

Sample.	Mg. of iodine per gram.	
I.....	...	1.899
I.....	...	1.899
II.....	1.963	1.957
II.....	1.942	1.943
III.....	1.702	1.714
III.....	1.723	1.724

The writer wishes to express his appreciation to Mr. A. W. Thomas for assistance during the course of this investigation.

Summary.

This paper on the determination of iodine establishes the conditions for the determination of iodine:

First, when present as a soluble iodide or in the uncombined form.

Second, when present with bromine, bromides, and chlorides.

Third, when present with interfering compounds, as copper, silver, mercury, nitrites, etc.

Fourth, when in organic combination.

Fifth, when present in small amounts, special reference being given to the determination of the iodine content of the thyroid gland, and to a qualitative test for the presence of iodine.

NOTES.

A Simple Automatic Mercury Pump.—This pump is made on the Töpler principle with a simple attachment whereby the mercury is raised and lowered by means of an ordinary water suction pump. It is shown in the diagram (Fig. 1) with the modification of the Töpler pump introduced by Antropoff.¹ A similar pump, but without this modification, has been in use for over four years and has proved satisfactory in every way.

A, B, C are capillary tubes, *C* being about 68 cm. long. The part *D* is shown in detail in Fig. 2. *E* is attached to a P_2O_5 tube and leads to the apparatus which is to be evacuated.

With the ordinary Töpler pump the evacuation is produced by alternately raising and lowering the mercury in *F*, by means of a movable vessel corresponding to *O* which is raised and lowered. In this pump the same result is attained by alternately increasing and diminishing the air pressure over the mercury in *O*. This is achieved in the following manner: By evacuating through *S* by means of a water suction pump, the mercury rises in *L* and *C*. At the same time the air pressure over the mercury in *O* diminishes. The mercury in *F* falls. When the mercury in *L* reaches the level x_1 the mercury in the cup *N* is drawn up through the

¹ *Chem. Ztg.*, **34**, 974 (1910).

capillary tube C into Q . Air then enters through the capillary tube C . The mercury falls in L , O and K , and rises in D , F and A ; the gas in F is thus driven out through the capillary B and the mercury in G into H .

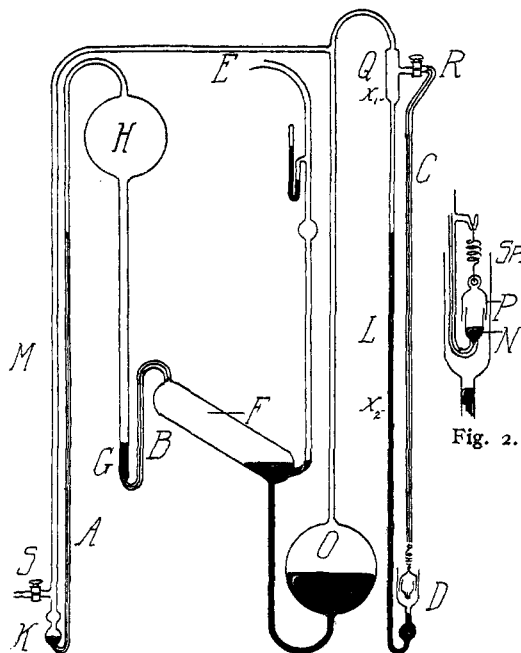


Fig. 1.

Fig. 2.

When the mercury in L falls to the level x_2 its level in D is up to the lower edge of the inverted cup P and thus closes the entrance to C . Since air is being drawn through C , P is drawn down into the mercury which overflows into and fills the cup N . The air supply is thus cut off and mercury again rises in L , C and O and falls in D , F and A , and the cycle is repeated. The volume of H is large in order that the pressure exerted by the gas driven over from F will not force the mercury in G back into F be-

fore the gas in H is drawn out through A , K and S by the water pump. The tap R regulates the rate at which air is allowed to enter the pump. Pumping may be stopped at any time by closing the tap S . One cycle is completed in from 30–60 seconds, depending on the efficiency of the water pump and the regulation of the tap R .

A great advantage of this pump over automatic pumps of the Sprengel type is that the outer air does not pass through the mercury used. Oxidation of the mercury and introduction of dust into the apparatus are thus avoided.

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Note on Amalgam Thermometers.—For the measurement of temperatures where great accuracy is not required, thermometers containing liquids have many advantages over the constant volume hydrogen or the platinum resistance thermometer. Unfortunately mercury can not be used at temperatures below -38° , and liquids such as pentane register very slowly, wet the capillary tube, etc., and consequently are much less accurate and convenient at low temperatures than mercury above its

freezing point. As we are engaged at present in investigating the properties of a number of bodies between -20° and -80° we have endeavored to construct suitable thermometers from various amalgams.

