Supporting Information

Microbial Metabolism Studies of Cyanthiwigin B

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X-ray analysis and crystal data for cyanthiwigin AE

Table 3. Fractional atomic coordinates and equivalent isotropic displacement parameters

Table 4. Anisotropic displacement parameters

Table 5. Selected geometric parameters

X-ray analysis and crystal data for cyanthiwigin AE

Crystal data

$C_{20}H_{28}O_3$

$M_r = 316.42$

Orthorhombic

$P2_12_12_1$

$a = 8.474$ (3) Å

$b = 10.392$ (3) Å

$c = 19.749$ (8) Å

$V = 1739.1$ (11) Å^3

$Z = 4$

$D_x = 1.208$ Mg m^{-3}

D_m not measured

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 2138 reflections

$\theta = 2.5$–$27.5°$

$\mu = 0.079$ mm^{-1}

$T = 102$ K

Fragment

Colorless

0.37 × 0.15 × 0.07 mm

Crystal source: local laboratory

Data collection

KappaCCD (with Oxford Cryostream) diffractometer

ω scans with κ offsets

Absorption correction: none

3928 measured reflections

2266 independent reflections

1871 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.042$

$\theta_{\rm max} = 27.5°$

$h = -10 \rightarrow 10$

$k = -13 \rightarrow 13$

$l = -25 \rightarrow 25$

intensity decay: <2%

Refinement

Refinement on F^2

$R[F^2 > 2\sigma(F^2)] = 0.042$

$wR(F^2) = 0.104$

$S = 1.055$

2266 reflections

217 parameters

H atoms treated by a mixture of independent and constrained refinement

w=$1/[\sigma^2(F_o^2) + (0.0492P)^2 + 0.2874P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\rm max} = 0.000$

$\Delta\rho_{\rm max} = 0.20$ e Å^{-3}

$\Delta\rho_{\rm min} = -0.20$ e Å^{-3}

Extinction correction: *SHELXL*

Extinction coefficient: 0.013 (3)

Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 3. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)

$$U_{\mathrm{eq}} = (1/3)\Sigma_i\Sigma_j U^{ij} a^i a^j \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
O1	0.5887 (2)	0.70968 (17)	0.32502 (9)	0.0311 (4)
O2	0.74155 (19)	0.59592 (18)	0.45590 (9)	0.0321 (4)
O3	0.18334 (19)	0.37630 (16)	0.64812 (8)	0.0254 (4)
C1	0.4835 (3)	0.6406 (2)	0.34565 (11)	0.0227 (5)
C2	0.3208 (3)	0.6357 (2)	0.32279 (12)	0.0232 (5)
H2	0.2787	0.6879	0.2877	0.028
C3	0.2374 (3)	0.5479 (2)	0.35754 (11)	0.0208 (5)
C4	0.3352 (2)	0.4858 (2)	0.41379 (11)	0.0195 (5)
H4	0.3326	0.3907	0.4063	0.023
C5	0.2617 (3)	0.5123 (2)	0.48550 (11)	0.0193 (5)
H5	0.2001	0.5942	0.4822	0.023
C6	0.3886 (3)	0.5317 (2)	0.54108 (11)	0.0208 (5)
C7	0.5024 (3)	0.6373 (2)	0.51573 (12)	0.0228 (5)
H7A	0.4411	0.7136	0.5014	0.027
H7B	0.5733	0.6636	0.5531	0.027
C8	0.5988 (3)	0.5891 (2)	0.45749 (12)	0.0237 (5)
C9	0.5063 (3)	0.5322 (2)	0.39864 (12)	0.0219 (5)
C10	0.1441 (3)	0.4041 (2)	0.50107 (12)	0.0225 (5)
H10A	0.0969	0.3751	0.4578	0.027
H10B	0.2035	0.3305	0.5201	0.027
C11	0.0104 (3)	0.4370 (3)	0.54969 (12)	0.0250 (5)
H11A	−0.0390	0.5179	0.5339	0.030
H11B	−0.0704	0.3685	0.5459	0.030
C12	0.0501 (3)	0.4529 (2)	0.62356 (12)	0.0245 (5)
C13	0.1941 (3)	0.5161 (2)	0.64768 (12)	0.0236 (5)
H13	0.189 (3)	0.551 (3)	0.6940 (13)	0.028
C14	0.3199 (3)	0.5845 (2)	0.60787 (12)	0.0235 (5)
H14A	0.4103	0.5967	0.6390	0.028
H14B	0.2783	0.6714	0.5977	0.028
C15	−0.0921 (3)	0.4521 (3)	0.66953 (12)	0.0334 (6)
H15A	−0.1467	0.3693	0.6655	0.050
H15B	−0.1640	0.5217	0.6564	0.050
H15C	−0.0580	0.4648	0.7165	0.050
C16	0.4835 (3)	0.4083 (2)	0.55485 (12)	0.0246 (5)
H16A	0.5725	0.4281	0.5848	0.037
H16B	0.5233	0.3737	0.5120	0.037

