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Attention: Vic Dobos

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Report Number JS0437

Quantitative X Ray Diffraction Examination of Gunn Point of Clay Samples

Introduction:

Six samples of clays were submitted for phase analysis using X-ray diffraction (XRD).

Sampling & Preparation:

The samples were crushed and prepared as

a. Unoriented powder mounts of the total sample. This is used for identification of all minerals in the sample and for SIROQUANT quantification.
b. Oriented clay mounts of the minus 2 micron clay fraction untreated, glcolated and heated. These mounts are used to help identify the clay minerals present.

Cation Exchange Capacity: A fraction of each sample was taken and sent for Cation Exchange Capacity, (CEC). This determines the amount of exchangeable cation in a sample. Exchange ions are either surface attached or inherent (though labile) components of the mineral structure. Surface CEC is negligible for all minerals of particle size > 2 µm, rising to about 4 meq/100g for kaolinite and true illite, reaching 30 meq/100 g for typical illite, and is of the order of 100 to 120 meq/100 g for smectite and vermiculite.

Weight Loss on Heating
By heating a bulk sample to specific temperatures, an estimate of some hydrous minerals can be obtained. Surface moisture is largely lost at 40°C (pre-treatment standard). Smectite loses adsorbed water at 110°C, vermiculite, at or below 350°C. Heating experiments provide a cross-check on mineral percentages derived from XRD, CEC and bulk chemistry

XRF major element analysis A further fraction was taken for XRF major element analysis. Chemistry is essential to help confirm mineralogy.

Analytical Method:

The XRD patterns were produced using a Bruker-AXS D4 XRD with copper radiation at 40 kV and 30 mA, over a range of 1.3 to $70^{o}2\theta$, with a 0.02 degree step and a 1 second per step count time. A graphite monochromator was used in the diffracted beam. The search/match was carried out with the aid of the Bruker Diffrac*plus* search/match software and the ICDD PDF-2 database.

Quantification was carried out using SIROQUANT which is a Rietveld based method and is at present widely accepted as the best instrumental method available for quantitative phase analysis.

Summary:
Clay minerals are often difficult to identify and positive identification of these minerals requires a number of tests.

Montmorillonite is a member of the smectite group of clays. Identification of a “typical” 2:2 layer smectite (montmorillonite) can normally be made by made by the behaviour of the position of the reflections in an untreated scan, a glcyolated scan and a heated scan. Glycolation increases the d spacing of the 001 basal reflection markedly and heating to 350 degrees C causes the structure to collapse.

The MMS series of samples do not swell as would be expected for a good montmorillonite. Because of this Dr R. A. Eggleton, the clay mineralogist working on this project recommended that cation exchange values (CEC) were measured for these samples.

A selection of XRD scans are listed below. These show the mineralogy of the MMS samples and scans of the Wyoming bentonite. The Wyoming sample illustrates the expansion of the 001 reflection clearly. This sample was prepared and run with the MMS samples as a standard to check the methodology. This showed confirmed results above.

A report written by Dr R. A. Eggleton is attached (Appendix 2).

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Appendix 1 XRF major element chemistry

	MMS1	MMS2	MM3	MMS4	MMS5	MMS6
SiO_2	47.61	51.75	55.67	52.91	57.01	56.62
TiO_2	1.52	1.00	1.05	1.00	0.96	1.03
Al_2O_3	30.12	23.72	20.91	24.02	15.73	20.27
Fe_2O_3	4.64	5.75	1.88	6.13	4.33	2.81
MnO	0.01	0.04	0.01	0.03	0.06	0.02
MgO	0.28	0.83	1.20	0.71	2.13	1.33
CaO	0.05	0.23	0.39	0.19	0.89	0.39
Na_2O	0.69	0.74	0.80	0.73	0.82	0.83
K_2O	0.53	1.20	0.76	1.17	3.21	1.63
P_2O_5	<0.01	0.11	<0.01	0.11	0.12	0.05
S	0.03	0.03	0.53	0.03	0.37	0.09
LOI	15.20	14.07	16.19	12.67	14.71	15.36
Total	100.68	99.48	99.39	99.70	100.33	100.42

