NP030519J

Crystal data and structure refinement of compound **4**

Crystal data

$C_{18}H_{18}O_5$
$M_r = 314.337$
Orthorhombic
$P2_12_12_1$
a = 9.1692 (2) A
b = 10.5530 (3) A
c = 15.8312 (5) A
$\alpha = 90.00°$
$\beta = 90.00°$
$\gamma = 90.00°$
V = 1531.87 (7) A^3
Z = 4
$D_x = 1.363$ Mg m^{-3}

Density measured by: not measured
fine-focus sealed tube
Mo $K\alpha$ radiation
$\lambda = 0.71073$ A
Cell parameters from 1978 reflections
$\mu = 0.096$ mm^{-1}
T = 298 K
Prism
Colourless
Crystal source: *Dendrolobium lanceolatum* (Dunn) Schindl

Data collection

KappaCCD
Absorption correction: none
16122 measured reflections
2008 independent reflections
1823 observed reflections
Criterion: >2sigma(I)

$\theta_{max} = 27.48°$
h = 0 →11
k = 0 →13
l = 0 →20

Refinement

Refinement on F^2
fullmatrix least squares refinement
R(all) = 0.0426
R(gt) = 0.0374
wR(ref) = 0.1050
wR(gt)= 0.1000
S(ref) = 1.040
2008 reflections
209 parameters
0 restraints
Only coordinates of H atoms refined

Calculated weights calc
$\Delta/\sigma_{max} = 0.000$
$\Delta\rho_{max} = 0.171e$ A^3
$\Delta\rho_{min} = -0.184e$ A^3
Extinction correction: *SHELXL*
$Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient = 0.033 (5)
Atomic scattering factors from International Tables Vol C Tables 4.2.6.8 and 6.1.1.4

Data collection: KappaCCD
Cell refinement: HKL Scalepack (Otwinowski & Minor 1997)
Data reduction: Denzo and Scalepak (Otwinowski & Minor, 1997)
Program(s) used to solve structure: *SIR*97(Cascarano al.,*Acta Cryst.*,1996,A52,C-79)
Program(s) used to refine structure: *SHELXL*-97 (Sheldrick, 1997)

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters ($Å^2$)*
$U_{eq} = 1/3\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \boldsymbol{a}_i.\boldsymbol{a}_j$.

	x	y	z	U_{eq}	Occ
O2	1.33327 (18)	0.12024 (15)	0.52223 (11)	0.0485 (4)	1
O8	0.53430 (16)	-0.15351 (17)	0.33280 (11)	0.0479 (4)	1
O10	0.95729 (18)	-0.40425 (15)	0.39623 (13)	0.0502 (4)	1
O9	0.66398 (19)	-0.39377 (15)	0.35653 (11)	0.0448 (4)	1
C1A	1.0055 (2)	0.05093 (19)	0.39714 (13)	0.0316 (4)	1
C11	0.9707 (2)	-0.1806 (2)	0.39417 (14)	0.0372 (4)	1
C4A	0.9931 (2)	0.1639 (2)	0.35133 (12)	0.0335 (4)	1
C9	0.7413 (2)	-0.2826 (2)	0.36663 (14)	0.0365 (5)	1
C5	0.8701 (3)	0.1822 (2)	0.28885 (15)	0.0438 (5)	1
C8	0.6774 (2)	-0.1644 (2)	0.35826 (13)	0.0363 (5)	1
C1	1.1208 (2)	0.04058 (19)	0.45569 (14)	0.0343 (4)	1
C11A	0.9088 (2)	-0.06027 (19)	0.38475 (12)	0.0329 (4)	1
C3	1.2107 (2)	0.2467 (2)	0.41854 (14)	0.0370 (5)	1
O3	1.32066 (19)	0.33137 (15)	0.43195 (12)	0.0503 (4)	1
C4	1.0954 (2)	0.2614 (2)	0.36259 (14)	0.0368 (4)	1
C7A	0.7571 (2)	-0.0517 (2)	0.36828 (13)	0.0345 (4)	1
C2	1.2215 (2)	0.13568 (19)	0.46566 (13)	0.0353 (4)	1
C7	0.6761 (2)	0.0688 (2)	0.36619 (15)	0.0420 (5)	1
C6	0.7251 (3)	0.1750 (2)	0.33182 (17)	0.0453 (5)	1
C10	0.8891 (2)	-0.2900 (2)	0.38654 (14)	0.0373 (5)	1
C14	0.6593 (4)	-0.4378 (3)	0.27162 (18)	0.0650 (8)	1
C13	0.4263 (3)	-0.2012 (3)	0.3875 (2)	0.0658 (8)	1
C12	1.3150 (3)	0.4497 (3)	0.3902 (2)	0.0619 (8)	1
H2	1.3739	0.1886	0.5297	0.058	1
H10	0.8959	-0.4589	0.4056	0.060	1
H11	1.0728	-0.1890	0.4064	0.045	1
H5A	0.8794	0.2631	0.2616	0.053	1
H5B	0.8742	0.1162	0.2472	0.053	1
H1	1.1303	-0.0347	0.4892	0.041	1
H4	1.0869	0.3389	0.3312	0.044	1
H7	0.5806	0.0738	0.3908	0.050	1
H6	0.6657	0.2497	0.3362	0.054	1
H14A	0.6046	-0.5152	0.2686	0.078	1
H14B	0.7564	-0.4522	0.2511	0.078	1
H14C	0.6127	-0.3743	0.2376	0.078	1
H13A	0.3318	-0.1872	0.3630	0.079	1
H13B	0.4318	-0.1591	0.4412	0.079	1
H13C	0.4415	-0.2904	0.3952	0.079	1
H12A	1.3985	0.4994	0.4056	0.074	1
H12B	1.2277	0.4936	0.4064	0.074	1
H12C	1.3148	0.4364	0.3302	0.074	1