Table 3. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2) (continued)

H16C	0.4152	0.3444	0.5766	0.037
C17	0.6029 (3)	0.4254 (2)	0.36386 (14)	0.0298 (6)
H17A	0.7037	0.4607	0.3483	0.045
H17B	0.5438	0.3918	0.3250	0.045
H17C	0.6226	0.3556	0.3962	0.045
C18	0.0689 (3)	0.5112 (2)	0.34233 (12)	0.0223 (5)
H18	0.0074	0.5167	0.3854	0.027
C19	0.0615 (3)	0.3716 (2)	0.31698 (12)	0.0265 (5)
H19A	0.1189	0.3645	0.2741	0.040
H19B	−0.0489	0.3468	0.3101	0.040
H19C	0.1098	0.3146	0.3506	0.040
C20	−0.0075 (3)	0.6011 (3)	0.29091 (12)	0.0297 (6)
H20A	−0.0018	0.6899	0.3075	0.044
H20B	−0.1182	0.5767	0.2846	0.044
H20C	0.0485	0.5946	0.2476	0.044

Table 4. Anisotropic displacement parameters (Å^2)

	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O1	0.0334 (10)	0.0289 (9)	0.0310 (10)	−0.0132 (9)	−0.0026 (8)	0.0042 (8)
O2	0.0180 (8)	0.0402 (10)	0.0380 (10)	−0.0027 (8)	−0.0025 (7)	0.0096 (9)
O3	0.0285 (9)	0.0226 (8)	0.0251 (9)	−0.0016 (8)	−0.0039 (7)	0.0027 (7)
C1	0.0266 (12)	0.0198 (11)	0.0219 (12)	−0.0026 (11)	0.0016 (9)	−0.0005 (10)
C2	0.0243 (12)	0.0218 (11)	0.0237 (12)	−0.0021 (11)	−0.0027 (10)	0.0029 (10)
C3	0.0221 (11)	0.0190 (11)	0.0214 (11)	0.0002 (10)	0.0021 (9)	−0.0049 (9)
C4	0.0192 (10)	0.0157 (10)	0.0238 (12)	−0.0025 (9)	0.0009 (9)	−0.0002 (9)
C5	0.0195 (10)	0.0176 (11)	0.0209 (11)	0.0007 (10)	−0.0007 (9)	0.0007 (9)
C6	0.0196 (11)	0.0207 (11)	0.0222 (12)	0.0006 (10)	−0.0020 (9)	0.0001 (10)
C7	0.0200 (11)	0.0211 (11)	0.0273 (12)	−0.0021 (11)	−0.0042 (9)	−0.0004 (10)
C8	0.0214 (11)	0.0210 (12)	0.0288 (13)	−0.0008 (10)	−0.0001 (10)	0.0085 (10)
C9	0.0192 (10)	0.0179 (11)	0.0285 (13)	0.0014 (10)	0.0035 (9)	0.0022 (10)
C10	0.0235 (11)	0.0251 (12)	0.0189 (11)	−0.0061 (10)	−0.0018 (9)	0.0016 (10)
C11	0.0199 (11)	0.0311 (13)	0.0241 (12)	−0.0043 (11)	−0.0027 (9)	0.0025 (11)
C12	0.0233 (11)	0.0263 (12)	0.0240 (12)	0.0018 (11)	0.0001 (9)	0.0012 (10)
C13	0.0274 (12)	0.0221 (12)	0.0212 (12)	0.0017 (10)	−0.0023 (10)	−0.0012 (10)
C14	0.0253 (11)	0.0219 (11)	0.0232 (12)	−0.0008 (10)	−0.0033 (9)	−0.0031 (10)
C15	0.0299 (13)	0.0462 (16)	0.0240 (13)	−0.0030 (14)	0.0023 (11)	0.0001 (12)
C16	0.0232 (11)	0.0230 (12)	0.0277 (13)	0.0026 (10)	−0.0019 (10)	0.0024 (10)
C17	0.0270 (12)	0.0252 (13)	0.0370 (14)	0.0024 (11)	0.0082 (11)	−0.0006 (11)
C18	0.0220 (12)	0.0238 (12)	0.0213 (12)	−0.0010 (10)	0.0006 (9)	0.0002 (9)
C19	0.0238 (12)	0.0302 (13)	0.0256 (13)	−0.0079 (11)	0.0003 (10)	−0.0009 (11)
C20	0.0224 (12)	0.0364 (14)	0.0302 (13)	0.0031 (12)	−0.0011 (10)	0.0066 (11)

