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Structure of a 1:1 Adduct of Azulene and 1-(Diethylamino)propyne

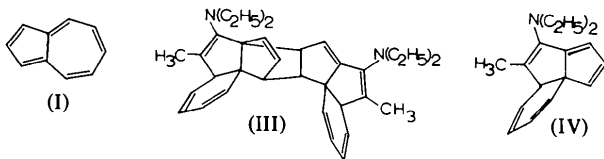
BY H. J. LINDNER, B. KITSCHKE, K. HAFNER AND W. UDE

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

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Abstract. C₃₄H₄₂N₂, triclinic, $P\bar{1}$, $a = 17.734(8)$, $b = 12.308(6)$, $c = 6.808(5)$ Å, $\alpha = 101.00(5)$, $\beta = 85.94(5)$, $\gamma = 106.63(5)^\circ$, $Z = 2$, $D_c = 1.140$ Mg m⁻³, $\mu(\text{Cu } K\alpha) = 0.42$ mm⁻¹. The structure, which was refined to $R = 0.108$ for 2148 independent reflections, identifies the compound as a Diels–Alder dimer in the crystalline state.

Introduction. Azulene (I), as the prototype of a non-benzenoid aromatic hydrocarbon, reacts with nucleophilic and electrophilic agents by substitution (Heilbronner, 1959). Recently, it was shown that (I) also undergoes thermally induced dipolar cycloadditions with electron-poor (Hafner, Diehl & Süß, 1976) and electron-rich alkynes such as 1-(diethylamino)propyne (II) (Hafner, Lindner & Ude, 1979). The structure of the adduct (III) formed by (I) and (II) at room temperature was determined by crystal structure analysis which reveals that (III) is the Diels–Alder dimer of two molecules of (IV).



Crystals of (III) were grown as clusters of colourless needles in the reaction mixture (Hafner *et al.*, 1979). 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 . 4295 reflections, $hk0$ to $hk5$, with $\theta \leq 60^\circ$ were measured in the θ – 2θ scan mode. The data were corrected for background and for Lorentz and polarization factors, but not for absorption.

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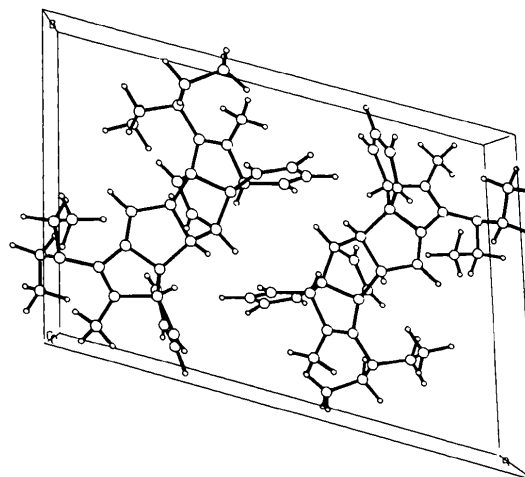


Fig. 1. The contents of the unit cell. View down c , with a horizontal and b vertical.

The structure was solved with *SHELX 76* (Sheldrick, 1977) by direct methods. The space group was established to be $P\bar{1}$ with one dimer in the asymmetric unit. Anisotropic refinement of the C and N atoms with geometrically positioned H atoms and 2148 independent reflections with $|F| > 2\sigma_F$ reduced R to 0.108. The highest peaks in the final density map did not exceed 0.40 e Å⁻³.

Discussion. The coordinates of the atoms are given in Table 1* and the molecular structure is shown in Fig. 1.

