

A TECHNIQUE FOR THE ISOLATION OF MONTMORILLONITE AND HALLOYSITE

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ABSTRACT

A technique is presented for the complete separation of montmorillonite and/or halloysite from other minerals occurring in artificial admixtures and natural clays. The technique is based on differences in specific gravity assumed by these minerals in a solution of alcohol and bromoform.

Techniques at present being employed for the quantitative determination of the clay minerals present in natural clays may be grouped under (a) those based on the characteristics of differential thermal graphs (1,2) (b) those based on the intensity of *x*-ray peaks (3) and (c) those based on chemical analysis in conjunction with other properties such as cation exchange (4). It is doubtful if any of these techniques afford results better than plus or minus five per cent, though some workers have claimed greater accuracy.

Methods so far used for the isolation of the clay minerals are less effective. The one most commonly employed, based on the preferred particle size for the minerals, generally permits a concentration, but rarely yields a complete separation for the required mineral, and moreover, fails to give a full particle size range for the mineral being isolated. More recently Buzagh and Szepesi (5) have presented a technique for the isolation of montmorillonites from mixed clay samples based on the stability of the sodium clay in an alcohol-water suspending medium.

In the present paper, the complete separation of montmorillonite and halloysite is demonstrated, utilizing the apparent specific gravity assumed by these minerals when suspended in a solution of ethyl alcohol and bromoform. The extension of the technique to include illite, kaolinite and quartz generally permits the concentration of these minerals, but, since particle size is an important factor, the results are less satisfactory.

EXPERIMENTAL TECHNIQUES

Determination of the Apparent Specific Gravity of Reference Minerals.

Two relatively pure samples of each of kaolinite, halloysite and montmorillonite and one of illite were used as reference minerals. A crystal of quartz was crushed to varying sieve sizes and subjected to the same procedure as that of the other minerals. The apparent specific gravities assumed by these minerals in a solution of alcohol and bromo-

form was determined by the following procedure. Samples of 500 mg. of each mineral, previously ground to minus 200 mesh, were placed in a centrifuge tube containing bromoform. The bromoform was then diluted with alcohol, thoroughly shaken for ten minutes on a flask shaker, allowed to stand a further ten minutes, then centrifuged. The process of

TABLE 1. REFERENCE MINERALS

Mineral	Location	Apparent S.G.
S. 1 Kaolinite	St. Ives, N.S.W.	2.34
S. 2 Kaolinite	Huber	2.37
S. 3 Halloysite	Eureka, Utah	2.17
S. 4 Halloysite	Bedford, Indiana	2.19
S. 5 Illite	Fithian, Illinois	2.27
S. 6 Montmorillonite	Wingen, N.S.W.	1.85
S. 7 Montmorillonite	Algeria	1.85
Quartz	Minus 270 Mesh	2.35
Quartz	200-270 Mesh	2.45

alcohol dilution was continued until the mineral just remained suspended. (Actually it was found that there was a range for the critical specific gravities and the values quoted below represent means.) The solution was filtered and the specific gravity determined by a micro-Westphal balance. Table 1 gives the localities of the reference minerals and their apparent specific gravities, and Table 2 the chemical analyses. Fig. 1 shows the differential thermal analysis curves for these reference minerals, as well as additional natural clays discussed later.

TABLE 2. CHEMICAL ANALYSES OF REFERENCE MINERALS

	S1	S2	S3	S4	S5	S6	S7
SiO ₂	43.1	45.1	43.6	44.3	51.1	51.6	59.7
Al ₂ O ₃	34.0	37.7	40.3	39.1	26.1	16.9	18.6
Fe ₂ O ₃	0.67	0.7	0.4	0.4	6.1	5.6	1.7
TiO ₂	5.73	1.4	0.1	0.1	0.6	0.4	0.03
CaO	—	—	—	—	—	3.63	1.11
MgO	—	—	—	—	—	1.33	—
K ₂ O	—	—	—	—	6.1	0.54	0.34
H ₂ O	12.30	13.9	14.7	13.4	7.3	11.82	6.09
H ₂ O—	3.00	0.9	2.5	4.0	—	8.59	9.12
Total	98.80	99.7	101.6	97.3	100.4	100.4	96.7

Separation of An Artificial Mixture of Clay Minerals

A sample of 100 mg. of each S1, S3, S5 and S6, together with a similar amount of quartz crushed to pass 150 mesh but retained on a 200 mesh sieve, was placed in a centrifuge tube of 23 mm. diameter containing a bromoform-alcohol solution with a specific gravity of 2.05. The tube and

TABLE 3. CHEMICAL ANALYSES OF NATURAL CLAYS

	I	II	III
SiO ₂	50.9	63.3	48.7
Al ₂ O ₃	24.4	16.7	15.6
Fe ₂ O ₃	3.61	4.75	7.48
TiO ₂	1.68	1.13	0.33
CaO	0.19	0.81	—
MgO	1.44	0.33	—
K ₂ O	0.84	2.28	1.12
H ₂ O	9.01	3.07	5.35
H ₂ O—	7.62	5.50	16.85
Total	99.7	97.9	95.4

I—Fullers earth from Dubbo, N.S.W.

