A NOTE ON THE ESTIMATION OF CHLORACETOPHENONE IN MIXTURE

by

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Iyengar and Raju[1] have reported a method of estimating chloracetophenone in a mixture with nitro-cellulose, as is used in tear gas grenades, after hydrolysis in alcoholic solution with sodium phenate and titration by Mohr's method. When this method was repeated by us it was noted that judging the end point was difficult on account of interference by products of hydrolysis. One is apt to overstep the end point and obtain high results. A control flask did not much help as the colour was apt to vary from flask to flask. Considerable improvement was effected by adoption of Volhard's method in lieu of Mohr's method in the titration of the hydrolysed chloride, with a few drops of toluene as the carrier liquid. The results obtained with chloracetophenone are given in Table 1. The chloracetophenone used was recrystallised from alcohol and had a melting point of 54·8°C[2].

TABLE 1

Estimation of Chloracetophenone

Serial No.	Description	Amount taken (gm)	Amount obtained (gm)	Difference %
1	Iyengar and Raju's Method with Mohr titration.	0·125	0·150	+12·0
		0·260	0·289	+11·15
		0·362	0·325	+10·25
2	Iyengar and Raju's Method with Volhard titration.	0·2600	0·2694	+3·6
		0·3033	0·3053	+0·66
		0·3791	0·3807	+0·42
		0·6058	0·6087	+0·48

Inspite of the encouraging results obtained by the above method when tried with chloracetophenone alone, the results obtained with mixtures of chloracetophenone and nitrocellulose as is present in tear gas grenades was too low (Sr. No. 1 of Table 2 below refers). It was therefore necessary to find out a method which will be applicable to the determination of chloracetophenone in mixture. It is known that chloracetophenone is hydrolysed by sodium alcoholate at room temperature in alcohol and alcoholic caustic soda after refluxing. Both these methods were tried as below:—

(*a*) *Sodium alcoholate Method*—A quantity of the mixture containing about 0·4—0·6 gms chloracetophenone and 25 ml. of sodium

alcoholate solution containing about 0·6 gm of sodium was added. After shaking for a few minutes, it was diluted with water and filtered. Filtrate made upto 100 ml after extracting the residue in the flask before each addition. 20 ml of solution was titrated for chloride by the Volhard Method using toluene as the collecting liquid.

(*b*) A quantity of the mixture containing about 0·4—0·6 gms of chloracetophenone was refluxed with 25 ml alcoholic soda containing 1 gm NaOH(N) for half an hour. The solution was cooled, diluted and filtered. The filtrate was made upto 100 ml washing the residue in the flask before each addition. 20 ml of the solution was titrated by the Volhard method using toluene as the collecting liquid.

Control blanks were carried out in all cases. The results obtained are given in Table 2.

TABLE 2

Estimation of Chloracetophenone in mixture

Serial No.	Method	Chloracetophenone		Difference %
		Taken (gms)	Found (gms)	
1	Modifi d Iyengar & Raju Method ..	0·5095	0·4992	—2·05
		0·6772	0·6557	—3·18
2	Sodium Ethoxide method	0·3581	0·3453	—1·08
		0·3672	0·3632	—1·09
		0·4808	0·4662	—3·04
		0·4225	0·4063	—2·61
3	Alcoholic soda method	0·4321	0·4315	—0·15
		0·4321	0·4273	+1·18
		0·4281	0·4273	—0·21
		0·4281	0·4261	—0·23

From the above results it will be seen that the alcoholic soda method gives the most reliable results for estimation of chloracetophenone in the mixture. The variations in the sodium ethoxide method are perhaps due to a reddish tinge that develops during hydrolysis which makes judgement of end point difficult.

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1. Iyengar & Raju, Sci. & Cult. 26, 236, 1960.

2. This appears to be a pure sample as it did not show any further rise in melting point by repeated crystallisation. Mixed melting point obtained from different batches and from different solutions was also the same. The value reported in literature 58°—59°C (Staedcal, Ber. 10, 230, 1870) appears to be too high.