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Crystal structure of (4aα,4bα,10aα,10bα)-2,3,4a,10b-tetrachloro-1,4-dioxo-1,4,4a,4b,5,6,7,8,9,10,10a,10b-dodecahydro-benzo[3,4]cyclobuta[1,2]cyclooctane, $(C_8H_{14})(C_6Cl_4O_2)$

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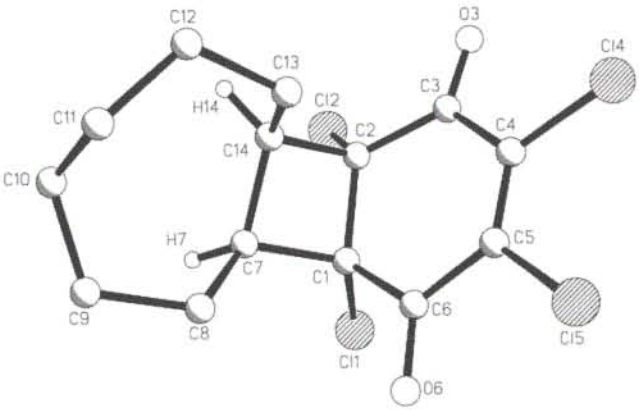

Abstract

$C_{14}H_{14}Cl_4O_2$, monoclinic, $P12_1/n1$ (No. 14), a = 12.717(2) Å, b = 9.2786(7) Å, c = 12.843(8) Å, β = 94.789(7)°, V = 1510.1 Å^3, Z = 4, $R_{gt}(F)$ = 0.059, $wR(F)$ = 0.063, T = 293 K.

Source of material

The title compound was prepared by irradiation of a solution of chloranil and cyclooctene in benzene at 293 K in a Rayonet photochemical reactor RPR-100 (λ = 350 nm) [1, 2].

Table 1. Data collection and handling.

Crystal:	colourless prism, size 0.4 × 0.5 × 0.6 mm
Wavelength:	Mo K_α radiation (0.71073 A)
μ:	7.80 cm^{-1}
Diffractometer, scan mode:	Siemens P4, ω
$2\theta_{max}$:	55°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	7293, 3469
Criterion for F_{obs}, $N(hkl)_{gt}$:	$F_{obs} > 3\,\sigma(F_{obs})$, 3026
$N(param)_{refined}$:	181
Program:	SHELXTL-plus [3]

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

Atom	Site	x	y	z	U_{iso}
H(7)	4e	0.4758(3)	0.1303(3)	0.1292(2)	0.08
H(8A)	4e	0.5966(3)	–0.0259(3)	0.1659(3)	0.08
H(8B)	4e	0.5696(3)	–0.0140(3)	0.2825(3)	0.08
H(9A)	4e	0.4285(4)	–0.0989(4)	0.1154(4)	0.08
H(9B)	4e	0.4736(4)	–0.1999(4)	0.2062(4)	0.08
H(10A)	4e	0.3025(4)	–0.1482(5)	0.2319(4)	0.08
H(10B)	4e	0.3252(4)	0.0174(5)	0.2246(4)	0.08
H(11A)	4e	0.3219(4)	–0.1214(5)	0.3958(3)	0.08
H(11B)	4e	0.4431(4)	–0.1073(5)	0.3838(3)	0.08
H(12A)	4e	0.3755(3)	0.0777(4)	0.4867(3)	0.08
H(12B)	4e	0.3090(3)	0.1328(4)	0.3863(3)	0.08
H(13A)	4e	0.4634(3)	0.2703(3)	0.4345(3)	0.08
H(13B)	4e	0.5299(3)	0.1377(3)	0.4023(3)	0.08
H(14)	4e	0.3818(2)	0.2509(3)	0.2711(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}
Cl(1)	4e	0.6523(1)	0.3199(1)	0.08102(8)	0.128(1)	0.0720(7)	0.0625(6)	–0.0175(6)	0.0418(6)	0.0006(5)
Cl(2)	4e	0.45853(9)	0.48969(9)	0.15844(8)	0.0915(7)	0.0441(5)	0.0760(6)	0.0021(4)	–0.0215(5)	0.0143(4)
Cl(4)	4e	0.69794(9)	0.4902(1)	0.52131(7)	0.0867(7)	0.0893(8)	0.0592(6)	0.0031(5)	–0.0066(5)	–0.0255(5)
Cl(5)	4e	0.82592(8)	0.2242(1)	0.44927(8)	0.0603(5)	0.0693(6)	0.0784(6)	0.0071(4)	–0.0064(5)	0.0154(5)
C(1)	4e	0.6033(3)	0.2705(3)	0.2008(2)	0.055(2)	0.042(1)	0.043(1)	–0.004(1)	0.009(1)	–0.002(1)
C(2)	4e	0.5282(2)	0.3766(3)	0.2516(2)	0.051(2)	0.035(1)	0.051(2)	0.001(1)	–0.001(1)	0.004(1)
C(3)	4e	0.5720(3)	0.4668(3)	0.3427(2)	0.053(2)	0.039(1)	0.054(2)	–0.000(1)	0.010(1)	–0.001(1)
O(3)	4e	0.5315(2)	0.5769(3)	0.3688(2)	0.078(2)	0.050(1)	0.080(2)	0.015(1)	0.006(1)	–0.013(1)
C(4)	4e	0.6660(3)	0.4062(3)	0.4055(2)	0.053(2)	0.047(2)	0.045(1)	–0.006(1)	0.004(1)	–0.003(1)
C(5)	4e	0.7210(2)	0.2942(3)	0.3746(2)	0.045(2)	0.045(2)	0.053(2)	–0.005(1)	0.003(1)	0.007(1)
C(6)	4e	0.6997(3)	0.2239(4)	0.2718(3)	0.048(2)	0.048(2)	0.066(2)	0.000(1)	0.011(2)	–0.010(2)
O(6)	4e	0.7568(2)	0.1300(4)	0.2451(3)	0.068(2)	0.095(2)	0.113(2)	0.027(2)	–0.001(2)	–0.043(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(7)	4*e*	0.5139(3)	0.1556(3)	0.1943(2)	0.055(2)	0.040(1)	0.046(2)	–0.002(1)	–0.000(1)	–0.001(1)
C(8)	4*e*	0.5427(3)	–0.0015(3)	0.2110(3)	0.067(2)	0.039(2)	0.077(2)	0.004(2)	0.014(2)	–0.004(2)
C(9)	4*e*	0.4500(4)	–0.1040(4)	0.1888(4)	0.094(3)	0.045(2)	0.102(3)	–0.014(2)	0.012(3)	–0.016(2)
C(10)	4*e*	0.3537(4)	–0.0738(5)	0.2482(4)	0.082(3)	0.081(3)	0.094(3)	–0.036(2)	–0.003(2)	–0.002(3)
C(11)	4*e*	0.3750(4)	–0.0656(5)	0.3654(3)	0.079(3)	0.081(3)	0.092(3)	–0.030(2)	0.004(2)	0.023(2)
C(12)	4*e*	0.3739(3)	0.0876(4)	0.4122(3)	0.071(2)	0.073(3)	0.067(2)	–0.013(2)	0.020(2)	0.009(2)
C(13)	4*e*	0.4645(3)	0.1887(3)	0.3887(3)	0.059(2)	0.050(2)	0.052(2)	–0.001(2)	0.008(1)	0.000(1)
C(14)	4*e*	0.4574(2)	0.2438(3)	0.2760(2)	0.042(2)	0.038(1)	0.056(2)	0.003(1)	0.004(1)	0.002(1)

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