long, hollow, flexible needles (Fig. 1). The solvent used is a 1.2% aqueous solution of acetic acid; the concentration of nitroguanidine is 5 g/l, and the cooling time, from 95° C to 30° C is 5 hr, with no agitation. The resulting needles are not single crystals but appear to be aggregates of crystals.

Photomicrographs of cross sections made by embedding the needles in plaster of Paris or very hard wax and grinding thin sections perpendicular to the long geometric axis of the needle are shown in Fig. 2. From a study of a large number of cross sections it appears that the boundaries of the cavity are always parallel to the external faces of the aggregate, and that the respective numbers of faces are the same. That is, if the cross section of the aggregate is hexagonal, the cavity will be hexagonal; or, if the cross section is square, the cavity will also be square. A photomicrograph of the cross section of an incompletely formed hollow aggregate is shown in Fig. 3.

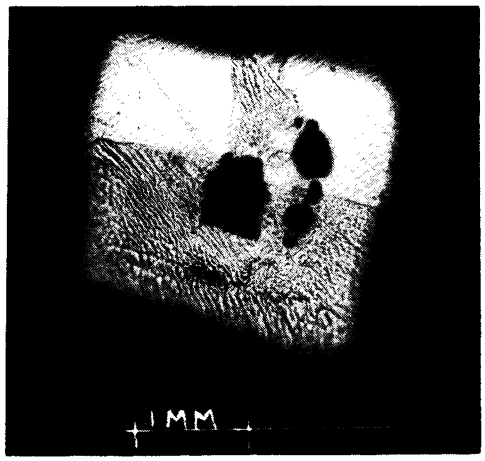

FIG. 4. Photomicrograph of the same cross section as shown in Fig. 2 on right, but after etching with water.

The presence of the cavity throughout the length of the aggregate is easily demonstrated in another way. If one end of the aggregate is dipped into an aqueous dye solution, the colored solution can be seen to ascend the bore of the aggregate, due to capillarity. Under the microscope a well-developed meniscus is readily seen. Frequently, the ends of the cavity are partially or completely closed because of crystallization, in which case it is necessary to trim the ends of the aggregate before the capillary can be revealed.

In any given crystallization under the conditions described, approximately 70%–80% of the needles will have well-defined cavities. If the crystallization is conducted from water rather than dilute acetic acid, the percentage of hollow aggregates is small (10%–15%). The influence of concentration or of the rate of cooling on this phenomenon has not been investigated. No effort was made to ascertain the minimum crystal size that still possesses the cavity; however, some aggregates with diameters as small as 0.2 mm have been hollow.

The formation of these hollow aggregates is probably due to a lengthwise contact twinning, involving two or three crystals. If the cross sections are carefully etched with water, groups of parallel striations appear, which are independent of the direction of grinding, and which appear to define the individual crystals composing the hollow needle (Fig. 4).

Cursory examination indicates that some of the long flexible needles of sublimed phthalic anhydride are also hollow.

Vacuum Differential Thermal Analysis[1]

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Differential thermal analysis is a technique for studying certain thermal characteristics usually of a solid complex substance. The procedure consists of heating the material under investigation at a constant rate to about 1000° C and recording the temperatures at which exothermic and endothermic reactions occur. In practice this is most readily accomplished by employing a metallic block with two holes, one of which is packed with the unknown substance, while the other is packed with an inert powder such as γ-alumina. A two-headed thermocouple, one head of which is imbedded in each powder, is then used to determine the direction and extent of the differential changes as the block is heated at a constant rate in a furnace.

During recent years differential thermal analysis has become a standard analytical procedure for the study of complex mixtures of minerals when resolution is difficult by conventional microscopic techniques or by x-ray methods. It has been especially useful in analysis of clays and other colloidal materials.

