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THE QUANTITATIVE ANALYSIS OF Cr, Ba, Sb, Ag, Zn, Co and Fe IN NORMAL HUMAN ENAMEL AND DENTINE BY NEUTRON ACTIVATION AND HIGH RESOLUTION GAMMA-SPECTROMETRY*

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In a previous investigation the concentration of some of the elements which give rise to short-lived radio-isotopes, Ca, Na, Al, Mg and Cl, were determined by means of instrumental activation analysis (Retief *et al*, 1970). The purpose of the present study was to continue the investigation of the concentration of the inorganic constituents in normal human enamel and dentine.

The concentrations of elements which produce radioisotopes with longer half-lives after irradiation have been investigated. Söremark and Lundberg (1964, 1967) determined the concentrations of six of these elements, Cr, Ag, Fe, Co, Pt and Rb, in normal human enamel and

dentine by means of neutron activation and gamma-ray spectrometric analysis. Subsequent to irradiation group separation of elements was carried out by employing a system of ion exchange columns. The activity was measured by means of a NaI(Tl) crystal connected to a 256-channel gamma spectrometer.

It was shown in an earlier publication (Retief *et al*, 1969) that four elements, Zn, Fe, Co and Sb, can be determined qualitatively in normal human enamel and dentine by instrumental activation analysis. In this investigation the same technique was used for the quantitative analysis of elements that give rise to long-lived isotopes in these tissues. The concentrations of Cr, Ba, Sb, Ag, Zn, Co and Fe were determined.

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EXPERIMENTAL METHODS

- (a) Preparation of the enamel and dentine samples. The samples were prepared as previously described (Retief *et al.*, 1970).
- (b) Procedure.

200 mg enamel and dentine dried to constant weight at 105°C were accurately weighed in separate quartz ampoules and sealed. Using analytical grade reagents, standard samples were prepared by weighing into quartz ampoules a fixed volume of a mixed solution containing known amounts of the seven elements to be determined and also a number of other elements. The standard liquid mixtures were then carefully evaporated to dryness, after which the ampoules were sealed.

Enamel and dentine samples were irradiated along with standards for 130 hours in a thermal neutron flux of approximately 2×10^{17} n.cm⁻² sec.⁻¹ in the poolside facility of SAFARI-1, an ORR type reactor of the Atomic Energy Board at Pelindaba. The activity was measured after a cooling period of approximately 28 days, by which time most of the active phosphorous had decayed (³²P, $t_{1/2} = 14.3$ days).

Gamma-ray emission was measured with the radioactive samples placed at 15 cm distance from the Ge(Li) detector—a 50 cm³ coaxial Ge(Li) diode (Princeton Gamma Tech.) connected to an uncooled TC 135M Tennelec preamplifier. The output pulses were amplified by a TC200 Tennelec amplifier and analysed by an Inter technique 4000 channel analyser (model SA 44). Data for peak

analysis was obtained on punched tapes by a Tally P-120. Yule's (1968) smoothed first derivative method was applied to obtain the peak counts under the photopeaks of interest.

The seven elements were measured by using the photopeaks of the respective radioisotopes as indicated in Table I.

RESULTS

The typical γ -spectra of enamel, dentine and standard samples are shown in Figs. 1, II and III respectively. It is clear that the 320 keV, 355 keV, 602 keV, 884 keV, 1115 keV, 1173 keV and 1292 keV photopeaks of ⁵¹Cr, ¹³²Ba, ¹²³Sb, ^{109m}Ag, ⁶⁵Zn, ⁶⁰Co and ⁵⁹Fe are well separated from the other peaks. These photopeaks can therefore be used as a measure of the chromium, barium, antimony, silver, zinc, cobalt and iron concentrations of irradiated enamel and dentine samples.

The concentration of these elements in normal enamel is shown in Table II and in normal dentine in Table III. The results are expressed in ppm based on the dry weight of the enamel and dentine, heated at 105°C to constant weight.

DISCUSSION

The enamel samples were obtained by chipping. Its separation from dentine by this method is a slow process and leads to its incomplete recovery. Further, its contamination by dentine is unavoidable especially if large amounts have to be collected for repeat analyses. Manley and Hodge (1939) developed a method designed to apply a centrifugal technique to the usual flotation procedure for the

TABLE I. Nuclear data of Cr, Ba, Sb, Ag, Zn, Co and Fe.

