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Crystal structure of 2-(3-acetyl-1-(4-bromophenyl)-5-((*R*)-4-chlorophenyl)-1,2,4-triazolo-4-yl)-2-deoxy-1,3,4,6-tetraacetyl-β-D-glucose, $C_{30}H_{31}BrClN_3O_{10}$

R. J. Abdel-Jalil*,[I], W. Voelter[II], C. Maichle-Mössmer[III], Y. Baqi[IV] and H.-J. Machulla[V]

[I] Hashemite University, Faculty of Science, Chemistry Department, Zarka, Jordan
[II] Universität Tübingen, Interfakultäres Institut für Biochemie, Hoppe-Seyler Straße 4, 72076 Tübingen, Germany
[III] Universität Tübingen, Institut für Anorganische Chemie, Auf der Morgenstelle 18, 72076 Tübingen, Germany
[IV] University of Bonn, Pharmaceutical Institute, Pharmaceutical Chemistry Poppelsdorf, Kreuzbergweg 26, 53115 Bonn, Germany
[V] Universitätsklinikum Tübingen, PET Zentrum, Radiopharmazie, Röntgenweg 15, 72076 Tübingen, Germany

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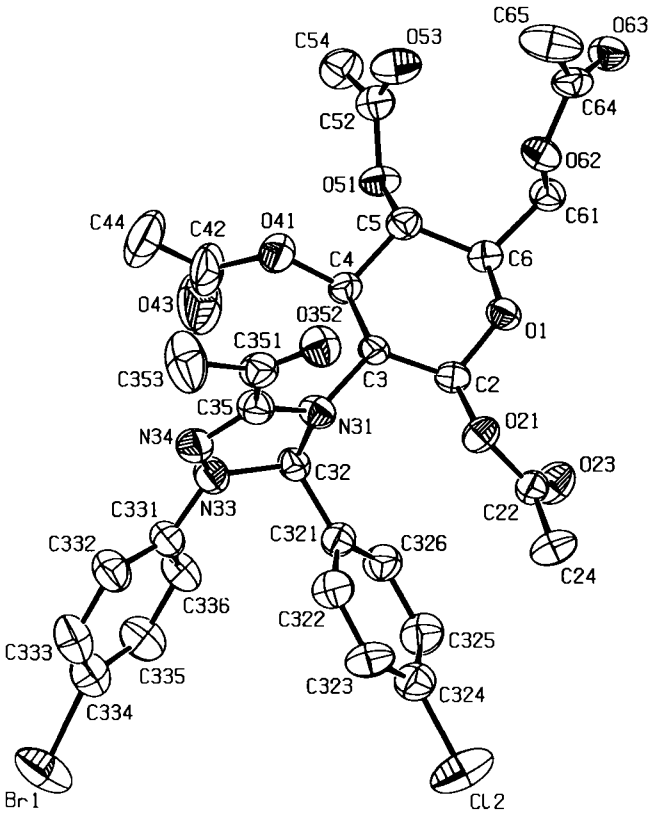

Abstract

$C_{30}H_{31}BrClN_3O_{10}$, orthorhombic, $P2_12_12_1$ (no. 19), a = 9.9317(8) Å, b = 11.1822(5) Å, c = 29.204(3) Å, V = 3243.3 Å^3, Z = 4, $R_{gt}(F)$ = 0.061, $wR_{ref}(F^2)$ = 0.172, T = 223 K.

Source of material

During our search towards developing novel methods for the construction of chiral heterocyclic systems, annulated on carbohydrate scaffolds [1], we synthesized the title compound by the direct interaction of 1,3,4,6-tetraacetyl-2-deoxy-[(4-chlorophenyl)-methyleneamino]-β-D-glucopyranoside [2] and 1-(4-bromophenyl)-hydrazono-1-chloropropanone [3] in refluxing ethanol in presence of triethylamine. The yellow product was purified by column chromatography. Pure single crystals, suitable for the X-ray structure analysis, were obtained from absolute ethanol as yellow needles (m.p. 413-414 K).

