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STUDY OF HEXAMINECOBALT (III) TRICARBONATOCOBALTATE (III) AS ANALYTICAL REAGENT: DETERMINATION OF CATECHOL

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The usefulness of hexaminecobalt (III) tricarbonatocobaltate (III) as analytical titrant has been extended for the successful determination of catechol in μg amounts. A study has been made through which the best acidic media – H_2SO_4 3N and 4N, HCl 1N and 2N, from various concentrations of H_2SO_4, HCl, $HClO_4$ and HOAC have been found out for this determination. Catechol in 4.0N H_2SO_4 medium can be determined within the range from 55.0 μg to 990.0 μg with a maximum error of + 3.03% for the later amount and in 2.0N HCl within the range from 99.0 μg to 925.0 μg with the maximum error of + 7.56% for latter amount. The results reported are quite useful and the method has been recommended for routine use.

Key words: Hexaminecobalt (III) Tricarbonatocobaltate (III); Catechol Determination.

INTRODUCTION

Due to the strong oxidizing behaviour of hexaminecobalt (III) tricarbonatocobaltate (III) it has been used for the determination of a variety of organic and inorganic compounds [1-6]. It is stable at pH 7.5 to 8.0 but in acidic range it gives divalent cobalt in which state it shows a redox potential of 1.8V [7]. In order to extend its usefulness in analytical chemistry, we have tried to use this titrant for the determination of catechol and some of the results of this investigation are reported here.

EXPERIMENTAL

Reagents

Ferrous sulphate. In 200 ml of distilled water containing 5.0 ml of concentrated H_2SO_4, were dissolved 7.0 g of ferrous sulphate ($FeSO_4$ $7H_2O$ – BDH), and the total volume was made to 250 ml with distilled water. The solution so prepared was calibrated against $K_2Cr_2O_7$ conventionally.

Potassium dichromate. To prepare a 0.1N solution, 4.904 g of the pure sample (Merck) was dissolved in one litre of water.

Catechol. Pure catechol (BDH), 250 mg, was dissolved in 250 ml distilled water and its normality was checked against $K_2Cr_2O_7$ using diphenylamine sulphonate as indicator and marking the end point with the appearance of the permanent violet blue colour. The solutions of low normalities were made by taking exact volume from the stock solution and diluting with distilled water. The normality of the solution was checked daily before use.

Sulphuric acid. The sulphuric acid used was A.R. Grade (BDH) and its different normalities were made by dilution method.

Hydrochloric acid. Like sulphuric acid, different concentrations of hydrochloric acid were prepared and used in this investigation.

A 1/40M Ferroin (Fluka) solution was used as redox indicator.

All other chemicals used were of A.R. Grade or of equivalent purity.

Hexaminecobalt (III) tricarbonatocobaltate (III). The solution of hexaminecobalt (III) tricarbonatocobaltate (III) was prepared according to the previously described method and it was standardized against ferrous sulphate with ferroin as indicator [7].

Shelf-life of hexaminecobalt (III) tricarbonatocobaltate (III). The study of the effect of temperature on the shelf-life of the titrant was carried out by keeping a portion at 20°, the second one at 25°, the third one at 35° and the fourth one at 40°C, and the normalities of these solutions were determined as described above.

The Determination of catechol. A definite aliquot of the catechol solution was taken in a conical flask, to which 20 ml of a definite concentration of either of the acids like H_2SO_4 or HCl or $HClO_4$ or HOAC were added and the solution was titrated against the standard hexaminecobalt (III) tricarbonatocobaltate (III) using ferroin as indicator. The end-point was reached when the colour of the reactants changed from red to pale blue.

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A blank titration was always run and the correction for the blank was made. The amount of the catechol found was calculated according to the conventional method. The procedure was repeated with various concentrations of H_2SO_4, HCl, $HC10_4$, and HOAC.

RESULTS AND DISCUSSION

According to the Table 1, in the case of the study of the effect of temperature on the normality of this titrant, when its various four portions were kept at 20°, 25°, 35° and 40° respectively the concentration decreased more at higher temperature. In other words, the higher the temperature the more is the decrease in concentration of the titrant or it is more stable at lower temperatures.

Table 1. Showing the shelf-life of hexaminecobalt (III) tricarbonatocobaltate (III) at 20°, 25°, 35° and 40° Initial strength of the titrant = 0.0060N

Temperature (°C)	Normality of the titrant (N) after 3 days
20	0.0060
25	0.0060
35	0.0058
40	0.0049

Sulphuric acid 0.5, 1.0, 2.0, 3.0, 4.0, 5.0, and 8.0 N; hydrochloric acid 0.1, 1.0, 2.0, 3.0, 5.0 and 7.0 N; perchloric acid 5, 10 and 20% and acetic acid 10, 30 and 50% were used as media for the searching of the best media for the quantitative determination of catechol with hexaminecobalt (III) tricarbonatocobaltate (III). It was observed that catechol could be determined quantitatively in 3N and 4N H_2SO_4, preferably with more accurate results in the later medium (Table 2). Similarly as shown in the Table 2, in 1N and 2N HCl it could also be determined but with more accuracy in the later case. In the case of $HC10_4$ and HOAC the various concentrations explored did not prove of any use for the purpose and hence they were discarded; the results are full of errors having no analytical importance (Table 2).

