

Appendix A

Equations and formulas used in the interpretation of characterization techniques of the mixed metal oxides nanoparticles.

1.

Spurr and Myers equation

$$F = \frac{1}{1 + 1.265 \frac{I_r}{I_a}}$$

Where F , I_r and I_a are such that

F = radial distribution of crystallographic phases,

I_r = strongest rutile intensity,

I_a = strongest anatase intensity

2. Scherer's formula

The size of the nanoparticles after a given calcination temperature will be calculated using the Scherer's formula

$$D = \frac{K\lambda}{(\beta_s - \beta_n) \cos \theta}$$

Where D , λ , β_s , β_n , θ and K are such that

D = average grain size

λ = wavelength with a constant value of 0.15418nm

β_s = the full-width half-maximum (FWHM) of the diffraction peak of the sample

β_n = instrumental broadening

θ = Bragg angle for the x-rays diffraction angle of 100% peak considered.

K = a constant equal to 0.91

Appendix B

3.2 Crystallographic phases

3.2.1 Identification of the mixed metal phases.

The XRD analysis determined a wide range of metal oxides mixed in the precursor powders produced by the resin-gel method of synthesis. The multi-phased precursor powders were composed of titanium oxides, copper oxides and complex copper titanium oxides of different polymorphic states. Eva software automatically search-matched and compared the sample patterns from a rapid method of identification of solid materials using the ICDD database that identifies the crystallographic phases of the precursor powders.⁶⁵ However the drawback associated with this identification process is that the compound must have been previously synthesised and have its analysed pattern entered into the database and the compound must be crystalline. The copper based oxides produced in the metal mixture nanopowders include the polymorph referred to as tenorite (CuO), the simple copper oxide (CuO), the dicopper monoxide polymorph called cuprite (Cu_2O), also a unique polymorphic oxide called paramelaconite (Cu_4O_3) and other copper oxides of similar crystallographic phases such as Cu_8O_6 and Cu_4O_2 .⁶⁶ However, the latter were in very small amounts so that they did not contribute to significant peaks in the patterns. This phenomenon is represented in figures below.

Titanium dioxide produced two polymorphs in their pure crystallographic phases. Their relative amounts were subject to their percentage ratios in the reaction mixture. The pure phases gave characteristic distinct peaks on the XRD patterns as shown in figures 3.2.1a, 3.2.1b and 3.2.1c below. Titanium and copper salts further reacted to produce mixed metal oxides of copper titanium oxide or titanium copper oxide. These oxides take the molecular forms Cu_3TiO_5 , Cu_3TiO_4 , $\text{Ti}_3\text{Cu}_3\text{O}$, $\text{Cu}_2\text{Ti}_4\text{O}$, $\text{Cu}_2\text{Ti}_2\text{O}_5$ and Cu_2TiO_3 . The occurrence of these ceramics varied with temperature and percentage composition of the respective metals.⁶⁷ Figure 3.2.1d below shows the variation in these mixed metal oxides.

Having identified the range of copper, titanium and copper titanium oxides, there were some peaks which did not identify with any of the oxides in the metal mixture. Detailed Eva search revealed that a peak occurring at 16.5° on the 2θ scale is due to an unknown phase of titanium based compound unidentifiable in the ICDD databases. The crystallographic phases identified in this analysis contributed to the peak patterns produced by the X-ray diffraction of the nanopowders. The relative abundance of each phase determined the intensity of each peak and in some instances resulting in peak broadening.⁶⁸ It is noticeable that some peaks are fingerprints of multiple phases of two or more compounds as shown in figures 3.2.1e and 3.2.1f. This

implies that these compounds diffracted X-rays at same angles hence occurring at the same peak position.^{28, 32, 69, 70} Figure 3.2.1d below illustrates this phenomenon and shows that tenorite, paramelaconite and all the phases of the mixed metal oxides of copper titanium oxides are on the same peak.

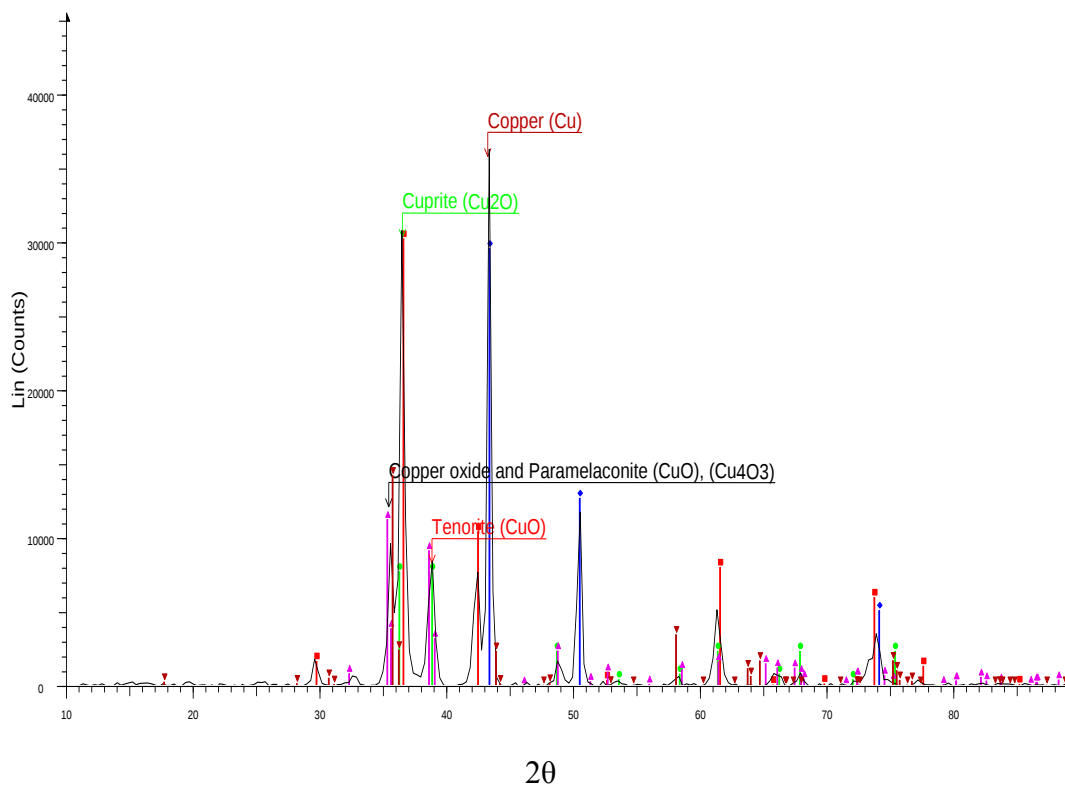


Figure 3.2.1a XRD pattern of non-calcined sample with composition 0% Ti: 100% Cu

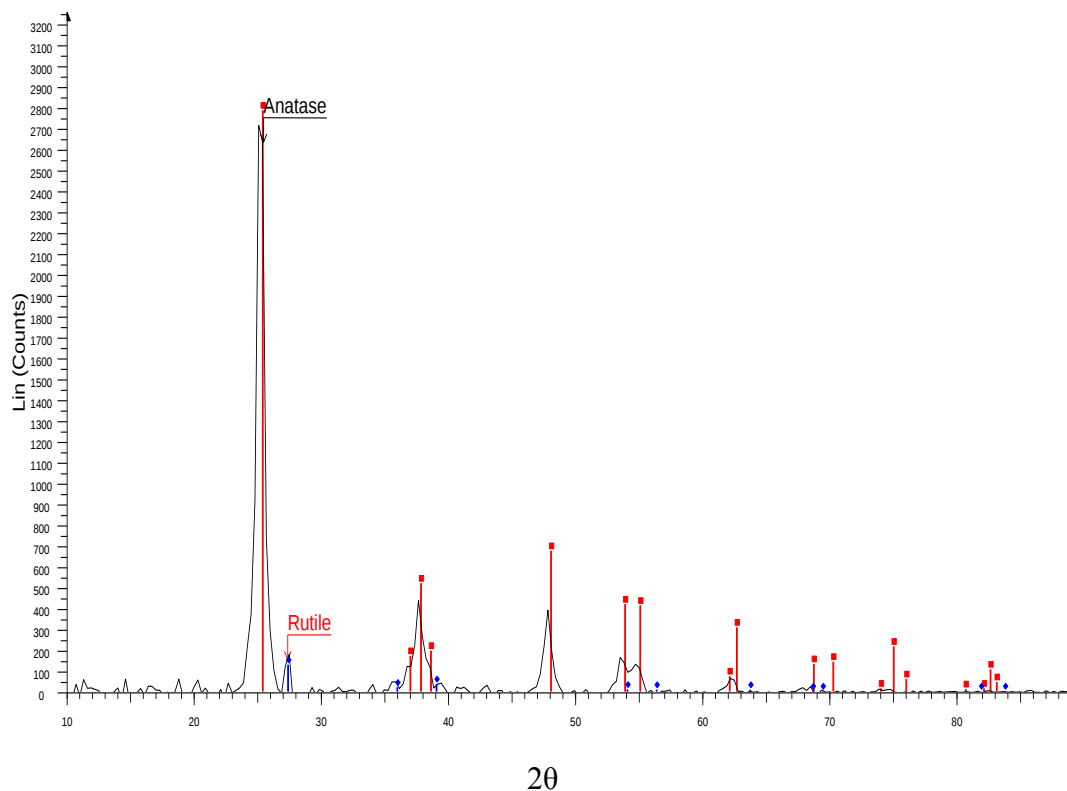


Figure 3.2.1b XRD pattern of a non-calcined sample with composition 95% Ti: 5% Cu

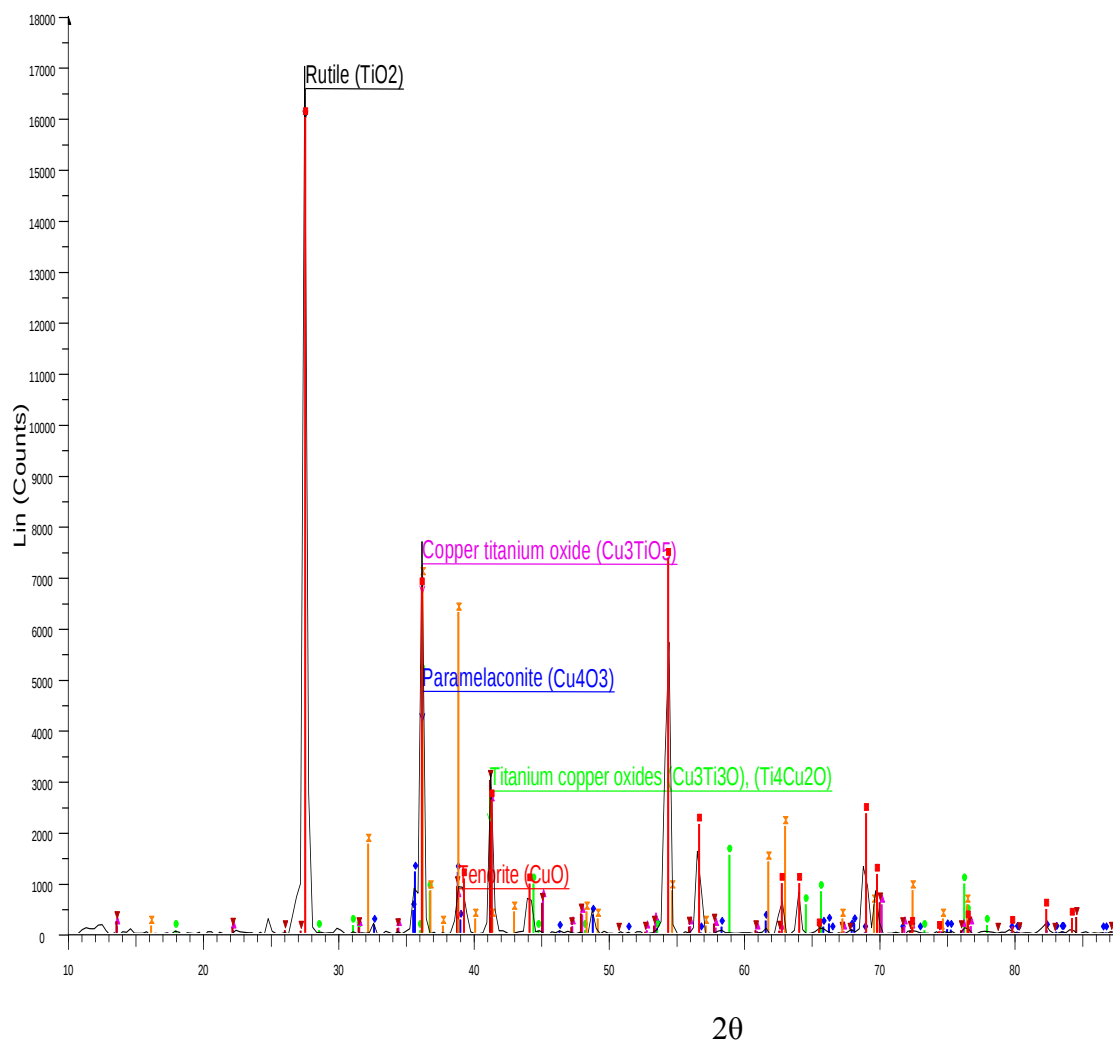


Figure 3.2.1c PXRD sample calcined at 900°C with composition 95% Ti: 5% Cu

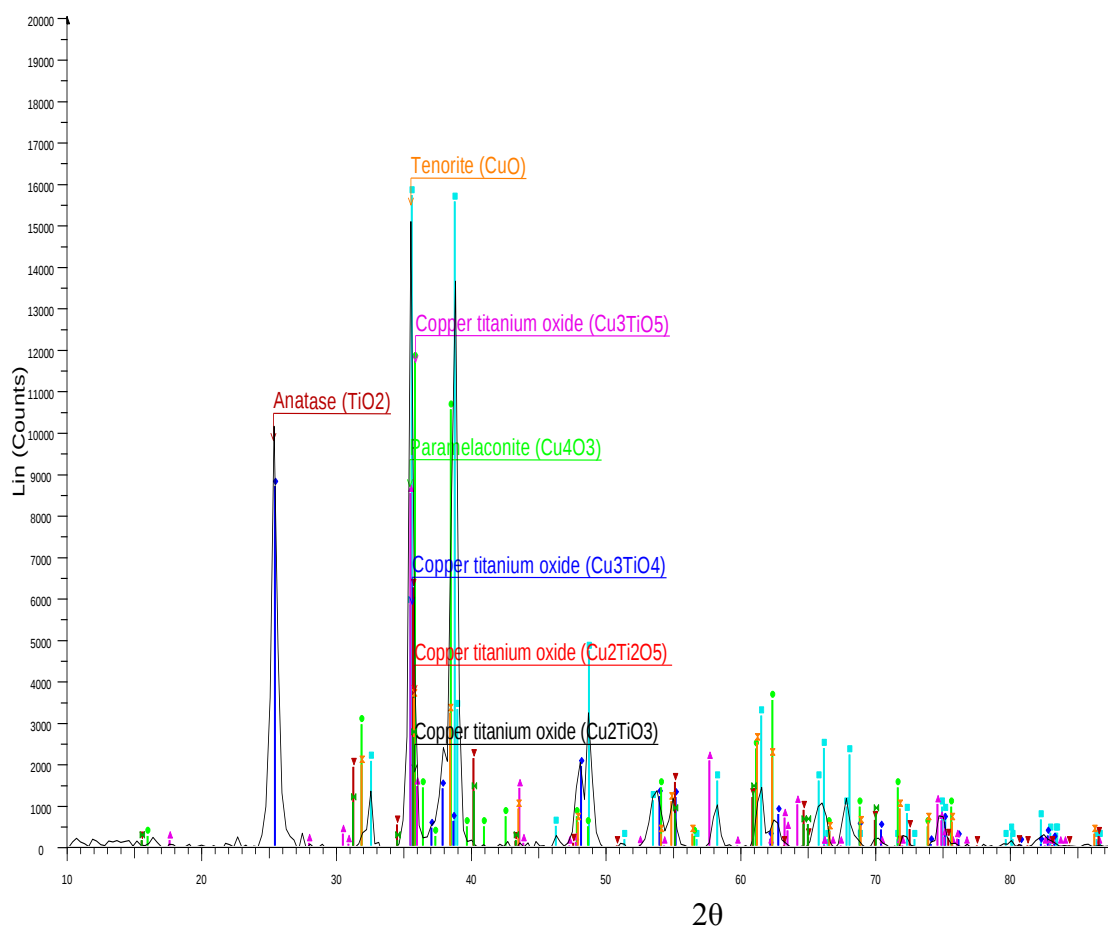


Figure 3.2.1d XRD sample calcined at 500°C with composition 40% Ti: 60% Cu

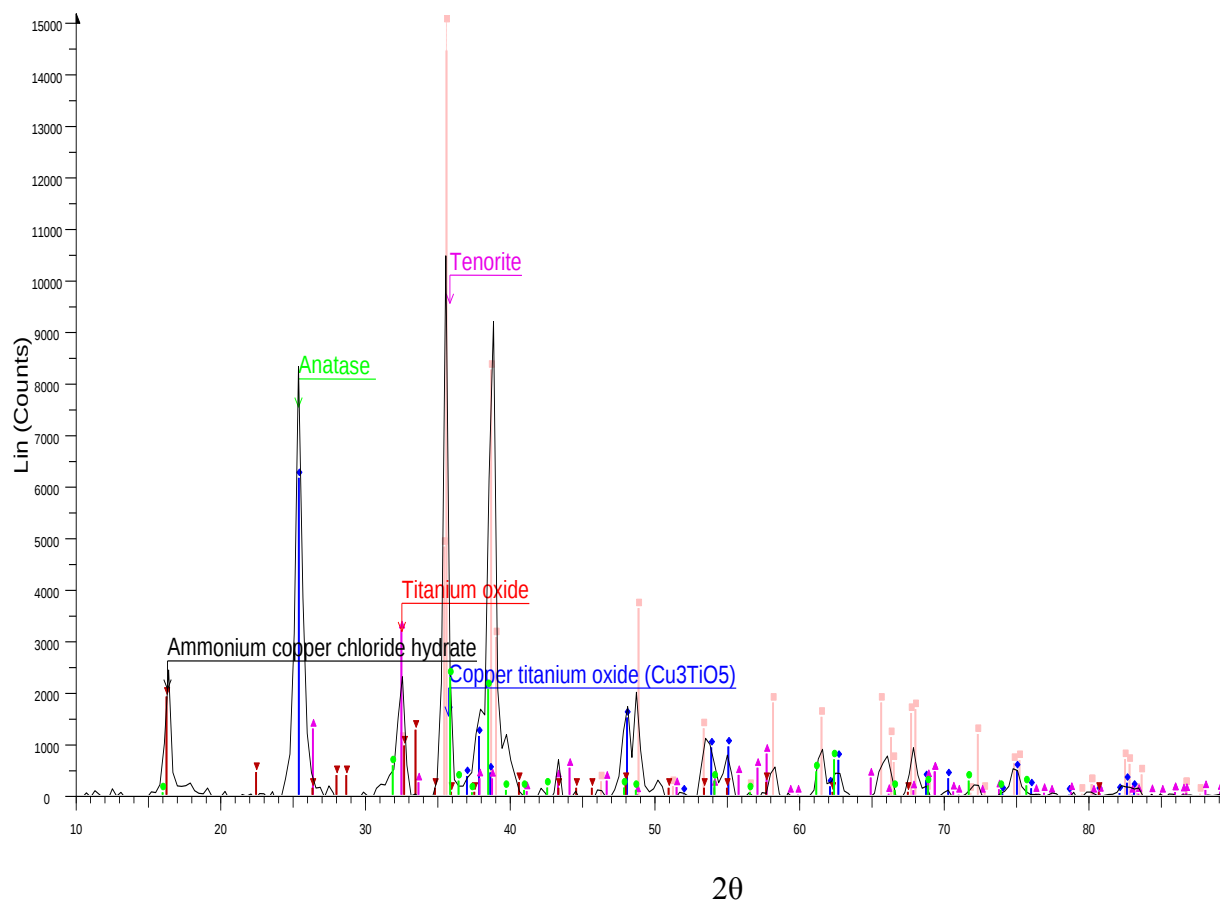


Figure 3.2.1e PXRD pattern of a non-calcined sample with composition 40% Ti: 60% Cu

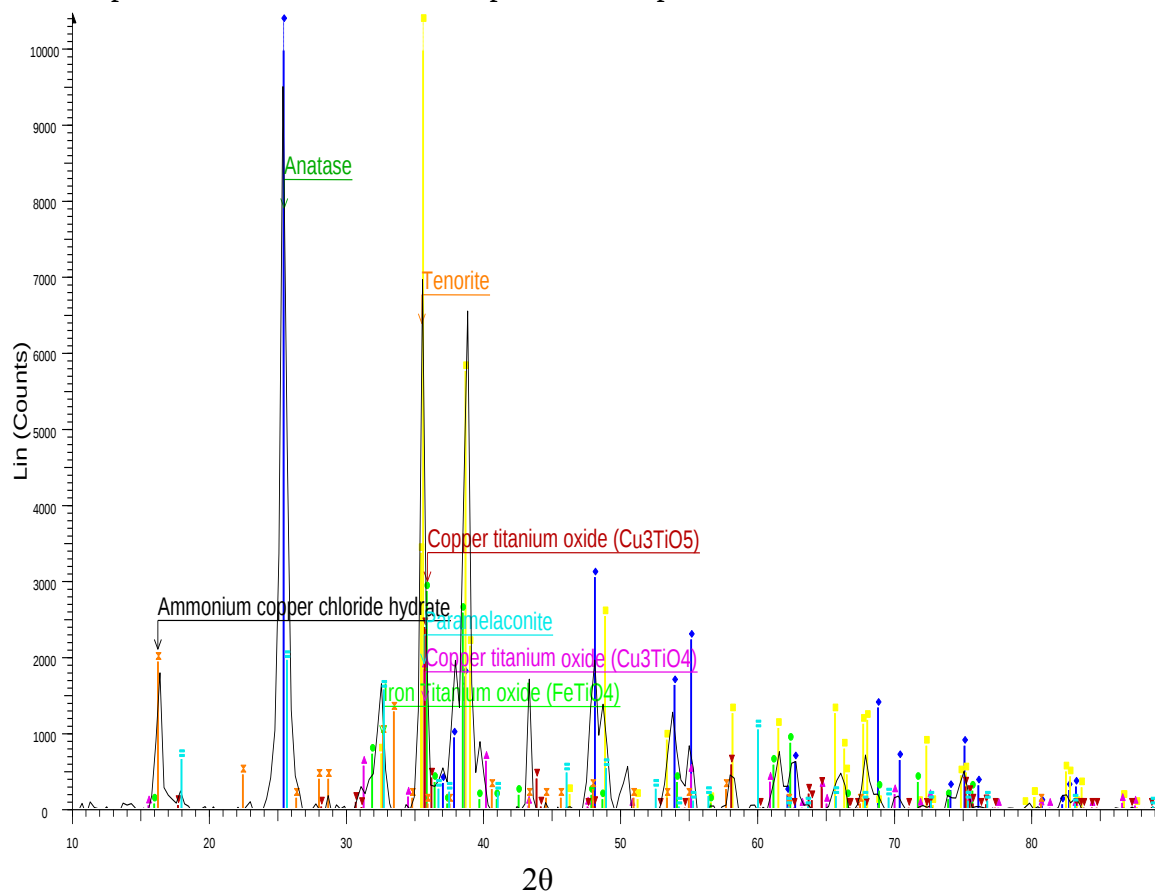


Figure 3.2.1f XRD pattern of a non-calcined sample with composition 50% Ti: 50% Cu

3.2.2 Trends in Crystallographic Phase Variation

Samples prepared for this research showed variation arising from the percentage composition varied in a decreasing order for copper and increasing order for titanium. The first sample in the series comprised of 100 % copper and 0 % titanium. In the non-calcined series, this sample produced a range of copper-based phases as shown in Figure 3.2.1 in Appendix B. There is also a peak showing pure copper. This peak is more intense than peaks due to copper oxides. This phenomenon is the same in the series calcined at 300°C. For the series calcined at 500, 700 and 900°C, the first peak pattern is showing only one pure phase, that is the tenorite with a peak so intense beyond 100 % without any other phases occurring as in appendices C, D and E. This implies that heat treatment of the precursor powders at temperatures above 300°C resulted in the copper oxide phases turning to tenorite.⁷¹ It also infers that the pure phase tenorite is more thermodynamically stable at high temperatures than other phases of copper oxides occurring in abundance at low temperatures.⁷²

The occurrence of the cuprite polymorph is shown to be subject to both temperature and percentage composition. Appendices B and C present this phenomenon in which it is occurring in the first three patterns of the non-calcined series with peak intensity diminishing as percentage copper decreased. In the series calcined at 300°C, cuprite occurred only in the first two patterns but had very low peak intensities. In the rest of the series, this polymorph did not form. This is attributable to the instability of cuprite at high temperatures and does not preferentially form in a random reaction in favor of other copper oxides.⁷³ Another phase of interest called paramelaconite (Cu_4O_3) occurred much in the same manner as tenorite except that its intensity was always considerably lower. It exhibited the same pattern as the tenorite does, as the copper percentage composition decreases. Its persistence shows that it is not temperature dependant.⁷⁴

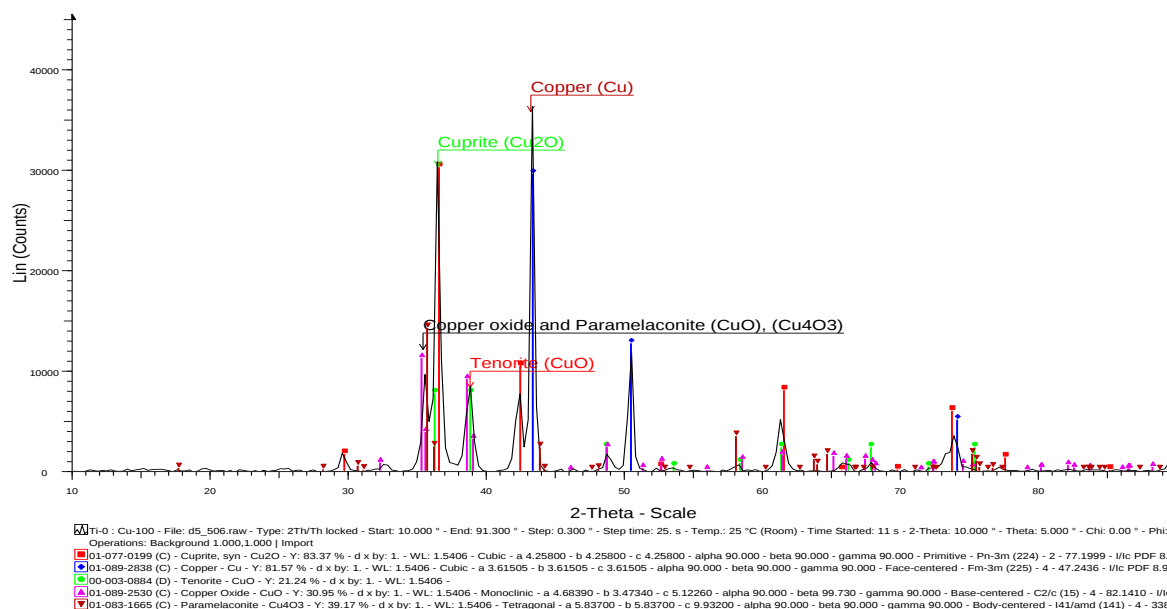
At the introduction of 5 % titanium, the intensity of the tenorite decreased across the whole temperature range with the formation of other phases of both copper and titanium oxides. There is a notable appearance of the anatase peak. This peak starts as a small peak increasing in intensity as titanium percentage composition increases. The occurrence of multiple phases on a single peak occurred from the initial introduction of titanium at 5 % until where percentage copper composition had diminished to 5 % and titanium increased to 95 %. At this composition a small peak of low intensity is identified as due to tenorite and high intensity peaks, due to either anatase only or both anatase and rutile. The last sample in all the series comprises of 100 % titanium and 0 % copper so that there are peaks due to pure phase Anatase (TiO_2). This trend in crystallographic phase variation occurred across the whole temperature ranges as in appendices A to F.

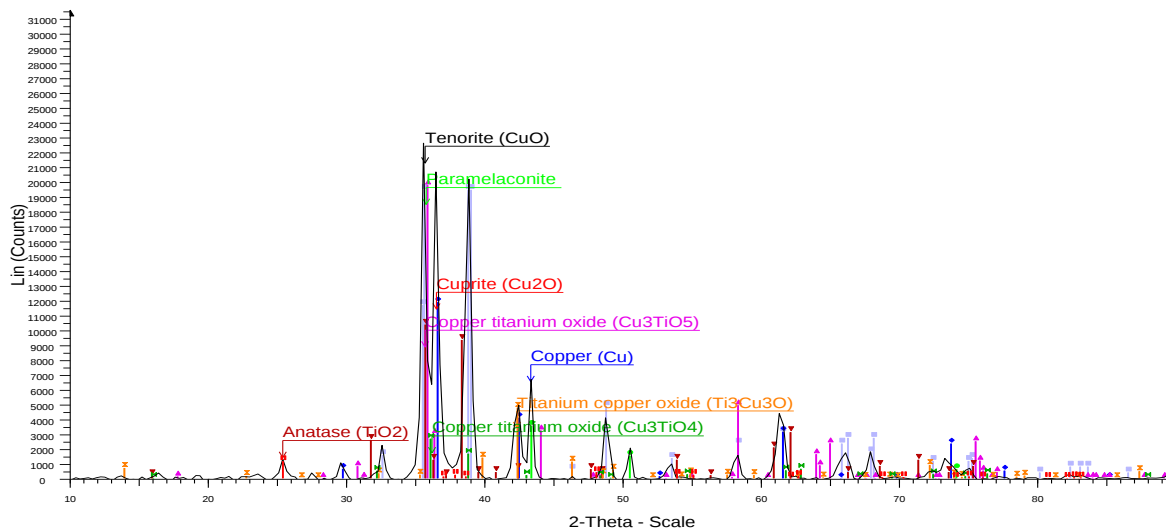
A new set of reflections corresponding to the unknown non-stoichiometric mixed-metal oxide product $\text{Cu}_x\text{Ti}_y\text{O}$ emerges and grows in intensity at the expense of the reflection from CuO and TiO_2 . Studying the evolution of the pattern as a function of temperature allows rapid identification of phase changes, which

cause marked changes in the pattern as the material adopts a new structure.⁷⁵ The wide range of copper titanium oxide polymorphs synthesised according to the temperature of calcination. The non-calcined series and the 300°C calcined series were composed of copper titanium oxides of the Cu_3TiO_5 , Cu_3TiO_4 and $\text{Ti}_3\text{Cu}_3\text{O}$ polymorphs. These phases occurred on the same peak as the Tenorite and paramelaconite peaks but with varying intensities. At 15 % Ti and 85 % Cu the polymorph $\text{Ti}_3\text{Cu}_3\text{O}$ vanished but the other two polymorphs continued at varying relative abundances until the percentage composition of copper diminishes to 0 %.

For the 500°C calcined series, a unique polymorph of the form $\text{Cu}_2\text{Ti}_2\text{O}_5$ was produced in addition to the polymorphs Cu_3TiO_5 , Cu_3TiO_4 and $\text{Ti}_3\text{Cu}_3\text{O}$. The phase's peak intensity initially increased with increase in Ti. However, after a significant decrease in copper percentage composition, its intensity diminished until it became indistinguishable. At 700°C and 900°C, the Cu_3TiO_5 polymorph is the most dominant copper titanium oxide, with other polymorphs occurring in small quantities and only appearing in the first two or three patterns of their respective series. In the rest of the series, the relative abundance of the Cu_3TiO_5 initially increased with increase of Ti percentage composition. However, with the continued decrease in Cu composition, its peak intensity started to diminish beyond the 50 % ratio until it was insignificantly low.

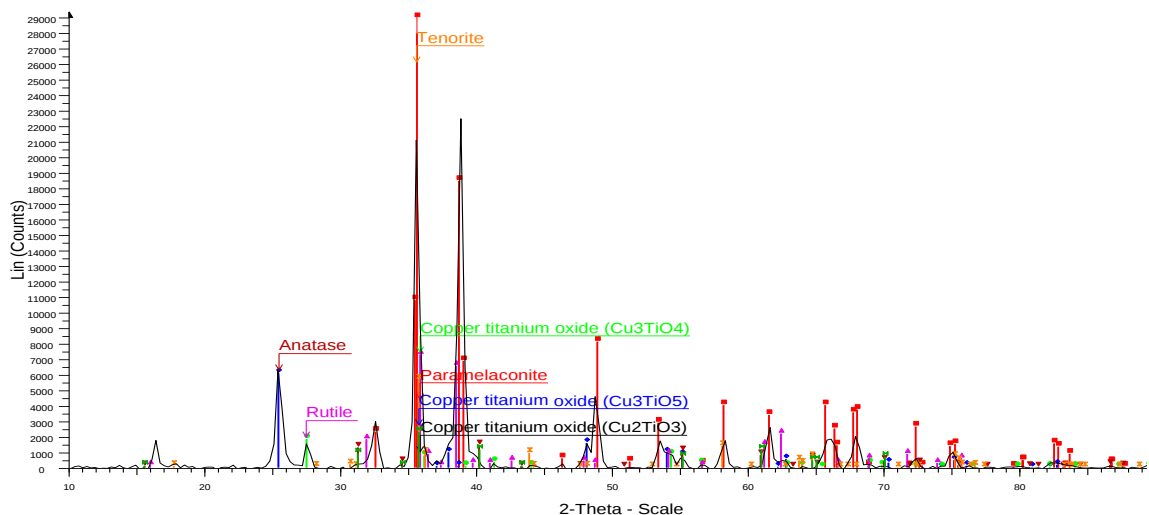
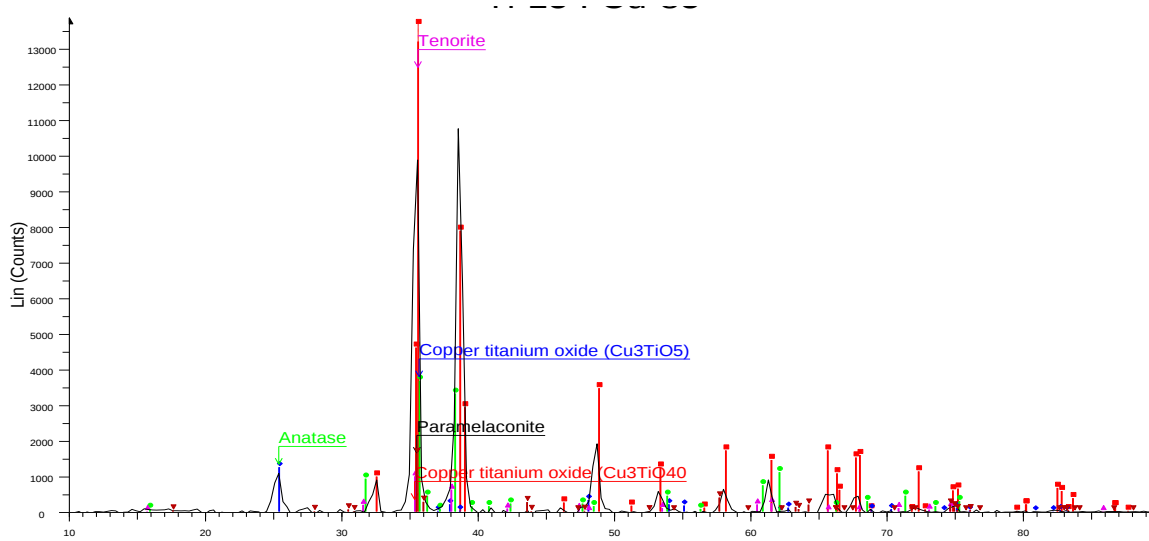
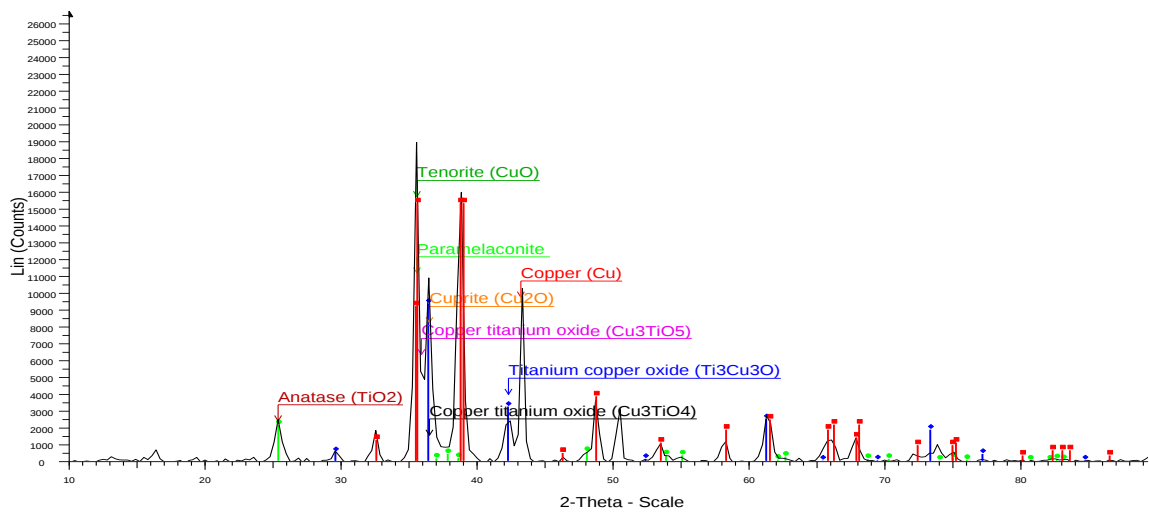
XRD Patterns of Resin-gel non – calcined precursor powders.

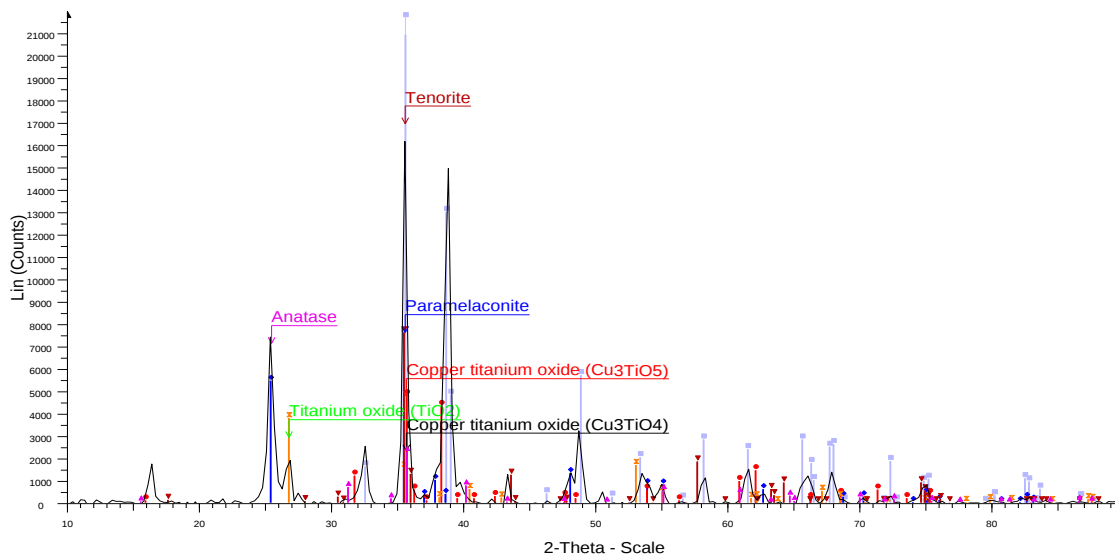




TI-5 : Cu-95 - File: d5_507.raw - Type: 2Th/Th locked - Start: 10.000 ° - End: 91.300 ° - Step: 0.300 ° - Ste
Operations: Background 1.000,1.000 | Import

00-041-0254 (D) - Tenorite, syn - CuO - Y: 86.11 % - d x by: 1 - WL: 1.5406 - Monoclinic - a 4.68500 - b 3	01-081-1612 (C) - Titanium Copper Oxide - Ti3Cu3O - Y: 21.06 % - d x by: 0.9854 - WL: 1.5406 - Cubic -
01-077-0199 (C) - Cuprite, syn - Cu2O - Y: 52.53 % - d x by: 1 - WL: 1.5406 - Cubic - a 4.25800 - b 4.258	00-027-0199 (I) - Copper Titanium Oxide - Cu3TiO4/Cu2O-CuO-TiO2 - Y: 11.81 % - d x by: 0.9729 - WL:
01-089-2838 (C) - Copper - Cu - Y: 15.66 % - d x by: 1 - WL: 1.5406 - Cubic - a 3.61505 - b 3.61505 - c 3	
01-071-0251 (C) - Paramelaconite - Cu16O14.15 - Y: 87.46 % - d x by: 1 - WL: 1.5406 - Tetragonal - a 5.	
00-018-0461 (D) - Copper Titanium Oxide - Cu3TiO5 - Y: 45.87 % - d x by: 0.9833 - WL: 1.5406 - Tetrago	
00-021-1272 (*) - Anatase, syn - TiO2 - Y: 5.10 % - d x by: 1 - WL: 1.5406 - Tetragonal - a 3.78520 - b 3.	





