The Geology and Engineering Properties of South African Calcretes

by F. Netterberg

Volume II

THE GEOLOGY AND ENGINEERING PROPERTIES OF SOUTH AFRICAN CALCRETES

(IN FOUR VOLUMES)

VOLUME II

AGE, PROSPECTING METHODS AND
ENGINEERING TEST METHODS

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F. NETTERBERG

VOLUME I: SUMMARY, CLASSIFICATION, DISTRIBUTION, COMPOSITION AND ORIGIN

VOLUME II: AGE, PROSPECTING METHODS AND ENGINEERING TEST METHODS

VOLUME III: ENGINEERING PROPERTIES AND SPECIFICATIONS FOR ROADS

VOLUME IV: APPENDICES AND MAPS

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CONTENTS PAGE

CHAPTER 5: AGE 313

5.1 LITERATURE SURVEY 314

5.1.1 Previous work on the ages of calcretes in Southern Africa 314

5.1.2 Ages of some calcretes outside Southern Africa 315

5.2 DEFINITIONS 317

5.3 METHODS OF AGE DETERMINATION APPLICABLE TO CALCRETES 319

5.3.1 Palaeontology 325

5.3.2 Artefacts 333

5.3.3 Stratigraphy, geomorphology and topography 333

5.3.4 Radiometric dating 341

5.3.5 The Radiocarbon dating of calcretes 353

5.3.6 Other methods 388

5.3.7 Methods of detecting calcretes forming at the present time 392

5.4 AGES OF CALCRETES IN SOUTHERN AFRICA 401

5.4.1 Pre-Pliocene calcretes 402

5.4.2 Pliocene calcretes 402

5.4.3 Calcified alluvial gravels and sands containing Acheulian artefacts 409

5.4.4 Calcretes associated with Middle Stone Age artefacts 412

5.4.5 Later stone age to present day calcretes 416

5.5 RATE OF CALCRETE FORMATION 420

5.5.1 Rates of calcrete formation from radiocarbon dating 420

PAGE

5.5.2 Evapotranspiration method for ages and rates of calcification 424

5.6 CORRELATION WITH AGES OF CALCRETES OUTSIDE SOUTHERN AFRICA 430

5.7 CONCLUSIONS 432

5.8 SCOPE FOR FURTHER INVESTIGATION 434

5.9 REFERENCES 441

CHAPTER 6: PROSPECTING METHODS 454

6.1 INTRODUCTION 455

6.2 LITERATURE SURVEY 455

6.3 FUNDAMENTAL FACTORS CONTROLLING THE DISTRIBUTION OF CALCRETE 455

6.3.1 Climate 456

6.3.2 Topography 457

6.3.3 Parent or host material 461

6.3.4 Biological factors 464

6.3.5 Time 464

6.4 REGIONAL PROSPECTING 468

6.4.1 Airphoto interpretation from conventional airphotos 469

6.4.2 Interpretation from diapositives 472

6.4.3 Colour photography 473

6.4.4 Infrared photography 473

6.4.5 Infrared imagery and other remote sensors 474

6.4.6 Geophysical methods 476

6.4.7 Geochemical methods 476

6.4.8 Plant indicators 477

PAGE

6.4.9 Zoological indicators 481

6.4.10 Drilling and excavation 481

6.5 DETAILED INVESTIGATION 484

6.5.1 Rapid probing 484

6.5.2 Overburden and calcrete colour, and overburden texture 485

6.5.3 Plant indicators 486

6.5.4 Hand augering and pitting 486

6.5.5 Geophysical methods 487

6.5.6 Bulldozing and ripping 491

6.6 CONCLUSIONS 492

6.7 SCOPE FOR FURTHER INVESTIGATION 493

6.8 REFERENCES 494

CHAPTER 7: ENGINEERING TEST METHODS 498

7.1 INTRODUCTION 499

7.2 PREPARATION OF THE SOIL FINES FOR DETERMINATION OF SOIL CONSTANTS AND MECHANICAL ANALYSIS 502

7.2.1 Comparison of methods 502

7.2.2 Effect of drying temperature 509

7.2.3 Suggestions for calcretes 512

7.3 LIQUID LIMIT 513

7.3.1 Comparison of methods 514

7.3.2 Accuracy and reliability of liquid limit determinations on calcretes 518

7.3.3 Suggestions for calcretes 523

7.4 PLASTIC LIMIT 525

7.4.1 Comparison of methods 526

PAGE

7.4.2 Application to calcretes 526

7.5 PLASTICITY INDEX 527

7.5.1 Factors influencing plasticity index determinations on calcretes 528

7.6 BAR LINEAR SHRINKAGE 530

7.6.1 Advantages of the bar linear shrinkage test 532

7.7 MECHANICAL ANALYSIS 535

7.7.1 Comparison of methods 537

7.7.2 Application to calcretes 539

7.8 AGGREGATE STRENGTH AND DURABILITY 547

7.8.1 Aggregate crushing and abrasion tests 548

7.8.2 Tests under wet conditions 552

7.8.3 A simple new pliers test 557

7.9 HARDNESS 561

7.9.1 Correlation between hardness and strength 562

7.9.2 A new pocket Mohs hardness tester 565

7.10 SELF-STABILIZATION 571

7.10.1 Prediction of self-stabilization 572

7.10.2 Detection of self-stabilization in road bases 584

7.11 SOLUBLE SALTS 586

7.12 MISCELLANEOUS METHODS 586

7.13 CONCLUSIONS 590

7.14 SCOPE FOR FURTHER INVESTIGATION 595

7.15 REFERENCES 597

CHAPTER 5

AGE

5.1 LITERATURE SURVEY

- 5.1.1 Previous work on the ages of calcretes in Southern Africa
- 5.1.2 Ages of some calcretes outside Southern Africa

5.2 DEFINITIONS

5.3 METHODS OF AGE DETERMINATION APPLICABLE TO CALCRETES

- 5.3.1 Palaeontology
- 5.3.2 Artefacts
- 5.3.3 Stratigraphy, geomorphology and topography.
- 5.3.4 Radiometric dating
- 5.3.5 The Radiocarbon dating of calcretes.
- 5.3.6 Other methods
- 5.3.7 Methods of detecting calcretes forming at the present time

5.4 AGES OF CALCRETES IN SOUTHERN AFRICA.

- 5.4.1 Pre-Pliocene calcretes
- 5.4.2 Pliocene calcretes
- 5.4.3 Calcified alluvial gravels and sands containing Acheulian artefacts.
- 5.4.4 Calcretes associated with Middle Stone Age artefacts.
- 5.4.5 Later stone age to present day calcretes

5.5 RATE OF CALCRETE FORMATION

5.6 CORRELATION WITH CALCRETES OUTSIDE SOUTHERN AFRICA

5.7/ ...

5.7 CONCLUSIONS

5.8 SCOPE FOR FURTHER INVESTIGATION

5.9 REFERENCES

5.1 LITERATURE SURVEY

5.1.1 Previous work on the ages of calcretes in Southern Africa

Previous work on the ages of Southern African calcretes has to a large extent been confined to a few sites of archaeological interest. Until recently in fact it was only at such sites that calcretes could be dated at all. The deposits of the lower Vaal river basin have perhaps received most attention. The first detailed work on the deposits was that of Söhnge, Visser and Van Riet Lowe in 1937, while Van Riet Lowe has described the situation as it was in 1952. His table on page 145 shows that calcification is considered to have taken place during Interpluvials after deposition of the Basal Older Gravels and prior to the red (Kalahari?) sand, between the South African Chelles-Acheul V and the Fauresmith I cultures and during oscillating wet and dry conditions in Middle Stone Age times. There has in the past been a tendency to assume dry conditions at times of calcification and to extend this inference to large tracts of country far removed from the site in question. The palaeoclimatic significance of calcretes and ferricretes has, however, since been questioned by Mason, Brink and Knight (1959) and Partridge and Brink (1967) have offered convincing alternative arguments other than climatic change for the varied textures of the deposits of the Vaal at Windsorton. There has also been a tendency by some to regard pedogenic materials as ordinary geological horizons from a datability point of view. It serves a purpose then, in the light of a better insight into their origin, to examine critically the evidence upon

which/ ...

which the ages of calcretes so far determined have been based, and to assess the extent to which these ages can be extended to Southern Africa in general in the form of distinct phases of calcification. This necessitates either dating as many calcretes as possible over the whole geographical region of interest, and moreover, every phase of calcification at each site, or the elimination of the possibility of variations in the other pedogenic factors at fewer selected sites. Only if nearly all the ages thus determined can be classified into distinct age groups can it then be said that the general area covered by the observations experienced distinct phases of calcification due to fluctuations of the aerial climate. The former method, that of dating a large number of calcretes, is probably the most reliable and in this way the contributions of the other pedogenic factors toward the production of calcretes under an aerial climate generally unfavourable to calcification can probably be ignored. It should, however, still preferably be supplemented by a critical assessment of the possible influence of past and present pedogenic factors at each site. However, this chapter only attempts to examine methods of dating applicable to calcretes and to classify these deposits in terms of age.

5.1.2 Ages of some calcretes outside Southern Africa

Price's (1933) description of the Reynosa Formation in South Texas is remarkably like that of our Kalahari Beds. His Upper Reynosa is chiefly made up of caliche rather like our Kalahari Limestone, while his Lower Reynosa could quite easily be taken for the rest of our Kalahari Beds (Passarge's (1904) Botletle Beds), while the Reynosa Plateau could easily be our Urinanib Plateau. Price decided on a probable age of Lower Pleistocene for the Reynosa caliche and a Pliocene age (on faunal evidence) for the Lower Reynosa, with some possibility of some of the

Formation/ ...

Formation being Cretaceous in age. Price, however, also fully realised that the caliche was probably of pedogenic origin and that it may still be forming in places. The Reynosa caliche proper was, however, considered to be Lowest Pleistocene.

Bryan and Albritton (1943), working in the Davis Mountains of West Texas recognised three stages of caliche formation, tentatively dated (on faunal and archaeological evidence) at 3,500 - 5,500 B.C., 1,200 - 1.400 A.D. and beginning about 1,880 A.D.

Bretz and Horberg (1949) concluded that the Ogallala caprock developed on the Middle and Early Pliocene Ogallala Formation of the Llano Estacado of south western New Mexico was probably Late Pliocene in age, but that caliche formation has continued intermittently on this and younger surfaces into Recent times. They also concluded that "the physiographic and geologic relations clearly indicate that the caliche varies in age from place to place and cannot be used as a stratigraphic horizon marker".

In Algeria, Durand (1963) concluded that certain powder calcretes (?) were probably formed during the Upper Villafranchian Period (= Calabrian II and associated with a pebble culture), certain floury nodules (nodules farineux) in red clays during the following Marine Regression (Acheulian I), hardpans chiefly during the Sicilian Marine Regression (Acheulian II), Tyrrhenian I (Acheulian III) and Tyrrhenian III (= Ouljian = Würm) Periods. Calcareous encrustations (encroûtements calcaires, possibly equivalent to the author's tufaceous calcrete) have probably been forming since Tyrrhenian II times up to the present.

Firman (1967), working in South Australia has arrived by various means at a Lower Pleistocene age for some nodular calcretes, a middle Middle Pleistocene age for the

Ripon calcrete, an upper Middle Pleistocene age for the calcrete in Bakara soils, an upper Upper Pleistocene age for the B horizons of soft nodular and platey calcrete in Loveday soils and a Recent age for some soft ropy and columnar calcretes.

It is apparent that overseas evidence is that calcretes are geologically very young. The oldest calcrete mentioned which has been recognised as such appears to be in the Lower Reynosa with a probable age of Pliocene. Calcretes in the same area may be of widely differing ages.

5.2 DEFINITIONS

By definition (Section 1.2), a calcrete must be composed of material of at least two ages, the authigenic carbonate and the allogenic original material cemented by the carbonate. There may also be several ages of calcification and calcretes in different stages of development in the same profile. Since calcification often occurs in alluvial sediments the allogenic material may also be composed of material of different ages.

The age of a calcrete is therefore defined as the age of the first onset of calcification.

The definition recognises that authigenic carbonate of more than one age may be present and that the calcrete may even still be forming at the present time. Where this information is known it should of course be added. The time of deposition of the allogenic material is also plainly of interest as well as the time of formation of any other authigenic components such as glauconite and other possibly datable minerals.

The age of each variety of calcrete described in Section 1.3.3 is defined as the age of the first onset of

calcification/ ...

calcification converting it from a less mature to a more mature state.

Thus the age of a nodular calcrete is the age of the centres of the largest nodules. The age of a honeycomb calcrete would be the age of the matrix joining the nodules together or, more strictly, the age of the matrix adjacent to the nodules, while the age of a hardpan would be the age of the matrix enclosing the nodules or, more strictly, the matrix furthest from the nodules. The age of a boulder calcrete cannot be satisfactorily defined, but the age of solution coatings on the lower surface of the boulder derived from the boulder itself may perhaps be a reasonable age. The age of calcareous and calcified soils can only be defined as the age of the carbonate matrix and no further subdivision between the two appears possible, except in the case of calcareous and calcified gravels, when the age of pebble coatings would define the age of the calcareous gravel and the age of the matrix furthest away from about a 2 mm skin around the pebbles would be the age of the calcified gravel. Dating the soluans in profiles like Plate 1.4 may yield useful information on the rate of weathering of basic igneous rocks.

It will be observed that an apparently paradoxical situation has now arisen. Due to the convention of quoting ages Before Present or Before Christ, the more a calcrete advances in its stage of development the "younger" it becomes. A boulder or hardpan calcrete is truly old in the sense that it has taken longer to reach that state of development than say the nodular calcrete below has taken to reach the nodular state. The boulder or hardpan is only "younger" than the nodular calcrete by convention in the sense that it attained its stage of development more recently than did the nodular calcrete.

5.3 METHODS OF AGE DETERMINATION APPLICABLE TO CALCRETES

The work which has so far been done on the ages of calcretes in Southern Africa has nearly all appeared in the Quaternary geological, palaeontological and archaeological literature. As a newcomer to these fields and without the benefit of any formal training or experience in any, the author experienced considerable confusion in trying to become acquainted with these fields. Terminologies have been altered or abandoned, different authors have used current terminologies in different ways (with or without redefinition), the long accepted climatostratigraphic subdivision of the Quaternary has recently been opened to serious doubt, and other time unit boundaries have been defined in different ways. No recent review seems to be available.

In sorting out the relevant Quaternary literature, Table 5.1 was prepared and is offered here as background information to this chapter on the ages of calcretes in Southern Africa. It was compiled after a study of Cooke (1946, 1952, 1957, 1963, 1967), Clark (1950, 1959, 1962), Leakey (1949), Leakey et al (1968) Pilbeam (1967), Mason (1962, 1967 and personal comm.), Dart (1964), Davies (1967), Butzer (1964), Flint (1959), Bishop (1967), Bond (1963), Tobias (1965), Van Zinderen Bakker (1967), Deacon (1966), Shotton (1967), and others, and further modified after discussion with Professor P.V. Tobias, Mr. Q.B. Hendey, Dr. R. Mason, Professor R. Inskeep, Professor G.W. Bond, Professor L. Wells, Dr. C.K. Brain and Miss E. Speed, all of whom kindly commented on some aspects of the draft table shown to them at the February 1969 Quaternary conference in Cape Town. The final overall synthesis is, however, to a large extent the opinion of the author, and it should not be assumed that the persons listed necessarily approve. The table attempts to correlate most features which may be useful in the dating of Quaternary deposits, as well as supplying background information essential to those not familiar

TABLE 5·1 TIME UNITS OF IMPORTANCE IN THE DATING OF QUATERNARY DEPOSITS IN SUB-EQUATORIAL AFRICA AND THEIR ISOTOPIC AGES.

GEOLOGICAL TIME UNITS			CLIMATO-STRATIGRAPHIC STAGE (2)	CLIMATE		ISOTOPIC AGE years	ARCHAEOLOGICAL TIME UNITS		PALAEONTOLOGICAL TIME UNITS		
ERA	PERIOD	EPOCH / AGE (STAGE) (1)		POSSIBLE RAINFALL	TEMPERATURE		MAIN TECHNOLOGICAL STAGES	CULTURAL UNITS	FAUNAL SPAN (STAGE)	HOMINIDS	SOME NON-HOMINID MAMMALS
CAINOZOIC	QUATERNARY	RECENT / RECENT	NAKURAN WET PHASE	WET	BECOMING COOLER	1,900 A.D. 0	IRON AGE	CHANNELLED AND STAMPED WARES	"RECENT"	NEGROID; BUSHMANOID, BUSHMEN AND OTHERS	
		RECENT / EPI-PLEISTOCENE	MAKALIAN WET PHASE	DRY WET	WARM	B.C. 9,000	LATER STONE AGE	WILTON, SMITHFIELD, NACHIKUFAN, TSHITOLIAN			
		UPPER PLEISTOCENE	MAIN GAMBLIAN PLUVIAL STAGE	DRY WET	COOL WARM	12,000 15,000	SECOND INTERMEDIATE; MIDDLE STONE AGE (LATER UPPER)	MAGOSIAN, LUPEMBO-TSHITOLIAN; LUPEMBAN	FLORISBAD-VLAKKRAAL	[First rock art] FISH HOEK MAN; TUIN PLAATS MAN; BOSKOP MAN	HOMO SAPIENS AFER
			THIRD INTERPLUVIAL	DRY	COOL WARM	>35,000? >40,000? >45,000?	FIRST INTERMEDIATE; MIDDLE STONE AGE (EARLIER LOWER)	SANGOAN, (FAURESMITH); PIETERSBURG; STILLBAY	POST OLDUVAI	[Stone spearheads Wooden handles] HOMO SAP. AFER; HOMO SAPIENS RHODESIENSIS (HOMO RHOD., SALDANHA MAN)	
		MIDDLE PLEISTOCENE	KANJERAN PLUVIAL; SECOND INTERPLUVIAL; KAMASIAN PLUVIAL	WET DRY WET?	COOL WARM	500,000?	EARLIER STONE AGE (PEBBLE CULTURES)	EARLIER MIDDLE LATER OLDOWAN (KAFUAN); ACHEULIAN (SOUTH AFRICAN CHELLES-ACHEUL I-V) (STELLENBOSCH)	VAAL-CORNELIA; SWARTKRANS; UPPER OLDUVAI	[Fire used or made] HOMO ERECTUS; HOMO SP. (TELANTHROPUS)	LYCYAENAS; CHALICOTHERIUM; SABRE-TOOTHS; (MASTODONS); (DINOPITHECUS); HIPPARION; GRIQUATHERIUM; GIANT PIGS; (ARCHIDISKODONS); (PALAEOLOXODONS); HOMIOCERAS; PELEROCERAS; EQUUS
		LOWER PLEISTOCENE	FIRST INTERPLUVIAL; KAGERAN PLUVIAL	DRY? WET?					STERKFONTEIN; LOWER OLDUVAI; OMO-KANAM	AUSTRALOPITHECINES; HOMO HABILIS	
	TERTIARY	PLIOCENE				2-3,000,000				RAMAPITHECUS	
		MIOCENE				12-13,000,000					
						25,000,000					

NOTES: (1) Obsolete terminology enclosed in round brackets (2) Use abandoned by most workers

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with Quarternary studies. Both current and obsolete terminology is therefore included. No up to date table of a similar nature was found in the literature. The main recent source of information not covered is the last Pan-African Congress, the proceedings of which have not as yet been published.

It is not intended to review in detail the state of Quarternary studies at the present time or even to discuss Table 5.1 in detail here. The following points of particular interest are, however, noted:

Apart from the insufficient number of radiometric dates, the uncertainty in the Pliocene/Pleistocene boundary is also due to the disagreement over its definition. Prior to 1948 it was defined as the beginning of the ice ages, widely thought to have begun about one million years ago. In 1948 the 18th Geological Congress defined it as the bottom of the Calabrian (=Villafranchian). It has also been defined on the basis of the appearance of the genera Archidiskodon, Bos, Equus and Camelus, on the basis of tectonics and on the basis of the first evidence of major climatic deterioration (See Butzer, 1964; Evernden et al, 1964; Flint, 1965; and Shotton, 1967). McDougal and Stipp (1968) have, however, concluded that the isotope dating evidence available strongly suggests that the two definitions currently in use (faunal-stratigraphic and climatic) are not equivalent. They considered that marked climatic cooling commenced 2.5 - 3.0 million years ago in mid-latitudes and possibly earlier than 4 million years in Antarctica, while the Calabrian "stratigraphically and faunally defined base of the Pleistocene in the type section in Italy seems to be reasonably well dated at about 2.0 million years." The climatic definition is thus of limited world-wide application. These appear to be basically European definitions and there does not appear to be any really suitable definition for Southern Africa. Flint (1965) has in fact stated that the position of the Pliocene-Pleistocene boundary is not known at all in South and East Africa. In East Africa it appears

to/ ...

to have been defined as the onset of the so-called Kageran Pluvial; however, the whole of the earlier part of the formerly accepted pluvial sequence is now open to serious doubt. It should be carefully noted then that the same deposit could in some cases be Pliocene or Lower Pleistocene depending on where the particular author concerned has placed the boundary. According to the older literature for example, the Kalahari Limestone (sensu stricto) would be Pliocene. Practically all the relevant work on the age of this horizon (if indeed it can be called such) is in fact in the older literature and it is suggested that if radiometric dates were available it might well be found that it would fall into the Lowermost Pleistocene according to Table 5.1. If the definition of the beginning of the Pleistocene is taken as the onset of climatic deterioration, the commencement of the formation of the Kalahari Limestone may possibly be a suitable definition for Southern Africa. If, as will be shown presently, the Limestone can probably be radiometrically dated, the definition would become a practical one. Due to the lack of radiometric dates at present, the old estimates of age are still used in this study, but it should be borne in mind that on an international scale the Kalahari Limestone and original Sands might well turn out to be Lowermost Pleistocene and Lower Pleistocene respectively.

Subdivisions of the Pleistocene have been made on both faunal and climatic arguments and disagreement is still present, (Butzer 1964). The East African pluvial/interpluvial climatostratigraphic stages have been widely applied in Southern Africa, but at the present time the whole sequence earlier than the second interpluvial appears open to serious doubt, and a more conventional geological subdivision on the basis of observation in place of interpretation is necessary (see Cooke 1957, 1963; Flint, 1959, and Bishop 1967). Van Zinderen Bakker (1967) has also stated that on the basis of pollen evidence, pluvials and non-

pluvials (a term preferred by some to interpluvials) were not everywhere synchronous. The climatostratigraphic stages in Table 5.1 must therefore be regarded as an undesirable basis for the subdivision of the Quaternary and the inferred relative rainfall prior to the second interpluvial as questionable. (See Bishop (1967) and also Partridge and Brink (1967) for a recent discussion of the validity of the South African type area for this sequence). Not so, however, are the temperature fluctuations, since these are based on pollen spectra (see Van Zinderen Bakker, 1967) and coincide very well with the later Quaternary fluctuations of Europe. In other words, the latest evidence is not that the pluvial/interpluvial sequence has been disproved, but that the interpretation of the evidence upon which it rests has been seriously questioned and that recent O^{18}/O^{16} and pollen data afford much more reliable evidence of temperature (as against rainfall) changes. It is of considerable interest to note that Emiliani's (1955) curve of tropical surface ocean temperature over the last 200,000 years based on O^{18} measurements agrees reasonably well with the Milankovitch (1930) effective solar radiation curve for lat. 65°N (see Fairbridge (1967) for a comparison). Two other O^{18} temperature curves by Van de Hamman et al (in Hendy and Wilson, 1968) and Hendy and Wilson (1968) covering the last 80,000 years in Holland and the last 40,000 years in New Zealand respectively also agree reasonably well with Milankovitch's curve. It would thus appear that the temperature changes which brought about the ice ages of the northern hemisphere were in fact qualitatively (at least) world wide. It is also of interest to note that all four curves show that the temperature has been decreasing for about the last 8-10,000 years. Inspection of Emiliani's and Milankovitch's curves show that cold troughs (corresponding to about a 5°C decrease in tropical surface ocean temperature from the present temperature of about 28°C) have occurred every 40-50,000 years. One can therefore state that the temperature in

Southern/ ...

Southern Africa (and the world) is probably decreasing at the present time and that, since the last cold trough occurred about 20,000 B.P., the next one is due between 20,000 and 30,000 A.D. Comparison shows that Milankovitch's and Emiliani's curves are in fair agreement with the temperature changes shown in Table 5.1 and that there is some tendency for the wet and warm phases to be associated. It should be noted that according to Cooke (1963), Brain's (1958) rainfall curve (modified by Robinson, 1961) deduced from cave deposits is the most realistic and objective rainfall interpretation in Southern Africa. Correlating on a faunal span basis, Brain's curve agrees reasonably well with the relevant parts of the climatostratigraphic sequence in Table 5.1. Bond's (1963) rainfall curve for Rhodesia is not quite the same as that indicated in Table 5.1 (the formerly accepted pluvial/interpluvial sequence), but it does not actually disagree (see also Van Zinderen Bakker (1967) above). Neither Bond's nor Brain's work rests on the dubious interpretation of river gravels and their results are therefore to be preferred. With respect to the oft-quoted opinion that the climate in Southern Africa is at present becoming drier, the Desert Encroachment Committee (1952) concluded that the hundred years or so of meteorological records available failed to show any significant long-term change in climate, though quite large and widespread changes in annual rainfall have occurred during this period.

The subdivision of the Pleistocene in sub-Saharan Africa into Lower, Middle and Upper ages appears to have been made on the basis of the supposed pluvial sequence, but not all authors have arrived at the same result in the case of the Lower/Middle and Middle/Upper Pleistocene boundaries. In all cases, and in general wherever there appeared to be a lack of formal definition, Table 5.1 was constructed so as to yield the best fit for all the data represented in it.

It should be noted that the term 'Neogene' has been used both for the Miocene plus Pliocene and for the Miocene,

Pliocene/ ...

Pliocene and Quaternary. It is not used in the table. The two uses of the term 'Recent' should be noted. Some authors use the term 'Epi-Pleistocene' for the period between the commencement of the Makalian wet phase and the commencement of the Nakuran wet phase. Others call the whole epoch beginning at the Makalian and ending at the present 'Recent' (or Holocene). 'Epi-Pleistocene' is used in the table for completeness.

In view of Partridge and Brink's (1967) paper placing the 200 ft gravels of the Vaal as probably Pliocene in age (see Section 5.4.2), with which view the author is in accordance, Cooke's (1963) table on page 181 forces the Sterkfontein faunal span to be placed right at the bottom of the Lower Pleistocene and even into the Pliocene. The length of the Swartkrans span is thus greatly lengthened. Professor P.V. Tobias (1969, personal comm.) is, however, of the opinion that the Sterkfontein material is not Pliocene in age.

The absolute ages less than 50,000 years were obtained by studying the C^{14} dates in Clark (1962) and Deacon (1966), rejecting any questionable ages and rounding off the figures to the nearest 1,000 years. The figures less than 40,000 years B.C. for the archaeological subdivisions are probably accurate to $\pm$ 1,000 years or even better in the case of the younger ages.* The 0.5 and 1.7 million year ages are K-A ages on Olduvai upper Bed 2 and Bed 1 respectively. Stone tools are probably the best dated 'fossils' in Southern Africa, but no K-A dates are available for Southern Africa.

The Burg Wartenstein symposium (Bishop and Clark, 1967) has recommended new terms ('industrial complex', 'industry' and others) in place of 'culture' and 'variant', but apparently 'culture' is to be retained, and this has therefore been used in the table. The symposium also recommended that the terms 'Earlier', 'Middle' and 'Later Stone Age' and

* MSA cultures may have been in existence before 51,000 BC. (Vogel: S.Afr. archaeol Bull, 24, 56).

'First' and 'Second Intermediate' be abandoned, without offering any substitutes. For want of the latter and in view of the C^{14} dates, these have been retained in the table. The terms 'Pebble Culture' and 'Chelles-Acheul' were also abolished. Superseded or abolished terminology is shown in round brackets in the table.

The non-hominid palaeontology was taken from Clark (1959) and their spans adjusted to fit the boundaries of the Pleistocene stages in Table 5.1. Cooke (1963 and 1967) should be consulted for more accurate information.

It should be noted that Table 5.1 is intended for background information and as an overall synthesis rather than as the most accurate correlation possible for individual objects and events; for the latter the particular authors in question should be referred to.

No attempt is made to correlate Table 5.1 with events elsewhere. A recent review of the dates of Pleistocene events in the northern hemisphere has been given by Shotton (1967).

5.3.1. Palaeontology

For the purposes of this study only organic remains such as bones, shells, teeth, etc. are considered as fossils. Artefacts are considered separately

While palaeontology is the classical way of determining the age of a deposit, the presence of a fossil in a calcrete only means that the calcrete is younger than the fossil. In the absence of any other evidence, the calcrete could in fact be anything from contemporary to very much younger than the fossil.

Calcretes containing useful fossils from a dating point of view are relatively rare. The only sites so far found where fossils have been of value are the Kalkbank,

Tuinplaats/ ...

Tuinplaats, Vaal river, Usakos, Elandsfontein, Melkbosch-strand and Swartklip occurrences. All these fossils have been mammals and the evidence provided by their remains will be presented in Section 5.4.

Mollusca

The mollusc and diatom remains common in tufaceous calcretes are mostly of extant types and are of relatively little use for dating purposes. Passarge's (1904) molluscs and diatoms for example only indicated an age of not older than Upper Tertiary. Rogers (1936) noted that there seemed to be two types of calcrete in the Kalahari, those with fossil water snails and those without, so that at least the fossils do provide some clue as to the origin of these deposits. The former type of calcrete is the author's tufaceous calcrete (equivalent to the Pfannenkalktuff, Schneckenkalk, diatomaceous pan limestone and vlei limestone of other authors). Rogers (1934) wrote that "the snail shells found in the limestones and sands of the Ngami-Linyanti-Botletle (Okavango) region include some striking forms, Melania, Lanistes, and Ampullaria, which do not seem to have been found in the Limpopo, Orange or Nossob valleys" and suggested that this may indicate that the Okavango once joined the Zambesi, and not the Limpopo or Orange as has been suggested. In 1936 Rogers stated that all the above snails plus Viviparus had been found in limestones of the Zambesi, Linyanti and Okavango, but had still not been found in the limestones of the Orange, Molopo and Okwa, nor in the limestones of the Ghanzi district. Rogers went on to say that a similar but less merked distinction between the mollusca of the Zambesi, Linyanti and Okavango and the Limpopo made a connection between the Okanvango and the Zambesi the more likely. Bond (1963) favoured a connection via the Kalahari between the Zambesi and the Limpopo. Rogers (1934) also mentioned that most of the snail fossils are found in the Molopo, Okwa, Botletle, etc. and some pans, and that none had been

found/ ...

found along the Weissrand.

Apart from Boettger in Passarge, determinations of molluscs in tufaceous calcretes have been given in Schultze (1907), Rogers (1907), Hermann (1910), Kent and Rogers (1947) and a few by Du Toit (1956). Succinea, Planorbis, Physa, Viviparus and Melania are perhaps the most common. Dr. H.H. Weinert (1968, personal comm.) found Succinea and Viviparus in calcretes near Soutpan in the Bloemfontein area. Tufaceous calcretes also occur in the plain of the Chengane river in Mocambique and cover the Alexandria limestones in the Cape (Du Toit, 1956). Mountain (1962) has recorded the land snail Achatina from aeolianite and tufa in the Port Alfred area and Haughton (1932) mentioned the occurrence of calcrete containing shells of the land snail Trigonepherus and mammalian and bird bones at Hoedjies Bay and in 1933 (in Amdurer, 1956) listed several molluscs from calcretes on the Cape Flats. Hendey (1967) has stated that "mammalian fossils embedded in calcrete occur within the present inter-tidal zone at various localities from Saldanha Bay to False Bay ...". A complete survey of the molluscs recorded from calcretes in Southern Africa has not been made, but would be a worthwhile project and would yield useful information on their environment of formation.

Of the profiles recorded by the author, the following profiles, mostly in tufaceous hardpans, contained mollusc shells: Profiles 17, 38, 58, 84, 89, 332, 336, 340, 345, 398, 399, 400, 401, 423, 426, 505, 513, 514, 518, 523, 524, 525, 526, 527 en 528. Several shells were kindly identified by Professor J.A. Van Eeden and are as follows (Table 5.2).

Microfossils

Insoluble residues of a number of calcretes were examined under binocular stero and polarising microscopes for mineralogy and microfossils. Specimens were prepared with dilute HCl. Only the -0.42 mm fraction (before acid

treatment/ ...

treatment) of the non-indurated calcretes was used. In the case of indurated calcretes a chip was merely broken off from one or more places. Oil, water or dilute HCl were used as mounting media.

TABLE 5.2 SOME FOSSIL GASTEROPODA IN CALCRETES AND OVERLYING SOIL

SAMPLE NUMBER	SAMPLE DESCRIPTION	GASTEROPODA IDENTIFICATION*
1554	In soil above calcrete	Achatina sp.
1594	In soil above calcrete	Achatina sp.
1720	Tufaceous hardpan	Succinea sp.
1723	Tufaceous hardpan	Dorcasia sp., Gulella sp.
1856	Tufaceous hardpan	Succinea sp.
1864	Tufaceous hardpan	Verocerastus sp.
1819	Tufaceous hardpan	Achatina sp.
1820	Boulder calcrete	Achatina sp.

*All of the snails listed are land snails, but succinea is very often associated with vegetation overhanging water.

Complete determinations were not made, as good survey of diatoms in tufaceous calcretes have been made by Reichelt (in Passarge, 1904 and Schultze, 1907) and Rogers (in Kent and Rogers, 1947). The main purpose was to merely determine the presence or absence of diatoms in view of their importance as palaeoenvironmental indicators and in lime stabilization (see Chapter 8). The results are shown in Table 5.3. A few semi-quantitative determinations on some nodular calcretes of particular interest are shown in Table 3.25.

Dr. K.A. Clauss kindly assisted with the identification of the Thecamoebians and the treatise edited by Moore

TABLE/ ...

TABLE 5.3 COMMON MICROFOSSILS PRESENT IN SOME CALCRETES

SAMPLE NUMBER	MICROFOSSILS IDENTIFIED				DIATOM GENERA IDENTIFIED
	DIATOMS	PHYTOLITHS	SPONGE SPICULES	OTHER	
1625	Nil	Rare	Nil	Nil	-
2325	Many	Probable	Many	Nil	Mostly *Epithemia*
2114	Nil	Nil	Nil	*Hoogenraadia*?, *Phryganella*, Unidentified	-
2321	Nil	Probable	Nil	Nil	-
2323	Nil	Nil	Nil	Nil	-
2224	Probable	Nil	Probable	Nil	-
1828	Many	Nil	Probable	Nil	-
2320	Possible fragments	Nil	Possible fragments	Nil	-
2324	Nil	Nil	Nil	Nil	-
2318A	One only	Possible	Nil	Nil	-
2318B	Nil	Nil	Nil	Nil	-
2322	Nil	Nil	Nil	Nil	-
2319A	Nil	Nil	Nil	Nil	-
2319B	Nil	Nil	Nil	Nil	-
1624	Nil	Nil	Nil	Nil	-
1601	Few	Few	Few	Nil	-
1659	Many	Many	Many	Nil	-
1602	Nil	Few	Few	Nil	-
1642	Few	Nil	Nil	Unidentified	*Epithemia argus*, *Mastogloia*, *Epithemia*

(1964)/ ...

(1964) was found invaluable here, as were the illustrations in Kent and Rogers (1947) in the identification of the diatioms, phytoliths and sponge spicules. The other treatises edited by Moore on the Mollusca and Protista (diatoms, etc.) were not consulted. Table 5.3 should not be regarded as showing all the classes or phyla of microfossils present in calcretes, but only the most common ones. As can be seen, calcretes vary widely in their microfossil content. The most common diatom noticed was probably Epithemia (Plate 5.1) perhaps followed by Mastogloia (Plate 5.2). Diatoms are common in tufaceous calcretes and uncommon in all others except certain calcretes (mostly nodular) in Ovamboland which have been found to stabilize exceptionally well with slaked lime for use in road bases. Sponge spicules are also more common in both these types. Phytoliths occur in small quantities in many calcretes. Two Thecamoebians, Phryganella sp. and a possible Hoogenraadia (probably Hoogenraadia Africana) (Plates 5.3 and 5.4) along with an unidentified specimen (Plate 5.5) were also identified in Sample 2114. Hoogenraadia probably indicates a Tertiary to Recent age and Phryganella a Pleistocene to Recent age (Moore, 1964).

In spite of the presence of diatoms (a class of the algae) in some calcretes, algal structures have not so far been confirmed in calcretes, although Passarge (1904) did make mention of "Algenrinde." Algal structures were suspected, however, in a silcrete on the lowermost terrace at Sambio on the Okavango river (Plate 5.6) and a specimen (Sample 1794) was later confirmed as such by Professor J.A. Van Eeden. The silcrete on this and to some extent on the other terraces as well seems to literally pass into calcrete both laterally and vertically at will. Lower terrace profiles are 362 and 365-368, but they do not convey the great variability of the section. A middle terrace profile is 36 and upper terrace profiles 358-361 and 364. Some of the soft nodular 'silcretes' at this site may be leached-out calcretes. Much of the similar material at Runtu next to and

below/ ...

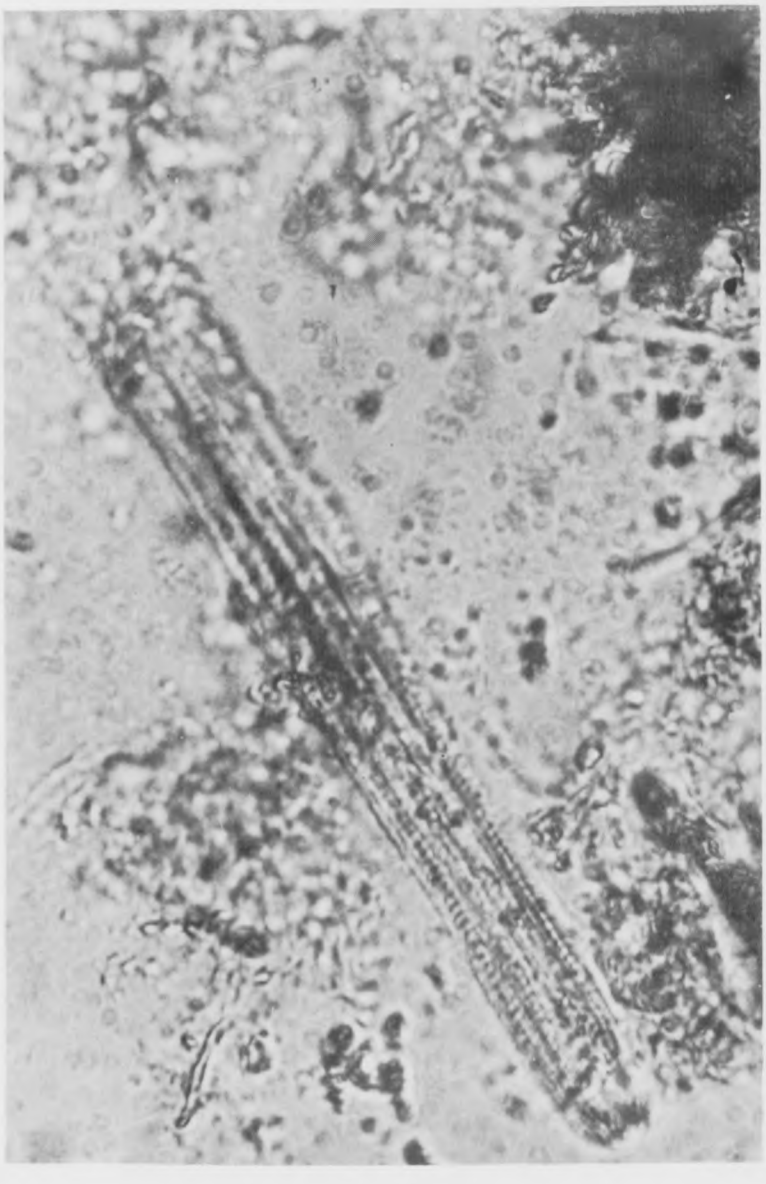


PLATE 5.1

Photomicrograph of Epithemia (X1000) from Sample 1642 (tufaceous hardpan) Ordinary light.

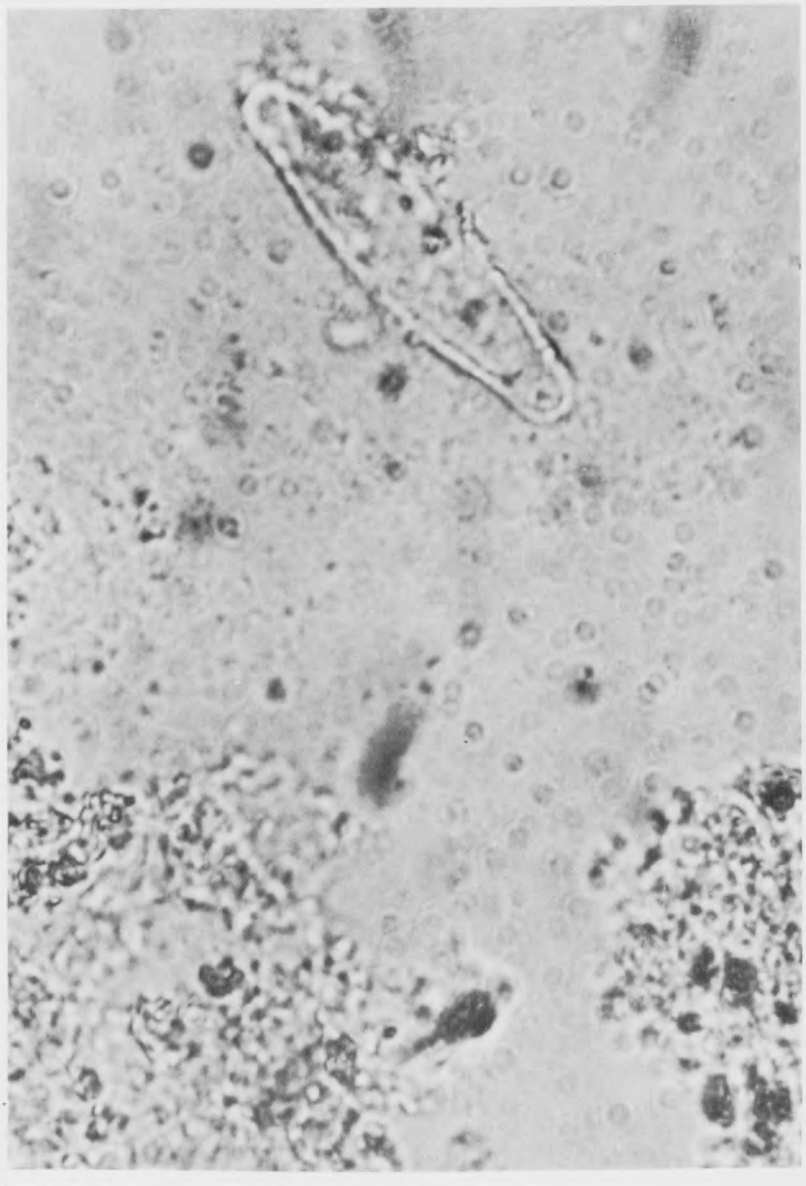


PLATE 5.2

Photomicrograph of a probable Mastogloia (X1000) from Sample 1642 (tufaceous hardpan). Ordinary light.

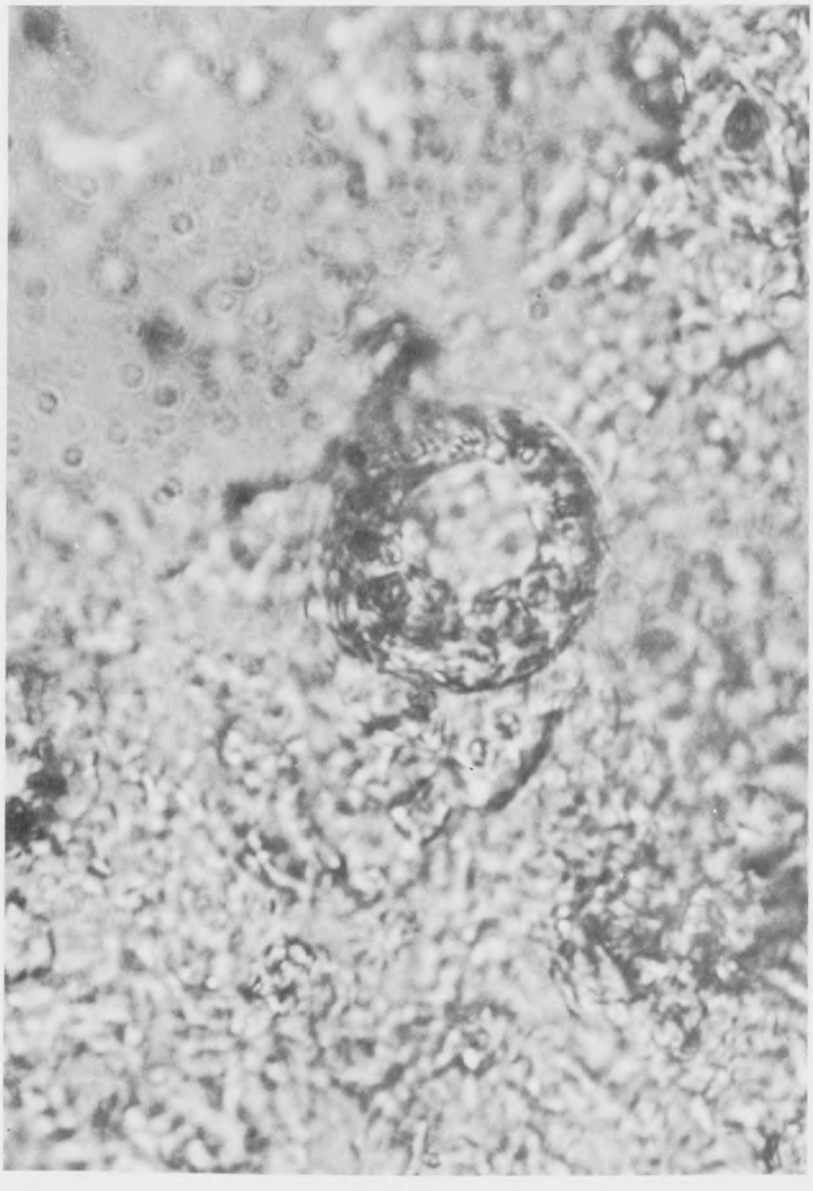


PLATE 5.3

Photomicrograph of a Phryganella (X1000) from Sample 2114 (nodular calcrete). Ordinary light.

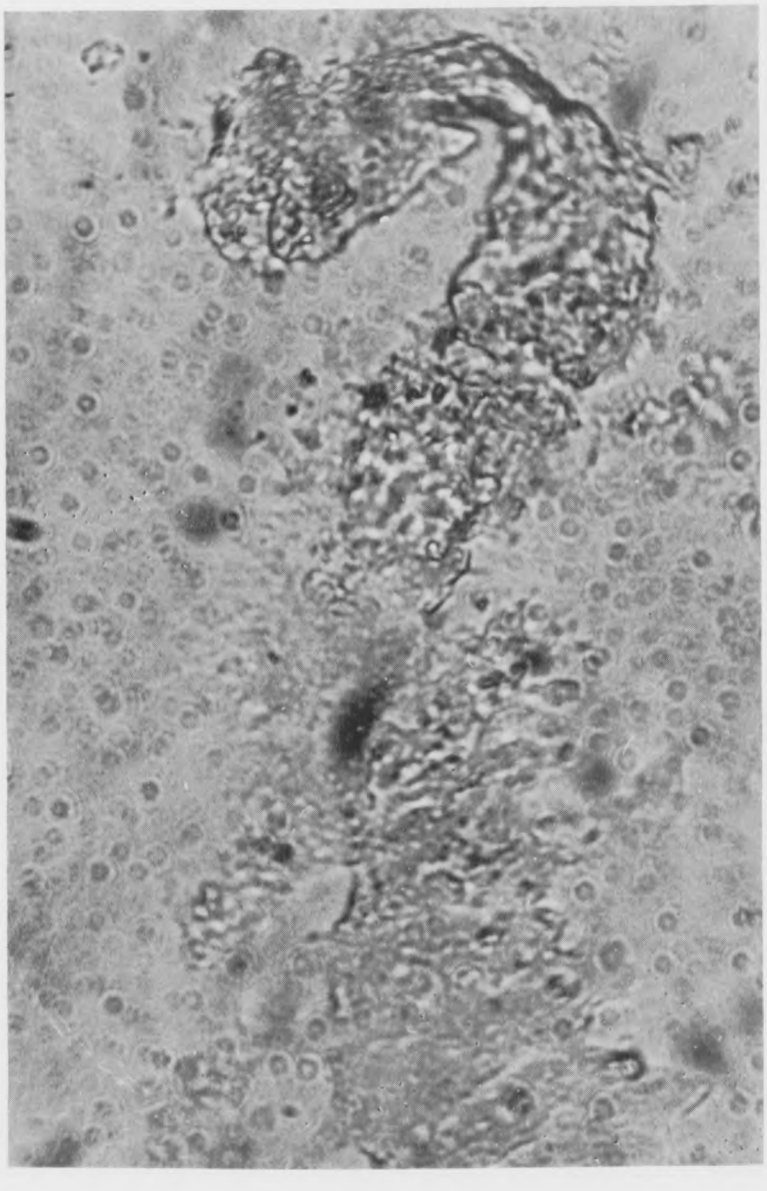


PLATE 5.4

Photomicrograph of a possible Hoogenraadia Africana (X1000) from Sample 2114 (nodular calcrete). Ordinary light.

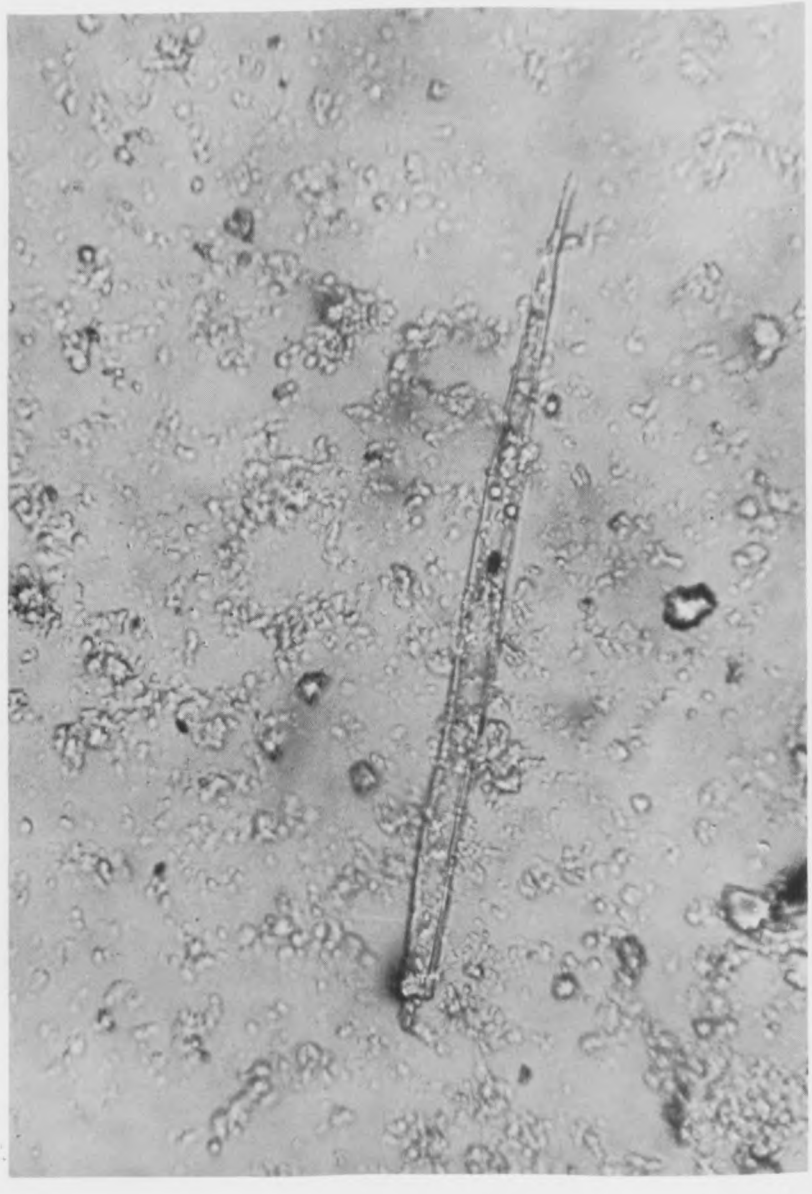


PLATE 5.5

Photomicrograph of an unidentified specimen (X200) from Sample 2114. Acicular tube or half-tube-like with pointed end and segmented wall in places. Not isotropic, probably of quartz. Maximum diameter 0.02 mm, length 0.5 mm. The specimen may perhaps be a phytolith which has recrystallised to quartz.



PLATE 5.6

Algal structures in silcrete at Sambio on the Okavango River.

below the Bantu Commissioner's house looks exactly like calcrete, but is non-calcareous. It may also be leached-out calcrete, but was logged as silcrete(?) in the field and its origin is still uncertain. Similar-looking material occurs in the 'bays' at the Elandsfontein fossil site near Hopefield. Several structures which look very much like algal stromatolites were noticed at the north end of the calcrete ridge at this site (Plate 5.7). They possessed a structure rather like a pile of saucers, concave sides upwards. Plate 5.7 bears a remarkable resemblance to Rajalu and Gowda's (1968) figure 12 of Oncolite, a subfamily of the Spongiostromata, thought to have lived in a shallow water intertidal marine environment. Oncolites possess a structure made up of "concentrically stacked spheroids", a description which fits the Elandsfontein examples very well. That the Elandsfontein specimens also formed in an intertidal environment is also not impossible, though their formation in a not too dissimilar interdune vlei or spring environment (possibly saline) close to the shore and at about sea level seems more likely (see Singer and Wymer (1968) for a recent description of the site). Before Rajalu and Gowda's paper was obtained, a specimen (Sample 2244) was shown to Dr. E. Plumstead, Dr. K.A. Clauss and Dr. S.H. Haughton, but though they thought an algal origin was likely, they were not able to state definitely that this was the case. Thin sections also yielded no positive evidence and X-ray diffraction analysis only showed the presence of calcite and quartz. The calcite was also low in Mg and was not the very high magnesian calcite which seems to be usually of algal origin.

After making a number of assumptions, Witty and Knox (1964) calculated that the grass opal phytoliths in the three grassland soils studied had taken 4,400 to 6,600 years to accumulate. Their method may be worthy of further investigation if data on the rate of production and destruction of grass opal in South African soils is available.

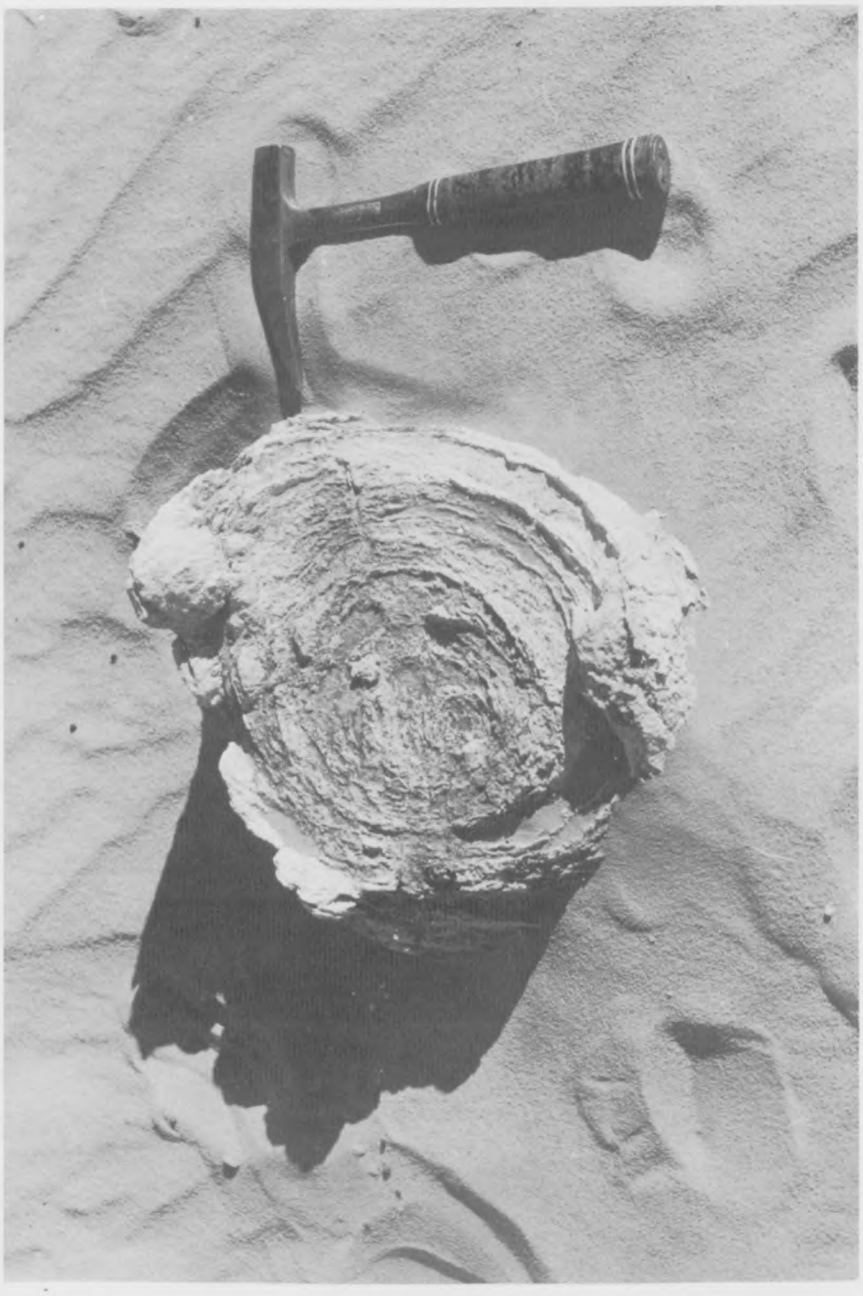


PLATE 5.7

Oncolite algal structure (?) in calcrete (?) at the Elandsfontein fossil site near Hopefield, Cape. Similar to Sample 2244.

No attempt has been made to find out if this information is available here, but Witty and Knox imply that it is not yet available for the United States.

Palynology

While it was not thought likely that pollen grains or spores would be preserved in the alkaline and usually dry environment of a calcrete, their potential value was considered to be sufficient to submit two silcretes, one quartzite (originally thought to be a silcrete), and eight calcretes to Dr. G.F. Hart for a preliminary analysis. His results are shown in Table 5.4.

TABLE 5.4 RESULTS OF PALYNOLOGIC ANALYSES ON SOME CALCRETES AND SILCRETES

SAMPLE NUMBER	ASSEMBLAGE NUMBER (DR. HART)	SAMPLE DESCRIPTION	ORGANIC MATERIAL IDENTIFIED
1599	4366	Nodular calcrete	Carbonized wood, grass
1828	4367	Tufaceous hardpan	Carbonized wood
1931	4368	Honeycomb calcrete	" "
1575	4369	Calcrete boulder or nodule	" "
1876	4370	Powder calcrete	" "
893	4371	Green dolocrete	" "
1627	4372	Boulder calcrete	" "
1833	4373	Red quartzite	" "
890	4376	White silcrete	Carbonized plant material
904	4377	Partly silicified calcrete	Barren
1690	4378	Brown silcrete	Carbonized plant material

Dr. Hart reported that no miospores were present in any of the samples and that "oxidation is presumed to have taken place during the formation of the sediments over a sufficiently long time period to cause almost total destruction of almost all organic material." No age determination was possible on any of the samples.

No pollen grains or spores appear to have been found in any other calcretes from Southern Africa and in fact the only recorded case appears to be that of Kosanke (in Swineford et al,1956) who reported the presence of "poorly preserved or corroded pollen grains" in some samples of the Pliocene pisolitic Ogallala caliche of the United States.

5.3.2 Artefacts

Whether rolled or unrolled, stone artefacts as a means of dating calcretes suffer from the same disadvantage as do fossils, namely that the presence of an artefact only indicates that the calcrete containing it is younger than the artefact. Care must be taken to ensure that the artefacts are not confined to the surface of the calcrete, having been cemented in by some later phase of calcification. This seems to be the cause in the case of the Basal Older Gravels of the Vaal and possibly also in the case of the Neuhof-Kowas archaeological occurrence in South West Africa.

Artefacts have the advantages of being rather more accurately dateable and less easily destroyed than fossils. Artefacts and fossils are most often associated with tufaceous hardpans.

5.3.3 Stratigraphy, geomorphology and topography

Stratigraphy

The position of a calcrete in the stratigraphic sequence often means little from the point of view of its

age. Once again, often only the original material can be dated by stratigraphic methods and calcification could have occurred at any time thereafter in one or more phases. In fact it is probably even possible that the carbonate could be completely leached out again without leaving any trace of its former presence. The boulder calcretes in Plates 1.19 and 5.12 seem to be in the process of being leached downwards.

The so-called Kalahari Limestone varies in age from place to place from Pliocene (or possibly Lowermost Pleistocene) to Upper Pleistocene and probably even younger, so that the common practice of using its upper surface to mark the top of the Kalahari Beds is misleading in some places. The deposits loosely known as the 'Kalahari Limestone' should not be regarded as a regional stratigraphic horizon. Passarge's (1904) subdivision of Botletle Beds and Kalahari Limestone may perhaps be better than the present practice of grouping both of them as the Kalahari Beds. For the purposes of this study the term 'Kalahari Limestone' will be taken to include only that calcrete of presumed Pliocene age plus any later calcification which has affected the Pliocene deposit in such a way that it cannot be readily separated from it stratigraphically (i.e. mainly crack fillings and coatings). Most of the calcrete shown as dark red (HA in legend) on Maps 2 and 4 is possibly Pliocene in age although this has by no means been proven, either in this study or, to the author's knowledge, in any other work. In many places what may be Pliocene calcrete is overlain by one or more calcretes which are plainly much younger in age.

Plate 5.12 (see Section 5.3.7) illustrates the danger of applying the Law of Superposition of Strata to all calcrete deposits. Even in the case of old river terrace deposits in which calcrete or calcified soil layers occur separated by non-calcareous material well below the influence

of rainfall infiltration and above the influence of the present dry water table (such as Profile 497 in the 100 ft gravels at Proksch Koppie at Windsorton) this law need not necessarily apply. It can, however, in such cases be taken that at least the calcretes must be fossil and not forming at the present time. A genuine unconformity would have to be established at the top of a calcrete layer, however, before it could be said that it was definitely older than the overlying material. The unconformity could be of the usual mechanical (erosional) type or of the chemical type such as is being produced by the boulder calcrete in Plates 1.19 and 5.12. The presence of successive distinct thin (less than 2 ft thick) hardpan layers overlying another variety of calcrete could probably also be taken, not necessarily as unconformities, but certainly as diastems and as indicating the validity of the Law of Superposition of Strata to this particular profile (as far as the hardpans are concerned). This is based on the assumption that pedogenic hardpan layers (probably all those less than 2 ft thick) only form close to a soil surface. Price (1933) considered all buried caliches to represent former emergence. It would be dangerous to conclude that this is invariably the case, however, since it has been shown that calcification can probably occur at practically any depth. The presence of hardpan could nevertheless be taken as indicating former emergence and at least a diastem. Boulder calcrete probably represents a true unconformity.

One of the few things that can be deduced from the stratigraphic position of a calcrete is that if it is outcropping it is surely fossil and not forming today. It is, however, quite possibly being leached out by rain to form a new calcrete below or a laminar horizon in the surface hollows. An important property of calcretes from the stratigraphic aspect is that they exhibit what could almost be called facies changes. Nodular calcretes may peter out sideways into scattered nodules or a calcareous soil or may

coalesce/ ...

coalesce into hardpan (Plate 5.8). Hardpans quite often degenerate sideways into boulders (Plate 5.9) and less often into honeycomb types. It is apparent from the sketches and descriptions of Brown (1965a, 1965b - both unpublished) that he has also noticed this property in the South West Kalahari.

Geomorphology and topography

Geomorphology affords a means of obtaining the maximum age of a calcrete by means of the land surface on which it is developed. It should be noted, however, that a land surface varies in age from place to place, decreasing in age from the coast inland. Land surfaces are normally named according to the time of their initiation at the coast.

By means of this technique Mabbutt (1955, 1957a), using the African erosion surface, correlated the Kalahari Limestone with a calcrete in Bushmanland containing an early Pliocene horse. Partridge and Brink (1967), by calculating the probable age of the Post-African erosion surface at Windsorton, arrived at a maximum age of some 4.8 million years for the Basal Older Gravels (200 ft gravels) resting on it.

