

## The Separation of the Isotopes He3 and He4 in the Liquid Phase

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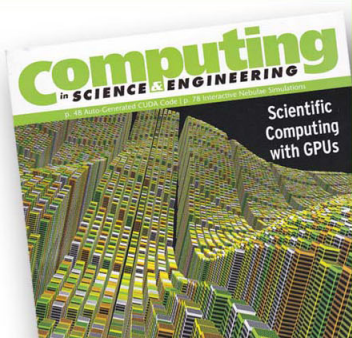
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## Letters to the Editor

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### Raman Tube Filters

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September 4, 1947

**S**ANNIE, Amy, and Paresky<sup>1</sup> have studied the use of several materials, including nitrobenzene and rhodamine 5G extra in alcohol, for the isolation of the 5358 mercury line as a source of Raman excitation. A modification of this procedure by Edsall and Wilson<sup>2</sup> consisted of 2 percent *p*-nitrotoluene and 1:50000 rhodamine 5 GDN extra (duPont) in alcoholic solution. Crawford and Horwitz<sup>3</sup> replaced the *p*-nitrotoluene by a Wratten 2-A gelatin filter wrapped around the Raman tube.

We have used the Raman apparatus described by Glockler and Tung<sup>4</sup> except that the polaroid has been omitted. One of the Raman tubes was provided with the filter under observation, while the other tube had no filter, and since the exposures were simultaneous, a direct comparison was obtained. The circular arrangement of the twelve General Electric A-H2 mercury vapor lamps made it desirable that we develop cylindrical plastic filters. Various plastic dopes were centrifugally cast on the inside of glass or methacrylate tubing of slightly larger diameter than that of the Raman tube. Methyl methacrylate molding powder dissolved in acetic acid and acetic anhydride, or in chloroform with a small amount of amyl acetate has been found convenient for use with rhodamine 5 GDN extra. After the dye or other absorbing material had been dissolved in the solvent, the molding powder was added. A number of coats of the dope were applied until the desired absorption characteristics were obtained. This procedure was found unsatisfactory when *p*-nitrotoluene was employed because the necessarily high concentration caused it to crystallize out from the polymer. Modifying the procedure used by Shenk, *et al.*<sup>5</sup> for infra-red filters, we found that 15 percent *p*-nitrotoluene solutions could be satisfactorily set. In view of its 90 percent transmission in the region 3800–6200, Beetle 230–8,<sup>6</sup> an unmodified urea-melamine-formaldehyde resin, was selected as the base. To prevent cracking and peeling it was plasticized with Rezyl 350–5,<sup>6</sup> an oxidizing-type phthalic alkyd. A suitable dope consisted of approximately 63 percent Beetle, 20 percent

Rezyl, 15 percent *p*-nitrotoluene, 1.5 percent Beckamine accelerator,<sup>7</sup> 0.5 percent Gui-A-Phene<sup>8</sup> antiskinning agent, and the desired amount of rhodamine. Using the latter plastic, we have applied up to 20 coats, and obtained thicknesses of about 1.6 mm for the finished filters. Such filters transmit up to 65 percent of 4358 and less than 10 percent of 4047, with very little continuous background on a benzene spectrum.

In order to reduce the number of reflecting surfaces, we have coated the Raman tube directly with the filter dope. The above techniques were used, except that the Raman tube was dipped or sprayed, and then placed on a slowly rotating spindle until the solvent had evaporated. Heat was applied to the thermo-setting resins by means of an infra-red lamp. 140 hours of direct exposure to a General Electric A-H2 produced no significant change in the transmission of these filters.

In addition, we have satisfactorily removed 4047 by means of a plastic dope furnished by General Aniline and Film Corporation, and also by using an Eastman 2-A gelatin filter. For the removal of continuous background, an Eastman X-3225 gelatin filter produced results similar to those obtained from solutions or plastic filters into which rhodamine 5 GDN extra had been incorporated.

We have found that plastic filters are not only more convenient when applied directly to the Raman tube, but also eliminate some of the extra reflecting surfaces created by several liquid filter jackets or separate solid filters, and they may prove to be more stable than solutions.

<sup>1</sup> C. Sannie, L. Amy, and V. Paresky, *Bull. Soc. Chem. France* **3**, 2018 (1936).

<sup>2</sup> J. T. Edsall and E. B. Wilson, Jr., *J. Chem. Phys.* **6**, 124 (1938).

<sup>3</sup> Bryce L. Crawford, Jr. and William Horwitz, *J. Chem. Phys.* **15**, 268 (1947).

<sup>4</sup> G. Glockler and Jo-Yun Tung, *J. Chem. Phys.* **15**, 112 (1947).

<sup>5</sup> John H. Shenk, Edwin S. Hodge, Robert J. Morris, Edward E. Pickett, and Wallace R. Brode, *J. Opt. Soc. Am.* **36**, 569 (1946).

<sup>6</sup> American Cyanamid and Chemical Corporation.

<sup>7</sup> Reichhold Chemicals, Inc.

<sup>8</sup> Southern Pine Chemical Company.

### The Separation of the Isotopes He<sup>3</sup> and He<sup>4</sup> in the Liquid Phase\*

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August 11, 1947

**A** PARTIAL concentration of the rare isotope He<sup>3</sup> present in atmospheric helium<sup>1</sup> with an abundance ratio<sup>2</sup> of  $1.22 \times 10^{-6}$  has been effected by a method involving the superfluid flow of liquid helium II. We have previously reported<sup>3</sup> that the transference of liquid helium II from one reservoir to another by means of superfluid flow through a supra-surface film effectively filters out the isotope He<sup>3</sup>, the superflow through films therefore being a process in which, at the temperature employed, He<sup>3</sup> atoms do not partake, as has been suggested by Franck.<sup>4</sup> Since these experiments<sup>3</sup> were restricted to flow through supra-surface films, it was of interest to observe whether a similar filtration takes place for superfluid flow through narrow channels in the bulk liquid. The results obtained are given below.

