University of California, Berkeley
College of Chemistry
X-ray Crystallography Facility

X-ray Crystal Structure Report

Sample ID: VM-iodolactone
X-ray ID: SARPONG12
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If you have any questions or concerns regarding the information contained in this report, please contact Dr. Antonio DiPasquale (adipasqu@berkeley.edu).

A colorless prism 0.12 x 0.10 x 0.10 mm in size was mounted on a Cryoloop with Paratone oil. Data were collected in a nitrogen gas stream at 100(2) K using phi and omega scans. Crystal-to-detector distance was 60 mm and exposure time was 5 seconds per frame using a scan width of 1.0°. Data collection was 100.0% complete to 67.00° in θ. A total of 19318 reflections were collected covering the indices, -8<=*h*<=11, -8<=*k*<=9, -24<=*l*<=24. 2781 reflections were found to be symmetry independent, with an R_{int} of 0.0247. Indexing and unit cell refinement indicated a primitive, monoclinic lattice. The space group was found to be P2(1)/c (No. 14). The data were integrated using the Bruker SAINT software program and scaled using the SADABS software program. Solution by direct methods (SIR-2004) produced a complete heavy-atom phasing model consistent with the proposed structure. All non-hydrogen atoms were refined anisotropically by full-matrix least-squares (SHELXL-97). All hydrogen atoms were placed using a riding model. Their positions were constrained relative to their parent atom using the appropriate HFIX command in SHELXL-97.

Table 1. Crystal data and structure refinement for sarpong12.

X-ray ID	sarpong12	
Sample/notebook ID	VM-iodolactone	
Empirical formula	C16 H18 I N O3	
Formula weight	399.21	
Temperature	100(2) K	
Wavelength	1.54178 Å	
Crystal system	Monoclinic	
Space group	P2(1)/c	
Unit cell dimensions	a = 9.6608(9) Å	α= 90°.
	b = 7.7675(7) Å	β= 91.435(3)°.
	c = 20.3906(19) Å	γ = 90°.
Volume	1529.6(2) $Å^3$	
Z	4	
Density (calculated)	1.734 Mg/m^3	
Absorption coefficient	16.536 mm^{-1}	
F(000)	792	
Crystal size	0.12 x 0.10 x 0.10 mm^3	
Crystal color/habit	colorless prism	
Theta range for data collection	4.34 to 68.18°.	
Index ranges	-8<=h<=11, -8<=k<=9, -24<=l<=24	
Reflections collected	19318	
Independent reflections	2781 [R(int) = 0.0247]	
Completeness to theta = 67.00°	100.0 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.2886 and 0.2416	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	2781 / 0 / 192	
Goodness-of-fit on F^2	1.116	
Final R indices [I>2sigma(I)]	R1 = 0.0196, wR2 = 0.0509	
R indices (all data)	R1 = 0.0199, wR2 = 0.0510	
Largest diff. peak and hole	0.865 and -0.547 $e.Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for sarpong12. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	1046(2)	1454(3)	6215(1)	12(1)
C(2)	1295(3)	-34(3)	5763(1)	14(1)
C(3)	2059(2)	-1355(3)	6207(1)	13(1)
C(4)	3069(2)	-2510(3)	5851(1)	13(1)
C(5)	4410(2)	-1584(3)	5654(1)	12(1)
C(6)	5125(2)	-980(3)	6291(1)	11(1)
C(7)	4245(2)	318(3)	6653(1)	10(1)
C(8)	2737(2)	-289(3)	6775(1)	11(1)
C(9)	1816(2)	1293(3)	6795(1)	12(1)
C(10)	1733(2)	2599(3)	7261(1)	14(1)
C(11)	894(2)	3992(3)	7120(1)	16(1)
C(12)	171(2)	4024(3)	6512(1)	14(1)
C(13)	5287(2)	-2787(3)	5244(1)	15(1)
C(14)	2921(2)	-1235(3)	7428(1)	13(1)
C(15)	4134(3)	-349(3)	7759(1)	13(1)
C(16)	-1373(3)	5483(4)	5777(1)	26(1)
N(1)	219(2)	2792(3)	6064(1)	14(1)
O(1)	4831(2)	604(2)	7308(1)	13(1)
O(2)	4510(2)	-359(2)	8325(1)	19(1)
O(3)	-628(2)	5428(2)	6396(1)	19(1)
I(1)	7125(1)	266(1)	6135(1)	12(1)

Table 3. Bond lengths [Å] and angles [°] for sarpong12.

