

## THE APPLICATION OF DRIFT SPECTROSCOPY TO THE MULTICOMPONENT ANALYSIS OF ORGANIC CHEMICALS ADSORBED ON MONTMORILLONITE

ROBERT W. PARKER<sup>1</sup> AND RAYMOND L. FROST<sup>2</sup>

<sup>1</sup> Queensland Department of Lands, Robert Wicks Research Station, PO Box 178, Inglewood, Q, 4387, Australia

<sup>2</sup> Centre for Instrumental and Developmental Chemistry, Queensland University of Technology  
PO Box 2434, Brisbane, Q, 4001, Australia

**Abstract**—Diffuse Reflectance Fourier Transform Infrared spectroscopy was used to monitor both molecular interactions and concentrations of volatile organic chemicals adsorbed on a commercial montmorillonite. Chemicals tested included propanoic acid, hexanal, heptanal, trimethylamine, dimethylsulfide and dimethyldisulfide. Diffuse Reflectance Fourier Transform Infrared spectroscopy had several advantages over other infrared techniques including ease of sample preparation, greater numbers of useful bands and the ability to detect both major and minor components from the same spectra. Evidence for the formation of organo-clay complexes was found for all chemicals except dimethylsulfide. Spectra of mixed chemicals on the clay showed numerous overlapping bands. Organic concentrations were determined by multicomponent analysis using a least squares curve fitting technique. Significant correlation ( $P < 0.01$ ) between actual and determined concentrations of added organics was obtained for all except dimethylsulfide. Here the weak spectral contribution appeared to be overshadowed by the strongly adsorbing montmorillonite with consequent decrease in sensitivity. Diffuse Reflectance Fourier Transform Infrared spectroscopy of organo-montmorillonite complexes could be used both as a means of studying molecular interactions and for the determination of adsorbed organic concentrations.

**Key Words**—Montmorillonite, Odor, Organic, Slow-release, Sorption.

### INTRODUCTION

Organo-clay complexes have a number of applications in everyday use, including catalysis, decolourization and in pharmaceutical, pesticide and paint preparations (Grim and Guven 1978). An additional application is the use of montmorillonite for the adsorption and desorption of volatile organics as a means of slow release odor generation. This application could be used for the generation of odors for use in attracting pest animals, in particular wild dogs.

Mixtures containing a wide range of organics such as carboxylic acids, amines, aldehydes, alcohols, alkyl aromatics, esters, ketones, terpenes and organo-sulphur compounds have been found to be attractive to coyotes in the United States of America (Prete et al. 1976; Bullard et al. 1978; Turkowski et al. 1983) and to dingoes in Australia (Mitchell and Kelly 1992). Controlling the rate of odor generated may be possible by initial adsorption of these chemicals onto montmorillonite, from which they would subsequently desorb over time. The rate of desorption would depend on the chemical interactions between the organic and the montmorillonite. Consequently, methods were needed for both the study of chemical interactions and the monitoring of the rate of desorption.

Studies of organo-clay complexes have been undertaken using a variety of techniques including X-ray diffraction (XRD) and infrared spectroscopy (Mortland 1970). Fourier Transform Infrared (FT-IR) spec-

troscopy has enabled the infrared spectra of powdered samples to be measured directly (Griffiths and Fuller 1982). One such technique, Diffuse Reflectance Fourier Transform Infrared (DRIFT) spectroscopy, has found increasing application in this field. Applying DRIFT to soils, Nguyen et al. (1991) found several advantages for DRIFT over pressed halide disc transmission spectra. These included minimal preparation, the ability to use neat samples, observation of useful overtone and combination bands, and that most bands were visible in a single DRIFT spectrum without sample dilution or spectral scaling. In addition, there was a greater number of discernible bands in the spectra and those bands, if not affected by 'inversion' or 'restahlen' effects, were generally more prominent than corresponding bands in transmission spectra. Consequently, DRIFT spectroscopy offered a number of features of use to this study, including ease of sample preparation, ability to detect minor components, information on organo-clay interactions, and through multicomponent analysis, a method of determining organic concentrations. Multicomponent analysis would benefit from the greater amount of spectral information present in the DRIFT spectra.

By using DRIFT spectroscopy, it was possible to study both the nature of the organo-clay interactions, and to also indirectly monitor the rate of odor generation by measuring the amount of organic chemical remaining. This paper reports on the application of DRIFT spectroscopy to the determination of several

potential attractant chemicals, namely dimethyldisulfide (DMDS), dimethylsulfide (DMS), hexanal, heptanal, propanoic acid, and trimethylamine (TMA) present as mixtures adsorbed on a commercial montmorillonite.

## METHODS

### Clay Material

The montmorillonite used in this study was obtained from Ipswich, Australia. Previous XRD analysis had shown it to be composed primarily of montmorillonite with trace amounts of dolomite and kaolin. As all preparation techniques may modify clay minerals, both chemical and mechanical pre-treatment of clays should be kept as mild as possible (Brown and Brindley 1980). In addition, extensive pre-treatment also carries a commercial penalty, in that it would have increased the cost of a controlled release formulation. Consequently, refinement of the commercial montmorillonite used in this study was restricted to fine grinding and drying to a constant weight at 130°C.

### Preparation of Samples

**MIXTURES.** Single component organic-clay stock standards were prepared by adding the neat organic liquid to the clay, blending by using a mortar and pestle, and further mixing by agitation. Standard mixtures were then prepared by adding together varying amounts of the stock standards and mixing by agitation. Samples were sealed and stored at -18°C between preparation and analysis. Two mixture sets each containing 4 organic chemicals were prepared, namely: 1) propanoic acid, hexanal, TMA, DMS; and 2) propanoic acid, heptanal, TMA, DMDS.