According to Table 3, the determination of catechol in 4.0N H_2SO_4 was possible with as low amounts of catechol taken as 55.0 μg to 495 μg and the maximum error of determination was + 0.40% with the initially taken amount of catechol as 495.0 μg. When the amount of the catechol taken increases beyond 660.0 μg and up to 990.0 μg the maximum error in the later case is + 3.03%. In the reaction mechanism, one molecule of catechol loses 2 electrons, whereas one molecule of cobalt (III) gains one electron and is reduced to Co^{+2}. The reaction rate between catechol and hexaminecobalt (III) tricarbonatocobaltate (III) in 4.0N H_2SO_4 as the medium, is very fast. Similarly in 2.0N HCl as the medium, catechol can be determined within the limits from 99.0 μg to 350.0 μg with a maximum error of -0.29% in the later case. The error of determination goes on increasing and thus limits the determination of catechol up to the amount of 350.0 μg. The rate of reaction between catechol and the titrant is reasonably fast like in the case of 4.0N H_2SO_4. The end point is quite sharp and persistant for a reasonable time.

Table 2. Effect of medium on the determination of catechol in various acids

	Medium (N)	Catechol taken (mg)	Catechol found* (mg)	Error (%)
H_2SO_4	0.5	0.240	0.282	+ 17.50
	1.0	0.240	0.256	+ 6.66
	2.0	0.240	0.256	+ 6.66
	3.0	0.206	0.211	+ 2.42
	4.0	0.206	0.205	– 0.48
	5.0	0.206	0.131	– 36.40
	8.0	0.206	0.127	– 38.34
HCl	0.1	481.0 μg	499.0 μg	+ 3.74
	1.0	481.0 "	490.0 "	+ 1.87
	2.0	481.0 "	485.0 "	+ 0.83
	3.0	481.0 "	502.0 "	+ 4.36
	5.0	481.0 "	519.0 "	+ 7.90
	7.0	481.0 "	439.0 "	– 8.73
$HC10_4$	5.0 %	0.481 mg	0.328 mg	– 31.80
	10.0 "	0.481 "	0.604 "	+ 25.57
	20.0 "	0.481 "	1.127 "	+ 134.30
HOAC	10.0 "	0.240 "	0.366 "	+ 52.50
	30.0 "	0.240 "	0.192 "	– 20.00
	50.0 "	0.240 "	0.091	– 62.08

*Average of 7 titrations.

Due to its being quite convenient, accurate, precise, sensitive and easy to handle, the method could be used for the routine analysis of catechol.

Table 3. The determination of catechol in 4.0N H_2SO_4 and 2.0N HCl [$(Co(NH_3)_6\ Co(CO_3)_3$ = 0.005N]

	Medium (N)	Catechol taken (μg)	Catechol found* (μg)	Error (%)
H_2SO_4	4.0N	55.00	55.05	+ 0.09
		137.50	137.50	± 0.00
		220.00	220.50	+ 0.23
		330.00	330.80	+ 0.24
		495.00	497.00	+ 0.40
		660.00	673.00	+ 1.96
		990.00	1020.00	+ 3.03
HCl	2.0N	99.00	99.15	+ 0.15
		195.00	194.50	− 0.25
		220.00	220.00	± 0.00
		250.00	249.50	− 0.20
		300.00	300.55	+ 0.18
		350.00	349.00	− 0.29
		396.00	404.00	+ 2.02
		594.00	616.00	+ 3.70
		750.00	795.00	+ 6.00
		925.00	995.00	+ 7.56

(continued. . . . .)

*Average of 7 titrations.

REFERENCES

1. M. Hanif, M. Saleem and Z. Sheikh, Pakistan J. Sci. Ind. Res., **17**, 91 (1974).
2. M. Hanif, M. Saleem, Z. Sheikh and J. Zyka, Mikrochim. Acta (Wien), 625 (1976 II).
3. M. Hanif and M. Saleem, Pakistan J. Sci. Ind. Res., **19**, 9 (1976).
4. M. Hanif, M. Ahmad and M.S. Chaudhry, Pakistan J. Sci. Ind. Res., **20**, 321 (1977).
5. M. Hanif, M. Ahmad, M.S. Chaudhry and Z. Sheikh, Pakistan J. Sci. Ind. Res., **20**, 255 (1977).
6. M. Hanif, I. Ijaz, M. Ahmad and S.H. Qureshi, Pakistan J. Sci. Ind. Res., **26**, 13 (1983).
7. J.A. Baur and C.E. Bricker, Anal. Chem., **37**, 1461 (1965).