

disregarding the escape of vapors of iodine and bromine and, after rinsing, add 60 cc. of fifth normal potassium iodate (or 12 cc. of the normal solution), 24 cc. of nitric acid and water to make about 250 cc. Proceed from this point precisely as in the analysis of a bromide (p. 278). Of course, when the solution is ready for titration, an aliquot part should be taken, if the amount of chlorine is so large as to make this step desirable.

The bromine may for many practical purposes be determined sufficiently well by difference, since the total quantity of impurities other than chlorine is usually much less than one per cent.

If accuracy is desired, the total free bromine and chlorine should be determined by an iodometric titration, the weighed substance being dissolved in a considerable excess of potassium iodide solution, and the liberated iodine titrated after feeble acidification. Correcting the volume of thiosulphate here required by that equivalent to the chlorine found, the difference gives the free bromine.

It is obvious that the accuracy of the method described for the determination of chlorine in bromine, or of any other process for this purpose, can not be shown by analyses of known mixtures, since the errors in preparing such a mixture must far exceed the analytical errors. It is only possible to show the agreement of duplicate analyses and the concordance of the analytical results with physical properties. The latter will be taken up in another paper.

As illustrative of the former, I take at random several analyses out of a large number, made by Dr. H. A. Carlton.

Sample 6706	Found % of Cl	2.445	2.380			
6806	“ “	2.069	2.072	2.052	2.32	
1220 dry	“ “	4.439	4.422			
1220 saturated with water		4.407	4.401			
1220 dried again		4.438	4.461			
5406		8.55	8.57			
424		5.00	4.98	5.04	5.06	

It is confidently believed that the methods here described for the determination of chlorine in bromine and bromides are a practical solution of this old analytical problem, suitable for routine analyses of either technical or scientific character and adapted to replace the indirect methods. It is a pleasant duty here to express my obligations to Dr. Carlton for his able assistance in the work.

St. Louis.

A CONVENIENT AIR-BATH AND HOT PLATE.

BY EDWARD DEMILLE CAMPBELL.

Received Jan. 2, 1907.

During the past ten years the small combined air-bath and hot plate to be described has been in use in this laboratory and has proved so conven-

extending 30 cm. above the cylinder. This wire is bent at right angles at the top and extends over the cylinder serving as a support on which to hang a thermometer. The outside of the cylinder is wrapped to a thickness of about 0.5 cm. with asbestos paper soaked with a solution of sodium silicate diluted with two volumes of water. Asbestos so wet with dilute sodium silicate can be easily formed into any desired shape and on drying becomes firm and hard. The top of the air-bath is covered by means of a 15 cm. split watch glass, one side of which is either perforated or provided with a notch to allow the insertion of a thermometer. When in use, the lower end of the cylinder usually rests directly upon the hot plate (D), but if in special cases it is desired to have a circulation of air in the bath, fine copper wires are twisted at four points around the sides of the cylinder in such a way that the cylinder is raised off the hot plate to an extent equal to the diameter of the wire. The hot plate (D) on which the air-bath may be placed consists of an ordinary seven-inch stove lid supported on the asbestos cylinder (E), which rests on the heavy asbestos board base (F). The asbestos cylinder (D) is made by cutting a strip of moderately heavy asbestos paper 4 cm. wide and long enough to form a cylinder 0.5 cm. thick and equal in external diameter to the diameter of the hot plate. Outside of this inner asbestos cylinder is wrapped a second piece of asbestos paper 4.5 cm. wide. This built-up asbestos cylinder which serves to support the hot plate is provided with four horizontal slots (G) 4 cm. long and 1 cm. wide which serve for the escape of the products of combustion. The built-up cylinder is soaked with dilute sodium silicate and is cemented to the heavy asbestos board base (F) by filling the inside angle (H) with a pulp made by disintegrating asbestos paper with dilute sodium silicate. The heavy asbestos board base (F) is perforated with a hole in the center 5 cm. in diameter to admit the flame of a Bunsen or Fletcher burner. The asbestos board base is supported on an ordinary iron stand, such as is used for supporting water baths.

The use of a heavy asbestos board base (F) underneath the hot plate prevents very largely the downward radiation of heat and enables a higher and more uniform temperature to be maintained on the entire surface of the hot plate than when this is not used. With a good burner there is no trouble in reaching a temperature of 375° in the air-bath and as the air-bath is covered with a watch glass, any changes taking place in material placed in the air-bath may be readily observed.

In the course of some research work in this laboratory, it has been shown that the oxalates of many of the rare earths such as thorium, cerium, lanthanum, neodymium and praseodymium may be completely dissociated into readily soluble basic and normal carbonates by heating to a temperature between 305° and 335° . The air-bath has also proved

very useful for heating or evaporating solutions, where a temperature higher than that attainable on a water bath is necessary and where it is necessary to avoid local overheating, which is so liable to occur if a dish rests directly on the hot plate. A set of ordinary water-bath rings may be substituted for the watch-glass cover, thus enabling any size of evaporating dish or casserole to be heated uniformly and to any desired temperature up to 375° .

Chemical Laboratory,
University of Michigan, Dec. 27, 1906.

THE INFLUENCE OF BASIC LEAD ACETATE ON THE ROTATION OF SUCROSE IN WATER SOLUTION.

BY FREDERICK BATES AND J. C. BLAKE.

Received January 7, 1907.

In the polarimetric estimation of sucrose in raw sugars an indefinite amount of basic lead acetate in solution is usually added to the sugar flask, before making up to volume, in order to precipitate the impurities more or less completely and thus clarify the sugar solution sufficiently for reading on the polariscope. The use of this reagent is almost universal, and it is, indeed, so powerful a clarifying reagent that the question as to whether it can be used in precision work on raw sugars is of the first importance. Excepting the error due to the volume of the precipitate which is formed by the reaction it is usually considered by commercial chemists that the results are not affected by the basic acetate.

The evidence obtainable from the literature as to the effect of basic lead acetate on the polarization of sucrose is conflicting and very unsatisfactory, the results usually being expressed in tenths of a per cent, with no indications that attention was paid to numerous minor sources of error. Von Lippmann's¹ conclusion, derived from the literature, is that basic lead acetate exerts no influence on the rotation of sucrose in water solution. We have found this conclusion to be in error. Its great influence on the specific rotation of levulose has long been known. Nevertheless, because of the relatively large amount of sucrose present in raw sugars as compared to the amount of levulose present, the effect of the basic lead acetate on the sucrose, though relatively less than its effect on levulose, still retains an equal importance.

The effect of basic lead acetate on the specific rotation of sucrose was overlooked by the early investigators because of the comparatively crude polarizing apparatus and methods used, and also because the temperature coefficient of pure sugar solutions was little known or entirely neglected.

¹ *Chemie der Zuckerarten* 2, 1185.