

# Technology Section

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## ESTIMATION OF SOME ORGANIC COMPOUNDS WITH HEXAMMINECOBALT(III) TRICARBONATOCOBALTATE(III) AS REDOX TITRANT

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**Abstract.** Hexamminecobalt(III)tricarbatocobaltate(III) has been successfully used for the estimation of hydroquinone, *p*-aminophenol and *p*-phenylenediamine even in  $\mu\text{g}$  amounts in 4.0N  $\text{H}_2\text{SO}_4$  as medium. The method described here can be practically used for the quantitative determination of the compounds.

In our previous communications<sup>1,2</sup> we had discussed the method of preparation of the solution of hexamminecobalt (III)tricarbatocobaltate(III) [ $(\text{Co}(\text{NH}_3)_6\text{Co}(\text{CO}_3)_3)$ ] and had reported the results of successfully using the titrant as a volumetric oxidimetric reagent for the estimation of iodide, alongwith effects of chloride and bromide on its determination, and tin(II) and effects of foreign ions like  $\text{Zn}^{2+}$ ,  $\text{Ag}^{1+}$ ,  $\text{Al}^{3+}$  and  $\text{Pb}^{2+}$ . The solution of the substance, prepared in saturated sodium bicarbonate, keeps its factor fairly constant thereby extending the scope of its use in volumetry.

The present work is an extension of the same work and a method for the estimation of hydroquinone, *p*-aminophenol and *p*-phenylenediamine with the titrant has been reported.

### Experimental

#### Reagents

**Hexamminecobalt(III)tricarbatocobaltate(III).** An appropriate solution was prepared from analytical grade compounds and its factor was checked according to the method discussed earlier.<sup>1</sup>

**Hydroquinone.** An approximately 0.01N solution of the substance was prepared (from Analar reagent) and its exact normality was checked against standard  $\text{K}_2\text{Cr}_2\text{O}_7$  solution.<sup>3</sup> Solutions of low concentrations were made by exact dilution of this stock solution. The normality of the solution was checked daily before use.

***p*-Aminophenol.** An approximately 0.01N solution of the A.R. grade substance (E. Merck) was prepared in water and its factor was determined with the standard solution of  $\text{K}_2\text{Cr}_2\text{O}_7$ .<sup>3</sup> Other solutions of low

concentrations were obtained by accurate dilution of the solution and the test solution was checked for its factor daily.

***p*-Phenylenediamine.** A 0.01N water solution was prepared from A.R. grade (BDH) compound and the factor was checked against a standard solution of  $\text{K}_2\text{Cr}_2\text{O}_7$ .<sup>3</sup> Further exact dilutions were made to get solutions of low normalities. The factor of the test solution was checked every day before use.

**Potassium Dichromate.** A 0.01N solution was obtained by dissolving an exactly weighed quantity of  $\text{K}_2\text{Cr}_2\text{O}_7$  G.R. reagent (E. Merck). Further exact dilutions were made to get solutions of low normalities and these solutions were used as such.

**Ferroin.** M/40 Ferroin (Fluka, Switzerland) was used as an indicator.

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

All officially calibrated glassware was used for various volumetric measurements.

**Procedure.** A definite aliquot (1-15 ml) of the solutions of any of the substances under test was taken in a 250-ml conical flask to which water and  $\text{H}_2\text{SO}_4$  were added in such a quantity as to make a total volume of about 50 ml, 4.00N with reference to  $\text{H}_2\text{SO}_4$ . About 1 drop of Ferroin indicator was added to the contents of the flask and the titration was carried out by adding the titrant solution, in small additions, from a 10-ml microburette graduated at 0.05 ml intervals. Contents of the conical flask were shaken while the addition of the titrant was being done. The end-point was reached by a sudden and sharp change of the colour of the indicator. The end-point colours for various substances are discussed at their respective places. The exact volume of  $\text{Co}(\text{NH}_3)_6\text{Co}(\text{CO}_3)_3$  used for the exact oxidation of any substance

was determined by subtracting the volume consumption of the titrant for blank from that the total. The whole procedure was followed for all the substances under examination.

