C(15)—N(1)—C(2)	108.9 (4)	C(14)—C(15)—N(1)	133.5 (5)
C(17)—N(1)—C(2)	123.8 (4)	C(16)—C(15)—N(1)	106.9 (5)
C(17)—N(1)—C(15)	126.8 (4)	C(16)—C(15)—C(14)	119.5 (5)
C(21)—N(2)—C(8)	121.3 (5)	C(11)—C(16)—C(3)	127.8 (5)
C(22)—N(3)—C(21)	118.7 (6)	C(15)—C(16)—C(3)	108.4 (5)
C(24)—N(3)—C(21)	123.1 (6)	C(15)—C(16)—C(11)	123.8 (5)
C(24)—N(3)—C(22)	117.9 (4)	C(18)—C(17)—N(1)	114.7 (5)
C(7)—N(6)—C(5)	109.8 (4)	C(19)—C(18)—C(17)	126.2 (6)
C(20)—N(6)—C(5)	111.4 (4)	N(2)—C(21)—O(1)	121.1 (5)
C(20)—N(6)—C(7)	109.7 (4)	N(3)—C(21)—O(1)	122.6 (6)
C(3)—C(2)—N(1)	109.9 (5)	N(3)—C(21)—N(2)	116.3 (6)
C(4)—C(3)—C(2)	135.2 (6)	C(23)—C(22)—N(3)	112.0 (6)
C(16)—C(3)—C(2)	105.9 (4)	C(25)—C(24)—N(3)	112.4 (6)
C(16)—C(3)—C(4)	118.6 (5)	C(33)—O(32)—C(31)	118.7 (6)
C(5)—C(4)—C(3)	110.1 (5)	C(34)—C(33)—O(32)	125.9 (6)
C(4)—C(5)—N(6)	109.3 (4)	C(42)—C(33)—O(32)	113.4 (6)
C(10)—C(5)—N(6)	107.4 (4)	C(44)—C(43)—C(38)	112.4 (5)
C(10)—C(5)—C(4)	116.9 (4)	C(45)—C(43)—C(38)	109.0 (5)
C(8)—C(7)—N(6)	111.0 (5)	C(45)—C(43)—C(44)	112.0 (6)
C(7)—C(8)—N(2)	112.4 (4)	O(46)—C(45)—C(43)	123.2 (7)
C(9)—C(8)—N(2)	110.2 (4)	O(47)—C(45)—C(43)	114.3 (6)
C(9)—C(8)—C(7)	111.3 (5)	O(47)—C(45)—O(46)	122.3 (6)
C(10)—C(9)—C(8)	125.0 (5)	C(53)—O(52)—C(51)	116.5 (6)
C(9)—C(10)—C(5)	121.1 (5)	C(54)—C(53)—O(52)	126.0 (5)
C(11)—C(10)—C(5)	115.7 (5)	C(62)—C(53)—O(52)	114.1 (6)
C(11)—C(10)—C(9)	122.9 (5)	C(64)—C(63)—C(58)	110.6 (5)
C(12)—C(11)—C(10)	127.0 (5)	C(65)—C(63)—C(58)	109.6 (4)
C(16)—C(11)—C(10)	117.0 (5)	C(65)—C(63)—C(64)	112.0 (5)
C(16)—C(11)—C(12)	116.0 (5)	O(66)—C(65)—C(63)	121.3 (6)
C(13)—C(12)—C(11)	120.9 (6)	O(67)—C(65)—C(63)	115.8 (5)
C(14)—C(13)—C(12)	122.3 (6)	O(67)—C(65)—O(66)	122.9 (5)
C(15)—C(14)—C(13)	117.5 (5)		

C(2)—C(3)—C(4)—C(5)	−158.5 (6)
C(3)—C(4)—C(5)—N(6)	−165.7 (4)
C(34)—C(33)—O(32)—C(31)	9.8 (8)
C(37)—C(38)—C(43)—C(44)	64.9 (8)
C(37)—C(38)—C(43)—C(45)	−59.8 (8)
C(54)—C(53)—O(52)—C(51)	4 (1)
C(57)—C(58)—C(63)—C(65)	−28.6 (8)
C(57)—C(58)—C(63)—C(64)	95.4 (8)

