Table 2. *Selected interatomic distances* (Å) *and bond angles* (°)

C(1)—O(1)	1·195 (6)	C(1)—O(1′)	1·327 (6)
O(1′)—C(1′)	1·443 (7)	C(1)—C(2)	1·466 (7)
C(2)—Br(2)	1·882 (5)	C(2)—C(3)	1·328 (7)
C(3)—N(3)	1·372 (7)	N(3)—C(4)	1·377 (7)
C(4)—O(4)	1·214 (5)	C(4)—C(41)	1·499 (7)

C(1)—O(1′)—C(1′)	117·7 (5)	O(1)—C(1)—O(1′)	123·1 (5)
O(1)—C(1)—C(2)	125·4 (5)	O(1′)—C(1)—C(2)	111·5 (5)
C(1)—C(2)—Br(2)	116·3 (4)	C(1)—C(2)—C(3)	123·2 (5)
Br(2)—C(2)—C(3)	120·4 (4)	C(2)—C(3)—N(3)	125·6 (5)
C(3)—N(3)—C(4)	123·0 (4)	N(3)—C(4)—O(4)	121·0 (5)
N(3)—C(4)—C(41)	116·2 (4)	O(4)—C(4)—C(41)	122·7 (5)

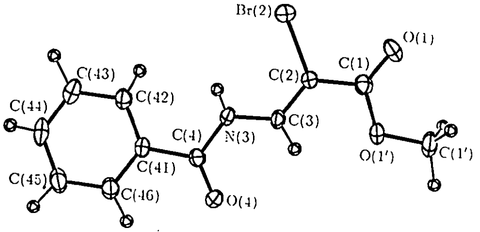

Fig. 1. Molecular structure and numbering scheme for $C_{11}H_{10}BrNO_3$. H atoms are labelled according to the atom to which they are bonded (Johnson, 1971).

thermal parameters for non-H atoms and isotropic thermal parameters for H atoms which were located from a difference map except for methyl H atoms which were included at their calculated positions (C—H 0·97 Å) and refined with a common isotropic thermal parameter. At convergence $R = 0{\cdot}033$, $wR = 0{\cdot}033$, $w = 1{\cdot}89/[\sigma^2(F) + 0{\cdot}0003F^2]$, $S = 1{\cdot}97$, $(\Delta/\sigma)_{\max} \le 0{\cdot}001$, $\Delta\rho_{\max} = 0{\cdot}41$, $\Delta\rho_{\min} = -0{\cdot}46$ e Å^{-3}; no extinction correction. Scattering factors for H, C, N and O given in *SHELX76* (Sheldrick, 1976) and those for neutral Br corrected for f' and f'' from *International Tables for X-ray Crystallography* (1974). All calculations on a SUN4/280 computer system. Atomic parameters are given in Table 1, selected parameters in Table 2* and the numbering scheme used is shown in Fig. 1, drawn with *ORTEP* (Johnson, 1971) at 25% probability ellipsoids.

Related literature. Owing to similarities in the spectroscopic data for isomers *A* and *B* it was necessary to characterize one of these by X-ray methods to determine the stereochemistry as *E* or *Z*. The structure determination of the title compound forms part of a wider study of the synthesis of halogenated amino acid derivatives (Burgess, Easton & Hay, 1989).

The Australian Research Council is thanked for support.

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, all bond distances and angles, and mean-plane data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52564 (9 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structures of 2,5,7,9-Tetranitro-8-oxo-2,5,7,9-tetraazabicyclo[4.3.0]nonane (I) and 2,5,7,9-Tetranitro-8-acetoxy-2,5,7,9-tetraazabicyclo[4.3.0]nonane (II)

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(*Received* 20 *June* 1989; *accepted* 8 *December* 1989)

