Investigation into mercury contamination in floor infill material taken from rooms 2.62/3, Rutherford Building.

Objective:

To investigate the nature of mercury contamination in floor infill material removed from between floor joists during 2004, and subsequently stored on campus awaiting disposal. Elemental mercury is a known contaminant, but initial investigations commissioned by IRAS (in connection with waste disposal) suggested that forms of mercury insoluble in hot, concentrated nitric acid may also be present. Information about the nature of such compounds may assist the Coggon Inquiry and on-going research into Lord Rutherford's working practices at University of Manchester during 1907-1919.

Report compiled by:

Dr Melanie Taylor, Head of Safety Services
Miss Catherine Davidge, University Safety Co-ordinator

March – Sept 2010.

Background

When rooms 2.62/3 in the Rutherford Building (then known as Coupland 1 Building) were refurbished in 2004, a fibrous material resembling cotton was removed from between the floor joists. It was known to have some radiological contamination and also to contain elemental mercury, some of which was clearly visible to those removing the waste. It was double-bagged and approx 120 "bin liners" were stored in a container, awaiting further testing and approvals for disposal.

During 2006-8, some samples were taken by IRAS Ltd for analysis, with a view to separating the mercury contamination from the waste, and disposing of two streams of waste – one with low level radioactivity and one with mercury contamination.

Analysis reports include:

Severn Trent Ltd Test Report IRAS/D4351, 21 March 2006

Severn Trent Ltd Test Report IRAS/D4354, 22 March 2006

Severn Trent Ltd Test report LL/358644/2006, 18 August 2006

NIRAS ref L080402 - Investigation into the effectiveness of acid washing and autodeposition for removing mercury and radioactive contamination from insulation material, dust and other debris, 10 October 2008

NIRAS ref L080402, 2nd report - Leaching of mercury from insulation material, dust and other debris, 27 October 2008

The results indicated an extremely wide range of mercury concentrations (in one set of data, 39.0 to 250,000 mg/Kg). There is clearly a very uneven distribution of contamination, as one might expect from spillages on the floor above trickling through gaps in floor covering and floor boards into the cotton fill material between the joists. Based on these reports, IRAS have suggested that other insoluble mercury compounds could be present.

Method for obtaining samples

Various enquiries were made of Health & Safety Laboratories and University academic schools about appropriate analysis techniques. Analysis for mercury and mercury compounds is known to be challenging, particularly in the absence of

any confirmed data about likely mercury compounds, and of any kind of control samples. One theory is that mercuric chloride ($HgCl_2$) may have been applied to the organic fibre as a fungicide/preservative, a known practice for biological and anthropological samples in the late 19th Century and early 20th Century. Historically, it was also used to enhance photographic images and could have been used in a Physics Lab.

After initial discussions with Dr E Armstrong (School of Chemistry) and Mr S Caldwell (School of Earth, Atmospheric and Environmental Sciences), X ray fluorescence (XRF) and X ray diffraction (XRD) were selected as possible analysis tools. XRF would provide an initial screening of samples to ensure sufficient presence of mercury to give a meaningful result using XRD, which requires samples of at least 5% mercury.

Samples of the bagged waste material were collected by Dr M Taylor, Ms C Davidge (University Safety Team) and Mr K Robinson (University Radiation Safety Team). The risk assessment for this work is in Appendix 1. A photographic record was also made, and illustrative photographs are included in Appendix 2.

To reduce the risk of contaminating the analysis equipment with radioactive particulates, waste bags were checked for surface activity and those with low counts were selected initially. Two bags were transported to a fume cupboard for opening and further selection of sample material – one containing mostly cotton fibre material (tag MO21), the other with more gritty waste and vacuum cleaner bag contents (tag MOV30). Once inside the fume cupboard, each bag was opened carefully, avoiding unnecessary generation of dust, and material scooped onto a shallow tray and spread out thinly. Radiation was monitored using RPS's Mini Instruments mini monitor g-m meter type 530 and Berthold LB 124, holding the probes as close to the surface of the material as possible without contaminating the instruments. Material giving counts over 10 cps was rejected for the purposes of selecting the sample for mercury analysis.

Samples were placed into 100ml polycarbonate tubs and gently pushed down. Before lids were secured, the Shawcity Mercury Vapour Indicator (MVI) was used to check for the presence of mercury vapour. Once covered and moved out of the fume cupboard, external surfaces were checked to ensure absence of radioactive contamination. Tubs were labelled MO21, 1-5 and MOV30, 1-3.

The original bags were resealed in the fume cupboard and placed into new bin liners. Once removed from the fume cupboard, surfaces were checked for radioactive contamination, found to be clean, and the bags were returned to the storage container.

Analysis for mercury and mercury compounds

Discussions about how to proceed with the analysis involved Dr Taylor, Ms Davidge and Mr Robinson from Health & Safety Services, and Dr P Lythgoe, Dr P Wincott, Prof D Polya (XRF) and Dr J Waters (XRD) from SEAES. The XRF technique involved preparation of a disc of compressed sample and concern was expressed about being able to prevent its disintegration during a 20 minute X ray bombardment. SEAES had experienced such failures with other work, particularly when samples contained metals which heated up during the process.

It was decided to proceed directly to using XRD, where sample preparation does not present the same risk to the equipment. The material is "glued" onto a slide using a petroleum jelly which does not interfere with X ray transmission.

Samples were prepared by Dr Jon Waters on 9 March 2010. Small quantities of grit or fibrous material were transferred into a mortar and ground up and homogenised. The gritty material was suspended in a quick drying solvent (amyl acetate), then transferred onto a slide. 4 slides were prepared initially (2 each from MOV30, sample 1 and MO21, sample 1). As previously noted, mercury vapour was detected above the opened sample pots.

Slides were also prepared from plasterboard core samples taken by ALControl as part of their investigation into mercury vapour concentrations prior to the programme of decontamination work carried out in rooms 2.62 and 2.63, April-Sept 2010. The method is included in their report reference 11826 amendment 1, dated 5 February 2010 but including a revisit on 3 March to resample plasterboard. Each sample taken was divided into 3 portions, a, b and c, with the University taking the c portions for in-house analysis (M10c, M15c, M16, 17c).

The XRD analysis results for the waste samples and the plasterboard core samples were provided by J Waters on 11 March, and are reproduced in Appendix 3. His commentary on these is reproduced in Appendix 4.

Conclusions and Recommendations

The analysis reports and accompanying email from Dr Waters suggest that further in-house analysis of the waste material would not be productive. The presence of calomel (HgCl) in one sample could be from spillage of a calomel electrode or other source, but its absence from other samples suggests that the wadding was not subject to widespread fungal or insecticide treatment.

In view of the difficulty in interpreting the Severn Trent analysis reports, insurmountable sampling errors and lack of definitive evidence of the presence of

mercury compounds in or on the floor wadding, it is recommended that further discussions are held with HSL about how to proceed.