An ω-scan width of $(1.21 + 0.30\tan\theta)^\circ$, an ω-scan rate of $4^\circ\ \text{min}^{-1}$ and background counts at the beginning and end of each scan, each for 50% of the total scan time, were used. The weak reflections, $I < 6\sigma(I)$, were rescanned (maximum of three rescans) and the counts accumulated to insure good counting statistics. Azimuthal scans of several reflections indicated no need for an absorption correction. The structure was solved by direct methods and refined by full-matrix least-squares methods. All the non-H atoms were refined anisotropically. The H atoms were introduced at calculated positions and refined as riding on the corresponding C atoms. Some H atoms were introduced only before the last cycles of refinement and after an accurate inspection of the structure. On the basis of C—O bond distances and consideration of intramolecular distances, which showed a network of hydrogen bonds, the following choices could be made: (*a*) the carboxyl group in molecule N*A* was considered undissociated and the H(44) atom was generated bound to O(47) and *trans* to C(43); (*b*) the carboxyl group in molecule N*B* was considered dissociated; (*c*) atom N(6) was considered protonated and H(66) was generated bound to N(6); (*d*) in the isopropyl alcohol, H(65) was generated bound to O(74) and *trans* to C(72); (*e*) the H atoms of the water molecule were neglected.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1985). Program(s) used to solve structure: *TEXSAN*. Program(s) used to refine structure: *CAOS* (Cerrini & Spagna, 1977). Molecular graphics: *TEXSAN*.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: NA1243). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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1,5-Bis[4,4-dimethyl-1-(4-methylphenyl)-5-(1-piperidinio)-1-penten-3-ylidene]carbonohydrazide Dichloride Methanol Solvate

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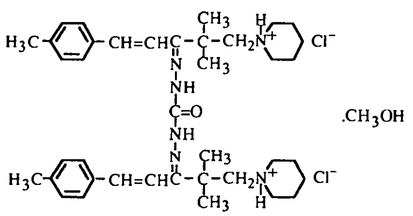
Abstract

The title compound, $C_{39}H_{58}N_6O^{2+}.2Cl^-.CH_3OH$, was synthesized as the lead molecule of a novel group of anticancer agents. The biscarbonohydrazide group in the molecule forms a complex conjugated resonance system involving the two 4-methylstyryl groups. Although the chemical bonding implies that the bonds of the molecule should have a mirror plane of symmetry, the molecule adopts an asymmetric bonding conformation.

Comment

Recently, Dimmock *et al.* (1996) described the syntheses and cytotoxicities of a novel group of biscarbonohydrazones of Mannich bases which displayed marked activity towards a number of murine and human tumours *in vitro*. The title compound, (I), which is four times as potent as the anticancer drug melphalan against a panel of 57 human tumour cell lines, is a very useful lead molecule for further molecular modifications in this series.



(I)

The biscarbonohydrazido group, together with atoms C4 and C4′, forms an essentially planar zigzag chain. Both piperidinium rings are in chair conformations and are approximately perpendicular to one another. The protonated N atoms of the two piperidinium groups form hydrogen bonds with the chloride Cl1 ion and the carbonyl O atom. All the C=N and C=C double bonds are in an *E* configuration. Comparison of the interatomic distances in the biscarbonohydrazido group with the expected values given by Allen *et al.* (1987) suggests considerable delocalization of the π bonds in this region and indicates that the biscarbonohydrazide is a conjugated resonance system.

The two methylstyryl groups rotate around the C2—C3 and C2′—C3′ bonds in order to reduce steric interactions with the H atoms on N3 and N3′, respectively. One azabutadiene unit, C1=C2—C3=N2, is twisted by 40.9 (6)° from the *s-cis* conformation and the other, C1′=C2′—C3′=N2′, is twisted by −159.9 (11)° from the *s-trans* conformation. Such large angle rotations should significantly reduce the conjugation in the azabutadiene groups. However, the bond distances C2—C3 and C2′–C3′ are comparable to the C—C single-bond distance of 1.472 (3) Å found in the azabutadiene group determined by Dimmock *et al.* (1995), suggesting that the two azabutadiene groups are still conjugated.

