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RESEARCH ESTABLISHMENT

LUCAS HEIGHTS

WORK WITH ALKALI METALS

by

P. P. Pugachevich

ZHURNAL FIZICHESKOY KHIMII 31 (9) 2140 (1957)

TRANSLATED BY

H. J. de BRUIN

Sydney, August, 1958.

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Summary

In this paper methods are described of purifying alkali metals in vacuo and production techniques for alkali metal amalgams. The glass devices used for these purposes have no vacuum taps, ground in stoppers or rubber tubes; thus the purity of the metals and amalgams is not affected by the vapour of the vacuum lubricant, water or otherwise. The alkali metal amalgams produced remained unaltered in quality through years of storage.

INTRODUCTION

The purification of alkali metals and the production of alloys or amalgams containing these metals is usually carried out in an inert (1) atmosphere or in vacuum (2). However, the suggestions made in the different instructions to accomplish this often requires complicated apparatus; many of these cannot guarantee the degree of purity, while sometimes uncertainty is shown, as for example in the recommendations of Strong (see (3) page 377).

The method of working with alkali metals suggested below, has been tested by the author for several years and was shown to be simple and reliable.

EXPERIMENTAL PROCEDURES

To purify sodium and potassium, a glass apparatus shown in Fig. 1 is used. The impure metal is first washed with benzol and its oxide crust removed; it is then transferred to funnel 1, tube 2 is sealed and the system evacuated. After preheating for quite some time while connected to a diffusion pump, the temperature in 1 is raised sufficiently for the metal, contained in the funnel, to melt and to run into tube 3, while the oxide is being filtered off, from where it is distilled into reservoir 4. Finally this container is sealed under vacuum at c —— c, its contents between 100 – 150° C are poured into the ampoules 5, each of which has a thin–walled bulb 6; the ampoules are sealed off from 4.

Work with the metal caesium, which is kept under an argon atmosphere in sealed ampoules or tubes with ground stoppers and vacuum grease, requires a different approach. This is explained by the fact that caesium at room temperature reacts violently with air, water vapour in the air or even the carbon dioxide.

Therefore to remove its oxide the ampoule with caesium is cooled in liquid nitrogen for 10 – 15 minutes and cut by touching with a heated glass rod, a mark made on the ampoule after it is blown but before it is cooled. The portion of the ampoule containing the caesium is quickly transferred into reservoir 1 (fig. 2); the reservoir is closed by means of a glass stopper with vacuum grease and immediately backing vacuum applied. Frost which covers the ampoule after it is removed from the liquid nitrogen rapidly disappears. After this reservoir 1 is slightly heated on the outside with the flame from a glassblower's hand–torch; the caesium melts, runs into ampoule 2 – with a thin walled bulb 3 – which is sealed off from reservoir 1.

To purify the caesium from its oxide and other impurities which could have fallen into ampoule 2, an apparatus is used shown in Fig. 3. After carefully baking the system under the application of high vacuum and freezing out in liquid nitrogen, the iron bob 1, enclosed in glass, is raised 6 – 8 cm. by means of solenoid 2, which is fed by an alternating current. By interrupting the current in the solenoid the bob falls and breaks the thin–walled bulb in the ampoule containing the caesium. After this, the bob is raised with the solenoid (its position is shown dotted in Fig. 3), the caesium is distilled off into manifold 3; finally the system is sealed off while still under vacuum along the lines c —— c and c_1 —— c_1; the alkali metal runs into ampoules, 4, which in turn are sealed off from the manifold.

Cleaned by this method the caesium has a bright surface of a golden colour and does not contain any visible traces of impurities.

To distill the alkali metals it should be borne in mind that they react with ordinary kinds of glass; the glass becomes brown and very brittle. Therefore the alkali metals should be distilled at the minimum temperatures, for sodium and potassium at 200 and 250°C respectively and for caesium at 350 – 400° C, since the latter does not visibly react with the glass even at this temperature. The alkali metals contained in ampoules 4 can be used to produce amalgams or alloys.

