strated that the formation of the complex salts from one another is a reversible reaction and that their relative stabilities can be determined by a spectroscopic method. Measurements will be reported in a later communication.

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[CONTRIBUTION FROM THE CHEMICAL DEPARTMENT, UNIVERSITY OF LONDON, KINGS COLLEGE]

A METAL-TO-GLASS JOINT

BY H. N. RIDYARD

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Introductory

The author desired to obtain a joint which would unite glass and silica, and hold a vacuum for long periods, at temperatures at which the usual vacuum cements soften and become useless.

McKelvy and Taylor[1] describe a method of soldering glass to metal, after giving an account of previous work on this subject. These authors describe tests of this joint made at high pressures, and state that it was found suitable for vacuum work, but do not give any data as to degree of vacuum obtained, etc.

Later, Dundon[2] stated that he was unable to make joints of this type more than 8 mm. in diameter, and described a method of making a joint by electrodeposition of copper between a platinized glass tube and another of metal.

More recently, Meyers[3] describes joints of the soldered type up to 41 mm. in diameter.

Experimental Part

Attempts to use the first mentioned (soldered) joint for high-vacuum work were unsuccessful, the joint leaking at pressures below 10^{-2} mm.

Examination of joints after they were taken apart, showed that the solder or tin adhered to the glass only in places, so that contact between glass and solder in the joint appears to have been incomplete, thus accounting for the leak.

Experiments were then conducted to obtain a continuous film of metal over the glass, but were at first unsuccessful. It was found, however, that hot zinc chloride, used as a flux, appeared to cause disintegration of the platinum film.

Attempts to obtain films on the glass by dipping platinized tubes into a bath of molten tin were also unsuccessful; but at length lead was substituted for the tin and, after necessary conditions were worked out, continuous

[1] McKelvy and Taylor, THIS JOURNAL, **42,** 1364 (1920).

[2] Dundon, *ibid.*, **45,** 716 (1923).

[3] Meyers, *ibid.*, **45,** 2135 (1923).

films of lead were obtained on glass. These lead-coated glass tubes were then soldered to metal as easily as another piece of metal and gave very strong joints, which proved to be excellent for high-vacuum work; and possessed the additional advantage that they could be taken apart by heating and soldered together again without difficulty.

The method of construction of this joint is given in detail below.

The glass tube is chosen to fit into the metal tube, leaving about 0.4 mm. space all around. Sharp edges are fire polished, and the glass is thoroughly cleaned by standing in sulfuric acid and dichromate, washed with water and dried by alcohol, which is preferably burnt off. When it is nearly cold a drop or two of a platinizing solution is placed on the tube and spread with a small glass rod (0.5 mm.). The tube is then rotated about 15 cm. above a Bunsen burner, at nearly full flame and about $^2/_3$ full air supply. The tube still rotated, is brought gradually lower, until all organic matter is burnt off, and is brought to the first sign of redness. It is then allowed to cool; too high a temperature at this stage gives a dull coating which does not take the lead film properly. If silica, hard glass, or glazed porcelain be substituted for the glass, however, the temperature may be somewhat higher, particularly in the first case. These details have been made complete as a perfect platinum mirror is essential to the formation of a good lead coating.

The platinizing solution may be any of the recognized ones,[1] but the following has been used very successfully, and possesses the merits of simplicity and cheapness.

Half a gram of platinum chloride is dissolved in the least amount of absolute alcohol, and the solution poured into 5 cc. of lavender spike oil while the mixture is shaken.

A bath of molten lead is then prepared, and maintained at a temperature only slightly above its melting point. The glass tube is rotated in the Bunsen flame for about one minute, until it attains a temperature about midway between that of the bath and that of the air; the oxides on the surface of the lead are skimmed off with a piece of tin plate so as to leave a perfectly bright surface, and the glass is dipped with a quick up and down motion of the hand. Considerable care is needed in this process. If the glass is too hot when it is dipped, or the bath is greatly above its melting point, the platinum is dissolved by the lead, and no film is formed. If the glass is too cold a very thick film of lead is formed which can readily be stripped off, the platinum remaining intact. Experience shows when the correct temperature is attained. Immersion of the platinized end in the bath for more than a fraction of a second also causes solution of the platinum, and failure to form a lead film. A good coating is quite bright, very thin, difficult to strip from the glass, and when so removed, removes the platinum also almost completely. The film thus appears to be formed as a result of the chilling effect of the glass, the platinum acting as a flux, and the preliminary heating of the glass being necessary to enable the lead to dissolve the platinum before solidifying. This may explain why tin could not be used in this way, the lower melting point of the tin lessening the range of temperature in which both this chilling effect and the solution of the platinum could be obtained. No attempts were made to determine accurately the limits of temperature of the glass tube when dipped, as this would need a rather elaborate apparatus, and a few trials show the amount of heating necessary.

