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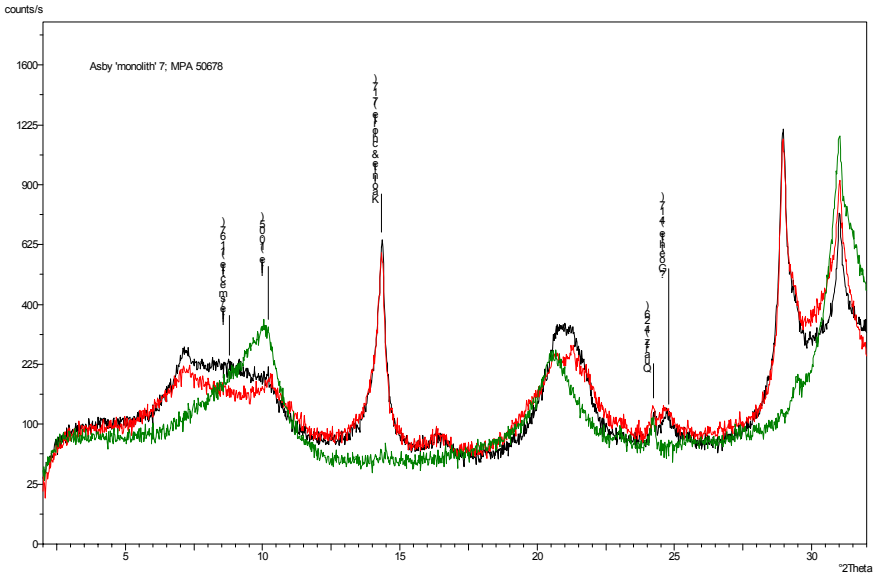
X-ray diffraction analysis of a palaeokarst sample from Asby, Cumbria

Integrated Geoscience Surveys (Northern Britain) Programme

Internal Report IR/02/010



BRITISH GEOLOGICAL SURVEY

INTERNAL REPORT IR/02/010

X-ray diffraction analysis of a palaeokarst sample from Asby, Cumbria

J A McKervey

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Front cover

X-ray diffraction traces for the < 2 micron fraction of the sample MPA 50678.

Bibliographical reference

MCKERVEY, J A. 2002. X-ray diffraction analysis of a palaeokarst sample from Asby, Cumbria. *British Geological Survey Internal Report*, IR/02/010. 10pp.

Keyworth, Nottingham British Geological Survey 2002

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TABLES

1 Introduction

This report presents the results of X-ray diffraction analysis of the sample Asby 'monolith' 7, a palaeokarst sample from pits in the Carboniferous limestones near Asby, Cumbria (Riding, 2002 for further details). The purpose of this report is to identify the mineralogy of the sample by X-ray diffraction analysis. In addition to X-ray diffraction analysis, a surface area determination was made using the 2-ethoxyethanol (EGME) method. The sample is listed in Table 1.

Table 1: Sample list

Sample	Lab code	Description
Asby 'monolith' 7; MPA50678	H140	orange/brown mudstone with some sub-angular rock fragments

2 Sample preparation

2.1 WHOLE-ROCK ANALYSIS

The sample was first disaggregated using a pestle and mortar and then approximately 5 g was hammer-milled to pass a 0.12 mm screen. Approximately 3 g of this material was micronised under acetone for 10 minutes to provide a finer and more uniform particle size. The powder was then back-loaded into a standard aluminium sample holder for XRD analysis.

2.2 <2 MICRON ANALYSIS

Approximately 15 g of the disaggregated sample was dispersed in ~ 150 ml of de-ionised water using a reciprocal shaker overnight followed by ultrasound treatment for ~ 4 mins. The suspension was then wet sieved on 63 μm and the >63 μm fraction dried and retained. The <63 μm fraction was placed in a measuring cylinder and allowed to stand. To prevent flocculation, 1 ml of 0.1M 'Calgon' (sodium hexametaphosphate) was added to each cylinder. After a period determined from Stokes' Law, a nominal <2 μm fraction (the 'clay' fraction) was removed and dried at 55°C. The 2-63 μm fraction was also collected, dried and retained. 100 mg of the <2 μm material was then re-suspended in ~3 ml of deionised water and pipetted onto a ceramic tile in a vacuum apparatus to produce an oriented mount. The mounts were then Ca-saturated using 2 ml 0.1M $CaCl_2.6H_2O$ solution and finally washed twice to remove excess reagent.

3 X-ray diffraction analysis

XRD analysis was carried out using a Philips PW1700 series diffractometer fitted with a cobalt-target tube and operated at 45 kV and 40 mA. The whole-rock samples were scanned from 3-50 °2θ at 0.7 °2θ/minute. The <2 μm oriented mounts were scanned from 2-32 °2θ at 0.55 °2θ/minute as air-dried mounts, after glycol-solvation and after heating to 550°C for 2 hours.

Diffraction data were analysed using Philips X'Pert software coupled to an International Centre for Diffraction Data (ICDD) database running on a Gateway personal computer system.