The methylstyryl groups are not planar. Rotations about C1—C6 and C1′—C6′ (see Table 2) give further conformational differences between the two chemically equivalent parts of the molecule. Surface charge distributions were calculated using the program *MACROMODEL* (Mohamadi *et al.*, 1990) and surface electrostatic potentials of the molecule were calculated by the program *DelPhi* (Sharp & Nicholls, 1989) to try to explain why the two methylstyryl groups show such a significant difference. No obvious explanation has been found. The N1′—H hydrogen bond to the carbonyl oxygen does impart an asymmetry to the molecule, which combined with the results of lattice packing, must somehow cause the observed configurational differences of the styryl groups.

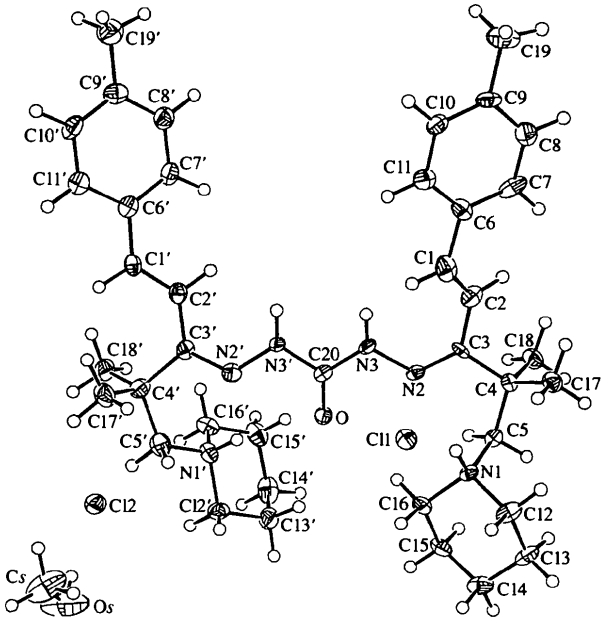


Fig. 1. An *ORTEPII* (Johnson, 1976) view of the title compound with non-H displacement ellipsoids drawn at the 50% probability level. For clarity, the H atoms are drawn as small spheres of arbitrary size.

Experimental

The crystals of the title compound were obtained by vapour diffusion using the solvents methanol and benzene.

Crystal data

$C_{39}H_{58}N_6O^{2+}.2Cl^-.CH_4O$
$M_r = 729.87$
Monoclinic
$P2_1/n$
$a = 18.019\,(2)$ Å
$b = 13.546\,(4)$ Å
$c = 18.092\,(3)$ Å
$\beta = 115.389\,(12)°$
$V = 3989.6\,(14)$ Å^3
$Z = 4$
$D_x = 1.215$ Mg m^{-3}
$D_m = 1.216$ Mg m^{-3}
D_m measured by flotation

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 8.5–14.5°$
$\mu = 0.20$ mm^{-1}
$T = 123\,(2)$ K
Prism
0.40 × 0.25 × 0.08 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\theta/2\theta$ scans
Absorption correction: none
5748 measured reflections
5161 independent reflections
3039 observed reflections
$[I_{net} > 2\sigma(I_{net})]$

$R_{int} = 0.017$
$\theta_{max} = 22.42°$
$h = -19 \rightarrow 17$
$k = 0 \rightarrow 14$
$l = 0 \rightarrow 19$
3 standard reflections
frequency: 120 min
intensity decay: none

Refinement

Refinement on F
$R = 0.056$
$wR = 0.055$
$S = 1.57$
3039 reflections
451 parameters
H atoms not refined
$w = 1/[\sigma^2(F) + 0.0003F^2]$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.42$ e Å^{-3}
$\Delta\rho_{\min} = -0.36$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

