Two Novel Alkaloids with a Unique Fused Hexacyclic Skeleton from *Daphniphyllum subverticillatum*

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Supporting Information Available

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1. General Experimental Paragraph
2. Copies of ^1H NMR, ^{13}C NMR and 2D NMR (^1H - ^1H COSY, HMQC, HMBC and NOESY) of deoxycalyciphylline B (**1**), deoxyisocalyciphylline B (**2**) and calyciphylline B (**3**), and ^1H NMR of isocalyciphylline B (**4**).
3. CIF file for X-ray analysis and tables of crystal data (crystal data and structure refinement, atomic coordinates, bond lengths and angles, anisotropic displacement parameters and hydrogen coordinates) for deoxycalyciphylline B (**1**) (labeled as **cd22231** for X-ray analysis).

General Experimental Paragraph. In x-ray crystallography, cell constants were determined by a least-squares fit to the setting parameters of 25 independent reflections measure on a Rigaku AFC7R four circle diffractometer employing graphite monochromated Mo-K α radiation (0.71073Å) and operating in the ϕ - ω scan mode. Data reduction and empirical absorption corrections (ψ -scans) were performed with the SHELXS-97 package.

deoxycalyciphylline B (**1**)
¹H-NMR

S3

H₂

S5

deoxycalyciphylline B (1)
H-H COSY

S6

deoxycalyciphylline B (1)
HMOC

S7

deoxycalyciphylline B (1)
HMBC

S8

deoxycalyciphylline B (1)
NOESY

6S

Hz

ppm

S10

171.601

deoxyisocalciphylline B (2)

^{13}C -NMR

85.689
77.347
77.229
77.028
76.710
73.170
60.047
58.586
48.947
48.056
41.880
40.800
35.470
32.272
31.456
30.547
28.398
26.269
23.512
22.363
21.740
14.265

CDCl_3

-127.01
-147.03

ppm

deoxyisocalciphylline B (**2**)

^{13}C -NMR (in CD_3OD)

S12

deoxyisocalciphylline B (2)
 $^{13}\text{C-NMR}$ (in CD_3OD , δ 20-65)

S13

deoxyisocalyciphylline B (2)
H-H COSY

S14

deoxyisocalyciphylline B (2)
HMQC

S15

deoxyisocalciphylline B (2)
HMBC

S16

deoxyisocalyciphylline B (2)
NOESY

S17

S19

calyciphylline B (3)
H-H COSY

S20

S21

calyciphylline B (3)
HMBC

S22

calyciphylline B (3)
NOESY

S23

ZH

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C1 C2 H2 112.4(10) . . ?
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H4A C4 H4B 110.4(14) . . ?
O2 C5 C21 105.17(14) . . ?
O2 C5 C6 102.86(12) . . ?
C21 C5 C6 111.90(15) . . ?
O2 C5 C8 111.82(13) . . ?
C21 C5 C8 114.68(14) . . ?
C6 C5 C8 109.71(13) . . ?
C12 C6 C5 116.92(14) . . ?
C12 C6 C7 110.60(15) . . ?
C5 C6 C7 108.72(13) . . ?
C12 C6 H6 108.4(9) . . ?
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N C7 C6 107.22(14) . . ?
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N C7 H7 109.5(8) . . ?
C9 C7 H7 109.1(8) . . ?
C6 C7 H7 108.0(8) . . ?
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C13 C8 C1 108.48(15) . . ?
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C9 C10 C17 103.08(18) . . ?

C9 C10 C11 110.45(17) . . ?
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 C12 C11 H11B 113.0(15) . . ?
 H11A C11 H11B 113(2) . . ?
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 C11 C12 H12B 109.3(10) . . ?
 H12A C12 H12B 112.9(13) . . ?
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 C8 C13 H13A 107.5(10) . . ?
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 C13 C14 H14A 110.0(11) . . ?
 C22 C14 H14B 108.3(12) . . ?
 C13 C14 H14B 111.0(13) . . ?
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 C2 C18 H16 106.5(9) . . ?