Ti-30 : Cu-70 - File: d5_512.raw - Type: 2Th/Th locked - Start: 10.000 ° - End: 91.300 ° - Step: 0.300 ° - Step time: 25. s - Temp.: 25 °C (Room) - Time Started: 11 s - 2-Theta: 10.000 ° - Theta: 5.000 ° - Chi: 0.00 ° - Phi: 0.00 °

Operations: Background 1.000,1.000 | Import

01-080-0076 (C) - Tenorite, syn - CuO - Y: 181.99 % - d x by: 1. - WL: 1.5406 - Monoclinic - a 4.67970 - b 3.43140 - c 5.13620 - alpha 90.000 - beta 99.262 - gamma 90.000 - Base-centered - C2/c (15) - 4 - 81.4014 - I/I

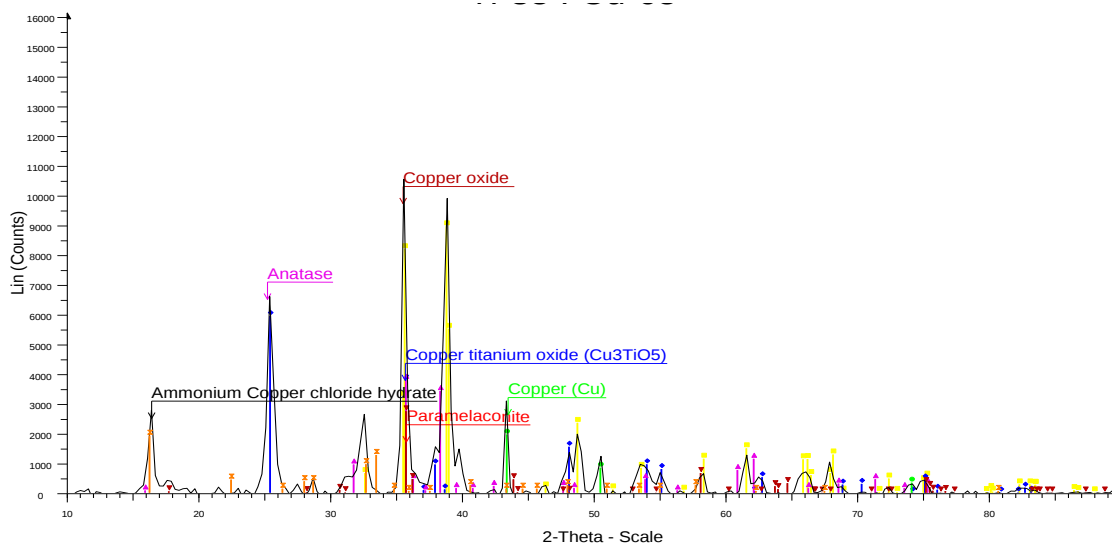
01-071-1166 (A) - Anatase - TiO₂ - Y: 33.76 % - d x by: 1. - WL: 1.5406 - Tetragonal - a 3.78420 - b 3.78420 - c 9.51460 - alpha 90.000 - beta 90.000 - gamma 90.000 - Body-centered - I41/amd (141) - 4 - 136.251 - I/I

00-018-0461 (D) - Copper Titanium Oxide - Cu₃TiO₅ - Y: 29.79 % - d x by: 0.9833 - WL: 1.5406 - Tetragonal - a 8.10000 - b 8.10000 - c 6.60000 - alpha 90.000 - beta 90.000 - gamma 90.000 - F22= 1(0.176)

01-080-1501 (A) - Titanium Oxide - TiO₂ - Y: 23.37 % - d x by: 1.0104 - WL: 1.5406 - Tetragonal - a 4.67000 - b 4.67000 - c 2.97000 - alpha 90.000 - beta 90.000 - gamma 90.000 - Primitive - P42/mnm (136) - 2 - 64.77

01-083-1284 (C) - Copper Titanium Oxide - Cu₃TiO₄ - Y: 14.00 % - d x by: 1. - WL: 1.5406 - Rhombo.H.axes - a 3.04000 - b 3.04000 - c 17.19300 - alpha 90.000 - beta 90.000 - gamma 120.000 - Primitive - R-3m (166)

01-083-1665 (C) - Paramelaconite - Cu₄O₃ - Y: 47.17 % - d x by: 1.0062 - WL: 1.5406 - Tetragonal - a 5.83700 - b 5.83700 - c 9.93200 - alpha 90.000 - beta 90.000 - gamma 90.000 - Body-centered - I41/amd (141) - 4 - 331



Ti-35 : Cu-65 - File: d5_513.raw - Type: 2Th/Th locked - Start: 10.000 ° - End: 91.300 ° - Step: 0.300 ° - Step time: 25. s - Temp.: 25 °C (Room) - Time Started: 12 s - 2-Theta: 10.000 ° - Theta: 5.000 ° - Chi: 0.00 ° - Phi: 0.00 °

Operations: Background 1.000,1.000 | Import

01-089-5898 (C) - Copper Oxide - CuO - Y: 85.06 % - d x by: 1. - WL: 1.5406 - Monoclinic - a 4.69000 - b 3.42000 - c 5.13100 - alpha 90.000 - beta 99.540 - gamma 90.000 - Base-centered - C2/c (15) - 4 - 81.1620 - I/I

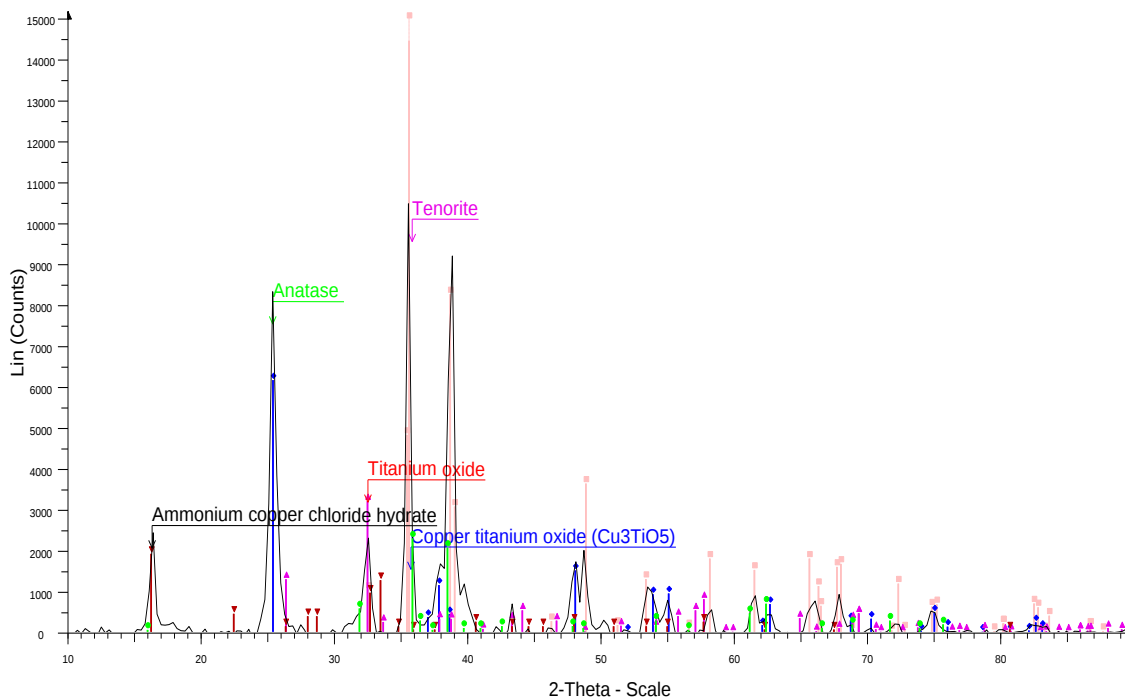
01-086-1157 (A) - Anatase, syn - TiO₂ - Y: 56.41 % - d x by: 1. - WL: 1.5406 - Tetragonal - a 3.78300 - b 3.78300 - c 9.49700 - alpha 90.000 - beta 90.000 - gamma 90.000 - Body-centered - I41/amd (141) - 4 - 135

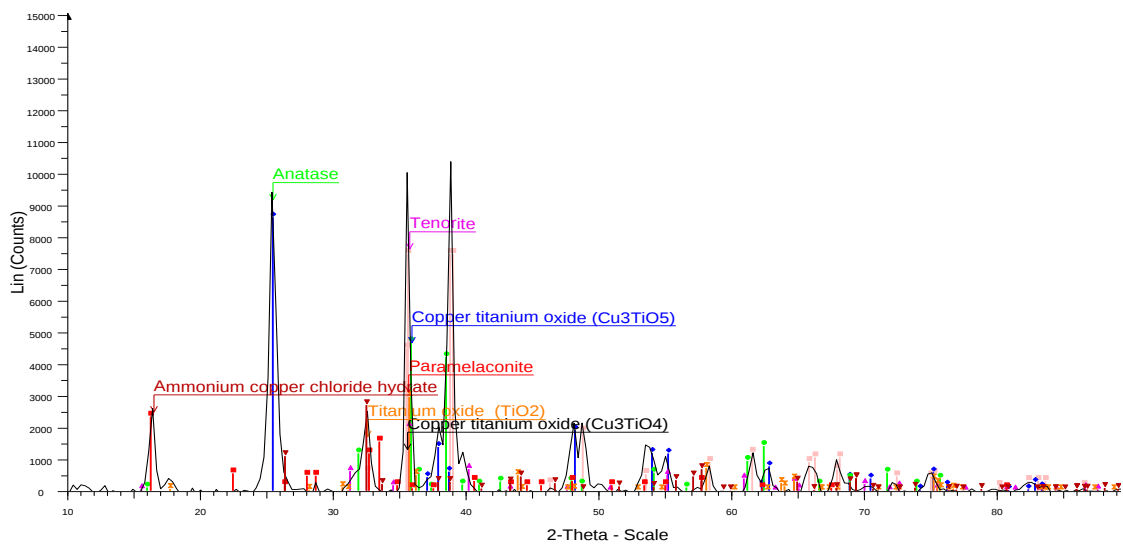
01-089-2838 (C) - Copper - Cu - Y: 18.56 % - d x by: 1. - WL: 1.5406 - Cubic - a 3.61505 - b 3.61505 - c 3.61505 - alpha 90.000 - beta 90.000 - gamma 90.000 - Face-centered - Fm-3m (225) - 4 - 47.2436 - I/I PDF 8.9

00-018-0461 (D) - Copper Titanium Oxide - Cu₃TiO₅ - Y: 36.08 % - d x by: 0.9833 - WL: 1.5406 - Tetragonal - a 8.10000 - b 8.10000 - c 6.60000 - alpha 90.000 - beta 90.000 - gamma 90.000 - F22= 1(0.176)

01-083-1665 (C) - Paramelaconite - Cu₄O₃ - Y: 26.00 % - d x by: 1. - WL: 1.5406 - Tetragonal - a 5.83700 - b 5.83700 - c 9.93200 - alpha 90.000 - beta 90.000 - gamma 90.000 - Body-centered - I41/amd (141) - 4 - 331

00-001-0211 (D) - Ammonium Copper Chloride Hydrate - (NH₄)₂CuCl₄·2H₂O - Y: 18.22 % - d x by: 1. - WL: 1.5406 - Tetragonal - a 7.58300 - b 7.58300 - c 7.95000 - alpha 90.000 - beta 90.000 - gamma 90.000 - 2 - 45





Ti-45 : Cu-55 - File: d5_515.raw - Type: 2Th/Th locked - Start: 10.000 ° - End: 91.300 ° - Step: 0.300 ° - St

Operations: Background 1.000,1.000 | Import

00-041-0254 (D) - Tenorite, syn - CuO - Y: 71.97 % - d x by: 1 - WL: 1.5406 - Monoclinic - a 4.68500 - b 3

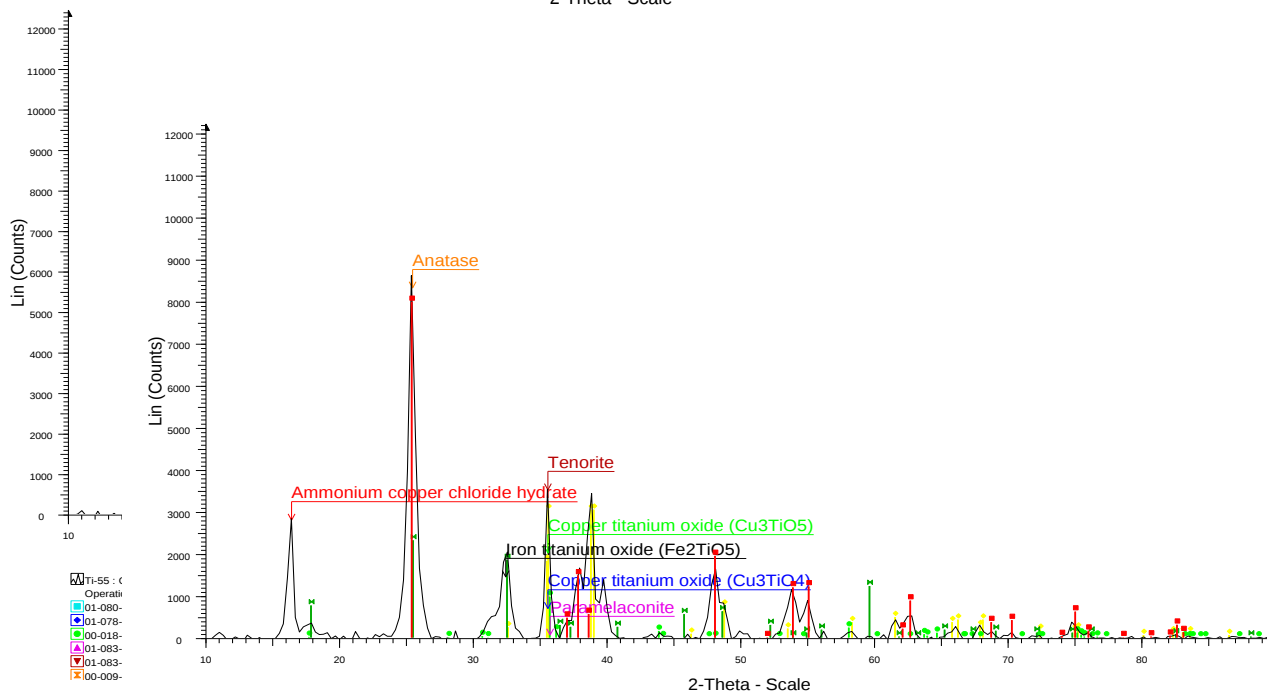
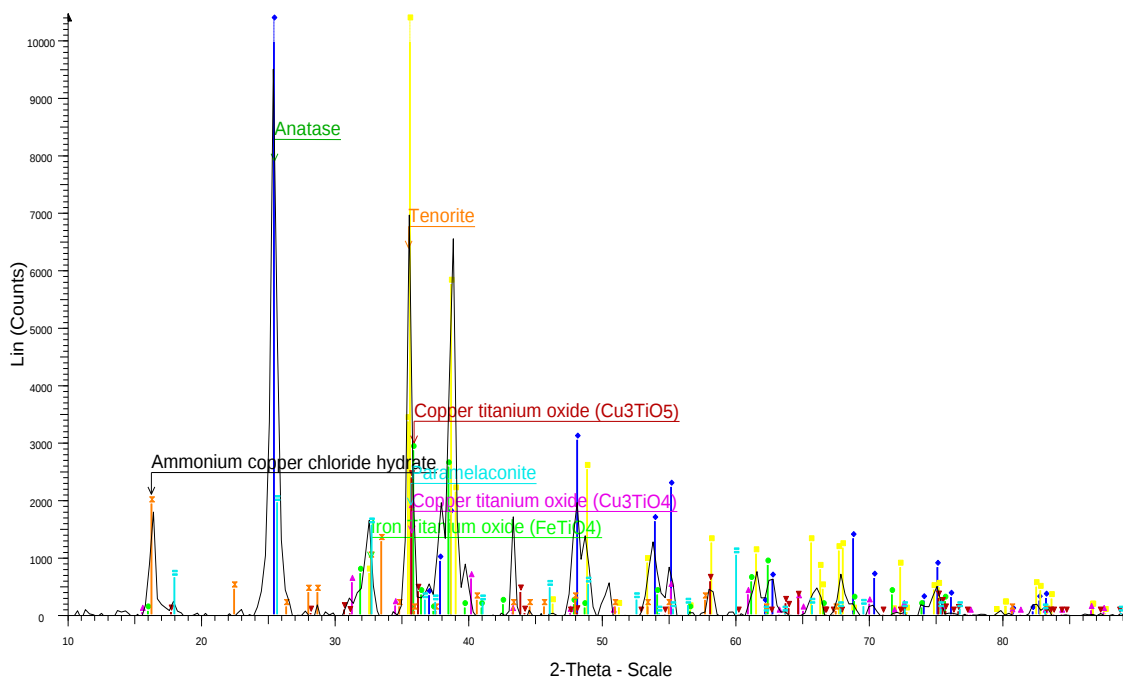
01-089-4921 (C) - Anatase, syn - TiO₂ - Y: 83.06 % - d x by: 1 - WL: 1.5406 - Tetragonal - a 3.77700 - b

00-018-0461 (D) - Copper Titanium Oxide - Cu₃TiO₅ - Y: 45.11 % - d x by: 0.9792 - WL: 1.5406 - Tetrago

01-083-1284 (C) - Copper Titanium Oxide - Cu₃TiO₄ - Y: 19.37 % - d x by: 1 - WL: 1.5406 - Rhombo.H.a

01-084-1750 (C) - Titanium Oxide - TiO₂ - Y: 25.97 % - d x by: 0.9687 - WL: 1.5406 - Orthorhombic - a 4.5

01-083-1665 (C) - Paramelaconite - Cu₄O₃ - Y: 28.50 % - d x by: 1 - WL: 1.5406 - Tetragonal - a 5.83700



Ti-60 : Cu-40 - File: d5_518.raw - Type: 2Th/Th locked - Start: 10.000 ° - End: 91.300 ° - Step: 0.300 ° - Step time: 25. s - Temp.: 25 °C (Room) - Time Started: 12 s - 2-Theta: 10.000 ° - Theta: 5.000 ° - Chi: 0.00 ° - Phi:

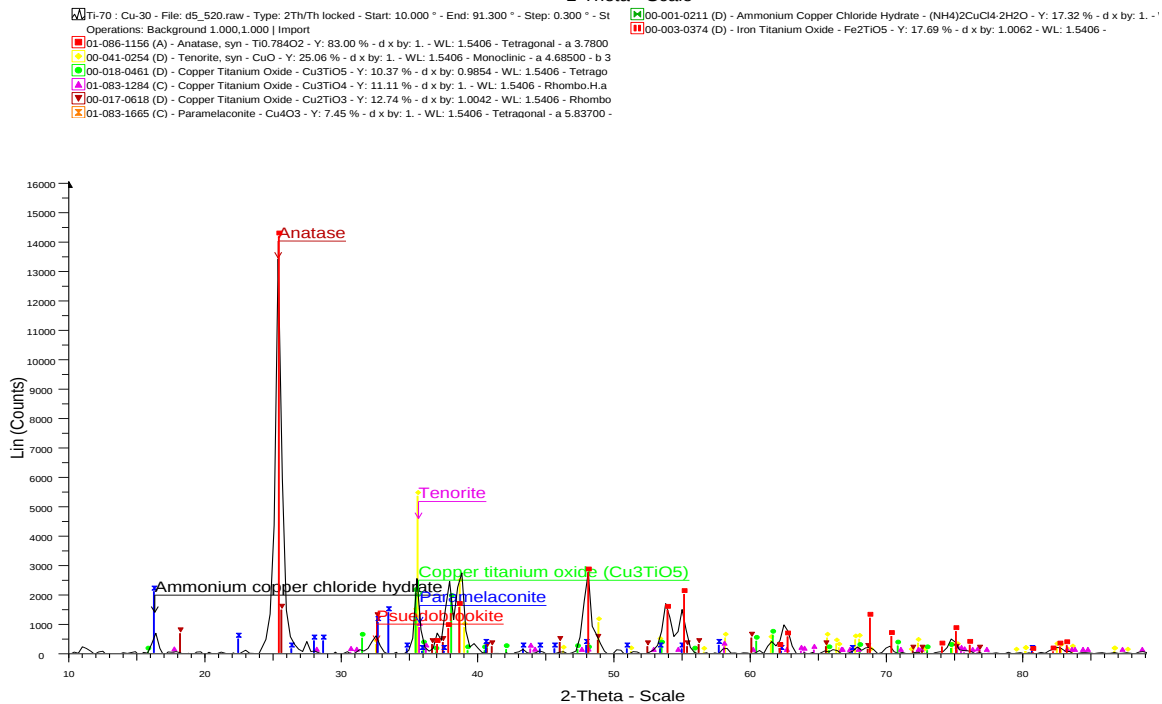
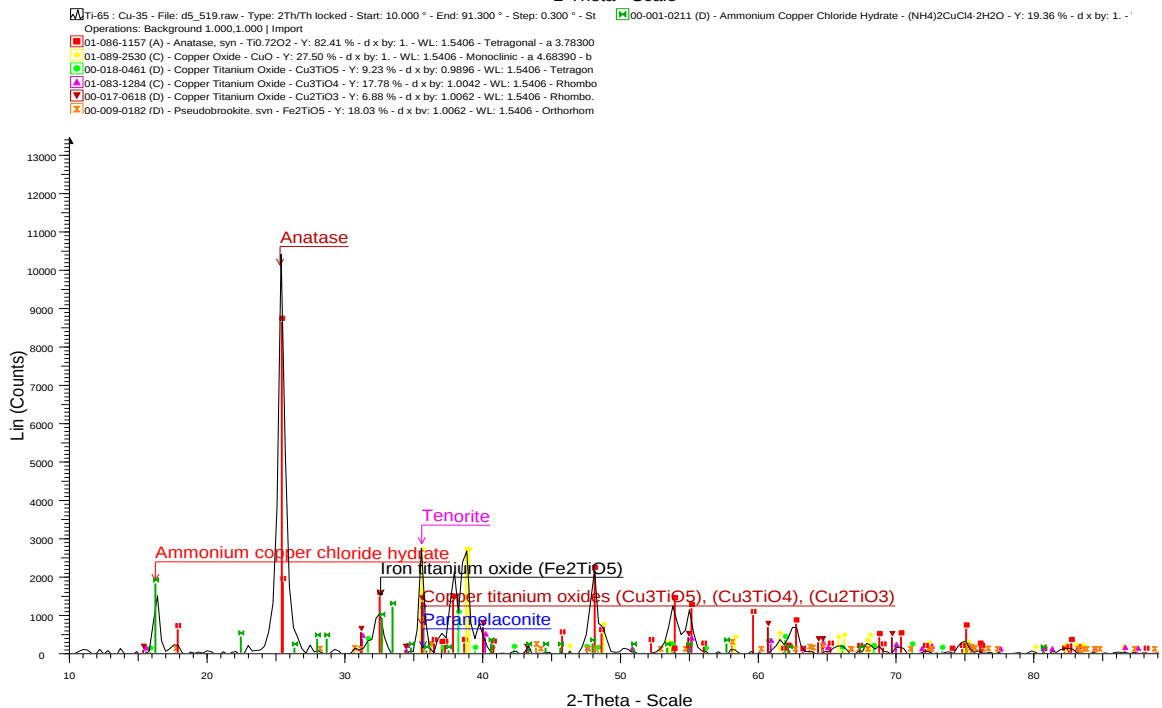
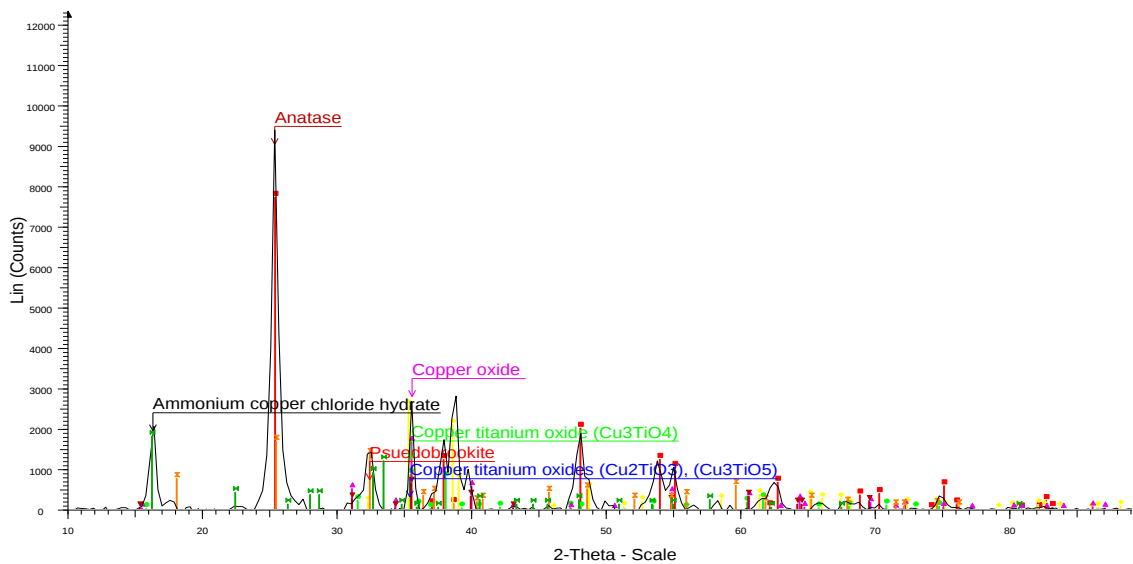
Operations: Background 1.000,1.000 | Import

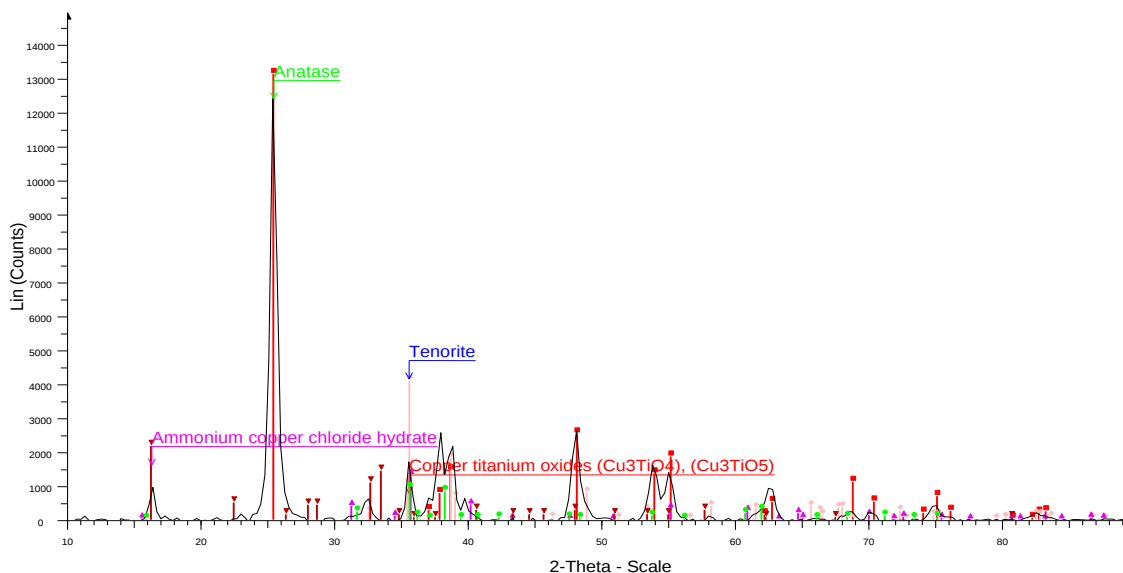
01-078-2486 (C) - Anatase, syn - TiO₂ - Y: 92.80 % - d x by: 1 - WL: 1.5406 - Tetragonal - a 3.78450 - b 3.78450 - c 9.51430 - alpha 90.000 - beta 90.000 - gamma 90.000 - Body-centered - I41/amd (141) - 4 - 136.268

00-041-0254 (D) - Tenorite, syn - CuO - Y: 35.34 % - d x by: 1 - WL: 1.5406 - Monoclinic - a 4.68500 - b 3.42300 - c 5.13200 - alpha 90.000 - beta 99.520 - gamma 90.000 - Base-centered - C2/c (15) - 4 - 81.1672 - I/c

01-083-1665 (C) - Paramelaconite - Cu₄O₃ - Y: 11.27 % - d x by: 1 - WL: 1.5406 - Tetragonal - a 5.83700 - b 5.83700 - c 9.93200 - alpha 90.000 - beta 90.000 - gamma 90.000 - Body-centered - I41/amd (141) - 4 - 338

00-003-0374 (D) - Iron Titanium Oxide - Fe₂TiO₅ - Y: 26.88 % - d x by: 1.0062 - WL: 1.5406 -





Ti-80 : Cu-20 - File: d5_522.raw - Type: 2Th/Th locked - Start: 10.000 ° - End: 91.300 ° - Step: 0.300 ° - Step time: 25. s - Temp.: 25 °C (Room) - Time Started: 11 s - 2-Theta: 10.000 ° - Theta: 5.000 ° - Chi: 0.00 ° - Phi

Operations: Background 1.000,1.000 | Import

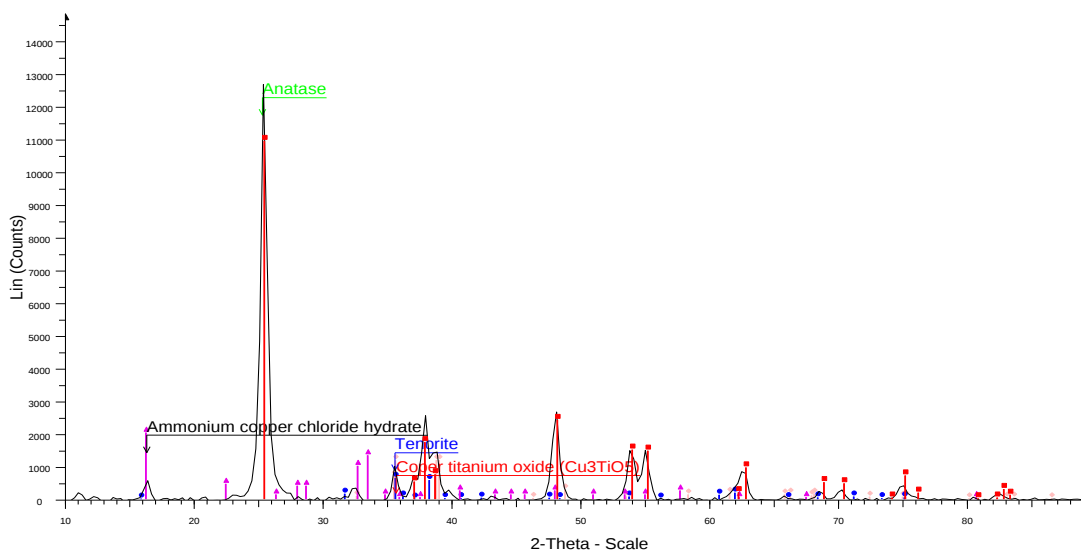
01-083-2243 (A) - Anatase, syn - TiO₂ - Y: 105.94 % - d x by: 1. - WL: 1.5406 - Tetragonal - a 3.78000 - b 3.78000 - c 9.51000 - alpha 90.000 - beta 90.000 - gamma 90.000 - Body-centered - I41/amd (141) - 4 - 135.88

01-080-0076 (C) - Tenorite, syn - CuO - Y: 32.99 % - d x by: 1. - WL: 1.5406 - Monoclinic - a 4.67970 - b 3.43140 - c 5.13620 - alpha 90.000 - beta 99.262 - gamma 90.000 - Base-centered - C2/c (15) - 4 - 81.4014 - I/c

00-018-0461 (D) - Copper Titanium Oxide - Cu₃TiO₅ - Y: 7.49 % - d x by: 0.9854 - WL: 1.5406 - Tetragonal - a 8.10000 - b 8.10000 - c 6.60000 - alpha 90.000 - beta 90.000 - gamma 90.000 - 433.026 - F22= 110.1760

01-083-1284 (C) - Copper Titanium Oxide - Cu₃TiO₄ - Y: 10.55 % - d x by: 1. - WL: 1.5406 - Rhombo H.axes - a 3.04000 - b 3.04000 - c 17.19300 - alpha 90.000 - beta 120.000 - gamma 120.000 - Primitive - R-3m (166

00-001-0211 (D) - Ammonium Copper Chloride Hydrate - (NH₄)₂CuCl₄·2H₂O - Y: 17.51 % - d x by: 1. - WL: 1.5406 - Tetragonal - a 7.58300 - b 7.58300 - c 7.95000 - alpha 90.000 - beta 90.000 - gamma 90.000 - 2 - 4



Ti-85 : Cu-15 - File: d5_523.raw - Type: 2Th/Th locked - Start: 10.000 ° - End: 91.300 ° - Step: 0.300 ° - Step time: 25. s - Temp.: 25 °C (Room) - Time Started: 11 s - 2-Theta: 10.000 ° - Theta: 5.000 ° - Chi: 0.00 ° - Phi

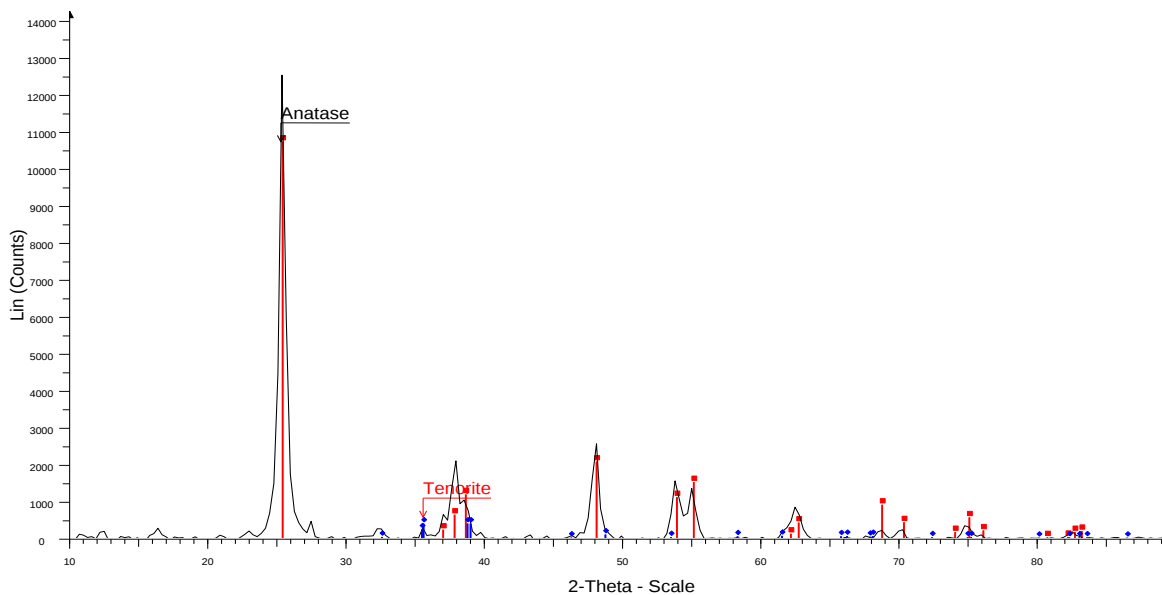
Operations: Background 1.000,1.000 | Import

01-089-4921 (C) - Anatase, syn - TiO₂ - Y: 86.47 % - d x by: 1. - WL: 1.5406 - Tetragonal - a 3.77700 - b 3.77700 - c 9.50100 - alpha 90.000 - beta 90.000 - gamma 90.000 - Body-centered - I41/amd (141) - 4 - 135.53

00-041-0254 (D) - Tenorite, syn - CuO - Y: 9.37 % - d x by: 1. - WL: 1.5406 - Monoclinic - a 4.68500 - b 3.42300 - c 5.13200 - alpha 90.000 - beta 99.520 - gamma 90.000 - Base-centered - C2/c (15) - 4 - 81.1672 - I/c

00-018-0461 (D) - Copper Titanium Oxide - Cu₃TiO₅ - Y: 5.11 % - d x by: 0.9854 - WL: 1.5406 - Tetragonal - a 8.10000 - b 8.10000 - c 6.60000 - alpha 90.000 - beta 90.000 - gamma 90.000 - 433.026 - F22= 110.1760

00-001-0211 (D) - Ammonium Copper Chloride Hydrate - (NH₄)₂CuCl₄·2H₂O - Y: 16.04 % - d x by: 1. - WL: 1.5406 - Tetragonal - a 7.58300 - b 7.58300 - c 7.95000 - alpha 90.000 - beta 90.000 - gamma 90.000 - 2 - 4

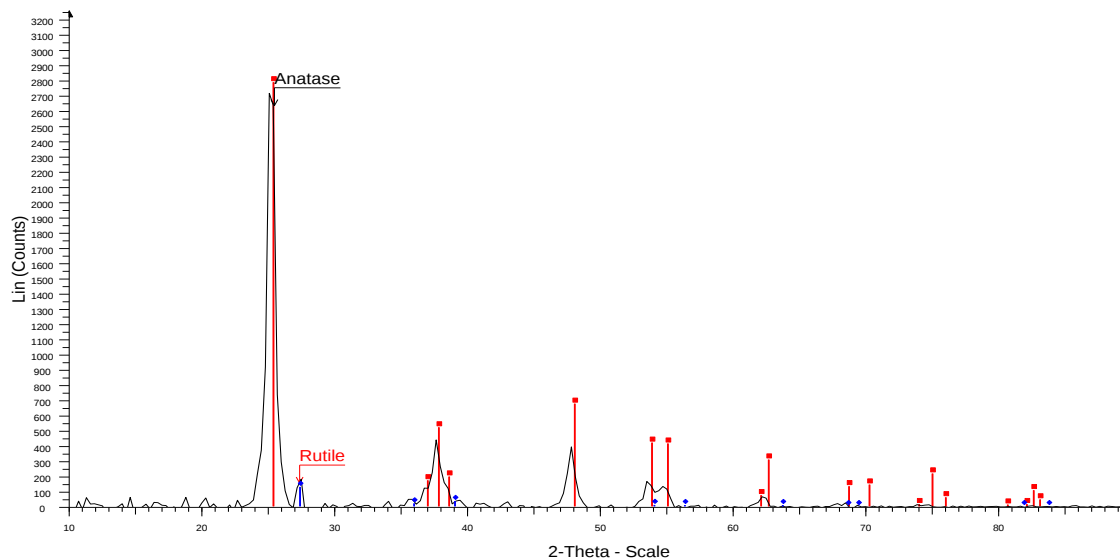


Ti-90 : Cu-10 - File: d5_524.raw - Type: 2Th/Th locked - Start: 10.000 ° - End: 91.300 ° - Step: 0.300 ° - Step time: 25. s - Temp.: 25 °C (Room) - Time Started: 11 s - 2-Theta: 10.000 ° - Theta: 5.000 ° - Chi: 0.00 ° - Phi

Operations: Background 1.000,1.000 | Import

01-083-2243 (A) - Anatase, syn - TiO₂ - Y: 85.69 % - d x by: 1. - WL: 1.5406 - Tetragonal - a 3.78000 - b 3.78000 - c 9.51000 - alpha 90.000 - beta 90.000 - gamma 90.000 - Body-centered - I41/amd (141) - 4 - 135.88

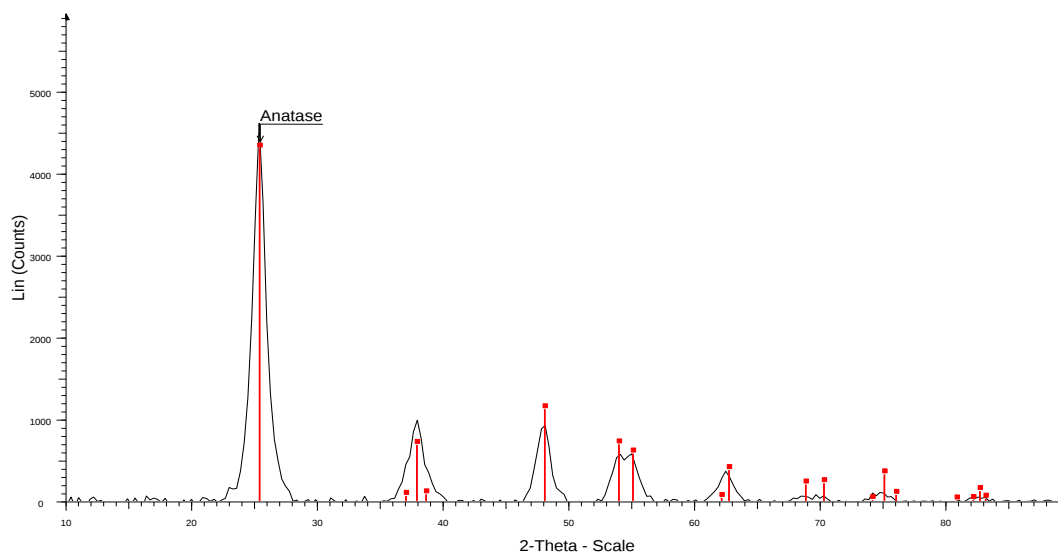
00-041-0254 (D) - Tenorite, syn - CuO - Y: 3.13 % - d x by: 1. - WL: 1.5406 - Monoclinic - a 4.68500 - b 3.42300 - c 5.13200 - alpha 90.000 - beta 99.520 - gamma 90.000 - Base-centered - C2/c (15) - 4 - 81.1672 - I/c



Ti-95 : Cu-K α - File: d5_525.raw - Type: 2Th/Th locked - Start: 10.000 ° - End: 91.300 ° - Step: 0.300 ° - Step time: 25. s - Temp.: 25 °C (Room) - Time Started: 12 s - 2-Theta: 10.000 ° - Theta: 5.000 ° - Chi: 0.00 ° - Phi: 0.00 ° - Operations: Background 1.000,1.000 | Import

01-071-1166 (A) - Anatase - TiO₂ - Y: 102.71 % - d x by: 1. - WL: 1.5406 - Tetragonal - a 3.78420 - b 3.78420 - c 9.51460 - alpha 90.000 - beta 90.000 - gamma 90.000 - Body-centered - I41/amd (141) - 4 - 136.251 - I

00-034-0180 (D) - Rutile, syn - TiO₂ - Y: 4.70 % - d x by: 1.0042 - WL: 1.5406 - Tetragonal - a 4.59300 - b 4.59300 - c 2.95900 - alpha 90.000 - beta 90.000 - gamma 90.000 - Primitive - P42/mnm (136) - 2 - 62.4220 - I

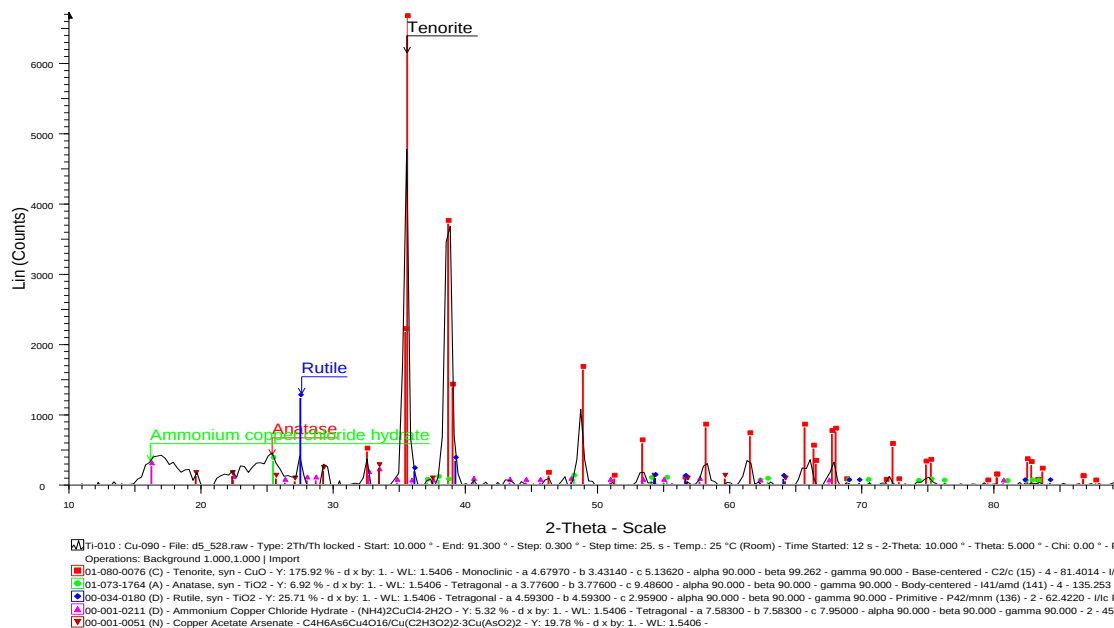
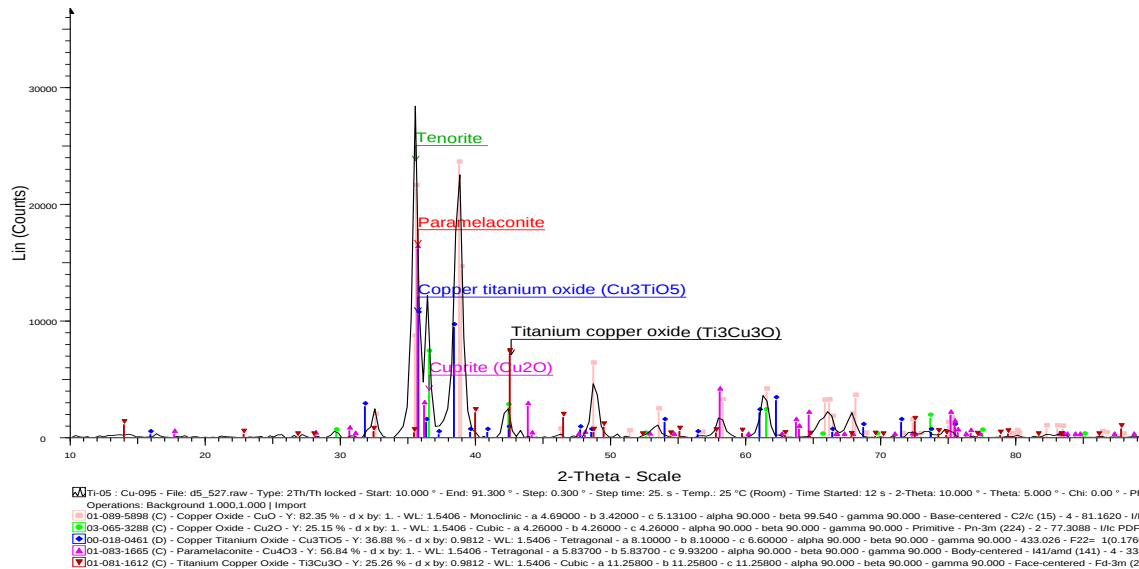