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34925 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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

	x	y	z
C(1)	0.2301 (5)	0.2366 (7)	0.503 (1)
C(2)	0.2458 (5)	0.1327 (8)	0.454 (2)
C(3)	0.2700 (6)	0.0810 (8)	0.601 (2)
C(4)	0.2686 (5)	0.1236 (7)	0.789 (2)
C(5)	0.2355 (5)	0.2263 (7)	0.866 (1)
C(6)	0.1460 (5)	0.1922 (7)	0.922 (1)
C(7)	0.1122 (5)	0.2646 (7)	0.859 (1)
C(8)	0.1686 (4)	0.3510 (7)	0.775 (1)
C(9)	0.1814 (5)	0.4593 (7)	0.744 (1)
C(10)	0.2660 (4)	0.5140 (6)	0.692 (1)
C(11)	0.3080 (4)	0.4186 (6)	0.704 (1)
C(12)	0.2404 (4)	0.3059 (7)	0.715 (1)
C(13)	0.1092 (6)	0.0925 (10)	1.020 (2)
N(14)	0.0307 (4)	0.2613 (7)	0.918 (1)
C(15)	0.0241 (6)	0.3655 (12)	1.065 (2)
C(16)	0.0677 (8)	0.3875 (14)	1.246 (2)
C(17)	-0.0159 (6)	0.2517 (10)	0.744 (2)
C(18)	-0.0221 (8)	0.1388 (11)	0.606 (2)
C(1A)	0.4504 (5)	0.6729 (7)	1.016 (1)
C(2A)	0.5263 (6)	0.7205 (8)	0.975 (2)
C(3A)	0.5610 (6)	0.7121 (9)	0.781 (2)
C(4A)	0.5155 (5)	0.6746 (8)	0.623 (2)
C(5A)	0.4272 (5)	0.6427 (7)	0.651 (1)
C(6A)	0.3920 (5)	0.7467 (7)	0.670 (1)
C(7A)	0.3306 (4)	0.7355 (6)	0.791 (1)
C(8A)	0.3118 (4)	0.6214 (7)	0.863 (1)
C(9A)	0.2837 (5)	0.5845 (7)	1.055 (1)
C(10A)	0.3119 (5)	0.4980 (7)	1.074 (2)
C(11A)	0.3629 (5)	0.4744 (7)	0.889 (1)
C(12A)	0.3958 (4)	0.6010 (7)	0.847 (1)
C(13A)	0.4265 (6)	0.8431 (8)	0.561 (2)
N(14A)	0.2872 (4)	0.8217 (6)	0.847 (1)
C(15A)	0.3029 (6)	0.8817 (8)	1.055 (2)
C(16A)	0.3859 (6)	0.9557 (9)	1.087 (2)
C(17A)	0.2026 (5)	0.7714 (9)	0.810 (2)
C(18A)	0.1847 (7)	0.7338 (10)	0.591 (2)

The Diels–Alder addition of the cyclopentadiene moieties forms the *endo* product. The bond lengths and angles (Fig. 2) are in the expected range with the exception of the long C(8A)–C(10) bond (1.64 Å). This bond, formed by the Diels–Alder addition, may be elongated because of the strain in the overcrowded region of the molecule.

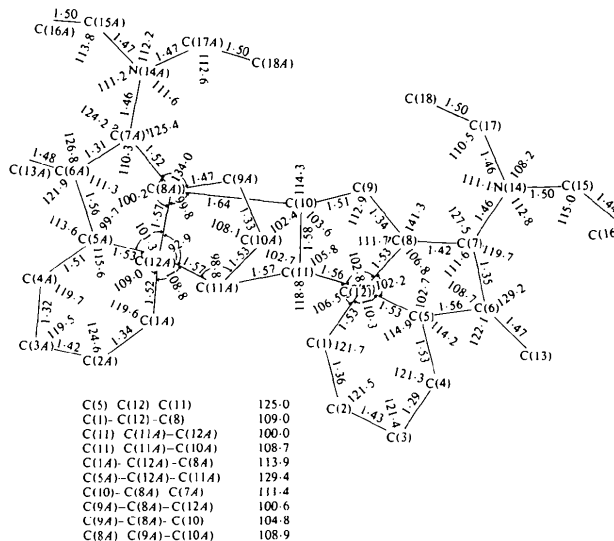


Fig. 2. Bond lengths (Å) and angles (°) ($\sigma_{xx} = 0.01$ Å, $\sigma_{xxx} = 0.8^\circ$; $x = C, N$).

The packing of the molecules (Fig. 1) gives no indication of intermolecular forces other than van der Waals interactions.

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