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A DRIFT Spectroscopy Study of Clays. Ground Movements: Shrink/Swell Project.

Urban Geoscience and Geological Hazards Programme
Internal Report IR/02/033

BRITISH GEOLOGICAL SURVEY

INTERNAL REPORT IR/02/033

A DRIFT Spectroscopy Study of Clays. Ground Movements: Shrink/Swell Project.

C. H. Vane & L. D. Jones

Key words

DRIFT, diffuse, reflectance, infrared, spectroscopy, shrinkage, swelling, clays.

Front cover

Shrinkage cracks in Gault clay, Arlesey, Bucks.

Bibliographical reference

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Foreword

This report is the published product of a study by the British Geological Survey (BGS). It refers to the work carried out on behalf of a Science Budget funded research project, under the Urban Geoscience & Geological Hazards Programme of the BGS.

Acknowledgements

A large number of individuals in the BGS have contributed to the project. This assistance has been received at all stages of the study. In addition to the collection of data, many individuals have freely given their advice, and provided the local knowledge so important to the project. Key staff have helped to review draft chapters of this report. Of the many individuals who have contributed to the project we would particularly like to thank the following:

Prof. M. G. Culshaw
Mr A. Forster

Contents

Summary

This report describes a study of a new technique for analysing the mineralogical composition of clay soils. It was carried out as part of a Science Budget programme of research for the Ground Movements: Shrink/Swell Project (ESB8390006703) which is itself part of the Urban Geoscience and Geological Hazards (UGGH) Programme.

This study uses a diffuse reflectance infrared spectroscopy (DRIFT) technique that can be an alternative to x-ray diffraction (XRD) values. Infrared spectroscopy was carried out on samples obtained from in-situ sampling techniques from quarries, pits and cliff-faces in the UK. The samples formed a representative range of clay soils from the Gault, Mercia Mudstone and Lambeth Group Formations.

A critical evaluation of the characteristic absorption features of the clay minerals most likely to be encountered, when testing the samples, was undertaken. Following this analysis, seventeen clay soils were tested using the DRIFT technique. Of these initial samples, nine were analysed in greater detail to ascertain their likely mineralogical composition.

The kaolinite and/or smectite content of the Newbury 1 sample was higher than the Newbury 2 and 3 samples, in contrast absorption bands indicative of kaolinite were not observed in the clay sample from Hemmington. In the soils from Arlesey the spectra were dominated by bands at 1429 and 2512 cm^{-1}, which originate from the carbonate mineral calcite. No absorption bands from organic matter were observed in any of the shrink swell soils, the most likely explanation being that the strong mineral bands overlap and mask peaks from organic matter.

1. Introduction

This study forms a part of the Ground Movements: Shrink/Swell Project (ESB8390006703) which is itself part of the Urban Geoscience and Geological Hazards (UGGH) Programme. The main aim of the project is to determine the shrinkage and swelling properties of UK clays and mudrocks and to investigate the relationships between them. One of the project's main objectives is to develop new techniques for determining the shrink/swell potential of a soil using spectroscopy, both in the laboratory and in the field. It is with this aim that this study was undertaken.

This report is concerned with the study of a new technique for analysing the mineralogical composition of clay soils. This study uses a diffuse reflectance infrared spectroscopy (DRIFT) technique that can provide an alternative to x-ray diffraction (XRD) values. This study was carried out on samples obtained from in-situ sampling techniques from quarries, pits and cliff-faces in the UK. The samples formed a representative range of clay soils from the Gault, Mercia Mudstone and Lambeth Group formations. All the samples were collected by members of the Shrink/Swell Project team (Table 1).

Diffuse reflectance (IR) fourier transform infrared spectroscopy (DRIFT) is an alternative technique to x-ray diffraction (XRD) for obtaining information on clay minerals. The inorganic and organic composition of soils can, in part, be determined using DRIFT. The principal advantage of the DRIFT technique over the pressed halide disc method is that samples require minimal preparation and the problem associated with absorption or exchange of the K ion into the clay structure is avoided. DRIFT spectra result from radiation from a non-mirror surface after it has undergone absorption, refraction, reflection and scattering. However, DRIFT spectra are subject to non-linear scaling of intensity, which decreases the intensity of strongly absorbing bands relative to weak bands. Such enhancement in the region of 2600-2500cm^{-1} can be useful in the identification of carbonate minerals.

Table 1

No.	Sample Name	Lithology	No.	Sample Name	Lithology
1	Newbury 1	Lambeth Group	11	Knoll Manor Farm	Lambeth Group
2	Newbury 2	Lambeth Group	12	Copyhold Farm	Lambeth Group
3	Newbury 3	Lambeth Group	13	Arlesey 1	Gault Clay
4	Enfield	Lambeth Group	14	Arlesey 2	Gault Clay
5	Hemmington	Mercia Mudstone	15	Leighton Buzzard	Gault Clay
6	Dorket Head 1	Mercia Mudstone	16	Shepshed 1	Mercia Mudstone
7	Dorket Head 2	Mercia Mudstone	17	Shepshed 2	Mercia Mudstone
8	Whitecliffe Bay	Lambeth Group	18	Quartz	Quartz
9	Newhaven	Lambeth Group	19	Kaolinite	Kaolinite
10	Michelmersh	Lambeth Group	20	Bentonite	Bentonite

2. Method

Sample preparation for DRIFT determinations was as follows: a 10 mg aliquot of pre-ground soil was dried for 2 h at 100 °C then mixed with 200 mg of Fourier Transform Infrared Spectroscopy (FTIR) grade potassium bromide (KBr). The sample was homogenised and immediately transferred to the stainless steel sample holder, its surface smoothed with a blade, and DRIFT spectra recorded. Diffuse reflectance infrared spectrometry (DRIFT) was performed using a Bio-Rad FTX3000MX series FTIR (Digilab Div., Bio-Rad Laboratories, Cambridge, MA) from 4000 and 400 cm^{-1}; the accuracy of the measurements was 2 cm^{-1}. Sample spectra were referenced against the KBr matrix, peak areas were obtained with the integration limits going to the constructed baseline.

3. Results & Discussion

A critical evaluation of the characteristic absorption features of the clay minerals most likely to be encountered, when testing the samples, was undertaken. Following this analysis, seventeen clay soils were tested using the DRIFT technique, the resulting spectra are shown throughout the text or in the Appendix. Of these initial samples, nine were analysed in greater detail to ascertain their likely mineralogical composition. The remaining eight samples will be analysed further at a later date, once the validity of the results has been determined.

3.1 Reference Materials

In order to identify minerals in clay soils a critical evaluation of the characteristic absorption features of the minerals most likely to be encountered is necessary. The minerals quartz, kaolinite and bentonite were selected as important reference materials for clay soils and their DRIFT spectra are shown in Figures 1-3.

The major absorption bands of quartz from 1159 cm^{-1} up to and including the peak at 695 cm^{-1} are assigned to stretching modes of vibration (O-Si and Si-O-Si). In contrast the bands equal to and less than 520 cm^{-1} may be attributed to bending deformation vibrations (O-Si) (Van der Marel and Beutelspacher, 1976). The doublet at 780, 799 cm^{-1} has been used for the identification of quartz in clay materials. The absence of absorption bands at ~3455 cm^{-1} and 1645 cm^{-1} (liquid H_2O) suggested that the KBr and quartz did not contain any adsorbed water.

