Multiple Thermal Analyses

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One of the most useful techniques in the study of clay minerals is differential thermal analysis, a procedure which can be used for both qualitative and semiquantitative analysis of many minerals. In this technique a two-headed thermocouple is employed. One head is inserted in an inert material which does not undergo either exothermic or endothermic reactions through the temperature interval to be studied; the other is placed in the mineral or mixture of minerals under test. With constant heating rate, any thermal reaction in the sample will be recorded as a "peak" or a "valley" in the otherwise straight-line curve, dependent on the nature of the heat change.

Thermal curves have recently been obtained from 6 samples simultaneously on one record sheet. On another sheet, driven by an electric motor synchronized with the multiple record motor, the temperature curve is drawn. The samples are mounted in a cylindrical resistance furnace. The pattern of the sample distribution in the vertical nickel block is shown in Fig. 1. The 6 samples to be tested are loaded in the outer holes, while the three inner holes are used for the inert material, purified alundum. The dotted lines indicate the connections between the two heads of the differential thermocouple. Dots "a" indicate the positions of the temperature-recording thermocouples. The heads of these couples are adjusted to the same height as the differential couples in the samples. The sample and alundum holes are 1/4 inch in diameter and 3/8 inch deep; the nickel block, 1 3/4 inch in diameter and 1 1/4 inch thick. A round cover of solid nickel, 1/4 inch thick, is placed on top of this specimen holder to shield the samples from direct radiation.

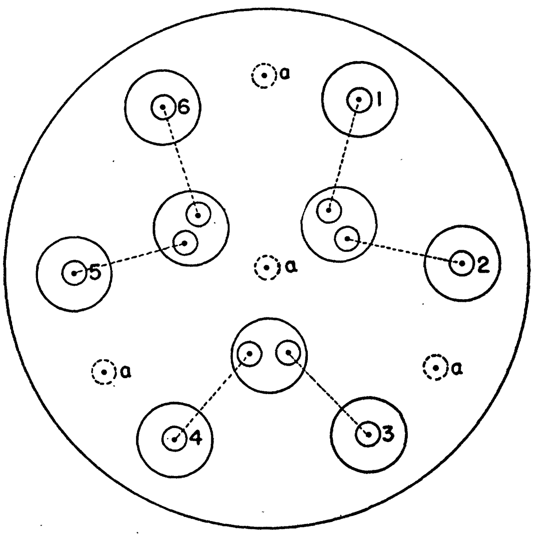

Fig. 1. Arrangement of samples in nickel block.

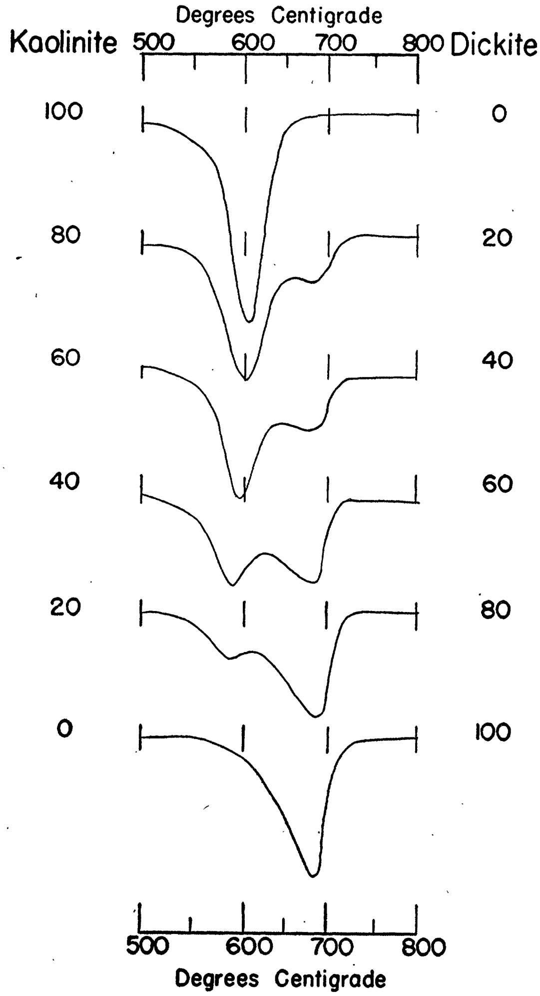

Fig. 2. Kaolinite-dickite mixtures.

The temperature thermocouple actuates a "micromax" recorder which is part of a program controller. A "speedomax," six-point, high-speed, electronic recorder with a maximum range of 3 mv is used to print the differential thermal curves.

Fig. 2 shows in a greatly reduced form a set of curves from one

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run for artificial mixtures of the clay minerals, kaolinite and dickite. Only the portions of the curves between 500° and 800° C. are shown in this diagram. The vertical coordinate for each curve indicates the relative intensity of endothermic reaction. The amplitude of the peaks is related to the percentage of the mineral present.