Abstract. (I): Perhydro-1,3,5,7-tetranitroimidazo[4,5-*b*]pyrazin-2-one, $C_5H_6N_8O_9$, $M_r = 322{\cdot}15$, trigonal, $P3_1$ (arbitrarily chosen over its enantiomorphic counterpart $P3_2$), $a = 10{\cdot}923$ (4), $c = 7{\cdot}888$ (4) Å, $V = 815{\cdot}7$ (2) Å^3, $Z = 3$, $D_x = 1{\cdot}969$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 0{\cdot}18$ mm^{-1}, $F(000) = 492$, $T = 295$ K, final $R = 0{\cdot}056$, $wR = 0{\cdot}054$ for 895 observed reflections. (II): Perhydro-1,3,5,7-tetranitroimidazo[4,5-*b*]pyrazin-2-yl acetate, $C_7H_{10}N_8O_{10}$, $M_r = 366{\cdot}21$, monoclinic, $P2_1/a$, $a = 11{\cdot}206$ (2), $b =$

0108-2701/90/061122-04$03.00

Table 1. *Atomic coordinates* (× 10^4) *and equivalent isotropic displacement coefficients* ($Å^2 \times 10^3$) *for* (I)

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

	x	*y*	*z*	U_{eq}
C(1)	1876 (7)	− 509 (7)	4525 (10)	29 (3)
N(2)	2617 (6)	544 (6)	3258 (10)	28 (2)
C(3)	4156 (7)	1282 (9)	3052 (11)	39 (3)
C(4)	4786 (8)	908 (7)	4527 (12)	35 (3)
N(5)	4117 (6)	933 (6)	6105 (9)	31 (2)
C(6)	2636 (7)	− 116 (7)	6247 (10)	28 (3)
N(7)	2456 (6)	− 1481 (6)	6794 (10)	34 (3)
C(8)	1918 (7)	− 2545 (7)	5605 (11)	33 (3)
N(9)	1820 (6)	− 1857 (6)	4112 (10)	32 (2)
N(10)	1890 (7)	1061 (6)	2346 (9)	31 (3)
O(11)	620 (5)	562 (5)	2637	37 (2)
O(12)	2571 (6)	1977 (6)	1336 (9)	47 (3)
N(13)	4581 (7)	2223 (7)	6918 (10)	39 (3)
O(14)	5712 (6)	3181 (6)	6410 (10)	54 (3)
O(15)	3862 (6)	2274 (6)	8051 (9)	52 (3)
N(16)	2447 (6)	− 1736 (8)	8555 (10)	41 (3)
O(17)	2772 (6)	− 722 (7)	9461 (9)	48 (3)
O(18)	2133 (7)	− 2921 (6)	8980 (10)	60 (3)
O(19)	1612 (6)	− 3744 (5)	5748 (9)	48 (3)
N(20)	983 (6)	− 2666 (6)	2793 (10)	33 (3)
O(21)	940 (7)	− 3768 (6)	2461 (10)	54 (3)
O(22)	366 (5)	− 2172 (5)	1999 (9)	43 (2)

Table 2. *Bond lengths* (Å) *and angles* (°) *for* (I)

C(1)—N(2)	1·430 (9)	C(1)—C(6)	1·537 (11)
C(1)—N(9)	1·479 (11)	N(2)—C(3)	1·465 (9)
N(2)—N(10)	1·384 (11)	C(3)—C(4)	1·509 (13)
C(4)—N(5)	1·450 (12)	N(5)—C(6)	1·445 (8)
N(5)—N(13)	1·393 (10)	C(6)—N(7)	1·468 (11)
N(7)—C(8)	1·376 (10)	N(7)—N(16)	1·416 (11)
C(8)—N(9)	1·430 (12)	C(8)—O(19)	1·184 (10)
N(9)—N(20)	1·375 (9)	N(10)—O(11)	1·232 (8)
N(10)—O(12)	1·202 (8)	N(13)—O(14)	1·220 (8)
N(13)—O(15)	1·209 (11)	N(16)—O(17)	1·213 (11)
N(16)—O(18)	1·209 (11)	N(20)—O(21)	1·210 (10)
N(20)—O(22)	1·225 (11)		

