Crystal structure of methyl-2-hydroxy-2-phenyl-3-(4-bromophenyl)-3-phenylamino-propanoate, $C_{22}H_{20}BrNO_3$

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isomer of the β -amino- α -hydroxyester and shows a torsion angle $O1-C2-C3-N$ of 60.6° . The environment of the amino N atom is not planar. The N atom is lying $0.23 \text{ \AA}$ out of plane of its three substituents. In the crystal the molecules are stacked in columns parallel to b axis. Weak intra- and inter-molecular hydrogen bonds are present.

Table 1. Data collection and handling.

Crystal:	colorless needle, size $0.1 \times 0.1 \times 0.6 \text{ mm}$
Wavelength:	Cu $K\alpha$ radiation ($1.54178 \text{ \AA}$)
μ :	30.26 cm^{-1}
Diffractionmeter, scan mode:	Bruker P4, $\theta/2\theta$
$2\theta_{\text{max}}$:	113.98°
$N(hkl)_{\text{measured}}, N(hkl)_{\text{unique}}$:	2753, 2650
Criterion for $I_{\text{obs}}, N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2130
$N(\text{param})_{\text{refined}}$:	259
Programs:	SIR97 [2], SHELXL-97 [3], SHELXTL [4]

Abstract

$C_{22}H_{20}BrNO_3$, monoclinic, $P12_1/c1$ (no. 14), $a = 17.643(1) \text{ \AA}$, $b = 5.935(1) \text{ \AA}$, $c = 18.782(1) \text{ \AA}$, $\beta = 90.13(1)^\circ$, $V = 1966.7 \text{ \AA}^3$, $Z = 4$, $R_{\text{gt}}(F) = 0.041$, $wR_{\text{ref}}(F^2) = 0.117$, $T = 293 \text{ K}$.

Source of material

The compound was prepared by a condensation of three components (methyl phenylglyoxylate, aniline and aromatic aldehyde) promoted by $TiCl_3$ /pyridine in anhydrous THF at room temperature. The details of the stereoselective synthesis and the proposed mechanism of reaction have already been described [1]. Colorless crystals were obtained by crystallization from a diethyl ether/hexane solution.

Experimental details

The hydrogen atoms being chemically relevant were refined isotropically, whereas the rest was treated as riding model.

Discussion

The crystal structure of the title compound was determined in order to understand the stereochemical feature of the two asymmetric centres at C2 and C3. The molecule was found to be the syn

Table 2. Atomic coordinates and displacement parameters (in $\text{\AA}^2$).

Atom	Site	x	y	z	U_{iso}
H(1N)	4e	0.132(2)	0.056(6)	-0.272(2)	0.04(1)
H(1O)	4e	0.230(3)	0.297(8)	-0.346(3)	0.07(2)
H(3)	4e	0.230(2)	-0.267(5)	-0.270(2)	0.030(8)
H(41)	4e	0.1768	-0.4924	-0.4744	0.076
H(42)	4e	0.2075	-0.2716	-0.5100	0.076
H(43)	4e	0.1268	-0.2740	-0.4753	0.076
H(6)	4e	0.3715	0.2925	-0.2910	0.058
H(7)	4e	0.5007	0.2482	-0.3007	0.065
H(8)	4e	0.5505	-0.0764	-0.3490	0.067
H(9)	4e	0.4707	-0.3632	-0.3826	0.071
H(10)	4e	0.3412	-0.3231	-0.3720	0.065
H(12)	4e	0.3363	-0.2980	-0.1865	0.054
H(13)	4e	0.4002	-0.1826	-0.0849	0.062
H(15)	4e	0.2661	0.3607	-0.0733	0.068
H(16)	4e	0.2016	0.2391	-0.1730	0.062
H(18)	4e	0.1602	-0.4151	-0.1779	0.068
H(19)	4e	0.0780	-0.5640	-0.0958	0.085
H(20)	4e	-0.0348	-0.3903	-0.0715	0.095
H(21)	4e	-0.0671	-0.0688	-0.1327	0.091
H(22)	4e	0.0133	0.0832	-0.2159	0.071