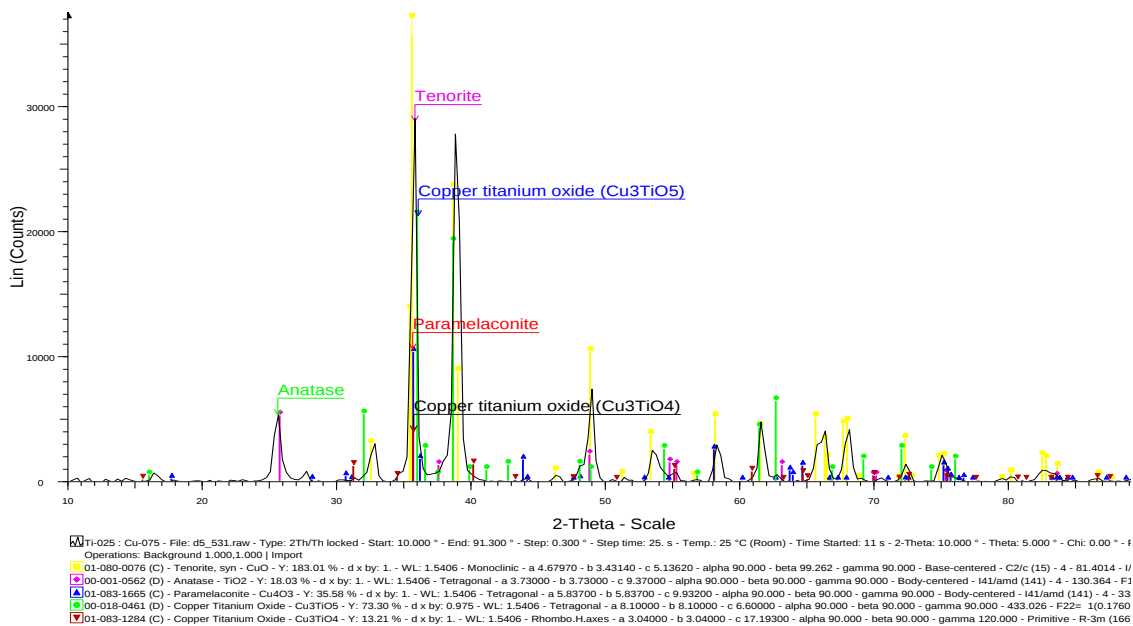
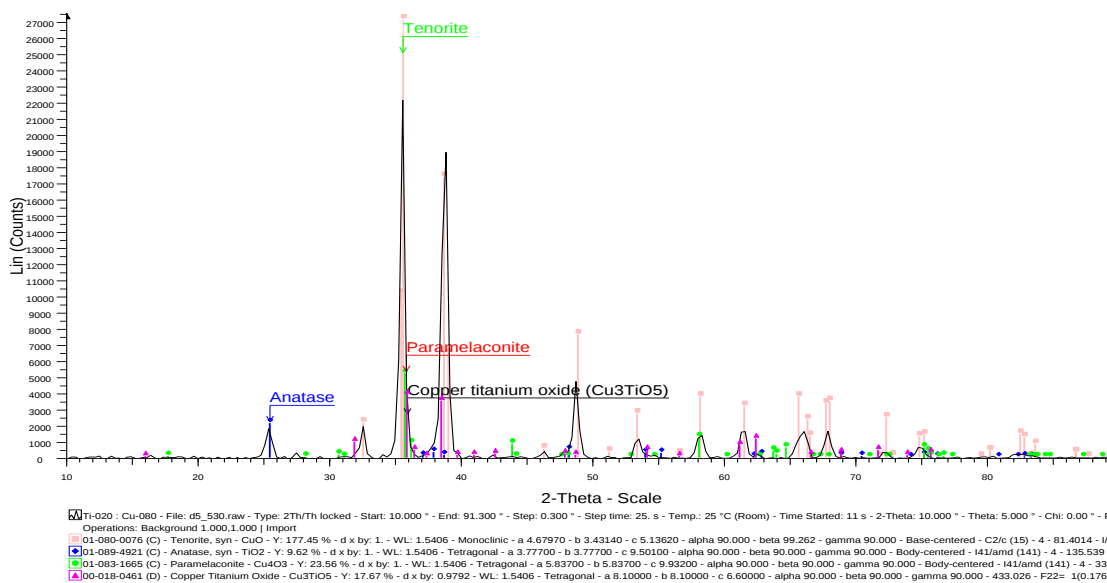
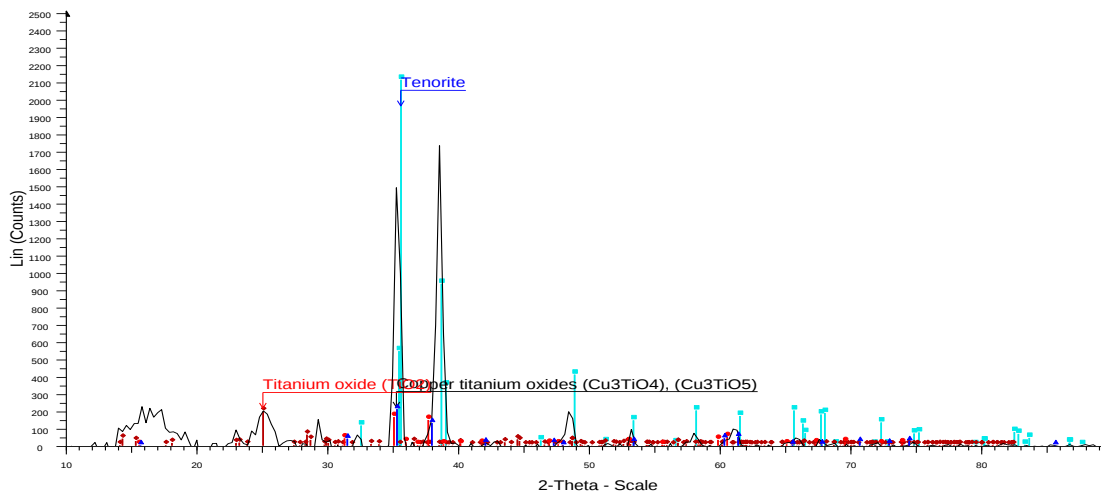
Ti-100 : Cu-K α - File: d5_526.raw - Type: 2Th/Th locked - Start: 10.000 ° - End: 91.300 ° - Step: 0.300 ° - Step time: 25. s - Temp.: 25 °C (Room) - Time Started: 11 s - 2-Theta: 10.000 ° - Theta: 5.000 ° - Chi: 0.00 ° - Phi: 0.00 ° - Operations: Background 1.000,1.000 | Import

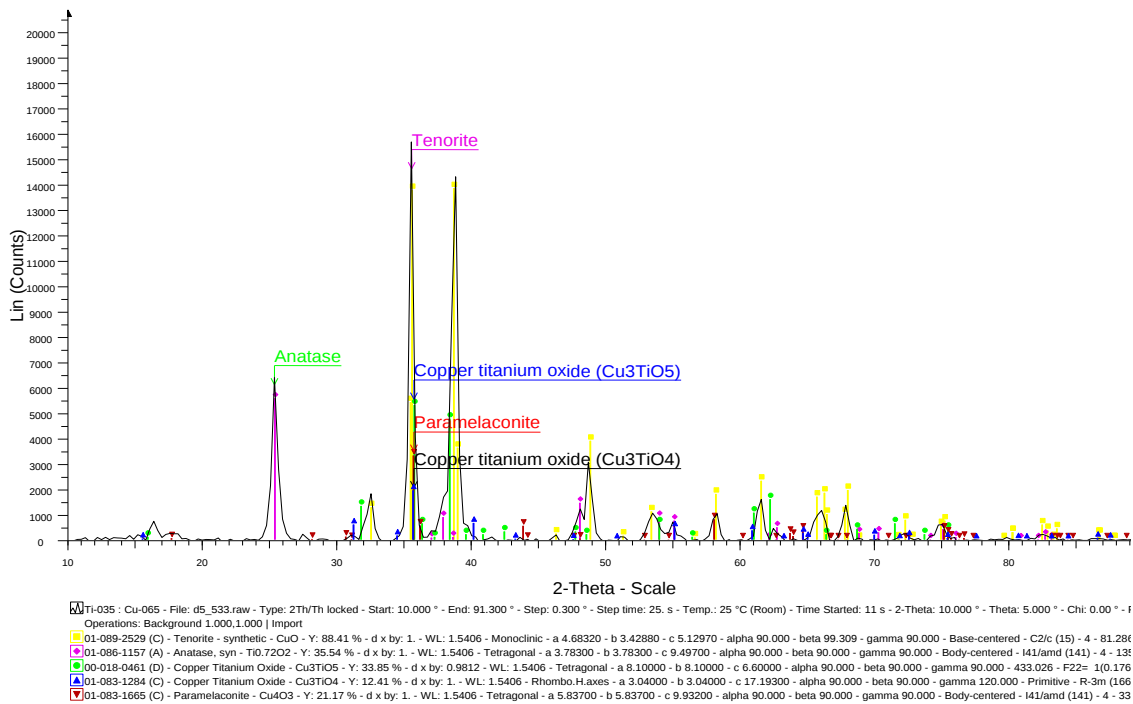
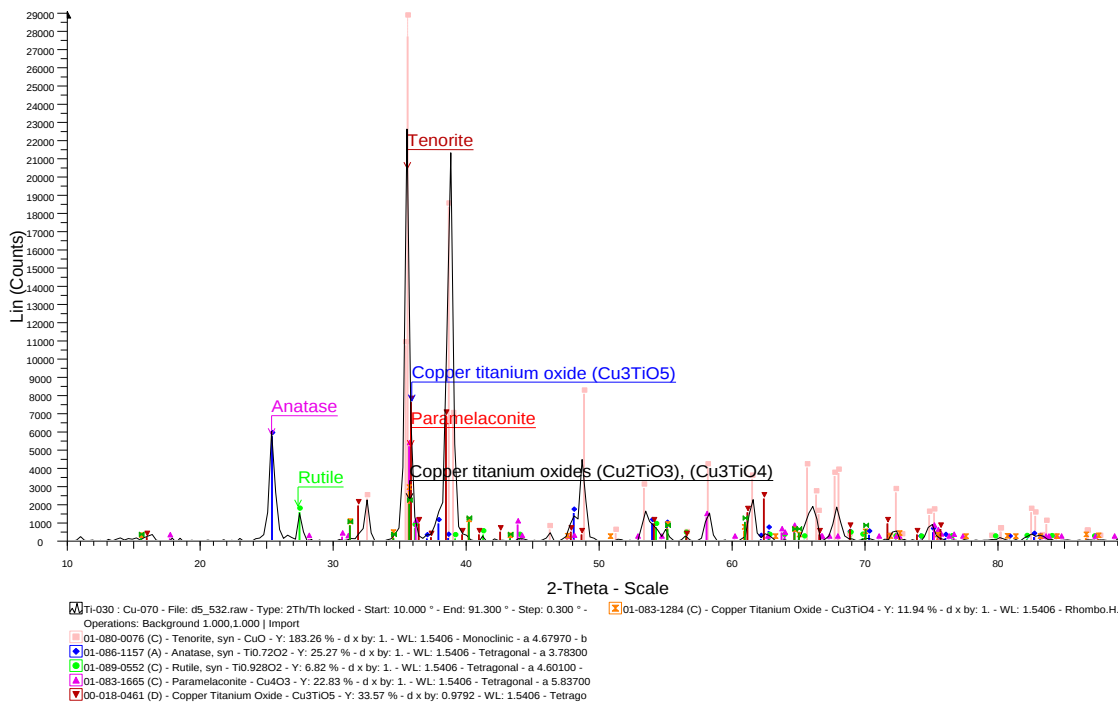
01-086-1157 (A) - Anatase, syn - TiO₂ - Y: 93.34 % - d x by: 1. - WL: 1.5406 - Tetragonal - a 3.78300 - b 3.78300 - c 9.49700 - alpha 90.000 - beta 90.000 - gamma 90.000 - Body-centered - I41/amd (141) - 4 - 136.251 - I

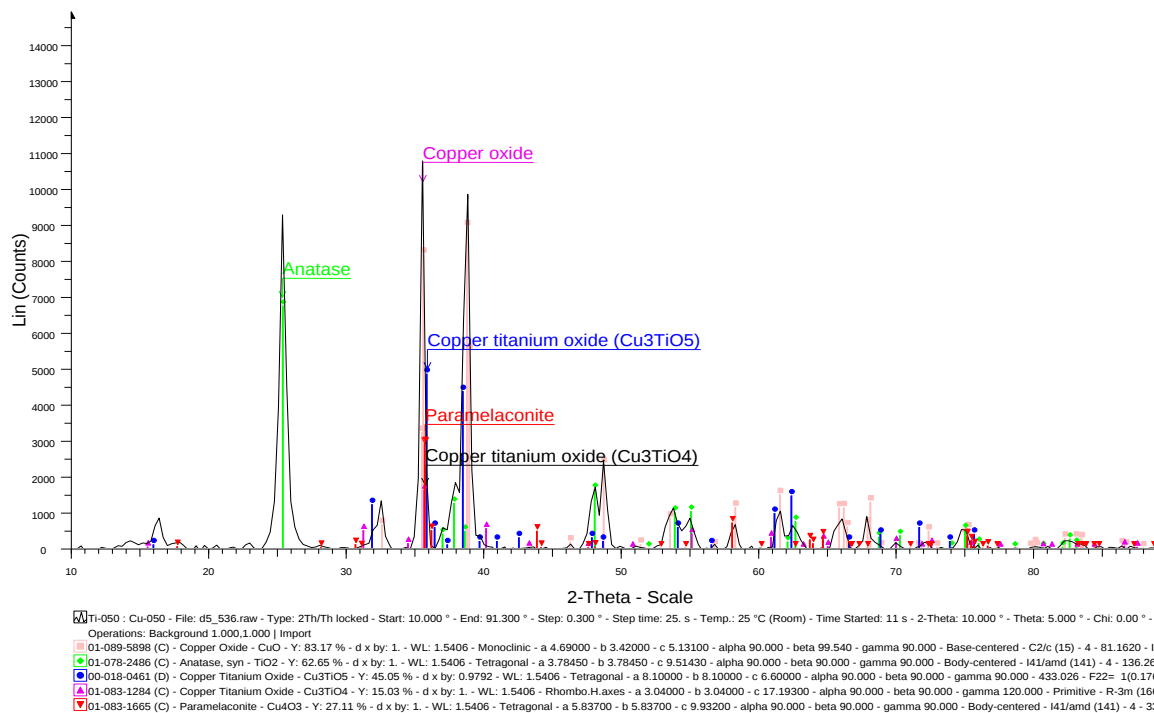
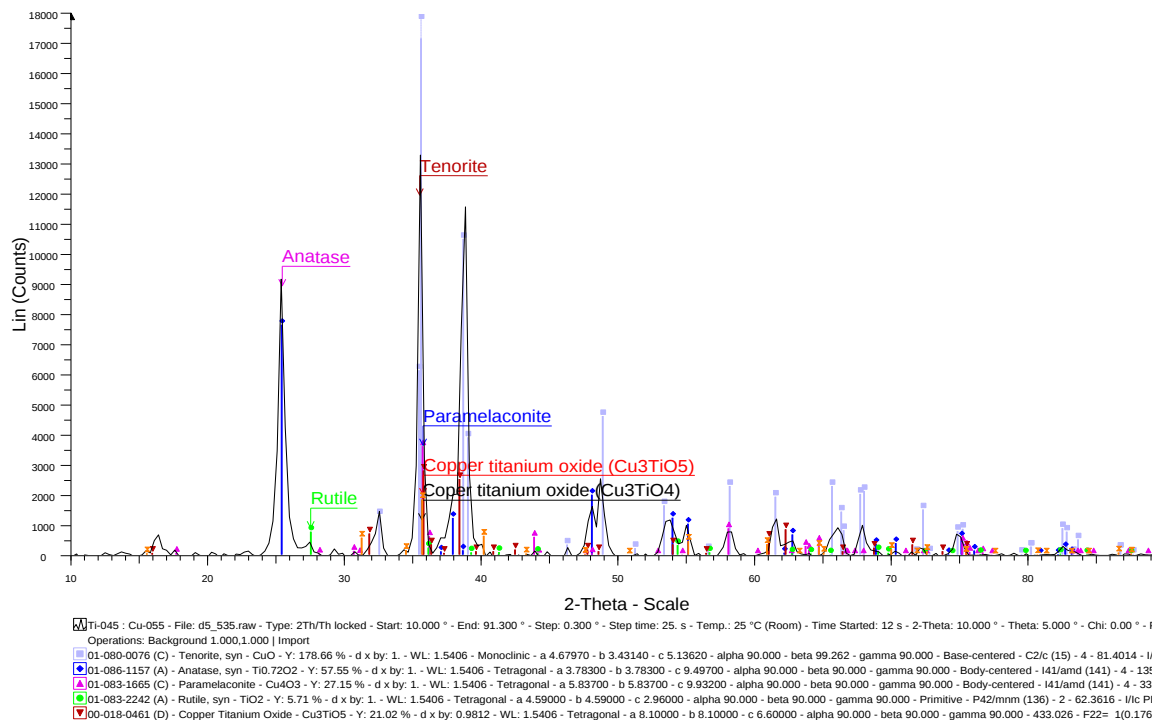
Appendix C

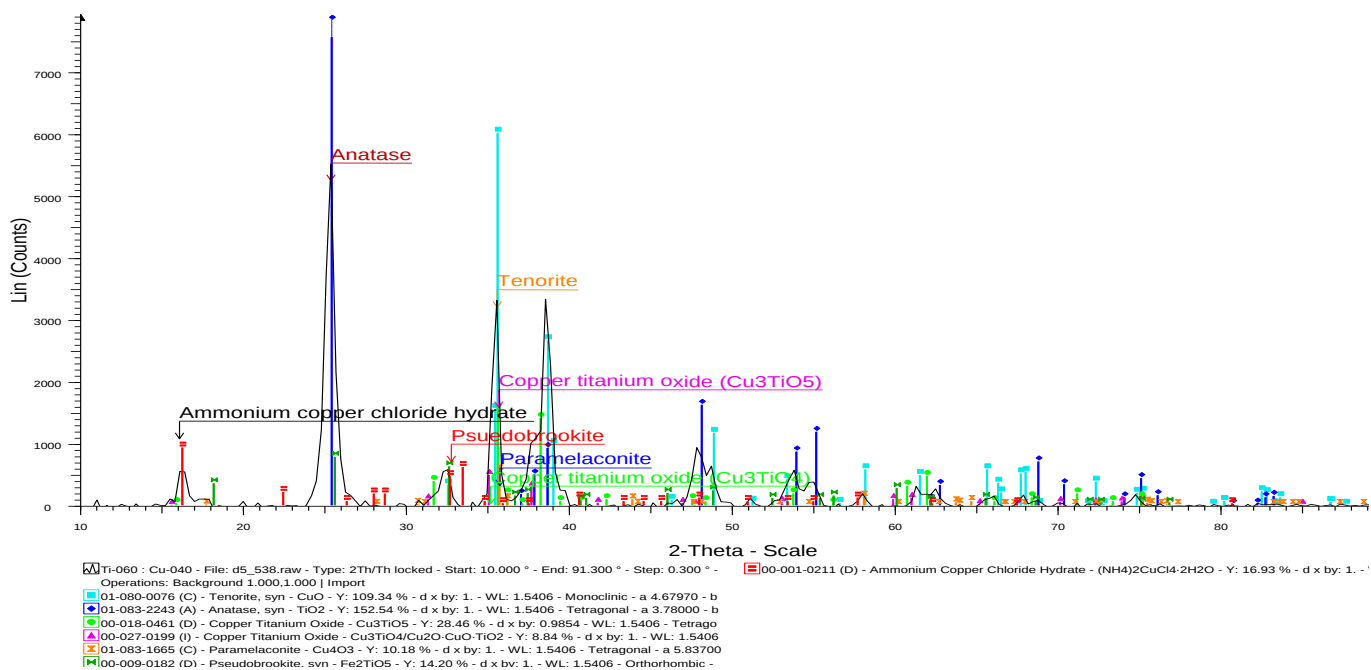
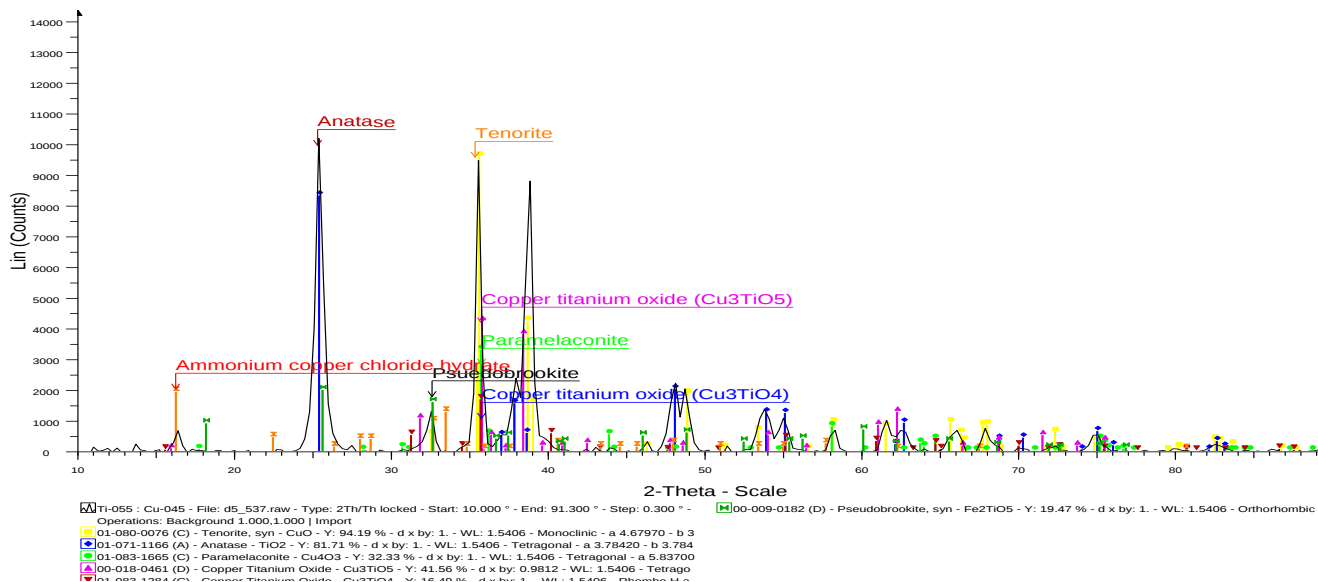
XRD Patterns of Resin-gel precursor powders calcined at 300 °C

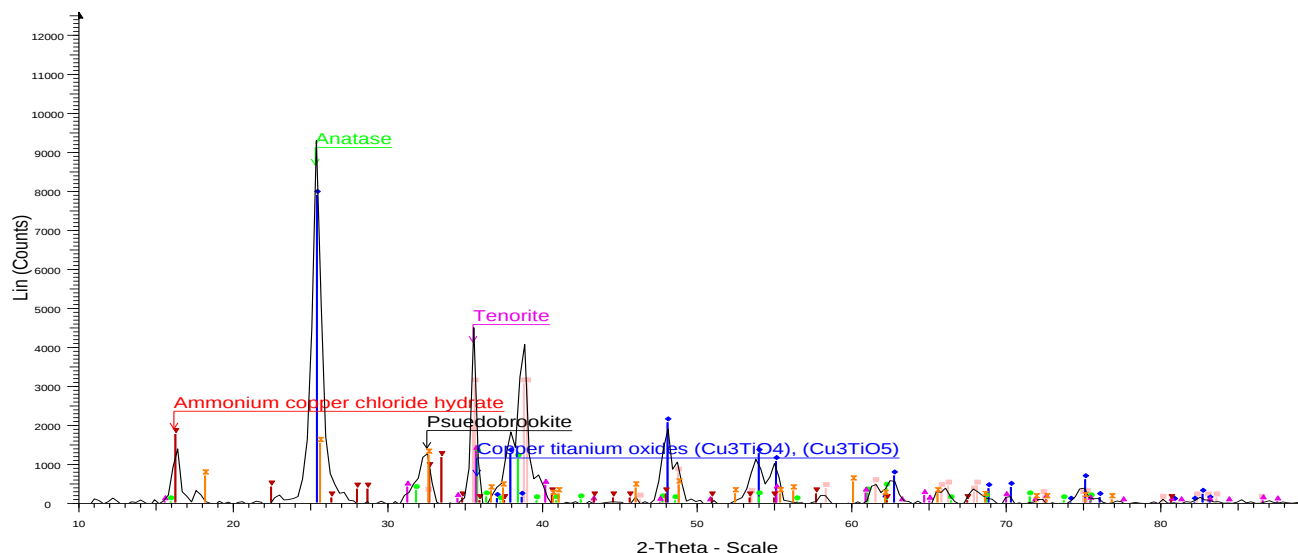




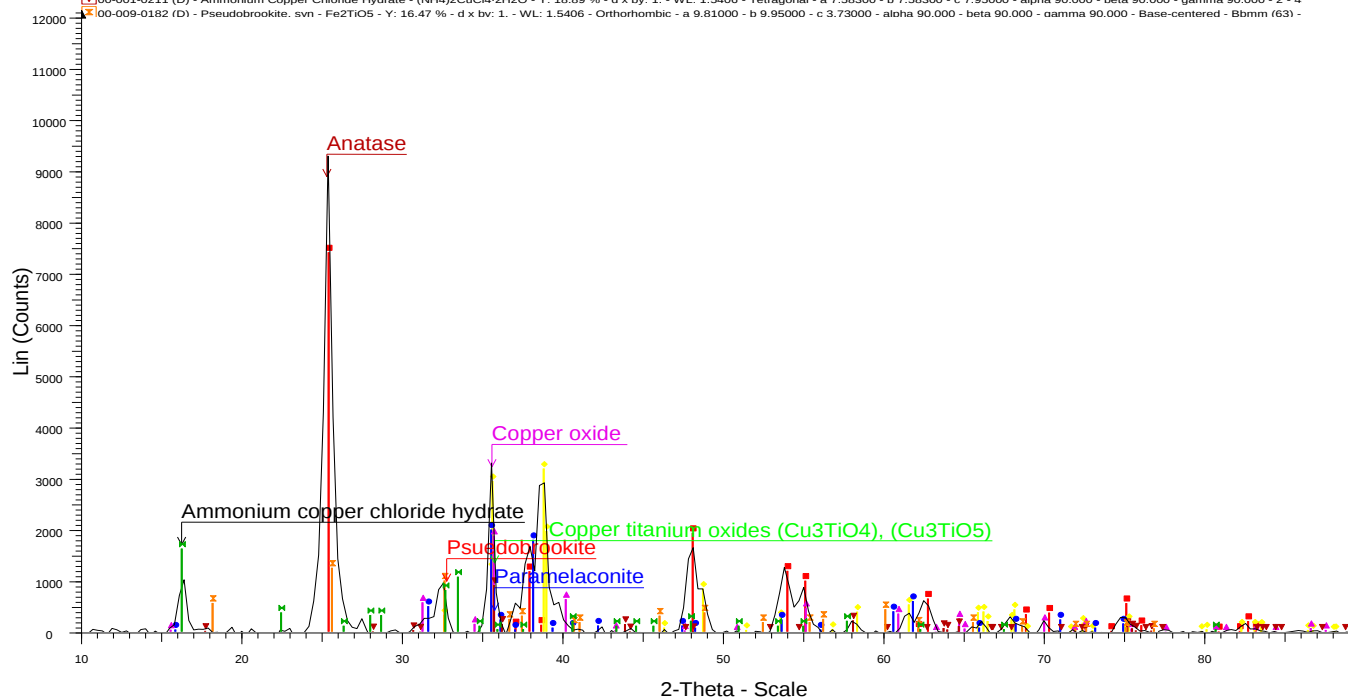




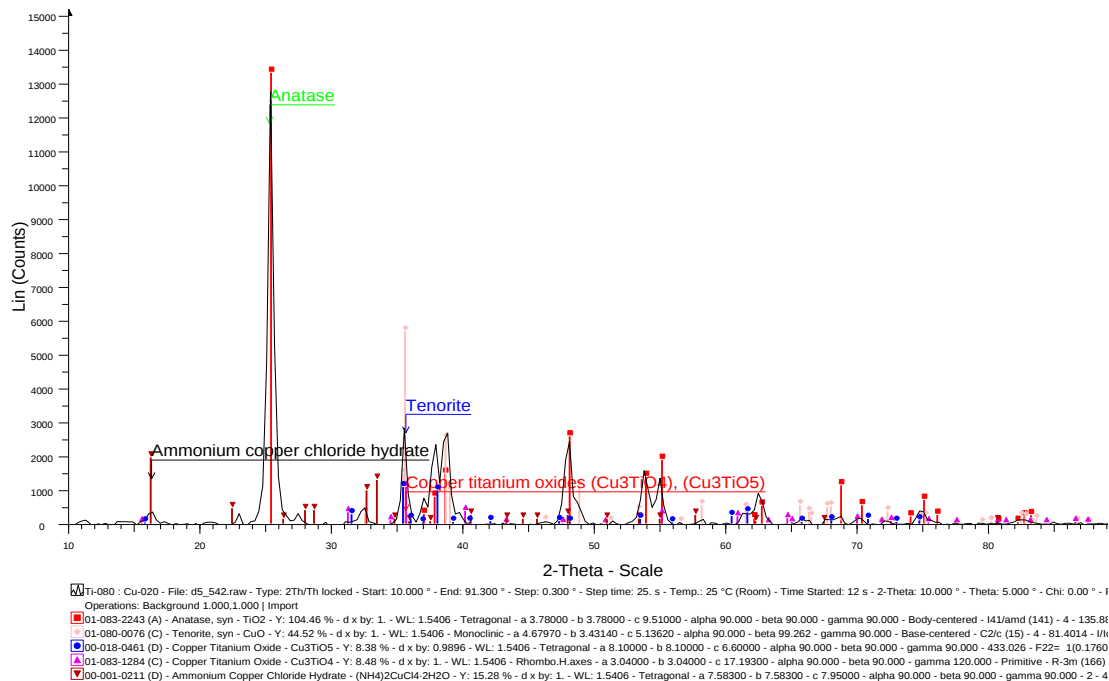
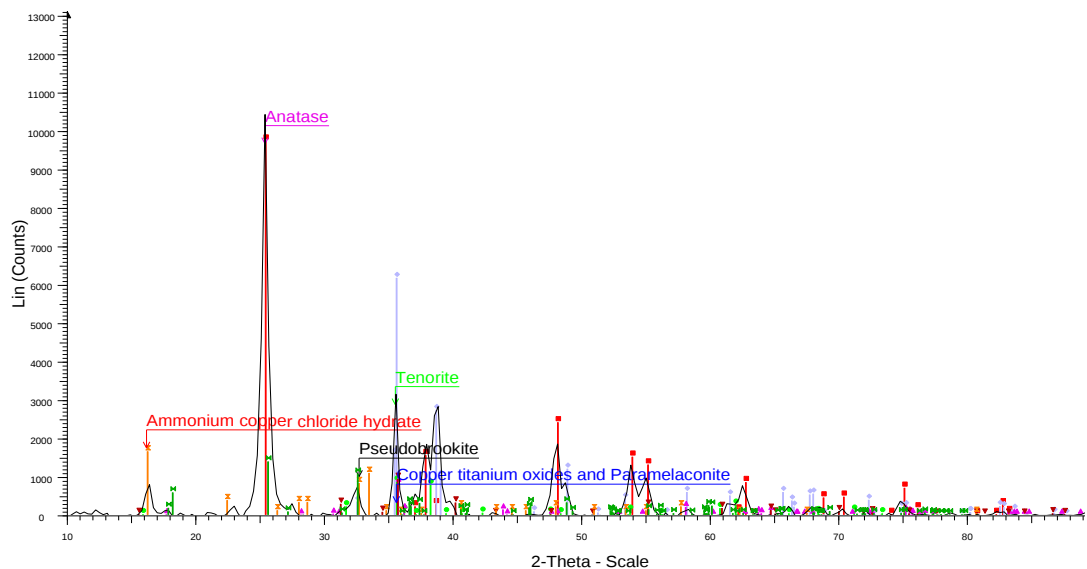


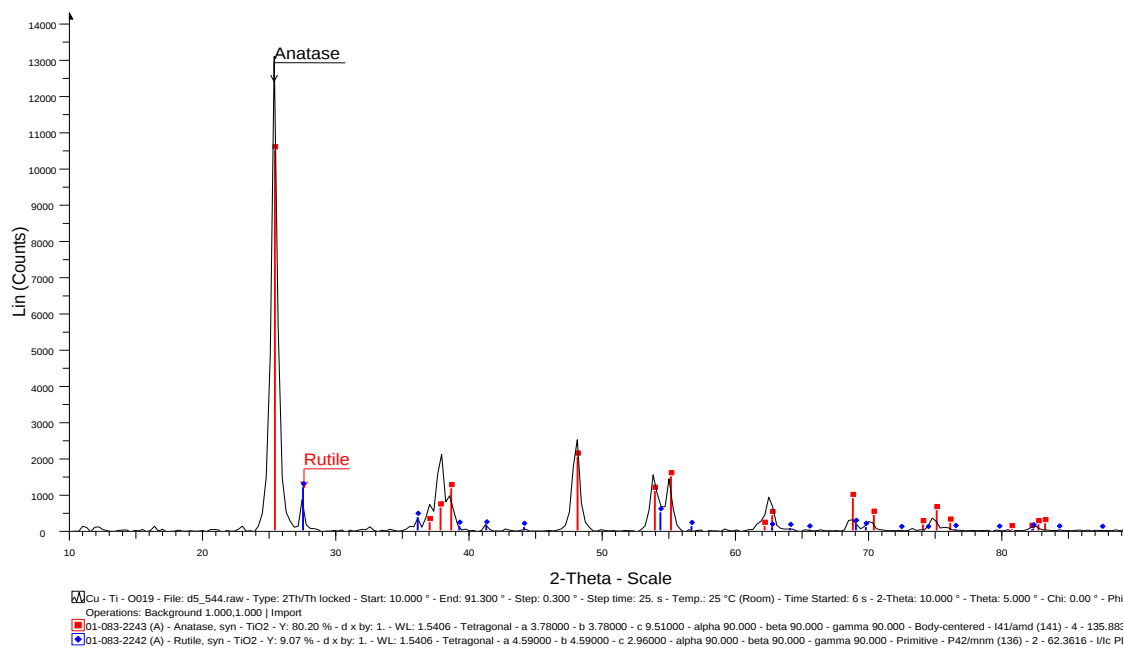
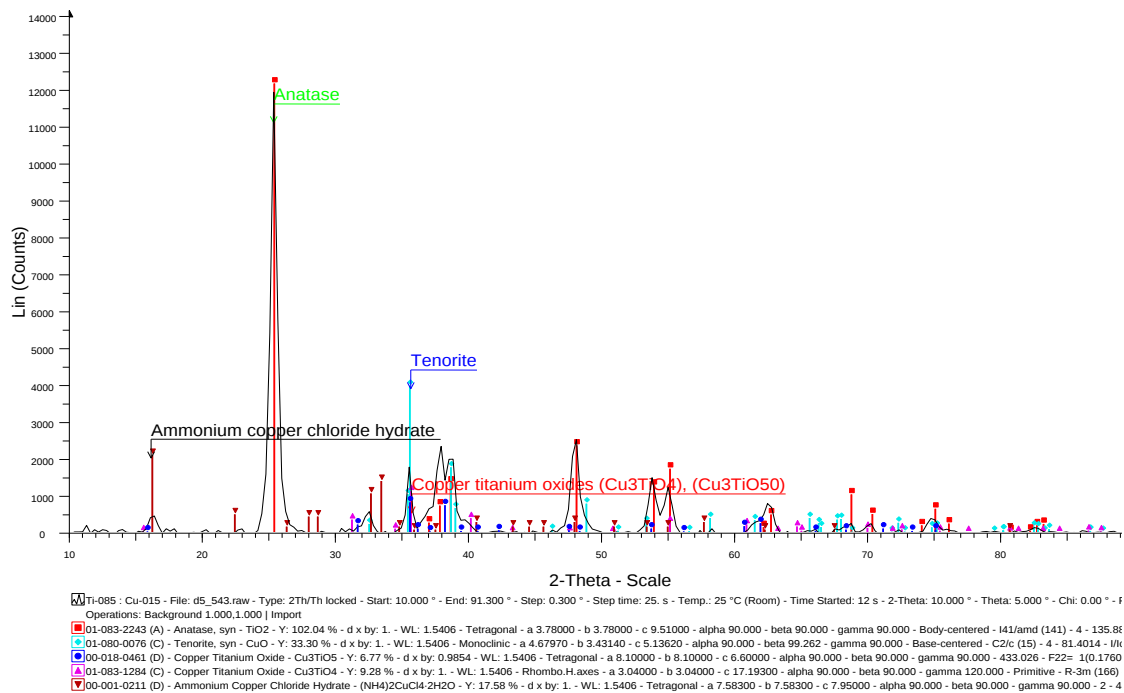


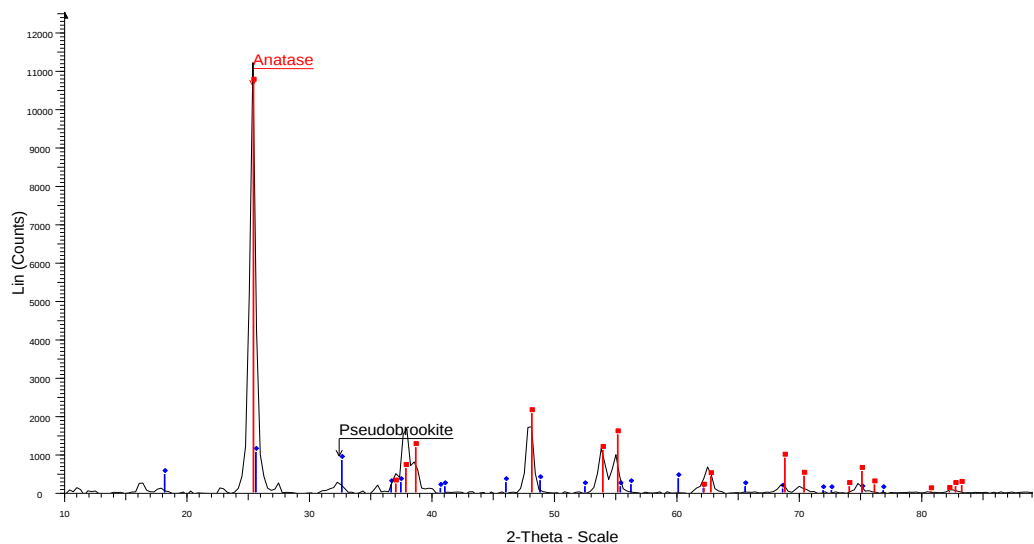
Ti-065 : Cu-035 - File: d5_539.raw - Type: 2Th/Th locked - Start: 10.000 ° - End: 91.300 ° - Step: 0.300 ° - Step time: 25. s - Temp.: 25 °C (Room) - Time Started: 11 s - 2-Theta: 10.000 ° - Theta: 5.000 ° - Chi: 0.00 ° - F
 Operations: Background 1.000,1.000 | Import
 00-041-0254 (D) - Tenorite, syn - CuO - Y: 32.90 % - d x by: 1. - WL: 1.5406 - Monoclinic - a 4.68500 - b 3.42300 - c 5.13200 - alpha 90.000 - beta 99.520 - gamma 90.000 - Base-centered - C2/c (15) - 4 - 81.1672 - I/k
 01-086-1157 (A) - Anatase, syn - TiO₂ - Y: 85.02 % - d x by: 1. - WL: 1.5406 - Tetragonal - a 3.78300 - b 3.78300 - c 9.49700 - alpha 90.000 - beta 90.000 - gamma 90.000 - Body-centered - I41/amd (141) - 4 - 135
 00-018-0461 (D) - Copper Titanium Oxide - Cu₃TiO₅ - Y: 13.39 % - d x by: 0.9812 - WL: 1.5406 - Tetragonal - a 8.10000 - b 8.10000 - c 6.80000 - alpha 90.000 - beta 90.000 - gamma 90.000 - 433.026 - F22c- 1(0.176
 01-083-1284 (C) - Copper Titanium Oxide - Cu₃TiO₄ - Y: 14.28 % - d x by: 1. - WL: 1.5406 - Rhombo.Haxes - a 3.04000 - b 3.04000 - c 17.19300 - alpha 90.000 - beta 90.000 - gamma 120.000 - Primitive - R-3m (166
 00-001-0211 (D) - Ammonium Copper Chloride Hydrate - (NH₄)₂CuCl₄·2H₂O - Y: 18.89 % - d x by: 1. - WL: 1.5406 - Tetragonal - a 7.58300 - b 7.58300 - c 7.95000 - alpha 90.000 - beta 90.000 - gamma 90.000 - 2 - 4
 00-009-0182 (D) - Pseudobrookite, syn - Fe₂TiO₅ - Y: 16.47 % - d x by: 1. - WL: 1.5406 - Orthorhombic - a 9.81000 - b 9.95000 - c 3.73000 - alpha 90.000 - beta 90.000 - gamma 90.000 - Base-centered - Bbmm (63) -



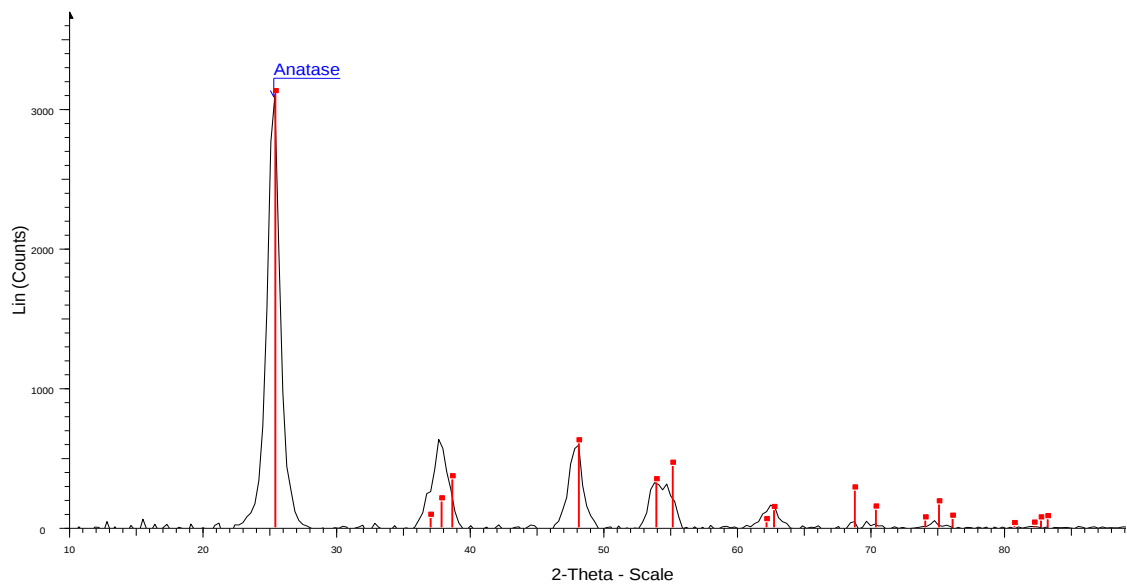
Ti-070 : Cu-030 - File: d5_540.raw - Type: 2Th/Th locked - Start: 10.000 ° - End: 91.300 ° - Step: 0.300 ° - 00-001-0211 (D) - Ammonium Copper Chloride Hydrate - (NH₄)₂CuCl₄·2H₂O - Y: 17.48 % - d x by: 1. -
 Operations: Background 1.000,1.000 | Import
 01-086-1157 (A) - Anatase, syn - TiO₂ - Y: 79.82 % - d x by: 1. - WL: 1.5406 - Tetragonal - a 3.78300
 01-089-5899 (C) - Copper Oxide - CuO - Y: 34.29 % - d x by: 1. - WL: 1.5406 - Monoclinic - a 4.68900 - b
 00-018-0461 (D) - Copper Titanium Oxide - Cu₃TiO₅ - Y: 21.45 % - d x by: 0.9875 - WL: 1.5406 - Tetrago
 01-083-1284 (C) - Copper Titanium Oxide - Cu₃TiO₄ - Y: 20.19 % - d x by: 1. - WL: 1.5406 - Rhombo.H.a
 01-083-1665 (C) - Paramelaconite - Cu₄O₃ - Y: 9.81 % - d x by: 1. - WL: 1.5406 - Tetragonal - a 5.83700 -
 00-009-0182 (D) - Pseudobrookite, syn - Fe₂TiO₅ - Y: 13.45 % - d x by: 1. - WL: 1.5406 - Orthorhombic -