N(2)—C(1)—C(6)	112·5 (5)	N(2)—C(1)—N(9)	111·8 (7)
C(6)—C(1)—N(9)	102·7 (7)	C(1)—N(2)—C(3)	123·6 (7)
C(1)—N(2)—N(10)	118·8 (6)	C(3)—N(2)—N(10)	116·9 (6)
N(2)—C(3)—C(4)	108·6 (6)	C(3)—C(4)—N(5)	110·7 (8)
C(4)—N(5)—C(6)	115·2 (6)	C(4)—N(5)—N(13)	118·9 (5)
C(6)—N(5)—N(13)	117·6 (7)	C(12)—C(6)—N(5)	112·8 (6)
C(1)—C(6)—C(7)	101·3 (6)	N(5)—C(6)—N(7)	110·9 (7)
C(6)—N(7)—C(8)	116·2 (7)	C(6)—N(7)—N(16)	118·2 (7)
C(8)—N(7)—N(16)	123·0 (7)	N(7)—C(8)—N(9)	103·3 (7)
N(7)—C(8)—O(19)	129·4 (8)	N(9)—C(8)—O(19)	127·3 (8)
C(1)—N(9)—C(8)	111·4 (7)	C(1)—N(9)—N(20)	119·3 (7)
C(8)—N(9)—N(20)	119·0 (5)	N(2)—N(10)—O(11)	118·1 (6)
N(2)—N(10)—O(12)	116·2 (7)	O(11)—N(10)—O(12)	125·7 (8)
N(5)—N(13)—O(14)	114·6 (8)	N(5)—N(13)—O(15)	118·1 (6)
O(14)—N(13)—O(15)	127·3 (7)	N(7)—N(16)—O(17)	115·3 (8)
N(7)—N(16)—O(18)	116·9 (8)	O(17)—N(16)—O(18)	127·7 (9)
N(9)—N(20)—O(21)	119·1 (8)	N(9)—N(20)—O(22)	116·0 (7)
O(21)—N(20)—O(22)	124·9 (7)		

10·757 (2), $c = 23{\cdot}026$ (4) Å, $\beta = 93{\cdot}7$ (1)°, $V = 2769{\cdot}8$ (7) $Å^3$, $Z = 8$ (two molecules per asymmetric unit), $D_x = 1{\cdot}756$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, $\mu = 0{\cdot}14$ mm^{-1}, $F(000) = 1504$, $T = 225$ K, final $R = 0{\cdot}062$, $wR = 0{\cdot}070$ for 1979 observed reflections. Both molecules have essentially the same conformation in that the five-membered ring is a flattened envelope and the six-membered ring is twisted. There is a *cis* junction between the two heterocyclic rings. Each of the rings contains a planar and a pyramidal N atom with the similarly configured N atoms on opposing sides of the ring system. There are several intermolecular N···O and O···O approaches between 2·78–2·95 Å. In (II) there is also a close O···C intermolecular approach of 2·98 Å.

Table 3. *Atomic coordinates* (× 10^4) *and equivalent isotropic displacement coefficients* ($Å^2 \times 10^3$) *for* (II)

