X-Ray Determination of the Structure of Acenaphtho[1,2-c]-1,2,5-thiadiazole

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Summary The structure of acenaphtho[1,2-*c*]-1,2,5-thiadiazole shows that the three independent molecules in the asymmetric unit are approximately planar and that the average bond distances in the thiadiazole ring are: S–N, 1·65 Å; C=N, 1·31 Å; C–C, 1·42 Å; the average angle ∠N–S–N is 99·4°.

CRYSTALS of acenaphtho[1,2-*c*]-1,2,5-thiadiazole obtained from the reaction of sulphur dichloride with acenaphthoquinone dioxime were supplied by Dr. Kurt Pilgrim. The yellow needles belong to the monoclinic space group $C2/c$ with unit-cell parameters: $a = 22{\cdot}836(8)$, $b = 18{\cdot}213(5)$, $c = 14{\cdot}604(3)$ Å, $\beta = 106{\cdot}53°$, $D_m = 1{\cdot}418$, $D_c = 1{\cdot}425$ g cm^{-3} for $Z = 24$.

Altogether, the intensities of 4911 independent reflections were measured with a Picker FACS-I diffractometer; of these, 3612 were considered to be non-zero using the criterion that $I > 1{\cdot}40\sigma(I)$. The structure was solved by application

of the computer program MULTAN.[1] The first E map revealed 40 of the 45 non-hydrogen atoms; a difference Fourier map led to the placement of the remaining heavy atoms. Three cycles of full-matrix least-squares refinement using isotropic temperature factors reduced the discrepancy index to 16·3%. Anisotropic refinement of only the sulphur atoms reduced this to 11·5% at which point refinement was terminated.

lengths of these bonds in the three molecules are 1·65, 1·31, and 1·42 Å respectively. The S–N and C=N bond distances in related compounds are: 1,2,5-thiadiazole-3,4-dicarboxamide (I)[2] (*X*-ray), 1·62 and 1·32 Å; 1,2,5-thiadiazole (II)[3] (electron-diffraction), 1·63 and 1·33 Å; 1,2,5-thiadiazole[4] (III) (microwave spectroscopy), 1·63 and 1·33 Å. The average N–S–N bond angle in the present work is 99·4°, while those in related compounds [(I)—(III)] are 98·8,

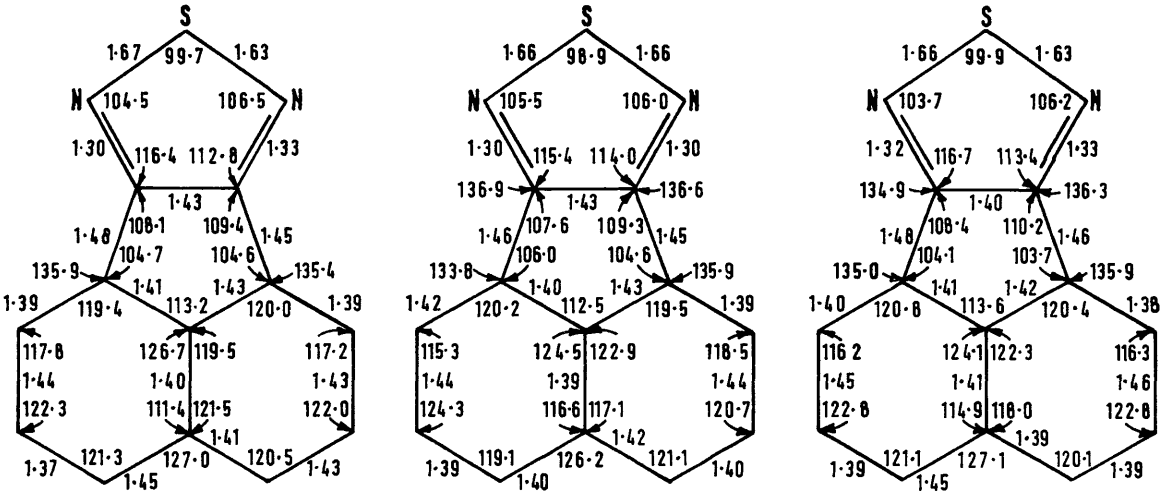

FIGURE. *Bond distances* (Å) *and bond angles* (°) *in the independent molecules in the asymmetric unit.*

Bond lengths and bond angles in the three independent molecules in the asymmetric unit are shown in the Figure. The standard deviations of the individual S–N, C=N and C–C bond distances are less than 0·01 Å and the average

99·4, and 99·6° respectively. Our results thus agree well with other studies.

(*Received, September 1st*, 1971; *Com.* 1520.)

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