



## United States Department of the Interior

BUREAU OF LAND MANAGEMENT  
National Minerals Testing Laboratory  
C/O Wind River/Bighorn Basin District  
101 S. 23<sup>rd</sup> Street  
Worland, Wyoming 82401



APR 28 2017

To: Shelby Cave, Hassayampa Field Office

From: Brent S. Bestram and Marilyn Wegweiser

Subject: X-ray Diffraction and Scanning Electron Microscope Test Results

X-ray diffraction (XRD) and a scanning electron microscope (SEM) were used to determine the mineralogical composition of eight samples with an emphasis being placed on the identification of erionite. Seven of the samples came from the Kirkland Quarry and one from a Highway 89 road cut according to information submitted with the samples. X-ray diffraction was accomplished by using a Rigaku Miniflex 600 diffractometer fitted with a copper tube. A Tescan VEGA 3 LMU scanning electron microscope was used to image the samples and look for erionite.

About 1 gram of sample is ground up in a sapphire-diamonite mortar and pestle until it has the consistency of talc. It is then packed into an aluminum sample holder and placed on the diffractometer for analysis. About 0.70 grams is needed to pack the sample holder. The parameters for the samples run are shown at the top of each scan. As seen on Scan-1 the first item given is the sample number, *PDO-SC-001*, followed by a note *KQ-sfc tuff base of unit*. Given parameters for the samples run are *3.0/56.0/0.02/2(sec)*, *Cu(40kV,15mA, I(p)=2502*, date tested and time that the scan was finished. The sample range was run from 3° to 56° Two-Theta (2θ). Scanning took place at step of 0.02° with a dwell of 2 seconds. In other words, the sample was rotated 0.02 hundredths of a degree every 2 seconds. Cu means the diffractometer is fitted with a copper tube and operates on the voltages and amps indicated. The *I(p)=2502* denotes the highest intensity peak on the scan. The date tested and scan time completion are the last two items shown. The peak line marker with the little circle on top indicates the highest intensity peak or 100% reflection for the minerals identified.

The primary mineral phases common to samples PDO-SC-001 through 007 (XRD Scans 1-7) are quartz, the feldspar group (includes the plagioclases) and clay from the smectite family that is predominately montmorillonite. A secondary mineral phase present in Scans 1-7 is a mica group mineral to which no specific mineral could be named with any degree of certainty. This is possibly due to the many polymorphs in which the mica clay minerals can occur. Specific minerals within the feldspar group are difficult to identify even if they occur in significant amounts due to their triclinic or monoclinic symmetry or a mixture of the two. Furthermore, the fact that they form solid solution series and can have numerous substitutions within the crystal lattice generates complex diffraction patterns. The blue line marker on Scans 1-7 designates where erionite peaks would occur if present in the sample. No erionite occurs in Scans 1-7.

In Scans 1, 2, 4, and 6, three peaks were identified as being consistent cristobalite. Although it would not be unreasonable to expect cristobalite occurrence in a tuff, the three peak hits are not conclusive as to its presence. A zeolite, known as chabazite, was definitely identified on Scan-6 in sample PDO-SC-006. Sufficient hits identified the feldspar group mineral albite on Scan-7 in sample PDO-SC-007. Returning to Scan-1, there is a broad bulge, if one ignores the sharp peaks, starting at about  $18^{\circ} 2\theta$  and ending at about  $29^{\circ} 2\theta$ . This is indicative of an amorphous substance that in this case is probably volcanic glass.

Scans 1A, 2A and 4A-7A definitely show the presence of an expandable clay of the smectite family. The smectites have a strong affinity for ethylene glycol (EG) which is adsorbed by the clay at the (001) face in the interlayer position causing a fixed expansion of the unit cell in the c-axis direction. Numbers shown in parentheses on the previously mentioned scans are Miller Indices that define the orientation of a crystal face or an internal crystal plane. Each of the "A" designated samples were placed in a desiccator containing an EG atmosphere for a minimum of 24 hours and x-rayed again. In each of the scans mentioned above, the air-dried sample was enhanced by the EG and moved to the left by expansion of the (001) face. As an example, Scan-1 is the air-dried sample and Scan-1A is the EG sample. In comparing the air dried sample to the glycolated sample, it is seen that the (001) reflection has increased from  $15.02 \text{ \AA}$  to  $17.38 \text{ \AA}$ . The d-spacing given in angstroms ( $\text{\AA}$ ) is equal to  $10^{-8}$  centimeters and is a measurement between atoms or planes of atoms. Conversely, the  $2\theta$  angle decreased from  $5.880^{\circ}$  to  $5.079^{\circ}$ .

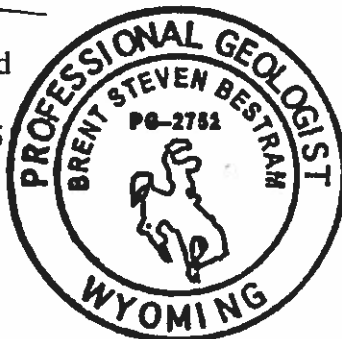
Scan-8, which is the highway 89 road cut, is straightforward. Three mineral phases are contained in sample PDO-SC-008, clinoptilolite, erionite and quartz. A secondary mineral phase is a mica group mineral but cannot be confirmed. Scan-9 is a compilation of scans 001-007 for easy comparison. A different perspective for comparing the seven scans is provided by the two enclosed 3D scans labeled as Scans 10 and 11. All of the 3D patterns shown on both scans have had the background noise removed.

Summarizing, samples 001-007 contained no erionite using x-ray diffraction. However, SEM imaging by Marilyn Wegweiser indicated a small amount of erionite in sample PDO-SC-005 at less than 2%. Considering only 0.7 grams of sample is used for x-ray diffraction, it is quite possible the sample did not contain any erionite or there was too little for the diffractometer to recognize. Marilyn's discussion on the results of the SEM imaging are attached.

If you have any questions regarding the test results, please give Marilyn Wegweiser or me a call at the laboratory, (307) 347-4135.



Brent S. Bestram, Geologist-retired  
Wyoming State Office  
Former CME #047 & CRME #025  
WY PG-2752



Enclosures

- 11 – XRD Scans
- 13 – SEM Images



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**MAY 02 2017**

To: Shelby Cave, Hassayampa Field Office

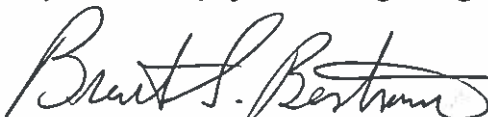
From: Brent S. Bestram

Subject: Asbestos Omission

The test results recently sent to you and dated April 28, 2017, unfortunately did not mention anything about asbestos. Judging from the literature at hand, it is my opinion that the name asbestos is a generic or nonprofessionals' term for a fibrous substance. Older names for asbestos are serpentine and chrysotile. Dana's New Mineralogy, 8<sup>th</sup> Edition (1997), identifies asbestos as clinochrysotile and provides a x-ray diffraction card numbered 00-022-1163. The JADE-9 x-ray diffraction software does not recognize asbestos as a mineral but does recognize the card number and brings up magnesium silicate hydroxide that is clinochrysotile.

The attached x-ray diffraction pattern compares clinochrysotile with sample PDO-SC-001. There is no match whatsoever. None of the eight samples you sent for analysis contains clinochrysotile.

If you have any questions regarding the above, please give me a call at the lab, (307) 347-4135.

  
Brent S. Bestram, Geologist-retired  
Wyoming State Office  
Former CME #047 & CRME #025  
WY PG-2752