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	$U_{\rm eq}$
Cl1	0.84117 (8)	1.05149 (10)	0.87536 (8)	0.0248 (9)
Cl2	1.26178 (8)	0.88721 (12)	0.61844 (9)	0.0340 (10)
O	1.0076 (2)	0.8464 (3)	0.8273 (2)	0.025 (3)
N1	0.7632 (2)	0.8543 (3)	0.7809 (2)	0.019 (3)
N2	0.9209 (2)	0.8073 (3)	0.9128 (3)	0.018 (3)
N3	1.0007 (2)	0.8429 (3)	0.9511 (2)	0.019 (3)
C1	0.9518 (4)	0.8517 (5)	1.0952 (4)	0.033 (4)
C2	0.9082 (3)	0.7859 (5)	1.0433 (4)	0.029 (4)
C3	0.8813 (3)	0.7839 (4)	0.9545 (3)	0.018 (3)
C4	0.7964 (3)	0.7405 (4)	0.9046 (3)	0.020 (3)
C5	0.7712 (3)	0.7499 (4)	0.8127 (3)	0.019 (3)
C6	0.9754 (3)	0.8496 (4)	1.1873 (3)	0.025 (4)
C7	0.9374 (3)	0.7865 (4)	1.2199 (4)	0.031 (4)
C8	0.9596 (3)	0.7872 (4)	1.3032 (4)	0.031 (4)
C9	1.0211 (3)	0.8495 (5)	1.3557 (3)	0.027 (4)
C10	1.0575 (3)	0.9127 (4)	1.3206 (3)	0.027 (4)
C11	1.0346 (3)	0.9130 (4)	1.2377 (3)	0.028 (4)
C12	0.6757 (3)	0.8927 (4)	0.7447 (3)	0.030 (4)
C13	0.6227 (3)	0.8419 (4)	0.6645 (3)	0.031 (4)
C14	0.6587 (4)	0.8509 (4)	0.6032 (3)	0.033 (4)
C15	0.7468 (3)	0.8145 (4)	0.6402 (3)	0.027 (4)
C16	0.7988 (3)	0.8672 (4)	0.7198 (3)	0.025 (3)
C17	0.7340 (3)	0.7883 (4)	0.9300 (3)	0.026 (4)
C18	0.7994 (3)	0.6288 (4)	0.9205 (3)	0.029 (4)
C19	1.0458 (4)	0.8505 (5)	1.4468 (4)	0.047 (5)
C20	1.0382 (3)	0.8610 (4)	0.9004 (3)	0.019 (4)
N1′	1.1313 (3)	0.8924 (4)	0.7442 (3)	0.026 (3)
N2′	1.1518 (3)	0.9289 (3)	0.8939 (3)	0.024 (3)
N3′	1.1168 (2)	0.8958 (3)	0.9435 (2)	0.021 (3)
C1′	1.3571 (3)	0.9812 (4)	1.0524 (3)	0.023 (4)
C2′	1.2847 (3)	0.9385 (4)	1.0098 (3)	0.024 (4)
C3′	1.2290 (3)	0.9508 (4)	0.9238 (3)	0.020 (4)
C4′	1.2554 (3)	0.9893 (4)	0.8593 (3)	0.028 (4)
C5′	1.1852 (3)	0.9836 (4)	0.7714 (4)	0.030 (4)
C6′	1.4089 (3)	0.9681 (4)	1.1394 (3)	0.024 (4)
C7′	1.3951 (3)	0.8957 (4)	1.1864 (4)	0.029 (4)
C8′	1.4454 (3)	0.8864 (5)	1.2694 (3)	0.030 (4)
C9′	1.5108 (3)	0.9508 (5)	1.3082 (3)	0.029 (4)
C10′	1.5259 (3)	1.0220 (4)	1.2616 (4)	0.033 (4)
C11′	1.4758 (3)	1.0314 (4)	1.1787 (4)	0.030 (4)
C12′	1.0708 (3)	0.9049 (5)	0.6560 (3)	0.028 (4)
C13′	1.0101 (3)	0.8210 (5)	0.6317 (3)	0.034 (4)
C14′	1.0531 (4)	0.7220 (5)	0.6449 (4)	0.038 (4)
C15′	1.1158 (4)	0.7135 (5)	0.7339 (4)	0.036 (4)
C16′	1.1766 (3)	0.7970 (4)	0.7584 (3)	0.029 (4)
C17′	1.2694 (4)	1.1023 (5)	0.8718 (4)	0.036 (4)
C18′	1.3339 (3)	0.9392 (5)	0.8641 (3)	0.034 (4)
C19′	1.5648 (4)	0.9390 (5)	1.3975 (4)	0.044 (4)
Osol	1.1841 (4)	1.0638 (4)	0.5021 (3)	0.097 (5)
Csol	1.2035 (5)	1.1422 (6)	0.5567 (5)	0.071 (6)

