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A New Spot Test for Oximes

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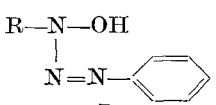
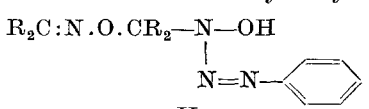
The class of organic reagents known as hydroxytriazenes (I) has been extensively studied in our laboratory[1]. The chelating properties of these compounds were first examined by *Elkins* and *Hunter*[2], later reported that the complexing properties of the so-called arylazo-bis-oximes (II) are extraordinarily similar to those of hydroxytriazenes.



I



II

(where R = an alkyl or an aryl group)

A study of arylazo-bis-oximes was therefore undertaken as an extension of the work on hydroxytriazenes. It was observed that these compounds can be prepared very easily by adopting the method described by *Mai*[3], which consists in coupling diazonium salts (1 mole) with oximes (2 moles) in dilute sodium hydroxide solution ($p_H \sim 10$) at 0° C. The reactions of these compounds with different metal ions are similar to those of hydroxytriazenes. However, the metal chelates of arylazo-bis-oximes are unstable toward heat and dilute mineral acids and thus were not considered suitable for gravimetric estimations of metal ions.

Arylazo-bis-oximes give an intense blue water-soluble complex with ferric ions. This reaction has been utilized for detecting microgram quantities of oximes on a spot-plate. A drop of alcoholic solution of an oxime is made to couple with a drop of diazonium salt in alkaline medium to give an arylazo-bis-oxime. This is indicated by the development of a blue colour with a drop of ferric chloride solution.

Procedure

A drop of the test solution (0.1% alcoholic solution of an oxime) is placed on a spot-plate and mixed with a drop of 5% sodium hydroxide solution. This is followed by a drop of diazonium salt (prepared from one drop of aniline, 1 ml of conc. hydrochloric acid and two drops of 1% sodium nitrite solution) and a drop of 1% ferric chloride solution. Appearance of an intense blue colour shows the presence of an oxime in the test solution; 50 μg of an oxime can be detected in 0.25 ml of total solution on a spot-plate by this method.

The oximes tested for by this procedure were: acetaldoxime, propionaldoxime, acetoxime, isobutyraldoxime, methyl ethyl ketoxime, diethyl ketoxime, crotonaldoxime, furfuraldoxime, piperonaldoxime, acetophenone oxime, benzophenone oxime and benzaldoxime. The test is performed in the absence of phenols and aromatic amines.

The test was not given by dimethylglyoxime, salicylaldoxime and α-benzoinoxime, which may mean that these compounds do not form their corresponding arylazo-bis-oximes. This may be due to absence of a free oxime group in these compounds because of intramolecular hydrogen-bonding.

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Summary

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Oximes couple in alkaline medium with diazonium salts to give arylazo-bis-oximes which develop an intense blue colour with ferric chloride; 50 μg of an oxime can be detected in 0.25 ml of total solution on a spot-plate by this method.

Zusammenfassung

Oxime Kuppeln in alkalischem Medium mit Diazoniumsalzen unter Bildung von Arylazo-bis-oximen, die ihrerseits mit Eisen(III)-chlorid eine intensive Blaufärbung geben. 50 μg Oxim können so in 0,25 ml Reaktionsgemisch auf der Tüpfelplatte nachgewiesen werden.

References

[1] *N. C. Sogani* et al., Analyt. Chemistry **28**, 81, 1616 (1956); **29**, 397 (1957); **33**, 1273 (1961); J. Indian Chem. Soc. **36**, 563 (1959); **37**, 53 (1960); **38**, 771 (1961); **43**, 703 (1966); Z. analyt. Chem. **203**, 181 (1964).

[2] *M. Elkins* and *L. Hunter*, J. Chem. Soc. London **1938**, 1346; **1940**, 653.

[3] *J. Mai*, Ber. dtsch. chem. Ges. **25**, 1685 (1892).

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