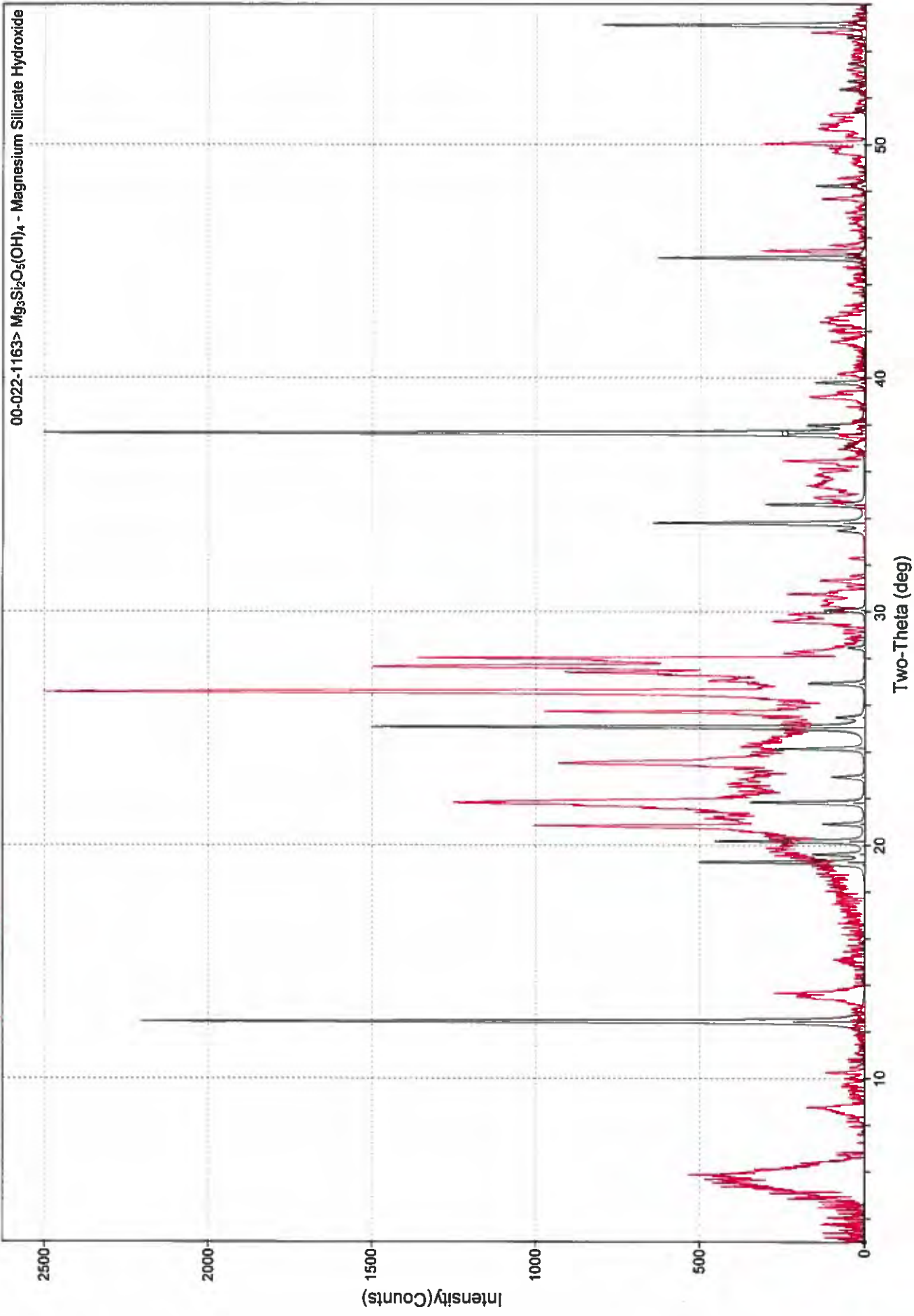


### Reference

Gaines, Richard V., Skinner, Catherine W., Foord, Eugene E., Mason, Brian and Rosenzweig, Abraham, 1997, Dana's New Mineralogy, 8<sup>th</sup> ed: John Wiley & Sons, Inc., pp.1428-1430.

Enclosure  
1-XRD Scan

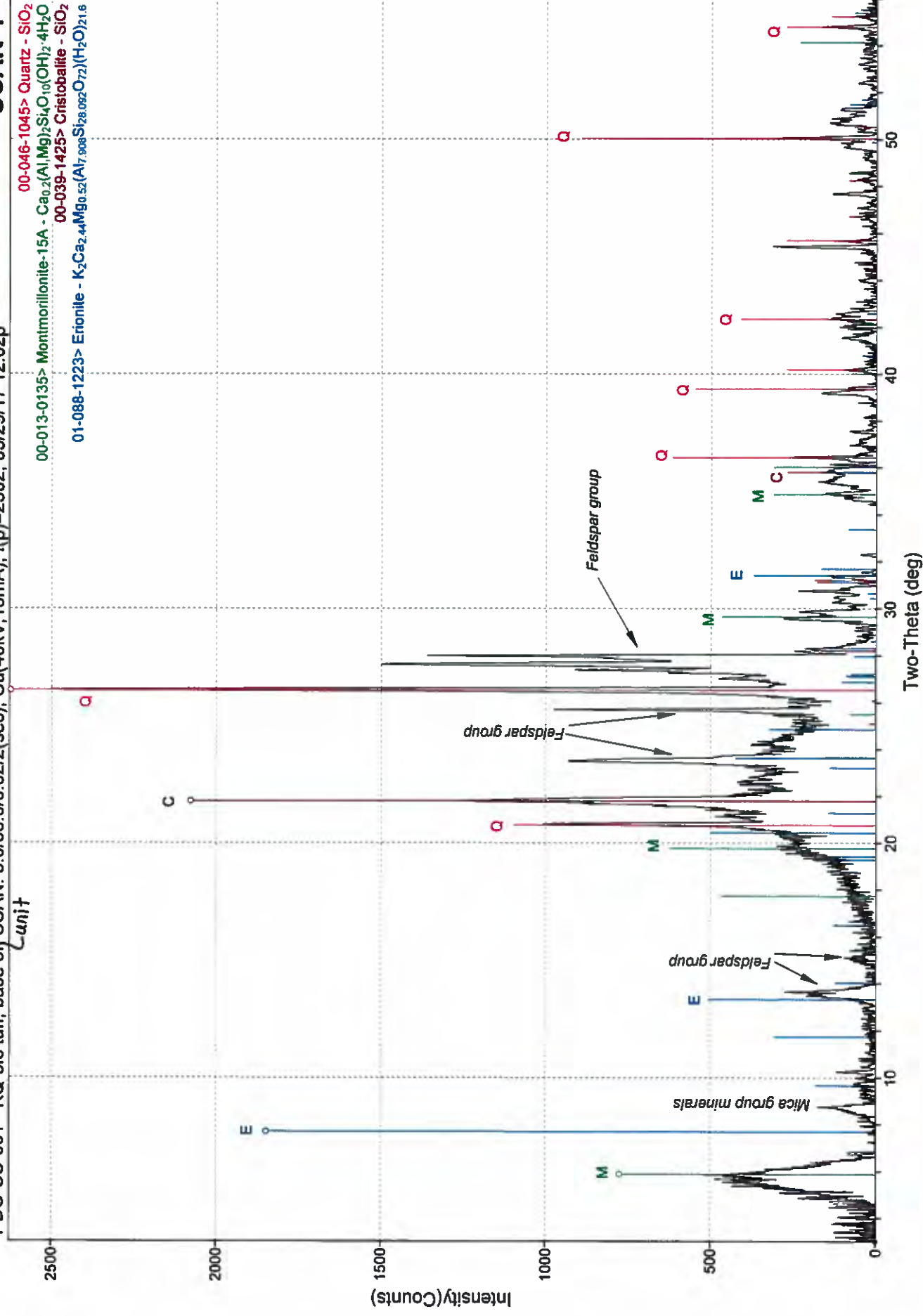
PDO-SC-001 - KQ-sfc tuff, base of unit



PDO-SC-001 - KQ-sfc tuff, base of, SCAN: 3.0/56.0/0.02/2(sec), Cu(40kV, 15mA), I(p)=2502, 03/29/17 12:02p

# SCAN-1

00-046-1045> Quartz - SiO<sub>2</sub>  
 00-013-0135> Montmorillonite-15A - Ca<sub>0.2</sub>(Al,Mg)<sub>2</sub>Si<sub>4</sub>O<sub>10</sub>(OH)<sub>2</sub>·4H<sub>2</sub>O  
 00-039-1425> Cristobalite - SiO<sub>2</sub>  
 01-088-1223> Erionite - K<sub>2</sub>Ca<sub>2.4</sub>Mg<sub>0.52</sub>(Al<sub>7.908</sub>Si<sub>28.092</sub>O<sub>72</sub>)(H<sub>2</sub>O)<sub>21.6</sub>



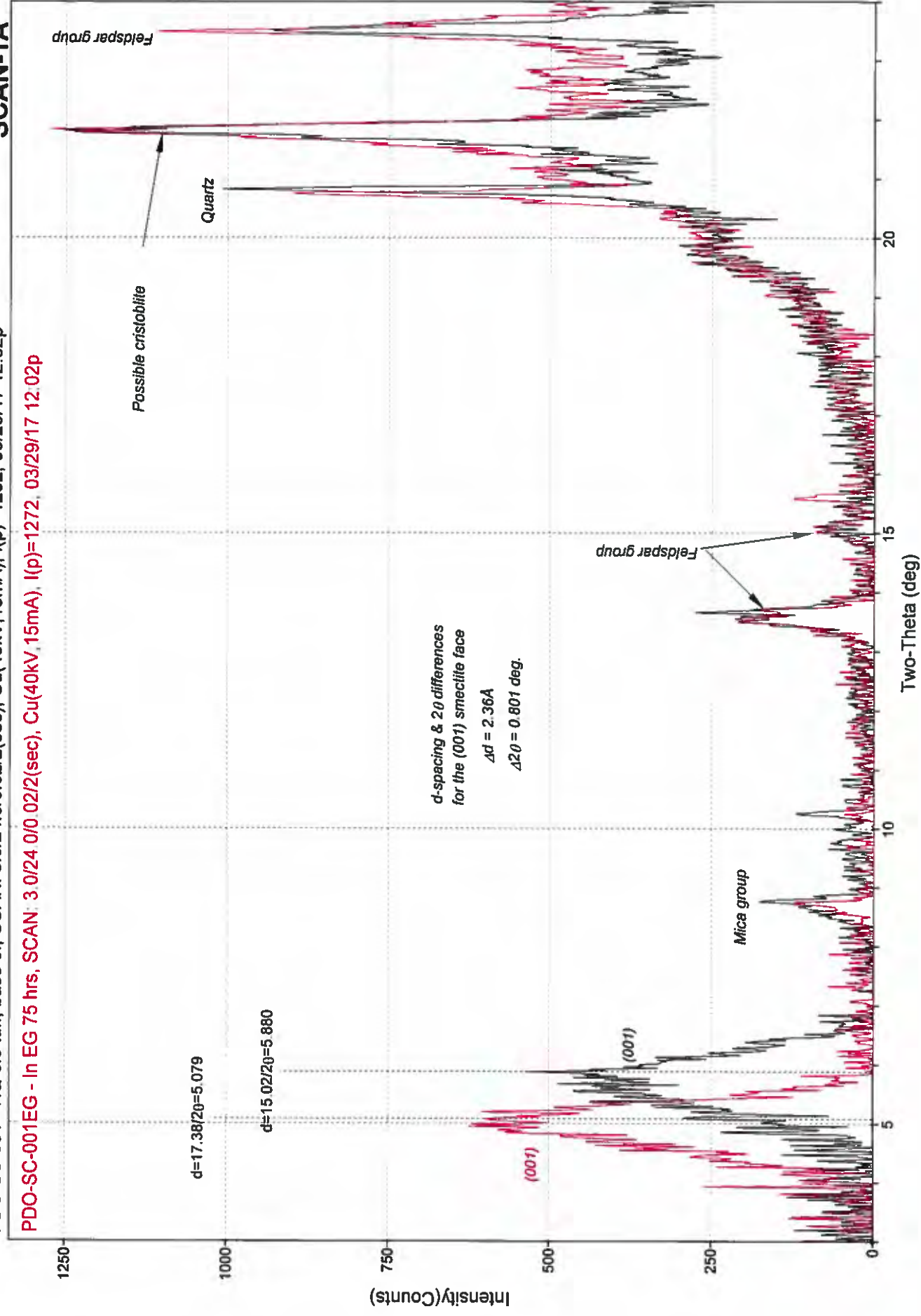


unit

PDO-SC-001 - KQ-sfc tuff, base of/SCAN: 3.0/24.0/0.02/2(sec), Cu(40kV,15mA), I(p)=1252, 03/29/17 12:02p

