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DISSOLUTION OF NaK

BY

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DISSOLUTION OF NaK

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ANALYTICAL CHEMISTRY DIVISION

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ABSTRACT

A rapid yet safe method for the dissolution of sodium-potassium alloys prior to chemical or instrumental analysis has been devised.

The alloy is submerged beneath the surface of an inert organic reagent and dissolved at a controlled rate by adding methanol drop-by-drop. A blanket of inert gas is maintained over the reaction flask.

The procedure has been successfully used to dissolve samples of NaK in the range of 0.05 to 25 grams.

[redacted]

DISSOLUTION OF NaK

Dissolution of sodium-potassium alloys prior to chemical or instrumental analysis is an extremely hazardous operation because of the great reactivity of NaK with common aqueous and organic reagents. Water reacts exothermally with the alloy with explosive violence and is obviously not recommended in any form or concentration for use with NaK. The use of organic reagents for dissolution is generally resorted to in such a situation. The rate of reaction of NaK with the lower alcohols is only slightly less than that with water, and the inflammability of these reagents presents an additional hazard. Those organic reagents, such as higher alcohol, which react fairly slowly with NaK are unsatisfactory since they produce by this reaction, alkali metal alcoholates which being slightly soluble in the alcoholic medium covers the NaK with a protective coating and retards the rate of dissolution. In general, the problem is to use a reactive solvent which yields a controllable reaction with NaK.

The conditions necessary for a rapid yet safe dissolution of NaK are essentially these:

(1) Exclusion of oxygen.

(2) An inert liquid, the specific gravity of which is less than that of NaK.

(3) Controlled addition of reactive reagent.

The complete exclusion of oxygen obviously eliminates the possibility of combustion. Submersion of the alloy beneath the surface of an inert liquid serves to prevent contact with air; the liquid also provides a medium in which dissolution can be conducted and the heat of reaction dissipated. Cautious addition of the solvent is necessary in order to control the rate of reaction.

An inert gas-filled dry box affords an oxygen-free atmosphere. In such an apparatus, methyl alcohol can be used to dissolve the NaK directly without an inert-liquid solvent medium. If an adequate dry box is available, this technique is probably the most satisfactory method known for dissolving NaK.

Since such a box is not always available, a procedure has been successfully formulated which embodies the principles outlined above. The sample of alloy, usually sealed in a glass container (metal containers, sealed with an organic wax, are often used), is placed in a beaker containing n-hexane to cover the sample completely. The n-hexane, which boils in the range of 60-65° C., is less dense than NaK, which therefore rests on the bottom of the beaker, and is, of course, inert to NaK at the temperatures involved. A funnel is inverted over the beaker (as shown in the accompanying figure) and a blanket of helium or argon provided over the reaction. Methyl or ethyl alcohol is added drop-wise, 2 to 3 drops per addition, until the reaction is complete. The alcoholate formed

is appreciably soluble in this medium and does not coat the unreacted NaK to any appreciable extent. Intermittent stirring with a glass rod eliminates any tendency of the formation of a coating. The dissolution is complete in a relatively short time without any noticeable increase in temperature of the reaction medium. When the reaction is complete, as noted by the absence of any unreacted NaK, an excess of alcohol is added as a safety measure. Water is then added to dissolve the alcoholate that has been formed. The two phases are then transferred to a separatory funnel and the heavier water layer separated.

If required, an aliquot of this phase may be titrated with standard acid for determination of sample weight.

This procedure has been successfully used to dissolve samples of NaK in the range of 0.05 to 25 grams.

Other precautions which are strongly recommended include, the use of plastic shield in front of the reaction vessel and the wearing of gloves by the operator.

Special care must be exercised when handling NaK that is heavily contaminated with oxides. Potassium is known to form the super-oxide, KO_2, which is the stable oxide at room temperature, in the presence of normal partial pressures of oxygen. Gilbert[1] states that the rather frequent explosions, which occur in handling potassium, can be attributed to the formation of super-oxide on the surface of the metal at room temperature. The cause of this explosion is not completely understood.

Such an explosion has been observed in this laboratory with a heavily oxide coated sample of NaK. The use of a dry box is apparently mandatory when dealing with such samples.

