

## CRYSTAL CHEMISTRY OF BOEHMITE

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**Abstract**—Thirty two boehmites, synthesized at temperatures ranging from room temperature to 300°C, were examined by scanning electron microscopy, transmission electron microscopy, electron diffraction, X-ray powder diffraction, differential thermal analysis, and infrared spectroscopy. The results show that boehmite exhibits a continuous gradation in crystallite size ranging from single octahedral layers or a few unit cells to about 65 unit cells in the y-direction. This conclusion suggests that the term pseudoboehmite is inappropriate for finely crystalline boehmite. Finely crystalline boehmite contains more sorbed water than coarsely crystalline boehmite; this water is commonly intercalated between octahedral layers, usually randomly but sometimes regularly. The regularly interstratified boehmite gives rise to a diffuse "long spacing" X-ray diffraction reflection. Calculated 020 X-ray diffraction peaks approximate closely those observed experimentally when a range of crystallite sizes is taken into account.

**Key Words**—Boehmite, Crystallite size, Pseudoboehmite, Synthesis, X-ray powder diffraction.

### INTRODUCTION

The primary purpose of this study was to ascertain the level of agreement that could be attained between experimental and calculated X-ray powder diffraction profiles for boehmite. Boehmite exhibits an extreme variation in crystallite size (Papée *et al.*, 1958; Hsu, 1967). Further, it can be synthesized from its elementary constituents so that its chemistry can be controlled. The substances that have been produced and studied here are probably more chemically pure and exhibit a wider range of crystallite sizes than any previously studied. X-ray powder diffraction data were supplemented by electron diffraction and microscopy, differential thermal analysis, and infrared spectroscopy in order to characterize fully synthetic boehmites and to discern the property variations among them.

Boehmite synthesized at low temperatures exhibits broad diffraction peaks, contains more water, and has a higher surface area than boehmite synthesized at high temperatures (Calvet *et al.*, 1953; Papée *et al.*, 1958). These differences prompted Calvet *et al.* (1953) to coin the term *pseudoboehmite* for this substance. The nature of pseudoboehmite, however, is not clear. Various interpretations of its structure and chemistry and aspects of its diffraction patterns noted by previous investigators must be explained before pseudoboehmite is understood. Papée *et al.* (1958) suggested that pseudoboehmite was not crystalline but had an atomic arrangement similar to boehmite with a short range order whose character is intramolecular or intramicellar. They further suggested that the broad diffraction peaks resulted from a poorly ordered arrangement of elementary sheet-like units. Hsu (1967) suggested that pseu-

doehmite was an incompletely dehydrated boehmite. Lahodny-Šarc *et al.* (1978) stated that the material they made at temperatures as high as 80°C was pseudoamorphous and not crystalline boehmite. Papée *et al.* (1958) noted that the intensities of the diffuse X-ray powder diffraction reflections did not decrease significantly at high angles, a feature common for gels. They noted that these high angle reflections were somewhat sharper than the low angle reflections. They also observed that the first reflection at the lowest diffraction angle, corresponding to the 020 reflection of boehmite, was absent from some patterns although the other reflections remained unchanged. The latter observation was particularly evident for synthetic samples that had been incompletely washed of salts. The d-spacing of the first pseudoboehmite peak is much larger than that of boehmite and has been attributed to excess water in interlayer positions, corresponding to interlayer water in clay minerals.

The present findings explain many of the observations made in previous studies and reconcile some of the conflicting interpretations. Also, a new feature of boehmites with small crystallite size has been discovered. The major conclusion is that differences in the X-ray diffraction patterns of boehmites can be explained mainly by variations in crystallite size and, therefore, any distinction between boehmite and pseudoboehmite is arbitrary.

### EXPERIMENTAL

#### *Synthesis procedure*

Boehmite was synthesized by the method of Hsu (1967) by adding 100 ml of a 0.6 N NaOH-4 N NaCl solution dropwise into a 250-ml beaker containing 100 ml of 0.2 M AlCl<sub>3</sub> over a period of 30 min. The solution was stirred constantly with a magnetic stirrer with the

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burette tip positioned close to the outer edge of the beaker to obtain maximum dispersion. To remove residual NaCl, the gelatinous precipitate was diluted immediately with 160 ml of distilled-deionized water, shaken vigorously, and centrifuged. The supernatant was decanted, and the process was repeated until the washings were chloride free as determined with a 10% silver nitrate solution. In some of the samples the first dilution was made with a 1:1 mixture of water and methanol. The alcohol wash caused no observable differences in the products.

Crystal growth was accomplished by heating the washed, gelatinous precipitates in sealed containers in ovens or furnaces preheated to the temperature desired. Polyethylene bottles were used up to 80°C, and hydrothermal pressure vessels were used above 80°C. Thirty-two samples were synthesized between room temperature and 300°C in ~25°C increments (Table 1). Sixteen samples were heated for 1 day; 15 samples were heated for 2, 4, 6, or 8 days; 1 sample was dried immediately after precipitation. Following heat treatment, the reaction vessels were quenched in water, and the gels were removed. Thirteen samples were freeze-dried; 19 samples were oven-dried at 125°C. After drying, all samples were ground for 15 min and stored in stoppered vials in a desiccator. The freeze-dried samples were ground mechanically; the oven-dried samples were ground by hand.