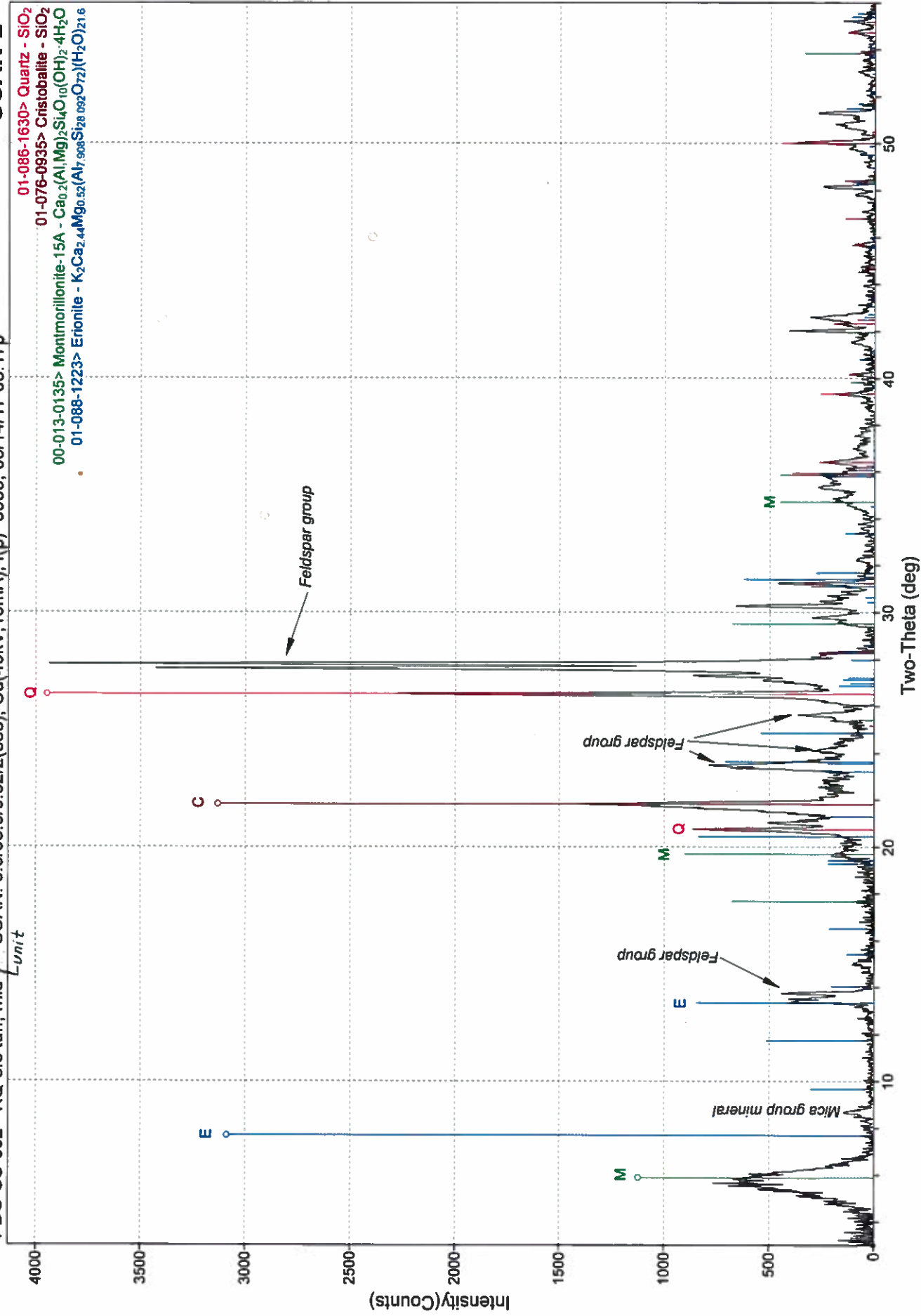
SCAN-1A

PDO-SC-001EG - In EG 75 hrs, SCAN: 3.0/24.0/0.02/2(sec), Cu(40kV,15mA), I(p)=1272, 03/29/17 12:02p



PDO-SC-002 - KQ-sfc tuft, mid 7 SCAN: 3.0/56.0/0.02/2(sec), Cu(40kV, 15mA), I(p)=3935, 03/14/17 03:17p

## SCAN-2



unit

# SCAN-2A

PDO-SC-002 - KQ-sfc tuff, mid SCAN: 3.0/23.0/0.02/2(sec), Cu(40kV, 15mA), I(p)=1360, 03/14/17 03:17p

PDO-SC-002EG - In EG 43 hrs, SCAN: 3.0/23.0/0.02/2(sec), Cu(40kV, 15mA), I(p)=1666, 03/16/17 01:28p

$d=17.73/2\theta=4.980$

$d=15.17/2\theta=5.821$

d-spacing & 2 $\theta$  differences  
for the (001) smectite face  
 $\Delta d = 2.56\text{\AA}$   
 $\Delta 2\theta = 0.841\text{ deg.}$

(001)

(001)

Feldspar group

Mica group mineral

Smectite or mica peak

Quartz

Possible cristobalite

Intensity(Counts)

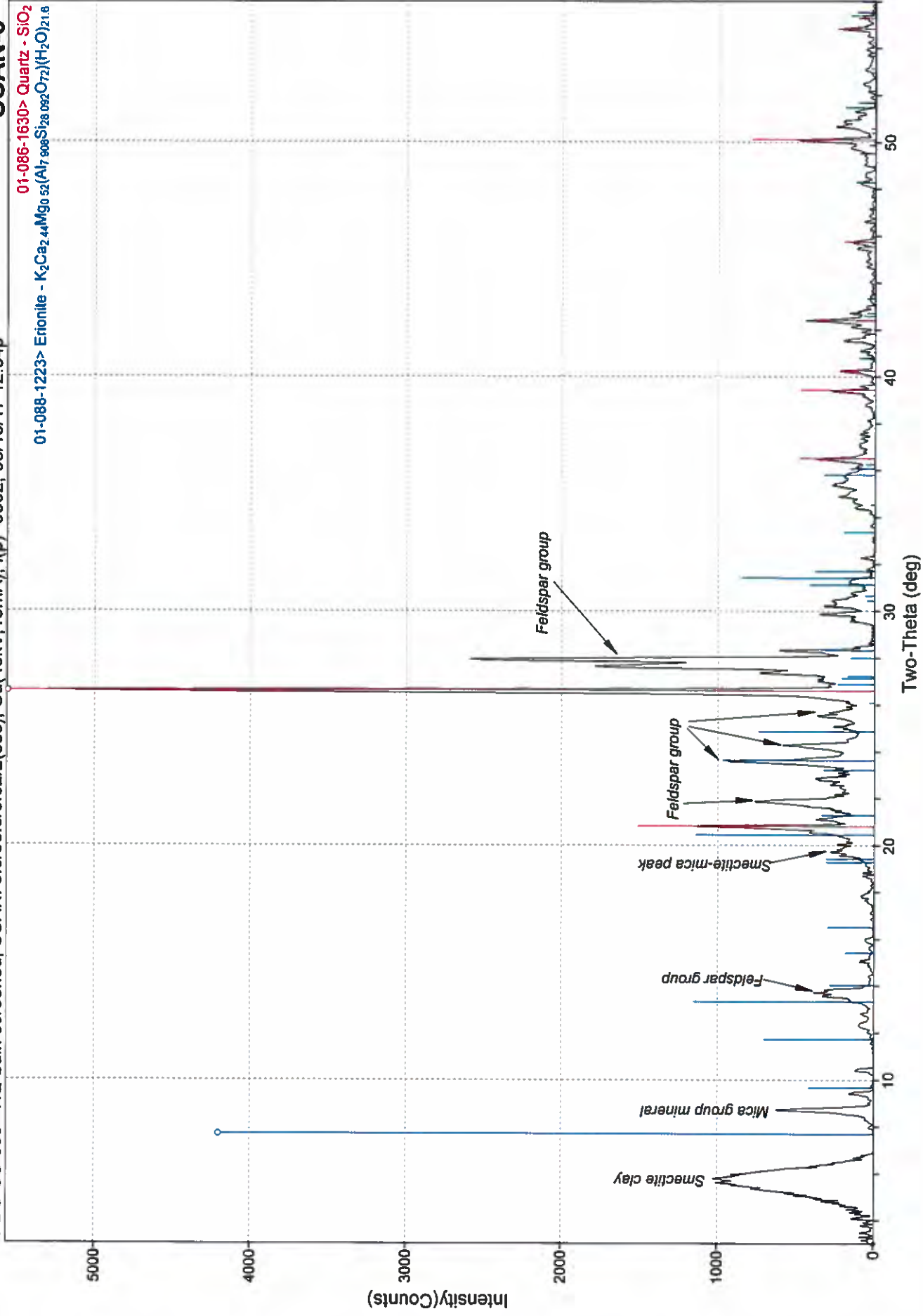
Two-Theta (deg)



