

August 1969

Brief 69-10241



# AEC-NASA TECH BRIEF



AEC-NASA Tech Briefs describe innovations resulting from the research and development program of the U.S. AEC or from AEC-NASA interagency efforts. They are issued to encourage commercial application. Tech Briefs are published by NASA and may be purchased, at 15 cents each, from the Clearinghouse for Federal Scientific and Technical Information, Springfield, Virginia 22151.

## Zone Purification of Potassium Chloride

### The problem:

Development of a direct procedure for purifying untreated KCl and removing two common interferences: Na and Br. Color-center investigations in alkali halides frequently require a controlled composition with respect to foreign alkali-metal ions and foreign halogen ions. These ions, occurring in unknown concentrations, interfere with optical, kinetic, and spin-resonance measurements.

### The solution:

A simple procedure for rapid removal and quantitative estimation of Na and Br in KCl. The procedure for purifying untreated stock-bottle KCl involves zone refining in a dilute halogen atmosphere. The distribution of Na and Br at concentrations of parts per million is followed by neutron-activation analyses. Computer-analyzed distributions in refined ingots yield  $K_{Na} = 0.53 \pm 0.03$  and  $K_{Br} = 0.72 \pm 0.02$ .

### How it's done:

For the zone melting of KCl in a controlled atmosphere a boat of trapezoidal cross section with 5° tapering sides was fabricated from quartz. The boat slipped with minimum clearance into a quartz tube, 26 mm in bore, that was coupled to a vacuum and gas handling system.

Reagent-grade KCl served as a starting charge. The loaded boat was inserted into the quartz environment tube, and the system was evacuated to  $1 \times 10^{-6}$  torr. Ambient temperatures were increased from 20° to 420°C over a 24-hour period during which the system pressure did not exceed  $1 \times 10^{-4}$  torr. Next a gaseous mixture of 2% HCl in 98% prepurified argon was passed over activated charcoal at dry-ice temperature and introduced into the environment tube. With the

ambient temperature maintained at 420°C, the system was evacuated and backfilled twice. The powder charge was then allowed to remain in contact with the static halogen atmosphere for 18 hours at 420°C.

Controlled dehydration and halide treatment are required to reduce hydroxide contamination in the KCl melt. Ingot adhesion to the quartz boat walls is prevented; thus boat breakage is obviated during early passages of the molten zone. A 2% mixture of the halogen in an inert gas accomplishes this without the bubble formation attendant on zone melting in a pure halogen atmosphere.

After a final backfill cycle, the dilute halide gas was permitted to flow over the boat at  $1 \text{ cm}^3/\text{min}$  in the direction of slow zone travel. Two molten zones were simultaneously passed through the 17-inch-long ingot at 2.0 inch/hr with an ambient temperature maintained at 540°C. The ratio of ingot length to molten-zone length was varied from 6.8:1 to 9:1.

Quantitative estimations of concentrations (ppm) of Na and Br in a KCl matrix were readily obtained by neutron-activation techniques. Solid chips weighing from 5 to 10 mg were irradiated for 72 hours at  $10^{13}$  neutrons per square centimeter per second (thermal). Possible surface contamination was removed by immersion of the irradiated chips in methanol. Separation of 15-hour (half life)  $\text{Na}^{24}$  from 34-hour  $\text{Br}^{82}$ , and removal of the 12-hour  $\text{K}^{42}$  background, proceeded on a 2-mm bore, by 10-cm long, column of zirconium tungstate. The KCl chip was placed atop the resin bed, and the column's tip was plugged temporarily. The sample was completely dissolved in a minimum amount of 0.01M  $\text{NH}_4\text{Cl}$  containing  $\text{Na}^{22}$  tracer. Less than 5 ml of 0.01M  $\text{NH}_4\text{Cl}$  was needed for complete elution of  $\text{Br}^{82}$ . After change in

the eluant concentration to 0.1M NH<sub>4</sub>Cl, it was possible to elute Na<sup>22,24</sup> with 10 ml of eluant with more than 95% recovery of the tracer.

Counting was done with a 3 by 3-inch NaI detector. At Na and Br concentrations of 10 µg/g, the standard deviation is ± 4%. The absolute error is estimated at no more than 10% on the basis of comparison with colorimetric and flame-photometric analyses at Na and Br concentrations higher by one to two orders of magnitude.

**References:**

For more details see S. Susman, *J. Chem. Phys.* 47(1), 83 (July 1967).

**Note:**

Inquiries may be directed to:  
Office of Industrial Cooperation  
Argonne National Laboratory  
9700 South Cass Avenue  
Argonne, Illinois 60439  
Reference: B69-10241

Source: S. Susman  
Solid State Science Division  
(ARG-10377)

**Patent status:**

Inquiries concerning rights for commercial use of this innovation may be made to:

Mr. George H. Lee, Chief  
Chicago Patent Group  
U.S. Atomic Energy Commission  
Chicago Operations Office  
9800 South Cass Avenue  
Argonne, Illinois 60439