River terraces afford reliable relative ages of much of the material lying on them. Neglecting colluvial effects, once again, however, they only determine the relative ages of the original allogenic materials with any certainty. Non-pedogenic calcretes formed by the river water can also be dated relatively by this means, but a pedogenic calcrete near the surface of any terrace can obviously have developed at any time subsequent to the formation of the terrace. The age of a terrace deposit only provides a maximum age for any calcrete in it. Older terraces may well exhibit several phases of pedogenic calcification while the youngest may possibly exhibit no calcification at all. Plainly,

young/ ...



PLATE 5.8

Nodular calcrete (?) grading from right to left via honeycomb calcrete into hardpan calcrete. A corner of the Hoba meteorite can be seen on the right hand side of the photograph. The meteorite is calcrete – encrusted and has apparently also crushed, compressed and distorted other limestone (calcrete?) underlying it (Luyten, 1929), so that C^{14} dating the calcretes would bracket the age for the fall of the meteorite Stony meterorites can be directly C^{14} dated, but the Hoba is a nickel-iron meteorite. (nickel-rich ataxite, containing only about 0.02% carbon: Spencer, 1932). Its date of fall is unknown, but it seems to have been discovered about 1920. See also Prior & Hey (1963). Iron meteorites can be dated using the C^{14}/Be^{10} ratio and the Cl^{36} method – see Section 5.8.



PLATE 5.9

Typical tufaceous hardpan degenerating from hardpan (Profile 399) on the left to boulders on the right (Profile 400). The hardpan in profile 401 (just behind the large bush in the left background) was C^{14} dated at about 12,000 years B.C. Terrace on west bank of the Runtu omuramba about 5 miles from Runtu on the old Grootfontein road via Karakuwisa.

young calcretes of the same age could occur on all the terraces. Furthermore, it is also apparent that calcretes of the same age need not necessarily occur in the same position in the profile relative to other deposits or calcretes on each terrace, nor need they be at the same stage of development. For example, a phase of pedogenic calcrete formation beginning today at Windsorton would encounter as parent materials boulder and hardpan calcretes, either outcropping or under a shallow red sand or gravel cover, on the Basal Older Gravels and calcified sand under a foot or so of loose red sand on the Younger Gravels II. The boulder and hardpans would thus tend to be leached down to form younger calcretes of any variety below, while a hardpan would form in the top foot or two of the calcified sands.

It is a general observation that older terraces are more heavily calcified than the younger ones and also tend to possess more of the better developed calcretes such as hardpans and boulders. This is well shown by some of the pans and omirimbi in Ovamboland and the Etosha Game Reserve. Figure 5.1 is a section across a shallow omuramba in the game reserve. A view across this feature from northwest to southeast is shown in Plate 6.1. The Highway Boring Machine is busy drilling Profile 32. Hardpan is only developed in the terrace on the southeast side, while honeycomb calcrete occurs on the terrace on the other side of the omuramba. On either side the calcrete degenerates to scattered nodules away from the omuramba and only nodular calcrete was found in the bed. Scattered ferricrete nodules were found in the lower parts of Profile 32 along with the calcrete nodules and scattered silcrete (or decalcified calcrete) nodules in Profile 28. In the field to the naked eye there appeared to be two terraces on either side of the omuramba, but this was not confirmed by the line of levels run across the omuramba with a telescopic alidade on which Figure 5.1 is based. The green calcareous sandstone is the uppermost well defined marker horizon occurring in the

area/ ...

FIGURE 5·1.

tion across shallow omuramba on trunk route 1/10 in Etosha
e Reserve 63 miles N.W. of border gate at farm Operet 312.

RG4-2987

area and was encountered in several other holes.

The Vaal river terraces at Windsorton also show a general increase in calcification with height of the terrace above the river, i.e. with increasing age. Profile 509 was taken close to the river in the lowest of Partridge and Brink's (1967) "Current Gravels". The whole profile was tested with acid and found to be non-calcareous. It could therefore be said that calcrete formation is not proceeding at the present time in this profile. The 40 ft gravels (also "Current Gravels"), also known as the Younger Gravels II, are represented by Profile 494 (Figure 1.3) at Riverview Estates which is quite heavily calcified. Profile 495 through the Younger Gravels II at Halliwell's Store is, however, much less heavily calcified and the degree of calcification here diminishes away from the river. Other profiles through the Current Gravels on the Windsorton side of the river (496 and 498) show only slight calcification. Profile 510 through the top six feet of the 70 ft gravels near the hotel, however, only seemed to possess about four feet of calcification. Towards the river (profile 511) this degenerated to very slight calcification only. The 100 ft gravels at Proksch Koppie (Profile 497) are more heavily calcified still and seem to have undergone several phases of calcification, but the 100 ft gravels at Profile 499 next to the road above the town are not calcified at all. Further away from the river next to the location, however, calcified residual weathered Ventersdorp andesite occurs at shallow depth (Profile 500). The 200 ft terrace gravels (Basal Older Gravels) at Suurkree (Profile 501) and at Ions' Claim (Profile 502, illustrated by Plate 1.3 and Figure 1.4, and Profiles 506-508) are generally very heavily calcified and also seem to have under gone several phases of calcification. Profiles 495, 497, 501 and the Ions' Claim area in general possessed bouldery tops to their profiles with makondo-type features being common. This

suggests/ ...

suggests that degeneration of the upper calcrete rather than active formation is proceeding at the present time in the upper part of these profiles. Active calcrete formation may, however, be proceeding lower down in the profiles.

While the Vaal profiles at Windsorton do show a general increase in calcification with age of the terrace, they also emphasise the variable degree of calcification found in deposits of supposedly the same age.

Mabbutt (1965, unpublished and personal comm.) has divided the area between Operet and Ondangua in South West Africa into the following "types of country" or physiographic settings (arranged in order of decreasing age):

(1) Flat or broadly and slightly undulating plain of leached white sands with localized depressions (oldest).

(2) Main and fossil drainage lines.

(3) Rims of large pans.

(4) Open clayey sand plain south of Ondangua.

(5) Oshana country around Ondangua.

(6) Fossil pan floors such as the Andoni Flats.

(7) Dune country of reddish sand of Kalahari type with streets and isolated depressions (youngest).

The types of calcrete Mabbutt noted occurring in these areas were as follows:

(1) "Loose clayey calcrete, green clayey sand with small calcrete nodules and opaline silica". (e.g. Profiles 4, 5, 171, 333 and 334 in depressions).

(2) Boulder calcrete, nodular calcrete and green sandy clay with scattered calcrete and siliceous nodules.

(e.g./ ...

(e.g. Profiles 246-257 from north to south along the road across the fossil drainage line in Plate 6.2).

(3) Silcrete, tufaceous hardpan, platey, bouldery and nodular calcrete. (e.g. Profiles 2, 313-320, 335-343).

(4) "Thin layer of concretions of small calcareous nodules locally making a continous slaggy layer". (e.g. Profiles 6, 170, 583 and 585 in depressions).

(5) Platey nodular calcrete and calcareous sand in depressions. (e.g. Profiles 7, 8, 11, 12, 131, 134 and 146 in depressions; Profiles 132, 133, 135, 137 and 161 on rises).

(6) Greenish calcareous clayey sand with calcrete nodules. (e.g. Profiles 310-312 on Andoni Flats).

(7) "Slaggy or nodular calcrete, locally as a hard layer" (e.g. Profiles 43 and 586 in depressions).

It can be seen that in general Mabbutt's calcrete types are corroborated by the observations and profiles of the author and do generally tend to become more well developed with age. An exception, however, is the Ondangua oshana area which later proved to contain well developed hardpan and nodular calcretes on rises. Radiocarbon ages of three samples (1598, 1603A and 1603B) are all equal to or older than about 14,000 B.C., which suggeststhat they formed prior to the last redistribution of Kalahari-type sand (2nd Intermediate?) which probably created the oshana country. Their appearance on the airphotos (Plate 6.3) does suggest a fossil occurrence, so that Mabbutt's order of ages is not necessarily invalidated. The only other exception is the flat plain (1) which should show well developed calcrete, but hardly does so. Either the old calcretes have been

leached/ ...

leached out or this area is much younger, perhaps the same age as (4). As a thorough investigation by the author around the depression known as Nel's borrow pit in which Profile 4 is situated failed to reveal any other better developed calcretes, the latter alternative may well be the case.

It would appear that the main effect of simple topography is to influence the stage of development of the calcrete though its influence on the soil texture and moisture regime. In some cases it can be seen to be a factor in the facies changes referred to earlier. Fossil calcretes may, however, influence the topography by resisting erosion and forming higher ground, for example the Weissrand east of Mariental and the long ridge in Plate 6.2. The latter calcrete both by its later stage of development and by its positive topography is almost certainly older than the last redistribution of the Sands of Kalahari type. A shallow water table could, however, have formed a non-pedogenic calcrete and caused the ridge-like feature after the redistribution. The depth to the water table on this ridge is unknown. A section constructed by drilling across two streets and a dune south of the Gaza line in the Okavango showed that the calcrete in the streets degenerated into a calcareous soil as it approached the dune and then disappeared entirely. It must therefore be younger than the dunes. There are at least two ages of calcrete in the streets, however. One variety is bouldery or hardpan and outcrops and the other is nodular and does not outcrop. The latter may be forming at the present time.

5.3.4 Radiometric dating

Only the radiocarbon method appears to have so far been applied to calcretes, although several methods seem to be applicable, or at least worth trying. A useful discussion of radiometric dating methods in general has been

given/ ...

given by Hamilton (1965), while Broecker (1965) and Shotton (1967) have reviewed most of the methods suitable for use on Pleistocene deposits and Aitken (1961) has discussed methods of interest to the archaeologist. Further useful reviews will be found in Faul (1954) and the I.A.E.A. symposium (1963).

All radiometric dating methods work on the following five principles:

(1) A parent atom A decays to a daughter atom B.

(2) The rate of disintegration from A to B (half life) is accurately known and is constant.

(3) The system remains closed after the event in question.

(4) The half life is not so long in relation to the age required that the amount of daughter B is too small to be measured.

(5) By determining A and B and knowing the half life the age can be calculated.

Errors can usually be traced to one or more of these factors:

(1) The system becomes open at some stage which may result in a loss (e.g. of A^{40}) or a gain (e.g. or C^{14}) of the isotopes being measured.

(2) Difficulty of measuring small quantities of the daughter B (or in some cases, e.g. C^{14}, the parent A). This determines the range of the method, the accuracy of which consequently tends to decrease at extreme ranges.

(3) Incorrect estimation of parent A (e.g. in C^{14} dating of carbonates).

Possible/ ...

Possible methods suitable for calcretes and terrestrial carbonates in general are the radiocarbon, ionium deficiency, uranium ^{234} decay, helium^{4}, potassium-argon, rubidium - strontium, lead ^{210}, protactinium - ionium, ionium excess, beryllium^{10}, strontium^{90}, aluminium^{26}, thermoluminescence and the argon^{40} - argon^{39} methods. Of these, the most promising for general use on calcretes are the radiocarbon, K-A, thermoluminescence, He^{4}, and ionium deficiency methods. The radiocarbon method is examined separately in more detail in Section 5.3.5.

The uranium^{234} decay method

The Th^{230}, U^{234}, He^{4} and Pa^{231} methods belong to the group known as the uranium disequilibrium methods. Th^{230}, U^{234} and He^{4} are daughter products of U^{238}, while Pa^{231} is a daughter of U^{235}. All these methods work on the principle that, as uranium is more readily soluble in natural waters than thorium or protactinium, the daughter products are enriched relative to the parents in the detrital phases of sediments, whereas the parents are enriched relative to the daughters in authigenic phases. If the original parent/daughter ratio is known the extent to which equilibrium has been re-established is a measure of the age of the system (Broecker, 1965).

The U^{234} method depends on the fact that U^{234} is more easily released during weathering than its parent U^{238}, resulting in an excess of U^{234} being incorporated into carbonate minerals deposited from natural waters. If the initial excess of U^{234} can be reliably estimated, the age of the carbonate can be determined. The potential range of the method is 50,000 - 1,000,000 years, but Broecker (1965) implied that the dates obtained with the method have so far been unreliable.

The thorium^{230} growth method

This method (also known as the ionium deficiency

method/ ...

method) has been applied to corals on the basis that they are formed with negligible Th^{230} (ionium), but with several ppm uranium. Thus, provided that the coral remained a closed system to isotopes of the uranium series, the extent to which Th^{230} has grown towards equilibrium with the uranium present is a measure of its age. Broecker (1965) quotes several ages obtained on coral and also gasteropods (plus one tufa) from Lakes Lahontan and Bonneville which are in quite good agreement with C^{14} ages. Broecker considers that the method is applicable to carbonates in general and (1966, private comm.) that it may well be applicable to calcretes. Its potential range is 0 to 200,000 years.

Prof. Broecker was contacted about dating some calcretes by this method and offered to try it on three calcretes. Samples 1962C from Kalkbank and 1940 and 1950 from Windsorton were accordingly submitted to him in March 1967. 1962C was taken from the bottom 3" to 4" portion of the lowermost hardpan layer (actually 3'9" - 3'10") at Kalkbank, (profile 514). The bottom 1" - 2" portion of the same bulk sample was C^{14} dated at 17,000 ± 400 years B.P. (Sample 1962B) so that the age of 1962C should be about 16,000 - 17,000 years. 1940 came from a depth of 4 ft in the calcified sand of Profile 494 (Plate 1.2, Figure 1.3) through the Younger Gravels II and should therefore be about 40,000 - 50,000 plus years B.C. in age (see Section 5.4.3). Sample 1950 came from a depth of 22 ft in the Basal Older Gravels at Profile 502 (Plate 1.3, Figure 1.4). 1950 is probably 2 to 5 million years old (see Section 5.4.2).

The $helium^4$ method

He^4 is a stable isotope and the method is limited in range only by the availability of suitably preserved material. It appears to be suitable for all carbonates and has given ages consistent with the Th^{230} method or with the Pleistocene time scale. It does not appear to have been

tried/ ...

tried on calcretes. Details of the method have been given given by Hurley (1954) and Damon and Green (1963).

The potassium-argon method

Potassium^{40} decays to argon^{40} by electron capture and β decay with a half life of 1.3 x 10^9 years. Ca^{40} is actually the major daughter product (89%), but this is of little use owing to the difficulty of estimating the small increment of radiogenic Ca^{40} in the presence of natural Ca^{40}. The range of the K-A method is limited only by the accuracy with which small amounts of radiogenic argon can be measured. This is equivalent to around 100,000 years at present, though Evernden and Curtis (1965, in Shotton, 1967) claim that their techniques allow assessments of the order of 2,500 years with an accuracy of 25% and that K-A dates on materials older than about 30,000 years are likely to be more accurate than radiocarbon dates. Shotton finds this difficult to accept. The assumption that no argon has been lost is usually valid if the mineral has not been heated above about 100°C, though apparently some diffusion does take place and some materials can contain an initial excess of radiogenic argon (which would cause the material to yield an age which is too high) - see Shotton (1967). Further details of the method have been given for example by Wasserburg (1954).

In dating sediments, whole rock dates are seldom used owing to the presence of detrital argon-containing minerals. Glauconite has been the most common authigenic mineral to have been dated so far, but it apparently does tend to yield slightly lower dates than expected (presumably due to some loss of radiogenic argon). It would appear that clays in general can also be dated and Lippolt and Gentner (1963) have had a measure of success in dating limestones and concluded that "K-A dating of clean limestones promises to be successful". The argon retention of calcite

is/ ...

is not known. In order to assess the possibilities of dating the older calcretes by K-A, four calcretes of interest were submitted to Mr. U. de Gasparis of the Bernard Price Institute for Geophysical Research who kindly determined K, Rb and Sr by X-ray fluoresence. The results are shown in Table 5.5

TABLE 5.5 APPROXIMATE POTASSIUM, RUBIDUM AND STRONTIUM CONTENTS OF FOUR CALCRETES

SAMPLE NUMBER	POTASSIUM ppm	RUBIDIUM ppm	STRONTIUM ppm
890	11,500	50	200
898	8,000	0	10,000
1940	3,500	9	300
1950	3,000	0	20-100

The ages of Samples 890 and 898 were unknown, but their stratigraphic position in Ovamboland suggested that they might be Upper Tertiary and they both contained small quantities of glauconite. 1940 came from the Younger Gravels II and 1950 from the 200 ft Basal Older Gravels at Windsorton. Neither were seen to contain glauconite, although 1940 was dolomitic. Commenting on the results, Mr. de Gasparis stated that the potassium contents of all four samples indicated that they should be dateable by K-A, provided of course that their ages were within the range of the method. Tables 3.1 - 3.6 show that whole rock samples of calcretes generally contain sufficient K to make them potentially datable by the K-A method. Some, if not most, of the potassium will of course be in the form of detrital minerals, particularly felspars and clays. The potassium content of the carbonate minerals and glauconites in these specimens has not been determined. The only known soluble

potassium/ ...

potassium contents of calcretes are given in Table 3.8, which were determined by extraction with cold concentrated HCl. The potassium contents are rather lower than any of the values in Table 5.5 and except for Sample 1881 may possibly be too low for dating. Any further work on the potassium content of the carbonate minerals in calcretes should employ exceedingly dilute acids to avoid attacking clay and other minerals. Alternatively, a mechanical separation method should be employed. A mechanical method will in any event be necessary in order to prevent argon loss if the sample is actually to be dated.

Recent developments in the K-A method now make it possible to date a mineral weighing only 5 milligrams after irradiation in a nuclear reactor. As some calcretes contain small amounts of apparently authigenic glauconite this method held great promise. Unfortunately, however, such small specimens can only be dated if they are at least 400 million years old (Dr. A.J. Miller, 1967; private comm.). At least 0.1 gm of separated potassium-bearing mineral is required in the case of specimens about 5 million years old.

In conclusion then, it can be said that glauconite-containing calcretes and calcareous sandstones (Samples 890, 898, 1638, 1639, 1693, 1725, 1728 and 2341) can probably be potassium-argon dated provided they are old enough, and in the case of non-glauconite-containing calcretes, cave breccias and travertines, it may be possible in some cases to date the calcite itself by this method. Clays might also give useful dates for the original material. Glauconitic calcretes probably include all of Passarge's (1904) green 'pan sandstones' (although Passarge was only able to identify the green mineral as "mica?"). Glauconite is responsible for the green colour of many of the deposits of the Kalahari Beds. The potential of this mineral in dating these deposits is obvious, but some work on its origin

under/ ...

under these conditions in relation to its stratigraphic position would be justified. For example, although glauconite occurs in abundance in the floor of the Etosha Pan, it could either be forming today in the (probably ideal) saline conditions of the pan or it could be derived from the underlying sediments. In any event, the Etosha Game Reserve and Ovamboland offer excellent opportunites for the study of glauconite formation or occurrence under terrestrial conditions.

Two recent papers on the use of glauconite in absolute dating are Polevaya et al (1961) and Hurley (1961). It would appear that while dates on this material are reasonably successful, they are usually 10-20% low.

The rubidium - strontium method

Rb^{87} decays to Sr^{87} with a half life of about 5 x 10^{10} years. The long half life means that only comparatively old material can be dated. The minimum age so far determined is about 10 million years, but the reliability of results in this region is apparently low. Rb does not form its own minerals,but replaces K.. As in K-A dating, minerals like biotite, orthoclase, lepidolite, muscovite, glauconite and potassium-bearing clays in general are used. Glauconite is the most common mineral used in dating sediments. Whole rock dates can only be used if detrital minerals containing inherited strontium can be corrected for. The dating of shales has been discussed by Peterman (1966) and he has concluded that if all the Sr was in authigenic minerals (unlikely) or if Sr-bearing detrital and authigenic minerals are thoroughly mixed, meaningful ages may be obtained. Further details of the method may be found for example in Ahrens (1954).

According to Mr. de Gasparis, the results in Table 5.5 indicate that Samples 898 and 1950 could not be dated by the Rb-Sr method, but that 890 and 1940 probably could

be dated if they were old enough. Dr. H.L. Allsopp has offered to try a Rb-Sr date on a calcrete if enough glauconite can be isolated, but unfortunately to date no calcrete of archaeological or stratigraphic significance has been found which contains glauconite. A possible exception may be the specimen from the Makarikari (Sample 2341) obtained from Professor G.W. Bond.

The Rb-Sr method should be readily applicable to the dating of the older glauconitic horizons of the Kalahari Beds.

Thermoluminescence

A rather ingenious method which unfortunately to date has not been noted for its reliability is thermoluminescence. It belongs to the group known as radiation damage methods, of which fission track dating is another example. All these methods work by attempting to measure the amount of damage done to a mineral lattice by the natural radiation to which it has been subjected. The age of the crystal can then be calculated after a further experiment to determine the damage done by known doses of similar radiation.

Briefly, the thermoluminescence method works as follows:

1. Natural radiation (apparently in practice mainly alpha particles) passing through a mineral is dissipated by the displacement of electrons (and sometimes even atoms). The electrons become held in semipermanent traps at higher energy levels.

2. If the mineral is heated the electrons returning to their sites emit their excess energy in the form of photons of light. The intensity of light emitted is a measure of the total radiation damage. A plot of intensity versus temperature is called a glow curve.

3./ ...

3. Natural glow curves of limestones yield peaks at 235 and 312°C. By comparing the intensity of the 312°C peak before and after irradiation with various known doses of alpha particles, the amount of damage affected by a known dose on the sample in question is assessed.

4. The age can then be calculated by measuring the natural alpha radiation of the sample.

Assumptions inherent in the method are that the relation between radiation dosage and damage has been constant during the history of the sample and that the irradiation rate is measurable and has also remained constant, or can be estimated.

The thermoluminescence of a sample of limestone depends on age, rate of irradiation, impurity content, thermal history and the pressure to which the sample has been subjected. Zeller (1954) concluded that thermoluminescence ages are only reliable when samples are selected with extreme care and that the method is restricted to fairly recent sediments or recent events. The age limitations of the method have not been stated in any of the works consulted, but this does seem to be a method that would be worth trying on calcretes.

Three samples of approximately known age (Samples 1962D, 1940 and 1950) were actually submitted to Mr. J. Hobbs at the University of Cape Town, but he later advised that he would after all be unable to attempt the determinations. This is a pity, since identical samples have been submitted to Professor Broecker for Th^{230} dating and the C^{14} age of 1962D is known. It may be worth contacting Dr. E.J. Zeller at the University of Kansas in this regard.

Further details of the method will be found in Zeller (1954), Holland (1954), Zeller et al (1957) and Zeller

and/ ...

Zeller and Ronca (1963). Ralph and Han (1966) have succeeded in dating pottery by this means. The samples of known age tested varied in age from 300 to 6,000 B.C. and they stated that, although the method was not fully proven, the precision could become comparable with C^{14} dating. (in Siegel) Siegel (1967) mentions that Angino and Grögler (1963) have prepared a bibliography on thermoluminescence totalling more than 600 entries.

Fission track dating

This method is similar in general principle to the thermoluminescence method. Fission of uranium within a mineral produces damage tracks which can be brought out by chemical etching. The tracks are counted andthe sample subjected to a known dose of neutrons. After recounting the tracks after the bombardment the age can then be calculated. The misidentification of tracks, their destruction by heat or weathering and the possible migration of uranium are some of the errors involved.

The method is still in its infancy and it would appear from Fleischer and Price (1964) that only mica, apatite, hornblende, zircon, uraninite, beryl and glasses, both natural and man-made, have so far been found to yield usable tracks. The one calcite sample examined yielded only shallow pits, though elsewhere these authors refer to Sippel and Glover as having counted fission tracks in calcite. No dolomite was examined. The only other known authigenic minerals in calcretes are barite and gypsum, but the one gypsum examined failed to reveal any tracks.

The overall age range of the method at 4 months to 300 million years plus (Fleischer and Price, 1964) is exceptional, though it does vary with the uranium content and in practice the available minerals thus severely limit its usefulness through their limited range of uranium content.

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In view of its exceptional age range it is most desirable that this method should be tried out on a number of calcretes and dolocretes. The method may already be applicable to phoscretes falling within the range 300-300,000 years.

Other radiometric methods

The uranium disequilium methods Pb^{210}, protactinium-ionium and ionium-thorium (ionium excess) methods appear to have possibilities in the dating of terrestial carbonates and should at least be investigated.

Be^{10}, with a half life of 2,500,000 years is produced by cosmic ray interaction in the atmosphere in a similar manner to C^{14}. This method has a range of 100,000 to 8,000,000 years. Lal et al (1960, in Hamilton, 1965) have shown the production rate of a number of potentially usable isotopes (H^3, Be^{10} and Si^{32}) to have been constant over the last few million years which, as in the C^{14} method, is a necessary requirement for their successful use.

Mr. C.J. Verwey (1965, personal comm.) has suggested that calcretes forming at the present time could be detected by their analysis for Sr^{90}, which only occurred in nature after the advent of atomic weapons. The strontium^{90} uptake of some clam shells has been investigated by Bonham (1965) and information should be available in the literature on the distribution of Sr^{90}.

Two further possibilities are the Al^{26} and Al^{26}/Be^{10} methods mentioned by Bowen (1966) and the new A^{40}/A^{39} method mentioned by Dr. A.J. Miller (1967, private comm.).

5.3.5 The radiocarbon dating of calcretes

The radiocarbon method of dating has been described for example by Libby (1955), Aitken (1961), Hamilton (1965) and Shotton (1967) and the ensuing brief discussion of the principles of the method has been compiled from their work.

Principles of the method

C^{14} is formed in the atmosphere at an altitude of some 5,000 ft by interaction between cosmic rays and nitrogen. The isotope C^{14} is unstable and with a half life of 5,730 years it decays by beta decay back to N^{14}. The C^{14} is then presumed to react with oxygen to form carbon dioxide (although the mechanism is apparently still obscure) and is thus incorporated into the natural carbon cycle. Within 4-10 years of its formation a C^{14} atom will normally be incorporated into some living matter deriving its carbon from the atmosphere. During the lifetime of the organism the C^{14} in it is normally in equilibrium with the atmospheric C^{14}, or in other words, after allowing for isotopic fractionation (about 3%) the ratio of the radioactive C^{14} to the stable C^{12} of the organism is the same as that of the atmosphere (about 10^{-12}). On its death, however, the exchange of all carbon ceases, the system theoretically becomes closed and the only change in the C^{14} content is through radioactive decay. At any time thereafter the time elapsed since the C^{14} uptake ceased and the present can be determined by measuring the present C^{14} content of a specimen of the object. In practice this is done by measuring the beta activity (the rate of disintegration of C^{14} in a fixed quantity of carbon) of the carbon in the specimen converted to carbon dioxide or a hydrocarbon gas and comparing it with a suitable modern standard which has itself been in equilibrium with the atmosphere. Standards in use are 19th century wood grown in about 1890 or the now practically

universal/ ...

universal equivalent standard of 95% of the activity of the NBS oxalic acid C^{14} standard. Such standards are chosen because of the Suess effect (dilution of atmospheric C^{14} by the burning of fossil fuels) and the atomic bomb effect (increase of atmospheric C^{14} from atomic explosions).

Two basic assumptions are inherent in the method:

1) Materials formed in the past had the same initial C^{14}/C^{12} ratio as materials forming at present (with allowance for the Suess and atomic bomb effects).

2) The material has remained a closed system to the introduction or loss of C^{14} other than loss by radioactive decay.

The first assumption is valid insofar as the C^{14}/C^{12} ratio of the atmosphere is thought to have been substantially constant back to the range of the method. It has been recently shown, however, (see Shotton, 1967) that variations have taken place within the last 6,000 years and that an error of up to perhaps 1,000 years due to this cause is possible. Radiocarbon dates of less than 6,000 years can be corrected for this effect. The first assumption is also valid in the case where the organism has been in equilibrium with the atmosphere. This is true in the case of most plants and animals and a few carbonates. Notable exceptions are plants which have derived part of their carbon from ancient limestone or coal and most carbonates (even bone and shell), which may yield ages which are too old by up to about 3,000 years. Whether the second assumption is valid depends on the nature of the substance being dated and the conditions it has experienced subsequent to it having become a closed system for the first time. Enrichment of the C^{14} in carbonates by exchange between the C^{12} in carbonates and the C^{14} of the atmosphere is a problem when carbonates are subjected to damp conditions. This

leads/ ...

leads to C^{14} ages which are too young by up to several thousand years.

While originally and primarily intended for organic substances, any carbon-containing substance can be dated by the C^{14} method provided it fulfills the above conditions or if corrections can be made for any violation thereof.

The usual range of the method of 70 - 40,000 years has recently been extended back to 70,000 years for organic materials only by pre-enrichment of C^{14} and the use of more sensitive counters. While carbonates up to 40,000 years old can be dated, Broecker (1965) has stated that C^{14} ages tend to be unreliable on carbonates more than 25,000 years old. Errors quoted with C^{14} ages are normally one standard deviation, i.e. a statistical counting error and not an absolute error. Ages are either quoted B.C. or B.P. (Before present, present being taken as A.D. 1950).

The errors inherent in the C^{14} method have been discussed for example by Shotton (1967), Hamilton (1965) and Aitken (1961) and it would appear that contamination by modern carbon is the most likely source of error. It is far more serious than for example contamination by ancient carbon. Contamination by modern carbon is a serious potential source of error for organic materials greater than 40,000 years old and for carbonates greater than 20,000 years old (Broecker, 1965). A useful-looking graph for assessing the effect of likely amounts of modern or "dead" carbon contamination is given in Olson (1963).

Application to calcretes

Provided a calcrete or a part thereof large enough to be separated and dated formed as a discrete event there is no theoretical reason why a meaningful C^{14} date should not be obtained. The possibility of the two basic assumptions of initial C^{14} equilibrium and no contamination

being/ ...

being violated in the case of calcretes are, however, very real. Calcretes are typically heterogeneous mixtures of carbonate, pre-existing soil and organic matter, all of different ages. In the older calcretes, crack fillings and coatings of probably different ages are the rule rather than the exception, while the mode of formation of many hardpans by a coalescence of nodules over a period of time ensures that this too contributes carbonate of varying age, as well as including both allogenic and authigenic carbon of organic and inorganic origin. Furthermore, roots of plants penetrate the calcrete and organic acids and CO_2 given off by them and decaying plant matter may also contribute to the problem.

At first sight then, the chances of obtaining meaningful dates on calcretes appear rather slim. Indeed, Bartlett (1951), in what are almost the only words which have so far been published on the general dateability of calcretes, stated that "Except under thoroughly understood conditions marl cannot be considered dateable ... neither can caliche", and left it at that. Most investigators seem to have heeded Bartlett's warning and at the present our knowledge of the state of initial C^{14} equilibrium and subsequent contamination in these materials has advanced very little further. It has been shown in Chapter 4, however, that the origin of calcrete is now sufficiently well understood to make dates on the various fractions meaningful. Different parts can be cut, sieved or dissolved out with acids and either the inorganic carbonate or the tiny fraction of dispersed organic matter dated. C^{14} laboratory techniques have also advanced and techniques for detecting and removing many contaminants are now available. Lastly, recent work in Europe and the United States on the C^{14} dating of lake tufas, cave travertines and ground waters has yielded valuable information on the initial C^{14} contents and the subsequent contamination undergone by these materials. Much of this information is relevant to the problem of

dating/ ...

dating calcretes. Another useful technique is that of mass spectrometer measurements of the ratio of the stable rare isotope C^{13} to that of the common isotope C^{12}, which enables estimates of the likely initial C^{14}/C^{12} ratio and subsequent contamination to be made.

The terminology in common use in the radiocarbon literature will now be defined. The initial C^{14}/C^{12} ratios of the different types of calcretes will then be estimated. Published C^{14} calcrete dates as well as those which have only appeared in the journal "Radiocarbon" will then be discussed and the first dates on South African calcretes presented.

Definitions and conventions

The literature on radiocarbon dating is most confusing to the uninitiated and, as no succint account of the principles, definitions and conventions in the right sort of detail was found, an attempt has been made to give this here.

The following definitions have been extracted from the C^{14} literature (mainly in "Radiocarbon") and are as followed in this work:

1. The activity, or C^{14} content of the sample (As) is normally expressed either as a percentage of the modern standard (now normally 95% NBS Aox) or as the difference in per mil from this standard (δC^{14}). Thus:

$$\delta C^{14} = \left(\frac{As - 0.95\ Aox}{0.95\ Aox}\right) \times 1000\ ^{o}/_{oo}$$

0.95 Aox is the measured activity of the standard corrected for isotopic fractionation in the laboratory when preparing the reference gas from NBS oxalic acid according to the formula given in the Editorial of "Radiocarbon", 3, (1961). By agreement, the standard gas has $\delta C^{13} = -19\ ^{o}/_{oo}$. δC^{14} is thus the measured

deviation/ ...

deviation of the activity of the sample from the corrected standard. It has however been used for both age-corrected and un-age-corrected deviations (see Editorial in "Radiocarbon," 1967: 9).

2. The content of the stable rare isotope C^{13} in the sample is normally expressed as a per mil difference from a standard, usually the average marine limestone or the Chicago PDB-1 Belemnite. The former consequently has a δC^{13} value of $0 \pm 3^{o}/oo$. The actual C^{13}/C^{12} ratio is $0.01123 \pm 3^{o}/oo$ (Nier, 1950; Craig, 1953; in Vogel, 1959). The formula for calculating δC^{13} takes a similar form to that used for calculating δC^{14}. The measurement requires the use of a mass spectrometer.

3. When the C^{13} content is known the sample can be corrected for isotopic fractionation during its formation in nature (known as normalising the same C^{13}/C^{12} ratio) by means of the formula

$$\Delta = \delta C^{14} - (2\delta C^{13} + 50)(1 + 0.001\delta C^{14})^{o}/oo.$$

Δ is then the per mil deviation of the C^{14} of the sample from the standard oxalic acid after correction of sample and standard "for real age, for the Suess effect and for normal isotopic fractionation" in nature and in the laboratory "and for deviations of C^{14} content of the age and pollution-corrected 19th century wood standard from that of 95% of the NBS oxalic standard" (Stuiver and Deevey, 1961). $\Delta = \delta C^{14}$ when $\delta C^{13} = -25^{o}/oo$. Δ should not be confused with the earlier (pre-1962) value of ΔC^{14}.

$$\Delta = \Delta C^{14} - \frac{\delta C^{14}}{20}$$

4. The age of the sample is then calculated from the standard radio active decay formula as follows:

$$\text{AGE} = -\left(\frac{t_{\frac{1}{2}}}{\ln 2}\right) \ln\left(\frac{1{,}000 + \Delta}{1{,}000 + \Delta_o}\right) \text{ years B.P. (Before 1950)}$$

Where $t_{\frac{1}{2}}$ is the half life of C^{14}, Δo is the normalised initial C^{14} content difference from the oxalic acid standard when the sample formed in per mil (i.e. the normalised value of $\delta_o C^{14}$) and Δ has already been defined. All dates published to date (late 1968) used the Libby half life of 5,568 years, but a change over to the latest value of 5,730 years appears imminent. Ages quoted according to the old value can be converted to the new value by multiplying them by 1.03. If the sample derived all its carbon from the atmosphere at the time of formation, then $\Delta o = 0$. This would be the case with most plants and animals, i.e. $\delta_o C^{14}$ would be equal to 0 (i.e. As = $0.95 A_{ox} = A_{1890}$ wood) and $\delta C^{13} = -25$. If the sample only contained a normalised 85% of the C^{14} content of the pre-bomb, pre-industrial revolution atmosphere at the time of formation, then $\Delta o = -150‰$. The value of Δo to be chosen depends on the origin of the sample itself and will be discussed in detail presently. For example though, Ostlund et al (1965) use $\Delta o = -50‰$ for samples deriving their carbon from sea water and $\Delta o = 0$ for samples deriving their carbon from the air (both apparently for samples which formed before A.D. 1960). Δo, however, varies also from place to place in the ocean and also with depth (see for ecample Broecker and Olson, 1961; and later work). ln x of course = $2.3026 \log_{10} x$.

5. It should be noted that the above would be the procedure only where mass spectrometer facilities are available to the C^{14} laboratory and used on their samples

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and standards. Many laboratories do not have such facilities or do not use them on routine samples owing to the cost. Most C^{14} ages published are therefore uncorrected for isotopic fractionation either in the laboratory or in nature. Many are probably corrected for fractionation in the laboratory at the present time, however, but very few for fractionation in nature. When no corrections at all are made, 0.95 Aox is simply 95% of the uncorrected measured value of the standard, δC^{13} is assumed to be -25°/oo (the mean value for plants), Δ then becomes equal to δC^{14} and Δo is assumed to be equal to 0 (i.e. the material was in C^{14} equilibrium with the atmosphere). Using $t_{\frac{1}{2}}$ = 5,568 years and simplifying, the age equation then becomes:

$$\text{AGE} = -18{,}520 \log \left(\frac{1{,}000 + \delta C^{14}}{1000} \right) \quad \text{years B.P.}$$

$$= -18{,}520 \log \left(\frac{\% \text{ modern } C^{14}}{100} \right) \quad \text{years B.P}$$

If the initial C^{14} content was less than 100% (1000°/oo), the actual figure is merely substituted for 100% or 1000°/oo in the denominator.

The initial C^{14} content of calcretes

The problem of the initial C^{14}/C^{12} ratio of calcretes (i.e. in choosing a value for $\delta_o C^{14}$ or Δo.) is caused by the fact they were almost certainly not in complete C^{14} equilibrium with the atmosphere at the time of formation. While no work appears to have been published on the C^{14} dating of calcretes, a fair amount of work has been carried out on the dating of cave travertines, loess, lake tufas and ground waters, both in Europe and in the United States. The work of the following authors has been drawn upon in compiling this discussion: Franke (1951), Deevey et al (1954), Münnich (1957), Broecker and Orr (1958), Franke et al (1959),

Brinkmann et al (1959), Munnich and Vogel (1959), Vogel (1959), Broecker and Olson (1959), Broecker and Walton (1959), Brinkmann et al (1960), Franke et al (1961), Vogel and Ehhalt (1963), Broecker and Kaufman (1965), Vidal et al (1966), and Degens (1967). The conclusions regarding calcretes are, however, those of this author's.

Suppose that water in C^{14} equilibrium with the atmosphere (taking A_{atm} = 100%, i.e. $\delta C^{14} = 0$, and $\delta C^{13} = -8^{o}/oo$) dissolves $CaCO_3$ from an ancient marine limestone containing only "dead" carbon (i.e. zero activity and $\delta C^{13} = 0$). The reaction can be represented by the equation:-

$$CaCO_3 + CO_2 + H_2O \rightleftharpoons Ca^{++} + 2\,HCO_3^-$$

When silicate rocks are weathered and dissolved the general reaction is (Broecker and Walton, 1959; Vogel and Ehhalt, 1963):

$$CaAl_2(SiO_4)_2 + 2\,CO_2 + H_2O \rightleftharpoons Ca^{++} + 2HCO_3^- + Al_2O_3 + SiO_2$$

The Al_2O_3 and SiO_2 would normally go to form clay minerals; this need not concern us here. For each mole of 'dead' $CaCO_3$ dissolved from the limestone one mole of CO_2 is required. The resultant solution will therefore have an activity of 50% of that of the atmosphere and a δC^{13} content of $-4^{o}/oo$. In the case of the silicate weathering, no dead carbon is added to the solution and it has a C^{14} and C^{13} content equal to that of the atmosphere. Humic acids and various salts can of course also contribute to the solution of $CaCO_3$, but the importance of the former is small (Vogel and Ehhalt, 1963). Salts such as NaCl without a common ion also increase the solubility of $CaCO_3$. Under saline conditions the activity of the carbon in solution could therefore drop below 50% and the δC^{13} content be

greater/ ...

greater (more positive) than $-4^{o}/oo$. If the weathering took place in the soil and the CO_2 of the soil air had a δC^{13} content of $-24^{o}/oo$ the bicarbonate solution derived from the limestone and silicate rock would have a C^{13} content of $-12^{o}/oo$ and $-24^{o}/oo$ respectively.

A small quantity of dissolved CO_2 is present in the system acting as a stabilizer (Vogel and Ehhalt 1963). Exchange then takes place between the carbon in solution and the carbon in the air around the solution according to the equation:

$$HCO_3^- \rightleftharpoons H_2CO_3 \rightleftharpoons CO_2\,(aq) \rightleftharpoons CO_2(gas)$$

Given long enough, complete equilibrium with the atmosphere would be reached, i.e. the activity of the bicarbonate in solution would be equal to that of the atmosphere ($\delta C^{14} = 0$). Degens (1967) states that fresh waters preferentially lose C^{12} and that in the final stage "the bicarbonate in fresh waters will be isotopically similar to the dissolved carbonates in the sea". In this case, $\delta C^{13} = 0$ and $\Delta o = -50^{o}/oo$. In the case where insufficient time is available for this before precipitation, the bicarbonate might attain a C^{14} content of anything between 50 and 100% modern. Where the air in contact with the solution contains more CO_2 than the atmosphere, this exchange is more rapid. Such conditions exist in the soil, as soil air contains up to 5% CO_2 (Brinkmann et al, 1960; Vogel and Ehhalt, 1963) as against the 0.03% by volume of atmospheric air. If the solution remains in contact with soil air having $\delta C^{13} = -24$ for long enough, δC^{13} of the bicarbonate can drop to around $-18^{o}/oo$.

Upon precipitation (apparently due to any mechanism) the $CaCO_3$ precipitated has the same C^{14} content (when normalised to a common C^{13}/C_{12} ratio - Broecker and Walton, 1959) as the bicarbonate in solution (Münnich and Vogel, 1959).

If/ ...

If the carbonate is precipitated in the soil, further exchange can then take place during the reprecipitation and resolution taking place as the moisture content and partial pressure of CO_2 fluctuates in the soil. (Münnich, 1957; Münnich and Vogel, 1959). This could also occur on any exposed surface subject to moisture changes.

This explanation for the increase in the initial C^{14} content above the 50% predicted by theory in the case of limestones is substantiated by measurements of C^{14} activity made by these workers on active cave formations, lake tufas and recent ground water bicarbonates. The actual initial C^{14} contents assumed by various workers in calculating the ages of these materials are shown in Table 5.6 ΔC^{14} figures have not been recalculated to Δ, since this requires a knowledge of δC^{14}.

The value of $\Delta o C^{14}$ of -47 used by Broecker and Orr (1958) for Lake Bonneville and Lahontan tufas was based on a measurement of one recently formed tufa in Pyramid Lake. In 1959, however, Broecker and Walton suggested that $\Delta o C^{14} = -120$ be used on Lake Bonneville materials and -50 for lake Lahontan materials. In 1965 Broecker and Kaufman were using "0.94 ± 0.06", for both lakes without making it clear whether this was a normalised figure or not (in this case it would either be 94% modern or $\Delta o = -60$ (not ΔC^{14}, although the difference between $\Delta o C^{14}$ and Δo would be negligible in this case). The figures for streams suggested by Broecker and Walton (1959) apply to any materials formed from the bicarbonate carried in streams. They subdivided the streams into those traversing mainly silicate rocks, those traversing mainly sedimentary rocks or limestones and those traversing an unknown sequence. The figures suggested are probably normalised and are shown as such, though they do not unfortunately make this clear. The figures given by Brinkmann, Munnich, Vogel en Ehhalt in their various papers are definitely not normalised. In this

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work all normalised figures are specifically referred to as such.

TABLE 5.6 INITIAL C^{14} CONTENTS ASSUMED BY VARIOUS WORKERS FOR TERRESTRIAL INORGANIC CARBONATES

AUTHOR	MATERIAL	C^{14} % MODERN	$\Delta_8 C^{14}$ ‰
Broecker & Orr (1958)	Lake tufa from Lakes Lahontan and Bonneville	99 ± 0.8	- 47 ± 8
Broecker & Walton (1959)	Lake tufa from Lake Bonneville	-	-120 ± 100
" "	Bicarbonate in Lake Lahontan	-	- 50 ± 50
" "	Streams on silicates	-	-100 ± 100
" "	Streams on limestones or sediments	-	-250 ± 150
" "	Streams on unknown rocks	-	-200 ± 200
Brinkmann et al (1959)	Groundwaters in Europe	85 ± 15	-
Munnich & Vogel (1959)	Cave waters & travertines; loess	85 ± 15	-
Brinkmann et al (1960)	One calcrete; groundwaters	85	-
Vogel & Ehhalt (1963)	Groundwaters in Egypt	$72.5^{+27.5}_{-22.5}$	-
" "	Groundwaters in Europe	85	-
Broecker & Kaufman (1965)	Tufa from Lakes Lahontan and Bonneville	94 ± 6?	Δ_o = -60 ± 60?
Broecker & Olson (1959)	Cave formations	70 ± 20	-
Geyh (1967)	Sinter and water	85	-

The European workers have constantly used a figure of 85 ± 15% modern based on ground water measurements. Some of their cave waters and travertines yielded, however, results up to 95% modern. Broecker and Olson's (1959) measurements on two recently formed cave materials yielded results of 71% and 85% modern respectively. From this and ground water measurements they suggested that most cave formations were formed with "70 ± 20% of the C^{14} concentration in modern wood". Two later measurements (Broecker et al, 1960) on apparently active cave travertines yielded C^{14} contents of 95.6 (calculated by this author) and 93.7% modern ($\Delta o C^{14}$ = -85 and -99°/oo.

Applying the foregoing results and arguments to calcretes, it is seen that their initial $C^{14}/_{C}12$ ratio will depend on:

1. The source of the carbonate (in many cases the solid geology).

2. Their mode of formation (pedogenic or non-pedogenic).

Pedogenic calcretes which have derived their carbonate from a weathered basic igneous rock such as the calcrete nodules in the black clays on the basalts and norites north of Pretoria should thus have been fully in C^{14} equilibrium with the atmosphere and should yield δC^{13} values of between about 8 and 18°/oo. No correction for initial C^{14} content should be necessary for these materials. Pedogenic calcretes which have formed in residual soil developed from sedimentary rocks such as the calcrete nodules in the residual expansive clay profiles of the Karroo System in the Orange Free State could have had initial C^{14} contents of anything from 50 to 100% modern. In view of the probably rapid rate of C^{14} exchange during pedogenic calcrete formation, however, a fairly high figure of about 85 - 90% is suggested. Such calcretes should yield C^{13} values of

between/ ...

between about 0 and 8°/oo. Thin calcrete crusts and soluans on and in outcropping dolomites such as those around Tsumeb would only be perhaps 70% modern, but those on and in weathered dolerites and basalts such as those common in the Karroo regions should be dateable without correction. Those on and in outcropping dolomite should yield δC^{13} values close to zero. Those on and in dolerites and basalts should yield values between about -4 and -10. Pedogenic tufaceous hardpans should be 95 - 100% modern and yield δC^{13} values close to zero or even slightly positive.

Depending largely on the origin of their carbonate then, pedogenic calcretes in general should be dateable with an accuracy varying from only the counting error to an error due to the difficulty of estimating the initial C^{14} content. The latter is not likely to exceed ± 20% for most calcretes, which corresponds to ± 2,000 years. This is plainly acceptable on the older calcretes and may well be comparable to the counting error. Errors of ± 15%, 10% and 5% in the estimation of the initial C^{14} contents are equivalent to ± 1,500, ± 800 and ± 400 years respectively and fortunately do not increase to any extent with the age of the sample.

Calcretes formed from ancient connate or magmatic water can plainly not be dated by the C^{14} method. The danger also exists that such waters may have picked up some young carbonate on their way to the surface and thus given rise to a spurious age. Most deposits of this type can, however, be readily recognised as spring tufas. From the study of the dates in the volumes of 'Radiocarbon' to be dealt with presently, it is apparent that dates on these materials are of no value.

Calcretes formed from ground water of long residence time will only yield the residence time of the water.

Residence/ ...

Residence times of 10,000 years are common (Vogel and Ehhalt, 1963). Care should thus be used in dating calcretes thought to be formed by capillary rise from shallow groundwater tables where the water is considered to have spent some time underground. Dating of both the dissolved bicarbonate in the water and the calcrete itself may yield a correction factor. Groundwater calcretes will probably yield C^{13} values of between about -5 and -10°/oo.

Calcified alluviums such as those of the Vaal should be readily dateable. Broecker and Walton's (1959) figure of 80 ± 20% modern should be applicable to the mixed catchment area of the Vaal. The C^{13} value of river water (and hence also of the calcified alluviums) probably varies from about -11 to -17°/oo at the source (depending on climate and geology in the source area) to about zero near the mouth, but to the author's knowledge no work has been done on this.

Dates obtained on the hardpan calcrete cappings of the calcified river terraces should, however, be interpreted with care, as though they are certainly pedogenic in origin, they will have derived half their carbon from the older carbonate of the terrace. In some cases the carbonate of the calcified alluvium may, however, not be much older than the calcrete. In these cases an equilibrium in excess of 90% modern can probably be expected. In others it might be only 80%, or even less in semiarid climates where evaporation is rapid and soil CO_2 low. Dating of the minimum ages of Later Stone Age tools embedded in the calcified Basal Older Gravels of the Vaal for example must therefore be approached with caution.

Use of C^{13} measurements

The European workers such as Brinkmann et al (1959) and Munnich and Vogel (1959) have used the C^{13} contents

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mainly as an indication of whether the sample in question is "normal" or not. If it is normal, its C^{13} content falls within the usual range of such materials as shown in Table 3.27 and the normal initial C^{14}/C^{12} ratio for that type of material can be assumed. A more reliable estimate of the initial C^{14} content is thus obtained. The United States workers (Broecker and co-workers) on the other hand have mainly used their C^{13} measurements to correct or "normalise" the actual C^{14} activity for isotopic fractionation as explained in the definition of Δ. It seems surprising though that they did not carry out more C^{13} measurements on their tufas. The only one that they seem to have measured was their recent C^{14} control, which gave a value of $\delta C^{13} = + 6.3^{o}/oo$. This was taken near a hot spring and was thus probably not representative of their other tufas, which would probably have δC^{13} values in Vogel's (1959, 1963) freshwater (presumably lacustrine) limestone range or quite likely even negative values in the groundwater limestone range as the dissociated HCO_3^- in the Pyramid lake yielded a value of $-5.9^{o}/oo$. Shell $CaCO_3$ from two rivers in California yielded values of -6.7 and $-4.7^{o}/oo$.

The best results on calcretes will plainly be achieved if both these approaches are used. Munnich and Vogel (1959) concluded that reasonably accurate ages could be obtained on freshwater limestones, especially when C^{13} measurements are available as controls and Broecker and co-workers have obtained good results on lake tufas.

Published C^{14} dates on calcretes

Notwithstanding the relatively favourable picture painted above, out of all the many thousands of C^{14} dates known, only 101 are known on calcretes (Appendix G), including the 10 dated in the course of this work. Of these, only 12 have appeared in the normal literature, all the rest having only appeared in

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the laboratory date lists in the journal "Radiocarbon". It is plain that their potential dateability and the possible errors involved have not been appreciated. This, coupled with the difficulty that would normally have been experienced in interpreting the dates obtained, appear to have been the reasons behind the few dates carried out and the fewer still published.

The only known dates published on calcretes are three by Brinkmann, Münnich and Vogel (1960), three by Buol and Yesilsoy (1964) and five by Ruhe (1965). With the addition of one date, Gile, Peterson and Grossman (1966) quote the same dates as Ruhe. Only Brinkman et al were trying to determine the dateability of calcretes, the other authors merely using the dates as part of other work on pedogenesis. Two other works which have not been obtained also apparently discuss some calcrete dates. These dates are LJ-601 and 602 in a Californian archaeological report and SI-270-276 in an unpublished thesis on the chronologies of playa lakes. Some details of these samples have, however, appeared in "Radiocarbon" and they will be dealt with in Appendix G.

Brinkman's first date (H 513-494, 9,360 years B.P.) was on a one centimetre thick crust of calcrete in basalt and weathered basalt soil from Tenerife. Unlike all the previous dates in Table G.1, this date is calculated on the assumption of an initial C^{14} content of 85% modern. The authors stated that subrecent calcretes are widespread in this area according to Kubiena (1956) and they regarded the date as fitting in with the general palaeoclimatic history of the area. The second sample (H 514-473, $>$ 25,000 years B.P.) came from a strongly calcified marl on the surface of the Tyrrhenian I terrace at Alicante, Spain (apparently equivalent in age to the Mindel-Riss Interglacial - about 300,000 B.P.). The authors regarded the age as not

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in conflict with the geological evidence. The third sample (H 511-487, about 30,000 years B.P.) came from a thin crust apparently coating the dish-shaped hollows in a calcrete conglomerate near Granada. The authors stated that the age disagrees with the pedological evidence for a young age put forward by Kubiena (private comm.). It is difficult to comment on this discrepancy, but from the description the material appeared to be the lining of a makondo or solution pothole in a calcified gravel and at ~30,000 years is, within experimental error, identical to W-788 (28,000 ± 1,500 years B.P.), from an exactly similar feature in the United States. This must surely be pure coincidence. The author regards these makondos as evidence of decalcification and the first onset of boulder calcrete formation. The calcrete is therefore fossil and not recent as suggested by Kubiena. The age of the makondo lining itself is, however, another matter and this could be contemporary or fossil.

The five dates of Ruhe are all on one profile and apart from one result all increase in age downwards from 5,725 years at 9 inches to 15,300 years at 18 inches. The discordant result of 18,300 years was at 12 inches. Organic carbon at 11 inches gave an age of 9,550 years while the enclosing inorganic carbonate yielded an age of 13,850 years. Beyond mentioning that the soil occurred on the Picacho surface in New Mexico of Late Pleistocene age, the validity of these dates was not discussed. Two other dates on a profile of 20,300 years for the Bbca horizon and >30,000 years for the Ccab horizon respectively were also mentioned. Ruhe did not state whether his dates were B.P. or B.C. and gave no laboratory date numbers. Buol and Yesilsoy's soil was a red desert soil in Arizona. They dated a weakly developed ca horizon between 38 and 44 inches at 2,300 years B.P. The top of another carbonate horizon lying between 60 and 96 inches was found to be 9,800 years old

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while the bottom was over 32,000 years old. Petrographic and other work led them to conclude that only the weak carbonate above a depth of 60 inches was pedogenically associated with the present soil surface. The C^{14} dates thus fit in with this view. No C^{14} laboratory sample numbers were given. With the addition of one date, Gile, Peterson and Grossman quote the same results as Ruhe. This date is also anomalous in that it does not fit into the sequence of increasing age with depth. These authors think that, as these two anomalous dates are whole soil dates, they are due to different ages and amounts of carbonate within certain horizons. This is indeed very likely to be the case. Regarding the ages obtained, the authors regard the absolute ages as "questionable" but the relative ages as valid. The calculated age of 1,300 years B.P. of the fine earth fraction of the uppermost carbonate horizon of the soil dated was considered reasonable with respect to the age of the land surface on which the soil is developed. A whole soil date on the 6-11 inch B22tca horizon (I-374) yielded an age of 5,725 years while the carbonate coating $>$ 2 mm pebbles yielded an age of 10,226 years. From the % gravel and fine earth they calculated the age of the fine earth carbonate to be 1,300 years. The soft upper laminae of their 11-12 inch K21m horizon yielded an age of 4,575 years, the hard lower laminae an age of 13,850 years and the adherent plugged horizon 18,300 years. A whole soil date on the 12-25 inch K22m horizon yielded a date of 15,300 years.

The nearest material to a calcrete radiocarbon dated in Africa appears to be the aeolianite described by Mountain (1966) and Deacon (1966). These authors dated a fossil footprint in aeolianite (dunerock) at East London by dating the comminuted shell fragments present after separating off as much of the secondary carbonate as possible. The date obtained of 29,090 years B.P. was in excellent agreement with the estimated age of about 30,000 years arrived

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at through the underlying Middle Stone Age implements. It is a pity that they did not also try to date the inorganic carbonate.

A preliminary conclusion that can be reached at this stage is that while perfectly reasonable dates have been obtained, either finite or beyond the range of the method, the authors had little check on the ages of the materials and little knowledge of the initial C^{14} contents. Only Brinkmann et al (1960) have gone so far as to use an estimated initial C^{14} content, which on the basis of C^{13} measurements seems reasonable enough. However, since their material was developed in weathered basalt, a figure higher than the 85% modern used was probable.

In order to check further just what work had been carried out on the radiocarbon dating of calcretes, all the known published calcrete dates plus those which appeared in "Radiocarbon" were collected together. The results and comments are shown in Appendix G. While only 101 calcrete dates are known, and virtually no work has been done on the dateability of calcretes, it is apparent from the dates themselves and the comments that at least finite and reasonable dates can be obtained. The samples too old to date also indicate that calcrete can survive without serious contamination by modern carbonate.

Radiocarbon ages of some South African calcretes

In order to assess the possibilities of C^{14} dating calcretes in Southern Africa, ten samples were chosen whose ages were approximately or relatively known. At the same time it was expected that information on the origin of calcretes would also be gained.

All samples were untouched by hand and carefully taken and prepared to ensure the minimum contamination. Nodular

calcrete/ ...

calcrete was sampled by first clearing the face of the profile back at least three inches with a geolocigal pick which had been cleaned of any adhering soil. In this way the pick itself was further progressively cleaned while using it for excavating. The sample was picked out directly into a container (a cooking pot was found useful for this) lined with several thicknesses of new aluminium foil with precautions being taken against material falling from above the level sampled. The sample was then carefully wrapped in a total of at least four layers of new foil, enclosed in a plastic bag and then placed in the usual canvas sample bag. Except when taking large samples about one cubic foot in size, hardpan samples were similarly treated. In the laboratory, however, the outer half to one inch of hardpan, all later infillings, voids, roots and termite channels were cut out with a carefully cleaned aircooled diamond saw. In all cases the parts which appeared to be the original nodules in the hardpan were cut out and used and the (usually lighter coloured) matrix avoided as far as possible. No material nearer a root, crack, void, or outer surface closer than half an inch to one inch was used. This necessitated cutting out several small pieces for each sample which were handled only with clean stainless steel tongs. All samples thus only contacted the atmosphere, the diamond saw, the tongs and the aluminium foil. The tongs and the saw were cleaned with hydrochloric acid, washed with distilled water and allowed to dry before being used on another sample. Only new foil was used. The only likely contamination of the samples was therefore that by microscopic roots (particularly in the nodular calcretes), insects, and, to a much lesser extent, by later carbonate infillings. In addition, Sample 1575 was not originally intended for dating and may have been contaminated with paper and by handling. It was too small to cut up any further. After a more detailed literature survey had been carried out it became apparent that organic carbon can be

readily/ ...

readily separated by the dating laboratory, so that only contamination by carbonate during handling is important. The precaution of using an aircooled, clean saw, clean tools, and avoidance of dust contamination in packing and cutting thus turned out to be worthwhile, while the avoidance of hand contact and careful wrapping in aluminium instead of polythene was unnecessary.

Ten samples were eventually submitted to Professor K. Kigoshi of Gakushuin University for radiocarbon dating of the inorganic carbonate fractions. Ages given are based on a half life of C^{14} of 5,568 years and he made no correction for isotopic fractionation in nature or any deviation of the initial C^{14}/C^{12} ratio from that of the NBS oxalic acid used as a standard, in other words he assumed that the samples had an initial C^{14} content of 100% modern. The location of the samples in question is shown in Table 5.8 and the dates are discussed below.

The dates as received from Professor Kigoshi are shown in the second column of Table 5.9. The same dates on the A.D./B.C. system are shown in the third column. The fourth column shows the estimated initial C^{14} contents and the fifth column the A.D./B.C. ages calculated according to these estimates, the errors shown being the calculated errors arising from the uncertainty in the initial C^{14} estimate. The corrected ages were obtained simply by subtracting the number of years equivalent to the probable deviation of initial C^{14} content from 100% (For example, in the case of sample 1575 the estimated initial C^{14} content is 85 $\pm$ 15%. The deviation is 15%, equivalent to 1,500 years, hence the corrected age is $>34,000 - 1,500 = >32,500 \pm 1,500$ years). The sixth column shows some C^{13} contents determined by Dr. W. Roether and the final column the normalised ages based on the estimated initial C^{14} contents shown. Both the measured activity and the estimated initial activity

were/ ...

TABLE 5.8 LOCATION OF RADIOCARBON SAMPLES

NIRR SAMPLE NUMBER	GAKUSHUIN CODE NO. GaK-	PROFILE NO.	DEPTH	GENERAL AREA	LAT. °S	LONG. °E	DATE COLLECTED
1575	972	146	~28'	Ondangua	17°56'	15°59'	15.7.65
1828A	974	401	1'1"-1'2"	Runtu	17°55'	19°46'	30.8.65
1828C	975	401	1'8"-1'9"	Runtu	17°55'	19°46'	30.8.65
1907	976	-	0.5"-1.0"	Farm Neuhof - Kowas 233	23°00'	18°00'	27.9.65
1961	979	513-514	1'1"-1'2"	Farm de Loskop 127	23°32'	29°15'	16.2.66
1962B	980	514	4'1"-4'2"	Farm de Loskop 127	23°32'	29°15'	16.2.66
1658A	982	195	~6"	Farm Vergenoeg 942	18°48'	17°06'	1.8.65
1598A	973	161	3'6"-4'0"	Ondangua	17°56'	15°59'	15.7.65
1603A	981	161	7'6"-8'0"	Ondangua	17°56'	15°59'	18.7.65
1603B	981b	161	7'6"-8'0"	Ondangua	17°56'	15°59'	18.7.65

TABLE 5.9 AGES ACCORDING TO DIFFERENT ASSUMPTIONS

SAMPLE NUMBER	APPARENT AGE AS RECEIVED		PROBABLE INITIAL C^{14} (% MOD.)	CORRECTED AGE (years A.D./B.C.)	δC^{13} ‰	NORMALISED CORRECTED AGE (years A.D./B.C.)
	B.P. (years)	B.C. (years)				
1575	>36,000	>34,000	85 ± 15	>32,500 - 1,500	-	-
1828A	14,500 ± 1,700	12,550	95 ± 5	12,150 ± 400	-	-
1828C	13,340 ± 290	11,390	95 ± 5	10,990 ± 400	-	-
1907	19,800 ± 450	17,850	85 ± 15	16,350 ± 1,500	-3.1	16,550 B.C.
1961	2,270 ± 100	320	95 ± 5	80 A.D. ± 400	-1.3	150 A.D.
1962B	17,000 ± 400	15,050	95 ± 5	14,650 ± 400	-	-
1658A	34,700 $^{+\ 4,300}_{-\ 2,800}$	32,750	85 ± 15	31,250 ± 1,500	-	-
1598A	16,700 ± 400	14,750	90 ± 10	13,950 ± 800	-2.3	13,950 B.C.
1603A	18,200 ± 450	16,250	90 ± 10	15,450 ± 800	-	-
1603B	25,200 ± 1,000	23,250	90 ± 10	22,450 ± 800	-1.0	22,500 B.C.

were normalised (i.e. Δ and Δo used) according to the C^{13} measurements. This refinement is probably not justified in view of the uncertainty involved in the choice of the initial activity. The normalised corrected age is theoretically the most likely age. The probable error ascribed to it is roughly the same as that in the case of the corrected age.

The errors in column two are altered slightly by using different half lives and $C^{14}/_{C}12$ ratio, but are not significant in comparison with the effect of the uncertainty involved in the choice of an initial $C^{14}/_{C}12$ ratio. The activity of Sample 1575 was less than twice the standard deviation above background and the minimum age given corresponds to three times the standard deviation.

Samples 1575 and 1828 were also examined for fossil pollens by Dr. G.F. Hart, with negative results (Table 5.4).

It can be seen that the most probable age, whether normalised or not, is somewhat younger than the apparent age.

In the discussion to follow, the error ascribed to each date is deliberately omitted from the date itself, since it arises from two independent sources. The statistical counting error is shown in column two of Table 5.9 and the probable error due to the uncertainty in the initial C^{14} content in column 5. The dates shown are the corrected or normalised corrected ages.

Sample 1575 >32,500 B.C.

Calcrete nodule or boulder from a well in an oshana bed on the east side of ondangua Airport Borrow Pit No.9 (the latter is shown in Plate 6.3). At the time, it was thought that calcrete was possibly forming in the oshana

beds/ ...

beds at this depth at the present time. There are two possible explanations for the age obtained. Either the calcite is in fact older than 32,500 years B.C. or its carbon has never been in equilibrium with the carbon in the atmosphere. The latter is unlikely, since the water table is shallow and is probably renewed every wet season by the annual flood ('efundja'), a feature of the oshana country. Even if the calcrete derived half its carbon from some ancient 'dead' source, its true age could not be less than 30,000 B.C. This apparently anomalous age caused this calcrete to be reappraised, and it is now considered to be a small calcrete boulder, a weathering relict of some old fossil calcrete which has been largely weathered away. The radiocarbon age is therefore accepted.

Samples 1828 A and C (A) 12,150 B.C.
(C) 10,990 B.C.