4 Surface area determination (EGME method)

Approximately 1.1g of the hammer-milled powder was weighed into an aluminium dish and placed in a desiccator containing anhydrous phosphorous pentoxide alongside a clay standard sample (Patterson Court Blue bentonite). The desiccator was evacuated and allowed to stand overnight before re-weighing the samples. The samples were then saturated with 2-ethoxyethanol and placed in the desiccator now containing dry calcium chloride. After approximately 1.5 hours the desiccator was evacuated and left overnight. The samples were then weighed and thus the weight of 2-ethoxyethanol absorbed was determined and the surface area calculated. Finally, a small correction was applied to the surface area based on the result of the clay standard.

Smectite has a surface area of 800 m^2/g whereas other clay minerals and quartz have surface areas typically less than 100 m^2/g and 1 m^2/g respectively. Such a difference in value means that the surface area of a sample can provide a useful estimate of its smectite content.

5 Results

Following identification of the mineral species present in the samples, whole-rock mineral quantification was achieved using the Reitveld refinement technique using Siroquant v.2.5 software. This method avoids the need to produce synthetic mixtures and involves the least squares fitting of measured to calculated XRD profiles using a crystal structure databank. Errors for the quoted mineral concentrations are probably ±5% accuracy for quartz; larger errors are possible for the quoted clay mineral, 'mica' and feldspar concentrations.

The results of X-ray diffraction analysis and the surface area determination are listed in Tables 2 and 3.

The sample is composed predominantly of quartz (~71%) and kaolinite (~15%). Minor amounts of feldspar (albite and k-feldspar), 'mica' (undifferentiated mica; however illite and a mixed-layer illite/smectite are present the <2 μm fraction), chlorite, ?hematite, ?pyrite and ?goethite are also present.

The surface area of the sample is 80 m^2/g, consistent with the clay mineral assemblage of kaolinite, chlorite, illite and a mixed layer illite/smectite.

(See the Appendix for annotated X-ray diffraction traces)

References

RIDING, J B. 2002. A palynological study of palaeokarst deposits near Asby, Cumbria. *British Geological Survey Internal Report*, IR/02/008.

Table 2: Summary of whole-rock analysis

Sample	Lab code	Whole-rock mineralogy (%)								Surface Area (m^2/g)
		Quartz	Albite	K-feldspar	'Mica'	Kaolinite	Chlorite	?Hematite	?Pyrite	
Asby 'monolith' 7, MPA 50678	H140	71	2	5	5	15	2	<1	<1	80

'mica'=undifferentiated mica species

Table 3: Summary of <2 μm X-ray diffraction analysis

Sample	Lab code	Clay mineralogy	Non-clay mineralogy
Asby 'monolith' 7, MPA 50678	H140	kaolinite, chlorite, illite and illite/smectite	quartz and ?goethite

