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A CONVENIENT VOLUMETRIC METHOD FOR THE DETERMINATION OF TITANIUM WITH COBALT (III) ACETATE

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Abstract. A volumetric method for the determination of titanium with Cobalt (III) acetate as redox titrant is described. Effects of foreign ions have also been studied.

Out of the various known volumetric methods for the determination of titanium, some procedures are cumbersome while others are either time consuming or less sensitive.[1–4] Therefore a simple, quick, precise and sensitive volumetric method for the determination of titanium has been urgently needed. Cobalt (III) acetate exhibits a very high redox potential which has been previously used for the determination of a variety of compounds.[5–16] With a view to extend the usefulness of cobalt (III) acetate as a volumetric titrant for devising some simple practicable method to determine titanium, these studies were undertaken and some of the results are reported here.

Experimental

Reagents

Cobalt (III) Acetate. The solution of cobalt (III) acetate was prepared by the anodic oxidation of cobalt (III) acetate using the method of Sharp and White[6] modified by Hanif *et al.*[11] The final solution is standardized using standard ferrous sulphate.[7]

Titanium (III) Solution. Appropriate solution of titanium (III) is obtained by taking 0.7 g TiO_2, 25 ml conc H_2SO_4 and 8 g of $(NH)_2SO_4$ in a beaker and heating the contents at low temperature to get a clear solution, which is further diluted with 4N H_2SO_4 to get 1 litre final solution. Titanium (IV) in this solution is reduced to trivalent state with Zn/Hg amalgam and standardized against $KMnO_4$.[2]

Apparatus

Potential measurements are made with a potentiometer (W.G. Pye, Cambridge) using platinum and saturated calomel electrodes as indicating and reference electrodes respectively.

All officially calibrated glassware was used for various measurements.

Procedure

A definite aliquot (0.5-25ml) of titanium (III) is taken in a 250 ml beaker, to which water and H_2SO_4 are added to prepare 100 ml of solution, which is 1N with respect to H_2SO_4. Titration is carried out by adding cobalt (III) acetate solution from 10 ml microburette graduated at 0.02 ml intervals. Thorough stirring of the reactants is done throughout the course of the titration with electromagnetic stirrer. Addition of the titrant near and after equivalence point is made in 0.02 ml lots. The volume consumption of the titrant is read from the graph showing ml of the titrant versus mV. Platinum and saturated calomel electrodes are thoroughly washed with distilled water after each titration.

Results and Discussion

The reaction proceeds according to the following equation:

$$Ti^{3+} + Co^{3+} \rightarrow Ti^{4+} + Co^{2+}$$

According to the reaction one mol of titanium (III) loses one electron and is oxidized to tetravalent titanium whereas one mol of trivalent cobalt gains one electron and is consequently reduced to divalent state. The reaction is fast and potential, it stabilizes at once after each addition of the titrant. The stabilization of potential even before at, and after equivalence point is reasonably fast. In order to look for the oxidizing effect of atmospheric oxygen on Ti^{3+} a special assembly is used,[14] but according to observations the use of simple procedure as detailed earlier is quite satisfactory for normal work. Out of the various media used for the quantitative oxidation of Ti^{3+} to Ti^{4+}, the best results are obtained in 1N H_2SO_4. According to some of the results reported in Table 1, Ti^{3+} can be conveniently determined from 310.50 μg to 9.88 mg with a maximum error of −3.38%. With further decrease in the amount of Ti^{3+} taken, the error increases to yield results loaded with greater error.

Effect of Diverse Ions

Copper and magnesium virtually do not interfere upto the levels reported in Table 2. They even do not effect the rate of reaction between Co^{3+} and Ti^{3+}, whereas iron (divalent and trivalent), vanadium, aluminium and calcium do interfere.

On the basis of these investigations, it is claimed that the method is simple, precise, sensitive and accurate and therefore, can be conveniently used for

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TABLE 1. DETERMINATION OF TITANIUM.

Titanium given	Titanium found	Error %	$\frac{\triangle mV \text{ for } 0.02 \text{ ml}}{\triangle ml \text{ of } Co^{3+} (0.01N)}$
301.50 μg	291.30 μg	—3.38	4.22
659.00 "	655.00 "	—0.61	2.26
1.98 mg	1.98 mg	±0.0	0.94
3.30 "	3.30 "	±0.0	0.95
4.22 "	4.21 "	—0.24	1.15
6.59 "	6.54 "	—0.76	0.91
9.05 "	9.05 "	±0.00	0.29
7.90 "	7.92 "	+0.25	0.06
9.88 "	9.89 "	+0.10	0.12

TABLE 2. DETERMINATION OF TITANIUM IN THE PREESNCE OF FOREIGN IONS.

Titanium given mg	Copper added mg	Magnesium added mg	Titanium found mg	Error %
1.25	2.19	—	1.24	—0.80
1.25	6.57	—	1.24	—0.80
1.25	10.95	—	1.24	—0.80
1.25	15.33	—	1.24	—0.80
1.25	21.90	—	1.24	—0.80
1.30	—	3.94	1.31	+0.77
1.30	—	11.83	1.31	+0.77
1.30	—	19.72	1.31	+0.77
1.30	—	27.61	1.31	+0.77
1.30	—	39.44	1.31	+0.77
1.30	—	78.88	1.31	+0.77

the determination of titanium even in the presence of copper and magnesium.

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