Stable Isotope	Abundance %	Activation cross-section (Barns)	Daughter Radio-nuclide	Half-life Radio-nuclide	γ -ray photopeak measured keV	Other prominent γ -ray photopeaks keV
⁵⁰ Cr	4.31	15.9	⁵¹ Cr	27.8d	320	—
¹³² Ba	0.097	0.15	¹³² Ba	10.7y	355	294
¹²³ Sb	42.75	2.5	¹²³ Sb	60.3d	602	—
^{109m} Ag	48.65	3.5	^{109m} Ag	253d	884	660,766,940
⁶⁵ Zn	48.89	0.47	⁶⁵ Zn	245d	1115	—
⁶⁰ Co	100	17	⁶⁰ Co	5.2y	1173	1332
⁵⁹ Fe	0.33	1.2	⁵⁹ Fe	45.1d	1292	1098

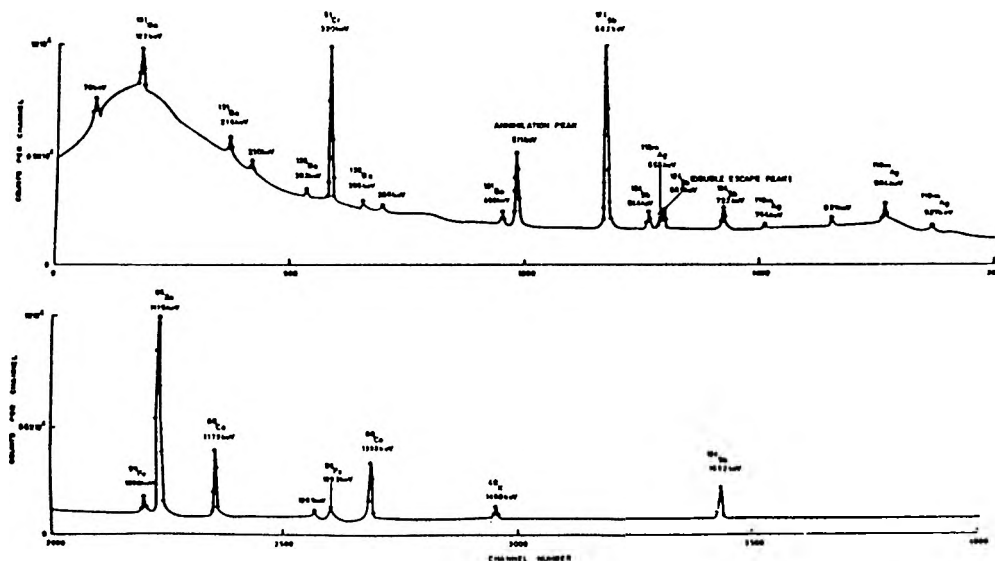


FIG. III. Gamma spectrum of irradiated standard.

TABLE II. The Concentration of Cr, Ba, Sb, Ag, Zn, Co and Fe in Normal Human Enamel.

Element	Number of samples analysed	Mean conc. p.p.m.	Standard error of mean	Standard Deviation	95% Confidence Interval for the true mean
Cr	6	1.02	0.21	0.51	0.45 - 1.49
Ba	7	125.11	8.97	23.68	104.84 - 145.38
Sb	9	0.96	0.23	0.69	0.44 - 1.48
Ag	9	0.56	0.10	0.29	0.34 - 0.78
Zn	8	263.42	5.23	14.80	251.60 - 275.24
Co	10	0.13	0.04	0.13	0.04 - 0.22
Fe	9	118.27	23.88	71.65	64.29 - 172.25

TABLE III. The Concentration of Cr, Ba, Sb, Ag, Zn, Co and Fe in Normal Human Dentine.

