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Crystal structure of 4-(acetoxymethyl)-6-(3-acetyl-3-(4-fluorophenyl)thioureido)cyclohex-4-ene-1,2,3-triyl triacetate, $C_{24}H_{26}FN_2O_9S$

Table 1: Data collection and handling.

Crystal:	Block, clear light colorless
Size:	$0.35 \times 0.26 \times 0.2$ mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	0.17 mm ⁻¹
Diffractometer, scan mode:	Bruker SMART, φ and ω -scans
$\theta_{\max}$, completeness:	25.5°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	21185, 5147, 0.033
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 4151
$N(\text{param})_{\text{refined}}$:	339
Programs:	Bruker programs [1], SHELX [2, 3]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.3530(3)	1.0512(3)	0.67981(15)	0.0409(7)
H1	0.3592	1.0476	0.7269	0.049*
C2	0.3265(3)	1.1702(3)	0.66051(15)	0.0419(7)
H2	0.3650	1.2276	0.6827	0.050*
C3	0.2520(3)	1.1981(3)	0.61397(16)	0.0417(7)
C4	0.1797(3)	1.1127(2)	0.57758(15)	0.0415(7)
H4	0.0964	1.1180	0.5913	0.050*
C5	0.2230(3)	0.9940(3)	0.58784(15)	0.0403(7)
H5	0.2936	0.9799	0.5613	0.048*
C6	0.2520(3)	0.9753(2)	0.65820(15)	0.0412(7)
H6	0.1812	0.9912	0.6842	0.049*
C7	0.2350(3)	1.3189(3)	0.59576(19)	0.0516(9)
H7A	0.2694	1.3676	0.6283	0.062*
H7B	0.2734	1.3345	0.5548	0.062*
C8	0.0722(4)	1.4440(3)	0.59106(18)	0.0611(10)
C9	-0.0603(4)	1.4493(4)	0.5804(3)	0.0884(14)
H9A	-0.0821	1.5236	0.5670	0.133*
H9B	-0.1008	1.4311	0.6198	0.133*
H9C	-0.0825	1.3964	0.5475	0.133*
C10	0.0844(4)	1.1481(4)	0.4762(2)	0.0679(11)
C11	0.1125(5)	1.1723(5)	0.4067(2)	0.1087(19)
H11A	0.1002	1.2507	0.3982	0.163*
H11B	0.0611	1.1286	0.3795	0.163*
H11C	0.1939	1.1531	0.3981	0.163*
C12	0.1502(4)	0.8413(3)	0.5237(2)	0.0675(11)
C13	0.0488(4)	0.7619(4)	0.5168(3)	0.0916(15)
H13A	0.0759	0.6941	0.4965	0.137*
H13B	-0.0122	0.7957	0.4908	0.137*
H13C	0.0171	0.7445	0.5586	0.137*
C14	0.2591(3)	0.8120(3)	0.7241(2)	0.0608(10)
C15	0.2994(5)	0.6932(4)	0.7252(3)	0.1016(18)
H15A	0.2998	0.6663	0.7688	0.152*

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Abstract

$C_{24}H_{27}FN_2O_9S$, orthorhombic, $P2_12_12_1$ (no. 19), $a = 11.2301(12)$ Å, $b = 11.9074(13)$ Å, $c = 20.738(2)$ Å, $V = 2773.1(5)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0389$, $wR_{\text{ref}}(F^2) = 0.1022$, $T = 296(2)$ K.

CCDC no.: 1871995

The crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

The title compound was synthesized with a total yield of 95% starting with (1S,2S,3R,6S)-6-amino-4-(hydroxymethyl)