C20 C18 H16 106.7(9) . . ?
 N C19 C18 103.39(16) . . ?
 N C19 H19A 112.2(10) . . ?
 C18 C19 H19A 109.3(10) . . ?
 N C19 H19B 112.2(11) . . ?
 C18 C19 H19B 114.8(11) . . ?
 H19A C19 H19B 105.1(15) . . ?
 C18 C20 H20A 114.4(15) . . ?
 C18 C20 H20B 108.8(17) . . ?
 H20A C20 H20B 108(2) . . ?
 C18 C20 H20C 113.2(15) . . ?
 H20A C20 H20C 108(2) . . ?
 H20B C20 H20C 105(2) . . ?
 C5 C21 H21A 108.9(11) . . ?
 C5 C21 H21B 106.5(11) . . ?
 H21A C21 H21B 111.6(15) . . ?
 C5 C21 H21C 111.9(11) . . ?
 H21A C21 H21C 108.2(16) . . ?
 H21B C21 H21C 109.8(17) . . ?
 O1 C22 O2 119.14(19) . . ?
 O1 C22 C14 124.89(19) . . ?
 O2 C22 C14 115.79(18) . . ?

loop_

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 C3 C4 C8 C5 -159.88(16) ?
 N C1 C8 C13 166.74(16) ?
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 N C1 C8 C4 -78.86(17) ?
 C2 C1 C8 C4 36.74(18) ?
 N C1 C8 C5 44.9(2) ?
 C2 C1 C8 C5 160.50(14) ?
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 O2 C5 C8 C4 -138.12(15) ?
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 C6 C5 C8 C4 108.42(16) ?
 O2 C5 C8 C1 106.16(15) ?
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 C7 C9 C15 C16 -173.23(18) ?

C10 C9 C15 C16 -2.4(2) ?
C9 C15 C16 C17 -0.8(3) ?
C15 C16 C17 C10 3.7(4) ?
C9 C10 C17 C16 -5.0(4) ?
C11 C10 C17 C16 115.4(3) ?
C1 C2 C18 C19 37.88(19) ?
C3 C2 C18 C19 -76.4(2) ?
C1 C2 C18 C20 164.44(18) ?
C3 C2 C18 C20 50.2(3) ?
C7 N C19 C18 161.93(14) ?
C1 N C19 C18 29.1(2) ?
C2 C18 C19 N -40.99(19) ?
C20 C18 C19 N -169.06(19) ?
C5 O2 C22 O1 153.94(16) ?
C5 O2 C22 C14 -30.6(2) ?
C13 C14 C22 O1 147.3(2) ?
C13 C14 C22 O2 -27.9(3) ?

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Table 1. Crystal data and structure refinement for cd22231.

Identification code	cd22231
Empirical formula	C22 H31 N O2
Formula weight	341.48
Temperature	293(2) K
Wavelength	0.71073 Å
Crystal system, space group	Orthorhombic, P2(1)2(1)2(1)
Unit cell dimensions	a = 7.2886(13) Å alpha = 90 deg. b = 8.5874(15) Å beta = 90 deg. c = 29.458(5) Å gamma = 90 deg.
Volume	1843.8(6) Å ³
Z, Calculated density	4, 1.230 Mg/m ³
Absorption coefficient	0.077 mm ⁻¹
F(000)	744
Crystal size	? x ? x ? mm
Theta range for data collection	2.47 to 28.25 deg.
Limiting indices	-9<=h<=9, -8<=k<=11, -39<=l<=38
Reflections collected / unique	11244 / 4290 [R(int) = 0.0612]
Completeness to theta = 28.25	97.2 %
Absorption correction	Sadabs
Max. and min. transmission	1.00000 and 0.56678
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	4290 / 0 / 350
Goodness-of-fit on F ²	0.879
Final R indices [I>2sigma(I)]	R1 = 0.0438, wR2 = 0.0802
R indices (all data)	R1 = 0.0650, wR2 = 0.0851
Absolute structure parameter	0.2(13)
Largest diff. peak and hole	0.217 and -0.182 e.Å ⁻³

