

DIFFERENCES IN APPARENT INTERSTRATIFICATION  
OF AN ORGANO-MONTMORILLONITE DISPERSED  
IN VARIOUS ORGANIC SOLVENTS  
I. X-RAY DIFFRACTION STUDY

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ABSTRACT

The degree and type of interstratification of a dimethyldioctadecylamine montmorillonite after dispersion in various organic solvents were studied by X-ray diffraction methods. Diffraction data from oriented films of the specimens were obtained at temperatures of 35°, 60°, and 110°C. From these data Fourier curves and Fourier transforms were prepared.

The organic solvents used were benzene, a mixture of 10 percent methanol and 90 percent toluene, dioxane, cyclohexanone, *n*-methyl-2-pyrrolidone, dimethyl formamide, butyrolactone, *n*-methylisobutyl ketone and acetone. These were classified according to various physical properties, such as the solubility of dimethyldioctadecylammonium chloride in each, the contact and wetting speed of each solvent on the organo-clay, surface tension, solubility of water in each, density, and others. A comparison of the physical properties with the Fourier curves and Fourier transform curves at various temperatures showed a general relation between the amount of apparent interstratification with the structure of the solvent and type of interaction between the solvent and the organo-montmorillonite.

INTRODUCTION

Many natural clays consist of particles in which layers of different types of clay minerals are stacked together. This property is called interstratification, and clay specimens having this property are generally referred to as mixed-layer clays (H. Van Olphen, 1963). During the last twenty-five years many interstratified clay minerals and laboratory prepared clay complexes have been characterized by the aid of X-ray diffraction and Fourier techniques (Brown, *et al*, 1951; MacEwan, 1956; McAtee and co-workers, 1956, 1958, 1964; Tettenhorst and Johns, 1966).

The present work was oriented toward a study of the effect of various organic solvents on the apparent interstratification of an organo-montmorillonite (dimethyldioctadecylammonium montmorillonite). After dispersing the organo-clay in the various organic solvents, oriented specimens were prepared which were then dried at temperatures of 35°, 60°, and 110°C. X-ray diffraction patterns of these specimens were obtained while holding the sample at these same temperatures. From the diffraction patterns, Fourier diagrams and Fourier transforms were constructed.

The Fourier diagrams were used to gain information with regard to the apparent number and thickness of the organic layers. The Fourier transforms yielded data regarding the apparent type of interstratification of

the organo-clay layers (MacEwan, 1956; Mering 1949; MacEwan *et al*, 1961). From this information, ideas were developed relating to the interaction of the organo-clay with the organic solvents.

It should be emphasized that throughout this work the authors realized that the limited number of X-ray reflections would lead to Fourier diagrams and transforms that could be used only in a limited way. It is believed, however, that the curves yield valuable information and that they can be used to show general trends and differences with regard to the interaction of the amine, clay and organic solvents.

#### MATERIALS AND EXPERIMENTAL METHODS

For this study a centrifuged Wyoming bentonite obtained from the Baroid Division of the National Lead Company, Houston, Texas, was used. X-ray diffraction data indicated this material consisted principally of the clay mineral montmorillonite. The organic amine and solvents employed were obtained from commercial sources. The amine was technical grade while the solvents were all cp grade materials.

The preparation of the organo clay was made by adding the desired amount of dimethyldioctadecylammonium chloride to homoionic sodium montmorillonite. This was then filtered and washed and then dried for forty-eight hours at 65°C. The sample was then ground to -200 mesh and the amount of organic retained on the clay was determined by loss on ignition. The sample used in this study contained 105 meq. of organic cation per 100 g. of dry clay minerals.

The organo-clay sample was dispersed by means of a Spex mill in several organic solvents: namely benzene, a mixture of 10 percent methanol and 90 percent toluene, dioxane, cyclohexanone, *n*-methyl-2-pyrrolidone, dimethyl formamide, butyrolactone, methylisobutylketone, and acetone. Oriented films prepared from the dispersed samples were dried at 35°, 60° and 110°C. A Philips High Angle Diffractometer using Ni filtered CuK $\alpha$  X radiation with a heating stage was used to obtain the diffraction patterns. During the X-ray examination, the temperature was kept constant at the desired temperature of 35°, 60°, or 110°C. by means of a West Temperature Controller. X-ray data on each specimen was obtained from at least duplicate runs and in many instances they were obtained in triplicate.

