various foods, in animals, and in man may be expected. Their mere detection is not necessarily cause for alarm. The important point is to be able to measure their levels in the biosphere in order to assure the safe development and use of nuclear energy (9).

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- 13 October 1959

Use of Long-Chain Polymers in **Preparation of Soil Extracts for Soil Testing**

Abstract. A method is described for the preparation of soil extracts for analysis which eliminates filtration. The procedure is based on the flocculating characteristics of very small amounts of polyacrylamides on inorganic and organic colloids. Amounts of plant nutrients extracted from soils by this method compare favorably with those obtained in filtered soil extracts.

The use of long-chain polymers (polyacrylamides) as flocculating agents in industrial processes has been described by Booth, Carpenter, and Hartjens (1) and by Linke and Booth (2). The application of this principle to soiltesting procedures appeared worthy of trial, in an effort to eliminate filtration in the preparation of soil extracts for the determination of inorganic nutrient elements available for plant growth.

Table 1. Comparison of results of analyses of soil extracts prepared by the method of the Pennsylvania Soil Testing Laboratory (PSTL) and by the new method (5).

Method	Item	P* (lb/acre)	K† (lb/acre)
PSTL	\overline{X}	11.3	162
New	\overline{X}	11.3	164
	t	0‡	1.050 [‡]
	df	28	65
	r	.9958§	.9920§

* N = 30. \dagger N = 67. \ddagger Not significant. nificant at the .001 level. § Sig-

Generally, extracts of soil samples are prepared by shaking the soil with an extracting solution or solvent, then filtering. A satisfactory extract is not always obtained by this method; filtering is often slow, and the resulting filtrate may be cloudy, thus interfering with subsequent analytical determinations.

Preliminary tests indicated that small amounts of commercially available long-chain polymers are highly effective in producing a clear supernatant extract of soils when the polymers are added to the usual extracting mediums. Under these conditions the flocculated soil samples would usually settle in 2 to 3 minutes, producing an extract superior to that obtained by filtration.

Tests with Superfloc 16 were good with soils high in clay, but a slight cloudiness resulted when the organic matter of the soil was high. Aerofloc 3171 gave the opposite results with these soils, but the use of equal amounts of the two polymers in the extracting solutions resulted in a highly satisfactory extract with both types of soils.

Numerous tests were made to determine the most satisfactory ratio of polymer to soil, and in general 0.5 mg to 5.0 gm of soil, or 1 part of polymer to 10,000 parts of soil, was found to give best results for routine work.

The procedure consisted first, of dissolving 0.25 gm of Superfloc 16 and an equal amount of Aerofloc 3171 in 500 ml of 0.753N sodium acetate solution adjusted to a pH of 4.8. The polymers dissolve in the acetate solution readily if they are mixed and slowly added to the acetate while it is being stirred by a mechanical stirrer. When all the polymers were dissolved, the solution was diluted to 1 lit. with the acetate. Then 416 ml of this solution was added to 20 lit. of 0.753N sodium acetate, and mixed. The resulting extracting solution was designated solution A and contained 0.25 mg of mixed polymers per 24 ml.

An additional solution was prepared by diluting 400 ml of the solution of mixed polymers to 800 ml with the 0.753N sodium acetate. This solution was designated solution B; 1 ml contained 0.25 mg of mixed polymers.

The soil extract was prepared by weighing or measuring 5 gm of a 20mesh, air-dry soil sample into a 50-ml Erlenmeyer flask containing 24 ml of the extracting solution A. The flask was shaken gently in a mechanical shaker for 5 minutes, 1 ml of solution B was added, and shaking was continued for exactly 30 seconds. The flask was removed from the shaker and allowed to stand a few minutes before aliquots for analysis were taken with an automatic pipet; care was exercised not to disturb the soil sample in the bottom of the flask when the sample was taken.

The soil sample must always be added to the extracting solutionnever the reverse.

When a cloudy extract was obtained in the procedure described above, 1 ml of solution B was added, and the mixture was shaken gently for a few seconds. A clear solution was usually obtained in this manner.

Extracts from a number of miscellaneous soils were prepared for analysis of phosphorus and potassium by the usual method in the Pennsylvania Soil Testing Laboratory and by the new polymer method. To increase the accuracy of the phosphorus determination. soil samples were weighed instead of measured. In all, 30 samples of soil with a phosphorus content ranging from 2 to $\overline{55}$ lb/acre were analyzed. A modified Deniges (3) procedure was used to develop color intensities, the colors being measured in a Bausch and Lomb No. 20 colorimeter, with matched tubes.

Potassium was determined in 67 soils containing from 30 to 325 lb of potassium per acre. The determination was carried out by means of a Perkin-Elmer flame photometer, with lithium chloride as an internal standard. The results are presented in Table 1, with appropriate statistical analysis.