Table 2. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for cd22231. U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U(eq)
O(1)	-2049(2)	9156(2)	2027(1)	69(1)
O(2)	301(2)	8761(1)	1574(1)	47(1)
N	4640(2)	6406(2)	975(1)	37(1)
C(1)	3286(3)	5692(2)	1283(1)	38(1)
C(2)	4267(3)	4259(2)	1476(1)	48(1)
C(3)	4908(4)	4760(2)	1955(1)	54(1)
C(4)	4355(3)	6448(2)	2019(1)	45(1)
C(5)	2294(2)	8424(2)	1569(1)	37(1)
C(6)	2821(3)	8718(2)	1075(1)	35(1)
C(7)	4763(2)	8100(2)	996(1)	34(1)
C(8)	2705(2)	6692(2)	1696(1)	38(1)
C(9)	5547(2)	8803(2)	571(1)	37(1)
C(10)	4280(3)	9328(2)	199(1)	47(1)
C(11)	2815(5)	10420(3)	387(1)	71(1)
C(12)	2648(3)	10377(2)	903(1)	50(1)
C(13)	994(3)	5954(3)	1911(1)	52(1)
C(14)	12(3)	7094(3)	2220(1)	61(1)
C(15)	7244(3)	9228(2)	496(1)	49(1)
C(16)	7460(4)	10056(3)	51(1)	66(1)
C(17)	5565(4)	10046(6)	-142(1)	109(1)
C(18)	5785(3)	3939(2)	1132(1)	52(1)
C(19)	6366(3)	5572(2)	1007(1)	49(1)
C(20)	7340(4)	2854(3)	1277(1)	72(1)
C(21)	3143(3)	9621(3)	1887(1)	46(1)
C(22)	-662(3)	8432(3)	1949(1)	52(1)

Table 3. Bond lengths [Å] and angles [deg] for cd22231.

O(1)-C(22)	1.209(2)
O(2)-C(22)	1.339(2)
O(2)-C(5)	1.481(2)
N-C(19)	1.450(2)
N-C(7)	1.458(2)
N-C(1)	1.475(2)
C(1)-C(2)	1.533(3)
C(1)-C(8)	1.548(2)
C(1)-H(1)	0.962(17)
C(2)-C(18)	1.526(3)
C(2)-C(3)	1.546(3)
C(2)-H(2)	0.971(17)
C(3)-C(4)	1.516(3)
C(3)-H(3A)	0.89(2)
C(3)-H(3B)	0.98(2)
C(4)-C(8)	1.547(2)
C(4)-H(4A)	0.972(18)
C(4)-H(4B)	0.998(18)
C(5)-C(21)	1.522(2)
C(5)-C(6)	1.527(2)
C(5)-C(8)	1.563(2)
C(6)-C(12)	1.517(2)
C(6)-C(7)	1.530(3)
C(6)-H(6)	0.945(15)
C(7)-C(9)	1.502(2)
C(7)-H(7)	0.984(15)
C(8)-C(13)	1.535(3)
C(9)-C(15)	1.308(3)
C(9)-C(10)	1.504(2)
C(10)-C(17)	1.504(3)
C(10)-C(11)	1.525(3)
C(10)-H(10)	0.91(2)
C(11)-C(12)	1.526(3)
C(11)-H(11A)	0.87(3)
C(11)-H(11B)	1.06(3)
C(12)-H(12A)	1.08(2)
C(12)-H(12B)	1.03(2)
C(13)-C(14)	1.517(3)
C(13)-H(13A)	0.991(19)
C(13)-H(13B)	0.95(2)
C(14)-C(22)	1.484(3)
C(14)-H(14A)	1.00(2)
C(14)-H(14B)	0.91(2)
C(15)-C(16)	1.499(3)
C(15)-H(15)	0.99(2)
C(16)-C(17)	1.493(4)