Appendix 1. Risk assessment for procedure to select and prepare samples from contaminated floor material.

General Risk Assessment Form

Date: (1) 16 Feb 2010	Assessed by: (2) Melanie Taylor	Checked / Validated* by: (3) Catherine Davidge	Location: (4) Container nr Radiation Stores RPS, Williamson Building	Assessment ref no (5)	Review date: (6)

Task / premises: (7)

Selection and preparation of samples from container with waste bags from Rutherford refurbishment of 2004, for X ray fluorescence

Activity (8)	Hazard (9)	Who might be harmed and how (10)	Existing measures to control risk (11)	Risk rating (12)	Result (13)
Entering container, examining bags, opening selected bags in lab	Radiation	MJT/CD/KR – exposure to α & β ionising radiation	In container, constant attendance by Kevin Robinson, RPS, with alpha and beta radiation detectors. Surface activity checks on boxes in container prior to opening them. Surface activity checks on waste bags. Primary selection of bags with low / background activity. Bags opened inside fume cupboard, behind sash and handled behind additional perspex screen. PPE – lab coat and nitrile disposable gloves worn. Once bags opened in fume cupboard, waste spread out on shallow tray, and monitored again. "Hot spots" rejected from sample material and returned to bag. Sample pots surveyed with meter on outer surfaces to check no contamination. Gloves checked for radioactive contamination before disposal via designated waste route.	Low	A

Entering container, examining bags, opening selected bags in lab	Mercury vapour and elemental mercury	MJT/CD/KR – Hg vapour MJT/CD – elemental Hg	Constant monitoring with MVI. Opening container doors for 20mins and venting, prior to investigative work (levels on earlier recce exceeded 400ug/m3 initially, falling rapidly). Once levels dropped to <25ug/m3, container re-entered. Once bags transferred to and opened in fume cupboard, presence of mercury vapour checked for selected material. Pots closed in fume cupboard. PPE worn – lab coat and nitrile disposable gloves to prevent contact with elemental mercury. Gloves disposed of through designated waste route.	Low	A
Entering container, examining bags, opening selected bags in lab	Asbestos	MJT/CD/KR	MJT checked with Asbestos Manager Lynn Irving, 16 Feb – presence of asbestos in this kind of material not anticipated. Analysis of underfloor material, 2nd floor, Rutherford Building obtained, 23/7/1997.	Low	A
Entering container, examining bags, opening selected bags in lab	General dust	MJT/CD/KR	At all times, bags and samples handled with care to minimise dust generation. Bags opened inside fume cupboard, behind sash and handled behind additional perspex screen. PPE – lab coat and nitrile disposable gloves worn. Gloves disposed of through designated waste route.	Low	T
Transferring bags from container to lab	Lifting/moving bags & equipment	MJT/CD/KR	Very heavy bags rejected for further sampling in container. Lighter bags selected and moved, well within capabilities of those moving them.	Low	A

Action plan (14)				
Ref No	**Further action required**	**Action by whom**	**Action by when**	**Done**
	None required.			

Appendix 2 : Selection of illustrative photographs

Photo 1: Bag containing Sample MO21 (cotton fibre material)

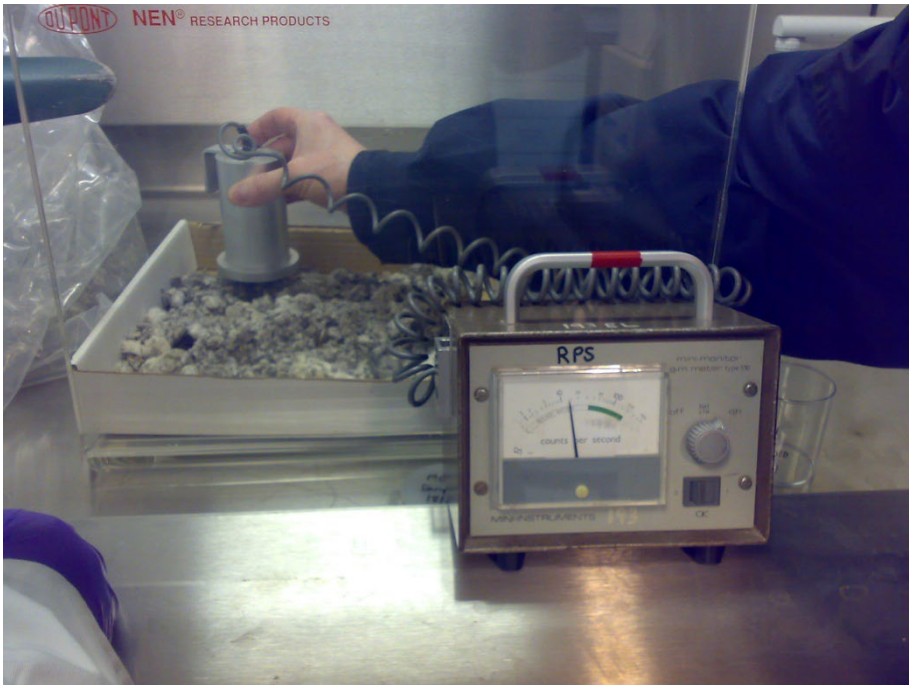


Photo 2: Monitoring contents of MO21 for radioactive contamination

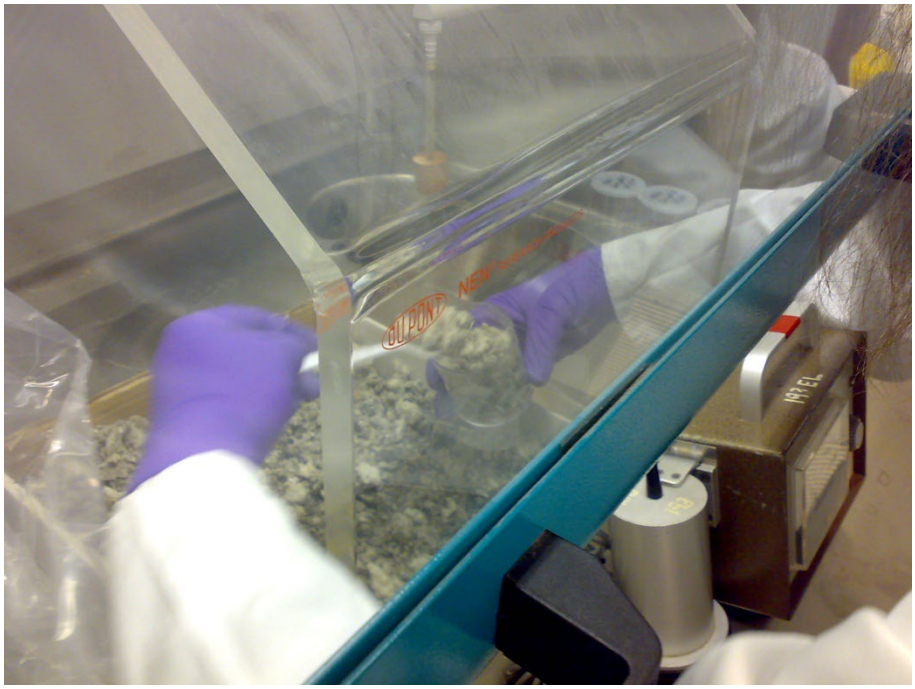


Photo 3: Sampling contents of MO21 into 100ml container

Photo 4: Typical of contents of bag MOV30 (from vacuum cleaner)

Photo 5: Typical plaster core sample taken by ALControl, 3 March 2010.