Of all the materials tried an 8.5% solution of thallium with a freezing point of -62° ,¹ has given us the only satisfactory instrument. Sodium, potassium, etc., lower the freezing point of mercury not more than $\frac{1}{4}10^{\circ}$, and no mixture investigated containing two or more metals remains liquid below -50° . The thallium thermometers have proved exceedingly useful down to -60° .

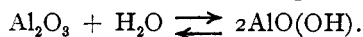
Since thallium oxidizes easily the amalgam must be filtered and the thermometers filled *in vacuo*. At -60° the amalgam flows, apparently, as easily as mercury, has been kept for six months without change, expands perfectly regularly, so that the temperatures plotted against those given by a platinum resistance thermometer are in a straight line.

D. MCINTOSH,
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Alumina as a Drying Agent.—In the course of an investigation on phosphonium compounds² a drying agent was necessary which would successfully dry both PH_3 and HI or HBr , since P_2O_5 proved useless for this purpose. Satisfactory results were obtained by employing Al_2O_3 prepared by igniting the hydroxide at a low temperature.

A test of the efficiency of this substance to take up moisture was made by passing air saturated with water vapor at room temperature, about 18° , through a tube containing about 7 grams of the oxide, and then through a second tube filled with P_2O_5 . The air was led through at the rate of about 2 or 3 bubbles per second. Both tubes were weighed from time to time.

The somewhat remarkable result obtained was that the P_2O_5 tube showed no perceptible gain until the Al_2O_3 had increased about 18% in weight, corresponding to the formation of the hydrate $\text{Al}_2\text{O}_3 \cdot \text{H}_2\text{O}$. The reaction is generally represented by the equation



The formation of the hydrate is accompanied by evolution of heat, hence increased temperature causes its dissociation into Al_2O_3 and H_2O .

It is thus seen that 1 gram of Al_2O_3 can practically completely absorb all moisture from approximately 10 liters of air saturated with water vapor at 18° .

G. P. Baxter and R. D. Warren³ investigated the drying properties of CaBr_2 , ZnBr_2 and ZnCl_2 , and from their results it is seen that compared

¹ Kurnakow, *Z. anorg. Chem.*, 30, 86 (1902).

² See page 877.

³ THIS JOURNAL, 33, 340 (1911).

with Al_2O_3 these substances are much inferior as desiccating agents. Al_2O_3 is also more effective than H_2SO_4 . A tube filled with Al_2O_3 can be used for an indefinitely long period, if from time to time it is heated with a smoky flame while air, previously led through H_2SO_4 , is passed through it.

To replace the usual P_2O_5 tube used in connection with mercury pumps it appears particularly suitable, since the tube used need never be renewed. A small tube of P_2O_5 following the Al_2O_3 would serve to indicate when the Al_2O_3 needed reheating. Its uses in many investigations are obvious.

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CORRECTION.

The Connection between Electrical Conductivity and Loss of Electrons by Metals.—Dr. Falk has kindly called my attention to the fact that Sir William Ramsay, in his Faraday lecture (*J. Chem. Soc.*, **93**, 787), pointed out the connection between the ease with which metallic elements lose electrons and their properties as conductors. I regret that I should have overlooked this when writing the note which appeared in the May Journal (THIS JOURNAL, **34**, 664).

W. A. NOYES.

LONDON, June, 1912.

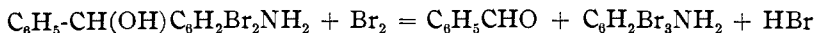
[CONTRIBUTION FROM THE CHEMICAL LABORATORY OF HARVARD COLLEGE.]

THE SPLITTING OF AMINOARYLCARBINOLS BY THE ACTION OF BROMINE.

BY LATHAM CLARKE AND RICHARD HARKNESS PATCH.

Received April 29, 1912.

It has been shown by Clarke and Esselen¹ that when 2,5-dibromo-4-aminobenzhydrol in chloroform solution is treated with bromine, a splitting takes place, whereby 2,4,6-tribromoaniline and benzaldehyde are produced:



Since the publication of the above noted preliminary paper, Clarke and Esselen have found that with aminobenzohydrols, the reaction is a general one, and an account of this research will appear in the near future in THIS JOURNAL.

The present writers have extended this research to aminoarylcaminols containing an aliphatic residue and also to tertiary carbinols. The latter are of especial interest, for if tertiary carbinols are split in a manner analogous to the splitting of secondary carbinols, then the color bases of triphenylmethane dyes would be broken down to substituted bromoanilines and ketones, thereby giving a method for the exact proof of constitution.

¹ THIS JOURNAL, **33**, 1135.