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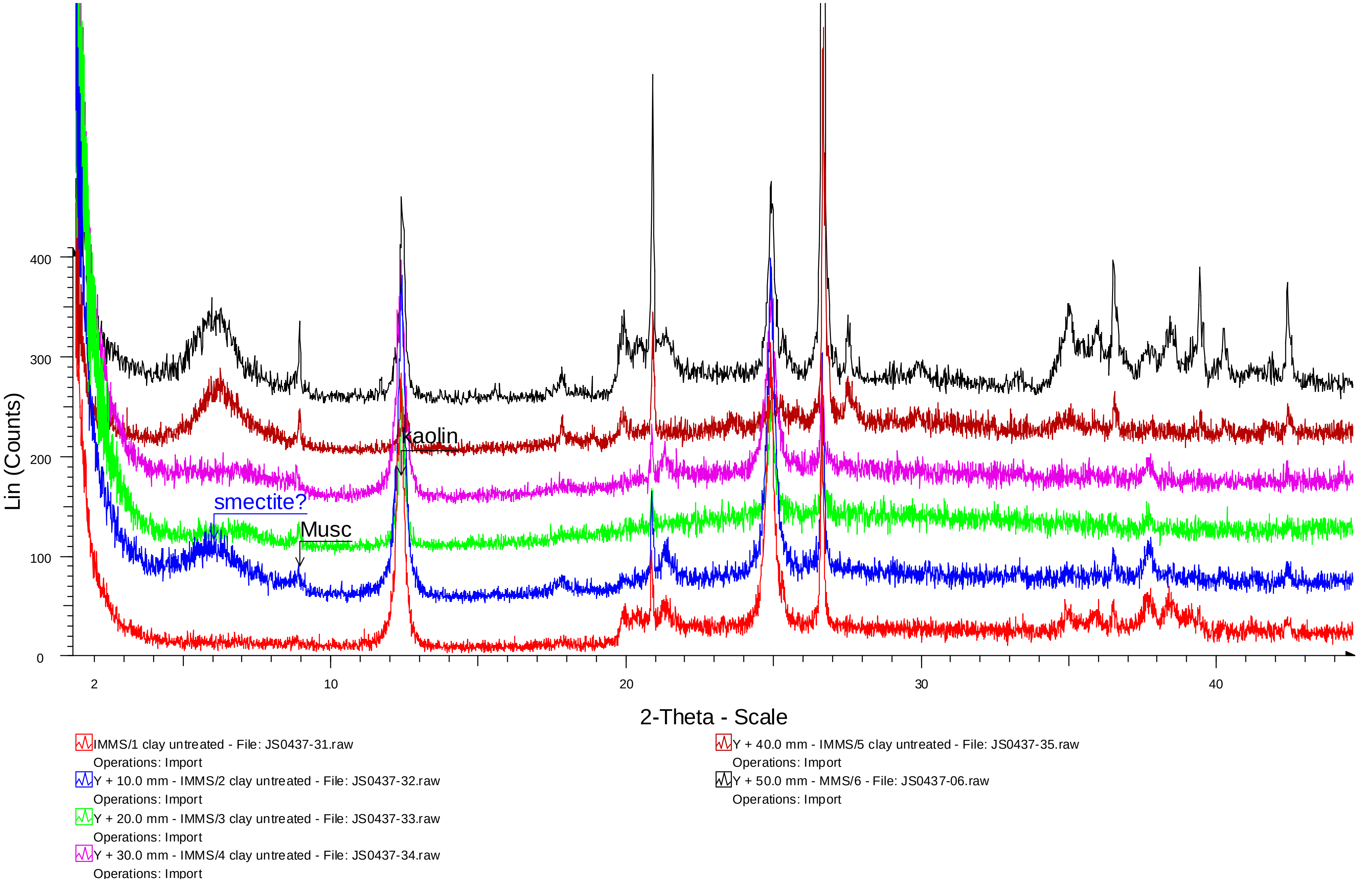
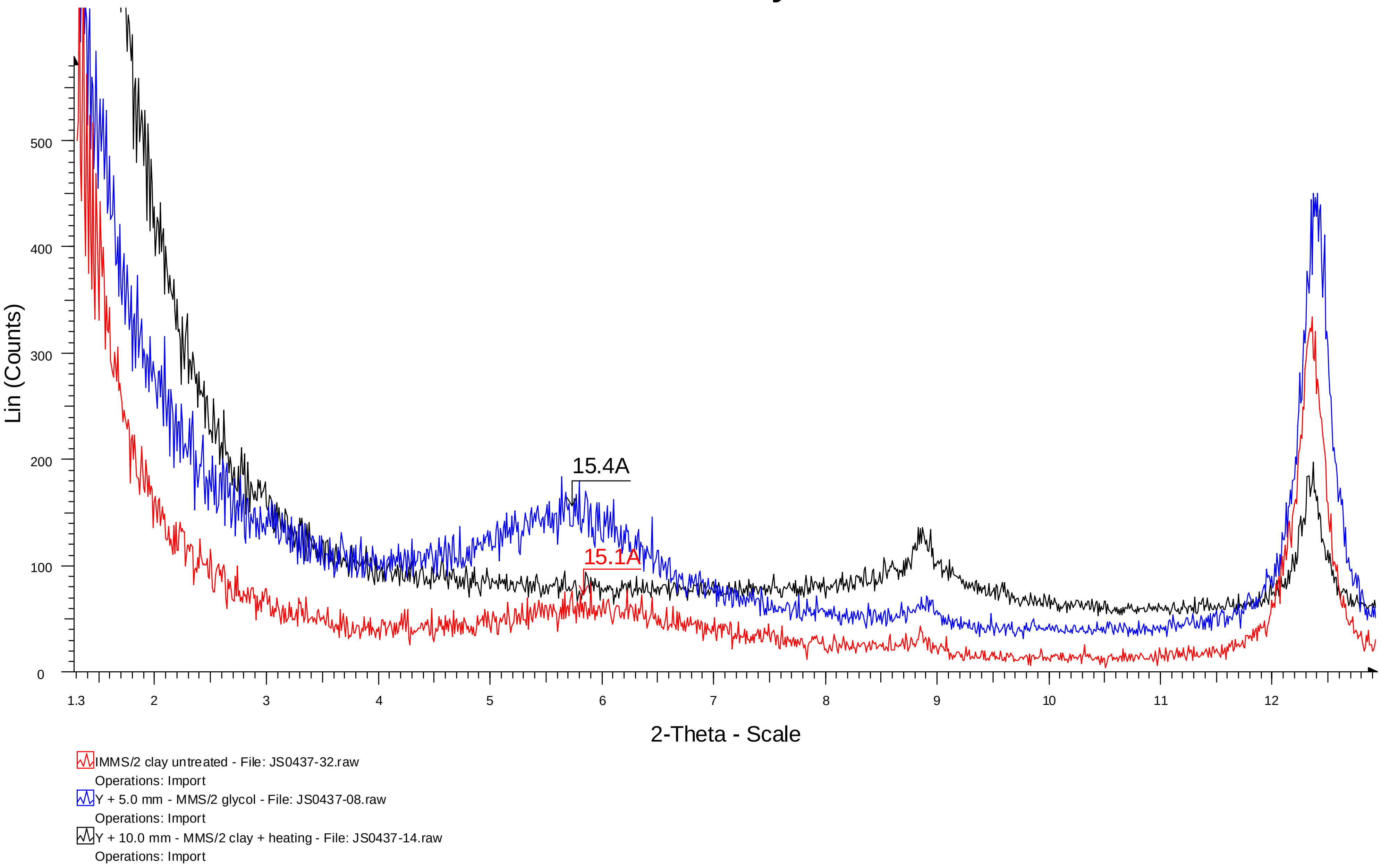
