

TABLE I
Fungicidal data
(Average percentage inhibition after 96 hours on *A. niger* at concentration 1000 ppm)

Compound	IIIa	IIIb	IIIc	IIId	IIIe	IIIf	IIIg	IIIh	Bavistin
	58.3	52.1	68.6	70.2	68.4	79.8	81.2	70.6	85.8

possibility the title azomethines were synthesised as per scheme given above and were tested against the fungal species.

Experimental

Melting points are taken in open capillary and are uncorrected. The analytical and spectral data of the compounds agreed with the expected values within the limits of experimental errors.

*N*²-Aryl(heteroaryl-2-aryl)benzyl imidochlorides(II)

The appropriate *N*-substituted amides (I) were converted into the corresponding imidochlorides; PCl_5 (0.12 M) was added to a suspension of appropriate amide (0.1 M) in petroleum ether and the reaction mixture was refluxed for 3-4 hours. The solvent and POCl_3 were removed under reduced pressure and the residual product was purified and used as such for further step.

*N*²-Aryl(heteroaryl-2-(2',4'-dihydroxyphenyl)-2-aryl)benzylazomethines (III)⁵

To a mixture of resorcinol (0.2 M) and appropriate imidochloride (1.25 M) in dry ether, an ethereal solution of anhydrous AlCl_3 (1.5 M) was added under continuous stirring. After keeping overnight, the ether was removed and the residue was decomposed with ice and dil. HCl. The product separating was filtered and recrystallised from aqueous ethanol. The yield of compounds varied within the range 47-65%. The compounds thus prepared were IIIa, m.p. 183°; IIIb, m.p. 121-22°; IIIc, m.p. 146-47°; IIId, m.p. 126°; IIIe, m.p. 136°; IIIf, m.p. 205°; IIIg, m.p. 192° and IIIh, m.p. 172°.

Fungicidal Screening

The antifungal activity was evaluated against two species of fungi, viz., *A. niger* and *A. flavus* by agar growth technique⁶ at three different concentrations 1000 ppm, 100 ppm and 10 ppm. The number of replication in each case was three. The percentage inhibition by various compounds are recorded in Table I.

The fungicidal data reveal that some of these compounds are as fungitoxic to *A. niger* as Bavistin at concentrations of 1000 ppm and 100 ppm (Nos. II, f,

g and h) but the same compounds are much less toxic to *A. flavus* at the above concentrations. At a concentration of 10 ppm only those compounds which have chloro- or methyl groups (Nos. IIIf, g and h) or those having heterocyclic moiety (Nos. IIIc and d) are more toxic to *A. niger* but not to *A. flavus*.

In general these compounds appear to be weaker fungicides.

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ESTIMATION OF INDIGOCARMINE BY BROMAMINE-B, DICHLORAMINE-B AND DIBROMAMINE-B

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AROMATIC sulphonyl haloamines have attracted the attention of analytical chemists as versatile redox titrants. The determination of indigocarmine (IC) is of considerable interest in view of its use as a redox indicator in analytical chemistry. A variety of

oxidants such as potassium dichromate¹, cerium (IV)², sodium vanadate³, chloramine-T⁴ and dichloramine-T⁴ have been shown to oxidize IC quantitatively under a prescribed set of experimental conditions. The present investigation reports the oxidimetric estimation of IC in solution with three sulphonyl haloamines, namely, bromamine-B (BAB), dichloramine-B (DCB) and dibromamine-B (DBB).

Materials and Methods

About 2 mM solutions of analar indigocarmine (E'Merck) were prepared by dissolving the required amount of the compound in appropriate solvents and buffers. Bromamine-B⁵, dichloramine-B⁶ and dibromamine-B⁷ were prepared by methods reported elsewhere. Approximately 0.05 N solutions of BAB in water, DCB and DBB in glacial acetic acid were prepared and standardized iodometrically. Standard buffer systems were employed. Analar grade chemicals were used in preparing other solutions. Absorbance measurements on oxidized indigocarmine solutions were carried out on a Beckmann Model DB spectrophotometer.

