TECHNICAL METHODS

THE ESTIMATION OF POTASSIUM BY A MICRO-METHOD

BY

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(RECEIVED FOR PUBLICATION, OCTOBER 6, 1947)

A method for the estimation of potassium using a silver cobaltinitrite complex was described previously (Cumings, 1939). The method as described below has overcome the disadvantages of the original method, which were the need for ashing sera and the rather difficult titration involved. The composition of the compound has been ascertained and will be indicated in this paper.

Breh and Gaebler (1930), after deproteinization of serum, added silver nitrate followed by the usual sodium cobaltinitrite reagent, and part of their procedure has been utilized. A colorimetric method is used in place of final titration for estimation of nitrite (Hoagland and Ward, 1942; see also Looney and Dyer, 1942).

Method

To 3.1 ml. of distilled water in a centrifuge tube are added 0.2 ml. serum followed by 0.3 ml. $2/3$ $N-H_2SO_4$ and 0.3 ml. 10 per cent w/v $Na_2WO_4 \cdot 2H_2O$; after mixing, 0.1 ml. of 5 per cent w/v $AgNO_3$ is added, and the contents mixed and centrifuged; 2 ml. of the supernatant fluid are placed in a centrifuge tube graduated at 10 ml., and 1 ml. of my original silver cobaltinitrite reagent is added (Cumings, 1939), and the tube centrifuged after standing for one hour. To the precipitate are added about 10 ml. of 50 per cent acetone, which is centrifuged after mixing, and this procedure is repeated once more, after which 1 ml. of 2.5 $N-NaOH$ is added to the precipitate and the tube placed in a boiling-water bath for five minutes. Distilled water is now added up to the 10 ml. mark. After mixing, the contents are centrifuged and 1 ml. of the supernatant fluid is diluted to 25 ml. with distilled water.

Five ml. and 10 ml. of this dilution are placed in test tubes made up to 10 ml. with water, and 1 ml. of 0.2 per cent sulphanilamide is added, followed by 0.2 ml. of 0.1 per cent N -(1-Naphthyl)-ethylenediamine hydrochloride.

A blank of 10 ml. of distilled water, and two

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standards of 10 ml. of distilled water containing 0.004 and 0.008 mg. of sodium nitrite, are treated in the same way, and all tubes are placed in ice or in a refrigerator as at this temperature there is a greater constancy of colour production. After cooling 0.2 ml. of 6 N -HCl are added to each tube, the contents mixed, and the tubes returned to the cold for fifteen minutes, after which time readings are taken in a colorimeter. (A King pattern photoelectric colorimeter has been found to be very suitable.) The pink to red colour is permanent for twenty-four hours.

A standard potassium solution should also be treated in the same manner as the serum by taking 0.2 ml. of a solution containing 51.7 mg. KNO_3 per 100 ml. (equivalent to 20 mg.K./100 ml.). With a visual colorimeter the calculation $\frac{x}{y} \times 20$ will give the result in mg./100 ml.

Results

Table I gives a few of the results obtained using this method, my original method, and that of King and others (1942).

TABLE I

COMPARISON OF RESULTS OF K IN SERUM BY THE METHOD OF KING AND OTHERS AND OUR METHODS

King	$K_3Ag_2Co_2Na(NO_2)_{11}$ method	
Mg./100 ml.	Original method mg./100 ml.	New method mg./100 ml.
18.0	21.0	19.5
25.3	26.6	26.6
24.1	24.0	25.0
18.0	19.0	20.7
27.0	25.6	26.3
20.4		21.6
20.6	21.3	21.6
19.2	21.6	19.8
19.0	17.0	18.0
40.0	41.0	42.0
(Haemolysed serum)		

Composition of Compound

Samples of the potassium silver cobaltinitrite compound were prepared from known amounts of potassium nitrate, dried at 100° C., and weighed. Separate quantities of the compound so prepared were then analysed for potassium, silver, cobalt, sodium, and nitrite, and Tables II and III show the results obtained. The analytical methods used are indicated in the appendix.

TABLE II
MOLECULAR WEIGHT OF COMPOUND FORMED
(Assuming molecule contains 3 atoms K)

KNO ₃ taken	Compound formed	Molecular weight
0.2636 g.	0.8695 g.	1020
0.0188 g.	0.0638 g.	1028

TABLE III
COMPOSITION OF COMPOUND

Constituent	Estimated (%)	Calculated (%)
K	10.94	11.40
Ag	20.1	21.05
Co	12.0	11.50
Na	2.2	2.24
NO ₂	52.8	53.80

The results found by experiment and by calculation are in sufficiently close agreement to substantiate the view that the formula $K_3Ag_2Co_2Na(NO_2)_{12}$ with a molecular weight of 1026 is correct.

Discussion

The method presented is quick and reliable, and can be performed on 0.2 ml. quantities of serum.

If much less serum were used the amount of precipitate would be so small that any losses during centrifugation would cause a relatively large error.

The final pink colour is very easy to match and the method involved is an extremely delicate one for the estimation of nitrite.

Summary

A method for the estimation of potassium in small amounts of serum is described.

The potassium silver cobaltinitrite compound formed has been shown to have the formula $K_3Ag_2Co_2Na(NO_2)_{12}$.

REFERENCES

- Breh, F., and Gaebler, O. H. (1930). *J. biol. Chem.*, **87**, 81.
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 King, E. J., Haslewood, G. A. D., Delory, G. E., and Beall, D. (1942). *Lancet*, **1**, 207.
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Appendix

ANALYTICAL METHODS USED

- Sodium:** King, 1946. *Micro-Analysis in Medical Biochemistry*, Churchill, London.
- Cobalt:** 1. Gravimetrically as $CoSO_4$.
 2. Colorimetrically with choline chloride and sodium ferrocyanide (King, 1946. *Micro-Analysis in Medical Biochemistry*, Churchill, London).
- Silver:** 1. Gravimetrically as $AgCl$.
 2. By titration with thiocyanate.
- Nitrite:** Application of Bratton-Marshall sulphonamide method, as used by Hoagland to determine nitrite.
- Potassium:** From titration of silver with thiocyanate. See original paper (Cumings, 1939) for rationale.