*Anal.* Calcd. for $C_{23}H_{22}O_{11}\cdot 0.5\ H_2O$: mol. wt., 492.6; C, 57.13; H, 4.80; $OCH_3$ (2), 12.8. Found: mol. wt. (titration, 482); C, 57.29; H, 4.93; $OCH_3$, 12.9.

**Tephrosinmonocarboxylic Acid.**—When isotephrosindicarboxylic acid was boiled for one-half minute with diphenyl ether, tephrosinmonocarboxylic acid was formed in essentially the same yield as recorded for tephrosindicarboxylic acid.[1]

As obtained from the cooled diphenyl ether solution upon the addition of methanol the acid melted at 266–268°, and when it was mixed with an authentic sample of tephrosinmonocarboxylic acid no depression of the melting point occurred. It also gave the characteristic scarlet color with ferric chloride that is obtained with tephrosinmonocarboxylic acid.

## Summary

A crystalline material, shown by its composition and certain of its derivatives to be an isomer of tephrosin, has been obtained from a sample of Peruvian cubé root. This substance has been designated as isotephrosin. The structural difference between tephrosin and isotephrosin exists in the orientation of the hydrogen and hydroxyl groups on carbons 7 and 8 in the structure given in the text.

WASHINGTON, D. C.

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# NOTES

## The Nitration of 4,4′-Dichlorodiphenyl

BY CLARENCE C. VERNON, A. REBERNAK AND H. H. RUWE

Shaw and Turner have recently published an article[1] which concerns in part the exhaustive nitration of 4,4′-dichlorodiphenyl. In it they have shown that the three nitro groups entered the 2,3′,5′ positions.

Without knowledge that the problem was being investigated elsewhere, this study has been in progress in this Laboratory for the past two years. The results attained are in agreement with those of Shaw and Turner, as the following brief account shows. Direct nitration of 4,4′-dichlorodiphenyl at 100°, using a mixture of fuming nitric and fuming sulfuric acids, resulted in the formation of a trinitro derivative with a melting point which corresponded to that of the 4,4′-dichloro-2,3′,5′-trinitrodiphenyl reported by Shaw and Turner. The yield was lower than that secured by them, but the product, precipitated by pouring the filtered reaction mixture over cracked ice, melted at 164–165° without further purification. Nitration of 4,4′-dichloro-2,3′-dinitrodiphenyl under these conditions resulted in the formation of the same compound, as was shown by the melting point of a mixture of the two.

The lability of the chlorine atoms was determined by hydrolysis with dilute sodium hydroxide solutions in the presence of metallic copper, and under quantitative conditions.[2] Only one chlorine atom was found to be

[1] Shaw and Turner, *J. Chem. Soc.*, 285–297 (1932).

[2] Hale and Britton, *Ind. Eng. Chem.*, **20**, 114 (1928).

labile. In the case of the 4,4′-dichloro-2,3′-dinitrodiphenyl, neither of the chlorine atoms was labile under these conditions. This was considered evidence supporting the conclusion that the third nitro group was in the 5′-position.

As a whole, our results confirmed the conclusions arrived at by Shaw and Turner. The fact that they were secured by somewhat different methods renders them still more valuable as supporting evidence.

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## Preparation of Diethylisopropylamine

BY SAUL CASPE

No appreciable reaction takes place between isopropyl bromide and diethylamine on boiling under atmospheric pressure. In the early experiments[1] it was found that a yield of 10% was obtained on heating these reactants for forty-two hours in the presence of copper and sodium bromide. The yield was increased to 30% by the use of an autoclave, with the above accelerators, at 140° for six hours. The reaction was promoted to an even greater extent by the presence of glycerol. A mixture of 123 g. of isopropyl bromide, 94.9 g. of diethylamine and 50 g. of glycerol was gently heated under reflux for seventy-two hours; the resulting amines were liberated with alkali, dried with potassium hydroxide and fractionally distilled, when 67 g. (60% of the theoretical amount) of a product boiling at 108° was obtained. A similar yield was obtained when the glycerol was replaced by an equal weight of ethylene glycol; with half that quantity of mannitol, the yield amounted to only 25 g.

Diethylisopropylamine is a colorless liquid, miscible in all proportions with water. Its specific gravity is 0.75.

*Anal.* Calcd. for $C_7H_{17}N$: C, 73.00; H, 14.80; N, 12.20. Found: C, 73.41; H, 14.82; N, 12.16.

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[1] W. F. Whitmore and S. Caspe, unpublished data, 1930.