#### EXPERIMENTAL RESULTS AND DISCUSSION

The general equation used to obtain the Fourier diagrams is as follows:

$$S = \sum F_o \cos \frac{2\pi ly}{d} \quad (1)$$

where  $S$  is the Fourier series,  $y = dx$ ,  $l$  is the order of reflection,  $F_o$  is the observed structure factor,  $d$  is the Bragg spacing, and  $x$  is the distance in Å from the symmetrical center of the clay structure.

The intensity,  $I$ , of a given Bragg reflection can be expressed as:

$$I = KF_o^2\theta TA \quad (2)$$

where  $K$  is an instrumental constant,  $\theta$  is the Lorentz-polarization factor,  $T$  is the temperature factor and  $A$  is the absorption factor. For the rela-

tively low  $2\theta$  angles employed the absorption factor leads to a negligible correction on the data. Likewise the temperature factor  $T$  was neglected since these experiments were carried out well below the melting points of the specimens. It is recognized that as the temperature increases a general decrease in the peak intensities would occur, but because of the qualitative nature of these experiments a temperature correction was not made. On rearranging, equation (2) becomes:

$$F_o^2 = \frac{I}{K\theta} \quad (3)$$

Tables 1, 2, and 3 give the X-ray diffraction data of the basal spacings of the organo-montmorillonite dispersed in the different solvents: dried and X rayed at  $35^\circ$ ,  $60^\circ$  and  $110^\circ\text{C}$ . Utilizing this data, one dimensional Fourier diagrams were constructed from which the apparent thicknesses and number of organic layers were calculated. The results of these calculations are given in Tables 4, 5, and 6.

Using the number of apparent organic layers as obtained from the Fourier diagrams, structure factor curves of  $F_o^2$  vs  $\sin \theta$  were constructed (McAtee, 1958). From these curves, values of  $F_c^2$  were obtained for use in Fourier transform calculations.

The cosine transform used was a summation formula shown as follows:

$$T_R = \sum_R \frac{I_R}{\theta(F_c)^2} \cos 2\pi\mu R^R$$

where  $T_R$  is essentially the Patterson line projection along the  $c$ -axis of the crystallites. This value is proportional to the mean number of layers at a distance  $R$  from an arbitrary layer chosen as the origin,  $I_R$  is the integrated intensity for each value of the reciprocal spacing,  $M_R$ ,  $\theta$  is the Lorentz-polarization factor and  $F_c$  is the theoretical structure factor for the number of layers of organic material under consideration. In this work, the structure factors employed were calculated by algebraically combining structure factors obtained for regular layered montmorillonite. For example, if the  $d_{001}$  value and the Fourier diagram suggested that the specimen was a mixture of the three- and four-layered montmorillonite, the corresponding synthetic three- and four-layer structure factor curve was calculated by algebraically averaging the curves for the three- and four-layer structure factor curves. This new curve of  $F_{3-4}$  vs  $\sin \theta$  was then used in the subsequent transform calculations. The purpose of this was to aid in the reduction of spurious peaks in the transform, since it was recognized that the number of diffraction lines being used was not sufficient to yield smooth curves.

TABLE 1. X-RAY DIFFRACTION DATA OF BASAL SPACINGS AND RELATIVE INTENSITIES OF ORGANO-MONTMORILLONITE AT 35°C.

