

A NOTE ON RESOKÆMPFEROL

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RESOKÆMPFEROL, 3:7:4'-trihydroxy flavone has not so far been found to occur in nature; however, the higher members, fisetin and robinetin and its ether, kanugin have been isolated from plants. The lower member 3:7-dihydroxy flavone is represented by its derivative, karanjin. Though the synthesis and study of resokæmferide, 3:7-dihydroxy-4'-methoxy flavone were reported by Heap and Robinson¹ and later by others,^{2, 3} the trihydroxy flavone, its trimethyl ether or acetate have not been described. Since they have been required in connection with other work done in this laboratory, they have now been made starting from ω -methoxy-resacetophenone and condensing it with anisic anhydride and sodium anisate according to the method of Allan and Robinson. The 7-hydroxy-compound first produced is methylated to yield the trimethyl ether and demethylated to form resokæmferol. Like the other members of this series this flavonol and its ethers exhibit strong fluorescence in solutions.

EXPERIMENTAL

7-Hydroxy-3:4'-dimethoxy flavone

ω -Methoxy-resacetophenone (2 g.) was condensed with anisic anhydride (9 g.) and sodium anisate (3 g.) by heating under reduced pressure at 170–80° for 3 hours. The reaction product was refluxed with 10% alcoholic potash (40 c.c.) for 20 minutes, the solvent distilled off, the residue dissolved in water and the deep brown solution saturated with carbon dioxide. The precipitated flavone was crystallised from alcohol when it separated out in the form of pale yellow prismatic needles melting at 248–50°. Yield, 1.5 g. (Found: C, 68.6; H, 5.0; $C_{17}H_{14}O_5$ requires C, 68.5; H, 4.7%.) It was sparingly soluble in alcohol and the solution did not exhibit any colour with ferric chloride. In concentrated sulphuric acid it showed weak blue fluorescence. The acetate of this crystallised from alcohol in the form of colourless needles melting at 148–49°.

3:7:4'-Trihydroxy flavone

The above 7-hydroxy compound (0.5 g.) was demethylated using acetic anhydride (3 c.c.) and hydriodic acid (5 c.c.). The trihydroxy flavone

crystallised from alcohol in the form of glistening greyish yellow stout rectangular prisms melting with decomposition at 280°. (Found: C, 62·8; H, 4·5; C₁₅H₁₀O₅, H₂O requires C, 62·5; H, 4·2%.) Its alcoholic solution exhibited a green fluorescence and gave a deep violet brown colour with ferric chloride. Its solution in aqueous alkali had a green fluorescence while that in concentrated sulphuric acid had a bright peacock blue fluorescence. It did not give a precipitate with lead acetate.

The triacetate of the flavonol crystallised from alcohol in the form of colourless needles melting at 159–60°.

3:7:4'-Trimethoxy flavone

The 7-hydroxy compound (0·2 g.) was methylated in acetone medium (25 c.c.) with dimethyl sulphate (0·5 c.c.) and potassium carbonate (5 g.). The trimethyl ether crystallised from alcohol as colourless rectangular plates melting at 146–47°. (Found: C, 68·8; H, 5·5; C₁₈H₁₆O₅ requires C, 69·2; H, 5·1%.) Its solution in concentrated sulphuric acid was yellow with a greenish blue fluorescence.

SUMMARY

Resokæmpferol, its tri-acetate and tri-methyl ether are prepared and described.

REFERENCES

1. Heap and Robinson .. *J. C. S.*, 1926, 2342.
2. Algar and Flynn .. *Proc. Roy. Irish Acad.*, 1934, B 42, 1.
3. Nadkarni and Wheeler .. *J. C. S.*, 1938, 1320.