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p-Cresyl 2,3-anhydro-5-deoxy-5-phthalimido-1-thio-α-D-lyxofuranoside

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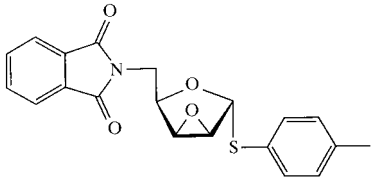
The crystal structure of the title compound, $C_{20}H_{17}NO_4S$, (I), was determined in order to compare the solution and solid-state conformations. The molecule was synthesized as a building block for incorporation into oligosaccharides comprised of conformationally restricted furanose residues. The furanose ring adopts an envelope conformation with the ring O atom displaced above the plane (an ^{O}E conformation). The pseudorotational phase angle (P) is 88.6° and the puckering amplitude (τ_m) is 31.5°. The C2—C1—S—C(Ph) torsion angle is −163.2 (2)°, which places the aglycone in the *exo*-anomeric effect preferred position. The C1—S—C14 bond angle is 99.02 (13)° and the plane of the cresyl moiety is oriented nearly parallel to the four in-plane atoms of the furanose ring envelope. The orientation about the C4—C5 bond is *gauche–gauche* [Bock & Duus (1994). *J. Carbohydr. Chem.* **13**, 513–543].



(I)

Experimental

p-Cresyl 1-thio-α-D-arabinofuranoside (0.5 g, 1.95 mmol) (D'Souza *et al.*, 2000), triphenylphosphine (1.28 g, 4.88 mmol) and phthalamide (0.43 g, 2.92 mmol) were dissolved in tetrahydrofuran (20 ml) and the solution was stirred and cooled to 273 K. Diethyl azodicarboxylate (0.77 ml, 4.88 mmol) was added dropwise over a period of 10 min and the reaction mixture was stirred for another 30 min as it warmed to room temperature. The solution was concentrated to dryness under reduced pressure and the residue was purified by column chromatography using 4:1 petroleum ether/ethyl acetate as the eluant to give 560 mg (78%) of the product as a colourless solid. The product was recrystallized from a 1:1 dichloromethane/hexane (m.p. 416–419 K).

Crystal data

$C_{20}H_{17}NO_4S$	D_x = 1.311 Mg m^{-3}
M_r = 367.41	Mo $K\alpha$ radiation
Monoclinic, $P2_1$	Cell parameters from 11686 reflections
a = 12.1660 (4) Å	θ = 2.66–25.04°
b = 5.5725 (2) Å	μ = 0.198 mm^{-1}
c = 14.5413 (4) Å	T = 150 K
β = 109.247 (2)°	Rod, colourless
V = 930.73 (5) Å^3	0.23 × 0.12 × 0.06 mm
Z = 2	

Data collection

Nonius KappaCCD diffractometer	R_{int} = 0.054
ω scans	θ_{max} = 25.04°
11 686 measured reflections	$h = -14 \rightarrow 14$
3119 independent reflections	$k = -6 \rightarrow 6$
2693 reflections with $I > 2\sigma(I)$	$l = -17 \rightarrow 17$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0458P)^2 + 0.3896P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.047	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2)$ = 0.102	$\Delta\rho_{max}$ = 0.21 e Å^{-3}
S = 1.086	$\Delta\rho_{min}$ = −0.19 e Å^{-3}
3119 reflections	Absolute structure: Flack (1983), 1286 Friedel pairs
237 parameters	Flack parameter = 0.00 (11)
H-atom parameters constrained	

The correct enantiomer was chosen based on the known absolute configuration. There is a region containing disordered solvent, which appears to consist of three peaks situated along a line. As it was difficult to obtain a satisfactory model of this region in terms of a recognizable molecule, the density in this area was accounted for by the *SQUEEZE* program (Sluis & Spek, 1990) of *PLATON* (Spek, 1999). This program modifies the observed structure factors by subtracting the contributions to them from the electron density in the disordered region. This region occupies a total of 84 Å^{-3} per unit cell and the electron density removed by the *SQUEEZE* procedure amounts to 5 electrons per unit cell. This disordered solvent molecule is located in a channel which runs parallel to the *b* axis.

Data collection: *COLLECT* (Nonius, 1999); cell refinement: *DENZO* (Otwinowski & Minor, 1997); data reduction: *DENZO*; program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993).

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