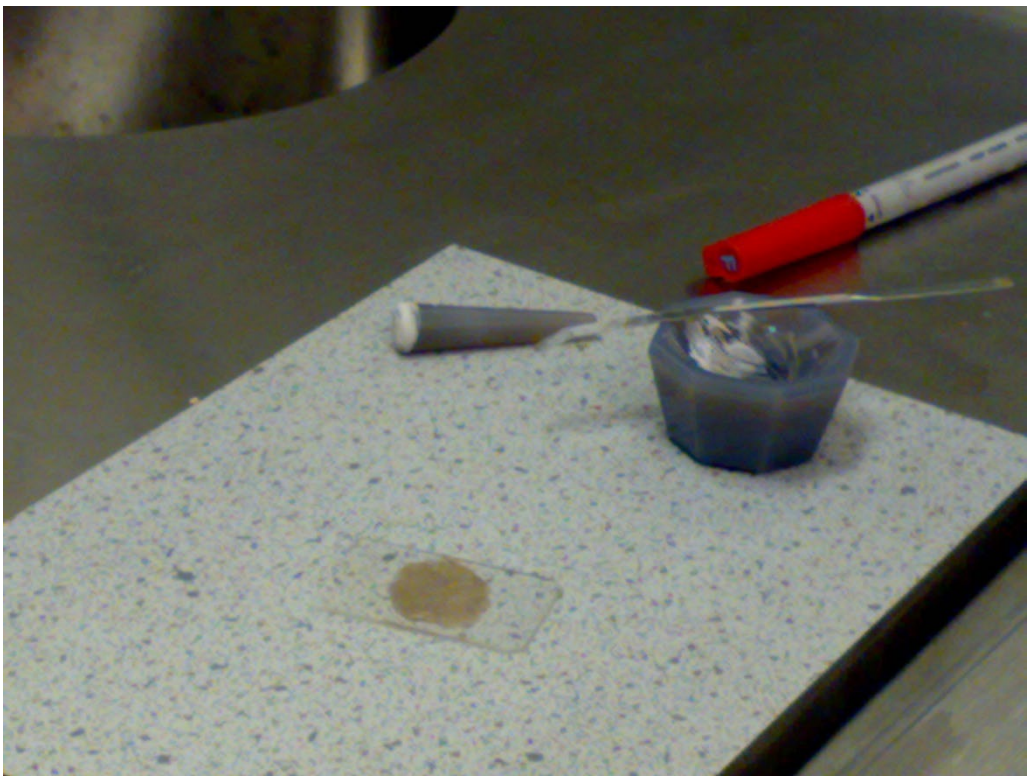
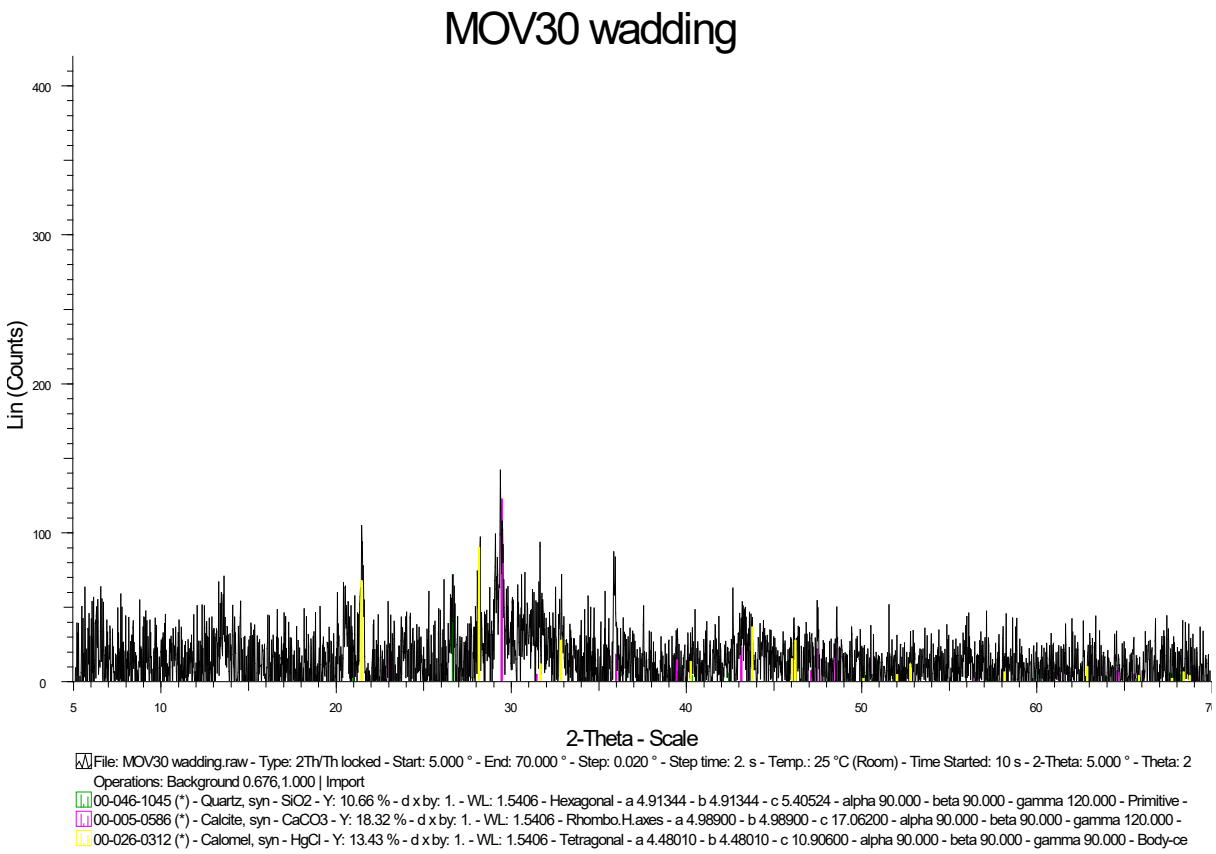
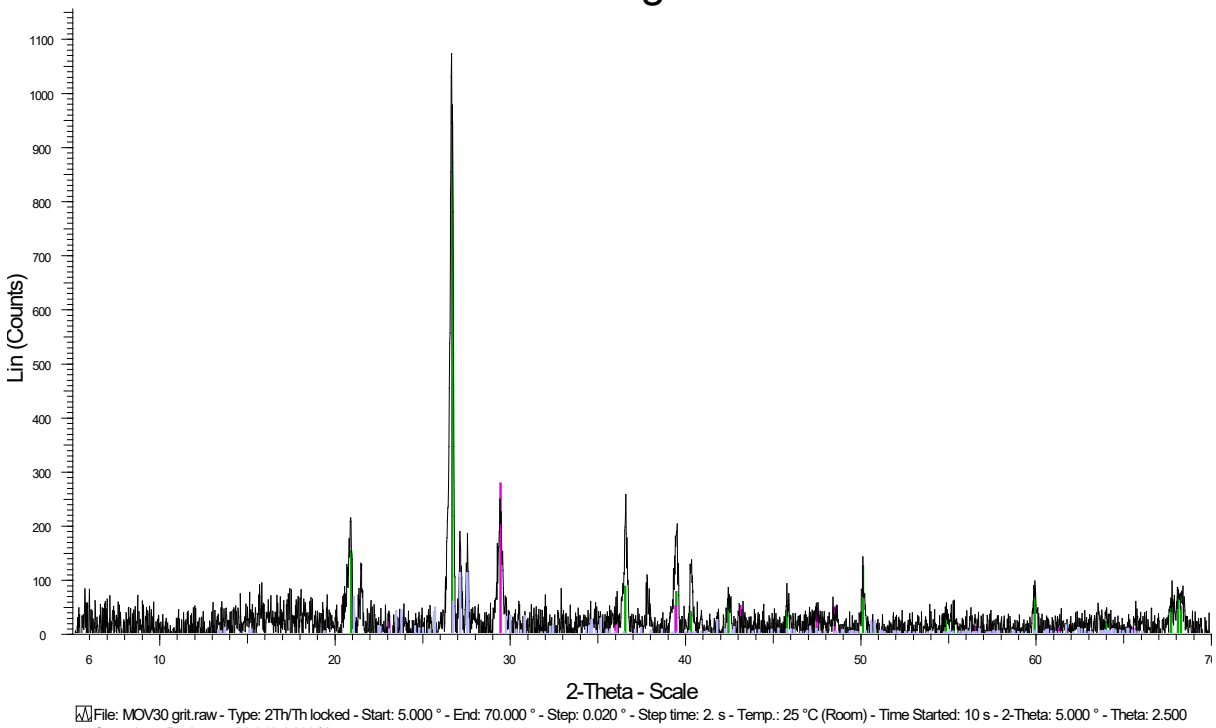


