Crystal structure of (*E*)-1,2-di-(2-bromo-5-*tert*-butyl-3,4-dimethoxyphenyl)ethene, $\{C_6H(OCH_3)_2[C(CH_3)_3]Br\}_2C_2H_2$

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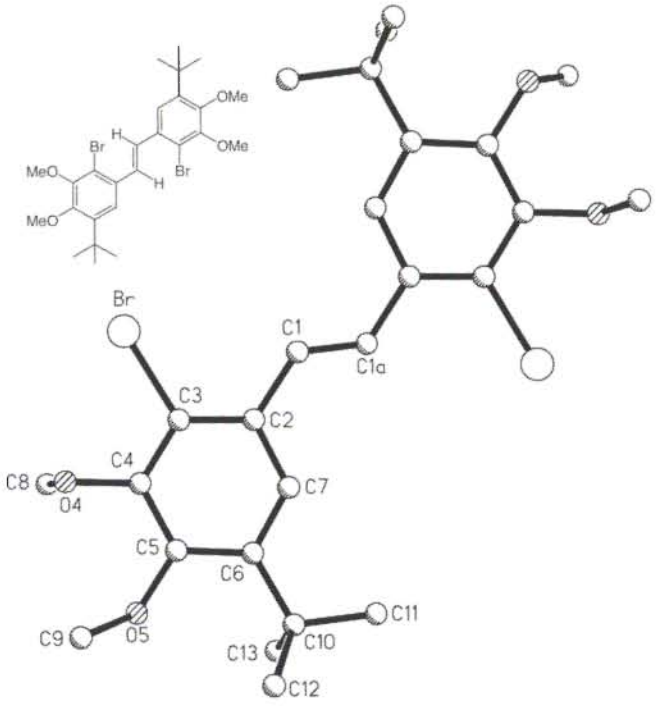

Abstract

$C_{26}H_{34}Br_2O_4$, monoclinic, $P12_1/c1$ (No. 14), $a = 9.657(1)$ Å, $b = 13.298(1)$ Å, $c = 10.654(1)$ Å, $\beta = 106.21(1)°$, $V = 1313.8$ Å^3, $Z = 2$, $R_{gt}(F) = 0.051$, $w\tilde{R}(F) = 0.047$, $T = 293$ K.

Source of material

The title compound, an intermediate for a new atroposelective approach for biaryl synthesis [1], was synthesized by oxidative 'dimerization' of the corresponding phosphonium periodate (prepared from the methyl derivative [2]), along with the (*Z*)-isomer.

Table 1. Data collection and handling.

Crystal:	pale yellow plate, size 0.75 × 0.85 × 0.25 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	31.10 cm^{-1}
Diffractometer, scan mode:	Siemens P4, ω
$2\theta_{max}$:	55°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	3100, 2801
Criterion for F_{obs}, $N(hkl)_{gt}$:	$F_{obs} > 3\ \sigma(F_{obs})$, 2261
$N(param)_{refined}$:	145
Program:	SHELXTL-plus [3]