Solvent	1st order $d(\text{Å})$	$I/I_0$	2nd order $d(\text{Å})$	$I/I_0$	3rd order $d(\text{Å})$	$I/I_0$	4th order $d(\text{Å})$	$I/I_0$	5th order $d(\text{Å})$	$I/I_0$	6th order $d(\text{Å})$	$I/I_0$	7th order $d(\text{Å})$	$I/I_0$	8th order $d(\text{Å})$	$I/I_0$	9th order $d(\text{Å})$	$I/I_0$	10th order $d(\text{Å})$	$I/I_0$	11th order $d(\text{Å})$	$I/I_0$	
Methyl isobutyl ketone	30.40	100	18.60	25.4	11.90	4.4	9.25	7.2	7.70	2.2	6.10	10.7	5.20	9.6	4.55	22.7	3.68	10.9	3.29	8.0			
Acetone	27.60	100	13.20	15.6	8.90	2.1	6.90	2.1	5.25	2.1	4.40	6.7	3.64	2.1	3.28	2.1							
Benzene	29.10	100	19.50	100	9.50	3.1		6.40	0.4	5.23	5.4	4.45	11.4	3.67	4.3	3.25	2.8						
10% MeOH+90% C <sub>6</sub> H <sub>6</sub>	23.20	100	12.30	20.5	8.40	1.4	6.13	2.2	5.08	4.0	4.35	6.5			3.40	4.8							
Butyrolactone	26.00	100	14.30	23.6	9.00	2.5		5.43	3.3	4.35	14.2	3.80	6.1	3.40	4.8								
N-methyl-2- pyrrolidone	25.70	100	13.40	17.1	9.10	1.8	6.50	3.8	5.30	5.4	4.40	10.2			3.40	4.8							
Dimethyl formamide	25.20	100	12.40	69.6	8.82	5.9	6.28	7.1	5.40	10.2	4.37	23.2	3.63	1.20	3.26	19.6							
Dioxane	32.40	100	16.50	45.6	11.70	10.2	8.60	3.4	6.20	5.9			4.60	27.6									
Cyclohexanone	32.20	100	17.40	39.7	11.50	6.7	8.90	2.4	6.70	1.1	5.90	1.9	5.10	5.0	4.50	6.9	3.50	2.6	3.25	4.6			

TABLE 2. X-RAY DIFFRACTION DATA OF BASAL SPACINGS AND RELATIVE INTENSITIES OF ORGANO-MONTMORILLONITE AT 60°C.

Solvent	1st order $d(\text{Å})$	$I/I_0$	2nd order $d(\text{Å})$	$I/I_0$	3rd order $d(\text{Å})$	$I/I_0$	4th order $d(\text{Å})$	$I/I_0$	5th order $d(\text{Å})$	$I/I_0$	6th order $d(\text{Å})$	$I/I_0$	7th order $d(\text{Å})$	$I/I_0$	8th order $d(\text{Å})$	$I/I_0$	9th order $d(\text{Å})$	$I/I_0$	10th order $d(\text{Å})$	$I/I_0$	11th order $d(\text{Å})$	$I/I_0$	
Methyl isobutyl ketone	31.50	100	15.50	24.6	10.50	1.2			6.46	1.1	5.54	6.6			4.62	4.1	3.45	2.1	3.23	2.2			
Acetone	31.80	100	15.75	55.9	11.20	2.1					5.54	2.6	4.72	6.5			3.50	1.5	3.26	1.1			
Benzene	31.50	100	16.00	26.4	11.00	2.3					5.46	2.9	4.70	8.3			3.50	3.7	3.22	1.8			
10% MeOH+90% C <sub>6</sub> H <sub>6</sub>	32.70	100	16.50	31.8	10.80	1.5					5.30	1.2	4.64	3.1									
Butyrolactone	28.20	100	14.30	11.4	9.38	1.3			5.62	2.0	4.59	4.8			3.46	1.2							
N-methyl-2- pyrrolidone	34.80	100	16.70	54.0	11.20	2.9					5.30	6.2	4.66	6.9	4.23	2.9							
Dimethylformamide	26.80	100	15.20	41.3			7.36	53.8	5.06	21.3	4.53	83.8	4.02	46.3	3.53	16.3							
Dioxane	37.00	100	18.20	49.3	12.00	4.9					5.70	2.4			4.79	2.7							
Cyclohexanone	35.60	100	17.20	64.3	11.70	17.5							4.95	2.25	4.46	20.8	4.20	19.3	3.32	5.0			

TABLE 3. X-RAY DIFFRACTION DATA OF BASAL SPACINGS AND RELATIVE INTENSITIES OF ORGANO-MONTMORILLONITE AT 110°C.

