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The mineralogy of mudrocks from the Lias Group of England

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The mineralogy of mudrocks from the Lias Group of England

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Key words

Lias Group, mineralogy, clay minerals, engineering geology, maturity, pyrite

Front cover

Coastal exposure of the Lias Group, Staithes, Yorkshire (photo. PRN Hobbs)

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Foreword

This report is the published product of a study by the British Geological Survey (BGS) and forms part of the fourth stage of the ongoing BGS programme, 'Engineering Geology of UK Rocks and Soils.' This report aims to provide mineralogical data to help explain the geotechnical behaviour of the UK Lias Group. This project follows similar projects dealing with soils and rocks of the Gault Formation, the Mercia Mudstone Group, and the Lambeth Group.

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Summary

This report describes the results of mineralogical analysis of a suite of sedimentary rocks from the Lias Group of England. The work was carried out as part of the ongoing BGS research programme, 'Engineering Geology of UK Rocks and Soils.'

The first part of the report gives an introduction to the Lias Group geology and a summary of previous mineralogical studies of these rocks. A summary of analytical methods employed (X-ray diffraction analysis and surface area determinations) is then provided and the results discussed with reference to their likely effect on the engineering performance of the Lias Group.

While analyses are in general agreement with previous studies, important differences in terms of swelling clay mineral species and carbonate content are highlighted between samples taken from the northern Cleveland Basin and the southern Worcester and Wessex Basins. Such differences in clay mineralogy also provide important information on the burial histories of the Lias Group across England.

The detection of pyrite in almost all the samples analysed together with sporadic gypsum and jarosite means that any concrete engineering sited in the Lias Group rocks risks corrosion by acidic groundwaters and the formation of thaumasite.

1 Introduction

The Lower Jurassic (Lias, or in strict lithostratigraphical terminology the Lias *Group*) of the UK forms a thin, approximately continuous northeast-southwest trending outcrop from Redcar on the Cleveland coast in the north through Lincolnshire, the Midlands, the Cotswolds and Somerset to Lyme Regis on the Dorset coast in the south. More isolated outcrops of the Lias also occur in west Somerset, south Wales, Cheshire, the Solway Basin and in Scotland (Sutherland, Gruinard Bay, Morvern and the Isles of Skye, Mull and Raasay). In general, the strata dip very gently towards the east or south-east beneath younger beds, and so the Lias is present at depth eastwards (downdip) of the main outcrop other than in the London area (Figure 1) (Cox *et al.*, 1999). It is predominantly formed of shallow marine intercalated mudstones, shales and muddy limestones which were deposited in an extensive epicontinental (or epeiric) sea during the major world-wide transgression that marked the end of the Triassic (Anderton *et al.* 1979). Thick mudstone sequences were deposited in a series of basins while thin calcareous and sandy deposits were formed in shallow shoals. Three structural highs developed, the Mendips, Moreton-in-the-Marsh and Market Weighton which separated areas of substantial subsidence and which would affect sedimentation throughout the Jurassic. The Lias is entirely marine and characterised by great lateral variations in thickness of sedimentation.

The Lias has been traditionally split into Lower, Middle and Upper divisions based initially on lithological character. The Lower and Upper Divisions are dominated by shales while the Middle Lias contains sandy shales, sandstones and oolitic ironstones. However, when greater emphasis was placed on the importance of fossils for correlation the traditional divisions Lower, Middle and Upper Lias became used variously as litho-, bio- or chronostratigraphical units and, as a result, positioning of their boundaries by different workers has been inconsistent and numerous other stratigraphical terms have been used to describe subdivisions of the Lias Group. In an attempt to rationalize the plethora of names, and clarify understanding, a revised scheme of lithostratigraphical classification for the Lias Group has recently been developed by the BGS with the support of the Geological Society of London (Cox *et al.*, 1999) which divides the Lias Group into no more than 12 formations (Figure 2). The outcrop has also been divided into four depositional areas - the Cleveland Basin, East Midlands Shelf, Worcester Basin (plus adjoining Bristol-Radstock Shelf) and Wessex Basin (including parts of Somerset and south Wales).

This study forms part of the fourth stage of the ongoing BGS programme, ‘Engineering Geology of UK Rocks and Soils’ and aims to provide mineralogical data to help explain the geotechnical behaviour of the UK Lias. This project follows similar projects dealing with soils and rocks of the Gault Formation, the Mercia Mudstone Group, and the Lambeth Group. Due to their high surface area, residual charge and interaction with water, clay minerals (and smectite in particular) are most frequently cited as the reason for the shrink-swell behaviour noted in many fine-grained sedimentary rocks. For this reason, this study principally aimed to evaluate the nature of the clay minerals present in the Lias Group samples.

However, this study also focussed on detecting the presence of sulphate-bearing species (e.g. pyrite, gypsum etc). Oxidation of pyrite in the environment leads to the formation of sulphuric acid which considerably reduces the pH of groundwater. Where such acidic groundwaters make contact with concrete engineering at low temperatures (e.g. foundations, motorway bridges etc), the main cementitious binder C-S-H is converted to thaumasite (a non-binding calcium carbonate silicate sulphate hydrate) resulting in deterioration and failure (e.g. Hobbs & Taylor, 2000; Bensted, 1999; Burkart, *et al.* 1999). Greater awareness of the potential problems that thaumasite can cause has arisen with the increased use of limestone fillers in cements, the common employment of limestone aggregates in concrete and the introduction of Portland limestone cements, together with the realisation that structural foundations of buildings are, on average, below ambient temperature and are within the optimum temperature range for thaumasite to be formed (Bensted, 1999). A knowledge of the presence of sulphate-bearing species in Lias Group sediments is therefore crucial to planning foundation construction in such strata.

2 Previous mineralogical studies of the Lias Group in England

In view of its relatively good coastal and quarry exposure, it is perhaps surprising that studies of the mineralogy of the onshore English Lias Group are relatively few. Several brief studies were carried out in the 1960's (e.g. Hallam, 1960; Cosgrove & Salter, 1966). These indicated that the Lias mudstones typically contain a clay mineral assemblage of illite, interlayered illite/smectite and smectite with minor kaolinite and chlorite.

Pye & Krinsley (1986) produced a petrographic, geochemical and mineralogical study of the Whitby Mudstone Formation from the Cleveland Basin using the then recent development of backscattered scanning electron microscopy. Using a combination of techniques, they differentiated three different facies; normal, restricted and bituminous. The normal facies (lower Grey Shale Member and upper Alum Shale Member) were rich in quartz, micas, chlorite and kaolinite, pyrite together with varying amounts of calcite and siderite (generally <10%) and traces of feldspar and carbonate-apatite. The restricted facies (upper Grey Shale Member) was mineralogically similar to the lower Grey Shale Member but with a lower siderite content. The same restricted facies (lower Alum Shale Member) contained more kaolinite but less quartz and chlorite than the restricted facies Grey Shales. The bituminous shale facies (~Mulgrave Shale Member) was found to be composed of quartz, kaolinite, mica, illite-smectite, chlorite, pyrite and calcite with subsidiary dolomite, feldspar and carbonate-apatite but no siderite. Textural evidence suggested that the authigenic mineralogy was predominantly early diagenetic and that the differences observed were due to changes in the prevailing sea bottom conditions.

As part of a site investigation for a low-level radioactive waste repository at Fulbeck, Lincolnshire, Bloodworth *et al.* (1987) carried out an extensive mineralogical and lithogeochemical study of the Lower and Middle Lias sequence of interbedded mudstones and limestones. Clay mineral assemblages were found to be dominated by illite with subordinate kaolinite, minor chlorite and interlayered illite/smectite (I/S). Surface areas for the mudstones varied from 112 to 172 m^2/g with a mean of 140 m^2/g. Evolved gas and X-ray diffraction analyses revealed that pyrite was ubiquitously developed throughout the interval, typically forming 1-2% but occasionally reaching over 20% in some limestone samples. Trace amounts of gypsum were only sporadically detected.

Mitchell (1992) carried out an XRD study of the clay mineralogy of a 200 m thick borehole sequence of Lias mudstones from the Copperhill Quarry, near Ancaster, Lincolnshire, in order to identify any potential clay 'marker' horizons or distinctive variations in clay mineral assemblage. The Lias here was found to be composed of the non-clays: quartz, mica and pyrite with traces of feldspar and calcite. Clay mineral assemblages were dominated by kaolinite with illite, chlorite and I/S. However, clay mineral abundances were based on a direct comparison of uncorrected peak intensity data.

A more recent study by Bessa & Hesselbo (1997) attempted to correlate the Lower Lias in southwest Britain using outcrop-based spectral gamma-ray spectroscopy. However, despite the fact that the gamma-ray signatures of such lithologies are predominantly determined by their clay mineralogy, no attempt was made to characterise the mineralogy of the mudstones. A similar study for the Cleveland Basin by van Buchem *et al.* (1992) presented limited clay mineralogical and geochemical data.