C(1)-N(1)	1.343(3)	C(8)-C(9)	1.518(3)
C(1)-C(9)	1.388(3)	C(8)-C(14)	1.528(3)
C(1)-C(2)	1.502(3)	C(9)-C(10)	1.394(3)
C(2)-C(3)	1.544(3)	C(10)-C(11)	1.377(3)
C(2)-H(2A)	0.9900	C(10)-H(10)	0.9500
C(2)-H(2B)	0.9900	C(11)-C(12)	1.408(3)
C(3)-C(4)	1.523(3)	C(11)-H(11)	0.9500
C(3)-C(8)	1.555(3)	C(12)-N(1)	1.325(3)
C(3)-H(3)	1.0000	C(12)-O(3)	1.353(3)
C(4)-C(5)	1.544(3)	C(13)-H(13A)	0.9800
C(4)-H(4A)	0.9900	C(13)-H(13B)	0.9800
C(4)-H(4B)	0.9900	C(13)-H(13C)	0.9800
C(5)-C(13)	1.525(3)	C(14)-C(15)	1.504(3)
C(5)-C(6)	1.530(3)	C(14)-H(14A)	0.9900
C(5)-H(5)	1.0000	C(14)-H(14B)	0.9900
C(6)-C(7)	1.522(3)	C(15)-O(2)	1.201(3)
C(6)-I(1)	2.190(2)	C(15)-O(1)	1.370(3)
C(6)-H(6)	1.0000	C(16)-O(3)	1.437(3)
C(7)-O(1)	1.456(3)	C(16)-H(16A)	0.9800
C(7)-C(8)	1.556(3)	C(16)-H(16B)	0.9800
C(7)-H(7)	1.0000	C(16)-H(16C)	0.9800

N(1)-C(1)-C(9)	124.7(2)	C(4)-C(3)-H(3)	107.5
N(1)-C(1)-C(2)	123.9(2)	C(2)-C(3)-H(3)	107.5
C(9)-C(1)-C(2)	111.3(2)	C(8)-C(3)-H(3)	107.5
C(1)-C(2)-C(3)	103.45(19)	C(3)-C(4)-C(5)	113.66(19)
C(1)-C(2)-H(2A)	111.1	C(3)-C(4)-H(4A)	108.8
C(3)-C(2)-H(2A)	111.1	C(5)-C(4)-H(4A)	108.8
C(1)-C(2)-H(2B)	111.1	C(3)-C(4)-H(4B)	108.8
C(3)-C(2)-H(2B)	111.1	C(5)-C(4)-H(4B)	108.8
H(2A)-C(2)-H(2B)	109.0	H(4A)-C(4)-H(4B)	107.7
C(4)-C(3)-C(2)	114.52(19)	C(13)-C(5)-C(6)	114.08(18)
C(4)-C(3)-C(8)	113.88(18)	C(13)-C(5)-C(4)	109.77(18)
C(2)-C(3)-C(8)	105.64(19)	C(6)-C(5)-C(4)	106.61(17)