During the course of studies on coals and organic shales it became evident that any simple analysis or set of analyses leads only to generalized indication of the structure and stability of the gross organic material. Difficulty is enhanced by the fact that these organic substances, derived to a great extent from colloidal gels, appear homogeneous under the microscope. Differential thermal analysis has proved after trial (*1–3*) to be a promising technique for such studies, but certain difficulties were encountered in determining the thermal characteristics of the organic complexes on account of combustion of the specimens. For this reason a new instrument for differential thermal anlysis was designed to operate under vacuum or inert atmosphere. This paper describes such a vacuum installation, its furnace, and control equipment. A typical record is illustrated in Fig. 1.

Previous differential thermal analytical studies have been carried out with magnesia-packed furnaces, but the

[1] The research on marine shales was sponsored by the American Petroleum Institute, and that on coals by the Nova Scotia Research Foundation.

[2] The authors wish to acknowledge suggestions by Mr. Coutland Pearsall on furnace design, and the cooperation of Mr. John Solo in construction of the furnace.

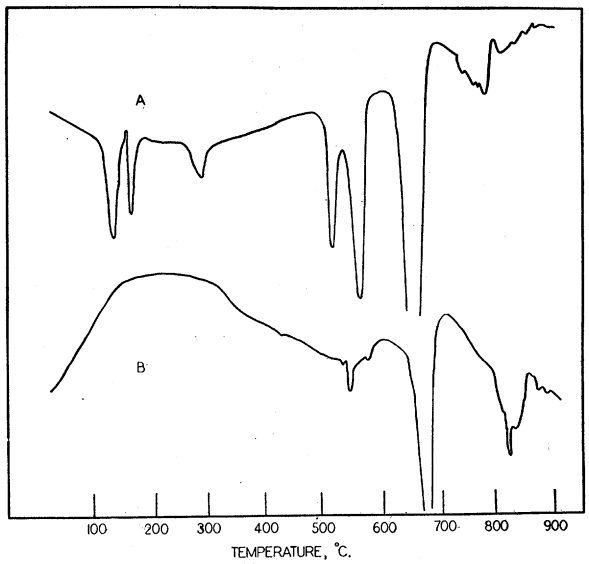

FIG. 1. Thermographic record of (A) pyritized coal from Greene County, Indiana (100 mg) and (B) pyrite (25 mg).

high thermal capacity of the packing has made it difficult to attain heating rates exceeding 10° or 12° C per min. For this reason, and because of the impracticability of attempting to evacuate a packed furnace, a vertical heating unit was designed with radiation shielding rather than conventional packing.

The furnace (Fig. 2) is constructed with a specially cast Alundum core 9 in. long, 2 in. inside diam, with a $\frac{1}{8}$ in. wall.[3]

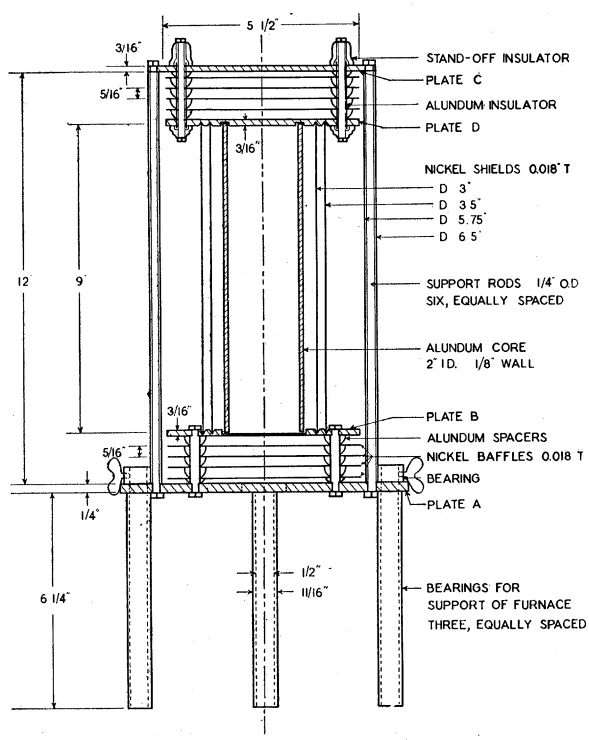

FIG. 2. Furnace used for thermographic analysis showing arrangement of radiation shields.