As a part of the ongoing BGS programme, 'Engineering Geology of UK Rocks and Soils', Kemp & Hards (2000) investigated the mineralogy of Lias samples from two site investigation boreholes sited near the M5 motorway in Gloucestershire. They found generally similar mineralogies to those described by previous workers with non-clay mineral assemblages typically composed of carbonates (calcite and ankerite), quartz, feldspar (K-feldspar and albite), 'mica', gypsum and pyrite. Clay mineral assemblages were generally formed of illite (c.40%), kaolinite (c.35%), smectite (c.20%) and chlorite (c.5%). However, smectite contents were found to increase at certain levels to c.30%.

3 Samples

Between 18th and 20th October and 14th and 16th November, 2000, field visits were made by BGS staff to several Lias Group outcrops in England. The aims of these visits were to examine the lithostratigraphy of the Lias Group across the country, to collect disturbed samples for geotechnical and mineralogical tests and to identify sites for the future collection of undisturbed samples for geotechnical tests.

A total of twenty seven disturbed, outcrop samples were submitted for mineralogical analysis. The first 'southern' batch were removed from quarry locations in Gloucestershire and Warwickshire and the Dorset coast representing the Worcester Basin, Wessex Basin and western edge of the East Midlands Shelf (Table 1) while the second 'northern' batch were taken from the Yorkshire coast between Staithes and Ravenscar to represent the Cleveland Basin (Table 2). Further details of the field sampling are given in Hobbs & Sumbler (2001).

4 Laboratory methods

4.1 GENERAL SAMPLE PREPARATION

Representative portions of each sample were separated, dried at 55ºC and jawcrushed. Approximately ¼ of the jawcrushed material was then hammer-milled to pass a 125 µm screen for surface area and whole-rock X-ray diffraction analyses.

4.2 SURFACE AREA DETERMINATION

Surface area determinations were performed on all samples using the 2-ethoxyethanol (ethylene glycol monoethyl ether, EGME) technique. The method is based on the formation of a monolayer of EGME molecules on the clay surface under vacuum. Aluminium dishes containing approximately 1.1 g hammer-milled sample/clay standard (Patterson Court Blue bentonite) were placed in a desiccator containing anhydrous phosphorus pentoxide. The desiccator was evacuated and allowed to stand overnight before the dishes were reweighed. The samples were then saturated with 2-ethoxyethanol and placed in a second desiccator containing dry calcium chloride. After 1½ hours, the desiccator was evacuated and left overnight. The sample was then rapidly re-weighed and the weight of 2-ethoxyethanol absorbed determined and the surface area calculated. Finally, a correction based on the Patterson Court Blue standard was applied.

Smectite has a surface area of c.800 m^2/g while 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.

4.3 X-RAY DIFFRACTION ANALYSIS

4.3.1 Preparation

In order to achieve a finer and uniform particle-size for whole-rock XRD analysis, approximately 3 g portions of the hammer-milled material was micronised under acetone for 5 minutes and dried at 55°C. The dried material was then disaggregated in a pestle and mortar and back-loaded into a standard aluminium sample holder for analysis.

Approximately 15 g subsamples of the jawcrushed material was dispersed in distilled water using a reciprocal shaker combined with treatment with ultrasound. The suspension was then sieved on 63 µm and the <63 µm material placed in a measuring cylinder and allowed to stand. In order to prevent flocculation of the clay crystals, 2 ml of 0.1M 'Calgon' (sodium hexametaphosphate) was added to each suspension. After a period dictated by Stokes' Law, a nominal <2 µm fraction was removed and dried at 55°C. 100 mg of the <2 µm material was then re-suspended in a minimum of distilled water and pipetted onto a ceramic tile in a vacuum apparatus to produce an oriented mount. The mounts were Ca-saturated using 2 ml 0.1M $CaCl_2.6H_2O$ solution and washed twice to remove excess reagent.

4.3.2 Analysis

XRD analysis was carried out using a Philips PW1700 series diffractometer fitted with a cobalt-target tube and operated at 45kV and 40mA. Whole-rock samples were scanned from 3-50 °2θ at 0.69 °2θ/minute. The <2 µm samples were scanned from 2-32 °2θ at 0.54 °2θ/minute as air-dry mounts, after glycol-solvation and after heating to 550°C for 2 hours.

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

4.3.3 Whole-rock quantification

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, calcite, dolomite and pyrite. Larger errors are possible for the quoted 'mica', chlorite, smectite, kaolinite, illite/smectite, gypsum, feldspar and jarosite concentrations.

4.3.4 XRD-profile modelling

In order to gain further information about the nature of the clay minerals present in the samples, modelling of the XRD profiles was carried out using Newmod-for-Windows™ (Reynolds & Reynolds, 1996) software.

Modelling was also used to assess the relative proportions of clay minerals present in the <2 µm fractions by comparison of sample XRD traces with Newmod-for-Windows™ modelled profiles. The modelling process requires the input of diffractometer, scan parameters and a quartz intensity factor (instrumental conditions), and the selection of different sheet compositions and chemistries. In addition, an estimate of the crystallite size distribution of the species may be determined by comparing peak profiles of calculated diffraction profiles with experimental data. By modelling the individual clay mineral species in this way, *mineral reference intensities* were established and used for quantitative standardization following the method outlined in Moore & Reynolds (1997).

5 Results

The results of whole-rock XRD and surface area analyses are shown in Table 3 and Figure 3. <2 µm clay mineral XRD analyses are summarised in Table 4 and Figure 4. Example labelled whole-rock and <2 µm clay mineral XRD traces are shown in the Appendix.

5.1 WHOLE-ROCK MINERALOGY AND SURFACE AREA

Up to thirteen different mineral phases were identified and quantified in the Lias Group samples. The presence of significant quantities of several different clay minerals and such complex mineralogies required many stages of refinement using the Siroquant software and resulted in mean chi-squared values of 4.5. Chi-squared values of <3 indicate a well refined pattern.

The non-clay mineralogy of the Lias Group rocks is composed of quartz, calcite, dolomite, feldspar (K-feldspar and albite), 'mica' (undifferentiated mica species), pyrite, gypsum and jarosite. The 'beef' sample is almost totally composed of calcite with minor contaminants. In overall terms, the remaining samples are predominantly composed of quartz (6-52%, mean 30%), calcite (nd-81%, mean 18%), 'mica' (8-41%, mean 28%) and kaolinite (2-22%, mean 14%). The remaining minerals typically form <3% but may reach more elevated levels in selected samples.

From whole-rock XRD analysis (Figure 3), it is clearly apparent that the samples from the East Midlands Shelf, Worcester and Wessex Basins are highly calcareous (nd-81%, mean 31%) when compared to the Cleveland Basin samples which often contain no carbonate species or are only poorly calcareous (nd-24%, mean 3%). Dolomite is also more common in samples from the south (nd-14%, mean 3%) than in the north (nd-3%, mean 1%). Similarly the samples from the Wessex and Worcester Basins contain discrete smectite while only interlayered illite/smectite was detected in the East Midlands Shelf and Cleveland Basin samples (see below). The southern batch, excluding the 'beef' sample, has a mean surface area of 110 m^2/g but a relatively large range of values from 27 to 203 m^2/g. Surface areas for the northern batch are smaller in comparison with a mean of 85 m^2/g and a range of 24 to 134 m^2/g.

Quartz contents are approximately similar for the Wessex and Worcester Basins (6-52%, mean 27%) and East Midlands Shelf and Cleveland Basin (22-47%, mean 33%) samples while feldspar contents (predominantly albite) are typically low (mean 2%) but reach 9% in a few samples. Of the sulphur-bearing species, pyrite appears to be commonly developed (nd-6%, mean 2%) throughout the Lias Group while gypsum and jarosite are more sporadic but may form up to 12% sample.

5.2 CLAY MINERALOGY

The Lias Group samples from the Cleveland Basin show relatively uniform clay mineral assemblages (Figure 4). Newmod-for-Windows modelling suggests that a typical <2 µm fraction is composed of 48% illite/smectite (I/S), 27% illite, 19% kaolinite and 6% chlorite. However, modelling of the XRD traces is hindered by the almost complete overlap of peaks from different clay mineral species. For example, modelling of the chlorite is largely based on the d_{003} (4.73Å) spacing as the d_{002} (7.1Å) and d_{004} (3.54Å) spacings are superimposed on the kaolinite d_{001} (7.1Å) and d_{002} (3.58Å) peaks. In addition to peak overlap problems, the broad peak profiles of the I/S, produced by its relatively small crystallite size, leaves only the d_{001} (c.11 Å) adequately resolved for modelling. The I/S component was therefore necessarily modelled using the air-dry diffraction trace.

Based on this limited data, modelling suggests that the I/S is 90% illite and 10% smectite R0 ordered interlayered clay which has a mean defect-free distance of 3 layers and a size range of 1 to 15 (10Å) units. Illite has a mean defect-free distance of 7 layers and a size range of 1 to 28

(10Å) units. Chlorite was estimated to have similar mean defect-free distance of 7 layers and a size range of 1 to 32 (14Å) units. Kaolinite has a mean defect-free distance of 11 layers but a size range of 1 to 58 (7Å) layers.

