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SELECTIVE DISSOLUTION OF MICAS FROM POTASSIUM FELDSPARS BY SODIUM PYROSULFATE FUSION OF SOILS AND SEDIMENTS

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ABSTRACT

The J. Lawrence Smith (1865) sodium pyrosulfate fusion is employed to decompose micas by removing octahedral cations so that their relics become readily soluble in the 0.5 *N* NaOH 150-second-boiling treatment of Hashimoto and Jackson (1960). Micas are thus selectively dissolved away from potassium feldspars, which are shown to be remarkably resistant to the treatment. The amount of K feldspars dissolved and the amount of exchange of Na from the $Na_2S_2O_7$ for K of potassium feldspars is compensated for in the residues by correction factors determined for various size fractions. The mica potassium is equal to initial potassium less corrected residual feldspar potassium.

INTRODUCTION

Removal of layer silicate minerals from quartz and feldspars in fine grained mineral samples is frequently desirable for the purpose of quantitative determination of the layer silicate content or for examining the components other than layer silicates. In quantitative mineralogical analysis, such a separation is helpful in the allocation of potassium and other elements to their mineral species. Potassium is a major plant nutrient occurring in both feldspars and micas in soils. The availability of potassium to plants is known to be largely determined by the mineral species in which it occurs and thus a differentiation of K feldspar from K mica is often desirable in mineralogical analysis as applied to soil fertility.

The methods available for separation of micas from feldspars have severe limitations. Specific gravity separation is not quantitative, since the two types of minerals have overlapping specific gravity ranges, which is particularly troublesome when particle sizes are below 50 microns. Most chemical methods involving dissolution have been aimed at dissolution of all nonquartz minerals (Hardy and Follett-Smith, 1931; Shaw, 1934; Trostel and Wynne, 1940; Durkan, 1946; Talvitie, 1951, Jophcott and Wall, 1955; Medicus, 1955; Jackson, 1956; Nagelschmidt, 1956; Shchekaturina and Petrashen, 1958). Feldspars, however, are only partly soluble in such treatments (Talvitie, 1951; Corey, 1952). A method for quantitative separation of potassium in feldspars from that in muscovite mica was proposed by Reynolds and Lessing (1962). It involves heating a sample at 825° C. for 2 hours, cooling it and boiling it in concentrated HCl for 2 hours. The treatment was reported to remove most of the potassium from muscovite in the fraction between 10 and 2 microns while the potassium content of a K feldspar remained unaffected.

Pyrosulfate fusion has long been employed for the decomposition of aluminosilicate minerals (Smith, 1865). Smith (1865) suggested the use of sodium pyrosulfate in preference to potassium pyrosulfate (which had previously been more commonly employed) since the sodium silicates formed during fusion are more readily dissolved than potassium silicates. Bisulfate is commonly used since it decomposes on heating, thus:

$$2\ NaHSO_4 \rightarrow Na_2S_2O_7 + H_2O$$

to form pyrosulfate. Water loss during the reaction causes considerable spattering. On further heating pyrosulfate decomposes in a quiet fusion liberating acid fumes, thus:

$$Na_2S_2O_7 \rightarrow Na_2SO_4 + SO_3$$

The fusion serves as a high temperature acid treatment, dehydroxylating kaolinite and removing octahedral cations from micas. For quartz determination as a residue in nonfeldspathic materials, Trostel and Wynne (1940) used a potassium pyrosulfate fusion. Astaf'ev (1958) employed sodium pyrosulfate fusion for quartz determination.

The method herein described involves fusion of a sample in sodium pyrosulfate, followed by HCl washing to remove cations which interfere with the subsequent dissolution of layer silicate relics in hot 0.5 N NaOH. Layer silicates less than 500 μ, with the exception of talc and pyrophyllite, were found to be completely soluble in the treatment, while quartz, feldspar other than plagioclase of high anorthite content, and some other silicates strongly resist the treatment.

Sodium pyrosulfate was used in preference to potassium pyrosulfate because it was found that potassium from potassium pyrosulfate was exchanged for sodium in plagioclase feldspars and some potassium was adsorbed in other minerals also. The cation exchange effect resulting with sodium pyrosulfate was less marked and, since a separation of K mica from K feldspars was the objective, possible introduction of potassium as a reagent was avoided.

