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AN *X*-RAY FLUORESCENCE METHOD FOR THE QUANTITATIVE DETERMINATION OF SMALL AMOUNTS OF MONTMORILLONITE IN KAOLIN CLAYS*

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During the course of an investigation of the kaolin deposits of the Coastal Plain of Southeastern United States it became evident that in order to better understand the genesis of the deposits a method was needed of measuring the small amounts of montmorillonite associated with the kaolinite. The method developed utilizes the difference in base exchange capacity of the two minerals. It consists of exchanging the Sr^{++} ion onto the montmorillonite, removing the excess strontium by dialysis, analyzing the exchanged sample for strontium by *x*-ray fluorescence, and computing the percentage of montmorillonite from these results according to a curve derived from known standards. This method makes the following assumptions:

1) The base exchange capacity of the sample is due primarily to montmorillonite, *i.e.* the base exchange capacity of the kaolinite is negligible in comparison to montmorillonite.
2) The base exchange capacity of the montmorillonite in the standards is the same as that of montmorillonite in the sample.

Preparation of Standards

Standards were prepared by mixing varying proportions of Wyoming bentonite with Georgia kaolin. The weighings were made after the clays had been ground to pass 200 mesh, dried at 105° C., and placed for several days in a constant humidity atmosphere produced by $CaCl_2 \cdot 6H_2O$. The clays were intimately mixed by passing the material through a 100 mesh sieve several times and by shaking vigorously in a jar between sievings.

Strontium Ion Exchange

To a one gram sample taken from the standard mixture and placed in a 75-ml. test tube, 50 ml. of 1N $SrCl_2$ solution is added. After stoppering, the tube is vigorously shaken and allowed to sit one hour. The sample is then transferred into $1\frac{1}{8}''$ diameter cellophane dialyzer tubing which has

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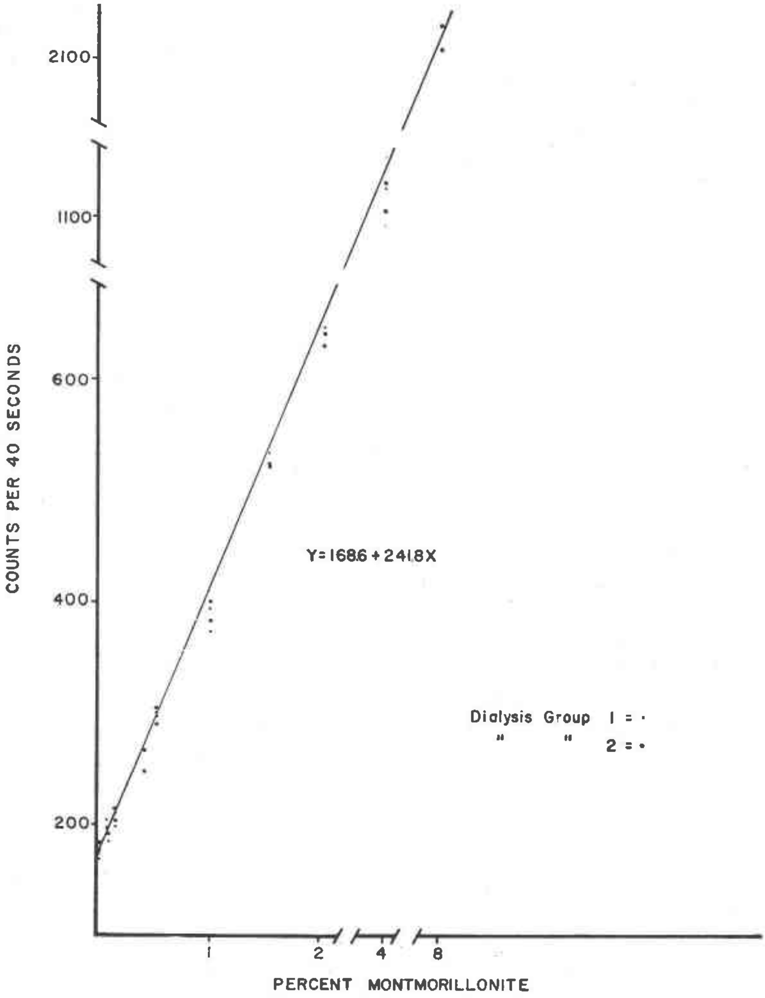

FIG. 1. Per cent montmorillonite versus counts per 40 seconds (x-ray fluorescence analysis of exchange Sr^{++}).

previously been cut into 15-inch lengths and tied at one end. The filled tube is then suspended from both ends in a 1-quart, wide-mouth mason jar which is filled with distilled water and allowed to remain undisturbed for 24 hours at which time the water is changed. While changing the water the sample is agitated by massaging the dialysis bag. This is repeated for 10 consecutive days. On the 11th day the tubing is left suspended in the jar and by use of a fan the amount of liquid in the tube is sufficiently reduced by evaporation to permit washing the sample from the tubing into a small jar for drying and storage. After drying the sample is pulverized and mixed in the jars by use of a rubber policeman and is then ready to be packed in holders for *x*-ray fluorescence analysis.

X-ray Fluorescence Analysis

For this work an XRD-5 General Electric *x*-ray unit with counting apparatus is used with a LiF analyzer crystal and a .005 collimator. With the tube operating at 50 KV and 50 MA, a 40 second background count is taken at about 29°2θ and subtracted from the average of two 40 second sample counts which are taken at a previously determined maximum intensity position for the $Sr^{+2}K\alpha$ wave length (about 25°2θ). A plot of counts versus per cent montmorillonite indicates the relationship to be essentially linear as shown in Fig. 1. For the construction of this regression line ten standards ranging from 0.00% to 8.00% montmorillonite were taken in duplicate and separately dialyzed as previously described. After dialysis two packings were made from each for the *x*-ray analysis.

A statistical analysis of the results shows that the differences in machine and dialysis error not significant at the .90 probability level. A combined regression line, $y=168.6+241.8x$, is constructed from which unknowns can be determined with a precision of approximately ±0.5% in the 0.5–8.0% range.

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THE CELL CONSTANTS OF ARTIFICIAL SIDERITE*

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The artificial siderite used to measure the cell constants was prepared in the following way. Equal molar quantities of solid $NaHCO_3$ and $FeSO_4 \cdot 7H_2O$ were placed in a 120 ml. capacity stainless steel bomb which was half filled with water. The sealed bomb was then heated to 200° C. After reaching 200° C., CO_2 was pumped into the bomb until a total pressure of 500 bars had been reached. The bomb was maintained at 200° C. and 500 bars for 3 days. This procedure served to stabilize the precipitated $FeCO_3$. The white precipitate was then removed, filtered, and dried in an oven at 100° C. It partly oxidized to a yellow-brown

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