PDO-SC-003 - KQ-bulk-screened, SCAN: 3.0/56.0/0.02/2(sec), Cu(40kV,15mA), I(p)=5302, 03/15/17 12:54p

## SCAN-3

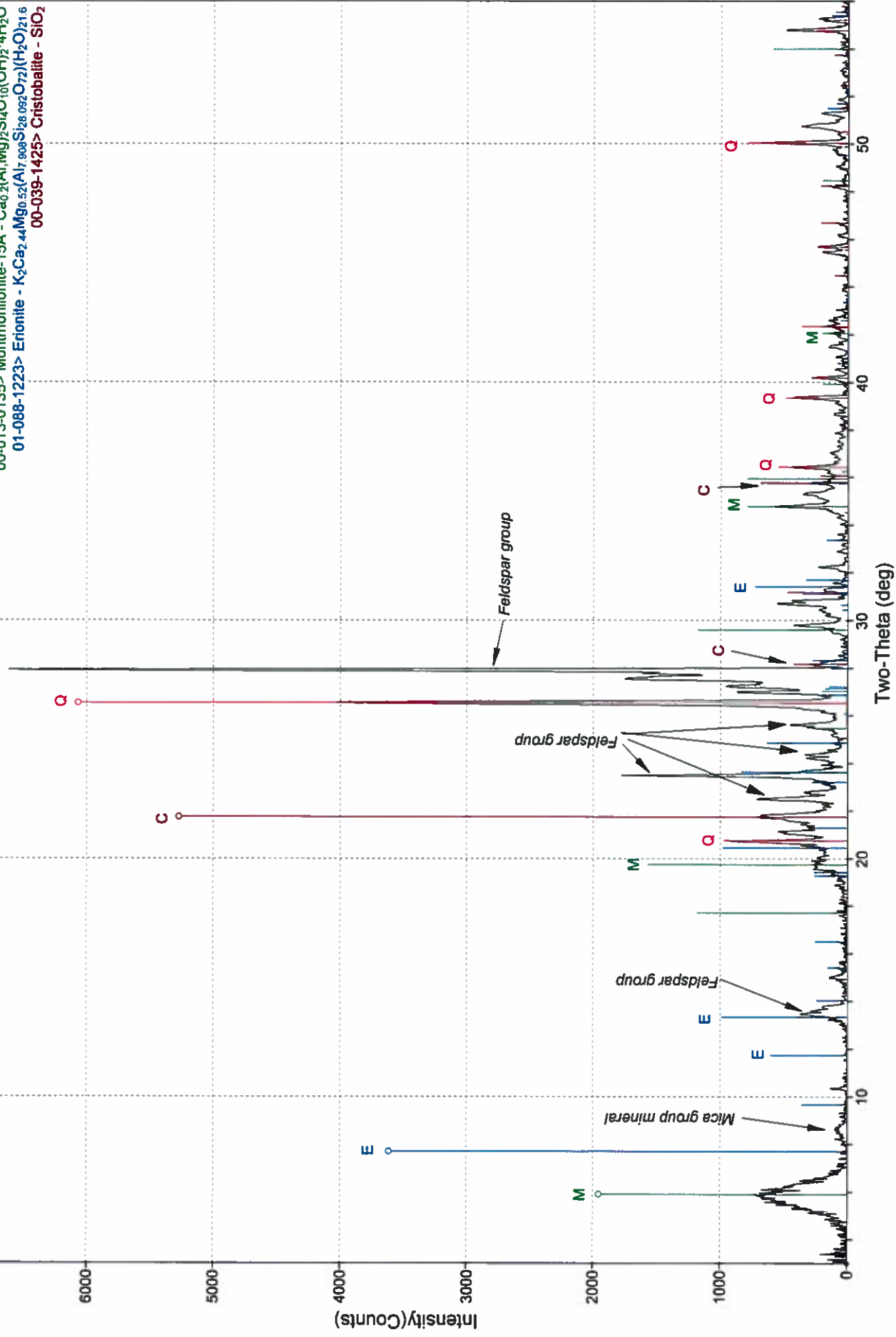
01-086-1630> Quartz -  $\text{SiO}_2$   
01-088-1223> Eriomite -  $\text{K}_2\text{Ca}_2\text{Mg}_{0.52}(\text{Al}_{7.908}\text{Si}_{28.092}\text{O}_{72})(\text{H}_2\text{O})_{21.8}$



PDO-SC-004 - KQ surge deposit, SCAN: 3.0/56.0/0.02/2(sec), Cu(40kV,15mA), I(p)=6614, 03/13/17 04:25p

## SCAN-4

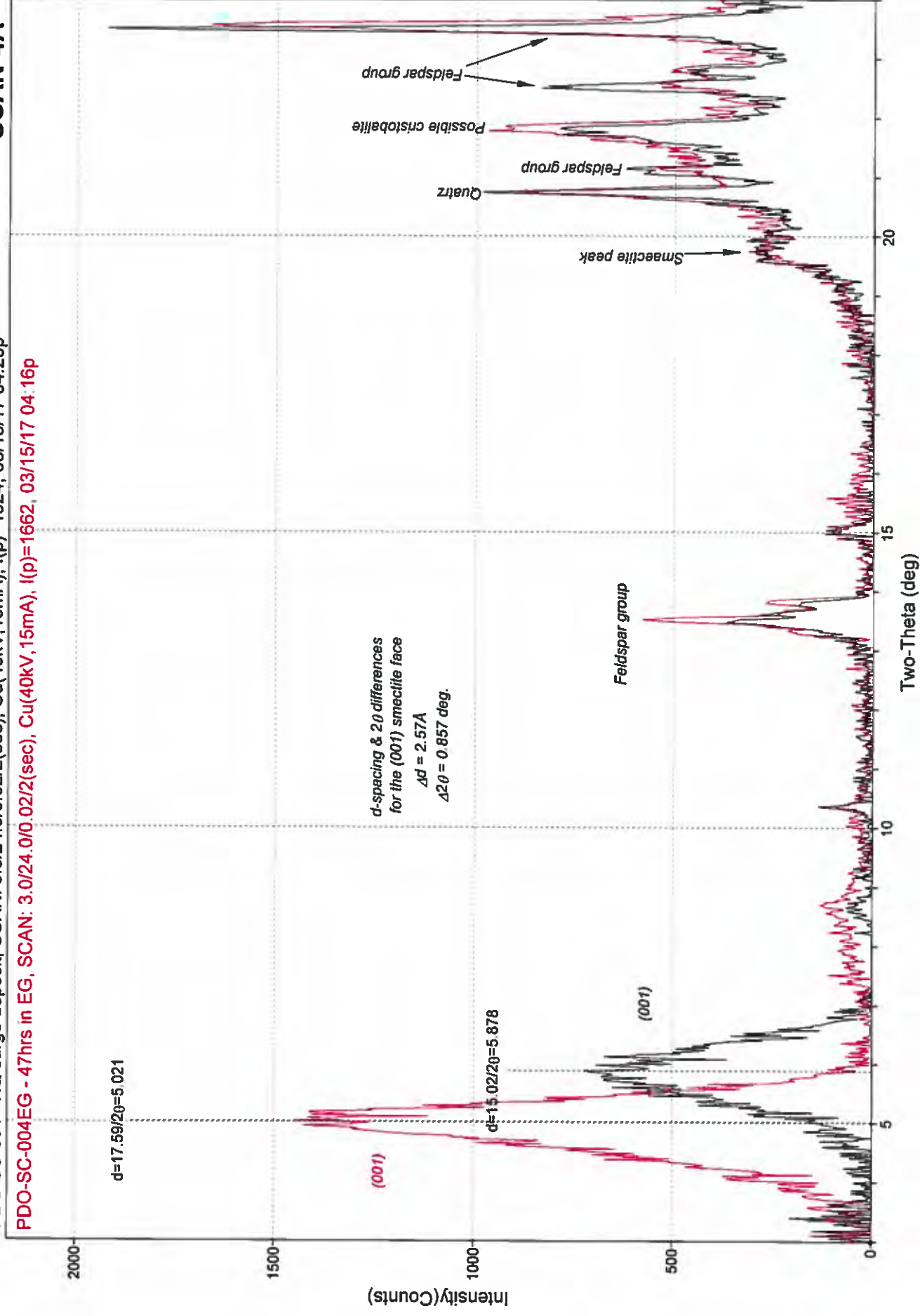
00-046-1045> Quartz -  $\text{SiO}_2$   
00-013-0135> Montmorillonite-15A -  $\text{Ca}_{0.2}(\text{Al,Mg})_2\text{Si}_4\text{O}_{10}(\text{OH})_2 \cdot 4\text{H}_2\text{O}$   
01-088-1223> Eriofonite -  $\text{K}_2\text{Ca}_2\text{Mg}_{0.52}(\text{Al}_{1.908}\text{Si}_{28.092}\text{O}_{72})(\text{H}_2\text{O})_{21.6}$   
00-039-1425> Cristobalite -  $\text{SiO}_2$



# SCAN-4A

PDO-SC-004 - KQ surge deposit, SCAN: 3.0/24.0/0.02/2(sec), Cu(40kV,15mA), I(p)=1924, 03/13/17 04:25p

