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Structure of 4:1 Sulfuric Acid–*N*,*N*'-(*p*-Phenylene)dibenzamide Complex, $C_{20}H_{16}N_2O_2.4H_2SO_4$

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Abstract. $M_r = 708{\cdot}66$, triclinic, $P1$, $a = 9{\cdot}747\,(1)$, $b = 10{\cdot}313\,(1)$, $c = 7{\cdot}879\,(1)$ Å, $\alpha = 108{\cdot}67\,(1)$, $\beta = 111{\cdot}41\,(1)$, $\gamma = 89{\cdot}20\,(1)°$, $V = 693{\cdot}8$ Å^3, $Z = 1$, $D_x = 1{\cdot}696$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71069$ Å, $\mu = 4{\cdot}128$ cm^{-1}, $F(000) = 366$, $T = 173$ K, $R = 0{\cdot}026$, 2877 reflections with $I \geq 2\sigma(I)$. The structure of the 4:1 sulfuric acid complex with PPDB shows a more planar conformation of the three rings than does the uncomplexed analogue. The carbonyl oxygens are protonated to give a sulfuric acid/bisulfate salt. The sulfuric acid and bisulfate groups form hydrogen-bonded layers, which alternate with sheets of PPDB molecules.

Introduction. Aromatic polyamides (aramids) form a class of polymers having exceptional thermal and mechanical properties. One member of the class, poly(*p*-phenyleneterephthalamide) (PPTA) has been commercialized by Du Pont as KEVLAR® aramid fibers. In order to gain insight into the structure of PPTA, the crystal structure of *N*,*N*'-(*p*-phenylene)dibenzamide (PPDB) has been determined (Harkema & Gaymans, 1977; Adams, Fratini & Wiff, 1978). It has been reported recently that certain aramids cocrystallize with solvent to form regular crystal solvates (Iovleva & Papkov, 1982). PPTA has been found to form a crystal solvate with sulfuric acid (Iovleva, Banduryan, Ivanova, Platonov, Milkova, Khanin, Volokhina & Papkov, 1979). In this paper we report the crystal structure of a 4:1 complex between sulfuric acid and PPDB.

Experimental. Colorless pyramidal crystal, *ca* $0{\cdot}25 \times 0{\cdot}25 \times 0{\cdot}40$ mm, obtained from slow cooling of supersaturated H_2SO_4 solution, sealed in capillary under N_2 in dry box; Syntex *R*3 diffractometer at low temperature (173 K). Lattice parameters from 23 reflections with $20 < 2\theta < 27°$ averaged with Friedel pairs; azimuthal scans indicated only statistical variation of intensities; no correction applied. 3456 data measured, $4 < 2\theta < 55°$; ω scans; h,k,l ± 13, ± 12, 10. Twenty reflections that exceeded the counting capacity of the detector omitted; data normalized for 10% fluctuation of intensity during collection (200 reflections collected during 6% standard intensity fluctuation omitted), 2877 unique data with $I > 2\sigma(I)$ (115 unobserved). Structure solved with some difficulty using direct methods (*MULTAN*; Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978) in space group $P\bar{1}$. However, an examination of the thermal ellipsoids of some of the sulfate oxygens prompted investigation of the acentric equivalent. The centric domination was broken by a series of Fourier refinements, and further full-matrix least-squares refinement then proceeded smoothly using space group $P1$. All hydrogen atoms obtained from difference electron density maps and refined isotropically with remaining anisotropic non-hydrogen atoms (refinement on F, 490 parameters) to give $R = 0{\cdot}026$, $R_w = 0{\cdot}032$ and $S = 1{\cdot}49$ with av. Δ/σ 0·1. Statistical weights employed, with $\sigma(F) \propto 1/[\sigma(I)^2 + (0{\cdot}03I)^2]$. Coordinates used correspond to enantiomorph with lowest R value. Largest residuals in final difference map (0·20–0·25 e Å^{-3}) correspond to density between carbon atoms in phenyl rings. Scattering factors from *International Tables for X-ray Crystallography* (1974) and included terms for anomolous scattering of sulfur (*International Tables for X-ray Crystallography,* 1972). All incidental programs (data reduction, Fourier, least squares) were written by one of us (JCC). Plots made with *ORTEP* (Johnson, 1976).*

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39712 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Discussion. Final atomic coordinates are listed in Table 1, bond lengths, angles and intermolecular distances in Table 2. A perspective view of the molecule together with the four nearest H_2SO_4 groups in the plane of the phenyl group is presented in Fig. 1. Two of the sulfuric acid molecules can be seen to have protonated the carbonyl oxygens of the amide groups. Thus, the structure is actually a sulfuric acid/bisulfate salt of PPDB. The arrangement of the ions, with the two sulfuric acid molecules at one end of the PPDB and the bisulfate ions at the other end, further reinforces the acentric symmetry of the unit cell.

