DETERMINATION OF THE INITIAL AND FINAL SETS OF PLASTER OF PARIS.

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ABSTRACT.

This communication is part of an investigation of dental plasters begun in 1929 with the assistance of Doctor A. W. Faulkner.. It deals with the setting points of plaster. The method of determination adopted in place of those usually in vogue uses the variation in conductivity measurements at the initial and final sets. These are the low and high points distinctly marked on the conductivity curves. The paper gives a description of the apparatus used and a tentative explanation for the form of the curves derived during the investigation.

During the course of an experimental investigation of the physical properties of plaster of paris for dental purposes, considerable difficulty was encountered when an accurate determination of the "set" was attempted. Since, with suitable treatment, plaster may be made to set almost at will, it soon became evident that a better method than those advocated would have to be found.

The official method presented by the A. S. T. M. for plaster is the same as that used in testing cements, viz.—the use of the Vicat needle. This was totally inadequate for accurate work and was therefore discarded.

Another method[1] used in some of the Canadian plaster mills as an easy and simple test was then tried. It is as follows:—"A piece of common window glass about eight inches square and 3/32 inch thick is used. One hundred grammes of the sample are mixed with enough water to make a paste of testing consistency previously determined. The paste is spread on the glass plate and flattened into a pat about 7/16 inch thick. The set of the plaster is considered complete when it separates from the glass of its own accord when the latter is flexed by pressure of the hands on the opposite edges of the

[1] Cole. *The Gypsum Industry of Canada.* Page 79.

plate. Upward pressure is applied by the index finger of one hand and downward pressure by the base of the thumb. The thumb of the other hand exerts downward pressure while the heel of the same hand presses upward. The plate is inverted while it is being flexed, and the time of setting is taken as the lapsed time for adding the water to the sample to when he pat leaves the plate."

For testing building plasters such methods as the above are perfectly satisfactory but in making up dental impression plasters with initial sets of one to one and a half minutes, and final sets of three, four or five minutes a better technique is necessary.

A conductivity method was found to answer the requirements. Two new dry cells, a microameter reading from 0 to 500, a cell to hold the plaster, and a switch were hooked up in series. The plaster container was constructed of two strips of hard rubber two and a half centimeters wide and eight centimeters long which formed the sides of the cell. The ends of the cell were squares of thin copper plate two and a half centimeters on the side and located five centimeters apart between the rubber strips, which were then clamped together and placed on a sheet of glass which formed the bottom of the container. Wires were soldered to the copper plates for attachment in the circuit. When the cell was filled with the plaster sample the microameter readings came well up on the scale.

Time was reckoned from the beginning of the mix to the final set, the initial set being also registered. The data derived from some sixty tests of raw and prepared plasters were extremely interesting. Two curves of prepared quick setting plasters are seen in Fig. I. In these curves it will be noted there is a rapid fall of the current to the point which marks the initial set of the plaster. After an interval, which varies in length according to the type of plaster used, the current begins to rise until it nearly, but not quite, reaches its original value. When the current has reached this highest point the final set may be considered complete for all practical purposes

although hydration is still actively under way. The plaster is no longer soft or friable but breaks with a clean, sharp fracture. The expansion of the plaster is just beginning. After another interval the current again drops, much more

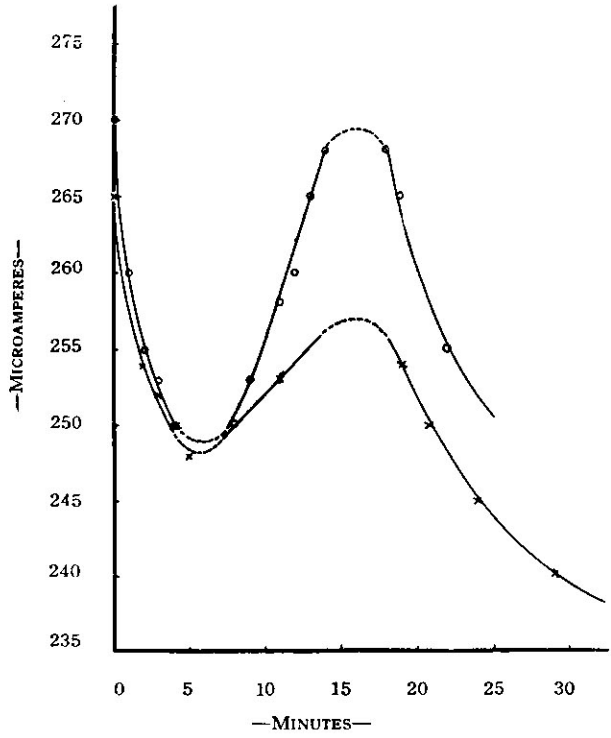

Fig. I.

slowly than before, and finally disappears completely as the insulator stage is reached. The time, as before, depends on the type of plaster under test.

It is difficult to explain the form of these curves, which, it may be said, is typical. A review of the literature indicates that the phase transformations of calcium sulphate and the properties of its dehydration products have remained a subject of controversy even to the present time. Possibly a plausible explanation may be found in the work of Davis[2].

[2] Davis, W. A. *Jour. Soc Chem. Ind.*, **26**, 727 (1907).

Davis has shown that when monoclinic gypsum is heated it first changes into a rhombic form before dehydration takes place. After dehydration the product is, of course, in the main the hemihydrate which is also rhombic in form. This reaction is strictly reversible. At this point it should be stated that van't Hoff and others have presented experimental evidence that before the expansion takes place in setting plaster a pronounced contraction occurs. Davis notes this fact and interprets it by showing that the preliminary contraction corresponds with the formation of the rhombic modification of gypsum, and the subsequent expansion with the conversion of the rhombic into monoclinic gypsum.

As a result of several comparative tests against the expansion apparatus it was found that the initial drop in the conductivity curve corresponds closely with the initial contraction in the expansion curve, or, as Davis puts it, to the change from the rhombic hemihydrate to the rhombic modification of gypsum. Why this change should be accompanied with such a sudden increase in the resistance of the plaster is a subject for further investigation.