

Article

Supplementary Materials: Vibrational Analysis of Benziodoxoles and Benzi-dazolotetrazoles

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1. Single Crystal X-ray Crystallography of I-Hydroxybenziodoxolone

Data were collected on a Rigaku XtaLAB Synergy-*i* Kappa diffractometer equipped with a PhotonJet-*i* X-ray source operated at 50 W (50kV, 1 mA) to generate Cu K α radiation ($\lambda = 1.54178 \text{ \AA}$) and a HyPix-6000HE HPC detector. Crystals were transferred from the vial and placed on a glass slide in type NVH immersion oil by Cargille. A Zeiss Stemi 305 microscope was used to identify a suitable specimen for X-ray diffraction from a representative sample of the material. The crystal and a small amount of the oil were collected on a MiTeGen 50 micron MicroLoop and transferred to the instrument where it was placed under a cold nitrogen stream (Oxford 700 series) maintained at 100 K throughout the duration of the experiment. The sample was optically centered with the aid of a video camera to insure that no translations were observed as the crystal was rotated through all positions. A unit cell collection was then carried out. After it was determined that the unit cell was not present in the CCDC database, a data collection strategy was calculated by CrysAlis^{Pro}[1]. The crystal was measured for size, morphology, and color.

Refinement details. After data collection, the unit cell was re-determined using a subset of the full data collection. Intensity data were corrected for Lorentz, polarization, and background effects using the CrysAlis^{Pro}[1]. A numerical absorption correction was applied based on a Gaussian integration over a multifaceted crystal and followed by a semi-empirical correction for adsorption applied using the program SCALE3 ABSPACK[2]. The programs SHELXT[3] was used for the initial structure solution and SHELXL[4] was used for refinement of the structure. Both of these programs were utilized within the OLEX2 software [5]. Hydrogen atoms bound to carbon atoms were located in the difference Fourier map and were geometrically constrained using the appropriate AFIX commands. The hydrogen atom (H3) was constrained to ride on oxygen O3 with a constrained O-H bond length of 0.86(2) \AA .

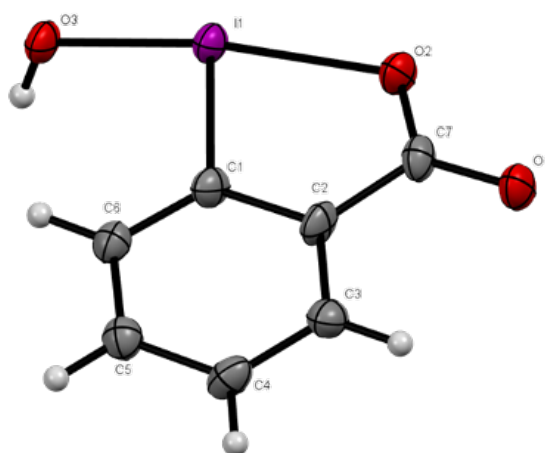


Figure S1. Displacement ellipsoid plot (50% probability level) of I-hydroxybenzidoxolone at 100.0(3)K. C, H, O, and I atoms are represented by gray, white, red, and purple ellipsoids, respectively.

Table S1. Crystal data and structure refinement.

Identification code	run21_02
Empirical formula	C ₇ H ₅ IO ₃
Formula weight	264.01
Temperature/K	100.0(3)
Crystal system	monoclinic
Space group	P2 ₁ /c
a/Å	12.8748(4)
b/Å	4.0112(2)
c/Å	14.0072(4)
$\alpha/^\circ$	90
$\beta/^\circ$	96.656(3)
$\gamma/^\circ$	90
Volume/Å ³	718.50(5)
Z	4
$\rho_{\text{calc}}/\text{g cm}^{-3}$	2.441
μ/mm^{-1}	34.652
F(000)	496
Crystal size/mm ³	0.151 × 0.036 × 0.017
Radiation	Cu K α (λ = 1.54184)
2 θ range for data collection/ $^\circ$	12.726 to 153.976
Index ranges	-16 ≤ h ≤ 16, -4 ≤ k ≤ 4, -16 ≤ l ≤ 17
Reflections collected	8790
Independent reflections	1437 [R_{int} = 0.0699, R_{sigma} = 0.0341]
Data/restraints/parameters	1437/1/104
Goodness-of-fit on F ²	1.098
Final R indexes [I ≥ 2 σ (I)]	R_1 = 0.0565, wR_2 = 0.1539
Final R indexes [all data]	R_1 = 0.0600, wR_2 = 0.1582
Largest diff. peak/hole / e Å ⁻³	2.07/-3.04

Table S2. Fractional Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters ($\text{\AA}^2 \times 10^3$). U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{IJ} tensor.