To prepare, for example, an amalgam, the molybdenum glass apparatus shown in Fig. 4 is made up, consisting of a mercury degasser 1, a reservoir 2 for the ampoule with alkali metal and a manifold 3 for the amalgam.

Reservoir 4 of the degasser is filled with a certain quantity of mercury through outlet 5, which is sealed and the system heated for several hours while being connected continually to a diffusion pump.

Manifold 3, reservoir 6 and also tubes 7 and 8 are heated to 450 – 500°C. Reservoir 2, containing the ampoule with alkali metal is heated between 150 – 300°C , dependent on the metal. The mercury in reservoir 4 is heated from 320 – 400°C, evaporating it rapidly, passing it through a water condenser, and collecting it in tube 9 from where it passes back into reservoir 4. By heating the mercury at a high temperature, exceeding the boiling point by 20 – 40°C, and also the mercury vapour in tube 8 between 450 – 500°C, the degassing and partial destruction of organic impurities in the mercury is promoted considerably.

A glass valve, 10, being spherically ground, prevents the penetration of mercury vapour into the left hand portion of the system.

After heating for 4 to 6 hours, mercury reservoir 6 and tubes 7 and 8 are cooled down to room temperature; then the mercury in 4 and reservoir 6 is heated to 370°C. The mercury condensing in tube 8 runs down, lifting the glass valve 10, thereby falling into 6. It can thus be seen from Fig. 4 that the degasser allows to select the middle fraction of the heated mercury.

When a sufficient quantity of mercury has been collected in reservoir 6, it is heated between 250 and 300°C, the mercury starts to distill, condensing in 7, running down 11. At the same time the ampoule with alkali metal contained in 2 is broken by means of the solenoid as has been described above, and the distillation into 12 is begun. The mercury distilling from 6 and running down from 7 and 12 dissolves the alkali metal to produce an amalgam collected in 11.

The distillation of the alkali metal should be carefully watched in order that the temperature does not rise above 200°C, because in the adverse case mercury vapour will penetrate into 2 and absorb the alkali metal. As a result an intermetallic compound is formed in the ampoule containing the alkali metal and the distillation of the metal stops. The mercury in 6 should distill so rapidly that while dissolving the alkali metal in it, the possibility is avoided of forming a very concentrated solid amalgam, where such an amalgam may block the constriction at c – c (Fig. 4), while during further disolution in mercury a large quantity of heat is evolved, which may lead to fracture of the glass tube.

When the distillation of the alkali metal and the mercury is finished, manifold 3, while still hot, is sealed off under vacuum from the apparatus at the lines c – c and $c_1 - c_1$, the contents of 11 are carefully mixed and the manifold turned through 180°. The amalgam is filtered through narrow capillaries, into 13 and by inclining the manifold poured into ampoules 14 which are then sealed off. Prepared in this way the amalgam may be kept for years without changing its characteristics.

If the amalgam is required to be transferred to an apparatus in which its properties are to be investigated, the system depicted in Fig. 5 is constructed. This system is subjected to the working vacuum after which ampoule 1 with the amalgam in reservoir 2 should be heated to 350°C and the remainder of the system to 450 – 500°C. After this the thin-walled bulb of the ampoule is broken by means of solenoid 3 and iron bob 4, and the amalgam filters through funnel 5 – which has a number of apertures – leading into the working apparatus; this is finally sealed and the amalgam contained in it is subjected to the investigation.

