

subjective evaluation of the reinforcing stimulus may provide an independent measure of the reinforcing value of a verbal reinforcer.

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References and Notes

1. J. Greenspoon, *Am. J. Psychol.* 68, 409 (1955).
2. This study was supported by an undergraduate research grant from the Social Science Research Council and by a grant from the Center for International Studies, Massachusetts Institute of Technology.
3. We would like to thank Sherman Tatz for the use of the questionnaire which he developed for a similar study.

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Influence of "Aging" on the Characteristics of an Electrodeless Discharge

"Aging" of a freshly prepared discharge tube—that is, maintaining the discharge for a period of time—at a constant applied potential V , has been found to affect the discharge characteristics markedly. The influence of aging on the conductivity of a low-frequency electrodeless discharge in iodine vapor is reported here. The details of the experimental set up are similar to those reported earlier by Saxena and me (2).

Aging decreases the discharge current i and the "threshold potential" V_m , namely, the potential at which the discharge becomes self-maintained. The conductivity decreases (i) very rapidly during the first few minutes, (ii) less rapidly during the next few minutes, and (iii) slowly until it attains saturation. Figure 1 shows a typical plot of the dis-

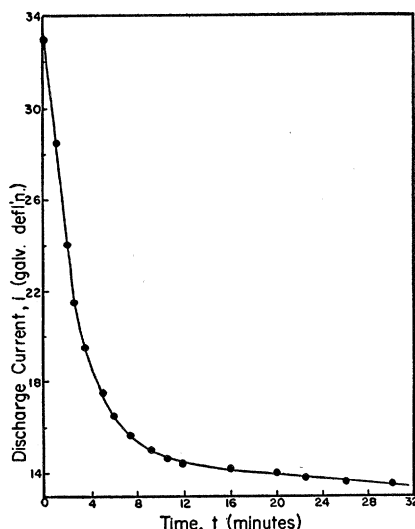


Fig. 1. Variation of discharge current with time.

28 SEPTEMBER 1956

charge current i versus time t in minutes. The equation proposed by Saxena *et al.* (3) ($\delta i = kt^{1/m}$) for similar observations in water vapor and iodine vapor under ozonizer discharge seems to hold good only during the afore-mentioned period (ii).

The decrease of the threshold potential on aging was observed even in the presence of excess of solid iodine and therefore cannot be ascribed to the pressure drop caused by adsorption of the vapor on the walls of the vessel. Aging reduces the width of the period of the discharge which is not self-maintained. In a particular experiment, the potential in the period that was not self-maintained was between 0.79 and 1.33 kv before aging, while it was between 0.79 and 1.06 kv after aging. Furthermore, aging is effective only when it is carried out at $V > V_m$; aging at $V < V_m$ has no appreciable effect on the discharge characteristics.

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References and Notes

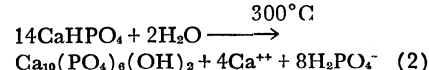
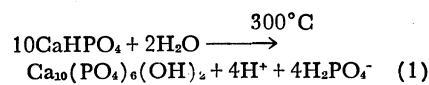
1. The work described was undertaken in the physicochemical laboratories of the Benares Hindu University, Benares, India. My thanks are due to S. S. Joshi for his kind interest in the work.
2. A. P. Saxena and C. N. Ramachandra Rao, *J. Sci. Research, Agra Univ.* 3, 207 (1954.)
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Preparation of Pure Hydroxyapatite Crystals

Naturally occurring hydroxyapatite exhibits a variable composition and contains impurities that have unknown effects on the structure and properties of the basic compound. A simple method for the production of pure, well-crystallized hydroxyapatite has long been needed. Methods of synthesis for this mineral have been reported in the past (1), but all of them produced impure and poorly crystallized products. This communication describes the preparation of pure hydroxyapatite suitable for x-ray diffraction, single-crystal studies.

The procedure is similar to an early preparation of hydroxyapatite by the hydrolysis of brushite ($\text{CaHPO}_4 \cdot 2\text{H}_2\text{O}$) (2). In the method outlined here, moneite (CaHPO_4), instead of brushite, is hydrolyzed to hydroxyapatite in a closed system. The reaction involved is probably one, or both, of the following.



An orthophosphoric acid solution (1 vol of reagent-grade 85-percent H_3PO_4 to 5 vol of distilled water) was saturated at room temperature with reagent-grade tribasic calcium phosphate. Well-crystallized CaHPO_4 was precipitated from the clear saturated solution by heating the solution nearly to its boiling temperature. The CaHPO_4 was filtered from the hot solution, washed thoroughly with distilled water, rinsed with absolute alcohol, and finally dried at 105°C . From 0.5 to 1.0 g of CaHPO_4 may be obtained from 100 to 150 ml of the saturated solution.

Pure well-crystallized $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$ was prepared from the reaction of 0.1 g of the CaHPO_4 with 10 ml of distilled water in a platinum-lined, Morey-Ingerson type hydrothermal bomb (3) at 300°C for 10 days. During this time, the system had an internal pressure of about 1250 lb/in.² owing to the vapor pressure of saturated steam at 300°C .

To obtain a complete reaction, it was necessary to use at least 10 ml of water for each 0.10 g of CaHPO_4 . Less than this ratio of water to CaHPO_4 resulted in a mixed product of $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$ and CaHPO_4 . Apparently the controlling factor for the hydrolysis is the final pH of the liquid. As long as this pH stays above 2.0 to 2.5, the reaction will proceed in the desired direction. Below a pH of about 2.0, the stable solid phase is CaHPO_4 for the reaction conditions used.

The presence of certain cation impurities can strongly influence the final product. During some preparations, the platinum lining developed cracks and Fe and Cr ions from the steel bomb were introduced into the water. When this happened, the final product contained a large proportion of well-crystallized whitlockite [$\beta\text{-Ca}_3(\text{PO}_4)_2$].

Under normal conditions, well-developed clear hexagonal dipyrnidal crystals of hydroxyapatite, which range up to about 0.3 mm in length, are produced. A spectrographic analysis showed the following amounts of impurities: 0.01–0.1 percent = Cu, Fe, Na, Pb, Si, Sr; 0.001–0.01 percent = Al, Ba, Cr, Mg, Ni, Pt; 0.0001–0.001 percent = Ag, Mn.

A petrographic examination showed the crystals to be uniaxial negative with indices of refraction: $\epsilon = 1.643 \pm 0.002$ and $\omega = 1.649 \pm 0.002$.

A method has been reported by Hayek, Lechtleiner, and Böhler (4) for obtaining well-crystallized hydroxyapatite by heating a finely divided hydroxyapatite with NaOH solution in a hydrothermal bomb. Although these investigators obtained well-formed crystals, the product was not as pure as might be desired, for these crystals contained at least 0.5 percent Na (5). Also, no attempt to remove the carbonate ion was made by Hayek and coworkers, and the influence of car-