**DILUTIONS AND PRESSED DISKS.** Dilutions were prepared by adding the required amounts of sample and potassium bromide (KBr), both finely ground, and then mixed by agitation. Pressed discs were prepared by diluting the sample to 2% w/w with KBr, blending, finely grinding using an agate mortar and pestle, and pressing into 10 mm diameter pellets at 8 t under vacuum.

### Infrared Spectra

DRIFT spectra were obtained using a Perkin Elmer 1600 series Fourier Transform Infrared spectrometer. Samples were placed in a stainless steel sample cup (10 mm diameter by 3 mm deep), leveled with the top of the cup and located in a Perkin-Elmer diffuse reflectance accessory. Transmission infrared spectra of pressed KBr pellets were also obtained using this instrument. All spectra were recorded between 4400 and 450  $\text{cm}^{-1}$  over 16 scans at 4  $\text{cm}^{-1}$  resolution, with the sample compartment continually flushed with dry oxygen-free nitrogen for a period of 90 seconds prior and during scan collection. Background spectra were ob-

tained using finely ground KBr. All spectra displayed in this paper have been presented in absorbance mode, which in the case of the DRIFT spectra was  $\log_{10}$  (reflectivity) versus wavenumber.

### Multicomponent Analysis

Perkin Elmer "QUANT®" multicomponent analysis software was used to determine the concentration of adsorbed organics. The entire DRIFT spectrum, from 4400 to 450  $\text{cm}^{-1}$ , but excluding the regions of 2390 to 2280 and 674 to 660  $\text{cm}^{-1}$  (the ranges expected for vibrations arising from atmospheric carbon dioxide), was used.