Figure 2 shows the DRIFT spectrum for the clay mineral kaolinite. The four absorption peaks in the region 3600 to 3800 cm^{-1} are assigned to stretching vibrations in hydroxyl (OH) groups. Assignment of the hydroxyl groups has attracted considerable interest due to hydroxyl bands being sensitive probes for the characterisation of kaolin mineral structure (Frost and Johansson, 1998). The three high frequency peaks at 3653, 3668 and 3694 cm^{-1} arise from the outer hydroxyls located in the outer upper, unshared plane. The former two absorption peaks are attributed to coupled antisymetric and symmetric stretching vibrations whereas the assignment of the latter absorption peak is disputed within the literature (Russell, 1987). In contrast the peak at 3620 cm^{-1} is assigned to internal hydroxyls situated in the plane shared with the apical oxygens of the tetrahedral sheet (Russell, 1987). The degree of ordering within the kaolinite structure can be determined by comparison of the OH stretching region; the presence of a doublet at 3653, 3669, is typical of an ordered kaolinite. Other important absorption peaks are observed at 915 and 937 cm^{-1} which, may be attributed to hydroxyl deformation modes of inner surface and inner hydroxyls.

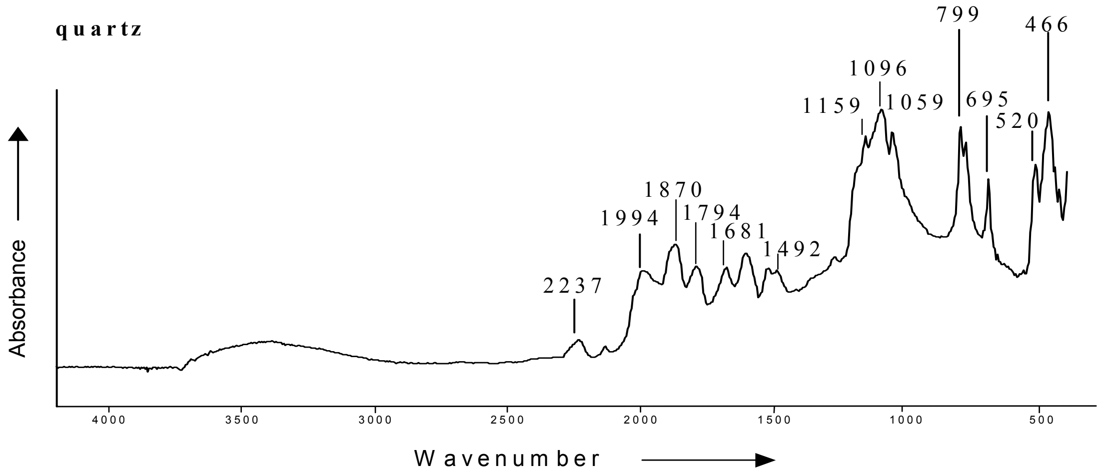


Figure 1. DRIFT Spectrum of Quartz

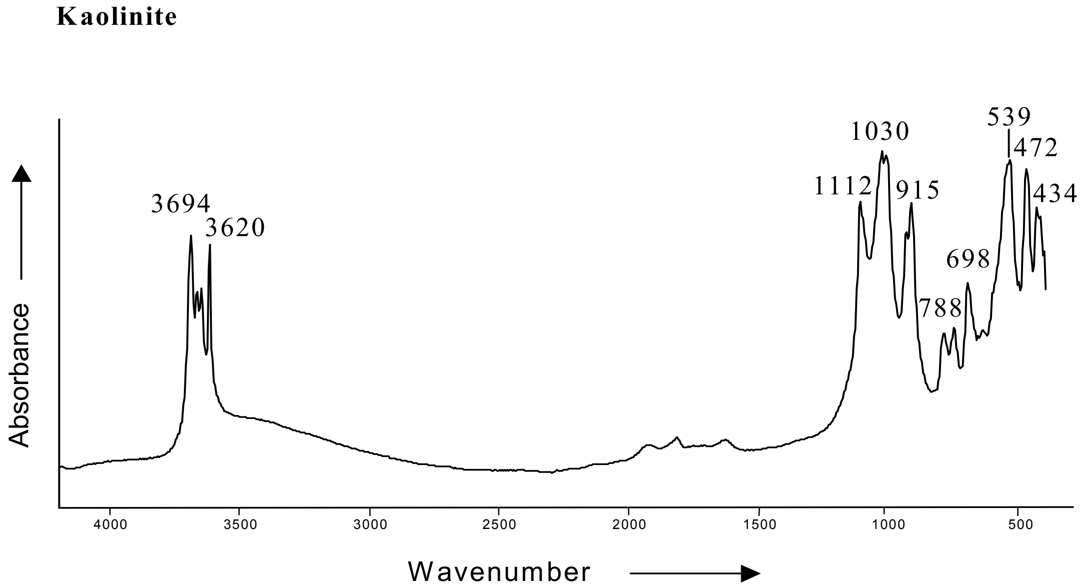


Figure 2. DRIFT Spectrum of Kaolinite

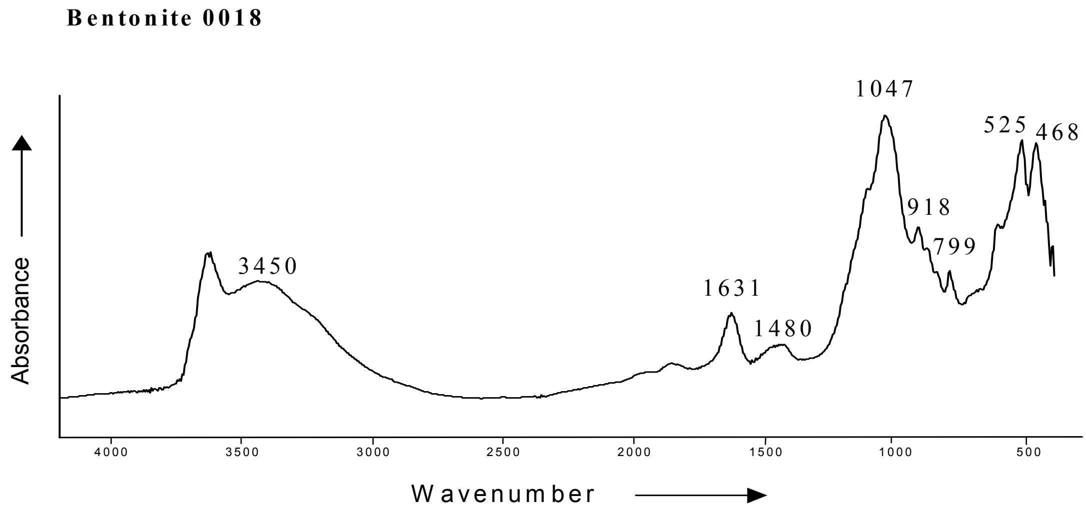


Figure 3. DRIFT Spectrum of Bentonite

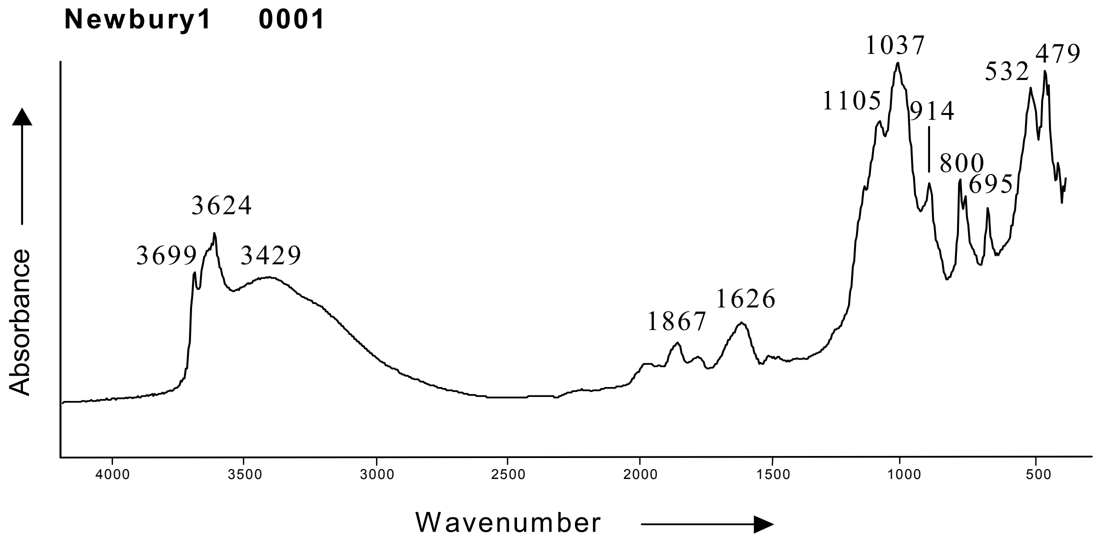
3.2 Newbury clay soils

The DRIFT spectra of the Newbury samples 1-3 are shown in Figures 4 a-c respectively. A comparison of the general spectral features suggested that all three samples have a similar mineral composition. The broad and poorly resolved hydroxyl stretching absorption bands between 3400-3630 cm^{-1} is typical of vibrations from alumino-silicates, namely smectite and illites. The two dominant OH-stretching peaks observed in the kaolinite reference material are apparent in all the Newbury samples at 3620 and 3697 cm^{-1} (Fig 2).