Solvent	1st order $d(\text{\AA})$	$I/I_0$	2nd order $d(\text{\AA})$	$I/I_0$	3rd order $d(\text{\AA})$	$I/I_0$	4th order $d(\text{\AA})$	$I/I_0$	5th order $d(\text{\AA})$	$I/I_0$	6th order $d(\text{\AA})$	$I/I_0$	7th order $d(\text{\AA})$	$I/I_0$	8th order $d(\text{\AA})$	$I/I_0$	9th order $d(\text{\AA})$	$I/I_0$	10th order $d(\text{\AA})$	$I/I_0$	11th order $d(\text{\AA})$	$I/I_0$
Methyl isobutyl ketone	31.80	100	15.90	37.8	11.20	2.2					5.40	2.9	4.66	6.5			3.56	1.4	3.18	1.3		
Acetone	28.90	100	14.00	51.8	9.48	1.8		5.67	4.1	4.73	12.1				3.50	4.1						
Benzene	30.50	100	14.70	51.9	10.00	4.2					5.47	3.4	4.77	9.5	3.56	3.9			3.21	3.0		
10% MeOH + 90% $C_6H_6CH_3$	32.00	100	16.00	36.3	11.20	2.5					5.50	0.4	4.73	3.9			3.57	1.5				
Butyrolactone	28.80	100	14.30	24.6	9.80	1.6		5.53	2.3	4.72	6.0		3.95	1.4	3.48	7.7	3.13	1.3				
N-methyl-2- pyrrolidone	34.20	100	16.90	41.6	11.30	3.7					5.73	2.4	4.87	2.6								
Dimethyl formamide	35.30	100	15.20	22.0				6.80	63.6	5.86	31.8		4.92	44.5					3.52	36.4	3.25	36.4
Dioxane	34.20	100	17.00	29.5	11.50	2.4					5.73	2.4	4.82	2.6								
Cyclohexanone	35.80	100	17.30	44.2	11.60	9.8							5.34	7.8	4.66	15.2	4.18	7.8	3.76	6.7		

TABLE 4. THICKNESS OF LAYERS FROM FOURIER CURVES AT 35°C.

Solvent	1st order $d(\text{\AA})$	No of layers	1st layer $d(\text{\AA})$	2nd layer $d(\text{\AA})$	3rd layer $d(\text{\AA})$	4th layer $d(\text{\AA})$	5th layer $d(\text{\AA})$	6th layer $d(\text{\AA})$	7th layer $d(\text{\AA})$
Methyl isobutyl ketone	30.40	4	5.60	4.80	4.80	5.60			
Acetone	27.60	4	4.30	4.70	4.70	4.30			
Benzene	29.10	4	5.10	4.60	4.60	5.10			
10% MeOH + 90% $C_6H_6CH_3$	23.20	4	3.00	3.80	3.80	3.00			
Butyrolactone	26.00	3	5.40	5.60	5.40				
N-methyl-2- pyrrolidone	25.70	4	4.00	4.10	4.10	4.00			
Dimethyl formamide	25.00	4	3.40	4.40	4.40	3.40			
Dioxane	32.40	4	6.50	4.90	4.90	6.50			
Cyclohexanone	32.20	7	3.60	3.40	3.40	1.80	3.40	3.40	3.60

TABLE 5. THICKNESS OF LAYERS FROM FOURIER CURVES AT 60°C.

Solvent	1st order $d(\text{Å})$	No of layers	1st layer (Å)	2nd layer (Å)	3rd layer (Å)	4th layer (Å)	5th layer (Å)
Methyl isobutyl ketone Acetone	31.50	4	5.80	5.20	5.20	5.80	
	31.80	3	7.60	7.00	7.60		
Benzene 10% MeOH + 90% $C_6H_5CH_3$	31.50	3	7.80	4.60	7.80		
	32.70	3	8.70	5.80	8.70		
Butyrolactone <i>N</i> -methyl-2-pyrrolidone Dimethyl formamide	28.20	3	6.90	4.80	6.90		
	34.80	5	6.00	4.10	5.00	4.10	6.00
	26.80	4	5.40	3.20	3.20	5.40	
Dioxane Cyclohexanone	37.00	3	10.50	7.40	10.50		
	35.60	4	8.20	4.80	4.80	8.20	

The calculated transforms for the various specimens dried at 35°, 60° and 110°C are shown in Figures 1, 2, and 3, respectively. The numbers in parentheses above the various peaks were obtained from the apparent thickness of the organic layers given in Tables 4, 5, and 6. For the purpose of these calculations it was assumed that the collapsed montmorillonite had a thickness of 9.6 Å.