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

O—C20	1.211 (7)	C20—N3′	1.375 (7)
N2—N3	1.389 (6)	N2′—N3′	1.375 (6)
N2—C3	1.280 (7)	N2′—C3′	1.292 (7)
N3—C20	1.373 (7)	C1′—C2′	1.331 (8)
C1—C2	1.288 (9)	C1′—C6′	1.457 (8)
C1—C6	1.533 (8)		
N3—N2—C3	120.7 (4)	N3—C20—N3′	111.4 (4)
N2—N3—C20	115.4 (4)	N3′—N2′—C3′	120.9 (4)
C2—C1—C6	125.2 (6)	C20—N3′—N2′	113.1 (4)
C1—C2—C3	128.5 (6)	C2′—C1′—C6′	125.9 (5)
N2—C3—C2	129.5 (5)	C1′—C2′—C3′	128.6 (5)
N2—C3—C4	114.7 (4)	N2′—C3′—C2′	123.1 (5)
C2—C3—C4	115.6 (4)	N2′—C3′—C4′	112.9 (4)
O—C20—N3	125.2 (5)	C2′—C3′—C4′	124.0 (4)
O—C20—N3′	123.3 (5)		
C2—C1—C6—C7	−13.7 (6)	C2′—C1′—C6′—C7′	11.4 (5)
C1—C2—C3—N2	40.9 (6)	C1′—C2′—C3′—N2′	−159.9 (11)
C6—C1—C2—C3	178.2 (11)	C6′—C1′—C2′—C3′	177.8 (10)
C3—N2—N3—C20	177.0 (9)	N3—C20—N3′—N2′	−170.7 (10)
N3—N2—C3—C4	−176.6 (9)	C3′—N2′—N3′—C20	−170.1 (9)
N2—N3—C20—N3′	−179.9 (9)	N3′—N2′—C3′—C4′	−178.1 (9)

All H atoms were placed in calculated positions on their corresponding O, N or C atom (O—H 0.85, N—H 0.90 and C—H 1.00 Å) and were not refined. The $U_{\rm iso}$ value of each H atom was assigned as be equal to the $U_{\rm eq}$ value of the attached non-H atom plus 0.01. The ω-scan width was $(0.90 + 0.35\tan\theta)^\circ$, with a θ-scan rate of 0.72–5.49° min^{-1}. The scan angle was extended 25% on each side of each peak for background measurement. Refinement was by full-matrix least-squares methods.

Data collection: *CAD*-4 *Operations Manual* (Enraf–Nonius, 1988). Cell refinement: *CAD*-4 *Operations Manual*. Data reduction: *DATRD*2 in *NRCVAX* (Gabe, Le Page, Charland, Lee & White, 1989). Program(s) used to solve structure: *SOLVER* in *NRCVAX*. Program(s) used to refine structure: *LSTSQ* in *NRCVAX*. Molecular graphics: *ORTEP*II (Johnson, 1976) in *NRCVAX*. Software used to prepare material for publication: *TABLES* (version of January 1994) and *UTILITY* in *NRCVAX*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: HA1170). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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5-(4-Hydroxyphenyl)tetrazole–Water (2/3)

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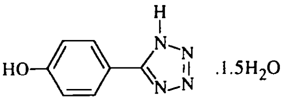
Abstract

The title compound, $C_7H_6N_4O.1.5H_2O$, is a key intermediate in the synthesis of mesogens, which are derivatives of phenyltetrazole. The tetrazole and phenyl rings are planar to within 0.007 (2) Å, with a dihedral angle of 14.8 (1)° between the planes.