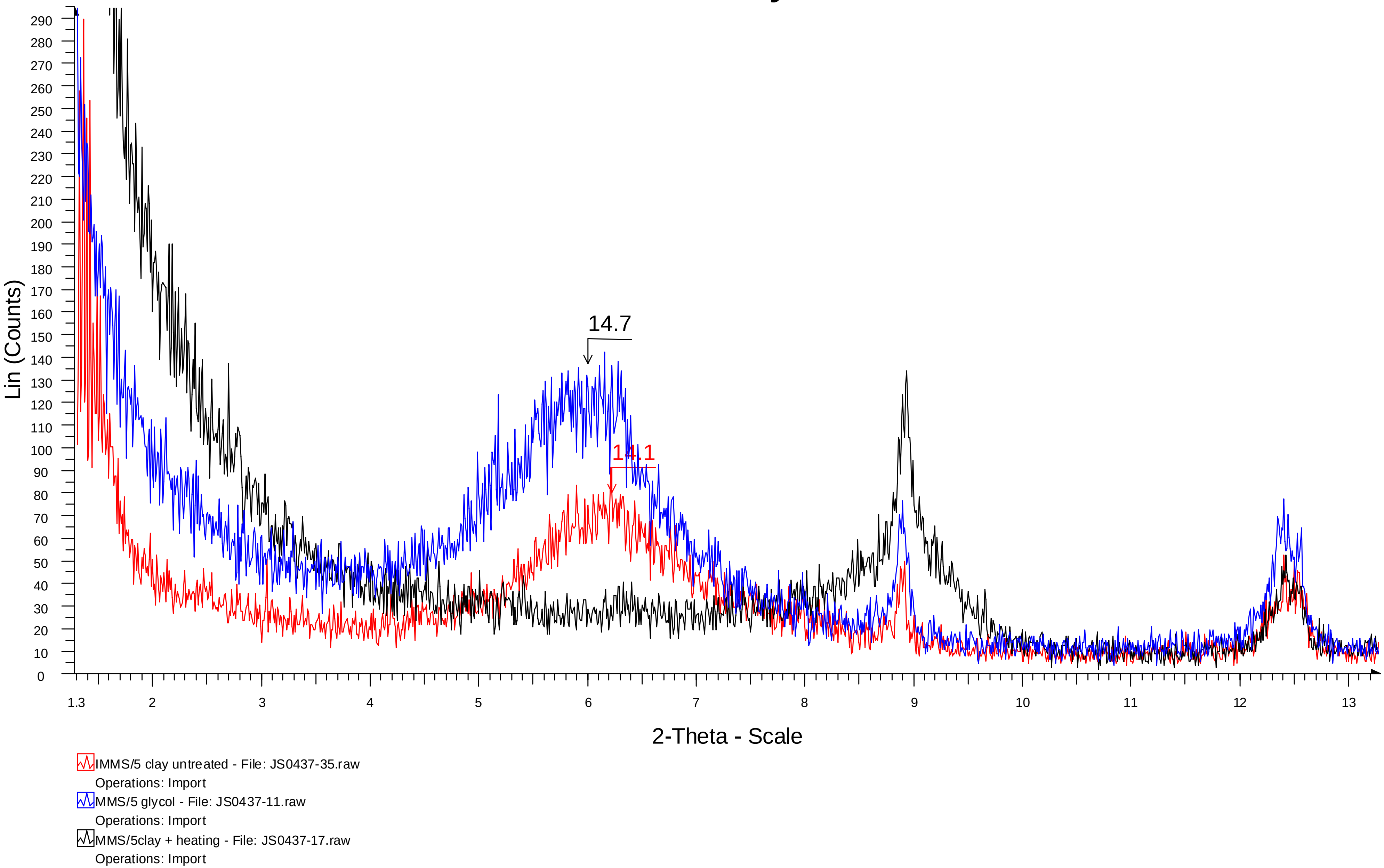
Composite scan Clay fraction untreated