#### Electron microscopy and electron diffraction

Undisturbed subsamples were examined with a Cambridge S4-10 scanning electron microscope to ascertain their morphology. An undisturbed portion of sample 32 was examined with a JEM6A transmission electron microscope to record an electron diffraction pattern to determine the relationship between crystallographic axes and crystal habit.

#### X-ray powder diffraction

X-ray powder diffraction (XRD) patterns with filtered Cu radiation were run on all samples. Experimental conditions are given by Christoph *et al.* (1979). The breadth of the 020 reflection of each pattern was measured at half-maximum intensity after subtracting the background; the  $2\theta$  position of each 020 reflection was measured at the midpoint of the chord at half-maximum intensity. The 100 reflection of powdered quartz was used as a standard for  $2\theta$  and as a measure of the instrumental broadening. The breadth ( $b$ ) of the 100 reflection of quartz was subtracted from the measured breadth of the 020 reflection ( $B$ ) of boehmite to give the pure diffraction breadth ( $\beta$ ), i.e.,  $\beta = B - b$ . The corrected  $2\theta$  and  $\beta$  values are given in Table 1.

#### Differential thermal analysis

A Dupont Model 900 DTA system was used to record the differential thermal analysis (DTA) patterns of 7

Table 1. Experimental data for synthetic boehmites.

Sample no.	Temp. (°C)	Duration (days)	Method of drying	020 reflection	
				Position (°2 $\theta$ )	Breadth (°2 $\theta$ )
1	20	0	O	12.90	5.45
2	20	1	F	12.35	5.66
3	20	1	O	10.80	5.73
4	20	2	O	13.86	3.85
5	20	4	O	13.73	3.81
6	20	8	O	13.70	3.75
7	43	1	F	13.30	3.67
8	60	1	F	12.56	5.66
9	68	1	O	13.80	3.05
10	68	2	O	13.66	3.93
11	68	4	O	13.75	3.65
12	68	8	O	13.84	3.41
13	80	1	F	13.47	3.54
14	100	1	F	12.71	5.55
15	100	4	F	13.54	4.18
16	125	1	F	13.96	2.46
17	125	1	O	14.12	2.45
18	125	2	O	14.30	2.05
19	125	4	O	14.40	1.39
20	125	8	O	14.46	1.31
21	150	1	F	14.32	1.20
22	175	1	F	14.40	0.70
23	200	1	F	14.39	0.50
24	200	1	O	14.48	0.77
25	200	2	O	14.49	0.88
26	200	4	O	14.51	0.48
27	200	8	O	14.46	0.62
28	250	1	F	14.45	0.27
29	300	1	F	14.46	0.05
30	300	1	O	14.52	0.10
31	300	2	O	14.53	0.10
32	300	6	F	—	—

F = Freeze-dried; O = Oven dried at 125°C.

020 breadth measured at half-maximum intensity and corrected for instrumental broadening; 020 position measured at midpoint of chord at half-maximum intensity.

samples. About 50 mg of sample and an equal amount of alpha alumina reference material was packed into glass tubes. Chromel-alumel thermocouples were used, and each sample was heated in a nitrogen atmosphere which was admitted into the heating chamber at a rate of 3 ft<sup>3</sup>/hr. The heating rate was 10°C/min, and the  $\Delta T$  sensitivity was constant for all runs.

#### Infrared spectroscopy

A Digilab Model FTS-14 spectrometer was used to record the infrared (IR) spectra of samples 7, 22, and 29. Sample 7 was dried at 200°C; samples 22 and 29 were dried at 65°C after which each was mixed with KBr and pressed into a disk.

## RESULTS

#### Electron microscopy and electron diffraction results

Samples 7, 14, 21, and 32 were examined by scanning electron microscopy. Crystals were seen only in sample

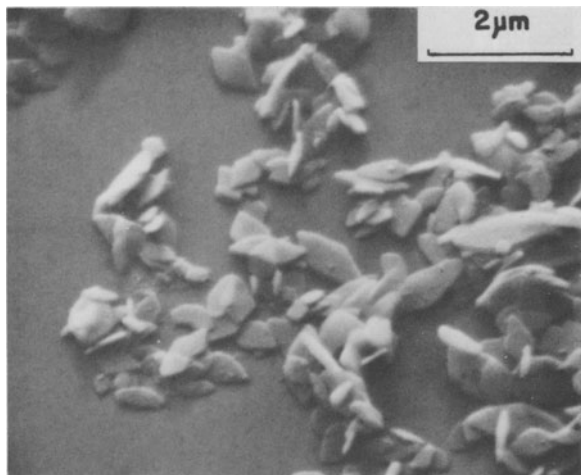


Figure 1. Scanning electron micrograph of boehmite sample 32.