The metal tube used was of brass, well tinned by being thoroughly cleaned with emery cloth, inside and out, on the lathe, heated, and dipped alternately into zinc chloride solution and molten tin. The tube was then washed thoroughly and dried, and the ends lined with solder, using 'Fluxite'[4] as the flux. The lead on the glass was warmed and coated with a film of Fluxite and the prepared end then inserted into the brass tube, both being heated to the melting point of the solder. Care must, of course, be taken not to melt the lead. More solder may be used if necessary; in fact, the glass tube may be treated exactly as if it were of lead.

The Fluxite appears to be completely soluble in ether, and may be removed from the interior of the joint by this solvent.

These joints were sealed to an oil pump and a mercury vapor pump (Kraus type) working in series; and to a McLeod gage, the arrangement of taps, etc., enabling the pressure to be determined in any joint independently.

TABLE I

TESTS OF METAL-TO-GLASS JOINT

Joint No. 1, 5 mm. in diameter, was tested over a period of nine consecutive days

Date	1	2	3	4	5	6	7	8	9
Pressure in mm. × 10^4 attained	..	20	10	..	9	5	..	4	4
Pressure in mm. × 10^4 evacuated to.	4	3	6	..	4	4	4	3	..

Later, this joint was tested over a period of two weeks, and rose during that period from 1×10^{-4} mm. to 1×10^{-3} mm. Later still it was evacuated to 10^{-6} mm., and rose at first to 1×10^{-4} mm. overnight, but later to 8×10^{-5} mm.

Joint No. 2, 2 cm. in diameter, consisted of a metal-to-glass, a metal-to-silica, and two De Khotinski cement joints. This could be evacuated to 10^{-4} or 10^{-6} mm., but rose overnight to 10^{-3} mm., and in longer periods to 10^{-1} mm. It was found, however, that a joint made with De Khotinski cement alone behaved in exactly the same manner—possibly owing to vapors evolved from the cement.

Joint No. 3, 6 mm. in diameter, was installed in an air oven, and evacuated at first at 85°, and later at 110–140°. This gave figures similar to those given by No. 1, above. It was then heated to 140° for three days, in which time the pressure rose from 4×10^{-4} mm. to 1×10^{-2} mm. It was then evacuated to 5×10^{-4} mm. at 140°, heated at 110° to 130° for 3 days, and allowed to stand in the cold for 4 days, when the pressure had risen to 3×10^{-3} mm. It was then evacuated to 10^{-6} mm. at 150° for four days, after which it rose overnight to 10^{-4} mm. This joint finally failed at 180° to 190°.

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[4] Fluxite, a commercial flux, of a greasy nature.

These joints thus hold a vacuum at least as well as a glass stopcock greased with Ramsay grease, and have the great advantages of holding at 150° and of not evolving any organic vapors when the flux is efficiently removed.

It has also been found possible to coat hard glass and porcelain with lead, but no attempts have been made to construct joints with these. There is no obvious reason, however, why such joints should not be as successful as those with glass and silica.

No difficulty has been experienced in constructing joints up to 16 mm. in diameter.

In conclusion the author wishes to acknowledge the assistance afforded by a maintenance grant from the Board of Scientific and Industrial Research.

Summary

A method is given for coating soft glass, hard glass, silica or glazed porcelain, with homogeneous lead films, and for soldering material so coated to metal. The method can be applied to sheet or tube. A metal-to-glass, or metal-to-silica joint made by this method will hold a vacuum up to 10^{-4} mm. at 150° for some days, and up to 10^{-6} mm. for shorter periods.

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[CONTRIBUTION FROM THE DEPARTMENT OF CHEMISTRY OF McGILL UNIVERSITY]

THE PROPERTIES OF PURE HYDROGEN PEROXIDE. IV. ACTION OF THE HALOGENS AND HALOGEN HYDRIDES

BY O. MAASS AND P. G. HIEBERT

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This paper deals with a further investigation of the properties of pure hydrogen peroxide. When dry hydrogen chloride is passed into anhydrous hydrogen peroxide, chlorine is liberated even at low temperatures.[1] This was unexpected as it was believed that dilute solutions of hydrochloric acid did not react upon hydrogen peroxide; in fact, hydrochloric acid has been recommended as a stabilizer to inhibit the spontaneous decomposition of hydrogen peroxide when kept in glass bottles. It was mentioned in the paper referred to above[2] that it would be interesting to determine the concentrations at which chlorine would be given off and the concentrations at which hydrochloric acid would act as a stabilizer.

Experiments carried out by the authors revealed from the first the fact that even at low concentrations hydrochloric acid would bring about the decomposition of fairly dilute solutions of hydrogen peroxide apparently without the liberation of chlorine. The investigation was therefore ex-

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[1] Maass and Hatcher, THIS JOURNAL, 44, 2472 (1922).

[2] Ref. 1, p. 2477.