C(16) -H(16A)	0.97(2)
C(16) -H(16B)	0.96(3)
C(17) -H(17A)	0.87(3)
C(17) -H(17B)	0.99(4)
C(18) -C(19)	1.510(3)
C(18) -C(20)	1.528(3)
C(18) -H(16)	1.052(17)
C(19) -H(19A)	0.989(18)
C(19) -H(19B)	0.949(19)
C(20) -H(20A)	1.00(3)
C(20) -H(20B)	0.98(3)
C(20) -H(20C)	1.03(3)
C(21) -H(21A)	1.002(19)
C(21) -H(21B)	1.01(2)
C(21) -H(21C)	0.93(2)
C(22) -O(2) -C(5)	118.70(13)
C(19) -N-C(7)	115.89(14)
C(19) -N-C(1)	109.54(13)
C(7) -N-C(1)	115.48(13)
N-C(1) -C(2)	104.50(15)
N-C(1) -C(8)	115.86(14)
C(2) -C(1) -C(8)	106.34(13)
N-C(1) -H(1)	109.4(9)
C(2) -C(1) -H(1)	111.4(9)
C(8) -C(1) -H(1)	109.2(9)
C(18) -C(2) -C(1)	103.67(14)
C(18) -C(2) -C(3)	115.91(19)
C(1) -C(2) -C(3)	104.84(15)
C(18) -C(2) -H(2)	108.8(9)
C(1) -C(2) -H(2)	112.4(10)
C(3) -C(2) -H(2)	111.0(9)
C(4) -C(3) -C(2)	107.36(16)
C(4) -C(3) -H(3A)	114.7(14)
C(2) -C(3) -H(3A)	108.8(13)
C(4) -C(3) -H(3B)	110.4(12)
C(2) -C(3) -H(3B)	112.3(11)
H(3A) -C(3) -H(3B)	103.4(18)
C(3) -C(4) -C(8)	105.12(16)
C(3) -C(4) -H(4A)	112.5(10)
C(8) -C(4) -H(4A)	111.7(9)
C(3) -C(4) -H(4B)	110.0(9)
C(8) -C(4) -H(4B)	106.9(9)
H(4A) -C(4) -H(4B)	110.4(14)
O(2) -C(5) -C(21)	105.17(14)
O(2) -C(5) -C(6)	102.86(12)
C(21) -C(5) -C(6)	111.90(15)
O(2) -C(5) -C(8)	111.82(13)
C(21) -C(5) -C(8)	114.68(14)

C(6) - C(5) - C(8)	109.71 (13)
C(12) - C(6) - C(5)	116.92 (14)
C(12) - C(6) - C(7)	110.60 (15)
C(5) - C(6) - C(7)	108.72 (13)
C(12) - C(6) - H(6)	108.4 (9)
C(5) - C(6) - H(6)	105.5 (9)
C(7) - C(6) - H(6)	106.0 (9)
N - C(7) - C(9)	112.94 (13)
N - C(7) - C(6)	107.22 (14)
C(9) - C(7) - C(6)	109.87 (14)
N - C(7) - H(7)	109.5 (8)
C(9) - C(7) - H(7)	109.1 (8)
C(6) - C(7) - H(7)	108.0 (8)
C(13) - C(8) - C(4)	108.83 (15)
C(13) - C(8) - C(1)	108.48 (15)
C(4) - C(8) - C(1)	101.21 (14)
C(13) - C(8) - C(5)	109.62 (15)
C(4) - C(8) - C(5)	115.16 (14)
C(1) - C(8) - C(5)	113.11 (12)
C(15) - C(9) - C(7)	127.82 (16)
C(15) - C(9) - C(10)	111.94 (16)
C(7) - C(9) - C(10)	119.62 (16)
C(9) - C(10) - C(17)	103.08 (18)
C(9) - C(10) - C(11)	110.45 (17)
C(17) - C(10) - C(11)	115.2 (2)
C(9) - C(10) - H(10)	108.7 (12)
C(17) - C(10) - H(10)	109.6 (12)
C(11) - C(10) - H(10)	109.5 (12)
C(10) - C(11) - C(12)	113.75 (18)
C(10) - C(11) - H(11A)	104.6 (19)
C(12) - C(11) - H(11A)	114.2 (19)
C(10) - C(11) - H(11B)	97.4 (16)
C(12) - C(11) - H(11B)	113.0 (15)
H(11A) - C(11) - H(11B)	113 (2)
C(6) - C(12) - C(11)	110.41 (17)
C(6) - C(12) - H(12A)	104.1 (10)
C(11) - C(12) - H(12A)	109.2 (10)
C(6) - C(12) - H(12B)	110.9 (11)
C(11) - C(12) - H(12B)	109.3 (10)
H(12A) - C(12) - H(12B)	112.9 (13)
C(14) - C(13) - C(8)	111.38 (18)
C(14) - C(13) - H(13A)	108.9 (10)
C(8) - C(13) - H(13A)	107.5 (10)
C(14) - C(13) - H(13B)	112.0 (12)
C(8) - C(13) - H(13B)	105.0 (12)
H(13A) - C(13) - H(13B)	112.0 (16)
C(22) - C(14) - C(13)	109.35 (18)
C(22) - C(14) - H(14A)	108.9 (12)
C(13) - C(14) - H(14A)	110.0 (11)