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REFERENCES

1. H. Brayer, "Handbook on Synthetic Inorganic Chemistry", IIL, M., 1956.

2. B.P.Bering, N.L.Pokpovskyi, Zhurn. Fiz. Khim. 7, 509, 1936.

3. D. Strong, "Practices in Modern Physical Laboratories." Gostekhizdat, M. – L., 1948.

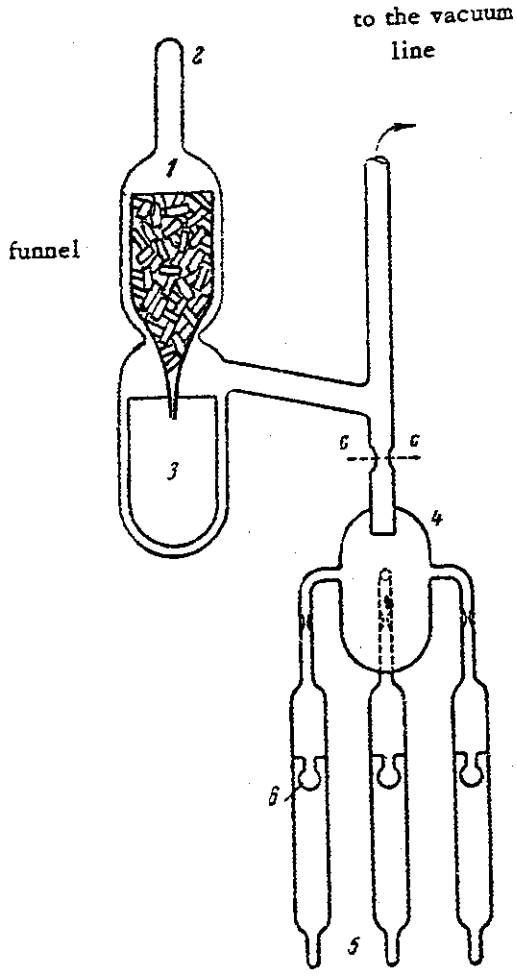