The clay mineralogy of the single sample from the south-western edge of the East Midlands Shelf (LGD1) is also composed of I/S, illite, kaolinite and chlorite. While the illite, kaolinite and chlorite appear to have similar characters to those already described for the Cleveland Basin, the air-dry profile for the East Midlands Shelf sample indicates a subtle shift in the I/S d_{001} to c.12Å. This indicates an increased smectite component in the interlayered species to perhaps 20%.

The Lias samples from the Wessex and Worcester Basins differ from their northern counterparts as they contain discrete smectite and no detectable illite/smectite (Figure 4). Although they are otherwise similarly composed of illite, kaolinite and chlorite, they display a greater range of clay mineral concentrations. However, modelling suggests that a typical <2 μm fraction is composed of 37% illite, 26% smectite, 25% kaolinite and 11% chlorite. Modelling of the 'southern' sample XRD traces is similarly hindered by the almost complete overlap of peaks from different clay mineral species. In addition to peak overlap problems, the broad peak profiles of smectite, produced by its relatively small crystallite size, leaves only the d_{001} (17.0 Å) adequately resolved for modelling. Illite has a marginally greater mean defect-free distance of 9 layers and a size range of 1 to 35 (10Å) units. Kaolinite and chlorite have approximately similar mean defect-free distances and size ranges to those models produced for the 'northern' samples. In comparison, smectite has a much smaller mean defect-free distance of 1.5 layers and a size range of only 1 to 5 (14.5Å) units. Despite the problems involved in modelling the traces, 'realistic' modelled profiles were generated (Figure 5).

6 Discussion

Mineralogical analysis of Lias Group samples from various sites representing a relatively large geographic and stratigraphic range have generally similar mineralogies to those described in previous studies (e.g. Kemp & Hards, 1999; Mitchell, 1992; Bloodworth *et al.*, 1987; Pye & Krinsley, 1986; Cosgrove & Salter, 1966). Non-clay mineral assemblages are typically composed of carbonates (calcite and dolomite), quartz, feldspar (albite and occasional K-feldspar), 'mica', pyrite, gypsum and jarosite. Clay mineral assemblages are generally formed of illite, smectite or illite/smectite, kaolinite and chlorite.

The relatively complex mineralogies of the Lias Group samples are difficult to quantify, even by employing state-of-the-art software modelling packages. For this reason the quoted mineral concentrations must be regarded with some caution. However, calculations using approximate values for %clay (from whole-rock XRD), the clay mineral concentrations from <2 μm XRD analysis and assuming theoretical surface area values for the individual clay minerals, reveal similar whole-rock surface area values to those determined empirically.

theoretical

$$theo.surface\ area = \left[\frac{\%\,clay}{100}\right] * \left[\left[\frac{\%\,smectite}{100} * 800\right] + \left[\frac{\%\,illite}{100} * 100\right] + \left[\frac{\%\,chlorite}{100} * 50\right] + \left[\frac{\%\,kaolinite}{100} * 15\right]\right] m^2/g$$

$$\text{e.g. sample LGD12, empirical surface area, } 116\ \text{m}^2/\text{g}$$

$$theo.surface\ area = \left[\frac{54}{100}\right] * \left[\left[\frac{26}{100} * 800\right] + \left[\frac{19}{100} * 100\right] + \left[\frac{17}{100} * 50\right] + \left[\frac{38}{100} * 15\right]\right] m^2/g$$

$$= 129\ m^2/g$$

$$where\ \%\,clay = \%'\,mica\,' + \%\,smectite + \%\,chlorite + \%\,kaolinite\quad from\ whole-rock\ XRD\ analysis$$

Nevertheless, this study has shown that Lias Group rocks of southern England (Wessex and Worcester Basins) and northern England (Cleveland Basin) have importantly different mineralogical characteristics. The mineralogy of the centrally-located, single sample from the East Midlands Shelf has an intermediate character, sharing similar characteristics to both northern and southern samples.

Southern rocks often contain large quantities of carbonate (principally as calcite with minor dolomite) while those from northern England contain little or no carbonate. [The only sample from northern England to contain appreciable calcite is the silt/sandstone from the Staithes Sandstone Formation.] The more calcareous nature of the southern rocks and sandier nature of those in the north has been noted previously but not explained by Anderton *et al.* (1979). Lithologically, both the analysed sample batches are dominated by mudstones with relatively few siltstones/sandstones and both were observed to contain fossil fragments. It would therefore appear that carbonate development is not lithologically controlled. Petrographic analysis is necessary to elucidate whether this apparent difference in mineralogy is due to different sediment source(s) or a different diagenetic overprint between the northern and southern rocks.

This study has also highlighted an important difference in the swelling clay species present in the Lias Group rocks. The samples from the Wessex and Worcester Basins contain discrete smectite whereas I/S (90% illite) is present in the East Midlands Shelf and Cleveland Basin samples. Newmod-for-Windows modelling suggests that all the clay minerals present in the Lias Group have small mean defect-free distances, typically <10 units thick. Such small crystallite sizes indicate that all species will provide an input to the surface area of the rock. However, the difference in swelling clay species does help to explain the larger surface area values for the southern batch (mean 110 m^2/g) compared with the northern batch (mean 85 m^2/g) despite the presence of more coarse-grained siltstone samples in the south. The smaller surface area of I/S compared to smectite might therefore be expected to produce a greater degree of swell-shrink behaviour in the southern strata. However, the high concentration of calcite in the southern samples 'dilutes' the effect of the smectite surface area. In trying to relate the mineralogy and the engineering properties of the Lias Group, it is therefore imperative not only to determine the quantity and type of clay minerals present but also the quantity of calcite present. It is also necessary to know whether the calcite is present as a cement which will influence engineering behaviour, and/or as shell fragments which would have a smaller effect. The only previous petrographic study of the Lias Group mudstones (Pye & Krinsley, 1986) suggests that calcite and siderite are present as randomly dispersed rhombs, irregularly shape grains and patches of intergranular cement in the more silty sediments. No primary biogenic carbonate was observed as forams, coccoliths or shell debris. Interestingly these authors also note that although the mudstones have a well-developed lamination and high degree of parallelism shown by micas and clay minerals, they are not notably fissile. During weathering they split into flaggy slabs rather than sheets. Such behaviour was attributed to the high proportion of authigenic minerals (carbonate, pyrite and kaolinite) which act as cements and bind adjacent laminae together.

The difference between the type of swelling clay present in the northern and southern samples also suggests differences in their burial histories. During burial of sedimentary sequences, the clay minerals contained in mudstones and shales undergo diagenetic reactions in response to increasing depth and temperature. Quantitatively, the most important change is the progressive reaction of smectite to form illite via a series of intermediate illite/smectite (I/S) mixed-layer minerals. In general, progressive changes are irreversible so that where basinal sequences have been inverted clay mineral evidence of the maximum burial depth is retained and can be used to estimate the amount of uplift. According to the Basin Maturity Chart of Merriman & Kemp (1996), the presence of I/S (90% illite) in the rocks from the Cleveland Basin suggests burial depths of perhaps 4 km, assuming a 'normal' geothermal gradient of 25-30°C/km. The I/S (80% illite) in the sample from the East Midlands Shelf therefore suggests shallower burial to perhaps 3 km while the presence of smectite and kaolinite suggest that the mudstones from southern

England are more immature and have only been buried to depths of less than c.3 km if the same geothermal gradient is assumed.

Vitrinite reflectance data for Middle Jurassic coals from the Cleveland Basin show reflectivities of c.0.85% and a rank equivalent to high volatile bituminous coals (Hemingway & Riddler, 1982). Barnard & Cooper (1983) used a combination of vitrinite reflectance and spore colouration indices to conclude that the Middle Jurassic had reached a maximum palaeotemperature of 95°C in the central part of the Cleveland Basin. Furthermore, c.80°C palaeotemperatures for the Middle Jurassic were obtained from fluid inclusion microthermometry from sphalerite grains (Hemingway & Riddler, 1982). Together these palaeotemperatures were taken to indicate a palaeo-depth of c.2.5 km for the base of the Middle Jurassic (Hemingway & Riddler, 1982). The 440 m thickness of the Lias Group in the Cleveland Basin thus produces a maximum depth of burial of c.3km. More recent and detailed modelling (Holliday, 1999) indicate that if the time of maximum burial was end-Cretaceous, between c.2200 and 3000 m of Late Jurassic to Late Cretaceous strata have been removed from the main basin depocentre, assuming a lack of overpressuring. If the time of maximum burial was during the mid-Cenozoic, the observed palaeotemperatures indicates that between 2300 and 3200 m of Late Jurassic, Cretaceous and Palaeogene strata have been removed. Again, if the thickness of the Lias Group is added, a maximum depth of burial of c.4 km is indicated and in agreement with the clay mineralogical data from this study. A combination of clay mineralogy, sonic log studies and palaeotemperature assessments suggest that the observed high palaeotemperatures for the Cleveland Basin can be accounted for by deep burial and there is no need to infer a local heating event.