PROCEDURE

A 200-mg sample of sand or silt sized fraction (dried at 110° C.) is weighed into a 50-ml vitreous silica crucible and 12 to 15 gm of $Na_2S_2O_7$ powder is added and mixed with the sample by means of a glass rod. The $Na_2S_2O_7$ is fused under a fume hood, a low flame being used at first until vigorous bubbling of the melt ceases. Thereafter the full flame of a Meker burner is applied. The fusion is complete when some Na_2SO_4 crystals appear on the surface of the melt while at full heat. The crucible is swirled while cooling in order to spread the melt on the crucible sides.

The solidified melt is transferred as a cake to a 150-ml beaker with 60 ml of 3 *N* HCl and the aid of a rubber tipped glass rod. The cake is slaked by gentle boiling and the resulting suspension is transferred to a 70-ml pointed centrifuge tube. The insoluble material is separated by centrifugation and the supernatant liquid discarded. Two further washings with 3 *N* HCl are employed to complete the transfer and to wash the residue, the supernatant liquid being discarded each time.

The residue from the tube is transferred to a 500-ml nickel or stainless steel beaker with 0.5 *N* NaOH and the total volume of NaOH in the beaker is made up to 100 to 150 ml. The suspension is brought rapidly to boiling, boiled for 2.5 minutes, and cooled rapidly in a cold water bath. The suspension is transferred to 70-ml pointed centrifuge tubes, the insoluble residue from the beaker being transferred quantitatively. The residue is separated by centrifugation and washed 3 times with 3 *N* HCl. It is then transferred to a tared platinum crucible (or Teflon beaker), dried at 110° C., and weighed.

Analysis for K_2O is carried out on the residue and also on the original sample dried at 110° C., according to the HF-$HClO_4$ method.

MATERIALS AND EXPERIMENTAL

Layer silicate samples used in testing the procedure included kaolinite from Georgia Kaolin Co.; muscovite from Marshall County, North Carolina, and biotite from Spruce Pine, North Carolina (Wards Scientific Est., Rochester, N. Y.); bentonite from Princeton, Br. Columbia; illite from Blaylock Formation, Oklahoma (courtesy C. J. Mankin); illite from Marblehead Quarry, Wisconsin; illite from Fithian, Illinois; chlorite from Cartersville, Georgia (Wards Scientific Est., Rochester, N. Y); and vermiculite from Libby, Montana (Wards Scientific Est., Rochester, N. Y.). Microcline (Wards Scientific Est., Rochester, N. Y.) and orthoclase were used as examples of potassium feldspars.

Muscovite and biotite samples were ground by prolonged treatment of flakes in suspension by a Waring Blendor. Other mineral samples were ground in an agate pestle and mortar. Silt fractions from two soils of Ireland, employed to illustrate the method, include Leitrim Daub, a Humic Gley soil formed from glacial till high in silt sized quartz, and Ballinree, a Brown Podzolic soil formed from granite containing appreciable feldspars. All samples were size-fractionated by the method of Jackson (1956).

The efficacy of the procedure outlined above was determined by applying it to the mineral fractions obtained and determining its effect on the potassium content of the minerals.

RESULTS

Layer silicates. Samples of kaolinite, muscovite, biotite, illite, glauconite vermiculite, montmorillonite and chlorite were completely dissolved by the treatment of the less than 500 μ fractions (Table 1). The fusion had to be carried to the point specified to ensure complete dissolution of these layer silicates; the 2 to 4 per cent residues with muscovite (Table 1) contained undetectable amounts of K ($<0.02\%$). Residues frequently occurred with the various samples but these were shown to consist mainly of quartz and feldspar as indicated by *x*-ray diffraction (Fig. 1) and by elemental analysis (Table 1). The high background intensity in the 7 to 18A portion of the *x*-ray diffractograms of the residues was shown to be a property shown by quartz and feldspar (Fig. 1) in the *x* ray spectrum used, and does not indicate layer silicates in the residue. Talc and quartz were found in the residue from the chlorite sample. Further studies of less than 500 micron talc and pyrophyllite specimens showed that both resist the treatment. *X*-ray diffraction studies indicated that the vermiculite sample from Libby, Montana (Wards Scientific Est., Rochester, N. Y.) consisted of interstratified 10A and 14A mixed layer silicate (MacEwan *et al.*, 1961, pp. 396–7). The quartz residue from this specimen, not clearly indicated for the original sample, is of interest. Quartz is sometimes noted in residues of certain biotites also.