A comparison of the present structure with the monoclinic PPDB-*m* (Harkema & Gaymans, 1977) and orthorhombic PPDB-*o* (Adams *et al.* 1978) forms

Table 1. *Fractional coordinates* ($\times 10^4$, $\times 10^3$ *for* H) *and isotropic thermal parameters*

$B_{\rm eq} = \frac{4}{3}\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j$ for non-hydrogen atoms.

	x	*y*	*z*	$B_{\rm eq}/B_{\rm iso}$(Å^2)
S(1)	4365	751	−2450	1·5 (1)
S(2)	−4360 (1)	−751 (1)	2425 (2)	1·5 (1)
S(3)	3551 (1)	−5166 (1)	1531 (1)	1·6 (1)
S(4)	−3848 (1)	5262 (1)	−2338 (1)	1·6 (1)
O(1)	1889 (3)	−2256 (3)	2721 (4)	1·9 (1)
O(2)	−1838 (3)	2228 (3)	−3116 (3)	1·7 (1)
O(11)	4981 (3)	−619 (2)	−2394 (3)	2·0 (1)
O(12)	4716 (3)	1643 (3)	−492 (4)	2·9 (1)
O(13)	2831 (3)	482 (3)	−3702 (4)	2·2 (1)
O(14)	5289 (3)	1324 (3)	−3273 (4)	2·4 (1)
O(21)	−2786 (3)	−439 (3)	3007 (4)	2·2 (1)
O(22)	−5015 (3)	440 (3)	3285 (4)	2·3 (1)
O(23)	−5161 (3)	−1346 (3)	335 (4)	2·7 (1)
O(24)	−4590 (3)	−1844 (3)	3297 (4)	2·8 (1)
O(31)	4436 (4)	−4395 (3)	946 (5)	4·5 (1)
O(32)	3458 (3)	−4278 (2)	3346 (3)	1·7 (1)
O(33)	2192 (3)	−5803 (4)	24 (4)	3·7 (1)
O(34)	4460 (5)	−6268 (3)	2173 (4)	5·0 (1)
O(41)	−3412 (3)	4236 (3)	−3709 (4)	2·5 (1)
O(42)	−2763 (3)	5948 (3)	−457 (4)	3·2 (1)
O(43)	−4447 (3)	6395 (2)	−3191 (4)	2·1 (1)
O(44)	−5192 (4)	4581 (3)	−2264 (4)	2·8 (1)
N(1)	−201 (3)	−1277 (3)	2338 (4)	1·2 (1)
N(2)	184 (3)	1174 (3)	−2936 (4)	1·3 (1)
C(1)	426 (4)	−2622 (3)	4468 (4)	1·3 (1)
C(2)	−1021 (4)	−3080 (3)	4143 (5)	1·7 (1)
C(3)	−1251 (4)	−3623 (4)	5422 (5)	2·2 (1)
C(4)	−78 (5)	−3694 (4)	7015 (5)	2·2 (1)
C(5)	1345 (5)	−3236 (4)	7344 (5)	2·2 (1)
C(6)	1617 (4)	−2707 (3)	6070 (5)	1·8 (1)
C(7)	711 (4)	−2040 (3)	3135 (4)	1·3 (1)
C(8)	−62 (3)	−671 (3)	995 (4)	1·2 (1)
C(9)	−1343 (4)	−701 (3)	−564 (5)	1·5 (1)
C(10)	−1281 (4)	−96 (4)	−1881 (5)	1·7 (1)
C(11)	66 (3)	539 (3)	−1595 (4)	1·2 (1)
C(12)	1348 (3)	557 (3)	−50 (4)	1·5 (1)
C(13)	1288 (3)	−40 (3)	1272 (4)	1·5 (1)
C(14)	−725 (3)	1976 (3)	−3646 (4)	1·3 (1)
C(15)	−499 (4)	2543 (3)	−5025 (4)	1·4 (1)
C(16)	926 (4)	2955 (3)	−4818 (5)	1·6 (1)
C(17)	1112 (4)	3460 (4)	−6164 (5)	2·1 (1)
C(18)	−116 (5)	3513 (4)	−7739 (5)	2·3 (1)
C(19)	−1536 (4)	3104 (4)	−7951 (5)	2·1 (1)
C(20)	−1732 (4)	2628 (4)	−6593 (5)	1·7 (1)
H(1N)	−99 (4)	−120 (3)	255 (5)	0·6 (6)
H(1O)	229 (6)	−286 (5)	299 (7)	3·2 (10)
H(2)	−174 (5)	−301 (4)	309 (6)	2·0 (8)
H(2N)	86 (5)	95 (5)	−324 (6)	2·5 (9)
H(2O)	−241 (7)	279 (6)	−351 (8)	5·1 (13)
H(3)	−206 (6)	−389 (6)	515 (8)	4·4 (13)
H(4)	−15 (5)	−402 (5)	801 (6)	3·4 (10)
H(5)	207 (5)	−333 (5)	843 (7)	3·7 (11)
H(6)	256 (5)	−242 (5)	621 (6)	2·3 (8)
H(9)	−220 (6)	−110 (5)	−60 (7)	3·3 (10)
H(10)	−219 (4)	−16 (4)	−303 (5)	1·7 (7)
H(11)	481 (6)	−85 (5)	−152 (7)	3·9 (11)
H(12)	228 (5)	107 (4)	23 (6)	2·3 (8)
H(13)	211 (4)	−7 (4)	228 (5)	1·4 (7)
H(14)	500 (9)	98 (8)	−424 (11)	8·4 (23)
H(16)	177 (4)	296 (4)	−373 (5)	1·2 (6)
H(17)	208 (5)	381 (4)	−601 (5)	1·9 (7)
H(18)	3 (5)	381 (5)	−864 (7)	3·2 (9)
H(19)	−249 (5)	312 (4)	−907 (6)	2·4 (9)
H(20)	−273 (6)	233 (5)	−676 (7)	3·2 (10)
H(24)	−455 (7)	−258 (7)	251 (9)	6·3 (16)
H(34)	458 (7)	−681 (6)	124 (9)	5·7 (14)
H(43)	−507 (5)	605 (4)	−430 (6)	2·1 (8)
H(44)	−526 (8)	491 (7)	−146 (10)	7·8 (22)