Element	Number of samples analysed	Mean conc. p.p.m.	Standard error of mean	Standard Deviation	95% Confidence Interval for the mean
Cr	8	1.99	0.30	0.84	1.32 - 2.66
Ba	7	129.05	20.72	54.69	82.23 - 175.87
Sb	10	0.69	0.12	0.41	0.41 - 0.97
Ag	6	2.18	0.34	0.84	1.41 - 2.95
Zn	9	172.81	3.71	11.14	164.52 - 181.20
Co	10	1.11	0.08	0.27	0.93 - 1.29
Fe	9	93.38	11.68	35.05	66.98 - 119.78

TABLE IV. Enamel—Comparison of results of other investigations with present study.

AUTHOR	ELEMENTS							METHOD
	Cr	Ba	Sb	Ag	Zn	Co	Fe	
Drea (2)	+	+	Not detected	+	+	Not detected	+	Direct current spectrum excitation
Cruickshank (1)	2 of 6							
Lowater and (4) Murray	Trace	Present		Weak	211-259 Trace		Present	Chemical Spectrographic
Söremark and (13) Samsahl					276			Neutron activation, chemical separation, Na I (Tl) detector
Söremark (11) and Lundberg	0.0037			0.0049		0.00024	338	Neutron activation, chemical separation, Na I (Tl) detector
Olsen (8) and Johansen					170			Atomic absorption. Ash weight
Swift (15)	20	100		0.05	5	0.7	210	Spark Source mass spectrometry. Cow's tooth enamel.
Hardwick (3) and Martin	10-100	10-100	<1	1-10	100-1000	10-100	100-1000	Mass spectrometry. Approximate estimation
Nixon, (7) Livingstone and Smith					190-542			Neutron Activation. Chemical separation and instrumental NaI (Tl) detector
Nixon, (6) Livingstone and Smith			0.005-0.67					Neutron activation, Ge(Li) detector
Present Investigation	1.02	125	0.96	0.56	263	0.13	118	Neutron activation, instrumental, Ge(Li) detector

TABLE V. Dentine—Comparison of results of other investigators with present study.

AUTHOR	ELEMENTS							METHOD
	Cr	Ba	Sb	Ag	Zn	Co	Fe	
Drea (2)	+	+	Not detected	+	+	Not detected	+	Direct current spectrum activation.
Cruickshank (1)	2 of 6							
Lowater & Murray (4)	Trace	Present		Weak	215-260 Trace		Present	Chemical Spectrographic
Söremark (14) and Samsahl					199			Neutron activation. Chemical separation, NaI (Tl) detector.
Olsen and (9) Johansen					220			Atomic absorption. Ash weight.
Söremark (12) and Lundberg	0.0049			0.0051		0.00034	110	Neutron activation. Chemical separation, NaI (Tl) detector.
Hardwick (3) and Martin	100-1000	10-100	<1	1-10	100-1000		100-1000	Mass spectrometry. Approximate Estimation.
Present Investigation	1.99	129	0.69	2.18	173	1.11	93	Neutron activation. Instrumental, Ge(Li) detector.

separation of enamel and dentine. The reagents they used cannot be obtained sufficiently pure to avoid contamination of the enamel and dentine prior to irradiation, which unfortunately excludes this method of separation when employing activation analysis. The authors know of no method of separation which will not lead to contamination of the samples prior to irradiation.

Interference from (n, α) and (n,p) reactions are of no consequence in this investigation.

The ampoules in which the samples and standards were irradiated contained antimony. As the weights of the ampoules and the antimony concentration in the quartz glass were known, the antimony concentration in the enamel and dentine were calculated.

With the exception of Zn and to a lesser extent Fe, there is a paucity of information in the literature regarding the concentration of the seven trace elements investigated. Tables IV and V compare the authors' findings with those of other workers; they obtained results in the neutron activation studies which differ considerably in the case of Cr, Ag and Co. Söremark and Lundberg (1964, 1967), employing destructive neutron activation analysis of enamel and dentine, found the concentration of these three elements to be much lower than that found in the present investigation. The marked variation in the results of these studies emphasises the need for further research.

SUMMARY

The concentrations of Cr, Ba, Sb, Ag, Zn, Co and Fe were determined in normal human enamel and dentine employing instrumental neutron activation analysis. Their mean concentrations in enamel are 1.02, 125, 0.23, 0.10, 263, 0.13, and 118 ppm respectively; and in dentine 1.99, 129, 0.69, 2.18, 173, 1.11 and 93 ppm. The need for further investigation is emphasised.

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