C(13)-C(5)-H(5)	108.8	C(12)-C(11)-H(11)	120.8
C(6)-C(5)-H(5)	108.8	N(1)-C(12)-O(3)	119.4(2)
C(4)-C(5)-H(5)	108.8	N(1)-C(12)-C(11)	124.7(2)
C(7)-C(6)-C(5)	111.63(18)	O(3)-C(12)-C(11)	115.9(2)
C(7)-C(6)-I(1)	106.46(14)	C(5)-C(13)-H(13A)	109.5
C(5)-C(6)-I(1)	113.19(14)	C(5)-C(13)-H(13B)	109.5
C(7)-C(6)-H(6)	108.5	H(13A)-C(13)-H(13B)	109.5
C(5)-C(6)-H(6)	108.5	C(5)-C(13)-H(13C)	109.5
I(1)-C(6)-H(6)	108.5	H(13A)-C(13)-H(13C)	109.5
O(1)-C(7)-C(6)	109.68(17)	H(13B)-C(13)-H(13C)	109.5
O(1)-C(7)-C(8)	104.09(17)	C(15)-C(14)-C(8)	104.07(18)
C(6)-C(7)-C(8)	114.42(18)	C(15)-C(14)-H(14A)	110.9
O(1)-C(7)-H(7)	109.5	C(8)-C(14)-H(14A)	110.9
C(6)-C(7)-H(7)	109.5	C(15)-C(14)-H(14B)	110.9
C(8)-C(7)-H(7)	109.5	C(8)-C(14)-H(14B)	110.9
C(9)-C(8)-C(14)	114.84(19)	H(14A)-C(14)-H(14B)	109.0
C(9)-C(8)-C(3)	102.41(18)	O(2)-C(15)-O(1)	120.4(2)
C(14)-C(8)-C(3)	115.53(19)	O(2)-C(15)-C(14)	129.9(2)
C(9)-C(8)-C(7)	108.10(18)	O(1)-C(15)-C(14)	109.68(19)
C(14)-C(8)-C(7)	101.33(18)	O(3)-C(16)-H(16A)	109.5
C(3)-C(8)-C(7)	114.90(19)	O(3)-C(16)-H(16B)	109.5
C(1)-C(9)-C(10)	118.5(2)	H(16A)-C(16)-H(16B)	109.5
C(1)-C(9)-C(8)	110.6(2)	O(3)-C(16)-H(16C)	109.5
C(10)-C(9)-C(8)	130.7(2)	H(16A)-C(16)-H(16C)	109.5
C(11)-C(10)-C(9)	118.3(2)	H(16B)-C(16)-H(16C)	109.5
C(11)-C(10)-H(10)	120.9	C(12)-N(1)-C(1)	115.6(2)
C(9)-C(10)-H(10)	120.9	C(15)-O(1)-C(7)	110.33(17)
C(10)-C(11)-C(12)	118.3(2)	C(12)-O(3)-C(16)	116.64(19)
C(10)-C(11)-H(11)	120.8		

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2$x 10^3)for sarpong12. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	7(1)	16(1)	14(1)	0(1)	-1(1)	0(1)
C(2)	11(1)	18(1)	14(1)	-3(1)	-6(1)	3(1)
C(3)	10(1)	14(1)	14(1)	-1(1)	-5(1)	-2(1)
C(4)	13(1)	11(1)	16(1)	-3(1)	-7(1)	0(1)
C(5)	14(1)	10(1)	10(1)	-2(1)	-5(1)	2(1)
C(6)	9(1)	11(1)	12(1)	0(1)	-3(1)	-1(1)
C(7)	10(1)	12(1)	8(1)	-1(1)	-5(1)	0(1)
C(8)	10(1)	13(1)	10(1)	-1(1)	-3(1)	-1(1)
C(9)	8(1)	14(1)	13(1)	1(1)	-1(1)	-1(1)
C(10)	12(1)	19(1)	12(1)	-1(1)	-2(1)	0(1)
C(11)	14(1)	16(1)	17(1)	-4(1)	-2(1)	1(1)
C(12)	9(1)	15(1)	17(1)	2(1)	0(1)	3(1)
C(13)	17(1)	14(1)	14(1)	-3(1)	-5(1)	5(1)
C(14)	14(1)	14(1)	12(1)	2(1)	-2(1)	-1(1)
C(15)	14(1)	12(1)	13(1)	-1(1)	-1(1)	3(1)
C(16)	27(2)	25(1)	24(1)	1(1)	-10(1)	13(1)
N(1)	11(1)	18(1)	14(1)	-1(1)	-2(1)	2(1)
O(1)	14(1)	17(1)	10(1)	-2(1)	-4(1)	-2(1)
O(2)	24(1)	22(1)	11(1)	-1(1)	-6(1)	-2(1)
O(3)	16(1)	19(1)	21(1)	-1(1)	-5(1)	7(1)
I(1)	9(1)	15(1)	11(1)	-1(1)	-3(1)	1(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for sarpong12.

	x	y	z	U(eq)
H(2A)	1874	316	5392	17
H(2B)	410	-511	5587	17
H(3)	1342	-2114	6402	15
H(4A)	2603	-2974	5451	16
H(4B)	3317	-3496	6138	16
H(5)	4156	-551	5384	14
H(6)	5281	-2003	6581	13
H(7)	4222	1431	6406	12
H(10)	2241	2532	7665	17
H(11)	805	4906	7425	19
H(13A)	6132	-2190	5116	22
H(13B)	4758	-3135	4849	22
H(13C)	5535	-3810	5503	22
H(14A)	2081	-1128	7693	16
H(14B)	3119	-2470	7357	16
H(16A)	-716	5439	5419	39
H(16B)	-1909	6553	5746	39
H(16C)	-2003	4496	5744	39