

# ACID DISSOLUTION OF CHLORITES: RELEASE OF MAGNESIUM, IRON AND ALUMINUM AND MODE OF ACID ATTACK\*

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**Abstract**—The acid dissolution of 8 chlorites in 2N Si-saturated HCl showed that there was no preferential dissolution of Al in octahedral positions and that the octahedral and tetrahedral sheets in the chlorite structure were equally attacked. An analysis of chemical data suggested a simple two-dimensional diffusion model in which the acid attack on the chlorite particles proceeded from the edges inward leaving a reacted layer which formed a diffusion barrier. Microscopic examination showed, however, that the acid attack was not simply an edge attack but started at any point where cracks, structural defects and weaknesses apparently predisposed sites to acid attack. It was also clear from the formation of etch figures and solution channels that the product layer at least partially dissolved or disintegrated during the progress of acid attack. The residue which remained after acid attack appeared to be an opaline amorphous hydrated silica as was indicated by index of refraction measurements. The orderly arrangement of etch figures on flakes of some chlorites reflected certain crystal symmetry elements. The results indicate that the acid dissolution technique to determine the ratio of Al in octahedral and tetrahedral positions can not be used as an aid in the calculation of structural formulae for chlorites.

## INTRODUCTION

IN STUDIES on the stability of chlorites in acid solutions (Brydon and Ross, 1966; Ross, 1967; Ross, 1968), it was found that a clinochlore from New York reacting with 2N HCl released Mg, Fe and Al at equal rates. According to the chemical data the reaction appeared to be diffusion controlled (Ross, 1967) but this was not confirmed by the combined data from X-ray and thermal analyses of the residues (Ross, 1968). These results were rather unexpected because one of the first studies on the acid dissolution of an orthochlorite by Brindley and Youell (1951) showed that the Al in octahedral positions dissolved at a faster rate than the Al in tetrahedral positions in the chlorite structure. Since then results of several acid dissolution studies on different phyllosilicates have been interpreted as indicating a faster dissolution rate of octahedral than of tetrahedral Al, (Cloos *et al.*, 1961; Gastuche *et al.*, 1960; Gastuche and Fripiat, 1962; Osthaus, 1954; Osthaus, 1956). Because these results suggested that octahedral and tetrahedral Al could be distinguished chemically by this method, the acid dissolution technique has been used as a tool to determine directly the ratio of octahedral to tetrahedral Al in the structure of phyllosilicates. In some acid dissolution studies the dissolution

rates were designated pseudo-first order (Cloos *et al.*, 1961; Miller, 1965; Osthaus, 1954; Osthaus, 1956) or pseudo zero order (Miller, 1965, 1968). In other studies the influence of particle shape on reaction rate was considered and the data appeared to fit a phase-boundary controlled reaction (Chaussidon and Valain, 1962; Gastuche *et al.*, 1960; Gastuche and Fripiat, 1962).

The purpose of this study was to (1) investigate whether different chlorites would dissolve in a similar manner as the clinochlore from New York or whether this clinochlore would be an exception in the dissolution behavior of chlorites, (2) compare the stability of different orthochlorites in acid, (3) examine the progress of acid attack on chlorite flakes under the microscope in an attempt to clarify the reaction mechanism.

## EXPERIMENTAL

The acid dissolution of eight chlorites was studied. Their chemical analyses are given in Table 1, as determined by X-ray spectrochemical analysis (Kodama *et al.*, 1967) except FeO which was determined by the method of Reichen and Fahey (1962). The required particle size fractions were obtained by filing the original rocks and passing the particles obtained through the appropriate sieves. The purity of the chlorites was checked by X-ray diffraction which indicated that chlorite

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Table 1. Chemical analyses of chlorites\*

	% based on oven-dry weight							
	1	2	3	4	5	6	7‡	8
SiO <sub>2</sub>	27.38	26.73	29.51	27.18	29.11	24.99	20.66	30.61
TiO <sub>2</sub>	0.00	0.03	0.03	0.10	0.97	0.95	n.d.§	0.74
Al <sub>2</sub> O <sub>3</sub>	20.32	20.78	15.90	21.02	21.22	21.46	21.57	18.95
Fe <sub>2</sub> O <sub>3</sub>	1.13	1.18	0.00	0.58	0.64	0.96	17.07	0.00
FeO	3.04	3.24	6.64	4.95	2.53	13.87	13.01	32.92
MgO	34.24	33.98	33.96	32.88	31.88	25.23	13.21	4.59
SrO	0.00	0.00	0.00	0.00	0.00	0.00	n.d.	0.00
CaO	0.03	0.10	0.13	0.03	1.04	0.08	n.d.	0.73
MnO	0.00	0.10	0.05	0.00	0.00	0.08	n.d.	0.86
K <sub>2</sub> O	0.03	0.03	0.03	0.03	0.80†	0.05	n.d.	0.04
Na <sub>2</sub> O	0.48	0.75	0.38	0.14	0.00	0.11	n.d.	0.07
H <sub>2</sub> O	13.34	13.11	13.38	13.08	11.80	12.24	13.71	10.43
	99.99	100.03	100.01	99.99	99.99	100.02	99.23	99.94

\* Particle size fraction 140–200 mesh.