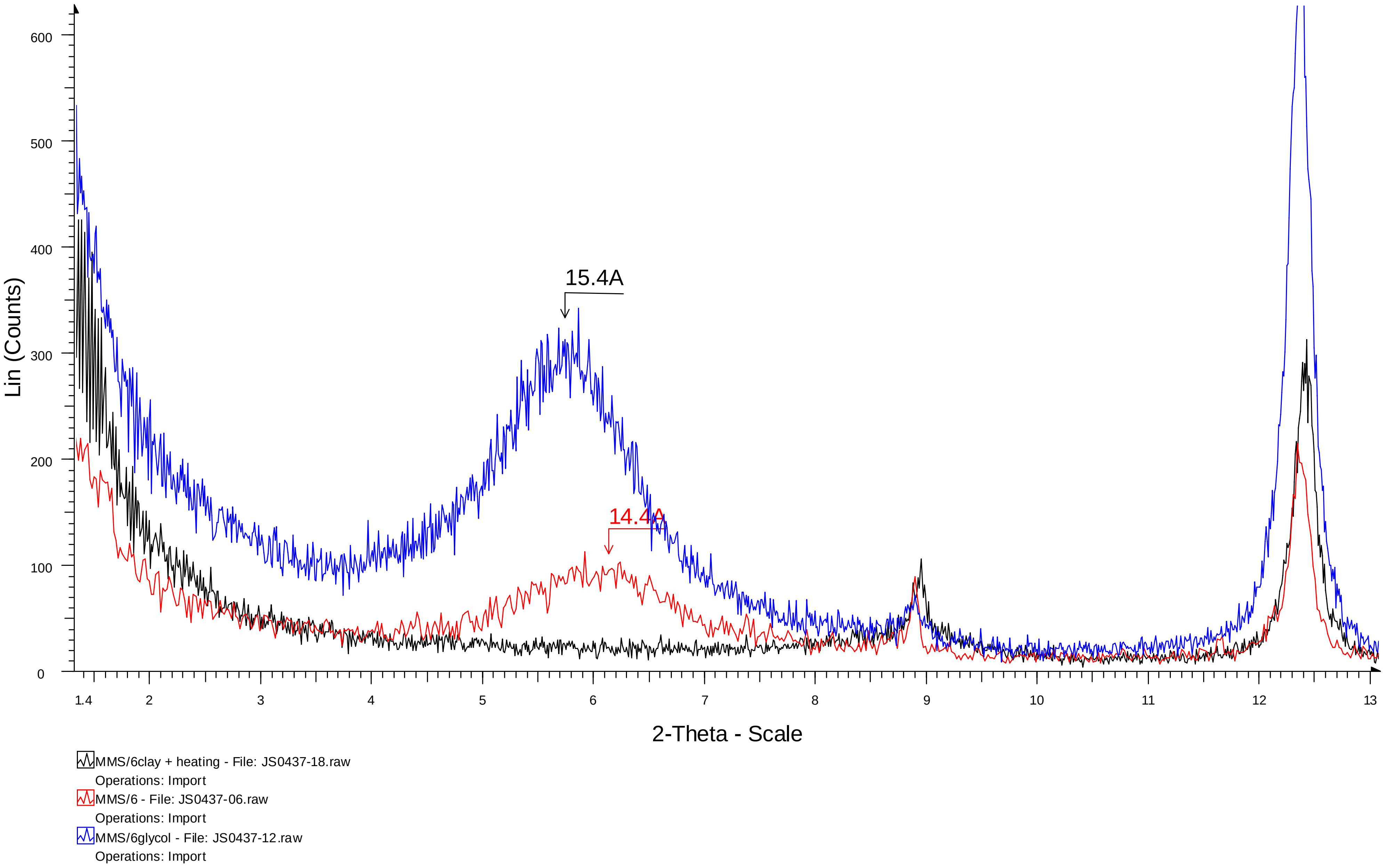
MMS 2 clays


Lin (Counts)
0
100
200
300
400
500
1.3
2
3
4
5
6
7
8
9
10
11
12
2-Theta - Scale
15.4A
15.1A
IMMS/2 clay untreated - File: JS0437-32.raw
Operations: Import
Y + 5.0 mm - MMS/2 glycol - File: JS0437-08.raw
Operations: Import
Y + 10.0 mm - MMS/2 clay + heating - File: JS0437-14.raw
Operations: Import

