University of California, Berkeley
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X-ray Crystallography Facility

X-ray Crystal Structure Report

Sample ID: KGardner01_Sarpong
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A colorless block 0.27 x 0.17 x 0.11 mm in size was mounted on a Cryoloop with Paratone oil. Data were collected in a nitrogen gas stream at 100(2) K using omega scans. Crystal-to-detector distance was 30.25 mm and exposure time was 0.50 seconds per frame using a scan width of 0.5°. Data collection was 100% complete to 74.000° in θ. A total of 19025 reflections were collected covering the indices -14<=h<=9, -17<=k<=17, -12<=l<=12. 3443 reflections were founded to be symmetry independent, with an R_{int} of 0.0375. Indexing and unit cell refinement indicated a primitive, monoclinic lattice. The space group was found to be P 21/c (No. 14). The data were integrated using the CrysAlis^{Pro} 1.171.41.109a software program and scaled using the SCALE3 ABSPACK scaling algorithm. Solution by intrinsic phasing (SHELXT-2015) produced a heavy-atom phasing model consistent with the proposed structure. All non-hydrogen atoms were refined anisotropically by full-matrix least-squares (SHELXL-2014). 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-2014.

Table 1. Crystal data and structure refinement for KGardner01_Sarpong.

Identification code	KGardner01_Sarpong	
Empirical formula	C22 H21 N O	
Formula weight	315.40	
Temperature	100(2) K	
Wavelength	1.54184 Å	
Crystal system	Monoclinic	
Space group	P 21/c	
Unit cell dimensions	a = 11.86970(10) Å	α= 90°.
	b = 14.3822(2) Å	β= 95.7760(10)°.
	c = 9.93520(10) Å	γ = 90°.
Volume	1687.45(3) $Å^3$	
Z	4	
Density (calculated)	1.241 Mg/m^3	
Absorption coefficient	0.585 mm^{-1}	
F(000)	672	
Crystal size	0.270 x 0.170 x 0.110 mm^3	
Theta range for data collection	3.743 to 74.445°.	
Index ranges	-14<=h<=9, -17<=k<=17, -12<=l<=12	
Reflections collected	19025	
Independent reflections	3443 [R(int) = 0.0375]	
Completeness to theta = 74.000°	99.8 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	1.00000 and 0.74274	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3443 / 0 / 217	
Goodness-of-fit on F^2	1.029	
Final R indices [I>2sigma(I)]	R1 = 0.0384, wR2 = 0.0970	
R indices (all data)	R1 = 0.0412, wR2 = 0.0983	
Extinction coefficient	n/a	
Largest diff. peak and hole	0.218 and -0.266 $e.Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for kgardner01_sarpong. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O(1)	1368(1)	2605(1)	6824(1)	26(1)
N(1)	2985(1)	3083(1)	5035(1)	18(1)
C(1)	2866(1)	3649(1)	6239(1)	21(1)
C(2)	1663(1)	3556(1)	6612(1)	24(1)
C(3)	1508(1)	2070(1)	5640(1)	26(1)
C(4)	2720(1)	2113(1)	5286(1)	21(1)
C(5)	3922(1)	3278(1)	4310(1)	17(1)
C(6)	3977(1)	4142(1)	3640(1)	17(1)
C(7)	3062(1)	4855(1)	3616(1)	17(1)
C(8)	1918(1)	4615(1)	3327(1)	19(1)
C(9)	1087(1)	5297(1)	3238(1)	22(1)
C(10)	1374(1)	6227(1)	3445(1)	22(1)
C(11)	2503(1)	6469(1)	3751(1)	21(1)
C(12)	3339(1)	5788(1)	3828(1)	19(1)
C(13)	4909(1)	4327(1)	2932(1)	18(1)
C(14)	5784(1)	3686(1)	2842(1)	18(1)
C(15)	6763(1)	3905(1)	2076(1)	19(1)
C(16)	6611(1)	4345(1)	817(1)	23(1)
C(17)	7519(1)	4494(1)	61(1)	27(1)
C(18)	8600(1)	4209(1)	560(1)	26(1)
C(19)	8768(1)	3786(1)	1819(1)	23(1)
C(20)	7861(1)	3640(1)	2576(1)	21(1)
C(21)	5701(1)	2827(1)	3477(1)	20(1)
C(22)	4787(1)	2630(1)	4205(1)	19(1)

Table 3. Bond lengths [Å] and angles [°] for kgardner01_sarpong.