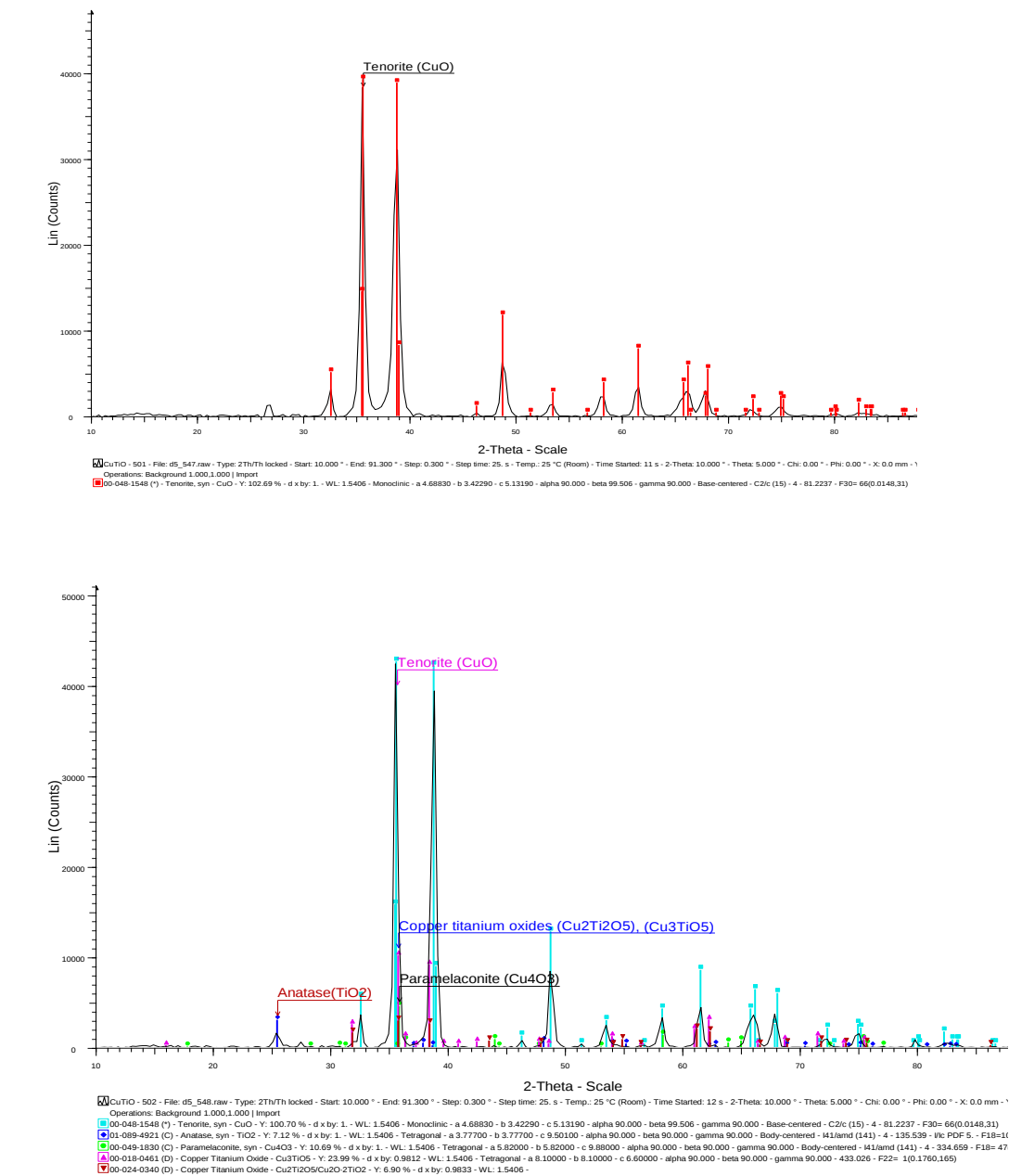
☒ Cu - Ti - O020 - File: d5_545.raw - Type: 2Th/Th locked - Start: 10.000 ° - End: 91.300 ° - Step: 0.300 ° - Step time: 25. s - Temp.: 25 °C (Room) - Time Started: 11 s - 2-Theta: 10.000 ° - Theta: 5.000 ° - Chi: 0.00 ° - Phi
Operations: Background 1.000.1.000 | Import
☒ 01-083-2243 (A) - Anatase, syn - TiO₂ - Y: 95.37 % - d x by: 1. - WL: 1.5406 - Tetragonal - a 3.78000 - b 3.78000 - c 9.51000 - alpha 90.000 - beta 90.000 - gamma 90.000 - Body-centered - I41/amd (141) - 4 - 135.88;
☒ 00-009-0182 (D) - Pseudobrookite, syn - Fe₂TiO₅ - Y: 9.39 % - d x by: 1. - WL: 1.5406 - Orthorhombic - a 9.81000 - b 9.95000 - c 3.73000 - alpha 90.000 - beta 90.000 - gamma 90.000 - Base-centered - Bbmm (63) - 4

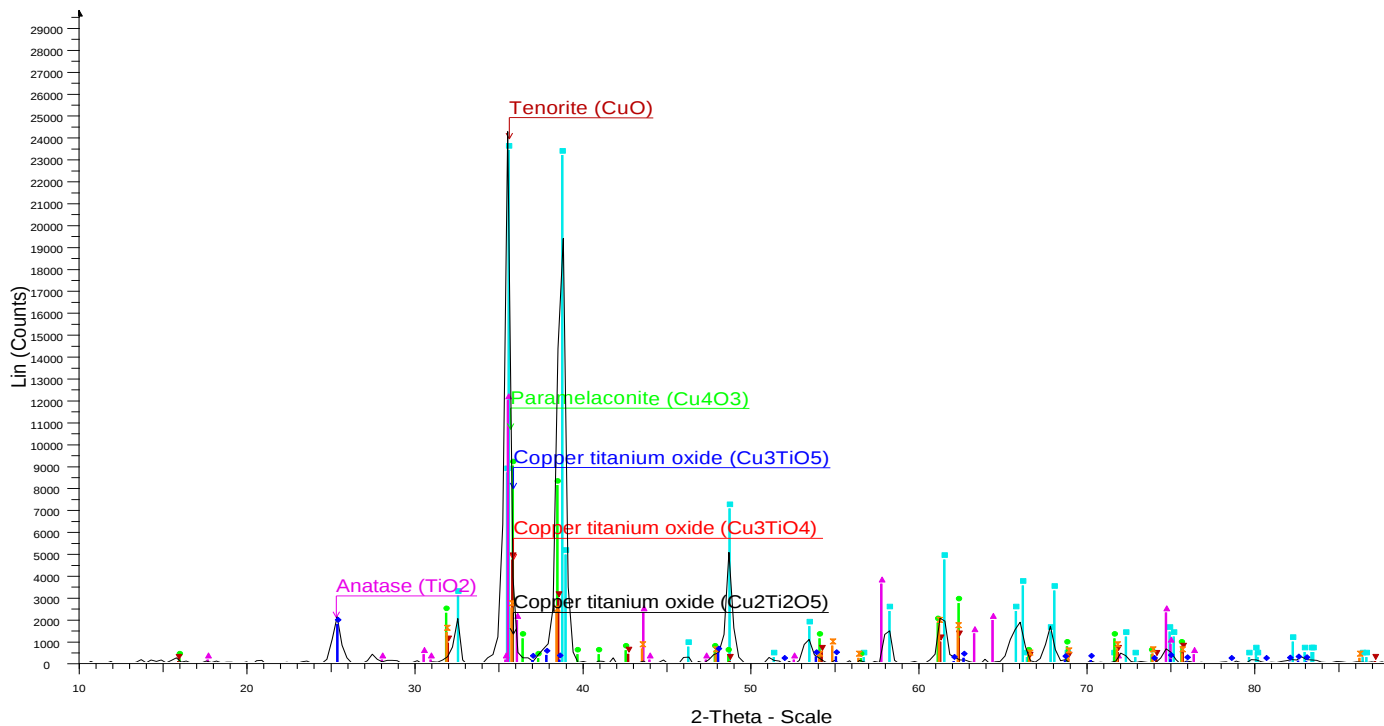
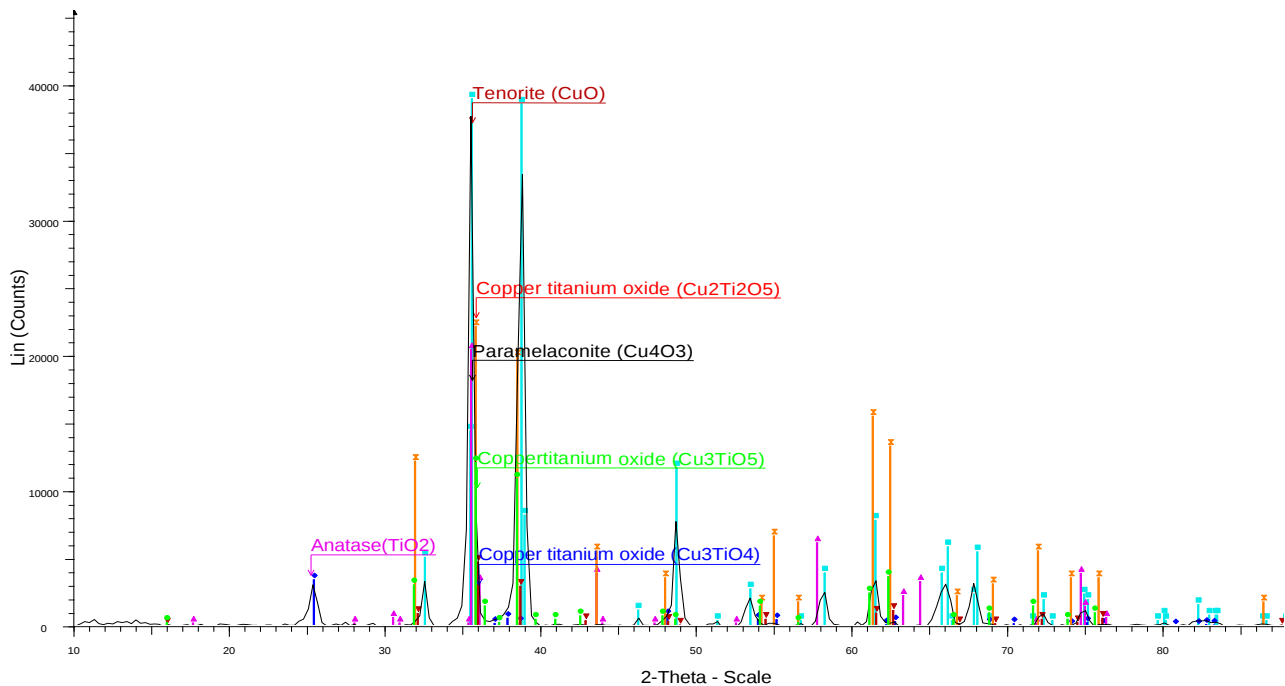


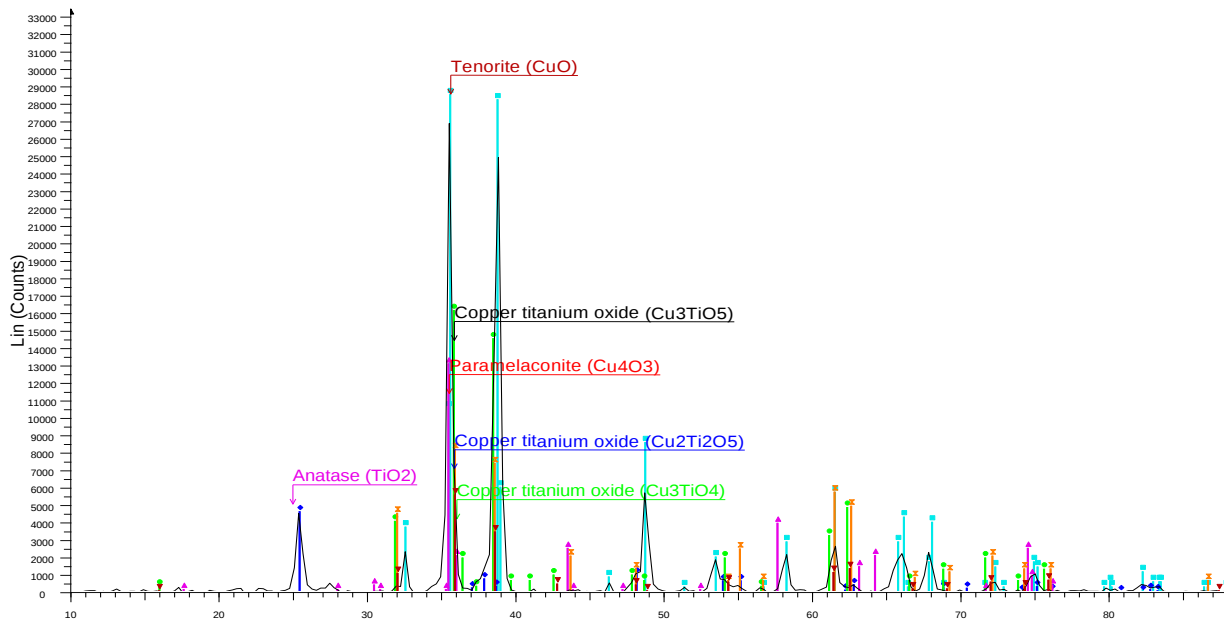
☒ Cu - Ti - O021 - File: d5_546.raw - Type: 2Th/Th locked - Start: 10.000 ° - End: 91.300 ° - Step: 0.300 ° - Step time: 25. s - Temp.: 25 °C (Room) - Time Started: 11 s - 2-Theta: 10.000 ° - Theta: 5.000 ° - Chi: 0.00 ° - Phi
Operations: Background 1.000.1.000 | Import
☒ 01-083-2243 (A) - Anatase, syn - TiO₂ - Y: 100.57 % - d x by: 1. - WL: 1.5406 - Tetragonal - a 3.78000 - b 3.78000 - c 9.51000 - alpha 90.000 - beta 90.000 - gamma 90.000 - Body-centered - I41/amd (141) - 4 - 135.88

APPENDIX D

Patterns of Resin-gel precursor powders calcined at 500 °C







00-CuTiO-505 - File: d5_551.raw - Type: 2Th/Th locked - Start: 10.000 ° - End: 91.300 ° - Step: 0.300 ° - Step time: 25. s - Temp.: 25 °C (Room) - Time Started: 11 s - 2-Theta: 10.000 ° - Theta: 5.000 ° - Chi: 0.00 ° - Phi: 0.00 ° - X: 0.0 mm - Y: 0.0 mm - Z: 0.0 mm - Operations: Background 1.000,1.000 | Import

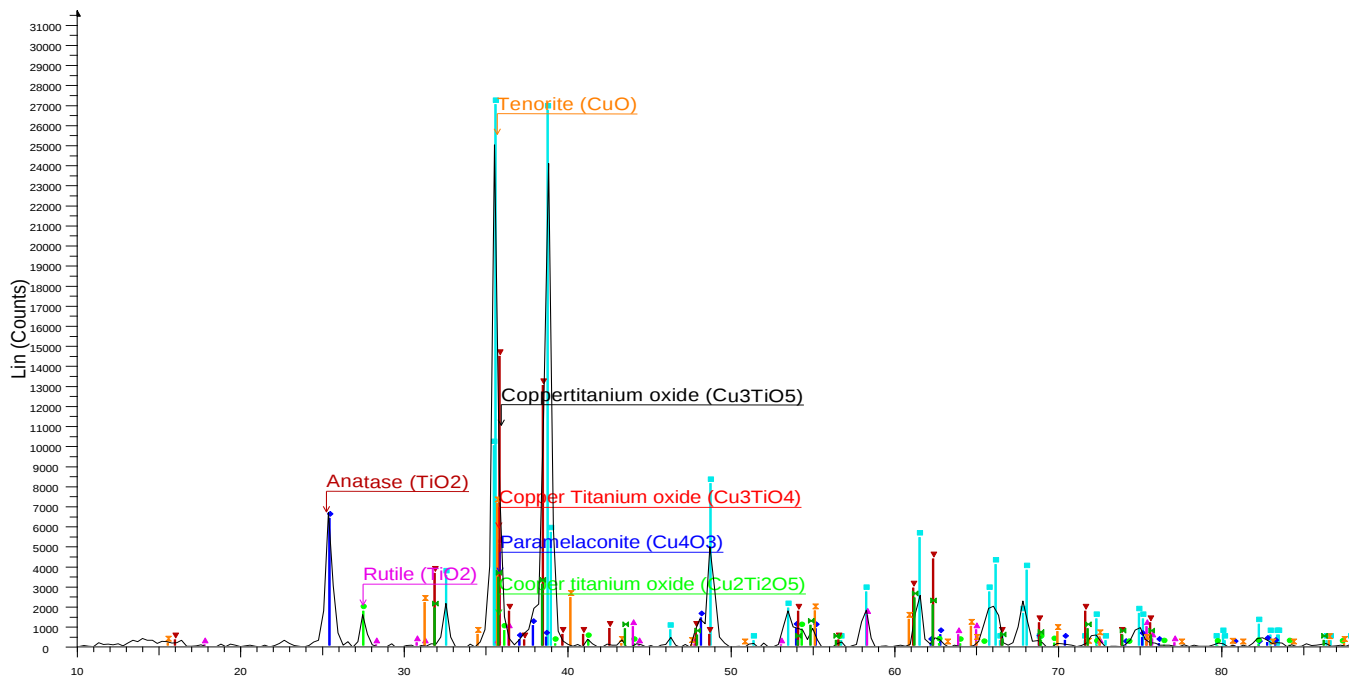
00-048-1548 (*) - Tenorite, syn - CuO - Y: 106.34 % - d x by: 1. - WL: 1.5406 - Monoclinic - a 4.68830 - b 3.42290 - c 5.13190 - alpha 90.000 - beta 99.506 - gamma 90.000 - Base-centered - C2/c (15) - 4 - 81.2237 - F30= 66(0.0148,31)

01-089-4921 (C) - Anatase, syn - TiO2 - Y: 17.16 % - d x by: 1. - WL: 1.5406 - Tetragonal - a 3.77700 - b 3.77700 - c 9.50100 - alpha 90.000 - beta 90.000 - gamma 90.000 - Body-centered - I41/amd (141) - 4 - 135.539 - I/c PDF 5 - F18=1

00-018-0461 (D) - Copper Titanium Oxide - Cu3TiO5 - Y: 60.19 % - d x by: 0.9792 - WL: 1.5406 - Tetragonal - a 8.10000 - b 8.10000 - c 6.60000 - alpha 90.000 - beta 90.000 - gamma 90.000 - 433.026 - F22= 1(0.1760,165)

00-049-1830 (C) - Paramelaconite, syn - Cu4O3 - Y: 48.74 % - d x by: 1.0104 - WL: 1.5406 - Tetragonal - a 5.82000 - b 5.82000 - c 9.88000 - alpha 90.000 - beta 90.000 - gamma 90.000 - Body-centered - I41/amd (141) - 4 - 334.659 - F1E

00-027-0199 (I) - Copper Titanium Oxide - Cu3TiO4/Cu2O-CuO-TiO2 - Y: 20.68 % - d x by: 0.9771 - WL: 1.5406 - Hexagonal - a 3.03540 - b 3.03540 - c 11.44000 - alpha 90.000 - beta 90.000 - gamma 120.000 - Primitive - P63/mmc (194)



00-CuTiO-506 - File: d5_552.raw - Type: 2Th/Th locked - Start: 10.000 ° - End: 91.300 ° - Step: 0.300 ° - Step time: 25. s - Temp.: 25 °C (Room) - Time Started: 11 s - 2-Theta: 10.000 ° - Theta: 5.000 ° - Chi: 0.00 ° - Phi: 0.00 ° - X: 0.0 mm - Y: 0.0 mm - Z: 0.0 mm - Operations: Background 1.000,1.000 | Import

00-048-1548 (*) - Tenorite, syn - CuO - Y: 108.17 % - d x by: 1. - WL: 1.5406 - Monoclinic - a 4.68830 - b 3.42290 - c 5.13190 - alpha 90.000 - beta 99.506 - gamma 90.000 - Base-centered - C2/c (15) - 4 - 81.2237 - F30= 66(0.0148,31)

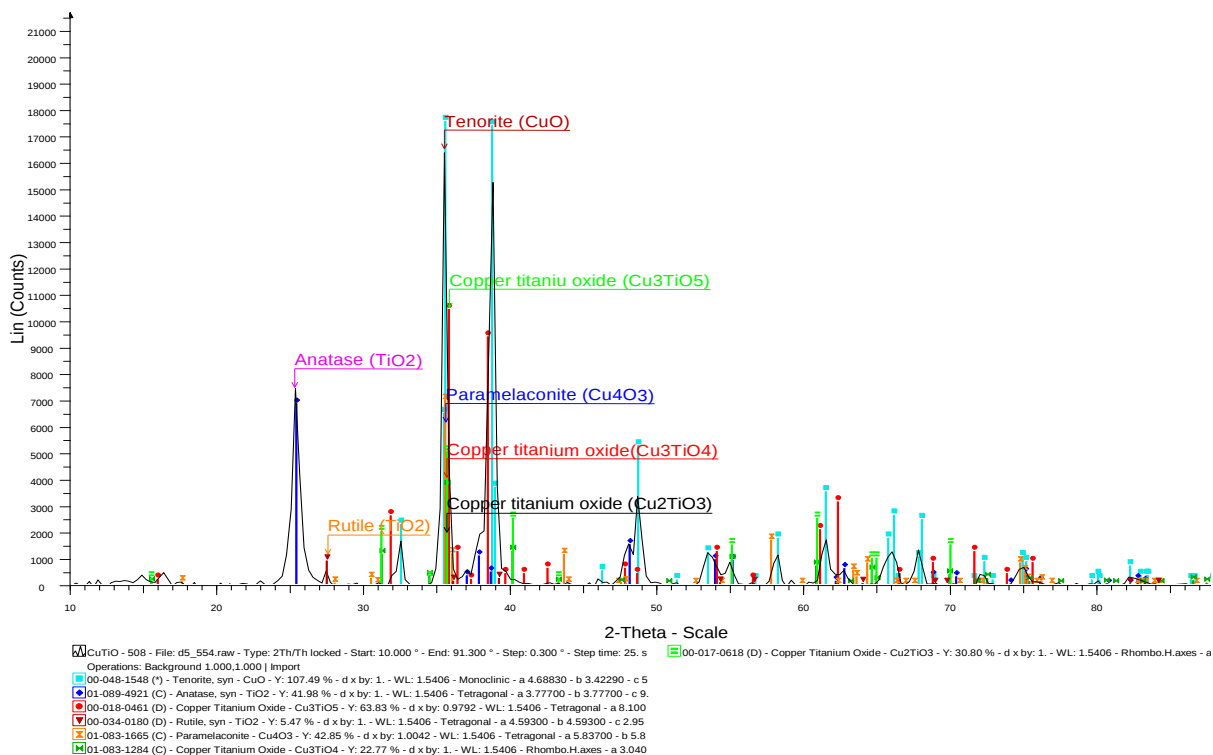
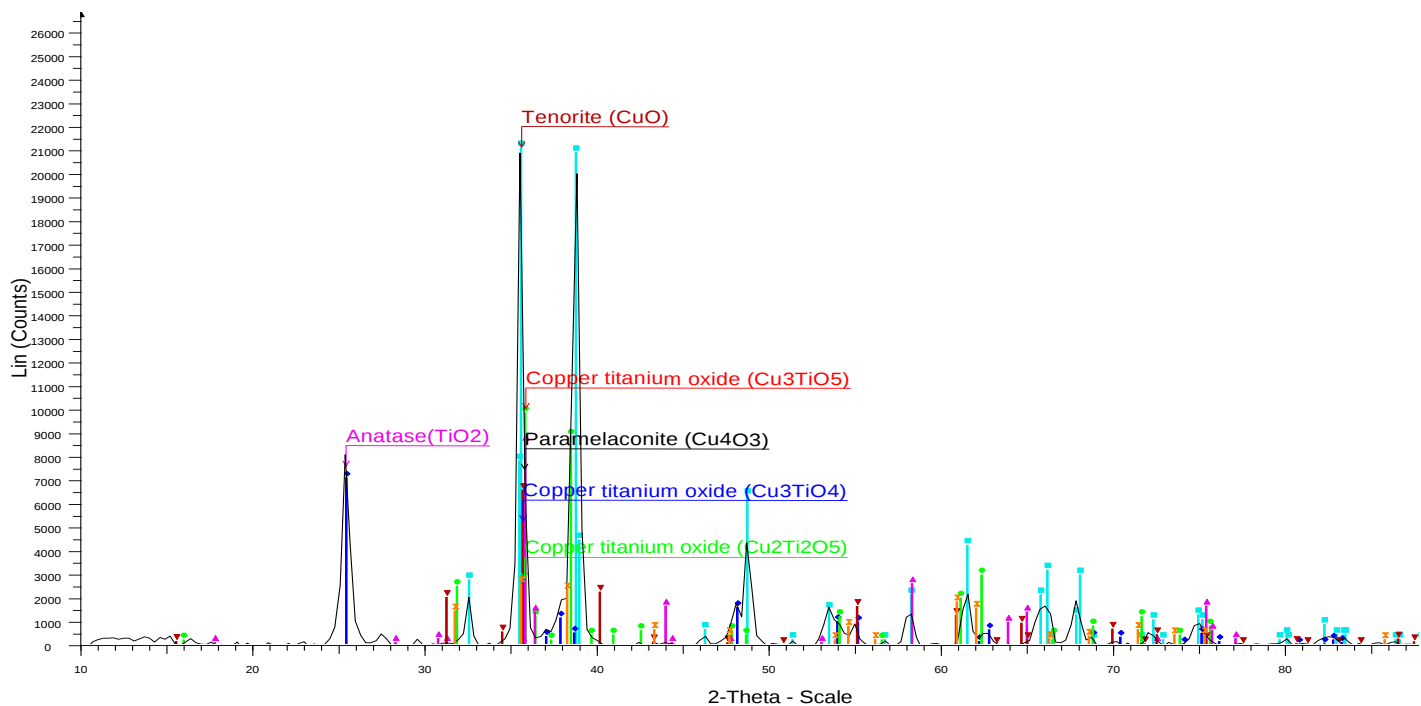
01-089-4921 (C) - Anatase, syn - TiO2 - Y: 25.49 % - d x by: 1. - WL: 1.5406 - Tetragonal - a 3.77700 - b 3.77700 - c 9.50100 - alpha 90.000 - beta 90.000 - gamma 90.000 - Body-centered - I41/amd (141) - 4 - 135.539 - I/c PDF 5 - F18=1

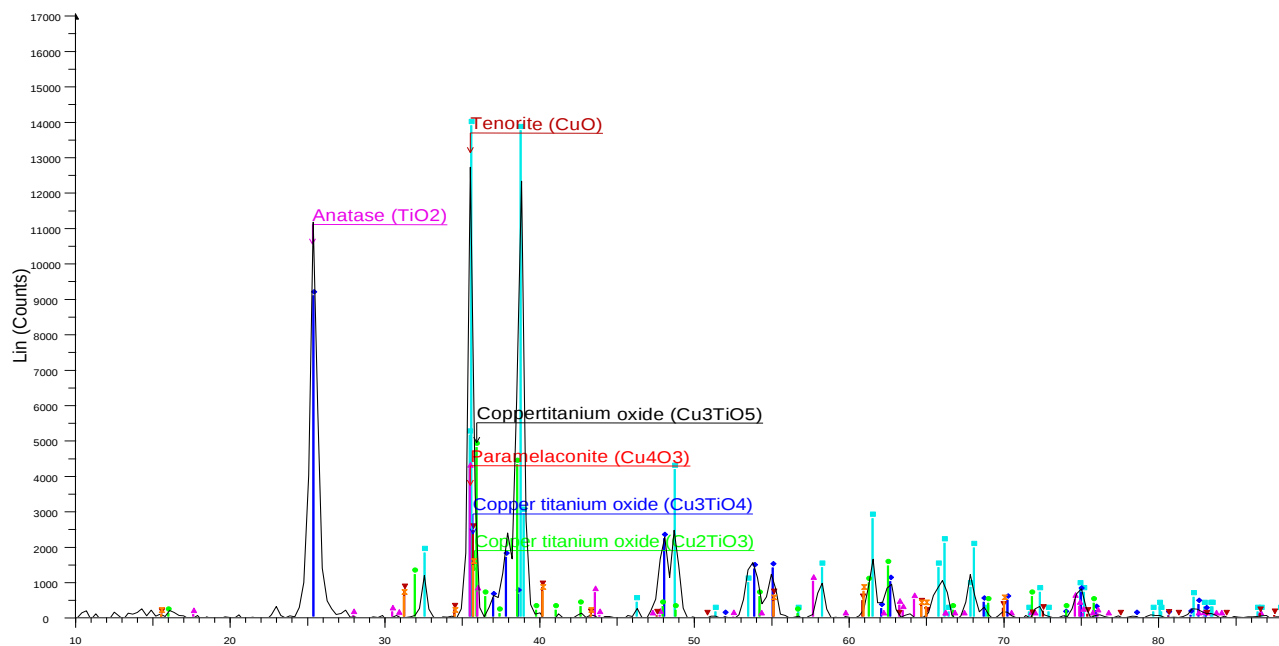
01-075-1750 (D) - Rutile - TiO2 - Y: 6.97 % - d x by: 1. - WL: 1.5406 - Tetragonal - a 4.59370 - b 4.59370 - c 2.95870 - alpha 90.000 - beta 90.000 - gamma 90.000 - Body-centered - I41/amd (141) - 4 - 135.539 - I/c PDF 5 - F18=1

00-049-1830 (C) - Paramelaconite, syn - Cu4O3 - Y: 20.31 % - d x by: 1. - WL: 1.5406 - Tetragonal - a 5.82000 - b 5.82000 - c 9.88000 - alpha 90.000 - beta 90.000 - gamma 90.000 - Body-centered - I41/amd (141) - 4 - 334.659 - F1E

00-018-0461 (D) - Copper Titanium Oxide - Cu3TiO5 - Y: 57.89 % - d x by: 0.9792 - WL: 1.5406 - Tetragonal - a 8.10000 - b 8.10000 - c 6.60000 - alpha 90.000 - beta 90.000 - gamma 90.000 - 433.026 - F22= 1(0.1760,165)

01-083-1284 (C) - Copper Titanium Oxide - Cu3TiO4 - Y: 28.42 % - d x by: 1. - WL: 1.5406 - Rhombo.H.axes - a 3.040





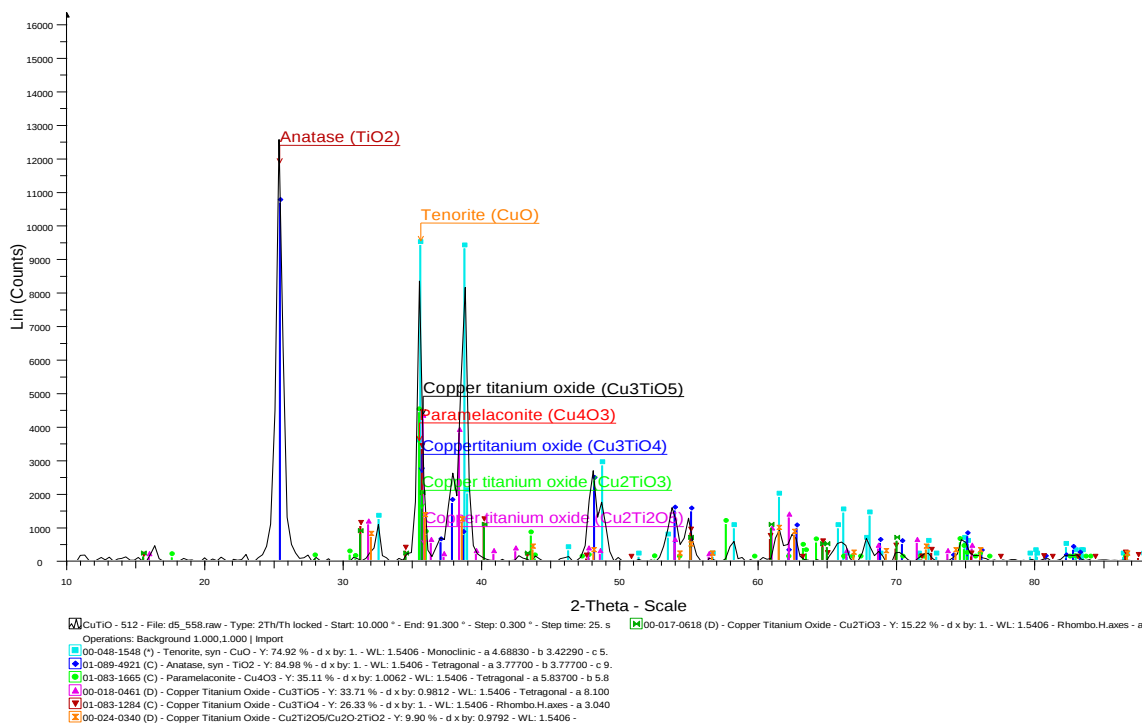
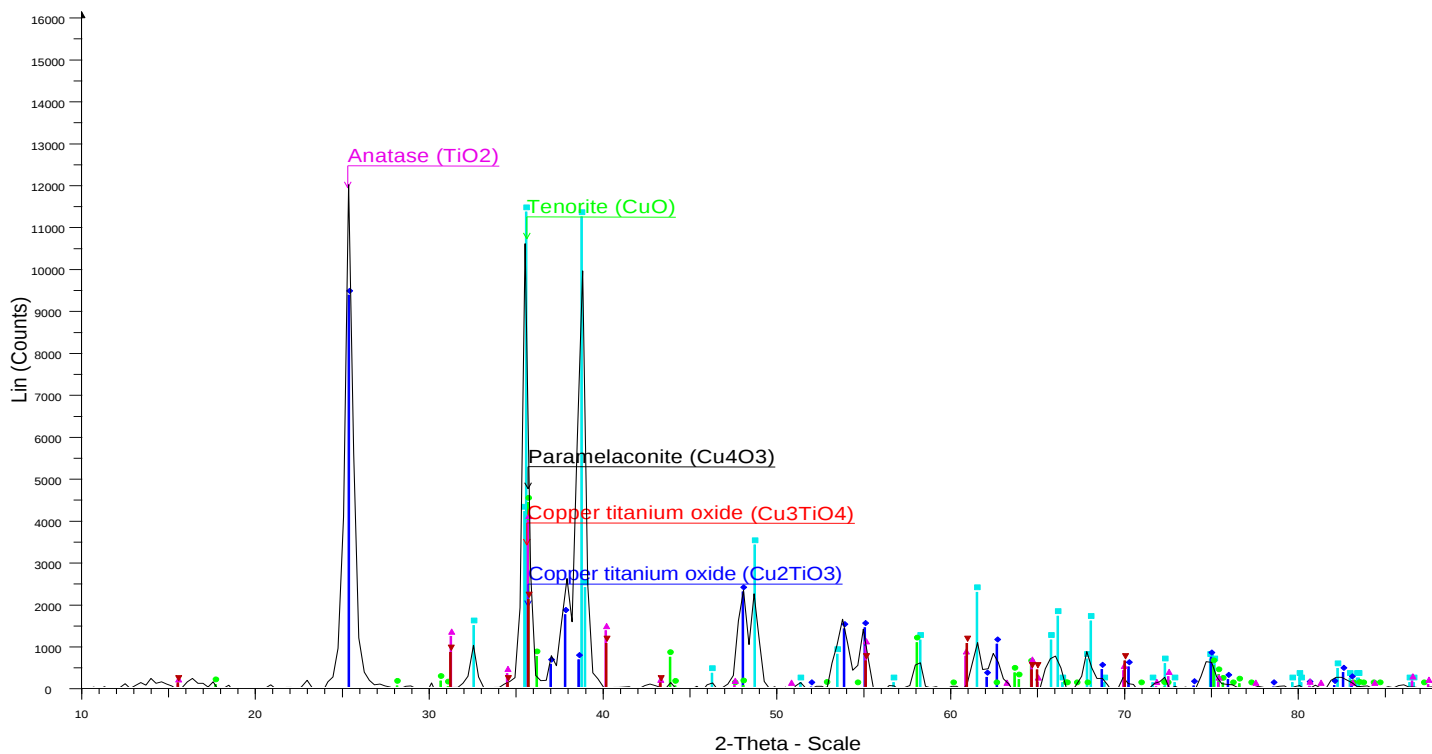
2-Theta - Scale

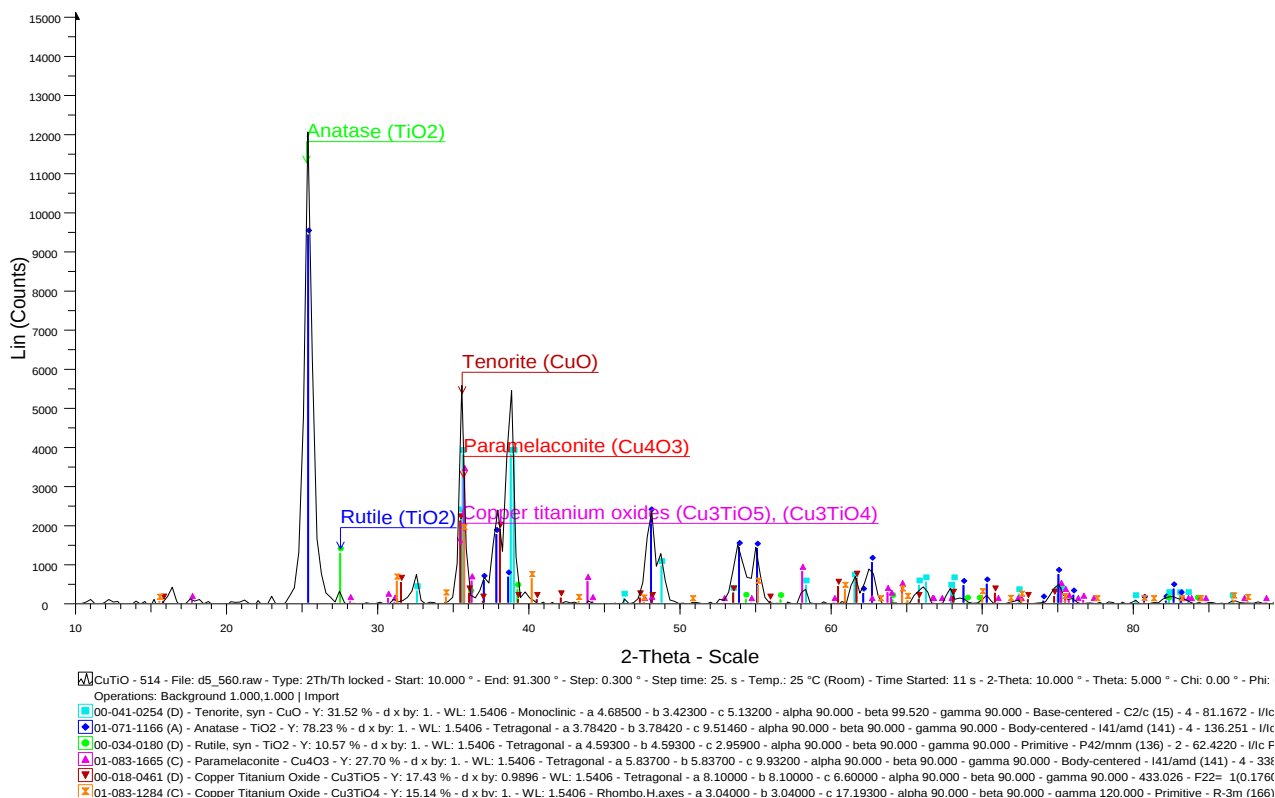
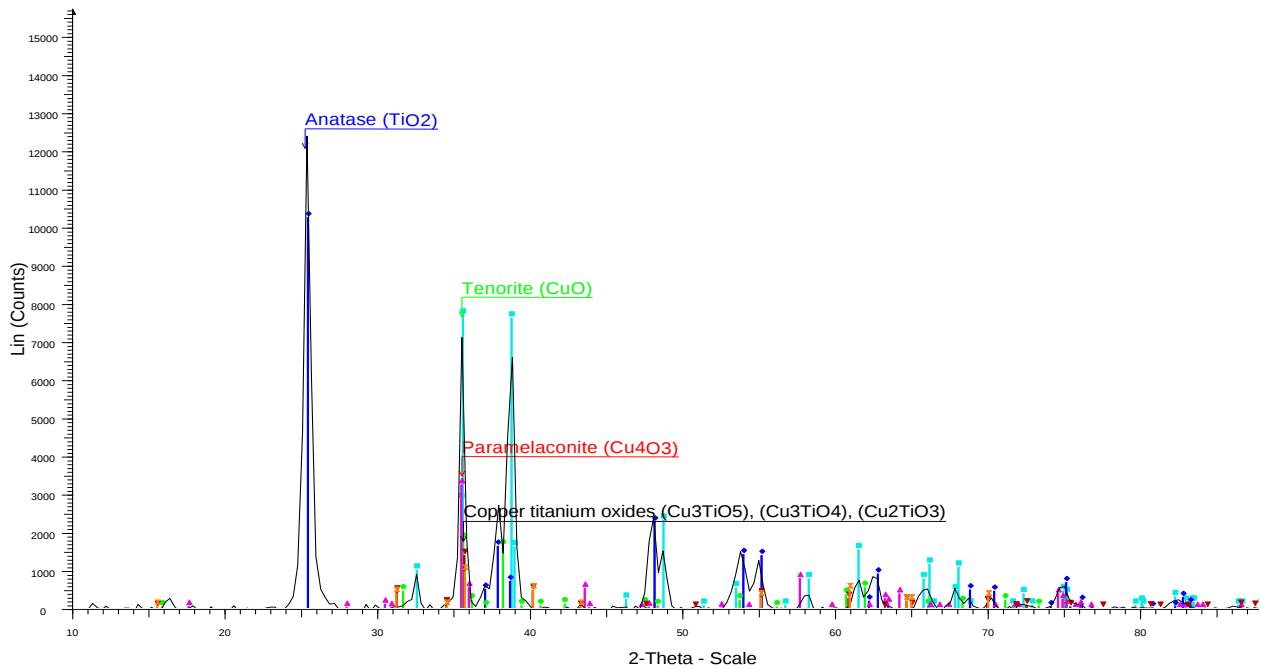
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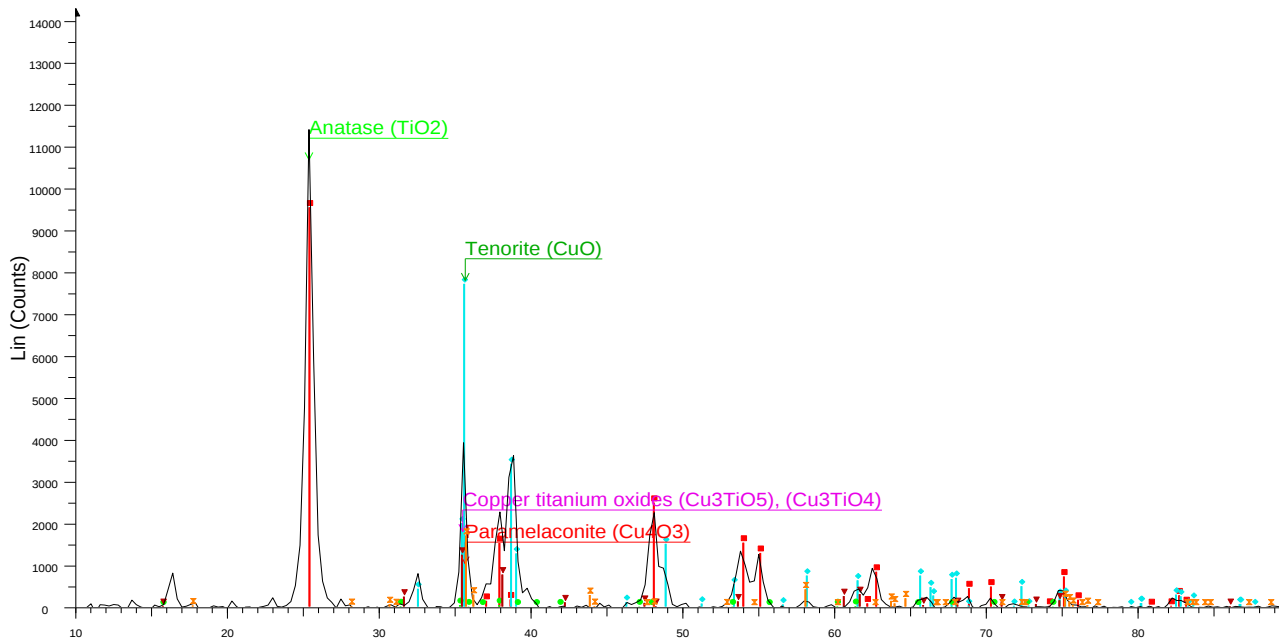
@Unit 1510 - File: d5_556.raw - Type: THzTf locked - Start: 10.000° - End: 91.300° - Step: 0.300° - Time: 25.5 - Temp.: 25° C (Room) - Time Started: 11.5 - 2-Theta: 10.000° - Theta: 5.000° - Chi: 0.00° - Phi: 0.00° - X: 0.0 mm - Y: 0.0 mm - Z: 0.0 mm
Operators: Background: d5_556.000.1.000 Import