† Allocated to phlogopite in the formula calculation

‡ Analysis according to methods of Jackson (1958). Sodium carbonate fusion of sample for determination of SiO<sub>2</sub> and HF-HClO<sub>4</sub> decomposition of sample for determination of Fe<sub>2</sub>O<sub>3</sub>, Al<sub>2</sub>O<sub>3</sub> and MgO. This sample contained about 18% magnetite which was removed using a magnet prior to this analysis.

§ Not determined.

7 contained an appreciable amount of pyrophyllite and also some magnetite. The pyrophyllite, which was present as relatively large flakes in the fine-grained chlorite rock, was removed under a binocular microscope and the magnetite was separated by a magnet. The structural formulae are shown in Table 2.

The acid dissolution techniques used in this study have been described in detail (Ross, 1967). The chlorites were treated with Si-saturated 2N HCl at 75°C for different lengths of time. The ratio of weight of chlorite to volume of acid was kept constant at 2:1 mg/ml (generally 200 mg; 100 ml). A few chlorites were treated also at 97°C and one at 30°C. The minerals were kept in suspension by a trickle of air and centrifuged immediately at the end of the reaction time. Mg, Fe and Al in the supernatant solutions were determined by atomic absorption spectrophotometry. The ratio of the amount of Mg, Fe and Al in the supernatant solutions to the total amount present in each untreated chlorite was designated as  $\alpha$ . In order to use the same analytical methods for the determination of the fractional and total amounts of Mg, Fe and Al, the total analyses for Mg, Fe and Al of the untreated chlorites were also carried out by HF-HClO<sub>4</sub> digestion (Jackson, 1958) followed by atomic absorption spectrophotometry.

Chlorite flakes, about 1.0 mm × 1.0 mm and 0.02 mm thick, or smaller, were cut from some of the flaky chlorite rock samples. The flakes were

treated with Si-saturated 2N HCl at 97°C for various lengths of time and then examined under the petrographic microscope.

## RESULTS

The  $\alpha$  values for Mg, Fe and Al followed a similar curve for each chlorite (Fig. 1). A comparison of the data in this Figure and in Table 1 indicates that except for chlorite 5, the dissolution rate increased with FeO + Fe<sub>2</sub>O<sub>3</sub> content. Because of the expected variation in the size of particles in the < 300 mesh size fraction, the dissolution data from this fraction were not suitable for further kinetic analysis. For this purpose the dissolution data from the 140–200 mesh (75–105  $\mu$ ) size fraction of three chlorites were used (Fig. 2). The  $\alpha$  values up to 0.6 for chlorites 1 and 8 and up to 0.8 for chlorite 4 followed the curve of the equation for a two-dimensional diffusion reaction (Holt *et al.*, 1962).

$$(1 - \alpha) \ln(1 - \alpha) + \alpha = \frac{(k)}{r^2} t$$

where  $k$  is the rate constant,  $r$  is the initial particle radius, and  $t$  is the reaction time. Although this equation is more soundly based than the equation given by Jander which was used in previous kinetic studies (Ross, 1967; Ross, 1968), the data appeared to fit both equations equally well. Sharp *et al.* (1966) also have pointed out the