The order in which the transforms have been placed follows the general trend with the simplest type of apparent layering and closest spaced layers and ending with the largest spaced and most numerous combinations. This order is general in nature since the trends at the various temperatures differed to some extent.

Attempts were made to correlate the type of transform obtained with some physical property of the organic solvent. It soon became apparent that no single physical property would correlate, thus combinations of

TABLE 6. THICKNESS OF LAYERS FROM FOURIER CURVES AT 110°C.

Solvent	1st order $d(\text{Å})$	No of layers	1st layer (Å)	2nd layer (Å)	3rd layer (Å)	4th layer (Å)	5th layer (Å)
Methyl isobutyl ketone Acetone	31.80	3	7.60	7.00	7.60		
	28.90	3	7.10	5.20	7.10		
Benzene 10% MeOH + 90% $C_6H_5CH_3$	30.50	3	7.00	6.80	7.00		
	32.00	3	8.60	5.20	8.60		
Butyrolactone <i>N</i> -methyl-2-pyrrolactone Dimethylformamide	28.80	4	4.70	4.90	4.90	4.70	
	34.20	3	9.50	5.60	9.50		
	35.30	5	6.90	3.40	5.20	3.40	6.90
Dioxane Cyclohexanone	34.20	3	9.50	5.60	9.50		
	35.80	4	7.80	5.30	5.30	7.80	

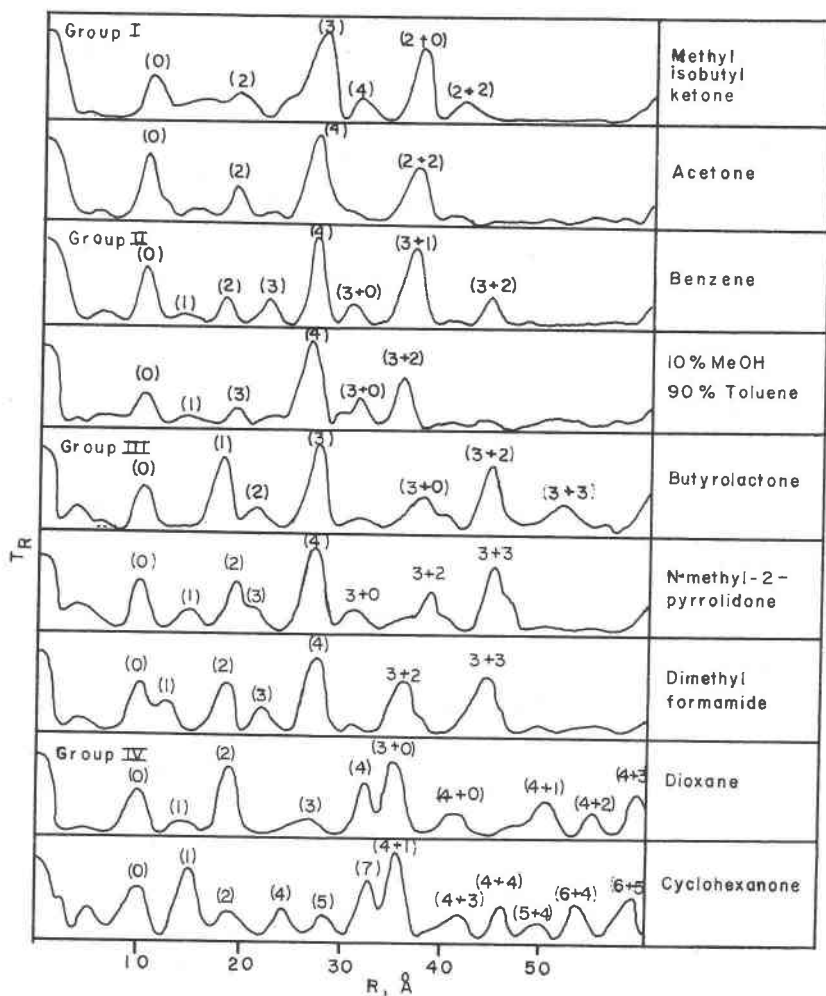


FIG. 1. Fourier transforms of the dispersed organo-montmorillonite at 35°C. Numbers in ( ) indicate number of apparent layers.

various factors were evaluated. It was also necessary to try and visualize the meaning of the transforms in terms of "apparent" number of organic layers and how this was related to the physical properties of the organic solvent.

