Triclinique
$P\bar{1}$
a = 8,8118 (4) Å
b = 11,6060 (6) Å
c = 12,1748 (7) Å
α = 115,234 (2)°
β = 107,660 (3)°
γ = 95,643 (3)°
V = 1035,1 (2) Å^3
Z = 2
D_x = 1,311 Mg m^{-3}
D_m pas mesurée

Paramètres de la maille à l'aide de 7400 réflexions
θ = 1,0–25,4°
μ = 0,082 mm^{-1}
T = 298 K
Cube
0,30 × 0,25 × 0,20 mm
Incolore

Collection des données

Diffractomètre KappaCCD détecteur bidimensionnel
Balayage φ
Correction d'absorption: aucun
7400 réflexions mesurées
3807 réflexions indépendantes

3211 réflexions avec $I > 3\sigma(I)$
R_{int} = 0,021
θ_{max} = 25,41°
$h = 0 \rightarrow 10$
$k = -14 \rightarrow 14$
$l = -15 \rightarrow 13$

Affinement

Affinement à partir des F
R = 0,050
wR = 0,078
S = 1,268
3211 réflexions
280 paramètres
Les paramètres des atomes d'hydrogène en position théorique

Statistique de comptage
$(\Delta/\sigma)_{max}$ = 0,040
$\Delta\rho_{max}$ = 0,58 e Å^{-3}
$\Delta\rho_{min}$ = −0,19 e Å^{-3}
Correction d'extinction: aucun
Facteurs de diffusion des Waasmaier & Kirfel (1995)

Tableau 1. *Paramètres géométriques (Å, °)*

N1—N2	1,404 (1)	C4a—C8a	1,387 (1)
N1—C13a	1,467 (1)	C10—C10a	1,521 (1)
N2—C3	1,293 (1)	C10a—C11	1,539 (1)
N4—C3	1,384 (1)	C10a—C13a	1,560 (1)
N4—C4a	1,435 (1)	C11—C12	1,522 (1)
N4—C13a	1,474 (1)	C12—C13	1,516 (1)
N9—C8a	1,424 (1)	C13—C13a	1,546 (1)
N9—C10	1,368 (1)		
N2—N1—C13a	111,5 (1)	C10—C10a—C13a	114,9 (1)
N1—N2—C3	105,5 (1)	C11—C10a—C13a	105,0 (1)
C3—N4—C4a	123,9 (1)	C10a—C11—C12	102,8 (1)
C3—N4—C13a	107,5 (1)	C11—C12—C13	107,1 (1)
C4a—N4—C13a	117,1 (1)	C12—C13—C13a	107,0 (1)
C8a—N9—C10	123,8 (1)	N1—C13a—N4	97,8 (1)
N2—C3—N4	113,2 (1)	N1—C13a—C10a	113,1 (1)
N4—C4a—C8a	121,0 (1)	N1—C13a—C13	116,6 (1)
N9—C8a—C4a	120,5 (1)	N4—C13a—C10a	113,1 (1)
N9—C10—C10a	120,0 (1)	N4—C13a—C13	111,8 (1)
C10—C10a—C11	118,3 (1)	C10a—C13a—C13	104,8 (1)

Les atomes d'hydrogène du groupement méthyl en position C26 ont été localisés sur la carte de densité différence en six positions de degré d'occupation égal à 0,5.

Collection des données: *Kappa-CCD Reference Manual* (Nonius, 1998). Réduction des données: *MAXUS* (Mackay *et al.*, 1998). Programme(s) pour la solution de la structure: *MAXUS*. Programme(s) pour l'affinement de la structure: *MAXUS*. Graphisme moléculaire: *ORTEP*II (Johnson, 1976). Logiciel utilisé pour préparer le matériel pour publication: *MAXUS*.

Des documents complémentaires concernant cette structure peuvent être obtenus à partir des archives électroniques de l'UICr (Référence: GS1040). Les processus d'accès à ces archives sont donné au dos de la couverture.

