

cesium (rubidium) carbonate that is needed to prepare 5 g of the alkali metal. Prepare a thoroughly triturated mixture of the calculated amount of the carbonate and a four-fold excess of powdered metallic zirconium. Put the reaction mixture into steel test tube *1* (what is a steel test tube needed for?) and place the latter into quartz vessels *2* (in the presence of your instructor, wear eye protection!).

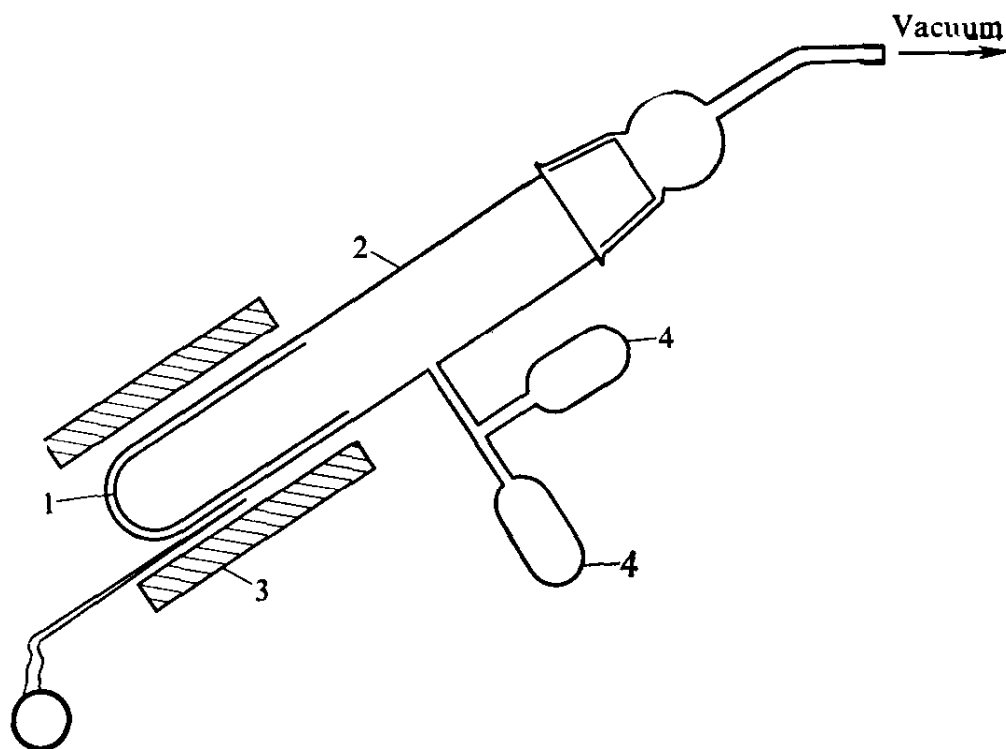


Fig. 114. Arrangement for preparing metallic cesium (rubidium)

Connect vessel 2 through a ground-glass joint to a vacuum system. When a vacuum of the order of  $10^{-3}$  mmHg is reached in the system, switch on furnace 3. Gradually raise the temperature in the furnace to  $500^{\circ}\text{C}$  and keep the apparatus under these conditions until a constant vacuum sets in with a value of  $10^{-3}$  mmHg. This indicates that the water vapour and gases have been removed from the system. Carefully, to prevent ejection of the reaction mixture, raise the temperature in the furnace to  $650\text{--}700^{\circ}\text{C}$ . Continue the heating until condensation of the metal vapour in ampoule 4 stops.

To purify the cesium (rubidium) from the admixture of the zirconium used for reduction, repeat the distillation from one ampoule into another one. For this purpose, carefully heat the alkali metal in the ampoule with a gas burner until boiling begins and perform distillation slowly (during 0.5 hour). After this, seal the ampoule with the metal. Write the equation of the reaction.

**2. Preparation of Lithium Hydride.** (Perform the experiment in the presence of your instructor!) Assemble an apparatus as shown in

Fig. 115. To prepare hydrogen, use a large freshly charged Kipp gas generator with a sufficient amount of acid. The latter must remain in the upper sphere even when it rises in the middle one above the zinc charge. Check the intensity of hydrogen evolution. Pour an acidified potassium permanganate solution into wash bottle 1 and a 96 % sulphuric acid solution into bottle 2. Fill U-tube 3 with glass wool mixed with phosphoric anhydride, and insert an iron tube into porcelain tube 5. Pour water into the crystallizer. Check the tightness of the entire apparatus.

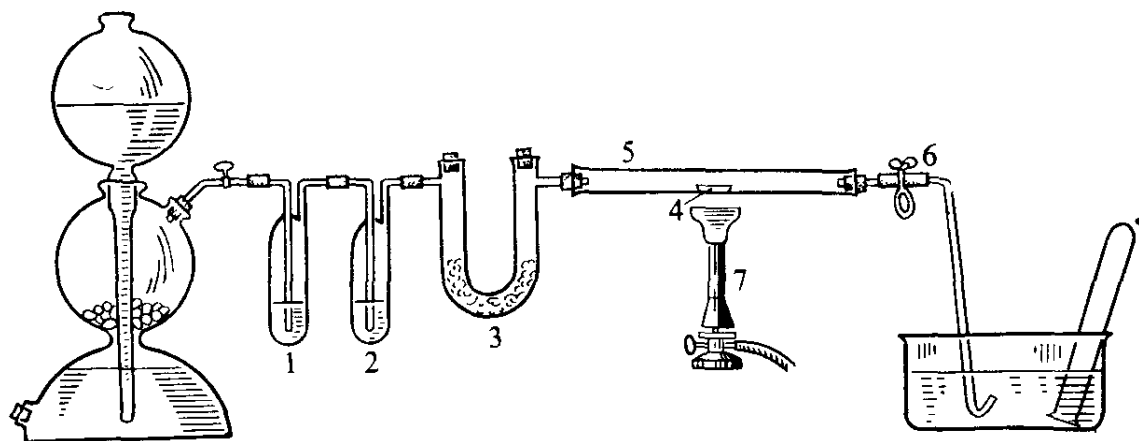


Fig. 115. Apparatus for preparing lithium hydride

Put 1 g of freshly cut metallic lithium cleaned of kerosene into iron boat 4 and place the latter into the middle part of the iron reaction tube. Again check the tightness of the apparatus and fill it with hydrogen. To do this, open clamp 6 and displace the air from the entire apparatus with a stream of hydrogen. Check the purity of the hydrogen evolving from the apparatus. Close the clamp and open the stopcock of the Kipp gas generator as far as it goes (not a single gas bubble should break through the washing liquids!). Put an asbestos sleeve onto tube 5 and begin to heat the tube where the boat with the metallic lithium is. Hold gas burner 7 with a flat "dovetail" nozzle in your hand while heating. At a temperature close to 500 °C, the lithium reacts with the hydrogen. Hydrogen bubbles begin to pass through wash bottles 1 and 2. If the reaction proceeds violently, stop heating the reactor, and leave the apparatus under the pressure of the hydrogen.

When the absorption of the hydrogen stops (how can this be determined?), cool the apparatus to room temperature in a hydrogen atmosphere. Write the equation of the reaction. Extract the boat from the tube. What is the colour of the product? See how lithium hydride reacts with water. What is the nature of the bond in a lithium hydride molecule?

**3. Preparation of Lithium Peroxide.** *a. Preparation of Lithium Hydroxide Monohydrate.* Put 5 g of lithium sulphate into a 400-ml