Feldspars. The dissolution effect the treatment had on different size fractions of microcline was determined. The percentage recovery of microcline by weighing ranged from 97.5 per cent in the 2 to 0.5 mm fraction to 64 per cent in the 2 to 0.2 micron fraction (Table 2). A lower percentage of potassium occurred in the residues than in the untreated samples, indicating an exchange of Na from pyrosulfate for K of feldspar, a stoichiometric exchange which was confirmed in several experiments (to be reported elsewhere). The exchange of Na for K increased with decrease in size fraction.

Based on the difference in per cent potassium between residue and sample, and on the amount of mineral dissolved, conversion factors were determined (Table 2) to convert residue K_2O to feldspar K_2O in the original sample. This factor, ranging from 1.03 to 1.73 for the different size fractions, corrects for both the dissolution (increasing much with the finer fractions) and for the Na-for-K exchange which occurred in the residue. The "conversion factor, residue K_2O to microcline" is based on the factor 5.9 for conversion of K_2O to endmember microcline (16.9 per cent K_2O in endmember K feldspar). It differs from 5.9, however, because of a reduction in the K_2O content of feldspars brought about by

Table 1. Solubility of Layer Silicate Minerals and K_2O Loss During $Na_2S_2O_7$-HCl-NaOH Treatment

Mineral and size fraction	K_2O content	Per cent residue	Per cent K_2O in residue	Per cent K_2O loss	Minerals in residue as shown by X.R.D.
Muscovite					
500-100μ	11.4	0.0	—	100	—
		2.3[1]	—	100	—
		4.4[1]	—	100	—
50-20μ	11.2	0.0	—	100	—
Biotite					
500-100μ	10.8	0.0	—	100	—
Illite					
(Fithian, Ill.)					
500-100μ	4.82	25.3	0.02	99.9	*Quartz*, feldspar
		25.1	0.03	99.8	*Quartz*, feldspar
Illite					
(Marblehead, Wis.)					
500-100μ	8.91	12.6	9.82	84.1	*Feldspar*, quartz
Illite					
(Blaylock, Okla.)					
500-100μ	6.52	2.2	0.00	100	*Quartz*
Glauconite					
(Madison, Wis.)					
500-100μ	4.9	28.5			
		26.8	1.21	93.2	*Quartz*, feldspar
-5μ	7.69	1.9	—	—	*Quartz*, feldspar
Kaolinite					
500-100μ	0.0	0.0	—	—	—
Vermiculite					
(Libby, Mont.)	5.81	7.1	0.00	100	*Quartz*
Chlorite					
(Cartersville, Ga.)					
500-100μ	0.12	3.4	0.00	100	*Talc*, quartz
Bentonite					
(Princeton, B.C.)					
500-100μ	0.05	4.5	1.21	0.0	Feldspar, quartz, cristobalite

[1] Fusions cut short of final appearance of crystals.