Table 2. Structural formulae, classification and source of chlorites

No.	Formula*	Name Hey's Classification†	Source and locality
1	$(Al_{0.94}Fe^{+3}_{0.06}Fe^{+2}_{0.25}Mg_{4.90})(Si_{2.63}Al_{1.37})O_{10}(OH)_8$	Sheridanite	Wards – Brinton's Quarry, Westchester, Pennsylvania, U.S.A., Labelled Clinchlore.
2	$(Al_{0.98}Fe^{+3}_{0.02}Fe^{+2}_{0.26}Mg_{4.89})(Si_{2.58}Al_{1.42})O_{10}(OH)_8$	Sheridanite	British Museum – Westchester, Pennsylv. U.S.A., BM 26647 Labelled Clinchlore‡
3	$(Al_{0.71}Fe^{+2}_{0.54}Mg_{4.94})(Si_{2.88}Al_{1.12})O_{10}(OH)_8$	Clinchlore var. Leuchtenbergite	British Museum – Fermatt, Valais, Switzerland, BM 42911 Labelled Pennine‡
4	$(Al_{1.00}Fe^{+3}_{0.04}Fe^{+2}_{0.38}Mg_{4.72})(Si_{2.62}Al_{1.38})O_{10}(OH)_8$	Sheridanite	Eckert – Tilly Foster Mine, Brewster, New York, U.S.A. Labelled Clinchlore.
5	$(Al_{1.18}Fe^{+3}_{0.05}Fe^{+2}_{0.22}Mg_{4.59})(Si_{2.69}Al_{1.31})O_{10}(OH)_8$	Sheridanite	Min. Unlim.§* – Singatse Range, Yerington, Lyon Co., Nevada, U.S.A. Labelled Clinchlore, var. Leuchtenbergite.
6	$(Al_{1.00}Fe^{+3}_{0.06}Fe^{+2}_{1.18}Mg_{3.80})(Si_{2.53}Al_{1.47})O_{10}(OH)_8$	Ripidolite	Min. Unlim.§* – Chester, Vermont, U.S.A. Labelled Prochlorite.
7	$(Al_{0.99}Fe^{+3}_{1.39}Fe^{+2}_{1.18}Mg_{2.13})(Si_{2.24}Al_{1.76})O_{10}(OH)_8$	Pseudothuringite	Wards – Chester, Massachusetts, U.S.A. Labelled Corundophyllite.
8	$(Al_{1.73}Fe^{+2}_{2.88}Mg_{0.74})(Si_{0.32}Al_{0.68})O_{10}(OH)_8$	Diabantite	Wards – Donna Ana Co., New Mexico, U.S.A. Labelled Thuringite.

\*Formula calculated according to the method described by Foster (1962) and based on theoretical  $H_2O$  (+).

†The same names apply according to Foster's (1962) classification except for chlorites 6, 7 and 8 which are designated sheridanite, ripidolite and chamosite, respectively, in Foster's classification.

‡Obtained through the courtesy of the British Museum. Specimen BM 26647 is the same chlorite as used by Brindley and Youell (1951) but was mistakenly referred to as being a Pennine from Binnenthal, Switzerland.