Taken from the one to two inch portion of the top and bottom of the tufaceous hardpan layer (Plate 1.13, Figure 1.10) in a pit a few yards from the borrow pit illustrated in Plate 5.9. The hardpan overlay silcrete flakes (Sample 1827) said by Miss. H.R. MacCalman (1966, private comm.) to be provisionally late Middle Stone Age/Second Intermediate in age. While no previous C^{14} dates are available for northern S.W.A. or southern Angola, Table 5.1 indicates a probable age for the flakes of some 12,000 - 15,000 years B.C. As the calcrete must obviously be younger than the flakes, its C^{14} ages are thus very reasonable. The occurrence was probably a factory site utilising the underlying silcrete as raw material.

These two C^{14} dates are the only calcrete dates known to the author where the upper sample yielded a greater age than the lower. Apart from a possible switching of the two samples, it is also apparent that the statistical counting

error/ ...

error on 1828A is sufficiently large to overlap the date obtained on 1828C on a one standard deviation basis. Another possible reason is that the lower part of the hardpan has become contaminated with younger carbon (50% from the upper part of the calcrete and 50% from the soil air, activity therefore 61% modern in this case) due to the weathering by downward leaching (boulder formation) which this profile is at present undergoing.

If a constant rate of growth can be assumed and the distance apart of the two samples taken to be exactly 7 inches, the above hardpan layer grew at the rate of one inch per 166 years.

In the case of these two samples, darker, supposedly relict nodules were cut out and these submitted for dating. However, in practice the older 'nodules' were never cut out entirely free of contaminating (presumably younger) matrix and the true C^{14} age of the 'nodules' could be even greater. A more sophisticated technique such as removal of the matrix with acid should be used in future.

Sample 1907 16,550 B.C.

This sample came from an outcropping, well cemented, but originally tufaceous hardpan layer next to a stream on the farm Neuhof - Kowas near Windhoek. The site was not examined in detail and no profile through the calcrete was available. More than one layer of hardpan was, however, present. Middle Stone Age artefacts embedded in the surface of the hardpan were quite common. A particularly good example was chosen and a specimen of calcrete with embedded artefact removed. The specimen is shown in Plate 5.10 and is an elliptical bifaced implement measuring 8 cms by 4½ cms. The actual calcrete dated was taken from the same level as and immediately adjacent to the artefact

which/ ...

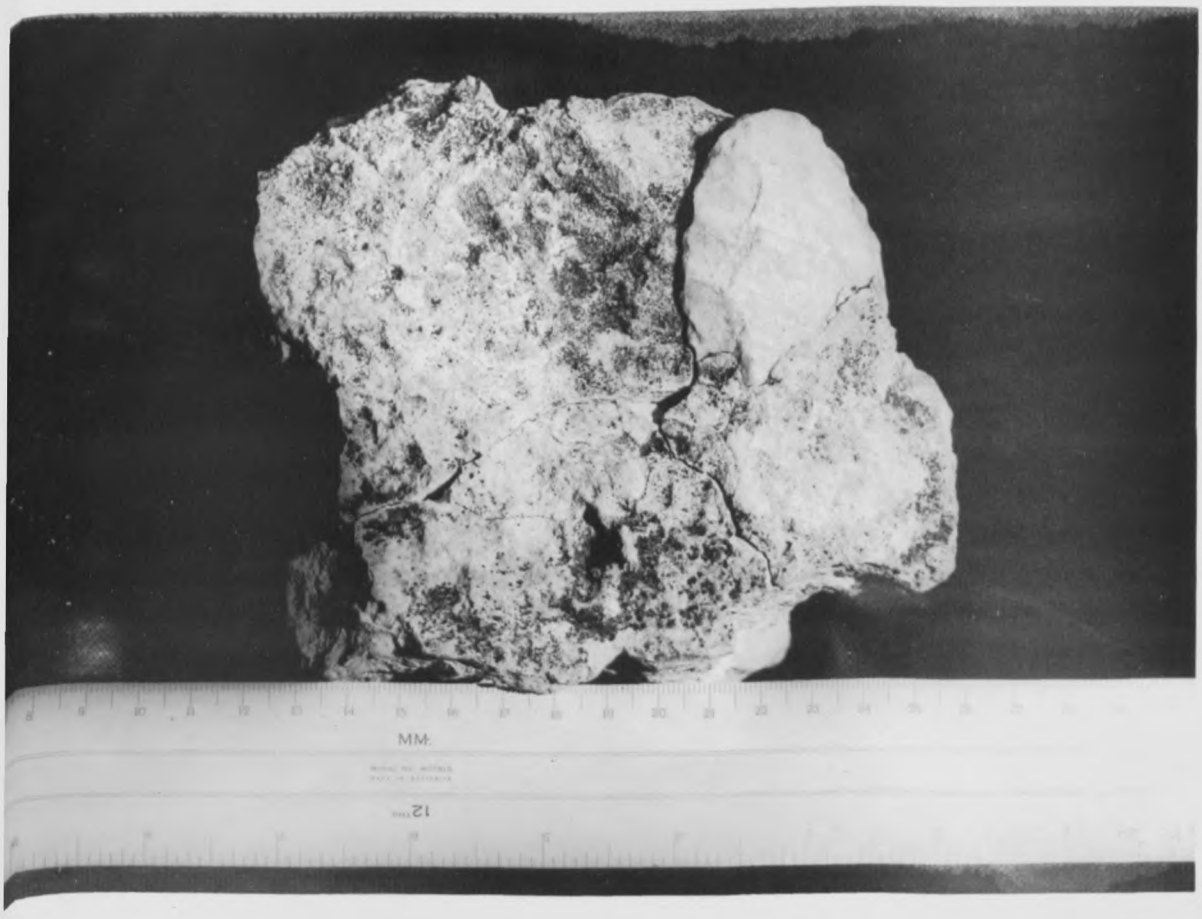


PLATE 5.10

Elliptical bifaced Middle or Later Middle Stone Age implement embedded in calcrete with an age of around 16,500 years B.C. (Sample 1907). The implement has an estimated length of 8 cm, a maximum breadth of 4½cm and a maximum thickness estimated at 1½ cm. It was not removed from the calcrete. *(Photo: Graphic Arts CSIR.*

(which is embedded to a depth of $2\frac{1}{2}$ cms), but avoiding the typical hard banded surface. The calcrete dated must therefore be younger than the artefact. This calcrete was homogeneous and did not show any of the original nodular structure. The owner of the farm, Mr. A. Viereck (1965, personal comm.) considered the artefact to belong to the Later Middle Stone Age. The artefact was also shown to Dr. R. Mason (1966, personal comm.) who considered it to belong to the Middle or Later Middle Stone Age. Dr. Mason also mentioned that similar artefacts are found with Later Lupemban or Rhodesian Stillbay assemblages both on the Witwatersrand and in Angola and that it was between about 15,000 and 30,000 years old.

Table 5.1 shows the C^{14} date to be very reasonable and it also falls within the range of Dr. Mason's estimate. Mr. Viereck considers the date to be reasonable, but a little on the old side. It is apparent from Deacon (1966) that not many C^{14} dates are available for even the main technological stages in South West Africa, so that the date must at present be regarded as satisfactory.

Sample 1961	150 A.D.
Sample 1962B	14,650 A.D.

Top of upper layer and bottom of lower layer, respectively, of calcrete hardpans from the Kalkbank archaeological site (Plate 5.11) where three hardpan layers overlie Upper Pleistocene fossils and stone tools belonging to the Middle or Late Pietersburg culture estimated to have an age of 13,000 - 28,000 years B.C. (Mason, 1962; p.63). The calcrete must therefore be younger than the fossils and artefacts.

Once again, the specimens dated were taken one to two inches in from the upper and lower surfaces, but no relict

nodules/ ...



PLATE 5.11

Part of the Kalkbank site north of Pietersburg, showing the three layers of slightly tufaceous hardpan. The upper one to two inches of the upper hardpan was C^{14} dated at about 100 years A.D. and the lower one to two inches of the lower hardpan at about 14,700 years B.C. Profiles 513 and 514 and Samples 1961 and 1962B were described and taken before the excavation was enlarged to that shown, but were at approximately this spot. A closeup of the opaline silica coating the upper hardpan is shown in Plate 4.3.

nodules could be seen and general specimens were used. If the hardpan growth is regarded as continuous (which it cannot be in this case, owing to the layers of dark soil separating the three layers) the un-normalised corrected ages used, and the samples are taken to be 36 inches apart, the overall thickness of hardpan formed at the rate of one inch per 410 years. This would make the calcrete in the upper surface some 560 A.D. and the calcrete in the lower surface some 15,060 years of age B.C. A faster rate than 410 years per inch is of course indicated for the profile. The actual banded surface coatings are probably also much younger than 560 A.D.

It is apparent that the lower C^{14} date is within the range of the estimated age of the artefacts. It may be worth noting that Mason (1966, personal comm.) considers that the bones at the site must have been covered rapidly to be as well preserved as they are.

Large samples 1-2 cubic feet in size were taken from each hardpan layer at this site and the radiocarbon samples later cut from these. The samples were taken where they could be prised and dug out with crowbars and picks and consequently were not quite vertically above each other, but were taken between Profiles 513 and 514 which were about 15 feet apart. The lower hardpans in 514 merged into one at 513.

The C^{13} and O^{18} measurements on Sample 1961 indicate that evaporation played a large part in its formation (Drs. J.C. Vogel and W. Roether, 1969; personal comm.).

Sample 1658A 31,250 B.C.

This calcrete was a piece of hard outcropping hardpan blasted out of a shallow trial hole from the proposed base borrow pit No. B.10 on the new Geluk-Namutoni road. The

actual/ ...

actual piece dated probably came from a depth of about six inches and as usual the relict nodules in the hardpan were cut out and as far as possible only these used for dating. This calcrete was chosen for dating because it fell within the northern area of the Kalahari Limestone and was thought to be Pliocene in age, a conclusion supported by the fact that this calcrete yielded the best aggregate crushing value (19%) of any hardpan yet tested. This calcrete was therefore thought to be undateable by the C^{14} method.

There are several possible explanations for the unexpected age of this calcrete:

1. This northern expanse of Kalahari Limestone may not be Pliocene in age. The only basis for putting its age at Pliocene was the fact that it occurred on the edge of the Kalahari Beds, it was well indurated and well developed, it outcropped (and was therefore fossil), and was included in the area shown as belonging to the African erosion surface on Partridge's (1968) map (Map 12). As has already been pointed out in Section 5.3.3, correlation of calcretes on stratigraphic grounds can be of dubious value. The work on which the area in question is correlated with the African surface has not been assessed. No fossils have been found in this area.

2. The calcrete dated may be a later one lying above the true Kalahari Limestone of Pliocene age: a section was unfortunately not available. In support of this possibility the author has seen implements said by Dr. R. Mason (1966, personal comm.) to belong to the Later Stone Age firmly embedded in the surface of the calcified Basal Older Gravels of the Vaal, themselves probably Pliocene in age.

3./ ...

3. The date may be incorrect. As has been pointed out by Broecker (1965), C^{14} ages on carbonates older than 25,000 years tend to be unreliable. From his results on page 747 and 748 it can be seen that a carbonate yielding an age of some 100,000 years by the Th^{230} method may only yield a finite age of some 30,000 years by the C^{14} method. The reason was not stated, but is presumably due to contamination by younger C^{14}. Contamination by younger carbonate is, however, not thought to be important in this particular case, as this calcrete was comparatively dense (water absorption only 1.6%).

In any event, it is apparent that the C^{14} date of this sample cannot at this stage be seriously questioned, and must be regarded as perfectly possible.

Sample 1598A	13,950 B.C.
Sample 1603A	15,450 B.C.
Sample 1603B	22,500 B.C.

These three samples all came from the same profile (161, Figure 5.2) in a proposed borrow pit (Ondangua Airfield No. 1 West) in a fossil drainage line west of Ondangua. The borrow pit in question is unfortunately well off the stereotriplet shown in Plate 6.3, but it occurred in an exactly similar situation to borrow pits 8 and 9. Sample 1598A was hardpan from 3'6" to 4'0" overlying nodular calcrete (Sample 1603). Sample 1603A represents the plus 10 U.S. mesh (+ 2 mm) fraction and 1603B the -10 mesh (-2 mm) fraction of the nodular calcrete from 7'6" to 8'0". The hardpan date (performed mostly on the original nodules in the hardpan, but with about 10% contamination by the matrix) shows that it formed after the nodules and proves part of the proposed sequence of calcrete development. The minus 10 mesh date also fits in with the proposed sequence of

calcrete/ ...



0'0"

1595 — Greyish brown, loose, dry, intact, calcareous, silty SAND with occasional ¼" hard calcrete nodules; roots; possibly aeolian.

0'10"

1596 — Light greyish brown, loose, dry, silty, sandy coarse, medium and fine GRAVEL; white, hard, nodular calcrete in above matrix; roots.

2'0"

1597 — White, stiff, dry HONEYCOMB CALCRETE; lenses out on west side of pit; roots.

3'6"

1598 — White, hard, dry, faintly very thinly bedded HARDPAN CALCRETE; consistency decreases downwards; original soil probably alluvial.

4'6"

Cream, loose, dry, faintly very thinly bedded silty, sandy, fine and medium GRAVEL; cream friable nodular calcrete; roots; original soil probably alluvial.

1603

10'0"+

FIGURE 5.2
PROFILE 161

Locality: Proposed Ondangua airfield borrow pit No 1 west.
Topography: Low rise on inside bend of oshana. Probably of partly alluvial and partly aeolian origin.
Vegetation: Bitterbos (Pechuel-Loeschea leubnitziae) and grass (not identified).
Date: 16/7/65
Scale: 2cms =1ft

calcrete development and presumably represents the age of the original calcareous soil in which the nodules grew. It seems surprising, however, that it was not contaminated by the younger carbon introduced by the process of nodule development. This may still have occurred of course, but there is no way of checking this.

Using the un-normalised corrected dates, 1598A and 1603A indicate a rate of formation of one inch per 31 years for the nodular calcrete. If 1598A is indeed contaminated by younger matrix, an even faster rate of formation is indicated. 1598 and 1603B suggest that it required some 8,500 years (or 177 years/inch) for profile 161 to develop from a calcareous sand to a hardpan/nodular calcrete profile.

It would be interesting to date the matrix of the hardpan and the nodules and matrix of the honeycomb and overlying nodular calcrete. It is thought that the nodular calcrete is being leached out to form the honeycomb calcrete under the present fairly high rainfall conditions at Ondangua.

CONCLUSIONS ON THE C^{14} DATING OF CALCRETES, LAKE OR STREAM TUFAS AND CAVE TRAVERTINES

1. All the 101 dates so far carried out on calcretes appear perfectly possible and none of the submitters or laboratories appear to have rejected any outright. Most of the dates have been carried out by the Arizona laboratory but they do not seem to have published any work on the dateability of calcretes.

2. Having regard to the mode of formation of calcretes, the few dates which gave anomalous (but not impossible) results can still be accounted for.

3./ ...

3. Nearly all calcrete samples taken down a profile increased in age with depth. A few taken from the surface of lava flows of differing age increased in age with the age of the lava flow (only relative ages of the flows were known). Three samples on marine terraces of different ages yielded ages in accordance with the relative ages of the terraces. These facts indicate that the C^{14} ages of calcretes should be at least relatively correct.

4. The very few calcrete samples on which the submitters seemed to have any check at all on the age agreed reasonably with the estimated ages.

5. It is considered that all pedogenic calcretes are formed with an initial C^{14} content of 85 $\pm$ 15% modern.

6. Depending on the origin of the carbonate in the calcrete in question a considerably more accurate estimate of the initial C^{14} content can probably be made.

7. Of the 101 calcrete dates found, 9 yielded ages of >30,000 years assuming an initial C^{14} content of 100%. This indicates that it is possible for calcretes to survive without large scale contamination by modern carbon.

8. The highest C^{14} content found so far in any calcrete was 90.4% modern and this was in the organic fraction. Only 3 yielded C^{14} contents of over 80% modern, two of which were on dispersed organic carbon. The highest inorganic carbonate C^{14} content was 85% modern. Only two samples out of the 101 were, however, actually described as in a possibly active state of formation.

9. The first ten C^{14} dates on calcretes from Southern Africa are presented. All results are perfectly

acceptable/ ...

acceptable and the three for which stone artefact checks were available yielded results in excellent agreement with the estimated age of the artefacts. All ten dates yielded ages compatible with the theory of calcrete development.

10. With certain reservations on the origin of the particular calcrete concerned, all pedogenic and many other calcretes should be dateable by the radiocarbon method to within ± 1,500 years plus laboratory errors, at least back to 25,000 years B.P. Provided contamination by modern carbon can be discounted, they should be dateable back to the range of the method with this uncertainty. By an evaluation of the origin of the carbonate concerned, this uncertainty can probably be reduced to ±500 years in some cases.

11. Further work on calcrete dating is desirable, however, and the form this could take is outlined in Section 5.8. The work can also be expected to yield valuable results in connection with the origin of calcrete, particularly if supplemented by C^{13} and O^{18} measurements.

12. No previous detailed consideration and work on the dateability of calcretes appears to have been published.

13. In view of the large number of archaeological and fossil occurrences associated with calcretes in Southern Africa, it is predicted that calcrete will become one of the most important dateable materials in Southern Africa.

14. The reliability of C^{14} dates on the inorganic $CaCO_3$ portion of dendritic and lithoid lake tufas formed by inorganic and algal processes has been established, notably by Broecker and co-workers. They have proven

beyond/ ...

beyond reasonable doubt that:

a) The initial C^{14} content was within a normalised 5% of that incorporated into terrestrial organic matter.

b) Errors due to contamination with modern carbon were in practice small (tufas 11,000 - 19,000 years old should have their ages reduced due to this by less than 1,500 years).

c) Agreement between co-existing inorganic and organic material was good.

d) The uncertainty of the age estimation due to difference in initial C^{14} content was only $\pm$ 500 years.

They also found that some thinolite tufas gave young ages, possibly due to recrystallisation.

15. C^{14} dating of cave travertines seems to be established in Europe, mainly due to the work of Brinkmann, Münnich and Vogel. Initial C^{14} contents of 85 $\pm$ 15% modern have been used by these workers for material having normal C^{13} contents (δC^{13} = -5 to -10°/oo in Europe). This corresponds to an age uncertainty of $\pm$ 1,500 years. Few checks on the actual ages of their materials seemed to be available, however.

16. The reliability of dates on lacustrine limestones in general, cliff limestone (dripkalk), and the apparent similarly formed stream tufas has not been assessed Several dates on lacustrine limestones were noticed in radiocarbon. An assumption of an initial C^{14} c tent of 85 $\pm$ 15% modern and a δC^{13} of around 0 $\pm$ seems justified, however. Further work should be to clarify this position as much higher (perhaps

initial/ ...

initial C^{14} contents) are possible in some of these materials and C^{13} contents might be variable.

5.3.6 Other methods

Leaching

The use of the thickness of the leached horizon in glacial drift and loess as a basis for age correlation in the United States has been critically reviewed by Flint (1949) in terms of the five pedogenic factors. The method works on the assumption that the carbonate in the upper portion of a calcareous soil is leached downwards by meteoric waters and deposited at a greater depth in the profile. With time the thickness of the leached zone thus increases to some limiting value. Flint concluded that provided certain conditions were met in the choice of sites and a statistical number of sites investigated, the depth of leaching method could differentiate between at least some pairs of drift sheets of different stratigraphic stages. He considered that correlation of two deposits of less than stage value was not likely to be reliable. He listed five conditions that should be met in the choice of sites of which comparable soil type and flat topographic situation were the most important.

It is apparent that some idea of the relative ages of carbonate horizons in areas such as the Springbok Flats and parts of the Orange Free State could probably be arrived at by this method.

Arkley (1963) has taken this sort of approach one step further and actually calculated minimum times for the present climate to produce the carbonate horizons in 13 soils on the Great Plains of the United States which were in agreement with the known geological ages of the parent materials. This he did by calculating the long term rate of water

movement/ ...

movement in the soil from climatic, suction/moisture content and soil density data and assuming two different carbonate solubilities. The solubility of the actual calcium plus magnesium carbonate in soils was calculated to be higher than that of pure calcium carbonate. This of course resulted in lower ages being obtained than when the pure $CaCO_3$ solubility was used.

This approach of Arkley's is very interesting and deserves further investigation. It would be extremely interesting for example to carbon date the carbonate in soils whose ages have been calculated by this method. Not only would this verify or disprove Arkley's method, but it would also provide valuable information on the true solubility of carbonate in soils. Such data seems to be scarce and Olsen and Watanabe (1959) have also found that the calcareous material in several soils was more soluble than calcite. They suggested that this might be due to the presence of more soluble, unstable forms of $CaCO_3$ in soils. If the C^{14} dates verify Arkley's method they would conclusively prove the downward leached origin of the carbonate in the profiles studied and the other assumptions inherent in his method. One of these, that upward translocation of carbonates is of insignificant importance, is of great interest as a possible means of calcification.

Evaporation

Some sort of estimate of the age of a calcrete forming at the present time can probably be made by consideration of the maximum rate that carbonate can be precipitated from solution in a profile undergoing evapotranspiration. In Section 5.5 it will be shown how an equation can be derived to give the time required for a given layer of calcrete to form. It may also be possible to apply this equation to calcretes forming at the present time in order to calculate their age. The method should be checked against various

calcretes/ ...

calcretes which have been C^{14} dated and shown to be single phase and still forming.

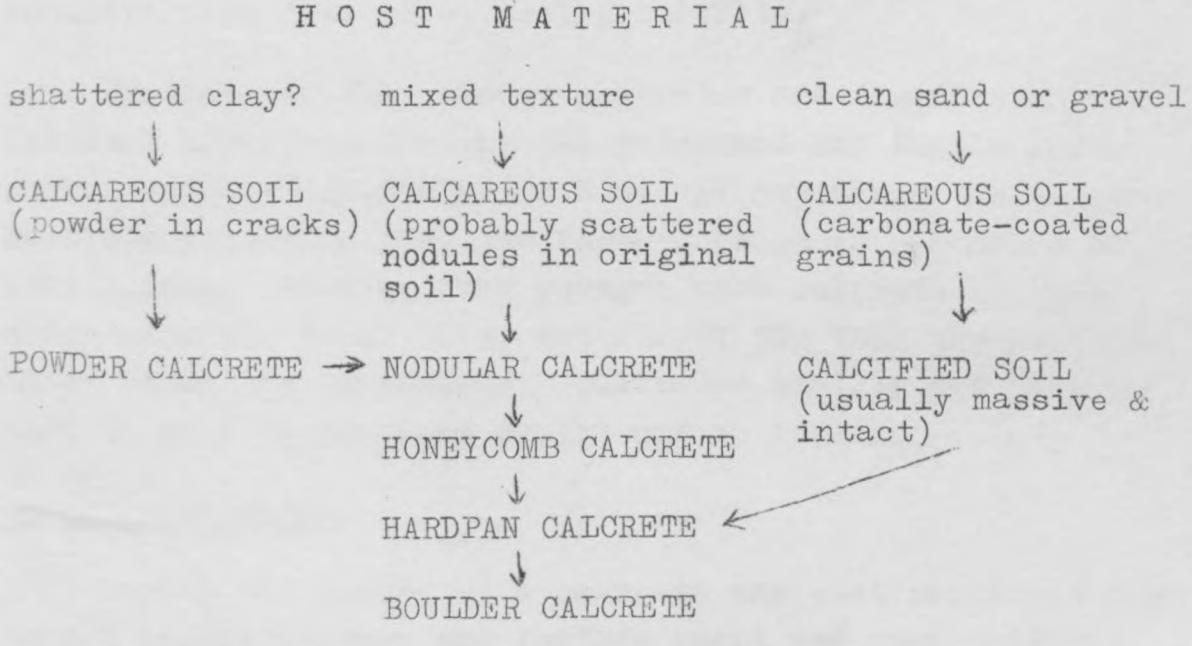
Extrapolation from laboratory experiments

The time required to form calcretes under accellerated laboratory conditions can probably be used to give some idea of the time required for natural calcretes to form, and hence some idea of their age.

Several other methods also exist whereby some idea of the relative ages of calcretes in close proximity to each other may be obtained. They must be applied with caution, since they can serve as guides only.

Stage of development

The theory of calcrete development predicts that calcretes and calcareous soils can be arranged in the following order from oldest to youngest:



using/ ...

using the convention that the youngest calcrete in any given area or profile is the one which attained its particular stage of development (i.e. boulder, hardpan or nodular, etc.) most recently.

The prediction of course holds good only for a single phase of calcification. Any subsequent phase of calcification might produce say a nodular calcrete in the same profile which would of course be younger than the boulder calcrete. This affords a means of recognising the presence of more than one phase of calcification.

Hardness

The further a calcrete proceeds along its developmental path the harder it becomes. Thus the nodules in a powder calcrete are usually friable, while boulder calcretes are almost invariably of hard Type 1 calcrete. Scattered hard Type 1 nodules (often enclosed by an equally hard brown and black banded crust) in say a predominantly Type 4 nodular calcrete are probably remnant nodules or (more likely) boulders from some pre-existing calcrete.

Examples of the hardest calcretes are the Pliocene Kalahari Limestone capping the Weissrand and Sample 1658, carbon dated at 31,250 years B.C. If anything, the later calcrete is harder than the former, which is weathered in many places. Another even younger hard calcrete is that which caps the Basal Older Gravels of the Vaal and encloses Later Stone Age implements. Calcretes plainly get just so hard (5 or 6 on the Mohs scale) and no harder.

Cement/sand ratio

During the growth of a calcrete the soil particles seem to get pushed further and further apart and even replaced by the carbonate matrix. Thus, in the hand specimen, boulder calcretes tend to exhibit few sand grains while

nodules/ ...

nodules are commonly quite sandy. This pushing apart of the original sand grains was also noticed during the laboratory experiments described in Section 4.3.4. While it probably occurs to all soil particles, the effect on the sand-sized particles is the one most easily observed in the hand specimen.

Distinctness

Brewer and Sleeman (1964) have pointed out that "glaebules with sharp external boundaries may have been transported or formed in situ, whereas those with diffuse boundaries have probably been formed in situ". This concept can be taken further and it is suggested that calcrete nodules with diffuse boundaries are likely to be at an earlier stage of development than those with sharp boundaries, in fact they are likely to be forming at the present time.

5.3.7 Methods of detecting calcretes forming at the present time

Before calcretes can be used as quantitative palaeoclimatic indicators, a correlation must first be established between calcretes which are forming at the present time and the present aerial climate and other soil-forming factors. It is not, however, easy to decide which calcretes are forming at the present time and Flint (1959) has stated that "the distribution within Southern Africa, of soils in which calcium carbonate is actively accumulating has not yet been defined" and goes on to suggest a tentative figure of 18 inches of rainfall. Apart from mentioning that "degraded ancient soils" can only be distinguished from actively forming soils through detailed examination, he gave no details of how this might be done. It is the purpose of this section then to suggest methods by which calcretes forming at the present time can be distinguished from those which are fossil. These methods can be listed

as/ ...

as follows:

1. State of equilibrium of the calcrete with its environment.

   Stratigraphic position
   Coexistence with certain other soils
   Composition

2. Radiometric dating

3. Calcification of recent man-made objects.

1. State of equilibrium of the calcrete with its environment

While calcretes probably never achieve a state of complete equilibrium with their environment, any that are grossly out of equilibrium must obviously be regarded as suspect. For example, the correlation between calcrete distribution and climate found in Section 2.3.1 is so good that gross exceptions to this must inevitably be suspected of being fossil. A detailed assessment of the other pedogenic factors at the site may provide the answer, but as the exact conditions under which calcretes can form are still imperfectly known, this should not be completely relied upon.

An example of disequilibrium with the present drainage system is the occurrence of calcretes such as those shown in Plate 6.2. They form a long, narrow, but low ridge, sometimes outcrop, but are mostly covered by red sand of Kalahari type. The only other calcretes in the vicinity occur associated with present-day drainage lines. The sand on either side of the ridge to a depth equivalent to well into the calcrete is exactly the same as that covering the ridge and yet it possesses no calcrete. In fact it is not even calcareous to a depth of at least six feet. Unless a shallow water table exists under the ridge, most of the calcretes on it must be regarded as fossil.

The/ ...

The following criteria can also be regarded as criteria of disequilibrium with the regional or local environment.

Stratigraphic position

If it is accepted that pedogenic calcretes, particularly hardpans, grow only in the soil a few feet below the surface, then any pedogenic calcrete which is outcropping is plainly fossil. Conversely, any pedogenic hardpan the upper surface of which is more than about two feet below the present day surface must also be regarded as fossil. At present only these wide limits can be suggested but, in time, once it is established which calcretes are still forming, it should be possible to define within narrow limits the depth of occurrence of any active calcrete occurring in a particular soil texture on flat land and under a particular aerial climate.

Many, if not most, of the calcretes in the zone receiving less than 22 inches (550 mm) of rainfall outcrop: the term 'outcrop' is used here in the sense that they have no soil covering of any sort, not necessarily that they project above the general ground surface. This leads to the important conclusion that many, if not most, calcretes in this zone are fossil. Another interesting point is that there is often no well defined transition zone, i.e. in many places hardpans or boulders outcrop up to say the 22" isohyet, but do not occur at all on the wet side of it. Where then are the actively forming hardpans? At the moment no entirely satisfactory answer can be given to this question. Honeycomb calcretes with calcareous fines can be taken to be actively forming hardpans, but they are rare. It is tempting to suggest then that there are none, or at least that very little hardpan formation is taking place in Southern Africa at the present time. This would seem to be impossible, however, as the range of climate encountered must surely be sufficient to ensure hardpan formation

somewhere/ ...

somewhere in Southern Africa. Perhaps a special change in climate is needed, such as the waning of a pluvial? This would decrease the depth of leaching in all say nodular calcrete profiles, which may well be a requirement of hardpan formation.

Whether these outcropping hardpan calcretes brought this situation on themselves (killing themselves off so to speak) by creating an horizon impermeable to water and thus conducive to the erosion and devegetation of the overlying soil or it was caused by an increase in aridity remains to be seen.

If most of the calcretes on the dry side of the 22 inch isohyet are fossil, then perhaps most of the calcretes on the wet side are active? This suggestion is supported by the fact that, in general, only calcretes at an early stage of development, such as discrete calcrete nodules in a calcareous soil matrix, occur in this zone. It seems fairly certain that this is indeed the case, and radiometric dating will provide the answer, but why then does one not encounter hardpan formation in this zone? The logical answer to this question is that the degree of leaching is sufficient to prevent it, in other words the calcretes in the area can develop just so far and no further. This is supported by the fact that the calcretes in clays in this zone are usually better developed than those (few) in sands.[xx] This brings us back to the suggestion that hardpan will only form in these soils if

a) there is a decrease in rainfall; or

b) there is an increase in temperature (which would increase evaporation); or

c) there is an increase in transpiration; or

d) there is an increase in overburden thickness; or

e) there is a decrease in overburden permeability (or

[xx] Clay probably retards both calcrete development as well as leaching.

possibly in the permeability of the calcrete horizon itself).

Another criterion which can be applied with pedogenic calcretes is that of whether or not they follow the present ground surface without outcropping. If a hardpan did this it would certainly at least be no older than the present ground level, while if a nodular calcrete with strongly calcareous fines (ratio of plus 40 U.S. mesh CO_2 to minus 40 mesh CO_2 not greater than 2.0?) were to do it, it would be reasonable proof of its contemporary nature.

Co-existence with certain other soils

The occurrence of calcretes and ferricretes together in the same horizon must be regarded with suspicion, although it should be noted that Mason, Brink and Knight (1959) have recorded the apparently concurrent formation of calcrete and ferricrete in the same profile. Scattered calcrete and ferricrete nodules occurring together in the same profile or horizon are quite common in the soils of the Orange Free State goldfields (see Section 2.3.2), but it is not known which of the two (if either) is fossil. The author believes that ferricrete - like materials can be formed by podsolisation, under impeded drainage conditions, or by laterisation. Scattered nodules of ferricrete and calcrete would thus be quite compatible in the same profile or possibly even the same horizon of some soils, but definitely incompatible in ferrisols, and ferrallitics or even in the normal profiles of fersiallitics.

The processes of silicification and possible ferruginisation of calcretes is too complex and vast a subject to be considered here. It will be suffice to note that silicification of Southern African calcretes seems to be accepted by at least three authors (Passarge, 1904; Rogers, 1907;

and/ ...

and Poldervaart, 1957) and that certainly decalcification and possibly silicification of some calcretes may be proceeding at the present time in the Runtu-Sambio area. The relations between calcrete, ferricrete, laterite, silcrete, dolocrete, phoscrete, and manganocrete have so far been insufficiently studied.

Composition

The theory of calcrete formation predicts that actively forming calcretes will have calcareous fines. This should hold true for any calcrete. The converse, however, that all fossil calcretes will have non-calcareous fines need not necessarily hold true. It is plain that only a calcrete which has been subjected to increased leaching will have fines deficient in carbonate. Examples of calcretes without or very deficient in calcareous fines are the boulder calcretes shown in Plates 1.19 and 5.12, the honeycomb calcrete shown in Plate 1.10 and a few nodular calcretes (see Section 3.3.4). Plate 5.12 (Profile 599) is a particularly interesting example. The calcrete boulders above the geological pick (the petrocalcic horizon of some pedologists?) are being leached downward to form younger calcretes below. The red sand (of Kalahari type?) matrix is non-calcareous above and on a level with the upper boulders, but is slightly calcareous below, while in the lower layers of the profile nodular and honeycomb calcretes (not very clearly shown by the photograph) occur intercalated between the weathering hardpan layers. The Pienaarsrivier-Springbok Flats area seems worthy of further study, since it seems probable that sites can be found where changes in the other pedogenic factors can be ruled out and that an increase of rainfall or decrease of temperature has been responsible for the latest features of this particular profile, though Dr. H. Von Moltitz Harmse (1969, personal comm.) has pointed out that an increase

in/ ...

in the permeability of the over burden would of course result in a similar effect. It is of interest to note that Wagner (1927) postulated that the Springbok Flats receives a higher rainfall at the present time than it did in the past. He cited evidence of stream rejuvenation, the dying out of trees typical of more arid areas such as the Kalahari and the establishment of moisture-loving species. As was pointed out earlier it seems very likely that the whole world is now in a phase of lower (and still decreasing) temperature. Due to a reduction in evaporation, a decrease in temperature would of course result in an increase in effective rainfall (see Section 2.3.1). As Maps 11A and 11B show, the general area north of Pretoria is an enclave of lower rainfall within an area of higher rainfall and is thus probably particularly sensitive to climatic change. It is also interesting to note that the calcretes in this area correlate with some climatic indices (see Section 2.3.1), but not with others. As the various indices emphasise different aspects of climate, further investigation along these lines may show whether temperature or rainfall or both have changed since the formation of the hardpan - which can probably be C^{14} dated. It is interesting to note that Table 5.1 shows that at least parts of Southern Africa are now colder than they were a few thousand years ago. The alternative explanation of Profile 599 is also in agreement with this.

The calcareous fines aspect is taken further in Section 3.3.4 and it seems that a nodular, powder or calcified sandwith a ratio of plus 40 mesh to minus 40 mesh CO_2 or $CaCO_3$ of less than about 2.0 could be active, while those with a ratio of more than 2.0 or possessing kaolinite in their fines are probably fossil.

A plot of carbonate content versus depth over the whole profile should also be indicative. Thus Singh and

Lal (1946) decided on the stage of development of their kankar profiles by consideration of the extent of leaching as measured by the $CaCO_3$ content of the horizon overlying the kankar. Development was assumed to increase with decreasing $CaCO_3$ content of the overlying soil until at maturity it became completely non-calcareous. While fundamentally sound, this method can plainly only be applied to calcretes which owe their origin to downward leaching.

Occasional concretion-like objects are found in nodular calcretes which appear to be hard Type 1 nodules covered by concentric, thin, alternating layers of lighter/darker brown and black hard material exactly similar to Sample 1028A. These objects are always rare and never make up a large proportion of any nodular calcrete. It is considered that they are not nodules at all, but small calcrete boulders relict from some pre-existing calcrete. The often reasonably concentric nature of the bands presumably indicates rotation during boulder formation.

It was initially thought that the aragonite/calcite ratio of calcretes might reveal contemporary calcretes if aragonite was a necessary step in the formation of calcrete. Extensive X-ray analyses, however, have so far failed to reveal aragonite in any calcrete within the detection limit of the apparatus (approx. 2%). A survey of the literature has also since revealed that it is theoretically unlikely that an aragonite stage is involved as a general step in calcrete formation, in spite of the suggestion made by Price (1933) and the petrographic evidence for its former existence in some calcretes (Appendix F).

It was also at first found that only some of the older calcretes (Passarge's (1904) 'pan sandstone?') were dolomitic and it was thought that the dolomite/calcite ratio might be useful as some sort of age indicator. Some green and brown sandy dolomitic nodules (Type 5 calcretes) thought

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to be possibly contemporary were, however, later found in the bed of the Omuramba Ovambo and in terraces around the edges of the Etosha Pan, suggesting that the dolomite was not a replacement feature.

2. Radiometric dating

A very young or more or less contemporary age obtained by C^{14} or some other radiometric method should be reasonable proof that a calcrete is still forming at the present time. The minimum age measurable by the conventional C^{14} technique is of the order of 70 years, but material formed subsequent to 1954 can be C^{14} dated to plus or minus about one year by the hydrogen bomb effect (Dr. J.C. Vogel, 1969; personal comm.). Avoidance of contamination may be a problem and, in the case of C^{14} dating, corrections for C^{14}/C^{12} disequilibrium, isotopic fractionation and variation of the C^{14} content of the atmosphere with place and time may be necessary.

Particularly attractive is the possibility of Sr^{90} analysis. Presence of this isotope in a calcrete should be conclusive proof that it is contemporary. A method for the determination of Sr^{90} has been given by the American Public Health Association (1965). Other bomb-produced isotopes might also be used. Tritium dating may also be possible, but, as calcium and strontium have very similar chemical properties, the Sr^{90} method holds most promise. Sr contents of calcretes are shown in Tables 3.1 to 3.8. The tritium contents of calcretes are unknown.

3. Calcification of recent man-made objects

Recent objects such as bottles, fence posts and the like which have been firmly cemented by calcrete should be sought for, while excavation of other recent man-made features such as graves and ruins might also be revealing

(graves/ ...

graves suggested by Professor H. Martin, 1964; personal comm.).

Later Stone Age artefacts such as those cemented into the top of the Basal Older Gravels of the Vaal are probably too old to be of much use for this purpose, but calcification of an iron age site would definitely be of interest.

None of these possibilites has been seriously investigated, but although a sharp lookout for any such objects was maintained during field investigations, none were found.

5.4 AGES OF CALCRETES IN SOUTHERN AFRICA

There are relatively few sites in Southern Africa where calcretes have been reliably dated and in most cases only maximum ages can be assigned with any certainty. The calcretes at these sites can be divided into those of pre-Pliocene age, those apparently of Pliocene age, those containing Acheulian artefacts, those containing or overlying Middle Stone Age artefacts and those which are forming at the present time. A few apparently fossil calcretes have also been found which contain Later Stone Age artefacts.

Only the ages of calcretes are considered in this work. The important cliff limestone deposits of the Campbell Rand described by Young (1925) and Peabody (1954) and the calcified cave breccias described for example by Brain (1957) are thus excluded from this discussion. Calcretes do occur in some of the above deposits, as both Young and Peabody speak of the deposits as being of composite origin, both vlei and cliff limestone being present. The fossil fauna, however, only apparently occur in cave deposits in the latter. Peabody has correlated the various travertines of the Campbell Rand with the Vaal deposits, the correspondence of which has been remarked upon by Cooke (1967), and further work should examine further the

possibilities/ ...

possibilities of this and the possibility that some of the calcified cave deposits might fall within the definition of a calcrete. For the purposes of this work, however, they are excluded.

5.4.1 Pre-Pliocene calcretes

Various calcrete-like materials occur intercalated in the Kalahari Beds in South West Africa and elsewhere. At present all that can be said regarding their age is that they must be post-Upper Cretaceous and pre-Pliocene in age.

Mabbutt (1952) has stated that the Tertiary Main Terrace deposits of the Ugab were laid down on calcrete-covered Khomas schists. This makes these calcretes older than the apparently Pliocene calcification of the Main Terrace deposits.

There is of course no reason whatever why calcretes should not be found in deposits older than the Kalahari Beds. Such calcretes undoubtedly do exist, but presumably due to their usually thin nature, they have been overlooked in the past. They may well also have become so altered and consolidated in many cases that they are now in any event indistinguishable from limestones.

5.4.2 Pliocene calcretes

Pliocene calcretes include the Kalahari Limestone and some calcified river terraces such as the Basal Older Gravels of the Vaal and the Main Terrace of the Ugab. These are the oldest calcretes in Southern Africa that have been dated with any degree of certainty.

The Kalahari Limestone

As was pointed out in Section 5.3.3, if the term 'Kalahari Limestone' is to have any stratigraphic significance

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at all it is essential that it be restricted to the calcretes of presumed Pliocene age and not extended to all the calcretes bordering the Kalahari or occurring near the top of the Kalahari Beds. While it is very convenient to lump together all the calcretes occurring in the belt bordering the southern and western Kalahari under the term 'Kalahari Limestone' it is considered that this is only stratigraphically justified as long as they are indistinguishable on an age basis. As soon as they become distinguishable the term should be restricted to those deposits of Pliocene age. In anticipation of this then, only those calcretes which were apparently initially calcified in the Pliocene Epoch are included here under the term 'Kalahari Limestone'.

Similarly, since the 'Kalahari Sand' is now regarded as not being everywhere of the same age, this term is restricted here to the original Kalahari Sands, equivalent to Cahen and Lepersonne's (1952, in Mabbutt, 1957a) Plateau Sands of the Congo. In the South West Kalahari the original Kalahari Sands are probably Pliocene in age and are unconformably underlain by the Kalahari Limestone. In deriving an age for the latter it is obviously thus also useful to consider the former. Following recent practice, where the age of an otherwise similar sand is uncertain or definitely post-lowermost Pleistocene, it will be referred to as 'sand of Kalahari type'.

Passarge (1904) suggested that the Kalahari Limestone started forming in Miocene and Lower Pliocene times and stopped forming and hardened during the Middle Pliocene. He also stated, however, that the mollusc and diatom evidence pointed rather to an Upper Pliocene age, but elsewhere in his work it appears that these fossils only reliably indicate that the Kalahari Limestone is no older than Upper Tertiary. Passarge regarded the Kalahari Sand

as/ ...

as lowermost Pleistocene in age.

Wagner (1916) considered that the dissection undergone by the Urinanib Plateau required a pre-Pleistocene age for the Kalahari Limestone.

Maufe (1930) considered the Kalahari Sand near Bulawayo to be Upper Tertiary or Pliocene in age. Thick deposits of calcrete such as occur along the Weissrand do not seem to occur in this area.

Du Toit (1933) considered that the Kalahari Sand was emplaced prior to the warping of the Kalahari-Rhodesia axis which latter took place about the close of the Pliocene, possibly cutting off the Okavango from the Limpopo. He also considered the Red Sands of the Pan Belt to be older than the Palaeolithic gravels of the Vaal. A late Tertiary date was therefore ascribed to the Kalahari Sands.

Wayland (1954, in Bond, 1963) differed from most other workers in regarding the Botletle Beds (the Kalahari Beds below the Kalahari Limestone) as early Pleistocene in age. Bond stated that, owing to their mostly unfossiliferous nature, their age must still be regarded as uncertain.

Mabbutt (1955, 1957a) regarded the upper surface of the Kalahari Limestone as an interior expression of the African erosion cycle. He correlated the Bushmanland, Great Namaqualand and Kalahari Limestone surfaces with this cycle. On the basis of the occurrence of the early Pliocene horse Notohipparion namaquense (Haughton, 1932; in Mabbutt) in Bushmanland, and further physiographic evidence, he has placed the Kalahari Limestone as probably early Pliocene in age and the original emplacement of the Kalahari Sands in the South West Kalahari in Upper Tertiary times. The more general use of the term "Plateau Sands" used by Cahen and Leppersonne (1952, in Mabbutt 1957a) in the

Congo/ ...

Congo for the original Kalahari Sands (as Mabbutt has done) or the restriction of the term "Kalahari Sands" seems highly desirable. Mabbutt (1955) stated further that the Plateau Sands unconformably overlie the Kalahari Limestone "but are themselves truncated by the end-Tertiary river plains and by the Kalahari valleys of a Pleistocene pluvial or pluvials".

Korn and Martin (1957) regarded the Kalahari Sand in South West Africa as having been deposited at the end of the Tertiary, but do not commit themselves as to the age of the Kalahari Limestone. Cooke (1957) accepted the age of the Kalahari Sand as probably Pliocene, while Bond (1963) accepts a Miocene or Pliocene age.

In connection with the apparently widespread distribution of sands of Kalahari type, Piaget (1963) has sounded a warning by concluding that the assumed aeolian soils of the Orange Free State are not aeolian at all, but are of autochthonous origin. He considered further that the so-called aeolian Kalahari Sand overlying Gravels II at Canteen Koppie east of Barkley West was derived mostly from local Ventersdorp Lava with the addition of some fluviatile material. Cooke (1957), applying Bond's (1957) criteria has also concluded that some of the assumed aeolian sand (calcified in places) of the Vaal basin has fluviatile as well as aeolian characteristics. It is apparent that there is much scope for the application of sedimentological techniques to Quaternary geology in Southern Africa after the manner of Bond (1957), Poldervaart (1957), Brain (1957) and Piaget (1963). Much useful information on techniques is given in Brewer (1964).

The 'Kalahari Limestone' around the Etosha Pan and around the southern rim of the Kalahari does not appear to have been dated in any way. Beyond implying that some of the calcrete at Witkop near the Hygap river may be very

young/ ...

young, Rogers (1907) and Du Toit (1907) also did not come to any conclusions regarding the age of the calcretes in the Gordonia-Mafeking area. No subsequent conclusions regarding the age of the calcretes in this area seem to have been published. Consideration of Partridge's (1968) landsurface map (Map 12) indicates that at least some of the calcretes around the southern egde of the Kalahari and around the Etosha Pan could be of the same age as the Kalahari Limestone of the Urinanib Plateau. Probably several ages of calcrete exist in these areas however, as indicated by the brecciated nature of some of the boulder and hardpan calcretes in the South West Kalahari and southwest of the Etosha Pan. The apparently anomalously young C^{14} age of Sample 1658A probably also indicates that subsequent phases of calcification and solution affected the pre-existing calcretes.

In conclusion then, it would seem that the consensus of opinion is that the Kalahari Limestone (sensu stricto) is probably Middle Pliocene in age with a probably maximum age of Early Pliocene and a probable minimum age of late Pliocene and pre-Kalahari Sand. The remarks made in Section 5.3 concerning the Pliocene/Pleistocene boundary should, however, be borne in mind here. The evidence for the age of the Limestone is, however, slender, being based on one mammal and some diatoms and molluscs, none of which were found in the Kalahari Limestone of the Urinanib Plateau-indisputably the best example of the limestone - and the observation that it is unconformably overlain by the Kalahari Sand. The fossils of course can only indicate maximum ages and the author has done too little work in the South West Kalahari to be able to comment on the unconformable nature of the contact between the Limestone and the Sands. Some features both there and southeast of the Etosha Pan, however, which perhaps in any other rocks would have been called unconformities, were considered to be the

solutional/ ...

solutional production of boulder calcretes and makondos and which could have taken place before or (more likely) after the deposition of the overlying sands of Kalahari type and the onset of a wetter climate.

Calcified river gravels

That the initial calcification of the Basal Older Gravels of the Vaal must have taken place soon after their deposition is apparent from the well-preserved state of the included Ventersdorp Lava fragments (Cooke, 1946). This is in accord with the hypothesis of calcification of these deposits outlined in Section 4.2.5. Clauss (1967) has also shown that the weathering of basic igneous rocks is retarded under conditions of high pH. The Older Gravels (also known as the Red Potato gravels, and as the Derived Gravels by Partridge and Brink, 1967) contain contemporary (Cooke, 1946) rolled Kafuan and Oldowan tools (Van Riet Lowe, 1952) while Van Riet Lowe (1953) has recorded unrolled Kafuan tools from the top 18 inches only of the Basal Older Gravels - apparently in recalcified Derived Gravels. Mason (1967), however, suggests that these artefacts are in fact Middle or Later Stone Age types. As the Derived Gravels are considered to be colluvium derived from the Basal Older Gravels (Partridge and Brink's 200 ft Gravels) (Cooke, 1946; Van Riet Lowe, 1952; Partridge and Brink, 1967) and, in view of Van Riet Lowe's finds, the Basal Older Gravels themselves must be pre-Oldowan and Kafuan, that is at least some 2 million years old. There are unfortunately no convincing fossil remains from these gravels (Partridge and Brink), although Mason (1967) has found two artefact-like objects of possible Earlier Stone Age type at Holpan, but on geomorphological grounds these authors consider that the Post-African surface at Windsorton on which the Basal Older Gravels rest is some 4.8 million years old. This figure was arrived at by assuming that the Post-African

nickpoint/ ...

nickpoint moved at a constant rate from its starting point at the coast during the Miocene some 15 million years ago to its highest point 400 miles above Windsorton today. This assumption is of course debatable, but the age yielded is nevertheless very acceptable.

The calcification of the Basal Older Gravels therefore probably took place some 2 - 5 million years ago, that is in Middle to Upper Pliocene times as the water table slowly sank in the alluvium, either due to a reduction in flow or degradation of the river bed. Cooke (1946) placed the calcification between the pebble cultures and the Chelles-Acheul I and after the formation of the Older Gravels, but there does not seem to be any reason why the calcification could not have taken place before the formation of the Older Gravels, as in Van Riet Lowe's (1952) table on page 145. A pre-Chelles-Acheul I date for the calcification is alone assured, but the author considers the Pliocene date most likely.

Mabbutt (1952) considered that the calcification of the Main Terrace deposits of the middle Ugab Valley was end Tertiary in age. They may well be contemporary with the Uis deposits mentioned to occur at the eastern foot of the Brandberg.

To summarise then, there is evidence that the calcification of the Kalahari Limestone (sensu stricto), the Basal Older Gravels of the Vaal, and the main terrace deposits of the Ugab may have been contemporary with each other, that is Pliocene in age. Some other deposits such as those of the Uis and perhaps the calcretes of the upper terrace of the Kunene near the Ruacana Falls may also fall into this category. No work seems to have been done on these deposits. The Pliocene of course lasted some 10 million years, so that only radiometric dating or some really good fossil finds will reveal whether these deposits are truly contemporary or not.

5.4.3 Calcified gravels and sands containing Acheulian tools

Calcretes (mostly calcified alluvial gravels and sands) containing Acheulian stone implements) occur at several river sites in Southern Africa. Some details of those which have been described are given below.

According to Cooke (1946) and others, South African Chelles-Acheul V+ (Mason's (1962) later Acheulian) artefacts occur in the lower parts of the calcified sands overlying the younger Gravels II and III of the Vaal, while Middle Pleistocene mammalian fossils have been found in the Younger Gravels I and II (Cooke, 1949; in Van Riet Lowe, 1952). This of course would only mean that the calcification is younger than the tools and fossils, but Cooke (1946) goes on to state that the tops of the calcified sands have been partly terraced, on which occur Fauresmith I tools. The calcification must therefore be post Chelles-Acheul V+ and pre-Fauresmith I. According to Table 5.1 this probably makes the date of calcification 40,000 - 50,000 years B.C., though recent work suggests that the MSA began prior to 51,000 B.C. It should be noted that Mason (1967 and personal comm.) doubts whether such accurate subdivision of the Chelles-Acheul culture is possible. It should be mentioned here that all the Younger Gravels, I, II and III (oldest to youngest) are equivalent to Partridge and Brink's (1967) Current Gravels. The latter authors regard their 70 ft terrace as being Middle Pleistocene in age. Mason (1967) has also found artefacts possibly belonging to the Earlier Stone Age in the calcified derived gravel of a calcified makondo (?) in the Basal Older Gravels at Holpan.

Many rivers in South West Africa such as the Fish, Auob, Nossob, Khan, Kuiseb, Swakop, Omaruru and Ugab

possess/ ...

possess calcified gravel terraces which Korn and Martin (1957) regarded as being equivalent to the Chelles-Acheul gravels of the Vaal, though implements have not been found in all these deposits. In the Fish river valley at Wasserfall for example "the calcified gravel is simply packed with Chelles-Acheul implements representing Stages III to V of the Vaal river. Tools of stages III and IV are always slightly rolled but those of stage V are never rolled". The only fossils which have so far been found in the South West African gravels are those from the Khan river at Usakos (Gevers, 1933; in Korn and Martin, 1957) which according to Haughton (in Korn and Martin) support the conclusion that these gravels correspond with the Chelles-Acheul gravels of the Vaal. Mabbutt (1952) reached the same conclusion for the Lower Terrace deposits of the middle Ugab valley.

In Zambia, Clark (1950) has recorded the finding of elephant remains (Palaeoloxodon darti Cooke) in calcrete associated with Earlier Stone Age tools.

Mabbutt (1957b) has recorded that heavily rolled Chelles-Acheul tools have been found in the calcified crusts of terraces of the Olifants river in the South West Cape.

Bond (1963) has mentioned the existence of kankar encrusted Acheulian artefacts at Lochard, north of Bulawayo and that none of the later artefacts were so encrusted. He considered that this period of calcification was fairly well fixed in time and gave a table suggesting general calcification between Later Acheulian and Sangoan times in Matabeleland. Bond also compiled a table from data in Wayland (1954) showing calcretes in Botswana falling between Earlier Stone Age and Fauresmith-Sangoan and also between Fauresmith-Sangoan and the Middle Stone age.

Some/ ...

Some of the calcretes at Elandsfontein (Profile 526) near Hopefield in the Cape may belong to this age group, as the fossils indicate a maximum age of some 50,000 - 60,000 years (Mr. R. Inskeep, 1966; personal comm.). The boulder calcretes in this profile through the calcrete ridge are certainly not forming at the present time. The artefacts at the site are "devoid of statigraphic separation" (Malan, 1962) although, according to Mabbutt (1957c), hand axes (Chelles-Acheul?) have been found in fossiliferous calcrete, while Stillbay tools have been found overlying the calcrete. It would appear that the fauna varies from Middle Pleistocene to Recent, but that "the archaeological record only shows one level with a large amount of faunal remains, associated with a South African Final Acheulian stone industry" (Singer and Wymer, 1968).

The only other site apart from the Elandsfontein one known to the author which is not alluvial is that at Doornlaagte in the Kimberley district (Profiles 504 - 505). The calcrete at this site was probably originally of the tufaceous hardpan variety associated with the edge of a pan. The associated artefacts were probably made during Middle to Later Acheulian times (Mason, 1966).

While all the calcretes of this group seem to have been nicely pigeonholed, it must be pointed out that in most cases only maximum ages have been established. The finding of a tool-bearing unconformity such as mentioned by Cooke (1946) for the Younger Gravels of the Vaal would be necessary to date the calcifications with any certainty. If the deposits were calcified by river water (as seems very likely) as it slowly dropped in the alluvium, the calcification would certainly be no younger than the maximum age of the next lower terrace. No Faresmith terrace has so far

been found in South West Africa (Korn and Martin, 1957), but these authors speak of the End-Pleistocene pluvial as being "represented by an erosion into the Chelles-Acheul Kanjeran gravels and by ubiquitous non-calcified gravels accompanying all the rivers as lower terraces". Martin (1964, personal comm.) is of the opinion that the Chelles-Acheul calcretes represent the last widespread phase of calcification experienced in South West Africa and that not much calcrete is forming there at the present time. His evidence for this are the facts that calcretes containing Middle Stone age implements have only been found in the areas of South West Africa receiving a rainfall of more than 400 mm, that the river terraces lower (younger) than Chelles-Acheul are not calcified and that calcrete is not found in Recent incisions.

5.4.4 Calcrete associated with Middle Stone Age artefacts

Of all the calcretes that can be dated, those associated with Middle Stone Age tools are probably the most common. Van Riet Lowe (1952) has remarked "It is a noteworthy fact that the subsoil throughout the greater part of South Africa - indeed throughout much of the sub-continent from the Equator to the Cape - frequently includes bands of pebbles and grits, ferricretes and calcretes, either singly or jointly which when they contain stone implements as they often do, show that these old, fossil land-surface were occupied, more often than not, by men of the Middle Stone Age when such cultures as the Pietersburg flourished in the Transvaal, the Stillbay in the Cape and the Natal Sangoan in Natal". He also stated that with a few exceptions the the associated fauna belong to living species.

In the Vaal river deposits, calcretes including Middle Stone Age tools occur overlying deposits characterised by

Fauresmith III implements (Van Riet Lowe, 1952). As these deposits outcrop in places they are probably fossil, but unfortunately there is as yet no evidence to show just when calcification actually took place. Van Riet Lowe has merely placed these calcretes in the Middle Stone Age.

In Natal, Davies (1951) has recorded the presence of a Chelles-Acheul handaxe and Fauresmith choppers in calcrete at Izwelihle in Tongoland and Middle Stone Age tools in mixed calcrete and ferricrete in a donga north of Nqutu.

Clark (1959) made mention of the finding of a skeleton associated with Later Middle Stone Age tools in calcrete at Tuinplaats north of Pretoria. No further details were given of the occurrence, but the skeleton is in the Transvaal Museum in Pretoria (Dr. C.K. Brain, 1968; personal comm.)

Clark (1950) placed the calcification in the Zambezi valley at the top of the Middle Stone Age, equivalent in age to the Rhodesian Stillbay culture, and Bond (1963) still accepts that calcification took place between Middle Stone Age and Magosian times in this area.

Fock (1968) has recently described a site at Rooidam west of Kimberley in which Fauresmith implements (apparently equivalent to Mason's (1962) Later Acheulian (?) are overlain by some eight feet of calcrete. This of course only means that the calcrete is younger than the implements. Rooidam is placed in this calcrete group merely because it is the most likely age of the calcrete.

At Kalkbank near Pietersburg (Profiles 512 - 516, 518), three modified tufaceous hardpan calcrete layers (Plate 5.11) occur overlying fossils and stone tools said to be Upper Pleistocene and Middle or Late Pietersburg respectively (Mason, 1962). Radiocarbon dating of the top and bottom of the calcretes yielded ages of 150 A.D. and 14,650 years B.C.

respectively/ ...

respectively (Samples 1961 and 1962B).

At Swartklip and Melkboschstrand near Cape Town, Late Pleistocene (Hendey, 1969) mammalian fossils occur in and below calcrete hardpans (Profiles 523 - 525). The fossils indicate maximum ages of about 30,000 years for the calcretes (Professor R. Inskeep, 1966; personal comm.). These calcretes are definitely fossil and not forming today.

Middle Stone Age implements have also been recorded from aeolianite (dunerock) at Isipingo Beach near Durban by Beater (1967) and at Bonza Bay near East London by Mountain (1945, in Beater) and at Nahoon near East London by Mountain (1966). The author has only observed aeolianite at one spot (Swartklip) where it was overlain by calcrete, but suggests that it may owe its origin to the same processes as do calcretes and it is hence included here for purposes of age considerations only. Pedogenic processes have certainly formed pedogenic calcretes in the aeolianite at Swartklip.

Most of the diatomaceous pan limestones (tufaceous hardpan calcretes) described by Kent and Rogers (1947) contain fossil mollusc shells as well as diatoms, but these authors concluded that neither the diatoms nor the molluscs were of any use for dating the deposits. Most were extant species and served only to show that geologically the deposits were quite young. Several deposits, however, were underlain by Middle Stone Age implements, often in a rolled condition. They therefore concluded that, with a few exceptions, the diatomaceous deposits in general were formed during the Third Wet Phase of the Pleistocene (Middle Stone Age). While this is probably true of the diatoms themselves, the author suggests, however, that the tufaceous hardpans themselves were formed at the end of the Middle Stone Age when conditions were probably becoming drier, or even in the Second Intermediate.

In/ ...

In South West Africa, Korn and Martin (1957) have recorded Fauresmith implements weathering out of a sandy (spring?) tufa in the Brandberg and Late Middle Stone Age implements weathering out of a spring tufa on the farm Doornkom in the Otjiwarongo district. Another similar deposit was stated to occur in the Maltahöhe district. Neither of the latter two springs are active today. The author has visited the Doornkom deposit and this certainly is a spring tufa and is readily distinguished from even a tufaceous hardpan calcrete.

In the Runtu area of South West Africa, about 5 miles from Runtu on the old road to Grootfontein along the omuramba, the author discovered a tufaceous hardpan calcrete overlying a rich deposit of silcrete flakes (Plate 1.13). A few of the artefacts were shown to Miss. H.R. MacCalman, who considered them provisionally to be late Middle Stone Age/Second Intermediate in age. Table 5.1 thus indicates an age of later than 12 - 15,000 years B.C. for the calcrete, while the radiocarbon results yielded 10,990 and 12,150 years B.C. for the bottom and top of the hardpan respectively.

Korn and Martin (1957) suggested that the Kalahari dunes assumed their present shapes in latest MSA times (Second Intermediate?). If this is indeed the case it probably brought calcrete formation in these areas to a stop about 12,000 B.C.

At Sambio, also on the Okavango river, calcretes were seen underlying ferricrete or ferruginished alluvial gravel which itself underlay red sand of Kalahari type which was probably partly hillwash (Profiles 358 - 360). Artefacts of all ages from MSA to Wilton occurred all mixed up (Miss H.R. MacCalman, 1965; personal comm.) in the ferricrete and sand. Father Hartman (1965, personal comm.) said,

however/ ...

however, that the Wilton tools occured chiefly in the red sand. The calcrete at this site appeared to grade both laterally and with depth into silcrete.

Another site in South West Africa possessing artefacts in calcrete is that at Neuhof-Kowas, some 70 miles south-east of Windhoek. Implements considered by Mr. A. Viereck (1965, personal comm.) to belong to the later Middle Stone Age were seen to occur embedded in the surface of an outcropping tufaceous hardpan. Radiocarbon dating of this calcrete (Plate 5.10) yielded an age of 16,550 years B.C.

Radiocarbon dating of three calcretes in Profile 161 not associated with artefacts at Ondangua in Ovamboland yielded ages of 13,950, 15,450, and 22,500 years B.C. Another date near Operet (Sample 1658) yielded a date older than 32,500 B.C.

In conclusion then, it can be said once again, that while in many cases only maximum ages have been established, the fact that these calcretes often outcrop and that most of those dated yielded finite C^{14} ages in the Middle Stone Age/Second Intermediate category does seem to indicate a widespread phase of calcification in early Second Intermediate times. The Kalkbank site is a special case of three hardpans and it seems that the top one has only stopped forming some 1,800 years ago. The bottom of the bottom hardpan, however, gave an age consistent with the underlying artefacts.

As only one site (Neuhof-Kowas) was on the dry side of the 400 mm zone in South West Africa, caution must be used in concluding that this phase of calcification affected all of South West Africa.

5.4.5 Later Stone Age to present dry calcretes

It is difficult to decide which calcretes are forming at the present time. The theory of calcrete formation

predicts/ ...

predicts that the nodules and nodular calcretes in the ares shown on the two calcrete maps (Maps 2 and 4) as possessing only nodules should be forming at the present time. This prediction could be verified by C^{14} dating of such nodules, their analysis for the presence of Strontium^{90} and the application of the other criteria suggested in Section 5.3.7. A C^{14} age of less than about one thousand years B.P. should suffice, depending on the initial C^{14} content. In the absence of information concerning the penetration of Sr^{90} in the soil, the absence of Sr^{90} could not be taken as evidence that they were not forming, but its presence would certainly indicate the contrary.

Korn and Martin (1937) and Martin (1964, personal comm.) were of the opinion that very little calcrete is forming at the present in the drier parts of South West Africa and that the last widespread calcification took place in Middle Pleistocene times. Evidence for this is that calcretes containing Middle Stone Age artefacts have only been found in the areas of South West Africa receiving a rainfall in excess of 400 mm per annum, that the river terraces younger than Chelles-Acheul are not calcified and that calcretes are not found in Recent incisions. Although the Neuhof-Kowas site is now known to possess Middle Stone Age calcretes, at least in their surface layer, after fieldwork in South West Africa and the Republic the author not only agrees with Korn and Martin, but suggests that most of the calcretes in Southern Africa as a whole receiving less than about 550 mm (22 inches) of rainfall are fossil. This conclusion is based largely on the fact that most hardpan calcretes not only outcrop, but that they outcrop right up to the border of the nodule zone (about the 550 mm isohyet), apparently without any regional honeycomb transition zone. As the 22" isohyet coincides quite well with the hardpan/nodule border, where then are hardpans and honeycomb types forming? The date of 150 years A.D.

for/ ...

for the top 1"-2" (Sample 1961) of the upper hardpan layer at Kalkbank suggests that it may still be forming at the present time. The theory of calcrete formation requires that an actively forming calcrete be in a calcareous matrix. This means that the honeycomb calcrete in Profile 483 (Plate 1.10) cannot be forming since there is no soil in the voids. In addition, it outcrops. The honeycomb calcrete in Profile 589 (Plate 4.7), however, does possess a calcareous soil matrix in its voids and could well be still forming. The nodular calcrete from 5'0" to 5'6" in Profile 26 is probably a young honeycomb type and is also probably still forming. One or two other honeycomb calcretes in the Operet area in South West Africa were also seen to fulfil the conditions of a calcareous matrix and not outcropping. It will be noticed that these are in the marginal rainfall zone for hardpan formation. Boulder calcretes in a non-calcareous red sand matrix (such as Plate 1.19) which are plainly being weathered away also exist in the same area, however, so that it is unlikely that any lower limiting value of rainfall can be arrived at.