Two significant improvements in the technique of thermal analysis are evident with this type of apparatus. First, there is saving in time by running 6 instead of a single specimen. In an 8-hour day, 18 samples can be run conveniently. Second, there are certain inherent advantages in the simultaneous recording of 6 samples.

Discrepancy in Analysis of Penicillin in Blood by the Oxford Cup Method as Revealed by the Paper Disc Technique

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It is well known that low results are obtained when penicillin is assayed in the presence of blood by the Oxford cup procedure. We have found that this is not the case when the filter-paper disc technique is used. Under the same conditions, the latter method gives results close to the theoretical.

In the experiment to be described, three solutions were prepared by adding 0.5 ml. of concentrated penicillin in phosphate buffer to 4.5 ml. of the following blood fractions: defibrinated whole blood, oxalated blood, and serum. In subsequent dilutions for assay purposes, the ratio of the blood protein was maintained at a constant level of 90 per cent by using as diluent 90 per cent blood fraction and 10 per cent phosphate buffer. The resulting solutions were assayed against a standard solution of penicillin in 0.11 M phosphate buffer of pH 7.34. The technique described by de Beer and Sherwood (*2*) was used for the paper disc assays. The Oxford cup assays, performed simultaneously on the same solutions, employed glass cylinders (5.7–5.9 mm. inside diameter and 9.9–10.4 mm. high) as reservoirs. All other details, such as the agar medium, the *Bacillus subtilis* seed, the incubation, etc., were identical for both procedures.

TABLE 1

Percentage Penicillin Found by Assay

Blood preparation	Paper disc method	Oxford cup method
Defibrinated rabbit blood	97.6	33.5
Oxalated rabbit blood	92.0	39.0
Rabbit serum	89.5	66.6

The initial concentration of penicillin for each of the blood preparations was 320 units/ml. Oxalated blood was prepared by adding 2 mg. of $K_2C_2O_4 \cdot H_2O$ to each ml. of blood.

The results of a typical assay are given in Table 1.

It will be observed that in every instance the results obtained by the Oxford cup method were low, whereas the values by the paper disc method were comparatively satisfactory. The slight losses in the latter case possibly may be due to a destructive action of the blood upon the penicillin. Such an action has been demonstrated by Bigger (*1*) and confirmed by us. We have found that blood containing penicillin solutions, when allowed to stand for a week or 10 days at refrigerator temperatures, suffered losses as high as 60 per cent as revealed by the paper disc technique. Thick paper discs under these circumstances appeared to be less reliable than those cut from thin filter paper.

Similar discrepancies between the disc and the cup technique were observed when dog blood was used instead of rabbit blood. E. T. Reese, of the J. T. Baker Chemical Company (personal communication), also has found that the disc method gives higher results than the cup method on samples of fermentation medium, but that the methods are in agreement on samples of commercial penicillin.

References

1. Bigger, J. W. *Lancet*, 1944, **247**, 400–402.
2. de Beer, E. J., and Sherwood, M. B. *J. Bact.*, 1945, **50**, 459–467.

A Simple Method for Studying Friction

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A spring which obeys Hooke's law has one end fastened to a horizontal plane and the other end fastened to a body so that displacement of the body along the plane produces a horizontal restoring force in the spring. When there is sufficient displacement to produce a restoring force of greater magnitude than the maximum static frictional force between the body and the plane, and the displacing force is then removed, the spring will move the body back along the plane. The body will continue in this motion until the kinetic frictional force exerted on the body by the plane absorbs all of the kinetic energy given to the body by the restoring force of the spring.

If the kinetic frictional force above is the only force which absorbs energy while the block is moving under the spring's influence, the time rate at which the energy is absorbed will be the same as that at which the sum of the potential and kinetic energies is decreasing in the system, since this is a nonconservative system. Thus, $f\frac{dx}{dt} = \frac{d}{dt}(\frac{1}{2}mv^2 + V(x))$, where $V(x)$ is the potential energy of the spring and $\frac{1}{2}mv^2$ is the kinetic energy of the body. Since all of the kinetic energy is absorbed by the kinetic frictional force and since the restoring force of the spring is linear, the magnitude of the kinetic frictional force will be equal to the average of the restoring forces acting on the body while it is in motion after one given displacement. (Integrating both sides of the above equation for the interval $x_2 - x_1$, over which the body moves after a given displacement, since the velocity is zero at the beginning and end of the interval, gives $f \doteq \frac{1}{2}(F_1 + F_2)$, where F_1 and F_2 are the restoring forces of the elongations x_1 and x_2 of the spring.)

This offers a very simple classroom method for measuring kinetic friction when a block slides on a horizontal plane. A spring balance is used as the spring. The sliding frictional force