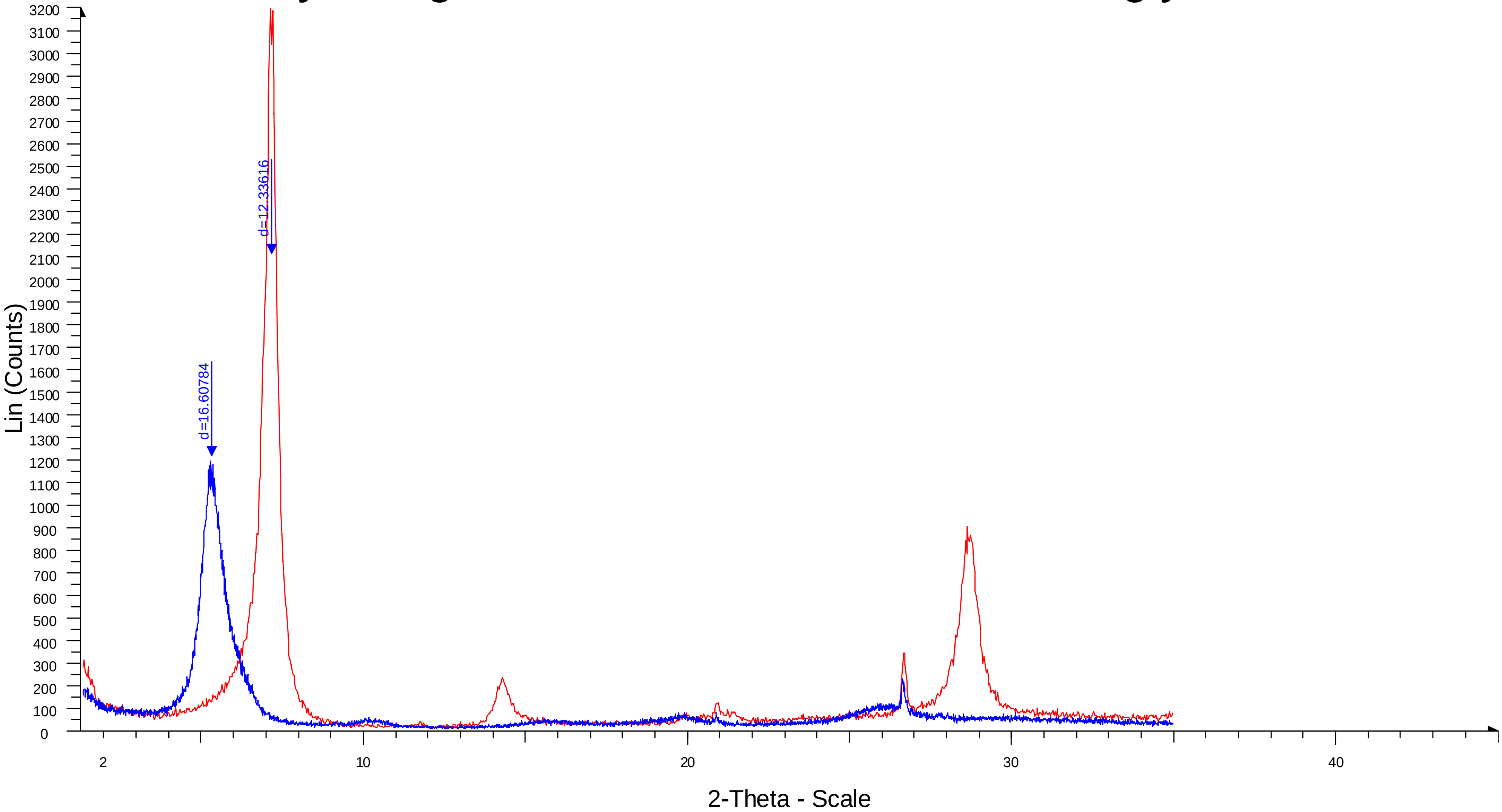
MSS 5 clays


Lin (Counts)
2-Theta - Scale
14.7
14.1
IMMS/5 clay untreated - File: JS0437-35.raw
Operations: Import
MMS/5 glycol - File: JS0437-11.raw
Operations: Import
MMS/5clay + heating - File: JS0437-17.raw
Operations: Import