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Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
H15B	0.3784	0.6883	0.7076	0.152*
H15C	0.2462	0.6482	0.6998	0.152*
C16	0.5511(3)	0.9605(3)	0.68278(15)	0.0424(7)
C17	0.6799(3)	0.9809(3)	0.58557(19)	0.0590(9)
C18	0.7937(4)	0.9455(4)	0.5534(2)	0.0810(14)
H18A	0.8603	0.9696	0.5789	0.122*
H18B	0.7984	0.9790	0.5113	0.122*
H18C	0.7952	0.8652	0.5493	0.122*
C19	0.7411(3)	0.8604(3)	0.67526(16)	0.0471(8)
C20	0.7272(3)	0.7470(3)	0.66562(18)	0.0554(9)
H20	0.6629	0.7203	0.6419	0.066*
C21	0.8092(4)	0.6728(3)	0.6913(2)	0.0680(11)
H21	0.8010	0.5958	0.6853	0.082*
C22	0.9016(4)	0.7148(4)	0.7255(2)	0.0738(13)
C23	0.9174(3)	0.8257(4)	0.7361(2)	0.0765(12)
H23	0.9817	0.8512	0.7602	0.092*
C24	0.8357(3)	0.9008(3)	0.7104(2)	0.0621(10)
H24	0.8449	0.9776	0.7168	0.075*
F1	0.9807(2)	0.6407(3)	0.75128(17)	0.1223(12)
N1	0.4674(2)	1.0179(2)	0.65179(12)	0.0432(6)
H1A	0.4813	1.0369	0.6125	0.052*
N2	0.6556(2)	0.9375(2)	0.64669(13)	0.0461(6)
O1	0.1088(2)	1.33768(19)	0.59092(12)	0.0551(6)
O2	0.1375(3)	1.5214(3)	0.59838(19)	0.0986(11)
O3	0.1874(2)	1.1378(2)	0.50974(11)	0.0528(6)
O4	−0.0111(3)	1.1406(4)	0.49946(18)	0.1088(13)
O5	0.12876(19)	0.91730(19)	0.57010(12)	0.0515(6)
O6	0.2413(4)	0.8374(3)	0.4945(2)	0.1198(16)
O7	0.2836(2)	0.85897(18)	0.66569(12)	0.0548(6)
O8	0.2122(3)	0.8618(3)	0.76675(15)	0.0772(8)
O9	0.6118(3)	1.0434(3)	0.55762(14)	0.0813(9)
S1	0.53935(8)	0.91842(8)	0.75889(4)	0.0576(3)

cyclohex-4-ene-1,2,3-triol, 1-fluoro-4-isothiocyanatobenzene and acetic anhydride as raw materials. Colorless crystals suitable for X-ray diffraction study were obtained from a trichloromethane solution.

Experimental details

The hydrogen atoms were assigned with common isotropic displacement factors $U_{\text{iso}}(\text{H}) = 1.2$ times $U_{\text{eq}}(\text{C, cyclohexene ring, benzene ring})$, and $U_{\text{iso}}(\text{H}) = 1.5$ times $U_{\text{eq}}(\text{C, methyl})$. The final refinement by using geometrical restraints, with $\text{C} - \text{H} = 0.98 \text{ \AA}$ (cyclohexene ring), $\text{C} - \text{H} = 0.96 \text{ \AA}$ (methyl), and $\text{C} - \text{H} = 0.93 \text{ \AA}$ (benzene ring). The Flack parameter (0.09(4)) was calculated based on 1519 quotients [3].

Discussion

Chiral glycosyl thioureas have been attracted attention because they can be served as chiral organocatalysts for many

reactions such as Michael additions [4] and Mannich reactions [5, 6]. Synthetic methods to chiral glycosyl thioureas were reported [7]. However, the crystal structure of chiral glycosyl thiourea, which is very important for the confirmation of the compound, is rarely reported.

As shown in the figure, the title compound features a six-membered cyclohexene ring that adopts a half-chair conformation with C1/C2/C3/C4 atoms in one plane. The bond length of C2–C3 is 1.322(3) Å, showing the characteristics of the double bond. Other C–C bond lengths of the cyclohexene ring are in the range of 1.499(4)–1.519(3) Å. Three acetoxyl substituents (CH₃CO₂–) occupy the equatorial bond positions, while the thiourea fragment occupies the axial bond position. The C17–O9 bond distance of 1.215(3) Å is longer than other four carbonyl groups, which may be caused by the stronger giving electrons ability of nitrogen than oxygen, and the formation of an intramolecular hydrogen bond (*cf.* the figure).

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