The organo-montmorillonite used contained 105 meq. of organic cation per 100 g. of dry clay. This is sufficient to yield a four layer system as calculated from the size and amount of the cation (Jordan, 1949). It is

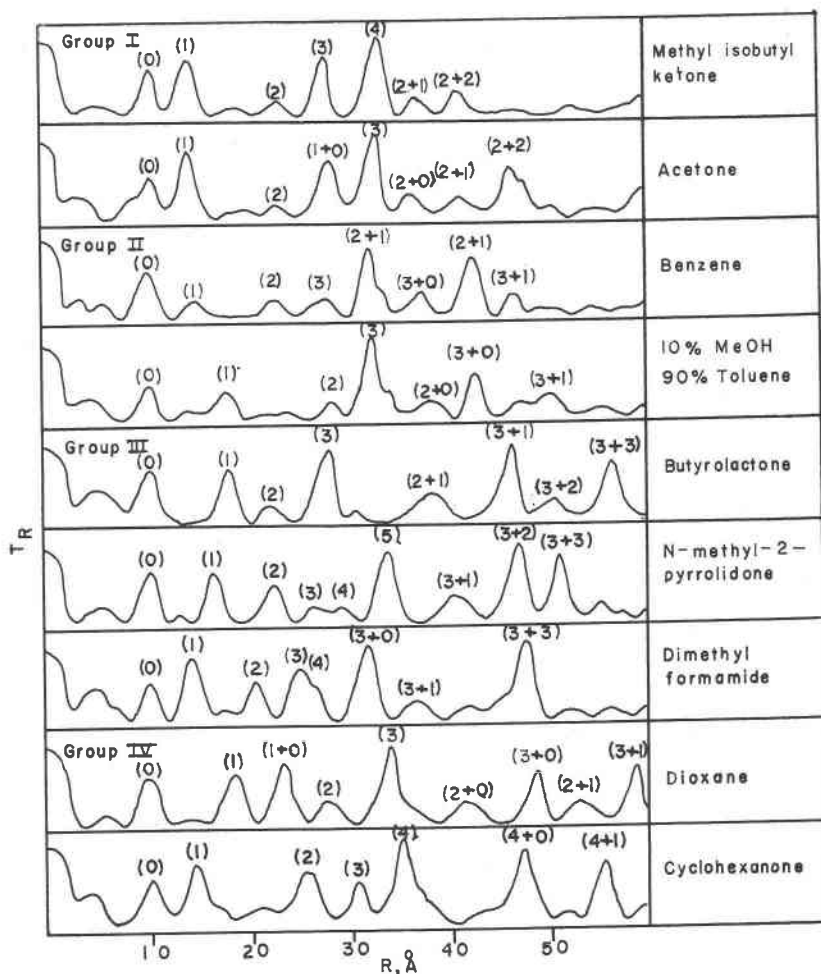


FIG. 2. Fourier transforms of the dispersed organo-montmorillonite at 60°C. Numbers in ( ) indicate number of apparent layers.

necessary, therefore, to take this fact into consideration in interpreting the Fourier transform curves.

For those cases where less than four organic layers are shown in the transforms (*e.g.*, 1, 2, or 3) this may indicate a packing of the organic to a close packed configuration. For an indicated number of layers greater than 4, it is interpreted to mean an open or loose packing of the organic chains. It is recognized, of course, that layers other than 4 can also mean clay plates containing less than or more than the average amount of



