1986

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The Structure of the Norbornadiene* Adduct of 5-Phenyl-1,3,2,4,6-dithiatriazine

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(Received 17 July 1985; accepted 29 October 1985)

Abstract. 10-Phenyl-1,8-dithia-9,11,12-triazatetracyclo-
[6.3.1.14.27]trideca-1(11),4,9,8(12)-tetraene, C_{14}H_{13}N_{3}S_{2}, M_r = 287.4, monoclinic, P2_{1}, a = 5.789 (2),
b = 10.064 (1), c = 11.109 (2) Å, β = 94.78 (2)°, V = 644.9 (4) Å³, Z = 2, D_x = 1.48 g cm\(^{-3}\), λ(Mo Kα) = 0.71073 Å, μ = 3.84 cm\(^{-1}\), F(000) = 300, T = 293 K, R = 0.037 for 1203 unique observed reflections. The norbornadiene bonds to the sulfur atoms in the exo-β orientation and the nitrogen atom between the sulfur atoms is displaced 0.802 (4) Å from the SNCNS plane. The C–N bond lengths are equal [1.335 (5) Å] and the S–N bond lengths in a very narrow range of values [1.637 (4)–1.652 (4) Å].

Experimental. Compound prepared by the reaction of norbornadiene with 1,3-dichloro-5-phenyl-1,3,2,4,6-dithiatriazine. Crystals obtained from acetonitrile solutions. White platelet data crystal 0.48 × 0.04 × 0.40 mm mounted on glass fiber. Intensities measured with Enraf–Nonius CAD-4 diffractometer, variable-speed o–2θ scans. Unit cell from least squares of angle data for 18 reflections with 2θ < 2θ < 30°. Analytical absorption correction based on crystal shape varied from 0.89 to 1.00. Data collected to sinθ/λ of 0.70 Å\(^{-1}\), −8 ≤ h ≤ 8, 0 ≤ k ≤ 14, 0 ≤ l ≤ 15. Three standard reflections (060, 224, 214) varied ±1.9% over 18·6 h of data collection; anisotropic drift correction applied. 2061 reflections measured, 1972 unique (R_{int} = 0.03), 769 reflections with I < 3σ(I) considered unobserved. Solved by direct methods using MULTAN11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982) and Fourier methods. Full-matrix least squares minimized $\sum w(\Delta F)^2$. H atoms refined with isotropic temperature factors, all other atoms refined anisotropically for 224 variables. R = 0.037, wR = 0.043, S = 1.09, where non-Poisson w\(^{-1} = [σ^2(I) + 0.0025F^2]/4F^2\). Final $\langle Δ/σ\rangle_{\text{max}} < 0.01$, $Δρ_{\text{max}} = 0.21 (5)$ and $Δρ_{\text{min}} = −0.24 (5) e Å\(^{-3}\) on final difference map. Atomic scattering factors and anomalous-dispersion corrections from International Tables for X-ray Crystallography (1974) and programs used were those of Enraf–Nonius (1982) SDP.‡ Table 1 gives the atom coordinates and Table 2 selected bond distances and angles. Fig. 1 shows the molecule with the numbering scheme.

‡ Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42630 (23 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
Related literature. Structures of three other derivatives of 5-phenyl-1,3,2,4,6-dithia triazine have recently been reported: PhCS₂N₃Cl₂ (Graham, Cordes, Oakley & Boeré, 1985), PhCS₃N₃ (Cordes, Oakley & Boeré, 1985) and (PhCS₂N₃)₂ (Boeré, French, Oakley, Cordes, Privett, Craig & Graham, 1985). Other norbornadiene adducts of S-N rings include Ph₂N₃S₂C₇H₈ (Burford, Chivers, Cordes, Laidlaw, Noble, Oakley & Swepston, 1982), Ph₃N₃S₃N₃C₇H₈ (Liblong, Oakley, Cordes & Noble, 1983) and (CF₃)₂PS₂N₃C₇H₈ (Roesky, Lucas, Noltemeyer & Sheldrick, 1984).

We thank the National Science Foundation, the State of Arkansas, the Research Corporation and the Natural Sciences and Engineering Research Council of Canada for financial support.

References


Table 1. Fractional atomic coordinates, isotropic thermal parameters, and their e.s.d.'s

$$B_{eq} = \frac{4}{3}(a^2B_{11} + b^2B_{22} + c^2B_{33} + ab^2\cos\beta)$$

Table 2. Selected bond distances (Å) and bond angles (°) and their e.s.d.'s

Fig. 1. ORTEPII diagram (Johnson, 1976) and atom-numbering scheme. Non-H ellipsoids at 30% probability level, H atoms given arbitrary radii.