Photo 6: Stages of slide preparation carried out by Dr J Waters, for XRF analysis.

Appendix 3 : XRD analysis results for waste samples (MOV30/MO21) and plaster core samples (M10c, M15c, M16c, M16c paper and M17c).

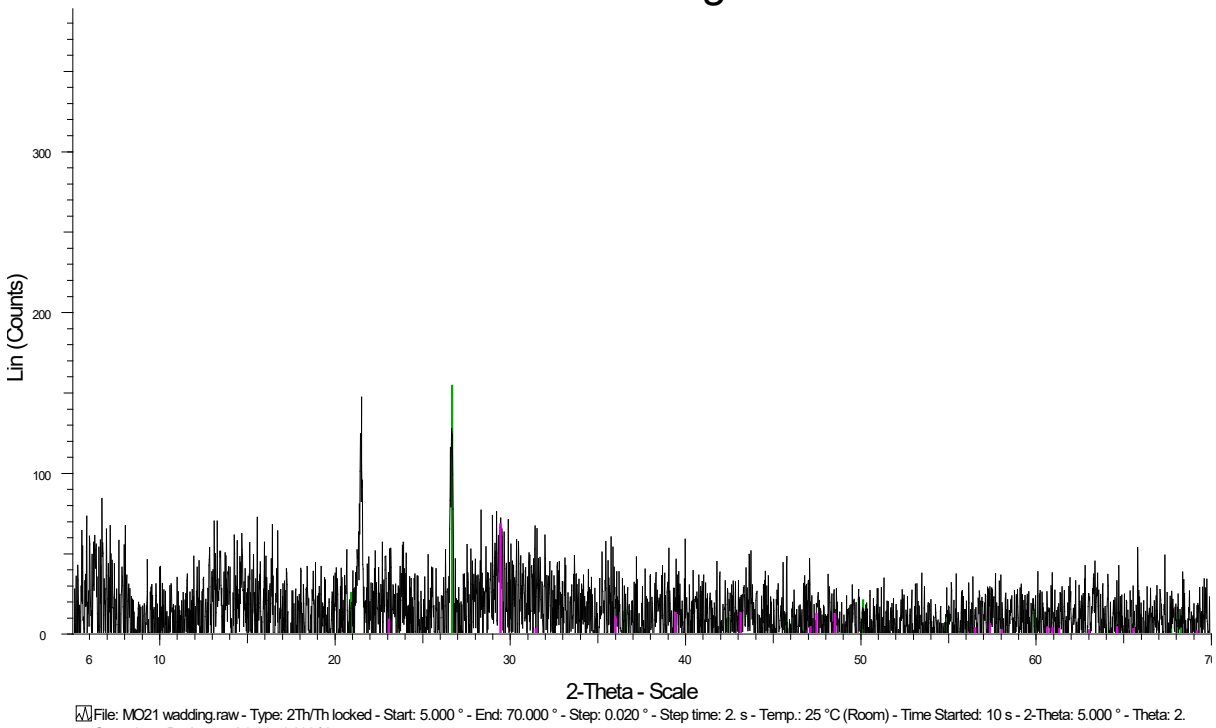


MOV30 grit



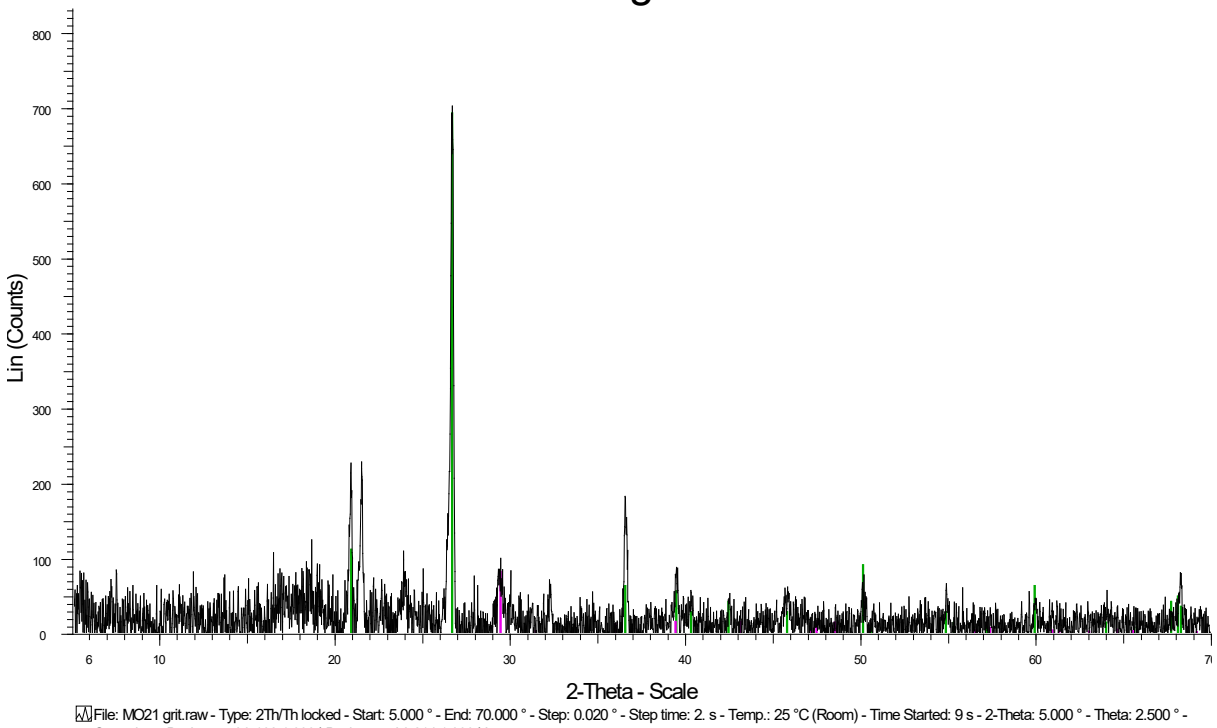
File: MOV30 grit.raw - Type: 2Th/Th locked - Start: 5.000 ° - End: 70.000 ° - Step: 0.020 ° - Step time: 2. s - Temp.: 25 °C (Room) - Time Started: 10 s - 2-Theta: 5.000 ° - Theta: 2.500
Operations: Background 0.813,1.000 | Import
00-046-1045 (*) - Quartz, syn - SiO2 - Y: 66.40 % - d x by: 1. - WL: 1.5406 - Hexagonal - a 4.91344 - b 4.91344 - c 5.40524 - alpha 90.000 - beta 90.000 - gamma 120.000 - Primitive -
00-005-0586 (*) - Calcite, syn - CaCO3 - Y: 19.41 % - d x by: 1. - WL: 1.5406 - Rhombo.H.axes - a 4.98900 - b 4.98900 - c 17.06200 - alpha 90.000 - beta 90.000 - gamma 120.000 -
01-077-0135 (C) - Microcline - K(Si0.75Al0.25)4O8 - Y: 7.78 % - d x by: 1. - WL: 1.5406 - Triclinic - a 8.57840 - b 12.96000 - c 7.21120 - alpha 89.700 - beta 115.967 - gamma 90.875 -

