| | | | |
|---|---|---|---|
| C2—S1—C5 | 91.9 (1) | C8*a*—S7*a*—C6*a* | 91.0 (3) |
| C3—C2—S1 | 110.6 (2) | C9*a*—C8*a*—S7*a* | 115.0 (4) |
| C2$^{i}$—C2—S1 | 120.9 (2) | C10*a*—C6*a*—S7*a* | 107.7 (6) |
| C5—C4—C41 | 124.7 (2) | C5—C6*a*—S7*a* | 120.8 (4) |
| C3—C4—C41 | 124.2 (2) | C8*b*—S7*b*—C6*b* | 87.2 (7) |
| C4—C5—S1 | 111.8 (2) | C9*b*—C8*b*—S7*b* | 113.3 (12) |
| C6*a*—C5—S1 | 115.5 (3) | C10*b*—C6*b*—S7*b* | 113.0 (9) |
| C6*b*—C5—S1 | 124.9 (5) | C5—C6*b*—S7*b* | 108.8 (7) |

| | |
|---|---|
| C4—C41—C42—C43 | −176.6 (2) |
| C41—C42—C43—C44 | 178.7 (2) |
| C42—C43—C44—C45 | −176.5 (2) |
| C43—C44—C45—C46 | 176.6 (3) |
| C44—C45—C46—C47 | −177.1 (3) |
| C45—C46—C47—C48*a* | −177.0 (3) |
| C45—C46—C47—C48*b* | 158.0 (6) |
| C46—C47—C48*a*—C49*a* | −177.6 (4) |
| C47—C48*a*—C49*a*—C50*a* | 147.7 (6) |
| C46—C47—C48*b*—C49*b* | −171.8 (7) |
| C47—C48*b*—C49*b*—C50*b* | −128.6 (9) |

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

The disorder of the thiophene ring and the decyl group were treated separately. First, the major peaks in the $\Delta F$ map were found around the outer thiophene ring after the first cycles of refinement. Two models were built for the outer thiophene ring and restraints (same 1,2 and 1,3 distances as in the inner thiophene with s.u.) were employed in the subsequent refinements. A free variable 2 was given to the occupation factor of one of the two models. The subsequent refinements gave the proportion 65.0 (4):35.0 (4) for the two models. For the decyl chain, two reasonable models for the last three C atoms were found from the $\Delta F$ map; a free variable was then given to the occupation factor of one of the two models. During the refinement, restraints of the 1,2 and 1,3 distances among atoms C47–C50 were applied. Suprisingly, a very similar proportion, 65:35, was obtained for the two models after refinement. Therefore, a common free variable was given to the two parts of disorder in the next refinement. The proportion was found to be 65:35 with an s.u. of 0.4. In the final refinement, the occupation factors were fixed at 0.65 and 0.35. All non-H atoms were refined anisotropically in the major conformer and isotropically, except for the S atoms, in the minor conformer. The same displacement parameter was applied to the three disordered atoms (C48–C50). H atoms were constrained to the parent site using a riding model and refined isotropically (C—H 0.93–0.98 Å). The isotropic displacement parameters were adjusted to $1.5U_{eq}$ for methyl C atoms or $1.2U_{eq}$ for non-methyl C atoms. A final verification of possible voids was performed using *PLATON* (Spek, 1995).

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *CAD*-4 *Software*. Data reduction: *NRC*-2 and *NRC*-2*A* (Ahmed *et al.*, 1973). Program(s) used to solve structure: *SHELXS*96 (Sheldrick, 1990). Program(s) used to refine structure: *NRCVAX* (Gabe *et al.*, 1989) and *SHELXL*96 (Sheldrick, 1996). Molecular graphics: *ORTEP*II (Johnson, 1976) in *NRCVAX*. Software used to prepare material for publication: *NRCVAX* and *SHELXL*96.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: FG1393). Services for accessing these data are described at the back of the journal.

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*Acta Cryst.* (1998). C**54**, 555–557

# ***trans*-2,3-Diiodo-2-butene-1,4-diol**

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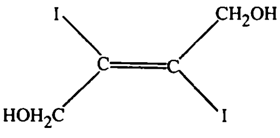
## Abstract

The crystal structure of *trans*-2,3-diiodo-2-butene-1,4-diol, $C_4H_6I_2O_2$, has been refined in space group $I4_1/a$, using data collected at 150 K. Bond angles around the $sp^2$-hybridized C atom show significant deviations from 120°.

