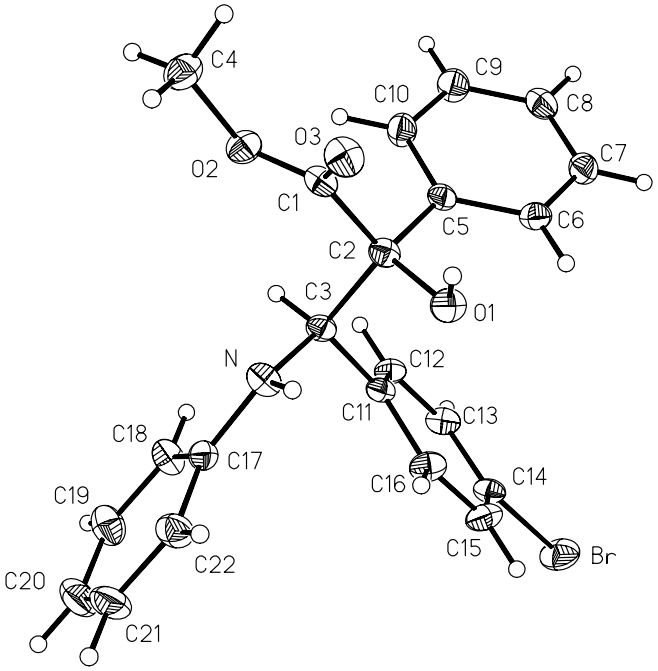
Crystal structure of methyl-2-hydroxy-2-phenyl-3-(4-bromophenyl)-3-phenylamino-propanoate, $C_{22}H_{20}BrNO_3$

L. Malpezzi*

Politecnico di Milano, Dipartimento di Chimica, Materiali e Ingegneria Chimica ″G. Natta″, Via Mancinelli 7, I-20131 Milano, Italy

Received March 12, 2004, accepted and available on-line May 4, 2004; CCDC no. 1267/1273



Abstract

$C_{22}H_{20}BrNO_3$, monoclinic, $P12_1/c1$ (no. 14),
a = 17.643(1) Å, b = 5.935(1) Å, c = 18.782(1) Å,
β = 90.13(1)°, V = 1966.7 Å^3, Z = 4,
$R_{gt}(F)$ = 0.041, $wR_{ref}(F^2)$ = 0.117, T = 293 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* 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 Å 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 × 0.1 × 0.6 mm
Wavelength:	Cu K_α radiation (1.54178 Å)
μ:	30.26 cm^{-1}
Diffractometer, scan mode:	Bruker P4, $\theta/2\theta$
$2\theta_{max}$:	113.98°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	2753, 2650
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2130
$N(param)_{refined}$:	259
Programs:	SIR97 [2], SHELXL-97 [3], SHELXTL [4]

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

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

* e-mail: luciana.malpezzi@polimi.it

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}
Br	4*e*	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	4*e*	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)	4*e*	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)	4*e*	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)	4*e*	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)	4*e*	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)	4*e*	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)	4*e*	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)	4*e*	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)	4*e*	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)	4*e*	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)	4*e*	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)	4*e*	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)	4*e*	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)	4*e*	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)	4*e*	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)	4*e*	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)	4*e*	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)	4*e*	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)	4*e*	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)	4*e*	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)	4*e*	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)	4*e*	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)	4*e*	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)	4*e*	–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)	4*e*	–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)	4*e*	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)

References

1. Clerici, A.; Clerici, L.; Porta, O.: A tricomponent reaction promoted by a titanium trichloride/pyridine system. Diastereoselective synthesis of β-amino-α-hydroxyesters. Tetrahedron Lett. **36** (1995) 5955-5958.
2. Altomare, A.; Burla, M. C.; Camalli, M.; Cascarano, G. L.; Giacovazzo, C.; Guagliardi, A.; Moliterni, A. G. G.; Polidori, P.; Spagna, R.: SIR97: a new tool for crystal structure determination and refinement. J. Appl. Crystallogr. **32** (1999) 115-119.
3. Sheldrick. G. M.: SHELXL-97. Program for the Refinement of Crystal Structures. University of Göttingen, Germany 1997.
4. Sheldrick, G. M.: SHELXTL/PC. Release 5.0. Siemens Analytical X-ray Instruments, Inc., Madison, Wisconsin, USA 1996.