#00-048-1548 (T) - Tenonite, spin - CuO - Y: 10.495° d - x by 1. - WL: 1.5406 - Monoclinic - C4_6B830 - B: 3.42290 - C: 5.13190 - alpha 90.000 - beta 90.000 - gamma 90.000 - Base-centered - C2/c (15) - 4: 81.2237 - FPO: 666 (0.148,31)
#01-078-2486 (C) - Anatase, spin - TiO2 - Y: 71.53° d - x by 1. - WL: 1.5406 - Tetragonal - C: 7.84540 - B: 3.78450 - C: 9.51430 - alpha 90.000 - beta 90.000 - gamma 90.000 - Body-centered - I41m (41d) - F22 - 4: 136.268 - V/c PDF 5 - F20=1
#01-088-0461 (D) - Copper Titanium Oxide - CuTiO3 - Y: 37.73° d - x by 1. - WL: 1.5406 - Tetragonal - a: 8.10000 - b: 8.10000 - c: 6.60000 - alpha 90.000 - beta 90.000 - gamma 90.000 - 432.026 - F22 - 10.1760,165)
#01-083-1665 (C) - Paramelanite, Cu2O3 - Y: 33.01° d - x by 1. 1.0062 - WL: 1.5406 - Tetragonal - a: 8.87300 - b: 8.87300 - c: 9.93520 - alpha 90.000 - beta 90.000 - gamma 90.000 - Body-centered - I41m (41d) - F41 - 4: 338.389 - V/c PDF 5
#01-1294 (C) - Copper Titanium Oxide - CuTiO4 - Y: 19.26° d - x by 1. - WL: 1.5406 - Rhombo-Hexagonal - a: 17.19500 - alpha 90.000 - beta 90.000 - gamma 120.000 - Primitive - R-3m (162) - 1: 137.603 - V/c F
#01-017-0618 (D) - Copper Titanium Oxide - Cu2TiO3 - Y: 11.46° d - x by 1. - WL: 1.5406 - Rhombo-Hexagonal - a: 3.03700 - b: 3.03700 - c: 17.19800 - alpha 90.000 - beta 90.000 - gamma 120.000 - Primitive - R (0) - 137.372 - F11: 220.0(2)

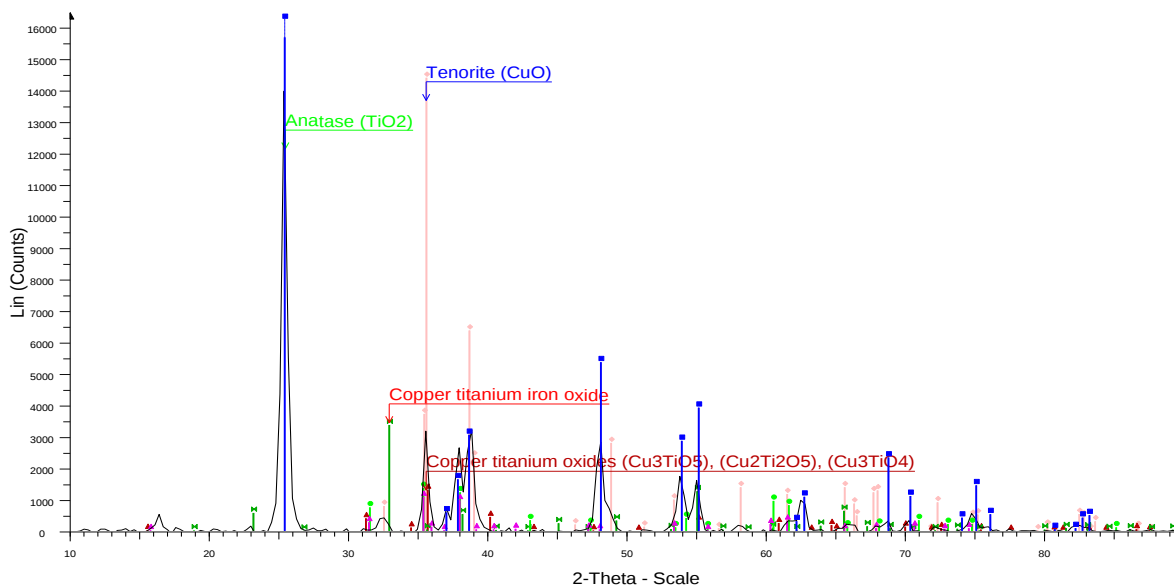
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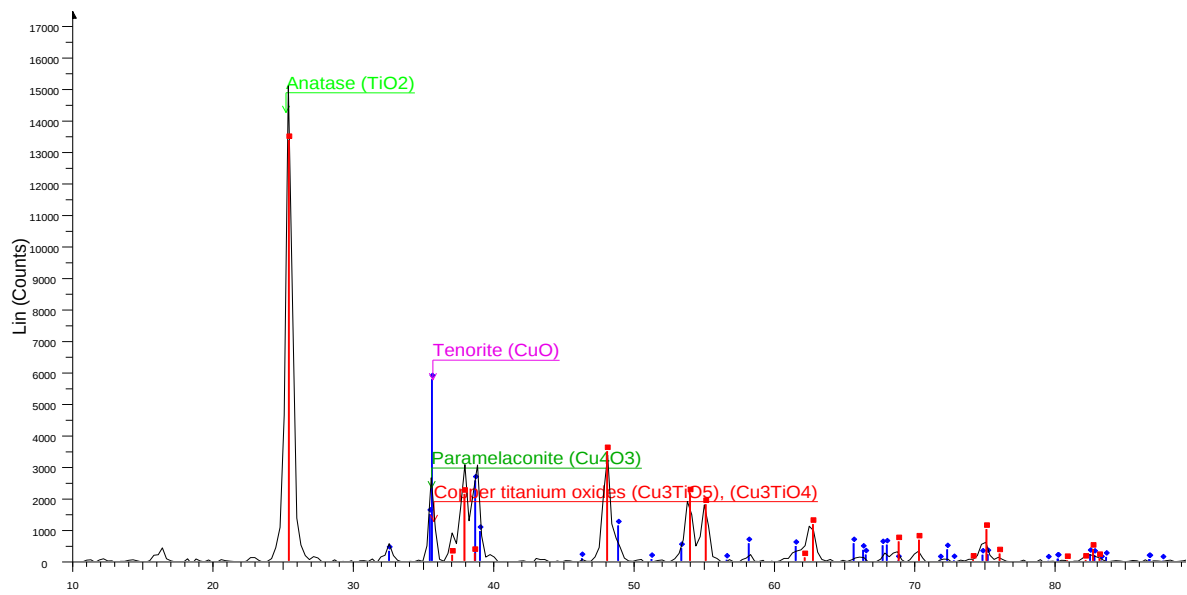




CuTiO - 515 - File: d5_561.raw - Type: 2Th/Th locked - Start: 10.000 ° - End: 91.300 ° - Step: 0.300 ° - Step time: 25. s - Temp.: 25 °C (Room) - Time Started: 11 s - 2-Theta: 10.000 ° - Theta: 5.000 ° - Chi: 0.00 ° - Phi:
 Operations: Background 1.000,1.000 | Import
 01-086-1157 (A) - Anatase, syn - TiO_2 - Y: 83.71 % - d x by: 1. - WL: 1.5406 - Tetragonal - a 3.78300 - b 3.78300 - c 9.49700 - alpha 90.000 - beta 90.000 - gamma 90.000 - Body-centered - I41/amd (141) - 4 - 135
 01-080-0076 (C) - Tenorite, syn - CuO - Y: 67.67 % - d x by: 1. - WL: 1.5406 - Monoclinic - a 4.67970 - b 3.43140 - c 5.13620 - alpha 90.000 - beta 99.262 - gamma 90.000 - Base-centered - C2/c (15) - 4 - 81.4014 - I/c
 00-018-0461 (D) - Copper Titanium Oxide - Cu_3TiO_5 - Y: 0.32 % - d x by: 0.9937 - WL: 1.5406 - Tetragonal - a 8.10000 - b 8.10000 - c 6.60000 - alpha 90.000 - beta 90.000 - gamma 90.000 - 433.026 - F22= 1(0.1760,
 00-027-0199 (I) - Copper Titanium Oxide - $\text{Cu}_3\text{TiO}_4/\text{Cu}_2\text{O}-\text{CuO}-\text{TiO}_2$ - Y: 10.84 % - d x by: 0.9896 - WL: 1.5406 - Hexagonal - a 3.03540 - b 3.03540 - c 11.44000 - alpha 90.000 - beta 90.000 - gamma 120.000 - Primiti
 01-083-1665 (C) - Paramelaconite - Cu_4O_3 - Y: 14.87 % - d x by: 1. - WL: 1.5406 - Tetragonal - a 5.83700 - b 5.83700 - c 9.93200 - alpha 90.000 - beta 90.000 - gamma 90.000 - Body-centered - I41/amd (141) - 4 - 33



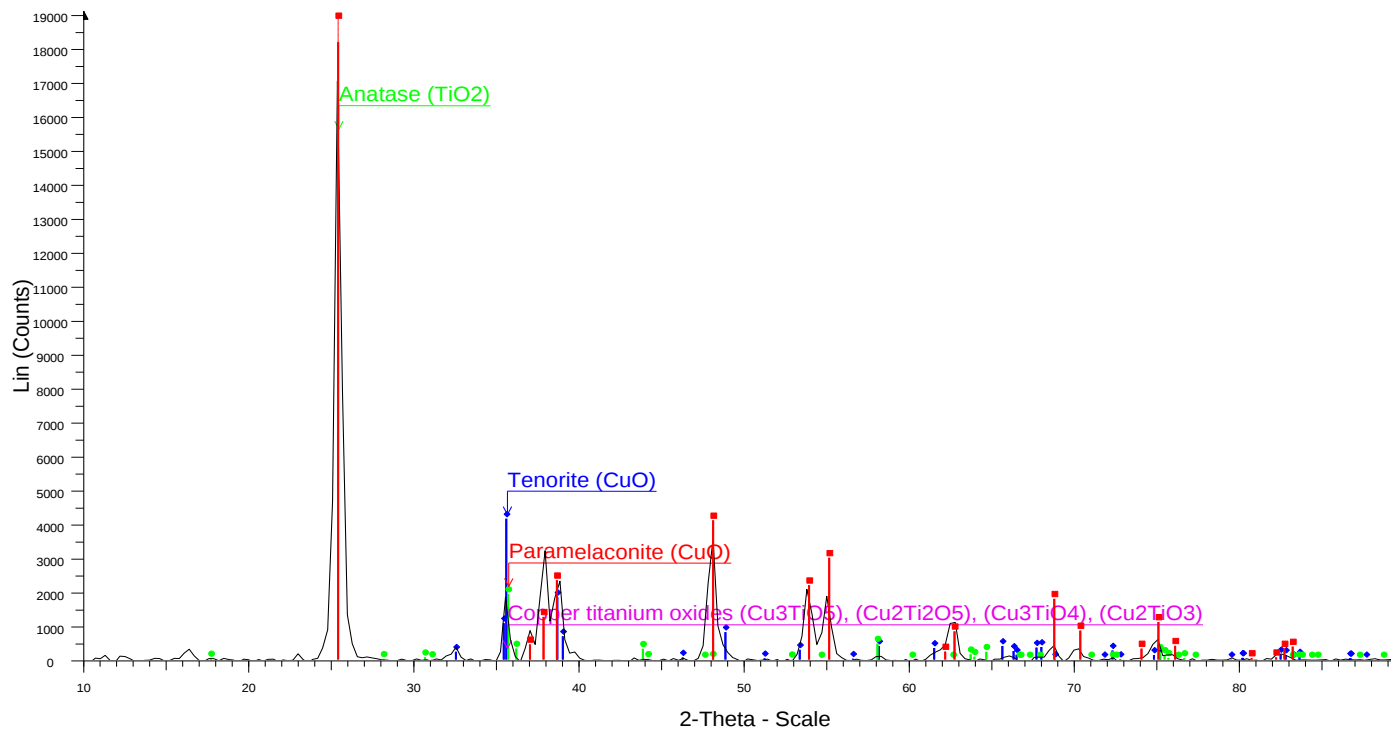
CuTiO - 516 - File: d5_562.raw - Type: 2Th/Th locked - Start: 10.000 ° - End: 91.300 ° - Step: 0.300 ° - Step time: 25. s - Temp.: 25 °C (Room) - Time Started: 11 s - 2-Theta: 10.000 ° - Theta: 5.000 ° - Chi: 0.00 ° - Phi:
 Operations: Background 1.000,1.000 | Import
 01-083-2243 (A) - Anatase, syn - TiO_2 - Y: 198.55 % - d x by: 1. - WL: 1.5406 - Tetragonal - a 3.78000 - b 3.78000 - c 9.51000 - alpha 90.000 - beta 90.000 - gamma 90.000 - Body-centered - I41/amd (141) - 4 - 135.88
 01-080-0076 (C) - Tenorite, syn - CuO - Y: 103.05 % - d x by: 1. - WL: 1.5406 - Monoclinic - a 4.67970 - b 3.43140 - c 5.13620 - alpha 90.000 - beta 99.262 - gamma 90.000 - Base-centered - C2/c (15) - 4 - 81.4014 - I/c
 01-083-1284 (C) - Copper Titanium Oxide - Cu_3TiO_4 - Y: 9.35 % - d x by: 1. - WL: 1.5406 - Rhombo H axes - a 3.04000 - b 3.04000 - c 17.19300 - alpha 90.000 - beta 90.000 - gamma 120.000 - Primitive - R-3m (166) -
 01-078-1627 (C) - Copper Titanium Iron Oxide - $\text{Cu}_0.66\text{Ti}_0.66\text{Fe}_0.66\text{O}_3$ - Y: 24.08 % - d x by: 1. - WL: 1.5406 - Cubic - a 9.43120 - b 9.43120 - c 9.43120 - alpha 90.000 - beta 90.000 - gamma 90.000 - Body-centered -
 00-024-0340 (D) - Copper Titanium Oxide - $\text{Cu}_2\text{Ti}_2\text{O}_5/\text{Cu}_2\text{O}-2\text{TiO}_2$ - Y: 9.73 % - d x by: 0.9937 - WL: 1.5406 -
 00-018-0461 (D) - Copper Titanium Oxide - Cu_3TiO_5 - Y: 7.73 % - d x by: 0.9917 - WL: 1.5406 - Tetragonal - a 8.10000 - b 8.10000 - c 6.60000 - alpha 90.000 - beta 90.000 - gamma 90.000 - 433.026 - F22= 1(0.1760,



■ CuTiO - 517 - File: d5_563.raw - Type: 2Th/Th locked - Start: 10.000 ° - End: 91.300 ° - Step: 0.300 ° - Step time: 25. s - Temp.: 25 °C (Room) - Time Started: 12 s - 2-Theta: 10.000 ° - Theta: 5.000 ° - Chi: 0.00 ° - Phi: 0.00 ° - Operations: Background 1.000,1.000 | Import

■ 01-086-1157 (A) - Anatase, syn - TiO₂ - Y: 88.60 % - d x by: 1. - WL: 1.5406 - Tetragonal - a 3.78300 - b 3.78300 - c 9.49700 - alpha 90.000 - beta 90.000 - gamma 90.000 - Body-centered - I41/amd (141) - 4 - 135

■ 01-080-0076 (C) - Tenorite, syn - CuO - Y: 38.23 % - d x by: 1. - WL: 1.5406 - Monoclinic - a 4.67970 - b 3.43140 - c 5.13620 - alpha 90.000 - beta 99.262 - gamma 90.000 - Base-centered - C2/c (15) - 4 - 81.4014 - I/c

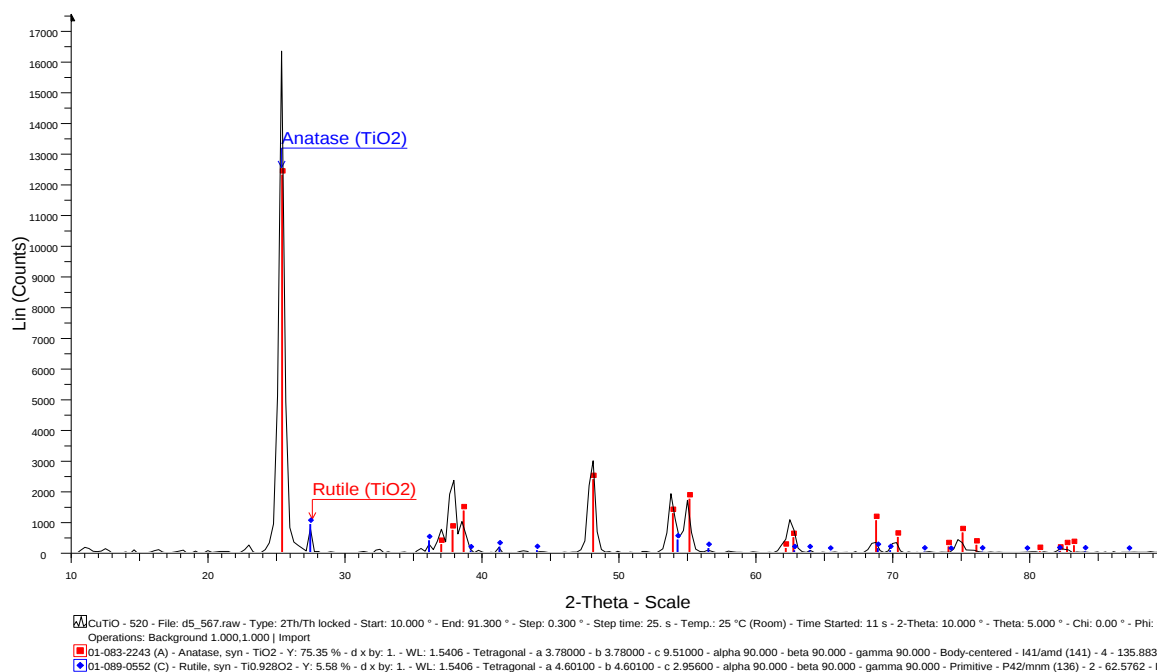
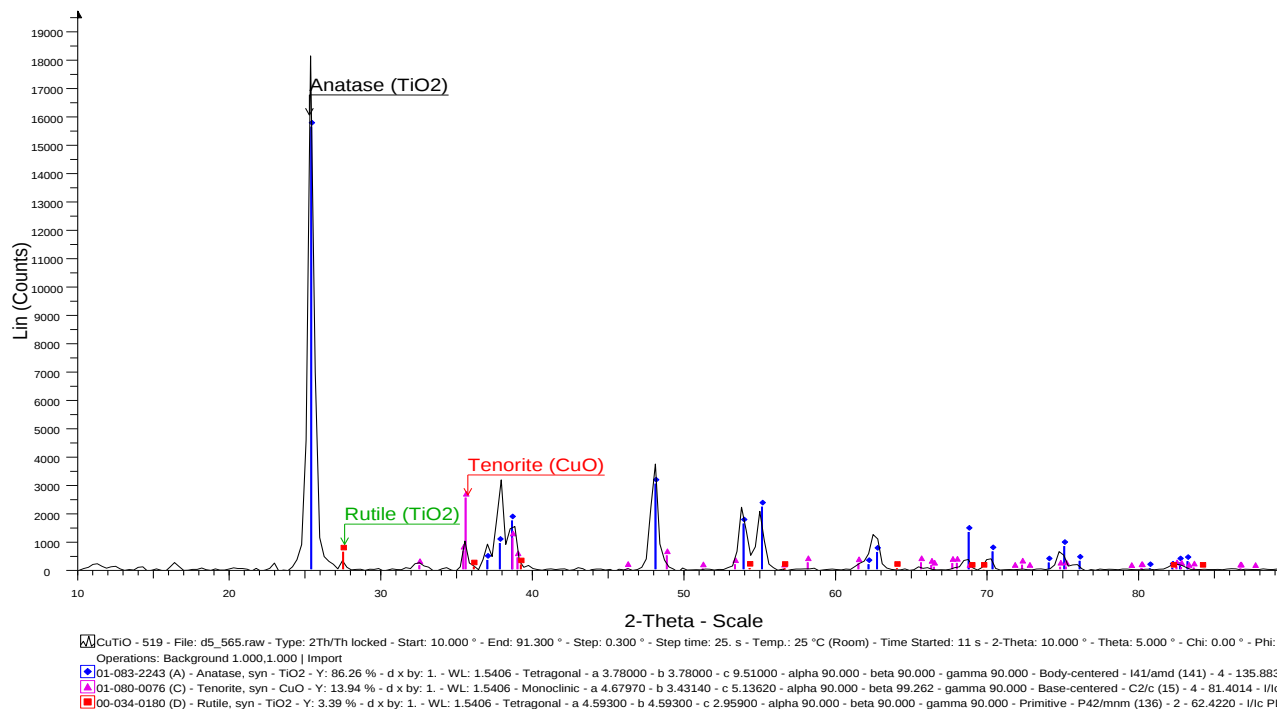


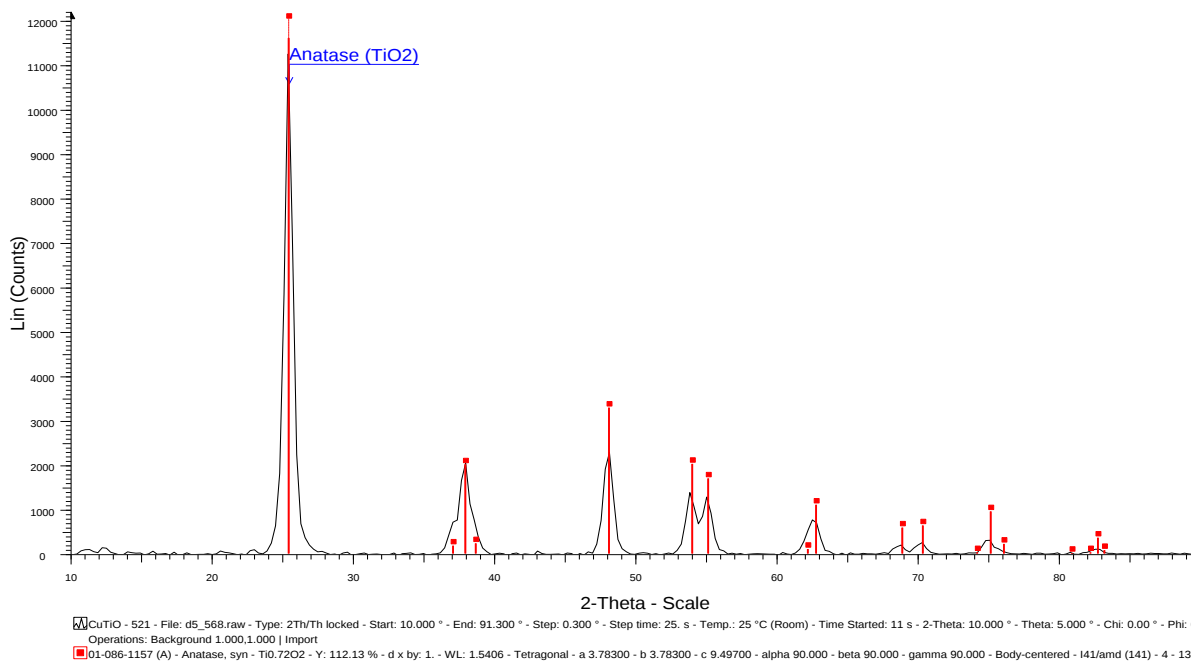
■ CuTiO - 518 - File: d5_564.raw - Type: 2Th/Th locked - Start: 10.000 ° - End: 91.300 ° - Step: 0.300 ° - Step time: 25. s - Temp.: 25 °C (Room) - Time Started: 12 s - 2-Theta: 10.000 ° - Theta: 5.000 ° - Chi: 0.00 ° - Phi: 0.00 ° - Operations: Background 1.000,1.000 | Import

■ 01-083-2243 (A) - Anatase, syn - TiO₂ - Y: 124.69 % - d x by: 1. - WL: 1.5406 - Tetragonal - a 3.78000 - b 3.78000 - c 9.51000 - alpha 90.000 - beta 90.000 - gamma 90.000 - Body-centered - I41/amd (141) - 4 - 135.88

■ 01-080-0076 (C) - Tenorite, syn - CuO - Y: 24.35 % - d x by: 1. - WL: 1.5406 - Monoclinic - a 4.67970 - b 3.43140 - c 5.13620 - alpha 90.000 - beta 99.262 - gamma 90.000 - Base-centered - C2/c (15) - 4 - 81.4014 - I/c

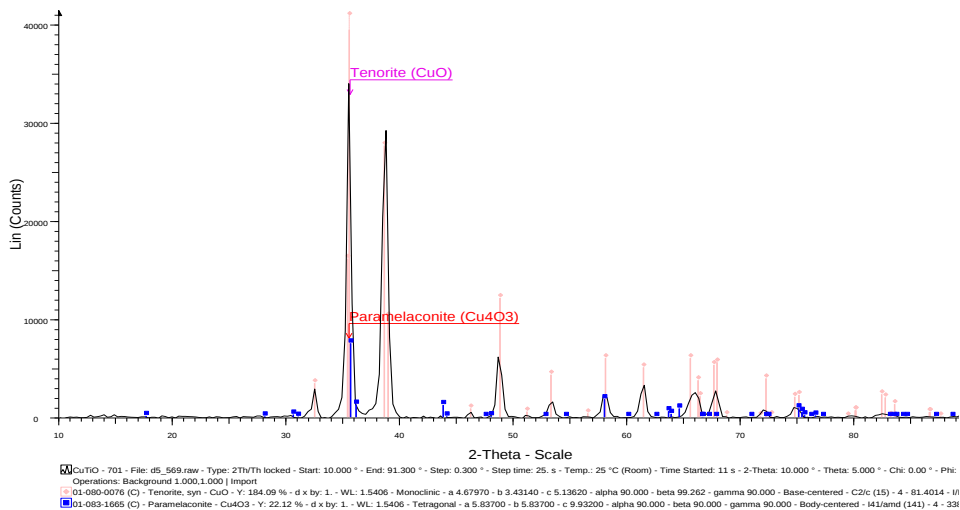
■ 01-083-1665 (C) - Paramelaconite - Cu₄O₃ - Y: 11.32 % - d x by: 1. - WL: 1.5406 - Tetragonal - a 5.83700 - b 5.83700 - c 9.93200 - alpha 90.000 - beta 90.000 - gamma 90.000 - Body-centered - I41/amd (141) - 4 - 331

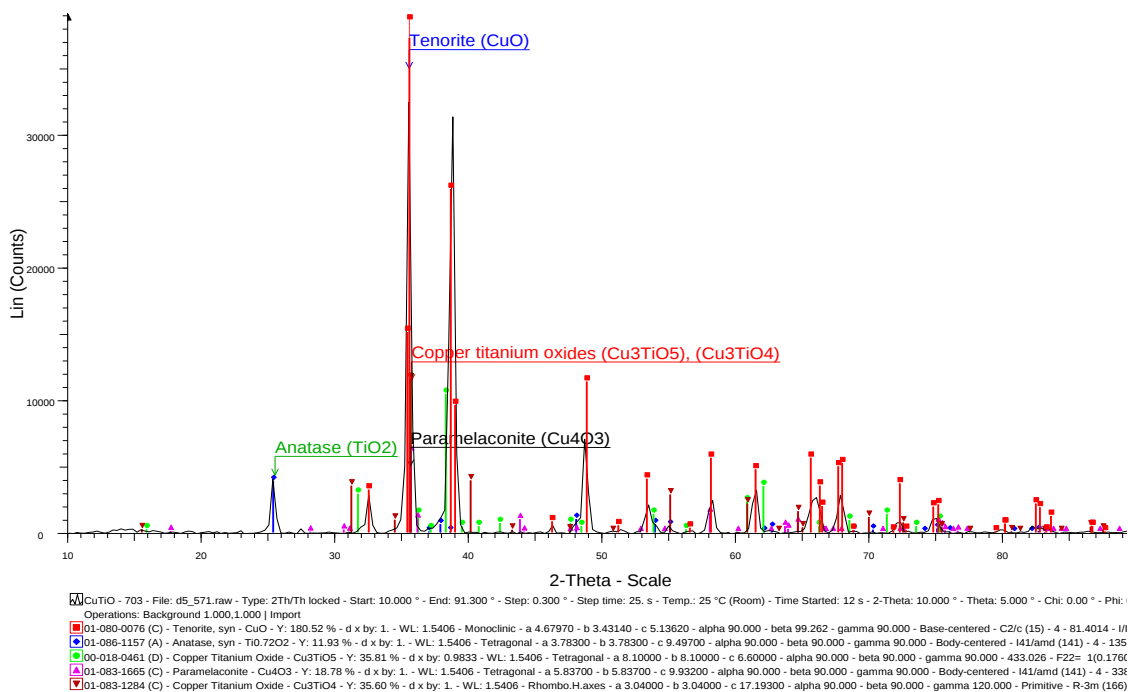
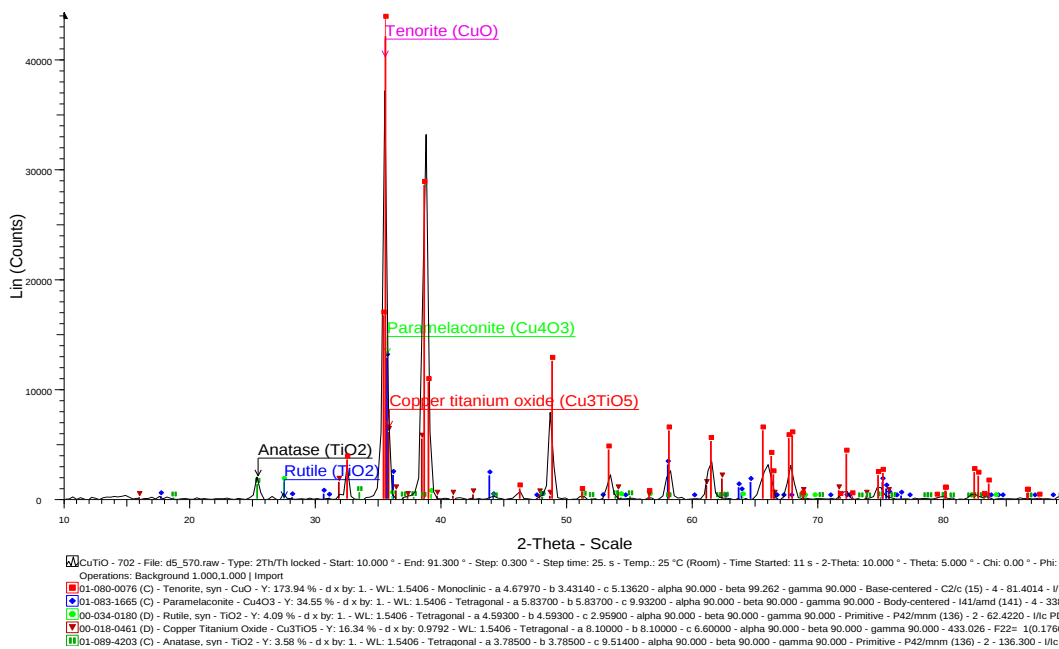


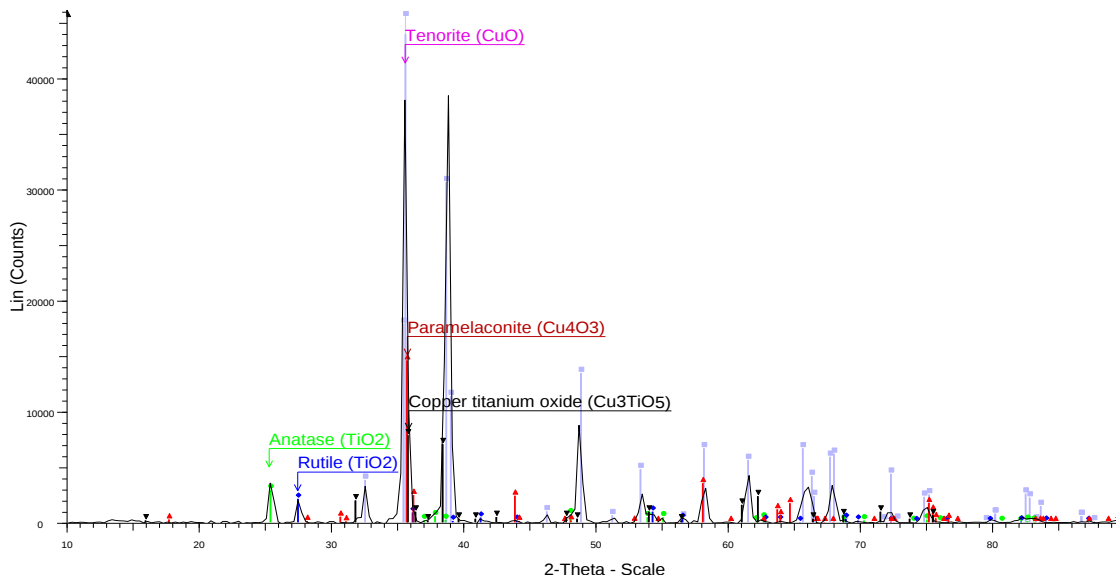


APPENDIX E

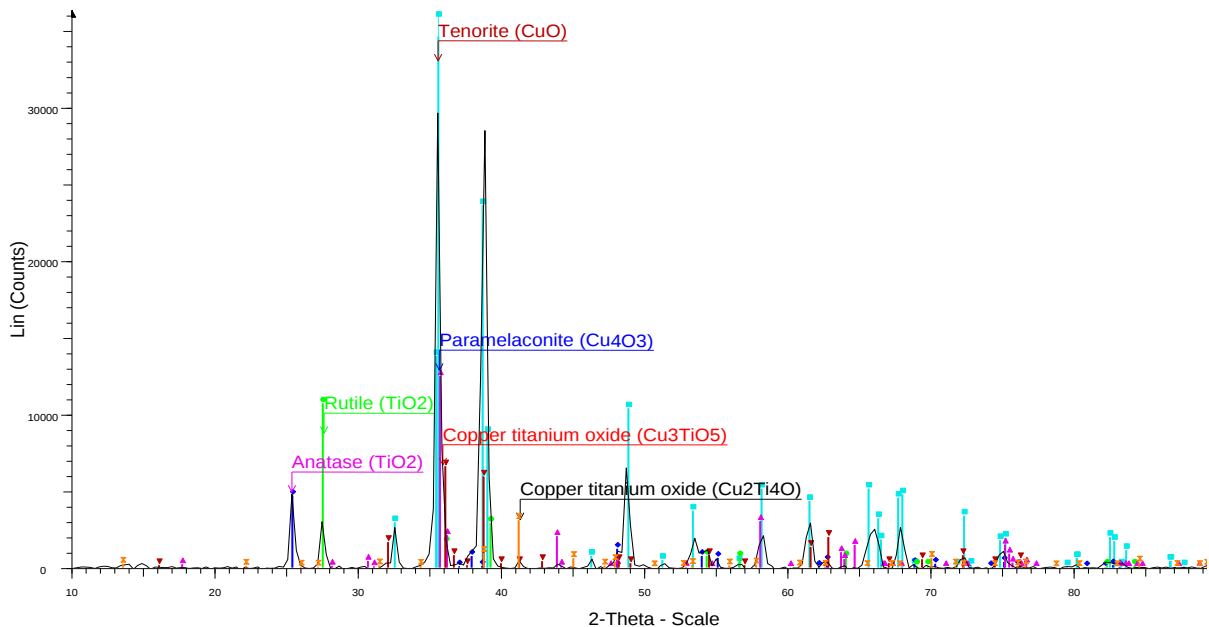
Patterns of Resin-gel precursor powders calcined at 700 °C



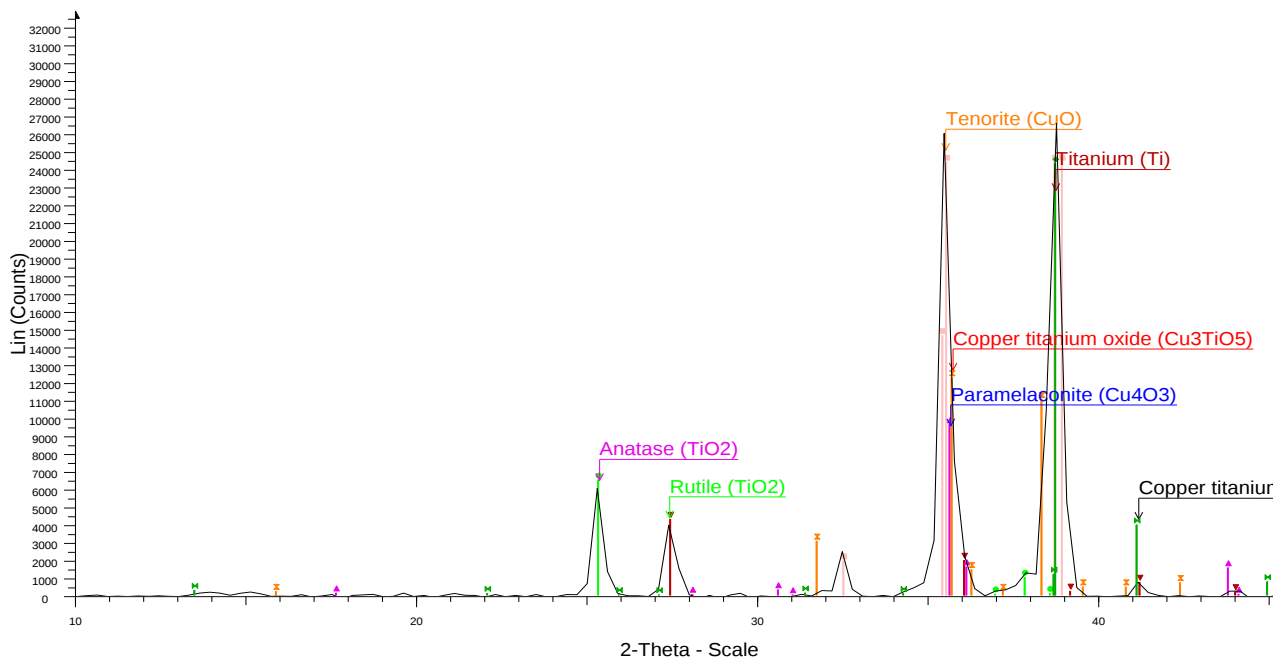




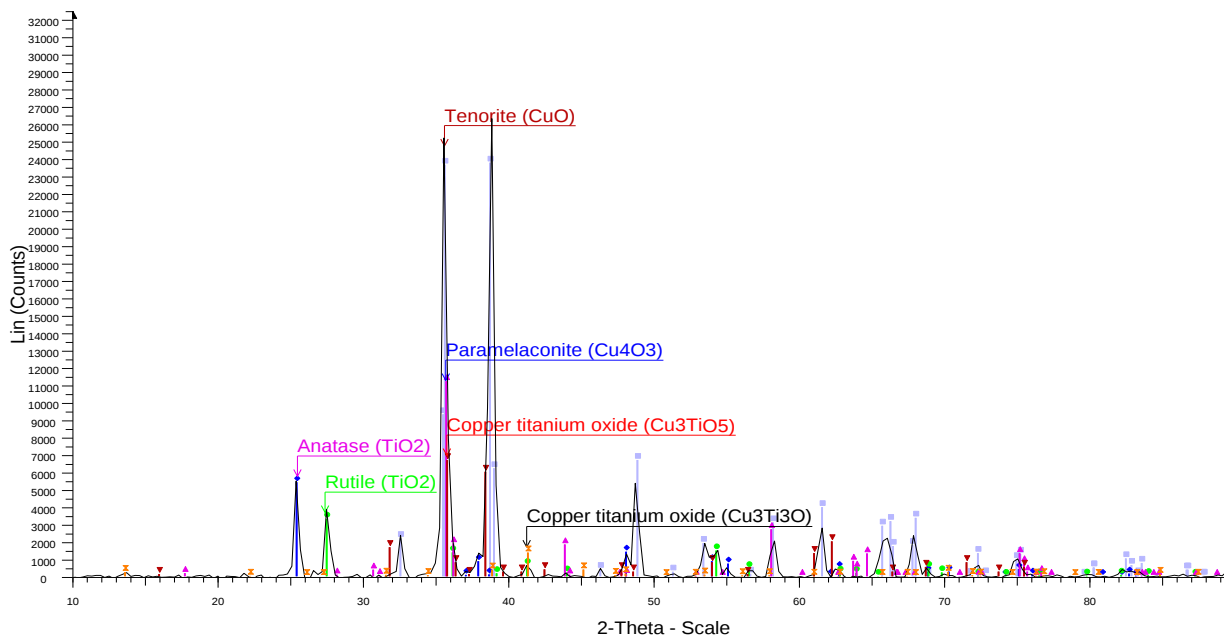
CuTiO - 704 - File: d5_572.raw - Type: 2Th/Th locked - Start: 10.000 ° - End: 91.300 ° - Step: 0.300 ° - Step time: 25. s - Temp.: 25 °C (Room) - Time Started: 12 s - 2-Theta: 10.000 ° - Theta: 5.000 ° - Chi: 0.00 ° - Phi: 0.00 °
Operations: Background 1.000,1.000 | Import
01-080-0076 (C) - Tenorite, syn - CuO - Y: 180.24 % - d x by: 1. - WL: 1.5406 - Monoclinic - a 4.67970 - b 3.43140 - c 5.13620 - alpha 90.000 - beta 99.262 - gamma 90.000 - Base-centered - C2/c (15) - 4 - 81.4014 - I/I
01-071-1166 (A) - Anatase - TiO2 - Y: 7.59 % - d x by: 1. - WL: 1.5406 - Tetragonal - a 3.78420 - b 3.78420 - c 9.51460 - alpha 90.000 - beta 90.000 - gamma 90.000 - Body-centered - I41/amd (141) - 4 - 136.251 - I/I
01-089-0552 (C) - Rutile, syn - TiO2 - Y: 5.46 % - d x by: 1. - WL: 1.5406 - Tetragonal - a 4.60100 - b 4.60100 - c 2.95600 - alpha 90.000 - beta 90.000 - gamma 90.000 - Primitive - P42/mmm (136) - 2 - 62.5762 - I/I
01-083-1665 (C) - Paramelaconite - Cu4O3 - Y: 37.95 % - d x by: 1. - WL: 1.5406 - Tetragonal - a 5.83700 - b 5.83700 - c 9.93200 - alpha 90.000 - beta 90.000 - gamma 90.000 - Body-centered - I41/amd (141) - 4 - 338
00-018-0461 (D) - Copper Titanium Oxide - Cu3TiO5 - Y: 20.38 % - d x by: 0.9812 - WL: 1.5406 - Tetragonal - a 8.10000 - b 8.10000 - c 6.60000 - alpha 90.000 - beta 90.000 - gamma 90.000 - 433.026 - F22= 1(0.176)