Subsidence history plots and hydrocarbon potential studies reveal much shallower depth of burial for the Lias Group in the Worcester and Wessex Basins. Chadwick & Evans (1995) used mudstone densities to suggest that 1650 m of overburden had been removed from the Mercia Mudstone Group in the Kempsey borehole, south of Worcester while perhaps 1200 m had been removed from the eastern part of the basin. It would therefore appear that the Lias Group has only been buried to perhaps 1.5 km in the Worcester Basin. Calculated organic maturity values of <0.50% R_0 (Ebukanson & Kinghorn, 1986) and organic geochemical analyses (Colter & Havard, 1981) for the Base Lias of the Wytch Farm oilfield, Dorset suggest organic immaturity. Although maturities are heavily influenced by the Purbeck-Isle of Wight Disturbance, burial-thermal history projections based on such data suggest a maximum burial of c.2 km and peak palaeotemperatures of c.75°C for the locations sampled in the Wessex Basin for this study.

Mineralogical analysis also suggests that pyrite is very commonly developed throughout the Lias Group, typically forming 2% rock. Pye & Krinsley (1986) observed authigenic pyrite occurring as framboids and larger euhedral crystals, particularly in the Mulgrave Shale Member of the Whitby Mudstone Formation. The other sulphur-bearing minerals, gypsum and jarosite are more sporadically developed but can form up to 12% rock. Jarosite and gypsum typically form as weathering products of pyrite. Although it is difficult to comment with such a small sample batch, stratigraphically it would appear that the Whitby Mudstone Formation together with the Blue Lias and Charmouth Mudstone formations show the greatest occurrence of sulphur-bearing species. Concrete engineering sited in rocks from these formations therefore potentially run the greatest risk of acid attack and thaumasite formation.

7 Conclusions

This mineralogical study of a suite of Lias Group sedimentary rocks has generally confirmed the findings of previous workers. However, the wide geographic and stratigraphic distribution of the analysed samples has provided important new information which will aid not only interpretation of the engineering behaviour of these rocks but also their diagenetic and geological histories.

The engineering properties of the UK Lias will be heavily influenced by its clay mineralogy and in particular whether discrete smectite or illite/smectite is present. This study has shown that rocks from the West Midlands and southern England (the Worcester and Wessex Basins) are likely to contain smectite and therefore have greater shrink-swell behaviour than those rocks from the East Midlands (East Midlands Shelf) and northern England (the Cleveland Basin). However, the degree of shrink-swell is moderated by the high carbonate content typically found in southern rocks compared to those in the north. As indicated by XRD modelling, the small crystallite size of the other clay minerals; illite, kaolinite and chlorite will also significantly contribute to any volume change and swell/shrink behaviour.

The type of swelling clay species present is also useful for determining the depth of burial for the Lias Group across England. The I/S (90% illite) present in the Cleveland Basin suggests a 4 km maximum depth of burial which corroborates earlier vitrinite reflectance-, fluid inclusion- and sonic velocity-based estimates. The greater proportion of smectite present in the I/S (80% illite) detected in the single sample from the East Midlands Shelf, suggests shallower burial to perhaps 3 km. However, the discrete smectite present in the Worcester and Wessex Basins indicates even shallower burial to no more than 2 km. Studies of basin maturity can therefore be used to predict likely engineering properties for the Lias Group rocks.

In order to further understand and predict the engineering properties of the mudstones, it is recommended that the distribution of clay minerals and their fabrics are studied in thin section and as rough stubs using a scanning electron microscope. Further work is also necessary to measure and relate the thickness of clay crystallites to engineering properties.

The very common presence of pyrite, together with gypsum and jarosite in the Lias Group means that concrete engineering sited in theses rocks potentially risk acid attack and thaumasite formation. The Whitby Mudstone Formation in the Cleveland Basin together with the Blue Lias and Charmouth Mudstone formations in the Worcester and Wessex Basins show the greatest occurrence of sulphur-bearing species.

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Table 1. Summary of samples from the East Midlands Shelf, Worcester Basin and Wessex Basin

Sample No.	Lab. code	Location	NGR	Basin	Stratigraphy		Detailed location	Description
					Formation	Member (zone)		
LGD1	G053	Harbury, Warks. (quarry)	SP 3862 5880	East Mids. Shelf	Blue Lias	Rugby Limestone	upper waterfall near entrance	Dark grey mudstone with shell frags.
LGD2	G054	Northcot, Blockley, Gloucs.(quarry)	SP 1795 3699	Worcester	Charmouth Mdst	N/A	Ibex zone	Dark grey mudstone with shell frags.
LGD3	G055	Northcot, Blockley, Gloucs.(quarry)	SP 1803 3404	Worcester	Charmouth Mdst	N/A	Ibex zone	Dark grey mudstone with shell frags.
LGD4	G056	Ware Cliff, Lyme Regis, Dorset (coast)	SY 3315 9138	Wessex	Blue Lias	(Angulata zone)	below Specketty Lst band	Dark grey, laminated mudstone
LGD5	G057	Ware Cliff, Lyme Regis, Dorset (coast)	SY 3337 9154	Wessex	Charmouth Mdst	Shales-with-Beef	upper semicostatum	Dark/pale grey, laminated mudstone
LGD5'beef'	G058	Ware Cliff, Lyme Regis, Dorset (coast)	SY 3337 9154	Wessex	Charmouth Mdst	Shales-with-Beef	upper semicostatum	Fibrous calcite
LGD6	G059	Stonebarrow Hill, Dorset (coast)	SY 3816 9264	Wessex	Charmouth Mdst	Belemnite Marl	2m above base	Medium grey siltstone
LGD7	G060	Cain's Folly, Stonebarrow Hill, Dorset (coast)	SY 3739 9288	Wessex	Charmouth Mdst	Black Ven Marl	below lowermost Pavior	Dark grey mudstone with shell frags.
LGD8	G061	Cain's Folly, Stonebarrow Hill, Dorset (coast)	SY 3739 9288	Wessex	Charmouth Mdst	Black Ven Marl	1.5m above Pavior Limestone	Dark grey, laminated mudstone
LGD9	G062	Seatown, Dorset (coast)	SY 4221 9162	Wessex	Dyrham	Eype Clay	4m below Eype nodule bed	Medium-dark grey mudstone
LGD10	G063	Watton Cliff, Dorset (coast)	SY 4529 9094	Wessex	Bridport Sand	Down Cliff Clay	against Eypemouth fault	Green siltstone
LGD11A	G064	Robins Wood Hill, Gloucester (quarry)	SO 835_ 149_	Worcester	Whitby Mdst	N/A	5m above Marlstone Rock Fm	Green mudstone with black 'root-like' material
LGD11B	G065	Robins Wood Hill, Gloucester (quarry)	SO 835_ 149_	Worcester	Whitby Mdst	N/A	(3m depth)(weathered)	Pale green siltstone
LGD12	G066	Robins Wood Hill, Gloucester (quarry)	SO 835_ 149_	Worcester	Dyrham	N/A	15m below Marlstone	Dark grey, laminated mudstone with shell frags.

Table 2. Summary of samples from the Cleveland Basin

Sample No.	Lab. code	Location	NGR	Stratigraphy Formation	Member (zone)	Detailed location	Description
LGD13	G187	Ravenscar (golf course)	NZ 9799 0173	Whitby Mdst	Alum Shale	S. cliffs, Robin Hood's Bay	Dark grey, laminated mudstone
LGD14	G188	Ravenscar (golf course)	NZ 9829 0211	Whitby Mdst	Mulgrave Shale	S. cliffs, Robin Hood's Bay	Dark grey, laminated mudstone, fossil frags.
LGD15	G189	Ravenscar	NZ 9778 0223	Redcar Mdst	Pyritous Shale (lower)	S. cliffs, Robin Hood's Bay	Medium to dark grey, laminated mudstone
LGD16	G190	Staithes	NZ 7880 1886	Cleveland Irst	3m below Avicula seam	S. harbour cliffs	Dark grey, laminated mudstone
LGD17	G191	Staithes	NZ 78565 18879	Staithes Sst		S. harbour cliffs	Pale/medium grey, massive slst/sst, fossil frags
LGD18	G192	Runswick Bay (cliff)		Whitby Mdst	Mulgrave Shale	'jet' workings 2m above beach	Dark grey, laminated mudstone, oxidised pyrite
LGD19	G193	Kettleness (cliff)	NZ 8318 1603	Whitby Mdst	Grey Shales	1m above base of W.M.F.	Dark grey, laminated mudstone
LGD20	G194	Kettleness (cliff)	NZ 8317 1599	Whitby Mdst	Grey Shales	4m above base of W.M.F.	Dark grey, laminated mudstone
LGD21	G195	Kettleness (former alum quarry)	NZ 8346 1586	Whitby Mdst	Alum Shale		Dark grey, laminated mudstone, oxidised pyrite
LGD22	G196	Kettleness (former alum quarry)	NZ 8321 1603	Whitby Mdst	Mulgrave Shale	5m below top of W.M.F.	Dark grey, laminated mudstone
LGD23	G197	Robin Hood's Bay (harbour)		Redcar Mdst	Ironstone Shale	S. of current sea-wall works	Dark grey, laminated mudstone
LGD24	G198	Boggle Hole	NZ 9644 0313	Redcar Mdst	Calcareous Shales (upper)	cliff near Stoupe Beck	Medium grey, laminated mudstone
LGD25	G199	Boggle Hole	NZ 9631 0307	Redcar Mdst	Calcareous Shales (lower)	wave-cut plat., Stoupe Beck	Dark grey, laminated mudstone

