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SHELF-LIFE AND STABILITY STUDIES OF HEXAMMINECOBALT(III) TRICARBONATOCOBALTAE(III)

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Abstract. Hexamminecobalt(III) tricarbonatocobaltate(III) as titrant keeps its factor fairly constant over a reasonable period. Hydrochloric acid from 1 to 8N, sulphuric acid from 0.5 to 10.0N, acetic acid from 10 to 80 and 2 to 10% and perchloric acid have proved quite suitable media for redox studies with this titrant.

Hexamminecobalt(III) tricarbonatocobaltate(III) $[Co(NH_3)_6Co(CO_3)_3]$, relatively a newer redox titrant, has been successfully used in these Laboratories for the determination of various inorganic and organic compounds.[1-5] Its redox potential, in various acid media, being reasonably high,[6] lends importance for its use as a potential titrant in volumetric analysis. Whereas studies on utilizing its high redox potential in volumetric measurements for various determinations are being carried out by us, we thought it necessary to investigate about its shelf-life and suitability of the media of hydrochloric, sulphuric, acetic and perchloric acids. The media proved to be quite suited to our previous investigations.[1-5]

Experimental

Reagents. The solution of hexamminecobalt(III) tricarbonatocobaltate(III) was prepared and its normality checked according to previously described method.[1]

Hydrochloric, sulphuric, acetic and perchloric acids were of analytical grade (Merck), and the other reagents were either of A.R. or equivalent purity.

Apparatus. A pH meter (Pye, Cambridge, England) with saturated calomel as reference and platinum as indicator electrodes was used. A grade, officially calibrated, glassware was used throughout these investigations.

Procedure. An aliquot (5 ml) of $Co(NH_3)_6$-$Co(CO_3)_3$ solution was measured out by a 5-ml burette, calibrated at 0.01 ml intervals, in a 250-ml beaker. Water and the required acid were added to make the total vlume to 50 ml which was finally of required strength with respect to the particular acid. The contents of the reaction vessel were stirred with a magnetic stirrer and the potential reading was recorded when the needle of the potentiometer became stable. Successive potential readings were recorded at regular intervals. The whole procedure was repeated for the acids and their respective required concentrations.

For studying the shelf-life of Co(III) solution a standardised solution was divided into two portions, each one of them kept in a stoppered bottle, one in the dark and the other on the laboratory bench. The normality of these test solutions was checked after regular intervals according to the method already described.[1]

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Results and Discussion

Hydrochloric acid (1–8N) is a very suitable medium and it has almost no effect on the reduction of Co(III). Although the strength of Co(III) solution starts decreasing in 8N HCl after about 20 min inverals yet the medium is quite suited to many determinations. Sulphuric acid (0.5–10.0N), acetic acid (10–80%) proved to be quite suitable media for oxdiation studies of various substances with Co(III) as these acids like HCl have no tendency of reducing the titrant (Table 1).

TABLE 1. POTENTIALS (mV) AGAINST TIME (min), FOR VARIOUS CONCENTRATIONS OF HYDROCHLORIC, SULPHURIC, ACETIC AND PERCHLORIC ACIDS, IN A TOTAL VOLUME OF 50 ml CONTAINING 5 ml OF 00.0046N $Co(NH_3)_6(CO_3)_3$ SOLUTION.

Acid	Time (min)	Potential (mV)
Hcl	0	1095
1N	20	1095
	40	1090
	60	1085
	80	1080
	90	1075
	130	1075
3N	0	1045
	20	1045
	40	1040
	60	1040
	80	1035
	90	1035
5N	0	1020
	10	1020
	20	1020
	40	1015
	60	1015
	80	1010
	90	1010
8N	0	940
	10	920
	20	905
	50	885
	80	880
	90	880

(Table 1 continued)

H_2SO_4		
0.5N	0	1400
	10	1400
	30	1400
	50	1395
	70	1390
	90	1380
2N	0	1400
	10	1400
	30	1400
	50	1395
	70	1395
	90	1390
6N	0	1400
	10	1400
	30	1400
	40	1400
	80	1395
	90	1395
8N	0	1400
	20	1400
	60	1400
	70	1395
	90	1395
CH_3COOH		
10%	0	1400
	5	1400
	15	1400
	30	1395
	50	1395
	80	1390
	160	1385
30%	0	1395
	30	1395
	50	1398
	90	1385
	120	1380
	140	1370
		1365
50%	0	1390
	5	1390
	20	1390
	50	1385
	80	1385
	120	1380
80%	0	1380
	10	1380
	50	1380
	80	1275
	30	1370
H_2SO_4		
10N	0	1400
	20	1400
	70	1400
	90	1395
HCl		
2%	0	540
	5	700
	15	630
	40	590
	60	580
	90	590
4%	0	885
	15	800
	35	630
	65	580
	120	515
$HClO_4$		
8%	0	900
	15	890
	40	800
	70	552
	90	505
	130	490
10%	0	930
	20	820
	30	805
	35	770
	65	760
	90	740
	125	500

(Continued)

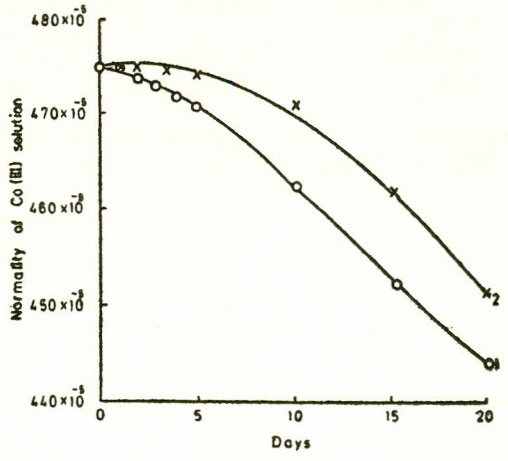

Fig. 1. Normality of Co(III) solution: (1) kept in light, and (2) kept in dark.

Water in the presence of perchloric acid reduces Co(III) and with increasing strength of the acid the rate of reduction is increased. When compared with other media like those of hydrochloric, sulphuric and acetic acids this medium (2–10% perchloric acid) stands relatively less acceptable for the purpose of its being used as a medium. Anyhow the reduction of the titrant, by water in the presence of various strengths of perchloric acid, is not as abrupt as to render it absolutely unfit for its use in oxidation studies.

Regarding shelf-life of the titrant it is evident that the solution kept in the dark is relatively more stable than the solution kept in the normal surroundings (Fig. 1). The change in normalities of both the lots is not so fast. After about five days the normalities start decreasing at quite a rapid rate. Within five days the slow rate of decrease of the strength of the titrant proves it to be reasonably suitable for determinations based on redox measurements.

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