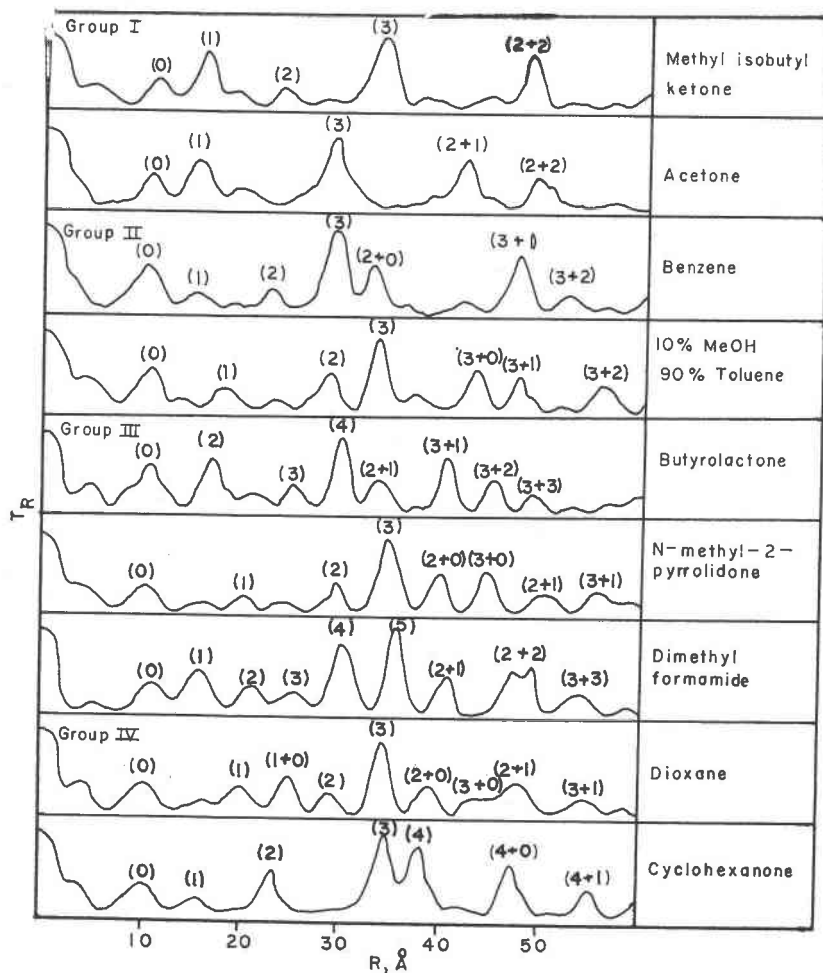


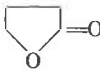
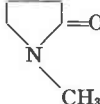
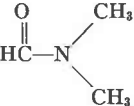
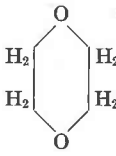
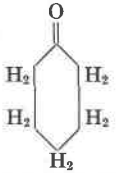


FIG. 3. Fourier transforms of the dispersed organo-montmorillonite at 110°C. Numbers in ( ) indicate numbers of apparent layers.

organic cation. However, it does not seem reasonable to assume this since the observed changes in the transforms would require a great deal of organic cation migration. Thus, from top to bottom of the Fourier transform curves shown in Figures 1, 2, and 3 the general trend is from a fairly close packed to a loose structure of the organic chains. Some of the curves at a given temperature appear out of place, but it is believed that the general trend is present.

Comparing the values found for the various properties in Table 7

TABLE 7. SELECTED PROPERTIES OF SOLVENTS

Solvent	$\epsilon$	$\gamma$	gel vol., cm <sup>3</sup>	b.p., °C	Amine solubility	Organo-clay penetration
<i>Group I</i>						
Methyl isobutyl ketone $\begin{array}{c} \text{O} \\ \parallel \\ \text{CH}_3\text{C}-\text{CH}_2\text{CH} \\ \quad \quad \quad \diagup \quad \diagdown \\ \quad \quad \quad \text{CH}_3 \quad \text{CH}_3 \end{array}$	13.1	25.4	0.9	118	vss	5 sec
Acetone $\begin{array}{c} \text{O} \\ \parallel \\ \text{CH}_3-\text{C}-\text{CH}_3 \end{array}$	21.5	22.0	0.6	56.5	vss	5 sec
<i>Group II</i>						
Benzene 	2.3	35.0	1.5	80.1	s	4 min
10% MeOH + 90% C <sub>6</sub> H <sub>5</sub> CH <sub>3</sub>  + CH <sub>3</sub> OH	5.4	29.2	1.9	108	s	4 min
<i>Group III</i>						
Butyrolactone 	39.0	42.0	0.58	206	ss	2 hrs
<i>N</i> -methyl-2-pyrrolidone 	1.2	41.2	0.9	202	ss	2 hrs
Dimethyl formamide 	16.6	35.2	0.6	152	ss	2 hrs
<i>Group IV</i>						
Dioxane 	2.2	36.9	0.6	101	fs	7 min
Cyclohexanone 	18.2	38.3	0.8	156	fs	1 min

