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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 μ g amounts. A study has been made through which the best acidic media – H₂SO₄ 3N and 4N, HCl 1N and 2N, from various concentrations of H₂SO₄, HCl, HClO₄ and HOAC have been found out for this determination. Catechol in 4.ON H₂SO₄ medium can be determined within the range from 55.0 μ g to 990.0 μ g with a maximum error of + 3.03% for the later amount and in 2.ON HCl within the range from 99.0 μ g to 925.0 μ g 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.

- 11.596 (1.196
- 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 (FeSO₄ 7H₂O - 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 $H_2 SO_4$ or HCl or HClO₄ 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_2 SO₄, HCl, HClO₄, 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) Image: Comparison of the
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 H₂SO₄, 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 HC10₄ 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 μ g to 495 μ g and the maximum error of determination was + 0.40% with the initially taken amount of catechol as 495.0 μ g. When the amount of the catechol taken increases beyond 660.0 μ g and up to 990.0

	Medium (N)	Catechol taken (mg)	Catechol found*	Error (%)
		(8)	(8)	
H ₂ SO 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
HC1	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
	1940 (J. 1987) (J.	나타 고테르크	- Mississi I	coleala (1911)
HClO 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

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

*Average of 7 titrations.

 μ g 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 H₂SO₄ as the medium, is very fast. Similarly in 2.0N HCl as the medium, catechol can be determined within the limits from 99.0 μ g to 350.0 μ g 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 μ g. The rate of reaction between catechol and the titrant is reasonably fast like in the case of 4.0N H₂SO₄. The end point is quite sharp and persistant for a reasonable time.

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

		Cat	Catechol		
	Medium	taken	found *	Error	
	(N)	(µg)	(µg)	(%)	
H ₂ SO 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	
HC1	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	

Table 3. The determination of catechol in 4.0N H_2SO_4 and 2.0N HCl [(Co(NH₃)₆ Co(CO₃)₃ = 0.005N]

continued....)

EAPER MENTAL

bymarene wheels (connection but) centre of the spin synthesize whee collected from a front finite spin file flat leaves were defed and turned to ash at 900⁴ which tools 5 hr

Wickereddare acid: Anilar Gode HCI was used for the preparation of solution for studie absorption studies without further purification.

Proposition of solution for acousts absorption studies 100 \pm of Gymmuna sylvestrp leaves were taken for experimental work. The ask of the above mentioned hard was prepared by burning the above mentioned hard was calig-heated oven (Thermolyne type 1306 Furnace). It was observed that higher temperature nucceases the amount of unsoluble materials. The test was first treated with 10% HCL then with 20% HCL and finally with ouncentrated HCL that the coloured material went into solution which was 8.5% of the total material About 1.5% of a white insoluble material temained. The solution was the solution which 250 ml by adding distilled water for atomic alignation and/w.

Measurements of absorption were carried out in the ketonic phase by using the Perkin Elmer, 180-B atondo absorption spectro-photometer with lamps having different wavelongths. The results are given in the Tenke 1.

5	94.00 61	6.00 +	3.70
7	50.00 79	5.00 +	6.00
9	25.00 99	5.00 +	7.56

*Average of 7 titrations.

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as gammar tant in the Pariph because the chewing of this herb, followed by the sufing of yar (inggery), gives no sense of sweetness. Gid physicus considered that by cating this herb the excessore amoent of the sweet reatorfal in the body will be constructed and diabetes cured. The real fact is that the presence of charanium in the herb is one of the major factors controlling diabetes atthough, there is no direct link between the loss of tasts and diabetes to direct link between the loss of tasts and diabetes of presents the taste of anget and all the toogue latter on. Effictive or the loss of tasts and diabetes of presents the taste of anget and all other saccharms to apprendix the taste of anget and all other saccharms abbitances was abolished [4]. This was later confirmed by Hooper [5] who observed that the leaf also had the latter only for one to two lears. On account of the property of gammar (from gar, raw sugar, and mar, to kill property all doing away the taste of sigar if has been given the name and doing away the taste of sigar if has been given the name signer descroying, Hoopen [6, 7], in 1887 carried out a facture of (C_3, H_{cd}) , quencifol, gymermic acid and hestricolen (C_3, H_{cd}) , quencifol, gymermic acid and papelied individually on to the taggies to fine observe house applied individually on to the taggies to fine observe house applied individually on to the taggies to the observe house applied individually on to the tangues to the observe house applied individually on to the tangues to the observe house gausia effect but no positive indications were fortheoring were

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