Crystal structure of (1*R**, 2*R**, 8*R**, 9*S**, 12*R**)-2,9-diiodo-13-oxa-bicyclo[6.4.1]tridec-5-en-12-ol, C₁₂H₁₈I₂O₂

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Abstract

C₁₂H₁₈I₂O₂, triclinic, $P\bar{1}$ (No. 2), $a = 10.297(3)$ Å, $b = 11.775(3)$ Å, $c = 12.337(4)$ Å, $\alpha = 93.8(2)^\circ$, $\beta = 96.8(3)^\circ$, $\gamma = 104.6(2)^\circ$, $V = 1430.1$ Å³, $Z = 4$, $R_g(F) = 0.062$, $R_w(F^2) = 0.163$, $T = 298$ K.

Source of material

The compound was prepared using literature procedures [1–3].

Discussion

The molecular structure of this compound possess a nine-membered ring trans-fused with a seven-membered ring. Only a C=C bond is in cyclodecane part of the molecule, namely C5=C6; all the other C–C bond lengths have value in the single bond range with C4–C5 and C6–C7 being Csp²–Csp³ bonds, while the rest are Csp³–Csp³, Csp³–O and Csp³–I. In the seven-membered ring all distances and angles are normal.

Table 1. Data collection and handling.

Crystal:	colorless prism, size 0.16 x 0.14 x 0.12 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	43.84 cm ⁻¹
Diffractometer, scan mode:	Siemens R3m/V, ω
$2\theta_{\max}$:	50.1°
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$:	5265, 4816
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 3942
$N(\text{param})_{\text{refined}}$:	286
Programs:	SHELXS-86 [4], SHELXL-93 [5]

Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso}
H(4B)	2i	0.5830(6)	0.4862(5)	−0.2978(5)	0.068
H(3B)	2i	0.4814(7)	1.0810(5)	0.1880(5)	0.073
H(1)	2i	0.6458(7)	0.8299(6)	0.1648(6)	0.038
H(2)	2i	0.7375(8)	1.0468(7)	0.0739(7)	0.047
H(3A)	2i	0.7140(9)	0.9044(8)	−0.0717(7)	0.053
H(3B)	2i	0.8679(9)	0.9656(8)	−0.0343(7)	0.053
H(1)	2i	0.9017(9)	0.7958(8)	0.0291(7)	0.054
H(5)	2i	0.7567(9)	0.6255(8)	−0.0157(7)	0.053
H(6A)	2i	0.5570(9)	0.5998(7)	−0.1297(7)	0.051
H(6B)	2i	0.5715(9)	0.7360(7)	−0.1187(7)	0.051
H(7A)	2i	0.4096(8)	0.5849(7)	−0.0158(7)	0.047
H(7B)	2i	0.5315(8)	0.6404(7)	0.0765(7)	0.047
H(8)	2i	0.3590(7)	0.7587(6)	−0.0340(6)	0.037
H(6)	2i	0.3839(8)	0.6953(7)	0.1777(7)	0.043
H(10A)	2i	0.2604(9)	0.8316(8)	0.2361(7)	0.053
H(10B)	2i	0.3176(9)	0.9084(8)	0.1448(7)	0.053
H(11A)	2i	0.445(1)	0.9613(8)	0.3278(7)	0.057
H(11B)	2i	0.493(1)	0.8467(8)	0.3101(7)	0.057
H(12)	2i	0.6601(9)	1.0043(6)	0.2813(6)	0.043
H(21)	2i	0.8291(8)	0.4989(6)	−0.5021(6)	0.036
H(20)	2i	0.5790(8)	0.3209(7)	−0.5229(7)	0.046
H(19A)	2i	0.5984(9)	0.2667(8)	−0.7013(7)	0.055
H(19B)	2i	0.5835(9)	0.3957(8)	−0.6898(7)	0.055
H(18)	2i	0.814(1)	0.332(1)	−0.7410(7)	0.068
H(13)	2i	0.895(1)	0.510(1)	−0.7829(8)	0.072
H(16A)	2i	0.656(1)	0.5657(9)	−0.7430(7)	0.070
H(16B)	2i	0.769(1)	0.6428(9)	−0.8023(7)	0.070
H(14A)	2i	0.896(1)	0.6505(8)	−0.6000(7)	0.060

Table 2. Continued.

Atom	Site	x	y	z	U _{iso}
H(14B)	2i	0.846(1)	0.7553(8)	−0.6457(7)	0.060
H(15)	2i	0.6506(8)	0.7028(6)	−0.5726(6)	0.043
H(17)	2i	0.8926(8)	0.7479(6)	−0.4343(7)	0.044
H(24A)	2i	0.6554(6)	0.6753(5)	−0.3377(5)	0.047

Table 2. Continued.

Atom	Site	x	y	z	U _{iso}
H(24B)	2i	0.7871(6)	0.7618(5)	−0.2726(5)	0.047
H(23A)	2i	0.9019(6)	0.6089(5)	−0.3117(5)	0.051
H(23B)	2i	0.8046(6)	0.5851(5)	−0.2228(5)	0.051
H(22)	2i	0.7789(9)	0.4161(7)	−0.3368(6)	0.045