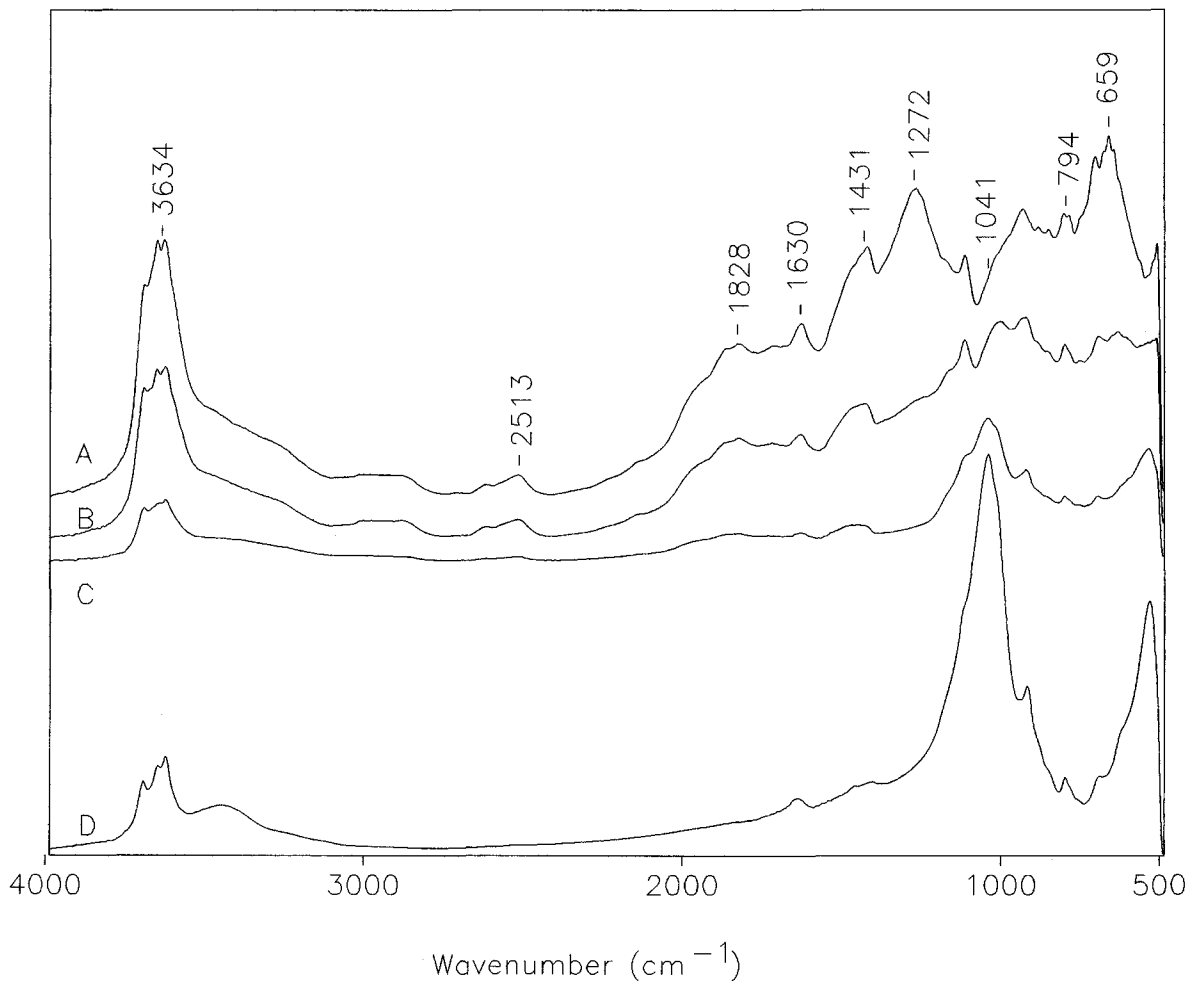
## RESULTS

### Clay Spectra

Figure 1 contains both a pressed KBr pellet and DRIFT spectra for the commercial montmorillonite used in this study. Previous XRD analysis showed this material to be composed of montmorillonite with traces of kaolin and dolomite, which was confirmed from the infrared spectra. The band corresponding to structural hydroxyl stretching contains 3 distinct peaks. The peaks at 3692 and 3647  $\text{cm}^{-1}$  were attributed to kaolin while the peak at 3621  $\text{cm}^{-1}$  was attributed to the montmorillonite, overlapping a similar peak in the kaolin spectrum (Russell 1987). Hydration vibrations, a broad band centred on 3445  $\text{cm}^{-1}$  assigned to OH stretch and a HOH deformation band at 1630  $\text{cm}^{-1}$  were also present. Vibrations at 2925 and 2854  $\text{cm}^{-1}$  could result from either carbonate (dolomite) or the alkyl CH stretch of contaminating organics or spectral overlap of bands from both sources. The DRIFT spectra show peaks at about 2616 and 2515  $\text{cm}^{-1}$  ( $\nu_1 + \nu_3$ ), corresponding to combination frequencies for dolomite reported by Nguyen et al. (1991). Additional bands due to carbonate (dolomite) occur at about 1824 ( $\nu_1 + \nu_2$ ), 1420 ( $\nu_3$ ), 870 ( $\nu_2$ ) and 715 ( $\nu_4$ )  $\text{cm}^{-1}$  in the DRIFT spectra. Farmer (1979) reported bands at 918, 890, 855-815, 805-795, 695, 627, 520-530, 468 and 438  $\text{cm}^{-1}$  for a Wyoming Bentonite; and bands at 937, 913, 790, 756, 697, 650, 543-535, 475-465, 433 and 416  $\text{cm}^{-1}$  for kaolin. Bands at 929, 880, 848, 802, 640 and 624  $\text{cm}^{-1}$  were attributed to the montmorillonite component of this clay, while bands at 782, 656, and 560  $\text{cm}^{-1}$  were attributed to kaolin (Farmer 1979). Bands at 538 and 466  $\text{cm}^{-1}$  were assigned to Si-O bending in the montmorillonite.

### Propanoic Acid-Clay Spectra

Figure 2 contains DRIFT spectra obtained for both neat and KBr diluted propanoic acid-montmorillonite mixtures. Additional peaks observed included alkyl CH stretching vibrations at 2989 and 2951  $\text{cm}^{-1}$  corresponding to alkyl CH stretching, and also a band at 1697  $\text{cm}^{-1}$  corresponding to C=O stretch which over-



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Figure 1. FT-IR spectra of commercial montmorillonite: (A) neat DRIFT, (B) 50% in KBr DRIFT, (C) 5% in KBr DRIFT, and (D) 2% in KBr disc.

laps the hydration HOH deformation band of the clay found at  $1630\text{ cm}^{-1}$ . Bands at  $1543$  and  $1240\text{ cm}^{-1}$  corresponded to the anti-symmetric and symmetric stretching of adsorbed propanate  $\text{C}=\text{O}$  stretch, and bands at  $1472$  and  $1383\text{ cm}^{-1}$  to the alkyl CH deformation. In addition, carbonate bands which appeared at  $2616$ ,  $2515$ ,  $1420$ ,  $870$  and  $715\text{ cm}^{-1}$  in the montmorillonite had moved to  $2646$ ,  $2561$ ,  $1412$ ,  $886$  and  $690\text{ cm}^{-1}$  with the addition of propanoic acid. Additional changes include broadening of the Si-O stretch band, and the formation of peaks at  $1008$ ,  $990$ ,  $802$  and  $750\text{ cm}^{-1}$ . Figures 3 and 4 contain neat DRIFT spectra for individual components of the two mixtures studied.