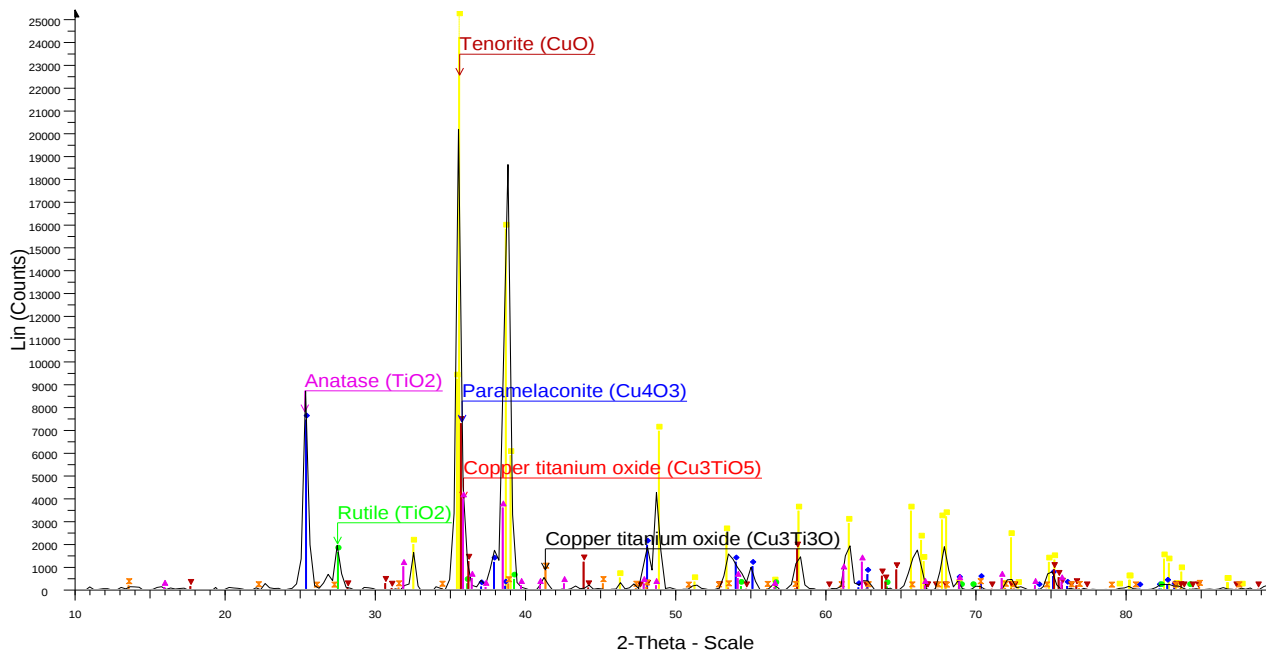
CuTiO - 705 - File: d5_573.raw - Type: 2Th/Th locked - Start: 10.000 ° - End: 91.300 ° - Step: 0.300 ° - Step time: 25. s - Temp.: 25 °C (Room) - Time Started: 12 s - 2-Theta: 10.000 ° - Theta: 5.000 ° - Chi: 0.00 ° - Phi: 0.00 °
Operations: Background 1.000,1.000 | Import
01-080-0076 (C) - Tenorite, syn - CuO - Y: 180.22 % - d x by: 1. - WL: 1.5406 - Monoclinic - a 4.67970 - b 3.43140 - c 5.13620 - alpha 90.000 - beta 99.262 - gamma 90.000 - Base-centered - C2/c (15) - 4 - 81.4014 - I/I
01-086-1157 (A) - Anatase, syn - TiO2 - Y: 15.78 % - d x by: 1. - WL: 1.5406 - Tetragonal - a 3.78300 - b 3.78300 - c 9.49700 - alpha 90.000 - beta 90.000 - gamma 90.000 - Body-centered - I41/amd (141) - 4 - 135
00-034-0180 (D) - Rutile, syn - TiO2 - Y: 36.10 % - d x by: 1. - WL: 1.5406 - Tetragonal - a 4.59300 - b 4.59300 - c 2.95900 - alpha 90.000 - beta 90.000 - gamma 90.000 - Primitive - P42/mmm (136) - 2 - 62.4220 - I/I
01-083-1665 (C) - Paramelaconite - Cu4O3 - Y: 42.11 % - d x by: 1. - WL: 1.5406 - Tetragonal - a 5.83700 - b 5.83700 - c 9.93200 - alpha 90.000 - beta 90.000 - gamma 90.000 - Body-centered - I41/amd (141) - 4 - 338
00-018-0461 (D) - Copper Titanium Oxide - Cu3TiO5 - Y: 22.24 % - d x by: 0.9729 - WL: 1.5406 - Tetragonal - a 8.10000 - b 8.10000 - c 6.60000 - alpha 90.000 - beta 90.000 - gamma 90.000 - 433.026 - F22= 1(0.176)
01-072-0444 (A) - Copper Titanium Oxide - Cu2Ti4O - Y: 10.41 % - d x by: 0.9958 - WL: 1.5406 - Cubic - a 11.43530 - b 11.43530 - c 11.43530 - alpha 90.000 - beta 90.000 - gamma 90.000 - Face-centered - Fd-3m (2)



☒ CuTiO - 706 - File: d5_574.raw - Type: 2Th/Th locked - Start: 10.000 ° - End: 46.000 ° - Step: 0.300 ° - Ste ☒ 01-072-0444 (A) - Copper Titanium Oxide - Cu₂Ti₄O - Y: 15.00 % - d x by: 0.9958 - WL: 1.5406 - Cubic -
 Operations: Background 1.000,1.000 | Import
☒ 00-041-0254 (D) - Tenorite, syn - CuO - Y: 91.94 % - d x by: 1. - WL: 1.5406 - Monoclinic - a 4.68500 - b 3
☒ 01-089-4913 (C) - Titanium - Ti - Y: 91.60 % - d x by: 1. - WL: 1.5406 - Cubic - a 3.28300 - b 3.28300 - c 3
☒ 01-086-1157 (A) - Anatase, syn - TiO₂O₂ - Y: 24.51 % - d x by: 1. - WL: 1.5406 - Tetragonal - a 3.78300
☒ 01-073-1782 (C) - Rutile, syn - TiO_{1.95} - Y: 16.22 % - d x by: 1. - WL: 1.5406 - Tetragonal - a 4.59300 - b
☒ 01-083-1665 (C) - Paramelaconite - Cu₄O₃ - Y: 36.29 % - d x by: 1. - WL: 1.5406 - Tetragonal - a 5.83700



☒ CuTiO - 706 - File: d5_575.raw - Type: 2Th/Th locked - Start: 10.000 ° - End: 91.300 ° - Step: 0.300 ° - Step time: 25. s - Temp.: 25 °C (Room) - Time Started: 5 s - 2-Theta: 10.000 ° - Theta: 5.000 ° - Chi: 0.00 ° - Phi: 0
 Operations: Background 1.000,1.000 | Import
☒ 01-089-2529 (C) - Tenorite - synthetic - CuO - Y: 90.43 % - d x by: 1. - WL: 1.5406 - Monoclinic - a 4.68320 - b 3.42880 - c 5.12970 - alpha 90.000 - beta 99.309 - gamma 90.000 - Base-centered - C2/c (15) - 4 - 81.286
☒ 01-086-1157 (A) - Anatase, syn - TiO₂O₂ - Y: 20.57 % - d x by: 1. - WL: 1.5406 - Tetragonal - a 3.78300 - b 3.78300 - c 9.49700 - alpha 90.000 - beta 90.000 - gamma 90.000 - Body-centered - I41/amd (141) - 4 - 135
☒ 01-089-0552 (C) - Rutile, syn - TiO₂O₂ - Y: 12.54 % - d x by: 1. - WL: 1.5406 - Tetragonal - a 4.60100 - b 4.60100 - c 2.95600 - alpha 90.000 - beta 90.000 - gamma 90.000 - Primitive - P42/mnm (136) - 2 - 62.5762
☒ 01-083-1665 (C) - Paramelaconite - Cu₄O₃ - Y: 42.65 % - d x by: 1. - WL: 1.5406 - Tetragonal - a 5.83700 - b 5.83700 - c 9.93200 - alpha 90.000 - beta 90.000 - gamma 90.000 - Body-centered - I41/amd (141) - 4 - 338
☒ 00-018-0461 (D) - Copper Titanium Oxide - Cu₃TiO₅ - Y: 25.38 % - d x by: 0.9812 - WL: 1.5406 - Tetragonal - a 8.10000 - b 8.10000 - c 6.60000 - alpha 90.000 - beta 90.000 - gamma 90.000 - 433.026 - F22= 1(0.176)
☒ 01-075-0400 (C) - Copper Titanium Oxide - Cu₃Ti₃O - Y: 5.18 % - d x by: 1.0104 - WL: 1.5406 - Cubic - a 11.24000 - b 11.24000 - c 11.24000 - alpha 90.000 - beta 90.000 - gamma 90.000 - Face-centered - Fd-3m (22)



CuTiO - 707 - File: d5_576.raw - Type: 2Th/Th locked - Start: 10.000 ° - End: 91.300 ° - Step: 0.300 ° - Step time: 25. s - Temp.: 25 °C (Room) - Time Started: 12 s - 2-Theta: 10.000 ° - Theta: 5.000 ° - Chi: 0.00 ° - Phi: 0.00 ° - Operations: Background 1.000,1.000 | Import

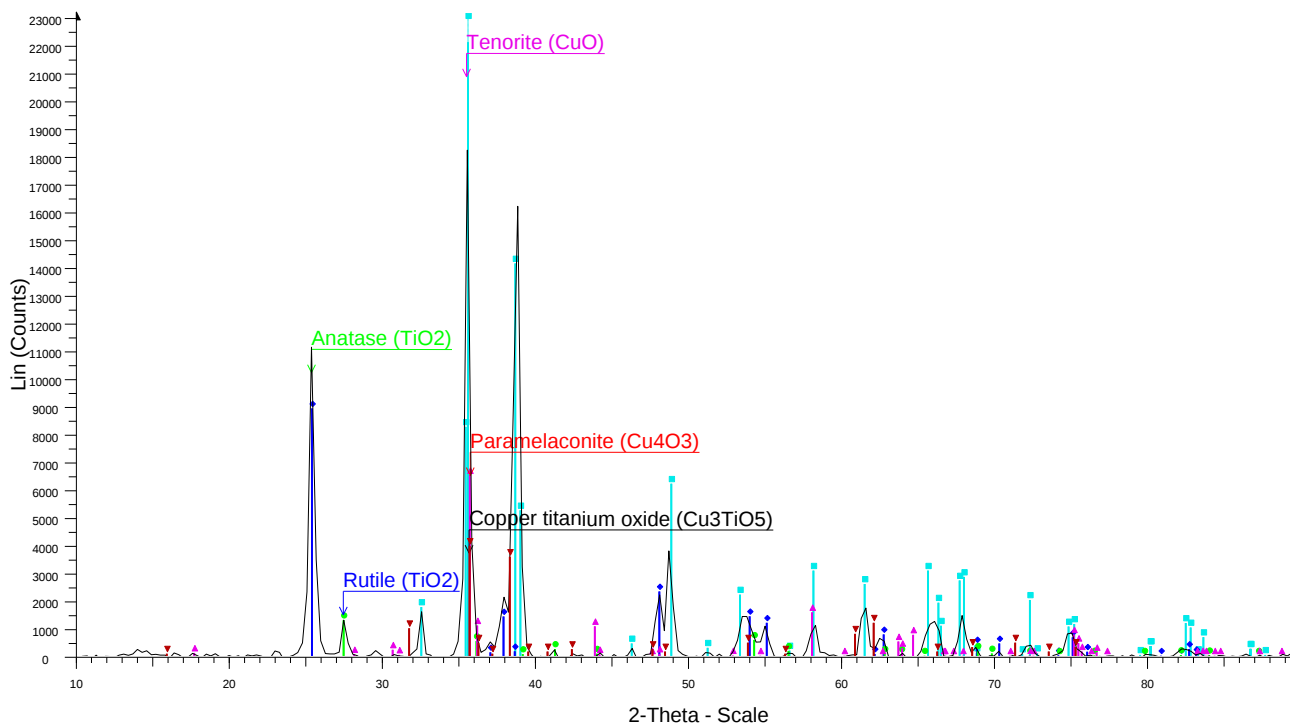
01-080-0076 (C) - Tenorite, syn - CuO - Y: 177.12 % - d x by: 1. - WL: 1.5406 - Monoclinic - a 4.67970 - b 3.43140 - c 5.13620 - alpha 90.000 - beta 99.262 - gamma 90.000 - Base-centered - C2/c (15) - 4 - 81.4014 - I/I

01-086-1157 (A) - Anatase, syn - TiO₂ - Y: 36.82 % - d x by: 1. - WL: 1.5406 - Tetragonal - a 3.78300 - b 3.78300 - c 9.49700 - alpha 90.000 - beta 90.000 - gamma 90.000 - Body-centered - I41/amd (141) - 4 - 135

00-034-0180 (D) - Rutile, syn - TiO₂ - Y: 8.04 % - d x by: 1. - WL: 1.5406 - Tetragonal - a 4.59300 - b 4.59300 - c 2.95900 - alpha 90.000 - beta 90.000 - gamma 90.000 - Primitive - P42/mnm (136) - 2 - 62.4220 - I/c Pt

00-018-0461 (D) - Copper Titanium Oxide - Cu₃TiO₅ - Y: 19.67 % - d x by: 0.9792 - WL: 1.5406 - Tetragonal - a 8.10000 - b 8.10000 - c 6.60000 - alpha 90.000 - beta 90.000 - gamma 90.000 - 433.026 - F22= 1(0.176)

01-083-1665 (C) - Paramelaconite - Cu₄O₃ - Y: 36.06 % - d x by: 1. - WL: 1.5406 - Tetragonal - a 5.83700 - b 5.83700 - c 9.93200 - alpha 90.000 - beta 90.000 - gamma 90.000 - Body-centered - I41/amd (141) - 4 - 338



CuTiO - 708 - File: d5_577.raw - Type: 2Th/Th locked - Start: 10.000 ° - End: 91.300 ° - Step: 0.300 ° - Step time: 25. s - Temp.: 25 °C (Room) - Time Started: 12 s - 2-Theta: 10.000 ° - Theta: 5.000 ° - Chi: 0.00 ° - Phi: 0.00 ° - Operations: Background 1.000,1.000 | Import

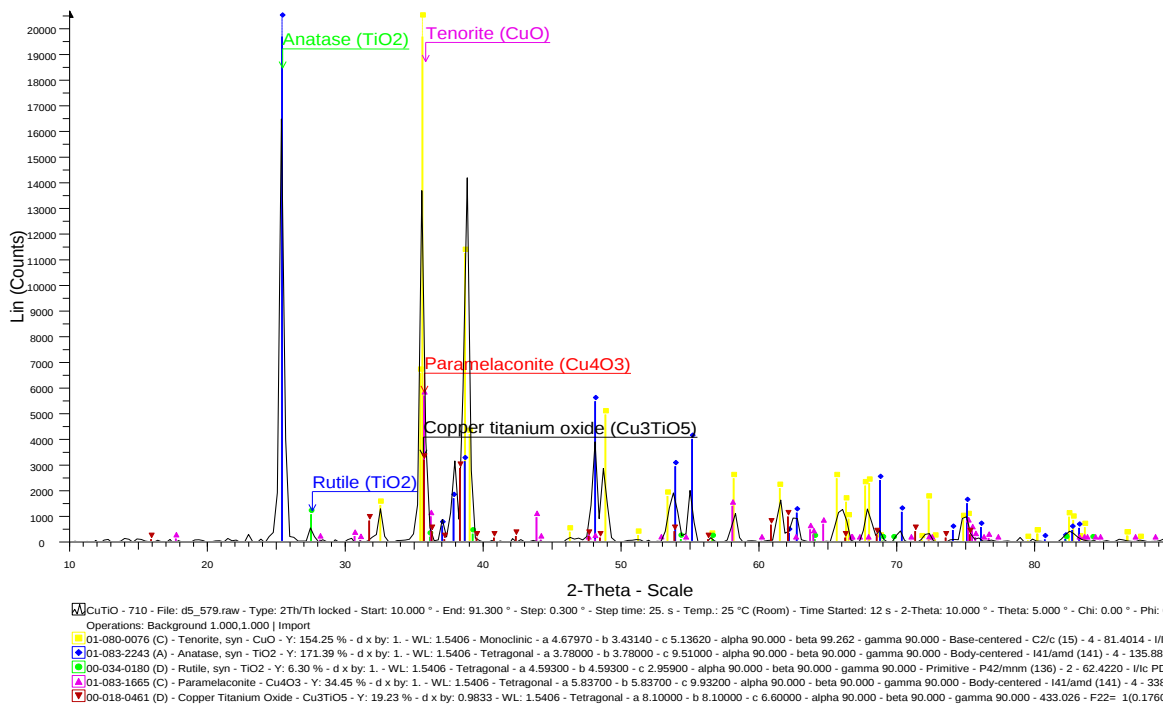
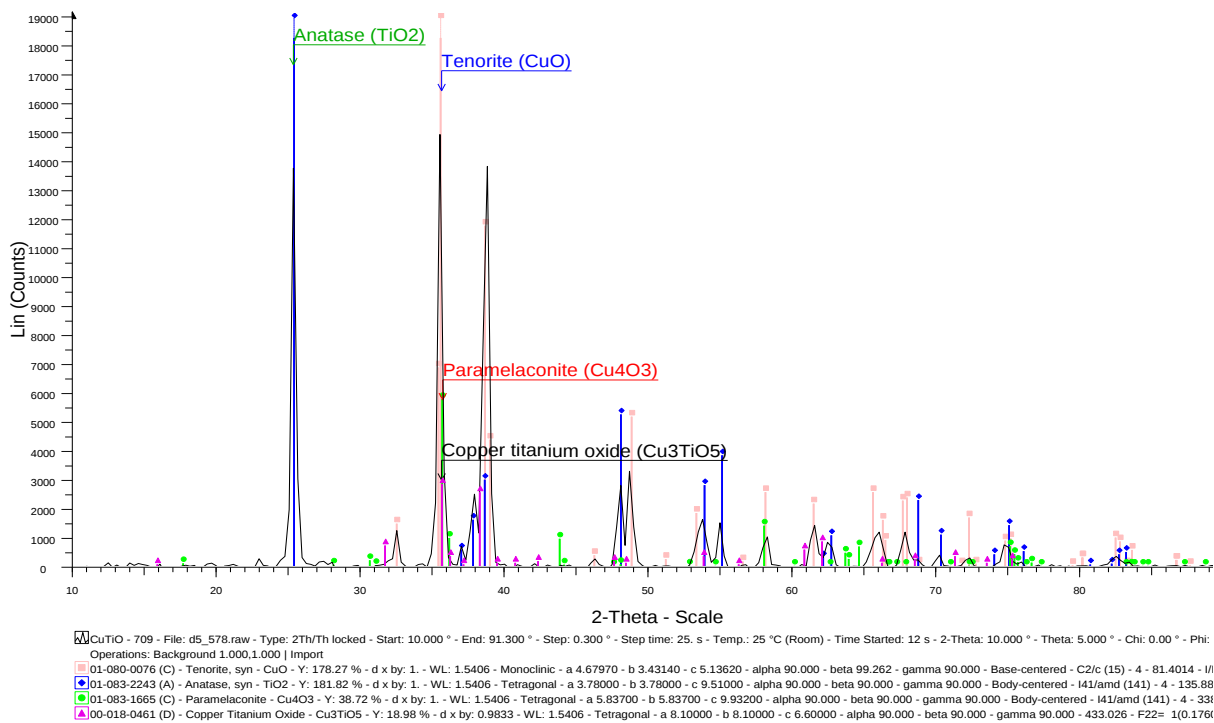
01-080-0076 (C) - Tenorite, syn - CuO - Y: 175.45 % - d x by: 1. - WL: 1.5406 - Monoclinic - a 4.67970 - b 3.43140 - c 5.13620 - alpha 90.000 - beta 99.262 - gamma 90.000 - Base-centered - C2/c (15) - 4 - 81.4014 - I/I

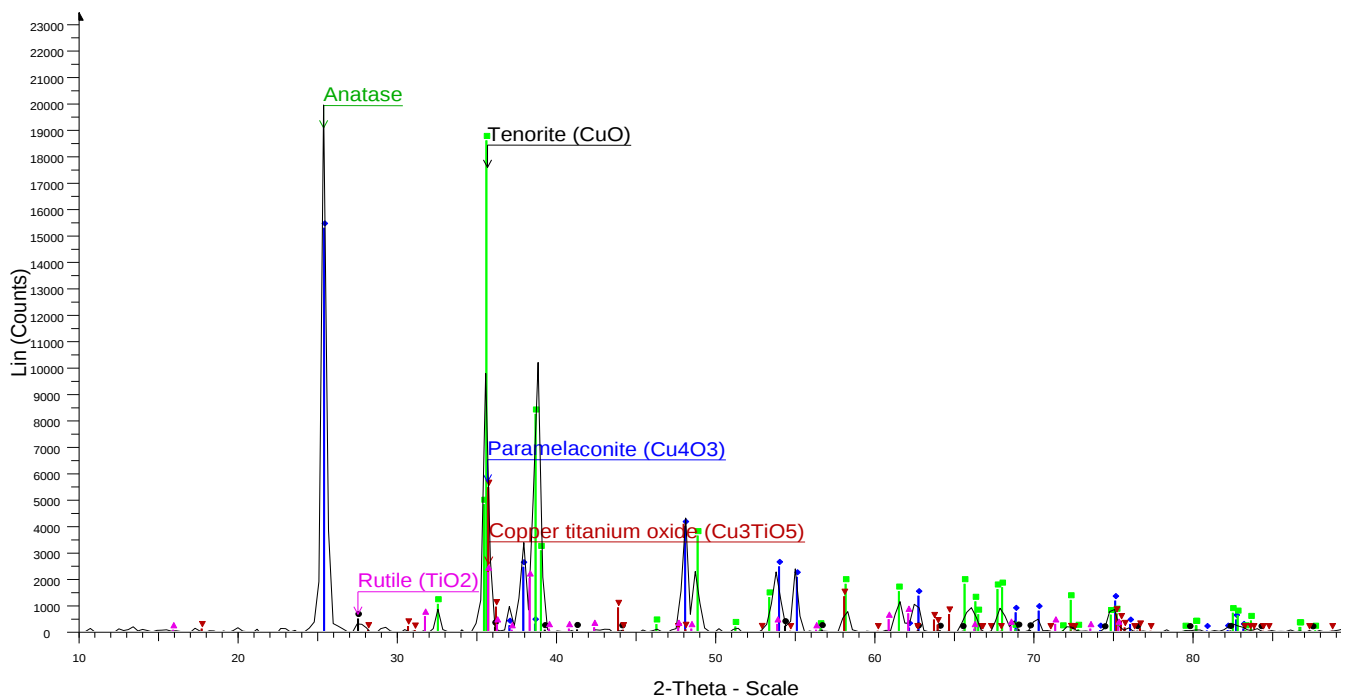
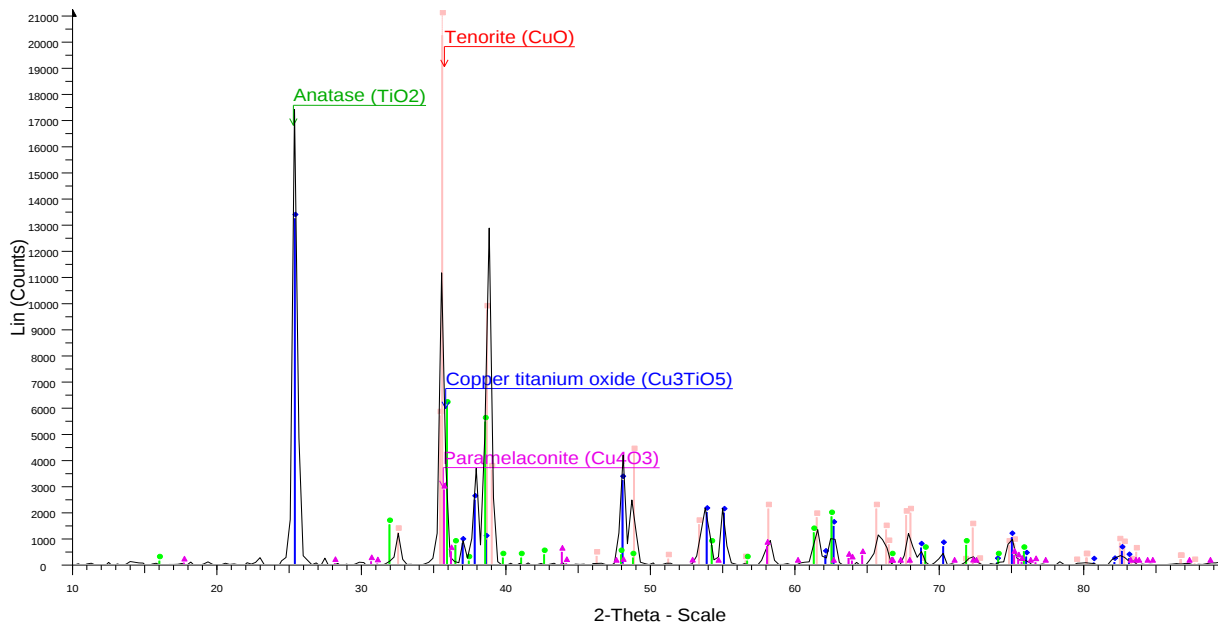
01-086-1157 (A) - Anatase, syn - TiO₂ - Y: 48.93 % - d x by: 1. - WL: 1.5406 - Tetragonal - a 3.78300 - b 3.78300 - c 9.49700 - alpha 90.000 - beta 90.000 - gamma 90.000 - Body-centered - I41/amd (141) - 4 - 135

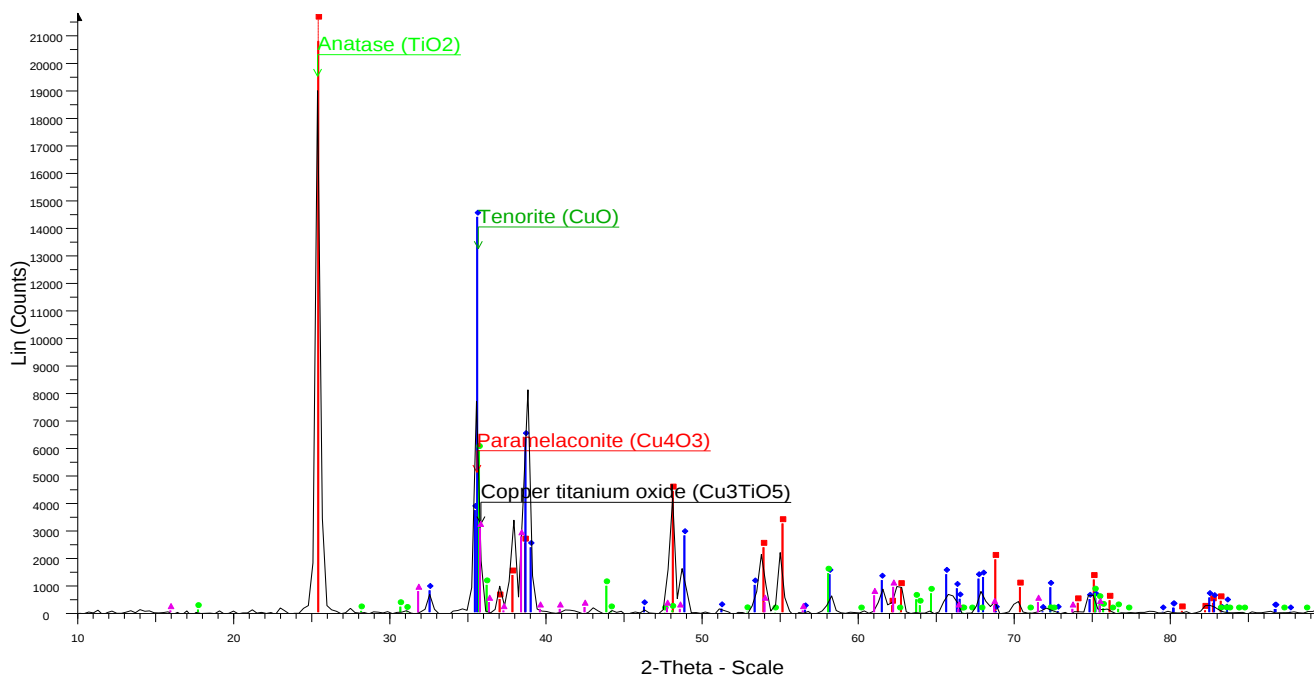
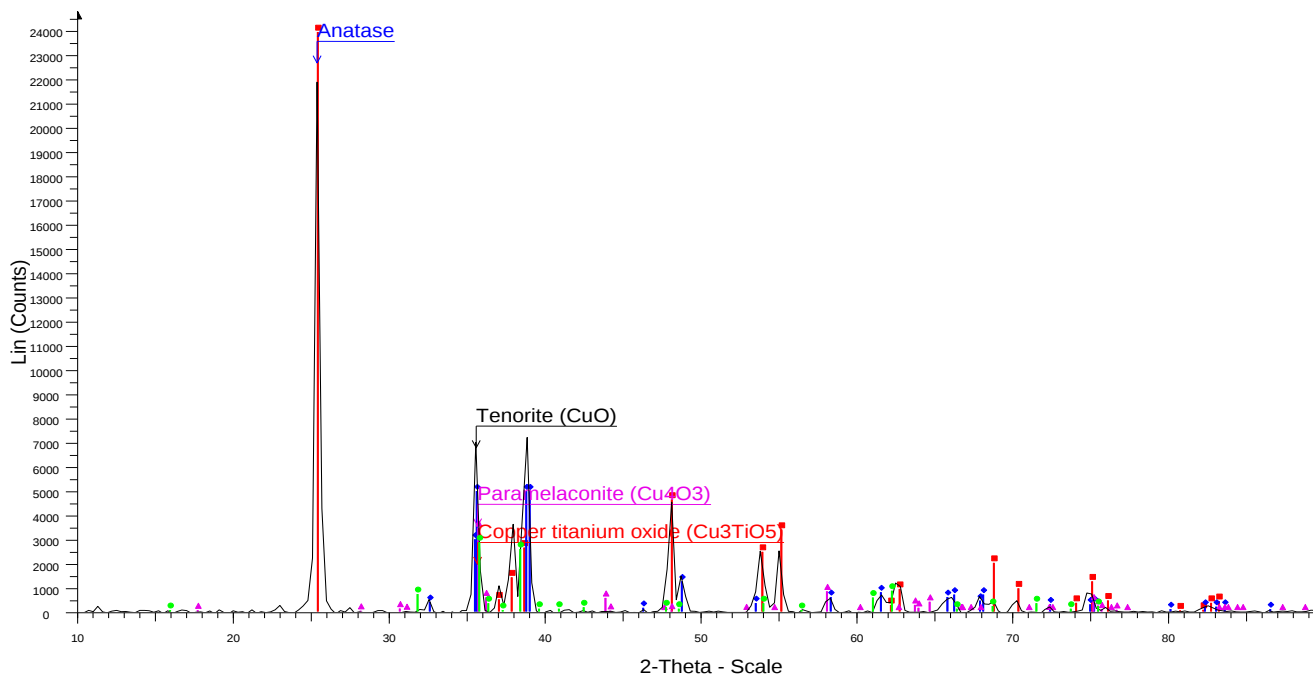
01-089-0552 (C) - Rutile, syn - TiO₂ - Y: 7.04 % - d x by: 1. - WL: 1.5406 - Tetragonal - a 4.60100 - b 4.60100 - c 2.95600 - alpha 90.000 - beta 90.000 - gamma 90.000 - Primitive - P42/mnm (136) - 2 - 62.5762 - I

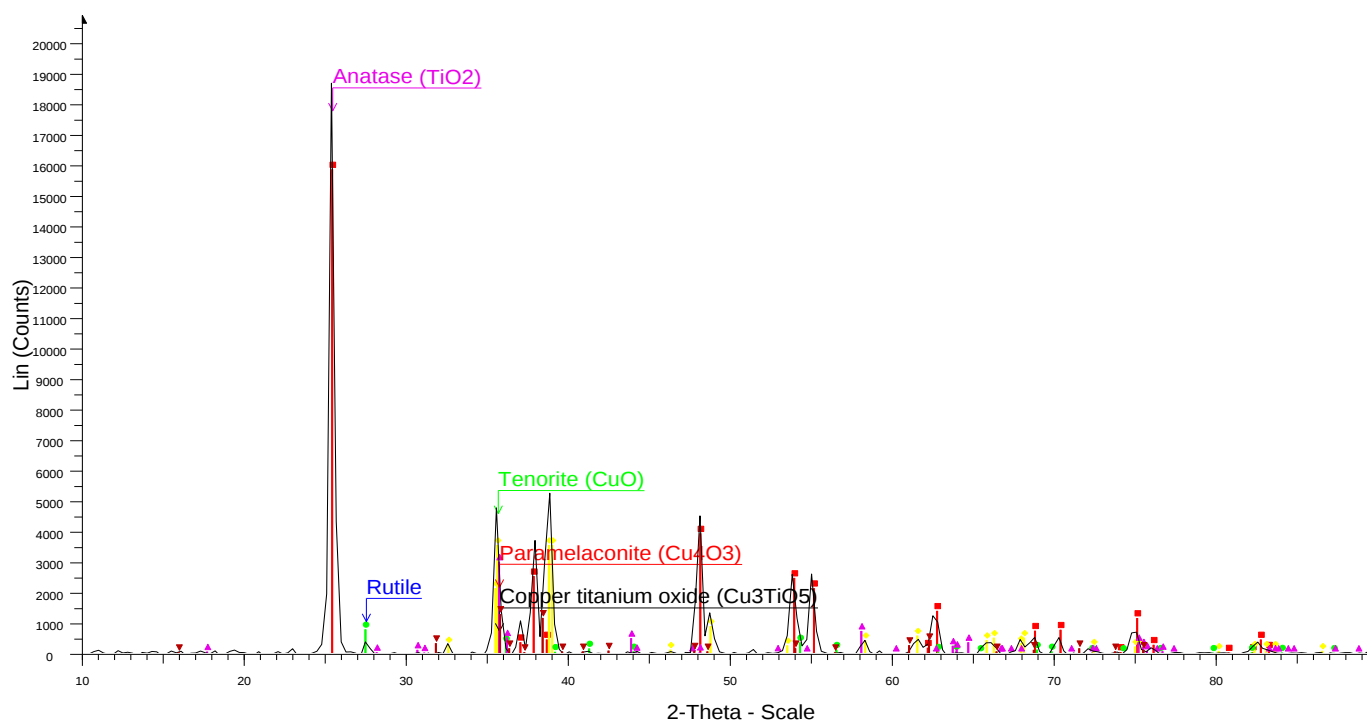
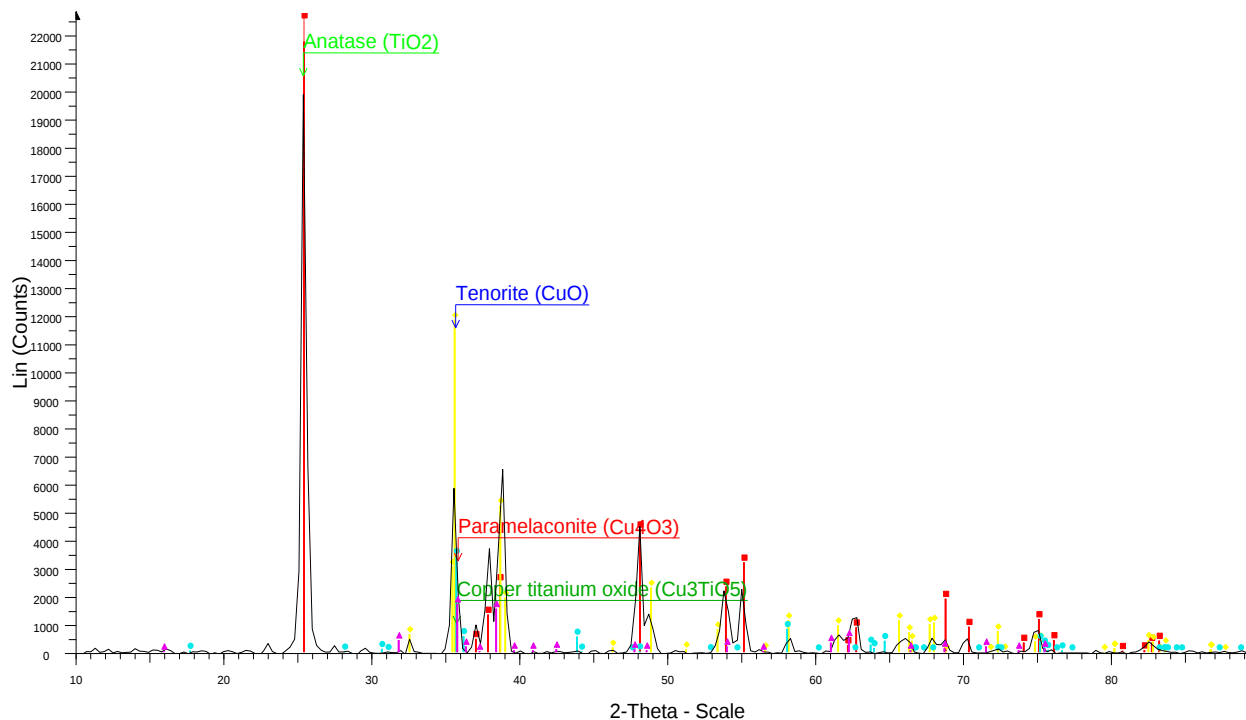
01-083-1665 (C) - Paramelaconite - Cu₄O₃ - Y: 35.73 % - d x by: 1. - WL: 1.5406 - Tetragonal - a 5.83700 - b 5.83700 - c 9.93200 - alpha 90.000 - beta 90.000 - gamma 90.000 - Body-centered - I41/amd (141) - 4 - 338

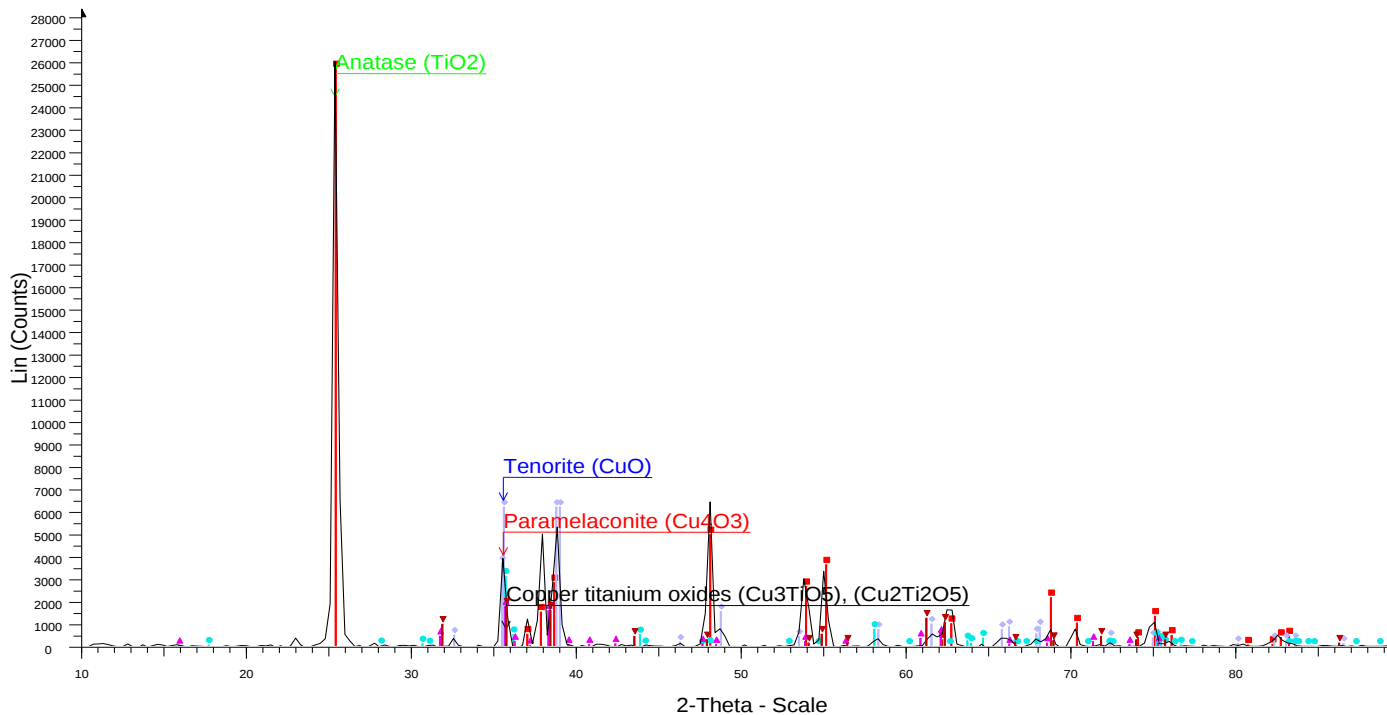
00-018-0461 (D) - Copper Titanium Oxide - Cu₃TiO₅ - Y: 21.79 % - d x by: 0.9833 - WL: 1.5406 - Tetragonal - a 8.10000 - b 8.10000 - c 6.60000 - alpha 90.000 - beta 90.000 - gamma 90.000 - 433.026 - F22= 1(0.176)