C(22) - C(14) - H(14B)	108.3 (12)
C(13) - C(14) - H(14B)	111.0 (13)
H(14A) - C(14) - H(14B)	109.3 (17)
C(9) - C(15) - C(16)	112.3 (2)
C(9) - C(15) - H(15)	126.4 (11)
C(16) - C(15) - H(15)	120.9 (11)
C(17) - C(16) - C(15)	103.45 (19)
C(17) - C(16) - H(16A)	111.6 (15)
C(15) - C(16) - H(16A)	107.6 (13)
C(17) - C(16) - H(16B)	112.0 (15)
C(15) - C(16) - H(16B)	116.0 (15)
H(16A) - C(16) - H(16B)	106 (2)
C(16) - C(17) - C(10)	108.98 (19)
C(16) - C(17) - H(17A)	121 (2)
C(10) - C(17) - H(17A)	118.4 (19)
C(16) - C(17) - H(17B)	106 (3)
C(10) - C(17) - H(17B)	116 (3)
H(17A) - C(17) - H(17B)	84 (3)
C(19) - C(18) - C(2)	101.39 (15)
C(19) - C(18) - C(20)	115.2 (2)
C(2) - C(18) - C(20)	117.56 (19)
C(19) - C(18) - H(16)	108.9 (9)
C(2) - C(18) - H(16)	106.5 (9)
C(20) - C(18) - H(16)	106.7 (9)
N-C(19) - C(18)	103.39 (16)
N-C(19) - H(19A)	112.2 (10)
C(18) - C(19) - H(19A)	109.3 (10)
N-C(19) - H(19B)	112.2 (11)
C(18) - C(19) - H(19B)	114.8 (11)
H(19A) - C(19) - H(19B)	105.1 (15)
C(18) - C(20) - H(20A)	114.4 (15)
C(18) - C(20) - H(20B)	108.8 (17)
H(20A) - C(20) - H(20B)	108 (2)
C(18) - C(20) - H(20C)	113.2 (15)
H(20A) - C(20) - H(20C)	108 (2)
H(20B) - C(20) - H(20C)	105 (2)
C(5) - C(21) - H(21A)	108.9 (11)
C(5) - C(21) - H(21B)	106.5 (11)
H(21A) - C(21) - H(21B)	111.6 (15)
C(5) - C(21) - H(21C)	111.9 (11)
H(21A) - C(21) - H(21C)	108.2 (16)
H(21B) - C(21) - H(21C)	109.8 (17)
O(1) - C(22) - O(2)	119.14 (19)
O(1) - C(22) - C(14)	124.89 (19)
O(2) - C(22) - C(14)	115.79 (18)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for cd22231.
The anisotropic displacement factor exponent takes the form:
 $-2 \pi^2 [h^2 a^{*2} U_{11} + \dots + 2 h k a^* b^* U_{12}]$

	U11	U22	U33	U23	U13	U12
O(1)	46(1)	92(1)	68(1)	-4(1)	17(1)	1(1)
O(2)	38(1)	62(1)	41(1)	4(1)	5(1)	-1(1)
N	47(1)	30(1)	34(1)	1(1)	0(1)	1(1)
C(1)	45(1)	37(1)	32(1)	5(1)	-8(1)	-12(1)
C(2)	62(1)	37(1)	45(1)	8(1)	-9(1)	-11(1)
C(3)	65(2)	55(1)	41(1)	15(1)	-9(1)	4(1)
C(4)	55(1)	52(1)	29(1)	3(1)	-9(1)	-6(1)
C(5)	34(1)	43(1)	34(1)	-1(1)	0(1)	-4(1)
C(6)	40(1)	34(1)	31(1)	0(1)	-1(1)	-5(1)
C(7)	41(1)	34(1)	28(1)	0(1)	-2(1)	-1(1)
C(8)	43(1)	42(1)	30(1)	5(1)	-5(1)	-9(1)
C(9)	45(1)	32(1)	32(1)	1(1)	5(1)	1(1)
C(10)	59(1)	45(1)	36(1)	5(1)	-3(1)	-4(1)
C(11)	78(2)	77(2)	57(1)	32(1)	11(1)	24(2)
C(12)	55(1)	41(1)	52(1)	7(1)	13(1)	11(1)
C(13)	54(1)	57(1)	45(1)	13(1)	2(1)	-14(1)
C(14)	50(1)	85(2)	48(1)	12(1)	14(1)	-13(1)
C(15)	51(1)	46(1)	50(1)	6(1)	10(1)	0(1)
C(16)	77(2)	60(2)	61(1)	16(1)	23(1)	-8(1)
C(17)	75(2)	185(4)	67(2)	75(2)	21(2)	29(2)
C(18)	66(1)	38(1)	51(1)	0(1)	-5(1)	3(1)
C(19)	56(1)	40(1)	50(1)	3(1)	5(1)	6(1)
C(20)	83(2)	45(1)	87(2)	6(1)	-2(2)	16(1)
C(21)	48(1)	49(1)	41(1)	-10(1)	3(1)	-2(1)
C(22)	40(1)	74(1)	43(1)	-7(1)	4(1)	-11(1)