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

Atom	Site	*x*	*y*	*z*	U_{iso}
H(1)	4*e*	0.35844	0.52778	–0.00775	0.08
H(7)	4*e*	0.6874(4)	0.4374(3)	0.2098(4)	0.08
H(8A)	4*e*	0.1365(6)	0.2133(4)	0.3593(6)	0.08
H(8B)	4*e*	0.2999(6)	0.1902(4)	0.3764(6)	0.08
H(8C)	4*e*	0.1986(6)	0.2180(4)	0.2382(6)	0.08
H(9A)	4*e*	0.4835(6)	0.3016(4)	0.6854(4)	0.08
H(9B)	4*e*	0.3649(6)	0.3524(4)	0.5718(4)	0.08
H(9C)	4*e*	0.5125(6)	0.4070(4)	0.6318(4)	0.08
H(11A)	4*e*	0.8828(5)	0.3542(4)	0.2939(5)	0.08
H(11B)	4*e*	0.9920(5)	0.3668(4)	0.4328(5)	0.08
H(11C)	4*e*	0.8911(5)	0.4563(4)	0.3692(5)	0.08
H(12A)	4*e*	0.7441(6)	0.3754(4)	0.6182(4)	0.08
H(12B)	4*e*	0.8081(6)	0.4690(4)	0.5645(4)	0.08
H(12C)	4*e*	0.9090(6)	0.3795(4)	0.6280(4)	0.08
H(13A)	4*e*	0.7320(6)	0.2071(4)	0.4964(5)	0.08
H(13B)	4*e*	0.8976(6)	0.2177(4)	0.5118(5)	0.08
H(13C)	4*e*	0.7898(6)	0.2024(4)	0.3728(5)	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	4*e*	0.15860(5)	0.44227(4)	0.10101(5)	0.0357(3)	0.0735(3)	0.0586(3)	0.0064(3)	0.0059(2)	0.0090(3)
C(1)	4*e*	0.4514(5)	0.4956(3)	0.0326(4)	0.043(2)	0.051(2)	0.042(2)	0.005(2)	0.010(2)	0.008(2)
C(2)	4*e*	0.4678(5)	0.4432(3)	0.1573(4)	0.041(2)	0.040(2)	0.041(2)	0.002(2)	0.012(2)	0.003(2)
C(3)	4*e*	0.3517(5)	0.4155(3)	0.2005(4)	0.032(2)	0.043(2)	0.042(2)	0.004(2)	0.008(2)	0.002(2)

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Table 3. Continued.

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(4)	4*e*	0.3702(5)	0.3633(3)	0.3165(4)	0.040(2)	0.041(2)	0.044(2)	–0.001(2)	0.018(2)	0.003(2)
O(4)	4*e*	0.2521(3)	0.3333(2)	0.3556(3)	0.038(2)	0.058(2)	0.066(2)	–0.000(2)	0.026(2)	0.009(2)
C(5)	4*e*	0.5093(5)	0.3453(3)	0.3978(4)	0.043(3)	0.037(2)	0.039(2)	0.003(2)	0.011(2)	0.002(2)
O(5)	4*e*	0.5247(4)	0.2942(2)	0.5135(3)	0.054(2)	0.053(2)	0.044(2)	0.010(2)	0.018(1)	0.011(1)
C(6)	4*e*	0.6312(5)	0.3708(3)	0.3577(4)	0.033(2)	0.039(2)	0.041(2)	0.001(2)	0.010(2)	–0.001(2)
C(7)	4*e*	0.6054(4)	0.4191(3)	0.2390(4)	0.033(2)	0.047(2)	0.041(2)	0.001(2)	0.012(2)	0.004(2)
C(8)	4*e*	0.2192(6)	0.2304(4)	0.3304(6)	0.063(4)	0.066(4)	0.125(5)	–0.016(3)	0.044(4)	0.010(3)
C(9)	4*e*	0.4668(6)	0.3429(4)	0.6087(4)	0.085(4)	0.086(4)	0.044(3)	0.015(3)	0.028(3)	0.011(3)
C(10)	4*e*	0.7839(5)	0.3468(3)	0.4413(4)	0.038(2)	0.058(3)	0.046(2)	0.007(2)	0.009(2)	0.001(2)
C(11)	4*e*	0.8980(5)	0.3845(4)	0.3785(5)	0.037(3)	0.093(4)	0.073(3)	0.001(3)	0.011(3)	0.007(3)
C(12)	4*e*	0.8142(6)	0.3974(4)	0.5756(4)	0.064(4)	0.084(4)	0.053(3)	–0.004(3)	–0.001(2)	–0.004(3)
C(13)	4*e*	0.8028(6)	0.2329(4)	0.4570(5)	0.058(4)	0.064(3)	0.092(4)	0.015(3)	0.004(3)	0.015(3)

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