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4-(Triphenylmethyl)phenol–triphenylphosphine oxide (1/1)

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Abstract

In the crystal structure of the title compound, $C_{25}H_{20}O{\cdot}C_{18}H_{15}OP$, the components are linked through O—H···O and C—H···O hydrogen bonds. The phenyl rings of triphenylphosphine oxide are close packed,

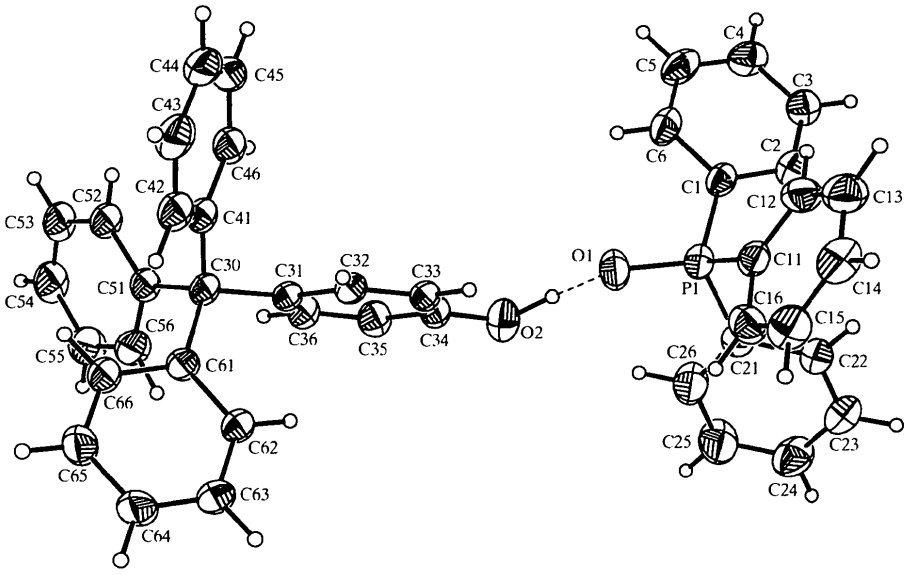
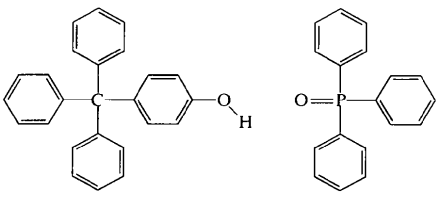


Fig. 1. Molecular diagram and numbering scheme of TPM-OH:TPPO (Siemens, 1994). Displacement ellipsoids are drawn at the 50% probability level for non-H atoms.

while those of 4-(triphenylmethyl)phenol are involved in a sextuple embrace type of packing.

Comment

The crystal structures of host–guest inclusion complexes of 4-(triphenylmethyl)benzoic acid with xylene and chlorobenzene guests were reported by us recently (Anthony *et al.*, 1998; Jetti *et al.*, 1998). In a continuation of these studies, the crystal structure of the 1:1 molecular complex of 4-(triphenylmethyl)phenol (TPM-OH) with triphenylphosphine oxide (TPPO; Etter & Baures, 1988), recrystallized from *o*-xylene, was examined. The structure of TPM-OH:TPPO shows the importance of strong hydrogen bonds (O—H···O) and weak interactions (C—H···O and phenyl···phenyl) in the stabilization of crystal structures.



TPM-OH TPPO

The asymmetric unit of TPM-OH:TPPO contains one molecule each of TPM-OH and TPPO (Fig. 1). The assembly of the molecular complex is mediated through an O—H···O hydrogen bond [O···O 2.633 (3) Å] between the phenol OH and the phosphine oxide P=O groups (Moreno-Fuquen *et al.*, 1998) (Fig. 2). The three

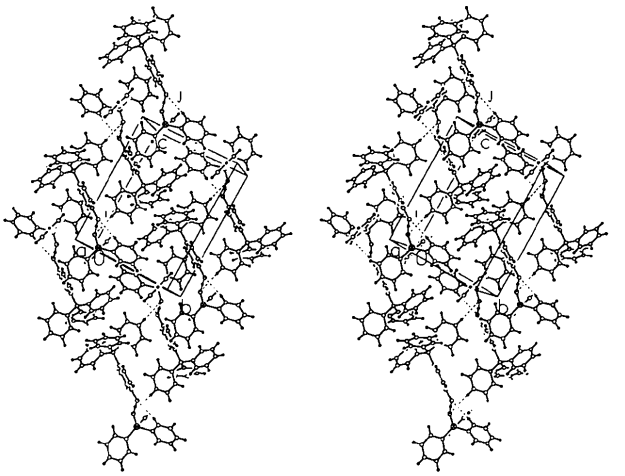


Fig. 2. Stereoview of TPM-OH:TPPO down [100], showing the O—H···O hydrogen bonds. O—H···O and C—H···O hydrogen bonds (*i* and *j*) are shown as dashed lines; interaction *k* is not shown for clarity. Notice the sextuple phenyl embrace of TPM-OH and the space-filling by TPPO molecules.