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

	x	*y*	*z*	U_{eq}
C(1)	4333 (6)	9446 (6)	3062 (3)	28 (3)
N(2)	5395 (5)	10080 (4)	2878 (2)	21 (2)
C(3)	6284 (6)	10434 (6)	3339 (3)	38 (3)
C(4)	6802 (6)	9291 (6)	3653 (2)	34 (3)
N(5)	5843 (6)	8396 (4)	3707 (2)	30 (2)
C(6)	4609 (8)	8730 (6)	3638 (2)	28 (3)
N(7)	4249 (5)	9620 (4)	4071 (2)	28 (2)
C(8)	3471 (7)	10608 (6)	3838 (3)	31 (3)
N(9)	3430 (5)	10288 (5)	3237 (2)	32 (2)
N(10)	5811 (7)	9752 (6)	2345 (2)	46 (3)
O(11)	6783 (6)	10167 (6)	2236 (2)	69 (3)
O(12)	5134 (5)	9133 (5)	2016 (2)	57 (2)
N(13)	6145 (8)	7218 (5)	3888 (2)	37 (3)
O(14)	7213 (6)	7037 (5)	4006 (2)	51 (2)
O(15)	5353 (5)	6441 (4)	3917 (2)	49 (2)
N(16)	4051 (5)	9200 (6)	4628 (2)	33 (2)
O(17)	3517 (4)	9897 (4)	4933 (2)	42 (2)
O(18)	4511 (4)	8196 (4)	4757 (2)	40 (2)
O(19)	4058 (4)	11752 (4)	3946 (2)	33 (2)
C(20)	3449 (10)	12654 (6)	4250 (3)	45 (4)
C(21)	4278 (7)	13743 (6)	4357 (3)	55 (3)
O(22)	2472 (7)	12525 (5)	4381 (3)	81 (3)
N(23)	2741 (5)	10956 (5)	2836 (2)	35 (2)
O(24)	2030 (4)	11678 (4)	3030 (2)	46 (2)
O(25)	2889 (4)	10735 (4)	2325 (2)	46 (2)
C(1′)	4138 (7)	5700 (7)	1261 (3)	37 (3)
N(2′)	3563 (7)	4510 (5)	1199 (3)	56 (3)
C(3′)	4122 (9)	3340 (7)	1358 (3)	73 (4)
C(4′)	5177 (7)	3578 (6)	1742 (3)	59 (3)
N(5′)	4998 (6)	4611 (5)	2142 (2)	50 (2)
C(6′)	4808 (7)	5799 (6)	1868 (3)	32 (3)
N(7′)	5930 (5)	6356 (5)	1728 (2)	35 (2)
C(8′)	6214 (7)	6326 (6)	1129 (3)	34 (3)
N(9′)	5083 (6)	5913 (5)	870 (2)	40 (2)
N(10′)	2406 (9)	4493 (7)	986 (3)	63 (3)
O(11′)	1948 (6)	3471 (6)	933 (3)	99 (3)
O(12′)	1870 (5)	5467 (7)	886 (2)	74 (3)
N(13′)	4396 (6)	4358 (9)	2638 (3)	66 (3)
O(14′)	4256 (6)	3216 (6)	2741 (2)	87 (3)
O(15′)	4100 (5)	5193 (7)	2917 (2)	83 (3)
N(16′)	6695 (6)	6834 (5)	2144 (2)	37 (3)
O(17′)	7645 (5)	7256 (4)	1987 (2)	49 (2)
O(18′)	6409 (4)	6795 (4)	2645 (2)	48 (2)
O(19′)	7093 (4)	5418 (3)	1040 (2)	33 (2)
C(20′)	8142 (7)	5758 (7)	811 (3)	39 (3)
C(21′)	8850 (7)	4675 (7)	676 (3)	58 (3)
O(22′)	8401 (5)	6813 (5)	746 (2)	70 (2)
N(23′)	4803 (9)	6289 (6)	302 (3)	54 (3)
O(24′)	5600 (6)	6800 (5)	54 (2)	66 (2)
O(25′)	3809 (6)	5990 (5)	102 (2)	66 (2)

Experimental. Values for molecule (II), whenever different, are given in parentheses. Colorless, 0·12 × 0·20 × 0·50 mm (0·35 × 0·12 × 0·04 mm) data crystal synthesized by Clifford Coon of the Lawrence Livermore Laboratory, Livermore, California. Automated Niclolet *R*3*m* diffractometer with incident-beam graphite monochromator; 25 (20) centered reflections within $16 \leq 2\theta \leq 29°$ ($17 \leq 2\theta \leq 70°$) used for determining cell parameters. Data corrected for Lorentz and polarization effects, but not for absorption. $2\theta_{max} = 50°$ (115°); range of *hkl*: $0 \leq h \leq 13$, $-13 \leq k \leq 11$, $-9 \leq l \leq 9$ ($-7 \leq h \leq 0$, $-10 \leq k \leq 0$, $-22 \leq l \leq 22$); standards 400, 040, 003 (200, 020, 0,0,10) monitored every 100 reflections with random

Table 4. *Bond lengths* (Å) *and angles* (°) *for* (II)