Table 5. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for cd22231.

	x	y	z	U(eq)
H(1)	2210(20)	5402(18)	1114(5)	31(4)
H(2)	3470(20)	3360(20)	1493(5)	34(4)
H(6)	2030(20)	8077(17)	901(5)	25(4)
H(7)	5520(20)	8407(16)	1256(5)	24(4)
H(10)	3730(30)	8470(20)	74(6)	58(6)
H(15)	8280(30)	9150(20)	712(6)	61(6)
H(16)	5150(20)	3419(19)	850(6)	39(4)
H(3A)	6100(30)	4580(20)	1980(6)	54(6)
H(4A)	4040(20)	6686(19)	2332(6)	38(4)
H(11A)	1820(40)	10160(30)	244(9)	102(11)
H(12A)	3810(30)	10970(20)	1051(6)	56(6)
H(13A)	160(20)	5660(20)	1661(6)	46(5)
H(14A)	870(30)	7490(20)	2457(7)	62(6)
H(16A)	7880(30)	11110(30)	116(7)	80(7)
H(17A)	5170(40)	10820(30)	-305(10)	104(9)
H(19A)	7170(30)	5990(20)	1249(6)	43(5)
H(20A)	8290(40)	2660(30)	1036(10)	102(9)
H(21A)	2850(30)	9330(20)	2208(7)	60(6)
H(3B)	4380(30)	4120(20)	2194(7)	62(6)
H(4B)	5360(20)	7148(19)	1911(6)	38(5)
H(11B)	3380(40)	11470(30)	264(9)	116(10)
H(12B)	1410(30)	10860(20)	998(6)	54(5)
H(13B)	1440(20)	5070(20)	2072(7)	58(6)
H(14B)	-960(30)	6640(20)	2357(7)	56(6)
H(16B)	8340(30)	9620(30)	-155(9)	91(8)
H(17B)	5660(60)	9500(60)	-436(16)	197(19)
H(19B)	7070(30)	5650(20)	737(6)	53(5)
H(20B)	6820(40)	1840(40)	1361(9)	110(10)
H(21B)	2590(30)	10660(20)	1804(6)	53(5)
H(20C)	8020(40)	3230(30)	1565(9)	103(9)
H(21C)	4420(30)	9660(20)	1857(6)	47(5)

Table 6. Torsion angles [deg] for cd22231.