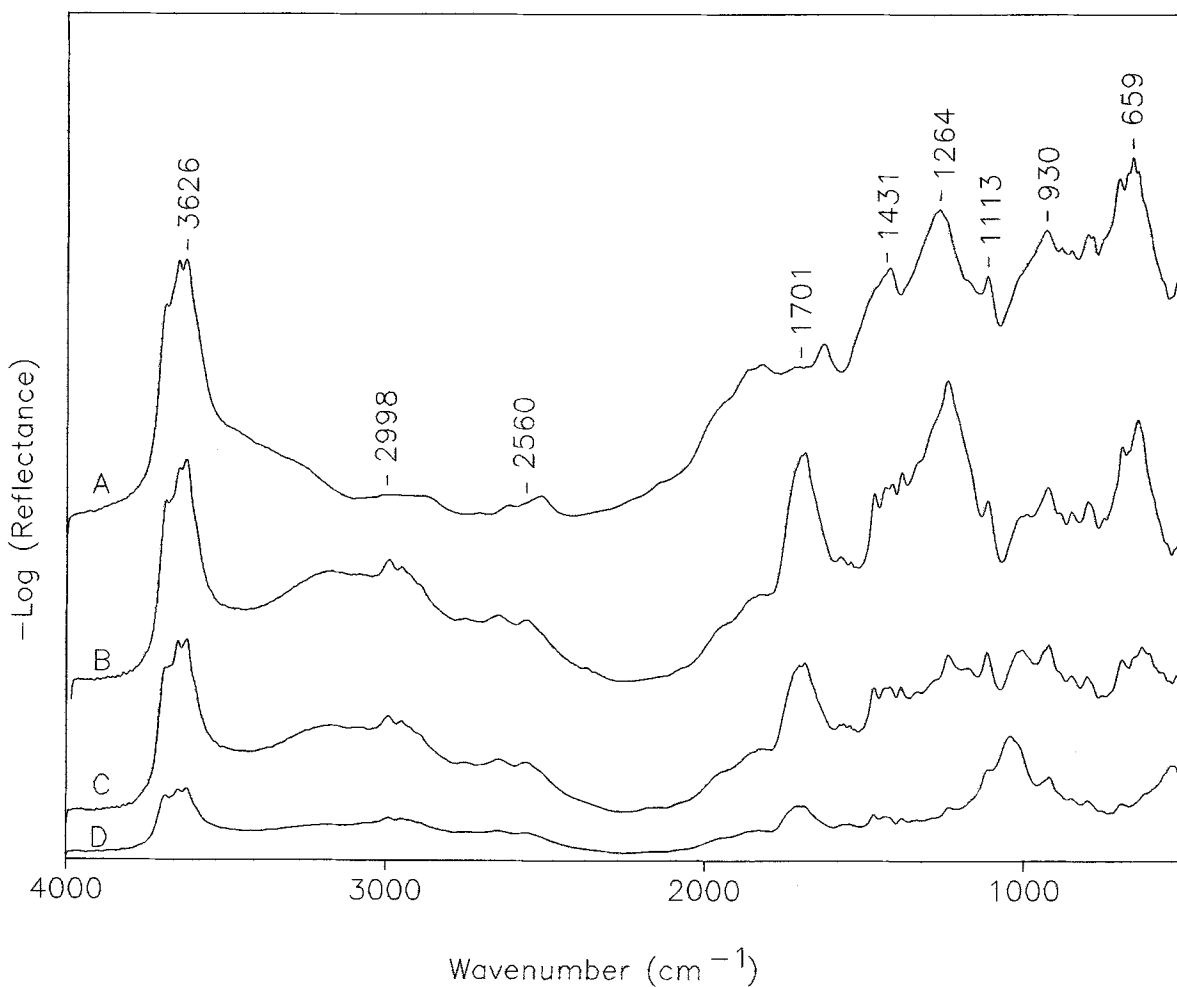
#### Hexanal-Clay Spectra

The neat DRIFT spectra of the hexanal-montmorillonite mixture shows several additional bands includ-

ing:  $2957$ ,  $2871\text{ cm}^{-1}$  (alkyl CH stretch);  $2755\text{ cm}^{-1}$  (aldehyde CH stretch);  $1713\text{ cm}^{-1}$  ( $\text{C}=\text{O}$  stretch); and  $1458$ ,  $1397$  and  $1373\text{ cm}^{-1}$  (CH deformation). Below  $1300\text{ cm}^{-1}$ , substantial band broadening occurred for peaks at  $1239$ ,  $991$ ,  $927$ ,  $847$  and  $796\text{ cm}^{-1}$ . Carbonate peaks observed at  $2616$ ,  $2520$ ,  $1832$ , and  $1419\text{ cm}^{-1}$  were relatively unchanged from similar positions for the blank clay.

#### Heptanal-Clay Spectra

The neat DRIFT spectra for the heptanal mixture was similar to that obtained for hexanal, with bands due to alkyl and aldehyde CH stretching vibrations at  $2931$ ,  $2861$  and  $2755\text{ cm}^{-1}$  respectively, the  $\text{C}=\text{O}$  stretch at  $1718\text{ cm}^{-1}$ , and CH bending at  $1460$  and  $1376\text{ cm}^{-1}$ . Again there was little change for the carbonate bands with peaks found at  $2623$ ,  $2527$ ,  $1814$ ,  $1418$  and  $718\text{ cm}^{-1}$ . Below  $1400\text{ cm}^{-1}$  there is broad-



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Figure 2. (A) neat DRIFT of commercial montmorillonite; and DRIFT spectra of 1.20 mol. kg<sup>-1</sup> propanoic acid adsorbed on commercial montmorillonite: (B) neat DRIFT, (C) 50% in KBr DRIFT, and (D) 5% in KBr DRIFT.

ening of the Si-O band with an additional band at 1201 cm<sup>-1</sup>, and size increases for the bands at 1000, 984, 848, 800, 618 and 530 cm<sup>-1</sup>.

#### Trimethylamine-Clay Spectra

The neat DRIFT spectra for the TMA mixture showed a band at 3240 cm<sup>-1</sup>, corresponding to NH stretching vibration, as well as the alkyl CH stretch bands at 2832 and 2783 cm<sup>-1</sup>. The hydration bands at 3432 (OH stretching) and 1647 cm<sup>-1</sup> (HOH deformation) have increased in size due to the addition of TMA in aqueous solution. The bands at 1456 and 1394 cm<sup>-1</sup> were due to alkyl CH deformation, while the band at 1474 cm<sup>-1</sup> resulted from the NH stretch of trimethylammonium ion and the shoulder at 1312 cm<sup>-1</sup> was assigned to the CN stretching vibration. The carbonate bands at 2525, 1828, 1418 and 712 cm<sup>-1</sup> re-