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Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> ₁₁	<i>U</i> ₂₂	<i>U</i> ₃₃	<i>U</i> ₁₂	<i>U</i> ₁₃	<i>U</i> ₂₃
Br	4e	0.39235(3)	0.20478(9)	0.01098(2)	0.0904(4)	0.1001(4)	0.0440(3)	0.0020(3)	−0.0082(2)	−0.0156(2)
N	4e	0.1418(2)	−0.0653(6)	−0.2603(2)	0.041(2)	0.049(2)	0.052(2)	0.008(1)	0.003(1)	0.013(2)
O(1)	4e	0.2392(2)	0.2566(4)	−0.3132(2)	0.062(2)	0.042(2)	0.050(2)	0.008(1)	0.002(1)	0.007(1)
O(2)	4e	0.2102(1)	−0.2628(4)	−0.4046(1)	0.059(2)	0.053(2)	0.044(1)	0.003(1)	−0.007(1)	0.001(1)
O(3)	4e	0.1945(1)	0.0972(5)	−0.4362(1)	0.060(2)	0.062(2)	0.052(1)	0.003(1)	−0.008(1)	0.017(1)
C(1)	4e	0.2165(2)	−0.0435(6)	−0.3946(2)	0.036(2)	0.056(2)	0.041(2)	0.001(2)	0.004(1)	0.008(2)
C(2)	4e	0.2563(2)	0.0260(5)	−0.3249(2)	0.043(2)	0.041(2)	0.041(2)	0.004(2)	0.004(1)	0.005(1)
C(3)	4e	0.2232(2)	−0.1054(6)	−0.2604(2)	0.037(2)	0.040(2)	0.041(2)	0.007(1)	0.005(1)	0.001(2)
C(4)	4e	0.1776(2)	−0.3309(7)	−0.4717(2)	0.077(3)	0.065(3)	0.049(2)	−0.002(2)	−0.008(2)	−0.005(2)
C(5)	4e	0.3420(2)	−0.0088(5)	−0.3311(2)	0.046(2)	0.044(2)	0.030(2)	−0.004(2)	0.003(1)	0.006(1)
C(6)	4e	0.3909(2)	0.1596(6)	−0.3097(2)	0.054(2)	0.052(2)	0.039(2)	0.001(2)	0.004(2)	−0.004(2)
C(7)	4e	0.4686(2)	0.1335(7)	−0.3158(2)	0.054(2)	0.063(2)	0.045(2)	−0.015(2)	−0.003(2)	0.000(2)
C(8)	4e	0.4984(2)	−0.0606(7)	−0.3439(2)	0.044(2)	0.070(3)	0.053(2)	−0.003(2)	0.005(2)	0.007(2)
C(9)	4e	0.4508(2)	−0.2302(7)	−0.3643(2)	0.044(2)	0.058(2)	0.077(3)	0.004(2)	0.007(2)	−0.009(2)
C(10)	4e	0.3730(2)	−0.2060(6)	−0.3580(2)	0.048(2)	0.046(2)	0.069(2)	−0.002(2)	0.003(2)	−0.005(2)
C(11)	4e	0.2632(2)	−0.0387(5)	−0.1919(2)	0.041(2)	0.038(2)	0.035(2)	0.005(1)	0.008(1)	0.004(1)
C(12)	4e	0.3222(2)	−0.1648(6)	−0.1641(2)	0.046(2)	0.047(2)	0.042(2)	0.012(2)	0.004(2)	−0.002(2)
C(13)	4e	0.3609(2)	−0.0959(7)	−0.1033(2)	0.048(2)	0.066(2)	0.041(2)	0.014(2)	−0.001(2)	0.003(2)
C(14)	4e	0.3400(2)	0.1025(6)	−0.0708(2)	0.055(2)	0.061(2)	0.032(2)	0.001(2)	0.009(1)	0.001(2)
C(15)	4e	0.2805(2)	0.2285(6)	−0.0962(2)	0.081(3)	0.047(2)	0.043(2)	0.013(2)	0.008(2)	−0.005(2)
C(16)	4e	0.2424(2)	0.1554(6)	−0.1561(2)	0.059(2)	0.048(2)	0.048(2)	0.017(2)	0.001(2)	0.001(2)
C(17)	4e	0.0953(2)	−0.1515(6)	−0.2067(2)	0.039(2)	0.047(2)	0.044(2)	−0.002(2)	−0.000(1)	−0.005(2)
C(18)	4e	0.1141(2)	−0.3444(6)	−0.1695(2)	0.045(2)	0.056(2)	0.070(2)	0.000(2)	0.006(2)	0.013(2)
C(19)	4e	0.0650(2)	−0.4326(8)	−0.1199(2)	0.065(3)	0.075(3)	0.071(3)	−0.012(2)	0.002(2)	0.019(2)
C(20)	4e	−0.0022(3)	−0.3308(9)	−0.1055(2)	0.063(3)	0.110(4)	0.066(3)	−0.019(3)	0.018(2)	0.010(3)
C(21)	4e	−0.0211(2)	−0.1389(9)	−0.1419(2)	0.045(2)	0.106(4)	0.077(3)	0.005(2)	0.018(2)	−0.014(3)
C(22)	4e	0.0269(2)	−0.0479(7)	−0.1920(2)	0.043(2)	0.069(3)	0.065(2)	0.010(2)	0.004(2)	−0.003(2)

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