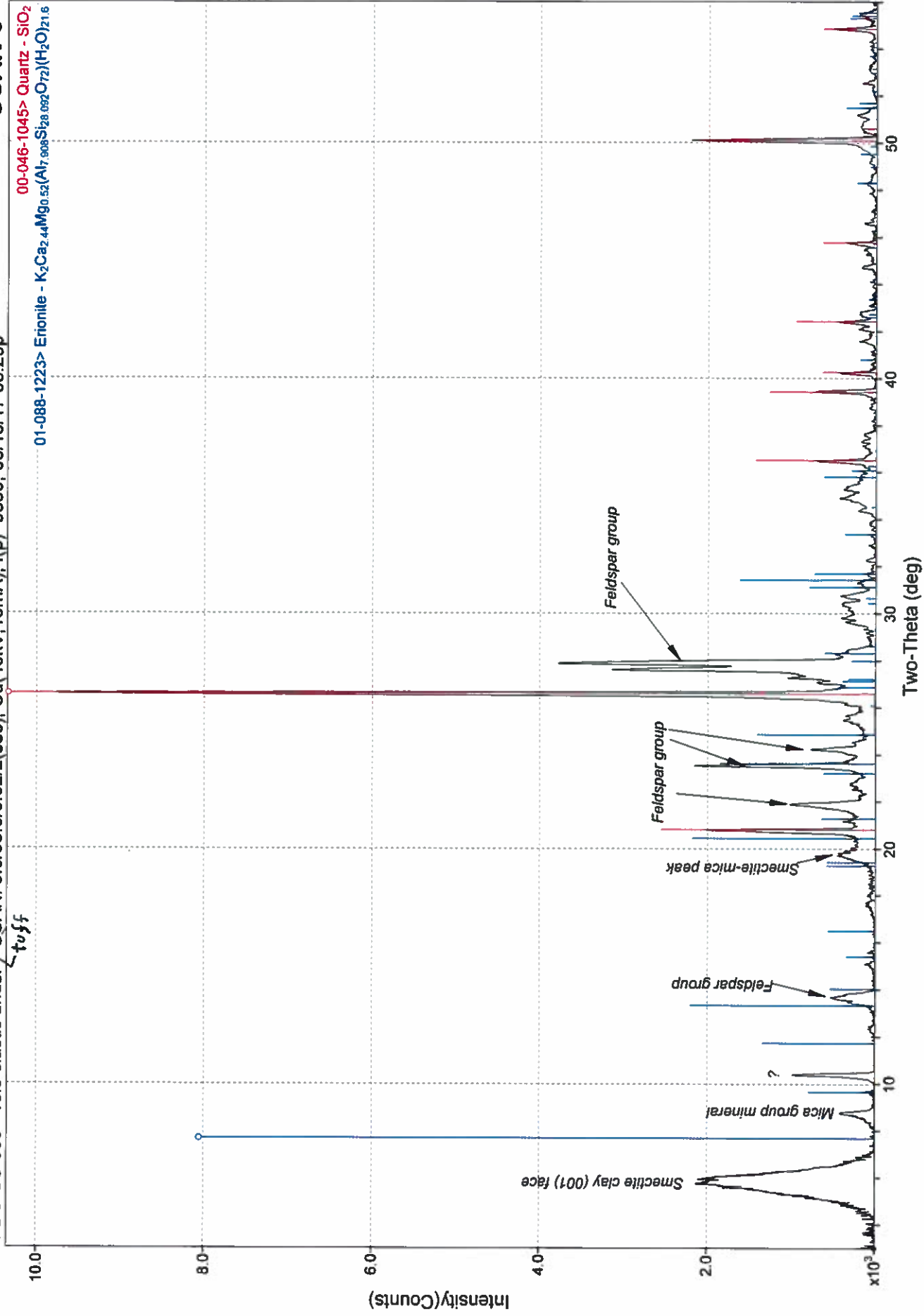
PDO-SC-004EG - 47hrs in EG, SCAN: 3.0/24.0/0.02/2(sec), Cu(40kV,15mA), I(p)=1662, 03/15/17 04:16p

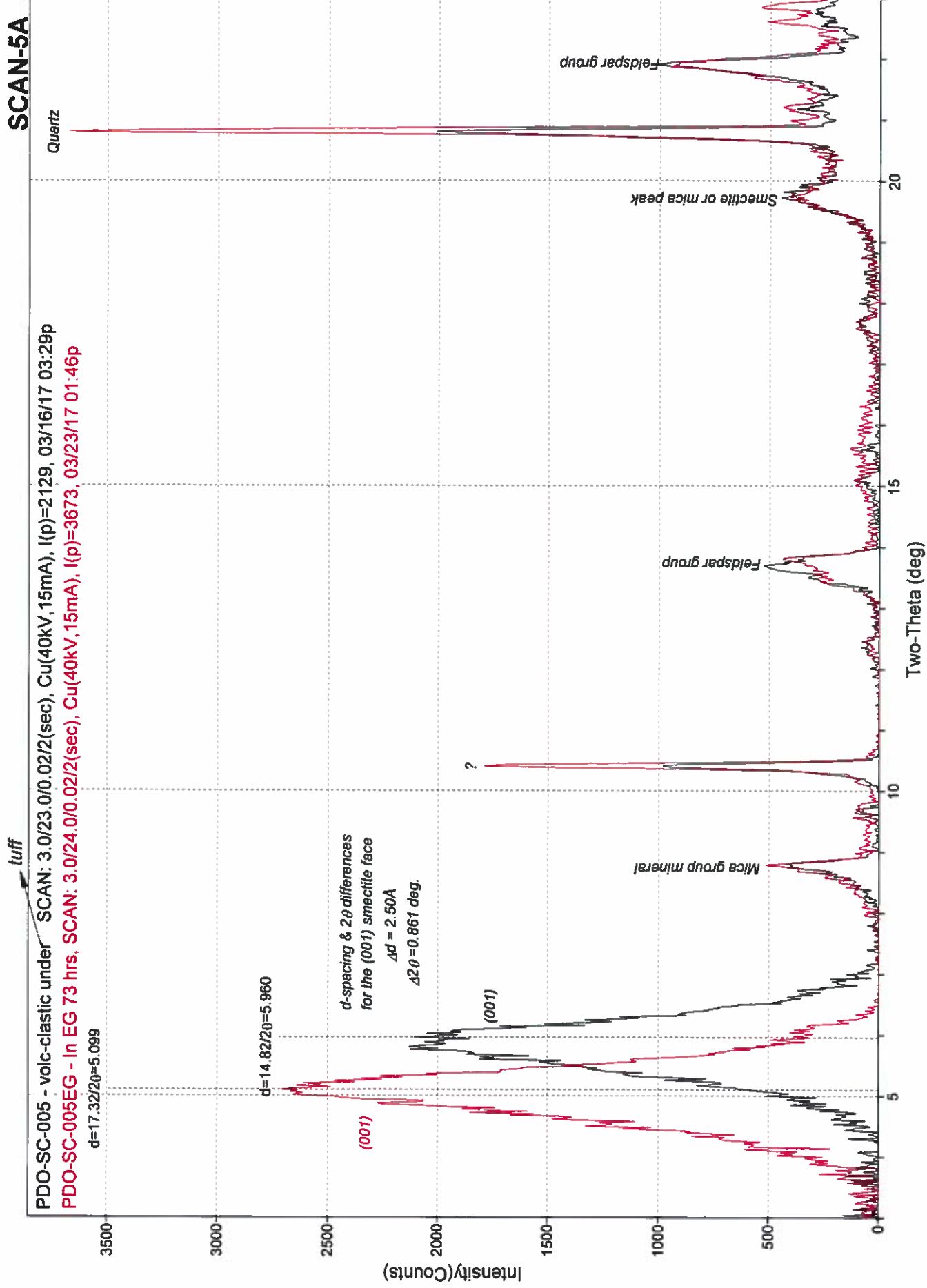


PDO-SC-005 - volc-clastic under tuff SCAN: 3.0/56.0/0.02/2(sec), Cu(40kV,15mA), I(p)=9859, 03/16/17 03:29p

## SCAN-5

00-046-1045> Quartz - SiO<sub>2</sub>  
01-088-1223> Erionite - K<sub>2</sub>Ca<sub>2</sub>4Mg<sub>0.52</sub>(Al<sub>7.908</sub>Si<sub>28.092</sub>O<sub>72</sub>)(H<sub>2</sub>O)<sub>21.6</sub>