Table 5. Selected geometric parameters (Å^2, ^{o})

O1—C1	1.215 (3)	C11—C12	1.506 (3)
O2—C8	1.212 (3)	C11—H11A	0.9900
O3—C13	1.455 (3)	C11—H11B	0.9900
O3—C12	1.464 (3)	C12—C13	1.465 (3)
C1—C2	1.452 (3)	C12—C15	1.509 (3)
C1—C9	1.550 (3)	C13—C14	1.504 (3)
C2—C3	1.343 (3)	C13—H13	0.99 (3)
C2—H2	0.9500	C14—H14A	0.9900
C3—C18	1.508 (3)	C14—H14B	0.9900
C3—C4	1.528 (3)	C15—H15A	0.9800
C4—C9	1.557 (3)	C15—H15B	0.9800
C4—C5	1.571 (3)	C15—H15C	0.9800
C4—H4	1.0000	C16—H16A	0.9800
C5—C10	1.534 (3)	C16—H16B	0.9800
C5—C6	1.550 (3)	C16—H16C	0.9800
C5—H5	1.0000	C17—H17A	0.9800
C6—C16	1.538 (3)	C17—H17B	0.9800
C6—C14	1.543 (3)	C17—H17C	0.9800
C6—C7	1.545 (3)	C18—C20	1.524 (3)
C7—C8	1.497 (3)	C18—C19	1.536 (3)
C7—H7A	0.9900	C18—H18	1.0000
C7—H7B	0.9900	C19—H19A	0.9800
C8—C9	1.522 (3)	C19—H19B	0.9800
C9—C17	1.541 (3)	C19—H19C	0.9800
C10—C11	1.524 (3)	C20—H20A	0.9800
C10—H10A	0.9900	C20—H20B	0.9800
C10—H10B	0.9900	C20—H20C	0.9800
C13—O3—C12	60.24 (15)	C9—C4—C5	119.23 (18)
O1—C1—C2	127.8 (2)	C3—C4—H4	107.3
O1—C1—C9	124.4 (2)	C9—C4—H4	107.3
C2—C1—C9	107.59 (19)	C5—C4—H4	107.3
C3—C2—C1	111.4 (2)	C10—C5—C6	113.82 (18)
C3—C2—H2	124.3	C10—C5—C4	108.04 (18)
C1—C2—H2	124.3	C6—C5—C4	112.70 (18)
C2—C3—C18	124.6 (2)	C10—C5—H5	107.3
C2—C3—C4	111.9 (2)	C6—C5—H5	107.3
C18—C3—C4	123.5 (2)	C4—C5—H5	107.3
C3—C4—C9	103.56 (17)	C16—C6—C14	110.04 (18)
C3—C4—C5	111.48 (17)	C16—C6—C7	108.84 (18)

Table 5. Selected geometric parameters (Å^2, ^{o})
(continued)

C14—C6—C7	105.06 (18)	C12—C13—C14	129.2 (2)
C16—C6—C5	112.34 (19)	O3—C13—H13	111.2 (16)
C14—C6—C5	112.97 (18)	C12—C13—H13	115.5 (15)
C7—C6—C5	107.22 (18)	C14—C13—H13	110.0 (16)
C8—C7—C6	110.62 (19)	C13—C14—C6	123.10 (19)
C8—C7—H7A	109.5	C13—C14—H14A	106.6
C6—C7—H7A	109.5	C6—C14—H14A	106.6
C8—C7—H7B	109.5	C13—C14—H14B	106.6
C6—C7—H7B	109.5	C6—C14—H14B	106.6
H7A—C7—H7B	108.1	H14A—C14—H14B	106.5
O2—C8—C7	123.0 (2)	C12—C15—H15A	109.5
O2—C8—C9	121.2 (2)	C12—C15—H15B	109.5
C7—C8—C9	115.83 (19)	H15A—C15—H15B	109.5
C8—C9—C17	110.28 (19)	C12—C15—H15C	109.5
C8—C9—C1	107.28 (19)	H15A—C15—H15C	109.5
C17—C9—C1	106.78 (19)	H15B—C15—H15C	109.5
C8—C9—C4	116.96 (19)	C6—C16—H16A	109.5
C17—C9—C4	110.96 (19)	C6—C16—H16B	109.5
C1—C9—C4	103.83 (17)	H16A—C16—H16B	109.5
C11—C10—C5	116.4 (2)	C6—C16—H16C	109.5
C11—C10—H10A	108.2	H16A—C16—H16C	109.5
C5—C10—H10A	108.2	H16B—C16—H16C	109.5
C11—C10—H10B	108.2	C9—C17—H17A	109.5
C5—C10—H10B	108.2	C9—C17—H17B	109.5
H10A—C10—H10B	107.3	H17A—C17—H17B	109.5
C12—C11—C10	117.96 (19)	C9—C17—H17C	109.5
C12—C11—H11A	107.8	H17A—C17—H17C	109.5
C10—C11—H11A	107.8	H17B—C17—H17C	109.5
C12—C11—H11B	107.8	C3—C18—C20	112.3 (2)
C10—C11—H11B	107.8	C3—C18—C19	110.0 (2)
H11A—C11—H11B	107.2	C20—C18—C19	110.1 (2)
O3—C12—C13	59.57 (14)	C3—C18—H18	108.1
O3—C12—C11	115.7 (2)	C20—C18—H18	108.1
C13—C12—C11	123.4 (2)	C19—C18—H18	108.1
O3—C12—C15	114.4 (2)	C18—C19—H19A	109.5
C13—C12—C15	118.2 (2)	C18—C19—H19B	109.5
C11—C12—C15	113.8 (2)	H19A—C19—H19B	109.5
O3—C13—C12	60.19 (15)	C18—C19—H19C	109.5
O3—C13—C14	121.3 (2)	H19A—C19—H19C	109.5

