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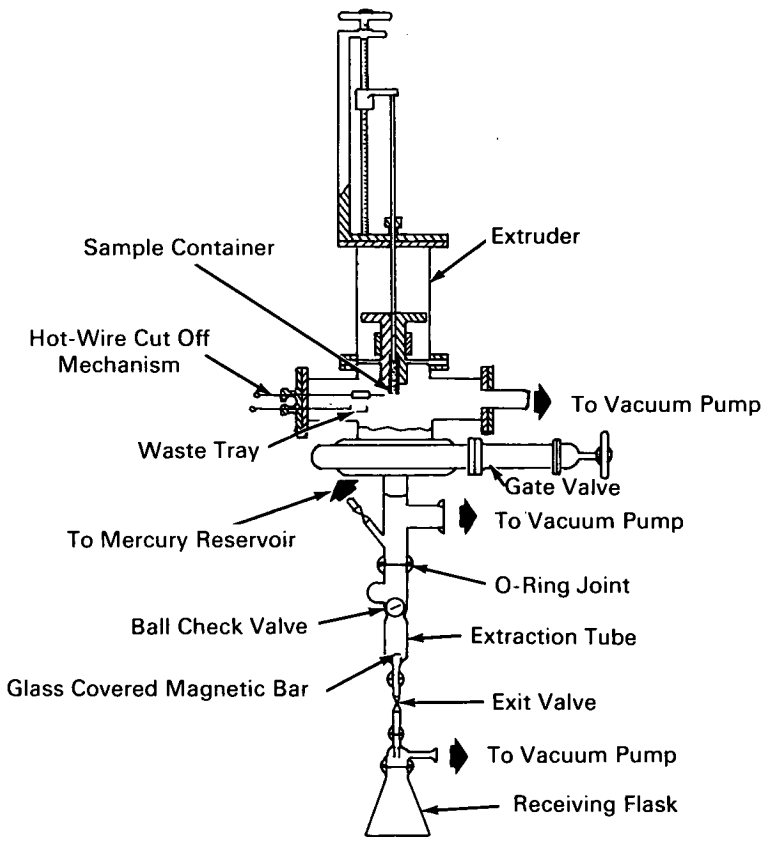
NASA TECH BRIEF

Apparatus Enables Accurate Determination of Alkali Oxides in Alkali Metals



The problem:

To devise an analytical test apparatus for determining the alkali oxide content of an alkali metal. The apparatus must prevent the inadvertent introduction of oxygen and moisture into the system during the sampling and analytical procedure. A reliable method for determining the oxygen content of alkali metals is important because alkali oxide impurities in alkali metals (even below 20 ppm in some cases), which have been used as heat transfer media, can promote corrosion attack on the walls of the heat transfer equipment.

The solution:

An evacuated apparatus in which the alkali metal is separated from the oxide by amalgamation with mercury.

(continued overleaf)

How it's done:

The apparatus consists of two major sections: an alkali metal extruder and an extraction section, connected by O-ring joints.

The sample container is a stainless steel tube 6 3/4-inches long by 5/16-inch inside diameter. It is vacuum filled with liquid alkali metal to be analyzed, capped, and stored under vacuum until used. For analyses, the sample tube is mounted in the extruder section of the apparatus. To perform an analysis, the entire apparatus is evacuated to a pressure between 2×10^{-5} and 4×10^{-6} torr. A small portion of the alkali metal is extruded from the sample tube, cut with the hot wire, and discarded into the waste tray. The analytical sample is then extruded, cut off, and dropped into the glass extraction tube. The extruder is valved off from the extraction tube and the ball check valve is seated in place to confine the subsequent amalgamation of the sample. A quantity of triple distilled mercury is then admitted to the extraction tube. After the amalgamation of the sample is completed, manipulation of a glass covered magnetic bar within the extraction tube removes all film from the tube walls. By means of an exit valve on the restricted end of the extraction tube, the amalgam is slowly admitted into the receiving flask. The exit valve is closed, and a second quantity (approximately 15 ml) of mercury is admitted to the extraction tube and the magnetic bar manipulation is repeated. The wash mercury is removed as before. A total of five similar washes is made to ensure complete removal of the alkali metal amalgam; 700 to 900 grams of mercury are used in all.

The extraction portion of the system (below the O-ring) is brought to atmospheric pressure and removed together with the amalgam receiving flask.

The alkali metal is extracted from the amalgam in the receiving flask with boiled distilled water. The hydroxide solution thus produced is titrated with standard acid to determine the weight of the alkali metal in the sample.

Any alkali oxide in the extraction tube is washed out with boiled distilled water. The resulting hydroxide solution is titrated with 0.005 N acid, using a microburette, to determine the amount of alkali metal oxide in the sample.

Notes:

1. This system was tested extensively to determine the oxygen content of samples of potassium. The results obtained showed good precision, independently of the time that the chemical components remained in the extraction system (up to 24 hours, the longest time studied).
2. The apparatus incorporates the following advantages over the standard Pepkowitz–Judd apparatus: (1) the entire system is maintained under a high vacuum, (2) the volume of the amalgamation reaction tube is small, (3) a ball check valve is used to contain the amalgamation reaction, (4) greased and waxed joints are eliminated, and (5) replicate samples are taken from a single sample tube.
3. Inquiries concerning this invention may be directed to:

   Technology Utilization Officer
   Lewis Research Center
   21000 Brookpark Road
   Cleveland, Ohio 44135
   Reference: B66-10296

Patent status:

This invention is owned by NASA, and a patent application has been filed. Royalty free, nonexclusive licenses for its commercial use will be granted by NASA. Inquiries concerning license rights should be made to NASA, Code GP, Washington, D. C. 20546.

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