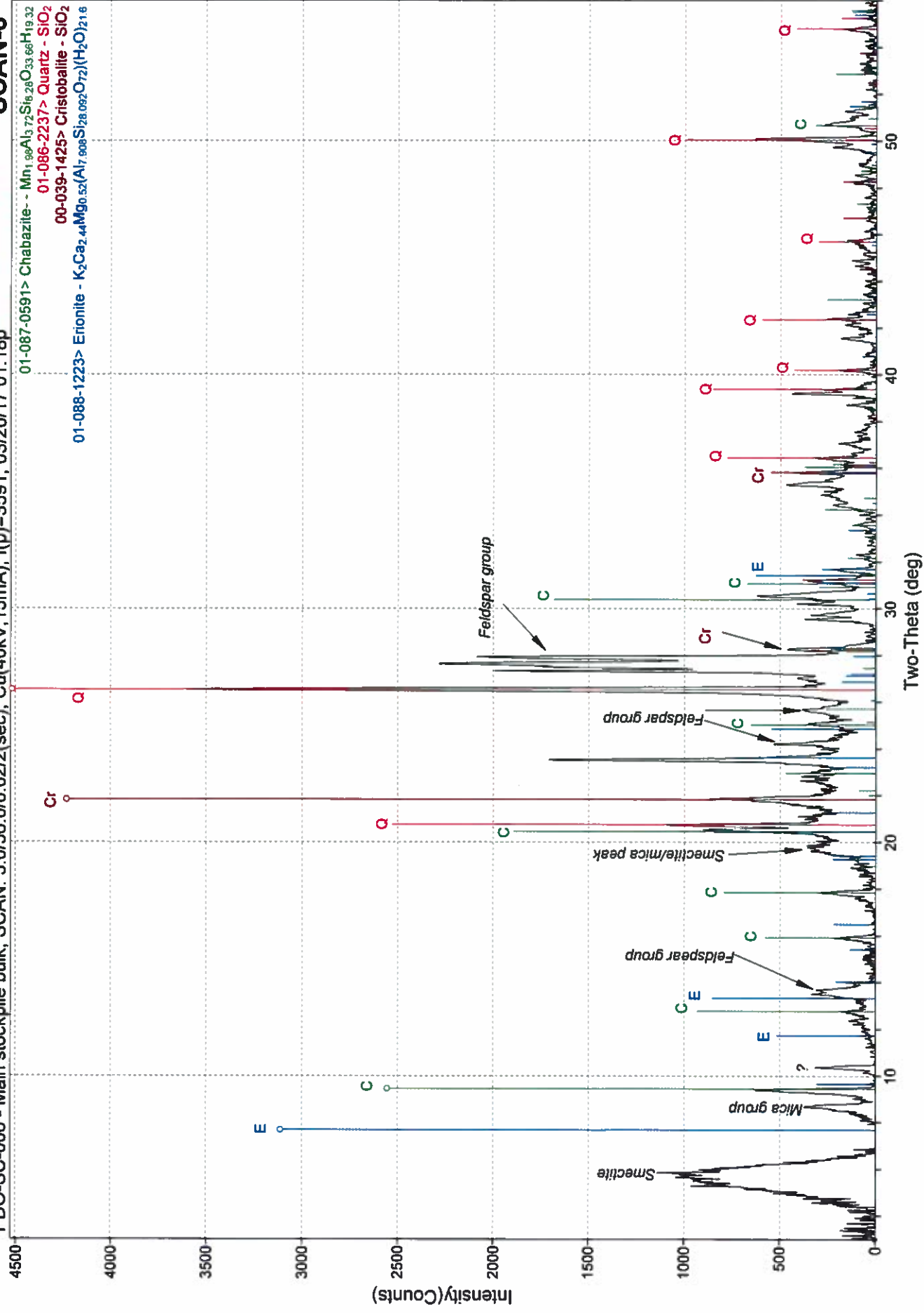




PDO-SC-006 - Main stockpile bulk, SCAN: 3.0/56.0/0.02/2(sec), Cu(40kV, 15mA), I(p)=3591, 03/20/17 01:18p

## SCAN-6

01-087-0591> Chabazite -  $\text{Mn}_{1.99}\text{Al}_{3.72}\text{Si}_{6.28}\text{O}_{33.66}\text{H}_{19.32}$   
01-086-2237> Quartz -  $\text{SiO}_2$   
00-039-1425> Cristobalite -  $\text{SiO}_2$   
01-088-1223> Erionite -  $\text{K}_2\text{Ca}_2\text{Mg}_{0.52}(\text{Al}_{7.908}\text{Si}_{28.092}\text{O}_{72})(\text{H}_2\text{O})_{21.6}$

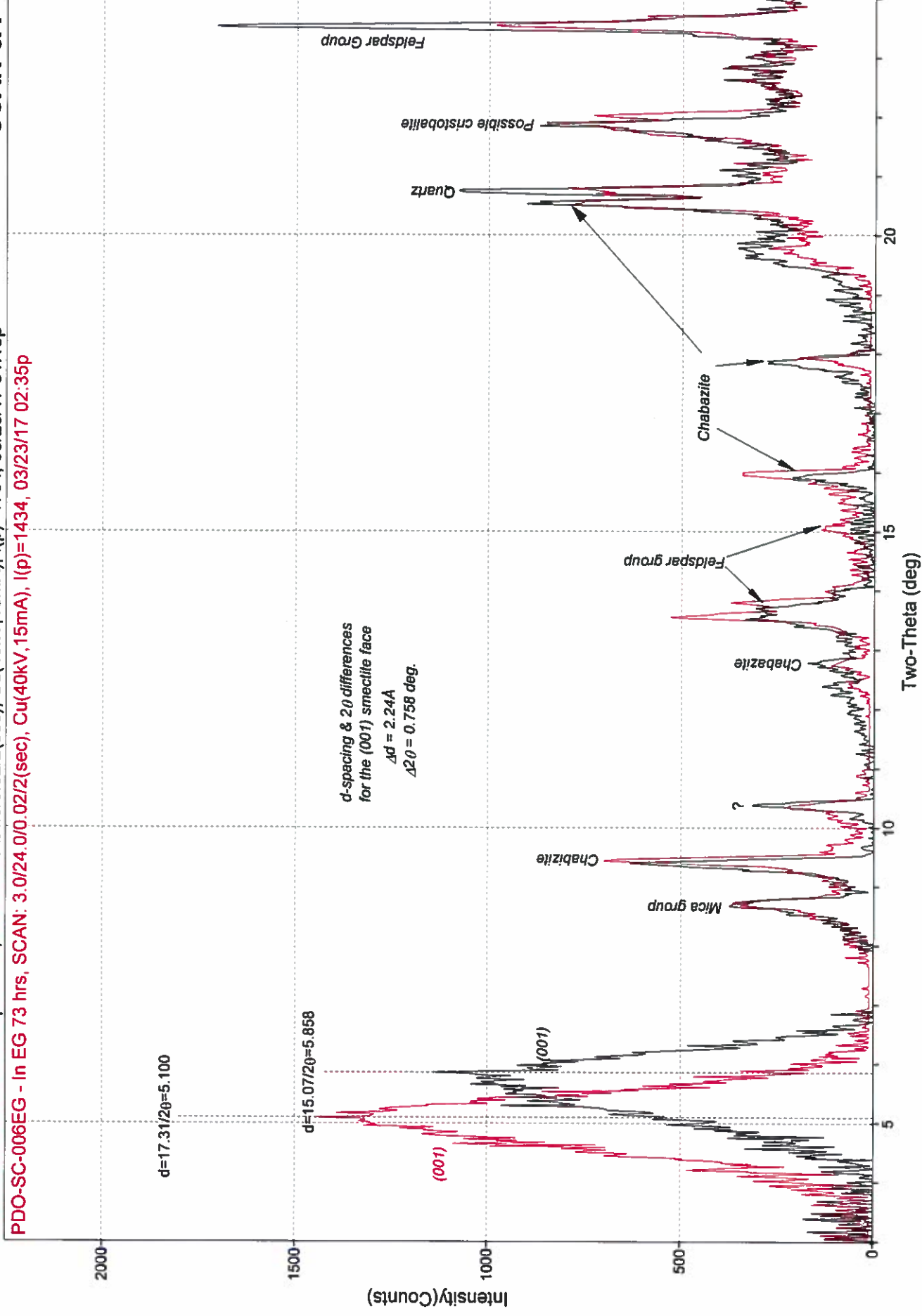




# SCAN-6A

PDO-SC-006 - Main stockpile bulk, SCAN: 3.0/24.0/0.02/2(sec), Cu(40kV, 15mA), I(p)=1704, 03/20/17 01:18p

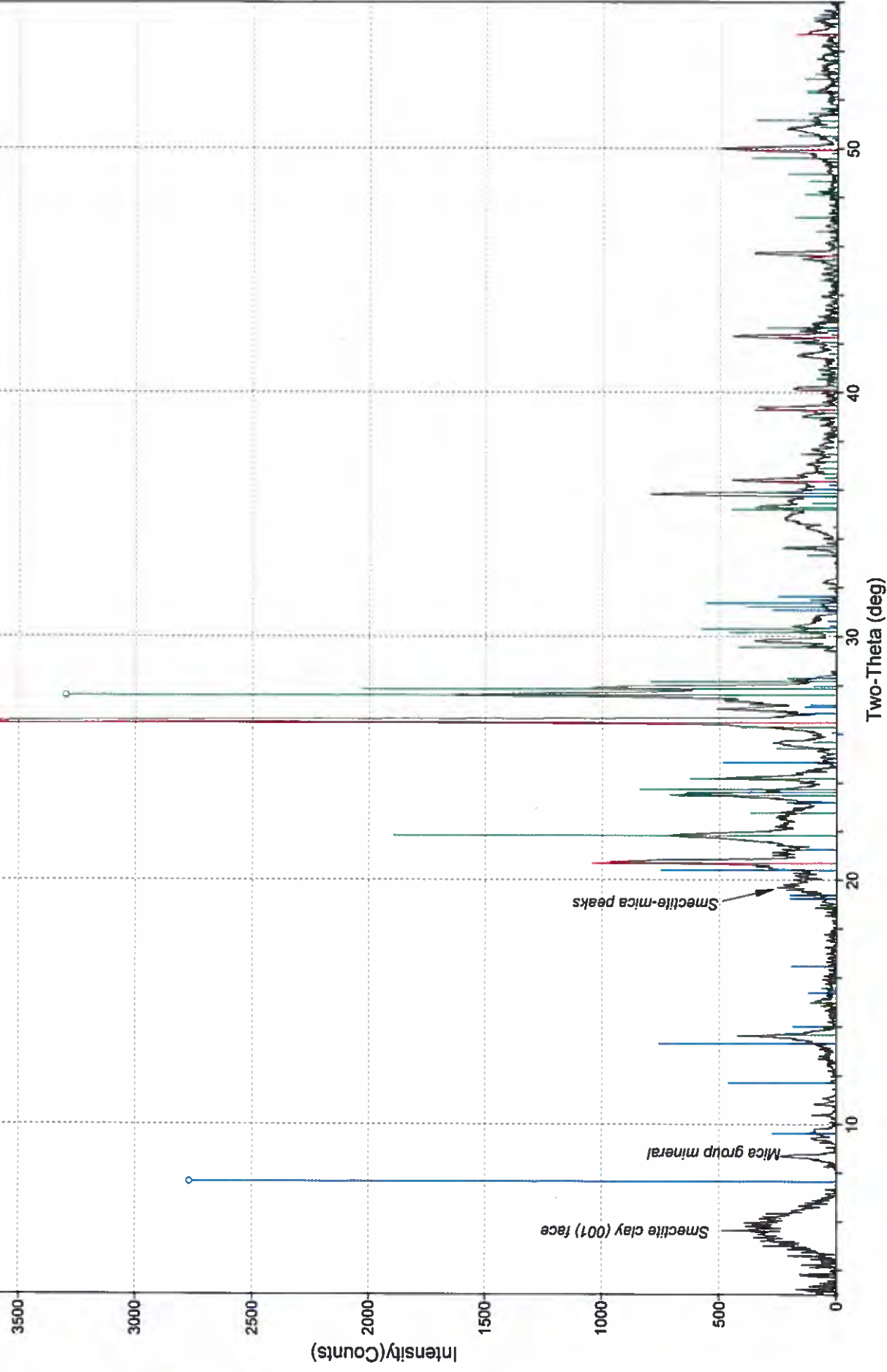
PDO-SC-006EG - In EG 73 hrs, SCAN: 3.0/24.0/0.02/2(sec), Cu(40kV, 15mA), I(p)=1434, 03/23/17 02:35p