## Comment

The title compound, (I), was prepared by iodine addition to the corresponding acetylene as described by Iserson

& Smith (1967). Addition of iodine to acetylenes results in *trans* isomers; however, *cis* products have also been reported (Hollins & Campos, 1979; Moreau & Commeyras, 1985). NMR studies of the stereochemical aspects of (I) were not successful (Priebe, 1997); therefore, a crystal structure analysis of (I) was undertaken.



(I)

There are no unusual bond lengths in this molecule. However, two of the three bond angles around the C1 atom show significant deviations from 120°, *i.e.* $C1^i—C1—C2 = 127.8 (1)°$ and $I—C1—C2 = 112.1 (1)°$ [symmetry code: (i) $\frac{3}{2} - x, \frac{3}{2} - y, -\frac{1}{2} - z$].

A search in the Cambridge Structural Database (Allen & Kennard, 1993) for diiodated double/aromatic bonds (no disorder, $R \leq 0.10$, atom coordinates reported) revealed a total of 15 structures, of which only two have an iodinated non-cyclic double bond, namely, tetraiodoethylene (Haywood & Shirley, 1977) and a pyrazine–tetraiodoethylene complex (Dahl & Hassel, 1968). The present structure thus presents the first diiodinated non-cyclic double bond.

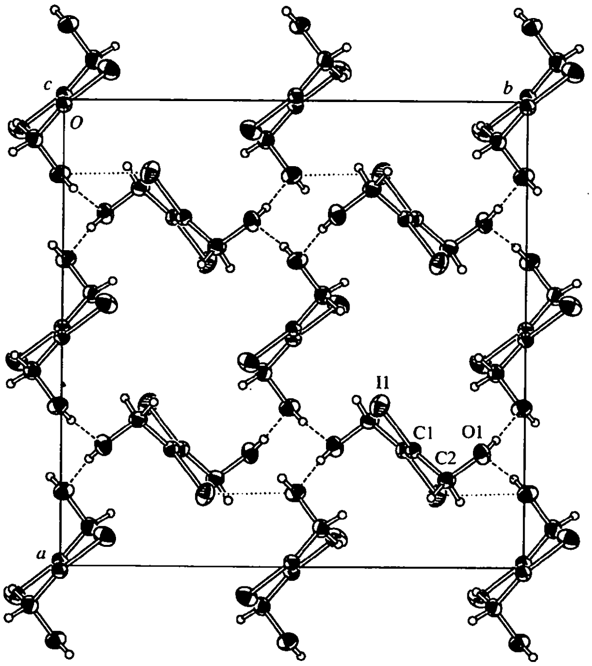


Fig. 1. Molecular packing diagram, with the atom numbering, of the *trans*-2,3-diiodo-2-butene-1,4-diol structure viewed along the *c* axis. Hydrogen bonds and charge-transfer interactions are illustrated using broken lines and dots, respectively. Displacement ellipsoids are drawn at the 75% probability level and H atoms are arbitrarily scaled.

There is only one type of hydrogen bond in the crystal, resulting in an infinite $\cdots O—H\cdots O—H\cdots$ chain, taking advantage of the cooperativity effect (Jeffrey & Saenger, 1991). Due to the fourfold screw axis, this chain constitutes a hydrophilic column with a hydrogen-bonded helix running parallel to the short crystallographic *c* axis (Fig. 1). The $O\cdots H$ distance is 1.87 Å and the $O—H\cdots O$ angle is 172°. The O atom is also engaged in a relatively weak charge-transfer interaction: $O\cdots I$ 3.429 (1) Å (the sum of the van der Waals radii is 3.55 Å).

The voluminous I atoms form a separate column along the *c* axis. The molecular packing arrangement is illustrated in Fig. 1.

## Experimental

Due to the instability of *trans*-2,3-diiodo-2-butene-1,4-diol in dimethyl sulfoxide when exposed to light, the crystallization was performed at room temperature in complete darkness. Crystals were grown by overnight diffusion of methanol into a saturated solution of *trans*-2,3-diiodo-2-butene-1,4-diol in dimethyl sulfoxide.

*Crystal data*

$C_4H_6I_2O_2$
$M_r = 339.89$
Tetragonal
$I4_1/a$ (second setting)
$a = 13.727 (1)$ Å
$c = 8.130 (1)$ Å
$V = 1531.9 (1)$ Å$^3$
$Z = 8$
$D_x = 2.947$ Mg m$^{-3}$
$D_m$ not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 8192 reflections
$\mu = 8.133$ mm$^{-1}$
$T = 150 (2)$ K
Block
$0.30 \times 0.30 \times 0.10$ mm
Colourless

*Data collection*

Siemens SMART CCD area-detector diffractometer
$\omega$ scan
Absorption correction: analytical *XPREP* (Siemens, 1996), followed by an empirical correction of intensities due to absorption by the glass fibre and glue (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.140$, $T_{max} = 0.428$