Table 5. Selected geometric parameters (Å^2, ^{o})
(continued)

H19B—C19—H19C	109.5	C18—C20—H20C	109.5
C18—C20—H20A	109.5	H20A—C20—H20C	109.5
C18—C20—H20B	109.5	H20B—C20—H20C	109.5
H20A—C20—H20B	109.5		
O1—C1—C2—C3	−179.6 (2)	C2—C1—C9—C17	106.1 (2)
C9—C1—C2—C3	5.0 (3)	O1—C1—C9—C4	173.2 (2)
C1—C2—C3—C18	−174.6 (2)	C2—C1—C9—C4	−11.2 (2)
C1—C2—C3—C4	3.8 (3)	C3—C4—C9—C8	130.6 (2)
C2—C3—C4—C9	−10.8 (3)	C5—C4—C9—C8	6.1 (3)
C18—C3—C4—C9	167.7 (2)	C3—C4—C9—C17	−101.7 (2)
C2—C3—C4—C5	118.6 (2)	C5—C4—C9—C17	133.8 (2)
C18—C3—C4—C5	−62.9 (3)	C3—C4—C9—C1	12.7 (2)
C3—C4—C5—C10	89.3 (2)	C5—C4—C9—C1	−111.8 (2)
C9—C4—C5—C10	−150.08 (19)	C6—C5—C10—C11	80.2 (2)
C3—C4—C5—C6	−144.05 (19)	C4—C5—C10—C11	−153.81 (19)
C9—C4—C5—C6	−23.5 (3)	C5—C10—C11—C12	−72.3 (3)
C10—C5—C6—C16	57.0 (3)	C13—O3—C12—C11	115.2 (2)
C4—C5—C6—C16	−66.4 (2)	C13—O3—C12—C15	−109.5 (2)
C10—C5—C6—C14	−68.2 (2)	C10—C11—C12—O3	−30.0 (3)
C4—C5—C6—C14	168.35 (18)	C10—C11—C12—C13	39.2 (4)
C10—C5—C6—C7	176.55 (18)	C10—C11—C12—C15	−165.5 (2)
C4—C5—C6—C7	53.1 (2)	C12—O3—C13—C14	−120.3 (3)
C16—C6—C7—C8	52.3 (2)	C11—C12—C13—O3	−102.4 (3)
C14—C6—C7—C8	170.08 (18)	C15—C12—C13—O3	103.3 (2)
C5—C6—C7—C8	−69.5 (2)	O3—C12—C13—C14	107.8 (3)
C6—C7—C8—O2	−127.9 (2)	C11—C12—C13—C14	5.4 (4)
C6—C7—C8—C9	53.3 (3)	C15—C12—C13—C14	−149.0 (2)
O2—C8—C9—C17	32.3 (3)	O3—C13—C14—C6	31.5 (3)
C7—C8—C9—C17	−148.8 (2)	C12—C13—C14—C6	−43.7 (3)
O2—C8—C9—C1	−83.7 (3)	C16—C6—C14—C13	−67.4 (3)
C7—C8—C9—C1	95.2 (2)	C7—C6—C14—C13	175.6 (2)
O2—C8—C9—C4	160.3 (2)	C5—C6—C14—C13	59.1 (3)
C7—C8—C9—C4	−20.8 (3)	C2—C3—C18—C20	−10.1 (3)
O1—C1—C9—C8	48.7 (3)	C4—C3—C18—C20	171.65 (19)
C2—C1—C9—C8	−135.65 (19)	C2—C3—C18—C19	113.0 (3)
O1—C1—C9—C17	−69.5 (3)	C4—C3—C18—C19	−65.3 (3)