Table 3. Summary of whole-rock XRD and surface area analyses

Sample	Location	%mineral													S.A.
		quartz	calcite	pyrite	dolomite	'mica'	K-feldspar	kaolin	chlorite	smectite	albite	gypsum	jarosite	illite/smectite	m^2/g
LGD1	Harbury, Warks. (quarry)	22	47	2	1	20	nd	4	nd	nd	1	nd	nd	3	114
LGD2	Northcot, Blockley, Gloucs.(quarry)	52	1	1	nd	22	nd	20	nd	1	3	nd	nd	nd	86
LGD3	Northcot, Blockley, Gloucs.(quarry)	38	1	1	nd	39	nd	21	nd	nd	nd	nd	nd	nd	97
LGD4	Ware Cliff, Lyme Regis, Dorset (coast)	13	51	4	5	22	nd	2	nd	nd	nd	3	nd	nd	137
LGD5	Ware Cliff, Lyme Regis, Dorset (coast)	22	20	5	1	31	1	9	nd	2	nd	8	1	nd	203
LGD5'beef'	Ware Cliff, Lyme Regis, Dorset (coast)	2	86	nd	nd	6	nd	1	nd	1	nd	1	3	nd	23
LGD6	Stonebarrow Hill, Dorset (coast)	10	56	2	12	17	nd	2	nd	nd	1	nd	nd	nd	77
LGD7	Cain's Folly, Stonebarrow Hill, Dorset (coast)	22	27	4	4	33	nd	10	nd	nd	nd	nd	nd	nd	123
LGD8	Cain's Folly, Stonebarrow Hill, Dorset (coast)	17	23	6	14	33	nd	6	nd	nd	nd	1	nd	nd	179
LGD9	Seatown, Dorset (coast)	44	3	1	nd	28	nd	15	nd	2	7	nd	nd	nd	98
LGD10	Watton Cliff, Dorset (coast)	51	21	nd	nd	17	1	5	nd	2	3	nd	nd	nd	106
LGD11A	Robins Wood Hill, Gloucester (quarry)	12	69	nd	nd	15	nd	4	nd	nd	nd	nd	nd	nd	77
LGD11B	Robins Wood Hill, Gloucester (quarry)	6	81	nd	nd	8	nd	2	nd	nd	nd	1	2	nd	27
LGD12	Robins Wood Hill, Gloucester (quarry)	40	nd	1	nd	29	nd	22	nd	3	3	2	nd	nd	116
LGD13	Ravenscar (golf course)	30	nd	5	nd	42	nd	21	nd	nd	nd	nd	nd	2	96
LGD14	Ravenscar (golf course)	28	3	6	nd	38	nd	21	nd	nd	nd	nd	nd	4	85
LGD15	Ravenscar	29	8	3	nd	37	nd	21	nd	nd	nd	nd	nd	2	86
LGD16	Staithes	36	nd	2	nd	36	nd	17	nd	nd	7	nd	nd	2	66
LGD17	Staithes	40	24	1	3	13	nd	10	nd	nd	9	nd	nd	nd	24
LGD18	Runswick Bay (cliff)	25	nd	3	nd	28	nd	10	nd	nd	1	10	12	11	78
LGD19	Kettleness (cliff)	39	nd	3	nd	31	nd	21	nd	nd	4	nd	nd	2	66
LGD20	Kettleness (cliff)	31	nd	2	nd	40	nd	18	nd	nd	nd	nd	nd	9	96
LGD21	Kettleness (former alum quarry)	32	nd	nd	nd	38	nd	20	nd	nd	nd	nd	nd	10	134
LGD22	Kettleness (former alum quarry)	24	nd	1	nd	36	nd	17	nd	nd	nd	8	4	10	117
LGD23	Robin Hood's Bay (harbour)	24	6	2	nd	42	nd	21	1	nd	nd	1	nd	3	100
LGD24	Boggle Hole	38	1	1	3	37	nd	17	nd	nd	1	nd	nd	2	79
LGD25	Boggle Hole	47	nd	nd	1	32	nd	15	nd	nd	1	nd	nd	4	76

Table 4. Summary of <2 µm clay mineral XRD analyses

Sample No.	Location	%clay mineral				
		smectite	illite/smectite	illite	chlorite	kaolinite
LGD1	Harbury, Warks. (quarry)	nd	22	50	10	18
LGD2	Northcot, Blockley, Gloucs.(quarry)	15	nd	23	18	43
LGD3	Northcot, Blockley, Gloucs.(quarry)	11	nd	27	17	45
LGD4	Ware Cliff, Lyme Regis, Dorset (coast)	16	nd	60	8	16
LGD5	Ware Cliff, Lyme Regis, Dorset (coast)	36	nd	29	10	24
LGD5'beef'	Ware Cliff, Lyme Regis, Dorset (coast)	na	na	na	na	na
LGD6	Stonebarrow Hill, Dorset (coast)	18	nd	66	5	11
LGD7	Cain's Folly, Stonebarrow Hill, Dorset (coast)	18	nd	38	17	27
LGD8	Cain's Folly, Stonebarrow Hill, Dorset (coast)	30	nd	32	11	27
LGD9	Seatown, Dorset (coast)	37	nd	17	17	29
LGD10	Watton Cliff, Dorset (coast)	71	nd	12	2	15
LGD11A	Robins Wood Hill, Gloucester (quarry)	30	nd	48	5	16
LGD11B	Robins Wood Hill, Gloucester (quarry)	21	nd	56	5	17
LGD12	Robins Wood Hill, Gloucester (quarry)	26	nd	19	17	38
LGD13	Ravenscar (golf course)	nd	52	23	6	19
LGD14	Ravenscar (golf course)	nd	45	29	5	21
LGD15	Ravenscar	nd	55	21	5	18
LGD16	Staithes	nd	42	29	10	19
LGD17	Staithes	nd	42	25	13	21
LGD18	Runswick Bay (cliff)	nd	36	40	4	20
LGD19	Kettleness (cliff)	nd	49	28	5	18
LGD20	Kettleness (cliff)	nd	56	26	4	14
LGD21	Kettleness (former alum quarry)	nd	48	27	4	20
LGD22	Kettleness (former alum quarry)	nd	54	21	4	21
LGD23	Robin Hood's Bay (harbour)	nd	46	25	6	24
LGD24	Boggle Hole	nd	45	31	7	17
LGD25	Boggle Hole	nd	51	31	5	13

Figure 2. Generalised outcrop map of the Lias Group in England and Wales showing the structural elements that controlled deposition (Cox *et al.*, 1999)

CLEVELAND BASIN (* to be formalised)

Formations	Members
Blea Wyke Sandstone	Yellow Sandstone Grey Sandstone
Whitby Mudstone	Fox Cliff Siltstone Peak Mudstone Alum Shale Mulgrave Shale Grey Shale
Cleveland Ironstone	Kettleness Penny Nab
Staithes Sandstone	
Redcar Mudstone	Ironstone Shale* Pyritous Shale* Siliceous Shale* Calcareous Shale*

EAST MIDLANDS SHELF: A. South (Moreton in Marsh to Leicester area), B. North (Leicester area to Market Weighton).

A. South		B. North	
Formations	**Members**	**Formations**	**Members**
Whitby Mudstone	pending	Whitby Mudstone	pending
Marlstone Rock	none	Marlstone Rock	none
Dyrham	pending	Charmouth Mudstone	Pecten Ironstone
Charmouth Mudstone	pending		
Blue Lias	Rugby Saltford Shale	Scunthorpe Mudstone	Frodingham Ironstone Foston Beckingham Granby Barnby Barnstone

WESSEX BASIN (including Central Somerset Basin) and South Wales

Formations	Members (Dorset coast)	Members (South Wales)
Bridport Sand	Down Cliff Clay	
Beacon Limestone	Eype Mouth Limestone Marlstone Rock	
Dyrham	Thorncombe Sand Down Cliff Sand Eype Clay	
Charmouth Mudstone	Green Ammonite Belemnite Marl Black Ven Marl Shales-with-Beef	
Blue Lias		Porthkerry Lavernock Shale St Mary's Well Bay

WORCESTER BASIN

Formations	Members
Bridport Sand	pending
Whitby Mudstone	pending
Marlstone Rock	none
Dyrham	pending
Charmouth Mudstone	pending
Blue Lias	Rugby Saltford Shale Wilmcote

Figure 3. Lithostratigraphic subdivision of the Lias Group (Cox *et al.*, 1999)

Figure 4. The whole-rock mineralogy and surface area of the Lias Group samples, ordered stratigraphically.