32. Most of them showed a bladed habit, elongate along  $z$ , and very thin along  $y$ . A separate portion of sample 32 was not freeze-dried but diluted manifold, air-dried, and photographed. The crystals in this sample (Figure 1) showed a different habit, similar to the rhombic, tabular habit of boehmite described by Mackenzie *et al.* (1971). The outline of most crystals is dominated by (201), whereas those shown by Mackenzie *et al.* (1971) are bounded by (101). Thus, the morphology of boehmite depends upon its method of preparation, although all crystals are thin, parallel to  $y$ .

An electron diffraction (ED) pattern (Figure 2, top) was obtained from a typical crystal (Figure 2, inset) of sample 32. The powder rings are due to metallic aluminum evaporated onto the sample for reference. The pattern shows 9 unique reflections which are indexed in the space group  $Amam$  (Figure 2, bottom). Cell dimensions determined from the pattern ( $a = 3.679 \text{ \AA}$ ,  $c = 2.799 \text{ \AA}$ ) are smaller than those from the X-ray diffraction results of Christoph *et al.* (1979) which are  $a = 3.6936 \text{ \AA}$ ,  $c = 2.8679 \text{ \AA}$ . The presence of  $hkl$  reflections suggests diffusion of intensity parallel to  $b^*$  owing to small crystal size and/or structural disorder. Comparable ED patterns of boehmite shown in Mackenzie *et al.* (1971) and Bosmans and Michel (1957) do not show  $hkl$  reflections. An ED pattern of synthetic lepidocrocite,  $FeO(OH)$ , which has a structure similar to or identical with boehmite, given by Mackenzie *et al.* (1971), shows 27 unique reflections, many of which are indexed as  $hkl$ .

#### X-ray powder diffraction results

XRD patterns showed no alumina hydrate other than boehmite. A series of patterns is shown in Figure 3 with crystallite size increasing from top to bottom. An indexed pattern recorded for sample 32 was shown in

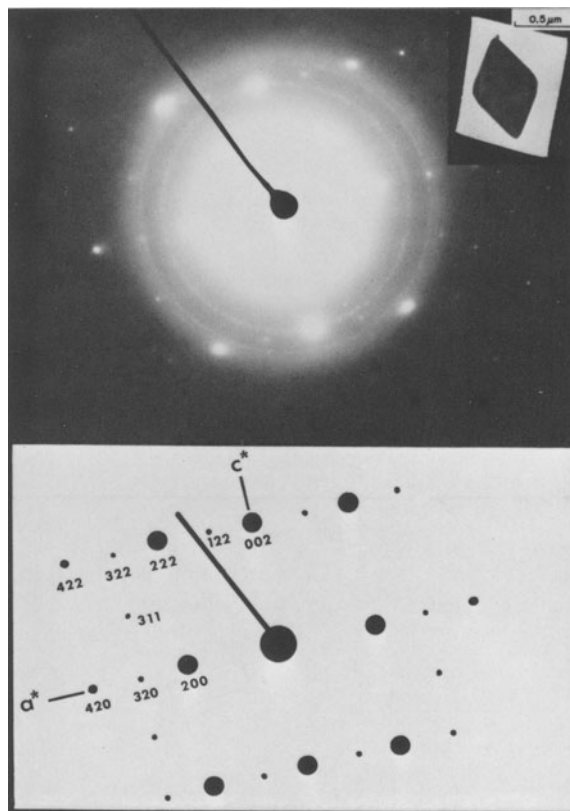


Figure 2. Electron diffraction (ED) pattern (top) obtained from a single crystal (inset) from sample 32. Rings are from metallic aluminum evaporated onto the crystal for reference. Schematic ED pattern (bottom) indexed in the space group  $Amam$ .

Christoph *et al.* (1979). The dashed vertical line at  $14.5^\circ 2\theta$  represents the position of the 020 reflection for boehmites synthesized at  $300^\circ\text{C}$ . An increased breadth of this reflection was accompanied by a shift of the peak position to small  $2\theta$  values, i.e., as crystallite size decreases along  $y$  the apparent  $d(020)$  increases. The 020 peak displacement as a function of the number of octahedral layers is due to the variation of  $F^2 \cdot L_p$  with  $2\theta$ , where  $F^2$  is the squared modulus of the structure factor and  $L_p$  is the powder Lorentz-polarization factor. The variation in position and breadth of the 020 reflection was extreme (Table 1), much larger than other layer structures such as clay minerals. A  $2^\circ 2\theta$  shift in position and breadths exceeding  $5^\circ 2\theta$  for the 020 reflection were measured on some samples synthesized below  $125^\circ\text{C}$ . A plot of position vs. breadth for the 020 reflection is shown in Figure 4. The progressive gradation shown by these XRD patterns indicates that any distinction between boehmite and pseudoboehmite is arbitrary. Pseudoboehmite is essentially boehmite with small crystallite size.

