Table 3. *Planarity of pyridine rings*

A plane was constructed by least-squares fitting of coordinates for C2, C3, C5 and C6. θ_1 is the angle between this plane and a vector from the C3/C5 midpoint to C4. θ_2 is the angle between the plane and a vector from the C1/C6 midpoint to N1. θ_{av} is, as defined by Triggle *et al.* (1980), the average of torsion angles C2—C3—C4—C5 and C3—C4—C5—C6. T and F refer to compounds in Triggle *et al.* (1980) and Fossheim *et al.* (1982), respectively.

Compound	θ_1 (°)	θ_2 (°)	IC$_{50}$(n*M*)*	θ_{av}(°)
Nimodipine	19·55	10·75	0·8	22·95
F-II	20·72	12·32	4	24·33
T-I	14·56	5·31	4·4	17·03
T-III	15·55	9·80	5·1	19·98
T-IV	25·90	15·17	35	30·16
F-VI	21·74	12·43	110	25·58
F-I	24·70	13·91	200	28·49
F-III	20·28	10·16	3200	23·72
T-II	23·92	14·56	50000	28·06

*IC$_{50}$ is for the tonic methylfurmethide response in guinea pig ileal longitudinal smooth muscle (Triggle & Janis, 1984) except for T-III, which is for CD in the same system (Triggle *et al.*, 1980). The IC$_{50}$ for either agonist is the same for T-III.

ment of structure/activity relationships. What is required is a comparison of the crystal structure of nimodipine with its structure in a membrane bilayer. The latter might be obtained by carrying out neutron scattering experiments with a series of selectively deuterated analogs of the parent compound.

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Structure of *N*-(2,2-Dinitro-1-methylethenyl)-2-bromo-1,1,2-trimethylpropanamine

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Abstract. $\mathrm{C_9H_{16}BrN_3O_4}$, $M_r = 310{\cdot}1$, triclinic, $P\bar{1}$, $a = 7{\cdot}013\,(1)$, $b = 9{\cdot}768\,(3)$, $c = 9{\cdot}969\,(2)$ Å, $\alpha = 73{\cdot}43\,(2)$, $\beta = 76{\cdot}79\,(2)$, $\gamma = 76{\cdot}07\,(2)°$, $V = 625{\cdot}8\,(3)$ Å^3, $Z = 2$, $D_x = 1{\cdot}646$ g cm^{-3}, λ(Mo $K\alpha$) $= 0{\cdot}71073$ Å, $\mu = 32{\cdot}5$ cm^{-1}, $F(000) = 316$, $T = 100\,(2)$ K, $R = 0{\cdot}040$ for 3038 unique observed reflections. An intramolecular N—H···O hydrogen bond [2·00 (3) Å] is formed with an oxygen of one of the nitro groups resulting in a relatively planar conformation of the molecule. A long C—C double bond and short adjacent C—N bonds indicate delocalization of double-bond character in the molecular plane. The second nitro group is twisted by 58·5 (3)° with respect to the molecular plane.

Introduction. Reaction of dibromodinitromethane with tetramethylethylene in acetonitrile yielded a complex mixture from which an unknown yellow solid was isolated as the major product (Boyer, Manimaran & Patterson, 1989). Since no one structure could be unequivocally assigned using conventional spectroscopic methods, the identity of the unknown has been determined by X-ray structure analysis.

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Experimental. Yellow rectangular prism, approximate dimensions 0·40 × 0·20 × 0·40 mm recrystallized from ethyl acetate. Enraf–Nonius CAD-4 diffractometer with graphite-crystal-monochromatized Mo $K\alpha$ radiation. All measurements were made at 100 (2) K with a locally modified Enraf–Nonius nitrogen-flow cooling system. Unit-cell dimensions and lack of systematic absences indicated either $P1$ or $P\overline{1}$ space group. Lattice constants determined by least-squares fit of 25 reflections with $50 \le 2\theta \le 60°$ measured on the diffractometer. Three-dimensional intensity data collected in ω:2θ scan mode to $[(\sin\theta)/\lambda]_{\max} = 0{\cdot}63$ Å^{-1}, total of 3678 independent reflections, 3038 observed with $I > 3\sigma$; $-8 \le h \le 8$, $-12 \le k \le 12$, $0 \le l \le 12$. Data corrected for Lorentz and polarization effects. Three standard reflections (512, 004 and 240) measured every 2 h during data collection showed no significant change in intensity. Empirical absorption correction based on ψ scans of the 285 and $\overline{2}\overline{6}\overline{4}$ reflections (transmission: max. 99·7, min. 89·9%). Structure solved by direct methods using the *MULTAN*11/82 series of programs (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982). Initial E map in space group $P\overline{1}$ contained 17 peaks corresponding to all non-H atoms. All H-atom positions were located on a subsequent difference Fourier map. Full-matrix least-squares refinement on F with weights $w = 4F_o^2[\sigma^2(I) + (0{\cdot}05F_o^2)^2]^{-1}$, anisotropic thermal parameters for non-H atoms, and isotropic thermal parameters for H atoms gave final $R = 0{\cdot}040$, $wR = 0{\cdot}051$ and $S = 1{\cdot}60$ for 3038 observations and 218 variables. In the final least-squares cycle, $(\Delta/\sigma)_{\max} = 0{\cdot}06$. Maximum and minimum peaks in difference Fourier map were 0·86 and $-1{\cdot}07$ e Å^{-3}, respectively, both located within 0·9 Å of the Br-atom position. Scattering factors and anomalous-scattering contributions from *International Tables for X-ray Crystallography* (1974). Enraf–Nonius *SDP* programs used (Frenz, 1978).