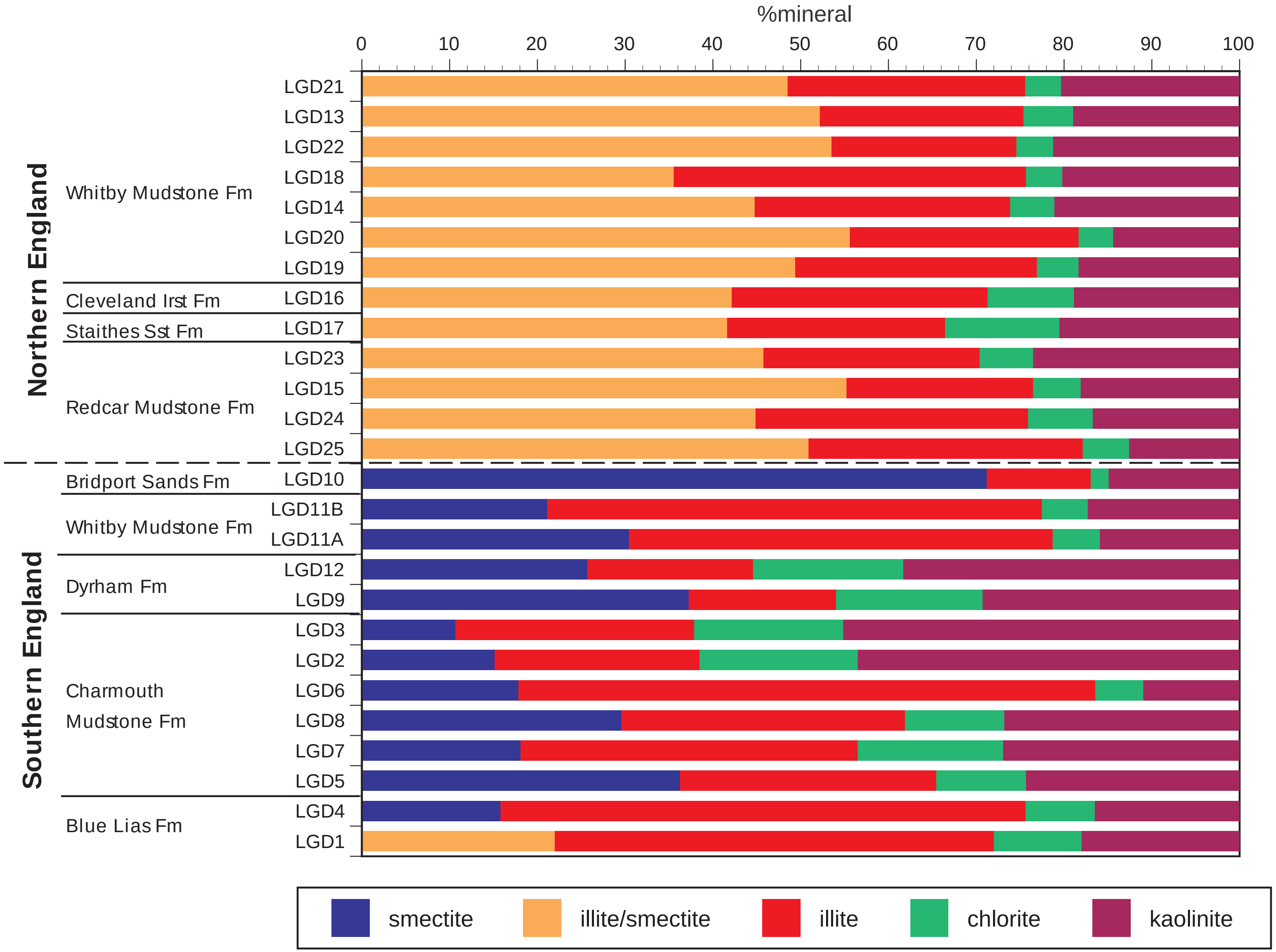


Figure 5. The clay mineralogy of the Lias Group samples, ordered stratigraphically.

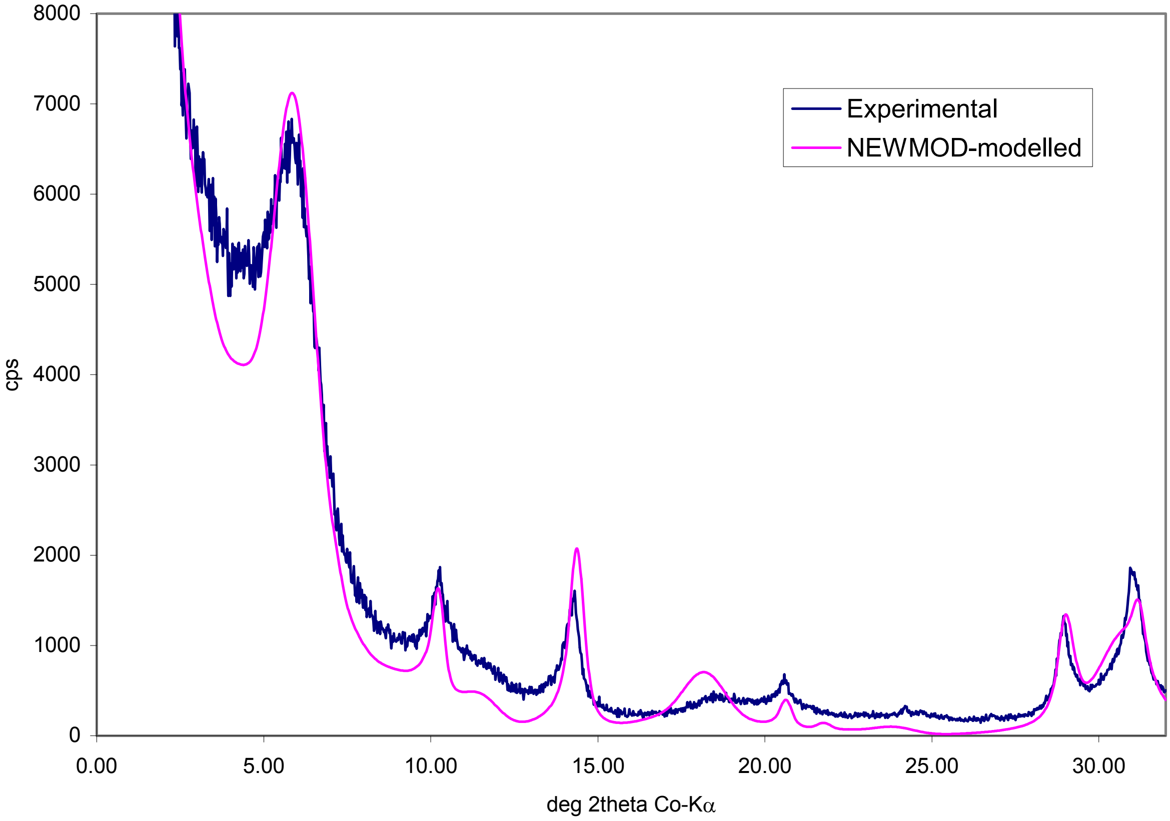


Figure 6. Comparison of experimental and NEWMOD-modelled XRD profiles. Ethylene glycol-solvated <2 µm oriented mount, sample LGD10, Watton Cliff, Dorset.

Appendix: X-RAY DIFFRACTION TRACES:

KEY

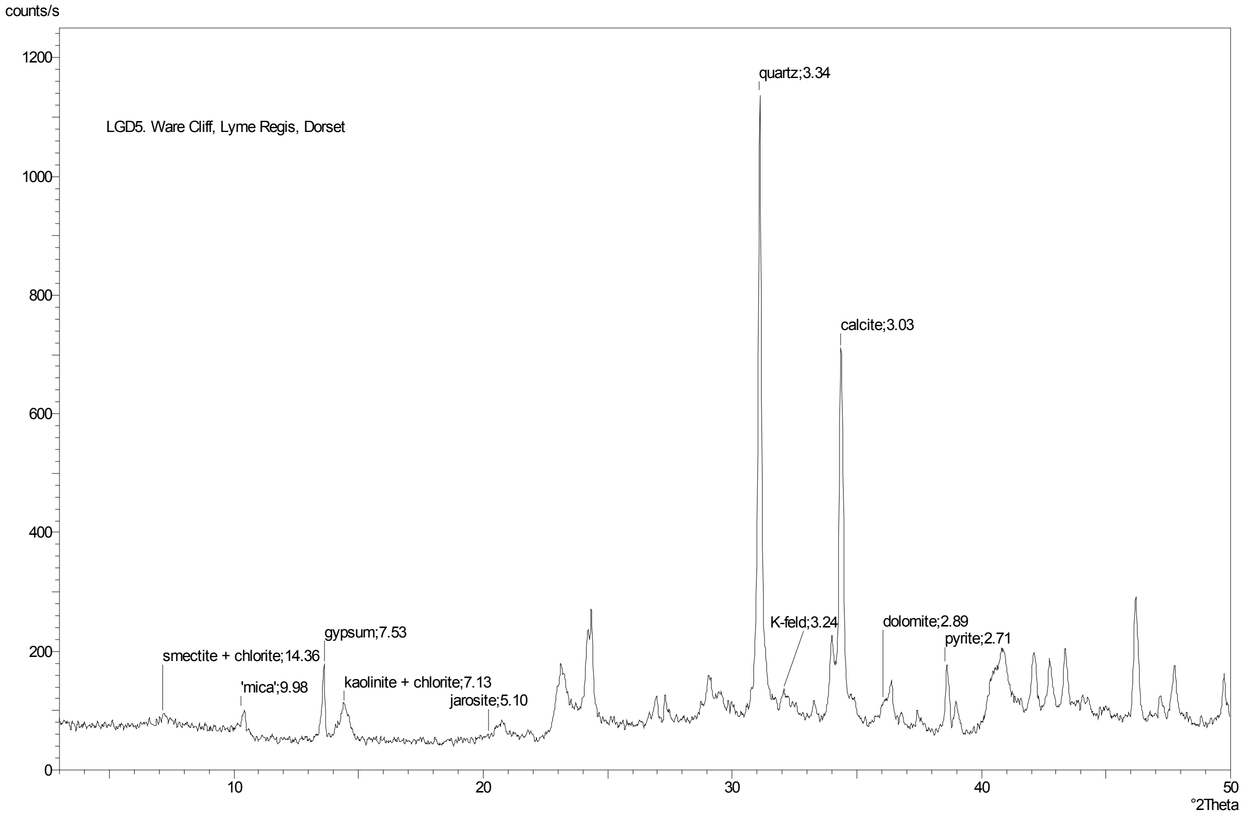
Vertical axis – Intensity (counts per second)