§\* Minerals unlimited

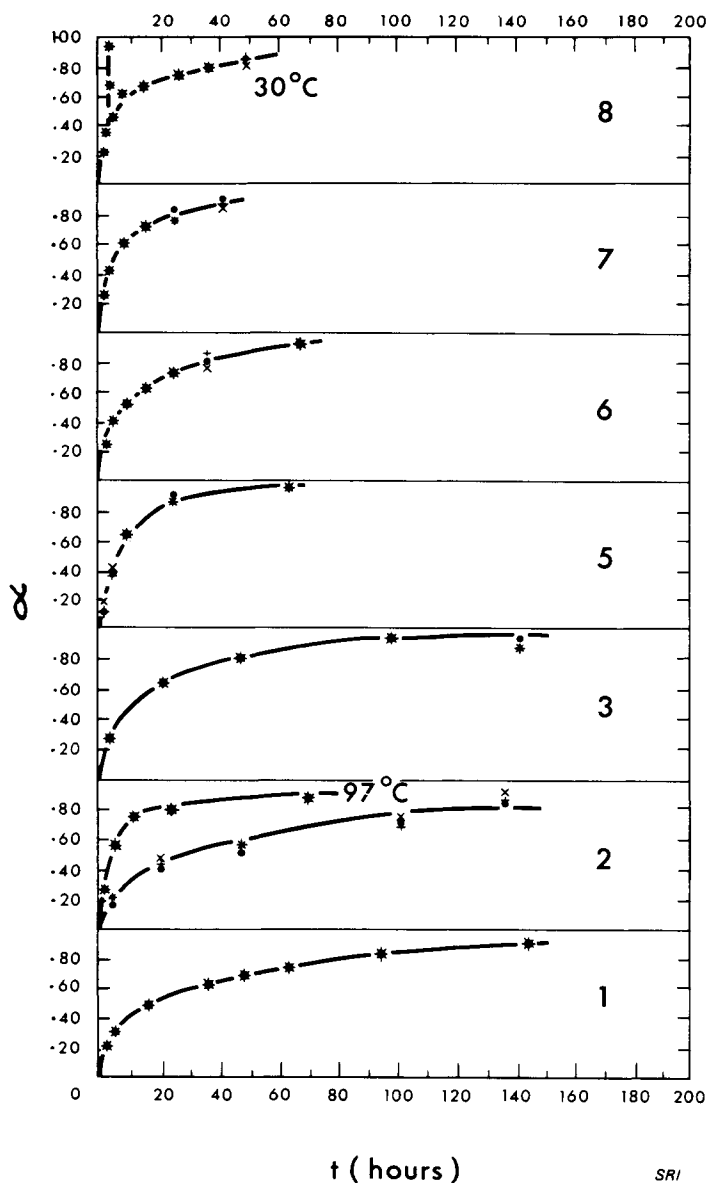
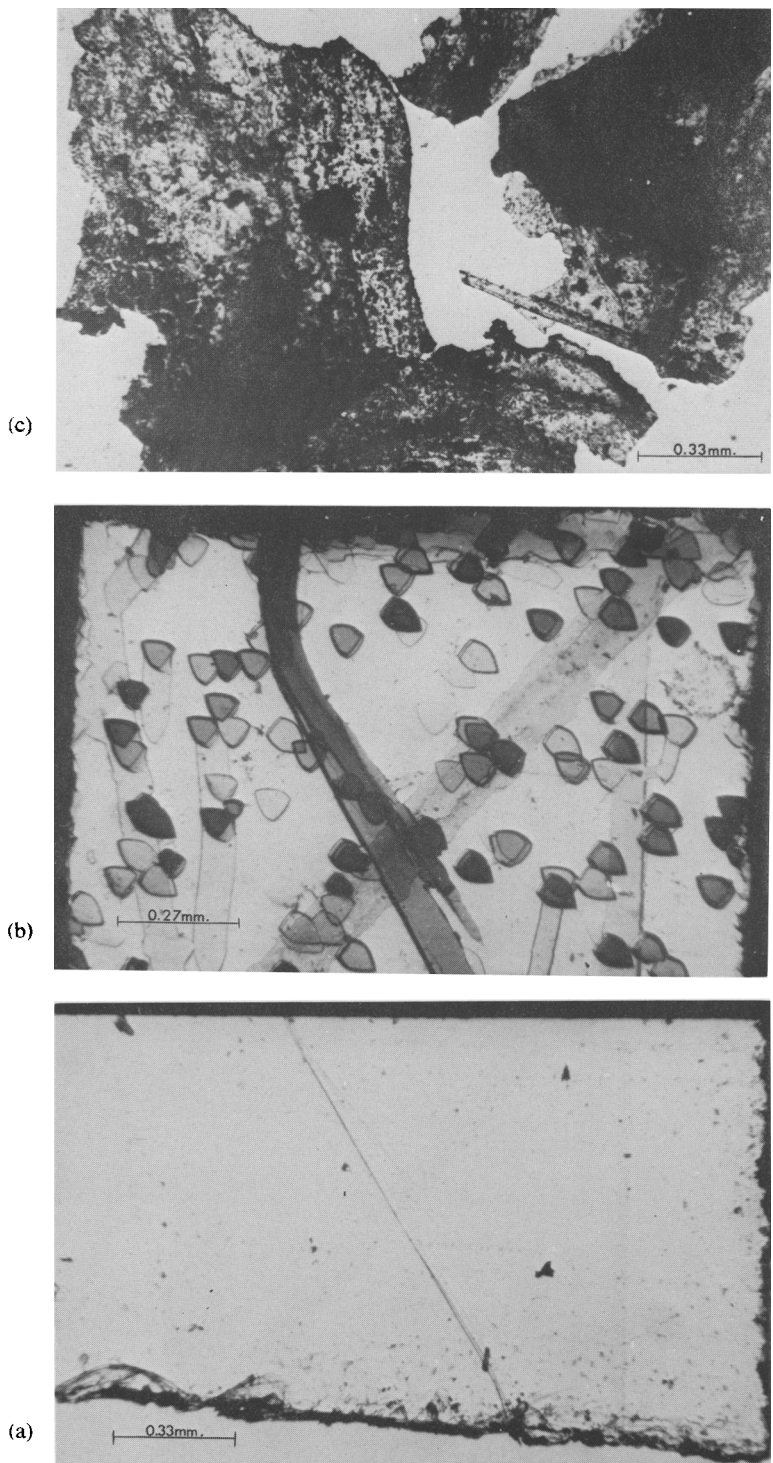


Fig. 1. Fraction dissolved ( $\alpha$ ) vs.  $t$  of Mg (\*), Fe (x), and Al (+) of the < 300 mesh fraction of chlorites reacting with 2N Si-saturated HCl. The symbol (\*) is due to superposition of the symbols for Mg, Fe and Al. Reaction temperature 75°C except where indicated otherwise. Size fraction of chlorite 2 at 97°C was < 100 mesh.

lack of differentiation between these types of equations for  $\alpha$  values below 0.7.