Occasional hardpan calcretes containing Later Stone Age implements have been found. One such site is that at Langverwacht 53 in the Schweizer Reneke district described by Kent and Rogers (1947). The vlei limestone (tufaceous hardpan) at this site has yielded Later Stone Age perforated stones and bolas as well as the usual mollusc and diatom remains. Teeth, claws and bones of large predators and buck were also found in tunnels in the limestone. The calcreted top of the Basal Older Gravels of the Vaal and Windsorton was also seen to include implements said by Dr. R. Mason (1965, personal comm.) to belong to the Later Stone Age. Mason (1967) has since recorded artefacts possibly of both Middle and Later Stone Age date from the upper 12 inches of the calcrete at Holpan near Windsorton. This calcrete was mostly of the hardpan variety, but was being

weathered/ ...

weathered towards the boulder stage and is probably not forming at the present time.

Bonds's (1963) table for Botswana shows calcrete nodules associated with black soils of Later Stone Age age and another table shows a sandy calcareous alluvium of Wilton age in the Victoria Falls area. This latter must be the same as the calcareous alluvium in Clark's (1950) table shownas being equivalent in age to the Lower Northern Rhodesian Wilton and the First Post-Pluvial Wet Phase.

In general it can be said that any calcrete up to and including the honeycomb stage could still be forming if it was in a strongly calcareous matrix. The author is unaware of any easy method to determine whether a hardpan is still forming. This problem is dealt with in Section 5.3.7. It should be noted that it is possible to have calcrete which is being weathered away and calcrete which is still forming in the same profile. A good example of this is Profile 599 (Plate 5.12) wherein the boulder calcrete near the top of the profile is being weathered away by downward leaching of rainwater. The carbonate leached out is undoubtedly being incorporated into one or more of the less developed types of calcrete below. Only the soil below the calcrete boulders was slightly calcareous. All the other underlying soil layers were also found to be calcareous. This indicates the danger of applying the Law of Superposition of Strata to calcretes.

To summarise, it would appear that the forms of calcification found in the general area receiving more than 500 - 550 mm (20 - 22 inches) of rainfall (chiefly nodular calcrete, nodules, soluans and calcareous soils) are probably forming at the present time. Calcretes possessing a strongly calcareous or calcified soil matrix in the area receiving less than 500 - 550 mm may still be forming at the present time provided they do not outcrop. There is

some/ ...



PLATE 5.12

Boulder calcrete being leached downwards to form new calcrete below. This is one case where the Law of Superposition of Strata does not apply. Profile 599, near Pienaarsrivier.

some evidence for a short phase of calcification which incorporated Later Stone Age artefacts into the surface of deposits like the Basal Older Gravels of the Vaal. It is actually probably dangerous to call this a "phase" at this stage on the basis of only two known occurrences. They could well be localised features.

5.5 RATE OF CALCRETE FORMATION

Little information is available on rates of calcrete formation. In discussing the caliche of Arizona, Breazeale and Smith (1930) mentioned that many cases of brick and gravel becoming cemented together in a few years or a layer of caliche forming below a regularly watered lawn were known from Tucson. They went on to state that caliche formation may be slow or fairly rapid, and succeeded in making a caliche-like material in a few weeks by evaporation of a calcium bicarbonate solution in a sample of soil kept in an oven.

While beach and dunerock have not been included in this study, they could probably be classified as calcretes, probably usually falling into the calcified sand category, and the lithification processes involved are probably also similar. It is apparent from the work of Frankel (1968) and others that the formation of beachrock may be quite rapid. Thus Daly (1924, in Frankel) reported the formation of 75 cm of beachrock in the intertidal zone in less than two years while Frankel and Emery et al (1954, in Frankel) have reported finding World War II debris well demented into beachrock.

5.5.1 Rates of calcrete formation from radiocarbon dating

By C^{14} dating the charcoal in some desert soils in New Mexico, Gile and Hawley (1968) found that very slight carbonate accumulation was possible in about 1,100 years.

Weak/ ...

Weak but morphologically distinct carbonate horizons were found in gravelly soils 1,100 - 2,100 years old and "weak" calcic horizons with common carbonate filaments on ped faces" in soils 2,200 - 4,600 years old. The upper horizons were not completely leached of carbonate after 2,200 years. The only other published rate of calcrete formation utilising radiocarbon dating is a figure of 1,000 years per inch mentioned by Buol and Yesilsoy (1964) for what this author would probably call a pedogenic calcareous soil and not a caliche or calcrete. They regarded this figure as reasonable, but did not state how they arrived at it. Calculation of the rate required from their 38" - 44" and 60" - 66" samples yields 348 years per inch. Even allowing for the fact that the age shown for the 38" - 44" sample ($\geq$2,300 years) does not agree with their figure of 79.6% modern C^{14} (assuming 100% modern initial C^{14}) and the presence of the discontinuity at 60", this does not account for the discrepancy (see also discussion of dates A397 to A399 in Appendix G).

Although they were performed as far as possible on the relict nodules in the hardpan in each case, the radiocarbon dates of Profiles 401, 513-514 and 161 give some indication of the rate of formation. If a constant rate of formation is assumed in each case, the relict nodules in Profile 401 grew at the rate of some 166 years per inch (0.006 inches per year). Owing to the manner of taking the samples at Kalkbank, it is not possible to state the exact vertical distance apart, but assuming a figure of 36", a rate of 410 years per inch (0.0024 inches per year) is derived for the rate of accumulation of the hardpan in this profile. The hardpan formation itself certainly was not formed at a constant rate as the intercalated soil layers show, but at least the two rates are of the same order. If the soil layers do represent unconformities, or at least diastems, the calcrete itself must obviously have formed at a faster rate than one inch every 410 years. As can be seen from Plate

5.11/ ...

5.11, the total thickness of hardpan may go up to 3'6". A rate of accumulation of 350 years per inch (0.0029 inches per year) is indicated in this case.

The relict nodules in the hardpan in Profile 161 yielded an age of 13,950 years B.C., while the nodules (+10 mesh) four feet below yielded an age of 15,450 years B.C. This represents a rate of accumulation of only 31 years per inch (0.0323 inches per year). This figure may be even lower owing to the possible contamination of the relict nodules in 1598A by younger matrix. If the -10 mesh date of 22,500 years is used a rate of 177 years per inch (0.0056 inches per year) is indicated - a figure very comparable with the 166 years per inch of Profile 401 (0.006 inches per year). It would be very interesting to date the other calcrete layers in this profile, both relict nodules and matrix, as well as different size fractions of nodular calcrete at different depths.

Further rates of calcrete formation were calculated where possible from the ages and descriptions of the samples in Appendix G. In most cases the descriptions were inadequate to decide whether the development could have been continous between the sampling points and the dates have been taken at their face value. It was also seldom clear what portion of the sample (e.g. relict nodules or matrix) was being dated and the chances are that most are 'whole rock' dates. Some of the submitters may go into these questions if and when they properly publish the dates as part of their other work. Until then the following figures, though interesting, must be used with caution (Table 5.10).

It can be seen that the calculated rates vary from 31 to 1,000 years per inch (0.0323 to 0.0010 inches per year). It cannot in most cases, however, be said which rates are continuous and which are discontinuous. The 1828 A, C rate

of/ ...

TABLE 5.10 RATES OF CALCRETE FORMATION CALCULATED FROM RADIOCARBON DATES

DATE OR SAMPLE NUMBER	RATE OF FORMATION	
	YEARS PER INCH	INCHES PER YEAR
A-224,222	1,000	0.0010
A-240,239	33	0.0303
A-249,248	114	0.0088
A-262,263	>85	<0.0118
A-259,250	24?	0.0417?
A-272-273	590	0.0017
A-397,399	348	0.0029
A-692,693	500	0.0020
1828 A, C	166	0.0060
1961, 1962 B	410	0.0024
1598 A,1603 A	31	0.0323
1598 A,1603 B	177	0.0057

of 166, the 1598 A, 1603 A rate of 31 and the 1598A, 1603 B rate of 177 years per inch could well be continuous. The 1961, 1962B rate is almost certainly not continuous. It can be expected of course that the rate of calcrete formation is likely to vary widely. Plotting these few results on a histogram shows two significant peaks, at 0-200 years per inch and 300 to 600 years per inch. The significance of this is not clear and the number of results are in any event too few to draw any statistically valid conclusions. It would appear though that a rate of formation much slower than 200 years per inch (0.005 inches per year) is probably unlikely. 30-600 years per inch (0.033-0.0017 inches per year) might be the most objective estimate. Strontium^{90} and other isotopes might also be useful in determining rates of calcification.

Three/ ...

Three figures of interest for other possible calcretes or calcrete-like materials (subject to the same limitations as discussed above for calcretes) also compiled from the dates in Appendix G are 312 years per inch (0.0032 inches per year) for a "white marl" (SI-273,274), 120 years per inch (0.0083 inches per year) for a "playa carbonate" (SI-270, 271) and 23 years per inch (0.0434 inches per year) for a pedogenically altered spring tufa (A.468, 467).

The author has not carried out a literature survey of the rates of formation of spring carbonates, but it would appear that they can form at much faster rates than can calcretes, thus Gevers (1932) mentioned a rate of $CaCO_3$ accumulation of about one inch every three weeks in the borehole pipes of the Pahl spring at Windhoek and the author knows of a road culvert near Outjo which has to be cleaned out several times a year to remove the carbonate deposited by spring water flowing through it.

5.5.2 Evapotranspiration method for ages and rates of calcification

The method of Arkley (1963) described in Section 5.3.6 and the evaportranspiration method to be described are also applicable to the calculation of rates of formation. Arkley's method will not be dealt with further here, but one example of the calculation of rates of formation assuming evapotranspiration to be the major factor in calcification will be given.

It is instructive to attempt to calculate the time required for the formation of a calcrete by considering the maximum and minimum rates at which carbonate can be precipitated by evapotranspiration. This requires assumptions to be made concerning the rate of evapotranspiration from the surface in question and the amount of carbonate in solution. A third assumption is that all and only the carbon-

ate/ ...

ate in solution is precipitated. This latter assumption may not be fully valid.

Consider a vertical column of one square centimetre cross section cut out of a calcrete layer having an average dry density of γ_D gms/cc and a thickness of H cms. Let the calcrete column possess an average $CaCO_3$ content of C% by weight. The column then contains $H\gamma_D C/100$ grams of $CaCO_3$ If the original aqueous carbonate solution contained P parts per million $CaCO_3$ and was evaporated at a rate of E cms/yr, 10^{-6} EP grams of $CaCO_3$ would be precipitated per year. Combining these two expressions gives the time required T in years for the calcrete layer to form:

$$T = \frac{H\gamma_D C}{EP} \times 10^4 \text{ years}$$

The application of this formula will be illustrated by attempting to calculate the time required for the ten inch thickness of tufaceous hardpan in Profile 401 (Plate 1.13, Figure 1.10) to form. The calcium carbonate content was 71% and the approximate dry density 1.3 g/cc. X-ray diffraction showed the carbonate to be in the form of calcite (Sample 1828). At the present time the calcrete occurs under a layer of silty sand one foot thick.

Since the X-ray results showed the sole carbonate in the calcrete in question to be calcite, it is assumed that solubilities can be estimated by means of Figures 4.2 - 4.8. The solubility of calcite in CO_2 - free distilled water and distilled water in equilibrium with the atmosphere at 22.4°C is thus about 13 and 56 ppm respectively. Both these figures will be used in the calculations.

The evaporation at the nearest village (Runtu) is not known and only rainfall figures have been published (626 mm per annum) by the Weather Bureau (1963). Annual average

evaporation/ ...

evaporation from a Symons and Class A tank at Katima Mulilo on the same latitude are 202 and 250 cms respectively while the rainfall is 662 mm (Dept. of Water Affairs, unpublished data). The annual average temperature at Runtu is 22.4°C (Weather Bureau, 1968; personal comm.). Evaporation must be much the same as at Katima Molilo and will be assumed to be $\frac{662}{626}$ = 1.06 times the figures for Katima Molilo. This makes the probable annual evaporation from Symons and Class A tanks at Runtu 214 and 264 cms respectively.

The next assumption, that of the actual evaporation figures to use, is a difficult one. This involves deciding on the probable environment of formation and the position of the water table. It was seen in Figure 4.1 that the effect of the latter is quite marked. It seems likely that the carbonate in this tufaceous hardpan was deposited in among vegetation in a small, shallow, muddy, reedy pool or series of small, muddy pools. The evaporation from a Symons tank probably approximates to this condition. The Symons tank is 6 ft square, 2 ft deep and is sunk into the ground with the water level kept approximately at ground level. Three inches of the sides project above the ground and the tank is painted black. Under the present climatic conditions an evaporation of 214 cms is thus a reasonable assumption for this condition, although if the pools were much bigger than a Symons tank the evaporation in practice would have been a little less, perhaps down to some 200 cms (see Section 4.2.2 for a discussion of evaporation in general). If the water table was maintained at the soil surface the evaporation could have been anything from 77 to 100% of the free water surface evaporation in the absence of vegetation. Similarly, if it can be assumed that the water table was no deeper than 1'10" (the bottom of the hardpan) the evaporation could have been anything from 13 to 70% of the free water surface evaporation from a Symons type tank. The

latter/ ...

latter figure seems to be the most reasonable figure to use for the muddy conditions which probably prevailed at the time of initial precipitation. This would also be a more reasonable figure to choose in view of the large transpiration losses. It corresponds to a figure of 150 cms/year.

The preceding two paragraphs naturally assume that the climatic conditions prevailing at the time of formation were the same as they are today. In view of the associated artefacts and the C^{14} ages of this calcrete discussed in the previous section the time of initial formation of this calcrete must be placed in Second Intermediate times. The climate in those times must have been a deal drier than at the present if this represents the time of the last redistribution of the sands of Kalahari type in this area as implied by Korn and Martin (1957). The sand in this area (and in general in the parts of the Okavango visited north of the Gaza line), however, does not exist as distinct longitudinal (seif) dunes. Distinct longitudinal dunes only exist south of the Gaza line, their amplitude and wavelength respectively decreasing and increasing rapidly north of this line. Flint (1959) has suggested rough annual maximum rainfall figures of 10 inches (254 mm) and 16 inches (406 mm) for areas of active longitudinal dune formation and other aeolian sand movement respectively, with the proviso that these figures may be higher in areas of strong seasonal rainfall. Clark (1962b) has suggested a possible limit of 28 inches (700 mm) for general sand movement in the Lunda area of North East Angola if the rainfall was more seasonal than it is at present. A certain amount of aeolian sand movement is certainly taking place at the present time in Ovamboland during the dry season under conditions of some 500 mm or more, although seif dune formation is probably not taking place anywhere in the <u>interior</u> of South West Afria north of 20°S. It seems unlikely then that the rainfall dropped below 10 inches (254 mm) at Runtu during the Second Intermediate/ ...

mediate. Of the stations listed in unpublished data of the Dept. of Water Affairs as possessing Symons tanks and receiving more than 9 inches of rainfall (228 mm), the highest average annual Symons evaporation was recorded at Windhoek (240 cms or 94.65 inches). It seems unlikely then that the Symons evaporation went much higher than 240 cms per annum at Runtu during the Second Intermediate. The highest recorded average annual Symons evaporation in Southern Africa occurs at Keetmanshoop (300 cms or 118.5 inches) which receives 129 mm (5.1 inches) of rainfall. This latter figure of evaporation probably therefore represents something like the maximum possible figure for Runtu in those times.

Table 5.11 shows the calculated time required for the formation of the 10 inch hardpan layer in Profile 401 according to the various assumptions made.

TABEL 5.11 CALCULATED TIME REQUIRED FOR THE FORMATION OF THE HARDPAN LAYER IN PROFILE 401

ANNUAL EVAPOTRANSPIRATION (E) cms	TIME REQUIRED (T) in years if water $CaCO_3$ content (P) was		
	13 ppm	56 ppm	100 ppm
150	12,000	2,760	1,550
214	8,420	1,960	1,100
240	7,520	1,740	980
300	6,020	1,400	790

It is seen that not only are all the figures in columns three and four of Table 5.11 of the same order as the time of 1,160 years derived from carbon dates, but that one is also in remarkably good agreement with this figure. If this

figure/ ...

figure derived by dating the older parts of the hardpan can be relied upon (the lower date may be too young - see date discussion), substituting it in the equation gives required concentrations of $CaCO_3$ in the original water of 84 and 67 ppm for evapotranspirations of 240 and 300 cms respectively. These are both quite reasonable figures, as even the Vaal river water at Warrenton contains up to 100 ppm $CaCO_3$ (Table 4.5). According to Figure 4.2, additions of 300 and 1,060 ppm NaCl would be required to raise the solubility of $CaCO_3$ from 56 to 67 and 84 ppm respectively. 1,060 ppm is only 0.1%, still quite suitable as drinking water for animals, and probably quite compatible with the salinity of shallow drinking holes in the semiarid environment. Decomposing vegetation in the pools may also have contributed sufficient CO_2 to raise the solubility above 56 ppm.

It is apparent that the times required on the assumption that the water only contained 13 ppm $CaCO_3$ (column two) are unreasonably large in comparison with the C^{14} figures. This indicates that the water must have contained more than 13 ppm $CaCO_3$ (extremely likely, in any event), and that, unless the water was extremely salty, CO_2 - loss must have played a part in precipitation, whether by pure inorganic means (heat or negative pore pressure) or by organic means (algae, plants and possibly bacteria) remains unknown. A study of the microfossils present should show whether they lived in saline or fresh water and C^{13} and O^{18} measurements would yield valuable information on the environment of deposition.

The following conclusions can be derived from this study:

1. On the basis of quite reasonable assumptions, a figure for the time required to deposit enough carbonate to form the hardpan in Profile 401 was calculated which

agreed/ ...

agreed well with a figure derived from carbon dates.

2. The original solution from which the carbonate was precipitated had a $CaCO_3$ content of at least 67 ppm. This indicates that either CO_2 - loss and/or saline conditions prevailed at the time of initial precipitation. It is assumed for simplicity that carbonate was only precipitated from true solution and that all carbonate in solution was precipitated.

3. If a climate like the present is assumed (using a Symons evaporation of 214 cms) the calcrete could have been formed in 1,160 years from a solution containing 95 ppm $CaCO_3$ - a perfectly plausible figure.

4. While there are too many imponderables for this method to yield a unique result, it may well yield some very interesting information if applied to other calcrete profiles which have been carbon dated. It would also be interesting to apply it to the thick calcified gravel deposits of the Vaal.

It should be noted that the picture of tufaceous calcrete hardpan formation described in this section has been somewhat oversimplified. In all probability the original carbonate was deposited in much the manner assumed, but was later reworked by pedogenesis into its present form. The fact that the time required for precipitation of the amount of carbonate present calculated by the two methods agrees so well may merely mean that no carbonate has been lost from the system. It is plain that many questions remain as yet unanswered. The lower C^{14} date is also suspect.

5.6 COMPARISON WITH AGES OF CALCRETES OUTSIDE SOUTHERN AFRICA

No attempt has been made to make a full comparison of the ages of Southern African calcretes with those elsewhere,

but/ ...

but a few observations were made which are of interest.

The Kalahari Limestone is probably equivalent in age to Bretz and Horberg's (1949) Ogallala caprock of New Mexico and Firman's (1967) Boston Bay and other silcretes in Australia. It may also possibbly be equivalent to Price's (1933) Upper Reynosa caliche of Texas and Durand's (1963) Villafranchian 'calcaires pulverulents' (powder calcretes) in Algeria, though both of these are given as Lower Pleistocene rather than Pliocene. Much depends of course on where the Pliocene/Pleistocene boundary is drawn. The Southern African calcretes of First Intermediate age could be equivalent to Firman's Ripon calcrete and/or "calcrete in Bakara soil" and Durand's 'croutes zonaires' (stratified or zonal crusts or hardpans). Calcretes equivalent in age to Firman's upper Lower Pleistocene "soil horizons of nodular carbonate accumulation" and Durand's 'nodules farineux' (floury nodules) do not seem to have been recognised in Southern Africa.

Second Intermediate calcretes could be equivalent to Firman's "B horizons of soft nodular and platy calcrete" and some of Durand's 'encroutements calcaires' (tufaceous hardpans (?) or tufas).

Durand implies calcification in Algeria as being continuous from Tyrrhenian II (Würm) times to the present day, while Firman shows "soft ropey and columnar masses of calcrete in about the Middle of the Recent period (apparently meant to be fossil).

This section is largely speculative and there are exceptions which cannot be fitted in, but it does indicate that at least some calcretes can be correlated with others elsewhere, particularly in Australia. When more reliable Pleistocene dates become available it would be interesting to attempt a more serious correlation in view of the possible palaeoclimatic implications.

5.7/ ...

but a few observations were made which are of interest.

The Kalahari Limestone is probably equivalent in age to Bretz and Horberg's (1949) Ogallala caprock of New Mexico and Firman's (1967) Boston Bay and other silcretes in Australia. It may also possibbly be equivalent to Price's (1933) Upper Reynosa caliche of Texas and Durand's (1963) Villafranchian 'calcaires pulverulents' (powder calcretes) in Algeria, though both of these are given as Lower Pleistocene rather than Pliocene. Much depends of course on where the Pliocene/Pleistocene boundary is drawn. The Southern African calcretes of First Intermediate age could be equivalent to Firman's Ripon calcrete and/or "calcrete in Bakara soil" and Durand's 'croutes zonaires' (stratified or zonal crusts or hardpans). Calcretes equivalent in age to Firman's upper Lower Pleistocene "soil horizons of nodular carbonate accumulation" and Durand's 'nodules farineux' (floury nodules) do not seem to have been recognised in Southern Africa.

Second Intermediate calcretes could be equivalent to Firman's "B horizons of soft nodular and platy calcrete" and some of Durand's 'encroutements calcaires' (tufaceous hardpans (?) or tufas).

Durand implies calcification in Algeria as being continuous from Tyrrhenian II (Würm) times to the present day, while Firman shows "soft ropey and columnar masses of calcrete in about the Middle of the Recent period (apparently meant to be fossil).

This section is largely speculative and there are exceptions which cannot be fitted in, but it does indicate that at least some calcretes can be correlated with others elsewhere, particularly in Australia. When more reliable Pleistocene dates become available it would be interesting to attempt a more serious correlation in view of the possible palaeoclimatic implications.

5.7/ ...

5.7 CONCLUSIONS

1) The age of any calcrete is defined as the age of the first onset of calcification which converted it from the stage of development immediately below to the stage to which it belongs at present. The usefulness or otherwise of this definition depends on the problem in hand.

2) By definition, fossils and artefacts found in a calcrete can only provide the age of the host material and not the age of the calcification. Maximum ages alone are thus assured.

3) The mollusc and diatom remains common in some calcretes are of little use in determining the ages of the calcretes except within wide limits. They do, however, indicate a maximum age for the Kalahari Limestone and provide useful information on the environment of deposition of the original material. Mammal remains have been rather more useful.

4) The Thecamoebians Phryganella and possibly Hoogenraadia have been identified from a calcrete at Lichtenburg. These are the only other fossils besides diatoms, sponge spicules, phytoliths, molluscs and occasional mammals to have so far been identified in calcretes.

5) Probable algal structures have been identified in a calcrete (?) and definite algal structures in a silcrete.

6) No fossil pollens have so far been found in calcretes in Southern Africa, though they have been reported from elsewhere.

7) Some calcretes may be dated roughly by means of land surfaces, while their topographic settings can be used to arrive at relative ages.

8) It is considered desirable to restrict the terms 'Kalahari Limestone' and 'Kalahari Sands' to those deposits of presumed Pliocene age. The start of formation of the former may be the best definition of the Pliocene/Pleistocene boundary in Southern Africa, however.

9) Caution should be employed in regarding calcretes as stratigraphic horizons.

10) The first radiocarbon ages of some South African calcretes have yielded very satisfactory results.

11) While further work is necessary, the indications are that pedogenic calcretes can be reliably dated by the radiocarbon method. The proving of calcretes as dateable material is of extreme importance to the understanding of the later Quaternary in Southern Africa.

12) Part of the theory of calcrete development is supported by the C^{14} dates.

13) Several other radiometric methods, particularly the ionium growth, helium accumulation, thermoluminescence, and potassium-argon methods should be applicable to many calcretes, even to those which do not contain glauconite.

14) A simple evapotranspiration method has been developed which gives ages and rates of formation of the same order as that determined by other methods.

15) Rates of calcrete formation between 30 and 1,000 years per inch (0.001-0.033 inches per year) have been calculated from C^{14} dates. About 200 years per inch (0.005 inches per year) is possibly the most common rate. The faster rates are likely to be the correct ones.

16) If they are classified as old as possible, calcretes in Southern Africa can be classified into the following

age/ ...

age groups: Pre-Pliocene, Pliocene, First Intermediate, Second Intermediate and Later Stone Age to present day. While further work is desirable, there thus appears to be evidence for fairly widespread phases of calcification over a large part of Southern Africa.

17) Calcretes of similar age to some of the Southern African calcretes also occur elsewhere in the world.

18) The evidence of age, distribution and theory suggests that ideal conditions for calcrete formation will be during waning pluvials as originally suggested by Rimann (1914) and Korn and Martin(1937), and not during interpluvials as normally suggested. A particular climatic sequence may thus be required for widespread calcrete formation, not just a dry climate.

19) Many of the calcretes occurring in the general area of Southern Africa receiving less than about 500 mm of rainfall are fossil and are not forming at the present time. This does not imply that there are no active calcretes in this area or that calcification is not proceeding elsewhere in the same profile as the fossil calcretes.

20) The development of criteria for distinguishing actively forming from fossil calcretes is of great importance.

5.8 SCOPE FOR FURTHER INVESTIGATION

1) There is probably much scope for work on the microfossil content of calcretes apart, perhaps, from diatoms. More calcretes should also be examined fro fossil pollens. A survey of all the molluscs so far recorded from calcretes would be useful.

2./ ...

2) There is much scope for the determination of the different ages of calcretes in the belt of calcrete commonly known as the Kalahari Limestone bordering the western and southern parts of the Kalahari as well as calcretes all over Southern Africa.

3) Much very valuable and essential work remains to be done on the radiocarbon dateability of calcretes. The form this could take is as follows:

a) Measurements of C^{14}/C^{12} C^{13}/C^{12}, O^{18}/O^{16} and Sr^{90}/Sr^{87} ratios should be carried out on calcrete nodules and calcareous soils in the 500-800 mm rainfall zone which appear to be forming at the present time. This should both establish the initial C^{14}/C^{12} and C^{13}/C^{12} ratios to use in dating pedogenic and non-pedogenic calcrete deposits and provide much information on their origin. Possibly suitable profiles are 531-533, 535-541; National Building Research Institute Profiles O.S.T.H.M. 3, B.R.P: 103A, 213, 81, 283, 26, 76, 249, 207, 56, 364, 371, 70 and 83 in the Orange Free State and Vereeniging; certain lower parts of Profiles 599 and 600 at Pienaarsrivier, National Institute for Road Research Profile RG3-1995 at Onderstepoort and possibly upper surface of Kalkbank calcrete in the Transvaal; in Ovamboland many of the shallow calcareous soils with scattered nodules occurring in the oshana country and the sand plain south of Ondangua at shallow depths should be suitable, as well as the nodules in the uppermost parts of profiles like 161. Pedologists may also be able to state the whereabouts of some apparently active calcareous profiles in South Africa.

b)/ ...

b) In the dating of nodular calcretes, dates should be determined on different sizes of nodules and the fines as well as the inner and outer portions of the larger nodules. This should establish rates of formation and whether whole calcrete dates can be used.

c) Dates on the relict nodules and the matrices in honeycomb and hardpan calcretes should be carried out. In cleaning the relict nodules consideration should be given to removal of the adhering matrix with acids. Steps (b) and (c) will finally prove or disprove the theory of calcrete development, provide information on rates of development and indicate which fractions should be used for dating purposes.

d) In the dating of banded and brecciated calcretes, all the different portions should be dated as they will certainly prove to be of different ages. If the brecciation and rehealing can be related to climatic changes these calcretes would prove unrivalled geological sources of information on the Quaternary. Thin sections of the indurated portions of all calcretes to be used for dating should be examined in order to assess the extent of contamination by older or younger carbonate. The contamination is often of a different carbonate and could be selectively dissolved out with acids, possibly even during the preparation of the counting gas (CO_2) in the radiocarbon laboratory.

e) Organic matter should be separated from the inorganic carbonate in calcretes and dates on both fractions compared. Professor H. Martin (private comm. via Professor R. Inskeep, 1966) has also suggested that roots encased in calcrete should

be/ ...

be dated. A site where they do occur is at the Melkboschstrand midden. In dating dispersed organic matter in calcretes it should be noted that one active soil profile in New York was found to contain organic matter varying in age from modern to 2,000 years (Olson, 1963). Olson has stated that, in general, equality of ages on different fractions of a sample argues strongly for no contamination and vice versa. This statement must plainly be applied with caution to cases involving soil organic matter. The alkali soluble organic matter (humic acids) is said to be nearly always native to the sample, but the author also doubts the validity of this statement. The ultrasonic method of McIver (1962) might be useful for the removal of organic matter from calcretes.

f) It would be interesting to date a profile through the calcified Basal Older Gravels of the Vaal at Windsorton which have Later Stone Age tools cemented into the surface. The dates should be less than 10,000 years B.C. in the upper few inches and then increase rapidly to beyond the dateable limit.

g) Dates on fossil mollusc shells in tufaceous calcretes should be compared with dates on the inorganic carbonate. In preparing shells, only the inner layers should be dated, the outer layer being removed with acid. It would appear from a cursory examination of the shell dates published in "Radiocarbon" that certain genera are more reliably dated than others. Physa and Anodonta seem to be particularly popular. Anodonta does not seem to have been recorded from Southern African calcretes, but Physa is quite common. Olson (1963) concluded that, in general, shell dates below 25,000 years

are/ ...

are much more reliable than the literature suggests, especially when acid leaching is employed. Even those over 25,000 years are often satisfactory. Tufaceous hardpans (vlei limestone, diatomaceous pan limestone, Pfannenkalktuff, Schneckenkalk) offer the most promise for C^{14} dating research due to their probably high initial C^{14} content, high organic matter content and their generally fossiliferous nature. In addition, these are usually the types associated with artefacts and mammalian fossils, so they are of the most importance in Quaternary studies.

h) Professor H. Martin (private comm. via Professor R. Inskeep, 1966) has suggested that the layer of calcareous sinter sealing a layer of charcoal dated at 3,300 years associated with the earlier (?) Smithfield (Korn and Martin, 1957) in the Phillips cave should also be dated. While this is probably not a calcrete, dates on materials like these would be invaluable in estimating initial C^{14} contents for use on the many important calcified cave deposits in this country.

i) Some sites at which C^{14} dating of calcretes would be of interest, both in order to prove the dateability of calcrete and in connection with the determination of the ages of the associated fauna or artefacts, are as follows:

Swartklip)
Melkboschstrand) and other faunal sites on the
Elandsfontein) Southwest Cape coast.

Langverwacht 53, near Schweizer Reneke

Doornlaagte, near Kimberley

Vaal river deposits with artefacts from Chelles-Acheul to Later Stone Age.

Olduvai/ ...

Olduvai Gorge (calcrete between Beds V and VI).
Olifants river deposits.
Fish, Nossob and other calcified river deposits in South West Africa.
Tuinplaats skeleton near Pretoria.
Aloes near Port Elizabeth?
Rooidam near Kinberley.
Lochard near Bulawayo.
Bultfontein 1904 on the Springbok Flats.

In addition, other interesting objects could be dated such as the minimum date of the fall of the Hoba meteorite. Stony meteorites seem to be the only type amenable to direct C^{14} dating (see Suess and Wanke, 1962), but Kohman and Goel (1963) have dated irons by means of their C^{14}/Be^{10} ratios. The Cl^{36} method also seems to be applicable to irons.

4) Application of the evapotranspiration and Arkley's (1963) leaching age methods should be made to dated calcrete profiles. This should provide information on the climate prevailing at the time of formation if reasonable estimates of the initial carbonate content of the soil water can be arrived at.

5) Other radiometric methods, particularly those mentioned in Conclusion 13, should be tried out on terrestrial carbonates in general. Of particular interest would be the dating of the Australopithecine-bearing cave deposits. No isotopic dating has been carried out so far on any of the Tertiary or Lower Pleistocene deposits of Southern Africa.

6) The calcretes at the sites listed in Section 5.4 should be more critically examined in order to arrive at minimum ages as well as the already known maximum

ages/ ...

ages afforded by the artefacts and fauna.

7) When more information is available, it may be profitable to attempt correlation with calcrete in other countries, particularly in the Southern Hemisphere.

8) When the isotopic evidence on actively forming calcretes is gathered, an attempt should be made to define the climatic and other conditions under which calcretes of various types can form.

9) Much work remains to be carried out on rates of calcification. C^{14} dating seems to be an invaluable tool in studies of this sort. Care should be taken in selecting the particular portion of calcrete to be dated if meaningful results are to be obtained. Much scope exists for the application of proper sedimentological and pedological techniques to the problems of Quaternary geology in Southern Africa.

10) The criteria suggested for distinguishing actively forming from fossil calcretes should be critically evaluated, using C^{14} and Sr^{90} for example. Further criteria should be evolved.

11) By carrying out both C^{14} and O^{18} measurements on the various carbonate portions of an old calcrete, a qualitative palaeotemperature curve can probably be constructed. Much of the reasoning of Hendy and Wilson (1968) with respect to the use of speleothems for this purpose will also apply to calcretes though owing to the likelihood of their temperature of formation being influenced by depth and vegetation, they will be less useful than speleothems.

5.9/ ...

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CHAPTER 6

PROSPECTING METHODS

6.1 INTRODUCTION

6.2 LITERATURE SURVEY

6.3 FUNDAMENTAL FACTORS CONTROLLING THE DISTRIBUTION OF CALCRETE

- 6.3.1 Climate
- 6.3.2 Topography
- 6.3.3 Parent or host material
- 6.3.4 Biological factors
- 6.3.5 Time

6.4 REGIONAL PROSPECTING

- 6.4.1 Airphoto interpretation from conventional airphotos
- 6.4.2 Interpretation from diapositives
- 6.4.3 Colour photography
- 6.4.4 Infrared photography
- 6.4.5 Infrared imagery and other remote sensors
- 6.4.6 Geophysical methods
- 6.4.7 Geochemical methods
- 6.4.8 Plant indicators
- 6.4.9 Zoological indicators
- 6.4.10 Drilling and excavation

6.5 DETAILED INVESTIGATION

- 6.5.1 Rapid probing
- 6.5.2 Overburden and calcrete colour and overburden texture

6.5.3./ ...

6.5.3 Plant indicators

6.5.4 Hand augering and pitting

6.5.5 Geophysical methods

6.5.6 Bulldozing and ripping

6.6 CONCLUSIONS

6.7 SCOPE FOR FURTHER INVESTIGATION

6.8 REFERENCES

6.1 INTRODUCTION

This chapter considers the location and proving of economically workable calcrete deposits. While wrtitten chiefly with road construction in mind, the methods outlined can also be applied to the location of calcrete for other purposes, such as cement or lime manufacture.

6.2 LITERATURE SURVEY

Apart from some work on airphoto interpretation which will be discussed under that heading, no work on the location of buried calcrete deposits for any purpose appears to have been published.

6.3 FUNDAMENTAL FACTORS CONTROLLING THE DISTRIBUTION OF CALCRETE

The first fundamental fact which the calcrete prospector must realise is that calcretes are not rocks, but soils. Their distribution is therefore not governed solely by geology, but by the five soil-forming factors of climate, topography, parent material, biological factors and time. Geology is in fact only a subfactor of parent material. The effect of the soil-forming factors on the distribution of calcrete has been discussed in detail in Sections 2.3 and 2.4 and, while this section is to some extent a recapitulation of the most important points, its main purpose is

to/ ...

to show how an understanding of the pedogenic factors can help in calcrete prospecting. Section 2.2.4 indicates how to make best use of the calcrete maps (Maps 2 and 4).

6.3.1 Climate

In Southern Africa, sufficiently well developed calcretes occurring in sufficient quantities to be economically workable for any purpose only generally occur in areas receiving a mean annual rainfall of less than about 550 mm (22 inches). As the carbonate part of calcretes is slightly soluble in water, only very weakly developed types are generally able to form in the wetter areas, while in the rare cases where further development is possible, the quantities formed are usually unworkably small. Higher temperatures, by increasing evaporation and decreasing the solubility of carbonate, increase the amount of rainfall required to achieve the same degree of leaching. In the hotter areas of Southern Africa economic calcrete deposits can occur under a maximum rainfall of about 625 mm (25 inches). Exceptions to this are very rare.

The seasonal distribution of rainfall is also important. Rainfall evenly distributed throughout the year will have a greater leaching potential than the same amount of rainfall confined mainly to a certain time of the year. Winter rainfall will also possess a greater leaching potential than summer rainfall.

Weinert (1964) has shown that the boundary between predominant disintegration and predominant decomposition agrees well with the N = 5 line. Disintegration is the dominant form of weathering where N is greater than 5 and decomposition the dominant form where N is less than 5. Since the plasticity of a calcrete depends to a large extent on the plasticity of the host material, this means for example that the PI of a calcrete in the residual Stormberg basalt soils of the Springbok Flats (N about 3?) will be

higher/ ...

higher than one at the same stage of development in the residual Stormberg basalt soils of the area east of Mariental (N about 30).

There is some tendency for the depth at which calcrete occurs to increase with rainfall (Figure 2.3), yielding some idea of the prospecting depth required under any given rainfall.

Other factors which affect the macro or micro climate close to the ground will also affect the distribution of calcrete. One rather interesting factor is the hydrogen sulphide gas blown inland along the coast of parts of South West Africa which has converted the coastal calcretes to gypsum or gypseous calcretes, a most undesirable feature as far as cement or lime stabilization is concerned.

6.3.2 Topography

Calcretes generally form on flattish or gently sloping land where moisture is able to linger, weather the parent material, release calcium and magnesium and, eventually, to be evaporated in situ. The rate of runoff and mechanical erosion on steep slopes is too rapid for much calcrete to form. Calcretes forming cliffs like the Weissrand or high ground are invariably fossil, and are themselves being eroded. In this connection, calcretes should not be confused with cliff limestone (dripkalk).

Calcretes may often be found by a study of their relation to the topography, for example hardpan calcretes often occur on rises or cementing alluvial or pan terraces, and the calcretes with the hardest aggregate, best grading and lowest PI will normally be found on rises or slopes. Only a rather plastic calcareous or calcified soil with scattered nodules will usually be found in pan floors, though the omirimbis in the northern part of South West Africa often possess a better developed calcrete of the nodular type in their beds. This is possibly due to their

containing/ ...

containing water for shorter periods and possessing more sandy beds than do pans - probably more favourable conditions for rapid calcrete development. They may also be older than for example the present-day floor of the Etosha Pan which is hardly even calcareous, and which is saturated with saline water a few feet below the surface, even in winter.

According to the theory of calcrete development, calcrete grows from a calcareous soil via a calcified soil or nodular and honeycomb stage to a hardpan, and finally weathers to a boulder calcrete. This sequence, not always fully represented of course, is often found in omuramba and pan sections, and an idealised section is shown in Figure 6.1. The decrease of PI away from the centre is due to three factors: in a circular depression such as a pan the fine clay tends to get washed out of the banks into the centre and, in addition, as the calcrete gets older the fines seem to become selectively excluded from the carbonate fraction. A third reason is that the more active clay minerals tend to be broken down to less active types by virtue of their favourable situation in relation to leaching and oxidation.

An important point to notice is that as one prospects further up the bank on the first terrace, the overburden thickness over the tufaceous hardpan increases to a point which may be beyond the range of a short hand auger or the probing device (Figure 6.2), or the calcrete may even peter out altogether. One should not then be lured into thinking that this is the end of the calcrete deposit, as the hardest material is yet to come, and a hardpan or boulder calcrete of crusher run quality may occur on the next terrace under a shallow overburden.

Figure 6.1 also applies in a more general way to the types of calcretes occurring on land surfaces (Section 6.3.5) of increasing age, though these erosion cycles move relatively slowly, so that it is possible to have several

different/

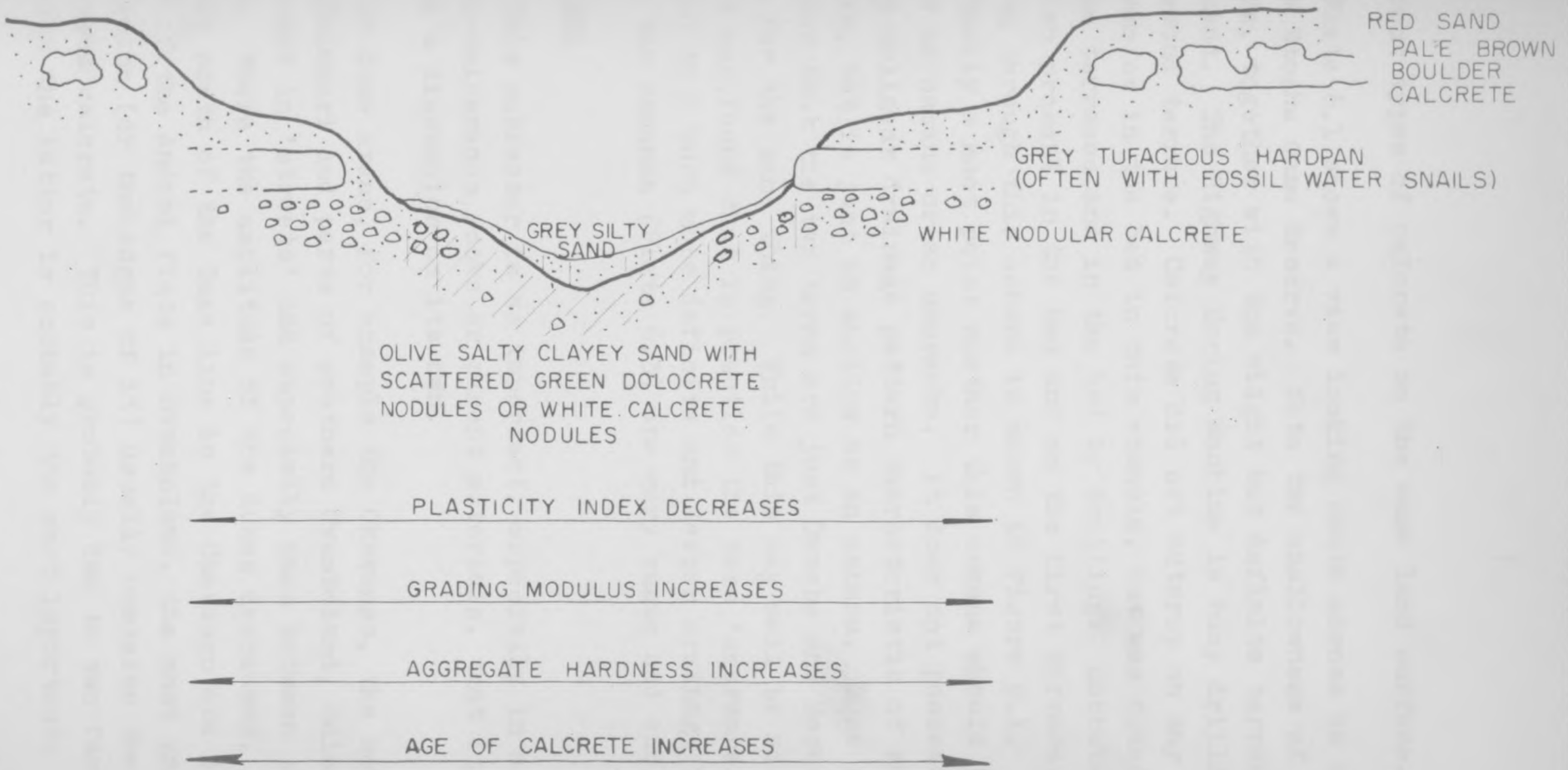


FIGURE 6·1

Idealised section across pan or omuramba

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different types of calcrete on the same land surface.

Plate 6.1 shows a view looking south across on oshana in the Etosha Game Reserve. Note the shallowness of the feature, together with the slight but definite terrace development. The Highway Boring Machine is busy drilling on the second terrace. Calcrete did not outcrop on any of the terraces or in the bed in this example, but was found on all the terraces and in the bed by drilling. Gabbabos was also present in the bed and on the first terrace. A section through this oshana is shown in Figure 5.1. It is actually a moot point whether this oshana should be called an oshana or an omuramba. It does not possess the almost colinear drainage pattern characteristic of so many oshanas, but is just as shallow as an oshana. Some persons consider that the two terms are just Ovambo and Herero words for the same thing. While this may well be so, the author has found that in practice the term 'omuramba' is applied to a much more definite and deeper drainage line, while the oshanas (Plate 6.3) are very vague and shallow.

Drainage

This subfactor is so important, especially in areas of non-calcareous rocks or parent materials, that it merits a discussion on its own.

In dune areas, for example the Okavango, the south west Kalahari and parts of southern Ovamboland, calcretes are found in 'streets' and especially pans between the dunes. Where the amplitude of the dunes decreases, for example north of the Gaza line in the Okavango and just north of the Andoni flats in Ovamboland, the most pronounced depression (or the edges of it) usually contains the best developed calcrete. This is probably due to two factors, of which the latter is probably the most important:

a) the decreased depth to a calcareous source rock,

b)/ ...



PLATE 6.1

View across an oshana or shallow omuramba in the Etosha Game Reserve north of Operet 312 on the then proposed Trunk Route 1/10. Note the slight but definite terrace development and lack of outcrops. Calcrete was, however, found to occur on all the terraces as well as in the bed.

b) the increased effective rainfall and inflow which allow deeper penetration and solution of carbonate, which may also contribute carbonate, and which brings in more fines, assisting in the production of a relatively impermeable horizon and a perched water table.

Contributory factors which speed calcrete growth in these depressions are probably the denser growth of shallow-rooted vegetation types resulting in increased moisture loss by transpiration and the consequent increase in CO_2 content of the soil atmosphere, resulting in an increase in the decomposition rate and in the greater solubility of carbonate. Some good aerial photographs illustrating these depressions have been given by Caiger (1964). Calcretes developed or apparently developed over granite for example were found to be usually associated with:

a) drainage lines (e.g. Profiles 554 and 559 in Swaziland, 476 near Aus, and 454 west of Usakos, and the area north of Beit Bridge in Rhodesia) or

b) basic intrusions (west of Usakos, e.g. Profile 454A) or

c) limestone outcrops upslope (east of Usakos, e.g. Profiles 456 and 457) or

d) mechanically eluviated form an older calcrete plateau (Profile 475) - probably a remnant of one of the Post African cycles (Oligocene and Miocene) of Partridge (1967) - still existing in parts of the area (between Luderitz and Aus).

The drainage factor probably entered into all of these cases to some extent; even the profiles developed over basic intrusions were generally in depressions. The importance of drainage is also made apparent by a reading of Van der Merwe's (1962) chapter on Desert Soils. Drainage is not only important in the arid and semiarid zone, however,

even/ ...

even in the subhumid zone it is the bottomland soils that are the most likely to be calcareous (see for example Figure 2.1 and Profiles 554 and 559).

In general, topography plays a large part in calcrete formation through its assistance in creating shallow perched or permanent water tables. In any area, the banks and terraces of all drainage features, whether they be pans, omurambas, oshanas or rivers are good places to look for calcrete. In some cases calcrete may only occur on one side of the drainage feature. Topography may also influence calcrete formation through its influence on the climate. Calcrete may only be found on the rain shadowed side of a mountain, for example.

6.3.3 Parent or host material

While calcretes form by growing in an existing soil, this soil may or may not provide the carbonate. It may in fact merely act as a host material in which the carbonate is precipitated. 'Host material' is therefore a better general term than 'parent material' in the case of calcretes. 'Parent Material' is really the special case of a host material which has also provided the carbonate.

Geology

The solid geology only determines whether or not calcrete will form when the soil profile is residual or the thickness of transported cover is reasonably thin. In such profiles, calcrete formation is likely over calcareous rocks such as limestone, dolomite, calcareous shales and mudstones or the more basic rocks like dolerite and basalt which release calcium and magnesium on weathering. Calcrete is not likely over rocks like sandstone and granite if the drainage is unfavourable, even in a favourable climate. Calcretes also sometimes mark faults and dykes, probably in many cases largely due to shallow water table conditions.

Soil/ ...

Soil cover

A depth of soil cover within certain limits seems necessary for significant calcrete formation. Van der Merwe (1962) stated that the Kalahari Limestone forms only when the sand is less than about 15 ft thick, presumably due to the rainfall being insufficient to penetrate to the underlying rock and dissolve the carbonate. He also stated that at this depth the "incipient calcium carbonate layer is generally powdery and soft" and went on to state that "on the other hand where the rainfall is low or the sand cover is not deep enough to retain sufficient water and prevent it from being evaporated, the calcareous horizon also does not form". The lack of soil cover is undoubtedly one of the reasons for the scarcity of calcrete over the Stormberg Lavas near Mariental. The general observations of the author are in agreement with those of Van der Merwe, but it is not always possible to decide by field inspection alone whether a calcrete could be forming at the present time.

Another good example of the influence of soil cover is the case of the new Grootfontein-Tsumkwe road. Abundant calcrete occurs at each end of the road, but very little was found between about Kanovlei and a point close to Tsumkwe. What little was found was associated with pans and drainage lines. A study of an isopach map of the Kalahari Beds kindly made available by Mr. D.H. Fulkerson of Etosha Petroleum seems to provide the answer, however. Between a point about 25 miles east of Grootfontein and Kanovlei, the pre-Kalahari floor drops rapidly, reaching its maximum depth of 1,000 ft just before Kanovlei. (This may be evidence for the extension of the Waterberg fault towards Andara). Between Kanovlei and Tsumkwe the floor slopes evenly upwards and at Tsumkwe Stormberg basalt comes to within about 40 ft of the surface, with much thick calcrete to depths of at least 12 ft. This seems to explain the

abundance/ ...

abundance of calcrete in the Tsumkwe area, but does not explain the presence of a fair amount of calcrete between Kanovlei and the point towards Grootfontein at which the basalt once again comes near the surface. The presence of suitable drainage conditions and possibly the presence of a near-surface calcareous horizon in the Kalahari Beds is probably the reason for this. The 40 ft of sand and calcrete at Tsumkwe presumably also took just as long to accumulate as the 1,000 ft of sediments east of Kanovlei. It is likely that a rapid accumulation of say 15 ft of sand would prevent further calcrete formation, while a slower accumulation of the same amount over a weathered basic igneous rock would provide an ideal environment for calcrete formation. The depth of sand required to prevent calcrete formation by rain infiltration to a calcareous horizon and subsequent rise of the carbonate-bearing solutions could be calculated if the effective rainfall, evapotranspiration and field capacity of the soil profile were known.

One effect of the texture of the soil cover or the parent material is through its effect on the leaching rate. The more impermeable the soil, the more humid the climate under which the calcrete can form or survive and the shallower the calcrete horizon is likely to be. Under the same climate the rate of calcrete development would be slower in a less permeable soil than in a more permeable one.

A second effect of texture is on the actual type of calcrete formed and its plasticity. Calcretes developed in clays will possess high plasticity, while those developed in sands will be non-plastic.

Applying these principles to Map 4, it can be predicted that workable calcrete deposits will be extremely rare in the areas shown under the soil legend as rock and rock debris, and rare in all weakly developed and sub desert

soils/ ...

soils except those on calcareous crusts (the calcareous crusts are actually hardpan calcretes). Heavy clay soils like vertisols would be the best types to search for calcrete in the subhumid zone. These calcretes are likely to be rather plastic.

6.3.4 Biological factors

Vegetation plays a very important role in calcrete formation through transpiration but the best use of vegetation in calcrete prospecting is through the recognition of calciphilous plaats, which will be dealt with in a separate section. The only generalised statement that can be made at this stage is that calcretes which are not obviously fossil tend to be associated with bush, rather than trees.

The only other biological factor thought to play a rôle in calcrete formation is termites, but again, the recognition of white termitaries on aerial photographs is the best way of using them.

6.3.5 Time

If all the other pedogenic factors remain constant, the effect of time is to increase the stage of development of the calcrete. The oldest calcretes are therefore the hardest, the strongest, and possess the best grading and lowest PI (Figure 6.1). The highest river or pan terraces are the oldest and the calcretes on the highest terraces are usually the best developed and thickest.

If one or more of the other pedogenic factors (particularly climate and drainage) change with time, a fossil calcrete may result. Since they are not forming at the present time, fossil calcretes obviously need not occur under conditions favourable for calcrete formation, and when they do not outcrop can be extremely difficult to locate. Empirically, it is, however, found that even fossil calcretes obey the climatic correlations mentioned in Section

6.3.1 and do not occur over non-calcareous rocks like the Waterberg and Table Mountain sandstone and granite unless the drainage was once favourable. A climate roughly like that of the present must have been in existence for a sufficiently long time to have leached away most of the fossil calcrete (if any) which was once present in the presently wetter areas of Southern Africa, and of course the solid geology of Southern Africa has remained essentially constant for a far greater period than the age of the oldest known calcrete.

Fossil drainage features are just as useful sources of calcrete as are present-day drainage features and are often detectable on airphotos. They usually yield well developed calcretes. The fossil drainage line calcretes at Operet (Plate 6.2) are among the best developed calcretes yet found. The best calcretes in the Ondangua area are associated with the most pronounced drainage lines and seem to represent old oshana lines (Plate 6.3). The elements of the airphoto pattern of these two occurrences are given in Table 6.1. The Operet fossil drainage line calcretes represent former courses of the Omuramba Ovambo (Mountain, 1964). Fossil drainage lines in the Sahara have been discussed briefly by Conedera (1963) and work being carried out by Etosha Petroleum (Mr. D.H. Fulkerson, 1965; personal comm.) in the Etosha Basin may show up more fossil drainage lines which may yield important sources of calcrete for road making.

Some idea of the nature of the calcretes in a particular area may be obtained from a knowledge of the age of the landsurface on which they rest or form. The Kalahari Limestone (probably late Tertiary in age) resting on the African erosion surface is extremely thick and indurated, and along the Weissrand (Profile 130) has passed the hardpan stage and is undergoing disintegration. The calcrete on the probably post African surface east of Aus (Profile 475) is

much/ ...

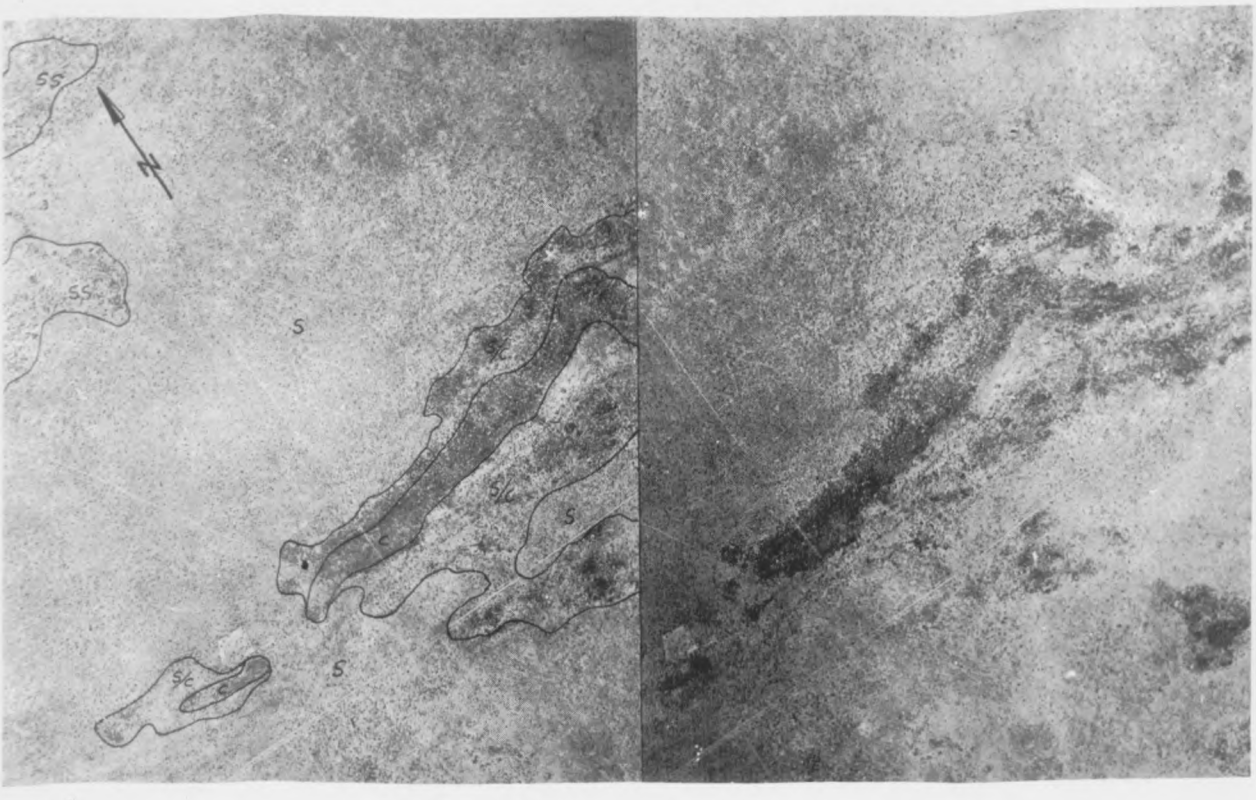


PLATE 6.2

Steropair showing an area of well developed boulder and nodular calcrete in an area of non-calcareous sand. The former probably represents a former course of the Omuramba Ovambo. Scale approximately 1:72,000. Interpretation after Mountain (1964). Airphotos No. 8325 and 8326 of Aircraft Operating Company's Job No. S.160/64 reproduced by Graphic Arts, CSIR.

TABLE 6.1 ELEMENTS OF THE PHOTOGRAPHIC PATTERN OF SOME CALCRETE DEPOSITS

AREA	SOURCE OF ELEMENTS	CALCRETE QUALITY	ELEMENTS OF FORM			ELEMENTS OF TONE AND TEXTURE			SOIL PROFILE
			TOPO-GRAPHIC	DRAINAGE	EROSIONAL	VEGETATION	LAND USE	SURFACE MATERIAL	
20 miles east of Kimberley	Brink and Williams (1964)	Fill	Gentle Slopes	Well integrated sub-parallel	Sheetwash and flattened V-section. Gulley erosion	Thornbush along gulleys	Partly cultivated	White tone; light grey in poorly drained depressions	Hillwash/ calcrete/ shale
Winters Rush - Koopmansfontein	Author, from Brink and Williams (1964)	Subbase	Flat lowlands	Very poor	-	Scattered bush	Pasture	Mottled dark and medium grey	Calcrete/ alluvium/ shale
Cape Plateau N E of Griqua-town	Author, from King (1964)	?	Plains	Swallow holes, colinear	-	Scattered bush	-	Light grey	Calcrete/ dolomite
Operet end of Operet-Andoni (Plate 6.2)	Author, from Mountain (1964)	Base, subbase	Low ridge	Itself probably a fossil drainage line	-	Dense forest	Pasture	Light grey	Calcrete and sand/ calcrete
Glen, near Bloemfontein	Author	Subbase	Plains, very gentle slopes	Poor	Vertical walled shallow gully	Scattered bush and grass	Pasture	Mottled medium and light grey	Aeolian sand/ calcrete
Operet 312	Author	Gravel road	Depressions, pans	Poor, colinear	Shallow oshanas	Scattered bush, close gabbabos and grass	Game reserve	Mottled light to medium grey and white (anthills and drainage)	Aeolian sand/ calcrete
South Edge of Andoni Flats, Etosha Game Reserve	Author	Gravel road (good)	Fossil pan terrace	-	-	Scattered gabbabos and grass	Game reserve	Medium grey	Aeolian sand/ calcrete
Andoni-Ondangua	Author	Gravel road	Pan	Nil	-	Gabbabos, grass, scattered bush	Game reserve	Mottled medium grey and white	Aeolian sand/ calcrete
Andoni Flats, Etosha Game Reserve	Author	Gravel road (poor)	Fossil pan floor	Nil	-	Grass tussocks	Game reserve	Mottled dark and medium grey	Aeolian sand/ calcrete & calcified soil
Ondangua (Plate 6.3)	Author	Subbase, base	Low rises at pinched out oshanas and inside oshanas bends	Oshanas colinear - sub-parallel	Oshanas shallow, gently sloping sides	Crops or bitterbos and scattered bush	Cultivated or pasture	Light to medium grey, white antheaps	Aeolian sand/ calcrete

TABLE 6.1 (CONTINUED)

AREA	SOURCE OF ELEMENTS	CALCRETE QUALITY	ELEMENTS OF FORM			ELEMENTS OF TONE AND TEXTURE			SOIL PROFILE
			TOPOGRAPHIC	DRAINAGE	EROSIONAL	VEGETATION	LAND USE	SURFACE MATERIAL	
Ondangua	Author	Gravel road (poor)	Oshana floors	Oshanas colinear - sub parallel	Oshana shallow, gently sloping sides	Grass tussocks	Poor grazing	Medium to dark grey	Alluvial soil/ calcified soil
Grootfontein - Runtu (south of Gaza line)	Author	Gravel road (good)	Streets between high seif dunes	Nil or colinear	Nil or shallow, gently sloping sides	Grass, scattered bush	Grazing	Light grey	Aeolian sand/ calcrete
Gaza Line (Grootfontein - Runtu)	Author	Gravel road (poor)	Street between poorly developed seif dunes	-	-	Grass, scattered bush	Game reserve	Medium grey	Aeolian sand/calcrete and calcified soil
Gaza Line (Grootfontein - Runtu)	Author	Gravel road (good)	Pan in street between poorly developed seif dunes	-	-	Grass, scattered bush	Game reserve	Light grey with medium grey rim	Aeolian sand/ calcrete
Bloemfontein - Soutpan	Mountain (1967) and author	subgrade, possibly subbase	Flat, tabular sand covered area between pans	No surface drainage. Rectangular subsurface drainage	Pans	Grass?	Grazing	Grey mottled lighter grey. White when outcropping	Sand/ calcrete
Vryburg - Terrafirma	Mountain (1967) and author	Gravel road (quality not stated)	Caps an ancient land surface. No relief	Converging on pans	Pans	Grass, scattered bush?	Grazing; partly cultivated?	White when outcropping	Aeolian sand/ calcrete

much thinner and has only reached the honeycomb stage, but is also undergoing disintegration, and neither of these two calcretes are forming today. Calcretes between Tsumeb en Namutoni are very well developed, while those between Namutoni and Ondangua are generally rather poorly developed. Examination of Partridge's (1967) map of cyclic landsurfaces (Map 12) shows the Tsumeb-Namutoni area to belong to the African landsurface (late Cretaceous) and the Namutoni-Ondangua area to belong to one of the post African landsurfaces (Oligocene and Miocene). It should be noted that the ages given to land surfaces are the dates of initiation at the coast. The young nature of the deposits was also one of the reasons suggested by Professor H. Martin (1964, personal comm.) for the paucity of calcrete development in Ovamboland. The reason for the paucity or absence of calcrete development in the weakly developed soils and the windblown sands in South West Africa is also at least partly attributable to their very young age. Apart from other factors, the shifting sands of the Namib for example have not yet had time to develop any soil horizons.

6.4. REGIONAL PROSPECTING

A materials survey can be divided into two phases; an initial phase whereby a relatively large area or strip on each side of the proposed road centreline is searched for potentially usable materials, and a final phase, in which the materials deposits found are investigated in detail to determine whether or not they are workable propositions. The first phase could be termed regional prospecting and the second phase detailed investigation.

The first step in regional calcrete prospecting should be to consult the calcrete map (Map 4). How to make the best use of this map is discussed in Section 2.2.4. Some idea of the types of calcretes to be expected could also

be/ ...

be gleaned from the use of the overlay of land surfaces (Map 12). After having gained some idea of what to expect, any geological, pedological or soil engineering maps of the general area on a larger scale could be turned to good account. The subsequent steps should involve airphote interpretation and field work, discussed in more detail in the following sections.