MMS 6

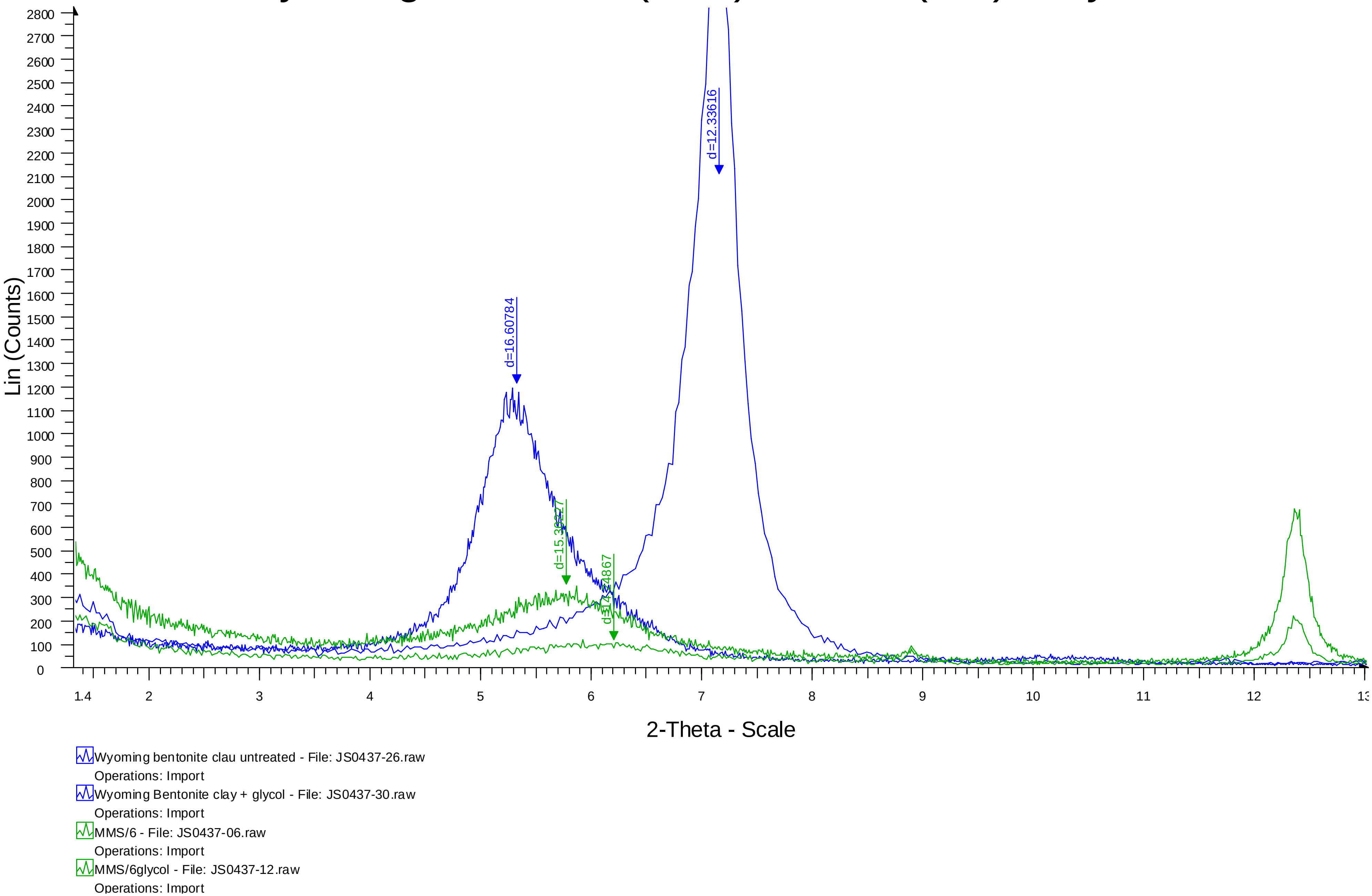

Lin (Counts)
0
100
200
300
400
500
600
15.4A
14.4A
1.4
2
3
4
5
6
7
8
9
10
11
12
13
2-Theta - Scale
MMS/6clay + heating - File: JS0437-18.raw
Operations: Import
MMS/6 - File: JS0437-06.raw
Operations: Import
MMS/6glycol - File: JS0437-12.raw
Operations: Import