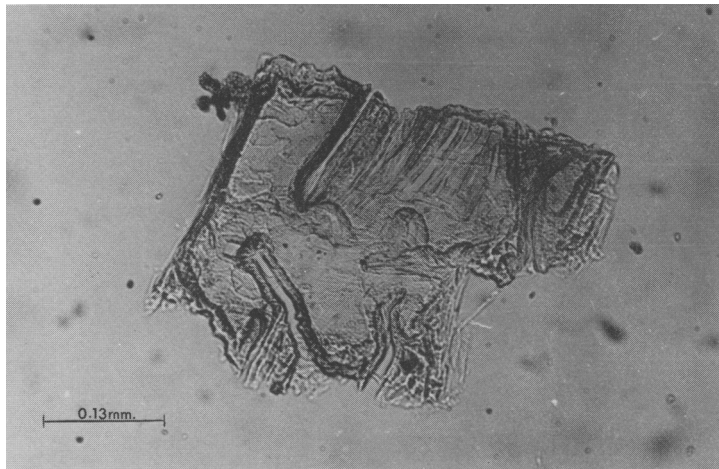
The effects of progressive acid attack on a single flake of chlorite 1 are illustrated in Fig. 3a, b and c. This chlorite is monoclinic, as determined from X-ray powder data, biaxial (+) with indices of refraction  $\alpha = \beta = 1.588$ ,  $\gamma = 1.592$  as determined by microscopic examination. The fresh

flake in Fig. 3a shows a relatively smooth edge, one well-defined crack and some diffuse fractures and numerous small specks of unknown origin. Acid treatment of this flake produced many triangular etch figures aligned diagonally across the flake (Fig. 3b). Microscopic examination showed that the etch figures are aligned parallel to the crystallographic  $X$ -axis and that the figures

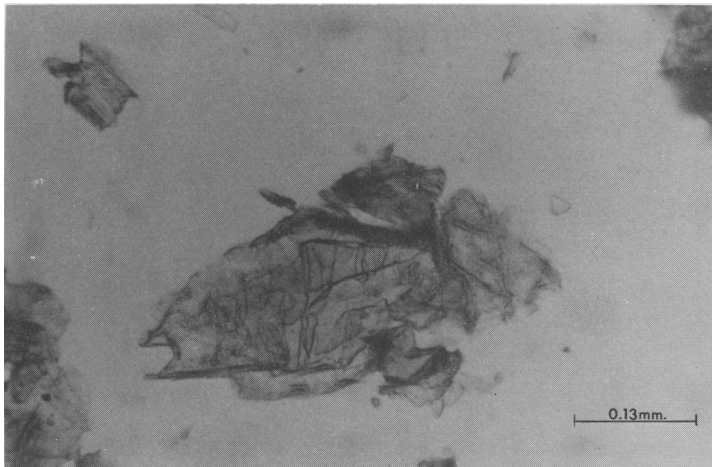


**Fig. 3.** Progress of acid attack on part of the same flake of chlorite 1. Treated with 2N Si-saturated HCl at 97°C. (a) Untreated. Indices of refraction  $\alpha = \beta = 1.588$ ,  $\gamma = 1.592$  (ordinary light); (b) Treated 14 days. Crossed nicols. Solution channels and triangular etch figures were discernible after 2 days reaction; (c) Treated 30 days. Isotropic fragments with index of refraction = 1.410 (Ordinary light).

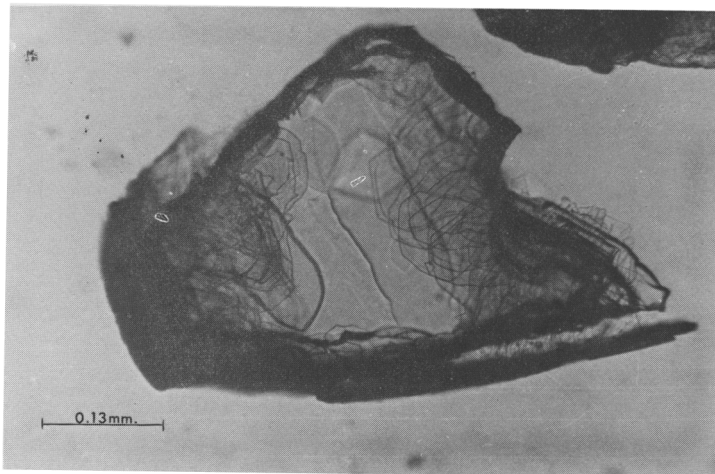
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**Fig. 4.** Flake of chlorite 3 treated 4 days. Solution channels producing ragged edges and eventually fragmentation (ordinary light).