phenyl groups of TPPO occupy the voids created by the O—H···O interlink and the trityl groups of TPM-OH are dove-tailed in a sextuple phenyl embrace (Dance & Scudder, 1996, 1998). The O—H···O hydrogen bond between the two components is strengthened by C—H···O interactions [C···O: (*i*) 3.730 (3), (*j*) 3.620 (3) and (*k*) 3.163 (3) Å]. In effect, the OH group donates one strong hydrogen bond and accepts two weak bonds, while the P=O group accepts one strong and one weak bond. Thus, if the weak interactions are also considered, the hydrogen-bond donor and acceptor capability of the

OH and P═O groups may be taken to be fully satisfied. It may be noted that the contacts from the phenyl C—H donor atoms of TPPO to the O2 atom of TPM-OH (interactions *j* and *k*) are shorter than that from the phenyl C—H of TPM-OH to the O1 atom of TPPO (interaction *i*). This is in agreement with a more general trend wherein the strength of a C—H···O hydrogen bond is more sensitive to donor acidity than it is to acceptor basicity, which is further enhanced, in this case, by co-operativity (Desiraju, 1996). It is expected that an analysis of this structure will be instructive in the design of wheel-and-axle host–guest systems (MacNicol *et al.*, 1996) with the triphenyl group as the wheel and a variable supramolecular axle (Jetti *et al.*, 1998).

Experimental

4-(Triphenylmethyl)phenol was prepared by diazotization of 4-(triphenylmethyl)aniline (Grimm *et al.*, 1986) followed by hydrolysis (Vogel, 1989). Triphenylphosphine oxide was obtained from Lancaster. Crystals suitable for single-crystal X-ray diffraction were grown by slow evaporation of equimolar amounts of 4-(triphenylmethyl)phenol and triphenylphosphine oxide from *o*-xylene.

Crystal data

$C_{25}H_{20}O{\cdot}C_{18}H_{15}OP$
M_r = 614.68
Triclinic
$P\bar{1}$
a = 9.3101 (8) Å
b = 12.4840 (11) Å
c = 14.7622 (14) Å
α = 89.988 (2)°
β = 89.819 (2)°
γ = 73.151 (2)°
V = 1642.1 (3) Å^3
Z = 2
D_x = 1.243 Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 3350 reflections
θ = 2.19–28.52°
μ = 0.121 mm^{-1}
T = 143 (2) K
Rod
0.23 × 0.21 × 0.19 mm
Colorless

Data collection

Siemens SMART CCD area-detector diffractometer
Full-sphere data collection in ω at 0.3° scan width, 120 frames and Φ = 0°, and in Φ at 0.3° scan width, four runs with 600 frames, and ω = 135, 143, 156 and 169°
Absorption correction: empirical (*SADABS*; Blessing, 1995)
T_{min} = 0.973, T_{max} = 0.977

14 578 measured reflections
6453 independent reflections
4581 reflections with $I > 2\sigma(I)$
R_{int} = 0.032
θ_{max} = 28.52°
h = −12 → 7
k = −7 → 16
l = −17 → 18

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.064
$wR(F^2)$ = 0.167
S = 1.086
6453 reflections
453 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0890P)^2 + 0.3827P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.47 e Å^{-3}
$\Delta\rho_{min}$ = −0.37 e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Hydrogen-bonding geometry (Å, °)*

D—H···A	D—H	H···A	D···A	D—H···A
O2—H1···O1	1.19 (5)	1.44 (5)	2.633 (3)	179 (4)
C15—H15···O2^{i}	0.96	2.40	3.163 (3)	136
C26—H26···O2	0.96	2.69	3.620 (3)	164
C64—H64···O1^{ii}	0.96	2.79	3.730 (3)	168

Symmetry codes: (i) $x-1, y, z$; (ii) $2-x, -y, 1-z$.

It should be noted that the e.s.u.'s of the cell dimensions are probably too low; they should be multiplied by a factor of 2 to 10. Furthermore, the cell angles α and β are close to 90°, indicating an apparent monoclinic symmetry. However, a closer examination of the crystal packing revealed that there is no pseudosymmetry in the structure and that the cell system is triclinic. The H atoms of the phenyl groups were generated at idealized geometries and refined isotropically using a riding model. The H atom of the OH group was located from a difference Fourier map and was refined isotropically.

Data collection: *SMART* (Siemens, 1996). Cell refinement: *SAINT* (Siemens, 1996). Data reduction: *SAINT*. Program(s) used to solve structure: *SHELXTL* (Siemens, 1994). Program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997). Molecular graphics: *SHELXL*97. Software used to prepare material for publication: *SHELXL*97.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: BK1472). Services for accessing these data are described at the back of the journal.