In general, an increased heating time or temperature

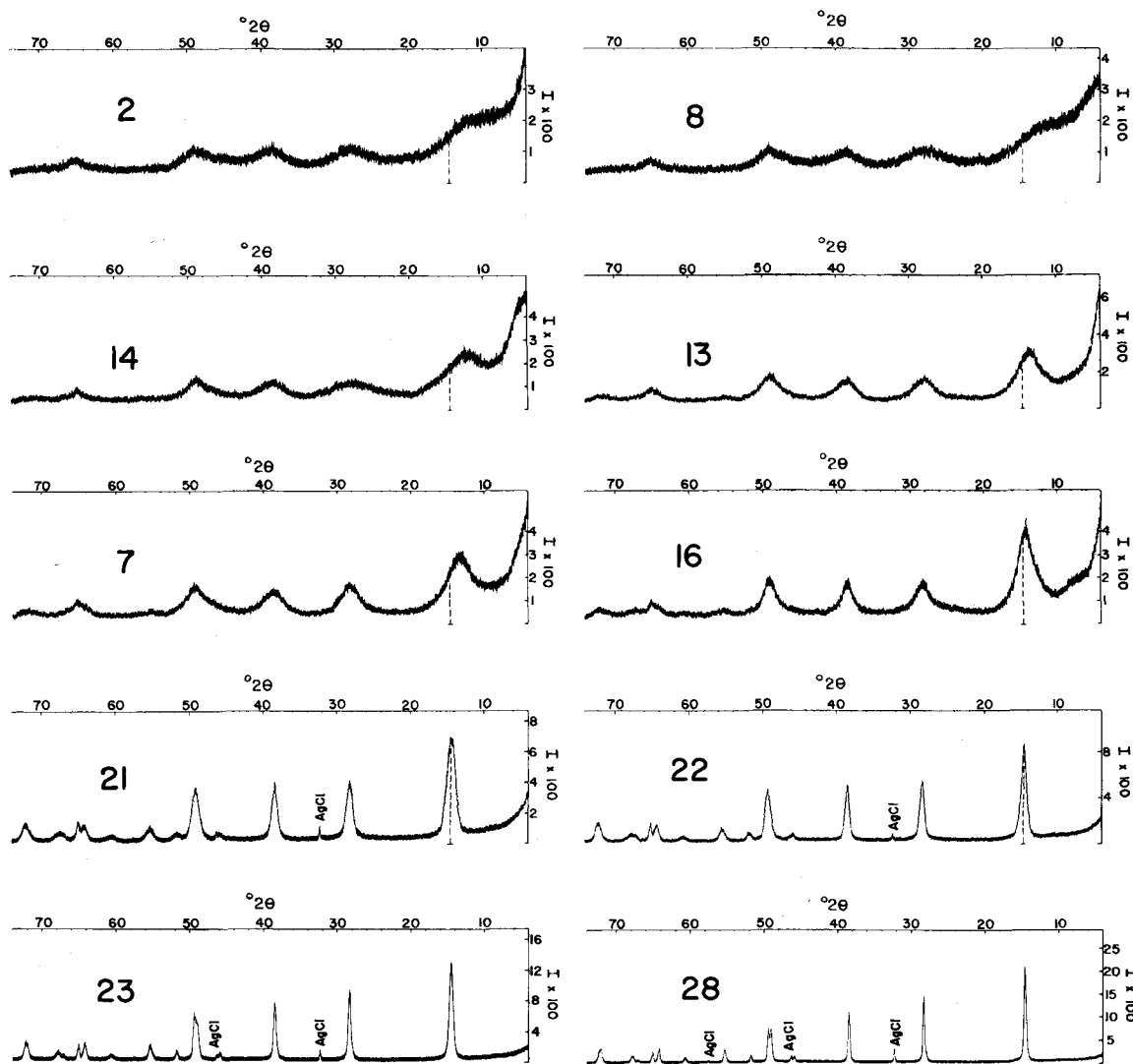


Figure 3. X-ray powder diffraction patterns of synthetic boehmites with  $\text{CuK}\alpha$  radiation. Intensity scales are counts/sec  $\times$  100. The dashed vertical line at  $14.5^\circ 2\theta$  represents the 020 position of the samples synthesized at  $300^\circ\text{C}$ .

of synthesis produced larger crystallites (Table 1). Notable exceptions are samples 8, 14, and 15. Whether the data for these indicate lack of control of an unsuspected variable during synthesis, drying, and handling, or a factor inherent in this system is not known. The XRD patterns presented by Lahodny-Šarc *et al.* (1978) also show more diffuse peaks for a sample prepared at  $100^\circ\text{C}$  compared with one prepared at  $80^\circ\text{C}$ .

All XRD peaks for samples synthesized at low temperatures ( $<125^\circ\text{C}$ ) were uniformly broad and reasonably symmetrical. The high angle reflections were not sharper than the 020 reflection as noted by Papée *et al.* (1958) which suggests that these samples are composed of crystallites that are small in all directions, not just along  $y$ . Tailing of non-basal reflections to high angles, as shown by smectites, was not observed for boehmite,

indicating that small crystallite size, and not layer disorder, was responsible for peak broadening in boehmite.