Sufficient 16-gauge Chromel-A wire is wound onto the core to make the power input approximately 2 kw at 110 v a-c. The core is shielded by four cylinders constructed of 0.018-in. sheet nickel. On top and bottom, shielding is by four disks made of the same material. Electrical connections within the furnace are made with stainless steel lugs and 1-mm platinum wire. For electrical insulation ceramic fish-spine beads are strung over the leads.

All steel parts of the furnace are machined from material obtained from the Rustless Iron and Steel Corporation, Baltimore, Maryland. Steel type 446, containing 23.00% to 30.00% chromium and a maximum of 1% nickel, and type 309, containing 22.00% to 26.00% chromium and 12.00% to 14.00% nickel, are used interchangeably, depending upon availability. These steels were chosen because of their resistance to scaling and to sulfur at high temperatures.

The furnace is mounted on three posts, 18 in. long, screwed into an 18 in. × 18 in. × $\frac{1}{2}$ in. brass plate. A groove $\frac{3}{16}$ in. deep and of width to accommodate an inverted 12 in. × 24 in. Pyrex jar is machined into the plate. To prevent failure of the glass jar, its flat bottom is rounded and then the entire jar is carefully annealed. By setting the plate on 2-in. adjustable legs and screwing and silver soldering a brass line into a hole through the plate, a means is afforded for complete evacuation of jar and furnace or for replacement of the air by an inert atmosphere such as helium. Electrical leads to the furnace and all thermocouple leads are insulated from the base plate by means of Kovar-to-glass seals obtained from the Stupakoff Ceramics and Manufacturing Company, Latrobe, Pennsylvania.

The block (Fig. 3) in which analysis is carried out is machined from type 446 or type 309 steel, has a diameter of 1 in. and has a length of $\frac{3}{4}$ in. Its lower end has a groove $\frac{1}{32}$ in. deep to enable the block to set firmly in place on a ceramic 1 in. outside diam × 12 in. furnace core which serves as a support post. This post is firmly fixed onto the base plate by means of a tightly fitting brass sleeve. The furnace bearings are of such length that when they rest on the brass plate, the heating block is exactly centered in the furnace.

The heating rate of the furnace is controlled from a Chromel-Alumel thermocouple junction placed between the center two windings of the spiral heating element. The output of the thermocouple is fed into a Leeds and Northrop Micromax controller equipped with an auxiliary device which enables variation of the heating rate of the furnace from 1° to 50° C per min. A heating rate of 12° C per min has been chosen for all comparative work in this laboratory. Additional control devices enable a constant cooling rate and "soak" of a specimen at a predetermined temperature.

Block temperature is measured by a Chromel-Alumel junction placed in the center hole of the block. The output of the thermocouple is recorded on a Brown recorder.

[3] Prof. F. Vinal of the M.I.T. Ceramics Division was instrumental in obtaining this core.

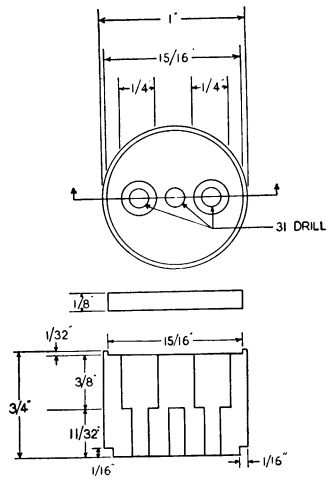

FIG. 3. Sample block.