Wyoming Bentonite, red untreated, blue glycol

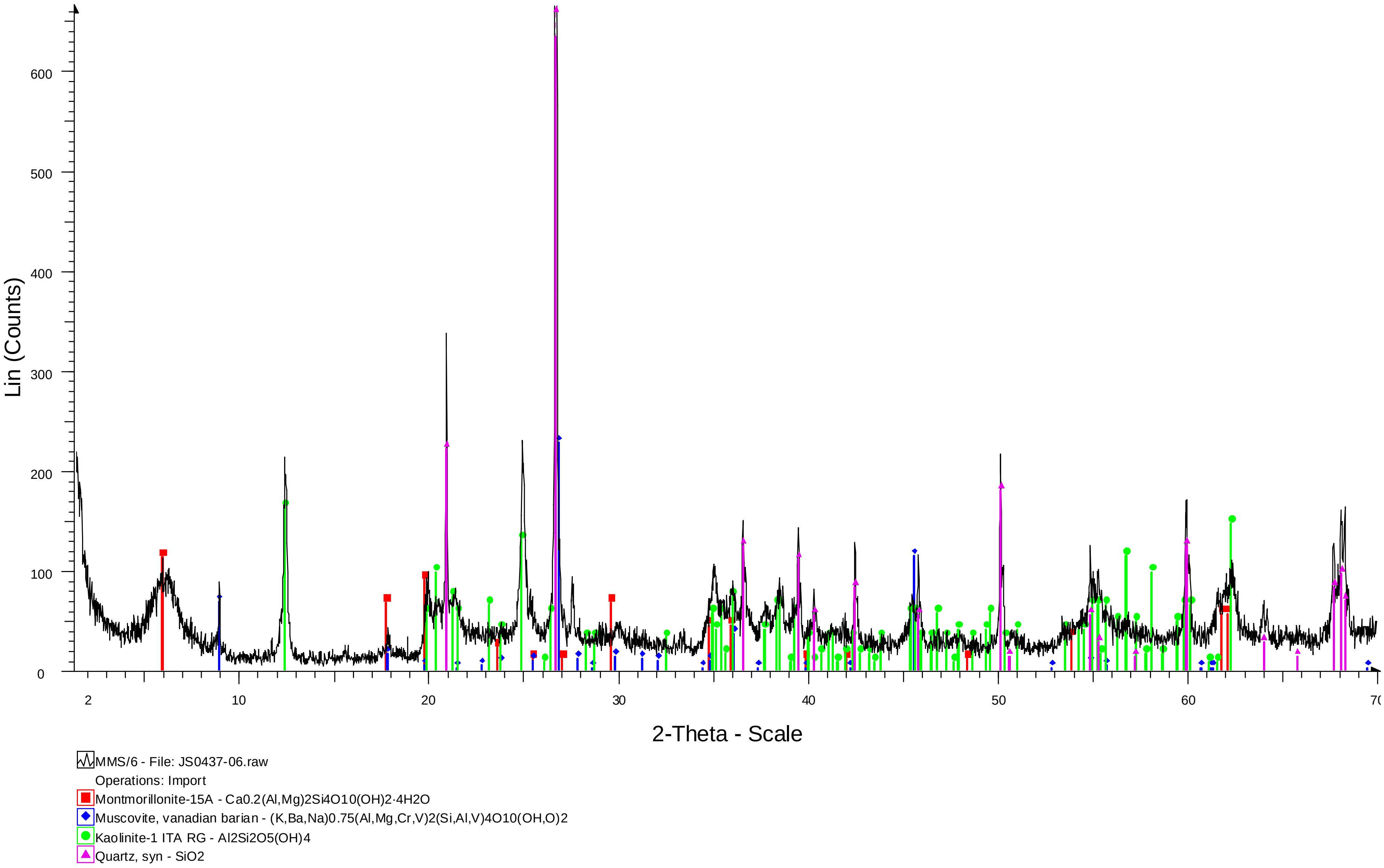


Wyoming bentonite clau untreated - File: JS0437-26.raw
Operations: Import
Wyoming Bentonite clay + glycol - File: JS0437-30.raw
Operations: Import

Wyoming Bentonite (blue)/MMS 6 (red) Clays



MMS 6 total,



Lin (Counts)
0
100
200
300
400
500
600
2
10
20
30
40
50
60
70
2-Theta - Scale
MMS/6 - File: JS0437-06.raw
Operations: Import
Montmorillonite-15A - Ca0.2(Al,Mg)2Si4O10(OH)2·4H2O
Muscovite, vanadian barian - (K,Ba,Na)0.75(Al,Mg,Cr,V)2(Si,Al,V)4O10(OH,O)2
Kaolinite-1 ITA RG - Al2Si2O5(OH)4
Quartz, syn - SiO2

MMS 6 total with vermiculite and smectite reflections