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

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
I(1)	2i	0.13856(6)	0.63730(6)	0.06184(6)	0.0446(4)	0.0502(4)	0.0738(5)	−0.0025(3)	0.0114(3)	0.0000(3)
I(2)	2i	0.92695(6)	1.00621(7)	0.22032(5)	0.0457(4)	0.0720(5)	0.0540(4)	−0.0058(3)	0.0031(3)	−0.0105(3)
I(3)	2i	0.78718(8)	0.22784(5)	−0.50253(6)	0.0822(5)	0.0346(4)	0.0714(5)	0.0316(3)	0.0079(3)	0.0032(3)
I(4)	2i	0.76615(8)	0.91720(5)	−0.45041(6)	0.0933(6)	0.0250(3)	0.0768(5)	0.0184(3)	−0.0062(4)	−0.0016(3)
O(131)	2i	0.5229(5)	0.8717(4)	0.0427(4)	0.043(3)	0.018(2)	0.031(3)	0.004(2)	0.007(2)	0.002(2)
O(132)	2i	0.6559(5)	0.5462(4)	−0.5277(4)	0.036(3)	0.023(2)	0.030(3)	0.007(2)	0.001(2)	−0.001(2)
O(4)	2i	0.5977(6)	0.4368(5)	−0.3414(5)	0.059(4)	0.036(3)	0.041(3)	0.009(3)	0.018(3)	0.004(2)
O(3)	2i	0.5576(7)	1.0771(5)	0.1782(5)	0.072(4)	0.026(3)	0.051(4)	0.018(3)	0.012(3)	−0.004(2)
C(11)	2i	0.6296(7)	0.9013(6)	0.1356(6)	0.035(4)	0.026(4)	0.032(4)	0.004(3)	0.005(3)	0.002(3)
C(21)	2i	0.7533(8)	0.9708(7)	0.0909(7)	0.045(5)	0.028(4)	0.039(4)	0.002(3)	0.005(3)	−0.001(3)
C(31)	2i	0.7883(9)	0.9132(8)	−0.0129(7)	0.042(5)	0.056(5)	0.039(5)	0.015(4)	0.014(4)	0.008(4)
C(41)	2i	0.8145(9)	0.7962(8)	−0.0008(7)	0.044(5)	0.057(6)	0.036(4)	0.018(4)	0.016(4)	−0.002(4)
C(51)	2i	0.7256(9)	0.6917(8)	−0.0286(7)	0.060(5)	0.047(5)	0.036(4)	0.033(4)	0.011(4)	0.003(4)
C(61)	2i	0.5814(9)	0.6701(7)	−0.0782(7)	0.060(5)	0.032(4)	0.034(4)	0.011(4)	0.007(4)	−0.008(3)
C(71)	2i	0.4837(8)	0.6545(7)	0.0082(7)	0.044(4)	0.026(4)	0.045(5)	0.006(3)	0.009(4)	0.000(3)
C(81)	2i	0.4242(7)	0.7588(6)	0.0307(6)	0.034(4)	0.018(3)	0.036(4)	0.001(3)	0.003(3)	0.005(3)
C(91)	2i	0.3433(8)	0.7423(7)	0.1278(7)	0.037(4)	0.028(4)	0.045(5)	0.007(3)	0.011(3)	0.010(3)
C(101)	2i	0.3355(9)	0.8517(8)	0.1942(7)	0.051(5)	0.042(5)	0.041(5)	0.012(4)	0.018(4)	0.006(4)
C(111)	2i	0.466(1)	0.9084(8)	0.2727(7)	0.064(6)	0.045(5)	0.033(5)	0.010(4)	0.016(4)	−0.003(4)
C(121)	2i	0.5838(9)	0.9765(6)	0.2221(6)	0.056(5)	0.024(4)	0.027(4)	0.008(3)	0.008(3)	0.001(3)
C(12)	2i	0.7358(8)	0.4712(6)	−0.4866(6)	0.041(4)	0.022(4)	0.032(4)	0.014(3)	0.006(3)	0.003(3)
C(22)	2i	0.6675(8)	0.3491(7)	−0.5475(7)	0.041(4)	0.026(4)	0.050(5)	0.014(3)	0.001(4)	−0.006(3)
C(32)	2i	0.6443(9)	0.3469(8)	−0.6709(7)	0.057(5)	0.032(4)	0.043(5)	0.011(4)	−0.005(4)	−0.009(3)
C(42)	2i	0.767(1)	0.387(1)	−0.7252(7)	0.071(6)	0.072(7)	0.032(5)	0.033(5)	0.010(4)	−0.014(4)
C(52)	2i	0.815(1)	0.496(1)	−0.7530(8)	0.075(7)	0.070(7)	0.040(5)	0.020(5)	0.025(5)	0.000(5)
C(62)	2i	0.753(1)	0.5954(9)	−0.7414(7)	0.096(8)	0.046(5)	0.031(5)	0.016(5)	0.012(5)	0.008(4)
C(72)	2i	0.816(1)	0.6739(8)	−0.6303(7)	0.063(6)	0.036(5)	0.046(5)	0.002(4)	0.010(4)	0.011(4)
C(82)	2i	0.7224(8)	0.6669(6)	−0.5445(6)	0.047(4)	0.023(4)	0.037(4)	0.008(3)	0.004(3)	0.010(3)
C(92)	2i	0.7955(8)	0.7415(6)	−0.4370(7)	0.044(4)	0.022(4)	0.043(5)	0.011(3)	0.003(3)	0.001(3)
C(102)	2i	0.7536(6)	0.6981(5)	−0.3310(5)	0.049(5)	0.028(4)	0.041(5)	0.012(3)	0.008(4)	−0.003(3)
C(112)	2i	0.8076(6)	0.5922(5)	−0.3004(5)	0.059(5)	0.037(5)	0.033(4)	0.019(4)	0.000(4)	−0.002(3)
C(122)	2i	0.7326(9)	0.4742(7)	−0.3633(6)	0.049(5)	0.029(4)	0.034(4)	0.010(3)	0.003(3)	0.005(3)

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