and stirring were continued until the sulphur had dissolved forming a brownish-red solution of sodium disulphide. The warm sodium disulphide solution was added dropwise with stirring, to a solution of 13.6 g (0.05 mole) of α,α'-dibromodiisopropylketone in 50 ml of dimethyl sulphoxide at room temperature. After the addition was complete, the reaction mixture was kept at 40° and stirred for 2 h; 500 ml of water were added and the mixture extracted ten times with ether. The ethereal extracts were dried over anhydrous magnesium sulphate, and the ether removed *in vacuo* leaving 8 g of a yellowish-red oil, containing a mixture of the mono-sulphide (II) and the disulphide (I) (identified through IR-spectra). The monosulphide together with some disulphide were removed by careful sublimation at 0.3 mm. The 3,3,5,5-tetramethyl-1,2-dithiolane-4-one was then collected at 37—39°/0.3 mm, n_D^{20} 1.5102, m.p. 13—14°. The yield was 3.1 g (35 %).

(Found: C 47.72; H 6.89; S 36.19. Calc. for $C_7H_{12}OS_2$ (176.31): C 47.69; H 6.86; S 36.38.)

Spectrochemical data: UV, $\lambda_{max}^{isooctane}$ 230, 253, 275, 302, 312, and 325 mμ.

IR, carbonyl absorption at 1735 cm^{-1} (liq. phase).

NMR, a single peak at τ = 9.26 ppm.

Acknowledgements. The authors are greatly indebted to Professor Arne Fredga for valuable discussions and for the facilities placed at their disposal. For the microanalyses we are indebted to Dr. Arthur Bengtsson of the Department of Analytical Chemistry, University of Uppsala. A grant from *AB Bofors, Nobelkrut*, Bofors, Sweden is gratefully acknowledged.

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Received April 1, 1963.

Dimorphism of N,N-Diethyl-*p*-toluenesulphonamide

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In performing the Hinsberg test on diethylamine, a reaction product was consistently obtained with m.p. 46° (in capillary tube) instead of as reported 60° [1]. Subsequently, a number of students working in the same room reported either one or the other melting point, but after a few weeks melting points of 60° were invariably found.

The low-melting compound is an unstable modification of N,N-diethyl-*p*-toluenesulphonamide as apparent from the following observations: Kjeldahl analyses of a number of preparations dried in various ways gave results within 2 % of the theoretical value. A sample of the low-melting compound enclosed in a sealed capillary tube showed m.p. 45.5—46° on a number of successive determinations; after heating to 62° and recrystallization the product melted at 59.5—60°. Melting point determination on a microscope hot-stage showed two different types of crystals; one melting at 45°, the other at 60°.

The low-melting modification is rapidly transformed in the presence of nuclei of the high-melting modification; thus in some cases melting point determinations performed on two successive days of a sample left openly in the laboratory revealed transformation. Only one example of N-*p*-toluenesulphonamide derivatives exhibiting dimorphism has been found in the literature, *viz.* that of N-butylaniline, [2] but in a number of cases inconsistent melting point data have been reported. These findings indicate, that polymorphism is a phenomenon that should not be overlooked, when *p*-toluenesulphonyl derivatives are used for the identification of amines.

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Received March 26, 1963.