* Correspondence author (e-mail: jalil@hu.edu.jo)

Experimental details

While the H atoms of the methyl ester groups were refined using restraints, the other H atoms were refined freely.

Discussion

The crystal structure proves the constitution and absolute configuration of the title compound. The absolute configuration could be determined reliably by a Flack parameter of 0.013(3). The new generated stereocenter C32 has *R*-configuration and the C=N bond length of the imine residue of the triazoline moiety (1.304(8) Å) lies within the normally accepted values. The molecules form layers via nonlinear intermolecular hydrogen bonds and are packed in a crossed arrangement, when viewed along the *b* axis. The pyranose moiety adopts a $4C_1$ conformation.

Table 1. Data collection and handling.

Crystal:	yellow needle fragment, size 0.10 × 0.15 × 0.25 mm
Wavelength:	Cu K_α radiation (1.54184 Å)
μ:	29.99 cm^{-1}
Diffractometer, scan mode:	Nonius CAD4, ω
$2\theta_{max}$:	129.64°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	7848, 5437
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 3955
$N(param)_{refined}$:	476
Programs:	SHELXS-97 [4], SHELXL-97 [5], PLATON [6]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(24A)	4*a*	0.5002	0.6860	1.2158	0.076
H(24B)	4*a*	0.5536	0.5824	1.2481	0.076
H(24C)	4*a*	0.4675	0.6885	1.2689	0.076
H(44A)	4*a*	0.1978	0.0725	1.0102	0.144
H(44B)	4*a*	0.2550	0.0073	1.0543	0.144
H(44C)	4*a*	0.3375	0.1128	1.0314	0.144
H(54A)	4*a*	−0.1687	−0.0319	1.1291	0.075
H(54B)	4*a*	−0.2343	0.0682	1.1604	0.075
H(54C)	4*a*	−0.2233	−0.0650	1.1785	0.075
H(64A)	4*a*	0.2192	−0.1683	1.3158	0.106
H(64B)	4*a*	0.3340	−0.0700	1.3159	0.106
H(64C)	4*a*	0.2716	−0.1142	1.2690	0.106
H(35A)	4*a*	0.7198	0.2146	1.0602	0.124
H(35B)	4*a*	0.5863	0.2139	1.0305	0.124
H(35C)	4*a*	0.6221	0.1027	1.0618	0.124
H(2)	4*a*	0.190(5)	0.446(4)	1.195(2)	0.01(1)
H(3)	4*a*	0.383(7)	0.270(6)	1.166(2)	0.05(2)

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(4)	4a	0.095(6)	0.294(5)	1.134(2)	0.03(2)
H(5)	4a	0.177(6)	0.106(5)	1.193(2)	0.02(1)
H(6)	4a	0.050(7)	0.311(6)	1.224(2)	0.04(2)
H(32)	4a	0.177(7)	0.467(5)	1.102(2)	0.04(2)
H(32A)	4a	0.510(7)	0.591(6)	1.100(2)	0.04(2)
H(32B)	4a	0.581(8)	0.773(6)	1.123(2)	0.05(2)
H(32C)	4a	0.197(7)	0.841(6)	1.163(2)	0.05(2)

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(32D)	4a	0.141(7)	0.640(6)	1.136(2)	0.05(2)
H(33A)	4a	0.474(6)	0.482(6)	0.983(2)	0.03(2)
H(33B)	4a	0.495(8)	0.662(7)	0.928(3)	0.07(2)
H(33C)	4a	0.149(8)	0.776(7)	0.986(2)	0.06(2)
H(33D)	4a	0.162(9)	0.612(7)	1.032(3)	0.07(3)
H(61A)	4a	0.085(6)	0.245(6)	1.301(2)	0.04(2)
H(61B)	4a	−0.002(7)	0.161(5)	1.269(2)	0.03(2)