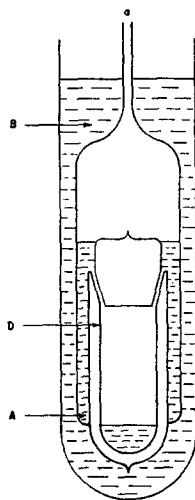


FIG. 1.

The experimental arrangement is illustrated in Fig. 1. The vessel *B* represents the main helium bath. Immersed in *B* is the vessel *A*, of which a small Dewar, *D*, formed an integral part. The top of *D* was closed off by an evacuated glass plug ground to fit the Dewar. Atmospheric helium<sup>6</sup> could be condensed in *A*, via the tube *a*. It was found that at 4.2°K the flow of helium through the ground joint at the top of *D* was negligible, even though the level of the liquid in *A* was well above the ground joint. From estimates of this minute flow, the channel width of the ground joint was assessed at 1 micron.

When the whole arrangement was cooled below the lambda-point, the liquid flowed rapidly from *A* into *D*, at 1.5°K the rate being 1.25 cc per minute, until the level in *A* fell just below the rim of the ground joint. From then on liquid would have flowed into *D* through the supra-surface film at a rate of approximately  $10^{-2}$  cc per minute. The liquid remaining in *A* was evaporated at this stage, and subsequently the liquid in *D* was evaporated and its vapor collected separately.

The abundance ratio of helium isotope He<sup>3</sup> in the vapor collected during two different runs was kindly measured for us by Professor Alfred O. Nier and Mr. L. T. Aldrich<sup>2</sup> to whom we wish to express our appreciation of their cooperation. The He<sup>3</sup>/He<sup>4</sup> ratio for *A* was  $1.85 \times 10^{-6}$  and  $1.55 \times 10^{-6}$  for the two runs, this increase over the normal concentration for atmospheric helium<sup>2</sup> depending on the initial volume condensed. For the vapor from *B* the average values were  $0.37 \times 10^{-6}$  and  $0.35 \times 10^{-6}$ .

One may conclude that the He<sup>3</sup> was at least partially filtered out by flow through the channel. It was difficult to estimate how complete this filtration might have been since in cooling from 4.2°K some liquid was condensed from the vapor already present in *D*. Calculation of this effect indicates that the filtration was almost complete, and certainly much more effective than might be concluded from direct calculation from the figures given above. Experiments to determine this effect more exactly are being pursued and will be given in detail elsewhere.

The method of filtration of the isotope He<sup>3</sup> by superfluid flow in the liquid phase, whether through supra-surface films<sup>3</sup> or through narrow channels in the bulk liquid, is one which, apart from its theoretical interest,<sup>4,5</sup> may lead to an isolation of the rare isotope in sufficient quantities for many applications. Such work is now in progress.

\* This work was assisted by Navy Contract No. N6onr-225.T.O.III between the Office of Naval Research and The Ohio State Research Foundation.

<sup>1</sup> L. W. Alvarez and R. Cornog, Phys. Rev. **56**, 379, 613 (1939).

<sup>2</sup> L. T. Aldrich and A. O. Nier, Phys. Rev. **70**, 983 (1946); see also H. A. Fairbank, C. T. Lane, L. T. Aldrich, and A. O. Nier, Phys. Rev. **71**, 911 (1947).

<sup>3</sup> J. G. Daunt, R. E. Probst, H. L. Johnston, L. T. Aldrich, and A. O. Nier, Phys. Rev. **72**, 502 (1947).

<sup>4</sup> J. Franck, Phys. Rev. **70**, 561 (1946).

<sup>5</sup> We are grateful to Linde Air Products for kindly providing large quantities of atmospheric helium.

## The Time Effect in the Fracture of Glass and Other Materials

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August 11, 1947

ONE of the important problems in the mechanical properties of glass as well as of other substances is that of the delayed time to fracture. Specimens which do not fail during the time it takes a load to build up to its maximum value may subsequently fracture under the steady application of the load even in the absence of the chemical effects of the atmosphere, which will be the case considered here.

Considerable data have been accumulated and a variety of methods of plotting functions of fracture stress against functions of time have been advanced in the hope that one of them would yield a straight-line relationship and thereby disclose some rational explanation of the phenomenon. Most of the methods of plotting seem to show about equally good approximations to a straight line. This may be because of the experimental errors involved as well as the limited extent of the data.

The author proposes to treat the problem from the point of view of chemical-reaction rate theory in which the breaking of bonds between ions or atoms is the product of the reaction. Although this method has been used before,<sup>1</sup> no account has been taken of either the rate at which the external loads are applied or of the effect of the breaking of bonds upon the remaining ones. The present treatment is an attempt to take both cases into account.

In the usual notation,<sup>2</sup> the net number of jumps of the flow unit per unit time (rate at which bonds are broken) in the presence of a potential gradient is given by:

$$\frac{dN}{dt} = \frac{2kT}{h} e^{-\frac{\Delta F^*}{RT}} \sinh \frac{w}{kT}, \quad (1)$$

where *N* is the number of bonds broken and *w* is the work done per flow unit in the process. The latter may be related to the force, *f*, necessary to break a bond through  $w = f(\lambda/2)$ , where  $\lambda$  is some distance through which *f* must act to break the bond.

Equation (1) may be written:

$$dN/dt = A \sinh Bf, \quad (2)$$