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Acta Cryst. (1999). C**55**, 1533–1534

2-(2,3:5,6-Di-*O*-isopropylidene-β-D-mannofuranosyl)-*N*,*N*-(pentamethylene)ethanethioamide†

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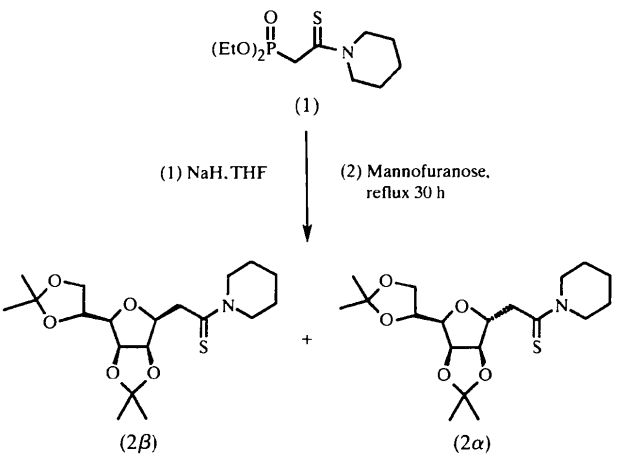
Abstract

The title compound, $C_{19}H_{31}NO_5S$, was prepared using a Wittig–Horner olefination followed by an intramolecular Michael addition. Two diastereoisomers were obtained in a 80:20 (β:α) ratio. The stereochemistry of the major anomer (β, thermodynamic) was based on NMR (^{13}C and ^{1}H) spectra. The present X-ray structure determination confirms the configuration of this product.

Comment

C-Glycosides, *e.g.* shodomycin (Barret & Broughton, 1986), are known for their potential biological activity. Among the methods described by Postema (1992) to synthesize these compounds, one described the Wittig–Horner olefination of a sugar derivative followed by a Michael addition (Ohrui *et al.*, 1975). We used this strategy for the synthesis of some mannofuranosyl ethanethioamides starting from the readily available corresponding thiocarbamoyl methylphosphonates (Bulpin *et al.*, 1994) (see Scheme below). The reaction of metallated (Na) 2-(diethylphosphono)-*N*,*N*-(pentamethylene)ethanethioamide, (1), with a protected sugar (2,3:5,6-di-*O*-isopropylidene-α-D-mannofuranose)



allowed the preparation of the title compound, (2), which was later converted into the corresponding dithioester (Sandrinelli *et al.*, 1998). The configurational assignment of compound (2) (β or α) was deduced from NMR spectra (^{1}H and ^{13}C) and by comparing the coupling constants of the two pure anomers. The X-ray structure determination reported herein confirms the configuration (β) and agrees with the assignment made previously by Mereyala *et al.* (1997). Two conformers of (2β) are observed in this arrangement. As shown in Table 1, their geometrical features exhibit only very slight differences (values for the second conformer are reported using prime notation). One conformer is represented in Fig. 1 (*ORTEP*II; Johnson, 1976). All the substituents of the furanose ring (C7, O3, O2 and C2) are *cis*. The dimethyldioxolane and furanose rings adopt envelope conformations (Merino *et al.*, 1997), with O1, C9 and C12 at the flaps. The thioamide linkage is planar, as expected (Van Roey & Kerr, 1981); see Table 1 for details.

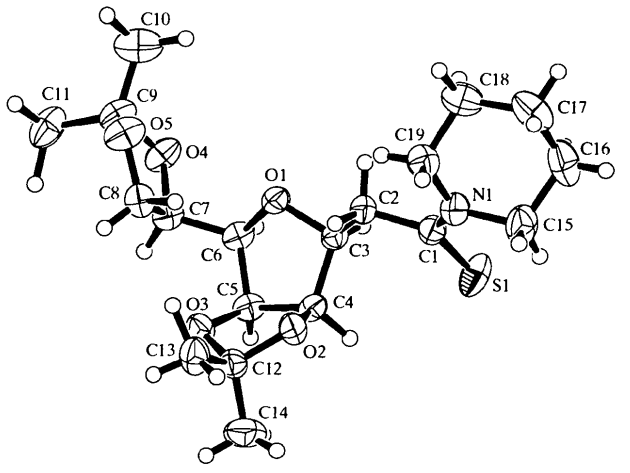


Fig. 1. An *ORTEP*II (Johnson, 1976) drawing of one of the two conformers shown with 45% probability displacement ellipsoids.

† Alternative name: 2,3:5,6-di-*O*-isopropylidene-1-(piperidin-1-ylthiocarbonylmethyl)-β-D-mannofuranose.