C(1)—N(2)	1·458 (11)	C(1)—C(6)	1·548 (11)
C(1)—N(9)	1·435 (11)	N(2)—C(3)	1·457 (11)
N(2)—N(10)	1·387 (10)	C(3)—C(4)	1·522 (12)
C(4)—N(5)	1·454 (11)	N(5)—C(6)	1·427 (13)
N(5)—N(13)	1·370 (10)	C(6)—N(7)	1·458 (11)
N(7)—C(8)	1·455 (11)	N(7)—N(16)	1·392 (10)
C(8)—N(9)	1·424 (10)	C(8)—O(19)	1·410 (11)
N(9)—N(23)	1·368 (10)	N(10)—O(11)	1·218 (12)
N(10)—O(12)	1·232 (10)	N(13)—O(14)	1·225 (12)
N(13)—O(15)	1·225 (11)	N(16)—O(17)	1·212 (9)
N(16)—O(18)	1·224 (10)	O(19)—C(20)	1·400 (11)
C(20)—C(21)	1·506 (13)	C(20)—O(22)	1·162 (14)
N(23)—O(24)	1·218 (10)	N(23)—O(25)	1·223 (9)
C(1′)—N(2′)	1·436 (12)	C(1′)—C(6′)	1·546 (12)
C(1′)—N(9′)	1·453 (12)	N(2′)—C(3′)	1·442 (12)
N(2′)—N(10′)	1·357 (14)	C(3′)—C(4′)	1·452 (14)
C(4′)—N(5′)	1·466 (12)	N(5′)—C(6′)	1·435 (11)
N(5′)—N(13′)	1·390 (11)	C(6′)—N(7′)	1·448 (12)
N(7′)—C(8′)	1·434 (11)	N(7′)—N(16′)	1·346 (10)
C(8′)—N(9′)	1·436 (12)	C(8′)—O(19′)	1·412 (11)
N(9′)—N(23′)	1·386 (10)	N(10′)—O(11′)	1·217 (12)
N(10′)—O(12′)	1·222 (12)	N(13′)—O(14′)	1·263 (13)
N(13′)—O(15′)	1·165 (12)	N(16′)—O(17′)	1·233 (11)
N(16′)—O(18′)	1·217 (9)	O(19′)—C(20′)	1·370 (12)
C(20′)—C(21′)	1·454 (13)	C(20′)—O(22′)	1·183 (11)
N(23′)—O(24′)	1·221 (12)	N(23′)—O(25′)	1·220 (13)