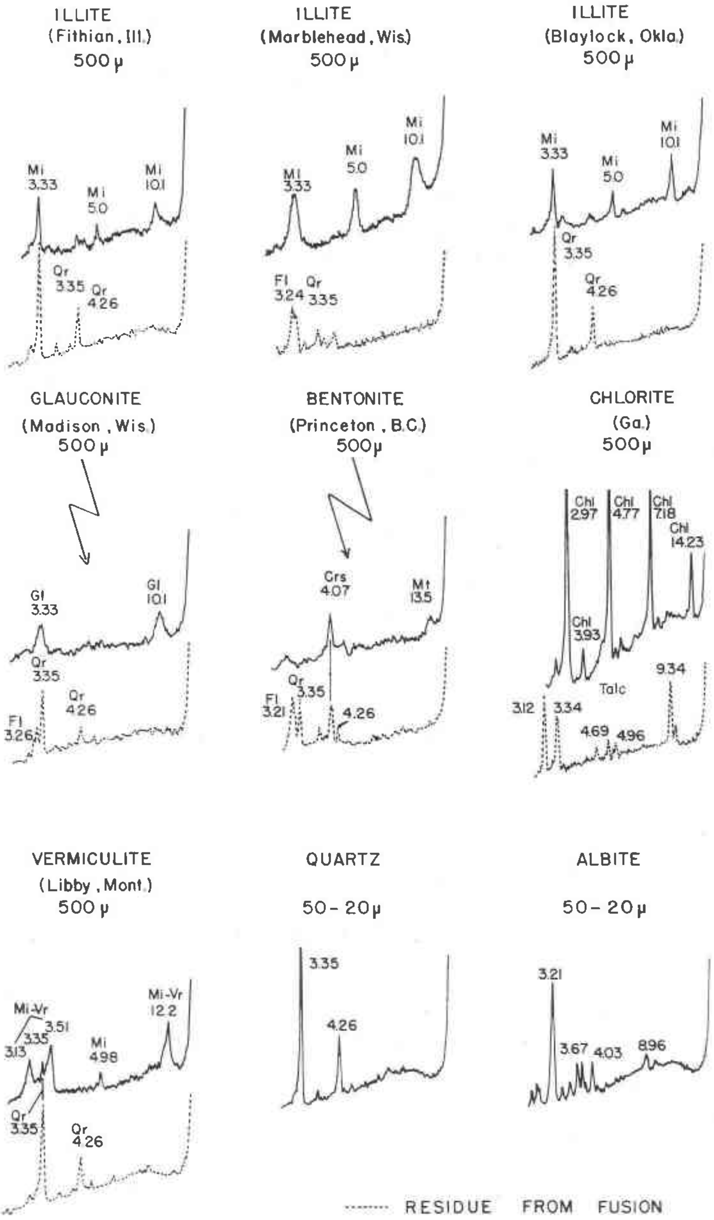

FIG. 1. *X*-ray diffractograms of layer silicate samples before and after $Na_2S_2O_7$-HCl-NaOH treatment showing removal of layer silicates and concentration of quartz, feldspar and talc impurities by the treatment. Quartz and feldspar *x*-ray diffractograms are included for comparison.

Abbreviations: Mi = mica; Qr = quartz; Fl = feldspar; Chl = chlorite; Gl = glauconite; Crs = cristobalite; Mt = montmorillonite; Vr = vermiculite.

TABLE 2. K_2O LOSS FROM MICROCLINE[1] DURING TREATMENT AND FACTORS FOR CONVERSION OF RESIDUE K_2O TO FELDSPAR K_2O AND MICROCLINE[1] FOR DIFFERENT SIZE FRACTIONS

Size fraction	Per cent K_2O in fraction	Per cent residue from treatment by wt.	Per cent K_2O in residue	Per cent K_2O lost of that originally present	Conversion factor, residue K_2O to feldspar K_2O	Conversion factor, residue K_2O to microcline
2-0.5 mm	14.7	97.5	14.7	2.5	1.026	6.05
500-50μ	14.7	96.5	14.7	3.5	1.036	6.11
50-20μ	14.0	96.0	14.0	4.0	1.042	6.46
20-5μ	13.6	93.6	13.2	9.1	1.10	7.01
5-2μ	12.7	84.4	12.2	18.9	1.23	8.42
2-0.2μ	11.4	64.0	10.3	42.2	1.73	13.16

[1] The microcline employed was not an endmember; it contained some sodium substitution for potassium and was equivalent to 87 per cent endmember microcline. The "Conversion factor, residue K_2O to microcline" in the above table multiplied by the per cent residue and by the per cent K_2O in the residue gives the endmember microcline content (87 per cent in this sample).

Na for K exchange, surface loss, and dissolution of feldspars during the procedure. An example of the derivation of these factors may be shown by considering the 5-2μ fraction:

$$\text{Conversion factor, residue } K_2O \text{ to microcline} = 5.9 \times \frac{14.7}{12.2 \times \frac{84.4}{100}} = 8.42$$

Orthoclase feldspar was found to behave similarly to microcline. Since microcline is far more abundant in nature than orthoclase, the microcline conversion factors are applicable generally.

Differentiation of mica K_2O from feldspar K_2O. The K_2O present in the residue generally represents feldspar K_2O. Correction factors are applied to correct for loss of K_2O from feldspars through dissolution and a noticeable exchange of Na for K during sodium pyrosulfate fusion. The mica K_2O content of the sample is derived by difference. Thus the K_2O content of the residue is increased by the appropriate conversion factor (Table 2) for the size fraction to obtain the feldspar K_2O content:

$$\begin{matrix}\text{Per cent} \\ \text{feldspar } K_2O\end{matrix} = \begin{matrix}\text{Per cent } K_2O \\ \text{in residue}\end{matrix} \times \frac{\text{Per cent residue}}{100} \times \begin{matrix}\text{Conversion factor, residue} \\ K_2O \text{ to feldspar } K_2O\end{matrix} \qquad (1)$$

For example, in the 50-20μ fraction of Ballinree soil (Table 3), initially containing 3.30 per cent K_2O, 72.2 per cent residue, and 2.95 per cent K_2O in the residue,

$$\text{Per cent feldspar } K_2O = 2.95 \times \frac{72.2}{100} \times 1.042$$
$$= 2.22$$

