Acta Cryst. (1980). B**36**, 758–759

Structure of a 1 : 1 Adduct of 4,6,8-Trimethylazulene and 1-(Diethylamino)propyne

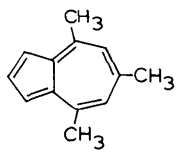
BY H. J. LINDNER, B. KITSCHKE, K. HAFNER AND W. UDE

Institut für Organische Chemie und Biochemie, Technische Hochschule Darmstadt, Petersenstrasse 22, D-6100 Darmstadt, Federal Republic of Germany

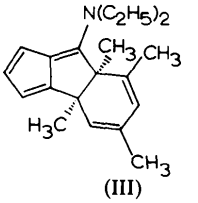
(*Received* 5 *November* 1979; *accepted* 10 *December* 1979)

Abstract. $C_{20}H_{27}N$, $P2_12_12_1$, $a = 16{\cdot}756\,(8)$, $b = 13{\cdot}305\,(8)$, $c = 7{\cdot}536\,(5)$ Å, $Z = 4$, $D_c = 1{\cdot}113$ Mg m^{-3}, μ(Cu $K\alpha$) = 0·41 mm^{-1}. The structure, which was refined to $R = 0{\cdot}070$ for 764 independent reflections, identifies the adduct as consisting of two five-membered rings *cis* connected to a non-planar six-membered ring.

Introduction. 4,6,8-Trimethylazulene (I) undergoes thermally induced cycloaddition with 1-(diethylamino)propyne (II) to form an adduct (III), which differs significantly from the structure of the product obtained by addition of (II) to azulene (Hafner, Lindner & Ude, 1979; Lindner, Kitschke, Hafner & Ude, 1980). By crystal structure analysis, the adduct was identified to be (III).



(I)



(III)

Crystals of (III) were grown from ether as brown prisms. The crystal system was determined from Weissenberg photographs. Intensities were collected on a Stoe two-circle diffractometer (Cu $K\alpha$ radiation) equipped with a graphite monochromator; the crystal was oriented along *c*. 1204 reflections, *hk*0 to *hk*5, with $\theta \leq 60°$ were measured in the θ–2θ scan mode. The data were corrected for background and for Lorentz and polarization factors, but not for absorption.

The structure was solved by direct methods and refined by full-matrix least squares with *SHELX* 76 (Sheldrick, 1977). Anisotropic refinement of the C and N atoms with geometrically positioned H atoms and 764 independent reflections with $|F| > 2\sigma$ reduced R to 0·070. The highest peak on the final difference map did not exceed 0·40 e Å^{-3}.

Table 1. *Positional parameters of the heavy atoms with e.s.d.'s in parentheses*

	x	*y*	*z*
C(1)	0·2670 (6)	0·3008 (8)	0·194 (2)
C(2)	0·2727 (7)	0·3160 (8)	0·360 (3)
C(3)	0·3374 (9)	0·2600 (9)	0·463 (2)
C(4)	0·3736 (6)	0·1804 (8)	0·383 (2)
C(5)	0·3513 (6)	0·1419 (6)	0·212 (2)
C(6)	0·4288 (5)	0·1152 (7)	0·109 (1)
C(7)	0·4554 (5)	0·2006 (6)	0·023 (1)
C(8)	0·5216 (6)	0·2418 (7)	−0·070 (1)
C(9)	0·4990 (6)	0·3374 (7)	−0·124 (2)
C(10)	0·4186 (6)	0·3572 (7)	−0·072 (2)
C(11)	0·3930 (6)	0·2744 (7)	0·025 (1)
C(12)	0·3171 (6)	0·2305 (7)	0·090 (1)
C(13)	0·2213 (9)	0·3834 (9)	0·474 (3)
C(14)	0·4360 (9)	0·1292 (9)	0·502 (2)
C(15)	0·2866 (6)	0·0591 (7)	0·237 (2)
C(16)	0·2677 (6)	0·1904 (8)	−0·062 (2)
N(17)	0·4649 (5)	0·0251 (5)	0·111 (1)
C(18)	0·4413 (7)	−0·0655 (7)	0·215 (1)
C(19)	0·4050 (8)	−0·1478 (7)	0·095 (2)
C(20)	0·5414 (6)	0·0151 (7)	0·014 (2)
C(21)	0·6097 (7)	0·0469 (9)	0·141 (2)