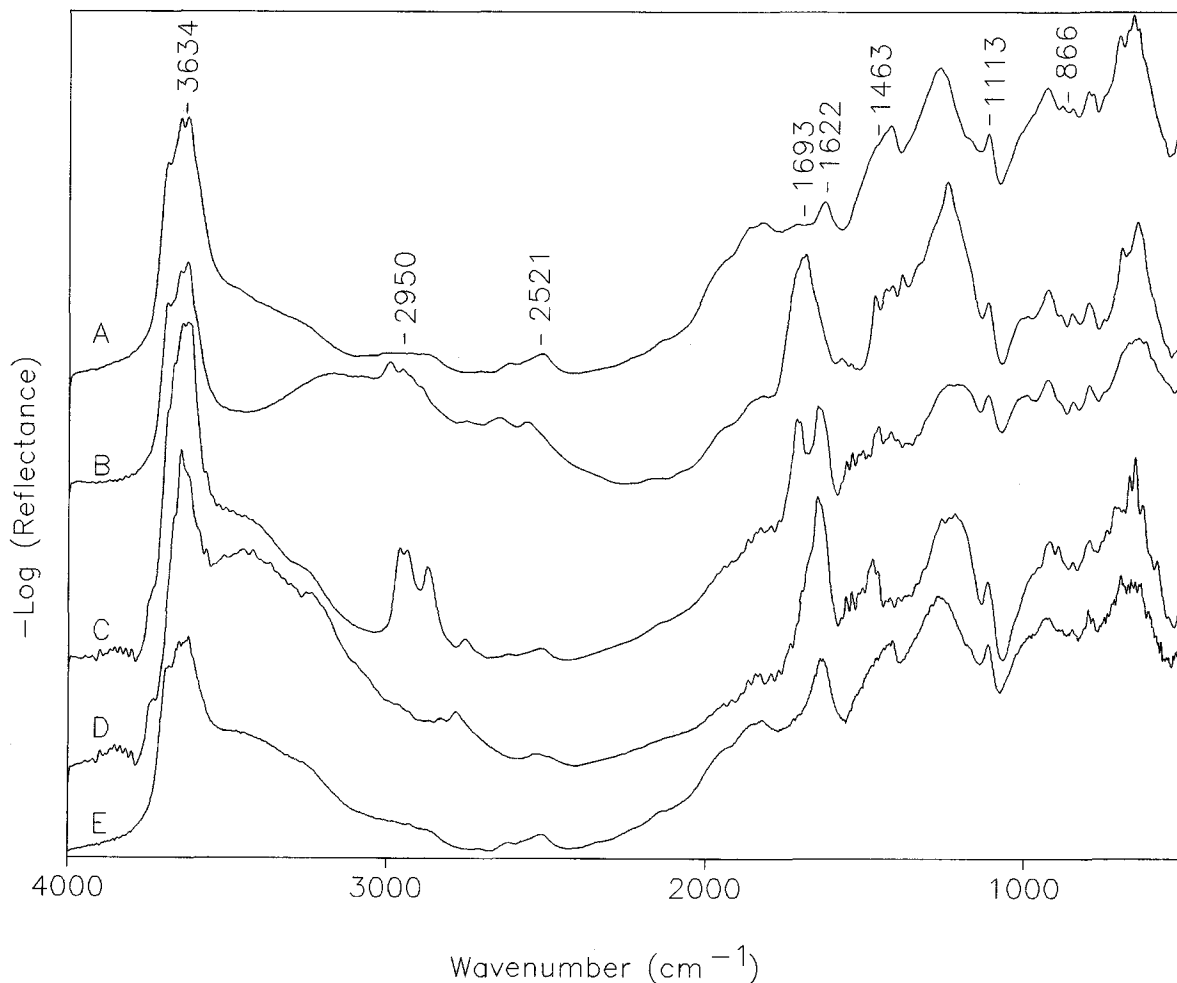
main unchanged. The Si-O stretch band was broadened with a second band appearing at 1216 cm<sup>-1</sup>.

#### Dimethylsulfide-Clay Spectra

The neat DRIFT spectra for the DMS mixture differed little from that of the substrate, with the only additional bands being weak ones at 2930, 1412 and 694 cm<sup>-1</sup>.

#### Dimethyldisulfide-Clay Spectra

The neat DRIFT spectra for the DMDS mixture contained additional bands at 2971, 2928, 2871 cm<sup>-1</sup> (CH stretching vibrations), 1447, 1371 cm<sup>-1</sup> (CH bending vibrations), and at 841, 689 and 523 cm<sup>-1</sup>, with the carbonate band positions unchanged from the blank clay.



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Figure 3. Neat DRIFT spectra: (A) commercial montmorillonite, (B) 1.20 mol. kg<sup>-1</sup> propanoic acid adsorbed on commercial montmorillonite, (C) 0.988 mol. kg<sup>-1</sup> hexanal adsorbed on commercial montmorillonite, (D) 1.19 mol. kg<sup>-1</sup> TMA adsorbed on commercial montmorillonite, and (E) 1.12 mol. kg<sup>-1</sup> DMS adsorbed on commercial montmorillonite.

### Mixture 1 and 2 Spectra

DRIFT spectra for both mixtures 1 and 2 (Figure 5) were similar, with a number of corresponding peaks. For example, mixture 1 had peaks at: 3220 cm<sup>-1</sup> (NH stretch); 2958, 2874 cm<sup>-1</sup> (alkyl CH stretch); 2752 cm<sup>-1</sup> (aldehyde CH stretch); 2539 cm<sup>-1</sup> (propanoic acid-dolomite); 1715 cm<sup>-1</sup> (aldehyde C=O stretch); and 1553 cm<sup>-1</sup> (asymmetric C=O stretch for adsorbed propanoic acid). Corresponding bands for mixture 2 were found at 3240, 2935, 2869, 2755, 2546, 1719 and 1549 cm<sup>-1</sup>.