Quartz has a number of distinct absorption bands at 695 and 799 cm^{-1} (Fig. 1) and these can be clearly observed in the Newbury samples spectra. The enhancement of weak overtone /combination absorption peaks in DRIFT spectra as compared to transmission IR spectra is a useful feature for the characterization of mineral mixtures in soils. For quartz, these absorption bands appear in the 1650-2000 cm^{-1} region, although other silicates can also yield weak absorption peaks in this region (Van der Marel and Beutelspacher, 1976). The three relatively strong absorption bands at 1977, 1867 and 1790 cm^{-1} are generally assigned to quartz (Fig. 4). The intensities of these absorption peaks have been shown to be independent of particle size in contrast to other intense absorption bands (Nguyen *et al.*, 1991).

The most intense absorption of the standard quartz occurred at 1096 cm^{-1} (Fig. 1), this overlaps with other absorption and forms part of the dominant peak centred at 1034 to 1037 cm^{-1} in each of the Newbury samples. The main absorption centred at 1030 cm^{-1} is probably derived from mixed alumino-silicate lattice vibrations in kaolinite and smectite. Although the DRIFT spectra of the Newbury soils are similar, they are not identical. The absorption at ~1105 cm^{-1} is probably from vibrations in kaolinite (Fig. 2). However, this peak is diminished in intensity relative to other peaks to such an extent that it forms a shoulder in Newbury 2 and Newbury 3 soils. In addition the sharp peak at 914 cm^{-1} is derived from vibrations in kaolinite and possibly smectite (Russell, 1987). Comparison of the Newbury 1 to 2 and 3 soils reveals a decrease in relative intensity of the peak at 914 cm^{-1}. One plausible interpretation of these different spectral features is that the Newbury 1 sample has a higher kaolinite and/or smectite content than Newbury 2 or 3 samples.

a



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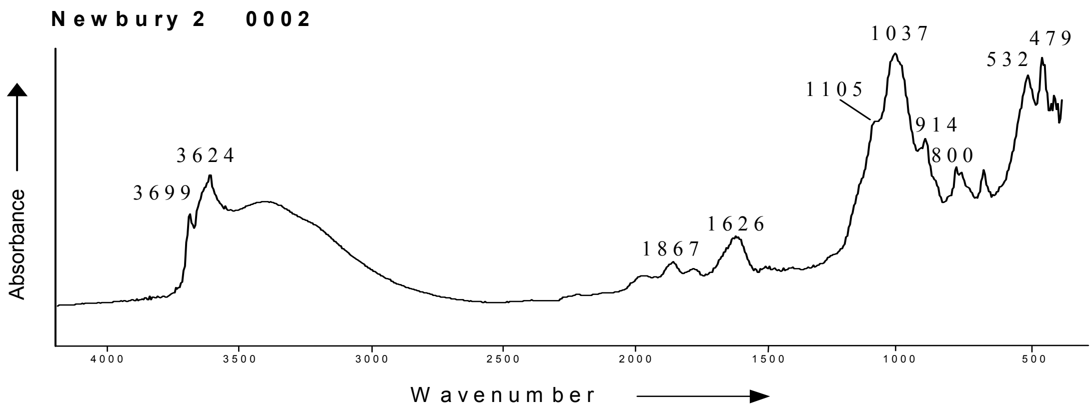

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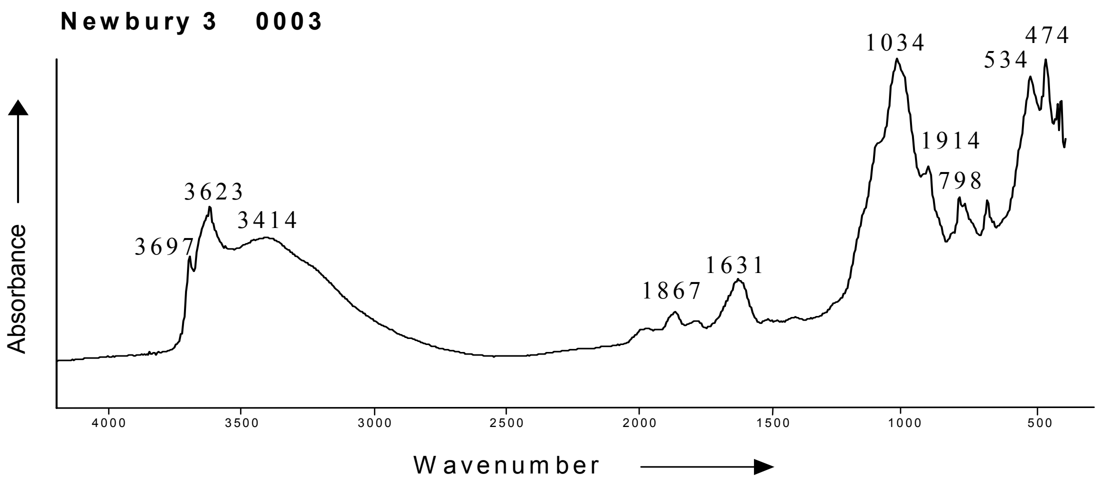

