

VOLUMETRIC ESTIMATION OF TIN(II) WITH HEXAMINECOBALT(III) TRICARBONATOCOBALTATE(III) USING FERROIN AS REDOX INDICATOR

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Abstract. A quick, precise and accurate method for the estimation of tin(II) with hexamminecobalt(III) tricarbonatocobaltate(III) as an oxidizing volumetric titrant has been evolved. Effects of foreign ions like Zn^{2+} , Ag^{+} , Al^{+3} and Pb^{+2} has also been reported.

In our previous communication¹ we have reported about our successfully utilizing the strong oxidizing power of hexamminecobalt(III) tricarbonatocobaltate(III), $Co(NH_3)_6Co(CO_3)_3$, for the quantitative determination of iodide and effects of interferences of chloride and bromide. Bricker *et al.*² were the first to use this redox titrant for the estimation of cerium (III), vanadium (IV) and iron(II). Probably no other reference except the two mentioned earlier, does exist in literature in which this compound might have been used as a redox titrant in volumetric analysis. The aqueous solution of the compound is unstable but it gives a stable solution when dissolved in a saturated solution of sodium bicarbonate thereby extending utility of the compound in volumetry. When $Co(NH_3)_6Co(CO_3)_3$ is dissolved in a medium having pH less than 7 the tricarbonate complex is decomposed to yield free cobalt(III) whereas if dissolved in a solution more alkaline than having a pH of about 8 a brown precipitate of cobalt(III) hydroxide is formed with the discharge of the original green compound.

Because of the high oxidizing power of cobalt(III), being liberated from the compound when dissolved in acidic medium, there always does exist the possibility of its use as a volumetric oxidizing titrant for the estimation of a variety of compounds. With these prospects in mind we have, in this publication, attempted at the estimation of tin(II) and observed the effects of interferences using this compound as a volumetric oxidizing titrant.

Experimental

Reagents

Hexamminecobalt(III)tricarbonatocobaltate(III). A saturated solution of sodium bicarbonate was prepared according to the method of Bricker *et al.*² observing precautions as mentioned earlier.¹ The factor of the solution was checked against ferrous ethylenediammonium sulphate tetrahydrate as primary standard.²

Water solutions of zinc sulphate ($ZnSO_4 \cdot 7H_2O$), silver nitrate ($AgNO_3$), aluminium chloride ($AlCl_3 \cdot 6H_2O$) and solution of lead acetate in glacial acetic acid were prepared (A.R. grade, E. Merck) and their

factors, wherever required, were checked conventionally. Solutions of low concentrations were prepared by exact dilutions of the standardised solutions.

Tin(II) Solution. An approximately 0.1N solution of tin(II) chloride was prepared by dissolving an appropriate amount of $SnCl_2 \cdot 2H_2O$ (E. Merck) in about 1.2M HCl and the factor of the prepared solution was determined with standard iodine solution using starch as indicator. Solutions of low concentrations were prepared by exact dilutions of this stock solution.

Ferroun. M/40 solution (Fluka, Switzerland) was used as indicator.

All other reagents used during this work were either of A.R. grade or equivalent purity.

Officially calibrated glassware was used during these investigations.

Procedure

A definite aliquot (5–25 ml) of the test solution was added to a titration flask to which water and hydrochloric acid were added to make a total volume of about 50 ml which was 1.0N with respect to HCl. Only 4 drops of Ferroun indicator were added to it and the titrant was added, in as small portions as possible, from a 10-ml microburette divided at 0.05 ml intervals. Vigorous stirring was avoided and a mild swirling motion to the reactants of the flask was given. End-point was reached when the colour changed from red to pale blue. Blank titrations were run similarly and the actual volume of the titrant consumed for the substance under test was obtained by subtracting the volume for blank from that for the total. For investigating effects of foreign ions, definite amounts of these substances, were added to the test solution and the above procedure was repeated.

