

1985

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International Union of Crystallography

Graham, J. B., Cordes, A. W., Oakley, R. T., & Boere, Rene T. (1985). Structure of 1,3-dichloro-5-phenyl-1{Greek letter lambda}4,3{Greek letter lambda}4,2,4,6-dithiatriazine. Acta Crystallographic C, Crystal Structure Communications, C41, 1835-1836.

<http://hdl.handle.net/10133/3808>

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Structure of 1,3-Dichloro-5-phenyl-1 λ^4 ,3 λ^4 ,2,4,6-dithiatriazine

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(Received 27 June 1985; accepted 21 August 1985)

Abstract. C₇H₅Cl₂N₃S₂, $M_r = 266.17$, triclinic, $P\bar{1}$, $a = 6.028$ (1), $b = 9.985$ (2), $c = 10.157$ (3) Å, $\alpha = 117.33$ (2), $\beta = 106.73$ (2), $\gamma = 90.31$ (1)°, $V = 513.3$ (4) Å³, $Z = 2$, $D_x = 1.72$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 9.85$ cm⁻¹, $F(000) = 268$, $T = 293$ K, $R = 0.032$ for 1547 unique observed reflections. The SNCNS portion of the ring is planar within 0.060 (2) Å and the third N is displaced 0.336 (2) Å from this plane on the side opposite the S-bonded Cl atoms. The S–N distances range from 1.581 (1) to 1.615 (2) Å and the N–C distances are 1.327 (2) and 1.344 (2) Å.

Experimental. Compound prepared by the reaction of chlorine gas with PhCN₂S₃ obtained from the reaction of PhC(NSiMe₃) with S₃N₃Cl₃. Crystals obtained from a 50:50 mixture of dichloromethane and hexanes. Air-sensitive parallelepiped data crystal 0.20 × 0.50 × 0.46 mm sealed in a glass capillary. Density not measured. Intensities measured with an Enraf–Nonius CAD-4 diffractometer using ω – 2θ scans of 4 to 16° min⁻¹ in θ . Unit cell determined from least-squares analysis of angle data for 20 reflections with $22 < 2\theta < 40^\circ$. Analytical absorption correction based on crystal shape varied from 0.67 to 1.00. Data collected to $\sin\theta/\lambda$ of 0.59 Å⁻¹, $-7 \leq h \leq 7$, $-11 \leq k \leq 11$, $0 \leq l \leq 12$. Four standard reflections (244, $\bar{4}\bar{4}\bar{4}$, $\bar{3}\bar{2}\bar{5}$, $3\bar{3}\bar{5}$) varied $\pm 2\%$; linear-drift correction applied. 1907 reflections measured, 1796 unique ($R_{\text{int}} = 0.01$), 247 reflections with $I < 3\sigma(I)$ considered unobserved. Two reflections (101, $\bar{1}\bar{3}\bar{1}$) which saturated the counter with their high intensities were omitted. 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 isotropically, other atoms anisotropically for a total of 147 variables. $R = 0.032$, $wR = 0.043$, $S = 1.64$, where non-Poisson $w^{-2} = \sigma(I) + 0.0016F^2$. Final $(\Delta/\sigma)_{\text{max}} < 0.03$, $\Delta\rho_{\text{max}} = 0.36$ (2) and $\Delta\rho_{\text{min}} = -0.35$ (2) 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 those

of Enraf–Nonius (1982) SDP.* Table 1 gives the atom parameters and Table 2 bond lengths and angles. Fig. 1 shows the molecule and numbering scheme.

* Lists of structure factors and anisotropic temperature factors have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42450 (18 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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