Figure 4. DRIFT Spectra of Newbury clays

3.3 Hemmington and Dorket Head 1

The Hemmington sample spectrum shown in Figure 5 shows broad OH stretching bands at 3412 and 3529 cm^{-1} in contrast to the sharp OH stretching kaolinite absorption bands previously observed in the Newbury samples 1, 2 and 3 (Fig. 4). The apparent absence of sharp peaks in the 3000-3600 cm^{-1} region suggested that kaolinite was not a major component of the Hemmington sample. No doublet at 780-800 cm^{-1} is apparent indicating that the quartz is not necessarily the dominant mineral. However, the peaks at 1990, 1868 and 1794 cm^{-1} are close to those observed in the quartz standard (Fig. 1). Therefore the presence of quartz is indicated in the spectrum (Fig 5). The weak absorption bands at 877, 714 and 1454 cm^{-1} indicate the presence of small amounts of carbonate minerals, namely calcite. The dominant band at 1005 cm^{-1} is at a lower frequency than for many clay soils. Previous FTIR studies have shown that orthoclase yields an intense absorption peak at 1005 cm^{-1} (Van der Marel and Beutelspacher, 1976). Therefore, the presence of a major peak at 1005 cm^{-1} is tentatively assigned to orthoclase. The Dorket Head 1 sample has a similar spectral pattern to the Hemmington sample, although the absorption bands at 876 and 1454 cm^{-1} have shifted to higher frequency of 879, 1480 cm^{-1} respectively (Fig 6). The presence of dolomite as opposed to calcite has been previously shown to cause such a shift to higher frequencies.

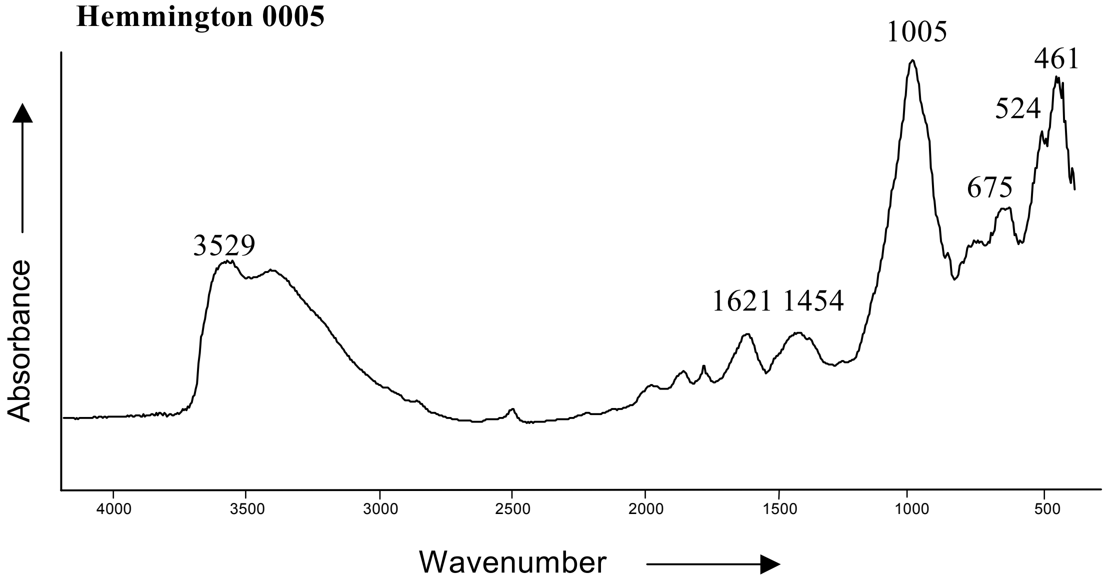

Figure 5. DRIFT Spectrum of Hemmington clay

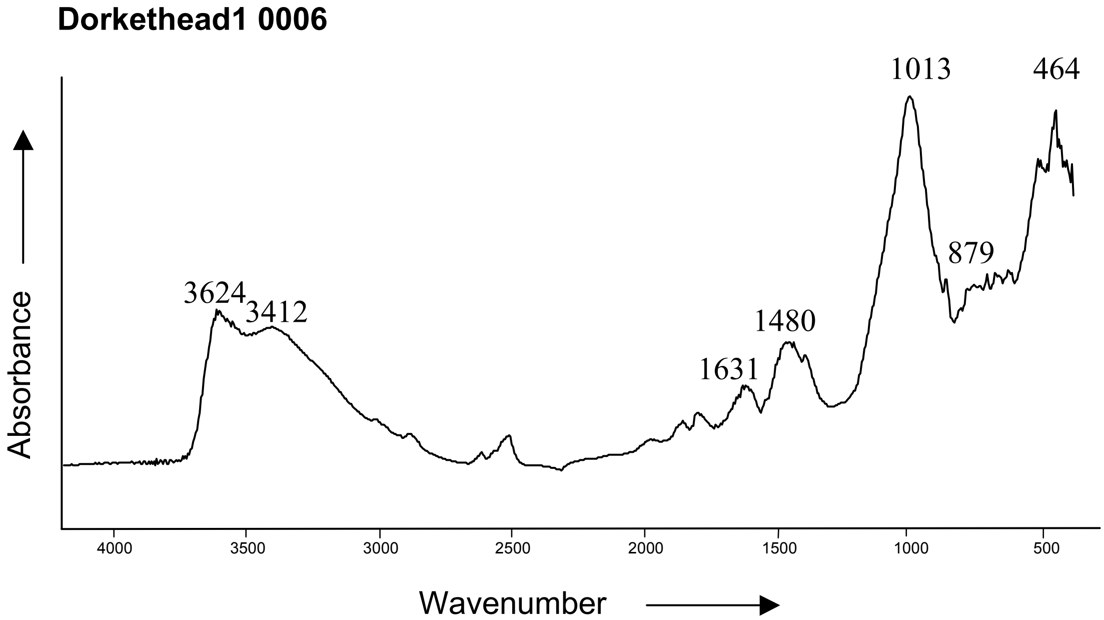

Figure 6. DRIFT Spectrum of Dorket Head 1 clay

3.4 Knoll Manor Farm

The spectrum of the sample from Knoll Manor Farm (Fig. 7) shows the sharp OH doublet of a kaolin mineral at 3622 and 3697 cm^{-1} (Fig. 2). The discreet band at ~915 cm^{-1} is assigned to hydroxyl deformation modes of inner surface and inner hydroxyls in kaolin. Comparison of kaolinite reference material and Knoll Manor Farm spectra also suggest that the peak at 1110 cm^{-1} may also be derived from kaolin vibrations (Fig.2 and Fig 7). The slight broadening under the 3620 cm^{-1} band as well as the relatively weak bands at 750 and 825 cm^{-1} indicate the presence of illite. Additional confirmation of illite is difficult due to overlapping bands from vibrations caused by other minerals (Russell, 1987). The doublet at 780 and 799 cm^{-1} is diagnostic of quartz. The absence of absorption bands at 2512, 2873 and 2983 cm^{-1} suggested that calcite was not present in the sample. The dominant absorption peak at 1032 cm^{-1} may arise from vibrations from kaolinite as well as quartz.

