# 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-$\beta$-D-glucose, $C_{30}H_{31}BrClN_3O_{10}$

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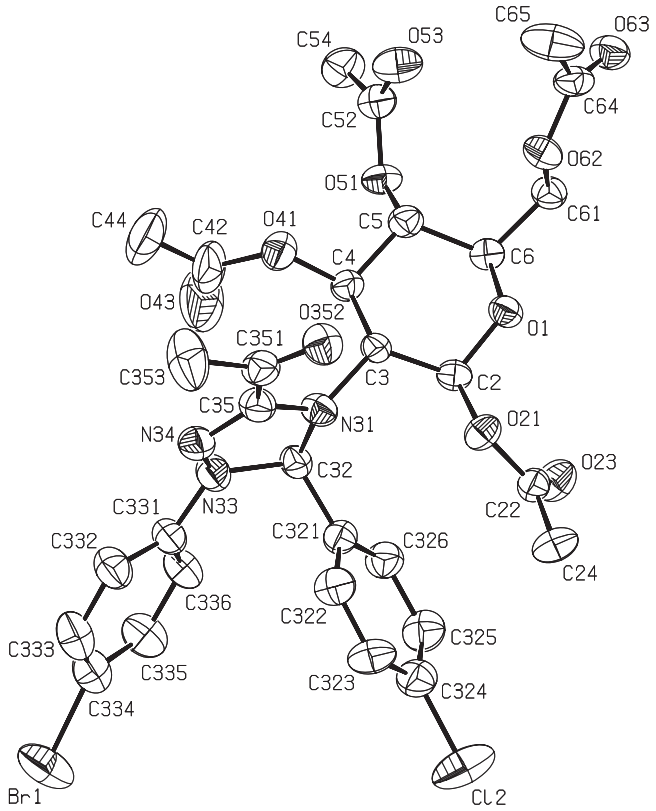
Received August 22, 2006, accepted and available on-line November 19, 2006; CCDC no. 1267/1878




**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]-$\beta$-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).

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**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 $K_\alpha$ radiation (1.54184 Å) |
| $\mu$: | 29.99 cm$^{-1}$ |
| Diffractometer, scan mode: | Nonius CAD4, $\omega$ |
| $2\theta_{max}$: | 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) | 4*a* | 0.095(6) | 0.294(5) | 1.134(2) | 0.03(2) |
| H(5) | 4*a* | 0.177(6) | 0.106(5) | 1.193(2) | 0.02(1) |
| H(6) | 4*a* | 0.050(7) | 0.311(6) | 1.224(2) | 0.04(2) |
| H(32) | 4*a* | 0.177(7) | 0.467(5) | 1.102(2) | 0.04(2) |
| H(32A) | 4*a* | 0.510(7) | 0.591(6) | 1.100(2) | 0.04(2) |
| H(32B) | 4*a* | 0.581(8) | 0.773(6) | 1.123(2) | 0.05(2) |
| H(32C) | 4*a* | 0.197(7) | 0.841(6) | 1.163(2) | 0.05(2) |

**Table 2.** Continued.

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

*Acknowledgments.* The authors are grateful for Deutsche Forschung Gemeinschaft (DFG) and Deutscher Akademischer Austauschdienst (DAAD) for granting a fellowship to R. J. A. and Y. B., respectively.

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