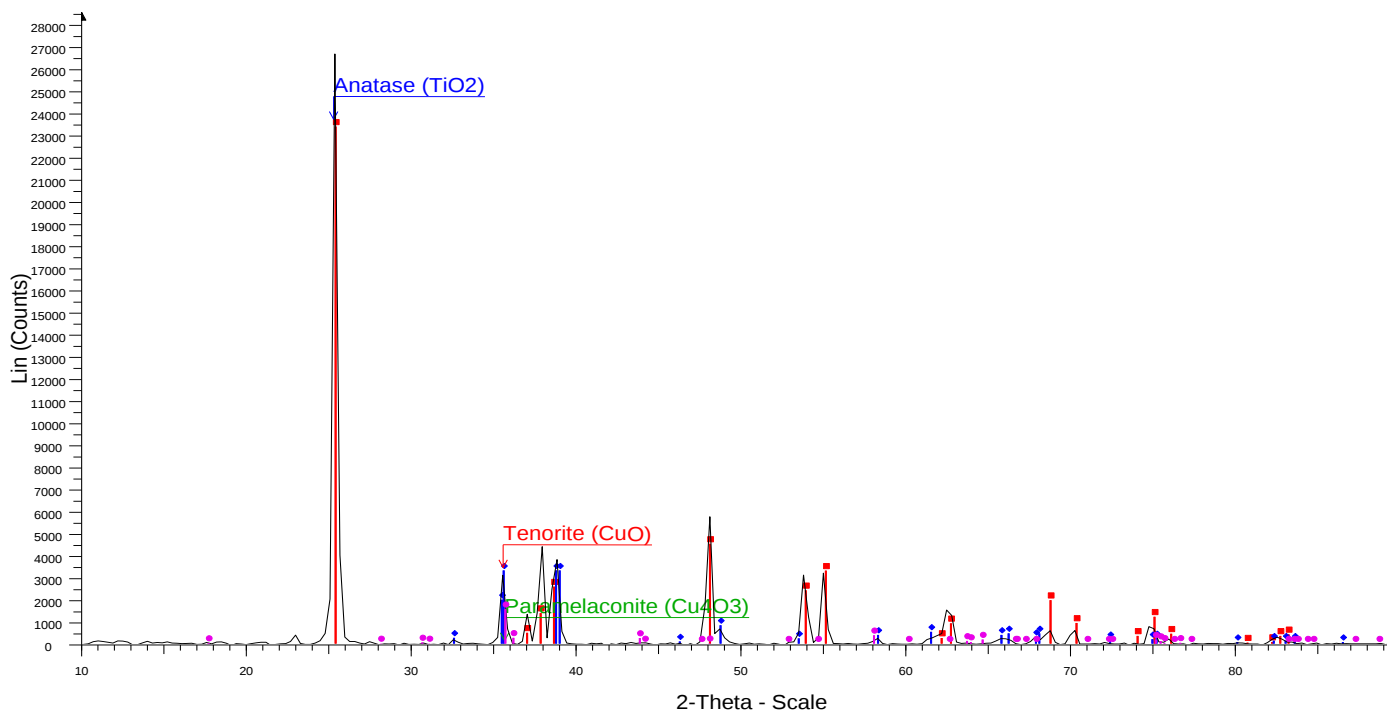




CuTiO - 717 - File: d5_586.raw - Type: 2Th/Th locked - Start: 10.000 ° - End: 91.300 ° - Step: 0.300 ° - Step time: 25. s - Temp.: 25 °C (Room) - Time Started: 11 s - 2-Theta: 10.000 ° - Theta: 5.000 ° - Chi: 0.00 ° - Phi: 0.00 ° - Operations: Background 1.000,1.000 | Import

Legend:

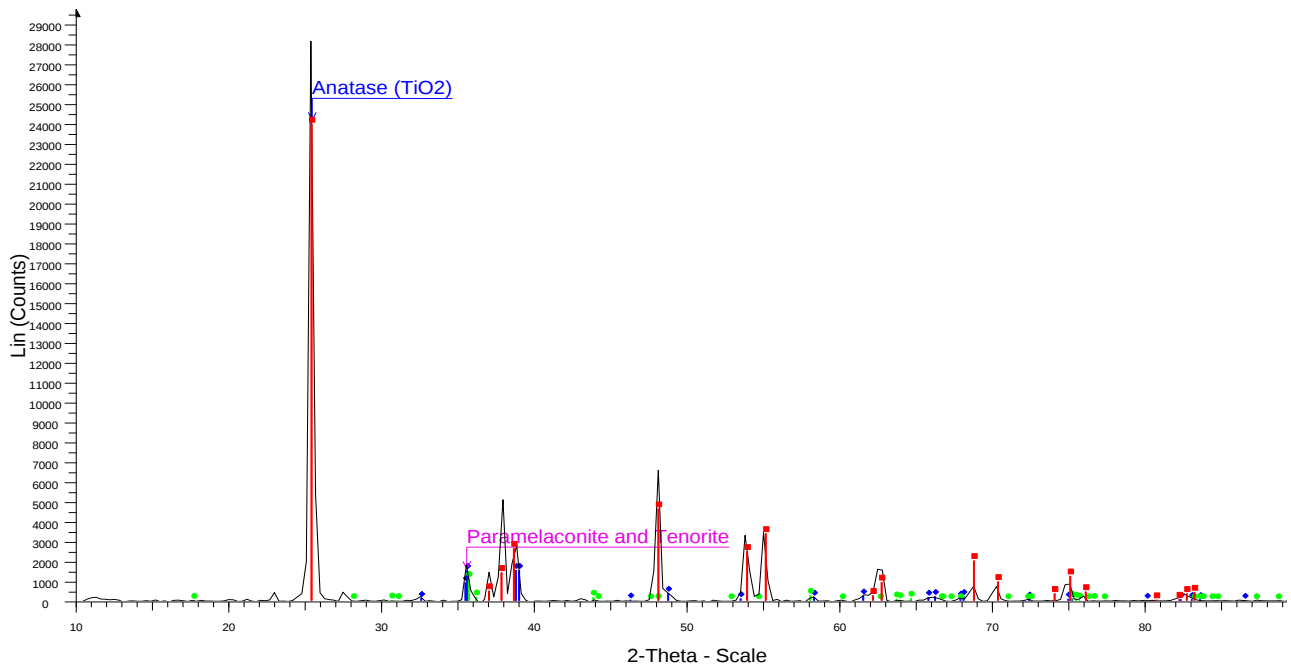
- 01-083-2243 (A) - Anatase, syn - TiO₂ - Y: 98.91 % - d x by: 1. - WL: 1.5406 - Tetragonal - a 3.78000 - b 3.78000 - c 9.51000 - alpha 90.000 - beta 90.000 - gamma 90.000 - Body-centered - I41/amd (141) - 4 - 135.883
- 00-041-0254 (D) - Tenorite, syn - CuO - Y: 23.82 % - d x by: 1. - WL: 1.5406 - Monoclinic - a 4.68500 - b 3.42300 - c 5.13200 - alpha 90.000 - beta 99.520 - gamma 90.000 - Base-centered - C2/c (15) - 4 - 81.1672 - I/c
- 01-083-1665 (C) - Paramelaconite - Cu₄O₃ - Y: 12.00 % - d x by: 1. - WL: 1.5406 - Tetragonal - a 5.83700 - b 5.83700 - c 9.93200 - alpha 90.000 - beta 90.000 - gamma 90.000 - Body-centered - I41/amd (141) - 4 - 338
- ▲ 00-018-0461 (D) - Copper Titanium Oxide - Cu₃TiO₅ - Y: 6.78 % - d x by: 0.9833 - WL: 1.5406 - Tetragonal - a 8.10000 - b 8.10000 - c 6.60000 - alpha 90.000 - beta 90.000 - gamma 90.000 - 433.026 - F22= 1(0.1760,
- ▼ 00-024-0340 (D) - Copper Titanium Oxide - Cu₂Ti₂O₅/Cu₂O·2TiO₂ - Y: 6.86 % - d x by: 0.9833 - WL: 1.5406 -



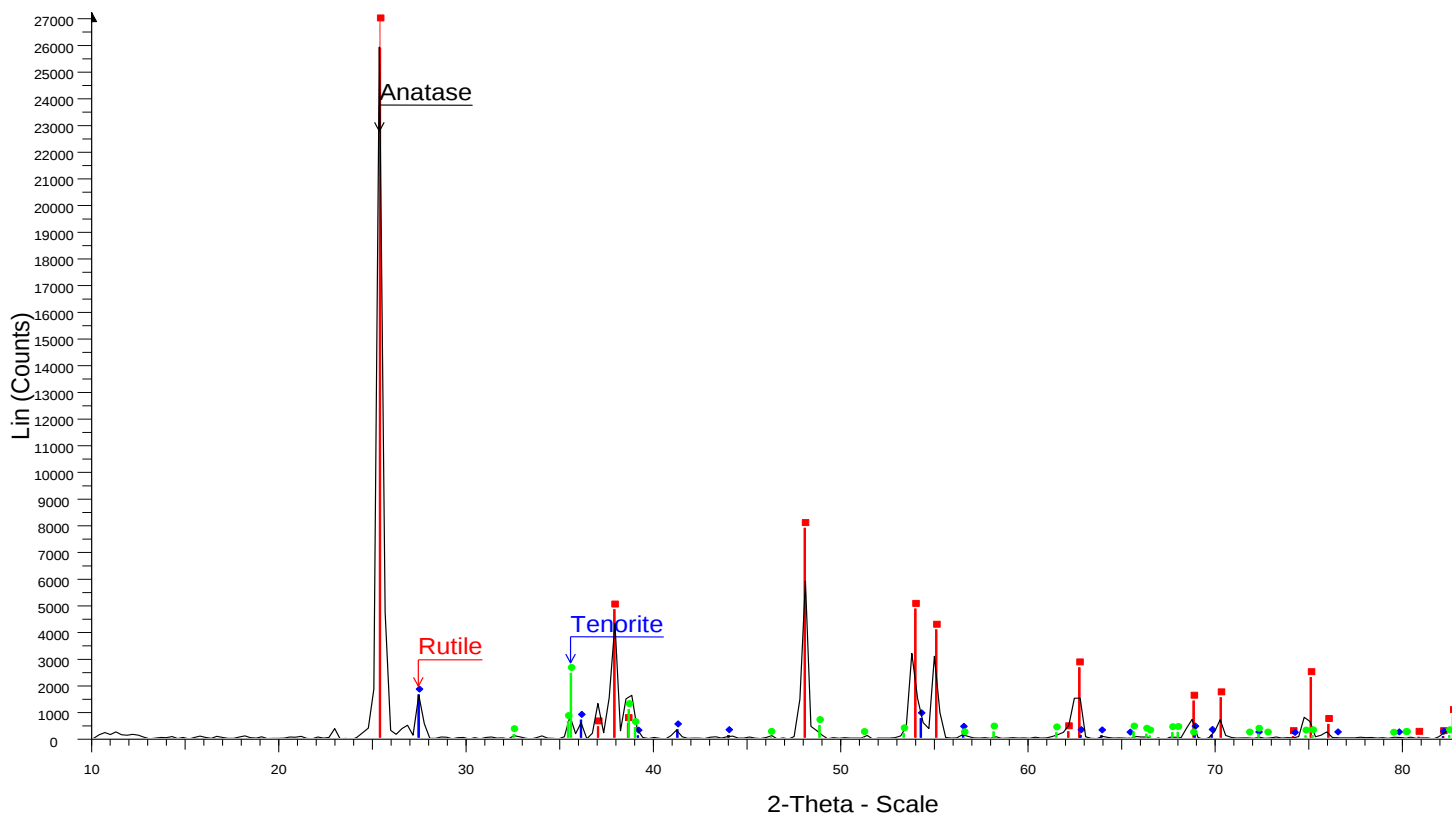
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Legend:

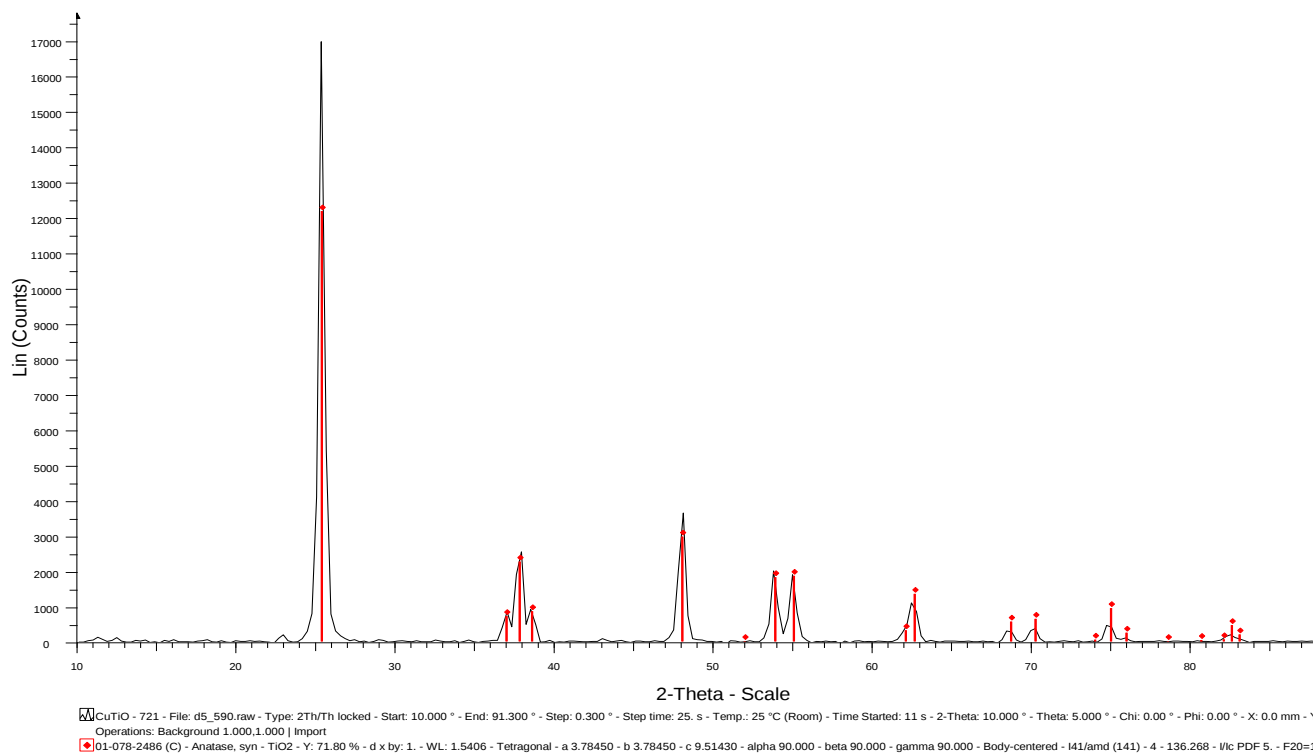
- 01-083-2243 (A) - Anatase, syn - TiO₂ - Y: 87.71 % - d x by: 1. - WL: 1.5406 - Tetragonal - a 3.78000 - b 3.78000 - c 9.51000 - alpha 90.000 - beta 90.000 - gamma 90.000 - Body-centered - I41/amd (141) - 4 - 135.883
- 00-041-0254 (D) - Tenorite, syn - CuO - Y: 12.41 % - d x by: 1. - WL: 1.5406 - Monoclinic - a 4.68500 - b 3.42300 - c 5.13200 - alpha 90.000 - beta 99.520 - gamma 90.000 - Base-centered - C2/c (15) - 4 - 81.1672 - I/c
- 01-083-1665 (C) - Paramelaconite - Cu₄O₃ - Y: 5.84 % - d x by: 1. - WL: 1.5406 - Tetragonal - a 5.83700 - b 5.83700 - c 9.93200 - alpha 90.000 - beta 90.000 - gamma 90.000 - Body-centered - I41/amd (141) - 4 - 338



CuTiO - 719 - File: d5_588.raw - Type: 2Th/Th locked - Start: 10.000 ° - End: 91.300 ° - Step: 0.300 ° - Step time: 25. s - Temp.: 25 °C (Room) - Time Started: 11 s - 2-Theta: 10.000 ° - Theta: 5.000 ° - Chi: 0.00 ° - Phi: 0.00 °
Operations: Background 1.000,1.000 | Import
01-083-2243 (A) - Anatase, syn - TiO₂ - Y: 85.22 % - d x by: 1. - WL: 1.5406 - Tetragonal - a 3.78000 - b 3.78000 - c 9.51000 - alpha 90.000 - beta 90.000 - gamma 90.000 - Body-centered - I41/amd (141) - 4 - 135.883
00-041-0254 (D) - Tenorite, syn - CuO - Y: 5.43 % - d x by: 1. - WL: 1.5406 - Monoclinic - a 4.68500 - b 3.42300 - c 5.13200 - alpha 90.000 - beta 99.520 - gamma 90.000 - Base-centered - C2/c (15) - 4 - 81.1672 - I/c I
01-083-1665 (C) - Paramelaconite - Cu₄O₃ - Y: 3.98 % - d x by: 1. - WL: 1.5406 - Tetragonal - a 5.83700 - b 5.83700 - c 9.93200 - alpha 90.000 - beta 90.000 - gamma 90.000 - Body-centered - I41/amd (141) - 4 - 338.

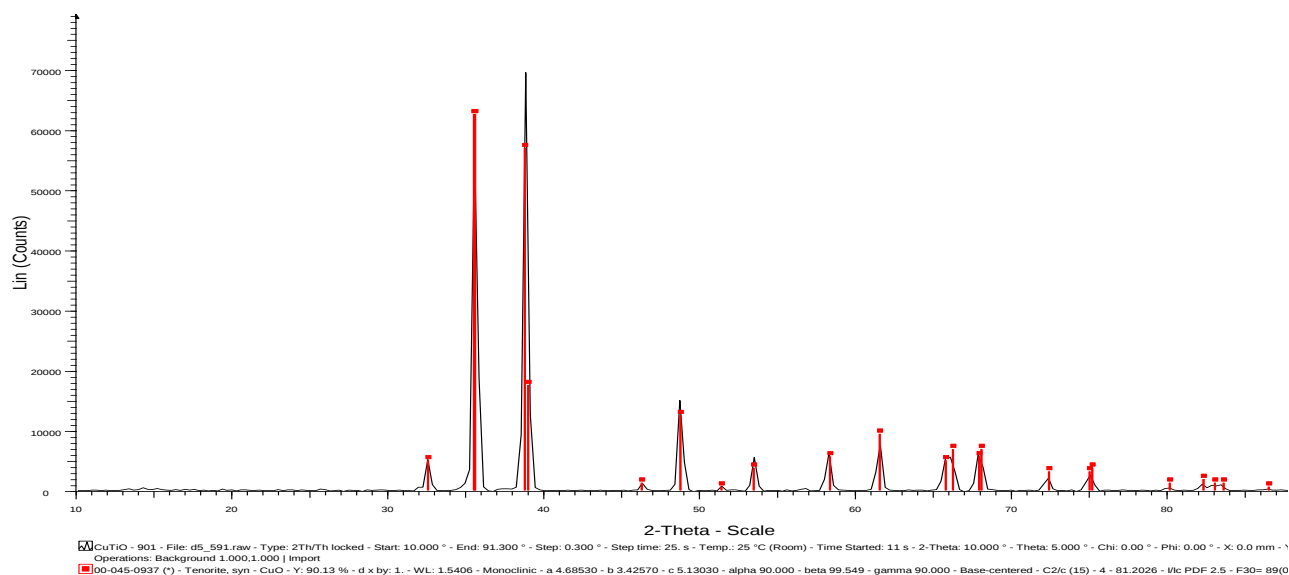


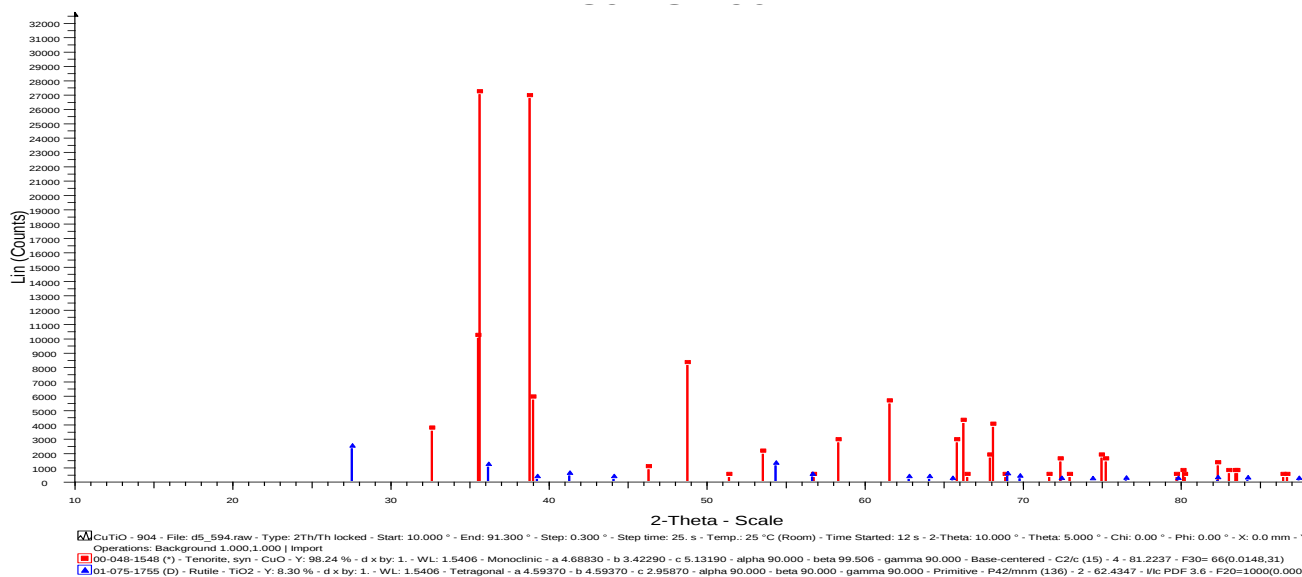
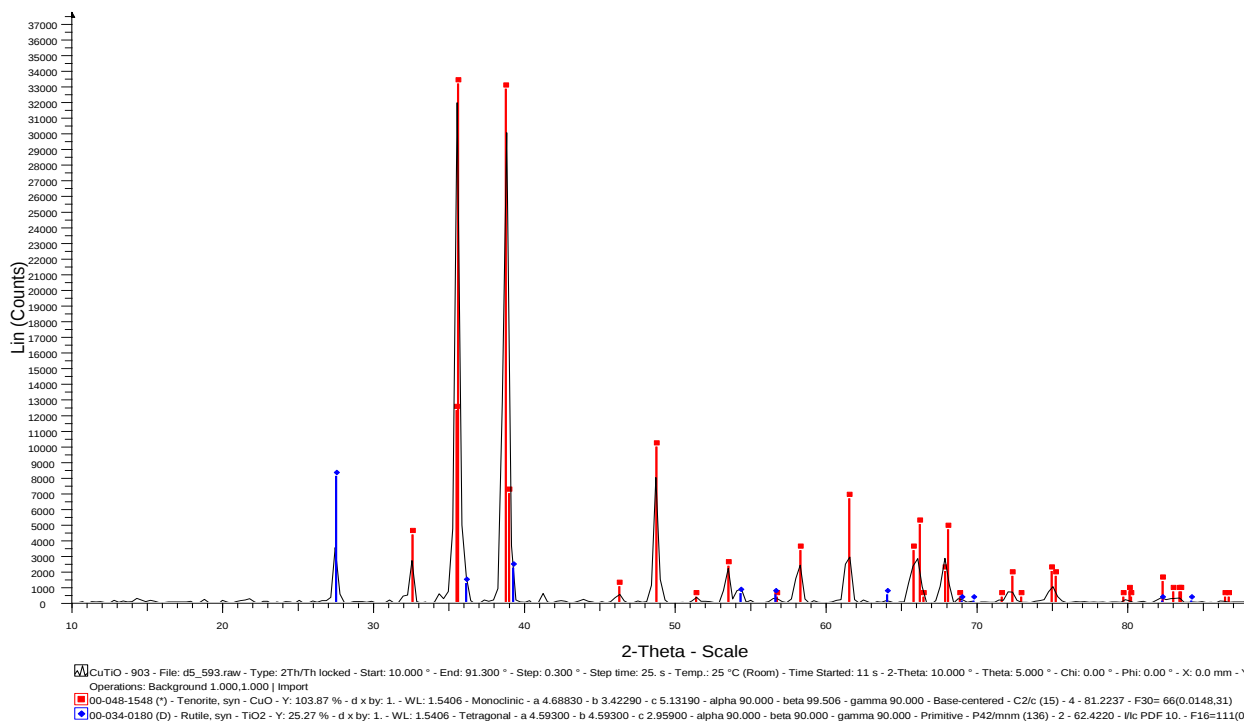
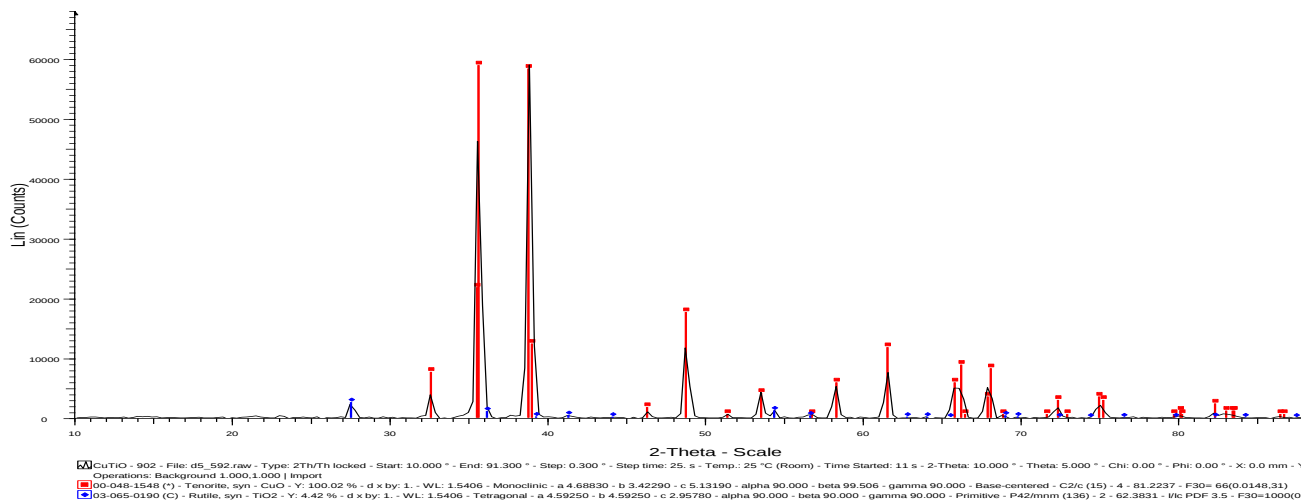
CuTiO - 720 - File: d5_589.raw - Type: 2Th/Th locked - Start: 10.000 ° - End: 91.300 ° - Step: 0.300 ° - Step time: 25. s - Temp.: 25 °C (Room) - Time Started: 11 s - 2-Theta: 10.000 ° - Theta: 5.000 °
Operations: Background 1.000,1.000 | Import
01-086-1157 (A) - Anatase, syn - TiO₂ - Y: 116.82 % - d x by: 1. - WL: 1.5406 - Tetragonal - a 3.78300 - b 3.78300 - c 9.49700 - alpha 90.000 - beta 90.000 - gamma 90.000 - Body-centered - I41/amd (141) - 4 - 135.883
01-089-0552 (C) - Rutile, syn - TiO₂ - Y: 6.27 % - d x by: 1. - WL: 1.5406 - Tetragonal - a 4.60100 - b 4.60100 - c 2.95600 - alpha 90.000 - beta 90.000 - gamma 90.000 - Primitive - P42/mnm (13) - 2 - 35.0912 - I/c I
01-080-0076 (C) - Tenorite, syn - CuO - Y: 9.38 % - d x by: 1. - WL: 1.5406 - Monoclinic - a 4.67970 - b 3.43140 - c 5.13620 - alpha 90.000 - beta 99.262 - gamma 90.000 - Base-centered - C2/c (15) - 4 - 81.1672 - I/c I

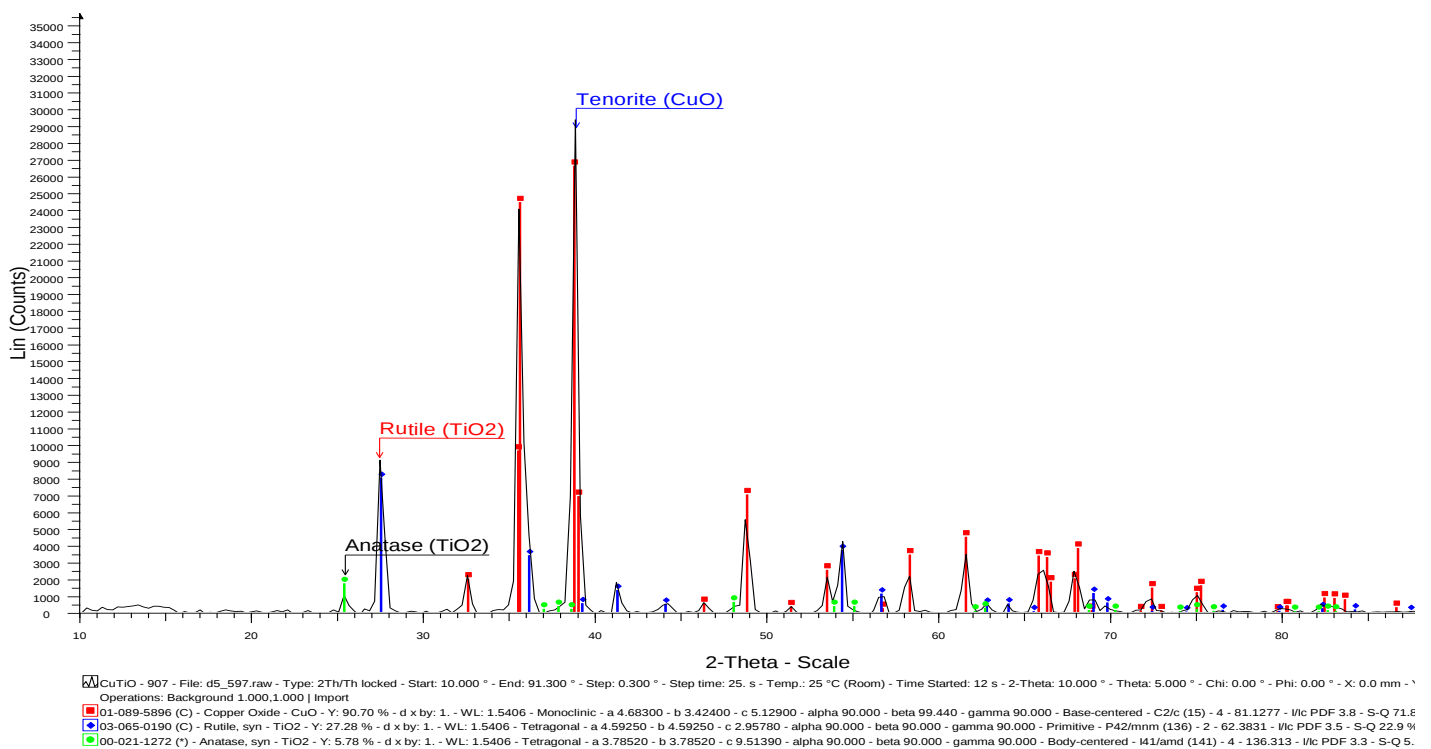
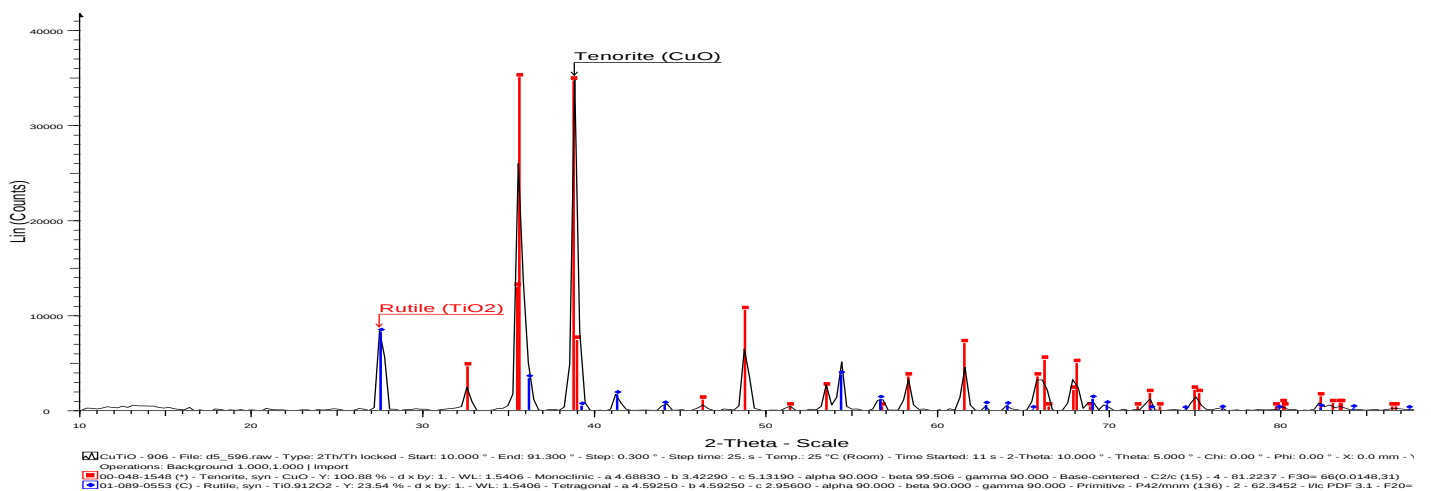
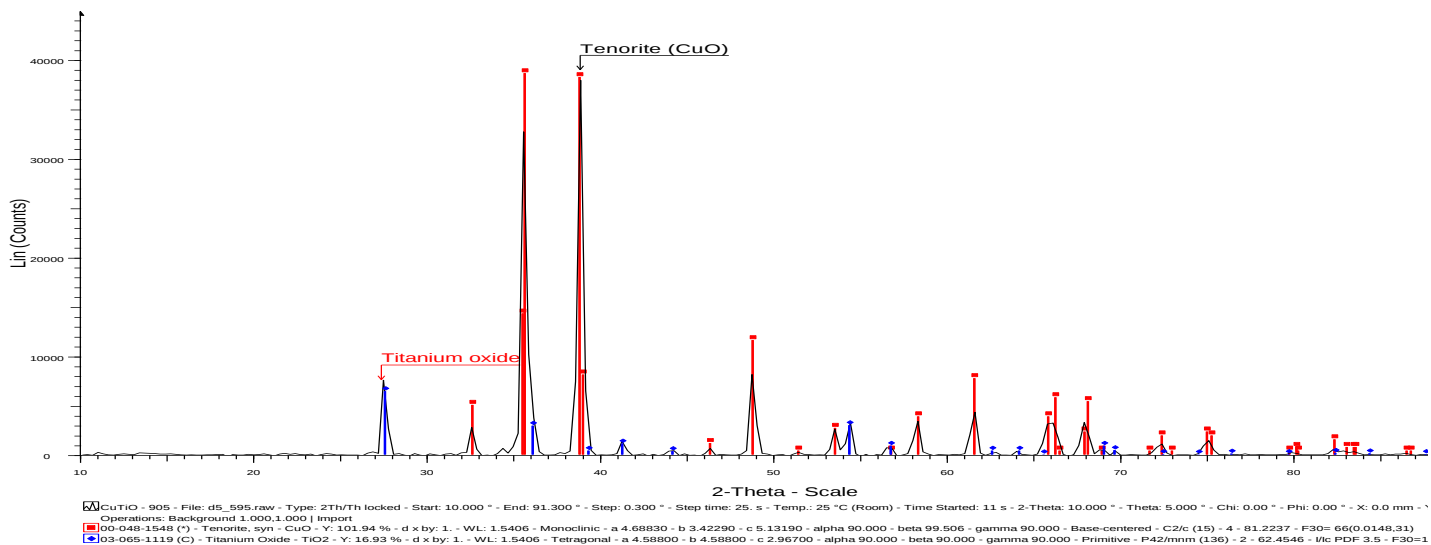


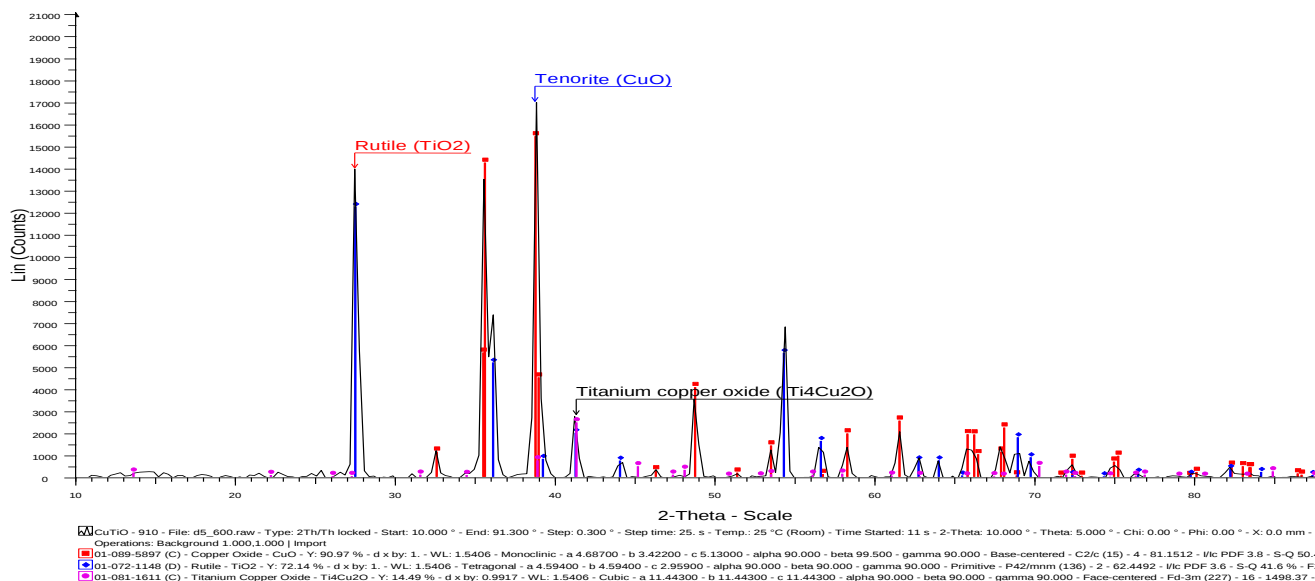
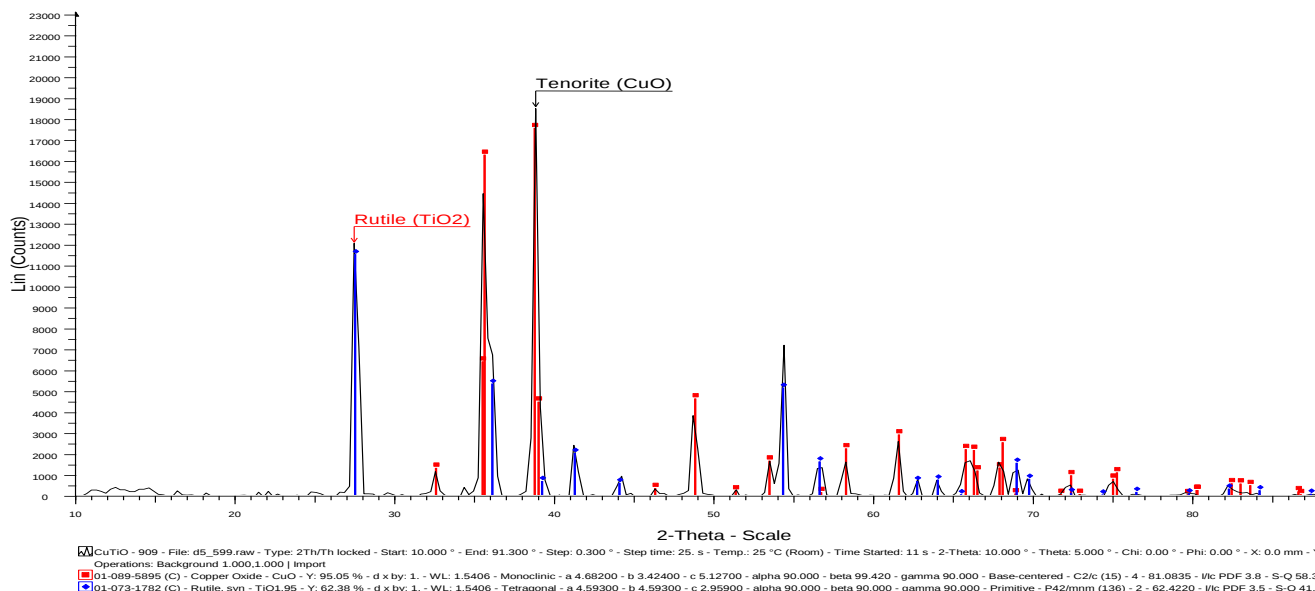
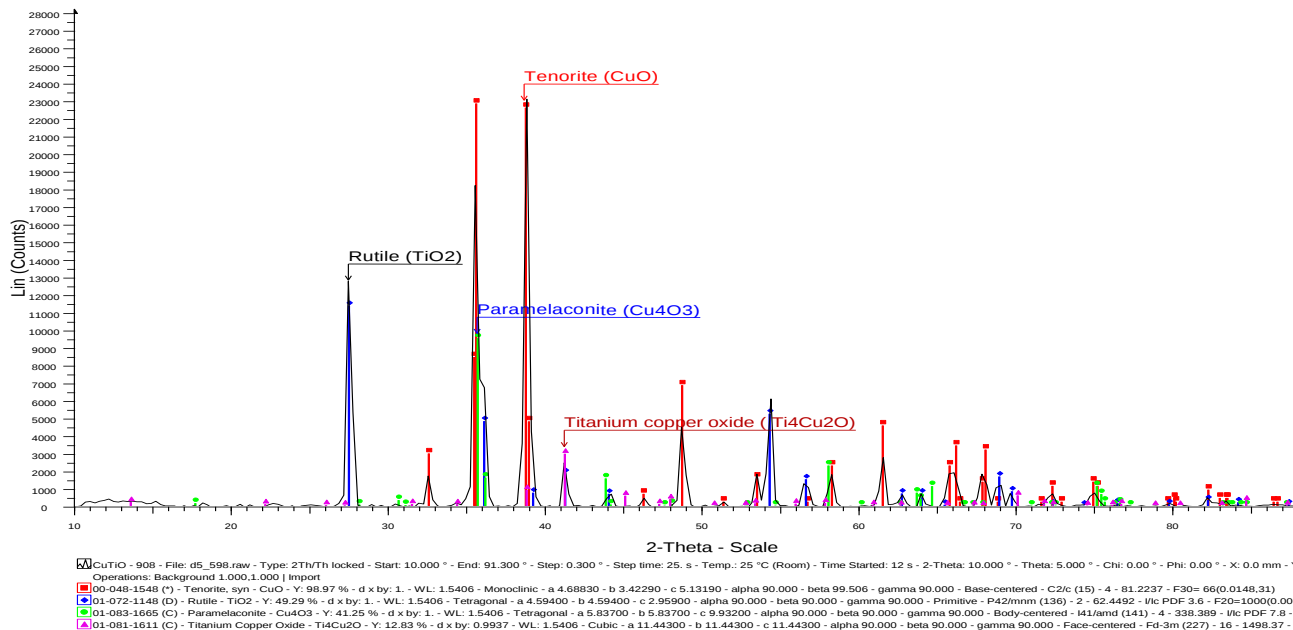
APPENDIX F

