

A NOTE ON THE PREPARATION OF QUINACETOPHENONE

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QUINACETOPHENONE was originally prepared by Nencki and Schmid by the interaction of quinol and glacial acetic acid in the presence of fused zinc chloride but the yield was rather poor.¹ Russell and Clark, following the same method, raised the yield to 25 per cent.² The ketone was also prepared from quinol diacetate through Fries migration. Contradicting the work of Heller³ it was shown by Rosenmund and Lohfert⁴ that the diesters of quinol do undergo the Fries migration and quinacetophenone was obtained in a yield of 23 per cent. Applying the same reaction, the substance was prepared in more satisfactory yields by Shahane,⁵ by Desai and Mavani⁶ and very recently by Amin and Shah.⁷ The best yield reported so far is that of Shahane, *viz.*, 76 per cent.

For the production of quinacetophenone through Fries reaction, so far only aluminium chloride has been used as the catalyst. It has now been found that using zinc chloride in place of aluminium chloride the ketone can be obtained in almost theoretical yields. The yield is dependent on the duration of treatment and reaches the maximum after 9 hours. Heating beyond this period results in the formation of some inorganic combination of the ketone.

For the preparation of the ketone in best yield, fused zinc chloride (10 g.) was dissolved by heating in glacial acetic acid (10 c.c.) and to this solution was added quinol diacetate (10 g.). The mixture was then mildly boiled under reflux under anhydrous conditions for 9 hours. It was then cooled and treated with 50 per cent. hydrochloric acid (100 c.c.) and left overnight. The deposited solid was then dissolved in 10 per cent. sodium hydroxide (100 c.c.), filtered in order to remove any of the original diacetate, if present, and from the clear alkaline filtrate, on acidification with hydrochloric acid, separated the ketone. The latter was subsequently filtered, washed with cold water and recrystallised from dilute alcohol. The ketone was obtained as colourless narrow rectangular plates melting at 201–02° and the yield

was almost theoretical. Its identity as quinacetophenone was confirmed through its oxime, melting at 149-50°.

REFERENCES

1. Nencki, M., and Schmid, W. .. *J. prak. Chem.*, 1881, **23**, 546.
2. Russell, A., and Clark, S. F. .. *J. Am. Chem. Soc.*, 1939, **61**, 2654.
3. Heller, G. .. *Ber.*, 1912, **45**, 2389.
4. Rosenmund, K. W., and Lohfert, H. .. *Ibid.*, 1928, **61**, 2602.
5. Shahane, R. Y. .. *Curr. Sci.*, 1941, **10**, 523.
6. Desai, R. D., and Mavani, C. K. .. *Ibid.*, 1941, **10**, 524.
7. Amin, G. C., and Shah, N. M. .. *J. Ind. Chem. Soc.*, 1948, **25**, 375.