Discussion. Final fractional coordinates for the non-hydrogen atoms are given in Table 1.* The numbering system used for the molecule may be found in Fig. 1. Bond distances, angles and torsion angles are listed in Table 2. A plot of the unit-cell contents is given in Fig. 2.

Table 1. *Table of positional parameters and their estimated standard deviations*

$B_{eq} = (4/3)[a^2\beta_{1,1} + b^2\beta_{2,2} + c^2\beta_{3,3} + 2ab(\cos\gamma)\beta_{1,2} + 2ac(\cos\beta)\beta_{1,3} + 2bc(\cos\alpha)\beta_{2,3}]$.

	x	y	z	B_{eq}(Å^2)
Br1	0·63241 (4)	0·21465 (3)	0·99389 (3)	1·861 (5)
O1	0·7095 (3)	−0·0373 (2)	0·7885 (2)	1·83 (4)
O2	0·7966 (3)	−0·2063 (2)	0·6716 (2)	2·25 (4)
O3	1·1945 (4)	−0·3264 (2)	0·6934 (3)	2·85 (5)
O4	1·2694 (3)	−0·1597 (2)	0·5037 (2)	2·59 (5)
N1	0·8387 (3)	−0·1139 (2)	0·7178 (2)	1·57 (4)
N2	1·1749 (3)	−0·2005 (2)	0·6232 (2)	1·83 (4)
N3	0·9713 (3)	0·1241 (2)	0·7660 (2)	1·31 (4)
C1	1·0336 (4)	−0·0908 (3)	0·6870 (3)	1·45 (4)
C2	1·0980 (4)	0·0263 (3)	0·7063 (2)	1·29 (4)
C3	1·3148 (4)	0·0346 (3)	0·6572 (3)	1·67 (5)
C4	1·0029 (4)	0·2568 (3)	0·7962 (3)	1·36 (4)
C5	0·7903 (4)	0·3468 (3)	0·8388 (3)	1·55 (5)
C6	0·7973 (5)	0·4685 (3)	0·9023 (3)	2·24 (6)
C7	0·6705 (5)	0·4030 (3)	0·7178 (3)	2·38 (6)
C8	1·1090 (4)	0·3504 (3)	0·6616 (3)	2·00 (5)
C9	1·1217 (4)	0·2139 (3)	0·9175 (3)	1·90 (5)

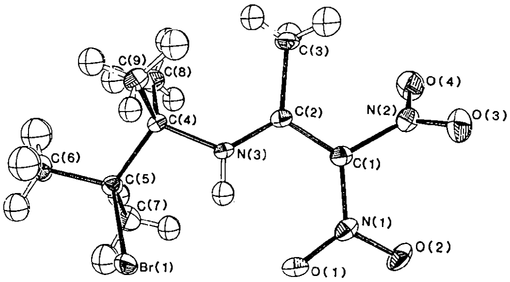

Fig. 1. *ORTEP* (Johnson, 1976) plot of the title compound showing the atomic numbering scheme. Thermal ellipsoids are drawn at the 50% probability level.

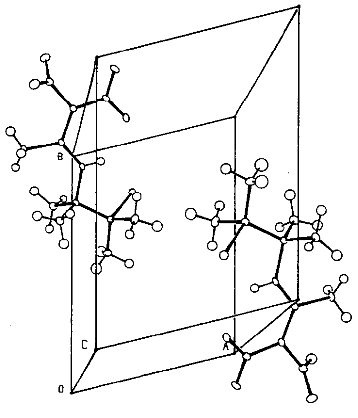

Fig. 2. Cell packing diagram.

The structure reveals that the title compound was formed from the starting materials with the incorporation of an acetonitrile solvent molecule. A formal double bond is located between C(1) and C(2), although the relatively long C(1)—C(2) bond distance [1·401 (3) Å] and short C(2)—N(3) and C(1)—N(1) distances [1·316 (3) and 1·389 (3) Å respectively] suggest considerable delocalization of the double bond. The C(1)—N(1) bond distance may be compared to the C(1)—N(2) bond distance of

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* Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and bond distances and angles involving H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51961 (42 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Table of bond distances* (Å), *bond angles* (°) *and torsion angles* (°)

Numbers in parentheses are estimated standard deviations in the least significant digits.