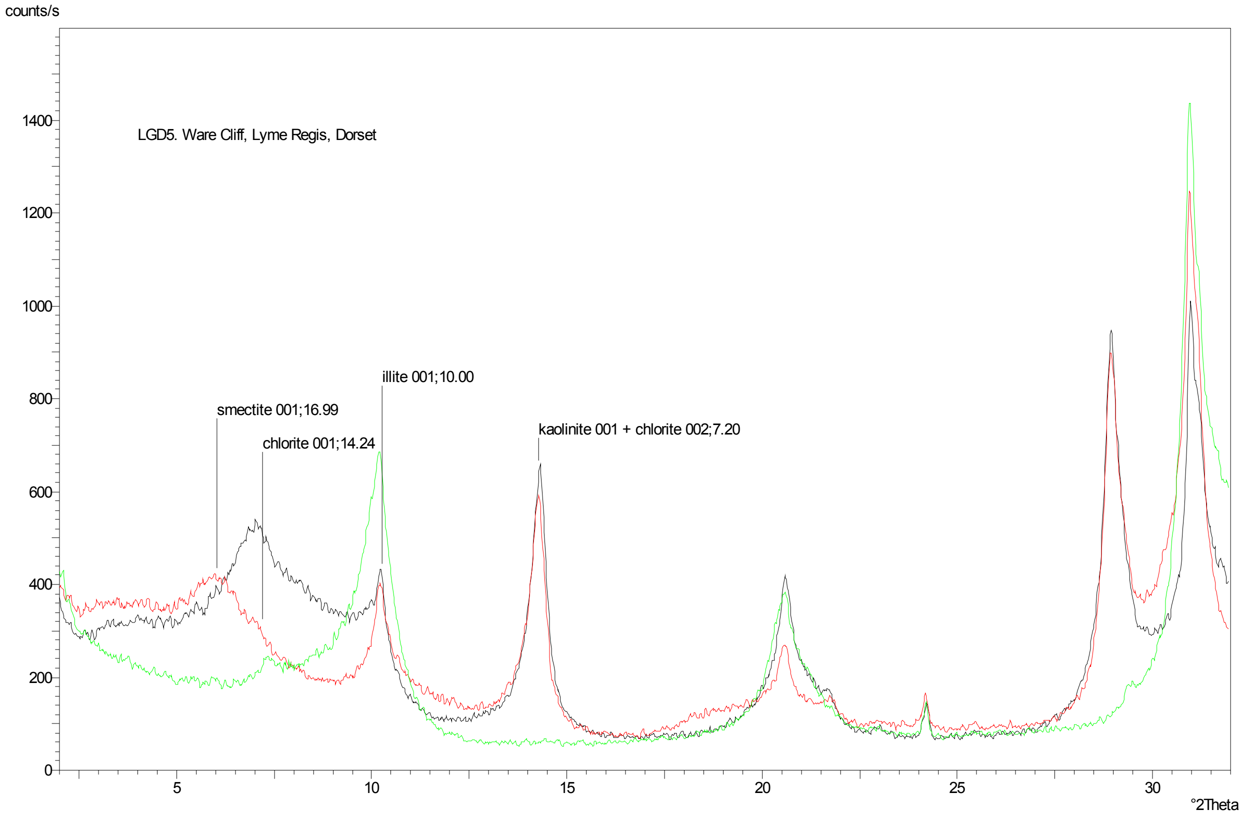
Horizontal axis - °2θ Co-Kα

For the whole-rock traces, only the most intense/diagnostic peak of each identified mineral is labelled.

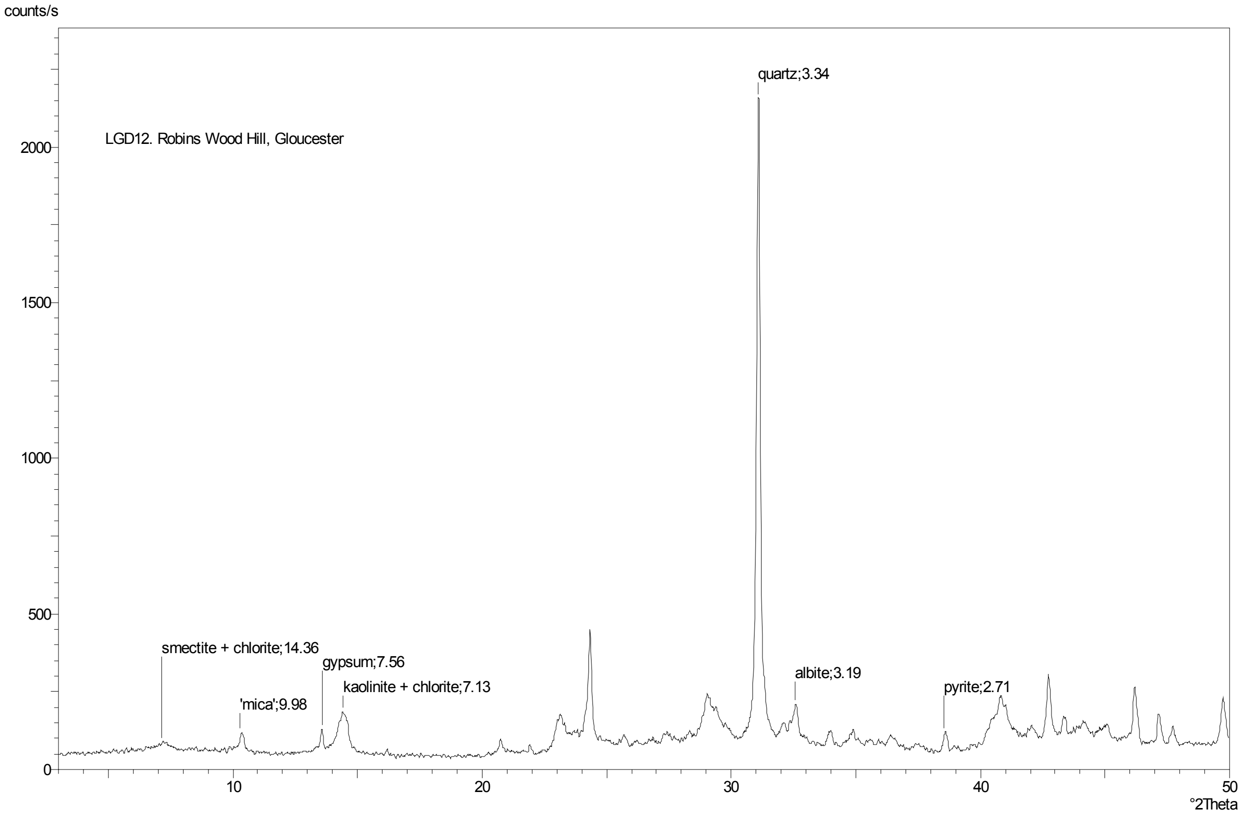
For the <2 µm traces. Black trace (air-dry), red trace (glycol-solvated) and green trace (heated 550°C/2 hours). Only the d_{001} of each identified clay mineral is labelled on the glycol-solvated trace.