XRD patterns were made to determine whether or not the  $2\theta$  and  $\beta$  of the 020 reflection were the result of sorbed water between octahedral layers. Samples were heated at  $65^\circ$ ,  $200^\circ$ , and  $350^\circ\text{C}$  for one day at each temperature and X-rayed in a sealed scatter shield which also contained a desiccant. Patterns of samples 2, 13, and 16 were unchanged and indicated that no water was present between the octahedral layers. When samples 7, 8, and 14 were heated at  $200^\circ\text{C}$ , the 020 peaks shifted to high  $2\theta$  values, and  $\beta$  decreased (Figure 4). The temperature-modified 020 peaks for these were measured as  $2\theta = 13.82^\circ$ ,  $\beta = 2.89^\circ$  for sample 7,  $2\theta = 12.95^\circ$ ,  $\beta = 4.80^\circ$  for sample 8, and  $2\theta = 13.11^\circ$ ,  $\beta = 4.12^\circ$  for

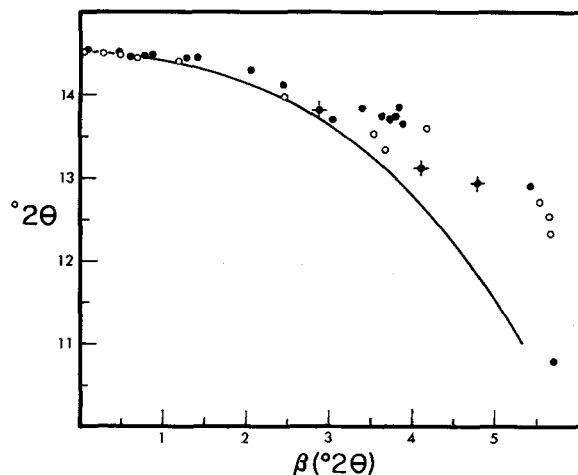


Figure 4. Position ( $2\theta$ ) vs. pure diffraction breadth ( $\beta$ ) of the 020 XRD peaks for synthetic boehmites for  $\text{CuK}\alpha$  radiation. Calculated relationship shown as solid line. Open circles represent experimental data from freeze-dried boehmites; closed circles represent experimental data from boehmites oven-dried at  $125^\circ\text{C}$ ; closed circles with crosses represent experimental data from freeze-dried boehmites subsequently heated to  $200^\circ\text{C}$ . The latter heat-treated samples are 7, 8, and 14 whose  $\beta$  values are  $2.89^\circ$ ,  $4.80^\circ$ , and  $4.12^\circ 2\theta$ , respectively. Experimental positions measured at the midpoint of the chord at half-maximum intensity; experimental breadths measured at half-maximum intensity and corrected for instrumental broadening.

sample 14. Sample 14 was unique in that it showed a broad peak at  $4\text{--}5^\circ 2\theta$  prior to heating (Figure 5). This "long spacing" peak disappeared on heating. An attempt to regenerate the "long spacing" by rehydration in a water-filled container for one day was unsuccessful. It is concluded that samples 7, 8, and 14 contained water intercalated between octahedral layers, randomly along  $y$  for 7 and 8, and regularly for 14.

XRD patterns of samples synthesized at  $100^\circ\text{C}$  or below and subsequently heated at  $350^\circ\text{C}$  showed no peaks. The patterns of these heated boehmites which had been rendered "X-ray amorphous" contrasted sharply with the broad but recognizable maxima observed in the patterns of the unheated materials.

Weak  $\text{AgCl}$  (cerargyrite structure) peaks were evident on all XRD patterns of samples synthesized in the silver-lined bombs at  $150^\circ\text{C}$  or higher. Evidently, the silver liners were not inert at these temperatures and reacted with remnant  $\text{Cl}^-$ . The presence of  $\text{Cl}^-$  suggests that the washing procedure was incomplete. However, no  $\text{NaCl}$  was detected in the XRD patterns. Chloride ions likely were incorporated in the gel which prevented their complete removal by the washing procedure. The maximum amount of  $\text{AgCl}$  was estimated to be less than 4%.

Calculated one-dimensional diffraction profiles along  $y$  were made to compare with the experimental data.

