the filtrates; but if (4) and (5) are coupled, the precipitate will have the composition  $Ag_5H(PO_4)_2$  which was obtained in the four cases previously quoted, the production of which can be expressed by the equation

(7) 
$$2Na_2HPO_4 + 5AgNO_3 = Ag_5H(PO_4)_2 + 4NaNO_8 + HNO_8$$
.  
 $Ag_3PO_4 \cdot Ag_2HPO_4$ 

For an expression which would account for the presence in the filtrate from the complete (?) precipitation of the silver, of both nitric acid and of phosphoric acid the following is suggested: (8)  $4Na_2HPO_4 + 6AgNO_3 = 2Ag_3PO_4 + 4NaNO_8 + HNO_8 + H_3PO_4$ 

It would appear then that the reaction between silver nitrate and disodium orthophosphate takes place in several distinct stages, and that the formation of a phosphate containing about 76 per cent. of silver is the most constant resultant. Also that free nitric acid and phosphoric acid remain in the solution from which no further precipitate can be obtained on the addition of the nitrate. It is our intention to further investigate this matter specially as regards the quantitative composition of these filtrates with a view to determining whether or not the last six equations suggested (Nos. 3 to 8) adequately express the reaction.

THE CHEMICAL LABORATORIES, THE UNIVERSITY OF TORONTO.

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## A CONSTANT TEMPERATURE BATH FOR LOW TEM-PERATURES.

BY A. GIVEN.

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THE usual temperature at which specific gravity determinations are made is  $15.6^{\circ}$  C. ( $60^{\circ}$  F.), and it was for this temperature that this bath was primarily intended, and at which the bath has been used, though modifications of it are in use at  $20^{\circ}$  C. The advantage of such a bath lies in the fact that the whole body of the pycnometer is immersed, after cooling to  $14^{\circ}$  C. or below, and therefore rises to the desired temperature more quickly and uniformly than in any other way. Moreover, the pycnometer can be left in the bath as long as may be desired without change



Fig. 1. Section on line A-B.

of temperature. Being held at such a relatively low temperature, the loss by evaporation is reduced to a minimum, and no appreciable change occurs in the density of the solution. The bath was built for the Bureau by a local tinsmith at a cost of  $$_{22.00}$ . The construction is shown in the drawing.

The apparatus consists of a double-walled bath, of which the inner wall and bottom are of copper, and the outside of galvanized iron, and a suitable stand. The space between the walls of the bath is packed with asbestos in order to insulate the contents of the bath as far as possible. In the bath is the copper ice-box I, of the same depth as the bath, extending to within  $1^{1}/_{2}$  inches of the bottom, and raised that much above the top of the bath to provide an overflow for the waste water from the melting ice. The wooden cover serves as an insulator. The remaining space in the bath is taken up by the perforated shelf

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P, which is fixed at the proper depth for the pycnometers or other vessels used. Above this, and fastened to the same hangers, is the narrow shelf S, which carries the thermometer and gas regulator. This regulator (Fig. 4) which was designed and made by Mr. B. J. Howard, of the Bureau of Chemistry, is one of the most necessary



Fig. 2. Section on line C-D.

parts of the apparatus, as it is the sensitiveness of the regulator which has contributed so much to the constancy of the temperature of the bath. The large bulb, I inch in diameter and 5 inches long, is filled with methyl alcohol, though toluene, having a higher coefficient of expansion, would perhaps be better. The regulator is connected with the gas supply, and with a Bunsen burner so adjusted that it will not strike back when the gas supply is nearly cut off. For maintaining an equal temperature throughout the bath some form of stirrer is necessary, and we have used with good results a gentle current of air, conducted to the bottom of the bath close beside the ice-box.

In starting the bath, the ice-box is first filled with water, and the rest of the bath with distilled water. Large pieces of ice are then placed in the ice-box. The stirrer is started, and to lower the temperature still faster, a clean piece of ice is placed on



the platform. When the thermometer shows that the temperature is about to fall below the desired point, the gas is turned on, and the piece of ice removed from the platform. A bath 18 inches square and 12 inches deep uses, in the hottest weather, about fifty pounds of ice in seven hours. As long as there are 300 or 400 grams of ice in the box, the temperature will remain constant, and if the ice-box is kept supplied, the thermometer shows no change of temperature for hours at a time. It is necessary to have a large volume of water in the bath in order that the temperature may not be affected by putting in and taking out the pycnometers.

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The following table gives the results of ten duplicate determinations of specific gravity on samples of whiskey. The tem-



Fig. 4. Temperature regulator: I, gas inlet; O, gas outlet; P, pilot flame supply; C, centering knobs of glass; R, rod of glass to strengthen the apparatus; A, alcohol reservoir; M, mercury trap.

perature at which the filling of the pycnometers was made remained constant at  $15.6^{\circ}$ .

Serial number.	Specific gravity.
14772	0.91139
	0.91136
14773	0.90820
	0,90804
14794	0.94395
0	0.94395
14805	0.91148
0.6	0.91141
14806	0.91274
	0.91275
14814	0.94021
0	0.94020
14842	0.94099
	0.94095
15005	0.94090
	0.94085
15000	0.94218
	0.94214
15007	0.94023
	0.94017

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It will be readily seen that the application of this bath is not confined to specific gravity determinations. At the same time that the one here described was built, another of the same surface area, but 22 inches deep, was constructed for use with the Roese fusel oil apparatus, which must be kept at constant temperature in order to get comparative results. Later, another, slightly smaller than the one described, was ordered for use with the Zeiss immersion refractometer. This has a basket of galvanized wire, bound with copper, instead of the perforated plate. The part of this basket next the ice-box is divided into compartments for holding four flasks. In front of these compartments is a strip of copper with holes for the small beakers used with the refractometer, under which is the strip of ground glass through which the light passes upwards. The wire frame for supporting the instrument is fastened to the sides of the basket, and the mirror for directing the light upward is attached below. The bath itself differs from those previously made in that the front side is of glass, to permit the entrance of light. This bath is kept at 20° C., and works just as perfectly as the one at 15.6° C.

The writer acknowledges suggestions from Messrs. Tolman, Howard and Munson, of this Bureau.

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## THE RAPID PRECIPITATION OF ANTIMONY IN THE ELEC-TROLYTIC WAY.

By Julia Langness and Edgar F. Smith. Received August 28, 1905.

THE BEST electrolyte for the determination of antimony is sodium sulphide, containing a small amount of sodium hydroxide. The polysulphides, frequently present, have been destroyed in various ways, one of the most convenient being by the addition of potassium cyanide, first used by A. Fischer,<sup>1</sup> and subsequently and wholly independently by Exner,<sup>2</sup> when describing his experiences in the rapid determination of metals with the aid of a rotating anode. His results in the case of antimony were exceedingly good, both from the point of rapid and complete pre-

1 Ber., 36, 2348.

<sup>2</sup> This Journal, 25, 896.