C(19)-N-C(1)-C(2)	-4.92(18)
C(7)-N-C(1)-C(2)	-137.96(15)
C(19)-N-C(1)-C(8)	111.71(17)
C(7)-N-C(1)-C(8)	-21.3(2)
N-C(1)-C(2)-C(18)	-20.93(18)
C(8)-C(1)-C(2)-C(18)	-143.98(15)
N-C(1)-C(2)-C(3)	101.05(17)
C(8)-C(1)-C(2)-C(3)	-22.0(2)
C(18)-C(2)-C(3)-C(4)	111.7(2)
C(1)-C(2)-C(3)-C(4)	-1.9(2)
C(2)-C(3)-C(4)-C(8)	25.0(2)
C(22)-O(2)-C(5)-C(21)	-70.88(19)
C(22)-O(2)-C(5)-C(6)	171.85(14)
C(22)-O(2)-C(5)-C(8)	54.20(19)
O(2)-C(5)-C(6)-C(12)	65.35(19)
C(21)-C(5)-C(6)-C(12)	-47.0(2)
C(8)-C(5)-C(6)-C(12)	-175.52(16)
O(2)-C(5)-C(6)-C(7)	-168.59(13)
C(21)-C(5)-C(6)-C(7)	79.01(18)
C(8)-C(5)-C(6)-C(7)	-49.46(18)
C(19)-N-C(7)-C(9)	72.65(19)
C(1)-N-C(7)-C(9)	-157.33(14)
C(19)-N-C(7)-C(6)	-166.18(15)
C(1)-N-C(7)-C(6)	-36.15(18)
C(12)-C(6)-C(7)-N	-155.45(14)
C(5)-C(6)-C(7)-N	74.90(16)
C(12)-C(6)-C(7)-C(9)	-32.36(19)
C(5)-C(6)-C(7)-C(9)	-162.01(13)
C(3)-C(4)-C(8)-C(13)	76.6(2)
C(3)-C(4)-C(8)-C(1)	-37.53(19)
C(3)-C(4)-C(8)-C(5)	-159.88(16)
N-C(1)-C(8)-C(13)	166.74(16)
C(2)-C(1)-C(8)-C(13)	-77.66(19)
N-C(1)-C(8)-C(4)	-78.86(17)
C(2)-C(1)-C(8)-C(4)	36.74(18)
N-C(1)-C(8)-C(5)	44.9(2)
C(2)-C(1)-C(8)-C(5)	160.50(14)
O(2)-C(5)-C(8)-C(13)	-15.03(17)
C(21)-C(5)-C(8)-C(13)	104.59(17)
C(6)-C(5)-C(8)-C(13)	-128.49(15)
O(2)-C(5)-C(8)-C(4)	-138.12(15)
C(21)-C(5)-C(8)-C(4)	-18.5(2)
C(6)-C(5)-C(8)-C(4)	108.42(16)
O(2)-C(5)-C(8)-C(1)	106.16(15)
C(21)-C(5)-C(8)-C(1)	-134.22(16)
C(6)-C(5)-C(8)-C(1)	-7.29(19)

N-C(7)-C(9)-C(15)	-97.1(2)
C(6)-C(7)-C(9)-C(15)	143.25(18)
N-C(7)-C(9)-C(10)	92.75(19)
C(6)-C(7)-C(9)-C(10)	-26.9(2)
C(15)-C(9)-C(10)-C(17)	4.6(3)
C(7)-C(9)-C(10)-C(17)	176.2(2)
C(15)-C(9)-C(10)-C(11)	-119.0(2)
C(7)-C(9)-C(10)-C(11)	52.6(2)
C(9)-C(10)-C(11)-C(12)	-15.5(3)
C(17)-C(10)-C(11)-C(12)	-131.8(3)
C(5)-C(6)-C(12)-C(11)	-167.7(2)
C(7)-C(6)-C(12)-C(11)	67.2(2)
C(10)-C(11)-C(12)-C(6)	-40.3(3)
C(4)-C(8)-C(13)-C(14)	87.9(2)
C(1)-C(8)-C(13)-C(14)	-162.80(17)
C(5)-C(8)-C(13)-C(14)	-38.8(2)
C(8)-C(13)-C(14)-C(22)	63.4(2)
C(7)-C(9)-C(15)-C(16)	-173.23(18)
C(10)-C(9)-C(15)-C(16)	-2.4(2)
C(9)-C(15)-C(16)-C(17)	-0.8(3)
C(15)-C(16)-C(17)-C(10)	3.7(4)
C(9)-C(10)-C(17)-C(16)	-5.0(4)
C(11)-C(10)-C(17)-C(16)	115.4(3)
C(1)-C(2)-C(18)-C(19)	37.88(19)
C(3)-C(2)-C(18)-C(19)	-76.4(2)
C(1)-C(2)-C(18)-C(20)	164.44(18)
C(3)-C(2)-C(18)-C(20)	50.2(3)
C(7)-N-C(19)-C(18)	161.93(14)
C(1)-N-C(19)-C(18)	29.1(2)
C(2)-C(18)-C(19)-N	-40.99(19)
C(20)-C(18)-C(19)-N	-169.06(19)
C(5)-O(2)-C(22)-O(1)	153.94(16)
C(5)-O(2)-C(22)-C(14)	-30.6(2)
C(13)-C(14)-C(22)-O(1)	147.3(2)
C(13)-C(14)-C(22)-O(2)	-27.9(3)

Symmetry transformations used to generate equivalent atoms:

Table 7. Hydrogen bonds for cd22231 [Å and deg.].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
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