The mica K_2O content is obtained by subtracting the feldspar K_2O, from equation (1) from the total initial K_2O content,

$$\text{Mica } K_2O = \text{Total initial } K_2O - \text{Feldspar } K_2O \qquad (2)$$

TABLE 3. RESULTS OF ANALYSIS OF SOIL SILT FRACTIONS BY $Na_2S_2O_7$-HCl-NaOH PROCEDURE

Size fraction	Per cent K_2O	Per cent residue from treatment	Per cent K_2O in residue	Per cent feldspar K_2O	Per cent mica K_2O	Per cent K-feldspar	Per cent K-mica
Ballinree soil—C_1 horizon, 26+inches							
50-20μ	3.30	72.3	2.91				
		72.1	2.99				
		72.2	2.95	2.22	1.08	13.8	10.8
20-5μ	4.35	30.0	2.66				
		30.4	2.52				
		30.2	2.59	0.86	3.57	6.0	35.7
5-2μ	4.42	19.7					
		20.1	2.57				
		19.9	2.57	0.63	3.79	4.1	37.9
Leitrim Daub soil—C_{1_g} horizon, 4–24 inches							
50-20μ	0.46	95.5	0.22				
		94.8	0.19				
		95.2	0.21	0.21	0.25	1.3	2.5
20-5μ	0.48	92.5	0.18				
		91.2	0.16				
		91.9	0.17	0.17	0.33	1.1	3.3
5-2μ	0.35	91.5	0.11				
		89.5	0.09				
		90.5	0.10	0.11	0.24	0.7	2.4

For example, in the above mentioned fraction,

$$\text{Per cent mica } K_2O = 3.30 - 2.22$$
$$= 1.08$$

The per cent mica is obtained by multiplying the resultant per cent mica K_2O by ($100/K_2O$ content of mica). The factor for converting mica K_2O to percentage mica is approximately 10 for the fine grained micas (Table 4) which have been depleted of potassium on cleavage planes and usually have appreciable iron and (or) sodium content (Jackson, 1956, p. 543).

TABLE 4. APPLICATION OF THE $Na_2S_2O_7$-HCl-NaOH PROCEDURE TO MIXTURES OF MICROCLINE, MUSCOVITE AND BIOTITE OF KNOWN COMPOSITION

Composition of sample		Per cent residue	Per cent K_2O in residue	Per cent feldspar K_2O	Per cent mica K_2O	Per cent K feldspar	Per cent mica
Components and size fraction	Per cent K_2O						
43.5% Microcline[1] 20-5μ 50% Muscovite 20-5μ	11.8	a) 47.35	13.1	6.8	5.0	43.5	50
		b) 47.25	13.1	6.8	5.0	43.5	50
43.5% Microcline[1] 20-5μ 50% Biotite 20-5μ	11.5	a) 46.35	13.2	6.7	4.8	42.8	48
		b) 46.55	13.2	6.8	4.7	43.2	47
43.5% Microcline[1] 20-5μ 25% Muscovite 20-5μ 25% Biotite 20-5μ	11.7	a) 46.05	13.2	6.7	5.0	42.8	50
		b) 45.85	13.2	6.7	5.0	42.8	50
29% Microcline[2] 20-5μ 33.3% Muscovite 20-5μ 33.3% Biotite 20-5μ	11.0	a) 30.7	13.1	4.4	6.6	28.2	66
		b) 30.5	1.32	4.4	6.6	28.2	66
Muscovite, 20-5μ	10.0	0	—	—	—	0	100
Biotite, 20-5μ	9.4	0	—	—	—	0	100

[1] and 6.5% plagioclase.

[2] and 4.3% plagioclase.

a) and b) are duplicate determinations.

$$\text{Per cent mica} = \text{Per cent mica } K_2O \times 10 \tag{3}$$

In the above example,

$$\begin{aligned}\text{Per cent mica} &= 1.08 \times 10\\ &= 10.8\end{aligned}$$

The per cent K feldspar is obtained by multiplying the per cent K_2O in the residue by the per cent residue and by a factor for conversion to microcline of the appropriate size fraction (Table 2).

$$\begin{matrix}\text{Per cent}\\ \text{K feldspar}\end{matrix} = \begin{matrix}\text{Per cent } K_2O\\ \text{in residue}\end{matrix} \times \frac{\text{Per cent residue}}{100} \times \begin{matrix}\text{Conversion factor, residue}\\ K_2O \text{ to microcline}\end{matrix}$$

From the above example,

$$\begin{aligned}\text{Per cent K feldspar} &= 2.95 \times \frac{72.2}{100} \times 6.46\\ &= 13.8\end{aligned}$$

The results of application of the method to soil mineral fractions are presented in Table 3 and the effect of the treatment on the *x*-ray diffractograms of the samples is shown in Figs. 2 and 3. The results of analysis of known mixtures of feldspars and mica are presented in Table 4.