Comment

As part of our synthetic and structural studies of conventional liquid crystals and metallomesogens which contain a heterocyclic ring (Gallardo & Merlo, 1993; Gallardo & Favarin, 1993; Gallardo & Begnini, 1995), the structure of the title compound, (I), has been determined and is presented here.



(I)

A rather small number of structures with a tetrazole ring have been described (Destro & Soave, 1995; Gowda, Rudman & Acharya, 1982; Bray & White, 1979; Bradbury *et al.*, 1992). The bond distances and angles in the title molecule are similar to the values found in another phenyltetrazole (Gallardo, Meyer & Vencato, 1995). In the tetrazole ring, the N2—N3 distance of 1.286 (2) Å is clearly a double bond, significantly shorter than the N1—N2 and N3—N4 single bonds of 1.360 (2) and 1.344 (2) Å, respectively. These values are consistent with those observed previously for the tetrazole ring.

The structure consists of almost planar molecules joined by hydrogen bonds to water molecules, resulting in layers extending along the *bc* face of the unit cell (Table 3). The r.m.s. deviation of atoms C1–C7, O1 and N1–N4 from the least-squares plane through them is 0.106 Å. The hydrogen-bond geometry around the O*W*2 water molecule is quite planar, as can likewise be seen by the r.m.s. deviation of 0.120 Å from the plane through O*W*1, O*W*2, N2^{iv} and N4^{ii} (see Table 3 for symmetry codes). The hydrogen bonds to the O*W*1 water molecule are in an approximately tetrahedral configuration.

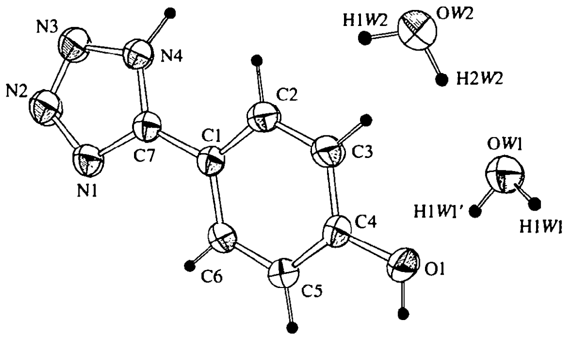


Fig. 1. An *ORTEP*II (Johnson, 1976) drawing of the molecular structure of (I). Displacement ellipsoids are drawn at the 50% probability level.

Experimental

The title molecule was prepared by treating 4-hydroxybenzonitrile with NaN_3 in NH_4Cl/DMF. The reaction mixture was then heated and refluxed for 3 h, cooled and poured into 200 ml of water and crushed ice. The crude tetrazole was separated as a solid and recrystallized from a 1:1 mixture of benzene and ethanol. ^1H NMR (200 MHz, DMSO-d_6): δ 7.05 (*d*, 3H, J = 8.4 Hz, aromatic system), 7.95 (*d*, 2H, J = 8.4 Hz, aromatic system).

Crystal data

$C_7H_6N_4O.1.5H_2O$
M_r = 189.18
Monoclinic
$C2/c$
a = 14.852 (1) Å
b = 9.910 (1) Å
c = 13.036 (2) Å
β = 113.749 (8)°
V = 1756.2 (3) Å^3
Z = 8
D_x = 1.431 Mg m^{-3}
D_m = 1.42 (2) Mg m^{-3}
D_m measured by flotation in CCl_4/benzene

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 25 reflections
θ = 8.59–13.92°
μ = 0.112 mm^{-1}
T = 293 (2) K
Prismatic
0.60 × 0.60 × 0.25 mm
Colourless

Data collection

Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
1625 measured reflections
1552 independent reflections
1334 observed reflections [$I > 2\sigma(I)$]

$R_{\rm int}$ = 0.0123
$\theta_{\rm max}$ = 25°
$h = -17 \rightarrow 16$
$k = 0 \rightarrow 11$
$l = 0 \rightarrow 15$
3 standard reflections
frequency: 60 min
intensity decay: −1.5%

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