6.4.1 Airphoto interpretation from conventional airphotos

In recent years this method has proved to be invaluable in materials surveys. The procurement of aerial photography and the fundamentals of its application to geology, soils and other problems has been discussed for example by Colwell et al (1960). Its application to soil engineering mapping in South Africa was first described by Brink (1962), though Schofield had described its use in materials surveys in Nyasaland in 1957. In 1964 King described the exact procedure involved in the use of airphotos for the location of borrow. The CSIR Research Report by Brink and Williams (1964) provides a recent review of the use of airphoto interpretation in soil engineering mapping in Southern Africa, while Caiger (1967) has described the method used by the South West African Roads Department. In essence the latter aims at the direct location of borrow materials by airphoto interpretation rather than the production of a map. A recent tendency on the part of some workers (Brink and Partridge, 1967) in South Africa has been to use the Land System - Land Facet approach to mapping which lends itself to data storage and retrieval.

The location of calcretes by airphoto interpretation has been shown to work well in the 'sandveld' of the Okavango and the south west Kalahari by Caiger (1964, 1968) and in southern Ovamboland and by Mountain (1964). In areas outside the Kalahari Beds, however, and occasionally even in this area, airphoto detection and delineation of calcretes is more difficult and less reliable. King (1964)

was able to differentiate between calcretes of greater and lesser plasticity between Okaputa and Otavi, and Caiger (1964) was able to differentiate between shoulder quality and inferior material near Sukses, but was unable to do so between Otjiwarongo and Okaputa. Mountain (1952) not surprisingly found that calcretes in the Vryburg area with high PIs were developed over Dwyka shale, while those developed over dolomite possessed consistently lower PIs. Mr. A.B.A. Brink (1964, personal comm.) was unable to detect at all some calcretes near Mariental. Caiger (1964) and Brink and Williams (1964) have stated that calcretes are among the most difficult materials to locate and delineate by means of airphoto interpretation. Uppal and Nanda (1968) noted that the kankar deposits in the alluvial plains of India usually occurred under a 2 - 3 ft soil overburden around the gently sloping edges of large depressions. Poor drainage and a lack of trees (the site being either cultivated or used for pasture) were also characteristics of the kankar sites. After thus establishing the airphoto pattern, they were able to achieve 80% success in predicting the occurrence of further deposits.

In an attempt to check these observations of other workers, the author plotted all known borrow pits for the proposed new Tsumeb-Namutoni road on the airphotos and, by looking for areas of similar airphoto patterns, suggested 44 sites where further materials of similar quality should be found or where the existing borrow pits should be improved by extension to one side. The consultants, Messrs. Ninham Shand and Partners, eventually dug trial holes at 21 of the sites. Of the 14 sites where calcretes of a certain quality were predicted, 4 were successful, 3 were completely unsuccessful, 5 yielded material somewhat inferior to that predicted, and insufficient information had been recorded to classify the remaining 3. Of the 7 improvements suggested, all but one were unsuccessful.

Plate/ ...

Plate 6.3 shows the borrow pits located without the use of airphotos for the Ondangua airport by the consultants, Messrs. Van Niekerk, Kleyn and Edwards. All the sites shown on the airphotos yielded calcrete of about natural base quality, but were used stabilized with lime for base and subbase. A stereoscope is unnecessary to predict where further materials of this quality will be found. At all other sites with dissimilar photo patterns, only very inferior calcretes and calcified or calcareous soils were found. Note also the white anthills on these photos.

Near Glen in the Orange Free State the author was able to delineate a calcrete deposit of roughly subbase quality on the airphotos by means of a characteristic mottled tone. Fossil drainage lines sometimes show up well on airphotos (Plate 6.2).

In order to determine which elements of the airphoto pattern are the most useful for the location of calcretes, a list of patterns for some calcrete deposits in various parts of the Republic and South West Africa was compiled (Table 6.1). There is a paucity of such data in the literature and most of the elements were compiled by the author from his own observations.

It can be seen that, in general, the elements of topography, drainage and tone and texture of surface material are the most useful. It would be misleading, however, to conclude that calcretes usually occur in depressions, possess a colinear drainage pattern and a mottled surface tone (all indicators of poor drainage), as the position of a calcrete deposit in relation to the present day topography and drainage depends mainly on its stage of development. Thus, while the younger calcretes are in fact associated with poor drainage conditions, the better developed ones tend to occur under better drained conditions, and a fossil calcrete deposit could possess virtually any elements

of/ ...

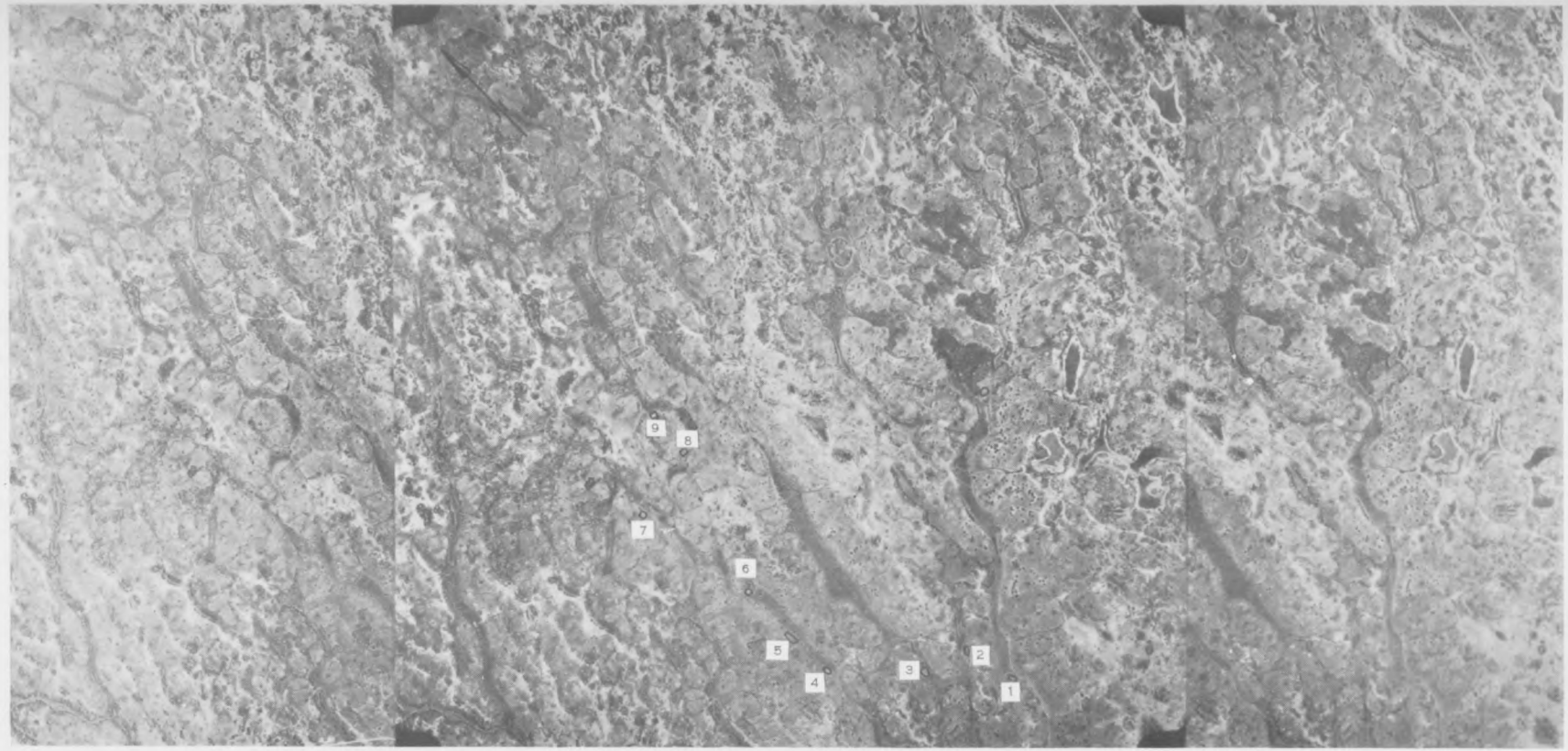


PLATE 6.3

Stereotriplet showing the location of base and subbase calcrete borrow pits for the Ondangua Airfield, South West Africa. Scale approximately 1:72,000. Borrow pits are shown approximately to scale and are numbered one to nine, according to the contract documents, with one unnumbered exception. Airphoto Nos. 8376–8378 of Aircraft Operating Company's. Job. No. S.160/64 reproduced by Graphic Arts, CSIR.

of the airphoto pattern.

In general, apart from possible small improvements in contrast which could be obtained by experimentation with different film/filter/camera combinations, there do not appear to be any improvements in the conventional techniques of airphoto procurement and interpretation which can be suggested to make calcretes more amenable to location by this method.

6.4.2 Interpretation from diapositives

Following up a suggestion by Mr. D. Kennedy (1965, personal comm.) the author attempted to make an assessment of the value of diapositives against conventional contact prints in airphoto interpretation for calcrete prospecting. Mr. Kennedy had pointed out that contact prints had a resolution of only 13 lines/ mm, while diapositives possessed the same resolution as negatives, namely about 48 lines/mm.

Fourteen glass diapositives on a scale of 1:36,000 and nine plastic diapositives on a scale of 1:72,000 were obtained covering known areas in the vicinity of the Operet-Ondangua and Grootfontein-Runtu roads in South West Africa. The diapositives were viewed by transmitted light on a light table with an Oude Delft scanning steroscope and compared with conventional 1:36,000 semi-matt and 1:72,000 glossy contact prints made from the same negatives.

It was found that the diapositives did definitely allow more detail to be seen at the maximum Oude Delft magnification of 4½X. Gabbabos and calcrete outcrops could be more easily detected and delineated on some diapositives, but others seemed to possess no advantage over contact prints. A higher magnification than 4½X may be required for their best utilisation.

Diapositives are made from the same negatives as contact prints and are about three times the price of the

latter/ ...

latter.

6.4.3 Colour photography

The advantages of colour airphotos in soils work have been discussed by Tator (in Colwell, 1960), Dominguez (in Colwell, 1960), Chaves and Schuster (1964), and Miles (1967). Due to the greater cost of film and processing, the cost of colour airphotos is two to four times that of black and white airphotos. Colour airphotos appear to possess definite advantages, however.

It has been noticed many times that the colour of the overburden is a fairly good guide to the quality of the calcrete, generally both the PI decreasing and the aggregate hardness increasing as the overburden colour changes from grey to red and as the ground rises away from the bed or edge of a drainage feature. The reason appears to be merely that the greater age of the calcrete under the redder soil implies both a more advanced stage of development and a better drained position, where the clay minerals have either been washed out or converted to the more inactive types. The good quality calcretes occurring in the fossil oshana lines around Ondangua are good examples of this. The use of ordinary colour airphotos and possibly camouflage detection film may then have some advantage in the search for buried calcrete deposits. "Camouflage detection film is also often better than panchromatic in tracing mineral deposits through plant patterns" (Tator, in Colwell, 1960).

6.4.4 Infrared photography

Ordinary panchromatic film is sensitive only to visible light, i.e. that section of the electromagnetic spectrum possessing wavelengths between about 0.3 and 0.7 micron. Infrared photography merely involves the use of film sensitive also (or mainly) to infrared light. Films

sensitive/ ...

sensitive to wavelengths up to about 1.2 microns are used. The sensitivity of the emulsion to infrared radiation means that the tone will depend to some extent on the temperature and emissivity of the object. The former is influenced by near surface bodies and the body will thus show up as a tonal difference on the infrared airphoto.

From the little information available on the subject (Colwell, 1960), it would appear that infrared photography is more sensitive to variations in drainage, moisture conditions and plant species than conventional panchromatic photography. This means that although buried calcrete deposits may result in tonal differences, these other factors may be even more important in the interpretation of infrared airphotos. Other advantages of infrared photography are a better haze penetration and sharper contrast than panchromatic photography.

6.4.5 Infrared imagery and other remote sensors

While all types of photography, imagery and conventional geophysical methods could be classed as remote sensors, only imagery and related methods are discussed in this section.

The types of remote sensors available and their applications have been briefly discussed by Kinsman (1965) and Fischer (1966). Of all the devices mentioned, the infrared imager, radar imager, infrared radiometer/spectrometer, microwave imager, microwave radiometer and the ultraviolet imager would appear to have possibilities in materials surveys in general, including the location of buried calcrete deposits. The infrared imager, radar imager, infrared radiometer/spectrometer, microwave radiometer, absorption spectrometer, radio frequency reflections and possibly the microwave imager are also sensitive to variations in soil moisture distribution. All these methods are unfortunately both in the developmental stage

and/ ...

and more or less 'classified' in the military sense. All are applicable to airborne use.

Of all these devices the most developed and most potentially useful appears to be the infrared imager. Principles and use of the technique have been described by Lattman (1963), Strangway and Holmer (1965) and others. Infrared imagery involves the use of a scanner sensitive to emitted infrared radiation having a wavelength of the order of 3 microns. The image is not photographed directly, but in common with other types of imagery is projected by means of a cathode ray tube and then photographed. The technique is far more sensitive to buried objects than infrared photography (it is believed that a pipe under six feet of concrete has been detected from the air) and it would appear that it should be easily capable of detecting buried calcrete deposits.

Some geological applications of radar imagery have been described by Cameron (1965), Simons (1965) and Rydstrom (1966). Interpretation of radar photos is based mainly on topography and possibly on differences in radar cross section reflection from different rock types. At present the method seems to have only been used for regional geological interpretation, but if correlation between the cross section reflection and rock type is established it could well become useful in materials surveying.

Multispectral sensing covering a large part of the electomagnetic spectrum will probably become the most useful technique of the future. Kinsman (1965) mentioned the use of a multisensor aircraft carrying gamma ray spectrometers, multiple camera systems and radar, infrared and ultraviolet scanners, most of which could be operated simultaneously.

Apart from other advantages, remote sensing devices do not generally appear to be affected by weather conditions.

6.4.6/ ...

6.4.6. Geophysical methods

Geophysical methods have not to the author's knowledge been used as regional prospecting tools in materials surveys for roads, although the resistivity and shallow seismic refraction methods are capable of indicating the presence of harder layers of any material at depth. They are, however, rather slow and are better suited to centreline surveys and the investigation of the in situ properties of a calcrete deposit. They are therefore discussed further under that heading.

Another geophysical method which has not been tried, but may be of value for regional prospecting is the electromagnetic (E.M.) method. This method consists of the detection of a conducting body by means of an induced electromagnetic field. The author has experience of the Crone method, which is very rapid (and does not require a topographic survey) and was able to detect unmineralised shear zones in granite by this means, presumably due to their higher moisture content. As calcretes are often associated with water, they may be detectable by this method in some cases. A type of E.M. device capable of detecting orebodies beneath highly conductive saline overburden has been described by Geyleynse and Barringer (1965).

The general principles of electromagnetic and other methods have been discussed for example by Dobrin (1960) and Eve and Keys (1954). Kent and Enslin (1965) have discussed the methods used in ground water prospecting in South Africa.

6.4.7 Geochemical methods

The author has experience of geochemical methods for copper, lead, zinc and nickel and considers that a method could be devised for calcretes, but that it would be too costly for calcrete prospecting. It may also not be possible to distinguish between calcareous soils and calcretes by this method. Work was not therefore carried out in

search of a geochemical method of calcrete location.

Calcretes often present problems in geochemical exploration for metals like those mentioned above, and some useful work could be carried out on this problem. It is believed that good results in calcrete areas have been obtained in Australia and Botswana.

6.4.8 Plant indicators

Of all the methods of calcrete prospecting, this is one of the most widely used. Hawke (1957) in Mogg (1961), has defined a plant indicator as "a species whose distribution is affected by the chemical constituents of an ore deposit" - in this case calcrete. McKinstry (in Mogg, 1961) mentioned further that metals may alter the foliage, and that certain plants can concentrate elements selectively from soil solutions. This latter characteristic has also been used in geobotanical prospecting and Mogg stated that plant sampling can be better than soil sampling due to the greater volume and depth of soil effectively sampled. He also mentioned the importance of the soil pH in controlling the species present and found that more than about 4 feet of transported soil cover masked the effect of the bedrock on the plant cover in the Vryburg area. The latter is in accordance with the author's findings in South West Africa, where it was found that the presence of the 'gabbabos' invariably meant the presence of at least a calcareous soil within about 5 feet of the surface.

The following list of plants indicative of at least a calcareous subsoil has been compiled (Table 6.2). The author is indebted to Mr. A. Barnard of the South West African Department of Agriculture for his assistance in compiling the list and in the identification of specimens submitted to him, and also to Mr. B. Maguire of the Bernard Price Institute for Palaeontological Research who supplied the names of the Botswana plants. Acknowledgement

is/ ...

is also due to Mr. W.B. Coetzee and Mr. Nel, who first drew the author's attention to the bitterbos, Professor H. Martin who mentioned the gabbabos, Mr. Jakobi who pointed it out, Mr. N. Smit who pointed out the Karroo wolbossie and the soetdoring, and Mr. A. Burger who confirmed the author's ideas on the distribution of some of the plants. The vaalbos was noticed by the author in the Okavango and identified by Mr. Barnard and also later pointed out by Mr. A.B.A. Brink near Christiana. The driedoring was also mentioned by Van der Merwe (1962).

The distribution of all of these is unfortunately incompletely known, although a search of the botanical literature has not been carried out. All of the above are known to be indicators of at least a calcareous subsoil in Southern Africa. Of these, the author only has personal experience of the gabbabos (Plates 6.4 and 6.5), vaalbos and bitterbos (Plate 6.6) and has found them to be very reliable. It should be noted that the absence of a plant indicator does not necessarily mean the absence of a calcareous subsoil. The presence of one of the above three plants has, however, been invariably found to indicate at least a calcareous subsoil.

In addition, Acacia reficiens (rooihaak) while not a calciphilous plant, is a water-loving one (phreatophyre), and the presence of shallow water itself in an arid or semiarid area is a likely indicator of calcrete. The rooihaak was the indicator at Profile 454, for example.

Table 6.2/ ...



PLATE 6.4

Gabbabos (Catophractes alexandri) in the Etosha Game Reserve, S.W.A. Probably the most reliable and widespread calcrete plant indicator in South West Africa.



PLATE 6.5

Gabbabos (Catophractes alexandri) in flower near Neuhof-Kowas, S.W.A. The gabbabos generally seems to grow to a larger size in the southern half of South West Africa.



PLATE 6.6

Dense growth of bitterbos (Pechuel – Loeschea leubnitziae) over a buried calcrete deposit developed in a fossil oshana course near Ondangua, S.W.A. A profile through the centre of this deposit is represented by Profile 161 (Figure 5.2).

TABLE 6.2 CALCIPHILOUS PLANTS OF SOUTHERN AFRICA

NAME	COMMON NAME	DISTRIBUTION
A. albornarginatum		
Acacia newbrownii	Soetdoring	S.W.A. south of lat. 19°S
Aizoon fruticosum	Basterganna	
A. leucorrhizum		
Aptosimum abietinum		Transvaal
Barberia rigida		
Boscia rehmanniana		Botswana
Catophractes alexandri	Gabbabos	S.W.A. receiving less than 20" rainfall
Enneapogon brachystachyus	Kalkgras	Botswana
Eriocephalus ericoides		Botswana, Karroo
Geigeria		Botswana
Grewia flava		Botswana
Grewia vernicosa		Botswana
Hermannia spinosa		
Justicia platysepala		
Kirkia wilmsii		Botswana
Leucosphaera bainesii	Karroo wolbossie	S.W.A. south of Lat. 19°30's. Karroo
Ornithoglossum calcicolum		
Osteospermum muricatum		
Pechuel-Loeschea leubnitziae	Bitterbos	Ovamboland receiving more than 20" rainfall
Petalidium oblongifolium		Botswana
Polygala seminuda		
Rhigozum trichotomum	Driedoring	S.Kalahari
Salsolatubercula var tomentosa	Ganna	
Sporobolus ioclados		
S. rangei		

TABLE 6.2 (CONTINUED)

NAME	COMMON NAME	DISTRIBUTION
Stipagrostis hirtigluma var patula		
Tarchonanthus camphoratus	Vaalbos	Okavango, S.Kalahari, Griqualand West
Terfezia	Truffle	Botswana
Tetragonia		
Zygophyllum suffruticosum	Skilpadbos	Botswana

Mr. Maguire also suggested the following possible calciphilous plants (Table 6.3).

TABLE 6.3 POSSIBLE ADDITIONAL CALCIPHILOUS PLANTS OF SOUTHERN AFRICA

NAME	COMMON NAME	DISTRIBUTION
Acacia detinens Various aloes Celtis kraussiana (rhamnifolia) Cephalocroton pechuelii Cleome diandra Corallocarpus sphaerocarpus Enneapogon scoparius Hermbstaedtia Schmidtia kalahariensis Vieten rehmanni	Aloes	N. Transvaal, Ngamiland, S.W.A.

When present in quantity, some calciphilous plants give rise to characteristic airphoto patterns. Mr. A.B.A. Brink (1966, personal comm.) was able to delineate calcrete occurrences in the Vryburg area by this means using the vaalbos, while the author was able to do likewise in the Stinkwater area of the Etosha Pan using the gabbabos.

Descriptions/ ...

Descriptions of the genera of most of the species mentioned in the tables, together with references for further details and some comments on distribution will be found in Phillips (1951).

6.4.9 Zoological indicators

While carrying out the field search for calcrete deposits, careful observation of the surface soil should be made for stray lumps of calcrete brought up by burrowing animals and insects. The spoils from their burrows should also be examined for traces of calcrete. These indicators can be just as useful and more positive than plant indicators.

White anthills often show up well on airphotos (e.g. between borrow pits 4 and 7 on Plate 6.3, but usually indicate a fairly plastic calcrete.

6.4.10 Drilling and excavation

The digging of trial pits is probably the most widely used prospecting method and, though obviously yielding much information, is slow and tedious. A labourer cannot excavate much more than a cubic yard of firm soil a day, so that, as the minimum surface dimensions of the hole are about 3 ft by 2 ft, it requires one to two man-days to excavate to six feet.

A 30" diameter power auger (Plate 6.7)capable of drilling to 20 feet was used for some of the holes in the field investigation in Ovamboland and was found to be excellent in the rapid and complete information it yielded, but being mounted on a 5 ton, 2 wheel drive truck (in this case) was both clumsy and expensive.

The bulldozer method used in some areas for regional prospecting is extremely wasteful and should not be condoned. This is plainly a machine for the final phase of the detailed investigation of a deposit.

Hand-held/ ...



PLATE 6.7

Cheshire Highway Boring Machine in action. Note size of spare auger bit lying on the ground and Oude Delft scanning steroscope set up on metal folding table in right foreground.

Hand-held power augers weighing about 150 lbs. are available and should prove very useful, but do not appear to have been used much, if at all, for calcrete prospecting. They yield rapid information, but require three persons for their operation and cannot drill through anything harder than that penetrable by a hand auger.

An ordinary hand auger was found to work very well except when the soil was very dry and sandy, when water had to be used to provide the soil with some cohesion. The Edelmann type with the sheet metal spiral point was found to be much faster than the usual Hardy Pick post-hole type in the soils encountered.

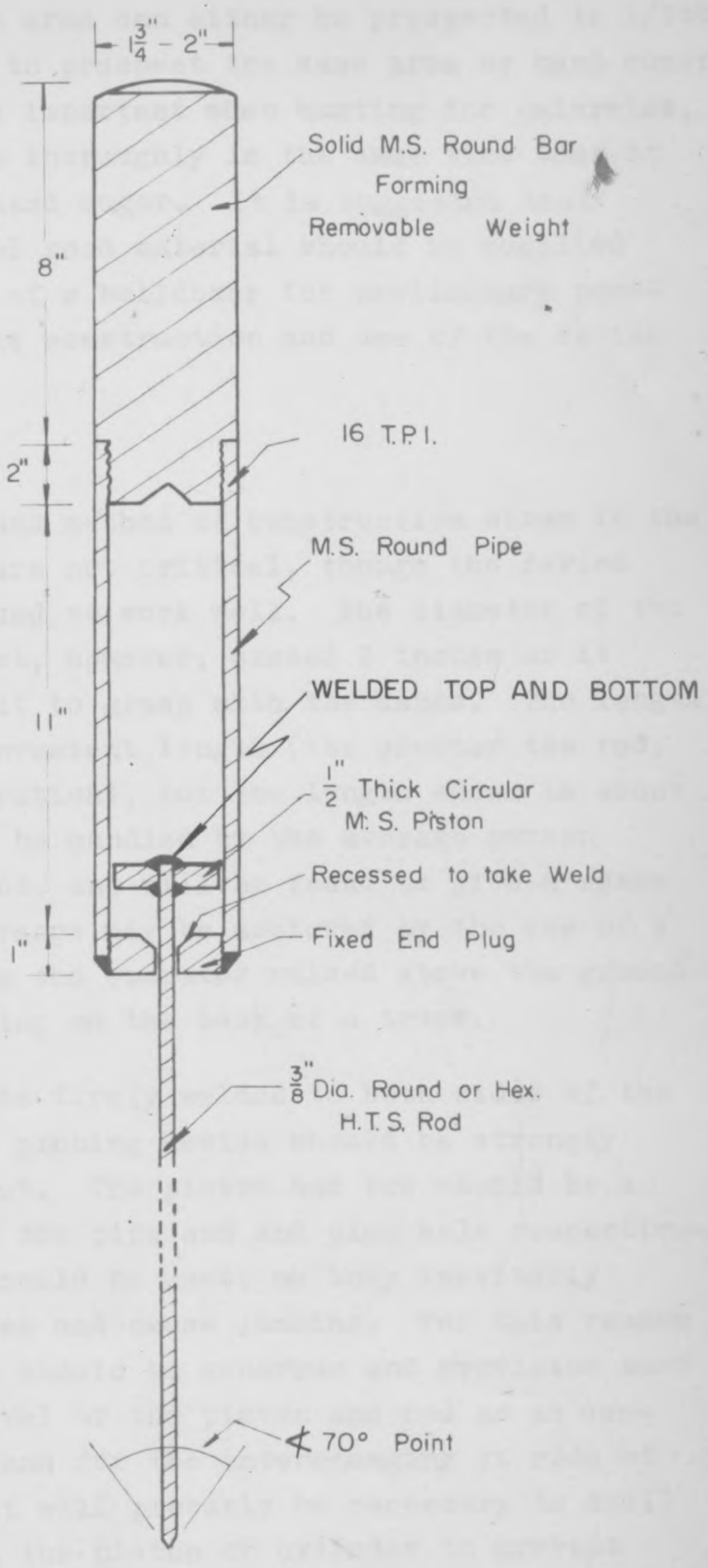
A rapid probing device invented by the late Senior Road Inspector at Upington, Mr. T. Van der Westhuizen, and unknown elsewhere, has been found by the author to be an excellent quick indicator of the presence of calcrete. Depending on the texture of the soil, it was found to require $\frac{1}{2}$ - 1 minute to complete a probe to 6 feet, which was 9 times as fast as the Edelmann hand auger. The probing device has been thoroughly tested out by the author who has found it to be unsurpassed as a preliminary indicator of calcrete within a usable depth. A drawing of the probing device appears in Figure 6.2. Using the attached pipe hammer, the rod is merely hammered into the ground until refusal or its maximum range is reached. It is then hammered upwards to effect a rapid withdrawal. Any calcrete present will have stuck to the point, while a few evenly spread shallow lateral saw cuts across the rod will allow the depth of penetration in loose calcrete to be estimated. The overburden thickness is of course also easily calculated. Though simpler, the probing device is in principle somewhat similar to the Linnemann auger used for Quaternary geological mapping in Europe for many decades.

The author has found this device to be cheap to make, foolproof, almost unbreakable and capable of indicating the location, extent, type, and overburden thickness of a

calcrete/ ...

NOTES:

1) DEPENDING ON THE PISTON/CYLINDER FIT IT MAY BE NECESSARY TO DRILL VENTILATION HOLES IN THE PISTON OR CYLINDER TO PREVENT AIR COMPRESSION.



SKETCH SECTION.

FIGURE 6·2

Rapid calcrete probing device

calcrete deposit. An area can either be prospected in 1/9th of the time required to prospect the same area by hand auger or, and possibly more important when hunting for calcretes, prospected 9 times as thoroughly in the same time that it would have taken by hand auger. It is suggested that prospectors for gravel road material should be supplied with a probe instead of a bulldozer for preliminary prospecting. Hints on the construction and use of the device are given below.

Construction

The dimensions and method of construction shown in the sketch (Figure 6.2) are not critical, though the device as shown has been found to work well. The diameter of the pipe hammer should not, however, exceed 2 inches as it then becomes difficult to grasp with the hands. The length of rod may be any convenient length (the shorter the rod, the easier the penetration), but the length shown is about the maximum that can be handled by the average person standing on the ground, and will be found to give a range of 6 ft. A greater range may be achieved by the use of a longer rod and having the operator raised above the ground, for example by standing on the back of a truck.

The rod should be firmly welded to both sides of the piston and the whole probing device should be strongly constructed throughout. The piston and rod should be a loose sliding fit in the pipe and end plug hole respectively. No lubricants should be used, as they inevitably collect sand particles and cause jamming. For this reason also, the clearances should be generous and provision must be made for the removal of the piston and rod as an assembly for cleaning and for the interchanging of rods of different lengths. It will probably be necessary to drill a few small holes in the piston or cylinder to prevent air compression.

The/ ...

The angle of the point should not be less than about 70° or it will rapidly become bent in service.

Use

The device is merely hammered into the ground to its full length or until refusal, upon which it is hammered upwards to affect a rapid withdrawal. Any calcrete encountered will have stuck to the point and, if penetrated to any depth, in the lateral saw cuts as well. A depth reading may be obtained from suitable marks on the rod or by subtracting the length of rod remaining above the ground from the known range of the device. It is unnecessary to clean the point unless bent, as it will normally clean itself after having penetrated about one foot of soil on the following probe.

The device will only refuse on very hard calcrete. While it may have difficulty in penetrating a dry clayey material, it is still faster than any hand auger.

It may be necessary to occasionally resharpen and straighten the rod in the field.

Due to the great speed with which probes may be made, it is recommended that the device be used for preliminary work before hand augering or pitting.

6.5 DETAILED INVESTIGATION

Once a calcrete deposit has been located, the next task becomes one of proving whether the deposit is of sufficient quality, extent and thickness and occurs at a sufficiently shallow depth to be economic to work. The results of this investigation will also indicate the economic haul distance.

6.5.1 Rapid Probing

The probe is able to determine the extent and depth of the calcrete deposit with great speed. With a little

experience/ ...

experience the type of calcrete can also be estimated from the resistance to penetration and the colour and texture of the deposit on the point. A rough guide is given below. (Table 6.4).

TABLE 6.4 INTERPRETATION OF PROBE RESULTS

PENETRATION RESISTANCE	POINT APPEARANCE		CALCRETE TYPE
Varies from almost none to refusal within the space of a few feet	No deposit (little resistance) or white or pale pink colour (refusal)		Loose sand interspersed with boulder calcrete
Refusal	White or pale pink colour	Cannot all be rubbed off with the fingers	Hard hardpan or boulder, probably unpickable
High	White or pale pink colour Pale mauve		Loose, hard nodular calcrete, stiff hardpan or calcified sand. Tufaceous hardpan
Fair to low	White		Powder calcrete
Low	White sandy deposit; can be rubbed off easily with the fingers		Calcareous soil

6.5.2 Overburden and calcrete colour and overburden type

The best graded, hardest and least plastic calcretes are usually found under a reddish sandy overburden on slopes or rises. Furthermore, reddish and brownish calcretes also invariably have lower PIs, are better graded and have harder aggregate than white, grey, pink or bluish

calcretes/ ...

calcretes. Bluish calcretes invariably have a high PI. Greenish ones often have a low PI, but are usually saline. This method of prospecting works well in the oshana country around Ondangua for instance. In the oshana beds, with their grey silty or clayey sand overburden, only olive green calcareous or calcified clayey sand with a few scattered soft nodules is found. The quality then increases away from the oshana to a nodular calcrete and then, when the reddish sand is encountered, a hardpan overlying hard nodular calcrete of low plasticity may be found.

6.5.3 Plant indicators

In the Ondangua area the best calcretes are not only found on low sandy rises with a reddish brown sandy overburden but, if uncultivated, also possess a prolific growth of bitterbos. If cultivated, the best muhango crop will be found.

Generally speaking, the more prolific the plant indicator, the more highly calcified the subsoil.

The author has not noticed any variation in the indicator itself, though according to Mr. A. Burger (1965, personal comm.) the gabbabos stem becomes darker over more powdery calcrete.

6.5.4 Hand augering and pitting

If the probe results indicate the possibilty of good calcrete below, it may be worth augering in order to obtain a sample, but in most cases the probe will be found sufficiently reliable to dispense with the hand auger and proceed directly to the next stage, that of pitting, or augering with a large diameter truck-mounted auger if one is available.

Owing to the extreme variability, both laterally and vertically, of most calcrete deposits it is advisable to pit very thoroughly.

6.5.5/ ...

6.5.5 Geophysical methods

During the last ten years the resistivity and shallow seismic refraction methods have found increasing application in materials surveying in the United States. The general principles of seismic and resistivity methods have been described for example by Eve and Keys (1954) and Dobrin (1960). Seismic methods in use for materials surveying in some states of the U.S.A. have been described by Irving (1965), Bigelow (1965), Malott (1965, 1967) and Lawson, Foster and Mitchell (1965), while resistivity methods have been described by Malott (1965, 1967) and Lawson, Foster and Mitchell (1965). Platts (1965) and Johnson (1967) have described some limitations of the resistivity method and geophysics in general in highway work. In Southern Africa, papers on a shallow seismic refraction instrument have been published by Kühn (1960) and Van Opijnen (1960), while Holden (1959) and Pinto Moreira (1959) have given details of the resistivity procedures used by the Rhodesian Roads Department and on a pipeline project respectively. The shallow seismic refraction and the resistivity methods have been successfully applied to calcretes in the South Africa by Barlows (Mr. J.M. Weaver, 1964, personal comm.) and the author, and by Mr. E. Matthews (1966, personal comm.) respectively, but these methods are only suitable for detailed investigations such as rippability and bearing capacity. (See Figures 6.3 and 6.4) and as a supplement to drilling in the delineation of subsurface horizons. This seismic method is also unable to determine the quality of a softer material underlying a harder - a common feature of calcrete deposits.

The Caterpillar Tractor Co. (1966) make use of the shallow seismic refraction method as an aid to the estimation of rippability and production rate and have published data relating the performance of various tractor/ripper

combinations/ ...

combinations to the seismic velocity of the material concerned. A material becomes more difficult to rip as the velocity increases. Caliches (calcretes) having velocities from 3,000 to 8,200 ft/sec may be readily ripped by a Caterpillar D9G with a No. 9B ripper, for example. If the seismic velocity of a calcrete is 6,000 ft sec for example, the above tractor/ripper combination should have an output of about 120 bank cu yds hr. Mr. F.A. Sabatier of Barlows (1967, personal comm.) stresses, however, that the published curves are used only as a guide, and that engineering judgement is still required.

The author has found the seismic velocities of South African calcretes to range from 1,350 ft/sec for a powder calcrete to 15,000 ft/sec for a very hard, intact hardpan. A total of 53 seismic traverses were made with a Viatec instrument, some for exploratory purposes, but mostly over already or subsequently known profiles. A time-distance graph for Profile 246 is shown in Figure 6.3 as an example. In this particular case an unusually good result was obtained, a wider scatter and less good depth correlation being more normal. It was not always clear whether this was due to the instrument being at fault or the natural variability of the calcrete. Reverse traverses were not normally found to be necessary. It was found that the different types of calcrete could be grouped roughly according to their seismic velocities (Table 6.5). Figures obtained at the same time for sands of Kalahari type are also shown for comparison.

Very erratic time-distance graphs were obtained in the case of boulder calcretes and in most cases a velocity could not be calculated. Possible velocities obtained varied between 5,000 and 10,000 ft/sec in some cases. Only 5 out of 21 nodular calcrete determinations fell below 2,000 ft/sec and it was uncertain whether these particular profiles had not been disturbed during bulldozing operations.

Table/ ...

FIGURE 6·3

Seismic transverse over a calcrete deposit.

RG4 - 2668

TABLE 6.5 SEISMIC VELOCITIES OF CALCRETES AND SOME OTHER MATERIALS

MATERIAL TYPE	SEISMIC VELOCITY (ft/sec)		NUMBER OF DETERMINATIONS
	RANGE	MEAN	
Boulder calcrete	Probably unreliable		6
Hardpan calcrete	3,850 - 15,00	6,680	11
Honeycomb calcrete	2,670 - 5,100	3,210	6
Nodular calcrete	1,200 - 3,080	2,220	21
Powder calcrete	1,350 - 4,300	2,490	11
Calcified soil	-	-	0
Calcareous sand	1,100 - 1,300	1,180	6
Red Kalahari-type sand	840 - 1,470	1,130	22

It is considered that nodular calcretes will normally fall within the range 2,000 - 3,000 ft/sec. The relatively high velocities of some powder calcretes emphasize their sometimes stiff in situ nature. The powder calcrete with a velocity of 4,300 ft/sec (Sample 2223) nevertheless broke down into material with a grading modulus of 0.6 when excavated with an RB23 mechanical excavator. Two further determinations over the same deposit yielded velocities of 2,700 and 2,300 ft/sec which are probably more realistic values. The calcareous sand determinations shown were on slightly clayey, redeposited Kalahari sands with varying degrees of calcification. Their consistencies varied from soft to firm. The Kalahari sands were more or less loose, but could usually just stand vertically in an excavation, probably due to some cementation by ferric oxide. When forming the topsoil of croplands they appeared slightly silty. The in situ density of the Kalahari sand forming

the/ ...

the subgrade of an experimental road section just north of the Andoni Flats in Ovamboland was about 95 lbs/cu/ft (Mr. C.P. Marais, 1966; personal comm.).

Allowing for experimental errors in Table 6.5, it is considered that the values shown in Table 6.6 will in most cases be found to hold and could be used to some extent for prospecting purposes. It would appear that calcretes having seismic velocities of less than about 3,000 ft/sec. can be bulldozed without ripping, while those with velocities above about 10,000 ft/sec cannot be ripped by any tractor/ripper combination at present in service, and will definitely have to be blasted. Blasting of calcrete hardpan can be difficult owing to dissipation of the blast by the softer layer below. The technique in this case is not to drill the shot holes completely through the hardpan.

TABLE 6.6 SEISMIC VELOCITIES OF TYPICAL CALCRETES

MATERIAL TYPE	SEISMIC VELOCITY ft/sec
Boulder calcrete	erratic
Hardpan calcrete	3,000 - 15,000
Honeycomb calcrete	3,000 - 4,000
Nodular calcrete	2,000 - 3,000
Powder calcrete	1,300 - 3,500
Calcified soil	2,000 - 5,000?

During a foundation investigation at Lichtenburg for Whites Portland Cement Company by Messrs. Van Niekerk, Kleyn and Edwards, calcrete hardpan samples for unconfined compression testing were obtained by diamond coring. A single resistivity traverse was also carried out over the same site along the line of the boreholes by Mr. E. Matthews of Whites. By kind permission of Mr. A. Albertyn of

Whites/ ...

Whites, Mr. J. Weaver of Van Niekerk, Kleyn and Edwards and Mr. Matthews, the results were made available to the author. The true resistivities at various depths were estimated and plotted against the unconfined compressive strength at that depth and the results are shown in Figure 6.4. Where the unconfined compressive results were found to vary widely over a short distance (a foot or so) the highest value was normally used, as it probably represented the intact state. While this approach is not fully objective, and the average strength should perhaps have been used, it is nevertheless considered that the results do serve to show that the resistivity method can be used to give an indication of the strength or bearing capacity of calcretes. The interpretation of field conductivity readings has been discussed by Grant and West (1965) and they state that the conductivity (or resistivity) is mainly dependent on the porosity and permeability of the rock and the conductivity of the fluid it contains, the mineralogy of the rock having little influence. Small amounts of clay can, however, increase the conductivity at low water contents. It will be shown later that the aggregate crushing strength of a calcrete is greatly dependent on its porosity. A plot of standard penetration results at the Lichtenburg site against resistivity gave very erratic results, which are not shown. This is thought to be due possibly to the few readings available and also to the standard penetration test probably not being particularly suitable for these materials.

6.5.6 Bulldozing and ripping

After the pitting programme and any geophysical work thought necessary, the final step is plainly to open up the deposit by ripping or blasting if necessary and dozing, scraping or excavating.

6.6/ ...

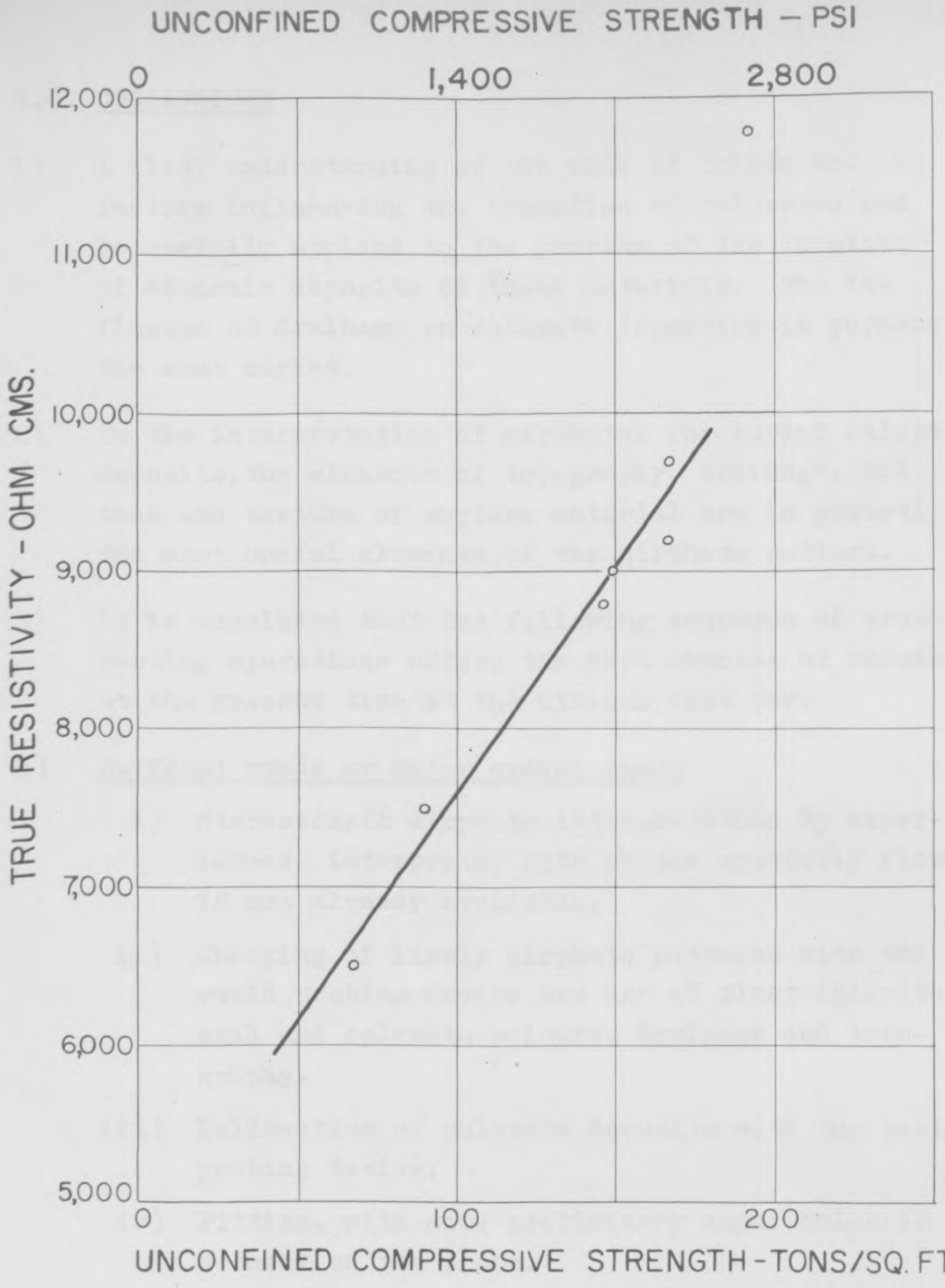


FIGURE 6·4

Relation between resistivity and unconfined compressive strength

RG - 240
RG4 - 2116

6.6 CONCLUSIONS

1) A clear understanding of the mode of origin and the factors influencing the formation of calcretes can be usefully applied to the problem of the location of economic deposits of these materials. The influence of drainage on calcrete formation is perhaps the most marked.

2) In the interpretation of airphotos for buried calcrete deposits, the elements of topography, drainage, and tone and texture of surface material are in general the most useful elements of the airphoto pattern.

3) It is concluded that the following sequence of prospecting operations offers the best chances of success at the present time at the minimum cost for:

a) Surfaced roads or major gravel roads

i) Stereoscopic airphoto interpretation by experienced interpreter with photos specially flown if not already available.

ii) Checking of likely airphoto patterns with the rapid probing device and use of plant indicators, soil and calcrete colours, drainage and topography.

iii) Delineation of calcrete deposits with the rapid probing device.

iv) Pitting, with some preliminary auger holes as a check on the probe.

v) Seismic survey for rippability if necessary.

vi) Opening up of deposit.

b) Minor gravel roads

i) Provision of airphotos, if available, to road superintendents or inspectors (one photo wide

coverage/ ...

coverage only), together with advice on air-photo navigation and non-stereoscopic interpretation. It is essential that persons using airphotos be able to pinpoint their positions in the field exactly on the airphotos. Even for an experienced user this can be quite difficult in areas like the Okavango and Ovamboland.

ii) Rapid probing of areas seen on the airphotos to have appearance similar to known good deposits.

iii) Use of plant indicators, soil and calcrete colours, drainage and topography.

iv) Opening up of deposit.

4) The use of colour and infrared airphotos, diapositives and infrared imagery (particularly the latter) appear to have promise for calcrete location. Various remote sensing devices still in the developmental stage also appear promising.

5) Although airphoto interpretation is an indispensable preliminary tool, it should not be completely relied upon for the location or delineation of calcrete deposits.

6) Apart from the use of diapositives and colour, camouflage detection or infrared films, no real improvements to the conventional techniques of airphoto procurement and interpretation for calcrete location can be suggested.

6.7 SCOPE FOR FURTHER INVESTIGATION

1) The use of colour and camouflage detection airphotos, diapositives and infrared photography and imagery should be properly investigated.

2/. ...

2) It would be interesting to try the Crone electromagnetic method over a few known calcrete deposits to see if they can be detected. The rapid probing device is, however, just as fast and much cheaper and easier to use. The electromagnetic method may be more rapid when exceptionally thick overburdens are encountered. In Ovamboland calcretes have been worked under overburdens as thick as thirteen feet.

3) A systematic survey of plant indicators for all pedogenic road materials and the provision of samples of these plants to those concerned with prospecting would be a worthwhile project.

4) Further work should be carried out on the resistivity method for the determination of strength or bearing capacity before it can be considered reliable.

5 A correlation between seismic velocity and strength or bearing capacity would also be useful. Both the resistivity and seismic methods take into account joints and cracks in the material.

6) More determinations of the seismic velocities of the various calcretes should enable a more accurate bracketing to be obtained. This grouping may also be possible by the resistivity method.

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CHAPTER 7

ENGINEERING TEST METHODS

7.1 INTRODUCTION

7.2 PREPARATION OF THE SOIL FINES FOR DETERMINATION OF SOIL CONSTANTS AND MECHANICAL ANALYSIS

7.2.1 Comparison of methods in use

7.2.2 Effect of drying temperature

7.2.3 Suggestions for calcretes

7.3 LIQUID LIMIT

7.3.1 Comparison of methods in use

7.3.2 Accuracy and reliability of liquid limit determinations on calcretes

7.3.3 Suggestions for calcretes

7.4 PLASTIC LIMIT

7.4.1 Comparison of methods

7.4.2 Application to calcretes

7.5 PLASTICITY INDEX

7.5.1 Factors influencing plasticity index determinations on calcretes

7.6 BAR LINEAR SHRINKAGE

7.6.1 Advantages of the bar linear shrinkage test

7.7 MECHANICAL ANALYSIS

7.7.1 Comparison of methods

7.7.2 Application to calcretes

7.8 AGGREGATE STRENGTH AND DURABILITY

7.8.1/ ...

7.8.1 Aggregate crushing and abrasion tests

7.8.2 Tests under wet conditions

7.8.3 A simple new pliers test

7.9 HARDNESS

7.9.1 Correlation between hardness and strength

7.9.2 A new pocket Mohs hardness tester

7.10 SELF-STABILIZATION

7.10.1 Prediction of self stabilization

7.10.2 Detection of self stabilization in road bases

7.11 SOLUBLE SALTS

7.12 MISCELLANEOUS METHODS

7.13 CONCLUSIONS

7.14 SCOPE FOR FURTHER INVESTIGATION

7.15 REFERENCES

7.1 INTRODUCTION

This chapter seeks to examine the applicability to calcretes of the standard test methods used in road engineering, to point out possible sources of error therein and to suggest improvements and new tests. Very little has been published on the subject and there is some evidence that plasticity indices and mechanical analyses may be unreliable guides to the performance of calcretes as road materials. Certain overseas authorities (the Texas Highway Department and the Federal Aviation Agency, for example) appear to allow a higher PI and poorer gradings for caliche* base courses than for other materials and in this country the Transvaal and Orange Free State Roads Departments relax their PI and grading requirements

somewhat/ ...

*United States caliches appear to be identical to South African calcretes.

somewhat when using calcretes for gravel roads. The term 'apparent PI' has been used by some when referring to the PI of calcretes and ferricretes, also considering that higher 'apparent PIs' could be allowed for these materials (Dr. H.H. Weinert, 1964; personal comm.). If for example the true PI is much lower than the apparent PI (obtained by testing in the normal way) it is obvious that one is over-designing and this of course is not good engineering. It serves a purpose then to examine not only the properties of calcretes, but also the methods used to determine those properties.

Virtually the only published work done on test methods for calcretes seems to be that of Gillette (1934). Gillette described the caliches of Texas and adjacent areas, grouping them into flourlike, semihard and hard caliches, and discussed test results on some roads built of these materials in Texas. Results on 30 samples of known satisfactory and unsatisfactory caliches were shown. He concluded that cementing value tests and chemical analyses were not generally significant indicators. While there was some tendency for the better caliches to have a lower cementing value and to be higher in calcium carbonate, the correlation was too erratic to be of great value. Gillette concluded further that the best test for caliches for bases under bituminous surfacings was the plasticity index, supplemented by the flocculation factor. Satisfactory materials were found to possess PIs of less than 15 and flocculation factors of less than 2.5. These specifications were most often met by the hard or semihard types. Other tests tried were mechanical analysis, liquid limit, shrinkage limit, shrinkage ratio, centrifuge and field moisture equivalent and volumetric change. It is interesting to note that he also stated that "shrinkage as indicated by either the volumetric change or its related constant, lineal shrinkage seems to be a minor factor in contributing to failure". Satisfactory

materials/ ...

materials had volumetric changes of less than 14% and liquid limits of less than 39% , though the converse was not the case, unsatisfactory materials having volumetric changes down to 3 and liquid limits down to 33. Unfortunately, no bar linear shrinkage figures were given; the test had probably not yet been devised, but linear (lineal) shrinkage is of course roughly a third of the volumetric shrinkage and formulae are available for its exact calculation from the volumetric shrinkage (see ASTM D427).

The United States Federal Aviation Agency (O'Fiel, 1944 and FAA, 1959) plainly consider that thorough slaking is necessary in the preparation of caliches for mechanical analysis and determination of soil constants. A slaking method is also followed by the Texas Highway Department (1966) for materials in general. Much caliche is used in this state. Both authorities also specify that the soil fines shall not be heated above 140°F (60°C) before Atterberg limit testing.

A short discussion of what a particular test actually measures and the variables affecting the results is often included in order to clarify and stimulate thinking as to the importance and meaning of the test when applied to calcretes. These discussions are not intended to be complete, but merely to make the reader realise that the full significance of some of the common tests is not properly understood, even today. The standard, stereotyped definitions given in many standard textbooks and methods tend to cause this fact to be lost sight of. Another point perhaps worth making at this stage is that "not all soils can be classified for roadbuilding purposes by physical tests alone. The chemistry of the soil must also be considered in many cases" (Rapp and Mizroch, 1940).

It should be noted that in soil mechanics work all moisture contents and additions of stabilizing agents like

lime/ ...

lime and cement are expressed as percentages of the oven dried (105-110°C) soil. A soil containing 10% moisture for example would contain 10 grams of water for every 100 grams of oven dried soil. As a conventional percentage it would contain only 9.1% moisture. Similarly, if a calcrete stabilized with 4% lime is referred to, it would mean that 4 grams of lime have been added to every 100 grams of oven dried soil.

Unless otherwise stated, throughout this work the term 'CBR' or 'Modified AASHO CBR' means the CBR at 0.1" penetration after 4 days' soaking on material compacted at Modified AASHO compaction and optimum moisture content.

7.2 PREPARATION OF THE SOIL FINES FOR DETERMINATION OF SOIL CONSTANTS AND MECHANICAL ANALYSIS

That fraction passing the U.S. No. 40 (B.S. No. 36) sieve (0.420 mm) is known in South Africa as the soil fines. In the United States this fraction seems to be known as the soil binder. This should not be confused with the South African term 'soil mortar', which is the fraction passing the U.S. No. 10 (2 mm) sieve.

7.2.1 Comparison of methods in use

All methods involve riffling or quartering of the bulk sample to the required size and drying. The drying temperatures specified vary considerably. British Standard 1377:1961 (p. 18) states that some tropical soils are even affected by air drying and that all soils to be used for plasticity or pH tests shall not be dried at above 50°C (122°F). In view of the comments of Wall (1959), it would appear that laterites may be the tropical soils referred to. ASTM D421-58 (1965) specifies air drying only, while AASHO T87-49, FAA P-210-2.2, AASHO T146-49 and TEX-101-E prohibit drying at temperatures above 140°F (60°C). ASTM D2217-66 makes provision for two procedures, one involving drying at less than 140°F

and/ ...

and the other involving keeping the soil sample at a moisture content greater than or equal to its natural moisture content. D.O.T. Method No. 1 (1958) allows oven drying to an unspecified temperature (presumably 105-110°C). NIRR Method A (1966) prohibits oven drying. The method given by RRL (1952) allows air drying or oven drying at 105°-110°C except in the case of organic or heavy clay soils, otherwise it is essentially the same as BS 1377:1961. Lambe (1951) stated that soils containing clay intended for Atterberg limit determinations should not have been dried to a moisture content less than the plastic limit. The next step is normally one of screening through a 0.420 mm sieve and the working of the +0.420 mm fraction with a rubber pestle to break up aggregations, but not discrete particles,and remove as much of the adhering clay as possible. This pulverization stage is not allowed in TEX-101-E (wet method). At this stage all methods can be divided into two groups, those which employ a period of slaking in water of the +40 mesh fraction to thoroughly disintegrate aggregations and remove adhering clay and those which do not. Wet methods include FAA P-210-2.2 (up to 72 hours). AASHO T146-49 (2-24 hrs), ASTM D2217-66 ("until the particle aggregations become soft"), TEX-101-E (2-24 hours) and D.O.T. (boiled for 1 minute). The slaking time normally required is the minimum time required to yield the maximum amount of fines (determined for example by TEX-102-E). Dry methods are AASHO T87-49, ASTM D421-58, D.O.T. (only the wet method permitted after October 1963) and NIRR. Dry methods sometimes employ a procedure involving two stages of sieving with sieves of different sizes and the working with a rubber pestle of each fraction retained (AASHO T87-49, B.S. D1377:1961); probably to ensure both maximum breakdown of aggregations, clay lumps and coatings, and as a protection for the finer sieve.

At this stage the material prepared by a dry method

is/ ...

is ready for use. In the wet methods the slaked material originally retained on the 40 mesh sieve would be washed on the sieve, the -40 mesh fraction allowed to settle, the clear supernatant liquid decanted or siphoned off and the slurry dried at less than 140°F (FAA P-210-2.2, AASHO T146-49, TEX-101-E) or 105-110°C (D.O.T.). In the D.O.T. method a settling time of only about 10 minutes is used, the suspension decanted, boiled to a paste and added to the slurry. In all methods the dried material is again broken up, passed through a 40 mesh sieve if necessary and added to the material obtained by dry screening. In the AASHO T146-49 and TEX-101-E methods the material retained on the 40 mesh sieve after slaking and washing through is dried and rescreened, the fraction passing being added to the other -40 mesh material.

It is apparent that preparation methods vary considerably and could give rise to different soil constants being obtained. Wet methods can normally be expected to yield more clay and thus higher constants than the dry methods, while the effect of drying is to reduce both the liquid limit and plasticity index, the soil effectively moving downwards parallel to the A line on the Casagrande plasticity chart. The effect of drying at temperatures up to 105°C is, however, usually small in the case of inorganic soils, and the plasticity can usually be restored by soaking for several days (Casagrande, 1932). Winslow and Gates (1963), however, found that some inorganic Indiana soils, particularly those containing appreciable quantities of montmorillonite, were quite badly affected even by air drying, let alone oven drying at 105°C. Rehydration periods of up to 24 hours considerably improved matters in both cases, though in neither case did two montmorillonite-rich soils revert completely to their original nondried PIs of 21 and 23. Some experiments conducted by Dr. H.H. Weinert (1969, personal comm.) showed that a rehydration period of

about/ ...

about 2 weeks under atmospheric conditions (or 6-8 hours under vacuum) was necessary to completely restore the original PI (60-70) of a montmorillonitic black clay after heating to temperatures up to 500°C. It should be particularly noted that the FAA specifically state that their wet method modification of AASHO T87 shall be used on caliches, whereas the standard dry method T87 is apparently permitted for all other materials, only for example the AASHO soil constant procedures being specified. In these procedures it is optional whether the dry (T87) or wet (T146) method of preparation is employed. The FAA has been using the same basic caliche specifications and test method since at least 1944, when they were mentioned by O'Fiel. The Texas method TEX-101-E is also a wet method and much caliche has been used in this state. In New Mexico on the other hand, caliche is also used (though possibly to a lesser extent than in Texas), but a wet method is not specified for any material, presumably thus making it optional as to whether AASHO T87 or T146 is employed. In this connection it may be worth mentioning that the materials and testing engineer of the New Mexico State Highway Commission, Mr. T. Cole (1966, private comm.), has stated that no extensive research on caliche has been carried out by the Commission. According to the Highway Research Board (1965, private comm.), most of the work on caliches in the United States has been carried out by the Texas Highway Department.

The effect of soil preparation method on soil constants and mechanical analysis has been studied by Gillette (1951) and McDowell (1966). Gillette's 17 materials were disturbed samples taken from the bases of existing roads and included 5 caliches. The caliches came from two areas in Texas and the remaining samples were from various parts of Texas, Oklahoma and Louisiana. The samples were split up and the wet preparation method AASHO T146-49 carried out by a Texas laboratory and the

dry/ ...

dry method AASHO T87-49 by an Oklahoma and a Louisiana laboratory. Liquid limits of the materials as a group by the wet method varied from 19 to 51 and PIs from 6 to 30. It was found that considerable differences resulted between the Texas laboratory's results using the wet method and both the other laboratories' results using the dry method. Lesser differences between the results of the two laboratories using the dry method were also found. In more detail, the following conclusions can be drawn from a study of Gillette's results:

1) Liquid limits were 2-11 percentage units higher by the wet method than by the dry method in the case of the caliches and 3-14 units higher in the case of the other materials.

2) Plasticity indices were 3-10 units higher by the wet method in the case of the caliches and 2-14 units higher in the case of the other materials.

3) Shrinkage limits were 1-6 units lower by the wet method in the case of the caliches and 6 units lower to 2 units higher in the case of the other materials.

4) In the case of the caliches, the wet method yielded field moisture equivalents and shrinkage ratios up to about 8 units and 0.03 respectively on either side of the dry method. The figures for other materials were up to 6 units higher and 10 units lower and about 0.06 on either side.

5) The differences in the mechanical analyses were also erratic. In the case of all materials neither method produced consistently higher or lower values than the other, in the 10 to 200 mesh size range, though differences of up to 20 percentage units were apparent. The wet method did, however, tend to produce more minus five micron material than the dry

method/ ...

method, so it is not surprising that it yielded the higher liquid limits and plasticity indices.

McDowell's (1966) study employed 42 samples of Texan base and subbase materials, seven of which were caliches. The samples were split in two and one half tested by the Bureau of Public Roads (BPR) and the other half tested by the Texas Highway Department (THD). The BPR employed the AASHO T87 dry and the AASHO T146 wet preparation methods and the THD employed the AASHO T87 dry and the TEX 101 wet preparation methods. The conclusions resulting from the study reported by McDowell are worth giving here in some detail in view of the scarcity of data pertaining to calcretes and the possibility of applying Texan caliche base specifications to Southern Africa. These conclusions are qualitatively mostly those of McDowell (who points out that they are not those of the Bureau of Public Roads), but the caliche details and most of the figures cited are those derived by the author from a study of McDowell's results. They can be listed as follows:

1) The agreement between the liquid limit and plasticity index results obtained by the BPR and the THD operators (using the hand liquid limit method) was fairly good in the case where both tested exactly the same minus 40 mesh material. The THD operators tended to obtain results about two percentage units higher than the BPR operators.

2) On all the materials, the liquid limit determined by the Texan hand method was about two units higher than that determined by the same laboratory using the liquid limit device.

3) The TEX wet preparation method usually caused the PI to be 0 to 2 units higher than that obtained when the AASHO dry method was used. Values -1 to +8

units/ ...

units higher were, however, obtained in the case of the materials in general, while the seven caliches were 0 to 4 units higher. Except for the value -1 above, the above figures were 2 units higher still when the THD operators determined the PI of the material prepared by the TEX wet method and BPR operators determined the PI of the material prepared by the AASHO dry method. This is in good agreement with conclusion(2) above.

4) Allowing for this tendency of the THD operators to obtain higher PIs, the TEX and AASHO wet preparation methods should cause reasonably comparable PIs to be obtained by the same operator.

5) The scatter of PI results on material prepared by the two laboratories by the AASHO dry method was larger than that obtained when the two wet methods were used.

6) The scatter of the values for the percentage passing 40 mesh was about twice as large when the dry method was used than when the two wet methods were used. The BPR operators obtained values varying from -1 to +29 percentage units higher than the THD operators due, McDowell concluded, to the large amount of discretion allowed the operator by AASHO T87 with regard to the degree of pulverization. The BPR operators were particularly prone to obtain high values on the caliches, presumably due to the often friable nature of these materials. The AASHO wet method determined at the BPR did, however, usually result in values 0 to 8 units higher than the TEX wet method (extremes -5 to +13, with caliches -1 to +9), again presumably because of the latitude allowed in the degree of pulverization before slaking in the AASHO method (this is prohibited in the TEX method). The BPR - determined AASHO dry method even tended to yield more minus 40 mesh material than the

TEX/ ...

TEX wet method (usually -1 to +9, but with extremes of -4 to +19, with caliches also -4 to +19 - again showing up their friable nature).

7) McDowell concluded that the AASHO methods yield more coarse soil binder particles than the TEX wet method and suggested that AASHO prohibit pulverization prior to slaking in the AASHO wet method and abolish the AASHO dry method, or somehow define the degree of pulverization to be used, both eminently sensible suggestions as far as the author's experience with calcretes is concerned.

McDowell's results confirm the more limited study of Gillette. General conclusions that can be made at this stage are that it is now possible to assess some of the differences likely to result from the different United States and South African preparation methods, that calcretes should probably not be heated above 140°F (60°C), that they are probably particularly prone to yield excessive minus 40 mesh material in the AASHO T87 and T146 preparation methods and that, like other materials, they are likely to yield higher liquid limits and PIs (and probably linear shrinkages) when prepared by a wet method due to the larger amounts of clay released.

7.2.2 Effect of drying temperature

In view of the fact that the D.O.T. (1958) method requires heating to 105°C, an experiment was carried out to assess the effect of heating at 105 and 50°C on calcrete soil fines. The five calcretes chosen represent nearly all the known clay mineral combinations in calcretes except the types containing kaolinite. A member of the latter group was also supposed to be tested, but insufficient material was available. All except 2223 were used for cement or lime stabilized base.

