

An Arrangement for Preparing and Transferring Amalgams without Contamination

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Amalgam electrodes may be used in studies of solution equilibria, for measuring the equilibrium concentration of the corresponding metal ion.¹ The preparation of the amalgam is not an easy task, however, especially if the system is sensitive to oxidation, and if high precision is desired.² This communication describes an arrangement for preparing and transferring an amalgam, avoiding any contact with stop-cock grease or air.

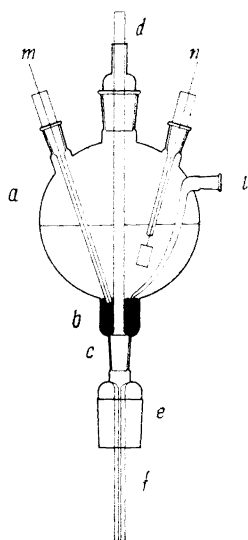


Fig. 1.

The main features of the apparatus are shown in Fig. 1. It is made of pyrex glass and consists of a three-necked round flask, in the lower part of which there is an extruded cylinder, ending in a standard ground socket. (a)=electrolysis vessel, (b)=pocket for Hg cathode. (If widely varying amounts of amalgam are desired in various experiments, one may make several flasks with different

pocket volumes). (c)=standard ground socket, (d) glass rod and standard ground cone, (e) standard ground cone for fitting tightly to the equilibrium vessel,³ which contains the solution to be studied. (f) a capillary tube which may have various shapes depending on the arrangement in the titration vessel. (m) cathode, (n) anode, (l) gas inlet tube.

Procedure. The apparatus is connected to the "equilibrium vessel" at (e), (f) is flushed with an inert gas from the titration vessel, and the plug (d) is inserted. The cathode pocket (b) is filled with mercury, after which the metal salt solution is introduced. Since Hg does not adhere to glass, the joint at (c) need not be greased. Grease should be avoided since it contains organic substances which may take part in redox reactions.

After the metal salt solution has been transferred, gas is bubbled through (l) which ends a little below the Hg surface. In this way the amalgam is also stirred and made homogeneous. Electrolysis is started by applying a d.c. voltage between the anode (n) and the cathode (m). If oxygen gas will be formed the anode may be placed in a separate vessel, connected to the cathode vessel by a salt bridge. Electrolysis is terminated when the amalgam is of the desired concentration. (d) is then cautiously turned and lifted until amalgam passes through (c), and the plug is shut again before the metal salt solution has reached the ground-glass joint. When a stop-cock of teflon or glass without grease was used instead of (c), (as suggested by Ref. 4), air invariably leaked in through the ends, so this method cannot be recommended. Amalgams of Tl, Pb, In, etc. have been prepared in this laboratory by means of the arrangement described here.

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