MO21 wadding



File: MO21 wadding.raw - Type: 2Th/Th locked - Start: 5.000 ° - End: 70.000 ° - Step: 0.020 ° - Step time: 2. s - Temp.: 25 °C (Room) - Time Started: 10 s - 2-Theta: 5.000 ° - Theta: 2.
Operations: Background 1.445,1.000 | Import
00-046-1045 (*) - Quartz, syn - SiO2 - Y: 25.51 % - d x by: 1. - WL: 1.5406 - Hexagonal - a 4.91344 - b 4.91344 - c 5.40524 - alpha 90.000 - beta 90.000 - gamma 120.000 - Primitive -
00-005-0586 (*) - Calcite, syn - CaCO3 - Y: 11.18 % - d x by: 1. - WL: 1.5406 - Rhombo.H.axes - a 4.98900 - b 4.98900 - c 17.06200 - alpha 90.000 - beta 90.000 - gamma 120.000 -

MO21 grit

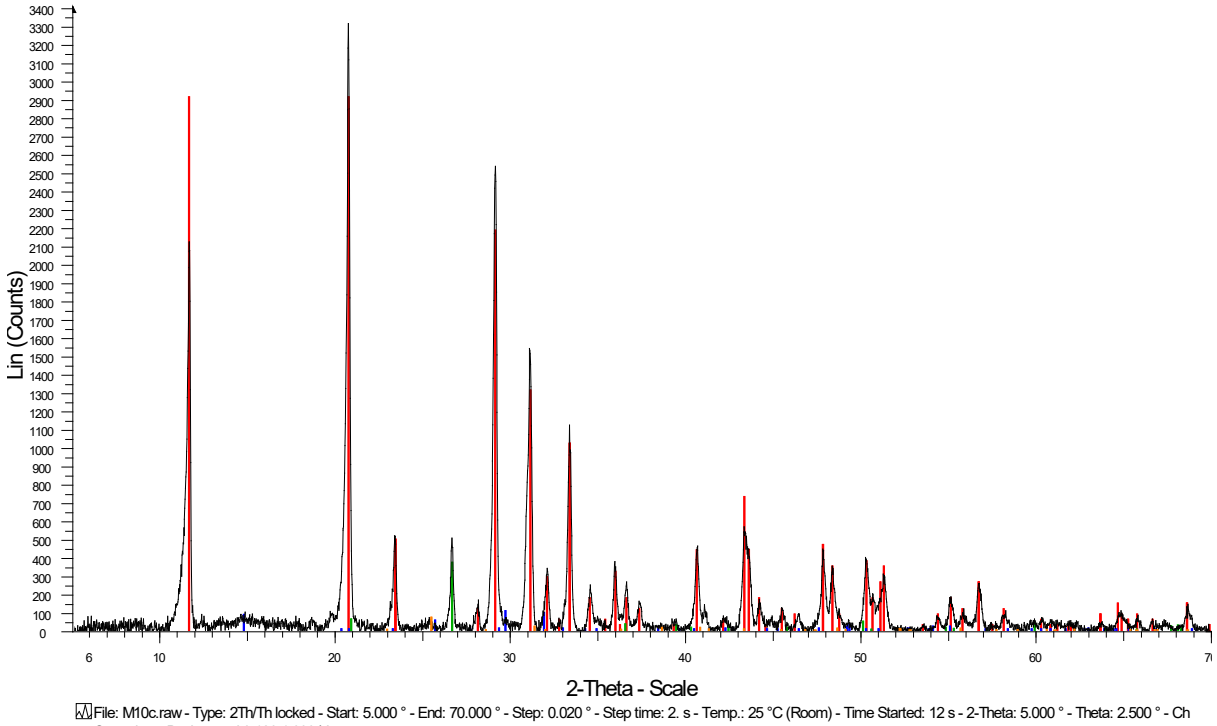


File: MO21 grit.raw - Type: 2Th/Th locked - Start: 5.000 ° - End: 70.000 ° - Step: 0.020 ° - Step time: 2. s - Temp.: 25 °C (Room) - Time Started: 9 s - 2-Theta: 5.000 ° - Theta: 2.500 ° - Operations: Background 3.162,1.000 | Background 0.214,1.000 | Import

