

The average binding capacity of these samples was 8 percent. There was no evidence of a delayed response.

In order to show further that irradiation had prevented a primary antibody response, six rabbits from the irradiated, immunized group were challenged with antigen 26 days after primary injection. No secondary response occurred in these animals.

Based on the relative percentage of each fraction of the total protein, the globulin fractions of sera from irradiated, immunized or nonirradiated, immunized rabbits showed little change. It was only when these groups were compared with the nonimmunized, irradiated control that differences became apparent. Relative percentages of the combined gamma and beta globulin fractions are plotted in Fig. 2. A difference between irradiated controls and irradiated, immunized animals was evident the 8th day after irradiation. The average difference between these groups on the 10th and 11th days was statistically significant ($P < .001$). Globulin levels in the irradiated, immunized animals paralleled those of normal, immunized rabbits during the period when

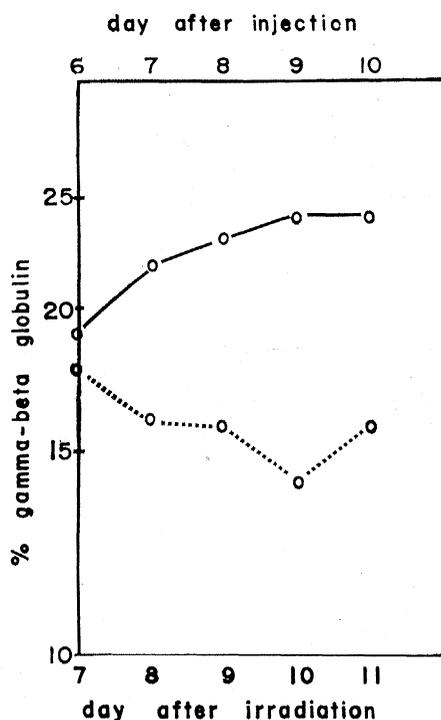


Fig. 2. Semilogarithmic plot of mean gamma-beta globulin levels in irradiated, immunized rabbits (solid line) and in irradiated, nonimmunized rabbits (dotted line). The values represent percentage of total serum protein. Minimum of ten rabbits per point. The immunized rabbits were injected 24 hours after x-irradiation.

sera from the latter contained antibodies to bovine serum albumin.

We conclude, therefore, that immunization was responsible for maintaining globulin levels in the irradiated animals. Since antibody was not detected we assume that the antigen-adjutant preparation injected (although containing 0.6 mg of DNA and 4 mg of rabbit hemoglobin) did not restore antibody formation as Taliaferro and Jaroslow (2) reported for depolymerized but not for polymerized DNA as used here. The very weak antigenicity of the adjuvant materials should eliminate these when the cause of the relative differences between the immunized and nonimmunized, irradiated rabbits is considered. The difference between the irradiated groups also indicates that permeability changes induced by radiation were not a factor.

The observed serum protein changes were slight since only a single injection of a small amount of antigen was employed. Nevertheless, antigen administration resulted in reversal of the expected loss in gamma-beta fraction and may be said to have exerted a protective effect on globulin levels. It is hoped that additional experiments with labeled amino acids will enable us to determine whether the synthesis of new (but non-antibody) globulin follows the immunization of irradiated rabbits as is suggested here.

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Four-Electrode Method for Measuring the Direct-Current Resistivity of Ice

Abstract. The improved system here described has been used successfully in systematic measurements of direct-current conductivity of ice doped with hydrofluoric and hydrochloric acids. The use of four electrodes allows continuous control of measurement reliability.

Direct-current-conductivity measurements are the basis for many other experimental techniques, such as studies of activation energy and the Hall effect. In the case of ice, however, such measurement is complicated by exceedingly pronounced electrode polarization effects.

It was thought possible that sandwich electrodes of a suitable type could be developed, in analogy to semiconductor techniques. Attempts to develop such electrodes were made by Mary Gourley of this laboratory as early as 1956 (1). The first truly usable sandwich electrode systems for ice were described and applied by Gränicher and his associates (2) and by C. Jaccard (3) of the ice research group at the Swiss Federal Institute of Technology. Jaccard's system consisted of polyvinyl chloride filter foils coated with gold under high vacuum and saturated with a dilute hydrofluoric acid solution. Some measurements on "pure" ice and ice samples made of a dilute hydrofluoric acid solution were published. No consistent results could be obtained with ice prepared from dilute hydrochloric acid (4) because an oxide film forms on a gold anode at the ice-electrode interface.

It was apparent that a broad experimental study of direct-current conductivities and activation energies of ice doped with different types of ionic impurities over a wide range of concentrations would fill a definite gap in ice research.

I have developed an improved four-electrode method that will work satisfactorily even with ice doped with hydrochloric acid. Separate circuits for measuring current and potential are used. Several hundred samples of ice doped with hydrofluoric and hydrochloric acids have been studied by means of this method. Concentrations ranged from about 10^{-6} to $3 \times 10^{-3}M$.

