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3-(Dimethoxythiophosphorylamido)-2-(2′,3′,4′-tri-*O*-acetyl-β-D-xylopyranos-1′-ylimino)-1,3-thiazolidin-4-one

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Key indicators

Single-crystal X-ray study
T = 113 K
Mean σ(C–C) = 0.004 Å
R factor = 0.040
wR factor = 0.084
Data-to-parameter ratio = 17.8

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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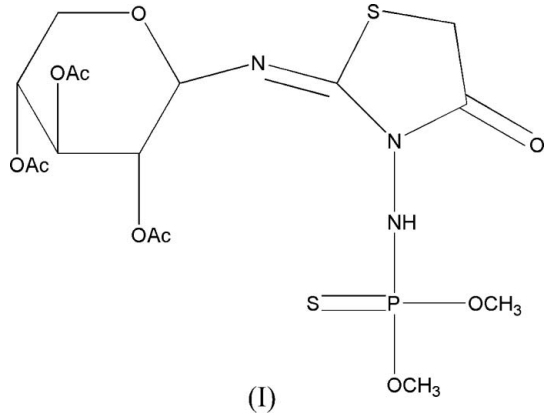
In the title compound, $C_{16}H_{24}N_3O_{10}PS_2$, the thiazolidine ring is approximately planar and the tetrahydropyran ring adopts a chair conformation. The crystal packing is stabilized by intermolecular N—H···O, C—H···S and C—H···O hydrogen bonds.

Comment

Thiazolidin-4-one derivatives are of great interest in pharmaceutical and agrochemical research owing to their biological properties. They exhibit anti-HIV (Barreca *et al.*, 2001), anticonvulsant (Kandeel & El-Latif, 2001), analgesic (Schenone *et al.*, 2001), herbicidal (Suzuki & Morita, 2003) activities *etc*. With the development of biomolecular science, many biological properties of carbohydrates have been discovered for *e.g.* cell recognition, cell growth, cell differentiation and signal transduction (Wells *et al.*, 2001). Thiazolidin-4-one derivatives as pharmacophores linked to carbohydrates could improve their bioactivity, biocompatibility and biodegradability. In view of these properties and as part of our ongoing studies in the area of agrochemical agents, we synthesized some novel thiophosphorylamido(glycosylimino)thiazolidin-4-one derivatives. The X-ray crystal structure determination of the title compound, (I), was undertaken to investigate the relationship between structure and herbicidal activity.



(I)

The molecular structure of (I) is shown in Fig. 1. The thiazolidine ring is approximately planar, with a maximum deviation of 0.101 (2) Å for C4. The dihedral angle between the thiazolidin-4-one ring (S1/N2/C3–C5) and the thiophosphorylamide (P1/N1/N2/S2) plane is 87.75 (7)°. The C5═N3 double bond length of 1.259 (3) Å is shorter than the corresponding length in 2-salicylideneamino-4,5,6,7-tetrahydrobenzo[*b*]thiophene-3-carbonitrile [1.281 (2) Å; Elerman & Elmali, 1998]. The C5—S1 distance of 1.765 (2) Å is shorter

than the C4—S1 distance of 1.814 (3) Å, as a result of the conjugation of the S atom with the C5═N3 double bond. The tetrahydropyran ring adopts a chair conformation.

The crystal structure is stabilized by N—H···O, C—H···S and C—H···O intermolecular hydrogen bonds (Table 2).

Experimental

Ethyl bromoacetate (4.5 ml, 3 mmol) was added to a solution of 1-dimethoxythiophosphoryl-4-(1′-*N*-2′,3′,4′-tri-*O*-acetyl-β-D-xylopyranosyl)thiosemicarbazide (0.947 g, 2.0 mmol) in CH_2Cl_2 (30 ml) with stirring. The mixture was refluxed for 12 h and the solvent was removed under vacuum. The crude product was purified by column chromatography using silica gel and eluted with ethyl acetate and petroleum ether (1:2) to afford compound (I) (yield 57%, m. p. 415 K). Single crystals of (I) suitable for X-ray diffraction were obtained by recrystallization from an ethyl acetate–petroleum ether (1:2 *v*/*v*) solution.

Crystal data

$C_{16}H_{24}N_3O_{10}PS_2$
M_r = 513.47
Monoclinic, $P2_1$
a = 7.517 (4) Å
b = 6.819 (3) Å
c = 22.574 (11) Å
β = 98.626 (7)°
V = 1144.0 (10) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.36 mm^{-1}
T = 113 (2) K
0.12 × 0.12 × 0.12 mm

Data collection

Rigaku Saturn diffractometer
Absorption correction: multi-scan (Jacobson, 1998)
T_{min} = 0.958, T_{max} = 0.958
12248 measured reflections
5317 independent reflections
4572 reflections with $I > 2\sigma(I)$
R_{int} = 0.040

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.084
S = 1.01
5317 reflections
299 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}
Absolute structure: Flack (1983), 2369 Friedel pairs
Flack parameter: 0.01 (6)

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O3^{i}	0.90 (1)	1.97 (1)	2.861 (3)	173 (3)
C1—H1B···S2^{ii}	0.98	2.73	3.666 (3)	161
C7—H7A···O9^{iii}	0.99	2.35	3.250 (3)	151
C14—H14A···O7^{iv}	0.98	2.44	3.293 (4)	145

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

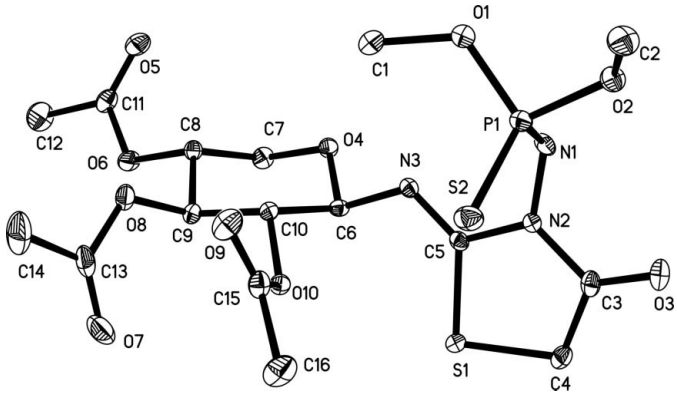


Figure 1
The molecular structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. For the sake of clarity, H atoms have been omitted.

The amino H atom was located in a difference map and refined with a distance restraint [N—H = 0.90 (1) Å]. The remaining H atoms were placed in calculated positions (C—H = 0.98–1.00 Å), and included in the final cycles of refinement using a riding model, with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C).

Data collection: *CrystalClear* (Rigaku/MSC, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1999); software used to prepare material for publication: *CrystalStructure* (Rigaku/MSC, 2005).

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