00-046-1045 (*) - Quartz, syn - SiO2 - Y: 43.88 % - d x by: 1. - WL: 1.5406 - Hexagonal - a 4.91344 - b 4.91344 - c 5.40524 - alpha 90.000 - beta 90.000 - gamma 120.000 - Primitive -

00-005-0586 (*) - Calcite, syn - CaCO3 - Y: 5.13 % - d x by: 1. - WL: 1.5406 - Rhombo.H.axes - a 4.98900 - b 4.98900 - c 17.06200 - alpha 90.000 - beta 90.000 - gamma 120.000 - Pr

M10c



File: M10c.raw - Type: 2Th/Th locked - Start: 5.000 ° - End: 70.000 ° - Step: 0.020 ° - Step time: 2. s - Temp.: 25 °C (Room) - Time Started: 12 s - 2-Theta: 5.000 ° - Theta: 2.500 ° - Ch Operations: Background 0.120,1.000 | Import

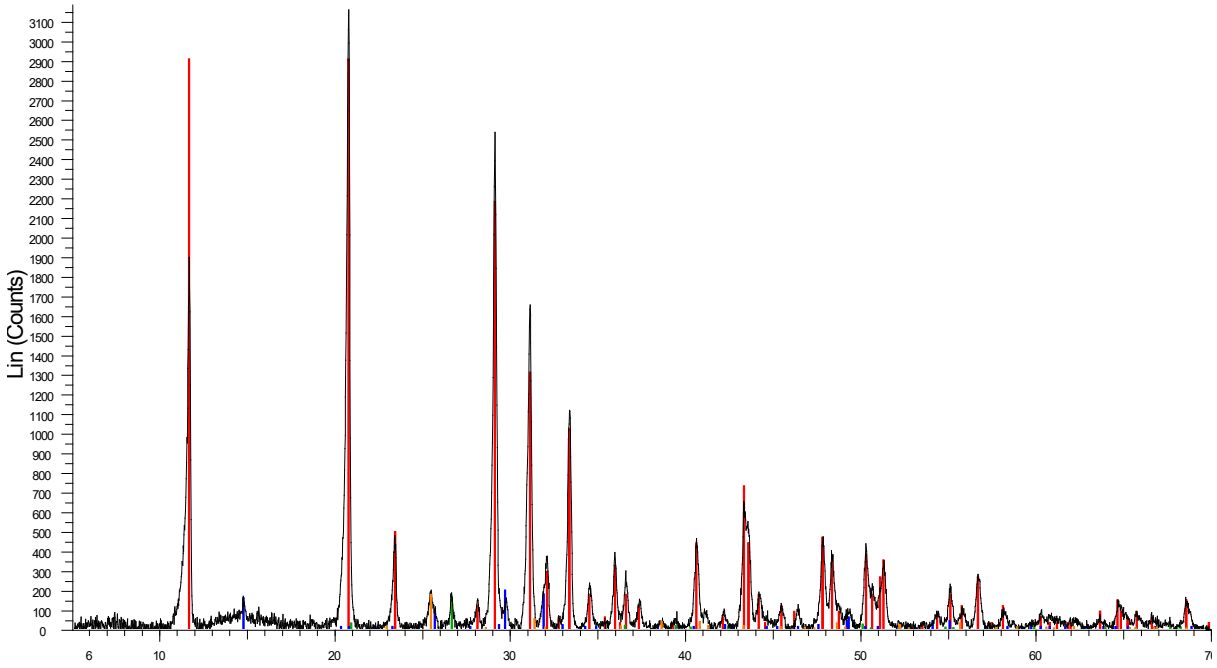
00-033-0311 (*) - Gypsum, syn - CaSO4·2H2O - Y: 82.71 % - d x by: 1. - WL: 1.5406 - Monoclinic - a 6.28450 - b 15.20790 - c 5.67760 - alpha 90.000 - beta 114.090 - gamma 90.000

00-041-0224 (I) - Bassanite, syn - CaSO4·0.5H2O - Y: 3.02 % - d x by: 1. - WL: 1.5406 - Monoclinic - a 12.02800 - b 6.93200 - c 12.69100 - alpha 90.000 - beta 90.183 - gamma 90.00

00-046-1045 (*) - Quartz, syn - SiO2 - Y: 10.48 % - d x by: 1. - WL: 1.5406 - Hexagonal - a 4.91344 - b 4.91344 - c 5.40524 - alpha 90.000 - beta 90.000 - gamma 120.000 - Primitive -

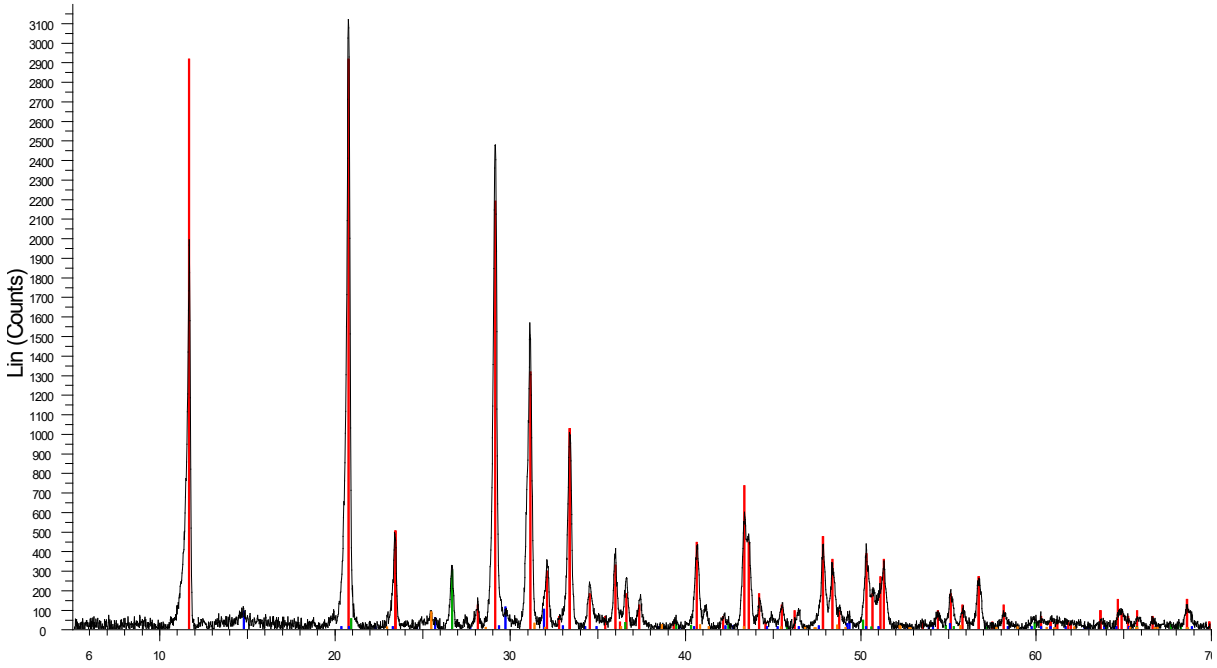
00-037-1496 (*) - Anhydrite, syn - CaSO4 - Y: 1.97 % - d x by: 1. - WL: 1.5406 - Orthorhombic - a 6.99330 - b 7.00170 - c 6.24110 - alpha 90.000 - beta 90.000 - gamma 90.000 - Bas