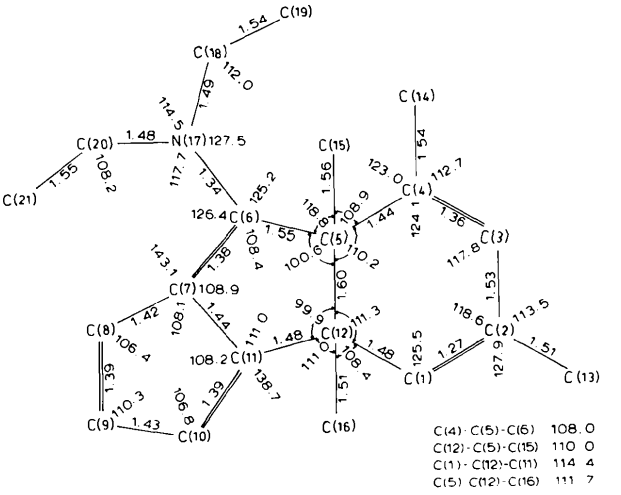


Fig. 1. The bond lengths and angles of (III) ($\sigma_{xx} = 0{\cdot}01$ Å, $\sigma_{xxx} = 0{\cdot}8°$; x = C, N).

0567-7408/80/030758-02$01.00

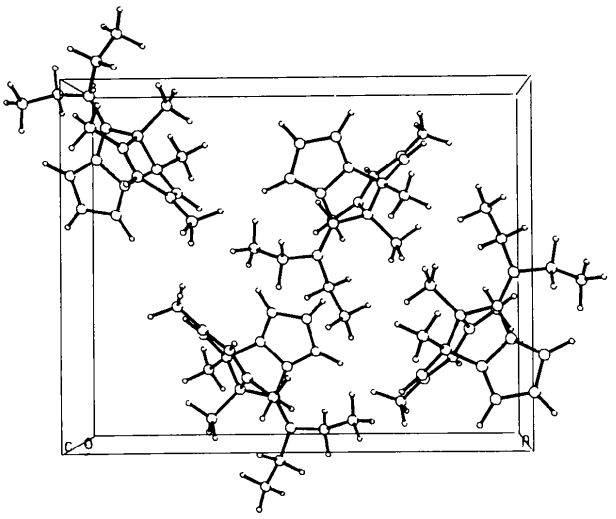


Fig. 2. The contents of the unit cell. View down *c* with *a* horizontal and *b* vertical.

Discussion. Fractional coordinates of the atoms are given in Table 1,* bond lengths and angles in Fig. 1. Fig. 2 shows the packing of the molecules. The non-planar six-membered ring [torsion angle C(4)–C(5)–C(12)–C(1) = 38°] is *cis* connected to the five-membered ring. The 6-(diethylamino)fulvene moiety is nearly planar; the formal double bond C(6)–C(7) is twisted by about 12°. The bond lengths of this part of the molecule are similar to those given for other fulvenes (Ammon & Wheeler, 1971). Between the molecules there are no intermolecular forces other than van der Waals interactions.

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34927 (7 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

We acknowledge financial support from the Deutsche Forschungsgemeinschaft and the Fonds der Chemischen Industrie.

References

AMMON, H. L. & WHEELER, G. L. (1971). *Chem. Commun.* pp. 1032–1034.

HAFNER, K., LINDNER, H. J. & UDE, W. (1979). *Angew. Chem.* **91**, 173–175; *Angew. Chem. Int. Ed. Engl.* **18**, 161–162.

LINDNER, H. J., KITSCHKE, B., HAFNER, K. & UDE, W. (1980). *Acta Cryst.* B**36**, 754–755.

SHELDRICK, G. M. (1977). *SHELX* 76. Program for crystal structure determination. Univ. of Cambridge, England.