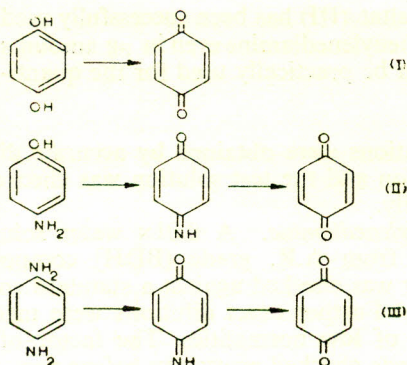
#### Calculations

Amount of the substance under test =  $N \times V \times E$  where  $N$ , normality of  $\text{Co}(\text{NH}_3)_6\text{Co}(\text{CO}_3)_3$ ;  $V$ , volume of  $\text{Co}(\text{NH}_3)_6\text{Co}(\text{CO}_3)_3$  used for the titer; and  $E$ , equivalent weight of the test substance.

#### Results and Discussion

The present studies are based on the principle that Cobalt(III), released from the latter half of  $\text{Co}(\text{NH}_3)_6\text{Co}(\text{CO}_3)_3$  in acidic medium, directly oxidises hydroquinone, *p*-aminophenol and *p*-phenylenediamine to their quinonide stage as their final entity.

The following equations represent the course of the reaction of the tested compounds with cobalt(III).



It has been observed in all the three cases that the reaction goes very smoothly and fast and one titration could be completed within about 5 min. The titrant was always added slowly with constant stirring of the reactants.

According to the results exhibited in Table 1 it is clear that hydroquinone, *p*-aminophenol and *p*-phenylenediamine from 8.25 mg to 55.00  $\mu\text{g}$ , 8.25 mg to 55.00  $\mu\text{g}$  and 8.10 mg to 55.00  $\mu\text{g}$ , respectively could be estimated with maximum respective error of 0.73, 0.72 and 0.93%. It has also been experienced that error is greater in higher concentration ranges of the test solutions while it drops in case of low concentrations of the substances. The end-point in all the three cases is marked by a very sharp change in colour which remains stable for a reasonable period. The end-point in all the cases, in mg ranges, is marked by pale yellow while in  $\mu\text{g}$  ranges it is pale blue.

Due to the fastness of the reaction, precision and accuracy observed in the results obtained, the method

TABLE 1. ESTIMATION OF HYDROQUINONE, *p*-PHENYLENEDIAMINE AND *p*-AMINOPHENOL IN 4N  $\text{H}_2\text{SO}_4$ .

Substance	Taken	Found*	Error (%)
Hydroquinone	1.10mg	1.10mg	—
	2.75	2.77	+0.73
	5.50	5.52	+0.36
	8.25	8.27	+0.24
	55.00 $\mu\text{g}$	55.02 $\mu\text{g}$	+0.04
	110.00 "	110.00 "	—
	275.00 "	275.00 "	—
	550.00 "	549.00 "	+0.20
	825.00 "	825.00 "	—
	<i>p</i> -Aminophenol	1.10 mg	1.10 mg
2.75 "		2.77 "	+0.72
5.50 "		5.51 "	+0.18
8.25 "		8.28 "	+0.36
55.00 $\mu\text{g}$		55.00 $\mu\text{g}$	—
110.00 "		110.00 "	—
165.12 "		165.13 "	+0.01
275.00 "		275.00 "	—
550.00 "		550.00 "	—
<i>p</i> -Phenylenediamine		1.08 mg	1.08 mg
	2.70 "	2.70 "	—
	5.40 "	5.41 "	+0.18
	8.10 "	8.11 "	+0.14
	54.00 $\mu\text{g}$	54.00 $\mu\text{g}$	—
	108.00 "	108.54 "	+0.50
	270.00 "	271.08 "	+0.03
	540.00 "	542.70 "	+0.50
	810.00 "	815.40 "	+0.66

\*Means of 7 titrations.

prescribed here could be used for practical purposes. The simplicity of the method could be realised from the fact that it does not require any special arrangement except that simple and officially calibrated glassware is required. The method is simple and easy to operate.

#### References

1. M. Hanif, M. Saleem and Z. Sheikh, *Mikrochim. Acta*, (submitted).
2. M. Hanif, J.I. Toor and S.H. Qureshi, *Pakistan J. Sci. Ind. Res.* (submitted).
3. M. Hanif, Ph.D. thesis, submitted to Charles University, Prague, 1968, p. 36.