Calculations

$$\text{Amount of tin (mg)} = N \times V \times E$$

Where N , normality of the titrant; V , volume of the titrant used for the titer; and E , equivalent weight of tin.

TABLE 1. DETERMINATION OF TIN(II).

Tin taken (mg)	Tin found* (mg)	Error (%)
3.05	3.06	+ 0.48
6.57	6.59	+ 0.41
13.14	13.15	+ 0.05
15.25	15.28	+ 0.25

TABLE 2. DETERMINATION OF TIN(II).

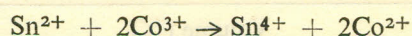
Tin taken (μg)	Tin found* (μg)	Error (%)
153.01	154.02	+ 0.66
306.00	309.00	+ 0.98
614.25	617.17	+ 0.48
767.03	772.22	+ 0.68

TABLE 3. EFFECT OF Zn^{2+} ON TIN(II) DETERMINATION.

Zinc added (mg)	Tin taken (mg)	Tin found* (mg)	Error (%)
0.49	1.89	1.90	+ 0.53
0.98	1.89	1.91	+ 1.06
4.90	1.89	1.96	+ 0.53
24.50	1.89	1.88	+ 0.53
49.00	1.89	1.91	+ 1.66

Results and Discussion

The estimation of tin(II) is based on the fact that tin is directly oxidized from divalent to tetravalent state whereas trivalent cobalt in itself is reduced to divalent state. The total reaction underlying this determination is represented by the following equation:



According to Tables 1 and 2, tin(II) can be easily estimated between the limits of 3.05 mg to 153.00 μg with a maximum error of 0.98%. The reaction is very fast and is marked by the sharp change of the indicator colour from red to colourless at end-point.

The presence of Zn^{2+} from about 4 times less to 25-folds more, Ag^{+} from about 6 times less to 15-folds more and Al^{3+} from about 5 times less to 21-folds more to that of Sn^{2+} concentration has got no significant interference on its determination. The reaction goes quite fast and the end-point is exhibited by a sharp change of indicator colour from red to pale blue. The results of these studies are exhibited in Tables 3-5.

*Average of 7 observations.

TABLE 4. EFFECT OF Ag^{+} ON TIN(II) DETERMINATION.

Silver added (mg)	Tin taken (mg)	Tin found* (mg)	Error (%)
0.46	3.04	3.08	+ 1.30
0.92	3.04	3.04	—
4.60	3.04	3.08	+ 1.30
23.00	3.04	3.08	+ 1.30
46.00	3.04	3.08	+ 1.30

TABLE 5. EFFECT OF Al^{3+} ON TIN(II) DETERMINATION.

Aluminium added (mg)	Tin taken (mg)	Tin found* (mg)	Error (%)
0.85	4.00	4.01	+ 0.25
1.71	4.00	4.01	+ 0.25
8.55	4.00	4.01	+ 0.25
42.75	4.00	4.01	+ 0.25
85.50	4.00	4.01	+ 0.25

TABLE 6. EFFECT OF Pb^{2+} ON TIN(II) DETERMINATION.

Lead added (mg)	Tin taken (mg)	Tin found* (mg)	Error (%)
0.82	2.42	0.59	— 75.00
1.63	2.42	0.49	— 79.70
8.15	2.42	0.16	— 93.40
40.75	2.42	No results	
81.50	2.42	No results	

The presence of lead hampers with Sn^{2+} determination to the extent that it becomes absolutely impossible to oxidise the substance with Co(III) . The results of these studies are shown in Table 6. The possible explanation for this interference could be the formation of lead chloride.

The rate of reaction between tin(II) and cobalt(III) is very fast and one titration could be done even in 2-4 min. The end-point is very sharp. The method reported here for tin(II) determination is very quick, precise and accurate with the added advantage that Sn^{2+} can be determined even in the presence of Zn^{2+} , Ag^{+} and Al^{3+} . Further work on the problem is in hand.

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References

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