Two types of heating were employed: heating of

the/ ...

the ordinary air dried material and heating after soaking for 24 hours. As the pH of a calcrete at 105°C is probably at least 10 (due to the loss of CO_2 from the soil moisture on heating and the formation of more OH^- ions), it was thought that wet heat might cause a greater effect than dry heat due to the greater possibility of attack on the clay minerals. The following method was employed: The soil fines (-40 mesh) were obtained by ordinary dry screening and carefully riffled into smaller portions. Only air drying was employed. This air dried material was then tested as such, after heating at 50°C for 36 hours, and after heating at 105°C for 24 hours. Further material was then soaked for 24 hours at about 1.5 times the liquid limit and portions dried at 50°C for 36 hours and at 105°C for 24 hours (the 36 hour drying had to be adopted because 24 hours at 50°C was insufficient). The ordinary NIRR method was employed for the liquid and plastic limits and the linear shrinkage except that all material was soaked for 24 hours at about 1-5 times the liquid limit before testing in place of the usual "overnight" soaking.

The full results of the experiment are given in Table J.1 in Appendix J and only the conclusions cited here. It was found that moist or dry heating at 105°C resulted in every case in a lowering of the liquid limits by up to 2 percentage units, the plasticity indices by 1 to 4 and, generally, in a raising of the plastic and shrinkage limits by up to 3 and 1 respectively. Linear shrinkage was relatively little affected, even allowing for the fact that it is usually only about half the PI. The maximum variation in the linear shrinkage from the air dry value was only 0.7. The behaviour of the volumetric shrinkages, shrinkage ratios, flow indices, toughness indices, absorption limits, and petrifaction degrees was erratic. The effect of heating at 50°C was generally similar to that at 105°C except that the trends were more subdued and the results more erratic, suggesting that the effect of heating at 50°C was becoming subordinate to

sampling/ ...

sampling and experimental error. Sample 2223 was quite badly affected by heating to even 50°C, however. While further work is necessary these limited results tend to support the American practice of allowing heating up to 60°C. The effect of ordinary air drying remains unknown. Most calcretes will be subjected to temperatures up to 70°C and an almost air dry state at some stage during construction or later in the road under the semiarid or arid climate in which they occur. Section 8.13.2 also provides some information as to the possible effect of heating at 105°C on calcretes. Twenty wetting and drying cycles appeared to exert a similar effect on some soil constants.

The reason for this effect is not known. Organic matter is the usual cause of a lowering of liquid limit and PI on heating, but Table C.9 shows that these five calcretes contain less than one percent organic matter. Samples 2114, 2116, 2223, and 2367 do contain some montmorillonite, but nothing like the content of Winslow and Gates' (1963) samples. While the clays in calcretes are all considered to be fully or oversaturated with Ca and/or Mg, it is just possible that the drying might have affected this, as Wooltorton (1954) has stated that a drying cycle is necessary for a reduction in plasticity and shrinkage by ion exchange to be fully effective. Even exchange of Ca for Mg can affect the liquid limit and PI of palygorskite[*] (Table 8.1). A strong possibility is that a development of structure or cementation took place on drying. This hypothesis is supported by the tendency of the shrinkage limit to increase on drying. Heating to 110°C would certainly not cause any decomposition of carbonates, but would cause a reversible water loss from gypsum and other salt hydrates, opaline silica and clay minerals, which do occur in calcretes. Unidentified colloidal matter may also be present in some calcretes. Whatever the reason, it is apparent that some calcretes of roadmaking quality (not the heavy clays so often used in Atterberg experiments) are quite

seriously/ ...

[*] Palygorskite is also known to lose its selective adsorption properties when heated above 88°C and to decrease in surface area above 95°C.

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seriously affected by heating, even to 50°C. It should also be noted that the drop in LL, PI and LS in Table J.1 would probably have been even greater had the samples not been presoaked before testing.

7.2.3 Suggestions for calcretes

It would appear from FAA practice on caliches, Texan practice in general and the two papers cited, that a wet preparation method is desirable, particularly if their relaxed caliche specifications are employed. The D.O.T. wet method with its one minute boiling time is probably the method most frequently employed in South and South West Africa. Whether a one minute boiling is equivalent to the "up to 72 hours" slaking at room temperature of the FAA method is unknown to the author, but it should in any event be superior to the dry methods. It would also appear that caliches are prone to a high degree of particle breakdown when pulverized dry to break up aggregations.

It would also appear that drying at temperatures of 105°C, or possibly even as low as 50°C, is undesirable and the D.O.T. method with its 105-110°C drying temperature and boiling dry procedure is probably introducing errors (probably a lowering of the soil constants), which may possibly not happen with other inorganic Southern African materials (except possibly ferricretes?).

In conclusion then it can be stated that the most desirable soil fines preparation methods for use on calcretes appear to be the wet methods involving drying at less than 140°F (60°C) such as the FAA P-210-2.2, TEX-101-E and AASHO T146-49. The Texas method is recommended. This is similar to AASHO T146 except that a preliminary dry pulverization is not permitted. Dry methods such as AASHO T87-49 (essentially the same as ASTM D421-58), BS 1377:1961 and NIRR must be regarded

as/ ...

as undesirable, though no actual figures are available comparing the various methods on South African materials. In view of the wide usage in South Africa of the D.O.T. method, a full investigation of the effect on calcretes of drying at 110°C and boiling for one minute instead of prolonged cold water slaking would be justified. Dry methods are also employed in South Africa, and it would also therefore be advisable to compare the two methods on calcretes. Some information may well be available in the records of roads departments, since some experimentation was presumably carried out before the D.O.T. method was instituted.

7.3 LIQUID LIMIT

The liquid limit (LL) of a soil is the moisture content at which the soil changes from a plastic to a liquid state. In mechanical methods it is the moisture content required to just cause shear failure of the remoulded soil at 25 blows in the standard liquid limit device. It is thus the moisture content at which the remoulded soil possesses a very small but definite shear strength (Casagrande, 1932) of 20-30 g/sq cm (0.28-0.42 psi) (Casagrande, 1958). Norman (1958) found values ranging between 8 g/sq cm (0.11 psi) and 16 g/sq cm (0.23 psi) when the liquid limit was determined using a device with a BS hardness base and 11-23 g/sq cm (0.16-0.32 psi) when a device with an American Micarta 221 base was used on the five clays used (liquid limits 41-72). Wiseman and Teferra (1965) found values ranging from 0.2-1.0 psi, the shear strength decreasing with the liquid limit. Each blow of the liquid limit device is equivalent to a shear

strength/ ...

strength of 1 g/sq cm (Casagrande, in Seed et al, 1964). The shear strength of a remoulded soil at the liquid limit is thus reasonably constant at around half a psi. According to Croney and Coleman (1954, in Seed et al; 1964) the negative pore water pressure at the liquid limit is about 4 g/sq cm (equivalent to a pF of 0.6). Seed et al did not state whether this was on the wetting or drying curve. Marsh (1967) obtained a pF of 0.04 (with a range from -0.9 to +0.5) for 13 Keuper Marls on the continuously disturbed line. It would appear that this was on the drying curve though this was not stated. In this connection it should be noted that the BS 1377 liquid limit method normally proceeds from the drier to the wetter state, so that a wetting-up pF is required.

The liquid limit is also held to represent the adsorption plus capillary capacity of a soil (Wooltorton, 1954).

A useful discussion of the history of the Atterberg limits has been given by Bauer (1959).

7.3.1 Comparison of methods in use

Liquid limit methods in use can firstly be differentiated on the basis of whether they employ a prior period of wet equilibration or not. Methods employing a prior soaking period are the new (October 1963) D.O.T. method (12 hours near LL) and the NIRR method (overnight at about 1.5 L.L.). The AASHO T89-60, the similar ASTM D423-66 methods (including the one-point and hand alternatives), the TEX-104-E mechanical and hand methods and the BS 1377:1961 standard and one point methods all use dry soil fines, though the latter standard mentions that a prior 24 hour wet equilibration period is advisable in the case of certain clayey soils.

The next step is that of mixing the dry soil with

water/ ...

water or remixing the previously seasoned soil. Specified mixing times vary from "several minutes" (TEX), "thoroughly" (ASTM and AASHO), at least five minutes per water increment (BS), 10 minutes (D.O.T.), and at least 10 minutes both before and after overnight seasoning (NIRR) to 40 minutes ("certain tropical soils", BS).

It will be noticed that the NIRR and D.O.T. methods must now start at a moisture content close to or above the liquid limit, while all the other methods (except the one point methods) start at a moisture content below the liquid limit.

After mixing, a portion of the wet sample is placed in the bowl of a standard liquid limit device. It should be noted here that the devices used in Britain (see BS 1377) and the United States (see ASTM 423 or AASHO T89) are not quite identical in all respects. Apart from small differences in dimensions, the BS 1377:1961 device is the only one which specifies the hardness of the rubber base. It can normally be recognised by the fact that, unlike the American device, it has no rubber feet and the base is made of more thinly laminated, softer rubber. The two devices are known to give slightly different results on the same sample. In experiments reported by Norman (1958) the hardness of the base was found to be important with, as might be expected, the liquid limit decreasing with increasing hardness of the base. A BS device fitted with an American Micarta 221 base and rubber feet was found to yield results 3-4 units lower than a device conforming to BS 1377:1948 on the 5 clays tested (liquid limits 41-72). The Westinghouse Micarta 221A base is the harder of the two bases and has been used by Casagrande (1958) since 1949 as a substitute for hard rubber. It is however, apparent, from the statements made by both these authors that the bases of both British and American devices are variable in hardness.

Similar/ ...

Similar results to those obtained by Norman were obtained in an experiment conducted by the S.W.A. Roads Department, wherein a BS device was found to yield liquid limits consistently higher by 1 to 5 units than an American device on 4 samples possessing liquid limits from 21 to 38 by the BS device (Mr. J.H. Caiger, 1968; personal comm.). The effect of the two devices on seven calcretes is shown in Table J.2 (Appendix J). In this case the BS device yielded results 1-2 units higher than the ASTM device. The hardnesses of the bases are given in Table J.3. The rubber feet of the ASTM device also make a difference (and are also known to vary in size). Dawson (1959) found that removal of the feet on his ASTM device reduced the liquid limit by 1 to 3 units depending on the hardness of the material the device was then placed on (glass or foam rubber). The American device is probably normally used in South Africa (see drawings in D.O.T., 1958), but the devices used at the NIRR and in this study are Clockhouse Type 7001, said to be British Standard types, though many dimensions do not correspond with BS 1377:1961 .

The next step involves cutting a groove in the wet soil cake with a standard grooving tool. The BS method uses the Casagrande grooving tool and all the other methods examined, including the D.O.T., employ the ASTM type. At the NIRR the ASTM type is also normally used, though the Casagrande type might be employed on sands and heavy clays. Casagrande (1958) has stated that the ASTM grooving tool "... is not satisfactory because it does not control the height of the groove with the necessary accuracy." The Casagrande and Hovanyi tools (very similar) possess sides which cut the soil cake to give an 8 mm groove depth whatever the thickness of soil placed in the cup. The ASTM tool is supposed to cut a 10 mm groove depth, but this also depends on the thickness of soil cake, since it does not

cut/ ...

cut off the top of the cake as the other two tools do. A study by Mitchell (1959) on a total of 32 soils from three different laboratories with liquid limits (using the Casagrande tool) varying between 13.8 and 54.7 showed that the Casagrande tool always yielded higher liquid limits by 0.0 to 2.5 percentage units than the ASTM type, probably largely due to the greater groove depth usually obtained with the ASTM tool. The difference did not appear to depend on the absolute value of the liquid limit (for example, the material which yielded the difference of 2.5 had a Casagrande tool value of 29.8). The Hovanyi tool was only tested on seven soils but yielded results essentially the same as the Casagrande tool. Mitchell concluded that the three tools should not be interchanged when testing for compliance with specifications. The ASTM type would probably be normally employed on the average road material in this country.

After cutting the groove the device is turned at the rate of two taps a second in all methods until the groove closes over a distance of about one centimetre to half an inch. A moisture content sample is then taken from the sheared material. In the one point methods the procedure is merely repeated at the same moisture content and after the moisture content is known the liquid limit may be read off from a chart, nomogram or special slide rule (see for example AASHO, BS and the ASTM symposium). In the AASHO method it is stated that a point between 22 and 28 blows yields a result with an accuracy comparable to the normal 3 point method, while a point between 15 and 40 blows yields an accuracy of $\pm$ 5% of the true LL. The classical method is the flow curve method described in detail by Casagrande (1932), employing at least 3 points in the range 15-35 blows (AASHO and ASTM) or at least 4 points in the range 10-50 blows (BS and NIRR). The major, and it is thought, an important difference between

the/ ...

the two methods is that the ASTM, AASHO and BS methods proceed from the drier to the wetter state, while the NIRR method employs mixing and drying under a lamp to proceed from the wetter to a drier state. In the Texas methods, dry soil or water is added until the groove closes at between 20 and 30 blows in the mechanical method, after which the liquid limit is estimated from a chart of flow curves. In the hand method, dry soil or water is added until the liquid limit is reached. In the D.O.T. method the moisture content is adjusted by adding more water or mixing until the liquid limit is reached. In other words, a sample could go through several cycles of rewetting and drying before reaching the liquid limit, though it is stated that it is better to work from a slightly too wet condition to the liquid limit.

Some of the factors affecting liquid limit results have been discussed by Casagrande (1932) and the ASTM symposium on Atterberg limits (1959). A recent statement in the AASHO and ASTM test procedures under referee tests is that results are affected by the time taken to make the test, the initial moisture content, and by the addition of dry soil to the seasoned sample. A strict procedure for referee tests is laid down. It is abundantly apparent from the literature that even small differences in even the mechanical methods cause significant differences in the test result when testing for compliance with specifications. Even the direction of grooving can significantly affect the result.

7.3.2 Accuracy and reliability of liquid limit determination on calcretes

The FAA and Texas procedures do not specify any special procedure to be followed when calcretes are tested. The FAA use the AASHO method and Texas only specify a one point and a hand method in their 1966

manual/ ...

manual and a hand method alone in their 1953 manual. The one point method appears to employ the chart developed by the Washington State Highway Department (see AASHO T89-60), though this is not stated. To the author's knowledge calcrete does not occur in any quantity in this state of the U.S.A.

Figures 7.1-7.5 show the flow curves of some calcretes and a few calcareous soils (891, 893 and 916) as determined by the NIRR method. It can be seen that in some cases the scatter of points about the best straight flow curve that could be drawn is rather large, giving rise to alternative possible flow curves. Some of these have been drawn in. Table 7.1 shows a detailed analysis of the flow curves (plus a few not shown in the figures). Columns two and three represent an attempt to assess the range over which the liquid limit could reasonably be interpreted from the points plotted. In doing this, preference was given to the points closer to 25 blows and apparently erratic points were rejected. The mean range was found to be 0.5, and in about 75% of cases this figure was bettered. A larger range would have been obtained if all the points determined for each sample had been used. Occasional samples gave rather ambiguous results. The most probable value for the liquid limit is shown in column 4 and the following two columns represent the maximum probable error of this figure. The mean maximum probable error is seen to be $^{+0.3}_{-0.2}$ with a standard deviation of $^{+1.0}_{-0.3}$. The standard deviation is, however, excessively influenced by a few very abnormal results and is misleading in this case. Inspection of the results shows that in about 85% of cases the maximum probable error was $^{+0.3}_{-0.2}$. In about 93% of cases the result fell within the range ± 0.5. The author is unaware of a similar statistical treatment being available for materials other than calcretes. While comparison is thus not possible, it would appear that this sort of accuracy

can/ ...

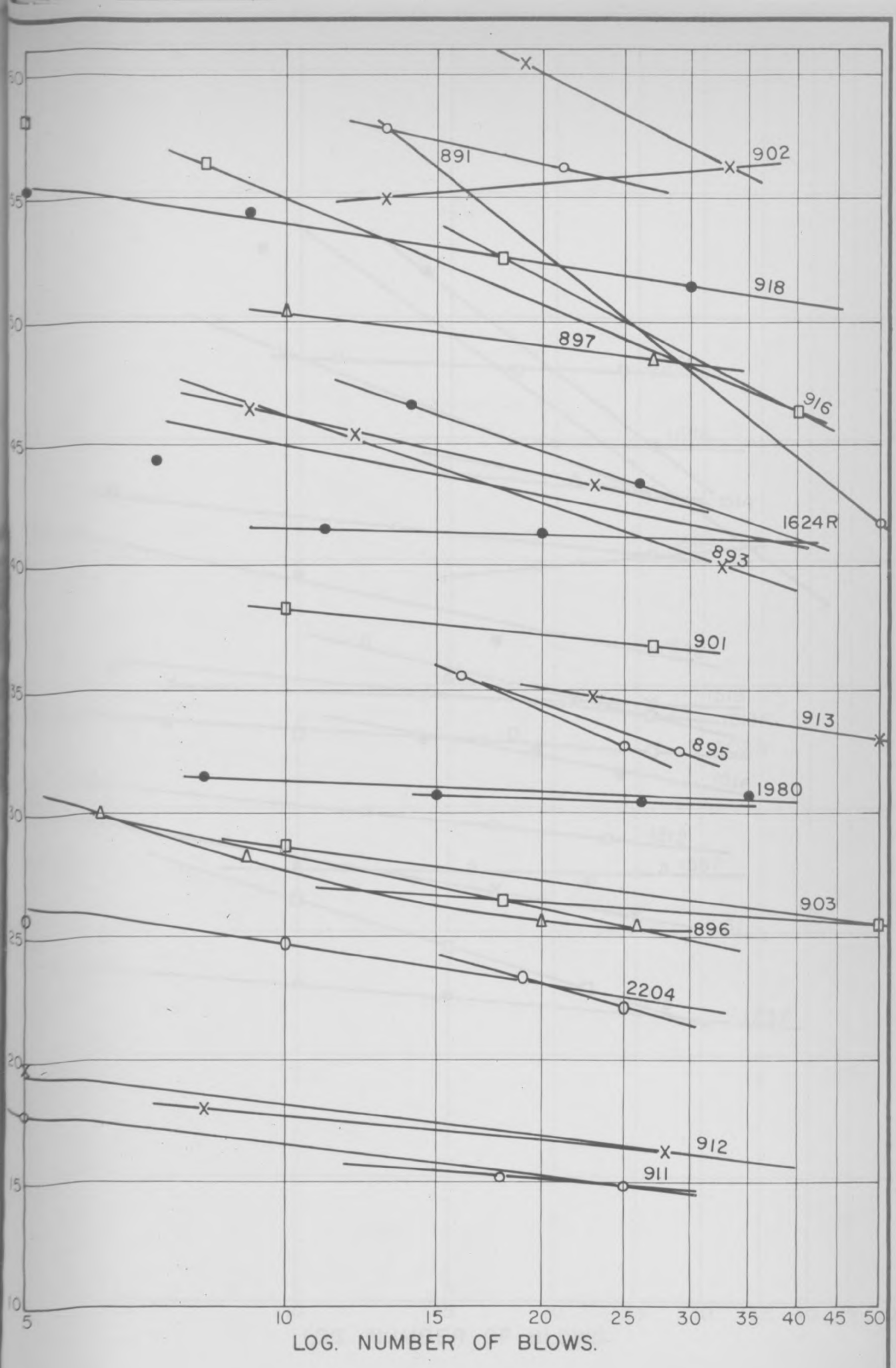


FIGURE 7·1

Some calcrete flow curves

RG4-3181

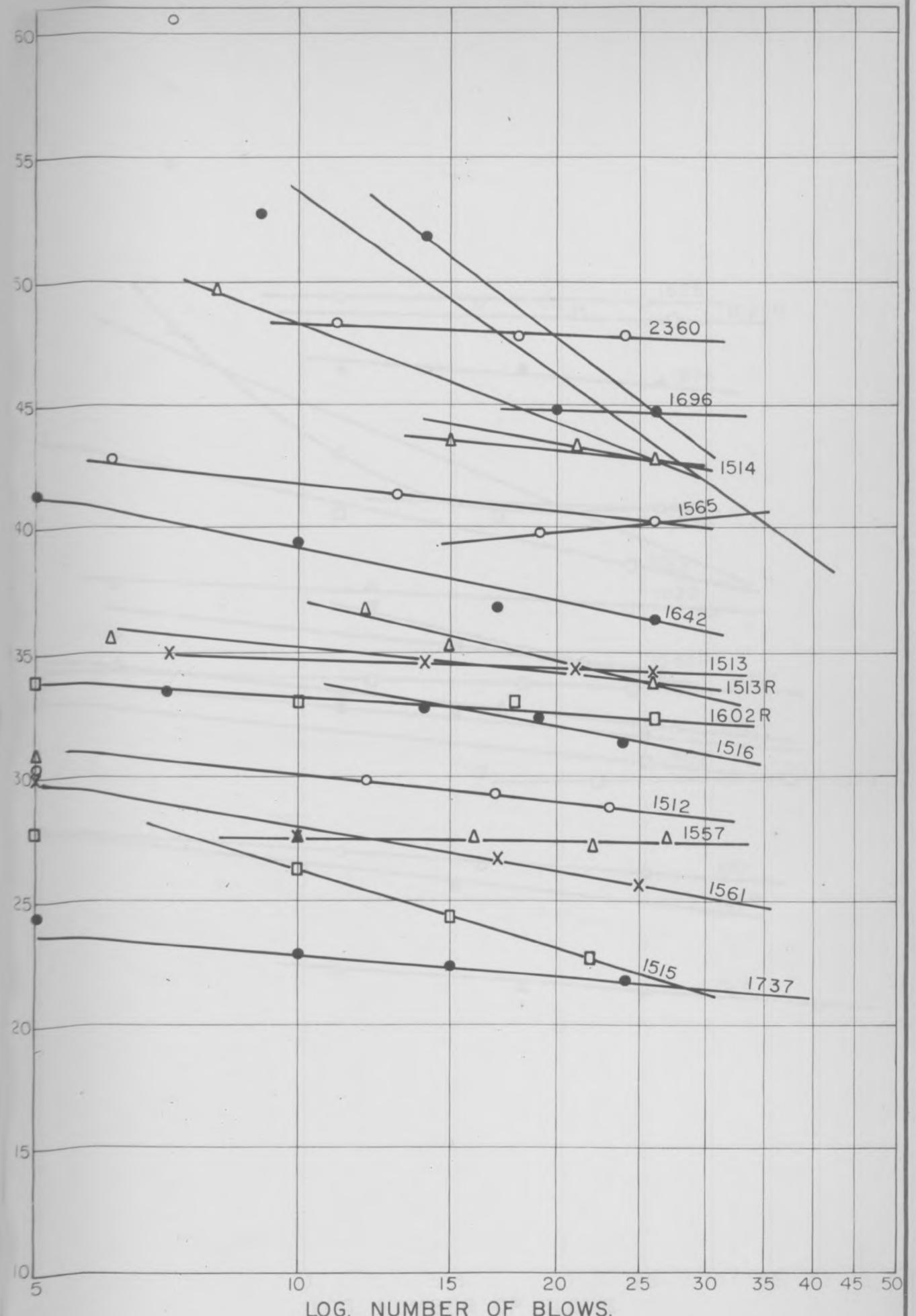


FIGURE 7·2

Some calcrete flow curves

RG4-3182

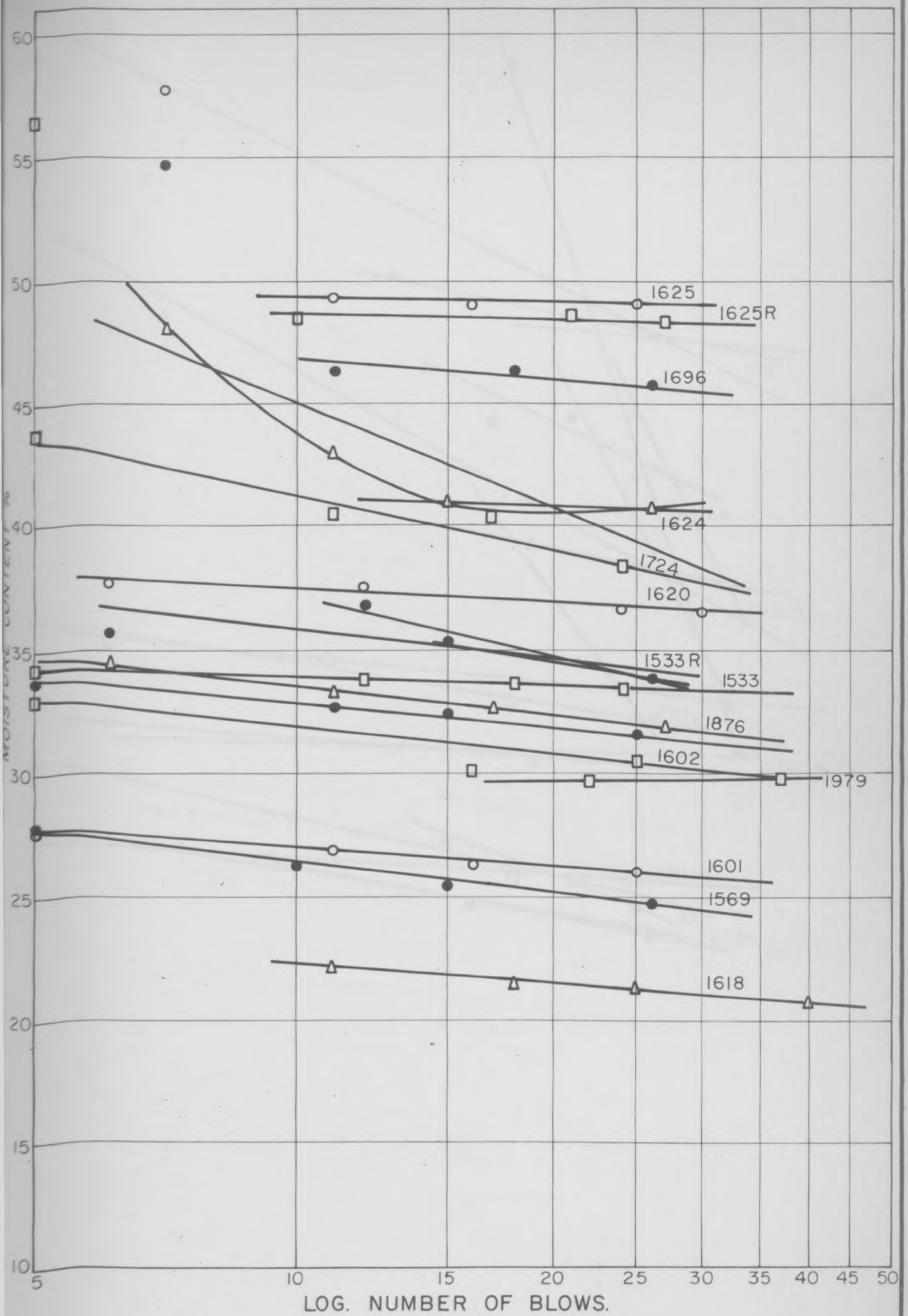


FIGURE 7·3

Some calcrete flow curves

RG4-3183

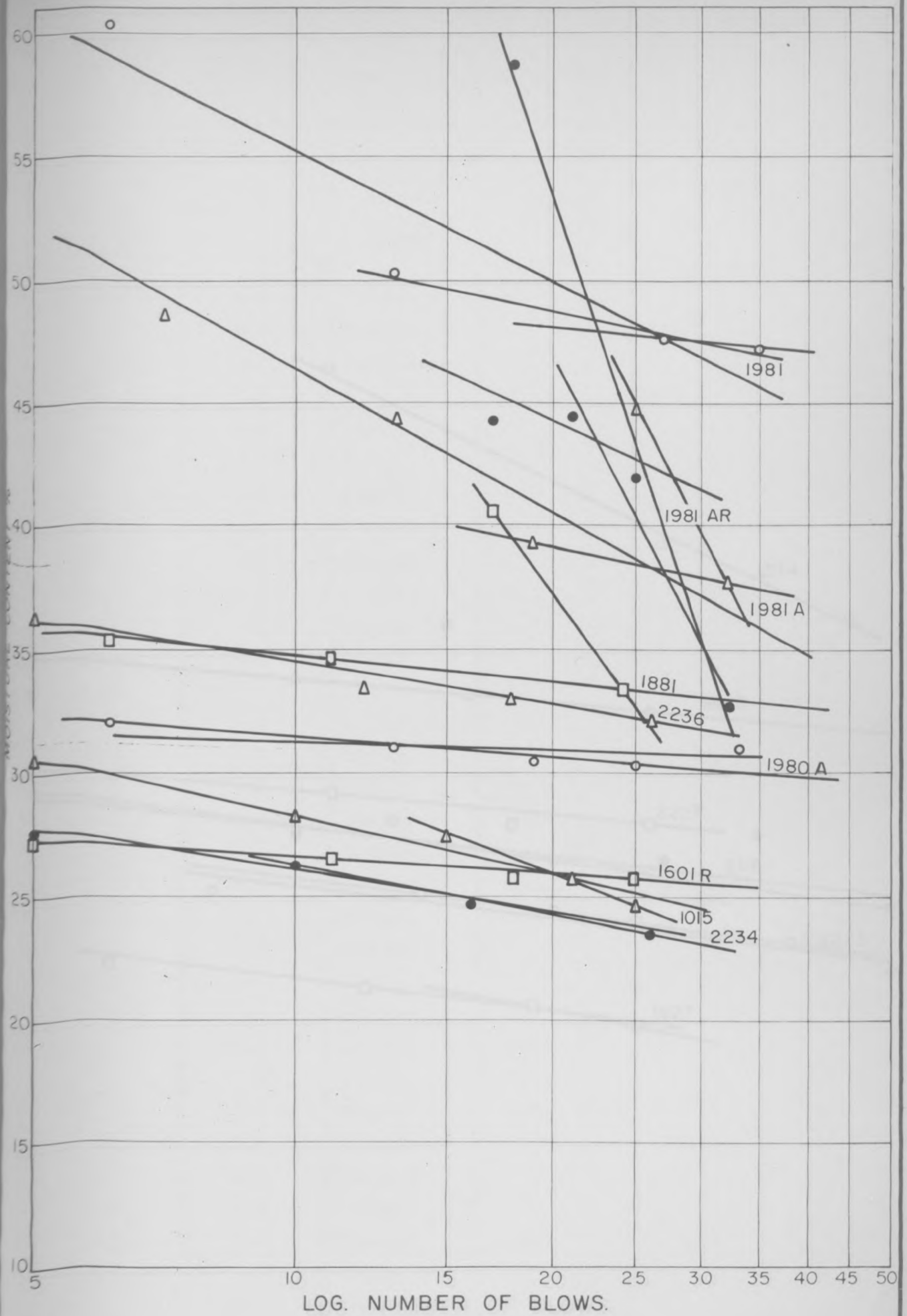


FIGURE 7·4

Some calcrete flow curves

RG4 - 3184

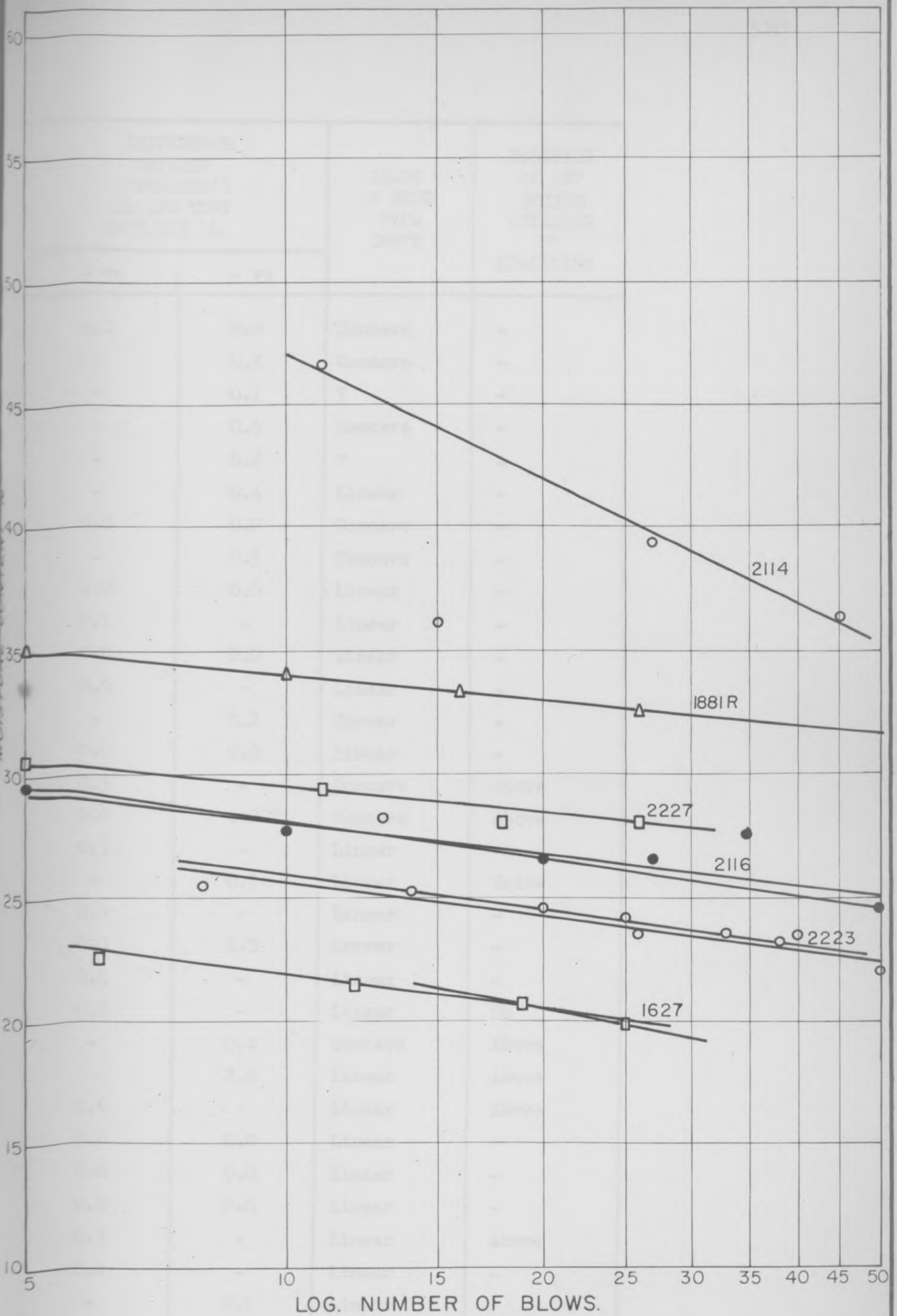


FIGURE 7·5

Some calcrete flow curves

RG4-3185

DIFFERENCE BETWEEN OPERATOR'S LL AND MOST PROBABLE LL.		SHAPE OF BEST FLOW CURVE	POSITION OF ANY POINTS OBTAINED BY REWETTING
+ ve	- ve		
0.0	0.0	Concave	-
-	0.3	Concave	-
-	0.1	?	-
-	0.5	Concave	-
-	0.2	?	-
-	0.4	Linear	-
0.0	0.0	Concave	-
-	0.3	Concave	-
0.0	0.0	Linear	-
0.1	-	Linear	-
0.0	0.0	Linear	-
0.9	-	Linear	-
-	0.2	Convex	-
0.0	0.0	Linear	-
0.3	-	Concave	Above
0.0	0.0	Concave	Above
0.3	-	Linear	-
-	0.5	Linear	Below
0.9	-	Linear	-
0.0	0.0	Convex	-
0.5	-	Linear	-
0.3	-	Linear	On
-	0.4	Concave	Above
-	2.4	Linear	Above
0.6	-	Linear	Above
0.0	0.0	Linear	-
0.0	0.0	Linear	-
0.0	0.0	Linear	-
0.3	-	Linear	Above
0.2	-	Linear	-
-	0.2	Linear	-
0.1	-	Linear	-
-	0.7	Linear	Above

TABLE 7.1 ANALYSIS OF CALCRETE FLOW CURVES

SAMPLE NUMBER	RANGE OF POSSIBLE LIQUID LIMIT	DIFFERENCE	MOST PROBABLE LIQUID LIMIT	MAX. POSITIVE ERROR	MAX. NEGATI ERROR
895	32.7 - 33.2	0.5	33.0	0.2	0.3
896	25.4 - 25.6	0.2	25.5	0.1	0.1
897	48.6 - 48.8	0.2	48.7	0.1	0.1
900	81.7 - 82.3	0.5	82.0	0.3	0.3
901	36.9 - 37.1	0.2	37.0	0.1	0.1
902	55.9 - 58.4	1.5	58.4	0.0	1.5
911	14.8 - 15.0	0.2	14.9	0.1	0.1
912	16.4 - 16.5	0.1	16.5	0.0	0.1
913	34.4 - 34.6	0.2	34.5	0.1	0.1
918	51.8 - 52.0	0.2	51.9	0.1	0.1
903	26.2 - 26.8	0.6	26.5	0.3	0.3
1015	24.7 - 25.1	0.4	24.7	0.4	0.0
1512	28.6 - 28.8	0.2	28.7	0.1	0.1
1513	34.1 - 34.3	0.2	34.2	0.1	0.1
1514	42.9 - 43.0	0.1	43.0	0.0	0.1
1557	27.3 - 27.5	0.2	27.4	0.1	0.1
1561	25.6 - 25.8	0.2	25.7	0.1	0.1
1565	40.1 - 40.3	0.2	40.3	0.0	0.2
1515	22.0 - 22.2	0.2	22.1	0.1	0.1
1516	31.2 - 31.4	0.2	31.3	0.1	0.1
1513R	33.7 - 33.9	0.2	33.8	0.1	0.1
1601	25.9 - 26.1	0.2	26.0	0.1	0.1
1624	39.3 - 40.8	1.5	40.8	0.0	1.5
1625	48.9 - 49.1	0.2	49.0	0.1	0.1
1696	45.7 - 45.9	0.2	45.8	0.1	0.1
1602	31.4 - 31.6	0.2	31.5	0.1	0.1
1533	33.3 - 33.5	0.2	33.4	0.1	0.1
1620	36.7 - 36.9	0.2	36.8	0.1	0.1
1533R	33.9 - 34.2	0.3	34.0	0.2	0.1
1618	21.1 - 21.3	0.2	21.2	0.1	0.1
1737	21.7 - 21.9	0.2	21.8	0.1	0.1
1876	31.8 - 32.0	0.2	31.9	0.1	0.1
1696R	43.9 - 45.1	1.2	44.7	0.4	0.1

TABLE 7.1 (Continued) ANALYSIS OF CALCRETE FLOW CURVES

SAMPLE NUMBER	RANGE OF POSSIBLE LIQUID LIMIT	DIFFERENCE	MOST PROBABLE LIQUID LIMIT	MAX. POSITIVE ERROR	MAX. NEGATIVE ERROR	
1979	29.7 - 30.5	0.8	29.7	0.8	0.0	
1980	30.5 - 30.8	0.3	30.5	0.3	0.0	
1980A	30.3 - 30.8	0.5	30.3	0.5	0.0	
1981	47.7 - 48.2	0.5	48.0	0.2	0.3	
1981A	38.5 - 45.8	7.3	38.7	7.1	0.2	
1981AR	40.5 - 43.5	3.0	42.5	2.0	1.0	
1881	32.7 - 33.3	0.6	33.3	0.0	0.6	
2234	23.6 - 23.8	0.2	23.6	0.2	0.0	
2236	32.1 - 32.3	0.2	32.1	0.2	0.0	
2360	47.6 - 47.8	0.2	47.7	0.1	0.1	
1569	24.8 - 25.0	0.2	24.9	0.1	0.1	
1624R	41.2 - 43.6	2.4	41.2	2.4	0.0	
1601R	25.7 - 25.9	0.2	25.8	0.1	0.1	
1602	32.2 - 32.4	0.2	32.3	0.1	0.1	
1625R	48.2 - 48.4	0.2	48.3	0.1	0.1	
1627	19.9 - 20.0	0.1	20.0	0.0	0.1	
2114	40.3 - 40.5	0.2	40.4	0.1	0.1	
2116	26.1 - 26.3	0.2	26.1	0.2	0.0	
2223	23.9 - 24.0	0.1	24.0	0.0	0.1	
2204	22.1 - 22.6	0.5	22.1	0.5	0.0	
1724	38.2 - 38.4	0.2	38.3	0.1	0.1	
1642	36.4 - 36.6	0.2	36.5	0.1	0.1	
1658	26.0 - 26.2	0.2	26.2	0.0	0.2	
2225	26.0 - 26.5	0.5	26.3	0.2	0.3	
2226	26.2 - 26.4	0.2	26.3	0.1	0.1	
2227	28.0 - 28.2	0.2	28.1	0.1	0.1	
MEAN		0.52	33.9	0.33	0.19	3
STANDARD DEVIATION		1.03	11.3	0.97	0.30	1
% STANDARD DEVIATION		198	33.2	293	158	3
RANGE		0.1 - 7.3	14.9 - 82.0	0.1 - 7.1	0.1 - 1.5	1
NUMBER OF RESULTS		59	59	59	59	5

†OPERATOR: Mr. E. Nash

OPERATOR: Mr. T.E. O'Connor (all other operator's liquid limits)

*OPERATOR:

†A zero dif

can probably be regarded as adequate. It is emphasised, however, that these results were obtained by what is considered to be an acceptable procedure of inspection and rejection of apparently erratic points. Greater errors are possible if the best straight line is merely drawn through all the points determined. Columns 7, 8 and 9 attempt to assess this. Column 7 shows the liquid limit as interpreted by the operator carrying out the determination. The next two columns show the difference between this and the most probable value shown in column 4. It can be seen that the operator's estimates are almost equally distributed on either side of the most probable value, or in other words there is no marked tendency for a consistently higher or lower value to be obtained by merely taking the best straight line than that obtained by a careful inspection and rejection of erratic points. In 31 cases out of 59 (53%) the operators' value was outside the maximum probable error considered reasonable. The actual magnitude of error likely is, however, in most cases not serious and in 80% of cases (47 out of 59) the operator obtained a liquid limit which was within $\pm$ 0.5 units of the most probable value. Errors of up to 2.5 units were found.

It is also of interest to note that in 10 cases out of 59 (17%) the best flow curve that could be drawn through the points was not linear on the semilog plot but a curve, concave upwards. Samples 896 and 1624 show this to the most marked extent. This tendency is not generally marked but could possibly affect the liquid limit in some cases. Which for example is the best straight line (and the true liquid limit) in the case of these two samples? If only the first three points are used in each case, liquid limits lower by $1\frac{1}{2}$ and about 4 respectively are obtained. The significance of a concave flow curve is unknown to the author, but it is suggested that it may be due to cementation during the drying inherent in the NIRR

liquid/ ...

liquid limit method. Rewetting and remixing of the dried out material has also contributed in some cases. This aspect will be considered in more detail presently. A few cases of convex flow curves are also apparent, though even the most marked case (2204) is much less marked than the concave curve of 1624, and this is probably not significant.

The scatter of the points about the flow curves was investigated further by examining the original records of the liquid limit determinations. It was found that wherever the test did not proceed smoothly from the wetter to the drier condition, but the material was rewetted to obtain a point to fill a gap, that point seldom fell on the line drawn through the other points (3 out of 22 cases), but nearly always fell above the line (17 cases out of 22, or 77%). Only in 2 cases out of 22 did points fall below the line. This could either be due to insufficient mixing after rewetting or to cementation during the drying inherent in the NIRR method, the mixing again being insufficient to completely annul all cementation. In either case this phenomenon holds serious possible consequences for the D.O.T. method, which involves drying and/or rewetting and remixing until the material flows at 25 blows. It is plain from Table 7.1 that this procedure is likely to result in liquid limits which are too high, perhaps by several units in some cases. In the NIRR method it also contributes towards the concave flow curves. The latter method does state that "the soil must be tested from wet to dry conditions" but this should be amended to specifically forbid rewetting.

7.3.3 Suggestions for calcretes

The effect of presoaking calcretes before carrying out the liquid limit determination has not been experimentally assessed. A 12 hour soaking period is in any event now standard in the D.O.T. method subsequent to October

1963,/ ...

1963, though just how widely this is followed is unknown. A letter from the Cape Roads Department on NIRR file RR/A35 stated that their testing of 89 samples (including dolerites and calcretes) had led to the finding that soaking for 24 hours resulted in the liquid limits of about half of the samples increasing by up to 2.5 units and about half decreasing by the same amount. A few samples exhibited no change. Presoaking was then abandoned by the Cape Roads Department. The delay due to soaking would be a serious hindrance to field laboratories engaged in construction control. A similar soaking period is in use at NIRR and has been suggested as an improvement to the ASTM method D 423 by Winslow and Gates (1963). This suggestion has not been adopted in the latest (1968) book of ASTM standards.

The effect of mixing time has also not been experimentally assessed. This will probably only be serious in cases where friable particles are present. The mixing time specified, such as the 10 minutes of the D.O.T. method, should be adhered to.

The effect of different liquid limit grooving tools on calcrete liquid limits is unknown, but should be comparable with other materials.

The NIRR flow curve method proceeding from the wetter to the drier state is considered to yield reliable calcrete liquid limits in all cases provided rewetting is prohibited and ordinary common sense employed in the selection of the best points to use in drawing the flow curve. In view of the rewetting effect and the occasional concave flow curve obtained on drying, it is considered that the D.O.T. method will tend to yield results which are too high (by several units in many cases). The extent of this effect will depend on whether the liquid limit is approached from the wetter or the drier state and the number of times rewetting has to be employed. Some

further/ ...

further work on this aspect would be justified. Several cycles of flow curves should be determined (a) by the usual NIRR method, (b) by progressively rewetting the same material and (c) by drying in the usual way again. Either some sort of a closed hysteresis loop with two roughly parallel sides something like a pF-moisture content loop or an open zig zag should be obtained, resulting in an increase of liquid limit with each cycle, presumably until some limiting value is reached. Whether the concave flow curve and rewetting effects are general phenomena or confined to calcretes is unknown.

The difference between liquid limits obtained by a flow curve method which involves proceeding from the wetter to the drier state and one which involves proceeding from the drier to the wetter state is unknown. If all other factors were equal, owing to the fact that, at the same moisture content, the pF and consequently also the shear strength, of a soil is higher when drying out than when wetting up, the method proceeding from the wetter to the drier state should tend to yield the higher liquid limit.

The application of the one point methods to calcretes has not been assessed. This could be readily done, however, by comparison of the best flow curves in Figures 7.1 - 7.5 with the Washington chart given in the TEX-104-E and AASHO T 89-60 methods and the other flow indices assumed by the other one point methods described in AASHO T 89-69, ASTM D 423 - 66 and BS 1377:1961.

7.4 PLASTIC LIMIT

The plastic limit is the moisture content at the boundary between the semi-solid and plastic states. In practice it is defined as the lowest moisture content at which the soil can be rolled into 1/8 inch diameter threads without the latter breaking up. According to

Wooltorton (1954) this represents roughly the minimum moisture content at which capillary moisture appears. According to Uppal (1966) the plastic limit of soils with plastic limits of 17 - 34% compacted to a bulk density of 1.6g/cc corresponds to a pF of 0.5 on the wetting curve and 1.5 on the drying curve. Marsh (1967) obtained a mean pF value of 3.06 (with a range from 2.7 to 3.6) for 13 Keuper marls on the continuously disturbed line - apparently while drying. Unlike the liquid limit, all soils do not possess the same shear strength at the plastic limit. According to the three figures available (Skempton and Northey, 1952) it seems to vary from about 10 (PL=32) to about 20 psi (PL=16), increasing with decreasing plastic limit and to be about 100 times the shear strength of the same soil at the liquid limit. A relative indication of the shear strength at the plastic limit may also be obtained from the flow index and the toughness index.

7.4.1 Comparison of methods

The AASHO T 90-61, ASTM D 424-54, D.O.T., TEX-105-E, BS 1377:1961 and the NIRR methods are all comparable though small differences do exist. Texas for example uses added dry or wet soil to adjust the moisture content and a plastic-faced plywood or linoleum surface instead of a ground glass plate, while the ASTM and AASHO methods specify the rate at which the thread shall be rolled. The FAA specifies the AASHO method.

The effect of these slight differences in procedure on the results obtained is unknown to the author, but they are almost certainly slight.

7.4.2 Application to calcretes

Several persons engaged in soil testing in South and South West Africa have mentioned that difficulty is often experienced in determining the plastic limits

of/ ...

of calcretes. This was usually attributed to the high silt content of many calcretes.

It is unfortunate that the plastic limit determinations for this project were not made in duplicate as they should have been (NIRR method C). The results would have yielded a useful assessment of the operator reproducibility on calcretes. Some work should be carried out on both the operator variability (i.e. the variability between different operators) and the operator reproducibility (i.e. the ability of a single operator to repeat his results) of plastic limit determinations on calcretes. Information for other materials is available in the literature.

7.5 PLASTICITY INDEX

The plasticity index (PI) is normally defined as the range of moisture content over which the soil is in a plastic state. This is of course numerically equal to the difference between the liquid and plastic limits. In view of what has been said regarding the liquid and plastic limits, perhaps the plasticity index should be more strictly defined as the range of moisture content over which the soil is in a plastic state when proceeding from the semisolid to the liquid states or vice versa. In all probability the two are slightly different. All the plastic limit methods examined would normally determine the plastic limit applicable to the drying out condition, but liquid limit methods vary in this respect. If this concept is valid, the NIRR method would determine the PI applicable to the drying out condition (LL→PL) while the other flow curve methods (ASTM, AASHO and BS) would determine the liquid limit applicable to the wetting up condition (PL→LL) and thus a PI intermediate between the drying out and the wetting up condition. The D.O.T. liquid limit method can involve wetting or drying and even several cycles of wetting and drying, and the PI can be expected to reflect this. Wooltorton (1947; in Wooltorton, 1954) has made a

most/ ...

most important contribution to the understanding of what a PI means when he stated that it is numerically equal to the volume change of 100 g of dry soil fines "when drying from the liquid limit to the plastic limit as illustrated by the shrinkage curve ....."

According to the Road Research Laboratory (1952), "the liquid and plastic limits are both dependent on the amount and type of clay in a soil, but the plasticity index is generally only dependent on the amount of clay present".

7.5.1 Factors influencing plasticity index determinations on calcretes

As the plasticity index is determined from the results of liquid limit and plastic limit determinations, it is plain that factors affecting these tests must also affect the PI. Apart from the personal factor, the factors affecting PI determinations on calcretes and probably on materials in general can thus be listed as follows:

1) Those arising from the preparation of the soil fines

   a) Whether dried from the natural moisture content.

   b) Drying temperature and possibly duration of this.

   c) Dry or wet method.

2) Those arising from the liquid limit determination

   a) Presoaked or non-presoaked and duration of the former.

   b) Mixing time and effort.

   c) Liquid limit device (dimensions and elastic constants of the base) and grooving tool, including accuracy of the height of drop,

weight/ ...

weight of cup, wear, cleanliness (even a drop of water at the contact point between the cup and base affects the results) and direction of grooving.

d) Relative humidity and temperature at the time of test.

e) Weight of soil paste in the cup.

f) Direction of moisture change (i.e. wetting or drying) and possibly the number of wetting and drying cycles (if any).

g) Addition of dry soil to the previously seasoned soil.

h) Soluble salts and gypsum (in material and/or in water if distilled water is not used) - cementation on drying out, flocculation, dispersion, solution, ion exchange and loss of moisture on heating.

i) Accuracy of moisture content determination (sometimes a problem in field laboratories).

3) Those arising from the plastic limit determination

Probably similar to 2 a), b), d), f), g), h) and i) above.

The practical quantitative importance of all of these factors is unknown, but under the appropriate circumstances all of them could possibly give rise to serious errors. Some of these factors have been discussed by Casagrande (1932). He found factors 1a, 1b, 2b, 2c and 2d (humidity only) to be important with some soils. See also the ASTM symposium on Atterbergs (1959). The most severe test method (i.e. that yielding the highest PI) for use on roadmaking materials would be that employing a wet

preparation/ ...

preparation method involving no or at most only air drying and presoaking. Even air drying is known to affect some soils. The most accurate liquid limit method will be a flow curve method employing at least four points, since one point is usually badly out.

Work on the effect of the D.O.T. liquid limit method, drying technique, wet versus dry preparation methods, different liquid limit devices and the operator variability and reproducibility in plastic limit determinations is particularly justified, since these are probably the most important factors in practice. It should be noted once again that the FAA specifically state in their caliche base course specification P.210 that their wet sample preparation method shall be used on caliches and that the material shall not be dried at temperatures exceeding 140°F (60°C).

7.6 BAR LINEAR SHRINKAGE

The linear shrinkage (LS) is the decrease in one dimension of a soil mass expressed as a percentage of the original dimension when the moisture content is reduced from the liquid limit to the shrinkage limit. The shrinkage limit is the maximum moisture content at which a reduction in moisture content does not cause any further shrinkage. The above definition of the linear shrinkage is the one used in Southern Africa and in Texas, but other initial moisture contents are also sometimes employed (e.g. the field moisture equivalent in the U.S.A.-D.O.T., 1958; AASHO, T92-60).

In practice, the linear shrinkage may either be calculated from the volumetric shrinkage or, apparently somewhat less accurately (Heidema, 1957; TEX-107-E), directly determined by the bar method. The volumetric shrinkage Vs is equal to $(w_1 - SL) R$ where w_1 is the initial moisture content (usually the liquid limit in

South/ ...

South Africa), SL is the shrinkage limit and R is the shrinkage ratio. The linear shrinkage Ls is then 100 $(1 - \sqrt[3]{\frac{100}{Vs + 100}})$, most conveniently read from a graph (Wintermyer et al, 1931). It should be noted that the graph given by Wintermyer et al is different from that given by Dawson (1959). The bar method is the one most commonly used in this country and simply involves the measurement of the shrinkage after oven drying the soil fines moulded into a small (15 cm x 1 cm x 1 cm) trough at the liquid limit.

That the linear shrinkage from the liquid limit is an acceptable control method has been shown by Heidema (1957) and Wall (1959). The former has also shown it to be statistically related not only to the plasticity index, but also to the liquid limit. If a perfect correlation exists between the results of two different tests, it means that both tests are measuring the same property (Van Rooyen, 1967). On reflection this is of course obvious. The plasticity index in fact measures the volumetric shrinkage of 100g of dry soil from the liquid limit to the plastic limit (see Wooltorton, 1954), while the linear shrinkage (as defined) measures the shrinkage in one dimension from the liquid limit to the shrinkage limit. It does not appear to have been previously realised that shrinkage and PI are theoretically related as follows:

$$PI = \frac{Vs}{R} - (PL-SL)$$

$$= \frac{Vs}{R} - SI$$

$$= \frac{100}{R}\left[\frac{1}{\left(1 - \frac{Ls}{100}\right)^3} - 1\right] - SI$$

(where SI is the shrinkage index) and

$$Ls = 100\left(1 - \sqrt[3]{\frac{100}{(PI + SI)\,R + 100}}\right)$$

by/ ...

by simple substitution in the two formulae for volumetric and linear shrinkage given in the previous section on the assumption that the material is dried from the liquid limit. It can be seen that the PI depends not only on the shrinkage, but on the shrinkage ratio and shrinkage index as well. A high PI is favoured by a low shrinkage ratio and a low or negative shrinkage index (SL>PL) for materials of equal volumetric (or linear) shrinkage. Using a plasticity index to measure shrinkage is plainly going about it the hard way. Heidema has even gone so far as to suggest that the bar linear shrinkage method is more accurate than the plasticity index method, and indeed there is little that can go wrong with it. The same certainly cannot be claimed for a PI determination. It is suggested then that more attention be paid to linear shrinkage than to plasticity index, in the case of calcretes at least. In view of the findings of Gillette (1934) already mentioned (Section 7.1) and the fact that the FAA and the THD still specify plasticity index determinations on calcretes (the THD apparently invented the bar linear shrinkage test) it is not at this stage suggested that the PI be abandoned, but merely that more attention be paid to shrinkage. The question of whether the shrinkage between the liquid and plastic limits or the shrinkage between the liquid and shrinkage limits is the more important factor in the performance of a road remains to the author's knowledge unanswered. In the arid and semiarid environment (which is where calcretes occur) it is almost certainly the latter.

7.6.1 Advantages of the bar linear shrinkage test

The advantages of bar linear shrinkage determinations over plasticity index determinations can be summarised as follows (partly after Heidema, 1957; and Wall, 1959):

1. The test is extremely rapid. Little preparation is required and since shrinkage stops at the shrinkage

limit/ ...

limit it is not even necessary to dry out the specimen completely. Speed is of the utmost importance in field control work. Much unsuitable material can be shifted and placed on the road in 24 hours.

2. It does not depend on the results of two other tests. It has also been shown in preceding sections that these other two tests (particularly the liquid limit) are fraught with numerous variables capable of influencing the results, particularly on calcretes.

3. No moisture content determination is required (a strong point in the case of field control laboratories). It is merely necessary to bring the initial moisture content somewhere near the liquid limit by a mechanical or hand method.

4. The cost of the test is less than 20% of that of the plasticity index test (Heidema).

5. It has been shown in the previous sections that the linear shrinkage seems to be less affected by heating than the PI.

6. The initial moisture content is not too critical. A few checks at groove closures of between 20 and 30 blows shows this to be also true for the 3 calcretes tested.

7. Hardly any apparatus is required. Use of a hand liquid limit method or starting from the field moisture equivalent even dispenses with the liquid limit device.

8. The personal factor plays a much smaller role in the linear shrinkage method than in either the liquid limit or plastic limit methods.

9./ ...

9. By using a specially calibrated rule for measuring the shrinkage (Heidema, TEX-107-E) all calculation is completely eliminated.

Regarding accuracy, Heidema (1957) stated that the Texas Highway Department found that the accuracy of the bar linear shrinkage method was about ± 2% of the value obtained and he suggested that it was more accurate than the PI. The same figure was mentioned by Dawson (1959), though he did not state whether it was ± 2% of the value obtained or ± 2 percentage units. He also stated that the bar method is a field method and that the most accurate method is calculation from the volumetric shrinkage. Wall (1959) stated that the bar method was repeatable to better than 1%. The latter author concluded that variation of the initial moisture content would not affect the result by more than about one part in 20.

Bowing and cracking of the specimens in the conventional troughs is sometimes encountered with the more plastic samples. This may be overcome by an initial air drying or by the use of Wall's special shrinkage slots which allow drying from two sides. Single troughs are normally used in South Africa, but the multiple troughs used by the Texas Highway Department would be advantageous when handling many samples.

The accuracy of the bar linear shrinkage test can probably be improved if necessary by more careful control of the initial moisture content (starting exactly from the liquid limit) or correcting for initial moisture content (the easiest would be by way of the number of blows required to close the groove in the liquid limit device), prevention of bowing and cracking (as described) and by use of the California 228-A measuring procedure.

Further work on the bar linear shrinkage test as a

possible/ ...

possible replacement for the PI in the arid and semiarid zone would be well worth while.

The statistical relation between plasticity index and linear shrinkage is discussed in Section 8.4.8.

7.7 MECHANICAL ANALYSIS

Mechanical analysis is of course only normally applicable to calcareous and calcified soils, powder calcretes and nodular calcretes. The following remarks would, however, also be applicable to honeycombs, hardpans and boulders used as crusher runs. The normal use of the term in the engineering sense implies the determination of the size-frequency distribution of particles ranging from almost 3 inches to about 2-5 microns in size. A full mechanical analysis would involve two separate tests; screening with sieves down to 200 (0.0029 inches, 0.074 mm) or 270 U.S. mesh (0.0021 inches, 0.05 mm) to deal with the coarser material and sedimentation analysis by the hydrometer or, rarely, the pipette method on material passing the 10 (2 mm) or 40 U.S. mesh (0.42 mm) sieve to deal with the finer material. The term 'mechanical analysis' is sometimes restricted to what is here termed sedimentation analysis. Dawson (1959) has stated that many engineers tend to over-emphasize the importance of sedimentation analysis and, in apparent agreement with this view, some roads laboratories do not run sedimentation analysis as a routine test. Amounts finer than the 200 mesh sieve are seldom if ever specified in base or subbase specifications and a wet screening method down to 200 mesh is probably all that is really necessary for roads use. Results are usually expressed as the cumulative percentage by weight passing various standard sieves, the U.S. standard sieve being normally employed in South Africa. Unless otherwise stated, all sieve sizes referred to in this work are U.S.

Calcretes/ .....

Calcretes may give trouble during mechanical analysis in three ways: by soft aggregate breakdown during sieving, by clay-coated aggregate and by flocculation during sedimentation analysis. The first difficulty may be largely overcome by use of a standard procedure, though much must inevitably be left to the operator's discretion. It would appear from McDowell's (1966) work that caliches may be particularly prone to aggregate breakdown in the dry preparation methods. This difficulty is still not fully resolved, however, since the aggregate strength of calcretes decreases with decreasing size of the aggregate. This means that the smaller particles are quite weak and possibly warrant the terms 'pseudosand' and 'pseudosilt' used by Ackroyd (1967) in connection with laterites in Nigeria. These particles probably break down during sieving and during dispersion by paddle-type dispersing devices, but may not break down in the road.

The problem of clay adhering to the larger particles can readily be overcome by the use of a wet sieving method, already standard practice in most laboratories.

Flocculation problems with all calcareous or saline materials seem to be common. This is probably due to adsorption of calcium or sodium ions on to some of the clay mineral surfaces, resulting in a partial neutralisation of their normally negative charge and consequent flocculation. The dispersing agents (antiflocculants) used, all possess anions with large negative charges and cations with small positive charges (e.g. the Na^+ and $(P_2O_7)^{4-}$ of tetrasodium pyrophosphate). Their dispersing action is presumably due to adsorption of the strongly negatively charged anions, thus restoring the overall negative charge on the clay particles. Methods which have been used to combat flocculation include use of the appropriate dispersant, increasing the amount of the dispersant used, and removal

of/ ...

of the carbonate with acid, or the more soluble salts by washing.

It should be noted that the usual mechanical analysis procedure results in a particle size distribution curve by weight, when what is really wanted is a particle size distribution curve by volume. The two are only the same if the bulk particle specific gravity (not G_s) of all the size fractions is the same. If for example the bulk particle specific gravity of a certain size fraction is higher than that of its neighbours on the particle size distribution curve, the curve by weight will overestimate the percentage of that size fraction present by volume. The concept of maximum compacted stability being governed by compacted dry density requires that a continuous Fuller-type curve by volume be attained for maximum stability. A curve by volume is inconvenient to determine, so curves by weight are usually employed, but this fact should not be lost sight of.

7.7.1 Comparison of methods

All mechanical analysis procedures employ sieving for the particles down to about 200 mesh and a sedimentation technique for the fines (-40 or -10 mesh).

Sieving techniques can be divided into two types, those which employ only dry sieving and those which employ wet sieving. Dry methods are BS 1377:1961 (6B) and AASHO T27-60 (when used without T11). Dry methods are widely recognised as only being suitable for clean granular materials containing little or no clay. Yoder (1959) for example has stated that "Gradation tests should always be made by washing the material through a nest of sieves down to the No. 200 mesh sieve. In no case should dry sieving ever be permitted". Wet methods are normally used by all roads authorities, including those in South Africa. Wet methods are D.O.T. (method 1) NIRR (method E), AASHO T11-60 and T27-60 combined (specified by the

FAA/ ...

FAA (1959) and New Mexico (1963) , TEX-110-E, AASHO T88-57 and ASTM D422-63.

Sedimentation methods used by roadmaking authorities are normally of the type employing hydrometers, since these are the quicker methods and are nowadays little inferior to the pipette method described for example in BS 1377:1961 (standard method). At this point some methods such as BS 1377 and D.O.T. (and often those used in pedological work) specify that any carbonates should be removed by acid treatment. While removal of soluble salts by washing is acceptable, it is plain that the carbonate should not be removed. The carbonate in calcretes is an essential and valuable component, with generally favourable rather than detrimental engineering properties. From this point onwards methods differ in their presoaking times, dispersion devices, choice of dispersing agents and sedimentation times. A detailed treatment is beyond the scope of this work and the following must suffice as a summary: Methods solely employing rotating paddle-type devices are the D.O.T. and BS 1377:1961 methods, while in the AASHO T88-57, ASTM D422-63 and TEX-110-E methods air dispersion using Wintermyer soil cups or the soil dispersion tube of Chu and Davidson is optional. The NIRR method employs the soil dispersion tube, which is more convenient to use than the Wintermyer cup. According to Davidson (1955), the air-driven soil dispersion tube of Chu and Davidson results in the best dispersion and least abrasion of soil particles in suspension. Davidson's results are particularly worthy of study since his samples contained up to 80% $CaCO_3$. Paddle-type devices (particularly when driven fast) tend to abrade the coarser particles, but are relatively inefficient in dispersion of the finer particles (particularly those finer than 20 microns- ASTM D422). As far as chemical dispersants are

concerned/ ...

