

assistance of Dr J. H. Reibenspies in the use of the crystallographic programs is greatly appreciated.

Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55809 (14 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: CR1030]

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Bis(3-chloro-2-pyridyl) Ditelluride

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Abstract

The entire ditelluride molecule is planar with an average deviation of its non-H atoms of 0.016 Å. It is located on a center of symmetry at $(\frac{1}{2}, 0, 0)$ with Te—Te_A distance 2.725 (1) Å and angle C(1)—Te—Te_A 85.8 (1)°. Each half of the molecule consists of a substituted pyridine ring with average C—N distance 1.334 (6) Å, C—C distance 1.38 (1) Å and angle C—N—C 117.5 (4)°. The ring is substituted in the 2-position [Te—C(1) distance 2.121 (3) Å] and in the 3-position [Cl—C(2) distance 1.736 (4) Å]. All of these values appear to be normal and there are no abnormally short intermolecular distances present in the structure.

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Comment

The preparation of the compound is described by Junk (1988). The solid is extremely deep blue in color (as opposed to the orange-to-red colors observed for aryl ditellurides) and the structure has been examined for any structural features that might be associated with its unusual color. The pyridine ring itself has dimensions slightly distorted from those of gaseous pyridine (Bak, Hansen-Nygaard & Rastrup-Anderson, 1958). The C(2)—Cl distance of 1.736 (4) Å is only slightly longer than expected (Drendel & Sundaralingam, 1985), as is the Te—Te_A distance of 2.725 (1) Å; but the angle Te_A—Te—C(1) of 85.8 (1)° differs greatly from those found in alkyl ditellurides [97–102° (Van den Bossche, Spirlet, Dideberg & Dupont, 1984, and structures reviewed therein)]. It seems likely that the blue color of the present molecule is associated with its extended planar structure and the possibility of interaction between its two pyridine rings since, in the aryl ditellurides, the aromatic rings are twisted from coplanarity and the possibility of extended conjugation is greatly reduced.

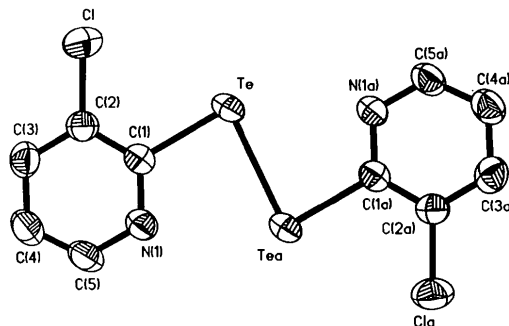


Fig. 1. Structure of bis(3-chloro-2-pyridyl) ditelluride drawn with thermal ellipsoids at 0.50 probability.

Experimental

Crystal data

C₁₀H₆Cl₂N₂Te₂
M_r = 480.3
 Monoclinic
*P*2₁/c
a = 7.298 (2) Å
b = 12.542 (3) Å
c = 7.944 (2) Å
 β = 116.05 (2)°
V = 653.3 (2) Å³
Z = 2

D_x = 2.442 Mg m⁻³

Mo *K* α radiation

λ = 0.71073 Å

Cell parameters from 25 reflections

θ = 4–11°

μ = 4.865 mm⁻¹

T = 296 K

Prism

0.30 × 0.20 × 0.20 mm

Dark blue

Data collection

Enraf–Nonius CAD-4 diffractometer

1779 observed reflections [*I* > 0]

$\omega/2\theta$ scans; ω -scan width
 $0.7^\circ + K\alpha$ separation;
 variable scan rate
 $2\text{--}20^\circ \text{ min}^{-1}$
 Absorption correction: none
 1989 measured reflections
 1989 independent reflections

$\theta_{\text{max}} = 30^\circ$
 $h = 0 \rightarrow 10$
 $k = 0 \rightarrow 17$
 $l = -11 \rightarrow 10$
 3 standard reflections
 frequency: 41 min
 intensity variation: negligible

Refinement

Refinement on F
 Final $R = 0.0351$
 $wR = 0.0577$
 $S = 1.76$
 1779 reflections
 73 parameters
 H-atom parameters not refined