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(2)	4a	0.2671(6)	0.3954(5)	1.1997(2)	0.028(3)	0.031(3)	0.039(3)	0.000(3)	0.000(3)	0.003(2)
C(3)	4a	0.3035(6)	0.3172(5)	1.1591(2)	0.034(3)	0.029(3)	0.029(3)	−0.003(2)	0.006(2)	0.000(2)
C(4)	4a	0.1782(6)	0.2440(5)	1.1464(2)	0.035(3)	0.031(3)	0.036(3)	−0.005(3)	0.003(3)	−0.002(2)
C(5)	4a	0.1227(6)	0.1762(6)	1.1867(2)	0.034(3)	0.035(3)	0.037(3)	−0.007(3)	−0.002(2)	0.001(3)
C(6)	4a	0.1124(6)	0.2565(5)	1.2295(2)	0.028(3)	0.029(3)	0.040(3)	0.001(3)	0.001(2)	0.000(3)
C(22)	4a	0.3563(7)	0.5694(5)	1.2341(2)	0.042(4)	0.034(3)	0.033(3)	−0.001(3)	−0.003(3)	−0.002(2)
C(24)	4a	0.4798(7)	0.6373(6)	1.2424(3)	0.046(4)	0.042(4)	0.064(4)	−0.010(3)	−0.006(3)	−0.007(3)
C(32)	4a	0.2821(6)	0.4789(5)	1.0968(2)	0.037(3)	0.035(3)	0.033(3)	−0.001(3)	0.006(3)	0.002(2)
C(35)	4a	0.4445(6)	0.3317(5)	1.0877(2)	0.042(3)	0.036(3)	0.043(3)	0.002(3)	0.004(3)	0.002(3)
C(42)	4a	0.176(1)	0.1723(9)	1.0686(3)	0.070(6)	0.098(7)	0.041(4)	−0.033(5)	−0.003(4)	−0.021(4)
C(44)	4a	0.248(1)	0.083(1)	1.0384(3)	0.091(7)	0.131(9)	0.066(6)	−0.036(6)	0.017(5)	−0.056(6)
C(52)	4a	−0.0451(6)	0.0236(5)	1.1802(2)	0.033(3)	0.034(3)	0.050(4)	0.002(3)	0.009(3)	−0.002(3)
C(54)	4a	−0.1796(7)	−0.0037(7)	1.1603(2)	0.039(4)	0.055(4)	0.056(4)	−0.012(3)	−0.004(3)	−0.003(3)
C(61)	4a	0.0817(7)	0.1868(5)	1.2721(2)	0.039(4)	0.032(3)	0.038(3)	0.004(3)	0.003(3)	0.002(3)
C(63)	4a	0.1471(7)	0.0002(5)	1.3036(2)	0.039(4)	0.034(3)	0.051(4)	−0.010(3)	−0.012(3)	0.006(3)
C(64)	4a	0.2523(8)	−0.0967(7)	1.3008(3)	0.061(5)	0.043(4)	0.107(7)	0.015(4)	0.001(5)	0.025(4)
C(321)	4a	0.3215(7)	0.6010(5)	1.1141(2)	0.040(3)	0.031(3)	0.036(3)	0.005(3)	−0.004(3)	−0.001(2)
C(322)	4a	0.4515(7)	0.6410(7)	1.1088(2)	0.039(4)	0.044(4)	0.055(4)	−0.001(3)	0.010(3)	−0.006(3)
C(323)	4a	0.4914(8)	0.7533(6)	1.1223(3)	0.041(4)	0.041(4)	0.076(5)	−0.007(3)	0.000(4)	−0.002(3)
C(324)	4a	0.3982(8)	0.8269(6)	1.1422(2)	0.061(5)	0.038(4)	0.052(4)	0.005(3)	−0.007(3)	0.004(3)
C(325)	4a	0.2651(8)	0.7906(6)	1.1482(3)	0.053(5)	0.043(4)	0.058(4)	0.014(3)	0.001(4)	−0.004(3)
C(326)	4a	0.2281(8)	0.6765(6)	1.1333(2)	0.044(4)	0.043(4)	0.047(4)	−0.002(3)	0.006(3)	−0.002(3)
C(331)	4a	0.3204(7)	0.5463(6)	1.0150(2)	0.049(4)	0.046(4)	0.035(3)	−0.007(3)	0.001(3)	0.005(3)