M15c



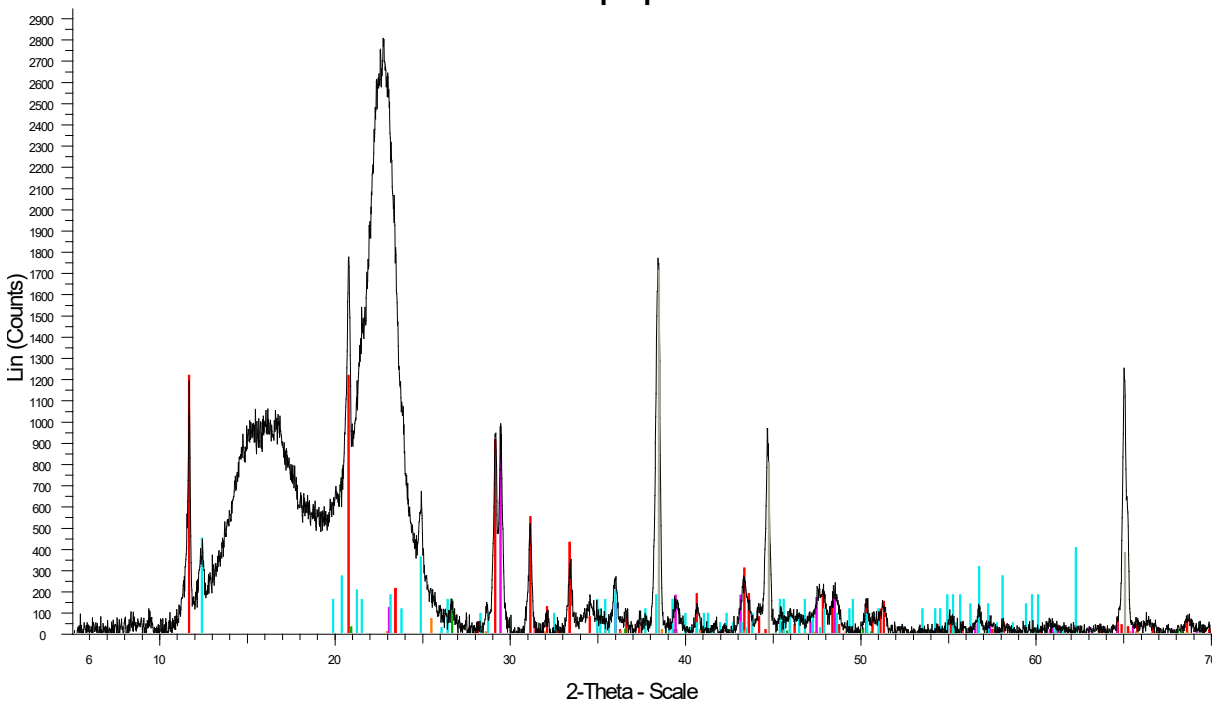
File: M15c.raw - Type: 2Th/Th locked - Start: 5.000 ° - End: 70.000 ° - Step: 0.020 ° - Step time: 2. s - Temp.: 25 °C (Room) - Time Started: 10 s - 2-Theta: 5.000 ° - Theta: 2.500 ° - Ch
Operations: Background 0.068,1.000 | Import
00-033-0311 (*) - Gypsum, syn - CaSO4·2H2O - Y: 86.59 % - d x by: 1. - WL: 1.5406 - Monoclinic - a 6.28450 - b 15.20790 - c 5.67760 - alpha 90.000 - beta 114.090 - gamma 90.000
00-041-0224 (I) - Bassanite, syn - CaSO4·0.5H2O - Y: 5.81 % - d x by: 1. - WL: 1.5406 - Monoclinic - a 12.02800 - b 6.93200 - c 12.69100 - alpha 90.000 - beta 90.183 - gamma 90.00
00-046-1045 (*) - Quartz, syn - SiO2 - Y: 4.88 % - d x by: 1. - WL: 1.5406 - Hexagonal - a 4.91344 - b 4.91344 - c 5.40524 - alpha 90.000 - beta 90.000 - gamma 120.000 - Primitive -
00-037-1496 (*) - Anhydrite, syn - CaSO4 - Y: 5.19 % - d x by: 1. - WL: 1.5406 - Orthorhombic - a 6.99330 - b 7.00170 - c 6.24110 - alpha 90.000 - beta 90.000 - gamma 90.000 - Bas

M16c



File: M16c.raw - Type: 2Th/Th locked - Start: 5.000 ° - End: 70.000 ° - Step: 0.020 ° - Step time: 2. s - Temp.: 25 °C (Room) - Time Started: 7 s - 2-Theta: 5.000 ° - Theta: 2.500 ° - Chi:
Operations: Background 0.120,1.000 | Import
00-033-0311 (*) - Gypsum, syn - CaSO4·2H2O - Y: 87.32 % - d x by: 1. - WL: 1.5406 - Monoclinic - a 6.28450 - b 15.20790 - c 5.67760 - alpha 90.000 - beta 114.090 - gamma 90.000
00-041-0224 (I) - Bassanite, syn - CaSO4·0.5H2O - Y: 3.19 % - d x by: 1. - WL: 1.5406 - Monoclinic - a 12.02800 - b 6.93200 - c 12.69100 - alpha 90.000 - beta 90.183 - gamma 90.00
00-046-1045 (*) - Quartz, syn - SiO2 - Y: 8.76 % - d x by: 1. - WL: 1.5406 - Hexagonal - a 4.91344 - b 4.91344 - c 5.40524 - alpha 90.000 - beta 90.000 - gamma 120.000 - Primitive -
00-037-1496 (*) - Anhydrite, syn - CaSO4 - Y: 2.42 % - d x by: 1. - WL: 1.5406 - Orthorhombic - a 6.99330 - b 7.00170 - c 6.24110 - alpha 90.000 - beta 90.000 - gamma 90.000 - Bas