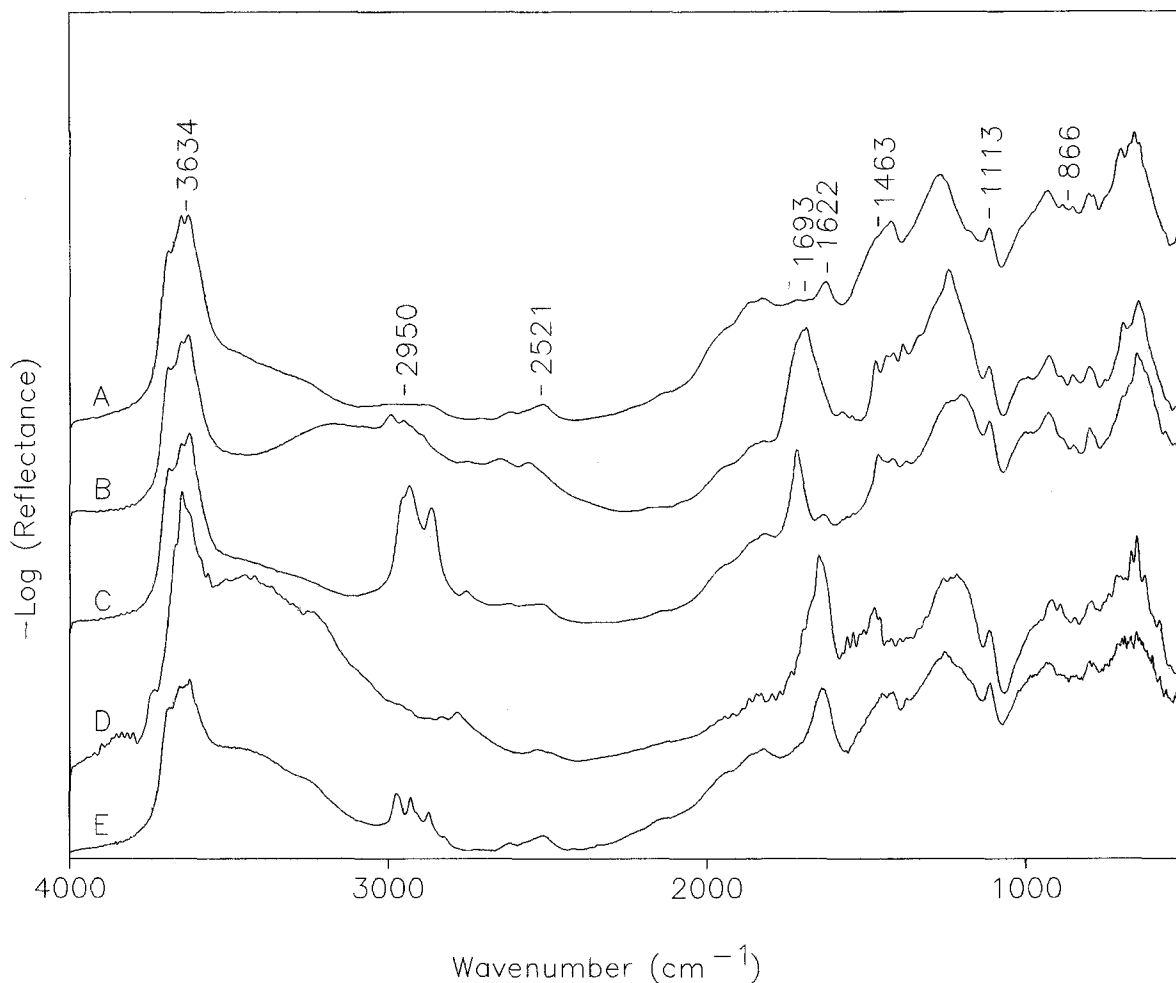
### Multicomponent Analysis

Multicomponent analysis software (Perkin Elmer QUANT<sup>®</sup>) was used to determine component concentrations on a series of prepared samples. Table 1 contains the summary statistics.

### DISCUSSION

#### Clay Spectra

It is obvious from Figures 1 and 2 that more spectral features could be observed in the neat DRIFT spectra than from the transmission spectra of the pressed KBr pellet or the DRIFT spectra of KBr diluted samples. Below 1400 cm<sup>-1</sup> the peaks in the neat DRIFT spectra did show inversion, which could be alleviated by dilution with KBr (Figure 1C), but with loss of sensitivity. For example, the minor band observed at 1113 cm<sup>-1</sup> in the neat DRIFT spectra was overlapped in the KBr pellet spectra by the broad montmorillonite Si-O stretching vibration at 1037 cm<sup>-1</sup>. Nevertheless, peak inversion proved more of a problem to band recognition and assignment than to multicomponent analysis, where the increased sensitivity and digital data han-



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Figure 4. Neat DRIFT spectra: (A) commercial montmorillonite, (B) 1.20 mol. kg<sup>-1</sup> propanoic acid adsorbed on commercial montmorillonite, (C) 0.709 mol. kg<sup>-1</sup> heptanal adsorbed on commercial montmorillonite, (D) 1.19 mol. kg<sup>-1</sup> TMA adsorbed on commercial montmorillonite, and (E) 0.751 mol. kg<sup>-1</sup> DMDS adsorbed on commercial montmorillonite.

dling of FT-IR compensated for small signal differences, inverted peaks, and 'reststrahlen effects'. The sensitivity of the DRIFT spectra for minor components was evident from the DRIFT spectra of the clay that demonstrated clearly the trace level of carbonate by the presence of the combination bands ( $\nu_1 + \nu_2$ ) at 2616 and 2515 cm<sup>-1</sup>. These bands were not evident in the transmission spectrum of the KBr pellet. Using these bands the carbonate could be further identified as dolomite (Nguyen et al. 1991).

