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Redetermination of 1,5-Dichlorocyclotetra-(azathiene) at 120 K

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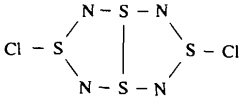
Abstract

The structure of the title compound (3,7-dichloro-1*S*-bicyclo[3.3.0]tetraazathiatetraene, $S_4N_4Cl_2$) has been redetermined at 120 K. The original structure determination was performed using photographic data obtained at ambient temperature [Žák (1981). *Acta Cryst.* B**37**, 23–26].

Comment

The original structure determination of 1,5-dichlorocyclotetra(azathiene), (I), was performed using photographic data obtained at ambient temperature (Žák, 1981). The compound, however, was not stable under X-rays and the data were merged from several crystals. Although there can be no doubt about the correctness of the structure, the accuracy of the derived structural parameters was affected accordingly. We considered it useful, therefore, to redetermine the structure from accurate diffractometer data collected at low temperature.



(I)

The overall geometry remains the same but there are significant differences in individual bond lengths and angles. Especially affected are the S—Cl distances [2.139 (1) and 2.446 (1) Å *versus* 2.183 (1) and 2.179 (1) Å in the original study], and the transannular S1···S3 distance [4.037 (1) *versus* 3.859 (2) Å in the original study]. However, it is not clear whether these differences are side effects of the structure determination at low temperature or whether they originate from inaccurate structural parameters of the ambient-temperature structure determination.

The molecules are bound into a three-dimensional network by weak S1···Cl1 [3.351 (1) Å] and S4···Cl2 [3.390 (1) Å] intermolecular interactions.

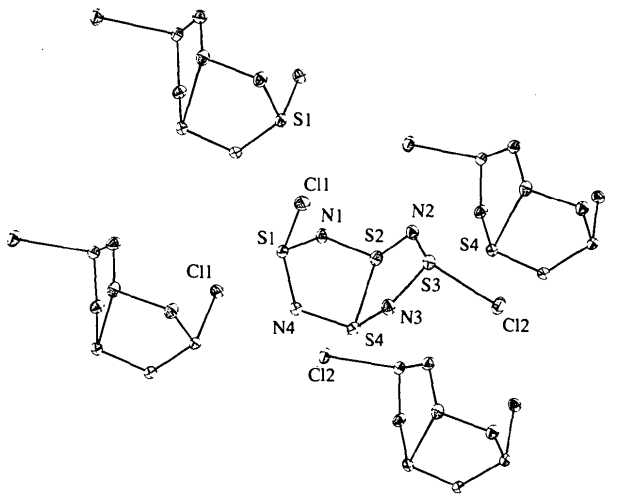


Fig. 1. Packing of the molecules of the title compound showing contacts less than the sum of the van der Waals radii. Displacement ellipsoids are plotted at the 50% probability level.

Experimental

Crystal data

$S_4N_4Cl_2$
M_r = 255.18
Monoclinic
$P2_1/n$
a = 9.344 (2) Å
b = 6.509 (2) Å
c = 12.764 (3) Å
β = 108.53 (2)°
V = 736.1 (3) Å^3
Z = 4
D_x = 2.303 Mg m^{-3}
Mo $K\alpha$ radiation
λ = 0.7107 Å
Cell parameters from 25 reflections
θ = 8.39–19.62°
μ = 1.936 mm^{-1}
T = 120 (2) K
Prism
0.50 × 0.50 × 0.30 mm
Yellow

Data collection

Kuma KM-4 κ-axis diffractometer
ω–2θ scans
Absorption correction: ψ scan (*ABSELI*; Kuma, 1991)
T_{min} = 0.138, T_{max} = 0.153
3193 measured reflections
2151 independent reflections
1798 observed reflections [$I > 2\sigma(I)$]
R_{int} = 0.0336
θ_{max} = 30.07°
$h = -12 \rightarrow 4$
$k = 0 \rightarrow 9$
$l = -17 \rightarrow 17$
2 standard reflections monitored every 100 reflections
intensity decay: none

Refinement

Refinement on F^2
$R(F)$ = 0.0253
$wR(F^2)$ = 0.0740
S = 1.053
2150 reflections
91 parameters
$w = 1/[\sigma^2(F_o^2) + (0.0358P)^2 + 0.2458P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.440 e Å^{-3}
$\Delta\rho_{min}$ = −0.421 e Å^{-3}
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{eq} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
S1	0.16701 (5)	0.12979 (6)	0.21103 (3)	0.01276 (10)
S2	−0.07637 (5)	0.23803 (7)	0.28033 (3)	0.01410 (10)
S3	−0.26401 (5)	0.31018 (7)	0.06509 (3)	0.01306 (10)
S4	−0.11641 (5)	−0.05153 (7)	0.15285 (3)	0.01341 (10)
N1	0.1015 (2)	0.2425 (2)	0.29838 (12)	0.0147 (3)
N2	−0.1668 (2)	0.3959 (2)	0.18263 (12)	0.0159 (3)
N3	−0.2057 (2)	0.0900 (2)	0.04769 (12)	0.0149 (3)
N4	0.0595 (2)	−0.0635 (2)	0.16459 (13)	0.0148 (3)
Cl1	0.12546 (5)	0.34292 (7)	0.07709 (3)	0.01573 (10)
Cl2	−0.48851 (5)	0.24905 (7)	0.08844 (4)	0.01849 (11)

Table 2. *Selected geometric parameters* (Å, °)

S1—N4	1.601 (2)	S3—N3	1.574 (2)
S1—N1	1.609 (2)	S3—N2	1.588 (2)
S1—Cl1	2.1392 (7)	S3—Cl2	2.2461 (7)
S1···Cl1^{i}	3.3508 (9)	S4—N4	1.605 (2)
S2—N1	1.604 (2)	S4—N3	1.623 (2)
S2—N2	1.630 (2)	S4···Cl2^{ii}	3.3901 (10)
S2—S4	2.4399 (8)	S1···S3	4.037 (1)
N4—S1—N1	106.44 (8)	N4—S4—N3	110.31 (8)
N4—S1—Cl1	106.29 (6)	N4—S4—S2	92.28 (6)
N1—S1—Cl1	104.22 (6)	N3—S4—S2	92.45 (6)
N1—S2—N2	111.09 (8)	S2—N1—S1	119.79 (10)
N3—S3—N2	109.50 (8)	S3—N2—S2	120.20 (10)
N3—S3—Cl2	103.71 (6)	S3—N3—S4	120.67 (9)
N2—S3—Cl2	102.42 (6)	S1—N4—S4	120.00 (10)

Symmetry codes: (i) $\frac{1}{2}-x, y-\frac{1}{2}, \frac{1}{2}-z$; (ii) $-\frac{1}{2}-x, y-\frac{1}{2}, \frac{1}{2}-z$.

Low-temperature data were collected on a Kuma KM-4 κ-axis diffractometer equipped with modified Nonius low-temperature device.

Data collection: *Kuma KM-4 Software* (Kuma, 1991). Cell refinement: *Kuma KM-4 Software*. Data reduction: *Kuma KM-4 Software* (*DATAREDC*). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP* (Johnson, 1965). Software used to prepare material for publication: *SHELXL*93.

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Lists of structure factors, anisotropic displacement parameters and complete geometry have been deposited with the IUCr (Reference: HU1126). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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