The differential thermocouple is made from two leads of 28-gage Chromel wire connected by a short length of 28-gage Alumel wire. The fine wire is used in order to eliminate conduction of heat away from the specimen and the short length of connecting Alumel wire is used for the same reason. The output of the differential thermocouple is fed through a resistance box into a galvanometer having a short time constant. The position of a light beam reflected from the mirror of the galvanometer suspension is recorded on a Beckman Photocell Recorder (National Technical Laboratories, Pasadena, California).

For automatic operation of the installation, the control unit, recorders, and galvanometer light are wired through a Type T-27 General Electric time switch which may be adjusted to start the run and then to turn off the installation (see Fig. 4) after a preset time interval.

Samples of 25 mg to 100 mg of material, ground to pass a 200-gage screen, are being used for the analysis. For maximum effectiveness the samples are packed immediately around the thermocouple junction. Using specially designed tools, it is possible to pack the material consistently into the sample hole at 530 psi, while at the same time assuring horizontal and vertical centering of the thermocouple junction.

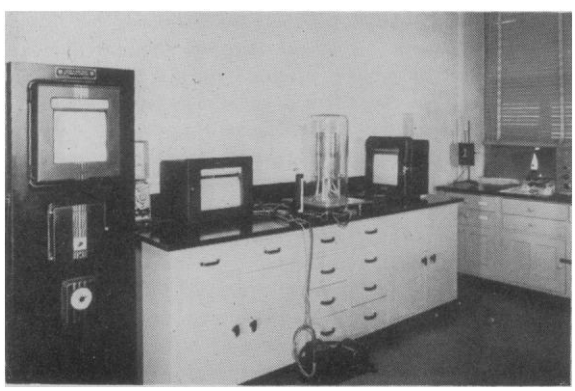

FIG. 4. Photograph of installation for thermographic anaylsis showing furnace, controller, recorder, galvanometer, time switch, vacuum gage, and pump.

The sensitivity of the differential recording installation is approximately ± 0.15 millivolt. This sensitivity can be increased by the substitution of a more sensitive galvanometer, a procedure that should enable the analysis of samples of smaller size.

During current operating practice with organic materials, the bell jar is evacuated to 1 mm of Hg and pump operation is continued throughout the experiment. The analysis of clays or inorganic samples can be made without the use of vacuum.

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Developmental Failure of the Pituitary in Amphibian Embryos Treated with Sugar

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A striking syndrome of abnormalities was obtained in embryos and larvae of the Pacific tree toad, *Hyla regilla*, by immersing late blastulae or beginning gastrulae, unremoved from the jelly, in a 10% solution of sugar (sucrose) for 14–20 hr at room temperature. Gastrulae so treated showed retardation of early gastrular movements and complete inhibition of gastrulation beyond the large yolk-plug stage. An exposure longer than 24 hr was usually lethal. Neurulae, developing from gastrulae returned to pond water, exhibited varying degrees of foreshortening of the archenteron and, correspondingly, of the medullary plate. Abnormalities frequently observed in the tail-bud stage included delayed formation and reduced size of the stomodeum, partial or complete fusion of nasal and sucker placodes, small irregular optic vesicles, and dorsal bending of the body. Larvae were characterized by the following features: albinism of the type caused by a deficiency of the melanosome-expanding hormone of the intermediate lobe of the pituitary; monorhina; reduction or complete absence of mouth parts; fused suckers; reduction in size and irregularities in form of eyes; partial or complete *situs inversus* of the gut; circular swimming movements owing to the dorsal bending of the body and tail; and little or no progress toward metamorphosis. All of the above abnormalities appeared in various combinations and to different degrees according to the length of treatment. Larvae developing from embryos immersed in the sugar solution for 14–16 hr showed mild symptoms; those exposed for 18–20 hr were highly abnormal. These observations have been made repeatedly and upon large numbers of animals. The cause of the anomalies has not yet been analyzed, but it would appear that because of the disturbance to gastrular movements—presumably an osmotic effect—the inductive role of the mesoderm was impaired,