DISCUSSION

Micas and most other layer silicates are removed by the treatment, and quartz and feldspars are thereby concentrated so that they can be more easily identified by *x*-ray diffraction and determined quantitatively.

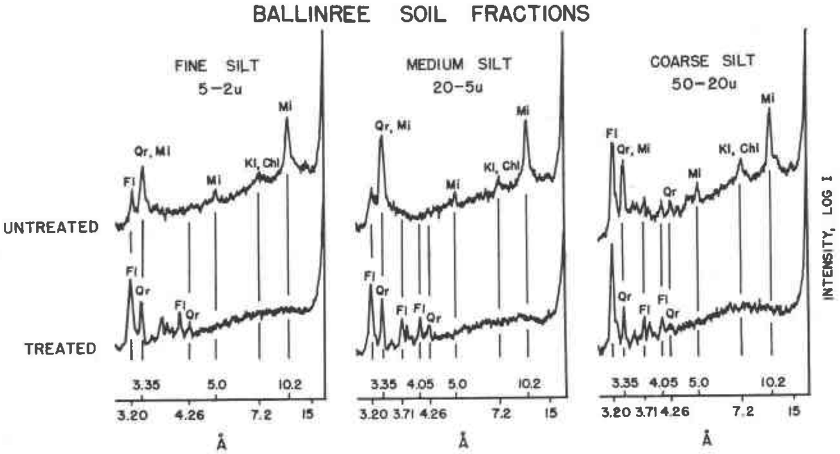

FIG. 2. *X*-ray diffractograms of Ballinree soil fractions before and after $Na_2S_2O_7$-HCl-NaOH treatment showing removal of mica by the treatment. The fractions were rich in plagioclase feldspars.

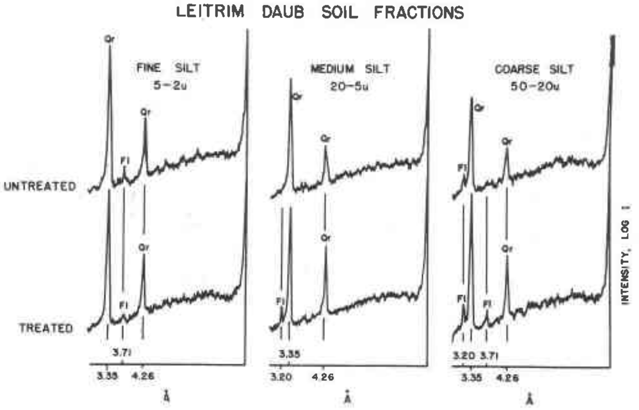

Fig. 3. X-ray diffractograms of Leitrim Daub soil fractions before and after $Na_2S_2O_7$-HCl-NaOH treatment. Na feldspar content was 7.5 per cent in the coarse silt, 6.3 per cent in the medium silt, and 3.2 per cent in the fine silt.

The method has been found to be extremely useful in the field of soil mineralogical analysis, particularly for silt fractions. It may also be applied to the sand fractions less than 500μ; however, optical methods are equally applicable in this size range. K feldspars are extensively dissolved in the coarse clay fraction. An estimate of microcline content in this fraction may, however, be obtained as seen in Table 2. Na feldspars may be determined in the residues by Na_2O determination and allocation to albite. The remaining residual mineral in soils is usually quartz and may be determined by subtraction of the feldspar content from the determined residue (Kiely and Jackson, 1965). Coarse clay quartz is relatively little attacked by the treatment and the method is accurate for quartz determination in this size fraction, even though feldspars can be determined only approximately in this fraction.

The precision of the method is very high in the coarser fractions (Table 2). In fractions greater than 5μ it is approximately ±2 per cent while in fractions less than 5μ it is approximately ±10 per cent.

The presence of other potassium bearing minerals of uncommon occurrence, e.g. leucite and nepheline, would interfere with this method for mica-feldspar differentiation. Use of x-ray diffraction analysis before and after the treatment is used to detect the presence of unusual minerals and thus to avoid introduction of this type of error.

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