factors for the H atoms were obtained from Stewart, Davidson & Simpson (1965). Values used to calculate the linear absorption coefficient are from *International Tables for X-ray Crystallography* (1974).* Figures were generated using *SHELXTL-PLUS* (Sheldrick, 1987). The positional and thermal parameters for non-H atoms are listed in Table 1, while the bond lengths and angles for the non-H atoms are listed in Table 2. The atom-labelling scheme is shown in Fig. 1.

Related literature. The title compound, 1,2-bis(1-naphthyl)cyclopentene, was prepared for studies on single-crystal-to-single-crystal photochemical transformations that would provide extended, non-aromatic π systems possibly suitable for non-linear optical applications (Chemla & Zyss, 1987). Photocyclization of 1,2-diphenylcyclopentene (Griffith, 1978) and 1,2-bis(2-naphthyl)cyclopentene (Wismontski-Knittel, Kaganowitch, Seger & Fischer, 1979) in solution and 1,2-bis(1-naphthyl)ethylene (Aldoshin, Alfimov, Atovnyan, Kaminsky, Razumov & Rachinsky, 1984) in the crystal have been reported.

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* Tables of crystallographic data and intensity collection/structure refinement information, anisotropic thermal parameters, H-atom positional parameters, bond lengths and angles involving the H atoms, structure-factor amplitudes, and a unit-cell packing diagram have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52303 (20 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of 3,4,5,6,7,8-Hexa-*O*-acetyl-1,2-dideoxy-2-*C*-nitro-D-*threo*-L-*talo*-octitol

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Abstract. $C_{20}H_{29}NO_{14}$, $M_r = 507{\cdot}4$, orthorhombic, $P2_12_12_1$, $a = 12{\cdot}950\,(3)$, $b = 19{\cdot}713\,(6)$, $c = 9{\cdot}942\,(10)$ Å, $V = 2538\,(3)$ Å^3, $Z = 4$, $D_x = 1{\cdot}328$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·7107 Å, $\mu = 0{\cdot}17$ mm^{-1}, $F(000) = 1072$, room temperature, final $wR = 0{\cdot}037$ for 1311 observed reflexions. Bond distances and angles are all within the expected ranges. The crystal structure is stabilized by C—H···O hydrogen bonds along [001] and [100].

Experimental. Single crystals in the form of colourless needles elongated along [001]. Dimensions of the crystal used 0·2 × 0·2 × 0·4 mm. Cell parameters were refined by least-squares fit from 25 reflections with $6 < \theta < 20°$ on an Enraf–Nonius CAD-4 diffractometer. Data collection by ω–2θ scan, up to $(\sin\theta)/\lambda = 0{\cdot}60$ Å^{-1}, using graphite-monochromated Mo $K\alpha$ radiation, 2602 unique reflections measured ($0 \leq h \leq 15$, $0 \leq k \leq 23$, $0 \leq l \leq 11$), 1311 with $I > 2\sigma(I)$ considered observed; three intensity standards ($1\bar{4}\bar{2}$; $\bar{1}42$; $\bar{4}\bar{2}1$) monitored at intervals of 60 reflections showed no significant variations ($< 2\sigma$) during data collection. Intensity data corrected for Lorentz–polarization factors; no absorption or extinction cor-

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Table 1. *Atomic coordinates and equivalent isotropic temperature factors* (Å² × 10³) *with e.s.d.'s in parentheses*

$$U_{eq} = \tfrac{1}{3}\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j \cos(\mathbf{a}_i,\mathbf{a}_j).$$