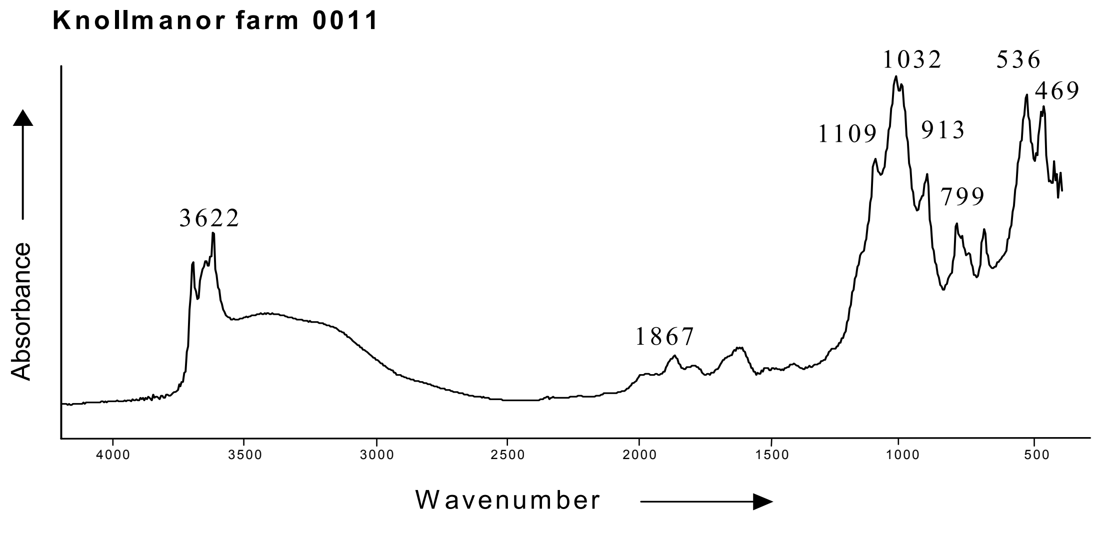

Figure 7. DRIFT spectrum of Knoll Manor Farm clay

3.5 Arlesey 1

The spectrum of the Arlesey sample is different from those discussed previously (Fig. 8). Absorption bands at 1429 along with those at 711, 1795 and 1877 cm^{-1} can be assigned to calcite, this is a plausible interpretation given that calcite is the most common carbonate mineral in clays. The relatively high abundance of the peak at 1434 cm^{-1} suggested that calcite is the major component of the soil. Other carbonate minerals (dolomite, aragonite) are not present or are present in very low amounts since these would shift the absorption at 1434 cm^{-1} to a higher frequency. Another diagnostic spectral feature for calcite is the presence of bands at 2512, 2873 and 2984 cm^{-1} these can be clearly seen in the Arlesey 1 sample spectra. Other minerals are indicated by the spectra namely kaolinite as suggested by the doublet at 3621 and 3697 cm^{-1}. The absorption bands at 780 and 798 cm^{-1} arise from quartz, however the relatively weak bands indicate that quartz is probably a minor component of the samples.

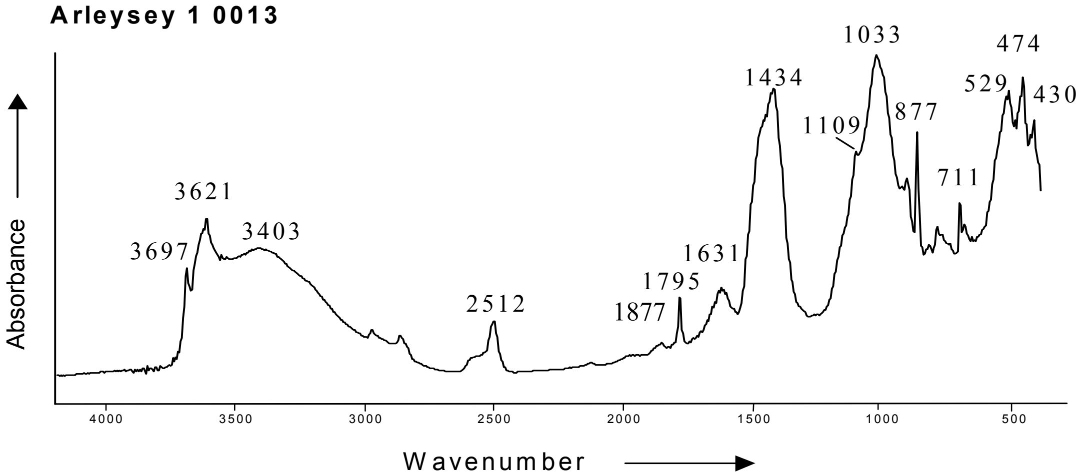

Figure 8. DRIFT Spectrum of Arlesey clay

3.6 Leighton Buzzard

The absorption bands at 3621 and 3697 cm^{-1} can be assigned to kaolinite with supporting bands at 914 and 750 cm^{-1} (Fig. 9). The prominent absorption band at 1434 cm^{-1} and bands at 877 and 2511 cm^{-1} as well as the weak absorption bands at 2873 and 2984 cm^{-1} provide strong evidence for calcite. Comparison of the Leighton Buzzard spectra (Fig. 9) to the Arlesey spectra (Fig. 8) reveal that the peaks at ~1430, 877, 2512, 2873 and 2984 cm^{-1} are diminished relative to other absorption bands in the former sample. This suggested that the calcite content of the Leighton Buzzard sample is lower than that of the Arlesey 1 sample. Nevertheless, calcite is an important component of the Leighton Buzzard soil. Kaolinite gives rise to distinct absorption bands at 3621 and 3697 cm^{-1} which can be clearly seen in the DRIFT spectrum (Fig. 9). The doublet at 780, 799 cm^{-1} and other bands at 695 and 1795 cm^{-1} suggested that quartz is also a major component of the soil.

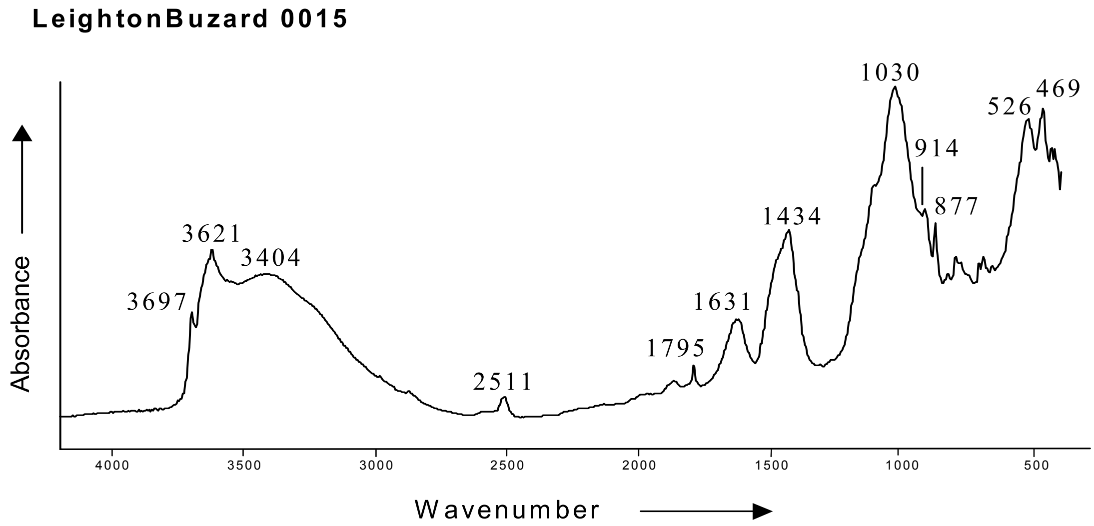

Figure 9. DRIFT Spectrum of Leighton Buzzard clay

3.7 Shepshed 2

The absence of the sharp OH doublet at 3620-3694 cm^{-1} and peaks at 698, 755 cm^{-1} suggested that kaolinite is not an important constituent of the Shepshed 2 sample (Fig. 10) . The strong absorption at 1466 cm^{-1} probably arises from a carbonate mineral. The presence of absorption bands at 729, 881, 2525 and 2627 cm^{-1} as well as 2898 and 3023 cm^{-1} suggest that the carbonate mineral is dolomite (Nguyen *et al.*, 1991). The doublet at 780 and 798 cm-1, which is diagnostic of quartz, is relatively weak in comparison to other absorption bands.