PDO-SC-007 - top tuff abv entry pitSCAN: 3.0/56.0/0.02/2(sec), Cu(40kV,15mA), I(p)=3673, 04/24/17 03:24p

## SCAN-7

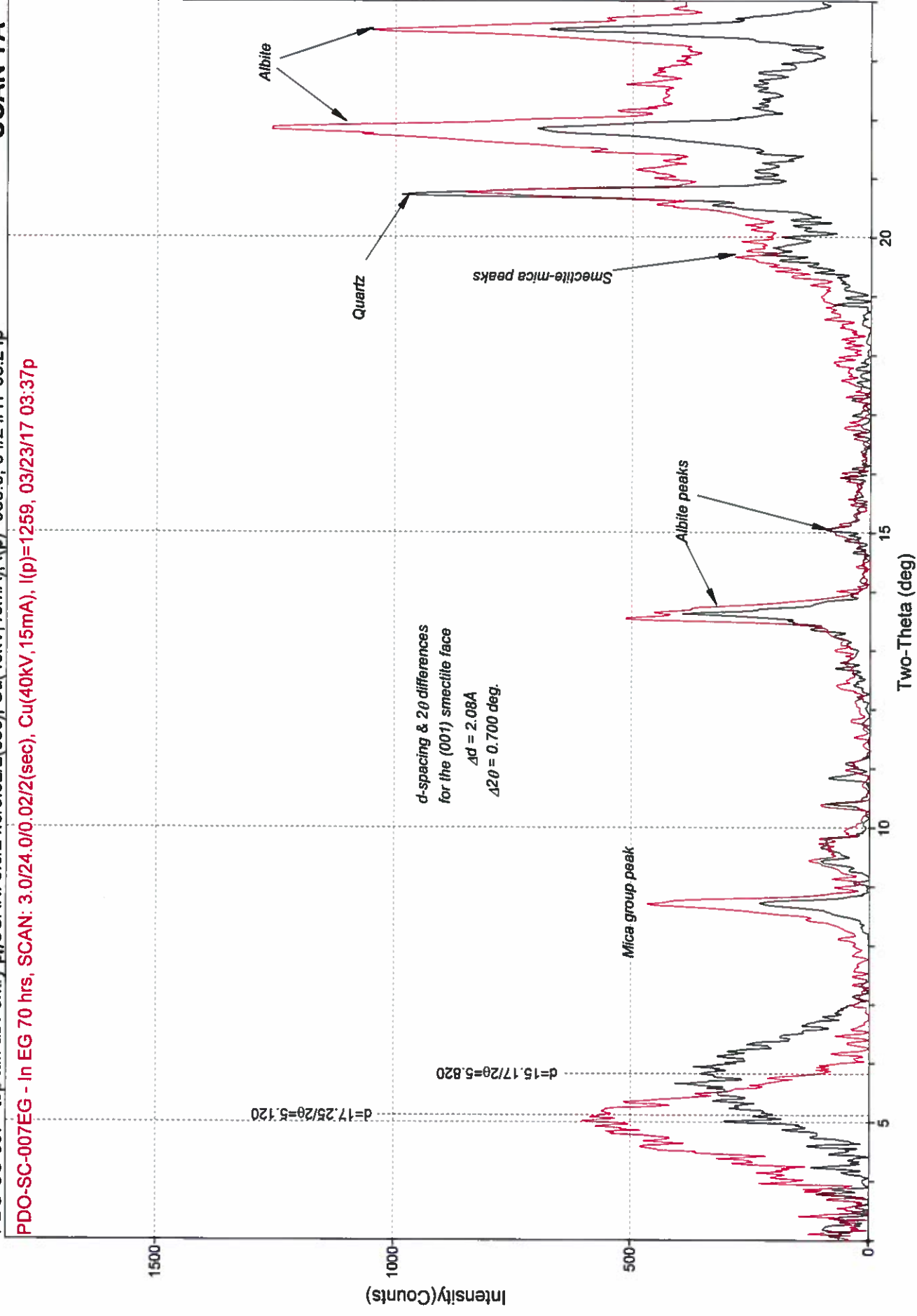
01-086-2237> Quartz -  $\text{SiO}_2$   
01-076-0926> Albite -  $(\text{Na}_{0.75}\text{Ca}_{0.25})(\text{Al}_1.26\text{Si}_{2.74}\text{O}_6)$   
01-088-1223> Erionite -  $\text{K}_2\text{Ca}_{2.4}\text{Mg}_{0.52}(\text{Al}_{7.98}\text{Si}_{28.092}\text{O}_{72})(\text{H}_2\text{O})_{21.6}$



PDO-SC-007 - top tuff abv entry pitSCAN: 3.0/24.0/0.02/2(sec), Cu(40kV, 15mA), I(p)=985.0, 04/24/17 03:24p

SCAN-7A

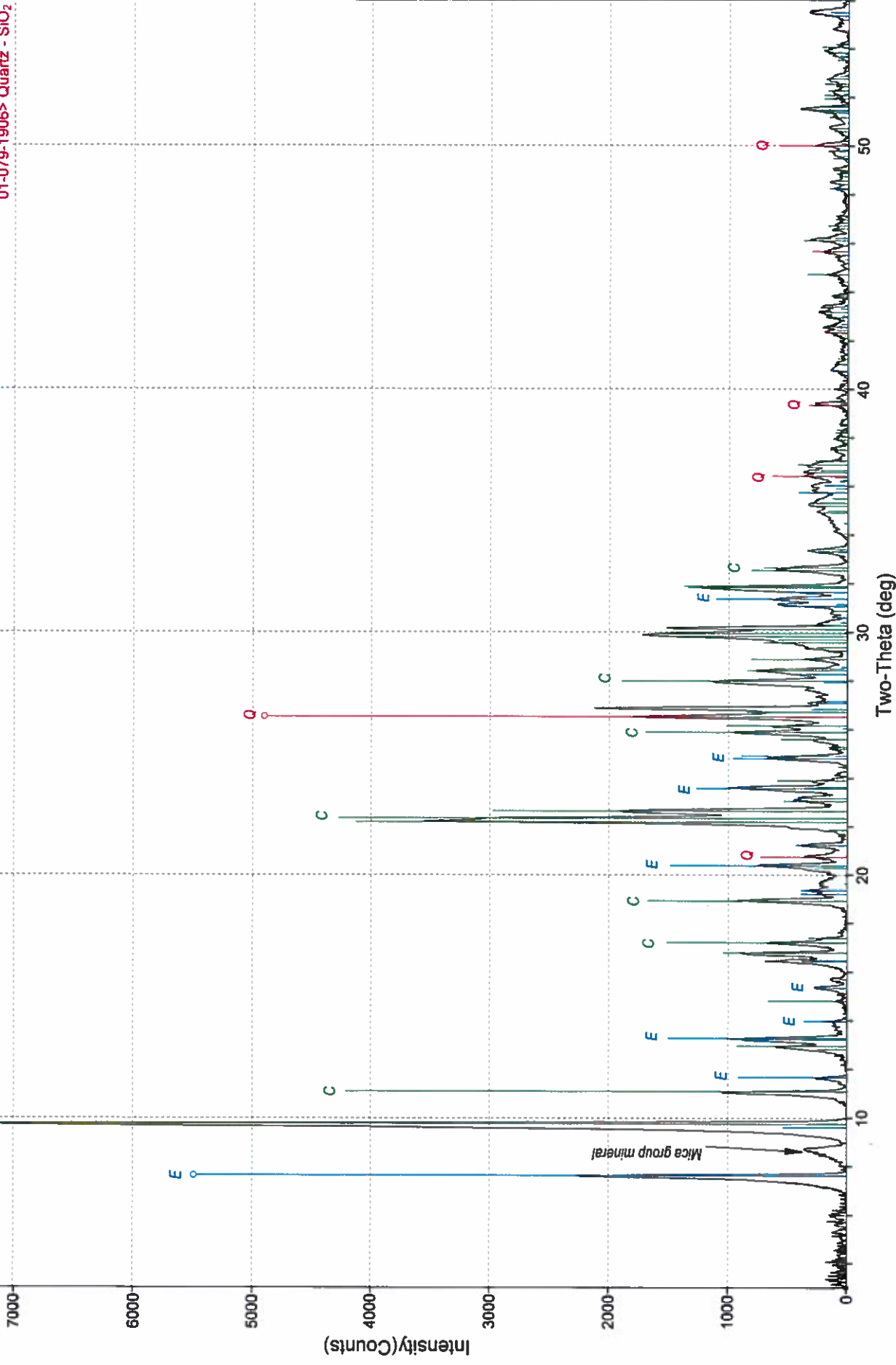
PDO-SC-007EG - In EG 70 hrs, SCAN: 3.0/24.0/0.02/2(sec), Cu(40kV, 15mA), I(p)=1259, 03/23/17 03:37p