(a)



(b)

**Fig. 5.** Flakes of chlorite 4 treated 6 days. Solution channels and hexagonal etch figures (ordinary light).

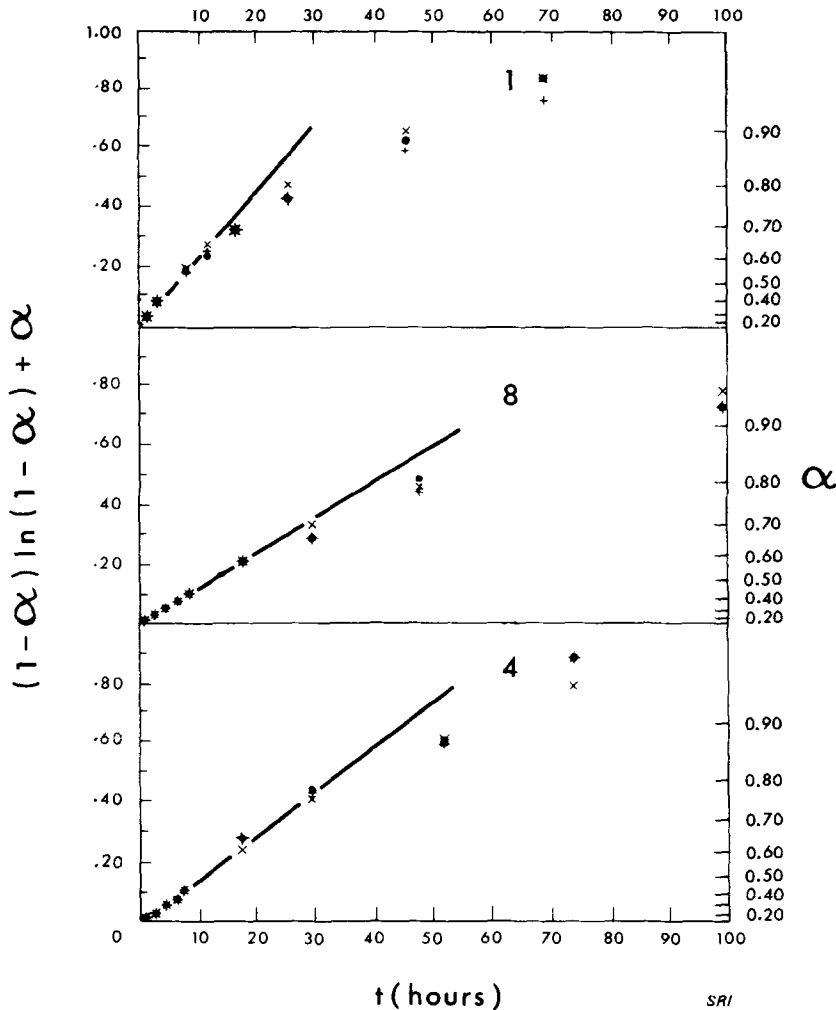


Fig. 2. Plots of  $\alpha$  and of  $(1-\alpha) \ln(1-\alpha) + \alpha$  vs.  $t$  of Mg (\*), Fe (x) and Al (+) of the 140–200 mesh fraction of chlorites reacting with 2N Si-saturated HCl. Reaction temperature 97°C for chlorites 1 and 4 and 30°C for chlorite 8.

oriented in one direction originated at the upper basal surface and those oriented in the opposite direction originated at the lower basal surface of the flake. Solution channels are prominent along cracks and fractures initially present in the untreated flake. Dissolution at the edges is also apparent especially along the top edge of the flake. In Fig. 3c fragments of the flake are shown which remained after acid attack. No etch figures are visible, and the fragments are isotropic with an index of refraction of 1.41 which is close to that of opal (Hydrophane)(Frondele, 1962). The features observed on the acid-treated flakes, as described above, could also be seen on acid-treated particles of the 140–200 mesh fraction of chlorite 1

although etch figures were smaller and less easily distinguished. Acid-treated flakes of chlorite 2 showed the same etch figures and solution channels as those described for chlorite 1.