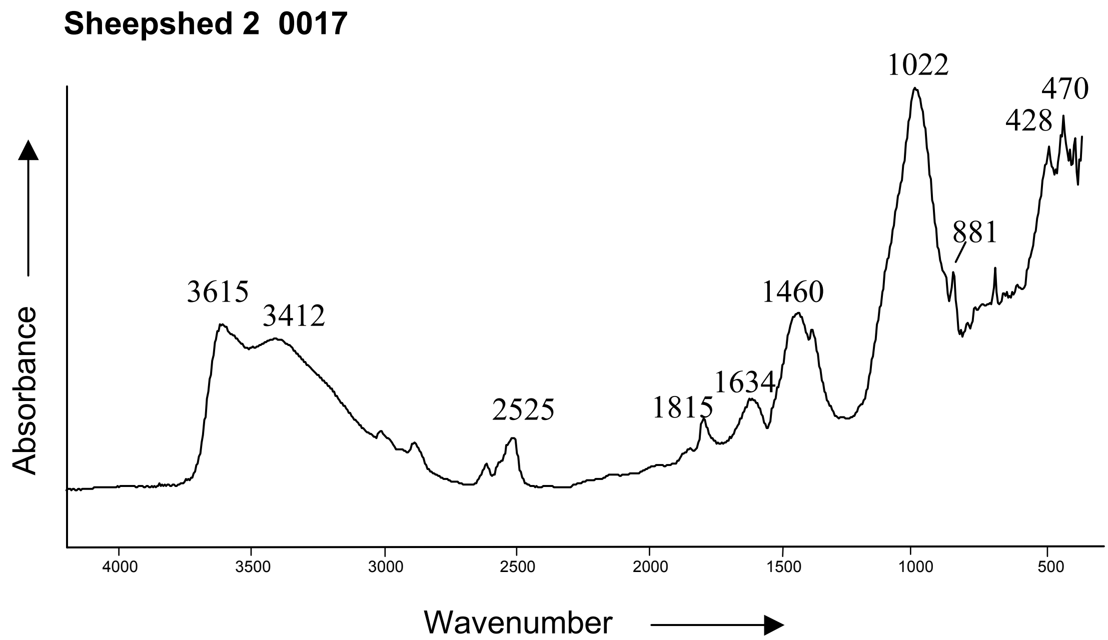

Figure 10. DRIFT Spectrum of Shepshed 2 clay

4. Conclusions

Nine samples of engineering soils known to contain shrink/swell clays and three reference materials were analysed using DRIFT spectroscopy. The major absorption bands originating from quartz included a doublet at 780 and 799cm^{-1} which, can be used as a marker for quartz in soils. Stretching modes of vibration from hydroxyl groups in the clay mineral kaolinite gave strong peaks in the region 3600-3800 cm^{-1} which were used as an indicator for kaolin in shrink/swell clay soils. Infrared spectroscopy was not adept at distinguishing between kaolinite and smectite, however in general DRIFT spectra can be used to differentiate the various clay minerals. The kaolinite and/or smectite content of the Newbury 1 sample was higher than the Newbury 2 and 3 samples, in contrast absorption bands indicative of kaolinite were not observed in the clay sample from Hemmington. Soils from Arlesey were studied by DRIFT spectroscopy, the spectra dominated by bands at 1429 and 2512 cm^{-1}, which originate from the carbonate mineral calcite. No absorption bands from organic matter were observed in any of the shrink swell soils, the most likely explanation being that the strong mineral bands overlap and mask peaks from organic matter. This pilot study shows that DRIFT spectroscopy can be used as an alternative technique to x-ray diffraction for obtaining qualitative information on clay soils. Future studies could characterise a wider variety of shrink swell soils and apply spectral subtraction techniques to aid assignments.

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Russell, J. D., 1987. Infrared methods. In: A handbook of determinative methods in clay mineralogy. (Ed. M. J. Wilson.). Ch 4, pp 133-173. (Blackie and Son: London).

6. Related Publications

Since the project started the following publications and reports have been generated:

Papers:
2000 Hobbs, P.R.N. and Jones, L.D. Clay Shrinkage Research at the British Geological Survey. *Proc. Slips, Shrinks and Swells - Clay Minerals and Geotechnics Conference. British Geological Survey, Keyworth. December 2000*

2000 Lu, P., Rosenbaum, M.S. and Jones, L.D. Fractal Behaviour of Particle Size and its Implications for Describing Volume-Change in Clays. *Proc. Slips, Shrinks and Swells - Clay Minerals and Geotechnics Conference. British Geological Survey, Keyworth. December 2000*

2000 Hobbs, P.R.N., Jones, L.D., Northmore, K.J. and Entwisle, D.C. Shrinkage Behaviour of some Tropical Clays. *Proc. Asian Conf. on Unsaturated Soils. Singapore. May 2000*

1998 Hobbs, P.R.N. and Jones, L.D. The Shrinkage and Swelling Properties of the Mercia Mudstone. *Proc. The Engineering Properties of the Mercia Mudstone. CIRIA Seminar. Pride Park, Derby. November 1998*

Technical Reports:
1999 Jones, L.D. and Hobbs, P.R.N. The shrinking and swelling properties of the Mercia Mudstone. *BGS Technical Report Series No. 98/14*

1998 Jones, L.D. and Hobbs, P.R.N. The shrinking and swelling properties of the Gault Clay. *BGS Technical Report Series No. 98/13*

1995 Hobbs, P.R.N. and Jones, L.D. Methods of testing for swelling and shrinkage of soils. *BGS Technical Report Series No. WN/95/15*

1995 Gostelow, T.P. Some geological aspects of clay swelling and shrinkage. *BGS Technical Report Series No. WN/95/16*

Other Reports:
2001 Jones, L. D. Determination of the Shrinking and Swelling Properties of the Clays of the Lambeth Group. *BGS Internal Report Series No. IR/01/54*

2000 Gunn, D. A. and Jones, L. D. Automatic Shrinkage Limit Apparatus: System Control Software. *BGS Internal Report Series No. IR/01/48*

2000 Jones, L.D. Results of a ‘user’ survey into the need for a greater understanding of shrink/swell behaviour. *BGS Project Note Series No. PN/00/03*

1998 Jones, L.D. Determination of the Shrinking and Swelling Properties of the Mercia Mudstone. *BGS Laboratory Report Series No. 98/03*

1998 Jones, L.D. Report on Visits to Lambeth Group Sampling Sites. *BGS Project Note Series No. 98/09*

1998 Jones, L.D. Sampling Techniques for UK Clay Soils. *BGS Project Note Series No. 98/06*

1997 Jones, L.D. and Cripps, A.C. Report on visit to various sites for sampling, for Mercia Mudstone Project (77BD). *BGS Project Note Series No. 97/10*

1996 Jones, L.D., Hobbs, P.R.N. and Cripps, A.C. Determination of the Shrinking and Swelling Properties of the Gault Clay. *BGS Laboratory Report Series No. 96/02*