Table 2. *Interatomic distances* (Å) *and angles* (°) *and intermolecular distances* (Å)

S(1)–O(11)	1·535 (2)	N(2)–C(14)	1·303 (4)
S(1)–O(12)	1·435 (2)	C(1)–C(2)	1·399 (4)
S(1)–O(13)	1·430 (2)	C(1)–C(6)	1·395 (4)
S(1)–O(14)	1·506 (2)	C(1)–C(7)	1·471 (4)
S(2)–O(21)	1·441 (2)	C(2)–C(3)	1·381 (5)
S(2)–O(22)	1·462 (2)	C(3)–C(4)	1·377 (5)
S(2)–O(23)	1·458 (2)	C(4)–C(5)	1·375 (6)
S(2)–O(24)	1·552 (3)	C(5)–C(6)	1·387 (5)
S(3)–O(31)	1·460 (3)	C(8)–C(9)	1·387 (4)
S(3)–O(32)	1·465 (2)	C(8)–C(13)	1·388 (4)
S(3)–O(33)	1·404 (3)	C(9)–C(10)	1·389 (4)
S(3)–O(34)	1·532 (3)	C(10)–C(11)	1·384 (4)
S(4)–O(41)	1·435 (3)	C(11)–C(12)	1·386 (4)
S(4)–O(42)	1·421 (3)	C(12)–C(13)	1·386 (4)
S(4)–O(43)	1·534 (2)	C(14)–C(15)	1·468 (4)
S(4)–O(44)	1·523 (3)	C(15)–C(16)	1·394 (4)
O(1)–C(7)	1·301 (4)	C(15)–C(20)	1·401 (4)
O(2)–C(14)	1·293 (4)	C(16)–C(17)	1·388 (4)
N(1)–C(7)	1·301 (4)	C(17)–C(18)	1·391 (5)
N(1)–C(8)	1·435 (3)	C(18)–C(19)	1·387 (5)
N(2)–C(11)	1·447 (4)	C(19)–C(20)	1·382 (4)

