Crystal structure of di(1*R*,4*S*)-1,7,7′-trimethyl-3-bromo-bicyclo[2.2.1]hept-2-ene carboxylate, $(C_{10}H_{14}Br)_2CO_3$

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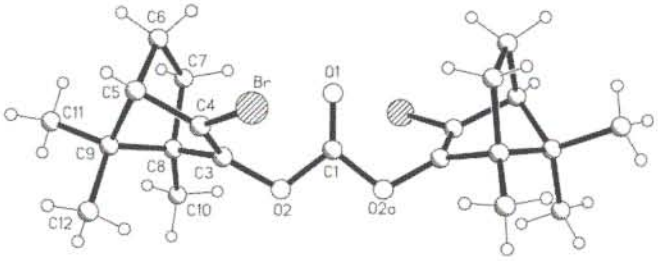

Abstract

$C_{21}H_{28}Br_2O_3$, tetragonal, $P4_122$ (No. 92), a = 7.848(1) Å, c = 34.945(7) A, V = 2152.3 Å^3, Z = 4, $R_{gt}(F)$ = 0.061, $wR(F)$ = 0.054, T = 293 K.

Source of material

The title compound was prepared by stirring a solution of (+)-*endo*-3-bromocamphor and *t*-BuOK at room temperature in THF for 3 hours. The mixture was added to 2 eq. of trifluoromethanesulphonic anhydride at 273 K, 1M aqueous HCl, and extracted with Et_2O. The combined organic layers were washed with water and brine. Rotoevaporation of the solvent left an oily residue which was purified by flash-chromatography (eluant hexane). The product partially crystallized on standing.

Table 1. Data collection and handling.

Crystal:	colourless plate, size 0.12 × 0.4 × 0.45 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	37.80 cm^{-1}
Diffractometer, scan mode:	Bruker AXS P4, ω
$2\theta_{max}$:	55°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	7203, 2469
Criterion for F_{obs}, $N(hkl)_{gt}$:	$F_{obs} > 3\ \sigma(F_{obs})$, 1750
$N(param)_{refined}$:	121
Program:	SHELXTL-plus [1]

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

Atom	Site	x	y	z	U_{iso}
H(5)	8*b*	1.1169(7)	0.2599(7)	−0.0258(2)	0.08
H(6A)	8*b*	0.8953(8)	0.2459(8)	−0.0711(2)	0.08
H(6B)	8*b*	0.8093(8)	0.3291(8)	−0.0352(2)	0.08
H(7A)	8*b*	0.8533(8)	0.4856(8)	−0.1037(2)	0.08
H(7B)	8*b*	0.7728(8)	0.5708(8)	−0.0674(2)	0.08
H(10A)	8*b*	1.0807(9)	0.7187(8)	−0.1221(2)	0.08
H(10B)	8*b*	1.1786(9)	0.8127(8)	−0.0892(2)	0.08
H(10C)	8*b*	0.9817(9)	0.8390(8)	−0.0940(2)	0.08
H(11A)	8*b*	1.0797(9)	0.3203(8)	−0.1132(2)	0.08
H(11B)	8*b*	1.2627(9)	0.2618(8)	−0.1009(2)	0.08
H(11C)	8*b*	1.2390(9)	0.4294(8)	−0.1247(2)	0.08
H(12A)	8*b*	1.3439(7)	0.5858(7)	−0.0322(2)	0.08
H(12B)	8*b*	1.3988(7)	0.5913(7)	−0.0753(2)	0.08
H(12C)	8*b*	1.4225(7)	0.4236(7)	−0.0515(2)	0.08

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	8*b*	1.01786(9)	0.48067(9)	0.04509(1)	0.0884(5)	0.1090(6)	0.0468(3)	0.0112(4)	0.0077(3)	0.0066(3)
O(1)	4*a*	0.7169(5)	0.7169(5)	0	0.060(2)	0.060(2)	0.107(5)	−0.002(3)	0.541(2)	−0.009(2)
C(1)	4*a*	0.8224(8)	0.8224(8)	0	0.053(3)	0.053(3)	0.068(5)	0.007(4)	0.478(3)	−0.002(3)
O(2)	8*b*	0.9829(4)	0.8091(5)	−0.0137(1)	0.057(2)	0.060(2)	0.073(2)	0.006(2)	0.012(2)	−0.011(2)
C(3)	8*b*	1.0248(7)	0.6488(7)	−0.0270(1)	0.049(3)	0.061(3)	0.047(3)	0.006(3)	0.003(3)	−0.004(3)
C(4)	8*b*	1.0509(6)	0.5099(7)	−0.0078(1)	0.055(3)	0.072(4)	0.043(2)	0.001(3)	0.000(2)	0.005(3)
C(5)	8*b*	1.0715(7)	0.3644(7)	−0.0357(2)	0.063(4)	0.060(3)	0.052(3)	0.015(3)	0.003(3)	0.001(3)
C(6)	8*b*	0.8961(8)	0.3426(8)	−0.0543(2)	0.082(5)	0.074(4)	0.067(4)	−0.008(3)	−0.014(4)	−0.014(4)
C(7)	8*b*	0.8691(8)	0.5085(8)	−0.0769(2)	0.074(4)	0.078(4)	0.060(3)	0.006(4)	−0.016(3)	−0.009(4)
C(8)	8*b*	1.0403(7)	0.6087(7)	−0.0697(1)	0.063(4)	0.058(3)	0.044(3)	0.012(3)	−0.004(3)	0.000(2)
C(9)	8*b*	1.1730(7)	0.4627(7)	−0.0673(1)	0.069(4)	0.063(4)	0.041(3)	0.010(3)	0.001(3)	0.001(3)
C(10)	8*b*	1.0734(9)	0.7586(8)	−0.0962(2)	0.095(5)	0.081(5)	0.062(3)	0.014(4)	0.004(4)	0.010(4)
C(11)	8*b*	1.1903(9)	0.3585(8)	−0.1052(2)	0.108(6)	0.097(5)	0.060(3)	0.022(4)	0.000(4)	−0.023(4)
C(12)	8*b*	1.3511(7)	0.5213(7)	−0.0555(2)	0.064(3)	0.099(5)	0.072(3)	0.020(4)	0.015(3)	−0.001(4)

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Reference

1. Sheldrick, G. M.: Program Package SHELXTL-plus. Release 4.1. Siemens Analytical X-Ray Instruments Inc. Madison (WI53719) USA 1990.