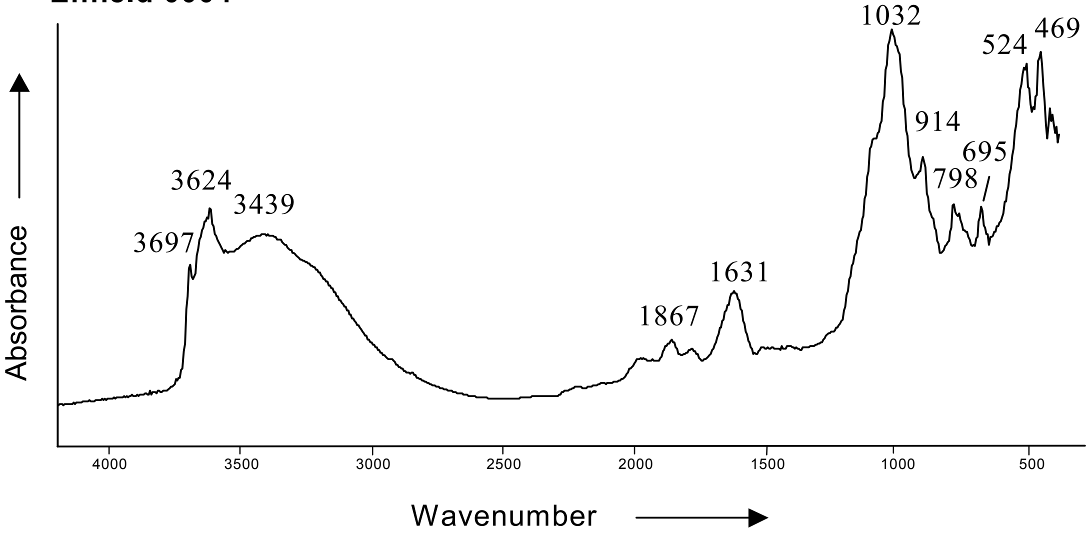
1996 Jones, L.D. and Cripps, A.C. Report on Preliminary Visits to Various Sites for Project 77BH. *BGS Project Note Series No. 96/19*

1996 Jones, L.D. and Cripps, A.C. Report on Visit (and Sampling) to Arlesey and Leighton Buzzard Brickpits for Project 77BH. *BGS Project Note Series No. 96/17*

Thesis:
1999 Jones, L.D. A Shrink/Swell Classification for UK Clay Soils. *Nottingham Trent University, Department of Civil & Structural Engineering.*

7. Appendix

Enfield 0004



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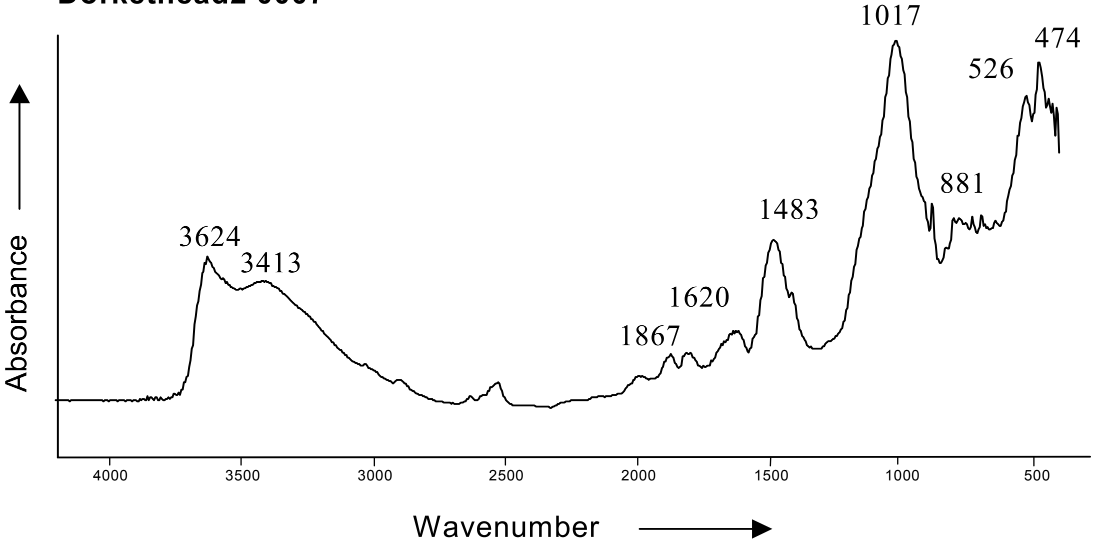

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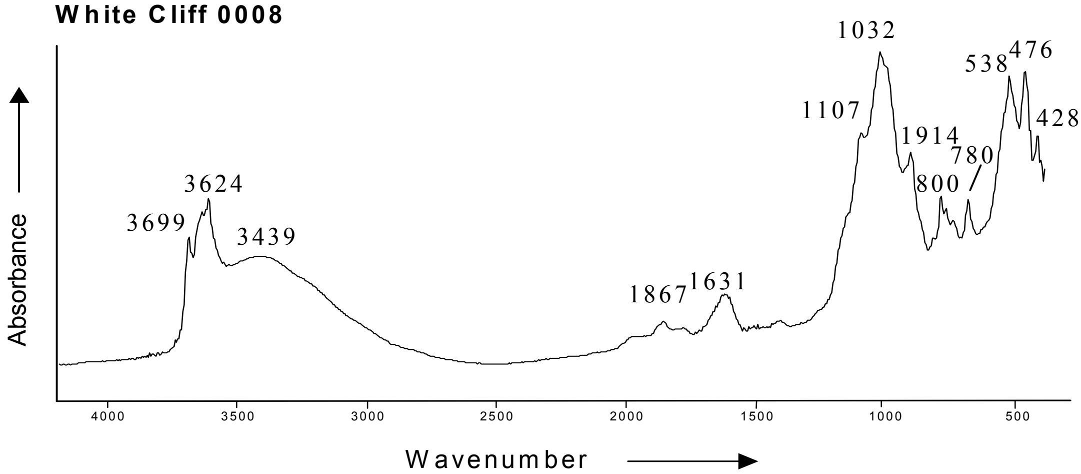

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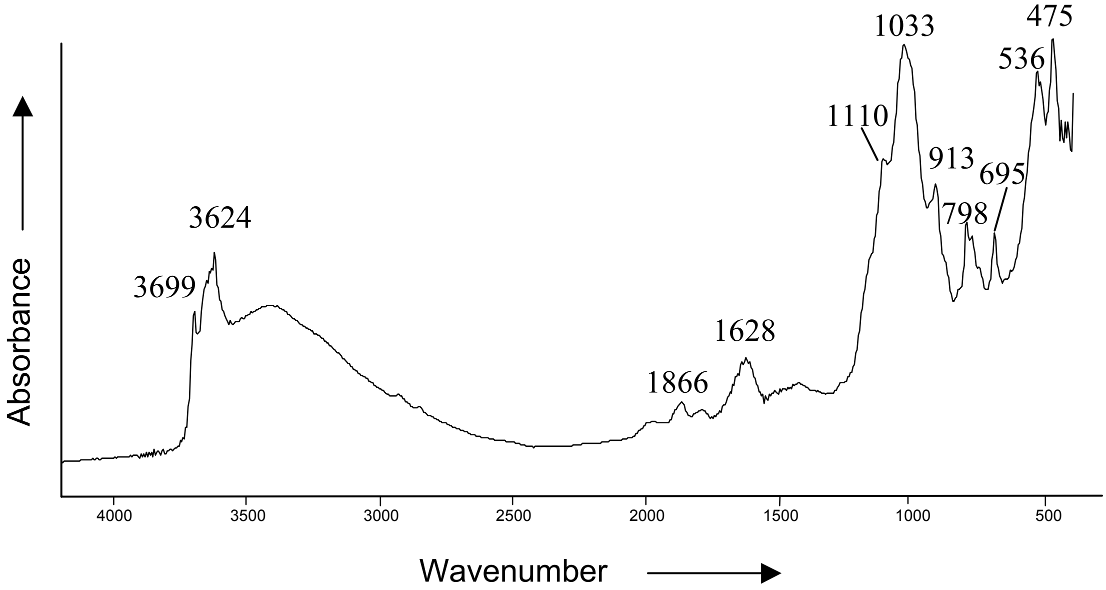