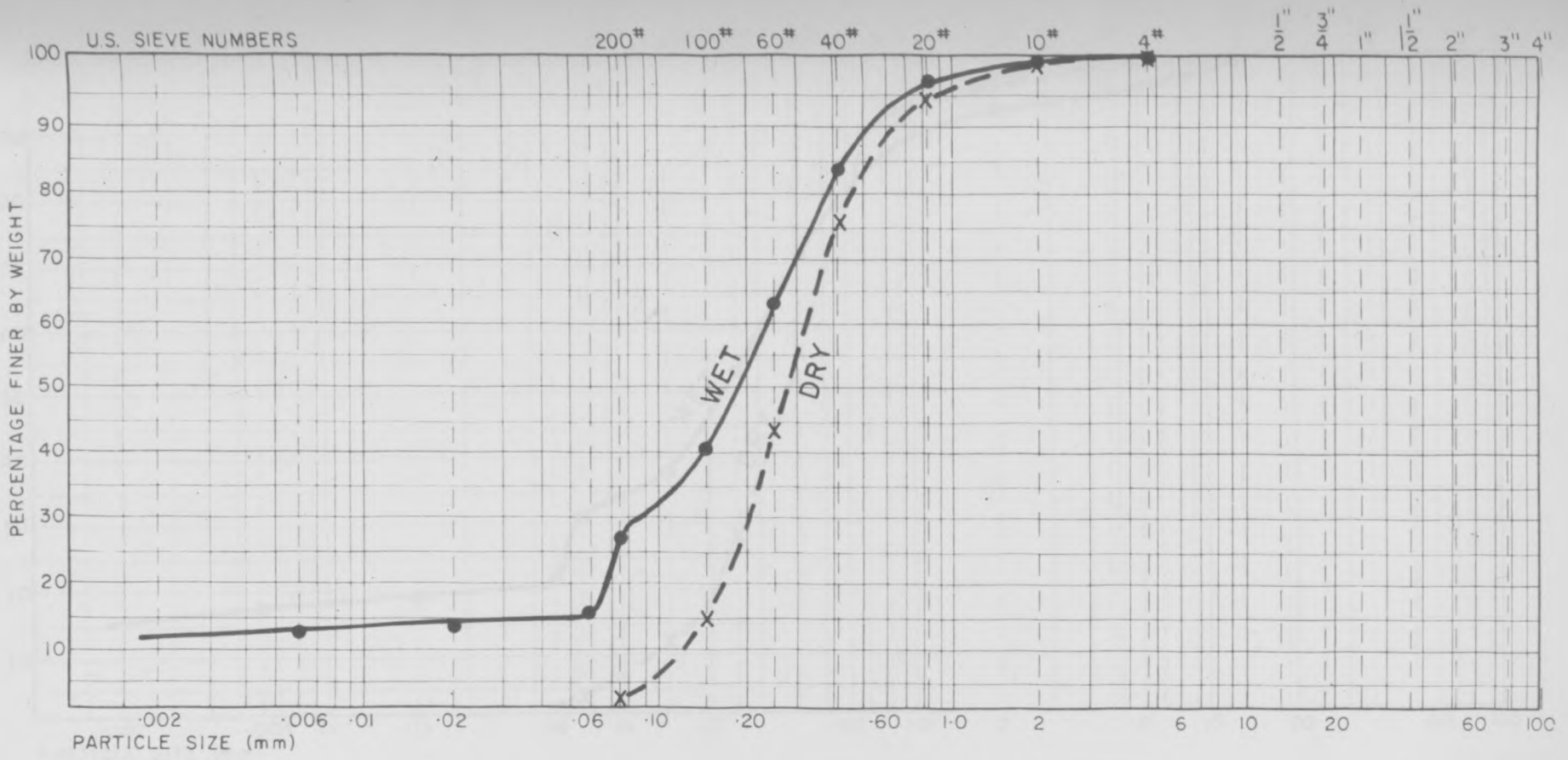
concerned, while it has been said that there is no single dispersant which is equally effective on all soils, the phosphates are generally conceded to be the best, with possibly sodium hexametaphosphate being the most universally applicable (see Wintermyer and Kinter, 1955; Davidson, 1955 and the ASTM symposium on particle size measurement, 1958). The NIRR method uses tetrasodium pyrophosphate while the ASTM and BS methods use sodium hexametaphosphate. The Texas method uses either sodium metasilicate or Na_2CO_3-buffered sodium hexametaphosphate, while the AASHO method also lists several optional dispersants (sodium hexametaphosphate, polyphos, trypolyphosphate and tetraphosphate). The D.O.T. method uses a mixture of sodium silicate and sodium oxalate. It would generally appear from the literature that sodium metaphosphate or hexametaphosphate are the best dispersants for calcareous soils and trisodium phosphate or tetrasodium phosphate best for lateritic soils.

Times allowed for sedimentation before hydrometer readings are taken vary widely. D.O.T. times tend to be the shortest, followed by NIRR times. The American methods all use the same times, which are longer still. All the factors previously discussed affect the times and most methods introduce corrections for the meniscus, dispersing agent, temperature and the specific gravity of the soil particles used. Briggs et al (1962; in Brewer, 1964) have pointed out that particle shape can affect the results obtained by about the same amount as variations in particle specific gravity.

7.7.2 Application to calcretes

Figures 7.6-7.10 show a comparison of wet and dry screening methods carried out on the author's behalf by Mr. E. Nash. In each case, analysis by the NIRR wet method yielded considerably more fines than a simple dry screening method. Samples 895, 913 and 916 also show

evidence/ ...

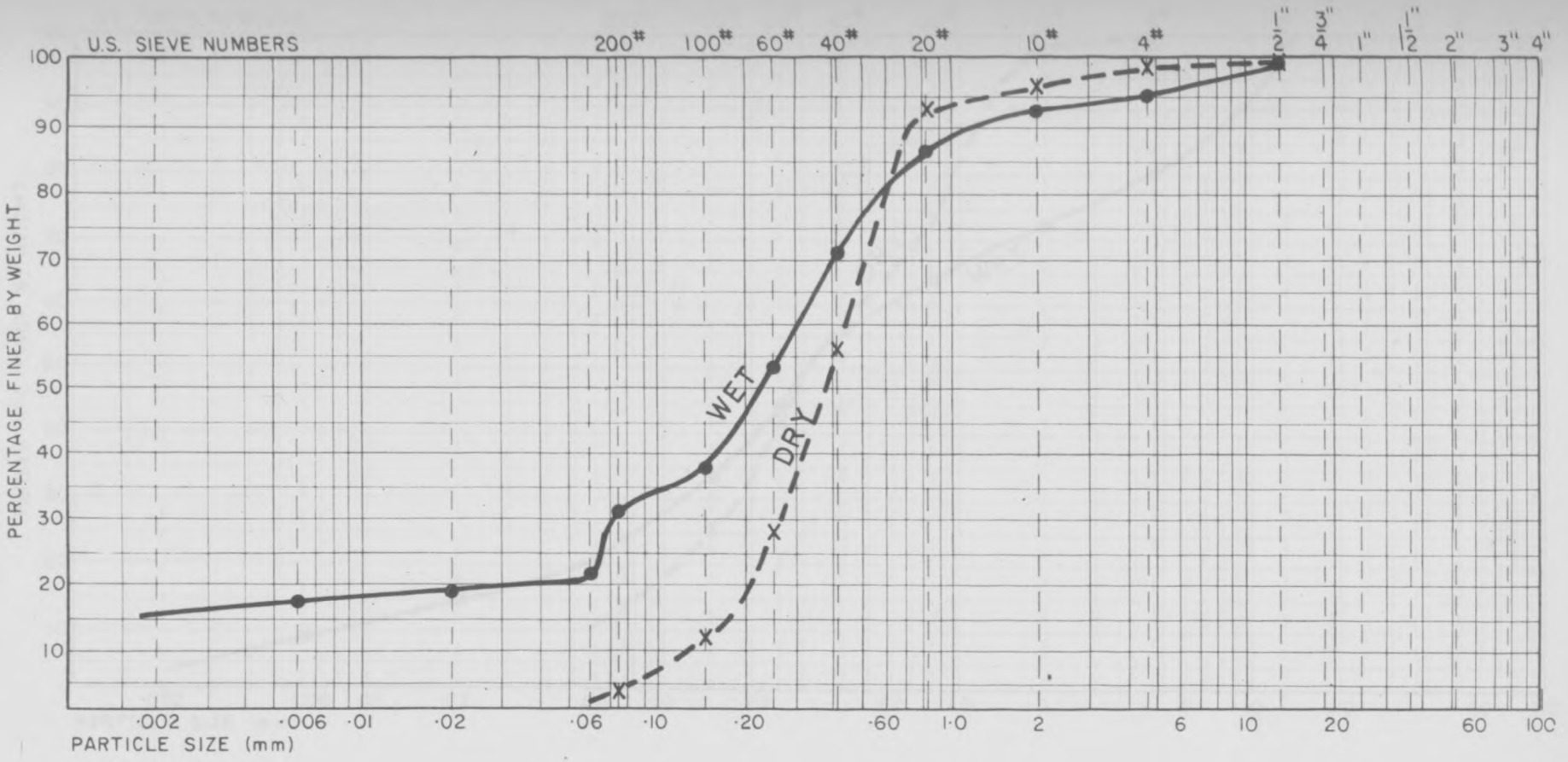


CLAY FRAC-TION	FINE	MEDIUM	COARSE	FINE	MEDIUM	COARSE	FINE	MEDIUM	COARSE
	SILT FRACTION			SAND FRACTION			GRAVEL FRACTION		

FIGURE 7·6

Comparison of wet and dry sieving methods on Sample 893 (calcareous clayey sand)

RG4-3186

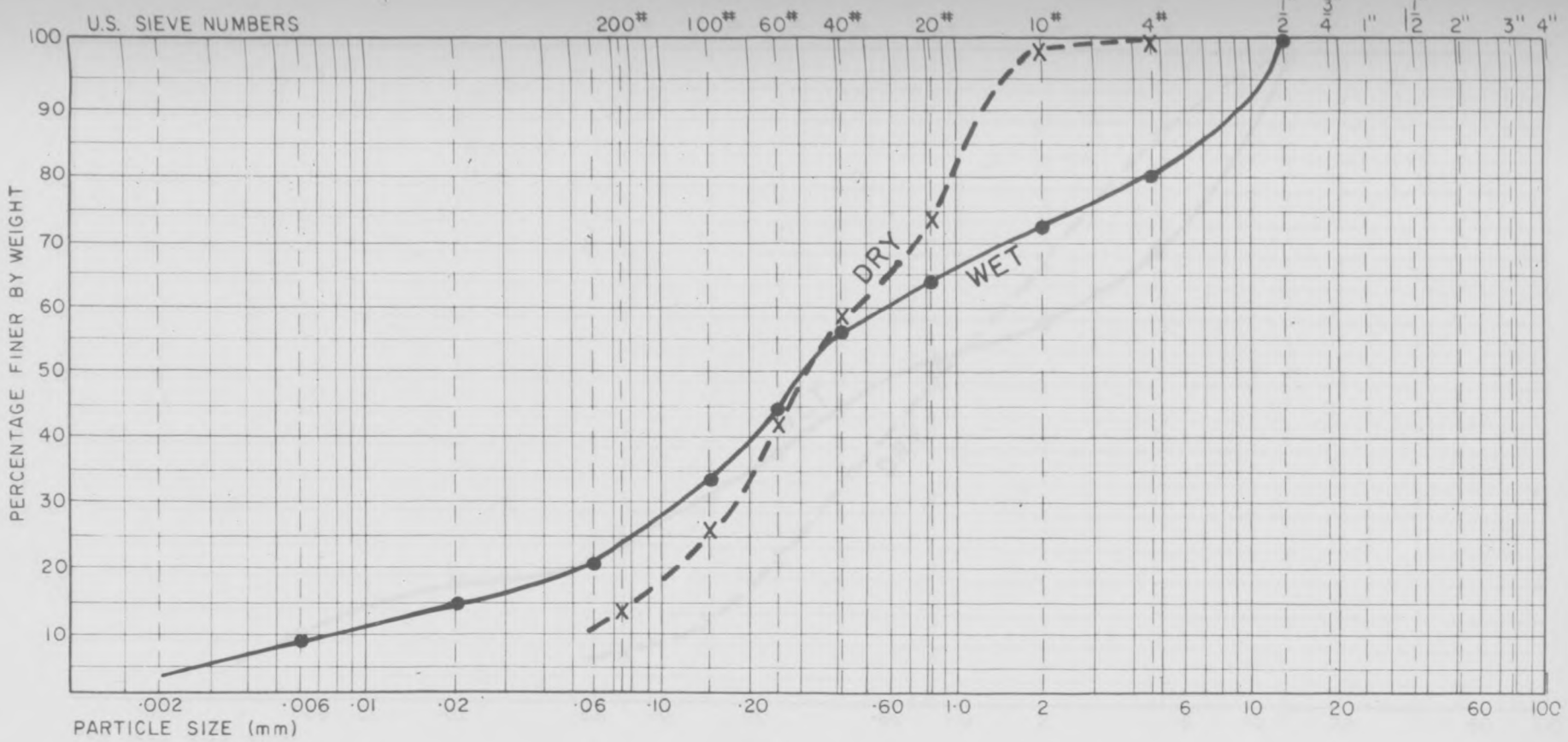


CLAY FRAC-TION	FINE	MEDIUM	COARSE	FINE	MEDIUM	COARSE	FINE	MEDIUM	COARSE
	SILT FRACTION			SAND FRACTION			GRAVEL FRACTION		

FIGURE 7·7

Comparison of wet and dry sieving methods on Sample 916 (calcareous clayey sand)

RG4 - 3187

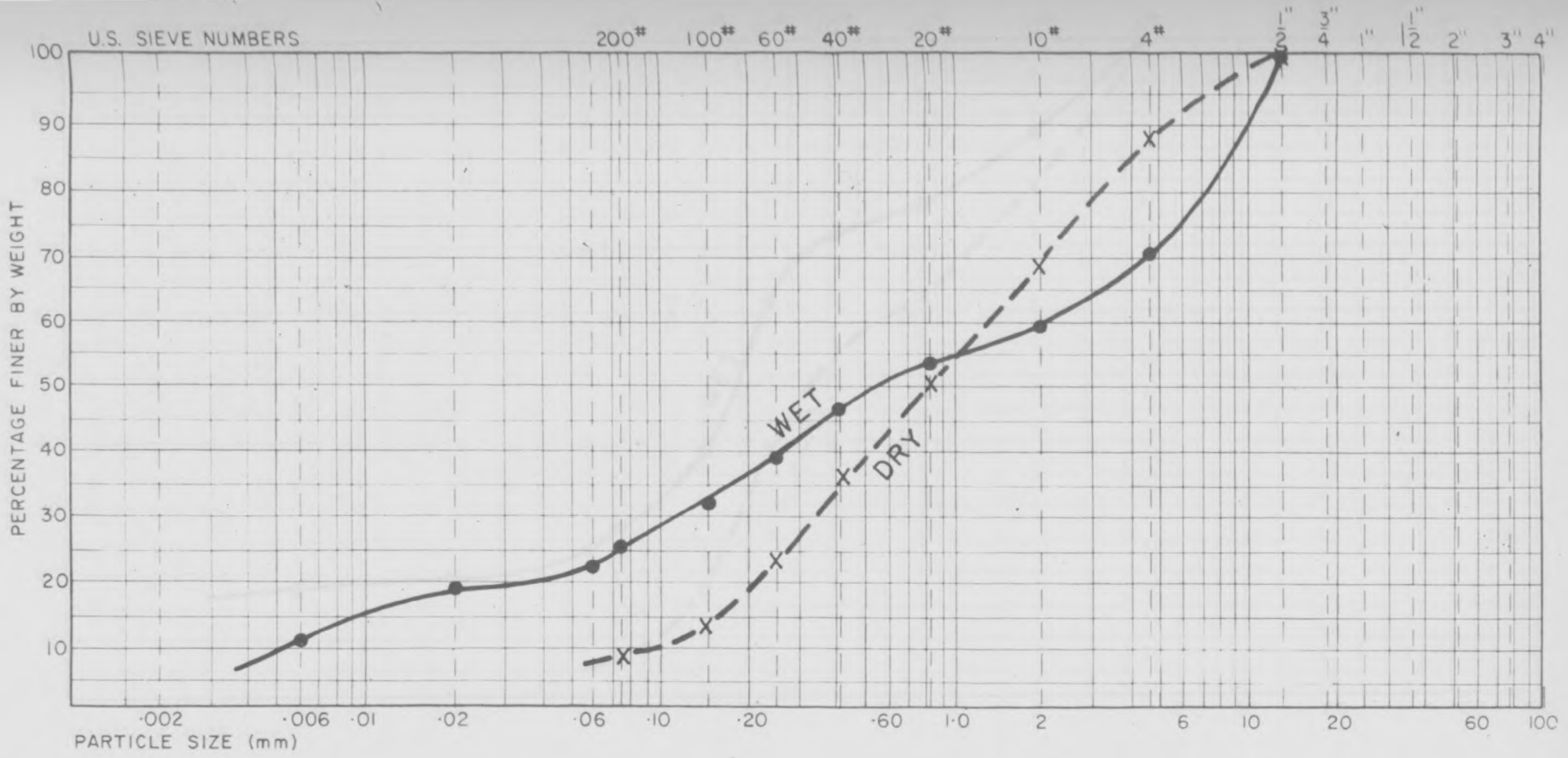


CLAY FRACTION	FINE	MEDIUM	COARSE	FINE	MEDIUM	COARSE	FINE	MEDIUM	COARSE
	SILT FRACTION			SAND FRACTION			GRAVEL FRACTION		

FIGURE 7·8

Comparison of wet and dry sieving methods on Sample 895 (calcified sand)

RG4-3188

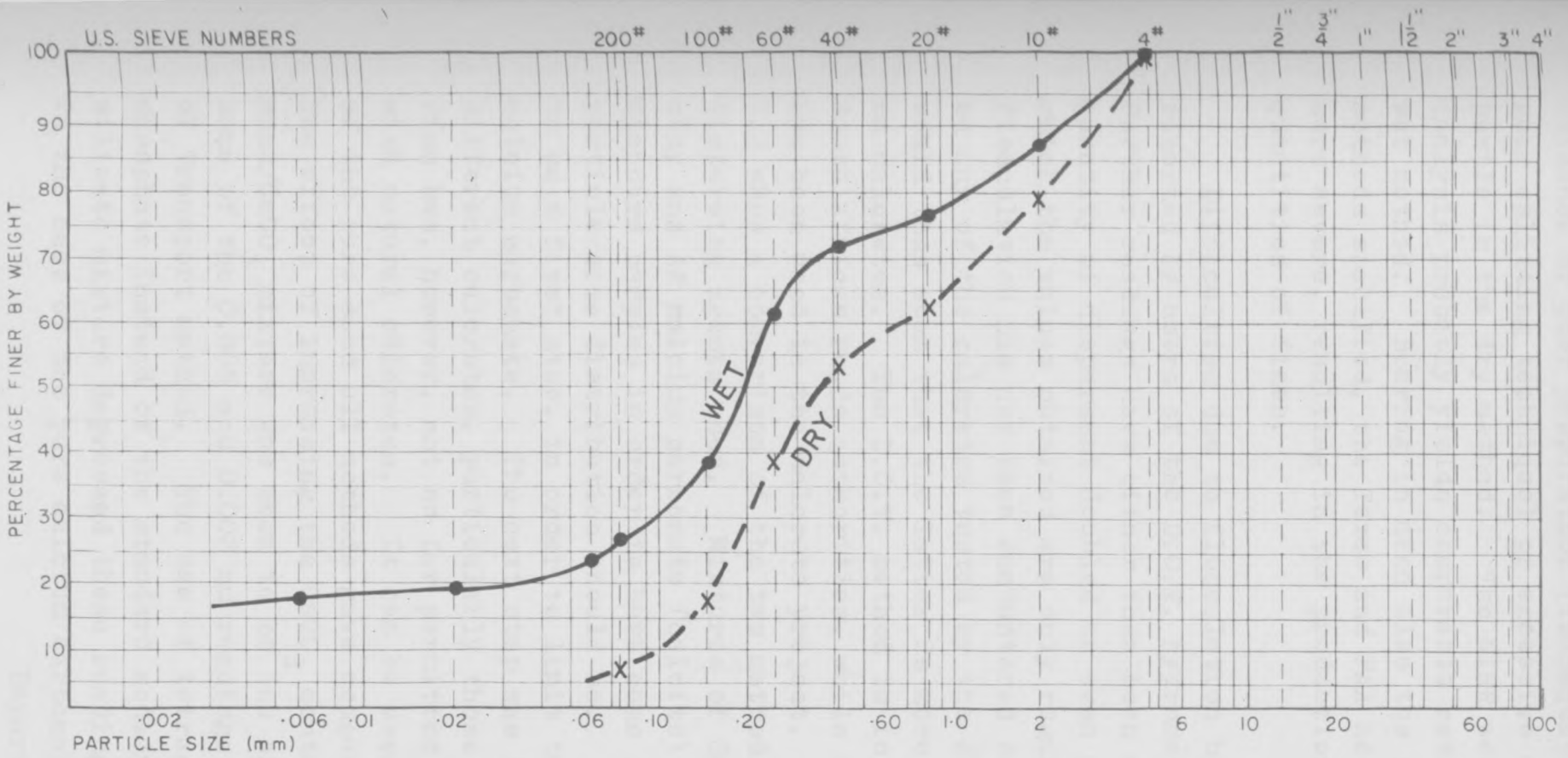


CLAY FRAC-TION	FINE	MEDIUM	COARSE	FINE	MEDIUM	COARSE	FINE	MEDIUM	COARSE
	SILT FRACTION			SAND FRACTION			GRAVEL FRACTION		

FIGURE 7·9

Comparison of wet and dry sieving methods on Sample 913 (nodular calcrete)

RG4-3189



CLAY FRACTION	FINE	MEDIUM	COARSE	FINE	MEDIUM	COARSE	FINE	MEDIUM	COARSE
	SILT FRACTION			SAND FRACTION			GRAVEL FRACTION		

FIGURE 7·10

Comparison of wet and dry sieving methods on Sample 918 (mixture of broken hardpan and calcareous sandy clay)

RG4-3190

evidence of poor preparation (inadequate mixing and/or poor splitting technique) or excessive use of the rubber pestle in the dry method. The NIRR wet method of sieve analysis probably yields comparable results to the D.O.T. wet method. Bearing in mind also the preparation methods specified, the Texas and FAA methods are possibly more severe, resulting in the production of greater quantities of fines.

Difficulties due to flocculation have often been reported by users of the D.O.T. hydrometer method. Further readings have either then been abandoned or the quantity of dispersant doubled or even quadrupled, upon which the values obtained are only regarded as approximate. Flocculation has not been encountered during this project on any of the calcretes tested by the NIRR method and it would thus seem that the latter is more suitable for use on calcretes. The D.O.T. method is in standard use by South African roads authorities, while the NIRR method has been used in the calcrete project. Tables 7.2 and 7.3 show a comparison of the two methods and the dispersing agents used. Mixtures of Onderstepoort black clay and AR calcium carbonate (calcite) were used as standard samples in order to have some idea what the true particle size distribution should be. This was intended to be a first step, in order to limit the variables to calcium carbonate. The next step was to be trials with different calcretes, particularly those known to be salty. Time has, however, not so far permitted the experiments with natural calcretes. It can be seen that in the case of the pure sand all methods gave comparable results. The effect of increasing the $CaCO_3$ content of the sand/$CaCO_3$ mixture was seen to be one of depression of some of the 0.006 and 0.002 mm readings in the Department of Transport method. The use of tetrasodium pyrophosphate instead of the standard sodium oxalate and silicate mixture depressed these readings still further. In the case of the pure calcium carbonate (A5 & A6), the

Department/ ...

TABLE 7.2 RESULTS OF HYDROMETER PARTICLE SIZE ANALYSES BY DIFFERENT METHODS.

Method 1) Calculated from results of Method 2 on A1, A5, A6, A10, AR6 and AR10.

2) Standard NIRR method using air dispersion and sodium pyrophosphate.

3) Method 2, but using sodium oxalate and silicate (10 ml).

3A) Method 2, but using sodium oxalate and silicate (40 ml).

4) Standard Department of Transport method.

5) Method 4 using sodium pyrophosphate and times as follows: 40 secs, 6 min. 64 min. and 7 hours.

6) Method 5 using Department of Transport's times. Figures bracketed are interpolated. (e.g. 0.006 mm reading from standard 0.005 mm reading).

SAMPLE NO.	METHOD	PERCENTAGE FINER THAN			
		0.06 mm	0.02 mm	0.006 mm	0.002 mm
A1 (Quartz sand)	1	4	4	4	4
	2	4	4	4	4
	3	4	4	4	4
	4	(7)	-	(4)	4
	5	5	5	4	4
	6	(5)	-	(4)	-
A2 (75% sand 25% $CaCO_3$)	1	26	24	12	5
	2	28	26	16	7
	3	28	27	15	6
	4	(28)	-	(12)	5
	5	14	11	5	2
	6	(13)	-	(6)	-
A3 (50% sand 50% $CaCO_3$)	1	48	43	21	6
	2	47	43	22	4
	3	49	45	24	6
	4	(45)	-	(14)	7
	5	50	40	8	4
	6	(44)	-	(5)	-
A4 (75% $CaCO_3$ 25% sand)	1	70	63	29	6
	2	68	63	31	7
	3	68	65	32	7
	4	(66)	-	(15)	7
	5	66	56	14	0
	6	-	-	-	-

Department of Transport method resulted in higher readings for the 0.002 mm fraction than the calculated and NIRR values. This was probably due to abrasion during dispersion of the relatively soft carbonate particles. The results of the black clay/$CaCO_3$ mixtures are rather erratic, but in general they indicate that flocculation has taken place in the Department of Transport method, resulting in very low readings, particularly in the 0.006 and 0.002 mm sizes. Sodium pyrophosphate in some cases gave some improvement over the oxalate and silicate, but the readings were still low. No flocculation was encountered in the standard NIRR method using air dispersion and 20 ml of an aqueous solution of 36 gms/litre of tetrasodium pyrophosphate ($Na_2P_4O_7.10H_2O$).

In an attempt to obtain better results, methods 1-3 were repeated in duplicate on fresh samples. The mean results are shown in Table 7.3. The differences between some of the calculated and observed values are again rather large, but are probably within the limits of experimental error. It would appear that sodium oxalate and silicate and sodium pyrophosphate are often equally effective as antiflocculants for calcretes in the NIRR method, though in two cases the oxalate and silicate did allow flocculation. Quadrupling the amount of dispersant used still gave reasonable results. It would further appear that the defect in the Department of Transport method is not so much the antiflocculant used, but rather the method of mechanical dispersion used (milkshake mixer-type device).

Table 7.4 shows the effect of an addition of 1% of an electrolyte (NaCl) to a mixture of 75% $CaCO_3$ and 25% Onderstepoort black clay. The suspension flocculated badly in cases 2 and 3, but an increase of sodium oxalate and silicate antiflocculant enabled better readings to be obtained. It would appear that a mixture of sodium oxalate and silicate is a more effective antiflocculant in the presence of NaCl than sodium pyrophosphate. The reason for

this/ ...

TABLE 7.3 EFFECT OF TWO DIFFERENT ANTIFLOCCULATION AGENTS ON THE NIRR METHOD

SAMPLE NO.	METHOD NO.	PERCENTAGE FINER THAN			
		0.06 mm	0.02 mm	0.006 mm	0.002 mm
AR6 ($CaCO_3$)	1	85	66	19	1
	2	85	66	19	1
	3	85	63	5	1
	3A	88	71	31	1
AR7 (75% $CaCO_3$ 25% black clay)	1	83	67	31	14
	2	86	71	42	18
	3	84	72	40	18
	3A	86	68	40	21
AR8 (50% $CaCO_3$ 50% black clay)	1	80	68	42	29
	2	84	78	48	28
	3	83	76	50	32
	3A	85	78	48	34
AR9 (25% $CaCO_3$ 75% black clay)	1	77	71	54	42
	2	81	73	54	45
	3	82	70	54	3
	3A	80	72	58	46
AR10 (Black clay)	1	75	70	65	56
	2	75	70	65	56
	3	75	71	63	53
	3A	81	74	66	57

Hydrometer analyses by Mr. T.E. O'Connor

this is not clear.

TABLE 7.4 EFFECT OF 1% NaCl ON RESULTS OBTAINED BY THE NIRR METHOD

SAMPLE NUMBER	METHOD NUMBER	PERCENTAGE FINER THAN			
		0.06 mm	0.02 mm	0.006 mm	0.002 mm
AR7 (75% $CaCO_3$, 25% black clay 1% NaCl)	1	83	67	31	14
	2	83	15	10	6
	3	85	70	19	1
	3A	89	73	40	21

Hydrometer analyses by Mr. T.E. O'Connor

It is difficult to draw satisfactory conclusions from the results of the experiments described, but in general it would appear that the NIRR method should give generally satisfactory results on calcretes except when large amounts (1%) of salts are present and that the standard D.O.T. method is generally less satisfactory in the presence of carbonates. On flocculation in the presence of an electrolyte in the NIRR method an increase in the quantity of dispersing agent (oxalate and silicate) was found to be the remedy. Reasonable results were still obtained. On flocculation in the D.O.T. method it is common practice either to take only the silt plus clay reading together or to increase the quantity of dispersing agent, upon which the results are not regarded as reliable. An alternative procedure which would be satisfactory is to wash the soil and remove the soluble salts in this way - they seldom make up more than 1% of the material by weight. It would appear that non-saline carbonate-containing materials may or may not cause flocculation in the D.O.T. method.

The average specific gravity of the solid particles (Gs)/ .....

(G_s) in the minus 40 mesh fraction of calcretes falls within the range 2.47 to 2.80. Most sedimentation methods assume a specific gravity of 2.65, though it is possible to correct for specific gravity (see for example Wintermyer et al, 1931; or ASTM D422). The effect of using an assumed specific gravity of 2.65 when the true value is 2.47 would cause the amount in suspension at any moment to be underestimated by about 4% of the value. Similarly, if the true value is 2.80, then the result reported would be too high by about 3%. These errors are not serious for most purposes. The particle shape of calcrete fines is roughly spherical, i.e. not significantly different from that of most other soils.* However, the question now arises as to which specific gravity should actually be used in sedimentation calculations. The effective specific gravity of a porous particle settling in water is probably not the specific gravity of solids at all, but is probably approximated most closely by the saturated surface dry specific gravity. Only when the particles are non-porous will this equal the specific gravity of solids. If the particles are not properly saturated, some value between the dry bulk and the saturated bulk specific gravity will apply. The data on the specific gravity of calcrete fines in Appendix J is unsatisfactory, but dry bulk particle specific gravities down to 1.6 and saturated values down to 1.9 have been obtained on some size fractions. Use of a value of 2.65 instead of a true value of 1.9 would result the percentage remaining in suspension at any one moment being underestimated by 35% of the value, which is plainly a serious source of error. In addition, the data in Appendix J suggests that the dry and saturated bulk specific gravities vary with size fraction, which would give rise to further errors. Theory suggests that below some critical size the dry and saturated bulk particle specific gravities should increase as the particle size decreases, becoming equal to the specific gravity of solids once the size of the elemental particles

which/ ...

*The particle shape varies though, for palygorskite and sepiolite are needle-shaped and the other common clays flake-shaped.

which go to make up the coarser fractions is reached (see Chapter 8) and there is a little evidence for this in Appendix J. If the effective specific gravity in the suspension of the coarse and fine sand is for example less than that of the silt plus clay sizes, the former will be underestimated and the latter overestimated (depending on the actual specific gravity used in the calculation).

It is apparent that further investigation of the accuracy of sedimentation methods when applied to calcretes is called for. Calcretes should first of all be thoroughly soaked before dispersing to ensure that the porous particles are saturated (soaking under vacuum as for the determination of G_s would speed things up). The correct specific gravity to use in the calculation should be experimentally determined (this could be done by using single-sized porous particles of definitely known solid, dry bulk and saturated bulk specific gravities), and the importance of variations of this value with size fraction assessed. Experimentation with different antiflocculating agents might be worthwhile. Sodium metaphosphate might well be found to be the most effective calcrete dispersant. Lastly, the accuracy of the final method decided upon should be checked against an absolute method (such as particle size measurements under a microscope). Many other methods for particle size characterisation besides the common hydrometer and pipette methods are available, though many require a knowledge of the particle specific gravity. Much useful information on particle size measurement is contained in the ASTM symposium of that name.

7.8 AGGREGATE STRENGTH AND DURABILITY TESTS

Aggregate tests can be conveniently divided into two groups, those which merely measure the dry strength of the aggregate in some way and those which make an attempt to predict its performance under conditions more nearly approaching those encountered in practice. Wet aggregate

crushing/ ...

crushing tests and the sulphate soundness tests can be placed in the latter category.

7.8.1 Aggregate crushing and abrasion tests

The test most commonly used for the assessment of the strength of stone for use in concrete, bituminous surfacings and base courses in South Africa is the aggregate crushing test, described for example in D.O.T. method no. 12 and BS 812. In this test a standard mould is filled with crushed aggregate and subjected to a load of 40 short tons at 4 tons/minute. The fines produced are then screened through a sieve, the size of which depends on the aggregate size used. The percentage fines passing the sieve is called the aggregate crushing value (ACV). It follows that stronger rocks have lower ACVs than weaker rocks. Putting a formula to Markwick and Shergold's (1946) Figure 3 shows the crushing strength in lb/sq inch to be roughly equal to $\frac{500,000}{ACV}$. Useful information on the test is contained in papers by Markwick and Shergold (1946) and Shipway (1964), and in BS 812:1967. Both authors found the results to be affected by flakiness, with elongated aggregates yielding somewhat poorer test results. Different aggregate sizes require different sizes of sieves for separating the fines and in some cases ($\frac{3}{4}$" and $\frac{1}{4}$") also different moulds. BS 812:1951 stated that larger aggregate usually yields a higher ACV than smaller aggregate.

Another aggregate crushing test which is becoming more common in South Africa is the 10% fines aggregate crushing test (10% F.A.C.T.) of Shergold and Hosking (1959) (see also BS 812:1962). Since then it has also been adopted as a national standard (SABS 647-1961). The test differs from the aggregate crushing test described above only in that a load giving only 7.5-12.5% fines is applied and the load in short tons required to yield 10% fines calculated by means of a formula. The test was

designed/ ...

designed mainly with weaker materials in mind (ACV more than about 30) since the latter compact under a 40 ton load and reduce the fines produced. The test is,however, equally applicable to both strong and weak materials. It suffers from the disadvantage that the operator is required as a first approximation to estimate the load required to produce 7.5-12.5% fines and may thus require more material than the ACV test. It is also slightly more trouble to carry out. There is no real advantage in its use on materials yielding ACVs of less than 30%. Stronger rocks obviously yield higher 10% F.A.C.T. values than weaker ones.

An assumption inherent in the 10% F.A.C.T. is that the plot of load in tons against the percentage fines produced in the test is linear between 7.5% and 12.5% fines and obeys the relation: 10% F.A.C.T. (i.e. the load required to produce 10% fines) = $14x/(y+4)$ where x is the applied load in tons and y is the percentage fines produced. Calcretes were not among the materials tested by Shergold and Hosking (1959) when they developed the test. While it was not expected that calcretes would deviate from the formula given above, as a precaution the data obtained from routine tests was examined. The results on some of the samples are shown graphically in Figure 7.11. It can be seen that in most cases the results calculated by means of the formula agree well with that indicated by a graphical solution. The only seriously deviant sample was 2507, which was a very inhomogeneous material, a gravelly hardpan consisting of very strong chert, quartzite and quartz gravel cemented in a much weaker calcrete matrix. Two results from 2114 and 2116 were also a little out, suggesting that the applicability of the formula to round particles (nodules in these cases) needs to be looked into. The formula may be satisfactory only for crushed,reasonably homogeneous material and a graphical solution would be the safest method. The crushing tests on which Figure 7.11

is/ ...

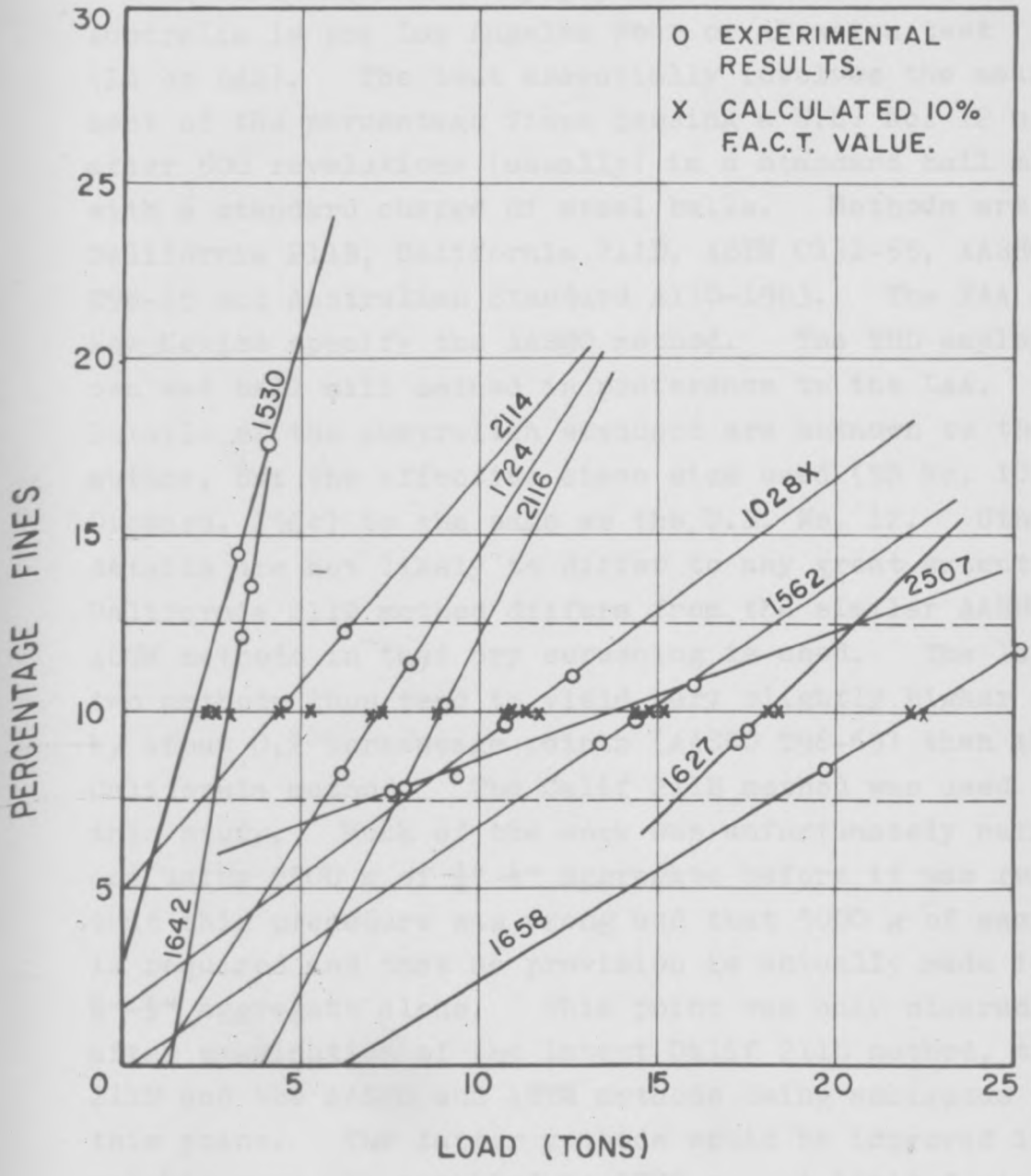


FIGURE 7·11

COMPARISON OF GRAPHICAL AND CALCULATED VALUES FOR 10% F.A.C.T. VALUE

is based were carried out by Mr. T.E. O'Connor.

A test which is very popular in the U.S.A. and Australia is the Los Angeles wear or abrasion test (LA or LAA). The test essentially involves the measurement of the percentage fines passing a U.S. No. 12 sieve after 500 revolutions (usually) in a standard ball mill with a standard charge of steel balls. Methods are California 211B, California 211D, ASTM C131-55, AASHO T96-65 and Australian Standard A130-1963. The FAA and New Mexico specify the AASHO method. The THD employs its own wet ball mill method in preference to the LAA. Details of the Australian standard are unknown to the author, but the effective sieve size used (BS No. 10, Orchard, 1964) is the same as the U.S. No. 12. Other details are not likely to differ to any great extent. The California 211B method differs from the similar AASHO and ASTM methods in that dry screening is used. The latter two methods thus tend to yield very slightly higher values by about 0.2 percentage points (AASHO T96-65) than the California method. The Calif 211B method was used in this study. Much of the work was unfortunately carried out using 2500 g of $\frac{3}{8}$"-$\frac{1}{2}$" aggregate before it was realised that this procedure was wrong and that 5000 g of sample is required and that no provision is actually made for $\frac{3}{8}$"-$\frac{1}{2}$" aggregate alone. This point was only cleared up after examination of the latest Calif 211D method, the 211B and the AASHO and ASTM methods being ambiguous on this point. The latter methods would be improved if the weights were all totalled to 5000 g. A limited study of the effect of the weight and grading of the sample is given in Table J.4. Very useful discussions of the factors influencing the LAA test had been given by Woolf and Runner (1935) and Orchard (1964). The former authors concluded that shape (sphericity) and to a much lesser degree roundness or angularity affected the test to quite a large extent, spherical or rounded aggregate yielding

less/ ...

less wear than elongated or angular aggregate. Even hand broken (more cubical) rock tended to yield less wear than crushed (more flaky) aggregate. Nodular calcretes are thus likely to yield less wear than many other materials of the same toughness or whatever is measured by the test. In the discussion of the latter paper Minty suggested that the LAA test measured toughness or tenacity and not hardness since the average values for limestone and quartzite were the same, while the hardness of these two rocks is of course quite different. Orchard concluded, however, that it is still not known precisely what properties of a stone are measured by either the LAA or the ACV tests. The LAA and ACV tests give results that are very nearly numerically equal (Markwick and Shergold, 1946; up to an ACV of 30, Shipway, 1964) BS 812:1951 stated that on most $\frac{3}{8}$"-$\frac{1}{2}$" aggregate the ACV is very close to the LAA, but that very soft aggregates tend to yield a lower ACV than LAA. The LAA and ACV tests are very reproducible, only one test being required to ensure a 90% chance that the result will be within $\pm$ 3% of the mean of a large number of tests (Road Research Laboratory, 1952). This is vastly superior to tests employing cylindrical specimens. The 10% F.A.C.T. test will be somewhat less reproducible than the ACV and LAA as it requires more operator skill in estimating the load to use and usually relies on a formula which assumes a certain relation between the load and the fines produced. There is some evidence that the LAA test is particularly suitable for carbonate aggregates (Aughenbaugh, 1963).

A test common in the Cape Province, but seldom seen elsewhere in the Republic is the Treton test. The only reference to it in the literature known to the author is that in Walker and Stewart (1943) and the remarks by Knight in the discussion of this paper. The test basically involves dropping a weight several times onto crushed aggregate, the Treton value being the percentage fines

produced/ ...

produced which passes a BS No. 10 sieve. The test is similar in principle though not in detail to the aggregate impact test described by the Road Research Laboratory (1952). Both the apparatus and the mode of operation are so simple that no field control laboratory need be without it. Walker and Stewart concluded that "in practice, the Treton (brittleness) and absorption of water tests together, will be sufficient in nearly every case" for the testing of road aggregates. It is surprising that the test is not more commonly used.

7.8.2 Tests under wet conditions

All aggregate strength tests are of course really also durability tests. It is obvious for example that, all other factors being equal, base aggregate with an ACV of 25% can be expected to undergo less degradation during excavation, stockpiling, placing, compaction and in service under traffic than one only possessing an ACV of 35. An important factor which most tests do not incorporate, however, is water. In practice, even a base course under a good bituminous surfacing is always damp, if not wet (around optimum moisture content), under most climates and at least some faces of surfacing chips, unless sealed in premix or covered with bitumen, are subjected to the normal climatic conditions prevalant in the area. The presence of water reduces strength due to a softening of bonds, pore water pressure build-up, lubrication, and an accelleration of decomposition. The weakened nature of the aggregate thus also makes it more susceptible to disintegration during the movement of flexible base courses under traffic. It follows from theory, and is substantiated to some extent by practical experience, that degradation is reduced in stabilized and concrete bases and that less severe aggregate strength specifications can be used in such cases. Many U.S. states find an LAA of 50 (and even 66 - South Carolina) satisfactory/ ...

satisfactory for concrete pavement aggregates (Erickson, 1960). Erickson, after a nation-wide survey in the United States reported that 36 out of the 46 states that replied had experienced degradation in base courses during service and that in seven states it was a serious problem. "Thirty replies reported that the Los Angeles rattler test, 21 the sulfate soundness test, and 13 the freeze-and-thaw test, did not differentiate degrading aggregates from good aggregates". Tests reported as helpful include a modified (but dry) Los Angeles using no steel charge (Erickson, 1958; Ekse and Morris (1960); soil constant determinations and before and after gradings carried out on the fines; wet tumbling and calculation of a degradation factor based on breakdown and sand equivalent of fines (Minor, 1960); wet ball mill tests (TEX-116-E and California 210-C) and others. Symptoms of degradation failures are initial longitudinal cracking followed by alligator cracking and potholing. Wet, plastic fines were encountered under alligator cracked surfacings where the base had been clean on laying (Minor, 1960). Ekse and Morris suggested that their test should be particularly suitable on soft materials like shale and caliche.

Degradation during compaction can also occur. This has been studied by Shergold (1954; in Aughenbaugh, 1963) in the case of surface dressings and by Aughenbaugh in the case of soil-aggregate base courses. Most degradation occurs during the first pass of the compactor and is negligible after about 8 passes. Most degradation occurs at the top of the layer being compacted. Well-graded materials degrade the least and single-sized materials the most. Degradation may thus decrease or increase the maximum density attainable. Aughenbaugh concluded that "no one laboratory physical test or combination of tests will successfully evaluate all aggregate types on all occasions for construction degradation" and that

"petrographic/ ...

"petrographic analysis is the best and only reliable means of evaluating aggregates for construction compaction degradation". Of the physical tests tried, the Los Angeles abrasion at 500 revolutions (preferably without the steel shot) was found to be the best on the 4 materials studied (3 of which were carbonate aggregates)). Water absorption and bulk density were also useful.

Sulphate soundness tests

The best known test for the durability of road aggregates is the sulphate soundness test (sometimes rather misleadingly called a weathering test). The aggregate size usually employed in South Africa is $\frac{3}{4}$"-1" (D.C.T., 1958; SABS 647-1961) but a mixture of various sizes may be tested by screening it into various sizes which are kept apart during the test. In such a case a weighted mean loss for the whole sample is eventually reported (see ASTM C88-61T and AASHO T104-65). Some tests use sodium sulphate (D.O.T., 1958), some magnesium sulphate (D.O.T. subsequent to October 1963; SABS) and in others the use of either salt is optional (the identical AASHO and ASTM tests). The two salts yield very different results,however, the magnesium sulphate test apparently yielding a loss of about 50% more than that of a sodium sulphate test (ASTM D692-59T). The tests basically involve soaking the aggregate in a saturated sulphate solution for 16-18 hours (AASHO and ASTM), 19 hours (D.O.T. and SABS) at 21°C (AASHO and ASTM), 25°C (D.O.T.) or 23°C (SABS) after which the material is dried in an oven at 110°C for 4 hours (D.O.T.), $4\frac{1}{2}$ hours (SABS) or to constant weight (AASHO and ASTM). This procedure is then repeated until the required number of cycles have been attained, after which the material is washed free of sulphate and sieved. The percentage passing a sieve equal to (SABS and D.O.T.) or slightly finer than (ASTM and AASHO) the smaller sieve originally used to separate

the/ ...

the aggregate is then determined and this figure reported as the 'loss'. This latter procedure is to be preferred, since otherwise one piece of aggregate which say only loses a corner may pass through the larger sieve, thus yielding a large,but essentially spurious loss. Aggregates which say only break in half cannot really be considered as deleterious. This effect is well shown in Section 8.10.4. Even calcretes with relatively good 10% F.A.C.T. values perform poorly in the 20 cycle test. The significant point, however, is that the percentages passing all the finer sieves from ½" to 100 mesh are largely predictable from only the dry 10% F.A.C.T. value, whereas the percentage passing the ¾" sieve is not. In the case of a mixture of aggregate of different sizes tested by the AASHO or ASTM methods, the percentage loss for each fraction is then corrected (weighted) by multiplying by the percentage of the appropriate fraction originally present in the sample before testing. Addition of these corrected percentage losses then gives the total weighted average loss for the whole sample.

The sulphate soundness tests are lengthy, requiring about a day per cycle, relatively poorly reproducible and do not simulate weathering under conditions experienced in South Africa. Dr. H.H. Weinert (1964, personal comm.) and the author would prefer to see them replaced by a dry and soaked 10% F.A.C.T. test. The soundness tests have also been criticised by Orchard (1964) and Minty (1965) and the latter author has suggested that the 5 cycle Australian sodium sulphate test is too severe for Australian conditions. Certainly it was developed in a country (the U.S.A.) where climatic conditions are rather more severe than in either Australia or South Africa. It is believed that the Cape Roads Department have found the test useful in showing up poor shales which are not detected by a dry ACV. It is suggested that these shales should show up under a soaked ACV or 10% F.A.C.T.

Wet/ ...

Wet aggregate abrasion and crushing tests

Interest in wet tests has increased in recent years. Minor (1960) found that not only did the Los Angeles test not predict the degradation of certain Washington aggregates (mainly weathered basalts), but the results were downright misleading, some materials possessing LAAs as low as 12% causing failures and others with LAAs of 40-50% being satisfactory.

It is obvious, and Minor also stated this, that it is not only aggregate breakdown and the production of fines which must be considered, but also the nature of the fines. The fines produced in the normal LAA test are not, however, representative of those produced during degradation in service (Minor, 1960; Ekse and Morris, 1960) so that Atterberg limits on the fines are misleading, apparently being lower than those encountered in practice. Minor described a simple wet tumbling test which yielded results consistent with experience and Ekse and Morris and Erickson (1958) used a modified dry Los Angeles test without the steel charge, considering that the particle to particle abrasion gave results more consistent with practice. Erickson considered that freezing and thawing, and probably not traffic, was the cause of the degradation difficulties in Idaho. Two tests which on the basis of the foregoing should be particularly useful are the California 210-C wet shot rattler and the Texas 116-E wet ball mill. Both, however, do use some steel balls. The Texas Highway Department (1966) states that their wet ball mill test is more reliable than the Los Angeles test. The former test also has the advantage that it uses material in its natural state, i.e. it does not use just some size fractions.

A test in use for some time at the NIRR is the soaked 10% F.A.C.T. This is carried out in the usual

way/ ...

way except that the aggregate is soaked for 24 hrs and surface-dried prior to testing. The soaked strength expressed as a percentage of the dry strength is often very revealing. Loubser (1967) suggested that this should be at least 60% for shales, the dry values of which fell between 20 and 25 tons. Figures for calcretes are shown in Figure 8.16. It would be well worth while collecting a number of known good and poor materials (including some of the Cape's shales) and subjecting them to a dry and soaked 10% F.A.C.T. or ACV and the sulphate soundness test. A disadvantage of the test is that it does not normally produce sufficient fines for a plasticity index determination.

It is apparent, and this is also a conclusion of Erickson's (1960), that a test for the degradation of base course in service is needed and that existing tests, particularly the dry ones and even the sulphate tests, do not always provide the answer. Erickson suggested that a test for the whole material used was required, not just for a certain fraction of it. The Texas 116-E wet ball mill method and Minor's (1960) method seem particularly worthy of consideration.

7.8.3 A simple new pliers test

A new test, almost ludicrous in its simplicity, designed originally for nodular calcrete gravel road wearing courses has been described by Netterberg (1967). For reasons of economy, the soil constants and grading of gravel road materials are seldom tested in South and South West Africa, let alone the aggregate strength, but inspection of a number of failed or unsatisfactory calcrete gravel roads in South West Africa led the author to the conclusion that aggregate breakdown under traffic was the major cause involved. What was needed was a simple test which could be applied by any roadworker in the field

without/ ...

without recourse to laboratory testing.

As a solution of the problem of aggregate strength, a simple field pliers test is suggested for testing the strength of calcrete and other aggregate (particularly nodules). The test can be applied in the field by any roadworker armed with a standard pair of pliers and a knowledge of simple arithmetic. The method of test involves obtaining 100-200 pieces (if exactly 200 pieces are used the arithmetic is simplified even further) of the air dry $\frac{1}{2}$"-$\frac{3}{4}$" aggregate portion of the nodular calcrete gravel by sieving or hand picking and firstly trying to break the pieces between the thumb and forefingers, using both hands at once. The unbreakable pieces are then tested with a standard pair of pliers (Hazet 1850-3/7") in the concave serrated portion of the jaws. In both cases more or less maximum strength should be exerted, though this aspect does not appear to be particularly critical. The total percentage passing the fingers could be called the aggregate fingers value (AFV) and the percentage passing the pliers the aggregate pliers value (APV). The test as a whole could be called the aggregate pliers test (A.P.T.). While these names, particularly the first, perhaps sound a little silly, they are both brief and expressive.

In routine A.P.T. testing, the pieces broken in the pliers, but which could not be broken in the hands and the pieces unbroken by the pliers of all the samples tested were kept and their respective 10% F.A.C.T. values determined. It was found that the material unbroken by the pliers had a 10% F.A.C.T. value of 10 tons, while that unbreakable by the hands was 5 tons. No serious attempt has been made to carefully correlate the A.P.T. test with any other, but plotting what data is available on Figures 7.12 and 7.13 shows that the test can yield a fair idea of the dry 10% F.A.C.T. value of calcrete

nodules/ ...

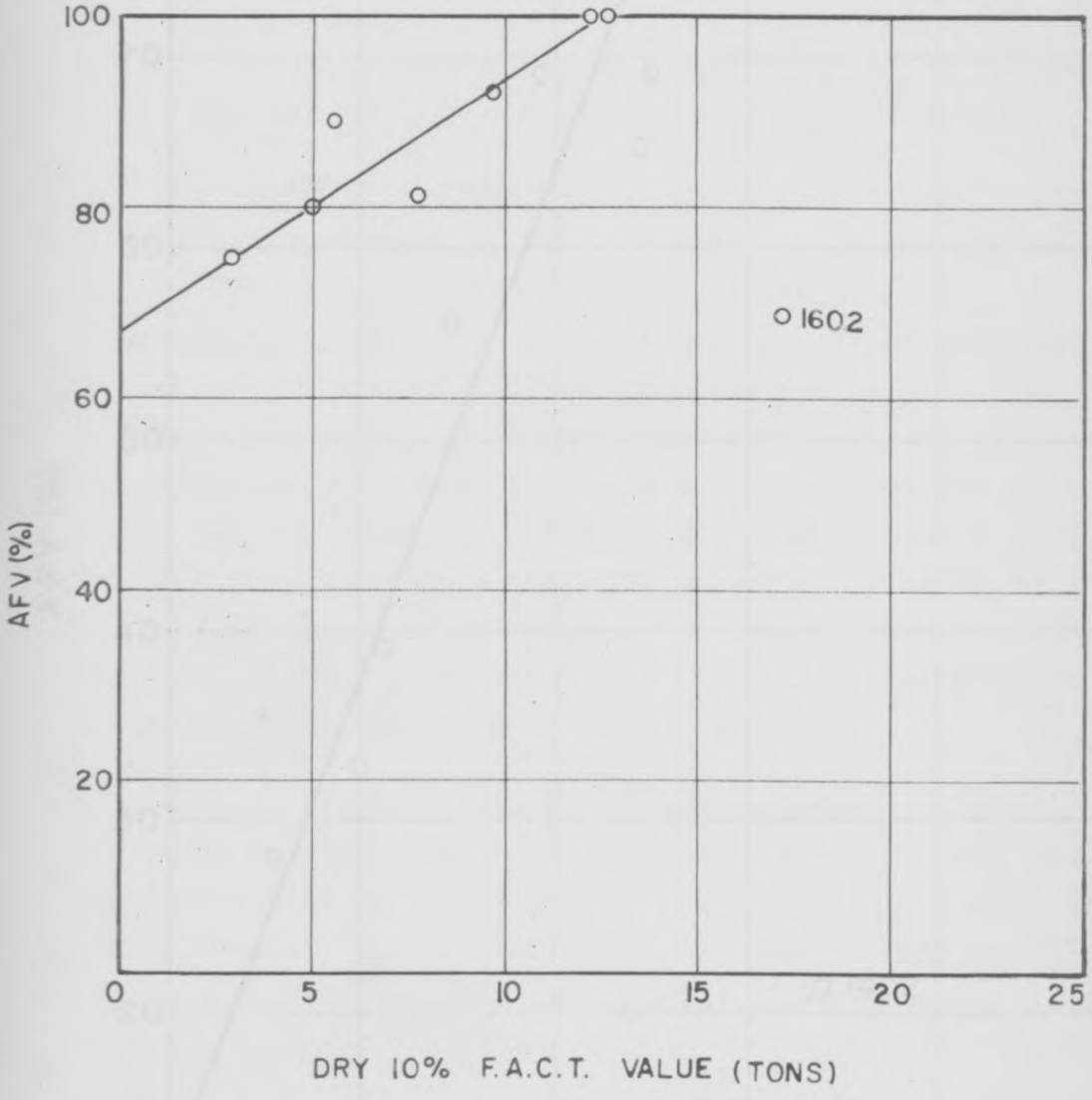


FIGURE 7·12

RELATION BETWEEN THE AFV AND 10% F.A.C.T. VALUE FOR CALCRETE NODULES.

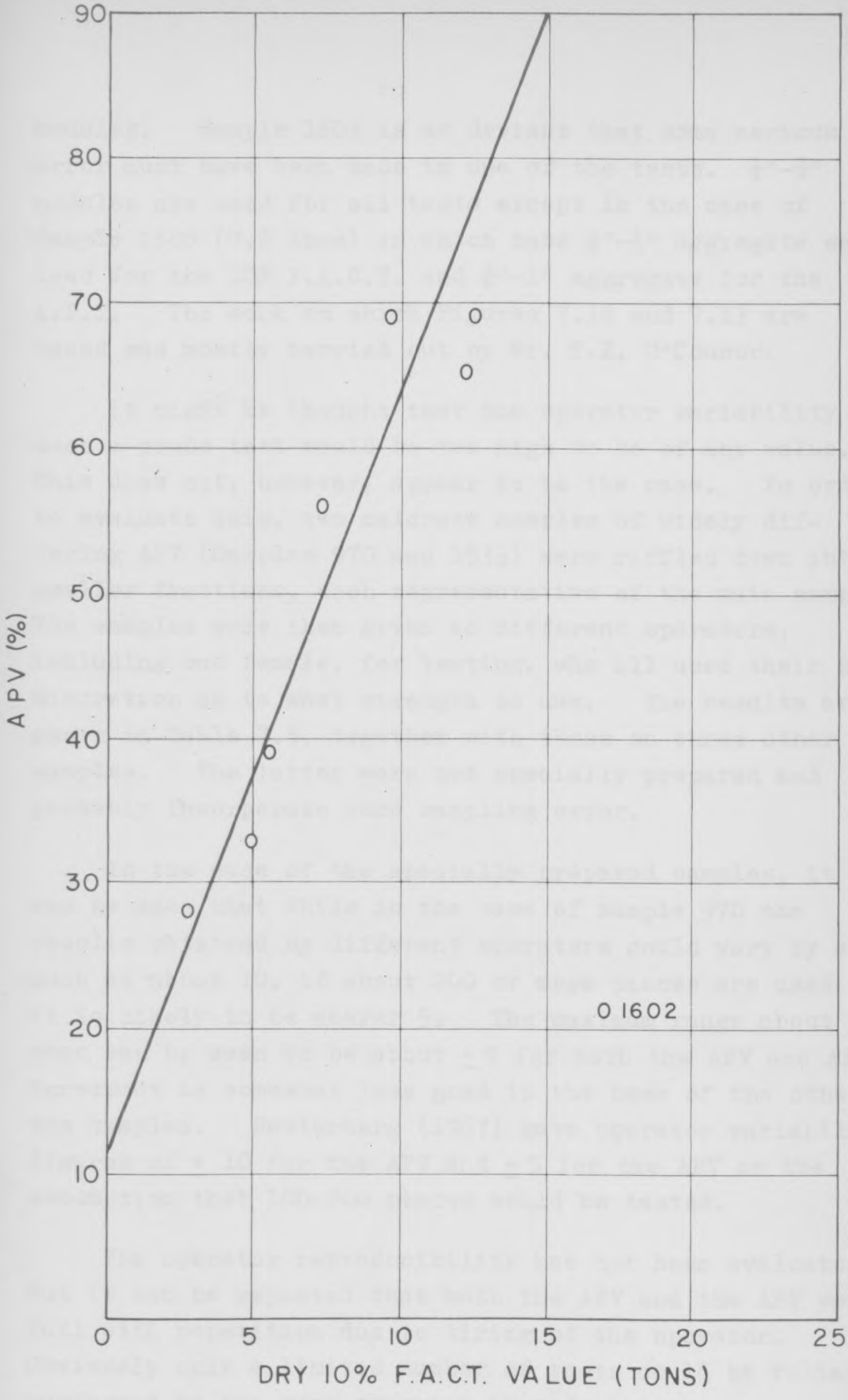


FIGURE 7.13
RELATION BETWEEN THE APV AND THE 10% F.A.C.T. VALUE FOR CALCRETE NODULES

nodules. Sample 1602 is so deviant that some serious error must have been made in one of the tests. $\frac{1}{2}$"-$\frac{3}{4}$" nodules are used for all tests except in the case of Sample 2588 (7.6 tons) in which case $\frac{3}{8}$"-$\frac{1}{2}$" aggregate was used for the 10% F.A.C.T. and $\frac{3}{4}$"-1" aggregate for the A.P.T. The work on which Figures 7.12 and 7.13 are based was mostly carried out by Mr. T.E. O'Connor.

It might be thought that the operator variability of such a crude test would be too high to be of any value. This does not, however, appear to be the case. In order to evaluate this, two calcrete samples of widely differing APV (Samples 970 and 1533) were riffled down into smaller fractions, each representative of the main sample. The samples were then given to different operators, including one female, for testing, who all used their own discretion as to what strength to use. The results are shown in Table 7.5, together with those on three other samples. The latter were not specially prepared and probably incorporate some sampling error.

In the case of the specially prepared samples, it can be seen that while in the case of sample 970 the results obtained by different operators could vary by as much as about 10, if about 200 or more pieces are used it is likely to be nearer 5. The maximum range about the mean can be seen to be about ± 5 for both the AFV and APV. Agreement is somewhat less good in the case of the other two samples. Netterberg (1967) gave operator variability figures of $\pm$ 10 for the AFV and ± 5 for the APV on the assumption that 100-200 pieces would be tested.

The operator reproducibility has not been evaluated, but it can be expected that both the AFV and the APV would fall with repetition due to tiring of the operator. Obviously only a limited number of tests could be reliably performed by the same operator in a day.

The/ .....

TABLE 7.5 EVALUATION OF OPERATOR VARIABILITY IN THE DETERMINATION OF THE AFV AND APV

SAMPLE NUMBER	OPERATOR	AFV (TOTAL % PASSING FINGERS)	% PASSING FINGERS BUT FAILING PLIERS	APV (% PASSING PLIERS)	NO. OF PIECES TESTED
970	Mr.K.Prinsloo	53	22	31	77
970	Mr.M.Loubser	46	10	26	77
970	Mr.M.Makumla	42	20	22	77
970	Miss D.Holton	42	10	31	77
1533	Mr.T.O'Connor	98	11	87	313
1533	Mr.F.Netterberg	96	10	86	324
1533	Mr.J.Whittaker	100	15	85	272
Maximum range of results		11	12	9	-
Maximum range about the mean		+7 -4	±6	+3 -6	-
978	Mr.T.O'Connor	87	32	54	148
978	Mr.C.Broadbent	94	35	59	110
1020	Mr.T.O'Connor	75	12	63	161
1020	Mr.C.Broadbent	97	19	78	74
1569	Mr.T.O'Connor	91	43	48	207
1569	Mr.C.Broadbent	92	37	55	109
Maximum range of results		22	7	15	-
Maximum range about the mean		±11	±4	±8	-

The choice of pliers was purely accidental and the one used came from the tool kit of the vehicle in use when the test was first thought of. The test could be made more severe if desirable (e.g. for base course) simply by the use of larger pliers. The pliers could be readily calibrated against an ACV or 10% F.A.C.T. The test was never intended for base course, but could perhaps find application as a field test or as a laboratory test when insufficient material is available for one of the more sophisticated tests. Most construction control laboratories lack a suitable press for an ACV or 10% F.A.C.T. In view of the surprisingly good correlation in Figure 7.13, it is suggested that a minimum APV of 65% would be a reasonable requirement for base course (equivalent to a 10% F.A.C.T. value of 10-12 tons) for most roads.