	x	y	z	U_{eq}
C1	0·6783 (7)	0·4475 (5)	−0·0329 (9)	42 (4)
C2	0·5785 (7)	0·4306 (5)	0·0466 (9)	37 (4)
C3	0·4981 (8)	0·3913 (5)	−0·0386 (9)	37 (4)
C4	0·4028 (7)	0·3746 (4)	0·0415 (9)	29 (3)
C5	0·3143 (7)	0·3444 (5)	−0·0477 (9)	33 (4)
C6	0·2158 (8)	0·3328 (5)	0·0310 (9)	39 (4)
C7	0·1297 (8)	0·2963 (5)	−0·0480 (10)	45 (4)
C11	0·7449 (8)	0·3879 (5)	−0·0736 (10)	55 (4)
C21	0·6079 (8)	0·4117 (6)	0·2828 (11)	43 (4)
C22	0·6244 (8)	0·3537 (5)	0·3809 (8)	48 (4)
C31	0·4936 (9)	0·4155 (6)	−0·2772 (11)	55 (5)
C32	0·4583 (8)	0·4706 (6)	−0·3778 (9)	76 (6)
C41	0·3508 (8)	0·4367 (6)	0·2429 (10)	44 (5)
C42	0·2999 (7)	0·5004 (5)	0·2873 (10)	58 (4)
C51	0·3455 (9)	0·2699 (7)	−0·2349 (12)	69 (6)
C52	0·3973 (8)	0·2067 (7)	−0·2731 (12)	87 (6)
C61	0·1970 (9)	0·3071 (7)	0·2662 (12)	59 (6)
C62	0·2358 (9)	0·2574 (6)	0·3676 (9)	69 (5)
C71	0·0278 (8)	0·3223 (6)	−0·2358 (12)	51 (5)
C72	0·0073 (9)	0·3736 (6)	−0·3454 (11)	83 (6)
N	0·7374 (9)	0·4958 (6)	0·0604 (10)	62 (5)
O11	0·8063 (7)	0·4706 (5)	0·1274 (8)	90 (4)
O12	0·7100 (7)	0·5553 (4)	0·0589 (9)	94 (4)
O21	0·6030 (5)	0·3832 (3)	0·1537 (6)	35 (2)
O22	0·5944 (6)	0·4696 (4)	0·3061 (7)	56 (3)
O31	0·4695 (5)	0·4386 (3)	−0·1470 (6)	43 (3)
O32	0·5390 (6)	0·3640 (5)	−0·2975 (7)	73 (4)
O41	0·3590 (5)	0·4355 (3)	0·1020 (6)	37 (3)
O42	0·3830 (5)	0·3920 (4)	0·3092 (6)	51 (3)
O51	0·3568 (5)	0·2828 (3)	−0·0998 (6)	42 (3)
O52	0·2978 (7)	0·3081 (5)	−0·3060 (7)	100 (5)
O61	0·2414 (5)	0·2882 (3)	0·1436 (6)	44 (3)
O62	0·1396 (6)	0·3527 (5)	0·2787 (8)	83 (4)
O71	0·0992 (5)	0·3452 (3)	−0·1499 (7)	55 (3)
O72	−0·0127 (6)	0·2689 (4)	−0·2253 (9)	96 (4)

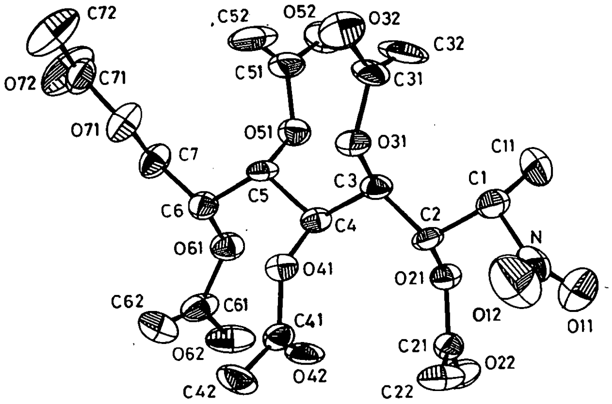

Fig. 1. An *ORTEP* view of the molecule with the atomic labelling scheme.

rections. Structure solved by direct methods with *MULTAN*80 (Main *et al.*, 1980); *E* map gave positions of the non-H atoms. H atoms located from difference synthesis and fixed at geometrically calculated positions. Refinement by full-matrix least squares with anisotropic thermal factors for all the non-H atoms. Final agreement factors $R = 0{\cdot}051$, $wR = 0{\cdot}037$; the function minimized was $\sum w||F_o| - |F_c||^2$ with $w = 1/\sigma^2(F)$; $S = 0{\cdot}82$. Max. shift/e.s.d. = 0·007. Final difference map showed residual electron density within −0·22 and +0·23 e Å⁻³. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). Calculations carried out on a VAX 780 computer at the computing centre of the University of Seville; crystallographic programs from the *XRAY*76 system (Stewart *et al.*, 1976) used throughout.