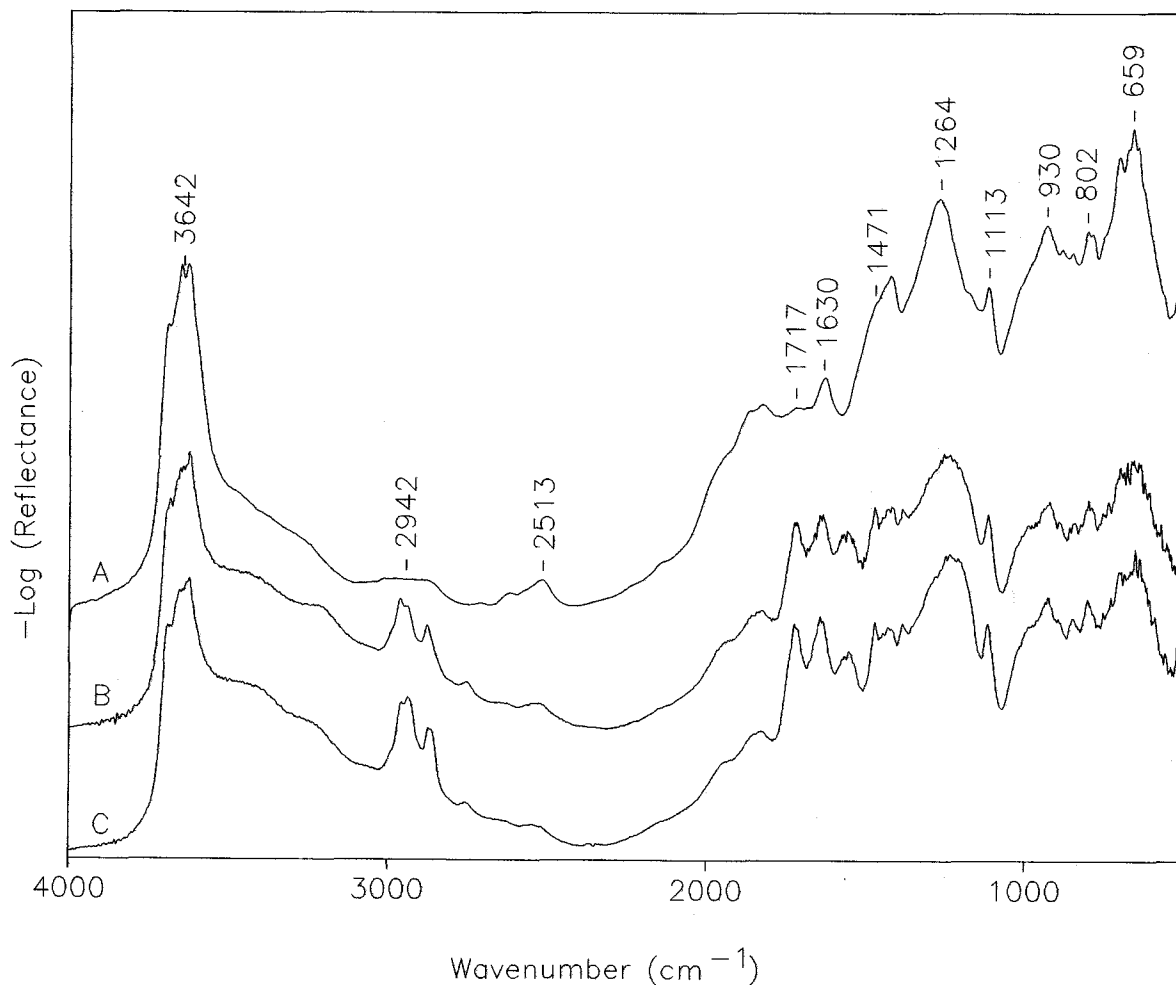
#### Propanoic Acid-Clay Spectra

The change to higher wavenumber of the carbonate bands indicates a reaction between the dolomite and propanoic acid with probable formation of a bicarbonate. For propanoic acid the C=O stretch vibration occurred at 1710 cm<sup>-1</sup> in the neat liquid (Sadler Re-

search Laboratories 1966), but was found at 1697 cm<sup>-1</sup> for the complex studied here. From the Brindley and Moll (1965) model, the propanoic acid was expected to form a short spaced complex within the interlayer with the zig zag of the alkyl carbon chain parallel to the silicate surface. In addition to this band, vibration coupling of the propanate anion resulted in the anti-symmetric stretching vibration at 1543 cm<sup>-1</sup>. This coupling resulted from the propanate anion bonding to montmorillonite through edge surface aluminium ions (Seiskind and Siffert 1972).

#### Hexanal/Heptanal-Clay Spectra

Both hexanal- and heptanal-clay complexes show a shift to lower wavenumber (1713 and 1718 cm<sup>-1</sup>) for the C=O stretching vibration, in contrast to the positions in the neat liquid at 1740 and 1730 cm<sup>-1</sup> (Sadler



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Figure 5. Neat DRIFT spectra: (A) commercial montmorillonite, (B) mixture 1 adsorbed on commercial montmorillonite to give final concentrations of: 0.269 mol. kg<sup>-1</sup> propanoic acid, 0.276 mol. kg<sup>-1</sup> hexanal, 0.252 mol. kg<sup>-1</sup> TMA, and 0.282 mol. kg<sup>-1</sup> DMS; and (C) mixture 2 adsorbed on commercial montmorillonite to give final concentrations of: 0.243 mol. kg<sup>-1</sup> propanoic acid, 0.247 mol. kg<sup>-1</sup> heptanal, 0.251 mol. kg<sup>-1</sup> TMA, and 0.259 mol. kg<sup>-1</sup> DMDS.

Research Laboratories 1969, 1973a). Band broadening below 1400 cm<sup>-1</sup> was also observed for both aldehydes. Theng (1974) suggested that aldehydes bonded to clay via carbonyl group interactions either directly with the exchangeable cations, or through a bridging water molecule. Such bonding was consistent with the changes observed here.

#### Trimethylamine-Clay Spectra

The DRIFT spectra of the trimethylamine mixture was characterized by the presence of NH bonding due to the presence of trimethylammonium ion. The TMA was added as an aqueous solution, resulting in the montmorillonite being intercalated by the trimethylammonium ion (CH<sub>3</sub>)<sub>3</sub>N<sup>+</sup>H, which was expected to react with the montmorillonite by replacing the exchangeable cation, calcium (Theng 1974).

#### Dimethylsulfide-Clay Spectra

The DRIFT spectra for the DMS complex shows few additional bands compared to the substrate. The Sadtler Reference Spectrum (Sadtler Research Laboratories 1975) for DMS contains weak bands at 2960, 2920, 2835, 1430, 1310, 1030, 970 and 690 cm<sup>-1</sup>. In this study the majority of these bands were not detectable in the presence of the strongly adsorbing montmorillonite. In addition, lack of change in the substrate band positions indicated a lack of chemical interaction between the DMS and the clay. This contrasts with ethers which show much more reactivity with montmorillonite (Theng 1974).

#### Dimethyldisulfide-Clay Spectra

In contrast to DMS, the DMDS complex displayed both additional bands and changes in substrate bands.