O(1)-C(3)	1.4292(15)
O(1)-C(2)	1.4325(14)
N(1)-C(5)	1.4124(13)
N(1)-C(4)	1.4578(14)
N(1)-C(1)	1.4661(14)
C(1)-C(2)	1.5169(15)
C(3)-C(4)	1.5161(15)
C(5)-C(22)	1.3988(15)
C(5)-C(6)	1.4135(15)
C(6)-C(13)	1.3958(14)
C(6)-C(7)	1.4918(14)
C(7)-C(12)	1.3930(15)
C(7)-C(8)	1.4030(14)
C(8)-C(9)	1.3875(15)
C(9)-C(10)	1.3912(16)
C(10)-C(11)	1.3877(16)
C(11)-C(12)	1.3915(15)
C(13)-C(14)	1.3982(15)
C(14)-C(21)	1.3947(15)
C(14)-C(15)	1.4859(14)
C(15)-C(16)	1.3969(16)
C(15)-C(20)	1.4003(15)
C(16)-C(17)	1.3907(16)
C(17)-C(18)	1.3897(17)
C(18)-C(19)	1.3879(17)
C(19)-C(20)	1.3900(15)
C(21)-C(22)	1.3917(15)

C(3)-O(1)-C(2)	110.04(8)
C(5)-N(1)-C(4)	118.17(8)
C(5)-N(1)-C(1)	116.83(8)
C(4)-N(1)-C(1)	110.61(8)
N(1)-C(1)-C(2)	108.72(9)
O(1)-C(2)-C(1)	111.73(9)

O(1)-C(3)-C(4)	111.20(9)
N(1)-C(4)-C(3)	107.70(9)
C(22)-C(5)-N(1)	121.81(9)
C(22)-C(5)-C(6)	118.79(9)
N(1)-C(5)-C(6)	119.38(9)
C(13)-C(6)-C(5)	118.75(9)
C(13)-C(6)-C(7)	118.28(9)
C(5)-C(6)-C(7)	122.94(9)
C(12)-C(7)-C(8)	118.52(10)
C(12)-C(7)-C(6)	119.98(9)
C(8)-C(7)-C(6)	121.45(10)
C(9)-C(8)-C(7)	120.38(10)
C(8)-C(9)-C(10)	120.61(10)
C(11)-C(10)-C(9)	119.35(10)
C(10)-C(11)-C(12)	120.22(10)
C(11)-C(12)-C(7)	120.91(10)
C(6)-C(13)-C(14)	122.58(10)
C(21)-C(14)-C(13)	117.89(10)
C(21)-C(14)-C(15)	120.95(9)
C(13)-C(14)-C(15)	121.14(10)
C(16)-C(15)-C(20)	118.22(10)
C(16)-C(15)-C(14)	121.19(10)
C(20)-C(15)-C(14)	120.54(10)
C(17)-C(16)-C(15)	121.10(10)
C(18)-C(17)-C(16)	119.94(11)
C(19)-C(18)-C(17)	119.68(10)
C(18)-C(19)-C(20)	120.32(10)
C(19)-C(20)-C(15)	120.71(10)
C(22)-C(21)-C(14)	120.65(10)
C(21)-C(22)-C(5)	121.30(10)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2$x 10^3)for kgardner01_sarpong. 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}
O(1)	25(1)	29(1)	28(1)	5(1)	11(1)	1(1)
N(1)	18(1)	17(1)	20(1)	0(1)	5(1)	0(1)
C(1)	21(1)	22(1)	19(1)	0(1)	4(1)	2(1)
C(2)	23(1)	26(1)	25(1)	3(1)	7(1)	4(1)
C(3)	22(1)	27(1)	29(1)	3(1)	6(1)	-4(1)
C(4)	21(1)	20(1)	24(1)	3(1)	5(1)	-1(1)
C(5)	15(1)	19(1)	17(1)	-1(1)	1(1)	-1(1)
C(6)	16(1)	18(1)	17(1)	-1(1)	1(1)	-1(1)
C(7)	17(1)	19(1)	15(1)	2(1)	3(1)	1(1)
C(8)	18(1)	19(1)	20(1)	1(1)	2(1)	-2(1)
C(9)	15(1)	24(1)	26(1)	1(1)	1(1)	0(1)
C(10)	20(1)	21(1)	26(1)	2(1)	3(1)	5(1)
C(11)	22(1)	17(1)	23(1)	1(1)	2(1)	-1(1)
C(12)	16(1)	21(1)	19(1)	2(1)	2(1)	-1(1)
C(13)	18(1)	17(1)	19(1)	0(1)	2(1)	-1(1)
C(14)	17(1)	20(1)	19(1)	-2(1)	2(1)	-1(1)
C(15)	18(1)	16(1)	23(1)	-4(1)	5(1)	-1(1)
C(16)	20(1)	24(1)	25(1)	0(1)	3(1)	-1(1)
C(17)	27(1)	30(1)	24(1)	3(1)	7(1)	-4(1)
C(18)	22(1)	28(1)	31(1)	-3(1)	11(1)	-6(1)
C(19)	17(1)	22(1)	31(1)	-5(1)	4(1)	-2(1)
C(20)	19(1)	20(1)	23(1)	-2(1)	3(1)	-1(1)
C(21)	17(1)	19(1)	23(1)	-2(1)	3(1)	2(1)
C(22)	19(1)	17(1)	22(1)	1(1)	2(1)	0(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for kgardner01_sarpong.