Fig. 1

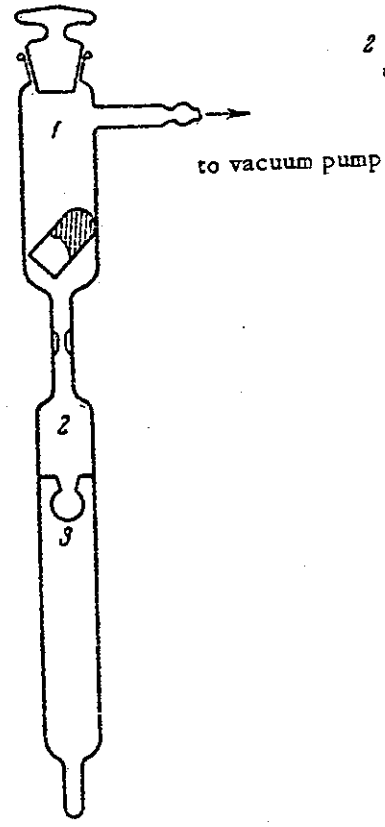

Fig. 2

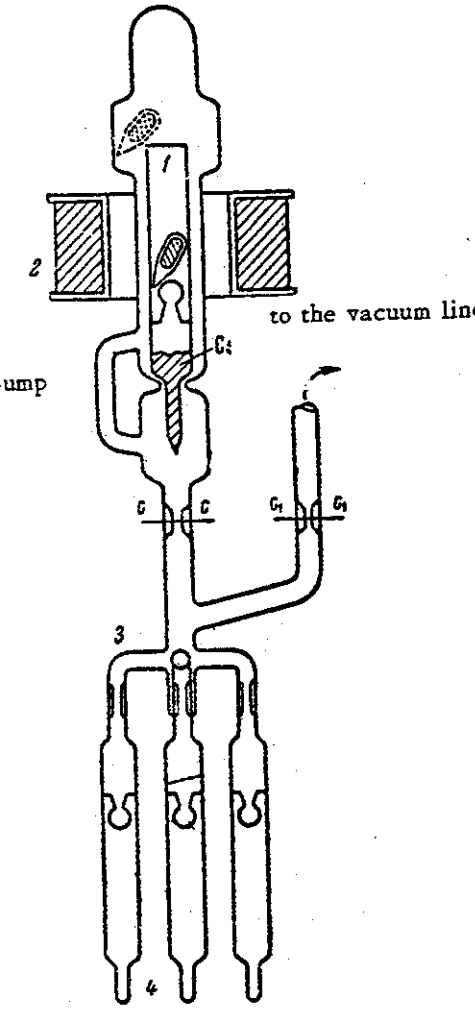

Fig. 3

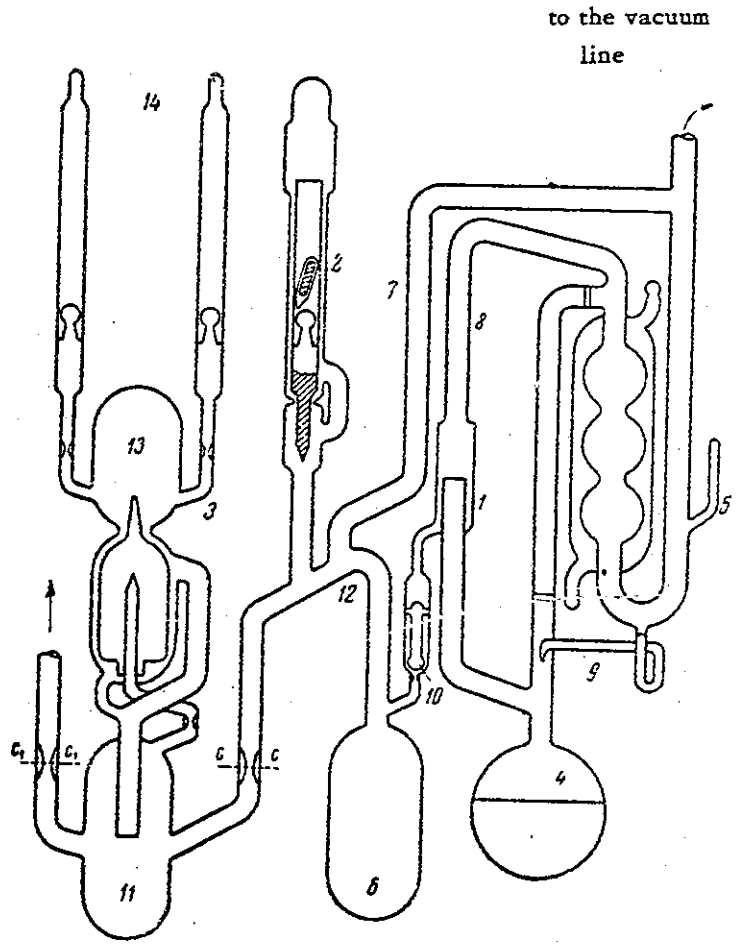
to the vacuum
line
14
13
3
2
7
8
1
5
12
10
9
4
6
11
C1
C1
C
C

Fig. 4

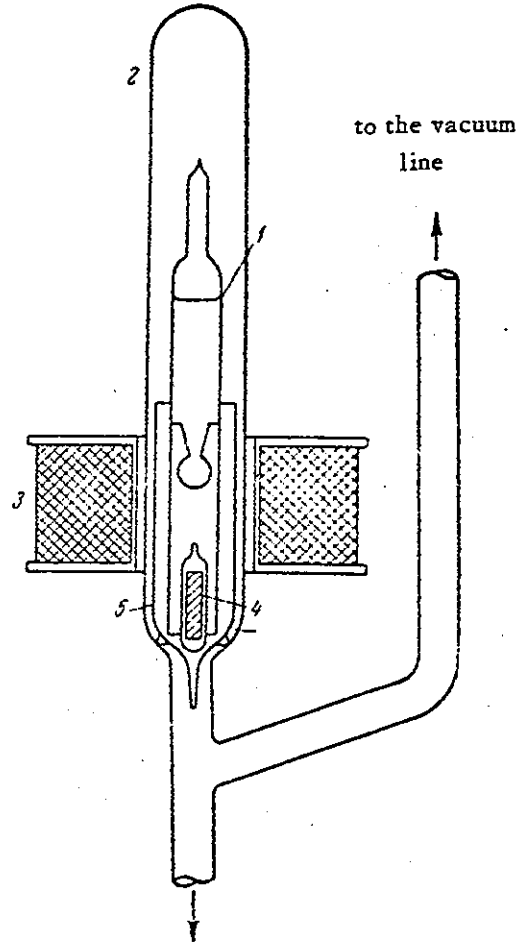
2
to the vacuum
line
1
3
5
4

Fig. 5