O(11)–S(1)–O(12)	108·1 (1)	O(2)–C(14)–C(15)	121·8 (3)
O(11)–S(1)–O(13)	109·3 (1)	N(1)–C(7)–C(1)	121·1 (3)
O(11)–S(1)–O(14)	102·7 (1)	N(1)–C(8)–C(9)	117·3 (3)
O(12)–S(1)–O(13)	116·5 (2)	N(1)–C(8)–C(13)	121·5 (2)
O(12)–S(1)–O(14)	107·8 (2)	N(2)–C(11)–C(10)	120·8 (2)
O(13)–S(1)–O(14)	111·5 (2)	N(2)–C(11)–C(12)	117·9 (3)
O(21)–S(2)–O(22)	111·5 (1)	N(2)–C(14)–C(15)	120·7 (3)
O(21)–S(2)–O(23)	114·0 (2)	C(2)–C(1)–C(6)	120·3 (3)
O(21)–S(2)–O(24)	107·5 (2)	C(2)–C(1)–C(7)	120·5 (3)
O(22)–S(2)–O(23)	111·7 (2)	C(6)–C(1)–C(7)	119·2 (3)
O(22)–S(2)–O(24)	104·8 (2)	C(1)–C(2)–C(3)	119·1 (3)
O(23)–S(2)–O(24)	106·7 (2)	C(2)–C(3)–C(4)	120·7 (3)
O(31)–S(3)–O(32)	108·9 (2)	C(3)–C(4)–C(5)	120·3 (3)
O(31)–S(3)–O(33)	112·5 (2)	C(4)–C(5)–C(6)	120·5 (3)
O(31)–S(3)–O(34)	107·7 (2)	C(1)–C(6)–C(5)	119·2 (3)
O(32)–S(3)–O(33)	115·8 (2)	C(9)–C(8)–C(13)	121·1 (3)
O(32)–S(3)–O(34)	101·8 (1)	C(8)–C(9)–C(10)	119·8 (3)
O(33)–S(3)–O(34)	109·3 (2)	C(9)–C(10)–C(11)	118·9 (3)
O(41)–S(4)–O(42)	118·6 (2)	C(10)–C(11)–C(12)	121·3 (3)
O(41)–S(4)–O(43)	108·4 (1)	C(11)–C(12)–C(13)	119·9 (3)
O(41)–S(4)–O(44)	106·2 (2)	C(8)–C(13)–C(12)	118·9 (3)
O(42)–S(4)–O(43)	106·0 (2)	C(14)–C(15)–C(16)	120·7 (3)
O(42)–S(4)–O(44)	112·0 (2)	C(14)–C(15)–C(20)	119·2 (3)
O(43)–S(4)–O(44)	104·7 (2)	C(16)–C(15)–C(20)	120·0 (3)
C(7)–N(1)–C(8)	126·4 (3)	C(15)–C(16)–C(17)	119·6 (3)
C(11)–N(2)–C(14)	125·8 (3)	C(16)–C(17)–C(18)	119·9 (3)
O(1)–C(7)–N(1)	117·5 (3)	C(17)–C(18)–C(19)	120·6 (3)
O(2)–C(14)–N(2)	117·5 (3)	C(18)–C(19)–C(20)	119·7 (3)
O(1)–C(7)–C(1)	121·4 (3)	C(15)–C(20)–C(19)	120·0 (3)

O(1)···O(32)	2·636 (3)	O(24)···O(31$^{\rm v}$)	2·630 (4)
O(2)···O(41)	2·620 (3)	O(31)···O(44$^{\rm iii}$)	2·568 (4)
O(11)···O(23$^{\rm ii}$)	2·540 (3)	O(32)···O(43$^{\rm vi}$)	2·618 (3)
O(12)···O(34$^{\rm iv}$)	2·566 (4)	O(13)···N(2)	2·887 (4)
O(14)···O(22$^{\rm i}$)	2·472 (4)	O(21)···N(1)	2·826 (4)

Symmetry code

(i) $1 + x, y, -1 + z$
(ii) $1 + x, y, z$
(iii) $1 + x, -1 + y, z$
(iv) $x, 1 + y, z$
(v) $-1 + x, y, z$
(vi) $1 + x, -1 + y, 1 + z$

Table 3. *A structural comparison of several N,N′-(p-phenylene)dibenzamide structures*

PPDB-*m* (monoclinic), PPDB-*o* (orthorhombic), DPTP (*N,N*-diphenylterephthalamide) and the protonated bisulfate PPDB complex. Ring *A* refers to the outermost phenyl ring and ring *B* is the central phenyl ring. (Distances in Å, angles in deg.)