Table 1. Regression analysis results for actual and determined concentrations for mixture 1 and 2 sample sets for mixture 1 and 2. Slope A, and y-intercept B values for the linear regression equation  $y = Ax + B$  are listed along with the correlation coefficient  $r$ ,  $t$  test value for regression  $t_r$ , and the mean percentage error with 95% confidence intervals.

Comp.	A	B	r	$t_r$	Mean % ( $\pm$ CI) error
Mixture 1. Results using raw DRIFT spectra (N = 11)					
Propanoic Acid	0.877	46.9	0.982#	15.6*	9.32 ( $\pm$ 6.40)
Hexanal	0.873	43.0	0.968#	11.5*	7.98 ( $\pm$ 11.9)
TMA	1.03	-23.6	0.984#	16.4*	-10.3 ( $\pm$ 11.6)
DMS	0.494	122	0.795#	3.80	9.20 ( $\pm$ 23.8)
Mixture 1. Results using KM transformed DRIFT spectra (N = 11)					
Propanoic Acid	0.989	19.6	0.989#	20.3*	8.89 ( $\pm$ 9.60)
Hexanal	0.866	35.5	0.963#	10.8*	2.69 ( $\pm$ 9.49)
TMA	0.990	-6.15	0.964#	10.8*	-3.77 ( $\pm$ 16.1)
DMS	0.0461	207	0.0920	0.278	5.55 ( $\pm$ 35.8)
Mixture 2. Results using raw DRIFT spectra (N = 13)					
Propanoic Acid	0.926	18.8	0.993#	28.4*	2.29 ( $\pm$ 6.04)
Heptanal	0.970	6.80	0.975#	14.6*	-0.499 ( $\pm$ 7.59)
TMA	0.939	22.1	0.985#	19.1*	8.84 ( $\pm$ 9.19)
DMDS	0.765	52.2	0.960#	11.4*	1.87 ( $\pm$ 12.5)

# Significant correlation ( $P < 0.01$ ).

\* Significant regression ( $P < 0.001$ ).

One particular change was the presence of methyl group CH deformation bands at 1447 and 1371  $\text{cm}^{-1}$ . Corresponding bands were found at 1456 and 1394  $\text{cm}^{-1}$  for TMA. Neither reference spectra contain bands for DMS in the region of 1371 nor 1394  $\text{cm}^{-1}$  for DMDS (Sadtler Research Laboratories 1973b, 1976). Their appearance suggested changes in electrical influence on the hetero-atom, and consequently the attached methyl group. In both cases an interaction was occurring between the clay and the organic chemical.

#### Mixture 1 and 2 Spectra

Mixture DRIFT spectra showed no evidence of additional interactions due to reactions between components. This indicated that the preparation of the mixtures by separate blending of component organo-clay complexes and then mixing these together ensured little interaction between the organic components. The DRIFT spectra for both mixture 1 and mixture 2 contained a large number of overlapping bands, with very few individual bands able to be assigned to one particular component. Consequently, a multicomponent analysis procedure would be required to obtain reliable quantitative concentration data.

#### Multicomponent Analysis

The multicomponent analysis software, QUANT<sup>®</sup>, used a least squares curve fitting technique. This technique determined the linear combination of the standard spectra which gave the best fit to the sample spectra using least squares criterion (Ford and Spragg 1985). To test the applicability of this method a series of test samples were prepared and analyzed. Determined values were compared with actual values cal-

culated from the amounts of organic initially added to the clay. Summary statistics for these comparisons are contained in Table 1. A least squares estimation was used to determine slope and intercept values for the following linear regression equation [1]:

$$\text{Measured Concentration} = A \times \text{Actual Concentration} + B \quad [1]$$

with ideal values of A and B being 1 and 0 respectively. The values obtained for DMS deviated strongly from these values, while for the remaining organics, the value of B was generally positive indicating an over estimation of concentration as zero concentration was approached. Mean percentage errors for measured concentrations ranged from -10.3 to 9.32%. While the DRIFT spectra for the mixtures appeared to be dominated by aldehyde vibrations, there was no significant variation between analytes for either mixture (single classification ANOVA,  $P < 0.05$ ). In addition, actual and measured values were significantly correlated ( $P < 0.01$ ) and exhibited highly significant regression ( $P < 0.001$ ) for all analytes except DMS. The poor sensitivity obtained for DMS was probably caused by the lack of distinct spectral features in the DRIFT spectrum in contrast to the strongly absorbing montmorillonite.

As DRIFT spectra do not exhibit a linear relationship between spectral data and concentration (Fuller and Griffiths 1978), the use of KM transformed spectra to achieve linearity for mixture 1 should have improved the results. This was not the case, with a comparison of corresponding percentage error means showing no significant difference ( $t$ -test,  $P < 0.05$ ) and no improvement in either regression or correlation. This in turn confirms the claim by Ford and Spragg



(1985) that this method can readily deal with non-linear spectra.

#### CONCLUSIONS

Despite the appearance of spectral inversion and 'restrahlen' effects in DRIFT spectra the use of such spectra offered several advantages to this study. These included ease of sample preparation and the ability to detect both major and minor spectral features, as well as the presence of a large number of spectral bands resulting in increased sensitivity for quantitative analysis. Most of the organics showed some evidence of interaction with the clay with both physi- and chemisorption occurring and DRIFT spectra could also be used to monitor the contribution of each type. Multi-component analysis of DRIFT spectra using a least squares curve fitting technique provided an effective means of measuring low concentrations (ca 10 mg·g<sup>-1</sup>) of propanoic acid, hexanal, heptanal, TMA, and DMDS, but not DMS, adsorbed as mixtures on a commercial montmorillonite substrate. Consequently this method has potential for indirectly monitoring mixtures of these chemicals on clay.

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