| | $B_{\text{eq}} = \frac{1}{3}(a^2B_{11} + b^2B_{22} + c^2B_{33} + acB_{13}\cos\beta)$ | | | |
|-------|--|-------------|-------------|-------------------------------|
| | x | y | z | $B_{\text{eq}}/B(\text{Å}^2)$ |
| Cl(1) | 0.0338 (1) | 0.60561 (7) | 0.38131 (7) | 4.47 (2) |
| Cl(2) | 0.3629 (1) | 0.61446 (6) | 0.15192 (6) | 4.54 (1) |
| S(1) | 0.19222 (9) | 0.39933 (6) | 0.33146 (5) | 3.05 (1) |
| S(2) | 0.46456 (8) | 0.41377 (5) | 0.15988 (5) | 2.82 (1) |
| N(1) | 0.0126 (3) | 0.2834 (2) | 0.1620 (2) | 3.18 (4) |
| N(2) | 0.2661 (3) | 0.2819 (2) | 0.0170 (2) | 3.49 (4) |
| N(3) | 0.4451 (3) | 0.4384 (2) | 0.3246 (2) | 3.03 (4) |
| C(1) | 0.0703 (3) | 0.2353 (2) | 0.0327 (2) | 2.64 (4) |
| C(2) | -0.0977 (4) | 0.1129 (2) | -0.1132 (2) | 2.83 (5) |
| C(3) | -0.3030 (4) | 0.0491 (2) | -0.1120 (3) | 3.36 (5) |
| C(4) | -0.4602 (2) | -0.0622 (2) | -0.2501 (3) | 4.17 (6) |
| C(5) | -0.4160 (5) | -0.1128 (3) | -0.3895 (3) | 4.56 (7) |
| C(6) | -0.2128 (5) | -0.0513 (3) | -0.3922 (3) | 4.51 (7) |
| C(7) | -0.0537 (4) | 0.0612 (2) | -0.2542 (2) | 3.67 (6) |
| H(3) | -0.338 (4) | 0.079 (2) | -0.023 (3) | 4.5 (6) |
| H(4) | -0.590 (5) | -0.097 (3) | -0.241 (3) | 6.3 (7) |
| H(5) | -0.531 (5) | -0.177 (3) | -0.477 (3) | 6.7 (8) |
| H(6) | -0.181 (4) | -0.083 (3) | -0.490 (3) | 6.2 (7) |
| H(7) | 0.076 (4) | 0.105 (3) | -0.255 (3) | 5.6 (7) |

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

| | | | |
|-------------------|-------------|-----------------|-------------|
| S(1)–Cl(1) | 2.180 (1) | Cl(1)–S(1)–N(1) | 100.49 (6) |
| S(1)–N(1) | 1.600 (1) | Cl(1)–S(1)–N(3) | 106.12 (6) |
| S(1)–N(3) | 1.600 (1) | N(1)–S(1)–N(3) | 112.16 (8) |
| S(2)–Cl(2) | 2.129 (1) | Cl(2)–S(2)–N(2) | 103.36 (6) |
| S(2)–N(2) | 1.581 (1) | Cl(2)–S(2)–N(3) | 103.56 (6) |
| S(2)–N(3) | 1.615 (2) | N(2)–S(2)–N(3) | 110.84 (8) |
| N(1)–C(1) | 1.327 (2) | S(1)–N(3)–S(2) | 118.41 (9) |
| N(2)–C(1) | 1.344 (2) | S(1)–N(1)–C(1) | 122.0 (1) |
| C(1)–C(2) | 1.475 (2) | S(2)–N(2)–C(1) | 122.1 (1) |
| C–C(phenyl) range | 1.375–1.394 | N(1)–C(1)–N(2) | 128.4 (2) |
| C–C(phenyl) mean | 1.383 | N(1)–C(1)–C(2) | 116.8 (2) |
| C–H range | 0.89–0.97 | N(2)–C(1)–C(2) | 114.8 (2) |
| C–H mean | 0.91 | C–C–C range | 119.3–120.4 |
| | | C–C–H range | 115–124 |

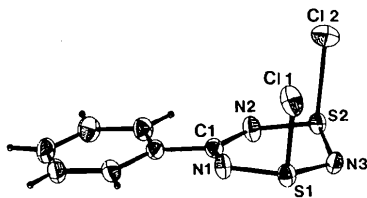


Fig. 1. ORTEP diagram (Johnson, 1976) and atom-numbering scheme. Non-H ellipsoids at 30% probability level, H atoms given arbitrary radius.

Related literature. Structures of three other derivatives of 5-phenyl-1,3,2,4,6-dithiatiazine are in preparation or have been published: $S_3N_3C_7H_5$ (Cordes, Oakley & Boéré, 1985), $PhCS_2N_3 \cdot C_7H_8$ (James, Craig, Cordes, Oakley & Boéré, 1985), and $(PhCS_2N_3)_2$ (Boéré, French, Oakley, Cordes, James, Craig & Graham, 1985). The title compound is a member of the series $N_3S_3Cl_3$ (Wiegiers & Vos, 1962), $PhCN_3S_2Cl_2$ (this report), $Ph_2C_2N_3SCl$ (Cordes, Hayes, Josephy, Koenig, Oakley & Pennington, 1984), and $Ph_3C_3N_3$ (Damiani, Giglio & Ripamonti, 1965). The heterocyclic ring has also been studied in $Me_2NCN_3S_2Cl_2$ (Hofs, Hartmann, Mews & Sheldrick, 1984) and $CF_3CN_3S_2Cl_2$ (Roesky, Schafer, Noltemeyer & Sheldrick, 1983).

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.

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