

LETTERS TO THE EDITOR

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THE BAND SPECTRUM OF CHROMIUM
CHLORIDE

THE band spectrum of Chromium Chloride which is prepared for the purpose, using a pure 'Kahlbaum' specimen of chromium, has been excited in the heavy current generator discharge maintained at 1500v, 1 A., in a specially designed quartz discharge tube. Five prominent groups of bands are obtained in the region λ 6400-5700. The bands are line-like and appear very similar to the system of $MnCl$, obtained in our laboratory, in the region λ 4000-3600. The bands show a complex intensity distribution, and are assigned to the electronic transition ${}^6\pi-{}^6\Sigma$, involving high multiplicity terms. The average separation between the components of the ${}^6\pi$ level is obtained as 44 cm.^{-1} , and the values of the vibrational frequencies for the lower and the upper states are :

$$\omega_e'' = 291\text{ cm.}^{-1}$$

$$\omega_e' = 362\text{ cm.}^{-1}$$

Details will be published shortly.

Andhra University, V. RAMA KRISHNA RAO.
Waltair,
February 18, 1949.

VANADAMETRY—PART I

Volumetric Estimation of Ferrous Salts in the Presence of Alcohols

Viswanadham and Gopala Rao¹ have shown that the reaction between ferrous salts and chromic acid induces the reaction between oxalic acid and chromic acid; if a solution of potassium dichromate is employed for the volumetric estimation of ferrous salts in the presence of oxalic acid, the amount of dichromate consumed will be found to be too high. Citric acid has also been shown to interfere by a similar induced mechanism. Gopala Rao and Viswanadham² have shown that the estimation of ferrous salts in the presence of oxalic and citric acids can be accurately carried out by titration with a solution of sodium vanadate.

Extensive investigations have now been initiated to demonstrate the wider application of sodium vanadate as a volumetric reagent and to bring out its exclusive features, if any, when compared with other reagents. We have now found that ferrous salts cannot be accurately estimated by potassium dichromate in the presence of alcohols like methyl, ethyl, isopropyl and *n*-butyl alcohols, the values obtained

being too high. This has been shown to be due to the fact that the reaction between ferrous salt and chromic acid induces the reaction between the alcohols and chromic acid. The excess dichromate solution consumed depends upon various factors, such as the speed of titration, the relative concentrations of ferrous salt and alcohol, the acid concentration, etc. The results recorded in the following table show that the estimation of ferrous salts in the presence of alcohols can be made with accuracy by using a standard solution of sodium vanadate in place of the dichromate solution.

TABLE I

Composition of solution		Amount of ferrous iron found by dichromate method	Amount of ferrous iron found by author's method
Amount of ferrous iron taken	Amount of alcohol solution		
milli mols.	milli mols. of methyl alcohol	milli mols.	milli mols.
0.2259	10.0	0.2526	0.2260
0.4517	10.0	0.5070	0.4518
0.4517	25.0	0.5596	0.4518
0.9034	10.0	0.9713	0.9036
	milli mols. of ethyl alcohol		
0.2397	20.0	0.2887	0.2384
0.4793	20.0	0.5720	0.4792
0.4793	50.0	0.6596	0.4792
0.9586	20.0	1.098	0.9560
	milli mols. of isopropyl alcohol		
0.2160	30.0	0.2352	0.2159
0.4319	30.0	0.4511	0.4319
0.4319	75.0	0.4840	0.4319
0.8638	30.0	0.8913	0.8638
	milli mols. of <i>n</i> -Butyl alcohol		
0.2186	20.0	0.2411	0.2188
0.4319	30.0	0.4511	0.4319
0.4319	75.0	0.4840	0.4319
0.8638	30.0	0.8913	0.8638

Sodium vanadate has thus some special advantages over potassium permanganate and dichromate as a volumetric reagent. It can be used for the estimation of ferrous salts in the presence of oxalic acid, citric acid and the alcohols, where potassium permanganate and potassium dichromate give too high results. Moreover, sodium vanadate solutions can be easily prepared and preserved over long periods without change in titre, unlike potassium permanganate. Ammonium vanadate supplied by Schering Kahlbaum, Merck or B. D. H. has been found to be quite pure. The requisite

quantity of the salt is weighed out carefully into a conical flask, dissolved in distilled water, a slight excess of pure sodium carbonate added and the solution boiled until all the ammonia is driven out. The resulting solution is cooled and transferred to a litre measuring flask and made up to the mark. The strength of the solution is checked up by titration against a standard solution of ferrous ammonium sulphate, using diphenylamine or diphenyl benzidine as internal indicator. A standard solution of sodium vanadate prepared in this way is remarkably stable, especially when containing a slight excess of sodium carbonate, about 0.1 per cent.

Detailed results are being published elsewhere.

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November 20, 1948.

1. Viswanadham and Gopala Rao, *Curr. Sci.*, 1943, 12, 327. 2. —, *Ibid.*, 1944, 13, 180.

REDUCTION OF NITRO GROUP TO AMINO GROUP BY 'HYDRO' IN ALKALINE MEDIUM

THE aromatic amino compounds are useful substances in synthesis as well as in industry. They are prepared by the reduction of the corresponding nitro compounds, the reducing agent used generally being a hydrogen-generating combination of metal and acid; the other reducing agents lead either to the production of hydroxyl-amines, azoxy or azo compounds.¹ Electrolytic reduction also gives different products depending upon the conditions used.²

In connection with other synthetic work, certain amino compounds (e.g., 5-amino-salicylic acid, 6-aminocresol, etc.) were required in quantity. The usual reduction by means of tin and hydrochloric acid did not lead to the desired product in satisfactory yield. Sidgwick and Callow have obtained *p*-amino phenol by incipient sodium hydro-sulphite using sodium sulphite and zinc.³

We thought of using sodium hydro-sulphite (hydro) ($\text{Na}_2\text{S}_2\text{O}_3$) directly as it is easily available and is being used in industry for the reduction of anthraquinone and indigoid derivatives to leuco compounds.⁴ The preliminary experiments were tried and the reduction yielded the