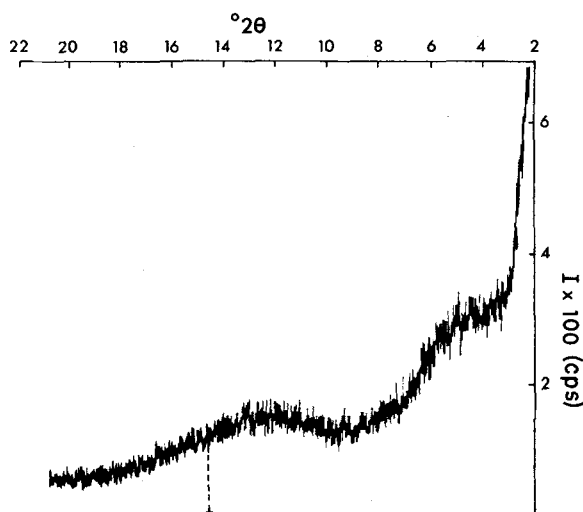


Figure 5. X-ray powder diffraction pattern with  $\text{CuK}\alpha$  radiation of sample 14 showing a "long spacing" peak at  $4\text{--}5^\circ 2\theta$ . The dashed vertical line at  $14.5^\circ 2\theta$  represents the 020 position of the samples synthesized at  $300^\circ\text{C}$ .

Relative intensities were computed at  $2\theta$  values from  $4^\circ$  to  $20^\circ$  in  $0.1^\circ 2\theta$  increments by multiplying the squared modulus of the Fourier transform by the appropriate powder Lorentz-polarization factor. The  $b$ -dimension,  $y$  coordinates of the individual atoms, and temperature factors were taken from Christoph *et al.* (1979). Exact agreement between calculated and observed data was not expected since these calculations assumed infinite extension in the  $xz$  plane, whereas the XRD data from samples grown at low temperatures indicated restricted growth in this plane. Better agreement is expected by profiling in three dimensions, and that analysis has now begun.

Diffraction profiles were calculated for crystallites which had as many as 60 unit cells along  $y$ . Because the boehmite unit cell contains two aluminum octahedral layers along  $y$ , computations were made at "half cell" increments also. The  $2\theta$  and  $\beta$  values for the 020 reflection were measured from the computed profiles as was done for the experimental patterns. The computed points were connected with the smooth line shown in Figure 4.

The theoretical curve essentially forms a lower boundary for the experimental points. Agreement between theory and experiment is good for large crystallites, i.e., the data at high  $2\theta$  and low  $\beta$ , but the agreement is poorer for small crystallites. To bring the theoretical and experimental data into closer accord, the calculations were modified to allow for (a) substitution of  $\text{Cl}$  for  $\text{OH}$  as suggested by the presence of  $\text{Cl}$  in the gels, (b) sorbed water molecules between octahedral layers, and (c) a distribution of crystallite sizes

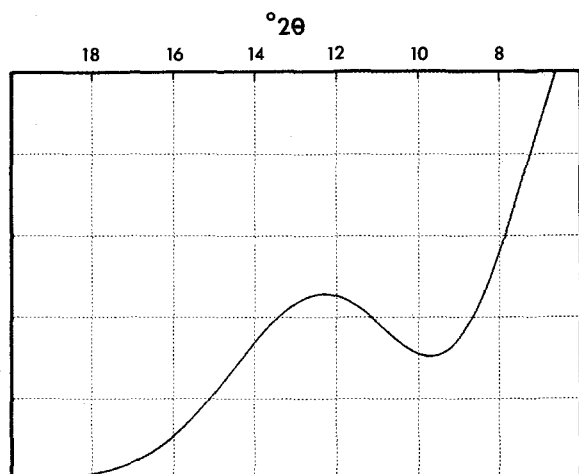


Figure 6. Calculated mean X-ray diffraction profile for  $\text{CuK}\alpha$  radiation along  $y$  for boehmite consisting of equal amounts of 1, 2, and 3 octahedral layers.

(thicknesses). Substitution of Cl for OH and intercalation of water between octahedral layers did not improve the agreement between experiment and theory. However, XRD patterns computed for a range of crystallite thicknesses gave theoretical points above the theoretical curve shown in Figure 4 and in the region of the experimental points. For example, the 020 peak (Figure 6) for equal amounts of 1, 2, and 3 octahedral layers gave  $2\theta = 12.52^\circ$  and  $\beta = 4.69^\circ$ . Another calculation gave  $2\theta = 11.95^\circ$  and  $\beta = 5.71^\circ$  for a model containing 75% of two octahedral layers and 25% of three octahedral layers. Results from calculated profiles indicate that boehmites whose  $\beta(020) \approx 5-6^\circ 2\theta$  consist of a significant number of crystallites which are composed of a single unit cell or a very few unit cells at most along  $y$ . Both single unit cells and single octahedral units apparently contribute to the diffraction pattern even though no 020 peak is present for the latter, only a smooth rise in intensity as  $2\theta$  decreases. The absence of the 020 peak observed by other investigators is likely due to the presence of a significant number of single octahedral units in their samples.