C(332)	4a	0.4188(8)	0.5512(7)	0.9815(2)	0.062(5)	0.054(4)	0.042(4)	0.008(4)	0.005(3)	0.011(3)
C(333)	4a	0.4202(8)	0.6463(8)	0.9507(2)	0.052(4)	0.082(6)	0.041(4)	0.002(4)	0.006(3)	0.009(4)
C(334)	4a	0.3238(7)	0.7347(7)	0.9526(2)	0.040(4)	0.074(5)	0.043(4)	−0.012(4)	−0.004(3)	0.008(3)
C(335)	4a	0.2209(8)	0.7229(7)	0.9843(3)	0.045(4)	0.061(5)	0.059(5)	0.008(4)	−0.002(3)	0.014(4)
C(336)	4a	0.2193(8)	0.6298(7)	1.0152(2)	0.042(4)	0.058(4)	0.042(4)	0.001(4)	0.007(3)	0.013(3)
C(351)	4a	0.5510(7)	0.2448(6)	1.0983(2)	0.047(4)	0.038(4)	0.047(4)	0.001(3)	0.008(3)	−0.002(3)
C(353)	4a	0.627(1)	0.1891(9)	1.0592(3)	0.094(7)	0.097(7)	0.056(5)	0.055(6)	0.019(5)	−0.001(5)
O(1)	4a	0.2365(4)	0.3190(3)	1.2368(1)	0.034(2)	0.036(2)	0.033(2)	−0.005(2)	0.001(2)	0.005(2)
O(21)	4a	0.3819(4)	0.4628(4)	1.2124(1)	0.030(2)	0.039(2)	0.045(2)	−0.003(2)	−0.004(2)	−0.007(2)
O(23)	4a	0.2430(5)	0.6005(4)	1.2437(2)	0.041(3)	0.050(3)	0.058(3)	0.002(2)	0.002(2)	−0.013(2)
O(41)	4a	0.2205(5)	0.1564(4)	1.1128(2)	0.055(3)	0.049(3)	0.042(2)	−0.007(2)	0.006(2)	−0.009(2)
O(43)	4a	0.0959(8)	0.2438(8)	1.0572(2)	0.111(6)	0.125(6)	0.055(4)	0.012(5)	−0.029(4)	−0.004(4)
O(51)	4a	−0.0106(4)	0.1387(4)	1.1741(1)	0.035(2)	0.030(2)	0.051(2)	−0.007(2)	−0.004(2)	0.003(2)
O(53)	4a	0.0271(5)	−0.0477(4)	1.1995(2)	0.043(3)	0.039(3)	0.079(3)	0.000(2)	0.000(3)	0.005(2)
O(62)	4a	0.1821(4)	0.0935(4)	1.2773(1)	0.035(2)	0.040(2)	0.042(2)	0.002(2)	0.000(2)	0.011(2)
O(63)	4a	0.0432(5)	−0.0055(4)	1.3247(2)	0.047(3)	0.044(3)	0.049(3)	−0.011(2)	0.002(2)	0.006(2)
O(352)	4a	0.5773(5)	0.2196(4)	1.1380(2)	0.050(3)	0.052(3)	0.049(3)	0.005(2)	−0.002(2)	0.002(2)
N(31)	4a	0.3578(5)	0.3824(4)	1.1198(2)	0.036(3)	0.035(3)	0.041(3)	0.002(2)	0.006(2)	0.005(2)
N(33)	4a	0.3277(6)	0.4575(5)	1.0492(2)	0.054(3)	0.041(3)	0.036(3)	0.007(3)	0.004(3)	0.003(2)
N(34)	4a	0.4318(6)	0.3768(5)	1.0468(2)	0.052(3)	0.032(3)	0.043(3)	0.004(3)	0.003(2)	0.002(2)
Cl(2)	4a	0.4470(2)	0.9681(2)	1.1593(1)	0.076(2)	0.043(1)	0.137(2)	0.003(1)	−0.018(2)	−0.024(1)
Br(1)	4a	0.3360(1)	0.86684(9)	0.91351(3)	0.0642(6)	0.0867(7)	0.0787(6)	−0.0110(5)	−0.0078(5)	0.0452(5)

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