17 735 measured reflections
2891 independent reflections
2646 reflections with $I > 2\sigma(I)$
$R_{int} = 0.023$
$\theta_{max} = 44.93°$
$h = -27 \rightarrow 19$
$k = -27 \rightarrow 27$
$l = -12 \rightarrow 16$

*Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)] = 0.027$
$wR(F^2) = 0.053$
$S = 2.239$
2891 reflections
39 parameters
H atoms constrained

$\Delta\rho_{max} = 1.103$ e Å$^{-3}$
$\Delta\rho_{min} = -0.711$ e Å$^{-3}$
Extinction correction: *SHELXTL* (Sheldrick, 1994)
Extinction coefficient: 0.0049 (2)

$w = 1/[\sigma^2(F_o^2) + (0.0086P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$

Scattering factors from *International Tables for Crystallography* (Vol. C)

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

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U^{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

| | $x$ | $y$ | $z$ | $U_{\rm eq}$ |
|---|---|---|---|---|
| I1 | 0.654070 (8) | 0.687482 (7) | −0.006829 (11) | 0.02098 (3) |
| O1 | 0.75744 (8) | 0.90831 (7) | −0.00683 (12) | 0.01740 (14) |
| C1 | 0.75028 (8) | 0.76063 (8) | −0.16952 (15) | 0.01362 (15) |
| C2 | 0.81343 (9) | 0.83352 (10) | −0.0813 (2) | 0.0171 (2) |

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

| | | | |
|---|---|---|---|
| I1—C1 | 2.122 (1) | C1—C1$^{\rm i}$ | 1.341 (2) |
| O1—C2 | 1.418 (2) | C1—C2 | 1.505 (2) |
| C1$^{\rm i}$—C1—C2 | 127.8 (1) | C2—C1—I1 | 112.1 (1) |
| C1$^{\rm i}$—C1—I1 | 120.1 (1) | O1—C2—C1 | 111.9 (1) |
| C1$^{\rm i}$—C1—C2—O1 | 117.9 (2) | I1—C1—C2—O1 | −62.2 (1) |

Symmetry code: (i) $\frac{3}{2} - x, \frac{3}{2} - y, -\frac{1}{2} - z$.

The asymmetric unit contains half a molecule. All atoms, except for the three H atoms, were refined anisotropically. The two ethylene H atoms were fixed in idealized positions, refining only the C—H distance with $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C2})$. The hydroxy H atom was localized using the *AFIX* card 147 of *SHELXTL* (Sheldrick, 1994) and $U_{\rm iso}$ was set to $1.2U_{\rm eq}({\rm O1})$ in the subsequent refinement.

Data collection: *SMART* (Siemens, 1995). Cell refinement: *SAINT* (Siemens, 1995). Data reduction: *SAINT*. Program(s) used to solve structure: *SHELXTL*. Program(s) used to refine structure: *SHELXTL*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: AB1505). Services for accessing these data are described at the back of the journal.

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*Acta Cryst.* (1998). C**54**, 557–559

# 9-(4-Dimethylaminophenyl)-3,3,6,6-tetramethyl-3,4,6,7,9,10-hexahydro-1,8(2*H*,5*H*)-acridinedione

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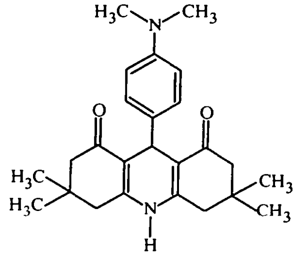
## Abstract

In the title compound, $C_{25}H_{32}N_2O_2$, the central ring in the acridine moiety is nearly planar, while the two outer rings adopt half-chair conformations. The phenyl ring is axial with respect to the central pyridine ring.

## Comment

Aminoacridinyl derivatives are biologically useful molecules, especially as potential anticancer agents. Acridines have been used as antibacterial agents for wound therapy (Neidle, 1979). Some of them fluoresce well as laser dyes in alcohol solvents (Selladurai *et al.*, 1990).

The bond lengths and angles of the title compound, (1), are similar to those of related acridine derivatives (Selladurai *et al.*, 1989, 1990; Sivaraman *et al.*, 1994, 1996). The O1 atom deviates significantly [−0.256 (2) Å] from the mean plane of ring I (Fig. 1), while the deviation of the O2 atom [−0.036 (5) Å] from the mean plane of ring III is small but significant. Phenyl ring IV is axial with respect to central pyridine ring II. The bonding geometry about each of the two N atoms is planar. Phenyl ring IV is nearly perpendicular to the mean plane of the acridine moiety. The buckling of the



(1)

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