Atom	x	y	z	U(eq)
I1	881.1(2)	3742.2(10)	6489.1(2)	22.5(3)
O1	2323(4)	2774(15)	3909(3)	28.6(10)
O2	1165(3)	2251(14)	4975(3)	26.2(9)
O3	905(4)	5656(13)	7790(3)	26.6(11)
C1	2358(5)	5777(16)	6352(5)	23.3(13)
C2	2716(5)	5250(20)	5452(4)	25.0(13)
C3	3686(5)	6464(16)	5299(5)	26.1(15)
C4	4296(6)	8172(18)	6034(5)	27.9(14)
C5	3907(6)	8669(15)	6923(5)	26.1(16)
C6	2930(5)	7502(19)	7086(4)	24.0(12)
C7	2039(5)	3338(16)	4719(4)	23.9(14)

Table S3. Anisotropic Displacement Parameters ($\text{\AA}^2 \times 10^3$). The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11} + 2hka^*b^*U_{12} + \dots]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
I1	22.8(4)	28.6(5)	17.1(4)	0.65(11)	6.1(2)	1.19(12)
O1	30(2)	39(3)	18(2)	0(2)	5.5(16)	-2(2)
O2	26(2)	36(3)	18.6(19)	1(2)	7.4(16)	-2(2)
O3	28(2)	37(3)	16(2)	0.5(17)	8.4(17)	5.3(19)
C1	23(3)	31(4)	17(3)	5(2)	6(2)	2(2)
C2	28(3)	32(4)	17(3)	3(3)	11(2)	3(3)
C3	22(3)	32(4)	23(3)	2(2)	2(3)	3(2)
C4	25(3)	27(4)	34(4)	-1(3)	12(3)	-1(3)
C5	28(4)	27(4)	23(3)	0(2)	2(3)	1(2)
C6	27(3)	24(4)	21(3)	0(3)	6(2)	2(3)
C7	32(3)	25(4)	14(3)	1(2)	4(2)	2(2)

Table S4. Bond Lengths.

Atom	Atom	Length/ \AA	Atom	Atom	Length/ \AA
I1	O2	2.274(4)	C1	C6	1.379(9)
I1	O3	1.975(4)	C2	C3	1.381(9)
I1	C1	2.098(7)	C2	C7	1.481(9)
O1	C7	1.252(7)	C3	C4	1.400(10)
O2	C7	1.296(8)	C4	C5	1.409(10)
C1	C2	1.408(8)	C5	C6	1.386(10)

Table S5. Bond Angles.

Atom	Atom	Atom	Angle/ $^\circ$	Atom	Atom	Atom	Angle/ $^\circ$
O3	I1	O2	167.52(19)	C3	C2	C7	123.0(5)
O3	I1	C1	90.9(2)	C2	C3	C4	119.9(7)
C1	I1	O2	76.8(2)	C3	C4	C5	119.5(7)
C7	O2	I1	114.1(4)	C6	C5	C4	121.4(7)
C2	C1	I1	114.7(5)	C1	C6	C5	117.6(6)
C6	C1	I1	122.5(4)	O1	C7	O2	122.9(6)
C6	C1	C2	122.8(6)	O1	C7	C2	120.8(6)
C1	C2	C7	118.2(6)	O2	C7	C2	116.2(5)
C3	C2	C1	118.8(6)				

Table S6. Torsion Angles.

A	B	C	D	Angle/ $^{\circ}$	A	B	C	D	Angle/ $^{\circ}$
I1	O2	C7	O1	-179.6(5)	C2	C3	C4	C5	-0.8(10)
I1	O2	C7	C2	1.9(7)	C3	C2	C7	O1	-0.8(10)
I1	C1	C2	C3	-179.3(5)	C3	C2	C7	O2	177.7(7)
I1	C1	C2	C7	-0.5(8)	C3	C4	C5	C6	0.0(10)
I1	C1	C6	C5	178.5(5)	C4	C5	C6	C1	1.2(10)
C1	C2	C3	C4	0.4(10)	C6	C1	C2	C3	0.9(11)
C1	C2	C7	O1	-179.5(7)	C6	C1	C2	C7	179.6(6)
C1	C2	C7	O2	-1.0(9)	C7	C2	C3	C4	-178.3(6)
C2	C1	C6	C5	-1.6(11)					

Table S7. Hydrogen Atom Coordinates ($\text{\AA} \times 10^3$) and Isotropic Displacement Parameters ($\text{\AA}^2 \times 10^4$).

Atom	x	y	z	U(eq)
H3	1320(50)	4440(170)	8170(50)	32
H3A	3939.31	6143.56	4694.76	31
H4	4968.17	8988.53	5935.12	33
H5	4322.6	9829.51	7420.29	31
H6	2663.27	7876.58	7681.54	29

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