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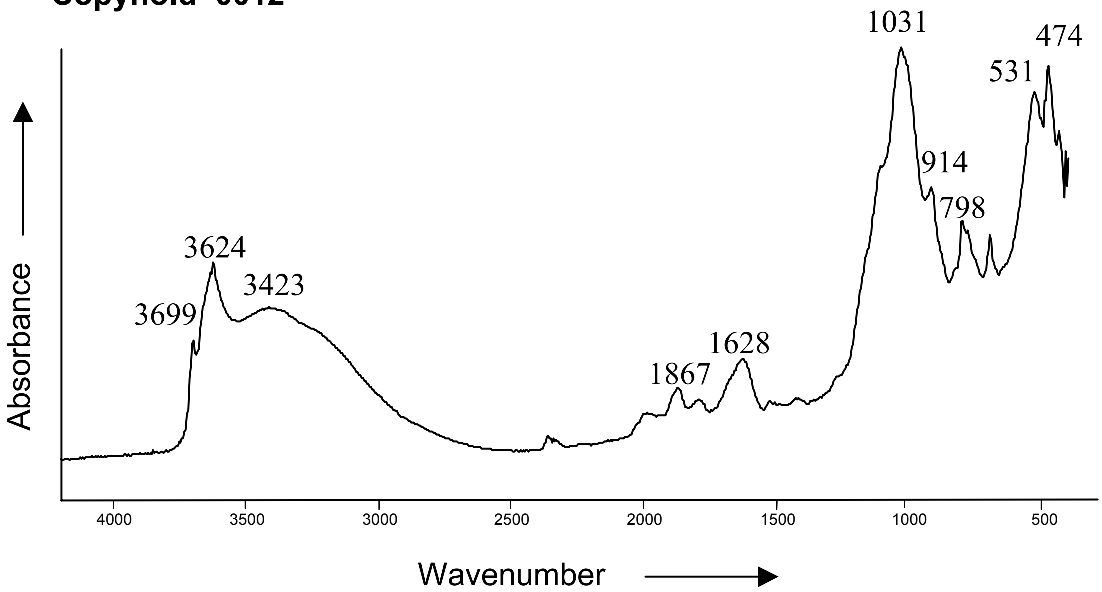

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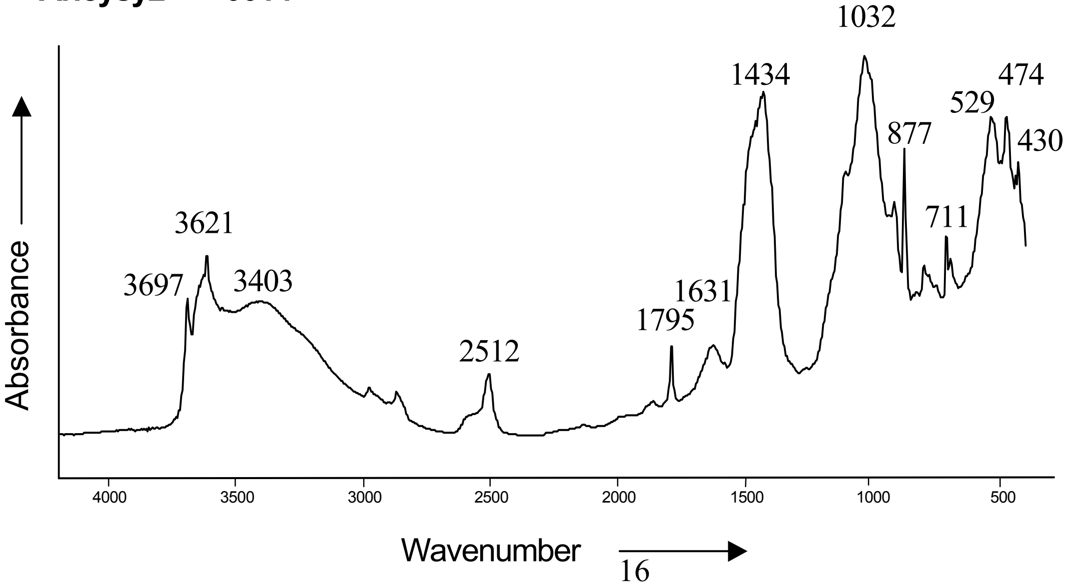

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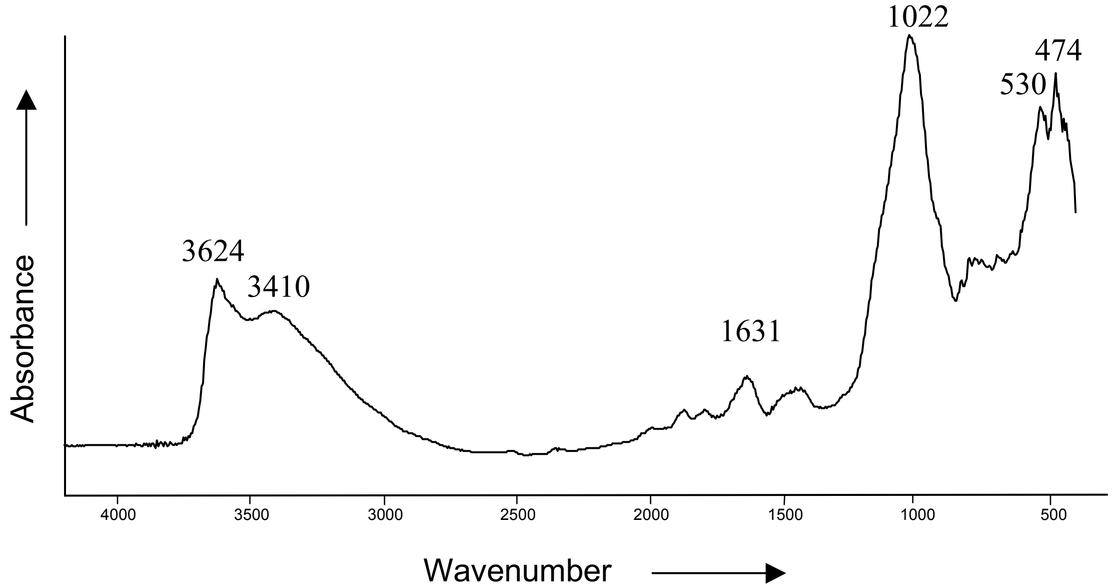

Michelmarsh 0019