NOTES.

The Detection of meta-*Orientation in Diamino-, Dinitro-, and Aminonitro-compounds.* By ADRIEN ALBERT.

IT has been confirmed (Albert and Large, *Nature*, 1938, **142**, 435) that *m*-diamines readily react with oxalic acid, glycerol, and zinc chloride to give fluorescing diaminoacridines. *o*- and *p*-Diamines do not produce a fluorescence. The reaction has been used for the detection or confirmation of *meta*-orientation in diamines. A solution of zinc chloride (15 g.) in glycerol (125 g.) at 100° was cooled to 60°, and powdered hydrated oxalic acid (10 g.) dissolved in it. The substance to be tested was added to 5 g. of the reagent in a test-tube, which was maintained at 160° for 10 minutes and then cooled. The presence or absence of fluorescence (always bright yellow-green) was noted either in bright daylight or alongside a filament lamp (60 watts). The following substances responded to the test; the minimum quantity that was plainly detected is given in mg. in parentheses: *m*-Phenylenediamine (0·4), 2 : 4-tolylenediamine (0·5), 1-chloro-2 : 4-diaminobenzene (1·0), *m*-aminodimethylaniline (0·8), 3-aminodiphenylamine (1·0), 3 : 3'-diaminodiphenylamine (1·0), *m*-aminoacetanilide (2·0), *m*-aminoformanilide (0·5), *NN'*-diacetyl-*m*-phenylenediamine (5·0), *NN'*-diformyl-*m*-phenylenediamine (0·5). As anticipated, the following substances produced no fluorescence: Aniline, *o*- and *p*-phenylenediamines, the toluidines, *m*-chloroaniline, *m*-anisidine, *m*-nitroaniline, *m*-aminobenzenesulphonic acid, *m*-aminobenzoic acid, *m*-dinitrobenzene.

Before the test is applied, the absence of a phenolic group should be ascertained, as this group (alone) interferes with the reaction.

The replacement of half the zinc chloride in the original reagent by its equivalent of stannous chloride permits of the detection of *meta*-orientation also in aminonitro-compounds, fluorescing diaminoacridines being again produced. The following results were obtained: 4-Nitro-*o*-toluidine (0·2), 6-nitro-*o*-toluidine (0·2), *m*-nitroaniline (1·0), 3-nitrodiphenylamine (1·0), 3 : 3'-dinitrodiphenylamine (1·0), 4-nitro-2-aminobenzenesulphonic acid (2·0), 4-nitro-*m*-2-xylidine (0·4), and its monoacetyl derivative (2·0).

In addition, several dinitro-compounds (for which this reagent is equally suitable) have been included, since, although the orientation of two nitro-groups in an aromatic compound is less likely to be in question, the test may usefully show that more than one nitro-group is present. *m*-Dinitrobenzene (1·0), 3 : 5-dinitrobenzoic acid (1·0), 2 : 4 : 6-trinitrobenzoic acid (2·0), 2 : 4-dinitrotoluene (1·0), 2 : 4-dinitroaniline (5·0), 2 : 4-dinitroanisole 0·6), and 1-chloro-2 : 4-dinitrobenzene (1·5) responded to the test.

The following compounds, as anticipated, proved negative: nitrobenzene, *o*- and *p*-nitroanilines, 3-nitro-*o*-toluidine, 4-nitro-*m*-toluidine, 5-nitro-*o*-toluidine, *m*-nitroanisole, *m*-nitrobenzoic acid, *o*- and *p*-dinitrobenzenes.

The reagent for testing aminonitro- and dinitro-compounds was prepared by dissolving zinc chloride (7·5 g.) and hydrated stannous chloride (14 g.) in glycerol (118·5 g.) at a somewhat higher temperature, but with the minimum of heating, and, after cooling to 60°, dissolving powdered hydrated oxalic acid (10 g.) in the mixture. The test was carried out precisely as before.

Both reagents should be preserved in stoppered bottles and decanted from any sediment before use.

The mechanism of the reaction is under investigation and the isolation and identification of several of the products of condensation have been achieved, the first four diamines mentioned

above giving respectively 2 : 8-diaminoacridine, 2 : 8-diamino-3 : 7-dimethylacridine, 3 : 7-dichloro-2 : 8-diaminoacridine and an isomeride, and 2 : 8-bisdimethylaminoacridine.

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The Alleged Optical Activity of o-*Toluidine*-3 : 5-*disulphonic Acid.* By P. P. HOPF and R. J. W LE FÈVRE.

THE resolution of the acid named in the title was reported by Sementzov (*Ukraine Chem. J.*, 1933, **8**, 193), a saturated chloroform solution of strychnine (1 mol)., when mixed with an alcoholic solution of the acid (½ mol.), giving an insoluble salt from which ultimately an acid was obtained showing a rotation of + 0·35° in 10% alcoholic solution in a 1 dcm. tube. Since it seemed possible that this optical activity could have been due to restricted intramolecular rotation of the type previously sought (J., 1933, 977), we repeated the above experiment. The *o*-toluidine-3 : 5-disulphonic acid was prepared according to Hasse (*Annalen*, 1885, **230**, 287) in an anhydrous state. The admixture of solutions made up as Sementzov described gave no immediate precipitate. On partial evaporation, however, a small fraction of *strychnine* salt crystallised, but the acid recovered (by treatment with sodium hydroxide and extraction with chloroform, etc.) from this or the mother-liquor was inactive (*i.e.*, α less than + 0·01° for a 2 dcm. tube and 1% solution). The strychnine salt of the disulphonic acid was prepared in quantity by addition of an excess of the acid to a chloroform solution of the base, brief warming, filtration, and evaporation. The deposit was recrystallised several times from alcohol without change in optical rotation ($[\alpha]_D^{18^\circ}$ + 21·0°, $c = 1$ in chloroform; to be compared with strychnine under the same conditions: $[\alpha]_D^{18^\circ}$ + 131°); it had m. p. 245° (decomp.) (Found: C, 62·1; H, 5·7. $2C_{21}H_{22}O_2N_2,C_7H_9O_6NS_2$ requires C, 62·6; H, 5·7%).—THE SIR WILLIAM RAMSAY AND RALPH FORSTER LABORATORIES, UNIVERSITY COLLEGE, LONDON. [*Received, January* 25*th*, 1939.]