REFERENCE

(1) Gilbert, Chem. Eng. News 26, 2604 (1948)

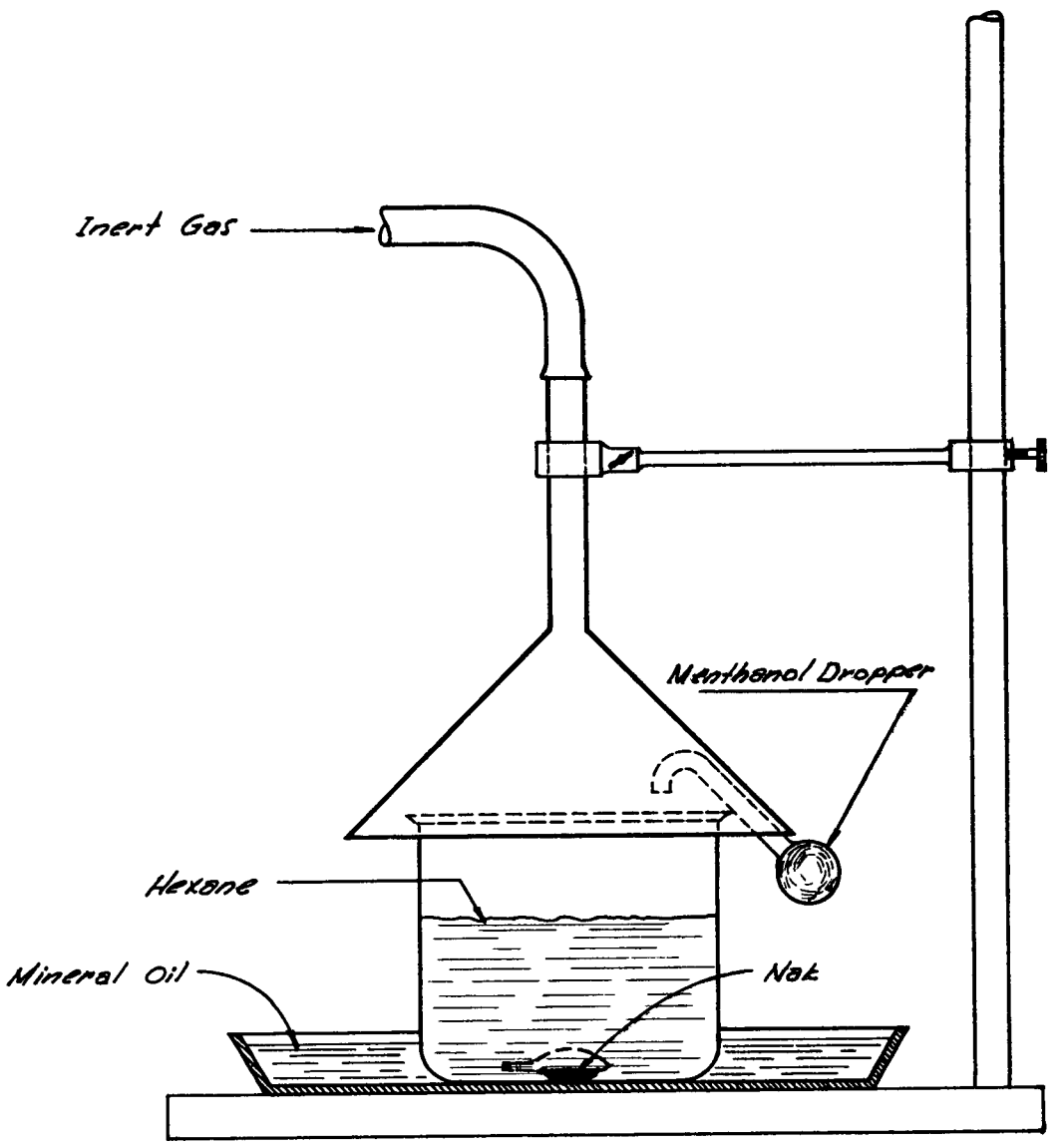

FIGURE I.

APPARATUS FOR DISSOLUTION OF NAK