Recommended Procedure

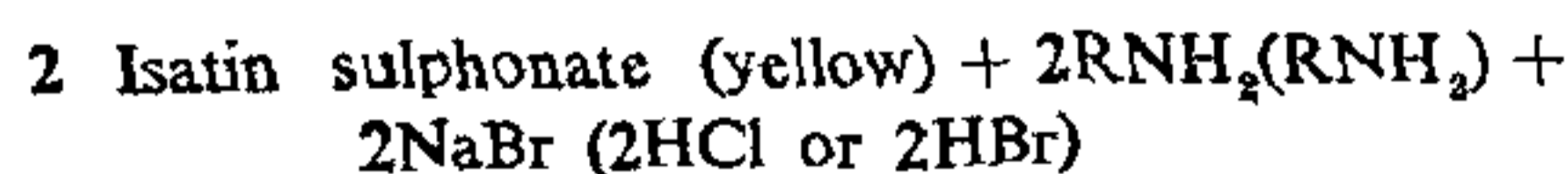
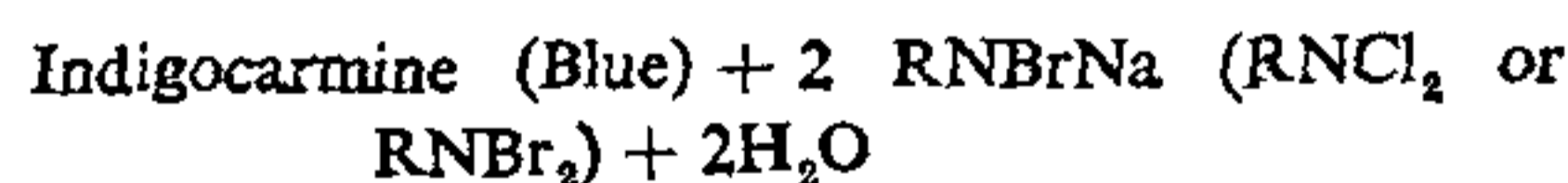
With BAB: Add 25 ml of 0.05 N BAB to aliquots of IC solution (2–60 μ moles) containing enough NaOH to give an overall normality of 0.01–0.1 N. After 30 minutes, estimate the unreacted BAB iodometrically.

With DCB: Add aliquots of IC solution (1–22 μ moles) to 25 ml of 0.05 N DCB. After 30 minutes, estimate the unconsumed DCB iodometrically.

With DBB: Add 25 ml of 0.05 N DBB to aliquots of IC in glacial acetic acid (1–22 μ moles). After 30 minutes, estimate the unconsumed DBB iodometrically.

Results and Discussion

From the results obtained for the estimation of IC with BAB, DCB and DBB, it is found that the error in recovery of the sample is within 1%. The stoichiometry of oxidation of indigocarmine to isatin sulphonate could be represented as



where R = C₆H₅SO₂.

The yellow isatin sulphonate solution has a λ_{max} at 410 nm (10 g ϵ_{max} = 2.8). It was of interest to estimate the amount of this product formed in solutions of indigocarmine oxidized by the sulphonyl haloamines. A calibration graph was prepared with isatin sulphonate and Beer's law was found to obey up to 20 μ moles. A solution of IC was treated with the oxidant and the excess oxidant was destroyed by thiosulphate

through the liberation of iodine. It was then made up to a known volume and absorbance was measured. Checks made for interference by other products of oxidation were found to be negative. From the results of the spectrophotometric analysis it is found that the experimental values obtained are in good agreement with those calculated based on the assumption that two moles of isatin sulphonate are formed per mole of indigocarmine. Benzene sulphonamide formed was detected by TLC⁶.

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STUDIES ON AMIDOALKYLATION REACTION: SYNTHESIS OF α -(AMIDO-ALKYL)-CYCLOHEXANONES AND THEIR CORRESPONDING THIOSEMICARBAZONES

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CYCLOHEXANONE was condensed with several N-hydroxyalkyl amides and imides, (*viz.*, N-hydroxymethyl/ethylphthalimide, N-hydroxymethylbenzamide, N-hydroxyethyl-2-aryl-quinazolin(3H)-4-one etc.) in the presence of conc. H₂SO₄ to give α -(amidoalkyl)-cyclohexanones in excellent yields. A part of this study has also been devoted to the synthesis of the thiosemicarbazone derivatives of these ketones with a view to testing their antiviral activity.

N-hydroxymethylphthalimide has been used widely as an electrophilic reagent¹. The stability of several N-methylol amides and imides toward heat and acid is sufficient to permit their use under drastic reaction conditions². In 1902 Tscherniac³ reported the condensations of N-methylol phthalimide with a series of