#### Differential thermal analysis

DTA patterns were made on 7 samples. The patterns are arranged in Figure 7 to depict a progressive gradation from one to the next. The arrangement from top to bottom is in order of increasing crystallite size as determined by XRD (Table 1). The low temperature endotherm centered at about  $150^\circ\text{C}$ , attributed to sorbed water, is an important feature of all patterns except those of samples 23 and 29. Its intensity decreases, in general, with an increase in both synthesis temperature and crystallite size. The low temperature endotherm in the patterns of samples 8 and 15 is merged with

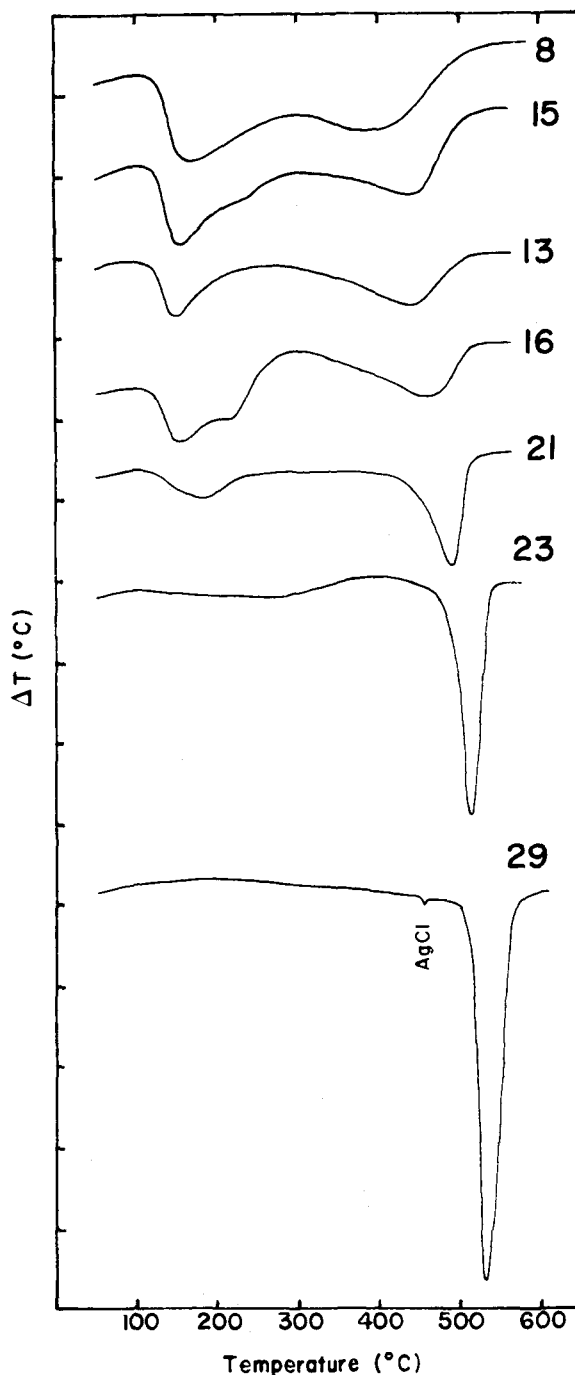


Figure 7. Differential thermal analysis patterns of synthetic boehmites. Increment on  $\Delta T$  scale is  $2^\circ\text{C}$ .

the high temperature endotherm suggesting a continuous gradation of bonding energy for the water. The very broad endotherm centered near  $275^\circ\text{C}$  in the pattern of sample 23 indicates the presence of strongly bound water molecules. This endotherm is absent in the pattern of sample 29.

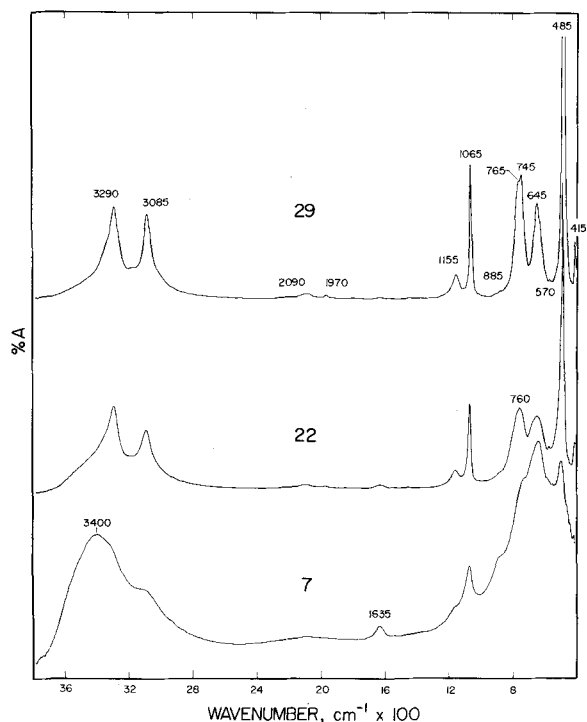


Figure 8. Infrared spectra of synthetic boehmites in KBr disks. Absorbance scale is linear.

The high temperature endotherm, representing the loss of hydroxyl groups, increases in intensity and shifts to higher temperatures from top to bottom. This variation is, in itself, an indication of an increase in crystallite size (Smothers and Chiang, 1958, p. 52). The progressive gradation of the patterns also suggests that any distinction between boehmite and pseudoboehmite is arbitrary.