N(2)—C(1)—C(6)	111·0 (6)	N(2)—C(1)—N(9)	113·0 (6)
C(6)—C(1)—N(9)	100·3 (6)	C(1)—N(2)—C(3)	116·4 (5)
C(1)—N(2)—N(10)	117·7 (6)	C(3)—N(2)—N(10)	117·3 (6)
N(2)—C(3)—C(4)	110·9 (6)	C(3)—C(4)—N(5)	108·3 (6)
C(4)—N(5)—C(6)	122·7 (6)	C(4)—N(5)—N(13)	118·0 (7)
C(6)—N(5)—N(13)	119·0 (7)	C(1)—C(6)—N(5)	111·1 (6)
C(1)—C(6)—N(7)	102·1 (6)	N(5)—C(6)—N(7)	113·5 (6)
C(6)—N(7)—C(8)	114·4 (5)	C(6)—N(7)—N(16)	119·0 (5)
C(8)—N(7)—N(16)	116·7 (6)	N(7)—C(8)—N(9)	99·3 (6)
N(7)—C(8)—O(19)	108·2 (6)	N(9)—C(8)—O(19)	111·5 (6)
C(1)—N(9)—C(8)	116·6 (6)	C(1)—N(9)—N(23)	121·2 (5)
C(8)—N(9)—N(23)	120·6 (6)	N(2)—N(10)—O(11)	116·4 (6)
N(2)—N(10)—O(12)	117·0 (7)	O(11)—N(10)—O(12)	126·5 (7)
N(5)—N(13)—O(14)	115·6 (7)	N(5)—N(13)—O(15)	118·9 (8)
O(14)—N(13)—O(15)	125·5 (7)	N(7)—N(16)—O(17)	116·5 (6)
N(7)—N(16)—O(18)	114·8 (6)	O(17)—N(16)—O(18)	128·5 (6)
C(8)—O(19)—C(20)	117·1 (6)	O(19)—C(20)—C(21)	107·8 (8)
O(19)—C(20)—O(22)	123·1 (7)	C(21)—C(20)—O(22)	129·2 (8)
N(9)—N(23)—O(24)	116·3 (6)	N(9)—N(23)—O(25)	116·2 (6)
O(24)—N(23)—O(25)	127·5 (6)	N(2′)—C(1′)—C(6′)	109·8 (6)
N(2′)—C(1′)—N(9′)	114·7 (6)	C(6′)—C(1′)—N(9′)	102·6 (6)
C(1′)—N(2′)—C(3′)	124·6 (8)	C(1′)—N(2′)—N(10′)	117·3 (7)
C(3′)—N(2′)—N(10′)	118·0 (7)	N(2′)—C(3′)—C(4′)	108·9 (7)
C(3′)—C(4′)—N(5′)	112·2 (7)	C(4′)—N(5′)—C(6′)	114·9 (6)
C(4′)—N(5′)—N(13′)	117·5 (7)	C(6′)—N(5′)—N(13′)	118·0 (7)
C(1′)—C(6′)—N(5′)	112·7 (6)	C(1′)—C(6′)—N(7′)	101·8 (6)
N(5′)—C(6′)—N(7′)	111·1 (6)	C(6′)—N(7′)—C(8′)	117·0 (6)
C(6′)—N(7′)—N(16′)	121·4 (6)	C(8′)—N(7′)—N(16′)	121·5 (7)
N(7′)—C(8′)—N(9′)	99·3 (6)	N(7′)—C(8′)—O(19′)	110·8 (6)
N(9′)—C(8′)—O(19′)	109·3 (6)	C(1′)—N(9′)—C(8′)	116·7 (6)
C(1′)—N(9′)—N(23′)	120·1 (7)	C(8′)—N(9′)—N(23′)	116·3 (7)
N(2′)—N(10′)—O(11′)	115·8 (8)	N(2′)—N(10′)—O(12′)	120·3 (8)
O(11′)—N(10′)—O(12′)	123·9 (9)	N(5′)—N(13′)—O(14′)	114·7 (8)
N(5′)—N(13′)—O(15′)	118·2 (9)	O(14′)—N(13′)—O(15′)	127·1 (8)
N(7′)—N(16′)—O(17′)	116·9 (6)	N(7′)—N(16′)—O(18′)	117·9 (7)
O(17′)—N(16′)—O(18′)	125·2 (6)	C(8′)—O(19′)—C(20′)	119·7 (6)
O(19′)—C(20′)—C(21′)	111·2 (7)	O(19′)—C(20′)—O(22′)	121·9 (7)
C(21′)—C(20′)—O(22′)	126·9 (8)	N(9′)—N(23′)—O(24′)	116·4 (8)
N(9′)—N(23′)—O(25′)	115·1 (7)	O(24′)—N(23′)—O(25′)	128·3 (7)

variation of 2·0% over data collection, θ–2θ mode, scan width $[2\theta(K\alpha_1) - 1{\cdot}0]$ to $[2\theta(K\alpha_2) + 1{\cdot}0]^\circ$, scan rate a function of count rate (8° min^{-1} minimum, 30° min^{-1} maximum); 3210 (3433) reflections measured, 962 (2436) unique, $R_{\text{int}} = 0{\cdot}049$ (0·032), 895 (1979) observed [$F_o > 3\sigma(F_o)$].

