

Nitration of Phthalide

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Nitration studies on phthalide have resulted in the isolation of 3-nitrophthalide, a compound hitherto unlisted in the literature.

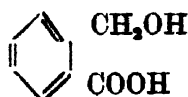
IN the present work a systematic study of nitration on phthalide is reported. The experiments of Kurbatow¹, Teppeme² and Tiroufflet³, were repeated and it was visualised that 6-nitrophthalide as the major product, which is in accordance with the findings of literature.

But with potassium nitrate in concentrated sulphuric acid at room temperature, a reagent used by Tiroufflet (*loc. cit.*), a mixture of products were obtained. The mixture, whose nature was acidic and neutral was readily separated and the components identified individually.

The acidic component

- (i) did not contain nitrogen,
- (ii) gave effervescence of CO₂, with sodium bicarbonate,
- (iii) observed melting point is 127°-8°,
- (iv) equivalent weight is 151.5.

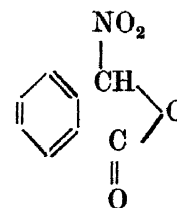
These correspond to the characteristics of *o*-hydroxy methyl benzoic acid reported in the literature.



The neutral component

- (i) showed the presence of nitrogen,
- (ii) had a melting point of 81°.

This did not coincide with any of the known nitro-derivatives of phthalide. Therefore a probable structure was postulated.



and the elucidation of the same thus implemented:

(i) On oxidation of the compound with alkaline potassium permanganate and subsequent acidification, yielded phthalic acid, which indicated the presence of -NO₂ in the side chain.

(ii) Found : C, 53.63; H, 2.79; N, 7.81; C₈H₅O₄N requires C, 53.58; H, 2.71; N, 7.76; hence confirmation of the structure. Further, the use of 3-nitrophthalide as a nitrating agent is recommended.

References

1. KURBATOW, *Ann.*, 1880, 202, 219.
2. T. TEPEME, *Rec. Trav. Chim.*, 1923, 42, 30.
3. J. TIROUFLET, (*Faculte, Sci. Rennes, France*), *Bull. Soc. Sci., Bretagne Special No. 26*, 1951, 7-122.