M16c paper



File: M16c paper.raw - Type: 2Th/Th locked - Start: 5.000 ° - End: 70.000 ° - Step: 0.020 - Operations: Background 0.120,1.000 | Import

00-033-0311 (*) - Gypsum, syn - CaSO4·2H2O - Y: 36.02 % - d x by: 1. - WL: 1.5406 -

00-046-1045 (*) - Quartz, syn - SiO2 - Y: 4.57 % - d x by: 1. - WL: 1.5406 - Hexagonal -

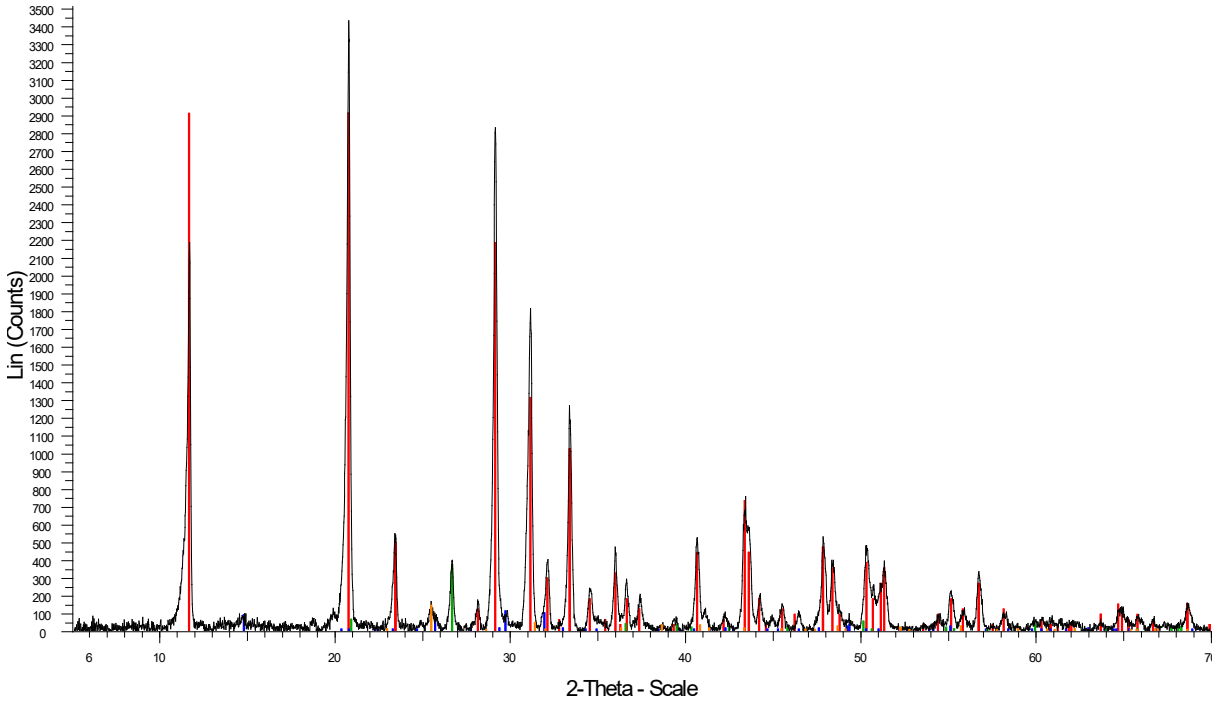
00-037-1496 (*) - Anhydrite, syn - CaSO4 - Y: 1.89 % - d x by: 1. - WL: 1.5406 - Orthorh

00-004-0787 (*) - Aluminum, syn [NR] - Al - Y: 50.77 % - d x by: 1. - WL: 1.5406 - Cubic

00-005-0586 (*) - Calcite, syn - CaCO3 - Y: 28.81 % - d x by: 1. - WL: 1.5406 - Rhombo.

00-014-0164 (I) - Kaolinite-1A - Al2Si2O5(OH)4 - Y: 13.17 % - d x by: 1. - WL: 1.5406 -

M17c



File: M17c.raw - Type: 2Th/Th locked - Start: 5.000 ° - End: 70.000 ° - Step: 0.020 ° - Step time: 2. s - Temp.: 25 °C (Room) - Time Started: 7 s - 2-Theta: 5.000 ° - Theta: 2.500 ° - Chi: - Operations: Background 0.120,1.000 | Import

00-033-0311 (*) - Gypsum, syn - CaSO4·2H2O - Y: 80.28 % - d x by: 1. - WL: 1.5406 - Monoclinic - a 6.28450 - b 15.20790 - c 5.67760 - alpha 90.000 - beta 114.090 - gamma 90.000

00-041-0224 (I) - Bassanite, syn - CaSO4·0.5H2O - Y: 2.93 % - d x by: 1. - WL: 1.5406 - Monoclinic - a 12.02800 - b 6.93200 - c 12.69100 - alpha 90.000 - beta 90.183 - gamma 90.00

00-046-1045 (*) - Quartz, syn - SiO2 - Y: 10.17 % - d x by: 1. - WL: 1.5406 - Hexagonal - a 4.91344 - b 4.91344 - c 5.40524 - alpha 90.000 - beta 90.000 - gamma 120.000 - Primitive -

00-037-1496 (*) - Anhydrite, syn - CaSO4 - Y: 3.81 % - d x by: 1. - WL: 1.5406 - Orthorhombic - a 6.99330 - b 7.00170 - c 6.24110 - alpha 90.000 - beta 90.000 - gamma 90.000 - Bas

Appendix 4

-----Original Message-----
From: John Waters [mailto:John.Waters@manchester.ac.uk]
Sent: 11 March 2010 12:18
To: Melanie Taylor
Cc: catherine.davidge@manchester.ac.uk;
david.a.barker@manchester.ac.uk
Subject: Re: Rutherford Building samples for XRD

Hi Melanie,

Here's the data. I'm afraid the assignment of the peaks wasn't exactly straight-forward.

The plasterboard samples have a peak at ~41o that I can't confidently match to anything in the database. The plasterboard paper sample has Al peaks because I remounted it in a different sample holder to get it to lie flat enough to scan. I used Al foil to raise the sample to the right height, without expecting nearly so much signal from the foil itself. The broad peaks in the pattern for the paper sample are from the paper itself.

The only solid evidence of mercury is in the MOV30 wadding, where we see calomel. One of the calomel peaks overlaps a background peak from the vaseline I used to mount the material on the slides (at 21.45o) but none of the other peaks for calomel are obvious in the other samples, so if it's there it's on the brink of the detection limit.
There are a few peaks which I can't assign with any confidence, which is nothing unusual. I can only auggest that you use a different technique to find out what elements are present, then I can use that data to help cut down the myriad possibilities and work out what those peaks relate to.

If you've any question, please ask away. The uxd files can be opened in excel (open excel first then the files and choose 'space separated'
values).

How should I go about disposing of the material on the slides?

John