Structures solved by direct methods. The least-squares refinement used the full-matrix program provided with the MicroVax version of the *SHELXTL* system (Sheldrick, 1980). $\sum w(|F_o| - |F_c|)^2$ minimized where $w = 1/[\sigma^2(|F_o|) + g(F_o)^2]$, $g = 0{\cdot}00023$ (0·0004), secondary isotropic extinction from $F_c^* = F_c/[1{\cdot}0 + 0{\cdot}002(p)F_o^2/\sin 2\theta]^{0{\cdot}25}$ where $p = 0{\cdot}004$ (1) [no extinction correction for (II)]. There were 199 (459) parameters refined: atomic coordinates for all atoms, anisotropic temperature factors for non-H atoms, H atoms were idealized, C—H = 0·96 Å, C—H—C = 109·5°, then allowed to ride (coordinate shift of attached C atom applied to H) on covalently bonded neighbors, $(\Delta/\sigma)_{\max} = 0{\cdot}01$ (0·3), $R = 0{\cdot}056$ (0·062), $wR = 0{\cdot}054$ (0·070), $S = 2{\cdot}17$ (2·41). Final difference Fourier excursions 0·33 (0·69) and −0·37 (−0·46) e Å^{-3}. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974).* The atomic numbering schemes for Tables 1–4 follow those shown in Figs. 1 and 2.

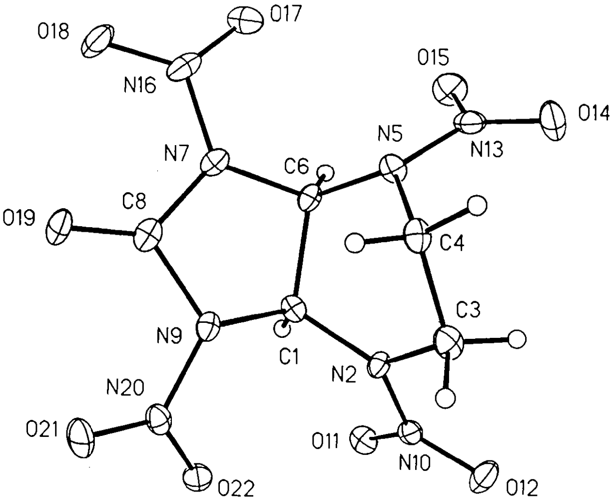

Fig. 1. Perspective drawing of (I). Thermal ellipsoids are drawn at the 20% probability level.

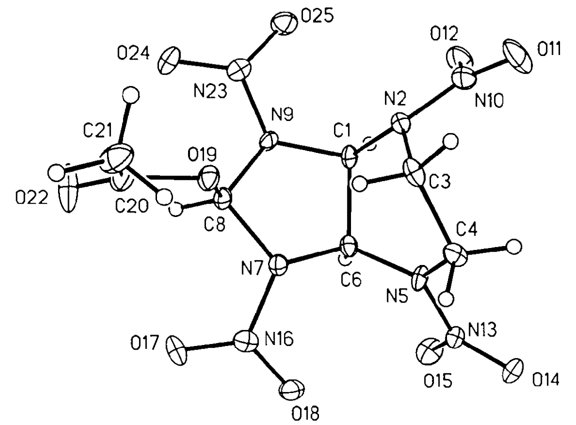

Fig. 2. Perspective drawing of (II). Thermal ellipsoids are drawn at the 20% probability level.

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* 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 52524 (16 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Related literature. A search of the most recent Cambridge Structural Database file did not produce any similar structures.

This work was supported in part by contract # N00173-86-M-R828 from the ONR Mechanics Division.

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Acta Cryst. (1990). C**46**, 1125–1127

Structure of (*S*,*S*)-3,6-Diphenyl-1,4-dioxane-2,5-dione

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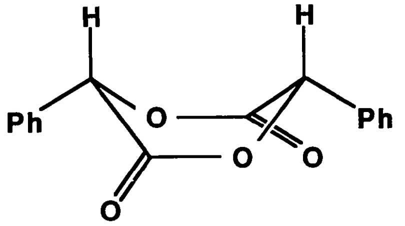
(*Received* 24 *October* 1989; *accepted* 4 *January* 1990)