Parameter	PPDB-*m*	PPDB-*o*	DPTP	$H_2PPDB^{2+}(.2HSO_4^-)$	
C—O distance	1·222 (4)	1·229 (2)	1·226 (2)	1·301 (4)	1·293 (4)
C—N distance	1·355 (4)	1·356 (2)	1·359 (2)	1·301 (4)	1·303 (4)
H(N)···H(*ortho*)	2·28	2·25	2·24	2·22 (4)	2·45 (4)
Amide···ring *A* angle	28·9	23·8	30·7	36·6 (4)	37·9 (4)
Amide···ring *B* angle	35·7	55·4	30·4	−41·2 (4)	−44·4 (4)
Ring *A* –ring *B* angle	64·4	79·2	60·9	5·7 (4)	8·2 (4)

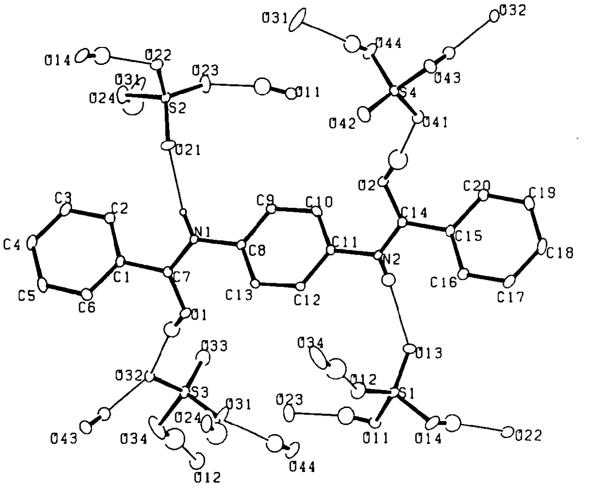

Fig. 1. Asymmetric unit of 4:1 sulfuric acid–*N,N′*-(*p*-phenylene)-dibenzamide complex showing the atom numbering scheme. Fine lines indicate hydrogen bonding.

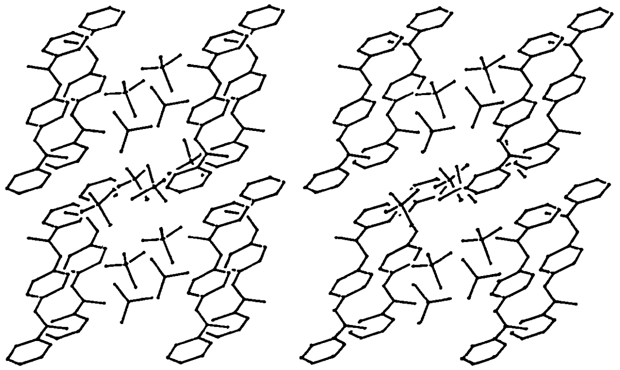

Fig. 2. Stereoscopic view of the packing in the crystal viewed along the *z* axis. Sheets of PPDB cation stacks are separated by sulfate/bisulfate layers.

of the neutral species and the structure of *N,N′*-diphenylterephthalamide (DPTP) (Harkema, Gaymans, van Hummel & Zylberlicht, 1979) is presented in Table 3.

The protonated oxygens cause an expected lengthening of the C—O bonds from 1·22 Å (av.) in the neutral compound to 1·30 Å (av.) here. In addition, the amide bond is shortened from 1·36 (av.) to 1·30 Å, indicating substantial increase of double-bond character.

Both previous studies of the neutral compound indicate a significant influence of crystal packing on the conformation, with a variation of 15° in the angles between the two phenyl rings. The orientation of the phenyl rings with respect to each other in the structure of the protonated PPDB is more parallel than in the unprotonated structure. In the neutral compound the phenyl rings are rotated by 64 and 79° from each other, while in the present structure the phenyl rings are nearly coplanar. However, as in the previous structure, the amide groups are severely rotated out of the plane of the phenyl groups.