As shown in Fig. 4, the dominant effects resulting from acid attack on flakes of chlorite 3 are solution channels which progressed from the edges inward resulting in very ragged edges and eventually in fragmentation of the flakes. Most acid-treated flakes of chlorite 4 appeared similar to the flake in Fig. 5a showing solution channels, but some flakes of this chlorite also showed etch figures (Fig. 5b). These etch figures are hexagonally shaped and appear to be less regular than the triangular etch figures observed in flakes of chlor-

ites 1 and 2. The solution channels in Fig. 5*b* also appear to progress from the edges inward although a few seem to originate at etch figures. Many of these solution channels overlap each other indicating that they are present at different basal cleavage planes within the flake.

#### DISCUSSION

The dissolution curves of the eight chlorites which ranged in composition from high Mg to high Fe content all had similar shapes. They apparently dissolved in the same manner as the clinocllore from New York (chlorite 4) which was studied previously (Ross, 1967; Ross, 1968). The data also indicated that the rate of dissolution increased with increasing Fe and decreasing Mg content.

As was found previously for chlorite 4 (Ross, 1967), Mg, Fe and Al were dissolved at the same rate from each of the different chlorites investigated in this study. In these chlorites, therefore, the acid dissolution technique did not distinguish octahedral and tetrahedral Al as had been shown by Brindley and Youell (1951). Although their experiment was repeated with a specimen of the same chlorite and under the experimental conditions as described by them, there was no evidence that Al was released at a different rate from Mg and Fe (Fig. 1, chlorite 2 at 97°C). No explanation can be offered for this difference in results except some unrecognized significant difference in specimen or experimental conditions. Considering specimen purity, it was found that the presence of some impurities may result in a different dissolution curve for Al than for Mg and Fe. Such results were obtained in preliminary experiments on the dissolution of chlorite 7. X-ray diffraction patterns of the residues at the various reaction times showed an increasing proportion of pyrophyllite. A pyrophyllite-free sample of this chlorite, however, gave the same rate curve for Al as for Mg and Fe (Fig. 1, chlorite 7). The results of this study, therefore, seem to justify the generalization that the acid dissolution technique does not distinguish between octahedral and tetrahedral Al of chlorites.

None of the residues from any of the acid treatments showed evidence of hydrated layer vermiculite-like or montmorillonite-like products. Thermal data obtained previously (Ross, 1968) and microscopic analysis of the residues indicated that the reaction product was an opaline amorphous hydrated silica. Consequently, there was no evidence of a preferential removal of the hydroxide sheet. In fact, all of the data and observations indicated that the octahedral and tetrahedral sheets of the chlorite layer were equally attacked.

On the basis of the chemical dissolution data alone, the mode of acid attack could be interpreted as following a model of a disk into which the reaction proceeds from the edge inward leaving an intact product layer which forms the diffusion barrier (Fig. 2; see also Ross, 1967). However, observation on chlorite flakes under the microscope showed that the acid attack was not simply an edge attack but started at any point where cracks, structural defects and weaknesses apparently predisposed sites to acid attack. It was also clear from the formation of etch figures and solution channels observed in this study as well as from thermal data obtained previously (Ross, 1968) that the product layer did not remain intact but at least partially dissolved or disintegrated during the progress of acid attack. Thus despite the fit of the chemical dissolution data to a simple two-dimensional diffusion model, the additional data and observations preclude any definite conclusions concerning the mechanism controlling the rate of reaction. These results show that, for the reactions encountered in this study, the fit of the data to a model up to 60 or 80 per cent of the reaction was not sufficient to establish the validity of the model. In regard to this Jacobs and Tompkins (1955) have pointed out that agreement between experimental results and rate equations, expressing  $\alpha$  as a function of  $t$ , deduced theoretically does not necessarily establish the validity of the model from which these equations are derived and that information about the reaction in addition to the  $\alpha$  versus  $t$  curve may sometimes be required. Carter (1961) has elaborated the precautions necessary to warrant any conclusions as to the validity of a model used in the analysis of data expressing  $\alpha$  as a function of  $t$ .

The arrangement of the etch figures on some chlorites appeared to reflect certain crystal symmetry elements. Thus the etch figures observed in chlorites 1 and 2 were always aligned parallel to the crystallographic  $X$ -axis. Also, the microscopic observation that etch figures originating at opposite basal surfaces were oriented in opposite directions may be interpreted as evidence for the centro-symmetry of this chlorite (Friedel, 1926). Finally, the difference in shape of the figures in chlorite 1 from those in chlorite 4 (Figs. 3*b* and 5*b*) indicates that these chlorites are different polymorphs.