#### Infrared spectroscopy

IR spectra were recorded on three samples (Figure 8). Band assignments for boehmite were made by Fripiat *et al.* (1967) and Russell *et al.* (1978). The IR patterns are similar with regard to the number of bands and their positions, but their intensity and breadth varies. The differences between the patterns is probably due to variations in particle size and to the presence of strongly bound water molecules in sample 7. The enhanced intensity of the band at  $485^{-1}$  cm compared with that in the pattern given by Russell *et al.* (1978) is due to the absorbance scale being linear and the elongation parallel to z of the freeze-dried crystals (V. C. Farmer, Macaulay Institute for Soil Research, Aberdeen, Scotland, personal communication).

#### CONCLUSIONS

The present data show that boehmite and pseudoboehmite are continuous in their structure and physical

properties and that any distinction between them is arbitrary. Pseudoboehmite is essentially finely crystalline boehmite which consists of the same or similar octahedral layers in the xz plane but lacks three-dimensional order because of a restricted number of unit cells along y. It consists of a significant number of crystallites which contain a single unit cell along y or single octahedral layers. It contains more water which is commonly intercalated between octahedral layers, normally randomly arranged, but sometimes regularly. The large breadth and high d(020) are a result primarily of a small number of octahedral layers although both the breadth and d-value of 020 are increased by water molecules between octahedral layers in some samples. Good agreement between calculated and experimental 020 XRD profiles is obtained when a range of crystallite thicknesses is taken into account. The term pseudoboehmite is inappropriate for finely crystalline boehmite and should be dropped from the literature.

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**Резюме**—Тридцать два бемиты, синтезированные при температурах, изменяющихся от комнатной до 300°C, были исследованы с помощью электронного сканирующего микроскопа, электронного трансмиссионного микроскопа, дифракции электронов, рентгеновского порошкового анализа, дифференциального термического анализа, и инфракрасной спектроскопии. Результаты показывают, что бемит проявляет сплошную градацию размеров кристаллитов, изменяющихся от простых октаэдрических слоев или немногих элементарных ячеек вплоть до примерно 65 элементарных ячеек по  $u$ -направлению. Этот вывод подсказывает, что термин псевдобемит является несоответствующим для тонко-кристаллического бемита. Тонко-кристаллический бемит содержит большее количество сорбированной воды, чем грубо-кристаллический бемит; эта вода обыкновенно находится между октаэдрическими слоями, обычно беспорядочно, но иногда регулярно. Регулярно внутринапластованный бемит приводит к рентгеновскому отражению с диффузионным “длинным расстоянием.” Когда берется во внимание диапазон размеров кристаллитов, расчетные 020 рентгеновские пики находятся в хорошем соответствии с экспериментальными величинами. [E.C.]

**Resümee**—Zweiunddreißig, im Temperaturbereich von Zimmertemperatur bis 300°C synthetisierte Boehmite wurden mittels Rasterelektronenmikroskopie, Transmissionselektronenmikroskopie, Elektronenbeugung, Röntgenpulverdiffraktometrie, Differentialthermoanalyse, und Infrarotspektroskopie untersucht. Die Ergebnisse zeigen, daß sich die Kristallitengröße des Boehmits allmählich ändert und von einzelnen oktaedrischen Lagen oder einigen Elementarzellen bis zu ~65 Elementarzellen in der  $y$ -Richtung reicht. Diese Schlußfolgerung deutet darauf hin, daß der Ausdruck Pseudoboehmit für feinkristallinen Boehmit unangebracht ist. Feinkristallisierter Boehmit enthält mehr adsorbiertes Wasser als grobkristallisierter; dieses Wasser ist gewöhnlich zwischen oktaedrische Lagen eingeschaltet und zwar meist statistisch, jedoch manchmal geordnet. Der regelmäßig wechselgelagerte Boehmit gibt eine diffuse “long spacing” Röntgenreflexion. Berechnete 020 Peaks liegen nahe bei den experimentell beobachteten, wenn man eine Variation der Kristallitengröße berücksichtigt. [U.W.]

**Résumé**—Trente deux boehmites, synthétisées à des températures s'étageant de température ambiante à 300°C ont été examinées par microscopie électronique balayante, microscopie électronique par transmission, diffraction électronique, diffraction poudrée aux rayons-X, analyse thermique différentielle, et spectroscopie infrarouge. Les résultats montrent que la boéhmite exhibe une gradation continue de la taille cristalline, s'étageant de couches octaédres simples ou de quelques mailles à à peu près 65 mailles dans la direction- $y$ . Cette conclusion suggère que le terme pseudoboéhmite est inapproprié pour la boéhmite finement cristalline. La boéhmite à fins cristaux contient plus d'eau sorbée que celle à gros cristaux; cette eau est souvent intercalée entre des couches octaédres, habituellement au hasard, mais parfois régulièrement. La boéhmite régulièrement interstratifiée produit une réflexion de rayons-X diffuse à “longs espacements.” Des sommets 020 de rayons-X calculés sont très proches de ceux observés expérimentalement lorsqu'un étagement de tailles cristallines est considéré. [D.J.]