Abstract. $C_{16}H_{12}O_4$, $M_r = 268{\cdot}27$, orthorhombic, $P2_12_12_1$, $a = 5{\cdot}103\,(3)$, $b = 9{\cdot}272\,(6)$, $c = 26{\cdot}50\,(3)$ Å, $V = 1254\,(2)$ Å^3, $Z = 4$, $D_x = 1{\cdot}42$ g cm^{-3}, λ(Mo $K\alpha$) $= 0{\cdot}7107$ Å, $\mu = 0{\cdot}9574$ cm^{-1}, $F(000) = 560$, $T = 198$ K, $R = 0{\cdot}0542$ for 886 reflections $[F_o \geq 4\sigma(F_o)]$. The absolute configuration is assigned on the basis of internal comparison to the (+)-mandelate moiety. Close C—H···O contacts are observed resulting in a two-dimensional network of H-bonded molecules lying parallel to *ab*. Atom H3 is involved in a three-center C—H···O-type hydrogen bond. The shorter H···O contact involves C3—H3···O8 ($1 - x$, $\frac{1}{2} + y$, $\frac{3}{2} - z$) with H3···O8 2·26 (6) Å, C3···O8 2·949 (7) Å and C—H···O 127 (4)°, while the longer contact involves C3—H3···O7 ($x - 1$, y, z) with H3···O7 2·41 (6) Å, C3···O7 3·143 (8) Å and C—H···O 132 (4)°.

Experimental. (1) was prepared by *p*-toluenesulfonic acid catalyzed self-condensation of *S*-(+)-mandelic acid in benzene at reflux with azeotropic removal of water (Pojman, 1988). After recrystallization from tetrahydrofuran, the material was sublimed *in vacuo* providing single crystals suitable for X-ray analysis. The data crystal was a colorless needle of approximate dimensions 0·03 × 0·13 × 0·54 mm. The data were collected on a Nicolet *R*3 diffractometer using a graphite monochromator and equipped with a Nicolet LT-2 low-temperature assembly. Lattice parameters were obtained from the least-squares refinement of 36 reflections with $12{\cdot}7 < 2\theta < 21{\cdot}3°$. The data were collected using the ω-scan technique (2444 reflections, of which 1323 were unique, $R_{\rm int} = 0{\cdot}0412$ from averaging *hkl* and $h\bar{k}\bar{l}$ reflections), with a 2θ range from 4·0–50·0°, and a 1·0° ω scan at a scan rate of 1–4° min^{-1} ($h = 0 \rightarrow 6$, $k = -11 \rightarrow 11$, $l = -31 \rightarrow 31$). Four reflections ($00\bar{6}$; $\bar{1}\bar{1}0$; $1\bar{2}3$; 021) were remeasured every 96 reflections to monitor instrument and crystal stability (maximum correction on I was < 4%). The data were also corrected for Lp effects but not for absorption. The decay correction and the data reduction were performed using *SHELXTL-PLUS* (Sheldrick, 1987). Reflections having $F_o < 4\sigma(F_o)$ were considered unobserved (437 reflections). The structure was solved by direct methods (Sheldrick, 1987) and refined by full-matrix least squares (Sheldrick, 1976). The non-H atoms were refined with anisotropic thermal parameters. All H-atom positions were obtained from a ΔF map and refined with isotropic thermal parameters. After several cycles of refinement, the $U_{\rm iso}$ for H3 refined to a negative value and, therefore, was fixed at 1·2 × $U_{\rm eq}$ of C3 for the final refinement cycles. In all, 228 parameters were refined. The function $\sum w(|F_o| - |F_c|)^2$ was minimized, where $w = 1/[\sigma(F_o)]^2$ and $\sigma(F_o) = 0{\cdot}5kI^{-1/2}\{[\sigma(I)]^2 + (0{\cdot}02I)^2\}^{1/2}$. The intensity I is given by $(I_{\rm peak} - I_{\rm background}) \times$ (scan rate); 0·02 is a factor to downweight intense reflections and to account for instrument instability and k is the correction due to Lp effects and decay. $\sigma(I)$ was estimated from counting statistics; $\sigma(I) = [(I_{\rm peak} + I_{\rm background})^{1/2} \times$ (scan rate)]. The final $R = 0{\cdot}0542$ for 886 reflections, with $wR = 0{\cdot}0487$ ($R_{\rm all} = 0{\cdot}0919$, $wR_{\rm all} = 0{\cdot}0557$) and goodness of fit = 1·250. The maximum $|\Delta/\sigma| < 0{\cdot}1$ in the final refinement cycle and the



(1)

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