The samples are of cylindrical shape, with a diameter of 30 to 35 mm and height of 8 to 10 mm. On each end surface is placed a circular disk of acid-resistant Whatman filter paper No. 40

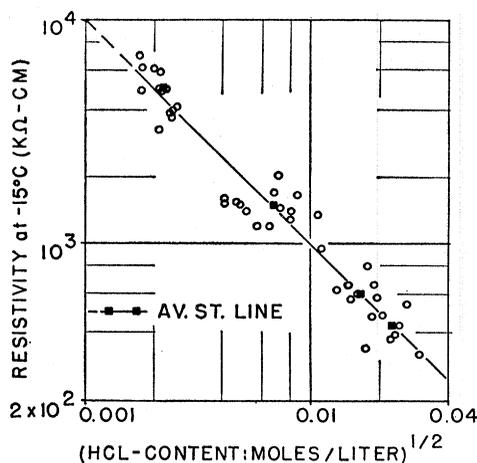


Fig. 1. Resistivities at -15°C plotted against the square roots of the concentrations for 43 ice samples doped with hydrochloric acid. The four points of the average straight line were computed by averaging melt concentrations and resistivities for each subgroup of samples prepared from the following four mother solutions: 0.01N, 0.005N, 0.001N, and 0.0001N.

or 41, on one side of which has been condensed an electrically coherent layer of finely divided palladium under vacuum, at pressure of about 10^{-3} mm-Hg. These disks, prior to use, are saturated in a solution of dilute hydrofluoric acid (at strength of about 100 to 1000 times the effective concentration of ionic impurities in the ice). Hydrofluoric acid is most suitable because it enters the ice structure easily and apparently does not stimulate anodic oxidation as actively as other acids do. A connection with the current circuit is provided by a circular electrode of spectrographic carbon, platinum, or palladium pressed against each palladium-coated disk. Care is taken to prevent an ice film from forming at this interface. An alternative method consists of growing ice samples on a clean palladium or platinum surface and using this undisturbed surface as the cathode, the anode being prepared in the manner described.

The potential electrodes are cross hairs made of 0.0126-inch or 0.02-inch platinum or palladium wire suspended in a circular Teflon frame and frozen into the sample during its preparation. Separation of these wires is 2.5 mm. The sample is thus divided into three sections of known thickness. The drop in potential across each section is measured by means of a Keithley electrometer, model 610. A comparison of these drops in potential suggests that the field-disturbing effect of the potential

electrodes may be neglected in the calculation of resistivities.

Samples were grown in a thin Teflon sleeve press-fitted onto a platinum or palladium disk about 5 mm thick. The solution to be frozen was degassed under vacuum (with a water aspirator). The cup was filled and frozen onto a refrigerated copper block. Average sample-growth velocity was about 1 mm/min. The freezing solution was hand-stirred with a Teflon rod. When the sample had reached the desired thickness the supernatant liquid was pipetted off and the free sample surface was rinsed with cold conductivity water. At once the palladium-coated paper disk and the upper current electrode were affixed, and the latter was weighted down with a weight of a few hundred grams until it was well frozen in. About 3 cm^3 of cold conductivity water were then added and frozen. The sample could then be taken out *in toto* and its lower surface could be frozen onto a similar electrode assemblage. After preparation, the samples were stored with shunted current electrodes at -15°C for at least 12 hours before measurements were made.

Currents in the range between 0.01 and 2 ma were obtained from batteries (floating circuit) or from an electronic power supply. Currents of several magnitudes were applied to a given sample, the magnitudes depending on the sample's resistivity; this, at -15°C , varies between 10^{10} ohm-cm for "pure" ice and a few hundred thousand ohm-cm for the most highly doped samples studied. If prepared properly, these electrodes showed a perfectly ohmic behavior and, equally important, insured a uniform potential gradient within the sample. Measurement procedures were carried out during as many as 3 hours before appreciable polarization occurred at the current electrodes. Reversal of polarity afforded an additional check on the reliability of results.

Current measurements were accurate to within 1 to 10 percent; those of potential drops to within about $\frac{1}{2}$ percent. Results from a series of measurements on a given sample with currents of the same magnitude or of different magnitudes (on the same day or on different days) placed the reproducibility roughly in a range between an average of ± 3.5 percent for samples of resistivity of 3×10^6 to 6×10^6 ohm-cm and an average of ± 10 percent for samples of resistivity of 3×10^5 to 6×10^5 ohm-cm.

The effective acid content of doped samples was determined by means of pH and conductivity measurements on the melt. The scatter shown in Fig. 1 is probably caused by two main factors: slight variations in the impurity-concentration gradient in different samples and the difficulty of avoiding some contamination of the melted sample prior to determination of its impurity content.

Measurements have been made at temperatures between -10° and -86°C in the experiments thus far completed.

The results indicate that the direct-current conductivity of ice at constant temperature is directly proportional to the square root of the acid concentration of the melted sample, as required by the mass-action law if conduction is due primarily to protons (see the "average straight line" of Fig. 1). Furthermore, the conductivity appears to be independent of the anion to a first approximation (5).

Note added in proof. After this report went to press, I learned of two papers (6) that describe such a series of experiments, carried out by a somewhat different method, for ice samples doped with several acids, bases, and salts. In particular, their results for HCl-doped ice agree well with those shown in Fig. 1.

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Natural Occurrence of Amino Acids in Virgin Crocidolite Asbestos and Banded Ironstone

The detection of fluorescent oils containing polycyclic aromatic hydrocarbons and amino acids in crocidolite and amosite asbestos but not in chrysotile has been reported by Harington (1). The present report provides further details about the amino acids found in natural association with virgin crocidolite asbestos taken from the North-