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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 1×10^{-6} torr. Ambient temperatures were increased from 20° to 420°C over a 24-hour period during which the system pressure did not exceed 1×10^{-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) Na^{24} from 34-hour Br^{82} , and removal of the 12-hour 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 NH_4Cl containing Na^{22} tracer. Less than 5 ml of 0.01M NH_4Cl was needed for complete elution of Br^{82} . After change in

the eluant concentration to 0.1M NH₄Cl, it was possible to elute Na^{22,24} 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 colorometric 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:

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Patent status:

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