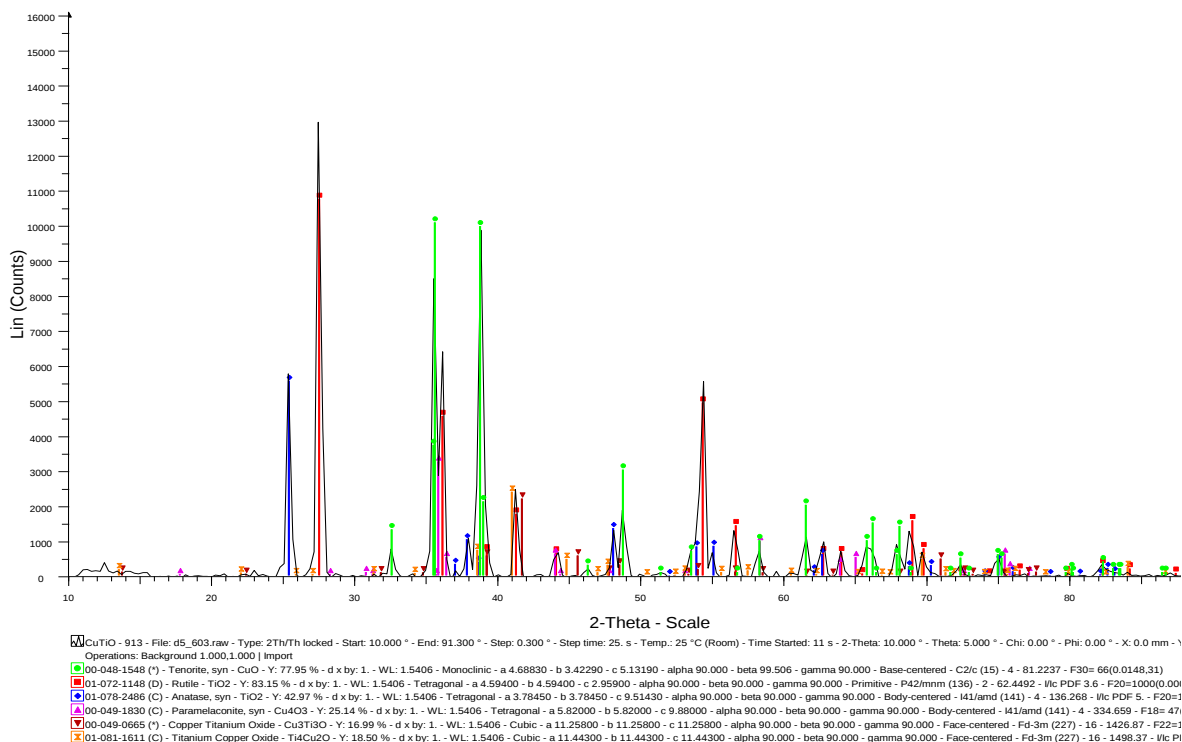
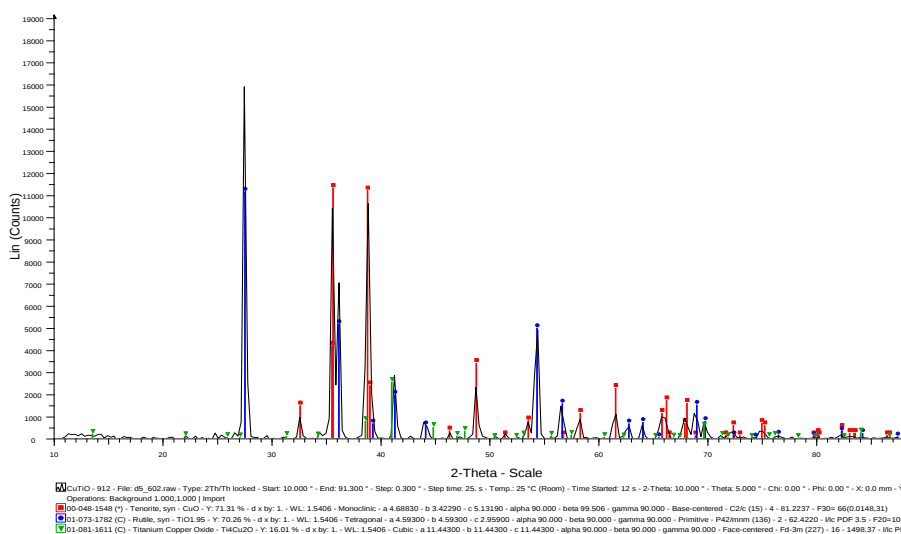
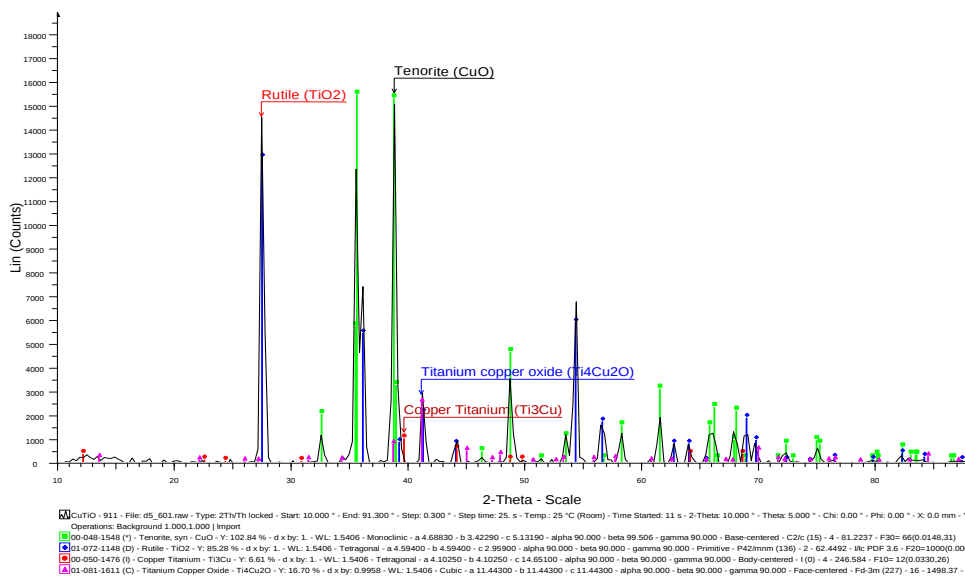
Patterns of Resin-gel precursor powders calcined at 900 °C

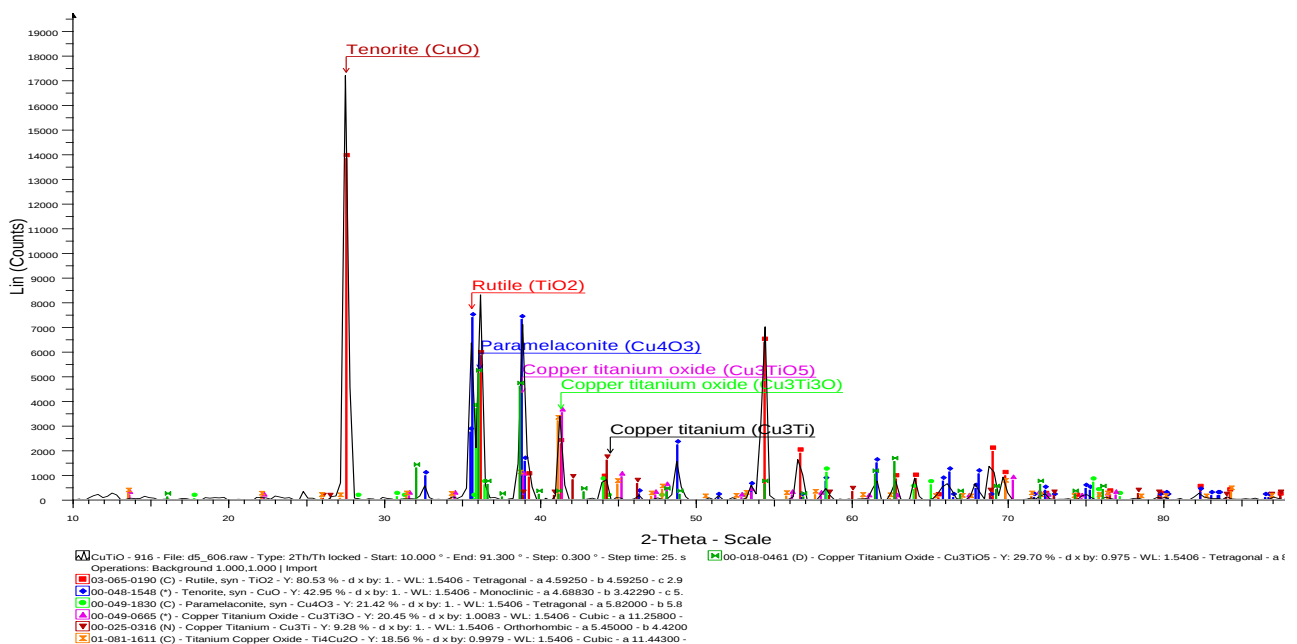
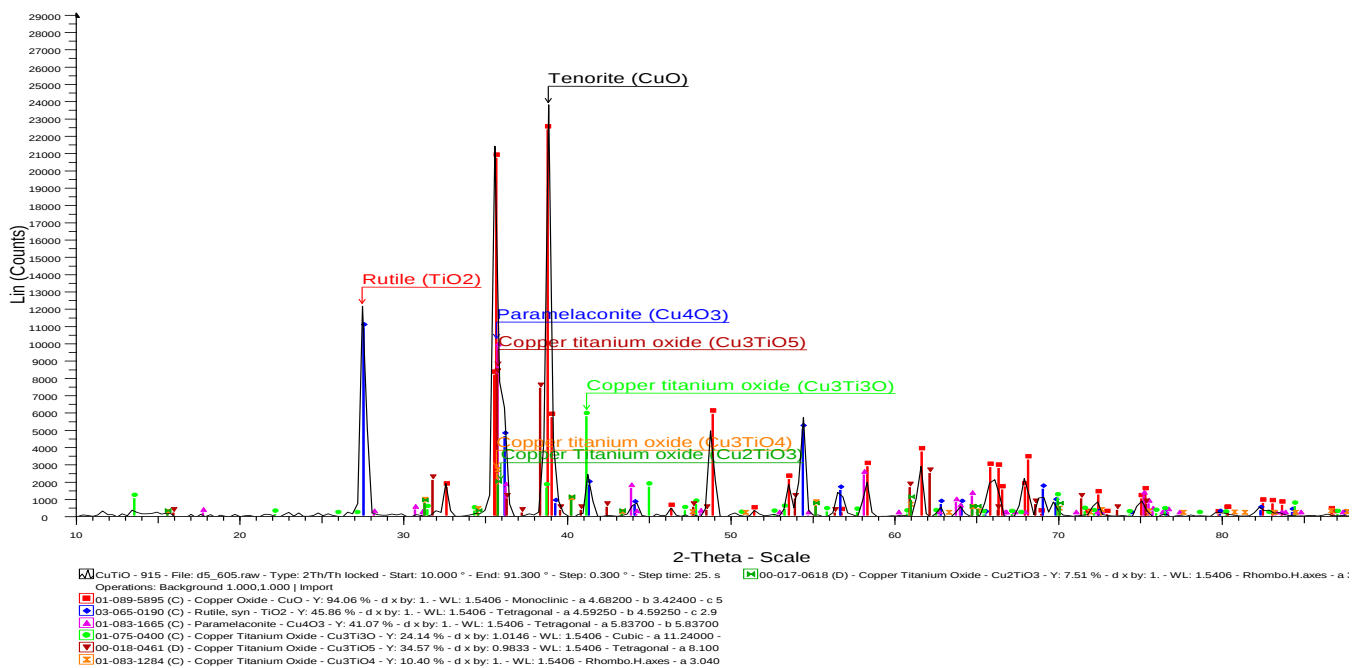
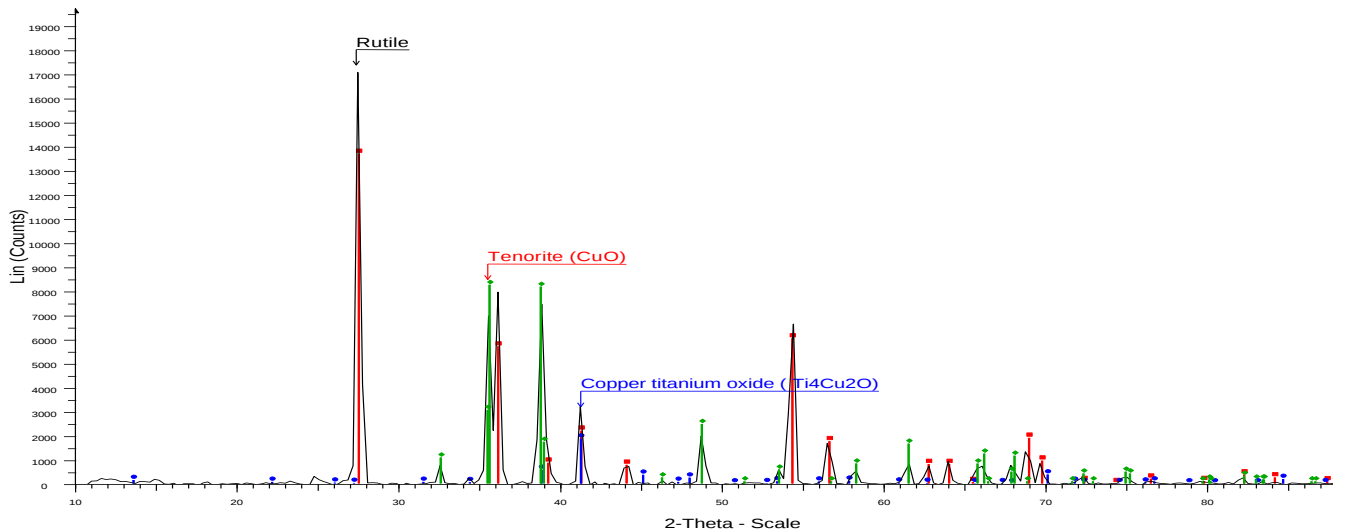


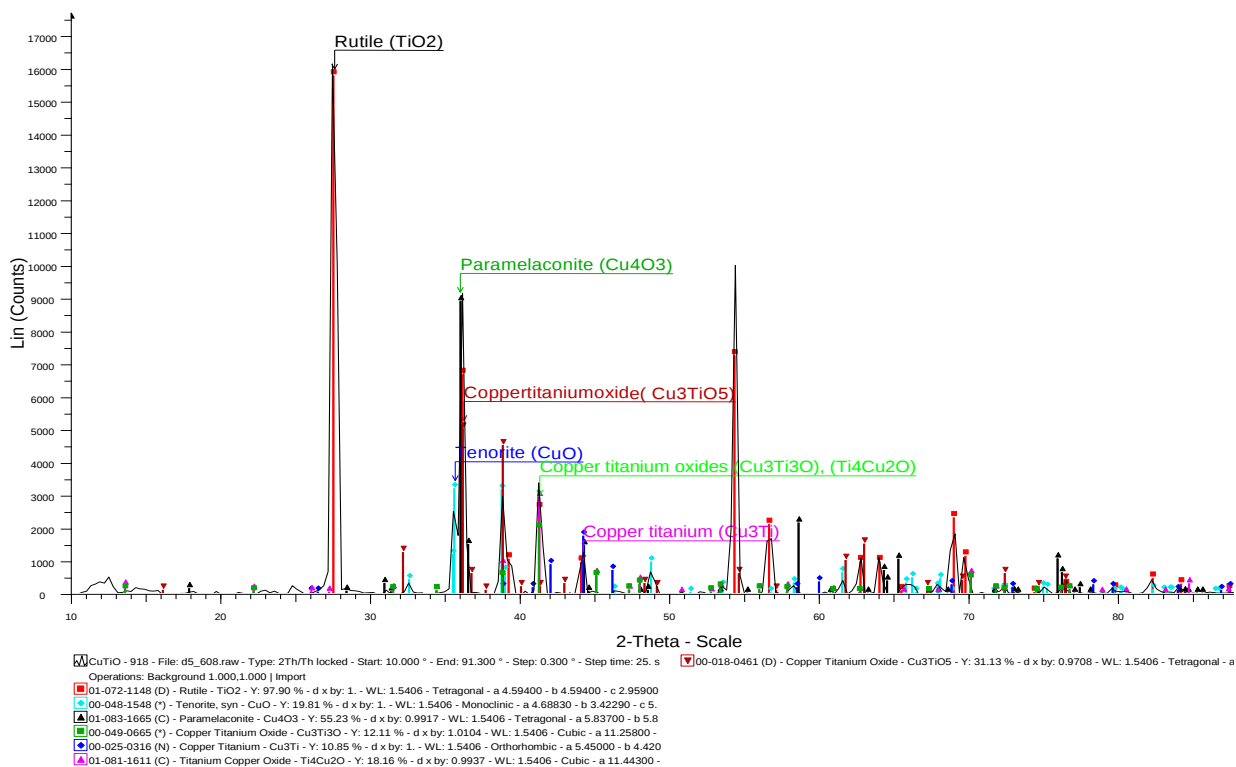
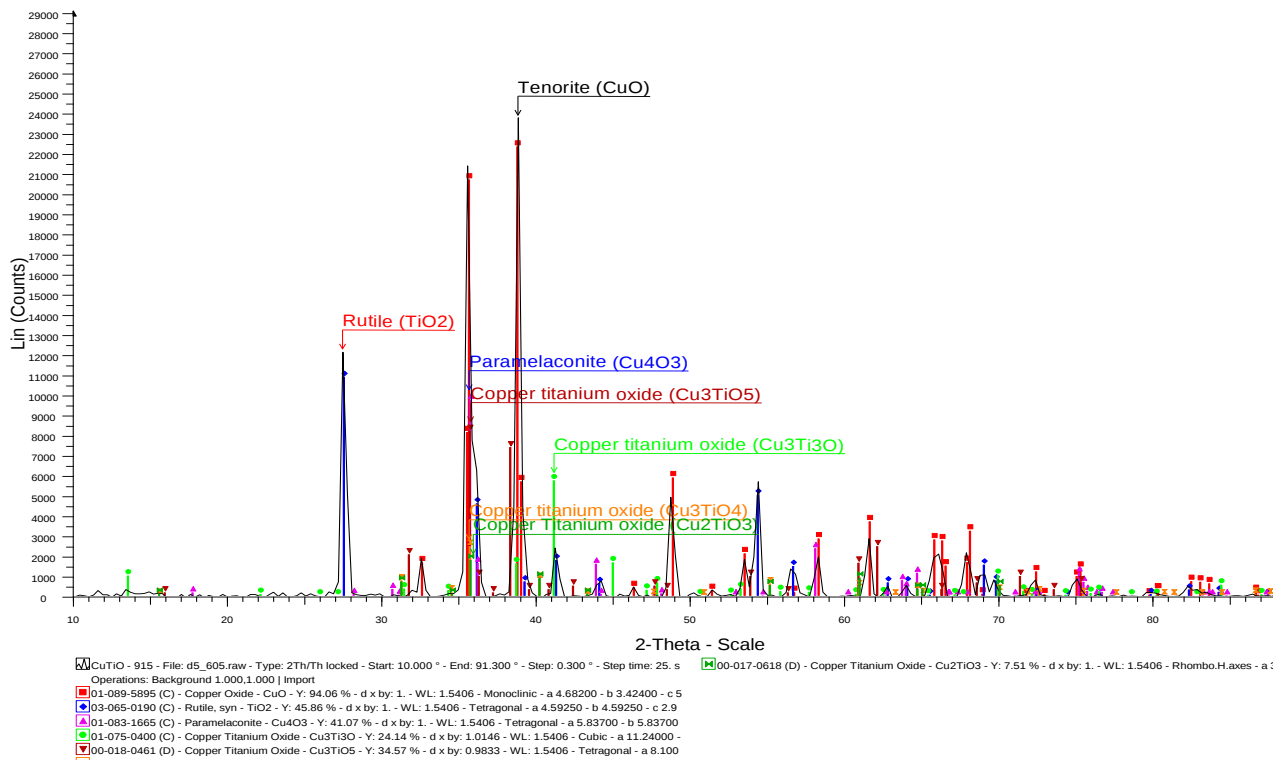


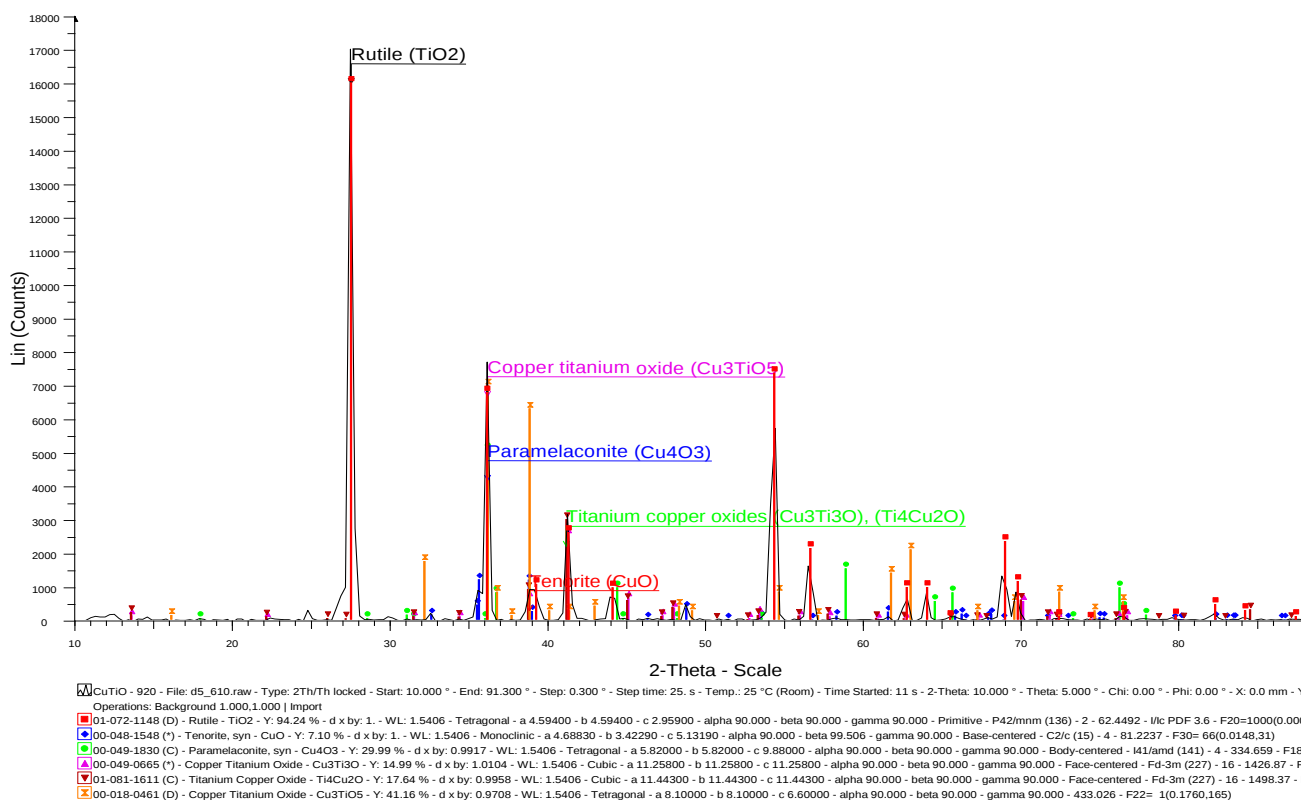
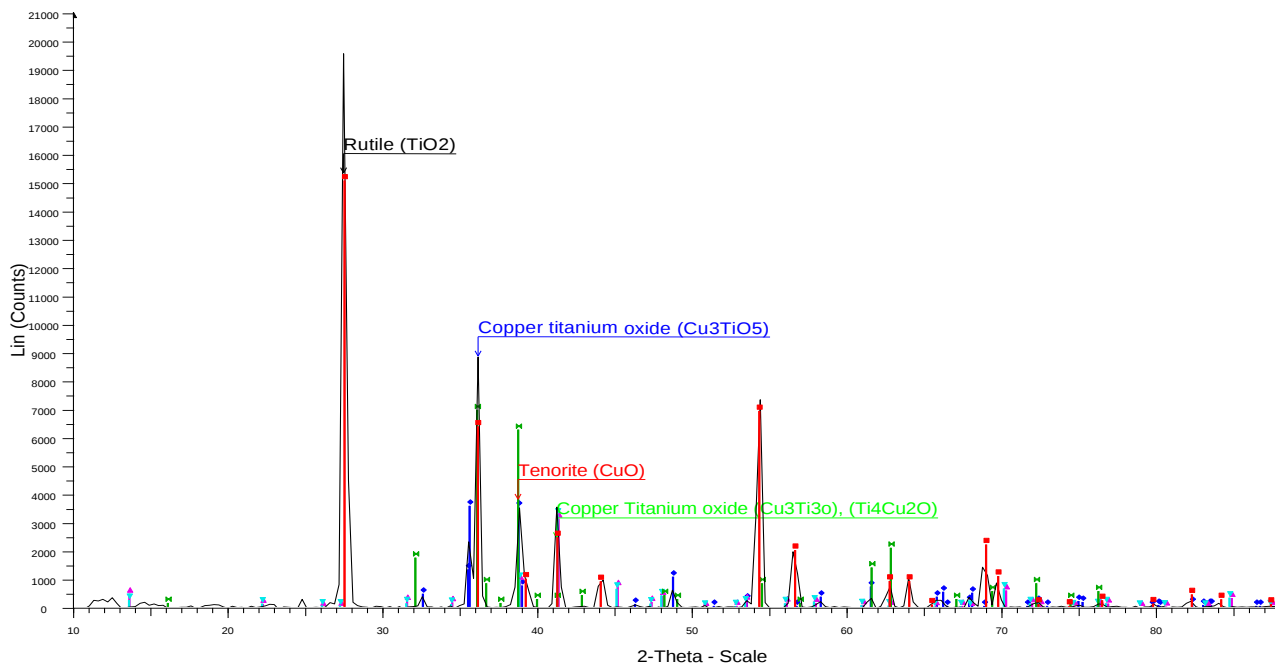


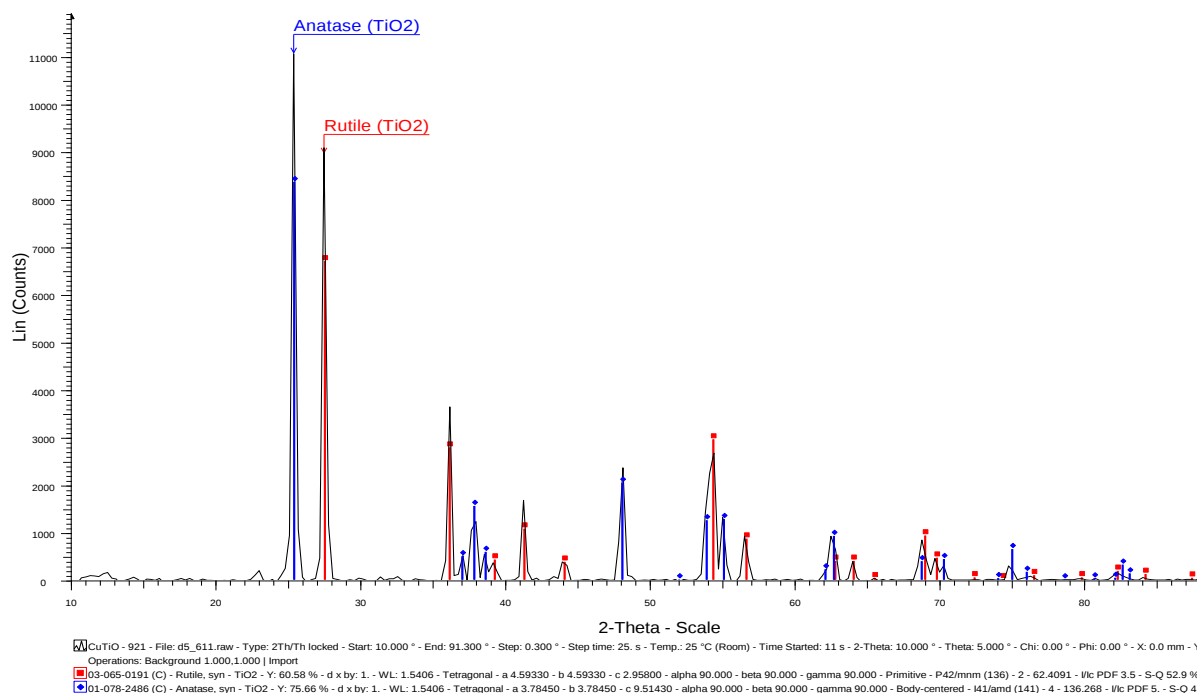












Appendix G

Particle size analysis data of mixed metal oxides nanopowders

Sample Composition	Phase	Non-calcined	300 °C	500 °C	700 °C	900 °C
	Tenorite (CuO)	6	8	8	9	10
	Amorphous	45				
	Copper (ii) oxide					
	Cuprite (Cu ₂ O)	48				
	Pure Copper					
	Paramelaconite (Cu ₄ O ₃)	4	9	10	19	

TABLE 4.1: PARTICLE SIZE ANALYSIS DATA FOR CRYSTALLOGRAPHIC PHASES IN THE PRECURSOR POWDER SAMPLE OF COMPOSITION 0 % TI: 100 % Cu .

Sample Composition	Phase	Non-calcined	300 °C	500 °C	700 °C	900 °C
5 % Ti: 95 % Cu						
	Tenorite (CuO)	7	8	40	45	45
	Cuprite (Cu ₂ O)	18	48			
	Paramelaconite (Cu ₄ O ₃)	4	7	17	18	
	Anatase	8	11	11	42	
	Rutile				3	4
	Copper titanium oxide (Cu ₃ TiO ₅)	5	8	8	19	

TABLE 4.2: PARTICLE SIZE ANALYSIS DATA FOR CRYSTALLOGRAPHIC PHASES IN THE PRECURSOR POWDER SAMPLE OF COMPOSITION 5 % Ti: 95 % Cu.

Sample Composition	Phase	Non-calcined	300 °C	500 °C	700 °C	900 °C
10 % Ti: 90 % Cu						
	Tenorite (CuO)	6	7	18	19	45
	Paramelaconite (Cu ₄ O ₃)	4	8	17	17	
	Anatase	11	11	41	42	
	Rutile		10			17
	Copper titanium oxide (Cu ₃ TiO ₄)	6		6	7	
	Copper titanium oxide (Cu ₃ TiO ₅)	6	7	7	10	

TABLE 4.3: PARTICLE SIZE ANALYSIS DATA FOR CRYSTALLOGRAPHIC PHASES IN THE PRECURSOR POWDER SAMPLE OF COMPOSITION 10 % Ti: 90 % Cu.

Sample Composition	Phase	Non-calcined	300 °C	500 °C	700 °C	900 °C
15 % Ti: 85 % Cu						
	Tenorite (CuO)	7	8	11	17	44
	Paramelaconite (Cu ₄ O ₃)	8	8	11	17	
	Anatase	7	16	41	42	
	Rutile				8	17
	Copper titanium oxide (Cu ₃ TiO ₄)	8	8	11		
	Copper titanium oxide (Cu ₃ TiO ₅)	7	11	11	11	

TABLE 4.4: PARTICLE SIZE ANALYSIS DATA FOR CRYSTALLOGRAPHIC PHASES IN THE PRECURSOR POWDER SAMPLE OF COMPOSITION 15 % Ti: 85 % Cu.

Sample Composition	Phase	Non-calcined	300 °C	500 °C	700 °C	900 °C
20 % Ti: 80 % Cu						
	Tenorite (CuO)	7	7	10	17	20
	Paramelaconite (Cu ₄ O ₃)	8	17	17	19	
	Anatase	7	11	42	42	
	Rutile				17	10
	Copper titanium oxide (Cu ₃ TiO ₄)	7	8	10	10	
	Copper titanium oxide (Cu ₃ TiO ₅)	5	6	7	7	

TABLE 4.5: PARTICLE SIZE ANALYSIS DATA FOR CRYSTALLOGRAPHIC PHASES IN THE PRECURSOR POWDER SAMPLE OF COMPOSITION 20 % Ti: 80 % Cu.

Sample Composition	Phase	Non-calcined	300 °C	500 °C	700 °C	900 °C
25 % Ti: 75 % Cu						
	Tenorite (CuO)	6	6	7	8	8
	Paramelaconite (Cu ₄ O ₃)	7	8	17	18	
	Anatase	8	10	11	17	
	Rutile	4	12	17	42	42
	Copper titanium oxide (Cu ₃ TiO ₄)	4	6	17		
	Copper titanium oxide (Cu ₃ TiO ₅)	5	5	6	8	
	Unknown Cu _x Ti _y O _z	17	17			

TABLE 4.6: PARTICLE SIZE ANALYSIS DATA FOR CRYSTALLOGRAPHIC PHASES IN THE PRECURSOR POWDER SAMPLE OF COMPOSITION 25 % Ti: 75 % Cu.

Sample Composition	Phase	Non-calcined	300 °C	500 °C	700 °C	900 °C
30 % Ti: 70 % Cu						
	Tenorite (CuO)	7	8	43	43	43
	Paramelaconite (Cu ₄ O ₃)	8	17	18	19	
	Anatase	3	7	8	11	17
	Rutile				42	42
	Copper titanium oxide (Cu ₃ TiO ₄)	2	8	10		
	Copper titanium oxide (Cu ₃ TiO ₅)	5	8	10	20	
	Unknown Cu _x Ti _y O _z	11	17			

TABLE 4.7: PARTICLE SIZE ANALYSIS DATA FOR CRYSTALLOGRAPHIC PHASES IN THE PRECURSOR POWDER SAMPLE OF COMPOSITION 30 % Ti: 70 % Cu.

Sample Composition	Phase	Non-calcined	300 °C	500 °C	700 °C	900 °C
35 % Ti: 65 % Cu						
	Tenorite (CuO)	17	43	43	43	43
	Pure Copper	1				
	Paramelaconite (Cu ₄ O ₃)	7	8	11	17	17
	Anatase	11	11	17	17	
	Rutile			17	42	42
	Copper titanium oxide (Cu ₃ TiO ₄)	7	11	12	18	43
	Copper titanium oxide (Cu ₃ TiO ₅)	5	6	8	10	
	Unknown Cu _x Ti _y O _z	7	10			

TABLE 4.8: PARTICLE SIZE ANALYSIS DATA FOR CRYSTALLOGRAPHIC PHASES IN THE PRECURSOR POWDER SAMPLE OF COMPOSITION 35 % Ti: 65 % Cu.

Sample Composition	Phase	Non-calcined	300 °C	500 °C	700 °C	900 °C
40 % Ti: 60 % Cu						
	Tenorite (CuO)	11	40	43	43	43
	Paramelaconite (Cu ₄ O ₃)	10	11	17	19	
	Anatase	8	11	11	17	
	Rutile					42
	Copper titanium oxide (Cu ₃ TiO ₄)	3	7	10	17	42
	Copper titanium oxide (Cu ₃ TiO ₅)	6	8	8	8	
	Unknown Cu _x Ti _y O _z	8	17			

TABLE 4.9: PARTICLE SIZE ANALYSIS DATA FOR CRYSTALLOGRAPHIC PHASES IN THE PRECURSOR POWDER SAMPLE OF COMPOSITION 40 % Ti: 60 % Cu.

Sample Composition	Phase	Non-calcined	300 °C	500 °C	700 °C	900 °C
45 % Ti: 55 % Cu						
	Tenorite (CuO)	41	41	43	43	43
	Paramelaconite (Cu ₄ O ₃)	11	17	18	19	
	Anatase	11	17	17	17	
	Rutile		8	17	42	42
	Copper titanium oxide (Cu ₃ TiO ₄)	5	6	8	12	42
	Copper titanium oxide (Cu ₃ TiO ₅)	5	6	8	43	
	Unknown Cu _x Ti _y O _z	7	11			

TABLE 4.10: PARTICLE SIZE ANALYSIS DATA FOR CRYSTALLOGRAPHIC PHASES IN THE PRECURSOR POWDER SAMPLE OF COMPOSITION 45 % Ti: 55 % Cu.

Sample Composition	Phase	Non-calcined	300 °C	500 °C	700 °C	900 °C
50 % Ti: 50 % Cu						
	Tenorite (CuO)	11	11	42	42	42
	Paramelaconite (Cu ₄ O ₃)	11	16	17	19	
	Anatase	11	11	17	17	
	Rutile					42
	Copper titanium oxide (Cu ₃ TiO ₄)	5	11	12		42
	Copper titanium oxide (Cu ₃ TiO ₅)	7	7	8	42	
	Unknown Cu _x Ti _y O _z	17	17			

TABLE 4.11: PARTICLE SIZE ANALYSIS DATA FOR CRYSTALLOGRAPHIC PHASES IN THE PRECURSOR POWDER SAMPLE OF COMPOSITION 50 % Ti: 50 % Cu.

Sample Composition	Phase	Non-calcined	300 °C	500 °C	700 °C	900 °C
55 % Ti: 45 % Cu						
	Tenorite (CuO)	11	17	17	42	42
	Paramelaconite (Cu ₄ O ₃)	17	17	17	17	
	Anatase	11	17	17	17	
	Rutile					42
	Copper titanium oxide (Cu ₃ TiO ₄)	4	11	11	18	42
	Copper titanium oxide (Cu ₃ TiO ₅)	7	7	10	11	
	Unknown TiO ₂ complex	8	11			
	Unknown Cu _x Ti _y O _z	8	17			

TABLE 4.12: PARTICLE SIZE ANALYSIS DATA FOR CRYSTALLOGRAPHIC PHASES IN THE PRECURSOR POWDER SAMPLE OF COMPOSITION 55 % Ti: 45 % Cu.

Sample Composition	Phase	Non-calcined	300 °C	500 °C	700 °C	900 °C
60 % Ti: 40 % Cu						
	Tenorite (CuO)	10	11	11	17	43
	Paramelaconite (Cu ₄ O ₃)	11	11	11	17	17
	Anatase	11	11	11	17	17
	Rutile					42
	Copper titanium oxide (Cu ₃ TiO ₄)	5	9	11	11	16
	Copper titanium oxide (Cu ₃ TiO ₅)	7	11	11	19	
	Unknown TiO ₂ complex	11	17			
	Unknown Cu _x Ti _y O _z	11	16			

TABLE 4.13: PARTICLE SIZE ANALYSIS DATA FOR CRYSTALLOGRAPHIC PHASES IN THE PRECURSOR POWDER SAMPLE OF COMPOSITION 60 % Ti: 40 % Cu.

Sample Composition	Phase	Non-calcined	300 °C	500 °C	700 °C	900 °C
65 % Ti: 35 % Cu						
	Tenorite (CuO)	11	11	42	42	43
	Paramelaconite (Cu ₄ O ₃)	10	11	16	17	17
	Anatase	11	11	17	17	
	Rutile			17	42	42
	Copper titanium oxide (Cu ₃ TiO ₄)	4	6	10	11	11
	Copper titanium oxide (Cu ₃ TiO ₅)	4	4	6	10	
	Unknown TiO ₂ complex	10	18			
	Unknown CuO complex	17	17			

TABLE 4.14: PARTICLE SIZE ANALYSIS DATA FOR CRYSTALLOGRAPHIC PHASES IN THE PRECURSOR POWDER SAMPLE OF COMPOSITION 65 % Ti: 35 % Cu.

Sample Composition	Phase	Non-calcined	300 °C	500 °C	700 °C	900 °C
70 % Ti: 30 % Cu						
	Tenorite (CuO)	11	11	42	42	43
	Paramelaconite (Cu ₄ O ₃)	6	11	16	17	17
	Anatase	6	11	11	17	
	Rutile					42
	Copper titanium oxide (Cu ₃ TiO ₄)	8	8	8	10	10
	Copper titanium oxide (Cu ₃ TiO ₅)	8	8	8	8	11
	Unknown TiO ₂ complex	8	17			
	Unknown CuO complex	17	17			

TABLE 4.15: PARTICLE SIZE ANALYSIS DATA FOR CRYSTALLOGRAPHIC PHASES IN THE PRECURSOR POWDER SAMPLE OF COMPOSITION 70 % Ti: 30 % Cu.

Sample Composition	Phase	Non-calcined	300 °C	500 °C	700 °C	900 °C
75 % Ti: 25 % Cu						
	Tenorite (CuO)	17	17	42	42	42
	Paramelaconite (Cu ₄ O ₃)		9	11	17	17
	Anatase	11	17	17	17	
	Rutile					43
	Copper titanium oxide (Cu ₃ TiO ₄)	6	7	7	8	9
	Copper titanium oxide (Cu ₃ TiO ₅)	6	7	7	8	18
	Unknown TiO ₂ complex	8	11			
	Unknown CuO complex	17	17			

TABLE 4.16: PARTICLE SIZE ANALYSIS DATA FOR CRYSTALLOGRAPHIC PHASES IN THE PRECURSOR POWDER SAMPLE OF COMPOSITION 75 % Ti: 25 % Cu.

Sample Composition	Phase	Non-calcined	300 °C	500 °C	700 °C	900 °C
80 % Ti: 20 % Cu						
	Tenorite (CuO)	8	11	11	42	42
	Paramelaconite (Cu ₄ O ₃)			11	12	12
	Anatase	8	11	11	17	-
	Rutile					42
	Copper titanium oxide (Cu ₃ TiO ₄)	6	6	6	11	18
	Copper titanium oxide (Cu ₃ TiO ₅)	5	6	6	17	17
	Unknown CuO complex	10	17			

TABLE 4.17: PARTICLE SIZE ANALYSIS DATA FOR CRYSTALLOGRAPHIC PHASES IN THE PRECURSOR POWDER SAMPLE OF COMPOSITION 80 % Ti: 20 % Cu.

Sample Composition	Phase	Non-calcined	300 °C	500 °C	700 °C	900 °C
85 % Ti: 15 % Cu						
	Tenorite (CuO)	11	17	42	42	42
	Paramelaconite (Cu ₄ O ₃)			11	17	17
	Anatase	17	11	17	17	
	Rutile					42
	Copper titanium oxide (Cu ₃ TiO ₄)		4	6	8	18
	Copper titanium oxide (Cu ₃ TiO ₅)	5	6	7	8	8
	Unknown (Cu _x Ti _y O _z)	8	17			

TABLE 4.18: PARTICLE SIZE ANALYSIS DATA FOR CRYSTALLOGRAPHIC PHASES IN THE PRECURSOR POWDER SAMPLE OF COMPOSITION 85 % Ti: 15 % Cu.

Sample Composition	Phase	Non-calcined	300 °C	500 °C	700 °C	900 °C
90 % Ti: 10 % Cu						
	Tenorite (CuO)	17	17	42	42	42
	Paramelaconite (Cu ₄ O ₃)				17	18
	Anatase	17	17	17	17	
	Rutile			10		17

TABLE 4.19: PARTICLE SIZE ANALYSIS DATA FOR CRYSTALLOGRAPHIC PHASES IN THE PRECURSOR POWDER SAMPLE OF COMPOSITION 90 % Ti: 10 % Cu.

Sample Composition	Phase	Non-calcined	300 °C	500 °C	700 °C	900 °C
95 % Ti: 5 % Cu		17	17	42	42	43
	Anatase	17	17	17	17	
	Rutile		10	17	42	43

TABLE 4.20: PARTICLE SIZE ANALYSIS DATA FOR CRYSTALLOGRAPHIC PHASES IN THE PRECURSOR POWDER SAMPLE OF COMPOSITION 95 % Ti: 5 % Cu.

Sample Composition	Phase	Non-calcined	300 °C	500 °C	700 °C	900 °C
100 % Ti: 0 % Cu	Anatase	6	6	11	17	41
	Rutile					43

TABLE 4.21: PARTICLE SIZE ANALYSIS DATA FOR CRYSTALLOGRAPHIC PHASES IN THE PRECURSOR POWDER SAMPLE OF COMPOSITION 100 % Ti: 0 % Cu.