The data included in Table 1 indicate that there is an extremely high correlation between the amounts of phosphorus and potassium found in the extracts prepared by these two procedures. Of particular interest is the fact that no phosphorus was occluded in the flocculated soil when the mixed polymers were added to the extracting solution. These results show that soil extracts prepared by this suggested procedure are as satisfactory for analysis as those prepared by filtering, and there are the added advantages that no filtering or refiltering is required, extracts are prepared more rapidly, and considerable cleaning of glassware is eliminated.

Tests were conducted with mixed polymers in distilled water, with normal neutral ammonium acetate, and with Bray's solution as extracting solutions, and satisfactory soil extracts were obtained, but no analyses of extracts were undertaken (4).

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- 5. The statistical data were prepared by Leon Johnson.

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Differential Thermal Study of Pyrosynthesis

Abstract. Equipment is described which provides a thermal record of the synthesis of sulfides and related minerals contained in easily constructed tubular glass vials. As an example, a temperature curve for the synthesis of galena (PbS) is given. Similarly derived temperatures of formation for eight other synthetic sulfides and selenides are reported.

The differential thermal analysis technique may be modified to provide information concerning the temperature at which crystallization takes place and at which other fundamental heat changes occur during mineral synthesis. A modification of equipment has been developed which provides a continuous differential record of exothermic and endothermic reactions as the temperature of a mixture of the components of a desired synthetic compound is increased at a linear rate. A record is obtained of the temperature, the magnitude, and the relative reaction rate of those physical-chemical processes which tend to give off or absorb heat. Among these processes are fusion, crystallization, chemical combination, dissociation, and the inversion of one structure to another. The equipment yields a continuous observable record during heating as well as cooling; this makes it possible to interrupt the cycle at any desired point for quenching, in order to study particular phases.

The apparatus (Fig. 1) consists of a thermal head containing Chromel-Alumel thermocouples and thermal wells which will accept sealed specimen vials of glass tubing 6 mm in diameter. A thermocouple is placed in a recess at one end of a tube, as shown in the sectional drawing. The recess is formed in the tube while one end still remains open and before the constituents are inserted. A dummy thermocouple is pressed against the end of the heated tube, forming a small depression into which an active thermocouple may be inserted.

The tubular sample vials are made of Pyrex, Vycor, or fused quartz, de-4 MARCH 1960 pending upon the terminal temperature required for the experiment. Each vial is evacuated by a mechanical vacuum pump during final sealing in order to guard against oxidation of the components. The space between the vials and the head metal is filled with 60-mesh Alundum in order to improve the thermal contact between the walls and the material in the vials. In this way, an attempt is made to provide an even, but still slightly incomplete, temperature flow between the thermal head and the constituents in the vial. Since the thermal conductivity of the Alundum is less than that of the metal head, the Alundum serves to prevent the mass of the head from masking the temperature effects produced in the vial. Although the thermal contact at the end of the tube between the thermocouple and the sample is relatively small, it has proved adequate to produce effective differential thermal records.

A similar thermal technique was described by Jensen (1) for the study of fusion points. However, the double tubes required in his work were found unnecessary in this study, and the fabrication of vials was simplified.

The instrumentation adopted was developed from differential thermal analysis equipment described by Kerr and Kulp (2) for work with clay minerals and related species. The major components consist of a 4000-watt electrical furnace; a program controller capable of maintaining a linear heating or cooling rate; a direct-current preamplifier for the differential thermocouples; and recorders for electromotive force and temperature. The program controller has a twoposition proportional section which provides a linear heating or cooling rate of 12.5° C/min from room temperature to 1050° C. A continuous furnace input produces a heating rate of approximately 28° C/min, with sufficient linearity for rapid testing of a sample.

Two ranges of preamplification provide a 5-mv deflection of the potential recorder, which has sensitivities of -5, 0, +5 mv. A low sensitivity of 0.5 mv is used on samples which have a large thermal reaction, and a high sensitivity of 0.25 mv is used on samples with a smaller reaction.

Figure 2 shows the formation of galena (PbS) by pyrosynthesis. An endothermic reaction appears at 110°C, which represents the melting point of sulfur, while an exothermic reaction shows initial deflection at 210°C. As the heat of the exothermic reaction is added to the temperature of the sample, the reaction proceeds at a faster rate and culminates in a peak deflection at 300°C. This point apparently represents the complete crystallization of galena, as indicated by supplemental x-ray studies. The return of the curve to the base line represents primarily the decay of the thermal gradient as the reaction is absorbed by the thermal mass of the head. In the synthesis of some minerals, final crystallization occurs during this period and is indicated by a comparatively slow return of the curve to the base line. The area of the curve and its configuration permit investigation of the chemical affinity of the constituents, relative bonding energy, amount of thermal energy released during crystallization, and rela-



Fig. 1. Thermal head and detail of specimen vial.