$w = 1/\sigma^2(F)$
 $(\Delta/\sigma)_{\text{max}} = 0.004$
 $\Delta\rho_{\text{max}} = 0.53 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\text{min}} = -1.59 \text{ e } \text{Å}^{-3}$
 Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

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

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

| | x | y | z | U_{eq} |
|------|------------|------------|-------------|-----------------|
| Te | 0.3195 (1) | 0.0295 (1) | -0.0017 (1) | 0.049 (1) |
| Cl | 0.1446 (2) | 0.1466 (1) | 0.2741 (2) | 0.071 (1) |
| N(1) | 0.7016 (5) | 0.0778 (3) | 0.3444 (5) | 0.048 (1) |
| C(1) | 0.4984 (5) | 0.0873 (3) | 0.2734 (5) | 0.042 (1) |
| C(2) | 0.4081 (6) | 0.1333 (3) | 0.3745 (6) | 0.045 (1) |
| C(3) | 0.5259 (7) | 0.1685 (3) | 0.5568 (6) | 0.053 (2) |
| C(4) | 0.7371 (6) | 0.1575 (3) | 0.6294 (6) | 0.057 (2) |
| C(5) | 0.8119 (6) | 0.1135 (4) | 0.5172 (6) | 0.055 (2) |

Table 2. Geometric parameters (Å , $^\circ$)

| | | | |
|----------------|-----------|----------------|-----------|
| Te—C(1) | 2.121 (3) | Te—TeA | 2.725 (1) |
| Cl—C(2) | 1.736 (4) | N(1)—C(1) | 1.340 (5) |
| N(1)—C(5) | 1.327 (5) | C(1)—C(2) | 1.370 (7) |
| C(2)—C(3) | 1.392 (6) | C(3)—C(4) | 1.396 (6) |
| C(4)—C(5) | 1.351 (7) | | |
| C(1)—Te—TeA | 85.8 (1) | C(1)—N(1)—C(5) | 117.5 (4) |
| Te—C(1)—N(1) | 118.0 (3) | Te—C(1)—C(2) | 120.7 (2) |
| N(1)—C(1)—C(2) | 121.3 (3) | Cl—C(2)—C(1) | 119.5 (3) |
| Cl—C(2)—C(3) | 120.0 (4) | C(1)—C(2)—C(3) | 120.5 (4) |
| C(2)—C(3)—C(4) | 117.6 (5) | C(3)—C(4)—C(5) | 117.5 (4) |
| N(1)—C(5)—C(4) | 125.6 (4) | | |

Preliminary examination and data collection were performed by the Molecular Structure Corporation (The Woodlands, Texas, USA) on a sample mounted on a glass fiber with epoxy cement. The structure was solved by Patterson methods and final calculations were carried out with the *SHELXTL-Plus* programs (Sheldrick, 1990). Full-matrix least-squares refinement of coordinates and anisotropic temperature factors for non-H atoms was carried out and H atoms were assigned idealized positions at 0.96 Å from the C atom to which they were attached. The intensities of 1989 reflections were measured, 91 of which were discarded because of systematic extinction.

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(3R,5R)-Tetrahydro-2H-1,4-thiazine-3,5-dicarboxylic Acid Monohydrate

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Abstract

(3R,5R)-Tetrahydro-2H-1,4-thiazine-3,5-dicarboxylic acid monohydrate, which is in the zwitterionic form in the crystal, shows a puckered chair conformation of the six-membered ring with S(1) and N(4) out of the C(2), C(3), C(5), C(6) mean plane by 0.972 and 0.648 Å, respectively. The ionized carboxyl group is axially oriented.

Comment

An improved method for the synthesis of the diastereomeric dimethyl diesters of tetrahydro-2H-1,4-thiazine-3,5-dicarboxylic acid has been recently reported by reacting methyl (*R,S*)-2,3-dibromopropionate with (*R*)-cysteine methyl ester (Paglialunga Paradisi, Pagani Zecchini, Torrini & Lucente, 1990). Taking into account the absolute configuration of