	x	y	z	U(eq)
H(1A)	3032	4309	6052	25
H(1B)	3407	3435	6999	25
H(2A)	1583	3916	7447	29
H(2B)	1133	3822	5878	29
H(3A)	995	2311	4872	31
H(3B)	1300	1415	5796	31
H(4A)	3242	1865	6042	26
H(4B)	2805	1734	4470	26
H(8)	1710	3981	3191	23
H(9)	315	5127	3034	26
H(10)	803	6693	3377	27
H(11)	2705	7102	3908	25
H(12)	4110	5962	4028	23
H(13)	4951	4911	2494	21
H(16)	5876	4545	472	27
H(17)	7401	4791	-796	32
H(18)	9220	4303	41	32
H(19)	9507	3595	2166	28
H(20)	7987	3357	3442	25
H(21)	6274	2372	3413	23
H(22)	4750	2044	4638	23

Table 6. Torsion angles [°] for kgardner01_sarpong.

C(5)-N(1)-C(1)-C(2)	162.48(9)
C(4)-N(1)-C(1)-C(2)	-58.46(11)
C(3)-O(1)-C(2)-C(1)	-57.32(12)
N(1)-C(1)-C(2)-O(1)	56.62(12)
C(2)-O(1)-C(3)-C(4)	59.23(12)
C(5)-N(1)-C(4)-C(3)	-161.57(9)
C(1)-N(1)-C(4)-C(3)	59.99(11)
O(1)-C(3)-C(4)-N(1)	-60.33(12)
C(4)-N(1)-C(5)-C(22)	-20.52(14)
C(1)-N(1)-C(5)-C(22)	115.39(11)
C(4)-N(1)-C(5)-C(6)	157.80(10)
C(1)-N(1)-C(5)-C(6)	-66.29(12)
C(22)-C(5)-C(6)-C(13)	-1.87(15)
N(1)-C(5)-C(6)-C(13)	179.76(9)
C(22)-C(5)-C(6)-C(7)	176.01(9)
N(1)-C(5)-C(6)-C(7)	-2.36(15)
C(13)-C(6)-C(7)-C(12)	-45.86(14)
C(5)-C(6)-C(7)-C(12)	136.25(11)
C(13)-C(6)-C(7)-C(8)	131.45(11)
C(5)-C(6)-C(7)-C(8)	-46.44(15)
C(12)-C(7)-C(8)-C(9)	0.87(15)
C(6)-C(7)-C(8)-C(9)	-176.48(10)
C(7)-C(8)-C(9)-C(10)	-0.55(17)
C(8)-C(9)-C(10)-C(11)	-0.39(17)
C(9)-C(10)-C(11)-C(12)	1.00(17)
C(10)-C(11)-C(12)-C(7)	-0.68(16)
C(8)-C(7)-C(12)-C(11)	-0.26(15)
C(6)-C(7)-C(12)-C(11)	177.13(10)
C(5)-C(6)-C(13)-C(14)	0.94(15)
C(7)-C(6)-C(13)-C(14)	-177.04(9)
C(6)-C(13)-C(14)-C(21)	0.81(15)
C(6)-C(13)-C(14)-C(15)	179.80(10)
C(21)-C(14)-C(15)-C(16)	136.38(11)
C(13)-C(14)-C(15)-C(16)	-42.57(15)

C(21)-C(14)-C(15)-C(20)	-41.03(15)
C(13)-C(14)-C(15)-C(20)	140.02(11)
C(20)-C(15)-C(16)-C(17)	1.74(17)
C(14)-C(15)-C(16)-C(17)	-175.73(10)
C(15)-C(16)-C(17)-C(18)	-0.42(18)
C(16)-C(17)-C(18)-C(19)	-0.74(18)
C(17)-C(18)-C(19)-C(20)	0.53(18)
C(18)-C(19)-C(20)-C(15)	0.85(17)
C(16)-C(15)-C(20)-C(19)	-1.95(16)
C(14)-C(15)-C(20)-C(19)	175.53(10)
C(13)-C(14)-C(21)-C(22)	-1.63(16)
C(15)-C(14)-C(21)-C(22)	179.39(10)
C(14)-C(21)-C(22)-C(5)	0.70(16)
N(1)-C(5)-C(22)-C(21)	179.42(10)
C(6)-C(5)-C(22)-C(21)	1.09(16)

Symmetry transformations used to generate equivalent atoms: