## Cascade cyclization and intramolecular nitrone dipolar cycloaddition and formal

## synthesis of 19-hydroxyibogamine

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1. X-ray data for compound 21



Figure S1. Crystal structure for **21** with thermal ellipsoids at 50% calculated displacement parameter.

Table S1. Crystal data and structure refinement for	<b>21</b> (oic266a).	
Identification code	oic266a	
Empirical formula	C27 H39 N3 O6	
Formula weight	501.61	
Temperature	100(2) K	
Wavelength	1.54178 Å	
Crystal system	Monoclinic	
Space group	P2 <sub>1</sub>	
Unit cell dimensions	a = 10.8594(4)  Å	<b>a</b> = 90°.
	b = 7.3763(3) Å	b=92.469(3)°.
	c = 15.5152(7) Å	$g = 90^{\circ}$ .
Volume	1241.65(9) Å <sup>3</sup>	
Z	2	
Density (calculated)	1.342 Mg/m <sup>3</sup>	
Absorption coefficient	0.772 mm <sup>-1</sup>	
F(000)	540	
Crystal size	$0.180 \text{ x} 0.060 \text{ x} 0.040 \text{ mm}^3$	
Theta range for data collection	2.851 to 65.991°.	
Index ranges	-12<=h<=12, -8<=k<=8, -17<=	=1<=18
Reflections collected	6309	
Independent reflections	3609 [R(int) = 0.0537]	
Completeness to theta = $65.991^{\circ}$	97.6 %	
Absorption correction	Semi-empirical from equivalen	its
Max. and min. transmission	0.93 and 0.84	
Refinement method	Full-matrix least-squares on F <sup>2</sup>	
Data / restraints / parameters	3609 / 1 / 329	
Goodness-of-fit on F <sup>2</sup>	1.011	
Final R indices [I>2sigma(I)]	R1 = 0.0451, wR2 = 0.0994	
R indices (all data)	R1 = 0.0679, wR2 = 0.1112	
Extinction coefficient	n/a	
Largest diff. peak and hole	0.185 and -0.182 e.Å <sup>-3</sup>	

CCDC 1858480

	Х	У	Z	U(eq)
O(1)	5183(2)	4682(4)	1099(2)	28(1)
O(2)	4010(2)	1967(4)	3607(1)	28(1)
O(3)	8214(2)	4857(3)	4335(2)	26(1)
O(4)	7446(2)	1799(4)	6845(1)	27(1)
O(5)	9113(2)	7041(4)	-276(1)	34(1)
O(6)	8660(2)	9740(4)	2361(2)	28(1)
N(1)	5499(2)	3553(4)	1856(2)	21(1)
N(2)	8706(2)	3635(4)	5005(2)	22(1)
N(3)	8096(3)	8598(4)	1685(2)	28(1)
C(1)	3174(3)	6074(5)	838(2)	29(1)
C(2)	3897(3)	4330(5)	803(2)	21(1)
C(3)	3467(3)	2841(5)	1393(2)	20(1)
C(4)	4273(3)	3292(5)	2216(2)	20(1)
C(5)	4304(3)	1751(5)	2867(2)	22(1)
C(6)	4704(3)	-27(5)	2501(2)	24(1)
C(7)	3804(3)	929(5)	1080(2)	23(1)
C(8)	4993(3)	294(5)	1556(2)	23(1)
C(9)	5969(3)	1783(5)	1540(2)	25(1)
C(10)	6130(3)	5962(5)	4158(2)	26(1)
C(11)	6944(3)	4325(5)	4095(2)	20(1)
C(12)	6644(3)	2806(5)	4718(2)	20(1)
C(13)	6956(3)	892(5)	4387(2)	23(1)
C(14)	8253(3)	357(5)	4717(2)	22(1)
C(15)	8226(3)	17(5)	5683(2)	24(1)
C(16)	7571(3)	3276(5)	5474(2)	20(1)
C(17)	9131(3)	1936(5)	4580(2)	27(1)
C(18)	7729(3)	1704(5)	6097(2)	20(1)
C(19)	10762(3)	10790(5)	2492(2)	27(1)
C(20)	9935(3)	9157(5)	2526(2)	25(1)
C(21)	10172(3)	7734(5)	1827(2)	23(1)
C(22)	9165(3)	8302(5)	1146(2)	23(1)
C(23)	8917(3)	6827(6)	484(2)	25(1)
C(24)	8485(3)	5085(5)	866(2)	28(1)

Table S2. Atomic coordinates (  $x \ 10^4$ ) and equivalent isotropic displacement parameters (Å<sup>2</sup>x 10<sup>3</sup>) for oic266a. U(eq) is defined as one third of the trace of the orthogonalized U<sup>ij</sup> tensor.

C(25)	9908(3)	5775(5)	2110(3)	34(1)
C(26)	8568(3)	5288(5)	1843(2)	26(1)
C(27)	7731(4)	6838(6)	2088(2)	32(1)

O(1)-N(1)	1.468(4)
O(1)-C(2)	1.474(4)
O(2)-C(5)	1.215(4)
O(3)-N(2)	1.459(4)
O(3)-C(11)	1.467(4)
O(4)-C(18)	1.215(4)
O(5)-C(23)	1.217(4)
O(6)-N(3)	1.459(4)
O(6)-C(20)	1.462(4)
N(1)-C(4)	1.479(4)
N(1)-C(9)	1.493(4)
N(2)-C(16)	1.482(4)
N(2)-C(17)	1.499(4)
N(3)-C(22)	1.475(4)
N(3)-C(27)	1.502(5)
C(1)-C(2)	1.509(5)
C(1)-H(11)	0.9800
C(1)-H(12)	0.9800
C(1)-H(1)	0.9800
C(2)-C(3)	1.516(5)
C(2)-H(13)	1.0000
C(3)-C(7)	1.541(5)
C(3)-C(4)	1.553(4)
C(3)-H(5)	1.0000
C(4)-C(5)	1.520(5)
C(4)-H(4)	1.0000
C(5)-C(6)	1.501(5)
C(6)-C(8)	1.530(5)
C(6)-H(3)	0.9900
C(6)-H(2)	0.9900
C(7)-C(8)	1.533(5)
C(7)-H(7)	0.9900
C(7)-H(6)	0.9900
C(8)-C(9)	1.527(5)
C(8)-H(8)	1.0000
C(9)-H(10)	0.9900

Table S3. Bond lengths [Å] and angles [°] for oic266a.

C(9)-H(9)	0.9900
C(10)-C(11)	1.502(5)
C(10)-H(14)	0.9800
C(10)-H(22)	0.9800
C(10)-H(21)	0.9800
C(11)-C(12)	1.524(5)
C(11)-H(20)	1.0000
C(12)-C(13)	1.545(5)
C(12)-C(16)	1.552(4)
C(12)-H(25)	1.0000
C(13)-C(14)	1.529(5)
C(13)-H(18)	0.9900
C(13)-H(17)	0.9900
C(14)-C(15)	1.521(4)
C(14)-C(17)	1.526(5)
C(14)-H(26)	1.0000
C(15)-C(18)	1.510(5)
C(15)-H(15)	0.9900
C(15)-H(16)	0.9900
C(16)-C(18)	1.514(5)
C(16)-H(19)	1.0000
C(17)-H(23)	0.9900
C(17)-H(24)	0.9900
C(19)-C(20)	1.504(5)
C(19)-H(38)	0.9800
C(19)-H(27)	0.9800
C(19)-H(37)	0.9800
C(20)-C(21)	1.539(5)
C(20)-H(36)	1.0000
C(21)-C(25)	1.540(5)
C(21)-C(22)	1.546(4)
C(21)-H(30)	1.0000
C(22)-C(23)	1.513(5)
C(22)-H(39)	1.0000
C(23)-C(24)	1.498(5)
C(24)-C(26)	1.522(5)
C(24)-H(29)	0.9900
C(24)-H(28)	0.9900

C(25)-C(26)	1.538(5)
C(25)-H(32)	0.9900
C(25)-H(31)	0.9900
C(26)-C(27)	1.520(5)
C(26)-H(35)	1.0000
C(27)-H(34)	0.9900
C(27)-H(33)	0.9900
N(1)-O(1)-C(2)	109.6(2)
N(2)-O(3)-C(11)	109.1(2)
N(3)-O(6)-C(20)	108.7(2)
O(1)-N(1)-C(4)	101.1(2)
O(1)-N(1)-C(9)	107.7(2)
C(4)-N(1)-C(9)	109.7(3)
O(3)-N(2)-C(16)	100.0(2)
O(3)-N(2)-C(17)	108.3(2)
C(16)-N(2)-C(17)	110.2(3)
O(6)-N(3)-C(22)	100.2(2)
O(6)-N(3)-C(27)	108.0(3)
C(22)-N(3)-C(27)	109.7(3)
C(2)-C(1)-H(11)	109.5
C(2)-C(1)-H(12)	109.5
H(11)-C(1)-H(12)	109.5
C(2)-C(1)-H(1)	109.5
H(11)-C(1)-H(1)	109.5
H(12)-C(1)-H(1)	109.5
O(1)-C(2)-C(1)	109.0(3)
O(1)-C(2)-C(3)	104.6(2)
C(1)-C(2)-C(3)	114.9(3)
O(1)-C(2)-H(13)	109.4
C(1)-C(2)-H(13)	109.4
C(3)-C(2)-H(13)	109.4
C(2)-C(3)-C(7)	112.9(3)
C(2)-C(3)-C(4)	99.4(3)
C(7)-C(3)-C(4)	108.8(3)
C(2)-C(3)-H(5)	111.7
C(7)-C(3)-H(5)	111.7
C(4)-C(3)-H(5)	111.7

N(1)-C(4)-C(5)	110.7(3)
N(1)-C(4)-C(3)	101.7(2)
C(5)-C(4)-C(3)	112.5(3)
N(1)-C(4)-H(4)	110.6
C(5)-C(4)-H(4)	110.6
C(3)-C(4)-H(4)	110.6
O(2)-C(5)-C(6)	124.3(3)
O(2)-C(5)-C(4)	122.0(3)
C(6)-C(5)-C(4)	113.6(3)
C(5)-C(6)-C(8)	107.6(3)
C(5)-C(6)-H(3)	110.2
C(8)-C(6)-H(3)	110.2
C(5)-C(6)-H(2)	110.2
C(8)-C(6)-H(2)	110.2
H(3)-C(6)-H(2)	108.5
C(8)-C(7)-C(3)	109.5(3)
C(8)-C(7)-H(7)	109.8
C(3)-C(7)-H(7)	109.8
C(8)-C(7)-H(6)	109.8
C(3)-C(7)-H(6)	109.8
H(7)-C(7)-H(6)	108.2
C(9)-C(8)-C(6)	107.4(3)
C(9)-C(8)-C(7)	110.1(3)
C(6)-C(8)-C(7)	107.8(3)
C(9)-C(8)-H(8)	110.5
C(6)-C(8)-H(8)	110.5
C(7)-C(8)-H(8)	110.5
N(1)-C(9)-C(8)	112.1(3)
N(1)-C(9)-H(10)	109.2
C(8)-C(9)-H(10)	109.2
N(1)-C(9)-H(9)	109.2
C(8)-C(9)-H(9)	109.2
H(10)-C(9)-H(9)	107.9
C(11)-C(10)-H(14)	109.5
C(11)-C(10)-H(22)	109.5
H(14)-C(10)-H(22)	109.5
C(11)-C(10)-H(21)	109.5
H(14)-C(10)-H(21)	109.5

H(22)-C(10)-H(21)	109.5
O(3)-C(11)-C(10)	108.5(3)
O(3)-C(11)-C(12)	105.1(2)
C(10)-C(11)-C(12)	114.1(3)
O(3)-C(11)-H(20)	109.7
C(10)-C(11)-H(20)	109.7
C(12)-C(11)-H(20)	109.7
C(11)-C(12)-C(13)	113.9(3)
C(11)-C(12)-C(16)	99.6(3)
C(13)-C(12)-C(16)	108.1(3)
C(11)-C(12)-H(25)	111.6
C(13)-C(12)-H(25)	111.6
C(16)-C(12)-H(25)	111.6
C(14)-C(13)-C(12)	109.6(3)
C(14)-C(13)-H(18)	109.7
C(12)-C(13)-H(18)	109.7
C(14)-C(13)-H(17)	109.7
C(12)-C(13)-H(17)	109.7
H(18)-C(13)-H(17)	108.2
C(15)-C(14)-C(17)	107.6(3)
C(15)-C(14)-C(13)	108.5(3)
C(17)-C(14)-C(13)	109.2(3)
C(15)-C(14)-H(26)	110.5
C(17)-C(14)-H(26)	110.5
C(13)-C(14)-H(26)	110.5
C(18)-C(15)-C(14)	107.8(3)
C(18)-C(15)-H(15)	110.2
C(14)-C(15)-H(15)	110.2
C(18)-C(15)-H(16)	110.2
C(14)-C(15)-H(16)	110.2
H(15)-C(15)-H(16)	108.5
N(2)-C(16)-C(18)	112.1(3)
N(2)-C(16)-C(12)	101.2(2)
C(18)-C(16)-C(12)	111.3(3)
N(2)-C(16)-H(19)	110.6
C(18)-C(16)-H(19)	110.6
C(12)-C(16)-H(19)	110.6
N(2)-C(17)-C(14)	111.7(3)

N(2)-C(17)-H(23)	109.3
C(14)-C(17)-H(23)	109.3
N(2)-C(17)-H(24)	109.3
C(14)-C(17)-H(24)	109.3
H(23)-C(17)-H(24)	107.9
O(4)-C(18)-C(15)	124.2(3)
O(4)-C(18)-C(16)	122.6(3)
C(15)-C(18)-C(16)	113.1(3)
C(20)-C(19)-H(38)	109.5
C(20)-C(19)-H(27)	109.5
H(38)-C(19)-H(27)	109.5
C(20)-C(19)-H(37)	109.5
H(38)-C(19)-H(37)	109.5
H(27)-C(19)-H(37)	109.5
O(6)-C(20)-C(19)	108.7(3)
O(6)-C(20)-C(21)	105.2(3)
C(19)-C(20)-C(21)	113.8(3)
O(6)-C(20)-H(36)	109.7
C(19)-C(20)-H(36)	109.7
C(21)-C(20)-H(36)	109.7
C(20)-C(21)-C(25)	113.6(3)
C(20)-C(21)-C(22)	99.3(3)
C(25)-C(21)-C(22)	108.3(3)
C(20)-C(21)-H(30)	111.6
C(25)-C(21)-H(30)	111.6
C(22)-C(21)-H(30)	111.6
N(3)-C(22)-C(23)	111.7(3)
N(3)-C(22)-C(21)	101.7(3)
C(23)-C(22)-C(21)	111.6(3)
N(3)-C(22)-H(39)	110.5
C(23)-C(22)-H(39)	110.5
C(21)-C(22)-H(39)	110.5
O(5)-C(23)-C(24)	124.4(4)
O(5)-C(23)-C(22)	122.0(4)
C(24)-C(23)-C(22)	113.5(3)
C(23)-C(24)-C(26)	107.6(3)
C(23)-C(24)-H(29)	110.2
C(26)-C(24)-H(29)	110.2

C(23)-C(24)-H(28)	110.2
C(26)-C(24)-H(28)	110.2
H(29)-C(24)-H(28)	108.5
C(26)-C(25)-C(21)	109.1(3)
C(26)-C(25)-H(32)	109.9
C(21)-C(25)-H(32)	109.9
C(26)-C(25)-H(31)	109.9
C(21)-C(25)-H(31)	109.9
H(32)-C(25)-H(31)	108.3
C(27)-C(26)-C(24)	108.2(3)
C(27)-C(26)-C(25)	109.0(3)
C(24)-C(26)-C(25)	107.8(3)
C(27)-C(26)-H(35)	110.6
C(24)-C(26)-H(35)	110.6
C(25)-C(26)-H(35)	110.6
N(3)-C(27)-C(26)	112.0(3)
N(3)-C(27)-H(34)	109.2
C(26)-C(27)-H(34)	109.2
N(3)-C(27)-H(33)	109.2
C(26)-C(27)-H(33)	109.2
H(34)-C(27)-H(33)	107.9

Symmetry transformations used to generate equivalent atoms:

	U <sup>11</sup>	U <sup>22</sup>	U <sup>33</sup>	U <sup>23</sup>	U <sup>13</sup>	U <sup>12</sup>
O(1)	26(1)	30(2)	26(1)	9(1)	1(1)	-6(1)
O(2)	32(1)	36(2)	16(1)	1(1)	2(1)	-3(1)
O(3)	24(1)	22(1)	31(1)	6(1)	1(1)	0(1)
O(4)	33(1)	32(2)	15(1)	2(1)	3(1)	4(1)
O(5)	41(2)	42(2)	18(1)	0(1)	4(1)	-8(1)
O(6)	23(1)	29(2)	33(1)	-8(1)	2(1)	2(1)
N(1)	22(1)	22(2)	19(1)	1(1)	2(1)	-2(1)
N(2)	20(1)	19(2)	25(1)	2(1)	1(1)	0(1)
N(3)	28(2)	24(2)	32(2)	-7(2)	1(1)	0(2)
C(1)	40(2)	22(2)	26(2)	2(2)	-2(2)	2(2)
C(2)	24(2)	25(2)	14(2)	0(2)	0(1)	-2(2)
C(3)	20(2)	21(2)	18(2)	0(2)	1(1)	-3(2)
C(4)	22(2)	19(2)	19(2)	-1(2)	2(1)	0(1)
C(5)	17(2)	26(2)	21(2)	1(2)	-2(1)	-4(2)
C(6)	28(2)	23(2)	22(2)	1(2)	-4(1)	-1(2)
C(7)	27(2)	18(2)	23(2)	-4(2)	0(1)	-3(2)
C(8)	26(2)	20(2)	22(2)	-5(2)	1(1)	2(2)
C(9)	22(2)	27(2)	26(2)	-3(2)	2(1)	3(2)
C(10)	33(2)	25(2)	20(2)	4(2)	3(1)	5(2)
C(11)	23(2)	20(2)	17(2)	-5(1)	1(1)	-2(1)
C(12)	18(2)	21(2)	22(2)	1(2)	1(1)	-1(1)
C(13)	25(2)	18(2)	25(2)	-3(2)	-6(1)	-1(2)
C(14)	26(2)	19(2)	21(2)	-3(2)	1(1)	3(2)
C(15)	31(2)	16(2)	25(2)	-1(2)	0(1)	4(1)
C(16)	23(2)	18(2)	21(2)	-3(2)	3(1)	-1(1)
C(17)	27(2)	23(2)	31(2)	-2(2)	9(2)	2(2)
C(18)	18(2)	20(2)	23(2)	1(2)	-2(1)	-1(1)
C(19)	31(2)	27(2)	22(2)	-6(2)	5(1)	-5(2)
C(20)	20(2)	34(2)	21(2)	2(2)	1(1)	-1(2)
C(21)	18(2)	25(2)	25(2)	0(2)	0(1)	-1(2)
C(22)	26(2)	22(2)	21(2)	1(2)	3(1)	-2(1)
C(23)	20(2)	30(2)	26(2)	1(2)	-2(1)	0(2)
C(24)	28(2)	25(2)	31(2)	-6(2)	-2(2)	-2(2)

Table S4. Anisotropic displacement parameters  $(Å^2 x \ 10^3)$  for oic266a. The anisotropic displacement factor exponent takes the form:  $-2p^2[h^2 \ a^{*2}U^{11} + ... + 2h \ k \ a^* \ b^* \ U^{12}]$ 

C(25)	37(2)	23(2)	40(2)	5(2)	-12(2)	-3(2)
C(26)	27(2)	24(2)	26(2)	3(2)	1(1)	-6(2)
C(27)	34(2)	30(2)	35(2)	-5(2)	15(2)	-9(2)

	Х	У	Z	U(eq)
H(11)	3070	6414	1442	44
H(12)	2362	5903	549	44
H(1)	3619	7037	547	44
H(13)	3880	3874	196	25
H(5)	2567	2947	1498	24
H(4)	3985	4438	2487	24
H(3)	4039	-937	2541	29
H(2)	5445	-482	2826	29
H(7)	3126	76	1193	27
H(6)	3922	951	451	27
H(8)	5303	-844	1291	27
H(10)	6696	1412	1905	30
H(9)	6238	1933	942	30
H(14)	6100	6339	4762	39
H(22)	5297	5664	3934	39
H(21)	6463	6953	3817	39
H(20)	6901	3853	3490	24
H(25)	5772	2881	4898	24
H(18)	6349	6	4591	28
H(17)	6912	882	3748	28
H(26)	8536	-752	4413	27
H(15)	7690	-1034	5799	29
H(16)	9066	-249	5922	29
H(19)	7304	4390	5782	24
H(23)	9963	1613	4818	33
H(24)	9191	2155	3953	33
H(38)	10753	11258	1901	40
H(27)	11605	10445	2675	40
H(37)	10466	11730	2879	40
H(36)	10023	8583	3110	30
H(30)	11016	7856	1602	27
H(39)	9400	9456	858	27

Table S5. Hydrogen coordinates (  $x\ 10^4$  ) and isotropic displacement parameters (Å  $^2x\ 10\ ^3$  ) for oic266a.

H(29)	9010	4067	686	34
H(28)	7624	4833	666	34
H(32)	10480	4930	1834	41
H(31)	10037	5664	2743	41
H(35)	8318	4137	2127	31
H(34)	6871	6545	1901	39
H(33)	7764	6973	2724	39

D-HA	d(D-H)	d(HA)	d(DA)	<(DHA)
C(4)-H(4)O(4)#1	1.00	2.58	3.540(4)	160.6
C(6)-H(3)O(4)#2	0.99	2.54	3.489(4)	161.4
C(9)-H(10)O(6)#3	0.99	2.54	3.480(4)	159.0
C(9)-H(10)N(3)#3	0.99	2.60	3.295(5)	126.9
C(10)-H(14)O(2)#1	0.98	2.58	3.557(4)	173.8
C(15)-H(15)O(2)#2	0.99	2.56	3.521(5)	162.3
C(16)-H(19)O(2)#1	1.00	2.58	3.550(4)	163.2
C(17)-H(23)N(2)#4	0.99	2.64	3.425(4)	136.6
C(19)-H(38)O(5)#5	0.98	2.60	3.569(4)	171.4
C(24)-H(29)O(5)#6	0.99	2.63	3.590(4)	164.1
C(27)-H(34)O(1)	0.99	2.57	3.489(5)	154.6
C(27)-H(34)N(1)	0.99	2.66	3.435(5)	135.1
C(3)-H(5)O(5)#7	1.00	2.66	3.284(4)	120.7
C(4)-H(4)O(4)#1	1.00	2.58	3.540(4)	160.6
C(6)-H(3)O(4)#2	0.99	2.54	3.489(4)	161.4
C(9)-H(10)O(6)#3	0.99	2.54	3.480(4)	159.0
C(9)-H(10)N(3)#3	0.99	2.60	3.295(5)	126.9
C(10)-H(14)O(2)#1	0.98	2.58	3.557(4)	173.8
C(15)-H(15)O(2)#2	0.99	2.56	3.521(5)	162.3
C(16)-H(19)O(2)#1	1.00	2.58	3.550(4)	163.2
C(17)-H(23)N(2)#4	0.99	2.64	3.425(4)	136.6
C(19)-H(38)O(5)#5	0.98	2.60	3.569(4)	171.4
C(24)-H(29)O(5)#6	0.99	2.63	3.590(4)	164.1
C(27)-H(34)O(1)	0.99	2.57	3.489(5)	154.6
C(27)-H(34)N(1)	0.99	2.66	3.435(5)	135.1

Table S6. Hydrogen bonds for oic266a [Å and °].

Symmetry transformations used to generate equivalent atoms:

#1 -x+1,y+1/2,-z+1 #2 -x+1,y-1/2,-z+1 #3 x,y-1,z #4 -x+2,y-1/2,-z+1 #5 -x+2,y+1/2,-z #6 -x+2,y-1/2,-z #7 -x+1,y-1/2,-z 2. <sup>1</sup>H and <sup>13</sup>C NMR spectra (Note: all alkenes were a mixture E:Z 5.5:1)







S-20











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190 180 170 160 150 140 130 120 110 100

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w.www.ww

90 80

70 60

20

50

40 30

i Yalad

10 ppm









![](_page_29_Figure_0.jpeg)

(crude material, taken on directly to next step)

![](_page_30_Figure_0.jpeg)

![](_page_31_Figure_0.jpeg)

![](_page_32_Figure_0.jpeg)