Table 2. *Bond distances* (Å) *and angles* (°) *in the molecule and in intermolecular hydrogen bonds with e.s.d.'s in parentheses*

C1—C2	1·550 (1)	C31—C32	1·546 (2)
C1—C11	1·513 (1)	C31—O31	1·407 (1)
C1—N	1·535 (2)	C31—O32	1·191 (2)
C2—C3	1·550 (1)	C41—C42	1·485 (2)
C2—O21	1·453 (1)	C41—O41	1·406 (1)
C3—C4	1·506 (1)	C41—O42	1·177 (1)
C3—O31	1·472 (1)	C51—C52	1·465 (2)
C4—C5	1·566 (1)	C51—O51	1·375 (1)
C4—O41	1·459 (1)	C51—O52	1·202 (2)
C5—C6	1·514 (1)	C61—C62	1·493 (2)
C5—O51	1·430 (1)	C61—O61	1·398 (1)
C6—C7	1·543 (1)	C61—O62	1·174 (2)
C6—O61	1·462 (1)	C71—C72	1·510 (2)
C7—O71	1·454 (1)	C71—O71	1·338 (1)
C21—C22	1·519 (1)	C71—O72	1·181 (1)
C21—O21	1·402 (1)	O11—N	1·218 (1)
C21—O22	1·170 (1)	O12—N	1·226 (2)
C11—C1—N	111·1 (8)	C32—C31—O32	129·5 (10)
C2—C1—N	103·9 (7)	C32—C31—O31	107·7 (9)
O2—C1—C11	116·4 (8)	O41—C41—O42	121·3 (9)
C1—C2—O21	109·2 (7)	C42—C41—O42	128·6 (10)
C1—C2—C3	112·9 (7)	C42—C41—O41	110·1 (9)
C3—C2—O21	103·0 (7)	O51—C51—O52	120·9 (11)
C2—C3—O31	104·6 (7)	C52—C51—O52	127·9 (12)
C2—C3—C4	111·8 (7)	C52—C51—O51	111·2 (10)
C4—C3—O31	108·6 (7)	O61—C61—O62	123·9 (11)
C3—C4—O41	110·9 (7)	C62—C61—O62	130·1 (11)
C3—C4—C5	112·6 (7)	C62—C61—O61	106·0 (9)
C5—C4—O41	105·2 (7)	O71—C71—O72	123·5 (11)
C4—C5—O51	104·2 (7)	C72—C71—O72	125·7 (11)
C4—C5—C6	112·4 (7)	C72—C71—O71	110·8 (9)
C6—C5—O51	112·5 (7)	C2—O21—C21	115·0 (7)
C5—C6—O61	107·2 (7)	C3—O31—C31	114·4 (7)
C5—C6—C7	114·6 (8)	C4—O41—C41	117·0 (7)
C7—C6—O61	105·9 (7)	C5—O51—C51	118·0 (8)
C6—C7—O71	104·0 (7)	C6—O61—C61	114·4 (8)
O21—C21—O22	124·6 (9)	C7—O71—C71	114·0 (8)
C22—C21—O22	128·4 (10)	O11—N—O12	127·5 (11)
C22—C21—O21	107·0 (8)	C1—N—O12	116·3 (10)
O31—C31—O32	122·7 (10)	C1—N—O11	116·2 (10)

	C—H	C···O	H···O	C—H···O
C32—H321···O11^{i}	1·059 (12)	3·262 (14)	2·597 (8)	120·3 (6)
C52—H522···O62^{ii}	1·058 (11)	3·350 (14)	2·410 (8)	147·3 (7)
C22—H222···O72^{ii}	1·092 (10)	3·374 (13)	2·450 (8)	141·6 (5)
C22—H223···O12^{iii}	1·051 (9)	3·308 (13)	2·654 (9)	120·1 (6)
C22—H223···O32^{iv}	1·051 (9)	3·389 (11)	2·505 (7)	141·3 (6)
C62—H623···O52^{iv}	1·059 (10)	3·489 (13)	2·711 (8)	130·1 (7)
C42—H422···O71^{v}	1·090 (10)	3·370 (12)	2·482 (7)	137·7 (6)
C72—H723···O12^{vi}	1·058 (11)	3·284 (14)	2·703 (9)	114·3 (6)