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**Résumé**—La dissolution acide de 8 chlorites dans 2N Si-saturé HCl a montré qu'il n'y avait pas de dissolution préférentielle de Al dans les positions octaédriques et que les plaques octaédriques et tétraédriques dans la structure de chlorite étaient également attaquées. Une analyse des données chimiques a permis de suggérer un simple modèle de diffusion tridimensionnelle dans lequel l'attaque de l'acide sur les particules de chlorite procédait des bords vers l'intérieur, laissant une couche ayant subi la réaction et formant une barrière de diffusion. L'examen microscopique a montré, toutefois, que l'attaque de l'acide n'était pas seulement une attaque au bord mais commençait en n'importe quel point où des craques, des défauts de structure ou des faiblesses prédisposaient apparemment les zones à l'attaque de l'acide. D'après la formation des figures gravées et des canaux de solution, il était aussi clair que la couche de produit se dissolvait ou se désintérait au moins partiellement au cours de l'attaque de l'acide. Le résidu qui restait après l'attaque acide était une silice opaline hydratée amorphe comme l'indiquait l'index des mesures de réfraction. L'arrangement régule des figures gravées sur les éclats de quelques chlorites reflétait certains éléments symétriques de cristaux. Les résultats indiquent que la technique de dissolution à l'acide pour déterminer le taux de Al dans les positions octaédriques et tétraédriques ne peut être utilisé pour venir en aide au calcul des formules structurelles pour les chlorites.

**Kurzreferat**—Die saure Lösung von 8 Chloriten in 2N Si-gesättigter HCl zeigte, dass eine bevorzugte Lösung von Al in oktaedrischen Stellungen nicht stattfand, und dass die oktaedrischen und tetraedrischen Schichten im Chloritgefüge gleich stark angegriffen wurden. Eine Analyse der chemischen Messwerte deutete auf ein einfache zweidimensionales Diffusionsmodell hin, in welchem der Säureangriff auf die Chloritteilchen von den Rändern aus einwärts erfolgte, unter Zurücklassung einer ausreagierten Schicht, die eine Diffusionsbarriere bildete. Mittels mikroskopischer Prüfung konnte aber gezeigt werden, dass der Säureangriff nicht bloss ein Randangriff war, sondern dass scheinbar Risse, Gefügefehler und sonstige schwache Stellen als bevorzugte Punkte für den Säureangriff

angesehen werden müssen. Ferner ging klar aus Ätzfiguren und Lösungskanälen hervor, dass im Laufe des Säureangriffs die Produktschicht sich zum mindesten teilweise löste und zerfiel. Der nach dem Säureangriff zurückbleibende Rückstand erschien, wie durch Messungen des Brechungsindex gezeigt werden konnte, als eine opalartige, amorphe, hydratisierte Silika. Die regelmässige Anordnung der Ätzfiguren auf den Blättchen gewisser Chlorite deutet auf Kristallsymmetrieelemente hin. Die Ergebnisse zeigen an, dass die Methode der Auflösung in Säure zur Bestimmung des Verhältnisses von Al in oktaedrischen und tetraedrischen Stellungen als Hilfsmittel bei der Berechnung von Strukturformeln von Chloriten ungeeignet ist.

**Резюме**—Изучение процесса кислотного растворения 8-ми хлоритов в 2N Si-насыщенной HCl показало, что при растворении отсутствует преимущественное вымывание Al из октаэдрических положений и что октаэдрические и тетраэдрические сетки хлоритовой структуры в равной степени подвергаются действию кислоты. Анализ химических данных позволил предположить простую двумерную диффузионную модель, при которой действие кислоты на хлорит начинается с краев частиц и в дальнейшем распространяется внутрь, что приводит к образованию поверхностного реакционного слоя, представляющего собой диффузионный барьер.

Однако, исследования под микроскопом показали, что кислота неравномерно воздействует на края частиц; действие кислоты начинается преимущественно с тех точек, где имеют место трещинки, структурные дефекты и другие нарушения, которые создают благоприятные условия для кислотного воздействия. Из анализа образующихся фигур травления и каналов растворения совершенно ясно следует также, что реакционный слой, по крайней мере частично, растворяется или разрушается под действием кислоты. Остаток, который получается после обработки кислотой, представляет собой, как было установлено измерением показателя преломления, опаловый аморфный гидратированный кремнезем. Упорядоченное расположение фигур травления на чешуйках некоторых хлоритов отражает определенные элементы симметрии кристаллов. Полученные результаты указывают на то, что метод кислотного травления, применяемый с целью определения относительного количества Al в октаэдрических и тетраэдрических положениях, не может быть использован для вычисления структурных формул хлоритов.