PDO-SC-008 - Hwy 89 roadcut, SCAN: 3.0/56.0/0.02/2(sec), Cu(40kV, 15mA), I(p)=7182, 03/23/17 12:53p

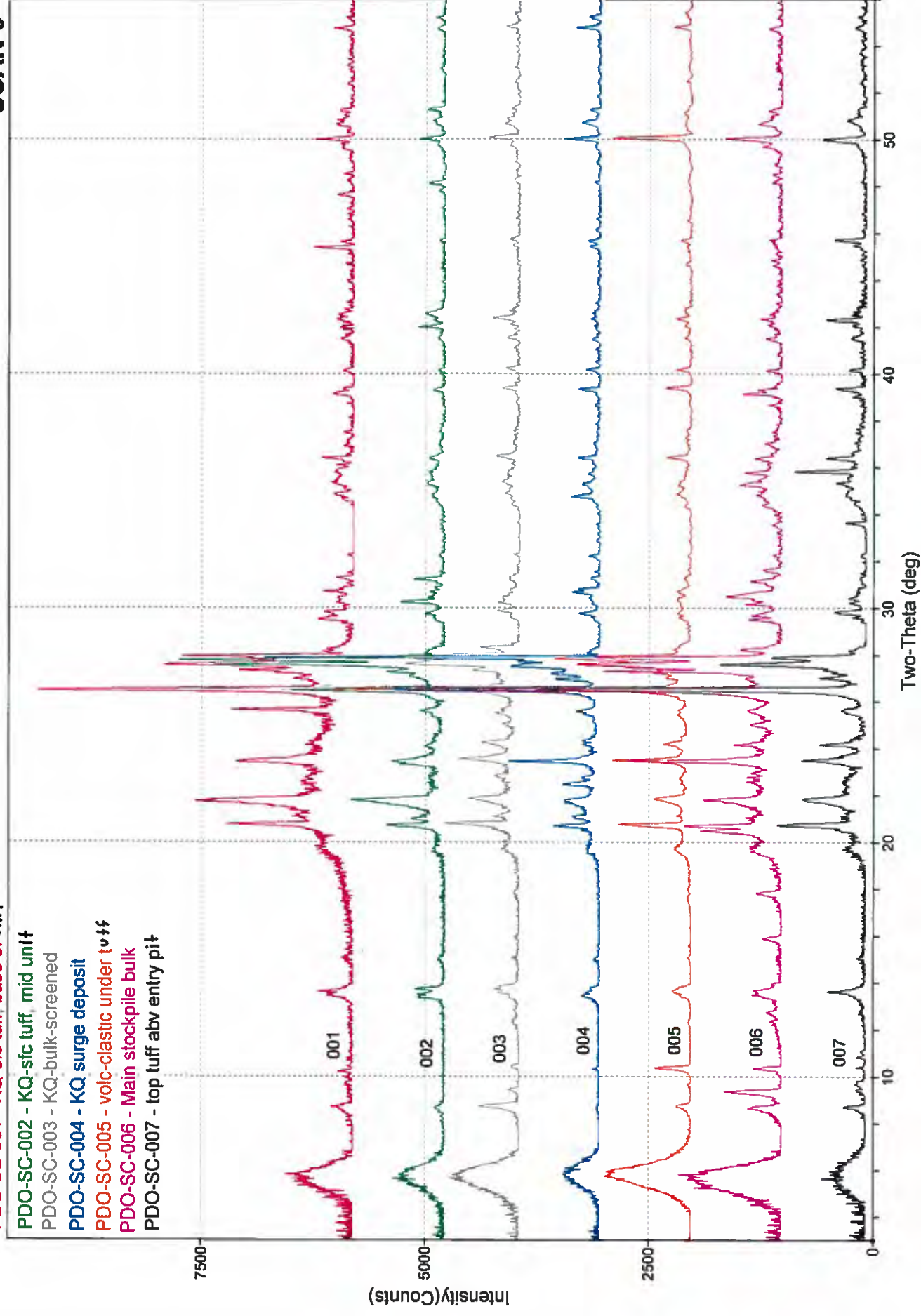
## SCAN-8

01-080-0464> Clinoptilolite -  $\text{Na}_{2.89}\text{K}_{0.37}\text{Mg}_{0.80}\text{Ca}_{0.64}\text{Ba}_{0.15}(\text{Al}_6\text{Si}_{29}\text{O}_{72})(\text{H}_2\text{O})$   
01-088-1223> Eriomite -  $\text{K}_2\text{Ca}_2\text{Mg}_{0.52}(\text{Al}_{7.90}\text{Si}_{26.09}\text{O}_{72})(\text{H}_2\text{O})_{21.6}$   
01-079-1906> Quartz -  $\text{SiO}_2$



PDO-SC-001 - KQ-sfc tuff, base of unit  
 PDO-SC-002 - KQ-sfc tuff, mid unit  
 PDO-SC-003 - KQ-bulk-screened  
 PDO-SC-004 - KQ surge deposit  
 PDO-SC-005 - volc-clastic under tuff  
 PDO-SC-006 - Main stockpile bulk  
 PDO-SC-007 - top tuff abv entry pit

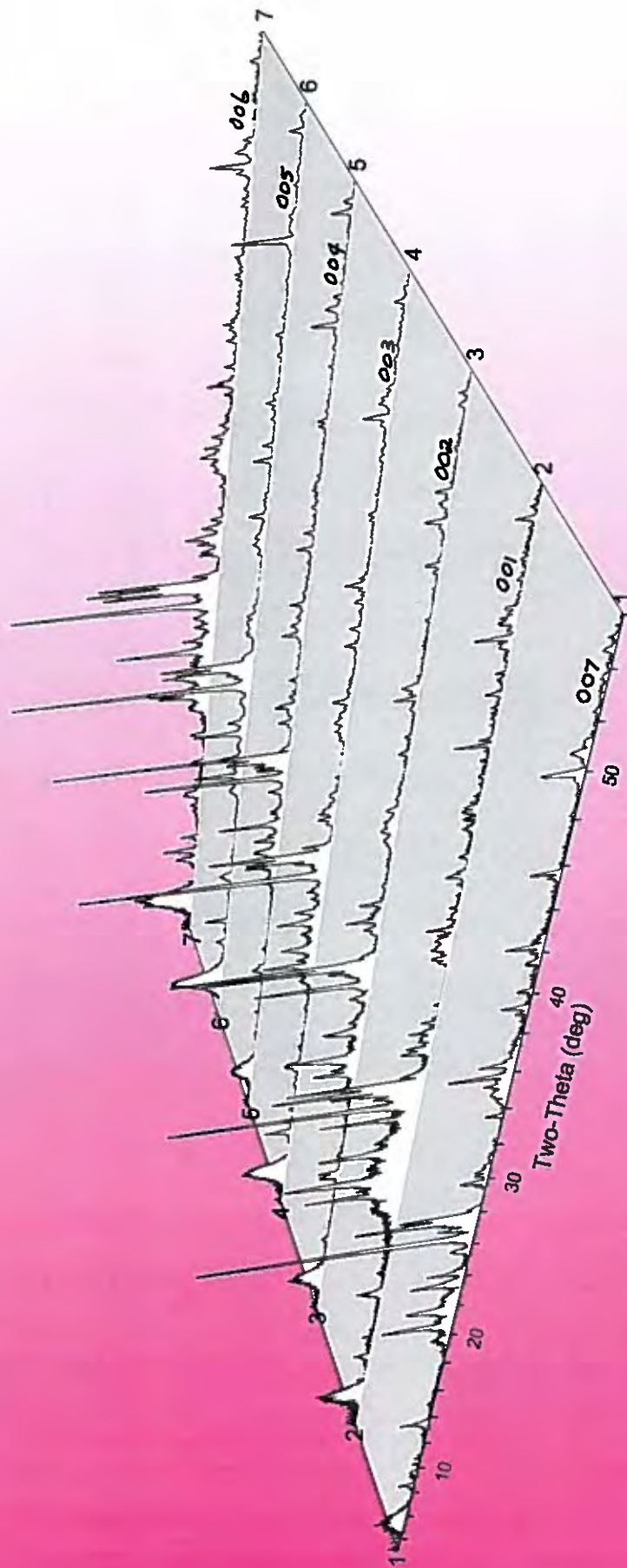
## SCAN-9





## SCAN-10

- (7) PDO-SC-006 - Main stockpile bulk
- (6) PDO-SC-005 - volc-clastic under tuff
- (5) PDO-SC-004 - KQ surge deposit
- (4) PDO-SC-003 - KQ-bulk-screened
- (3) PDO-SC-002 - KQ-sfc tuff, mid unit
- (2) PDO-SC-001 - KQ-sfc tuff, base of unit
- (1) PDO-SC-007 - top tuff abv entry pit





## SCAN-11

- (7) PDO-SC-006 - Main stockpile bulk
- (6) PDO-SC-005 - volc-clastic under tuff
- (5) PDO-SC-004 - KQ surge deposit
- (4) PDO-SC-003 - KQ-bulk-screened
- (3) PDO-SC-002 - KQ-sfc tuff, mid unit
- (2) PDO-SC-001 - KQ-sfc tuff, base of unit
- (1) PDO-SC-007 - top tuff abv entry pit