Symmetry codes: (i) $-x+\frac{1}{2}+1,\ -y+1,\ +z-\frac{1}{2}$ (ii) $+x+\frac{1}{2},\ -y+\frac{1}{2},\ -z$ (iii) $-x+\frac{1}{2}+1,\ -y+1,\ +z+\frac{1}{2}$ (iv) $+x,\ +y,\ +z+1$ (v) $-x+\frac{1}{2},\ -y+1,\ +z+\frac{1}{2}$ (vi) $-x+\frac{1}{2},\ -y+1,\ +z-\frac{1}{2}$.

The final atomic coordinates and equivalent isotropic thermal parameters for the non-H atoms are given in Table 1.* An *ORTEP* (Johnson, 1965) plot of the molecule with atom labelling is displayed in Fig. 1. Bond distances and angles are listed in Table 2 and values are all within expected ranges.

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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 52444 (10 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Related literature. For the preparation and characterization of the compound see Mancera, Rodríguez, Roffe & Galbis (1988). The title compound is an intermediate product in the synthesis of pyrazoles with the sugar nitro-olefins as dipolarophiles. Great difficulty is encountered in the crystallization of these compounds and crystals are poor in quality. Recently the structure of a related compound has been reported (Conde & Conde, 1989).

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Structure of Triphenylphosphine Oxide Hemihydrate

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Abstract. $C_{18}H_{15}OP.\frac{1}{2}H_2O$, $M_r = 287{\cdot}30$, orthorhombic, $Fdd2$, $a = 19{\cdot}794\,(18)$, $b = 32{\cdot}540\,(12)$, $c = 9{\cdot}459\,(6)$ Å, $V = 6092{\cdot}5\,(22)$ Å^3, $Z = 16$, $D_x = 1{\cdot}253$ g cm^{-3}, $\lambda(\text{Mo }K\alpha) = 0{\cdot}71073$ Å, $\mu = 1{\cdot}76$ cm^{-1}, $F(000) = 2416$, $T = 294$ K, $R = 0{\cdot}037$ for 1677 reflections with $I > 3\sigma(I)$. The water O atoms sit on a crystallographic twofold axis and are linked by hydrogen bonds to two of the oxide molecules with O···OW distances of 2·856 (3) Å. Considerable conformational flexibility is conferred on the molecule by the long P—C bonds (around 1·8 Å each).

Experimental. Two independent experiments intended to produce cocrystallization of triphenylphosphine oxide (TPPO) with a catechol-related compound (carried out by PWB) and racemic phenylalanine (carried out by JVS) gave crystals different from those of the reactants. In the latter case, mass spectroscopy of crystals of the hemihydrate obtained from 1:1 dimethylformamide/water indicated a molecular ion (m/e: 278 kg C^{-1}) corresponding to TPPO. Since the unit-cell dimensions had not been reported previously, crystal-structure determinations were undertaken independently.† Essentially identical results were obtained. PWB used Mo $K\alpha$ X-radiation and his determination involved more measurements and has lower e.s.d.'s than that of JVS who used Cu $K\alpha$ X-radiation. Since there are no inexplicable differences, the results to be described here are those of PWB. A poorly formed block measuring 0·80 × 0·70 × 0·50 mm was carefully carved away from a mass of crystals and was mounted on a glass fiber using an epoxy resin. X-ray data for this crystal were collected on an Enraf–Nonius CAD-4 diffractometer using graphite-monochromated Mo $K\alpha$ radiation. Lattice parameters and their e.s.d.'s were derived from the setting angles of 25 reflections ($30 < 2\theta < 37°$). The space group was determined from the systematic absences (hkl: $h + k$, $h + l$, $k + l = 2n$; $0kl$: $k + l = 4n$, $k,l = 2n$; $h0l$: $h + l = 4n$, h, $l = 2n$; $hk0$: $h,k = 2n$). 2037 unique reflections ($2 < 2\theta < 56°$; $0 < h < 26$, $0 < k < 43$, $0 <$

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† The structure of TPPO hemihydrate had been solved in the maximal non-isomorphous subgroup Cc by Israel Goldberg with unpublished results (Etter, 1989).

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