II—Shale underlying the bentonite deposit at Wingen, N.S.W.

III—Montmorillonite from the alteration of andesitic basalt, Barraba, N.S.W.

contents were shaken for ten minutes on a flask shaker, permitted to stand a further ten minutes and then centrifuged. The tube was stoppered throughout the whole procedure. The supernatant liquid containing the light fraction was decanted, and the clay removed, dried and *x*-rayed. It was found to be pure montmorillonite.

The above procedure was repeated on the remaining heavy fraction, using successively solutions with specific gravities of 2.20, 2.30 and 2.40; the light fractions representing halloysite, illite and kaolinite respectively. The halloysite fraction was found to be pure when *x*-rayed, but the illite, kaolinite and remaining quartz fraction showed mutual con-

TABLE 4. COMPOSITION OF NATURAL CLAYS

Sample No.	Montmorillonite	Halloysite	Remainder
I	6%	76%	Predominantly quartz with a little kaolinite
II	31%	—	Quartz with approx. 20% kaolinite
III	87%	—	Predominantly feldspar

tamination. In all cases, however, a concentration exceeding 80% was obtained.

Separation of Montmorillonite and Halloysite from Natural Clays

A modification of the above procedure was then applied to three natural clays. They were first dispersed in water using calgon as a dis-

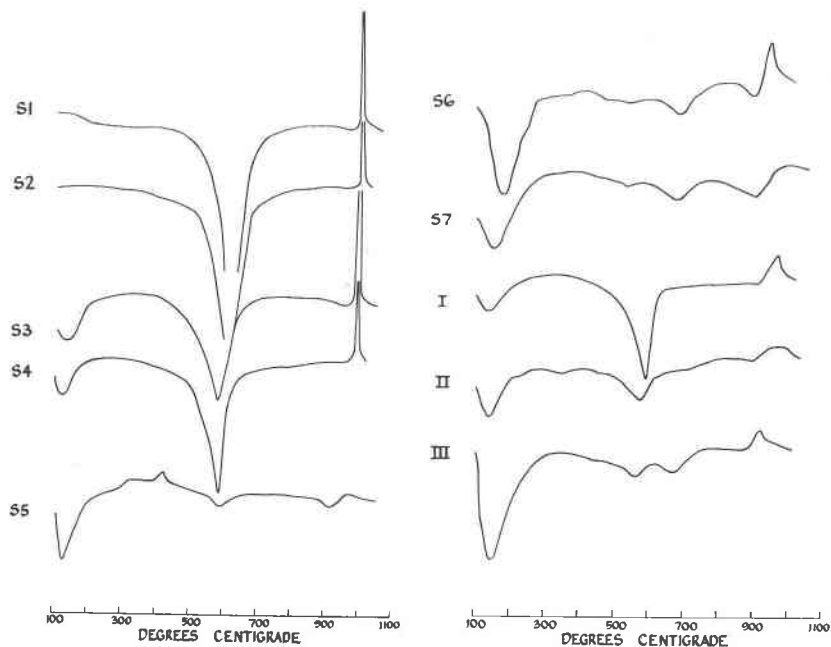


FIG. 1. Differential thermal analysis curves for reference minerals and natural clays. (See Tables 1 and 3).

persant, and then dried at 40° C. before being placed in a bromoform-alcohol solution of S.G. 2.00. A specific gravity of 2.27 was used for the recovery of the halloysite fraction.

DISCUSSION

The decrease in density (6) of the dried clay when treated with a bromoform alcohol solution is dependant on two factors. Firstly, the ability of the mineral to adsorb alcohol with a subsequent expansion of the lattice and, secondly, the particle size, which controls the specific surface of any given mineral. For montmorillonite and to a lesser extent halloysite, lattice expansion with the accompanying decrease in true

density is the predominating factor, but for kaolinite, illite and quartz, where expansion is insignificant, particle size is of greater importance. Consequently, the separation of montmorillonite and/or halloysite from mixed clay samples is possible by the technique outlined above, provided that other minerals occurring in the mixed samples do not have particles with diameters considerably smaller than the minerals being separated. The probability of this happening in the case of montmorillonite is remote, but with halloysite there is a strong possibility of one of the contaminants having such a particle size. In this event the value of the technique is doubtful.

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