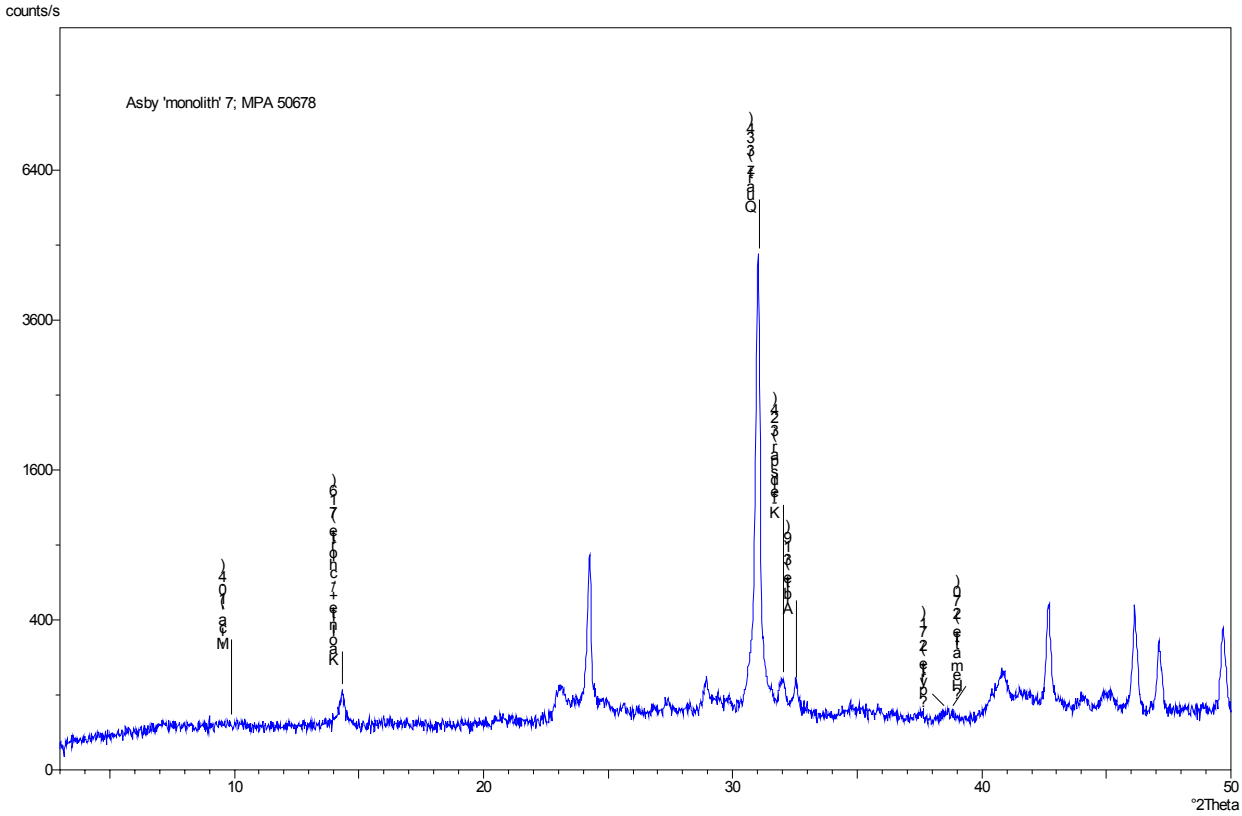
Appendix

Key to the whole-rock X-ray diffraction trace:

X-axis: °2θ Co-Kα
Y-axis: (Intensity) counts per second
Peak Labels: mineral and d-spacing (Å)
Blue trace: whole-rock powder

Key to the <2 μm diffraction traces:

X-axis: °2θ Co-Kα
Y-axis: (Intensity) counts per second
Peak Labels: mineral and d-spacing (Å)
Black trace: air-dried oriented mount
Red trace: glycol-solvated oriented mount
Green trace: heated (550°C for 2 hours) oriented mount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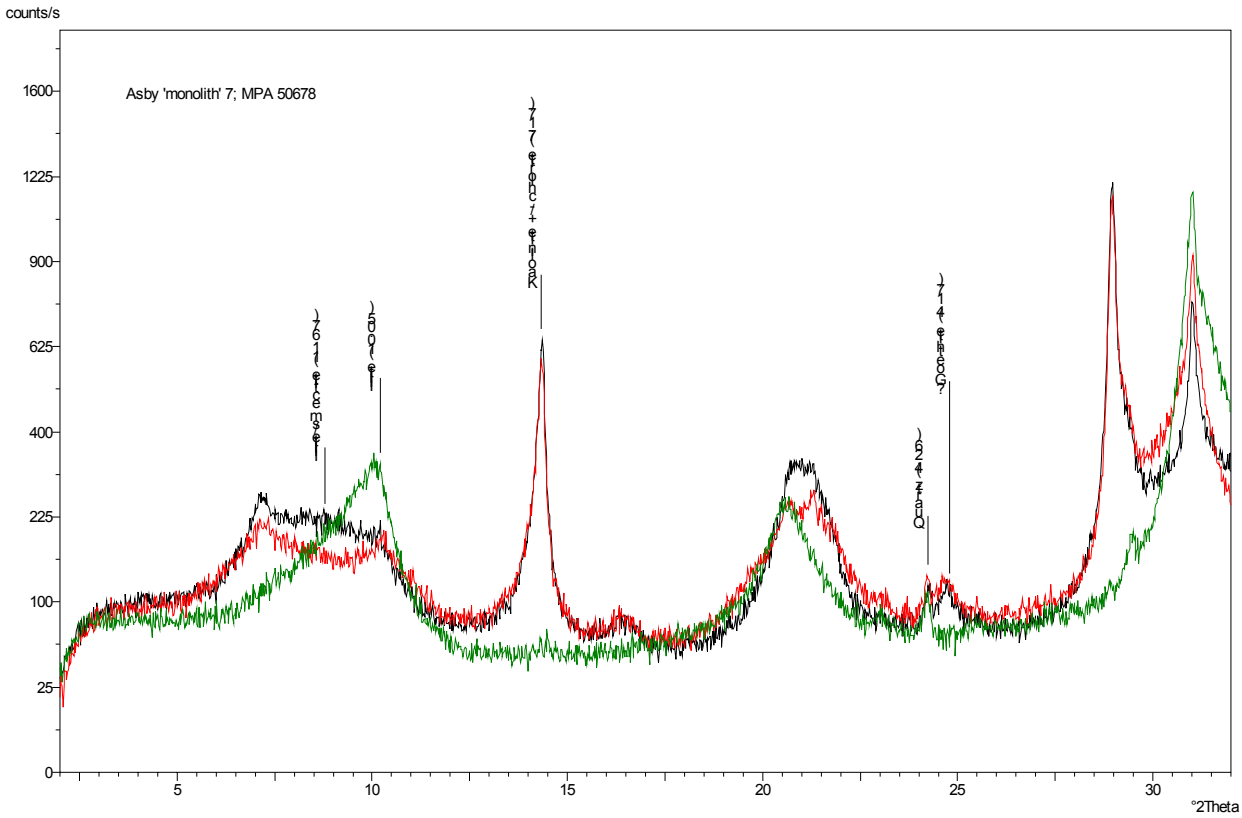
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100
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0
5
10
15
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25
30
°2Theta