$\epsilon$  dielectric constant at 20°C

$\gamma$  surface tension, dyne/cm

Penetration: time of complete penetration of the given solvent through a cake 5 mm thick of the organo-clay or montmorillonite

Solubility: vss, *very slightly soluble*; s, *soluble*; ss, *slightly soluble*; fs, *fairly soluble*.

with the Fourier transforms of Figures 1, 2, and 3 it will be noted that there is no one property that has the same general trend. In attempting to rationalize the meaning of the transforms the following types of interactions were considered: (1) organic amine with the solvent; (2) solvent with the clay surface, and (3) organic amine with the clay surface. Furthermore, since the specimens were dried films, the removal of the solvent from the organic amine and from the clay surface also must be considered.

Upon studying the transforms, it will be noted that they seem to fall into several general groups. That is, the samples dispersed in methyl isobutyl ketone and acetone in one group; benzene and toluene in a second group; butyrolactone, *n*-methyl-2-pyrrolidone and dimethyl formamide in a third group; and dioxane and cyclohexanone in a fourth group. It will be noted that, in general, the physical properties as given in Table 7 of a given group are similar. There are a number of anomalies such as the differences in boiling points within a group, but properties such as functional groups and structure, dielectric constant and solubility of the amine in the given solvent seem to be fairly consistent.

The general trends shown seem to be most closely related to the structure of the organic solvent being used. It will be noted that with the exception of dimethyl formamide the general change is from aliphatic to aromatic compounds and increasing from generally smaller to larger organic compounds. The significance of this may be in the drying mechanism. That is, the larger solvent molecules as they evaporate from the films leave a somewhat less ordered array or stacking within the clay plates. This would lead to the changes in the Fourier transforms noted. This trend does not appear to correspond in anyway to the boiling points of the solvents, therefore, probably is not related to residual solvent remaining in the specimens.

It is apparent that the solubility of the amine in the various solvents results in similar transform curves. This would be related to the interaction of the solvent molecules with the organic amine chains. This, of course, would also be related to the general structure of the solvent. Looking again to the various interactions that must be considered it is postulated that the trends in the Fourier transform curves for the four groups can be explained as follows:

1. Group I solvents lead to transforms showing the most regular curves since the organic amine chains are the least disturbed. Since the amine is only very slightly soluble in the solvent the interaction of the amine chains with the clay surface predominates. As the solvent evaporates the chains are attracted to the clay surfaces in a fairly orderly fashion, thus yielding transforms of uniform simple nature.

2. Group II solvents yield transforms showing a greater amount of

interlayering than do the Group I solvents because the amine molecules tend to dissolve into the solvent. On the other hand these solvents have low dielectric constants and probably do not interact with the clay surface to any extent (Farris, 1957). The net result is that the amine chains, which have van der Waals attraction for the clay surface, continue to become oriented parallel to the clay surface in a generally orderly manner. It will be noted also that the Group II solvents yield the largest values for the gel volumes, which also indicates the solvating effect between the solvent and amine. After evaporation of the solvent the amine chains probably reorient on the clay. Thus the transforms are not too dissimilar from the Group I solvents.

3. Group III solvents give transform curves with somewhat more interstratification of higher spacings than do the Group I and II solvents. The time of penetration of the solvent through the organo-clay as noted in Table I may be significant for this group. Note also that the organo-clay did not show any swelling in any of these solvents. Thus the slow penetration is not due to gel formation. The slow penetration apparently is due to just the right combination of factors that allows a very large amount of interaction between the solvent and the organo-clay, both with the amine molecules and with the clay surface itself. Upon evaporation of the solvent the amine-clay interactions apparently remain somewhat disrupted, leading to the Fourier transforms shown.

4. Generally large spacings shown by the Fourier transforms for Group IV solvents are probably a result of the relatively large size of these solvents. As the solvent molecules evaporate and push their way out from between the clay plates they apparently leave the amine chains in a generally disordered array; thus the larger spacings result in the transform curves.

5. As the various specimens are heated from 35°, to 60°, to 110°C, the Fourier transform curves show a general expansion of the organo-clay, probably due to the increased thermal motion for the amine chains. Even though this expansion takes place, the differences in the organo-layering caused by the different solvents continues in the same general way.

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