Application of the aggregate pliers test to the problem of aggregate strength specifications for calcrete gravel road wearing courses is made in Section 9.8. The suggested test limits correlate well with known performance.

7.9 HARDNESS

The term 'hardness' is often used in civil engineering circles when referring to rocks, when what is really meant is strength, it being assumed that the two are synonymous. Whenever the term 'hardness' is used in this work, it can be taken to mean just that, i.e. the property measured by a Mohs or Vickers hardness test. The former test, that of attempting to scratch the object being tested with the standard minerals of the Mohs hardness scale is well known and needs no further description here (see for example Read, 1948; or Dana, 1932). The Vickers hardness test described for example by Tourenq (1967) involves applying a loaded diamond pyramid to the surface being tested and measuring the shorter diagonal (not the depth) of the impression thus formed by means of the

calibrated/ ...

calibrated microscope attachment. Tourenq found that the zone of influence of the penetrating diamond extended to an area equivalent to about 5 times the diameter of the impression, that the intrinsic hardness of minerals was only measured at loads of 1 kilogram or more (i.e. at lower loads, especially those less than 200 grams, the hardness obtained also depended on the load employed), that surface hardness was increased by polishing and that all minerals were anisotropic as far as hardness was concerned. The relation between hardness according to the Mohs, Vickers and Knoop scales is shown in Table 7.6. The Knoop and Vickers values are seen in most cases to be very similar. The agreement between Taylor's (1949) and Tourenq's figures is very good, especially considering the fact that Taylor used a load of 50 grams and Tourenq one of 1,000 grams or more. A plot of Mohs hardness against Vickers hardness is shown in Figure 7.14. Apart from a change in shape at Mohs hardnesses of 4.2 and between 9 and 10 the relation is linear. The differences in hardness between the minerals are very roughly equal to 50 Vickers up to a Mohs value of 4 and 400 Vickers thereafter, up to 9. The reason for orthoclase failing to fall on the line is obscure. It does not appear to be due to chance.

7.9.1 Correlation between hardness and strength

That strength is at least related to hardness is of course an everyday observation. The work of Tourenq (1967) shows in fact that there is a linear relation between the two in the case of limestones and calcretes, respectively.

Tourenq found that the compressive strength in kilograms per square millimetre of very pure calcitic limestones was equal to one eighth of the Vickers hardness (also expressed in kg/mm^2). This is equivalent to a

strength/ ...

TABLE 7.6 HARDNESS OF MINERALS ACCORDING TO THE MOHS VICKERS AND KNOOP SCALES

MINERAL	MOHS VALUE	SURFACE TESTED	VICKERS VALUE TAYLOR (1949)	VICKERS VALUE TOURENQ (1966)	KNOOP VALUE (k100 SCALE) GILL & SPENCE (1962) MEAN	KNOOP VALUE (k100 SCALE) GILL & SPENCE (1962) RANGE	KNOOP VALUE HODGMAN (1961)
Talc	1	-	47	20	21	16- 35	-
Gypsum	2	Cleavage	60	69	54	42- 67	32
Calcite	3	-	-	110	132	114- 177	135
Calcite	3	⊥ optic axis	105	-	-	-	-
Calcite	3	// optic axis	145	-	-	-	-
Calcite	3	Cleavage	136	-	-	-	-
Fluorite	4	-	200	180	188	171- 199	163
Fluorite	4	Cleavage	175	-	-	-	-
Apatite	5	-	659	500	476	427- 514	430
Orthoclase	6	-	714	720	682	547- 729	560
Quartz	7	-	-	1280	958	889- 985	820
Quartz	-	Fused silica glass	480	-	-	-	-
Quartz	7	⊥ optic axis	1103	-	-	-	-
Quartz	7	// optic axis	1260	-	-	-	-
Topaz	8	-	1648	1620	1435	1391-1561	1340
Corundum	9	-	2085	2000	2004	1802-2250	-
Corundum	9	Synthetic sapphire	2720	-	-	-	-
Diamond	10	-	-	-	7000	5500-8500	7000

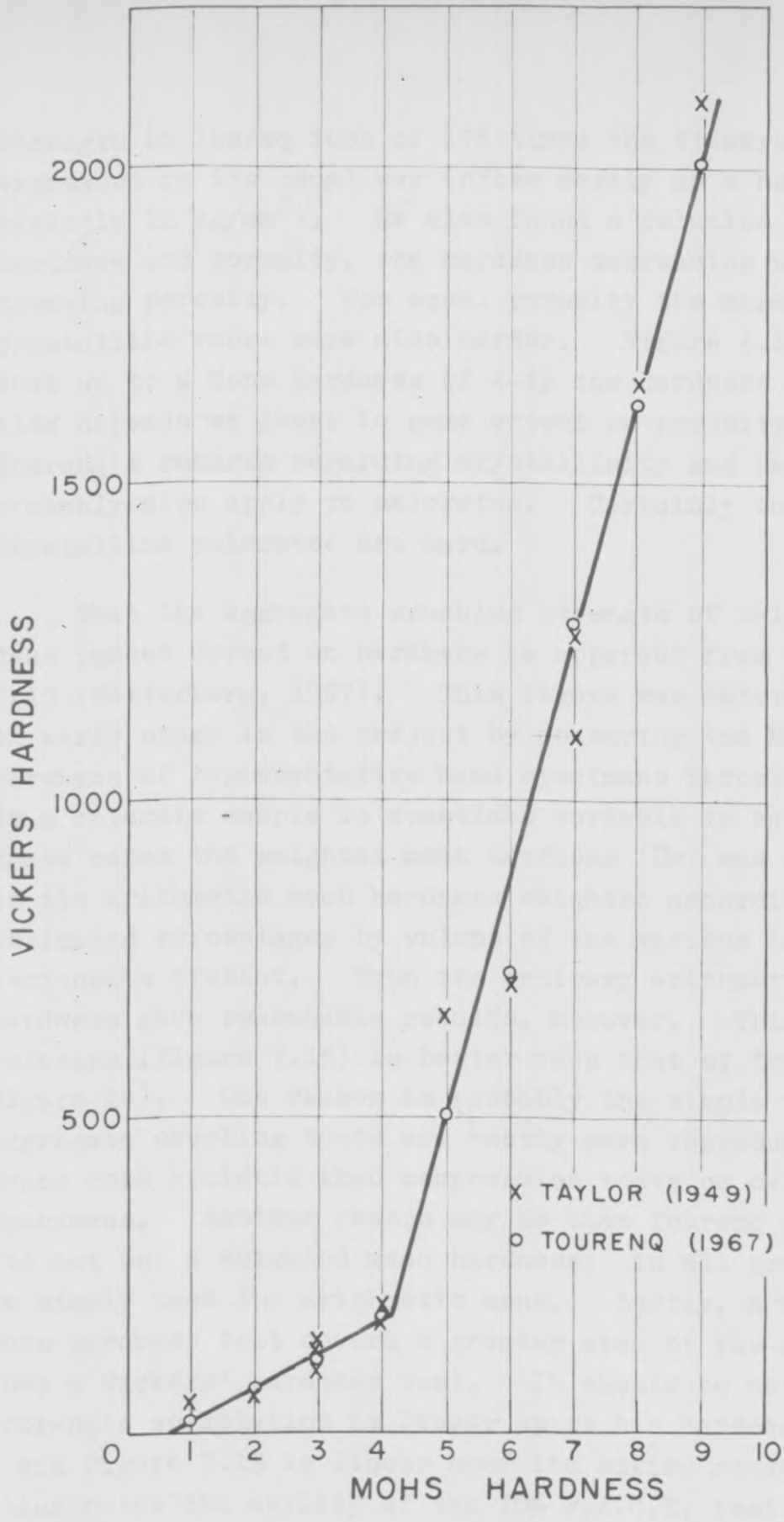


FIGURE 7·14

Relation between Vickers and Mohs hardness numbers for minerals

RG4-3192

strength in lbs/sq inch of 178 times the Vickers hardness expressed in its usual way (often merely as a number, but strictly in kg/mm^2). He also found a relation between hardness and porosity, the hardness decreasing with increasing porosity. For equal porosity the more finely crystalline rocks were also harder. Figure 4.13 shows that up to a Mohs hardness of 4-4½ the hardness of calcretes also depends at least to some extent on porosity or density. Tourenq's remarks regarding crystallinity and hardness probably also apply to calcretes. Certainly only cryptocrystalline calcretes are hard.

That the aggregate crushing strength of calcretes does indeed depend on hardness is apparent from Figure 7.15 (Netterberg, 1967). This figure was determined at an early stage in the project by measuring the Mohs hardness of representative hand specimens before crushing. As a calcrete sample is sometimes variable in hardness, in these cases the weighted mean hardness ($\overline{H}w$) was used, that is the arithmetic mean hardness weighted according to the estimated percentages by volume of the various hardness components present. Even the ordinary arithmetic mean hardness gave reasonable results, however. This correlation (Figure 7.15) is better than that of Tourenq (his Figure 14). One reason is probably the simple fact that aggregate crushing tests are vastly more reproducible and hence more reliable than compression tests on cylindrical specimens. Another reason may be that Tourenq apparently did not use a weighted mean hardness; in all probability he simply used the arithmetic mean. Lastly, a single Mohs hardness test covers a greater area of the sample than does a Vickers' hardness test. It should be noted that Tourenq's correlation is linear up to his hardness limit of 4 and Figure 7.15 is linear over its entire range. This illustrates the ability of the 10% F.A.C.T. test to cover a wide range of strength more satisfactorily than the ACV, which does not yield a linear correlation. Figure 7.16

shows/ ...

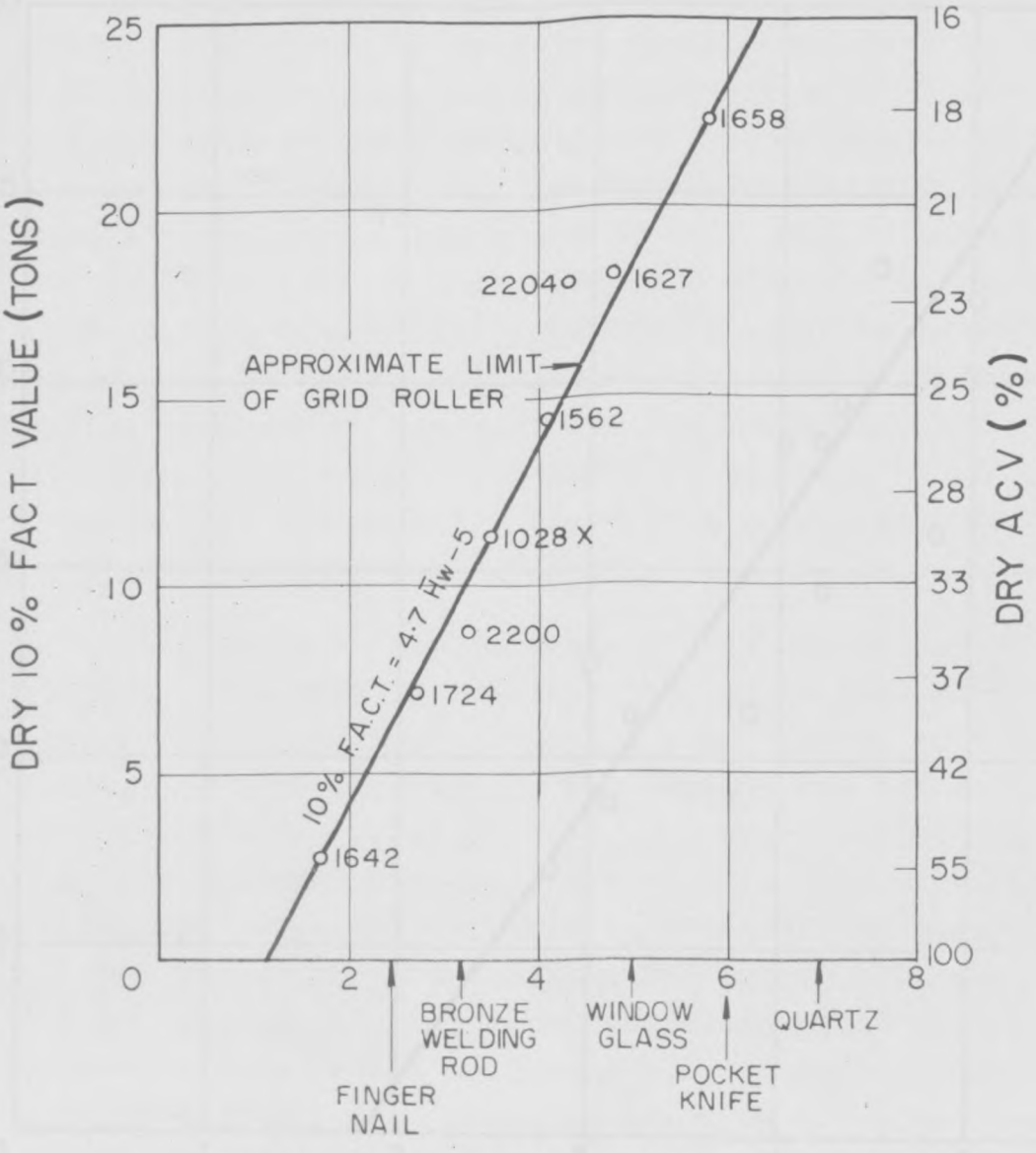


FIGURE 7·15

Relation between aggregate crushing strength and Mohs hardness by minerals on hand specimens before crushing.

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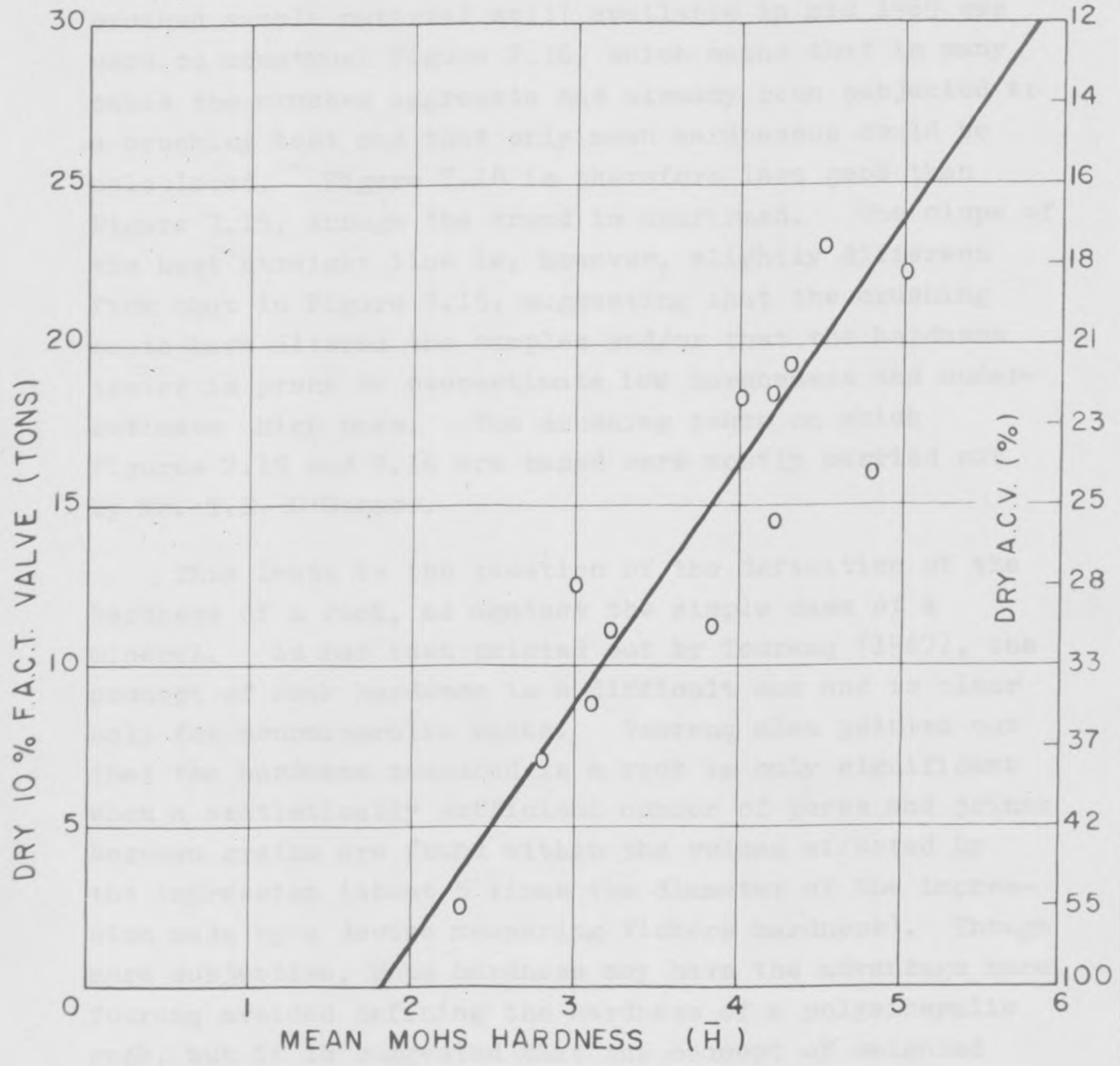


FIGURE 7·16

RELATION BETWEEN AGGREGATE CRUSHING STRENGTH AND MOHS HARDNESS BY POCKET HARDNESS TESTER ON CRUSHED AGGREGATE.

shows the relation between Mohs hardness and crushing strength found using the pocket hardness tester (to be described in the next section) on crushed aggregate. All crushed sample material still available in mid 1969 was used to construct Figure 7.16, which means that in many cases the crushed aggregate had already been subjected to a crushing test and that only mean hardnesses could be calculated. Figure 7.16 is therefore less good than Figure 7.15, though the trend is confirmed. The slope of the best straight line is, however, slightly different from that in Figure 7.15, suggesting that the crushing tests have altered the samples and/or that the hardness tester is prone to overestimate low hardnesses and underestimate high ones. The crushing tests on which Figures 7.15 and 7.16 are based were mostly carried out by Mr. T.E. O'Connor.

This leads to the question of the definition of the hardness of a rock, as against the simple case of a mineral. As has been pointed out by Tourenq (1967), the concept of rock hardness is a difficult one and is clear only for monomineralic rocks. Tourenq also pointed out that the hardness measured in a rock is only significant when a statistically sufficient number of pores and joints between grains are found within the volume affected by the impression (about 5 times the diameter of the impression made by a device measuring Vickers hardness). Though more subjective, Mohs hardness may have the advantage here. Tourenq avoided defining the hardness of a polymineralic rock, but it is suggested that the concept of weighted mean hardness as used on calcretes above may possibly be of wider application. Much would depend on the mode of measurement (e.g. Mohs or Vickers), the grain size and the strength of the intergranular bonds.

7.9.2 A new pocket Mohs hardness tester

The correlation between aggregate crushing strength

and/ ...

and hardness in Figure 7.15 was arrived at by determining the hardness of a 3 or 4 representative hand specimens removed from the sample before crushing to the $\frac{3}{8}$"-$\frac{1}{2}$" size used in the crushing tests. The ordinary Mohs hardness box of standard minerals was used for this purpose. While this is generally reasonably satisfactory, it is apparent that the mineral collection cannot be carried about in the pocket for field use in the same way that every geologist carries a hand lens. While a few common objects such as a finger nail, a bronze welding rod, a piece of window glass and a pocket knife can be useful aids, some of them are also inconvenient to use and carry about, as well as being variable in hardness. This led to the idea of making up a small, compact set of metals of different hardnesses to be carried on a key ring or made up like a set of feeler gauges. The latter design was eventually decided upon and a photograph of the prototype is shown in Plate 7.1.

The choice of suitable metals was made with the aid of Table 7.6, Tourenq's figures being used. The kind assistance of Mr. J.W. Borman and the late Dr. O.E. Lissner of the Metallurgy Division of the National Mechanical Engineering Research Institute of the CSIR in choosing the metals is acknowledged. The metals used are shown in Table 7.7. The shaping of the metals involved was carried out by the CSIR central workshop. The heat treatment required to attain the specified hardness was carried out by Mr. Borman, who can be consulted for details. The $6\frac{1}{2}$ blade should have been hardened to 900 Vickers, but 770 was the best that could be achieved. Tungsten carbides are available in Vickers hardnesses from about 1,100 to 2,000 and it is hoped that the production model will possess blades of Mohs hardnesses of 2 to 9 with a boart-tipped blade for 10.

After/ ...

TABLE 7.7 METALS USED IN POCKET HARDNESS TESTER

BLADE AND MOHS NUMBER	METAL	HARDENED TO: (VICKERS)
1	Pure aluminium	20
2	Phosphor bronze	69
3	Duralumin	110
4	Beryllium bronze	180
5	Spring steel	500
6	Tool steel	720
$\sim(6\frac{1}{2})$	High speed steel (SS 27/1W luna)	770

After assembling the device it was first tested against the standard minerals, the results being shown in Table 7.8.

TABLE 7.8 COMPARISON OF POCKET MOHS HARDNESS TESTER WITH THE STANDARD MINERALS

BLADE NUMBER	SCRATCHES	SCRATCHED BY	MEAN MOHS HARDNESS	REMARKS
1	Talc, gypsum(1)	Gypsum, finger nail, calcite	$2^{+\frac{1}{4}}_{-\frac{1}{2}}$	
2	Talc, gypsum, calcite(1)	Calcite, fluorite	$3^{+\frac{1}{4}}_{-\frac{1}{2}}$	Slightly softer than blade number 3
3	Gypsum, calcite(1)	Calcite(2) fluorite(2)	$3\pm\frac{1}{4}$	
4	Calcite, (fluorite(1,3))	Fluorite, apatite	$4\pm\frac{1}{4}$	
5	Fluorite, (apatite(3))	Apatite, orthoclase	$5\pm\frac{1}{4}$	
6	Apatite, orthoclase(1)	Orthoclase, quartz	$6\pm\frac{1}{4}$	Perhaps 5.8 or 5.9
$(6\frac{1}{2})$	Orthoclase, (quartz(3))	Quartz	$6\frac{1}{2}\pm\frac{1}{4}$	

(1) With loss of some metal (i.e.marks the mineral).
(2) With powdering of mineral.
(3) Mainly chipping away of edges, not a true scratch.

It/ ...

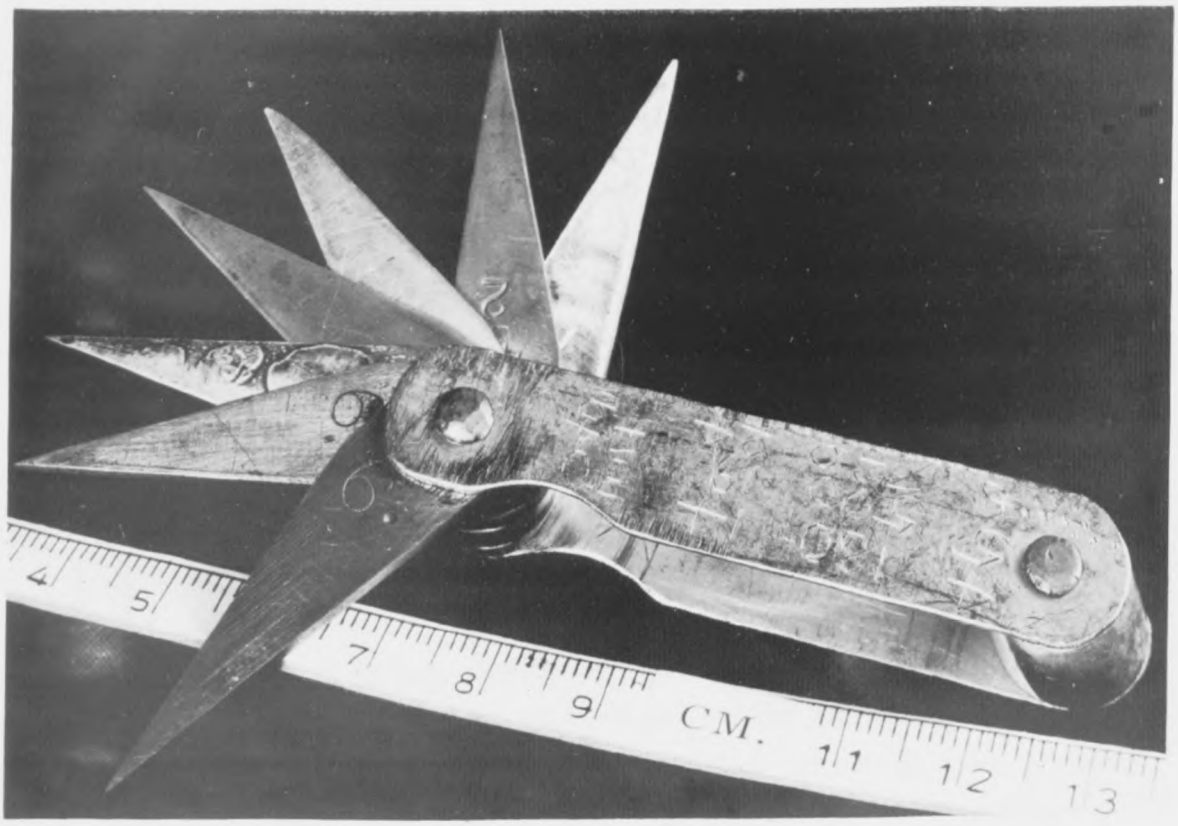


PLATE 7.1

Prototype pocket Mohs hardness tester. The blades are marked 1 to 6½ (6.) and the numbers scratched on the container are Mohs numbers and their equivalent Vickers numbers, 10% F.A.C.T. values and ACVs. The device is suitable for the determination of Mohs and Vickers hardnesses of minerals and metals as well as the aggregate crushing strengths of calcretes. Length when closed 6 cm (2½ inches), weight 40 grams (1½ oz.). *Photo: Graphic Arts, CSIR.*

It is apparent that although the Vickers hardnesses are as given by Table 7.6, blades number 1 and 2 are much too hard when tested against the minerals. No suitable metal for blade 1 has so far been found, but this blade would rarely be used in practice anyway. All the other blades are equal within experimental errors to the equivalent minerals. The following points worthy of note arose from this trial:

1) The tool in each case appeared slightly harder than the object. In other words the mineral (or blade) was both capable of just scratching and just being scratched by the blade (or mineral) of supposedly equivalent hardness. Trials of each standard mineral against a fragment of itself showed that each mineral was also able to just scratch and be scratched by a fragment of itself. Trials of the metal blades against another blade of the same hardness produced the same effect. The cause of this phenomenon is presumably due to the high stress at the point of contact. Both tool and object are probably deformed, but this is most noticeable in the case of the object. The metal blades generally appeared to be a little harder than the equivalent mineral (i.e. they scratched the mineral more easily than they are scratched by it), presumably because their points did not crumble and break under pressure. This tool effect diminished with increasing hardness and was hardly noticeable in the case of a $6\frac{1}{2}$ blade against another $6\frac{1}{2}$ blade. In the case of corundum against corundum it could only be observed under a stereo microscope.

2) The blades marked (due to a loss of metal) minerals and blades of greater hardness. They usually also slightly marked minerals of equal hardness while at the same time also slightly scratching them.

3)/ ...

3) Macroscopic, stereo microscopic and hand lens observations showed that it was important to distinguish between:

(a) genuine scratching (grooving by cutting),
(b) marking (removal of metal from blade),
(c) polishing (removal of oxide film from blade),
(d) brittle fracture of harder minerals under pressure from a softer blade, and
(e) indentation (grooving not by cutting but by shear failure or plastic flow due to pressure).

(d), (e) and to a lesser extent (c) could be mistaken for genuine scratches if the test was carelessly carried out. (d) seemed to be the predominant mode of scratching where minerals were scratched by other minerals. (e) probably often contributes to the genuine scratch.

4) It was concluded that the action of metals on minerals was sufficiently similar to that of minerals on minerals for metals to be used as substitutes for minerals in Mohs hardness testing.

The hardness tester was then tested against actual calcretes, the hardness of which had previously been determined by means of the standard minerals. The results are shown in Table 7.9.

It can be seen that the agreement between the two methods is quite good. Differences of up to half a unit are, however, possible, particularly in the case of very sandy calcretes. In all cases the hardness of the matrix was sought, and sand grains were disregarded, since at less than about 50% sand grains they merely float in the matrix anyway. Hardnesses on calcretes with more than 50% sand grains are probably unreliable and difficulty was usually experienced with those containing more than about

30%/ ...

TABLE 7.9 COMPARISON OF CALCRETE HARDNESSES DETERMINED WITH STANDARD MINERALS WITH THOSE OBTAINED WITH THE METAL POCKET HARDNESS TESTER

SAMPLE NUMBER	PERCENT SAND GRAINS(1)	MOHS HARDNESS(2)		DIFFERENCE
		BY MINERALS	BY METALS	
898	60	7	6.5	0.5
1000A	0	4.0	4.0	0.0
1000B	0	4.3	4.5	0.2
1007	10	3.2-4.2	3.0-4.0	0.2
1562A	10	5.0	4.8	0.2
1562B	60	4	3.5	0.5
1627	10	4.5	4.3	0.2
1638	60	6	6	0
1639	20	5.5	5.5	0.0
1658A	5	6.0	5.5	0.0
1693	5	5	5	0
1725	30	5	5	0
1728	50	6	6	0
1746	2	4	4	0
1810	20	4	4	0
1859	10	4.7	4.5	0.2
2223	0	2.5	2	0.5
2516	2	3 - 4.5	3 - 4.5	0.0
2516	2	3 - 4.5	3 - 4.5	0.0
2517	20	2.7	2.7	0.0
2517	10	2.7 - 4	2.7 - 4.5	0.5
2518	5	1.5	< 2	0.5
MEAN DIFFERENCE				0.2
RANGE				0.0-0.5
NUMBER OF SAMPLES TESTED				22

(1) Estimated in hand specimen (by volume).

(2) Of the carbonate matrix. An accuracy of $\pm$ 0.2 units is implied when decimals are stated. An accuracy of $\pm$ 0.4 is implied when only whole numbers are stated.

30% sand grains. In all cases a definite (be it only under a hand lens), reasonably continuous scratch was required. In doubtful cases the highest hardness was taken and it is suggested that this should be standard practice when using the hardness tester. In some cases sawn surfaces yielded different hardnesses to fractured surfaces. Very sandy calcretes yielded more consistent results when sawn surfaces were used.

Figure 7.16 confirms that the hardness tester can be used to estimate aggregate crushing strengths of calcretes. It may possibly also be usable on some other rocks, particularly reasonably homogeneous and fine grained rocks like limestones, dolomites, shales and possibly even fine grained igneous rocks. Coarse grained rocks might be amenable to the weighted mean hardness concept. The most accurate means of estimating hardness for crushing strengths is probably to take the weighted mean hardness of representative hand specimens before crushing.

7.10 SELF-STABILIZATION

Self-stabilization, or the improvement in strength said to be exhibited by some pedogenic materials, is a controversial subject. Some workers of wide experience in the road materials field have not observed this phenomenon. Others have, and Nascimento et al (1963 and 1965) have gone so far as to publish details of a test to predict this phenomenon. Materials said to exhibit self-stabilization include laterites, ferricretes and calcretes. The subject will be discussed in detail in Section 8.13 and this section will be confined to an examination of possible means of predicting and detecting this phenomenon. For the purposes of this section then, it will be assumed that self-stabilization of engineering significance does indeed take place.

7.10.1/ ...

7.10.1 Prediction of self-stabilization

Petrifaction degree

Nascimento's test is very simple. A dry soil pat of -40 mesh material is first prepared in an ungreased shrinkage limit mould and its shrinkage limit (SL) determined in the usual way. The pat is then allowed to absorb water by standing it on a porous plate which is submerged in water only to the top of the plate. After 24 hours the moisture content of the wet pat is determined. This is the absorption limit (AL). The petrifaction degree (PD) is then defined as:

$$PD = \frac{SL}{AL}$$

the theory being that for complete petrifaction (PD = 1), water would only be absorbed up to the shrinkage limit. In the 1965 paper the method was extended to include undisturbed samples. In neither paper was any PD limit suggested and it was merely stated that values of PD close to unity were thought to offer good prospects for petrifaction (self-stabilization) in practice. Remoulded PDs of nearly unity were obtained on some laterites. A study of Nascimento's results seems to indicate that PD values above about 0.37 may be significant, but by private communication (1969) Nascimento has stated that he has not yet found it possible to fix limits.

While Nascimento's method is theoretically sound, the following experiment was carried out as a check on it. The PDs of two calcretes were first determined in duplicate in the usual way, after which the tests were repeated, in duplicate, on material to which (a) 4% ordinary portland cement and (b) 4% high calcium lime (Rotalym) had been added. The specimens in their moulds were first cured for 3 days in a humid room nominally at 100% relative humidity and room temperature (about 20°C at the time)

before/ ...

before drying in the oven at 105-110°C and determining the petrifaction degree in the usual way. The results are shown in Table 7.10.

TABLE 7.10 PETRIFACTION DEGREES BEFORE AND AFTER STABILIZATION

SAMPLE NUMBER	ADDITIVE	CURING TIME (DAYS)	LIQUID LIMIT %	MEAN SL* %	MEAN AL %	MEAN PD
1569	None	0	24.9	19.8	39.6	0.50
	4% cement	3	-	34.7	43.2	0.81
	4% lime	3	-	31.5	33.6	0.95
2116	None	0	26.1	14.7	36.1	0.41
	4% cement	3	-	23.3	21.4	1.09
	4% lime	3	-	28.8	23.5	0.93

* Specific gravity method
Operator: Mr. T.E. O'Connor

It can be seen that in both cases the petrifaction degree was considerably increased by stabilization to values approaching or even in excess of unity. The method must therefore be regarded as valid as an indicator of cementation under the conditions of test. Petrifaction is accompanied by both an increase in the shrinkage limit and a decrease in the absorption limit. It is also interesting to note that the test differentiates lime reactive calcretes from non-lime reactive calcretes. Sample 1569 is known to react very well with lime to yield an extremely high CBR, while Sample 2116 is known to yield better strengths with cement than with lime (see Chapter 8). The stabilized specimens of 2116 at the absorption limit were much harder than those of 1569.

One problem with the test is that the material tends

to/ ...

to crack in the mould while drying or due to sticking on being removed. The result is that sometimes only small fragments weighing less than 10 grams are available for the determination, with a resultant loss of accuracy (the whole pats weigh around 30 grams). This also means that the ASTM D427 optional shrinkage limit method using the solid particle specific gravity has to be used, with a commensurate increased probability of error. Agreement between duplicate samples in Table 7.10 was poor in such cases, differences of up to 8 units in the shrinkage limit and 7 in the absorption limit being encountered. Cracking due to drying can probably be reduced or eliminated by slower drying, or more constant moisture conditions in the case of stabilized samples. The moulds cannot be greased since this would interfere with the water absorption, but coating them with some kind of plastic such as that used to form non-stick frying pan bottoms might be the answer. Such a plastic (Teflon?) is available in aerosol form under the trade name of Pot 'n Pan.

Another experiment was carried out to evaluate the effect of two different drying temperatures. The results are shown in Table 7.11.

The shrinkage limit for the appropriate specimen at 105°C was used in the calculation of the petrifaction degrees at 55°C. In retrospect, the shrinkage limit should have been determined in each case. Good agreement was obtained in the measurement of both shrinkage limit and absorption limit, the values obtained seldom differing by more than 1 unit.

It can be seen that the drying temperature seems to make little difference to the result. In some cases the use of CO_2- saturated water increased the petrifaction degree, but in others it apparently caused a decrease. The difference may be within experimental error and

therefore/ ...

TABLE 7.11 EFFECT OF DRYING TEMPERATURE AND CARBON DIOXIDE CONTENT OF MOULDING WATER ON PETRIFACTION DEGREE

SAMPLE NUMBER	DRYING TEMPERATURE °C	MOULDING WATER	LIQUID LIMIT %	MEAN SL* %	MEAN AL %	MEAN PD
2114	105	Fresh distilled	40.4	23.5	46.5	0.51
	105	CO_2- saturated	-	21.5	40.0	0.54
2116	105	Fresh distilled	26.1	17.5	46.5	0.38
	105	CO_2-saturated	-	16.0	40.0	0.40
	55	Fresh distilled	-	-	40.5	0.43
	55	CO_2-saturated	-	-	43.0	0.37
2223	105	Fresh distilled	24.0	23.5	46.5	0.51
	105	CO_2-saturated	-	24.0	48.5	0.50
	55	Fresh distilled	-	-	46.5	0.51
	55	CO_2-saturated	-	-	46.5	0.52

*Specific gravity method

therefore not significant. Theoretically, the CO_2-saturated water should have increased the solubility of carbonate during moulding so that more carbonate could be precipitated as a cement on drying, thus increasing the petrifaction degree. Soaking in ordinary water should not have dissolved all the bonds thus produced.

It is of interest to note that in all cases the absorption limit exceeded the liquid limit. By this criterion the three samples would probably be regarded as non-self-stabilizing by Nascimento et al. It must be remembered, however, that the liquid limit is determined in a continuously disturbed state, while the shrinkage limit and absorption limits are not. Further data for a number of

calcretes/ ...

calcretes is shown in Appendix H. These were all determined using ordinary distilled water and drying at 105°C. The highest PD obtained in this fashion was 0.79 (Sample 1567).

Table 7.12 shows the effect of wetting and drying cycles on the petrifaction degree. Samples were prepared as in Table 7.11 with distilled and CO_2- saturated distilled water and without removal from their moulds subjected to wetting and drying cycles. Each cycle consisted of 24 hours wetting with CO_2- saturated or plain distilled water and 24 hours drying at 105°C. Wetting consisted of keeping the pat wet, but not swimming in water by adding water from a wash bottle from time to time, taking care to cause the minimum disturbance to the pat. Ordinary distilled water was employed for the determination of the absorption limit in all experiments. Results missing from Table 7.12 are due to disintegration of the pats on attempted removal from the moulds.

It can be seen that 10 wetting and drying cycles caused the petrifaction degree to rise in every case, in two cases to approach unity. Sample 1569 yielded a value exceeding that attained with 4% cement (Table 7.10). In most cases the shrinkage limits rose by several units (up to 15 units in the case of 2114). It is known that the shrinkage limit normally rises when soil is stabilized with lime or cement. It would appear that all of these samples are potentially self-stabilizing if subjected to wetting and drying cycles (as distinct from only a drying cycle), particularly Samples 2114 and 2555. The CBR of the latter is known to improve with ordinary 7 day curing as for a stabilized sample from 83 to 149 (Mr. J. Caiger, personal comm.).

Pozzolanic PI

The plot of water content against the log of the

number/ ...

TABLE 7.12 EFFECT OF WETTING AND DRYING CYCLES ON THE PETRIFACTION DEGREE

SAMPLE NUMBER	NO. OF CYCLES	WATER	MEAN SL %	MEAN AL %	MEAN PD %
1569	0	Distilled	19.8	39.6	0.50
	10	Distilled	-	-	-
	10	CO_2-saturated	31.3	37.5	0.83
1602	0	Distilled	24.1	43.0	0.61
	10	Distilled	33.2	44.6	0.75
	10	CO_2-saturated	29.9	45.3	0.65
2114	0	Distilled	23.5	46.5	0.51
	10	Distilled	35.7	42.8	0.84
	10	CO_2-saturated	39.3	41.0	0.96
2223	0	Distilled	23.5	46.5	0.51
	10	Distilled	26.8	33.0	0.81
	10	CO_2-saturated	-	-	-
2367	0	Distilled	21.7	~52	0.42
	10	Distilled	-	-	-
	10	CO_2-saturated	27.0	40.0	0.68
2555	0	Distilled	35.6	22.2	0.63
	10	Distilled	22.5	27.2	0.83
	10	CO_2-saturated	31.1	32.0	0.97

Operators: Messrs. T.E. O'Connor and F. Netterberg

number of blows in the liquid limit determination to give the familiar flow curve is nothing more than a plot of water content against shear strength, which apparently varies from 0.2 - 1 psi at the liquid limit to 10 - 20 psi at the plastic limit (see Sections 7.3 and 7.4). This led Mr. A.A.B. Williams (1967, personal comm.) to suggest that if calcretes self-stabilized so as to possess a greater strength at the plastic limit than 20 psi, they must then also possess a lower true PI. This true PI he called the pozzolanic PI (PPI). If the flow curve remained straight on the semilog plot from the liquid limit to the plastic limit the pozzolanic PI is then:

$$PPI = PI \frac{2PI}{\log s_{PL}}$$

where PI is the measured PI and s_{PL} is the shear strength at the plastic limit.

In order to test this hypothesis, 4" x 2" cylindrical specimens of minus 40 mesh material from 2 nodular calcretes and one powder calcrete were compacted to 105 lbs/cu ft dry density at moisture contents corresponding to the plastic limit. The specimens were then cured for 7 days at constant moisture content and tested in unconfined compression. Some difficulty was experienced in maintaining the moisture content, but after several trials a layer of aluminium foil wrapping followed by a layer of grease was found to be reasonably effective and sufficient uncracked specimens were eventually obtained for testing. The results are shown in Table 7.13.

The shear strength is taken to be half the unconfined compressive strength.

While in no case was the moisture content at test exactly equal to the plastic limit, it is apparent that

the/ ...

TABLE 7.13 STRENGTH OF CALCRETE SOIL FINES NEAR THE PLASTIC LIMIT

SAMPLE NO.	PI %	I_t	PL %	MOISTURE CONTENT %	UCS psi	MEAN SHEAR STRENGTH psi	PPI %
2114	14.3	0.8	26.1	28.2 27.8 27.6	4.5 4.4 4.3	2.2	27.4
2116	10.7	~2.2	15.4	17.4 17.4 17.5	9.0 10.0 9.1	4.7	15.6
2223	6.5	1.4	17.5	16.9 16.7 16.3	7.8 8.5 9.7	4.4	9.7

Operators: Messrs. T.E. O'Connor and F. Netterberg

the three samples tested did not possess strengths anywhere near 20 psi. Their PPIs were thus greater than their measured PIs. It is of interest to note that the toughness index does place these three calcretes in their correct order as far as their strength at the plastic limit is concerned. All three calcretes possess comparatively low toughness indices, however. In view of this the pozzolanic PI formula was also expressed in terms of toughness indices. The toughness index I_t is equal to $\log \left(\frac{s_{PL}}{s_{LL}}\right)$ where s_{LL} is the cohesion at the liquid limit, here assumed to be 0.2 psi. The pozzolanic PI formula then becomes:

$$PPI = PI \frac{2}{\log (5 s_{LL} \text{ antilog } I_t)} = \frac{2\ PI}{I_t}$$

$$= \frac{2\ I_p}{I_t} \text{ using international symbols.}$$

When $I_t = 2$ (i.e. $s_{PL} = 100\ s_{LL}$), PPI = PI.

Inspection/...

Inspection of Tables H.1 to H.4 shows that many calcretes possess toughness indices of more than two. Values range from 0 to infinity. Taking Sample 1625 as an example (I_p = 21.3, I_t = 26.6) we get

$$PPI = \frac{2 \times 21.3}{26.6} = 1.6$$

This approach appears worthy of further study. The shear strengths of a number of calcretes of varying toughness index should be measured, both at the liquid and the plastic limit. Vane shear tests (or triaxials on the plastic limit specimens) should yield more reliable values of shear strength than unconfined compressive tests.

Curing

Mr. J.A. Caiger (1967, personal comm.) has found the CBR of some reddish nodular calcretes from South West Africa to increase from about 80 to about 110 or more on curing for 7 days as for a stabilized CBR. Not all the samples cured showed any increase, however, and some actually showed a decrease. The effect of wetting and drying cycles on one similar sample kindly provided by Mr. Caiger is shown in Section 8.13 (Sample 2555)
The Cape Roads Department (1956) has cured some calcretes from the Port Elizabeth area for 7 and 28 days with no increase in CBR. Of the two chief mechanisms postulated to account for self-stabilization in calcretes (oxidation of ferrous iron and solution and reprecipitation of carbonate), only stabilization caused by the oxidation of ferrous iron can be expected to be possibly detectable by mere curing.

Wetting and drying cycles

The effect of wetting and drying cycles in increasing the petrifaction degree and (usually) the shrinkage limit has already been shown. Several calcretes were compacted

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in CBR moulds and subjected to wetting and drying cycles. After cycling, nearly all specimens exhibited an increase in the 4 day soaked CBR, of up to three times the original value. The detailed results of the tests will be given in Section 8.13.2. It is suggested that any test attempting to predict self-stabilization should employ several cycles of wetting and drying, not just a single cycle as employed in the petrifaction degree test. The latter test would probably not detect any self-stabilization due to carbonate.

Da Silva et al (1967) have also noted that the CBR of four materials in Angola composed largely of ferric oxides increased (almost doubled) in CBR after 5 or 10 24 hour wetting and drying cycles. These materials possessed petrifaction degrees of 0.82-1.00 and shrinkage limits of 24-36.

It should be noted that an increase in wet strength after a drying cycle is not unknown in other soils. McDowell (1946) found that dry curing of compacted soils having PIs of less than about 15 could result in as much as a $2\frac{1}{2}$ fold increase in wet strength after dry curing and that this could probably also be realised during construction. The newly developed Texas Triaxial test incorporated an 8 hour period of dry curing at 140°F (60°C) for this reason and a modified version of this dry curing is still in use in the standard test (Texas Highway Department, 1966). McDowell's soils included some caliches. An increase in soaked CBR on drying at 60°C has also been noted by Zalazar (1963; in Barata, 1969) and Barata (1969) and an increase in triaxial value and a decrease in stabilometer value by Zalazar. It would appear that neither Zalazar's nor Barata's soils were calcretes.

Variation of the partial pressure of CO_2

An air tight pressure tank large enough to take

several/ ...

several CBR moulds was fitted with pipes to allow CO_2 to be bubbled into the water in it. CBR moulds containing compacted calcrete were placed in the tank and the CO_2 bubbled through for 30 minutes. The tank was then closed and pressurised with CO_2 to 5 psi. After 12 hours the pressure was released and the tank allowed to stand for a further 12 hours with the tap open to the atmosphere. This constituted one cycle. By this means the partial pressure of CO_2 in the system was varied from about one atmosphere to about 1.3 atmospheres, thus varying the solubility of the carbonate in the calcrete. After 10 cycles the CBR moulds were removed, soaked and tested. The only results available are on Sample 2114, two CBR moulds of which yielded Modified AASHO CBRs of 65 and 70 as against the usual 55-65 of this sample, a very small to negligible increase. Further experiments are planned employing larger partial pressure variations, between $P_{CO_2} = 3.2 \times 10^{-4}$ atmospheres (obtained by bubbling air through) and one or two atmospheres of CO_2 (obtained as outlined). Even lower partial pressures of CO_2 could be obtained by bubbling 'CO_2-free' air or any 'CO_2-free' gas through in place of ordinary air.

An increase of CBR in this experiment is considered to demonstrate the possible importance of solution and reprecipitation of carbonate as a self-stabilization mechanism at constant temperature and moisture content. The apparatus is maintained at 25°C.

Redox potential

Any material which can undergo self-stabilization due to oxidation of ferrous iron must be in a reduced state. Such a material will possess a negative redox potential ($E_{Red.}$ or E_H). If the redox potential is positive the system is in an oxidised state and the ferrous iron will tend to become oxidised. If the working and construction procedure is such that a material is excavated with a

negative/ ...

negative redox potential, this is more or less maintained until after the material is compacted, and then the material is allowed to oxidise to yield a positive redox potential (for example by drying out), then self-stabilization due to oxidation of ferrous iron is theoretically possible. An extremely reduced soil possesses a redox potential of about -500 millivolts, while a very well oxidised soil possesses a redox potential of about +800 millivolts (Jackson, 1956). It should be noted that just as the pH measures the intensity of H^+ and titration with OH^- is necessary to establish the amount present, so does $E_{Red.}$ measure the electron accepting intensity and titration with a strong oxidising agent such as $KMnO_4$ is necessary to determine the amount of reducing substance present. $E_{Red.}$ should therefore be a qualitative indicator of potential self-stabilization due to oxidation, and titration with $KMnO_4$ a quantitative measure.

The measurement of $E_{Red.}$ is a simple process no more complicated (apart from a larger equilibration time) than the measurement of pH. A redox electrode is used with any pH meter which also reads in millivolts. Metrohm make a convenient combined redox electroce. . Details of general methods may be found in most textbooks on inorganic chemical analysis, while Jackson (1956) gives a procedure for soils. It should be noted that $E_{Red.}$ will be affected by drying. Classification of materials by $E_{Red.}$ and pH might be revealing. The reduction (redox) potential of the soil scientist is the same as the reduction-oxidation potential and minus the oxidation potential ($E_{Ox.}$) of the chemist. The redox potentials of calcretes are unknown.

Upward leaching

Water was fed to the lower perforated surface of CBR moulds of compacted calcrete in an oven at 70°C via a constant head device adjusted to theoretically keep the water table level about half way up the compacted material. The combined water storage reservoir and constant head

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device was in the same oven as the CBR moulds, so that the whole experiment was roughly at constant temperature. The filter paper, perforated plate and annular weight were removed,and in this fashion water was caused to migrate upwards through the compacted material to evaporate near the surface. It was hoped to cause a cementation of the upper portion of the specimens in this fashion due to the solution of carbonate in the lower part of the material and its crystallisation near the surface, thus simulating what was thought to be happening in the road itself. To date only results on Sample 2114 are available. After 12 days in the oven and 4.5 litres of water had passed through two specimens (presumably about 2.25 litres each), the soaked CBR of the two specimens was determined as 67 and 90. The latter figure represents a significant increase on the usual 55-65 of this sample. Values at 0.2" penetration were 126 and 145, respectively, in comparison with the usual value of close to 100.

Other methods

Calcretes exhibiting curved flow curves and the re-wetting effect in the liquid limit test may possibly be self-stabilizing.

7.10.2 Detection of self-stabilization in road bases

Nascimento et al (1965) have extended their petrifaction test to undisturbed and compacted samples. Higher values of petrifaction degree on an undisturbed sample than on the remoulded or recompacted sample are considered to indicate self-stabilization.

A probably related phenomenon is the structure of undisturbed clay. Casagrande (1932) noted that the difference between the shrinkage limit of undisturbed and remoulded soil is a good estimate of the structure of the undisturbed soil. Soils possessing structure possess

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much higher undisturbed than remoulded strengths. Casagrande cited as an example some Laurentian clays which are so rigid at the liquid limit that they can carry a building load of 5 tons per square foot without excessive settlement.

Another more direct method would be to carry out in-situ CBRs on the base suspected of self-stabilization and then to excavate the material tested, break it up thoroughly to as near its original grading as possible and recompact it to its original dry density. If the CBR obtained on such material is much lower than the in-situ CBR, then self-stabilization must have taken place. This method of course brings with it all the difficulties inherent in an in-situ CBR. A possibly better way would be to core the base and subject it to undisturbed and recompacted triaxial or CBR testing. The snag with all these forms of testing, however, is aggregate size. A core about one foot in diameter would really be needed if 2 inch aggregate is present. Since the average base is only about six or eight inches thick this raises problems of height/diameter ratio.

Curvature meter or Benkelman beam measurements over a period of several years at the same sites might be revealing. Nuclear density measurements should be made at the same time to guard against any change in density. Anomalously low deflections or high radii of curvature obtained on unstabilized calcrete-based roads would also point to some cementation having taken place.

Roads built of substandard pedogenic materials which have nevertheless performed well for many years may be suspected of having undergone self-stabilization. All the other factors which might have affected the performance of the base (such as surfacing, traffic, climate, drainage, subbase and subgrade) should of course also be considered

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in such cases.

7.11 SOLUBLE SALTS

All calcareous soils, calcified soils, powder calcretes, nodular calcretes and honeycomb calcretes should be tested for soluble salts before use as base and subbase, particularly in the drier areas. The whole aspect of soluble salts is a complex one and is still imperfectly understood. It is dealt with in detail in Appendix D and by Netterberg (1969a, 1969b). Rapid conductivity testing (Section D.5) is suggested as a routine test, supplemented by the wetting/drying/brushing test (Section D.4.2) where cement or lime stabilization is to be employed on borderline samples or where the presence of gypsum or anhydrite is suspected, unless it can be shown by some other suitable method that the $CaSO_4$ content is below the prescribed limits. Limits for various salts will be found in Chapter 9, together with the specifications for different types of base.

A quick colorimetric test for sulphate capable of distinguishing between soils and waters containing more and less than 0.2% SO_4^{--} is the Calif 417-A. A 3:1 water: soil ratio is used, so that the test will detect some gypsum. An accurate gravimetric procedure is also given, as well as a rapid titration method for chlorides (Calif 422-A).

7.12 MISCELLANEOUS METHODS

Shrinkage limit

The shrinkage limit determined by the normal ASTM D427 method and the optional method employing the solid particle specific gravity differ somewhat. A comparison of results yielded by the two methods is shown in Table J.5. Differences between the two methods of 1 or 2 percentage units are common and differences up to around 7 possible. The specific gravity method usually tends

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to yield the higher result. This could possibly be due to values of G_s which are too high. The volumetric shrinkage method suffers from the disadvantage that a good, unbroken pat must be obtained. Unbroken pats weigh between 25 and 30 grams. A maximum loss of 0.5 g (i.e. about 2%) was allowed in calculating the results for Table J.5. All other results were rejected as not complying with the method.

Specific gravity and absorption

Data in Appendix J (Section J.6) suggests that the tests for specific gravity and absorption of fine aggregate ASTM C127, BS 812 and Calif 225-A are inaccurate on calcrete particles smaller than about 20 mesh. The results of a limited attempt to pursue the matter further are reported there, but unfortunately no particular method can be recommended at this stage.

California bearing ratio

Calcretes are not known to present any problems in CBR work. It should, however, be noted that the CBR of calcretes at 0.2" penetration is almost invariably higher than that at 0.1", especially at the higher compactive efforts like Modified AASHO. A number of materials experts spoken to confirmed that this is a feature exhibited by many materials of reasonably good quality (the poorer materials tend to decrease in CBR with penetration). This is a significant observation, since it would seem that it is normal South African practice to take the 0.1" CBR as the design CBR. Significant savings could, however, be made by looking at the CBR results at higher penetrations, since at least three overseas authorities have stated that if the CBR at 0.2" is higher than that at 0.1", the test should be repeated and if confirmed the 0.2" CBR used for design purposes (Road Research Laboratory, 1952; Woods, 1960; Dawson, 1959). The 0.2"

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and 0.3" CBRs are always determined in normal practice anyway, so that the information is available.

The reverse is normally the case with cement or lime stabilized calcretes, the CBR at 0.1" penetration normally being higher than that at 0.2", especially at the lower compactive efforts.

The reason for this behaviour is probably simply a matter of density and the brittle cohesion developed by stabilized materials.

The EDTA method for the control of lime and cement distribution during stabilization is unsuitable for use on calcretes, as is any other method which works by determining Ca. A method which determines OH^- in the presence of CO_3^{--} such as the Calif 338-D (Parts II and IV) would, however, be suitable. It is believed that a rapid conductivity method also exists. The variation in salinity of some calcretes may be sufficient to interfere with the reliability of this method. Some calcretes are thought to react so rapidly with lime that none of these tests for free lime or Ca would be of any value in such cases.

Moisture equilibration

Owing to the relatively high porosity of calcrete grains, moisture is held both in and around the particles. In all tests involving the addition of water, sufficient equilibration time should be allowed to ensure that the porous particles can take up moisture if reproducible results are to be obtained. Drying times should always be adequate for the same reason.

Testing of lime stabilized calcretes

Calcretes which yield high CBRs when stabilized with lime should be treated as cement stabilized materials for purposes of specifying the delay allowable between wet mixing and compaction and the curing procedure to be

adopted/ ...

adopted. The speed of reaction of such reactive calcretes is at least equal to, if not greater than that of cement stabilized materials and similar cracking problems can also be expected.

Testing of lime and cement stabilized materials in general

The testing of stabilized materials is too vast a subject to consider in any detail here and little work has been carried out in this field during the course of this project. Useful sources of reference on stabilization in general, including the testing of stabilized materials, are Road Research Laboratory (1952), Woods (1960), Portland Cement Association (1956) and Highway Research Board (1961 and 1961a). One point may be worth making regarding the durability testing of stabilized materials. Several tests are used for this purpose in the United States, the AASHO T 135 wetting and drying and the AASHO T 136 freezing and thawing tests perhaps being the most well known. Durability testing of this sort is not normally carried out in South Africa, but may be worth considering in some cases. In Australia, Smith (1962) found that the wetting and drying test showed up a tendency for a salt-containing cement stabilized material to disintegrate better than CBR and compressive strength tests. It may thus have predicted the disintegration of the lime stabilized calcrete base described by Netterberg and Jamieson (1968), in fact the failure symptoms were in both cases remarkably similar. In the United States the FAA (1964) for example specify both freezing and thawing and wetting and drying tests for soil cement (Item P.301) and cement treated (Item P.304) base course, but state that the freezing and thawing test may be omitted in areas where there is no frost penetration. As durability testing has always been regarded as the 'proof' test and all other methods to be short cuts, it is apparent that the short cut methods should always be

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devices appear to yield higher liquid limits by up to several percentage units than ASTM devices. The effect of presoaking and excessive mixing on the liquid limits of calcretes is unknown. The NIRR flow curve method proceeding from the wetter to the drier state is considered to yield reliable results in most cases (± 0.5 units about the true liquid limit for the method in more than 90% of cases) provided rewetting is prohibited and the points to be used in drawing the flow curve are carefully selected. Some 17% of calcrete flow curves are not linear on the semilog plot, but are slightly curved, concave upwards. In view of the rewetting effect and the occasional concave flow curve it is considered that the Department of Transport method will tend to yield results on the high side of the NIRR method (by several units in many cases). Experimental verifaction is, however, still lacking.

4) Difficulties are said to be encountered in the determination of plastic limits on calcretes. The operator variability and reproducibility of such tests on calcretes have not been experimentally assessed.

5) In view of the difficulties with the liquid limit previously mentioned, plasticity indices of calcretes determined by the Department of Transport method are likely to be on the high side. The term 'apparent PI' has been used by at least one consulting engineer when referring to calcrete PIs.

6) Bar linear shrinkage determinations are so rapid, easy and inexpensive to carry out and so little can go wrong with them that it is amazing that they have not displaced the plasticity index as the most useful soil constant. By comparison, a PI determination is

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so fraught with possible sources of error and is so time consuming that it is difficult to see how it can compete with bar linear shrinkage. As the correlation coefficient between the two tests is usually high enough to indicate that they both measure essentially the same property, it is plain that to use a PI to measure linear shrinkage is going about it the hard way. It is suggested then that more attention should be paid to the linear shrinkage of calcretes (at least) than to the PI. The value of the California sand equivalent and cleanness value tests as substitutes for Atterberg limits on calcretes has not been assessed.

7) A theoretical equation relating the linear shrinkage and plasticity index has been developed:

$$PI = \frac{100}{R}\left[\frac{1}{(1-\frac{Ls}{100})^3} - 1\right] - SI$$

where PI = plasticity index, R = shrinkage ratio, Ls = linear shrinkage and SI = shrinkage index. The equation has not yet been experimentally verified.

8) Mechanical analyses are not always accurate measures of the particle-size distribution of calcretes. This is considered to be due to four factors:

a) great variability of particle strength, not only between calcretes, but within the same sample, leading to soft aggregate as well as aggregation breakdown during sieve analysis and sample preparation (most calcretes?),

b) variation of bulk particle specific gravity with size fraction (most calcretes?),

c) clay-coated aggregate (some calcretes), and

d)/ ...

d) flocculation during sedimentation analysis (many calcretes).

A standard wet sieving method overcomes the second difficulty and should at least yield comparable results in the first. Calcretes may be washed to remove highly soluble salts like NaCl, but the carbonate should not be removed prior to sedimentation analysis. Experiments with the NIRR and Department of Transport hydrometer methods did not lead to any hard and fast conclusions, but it would generally appear that the NIRR method is less prone to calcrete flocculation problems than the D.O.T. method. Owing to the apparent high particle porosity of calcrete fines, the use of G_s, the solid particle specific gravity, probably underestimates the amount in suspension at any time. The correct specific gravity to use is probably the saturated surface dry specific gravity, but no method has been found to determine it.

9) The importance of aggregate strength testing under soaked conditions and the determination of soil constants on the fines produced is emphasized. There is evidence in the literature that the fines produced during normal Los Angeles testing are different (less plastic) than those produced by degradation under service conditions. Experience in the United States is that the Los Angeles and sulphate soundness tests are now always successful in predicting aggregate degradation in service.

10) A simple and inexpensive new pliers test may be applied in the field by any roadworker to the problem of aggregate degradation on calcrete gravel roads. Operator variability is satisfactory, correlation with the dry 10% F.A.C.T. test reasonable, and correlation with the known performance of calcrete wearing courses good.

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11) A good correlation has been established between the Mohs hardness and the aggregate crushing strength (10% F.A.C.T. or ACV) of calcretes. The relation is 10% F.A.C.T. (tons) = 4.7 $\overline{H}_w$ - 5, where $\overline{H}_w$ is the weighted mean Mohs hardness of the calcrete tested.

12) A simple pocket Mohs hardness tester employing metals instead of minerals and no larger and heavier than a small pocket knife has been designed and constructed. The device is applicable to the determination of Mohs and Vickers hardnesses of minerals and metals and the direct determination of the aggregate crushing strengths of calcretes. The range of the prototype model is from Mohs 2 (Vickers 20) to Mohs 6½ (Vickers 770). Its potential range is up to a Mohs hardness of 9 (Vickers 2000) or 10 (Vickers 7000?). Good agreement was obtained in trials of the prototype tester against the standard minerals of the Mohs scale and against calcretes of known hardness as determined by the minerals.

13) Several tests appear to hold promise of being able to detect self-stabilizing materials. These include the petrifaction degree and subjecting petrifaction degree pats or samples compacted in CBR moulds to wetting and drying cycles. Even merely curing them as for stabilized materials has sometimes been known to increase the CBR. Another promising method is the application of redox potentials.

14) The detection of significant self-stabilization in road bases is more difficult, but possible methods include the comparison of undisturbed and recompacted petrifaction degrees, shrinkage limits and strength tests, as well as long term curvature or deflection measurements.

15) The EDTA method for the determination of cement and

lime/ ...

lime admixtures is unsuitable for use on calcretes. A probably suitable method is the Calif 338-D, which determines OH^- in place of Ca^{++}.

16) Calcareous soils, calcified soils, nodular calcretes, powder calcretes and honeycomb calcretes should always be tested for soluble salts before use as base, subbase, binder or premix filler. Rapid conductivity testing is suggested as a routine test, supplemented by durability testing when necessary.

17) Calcretes which react with lime to yield high CBRs should be treated as cement stabilized materials for the purposes of allowable delays in testing and construction.

7.14 SCOPE FOR FURTHER INVESTIGATION

1) The effect of drying temperature on the soil constants of calcretes should be further investigated.

2) A comparison of the effect of the S.A. Department of Transport, Texas TEX-101-E, FAA P-210 and a dry soil fines preparation method on South African calcretes is needed.

3) There is scope for work on the effect of presoaking and time on the liquid limit of calcretes.

4) The importance of the rewetting effect and concave flow curves on liquid limits obtained by the Department of Transport method should be experimentally assessed. Details are given in Section 7.3.3. Some of Croney and Coleman's (1954) work on the behaviour of the pF-moisture content curve with wetting and drying might be relevant here.

5) The effect of the initial moisture state and the direction in which the liquid limit test proceeds (i.e. from wet to dry or from dry to wet) should be

experimentally/ ...

experimentally assessed. The well known pF-moisture content hysteresis effect may well be reflected in the test.

6) The application of the one-point and hand liquid limit methods to calcretes should be assessed.

7) The operator variability and reproducibility of plastic limit determinations on calcretes should be experimentally assessed.

8) A full study of the bar linear shrinkage test as a possible replacement for the PI in the arid and semiarid zone should be undertaken.

9) It may be worth trying Castro's (1963; in Nascimento et al, 1963) swelling test on calcretes.

10) An attempt should be made to experimentally verify the theoretical formula derived relating plasticity index and linear shrinkage. This could easily be done using existing test results.

11) The California sand equivalent and cleanness value tests may be reliable substitutes for a PI on calcretes.

12) Work on wet aggregate strength testing methods for materials in general is needed. The method should preferably lead to particle-particle abrasion and the production of sufficient fines for a plasticity index or linear shrinkage determination.

13) Much interesting work could be carried out on the concept of rock hardness as against mineral hardness. The suggestion that the hardness of a rock is the weighted mean Mohs hardness of its components should be examined.

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14) Much interesting work remains to be done on means of predicting self-stabilizing properties in the laboratory and the detection and measurement of self-stabilization in practice. While self-stabilization has been induced under laboratory conditions, no fully documented account of significant self-stabilization in practice appears to have been published.

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