This twisting can be understood in terms of the steric interaction of the amide hydrogens with the *o*-phenyl hydrogens. This interaction, ~2·2 Å, gives rise to an approximately 30° torsion angle about the phenyl–amide bonds. In the neutral compounds the torsional angles about the plane of the amide groups are +30 and +30° yielding a ~60° ring–ring angle. However, in the protonated structure, the opposite conformer with torsion angles +37, −42° is adopted, yielding the planar structure. In addition to packing considerations, this result is not inconsistent with the increased steric interference of the protonated oxygen with the opposite *ortho*-hydrogen.

The S—O distances range from 1·404 (3) to 1·552 (2) Å and are also consistent with the hydrogen coordination (Table 2). In the crystal, the sulfuric acid and bisulfate groups are arranged in layers (Fig. 2) with an extensive network of hydrogen bonding. These layers alternate with sheets of PPDB consisting of stacks of the molecules, in which the phenyl rings from adjacent molecules align in a *B* (inner) to *C* (outer) to *A* (outer) ring stacking pattern. The perpendicular ring–ring distances are *BC* ~3·5, *CA* ~3·6 and *AB′* ~3·5 Å. The alternating sulfate–PPDB layers are interconnected by strong amide to sulfate hydrogen bonds.

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Isobaimonidine, $C_{27}H_{45}NO_3$: A New Alkaloid from *Fritillaria imperialis* L.

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Abstract. $M_r = 431{\cdot}7$, monoclinic, $P2_1$, $a = 12{\cdot}048$ (2), $b = 7{\cdot}292$ (1), $c = 14{\cdot}408$ (3) Å, $\beta = 101{\cdot}54$ (4)°, $V = 1240{\cdot}2$ Å^3, $Z = 2$, $D_m = 1{\cdot}15$, $D_x = 1{\cdot}16$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, $\mu = 0{\cdot}57$ mm^{-1}, $F(000) = 476$, $T = 293$ K, $R = 0{\cdot}042$ for 1195 unique observed reflections. The cyclohexane rings were found to have normal chair conformations except for ring *D*, which exists in a distorted boat form. There is a considerable amount of flexibility in the cevanine skeleton and this flexibility may be characterized by rotation about the C(14)—C(15) bond.

Introduction. Imperialine, verticine, verticinone and a new alkaloid have been isolated from *Fritillaria imperialis* L. (Mašterová, Kettmann, Majer & Tomko, 1982). On the basis of chemical properties, spectral evidence and preliminary X-ray examination the structure of this new compound was assumed as 3α,6α,20β-trihydroxy-5α-cevanine. In this paper a full description of the structural and stereochemical properties of the alkaloid is given.

Experimental. Crystallization from chloroform/acetone solution, colourless plate-like crystal, 0·40 × 0·30 × 0·05 mm, D_m by flotation in aqueous KI solution; Syntex $P2_1$ diffractometer; unit-cell parameters by least-squares refinement of 18 reflections, $15 < \theta < 35°$; intensity data (h 0 to 11, k 0 to 7, l −14 to 13) collected with Cu $K\alpha$ radiation using θ–2θ scanning mode, each reflection scanned 1° (in 2θ) above and below $K\alpha$ doublet, background to scan time ratio 1·0; two standard reflections measured every 98 reflections, no significant systematic fluctuation; intensities not corrected for absorption; 1406 unique reflections, $3 < \theta < 55°$, 1195 with $I \geq 1{\cdot}96\sigma(I)$ considered observed and included in refinement; structure solved by direct methods using *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978) and refined by Fourier and block-diagonal least-squares methods; difference electron density map showed positions of all H atoms, refinement then continued on all positional parameters, anisotropic thermal parameters for non-H atoms and isotropic thermal parameters for H; in final cycle $R = 0{\cdot}042$, $R_w = 0{\cdot}044$ for all observed reflections, max. Δ/σ 0·25, function minimized $\sum w(\Delta F)^2$, where $w = 1$ if $|F_o| < 35$ and $w = 35/|F_o|$ if $|F_o| \geq 35$; final difference map showed no maxima greater than 0·3 e Å^{-3}; scattering factors for neutral atoms from *International Tables for X-ray Crystallography* (1974); all calculations except *MULTAN* carried out with NRC program package (Ahmed, 1970).

Discussion. Refined positional parameters of non-H atoms and equivalent isotropic *B*'s are listed in Table 1.* Numbering of the atoms is shown in Fig. 1, which also displays configurational and conformational

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and torsion angles of the ring system have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39728 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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