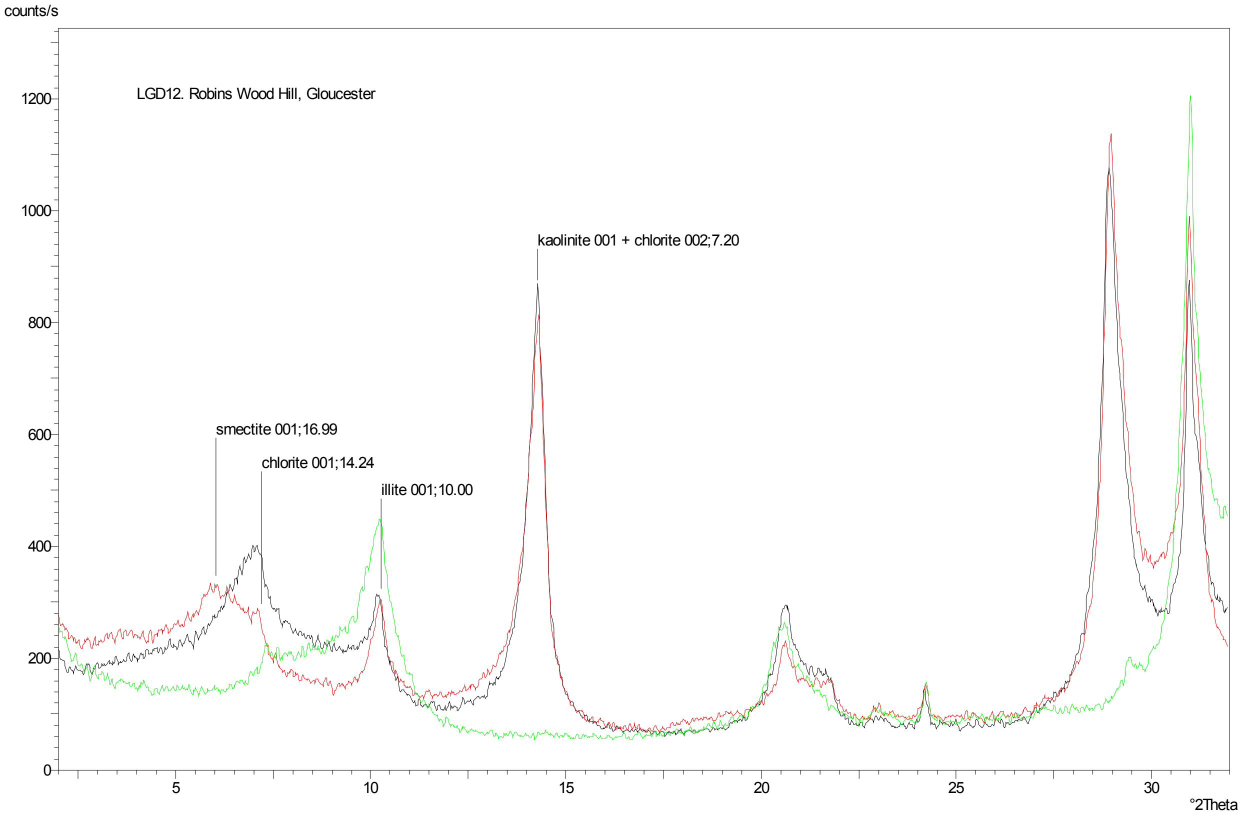
WESSEX BASIN



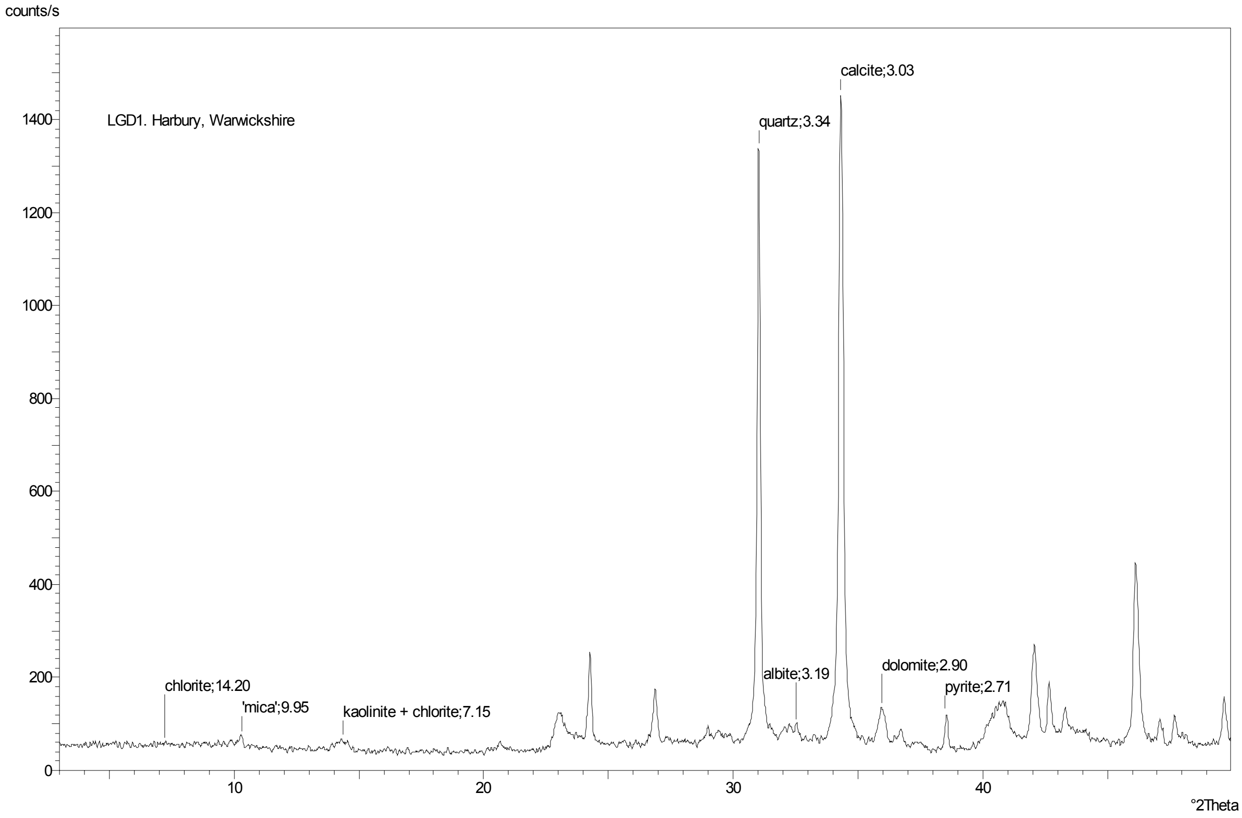


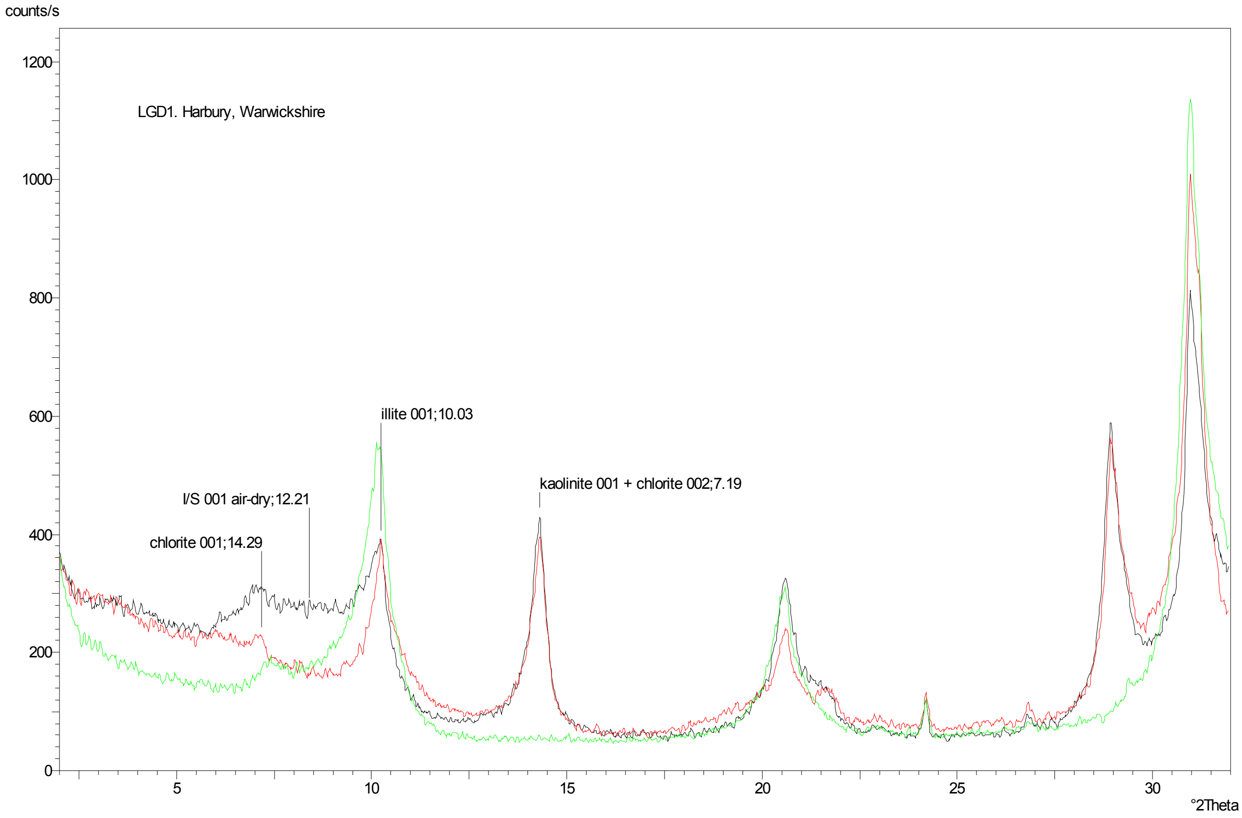
WORCESTER BASIN





EAST MIDLANDS SHELF





CLEVELAND BASIN