Br1—C5	2·012 (2)	N3—C4	1·487 (2)
O1—N1	1·249 (2)	C1—C2	1·401 (3)
O2—N1	1·242 (2)	C2—C3	1·501 (3)
O3—N2	1·222 (3)	C4—C5	1·571 (3)
O4—N2	1·230 (3)	C4—C8	1·537 (3)
N1—C1	1·389 (3)	C4—C9	1·528 (3)
N2—C1	1·457 (3)	C5—C6	1·513 (3)
N3—C2	1·316 (3)	C5—C7	1·525 (3)

O1—N1—O2	121·9 (2)	N3—C4—C5	106·7 (2)
O1—N1—C1	118·4 (2)	N3—C4—C8	110·3 (2)
O2—N1—C1	119·6 (2)	N3—C4—C9	109·9 (2)
O3—N2—O4	124·4 (2)	C5—C4—C8	107·9 (2)
O3—N2—C1	117·7 (2)	C5—C4—C9	110·7 (2)
O4—N2—C1	117·9 (2)	C8—C4—C9	111·2 (2)
C2—N3—C4	130·3 (2)	Br1—C5—C4	108·4 (1)
N1—C1—N2	113·1 (2)	Br1—C5—C6	105·1 (2)
N1—C1—C2	126·5 (2)	Br1—C5—C7	105·8 (2)
N2—C1—C2	120·4 (2)	C4—C5—C6	112·7 (2)
N3—C2—C1	120·5 (2)	C4—C5—C7	113·1 (2)
N3—C2—C3	122·0 (2)	C6—C5—C7	111·1 (2)
C1—C2—C3	117·6 (2)		

O1—N1—C1—N2	171·3 (4)	N1—C1—C2—N3	4·0 (3)
O1—N1—C1—C2	−11·4 (4)	N1—C1—C2—C3	−176·3 (3)
O2—N1—C1—N2	−10·7 (3)	N2—C1—C2—N3	−178·9 (3)
O2—N1—C1—C2	166·6 (4)	N2—C1—C2—C3	0·8 (4)
O3—N2—C1—N1	−62·0 (3)	N3—C4—C5—Br1	52·0 (2)
O3—N2—C1—C2	120·6 (3)	N3—C4—C5—C6	167·9 (3)
O4—N2—C1—N1	119·5 (4)	N3—C4—C5—C7	−65·0 (4)
O4—N2—C1—C2	−57·9 (4)	C8—C4—C5—Br1	170·5 (2)
C4—N3—C2—C1	−179·3 (3)	C8—C4—C5—C6	−73·6 (3)
C4—N3—C2—C3	1·0 (3)	C8—C4—C5—C7	53·5 (3)
C2—N3—C4—C5	168·4 (2)	C9—C4—C5—Br1	−67·5 (2)
C2—N3—C4—C8	51·4 (3)	C9—C4—C5—C6	48·4 (3)
C2—N3—C4—C9	−71·6 (2)	C9—C4—C5—C7	175·5 (3)

1·457 (3) Å and to an average value of 1·462 (33) Å for 2236 bond distances between sp^2-hybridized C atoms and nitro-group N atoms extracted from the 1986 release of the Cambridge Structural Database (Allen *et al.*, 1979). Two structures containing the 1,1-dinitroethylene fragment have been solved recently (Gilardi, 1988), both also showing evidence of reduced double-bond character in the C—C bonds.

The molecular structure contains an intramolecular hydrogen bond between H(16) and O(1) with an H(16)···O(1) distance of 2·00 (3) Å and N(3)—H(16)···O(1) angle of 131·9 (9)°. As a result, the O(1)—N(1)—O(2) nitro group is coplanar with an approximate plane defined by the backbone atoms, C(1)—C(2)—N(3)—C(4)—C(5)—C(6).

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Topochemical Studies. XI.* Structures of α-Acetylaminocinnamic Acid Dihydrate, its Photodimer and the Tetrahydrate of the Photodimer

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Abstract. α-Acetylaminocinnamic acid dihydrate (AC2), $C_{11}H_{11}NO_3.2H_2O$, $M_r = 241{\cdot}24$, monoclinic, $P2_1/a$, $a = 18{\cdot}286$ (2), $b = 6{\cdot}0823$ (3), $c = 11{\cdot}415$ (1) Å, $\beta = 105{\cdot}946$ (8)°, $V = 1220{\cdot}7$ (2) Å^3, $Z = 4$, $D_m = 1{\cdot}32$ (1), $D_x = 1{\cdot}312$ Mg m^{-3}, $\mu = 0{\cdot}89$ mm^{-1}, $F(000) = 512$, $R = 0{\cdot}047$ for 1936 unique reflections; photodimer, 1,*c*-3-diacetylamino-*t*-2,*c*-4-diphenylcyclobutane-*r*-1,3-dicarboxylic acid tetrahydrate (ACD4), $C_{22}H_{22}N_2O_6.4H_2O$, $M_r = 482{\cdot}48$, monoclinic, $P2_1/a$, $a = 19{\cdot}153$ (2), $b = 6{\cdot}0987$ (4), $c = 10{\cdot}2964$ (8) Å, $\beta = 106{\cdot}424$ (7)°, $V = 1153{\cdot}6$ (2) Å^3, Z

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* Part X: Kashino, Oka & Haisa (1989).

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