Table 2. *Anisotropic displacement parameters (A^2)*

	U_{11}	U_{12}	U_{13}	U_{22}	U_{23}	U_{33}
O2	0.0436 (9)	-0.0063 (7)	-0.0227 (8)	0.0403 (8)	0.0042 (7)	0.0618 (10)
O8	0.0296 (7)	-0.0092 (7)	-0.0099 (7)	0.0566 (10)	0.0173 (8)	0.0575 (10)
O10	0.0404 (9)	0.0000 (7)	0.0050 (9)	0.0312 (8)	0.0052 (8)	0.0790 (12)
O9	0.0476 (9)	-0.0155 (7)	-0.0003 (8)	0.0385 (8)	0.0031 (7)	0.0484 (9)
C1A	0.0278 (8)	-0.0010 (8)	0.0005 (8)	0.0322 (9)	-0.0008 (8)	0.0349 (10)
C11	0.0308 (9)	-0.0021 (9)	-0.0007 (9)	0.0370 (10)	-0.0011 (9)	0.0438 (11)
C4A	0.0314 (10)	-0.0004 (8)	-0.0021 (8)	0.0349 (10)	0.0000 (8)	0.0342 (9)
C9	0.0359 (10)	-0.0090 (9)	-0.0005 (9)	0.0350 (10)	0.0022 (8)	0.0385 (10)
C5	0.0448 (12)	-0.0067 (10)	-0.0120 (9)	0.0428 (11)	0.0087 (10)	0.0436 (11)
C8	0.0301 (10)	-0.0037 (9)	-0.0047 (8)	0.0412 (11)	0.0055 (9)	0.0376 (10)
C1	0.0329 (9)	-0.0010 (8)	-0.0021 (8)	0.0317 (9)	0.0018 (8)	0.0383 (10)
C11A	0.0321 (9)	-0.0025 (8)	0.0006 (8)	0.0331 (10)	-0.0002 (8)	0.0335 (10)
C3	0.0330 (10)	-0.0055 (8)	-0.0004 (8)	0.0341 (10)	-0.0026 (9)	0.0440 (11)
O3	0.0452 (9)	-0.0150 (8)	-0.0152 (8)	0.0397 (8)	0.0073 (8)	0.0661 (11)
C4	0.0361 (10)	-0.0034 (9)	-0.0010 (9)	0.0338 (10)	0.0032 (9)	0.0405 (10)
C7A	0.0314 (9)	-0.0029 (9)	-0.0023 (8)	0.0357 (10)	0.0009 (8)	0.0363 (10)
C2	0.0323 (10)	0.0008 (8)	-0.0059 (8)	0.0349 (10)	-0.0018 (8)	0.0388 (10)
C7	0.0300 (10)	0.0017 (10)	-0.0037 (10)	0.0435 (12)	0.0017 (10)	0.0524 (12)
C6	0.0367 (11)	0.0030 (10)	-0.0138 (10)	0.0390 (11)	0.0067 (11)	0.0601 (13)
C10	0.0358 (10)	-0.0010 (8)	0.0022 (9)	0.0312 (10)	0.0011 (8)	0.0448 (11)
C14	0.0742 (18)	-0.0299 (16)	-0.0021 (14)	0.0633 (17)	-0.0120 (14)	0.0576 (15)
C13	0.0376 (12)	-0.0010 (14)	0.0085 (13)	0.084 (2)	0.0220 (17)	0.0757 (19)
C12	0.0590 (15)	-0.0184 (13)	-0.0172 (15)	0.0459 (13)	0.0155 (13)	0.0808 (19)

Table 3 . *Geometric parameters (A, °)*

O2—C2	1.371 (2)	O3—C12	1.414 (3)
O8—C8	1.377 (2)	C7A—C7	1.472 (3)
O8—C13	1.408 (3)	C7—C6	1.325 (3)
O10—C10	1.367 (3)	O2—H2	0.8200
O9—C9	1.380 (2)	O10—H10	0.8200
O9—C14	1.423 (3)	C11—H11	0.9600
C1A—C4A	1.400 (3)	C5—H5A	0.9600
C1A—C1	1.410 (3)	C5—H5B	0.9600
C1A—C11A	1.484 (3)	C1—H1	0.9600
C11—C10	1.380 (3)	C4—H4	0.9601
C11—C11A	1.399 (3)	C7—H7	0.9599
C4A—C4	1.404 (3)	C6—H6	0.9600
C4A—C5	1.513 (3)	C14—H14A	0.9600
C9—C8	1.384 (3)	C14—H14B	0.9600
C9—C10	1.394 (3)	C14—H14C	0.9600
C5—C6	1.495 (3)	C13—H13A	0.9600
C8—C7A	1.405 (3)	C13—H13B	0.9601
C1—C2	1.373 (3)	C13—H13C	0.9600
C11A—C7A	1.418 (3)	C12—H12A	0.9600
C3—O3	1.364 (2)	C12—H12B	0.9601
C3—C4	1.388 (3)	C12—H12C	0.9599
C3—C2	1.393 (3)		
		C11—C10—C9	120.0 (2)
C8—O8—C13	117.40 (19)	C2—O2—H2	109.2
C9—O9—C14	113.74 (19)	C10—O10—H10	109.1
C4A—C1A—C1	117.85 (18)	C10—C11—H11	118.0
C4A—C1A—C11A	123.82 (18)	C11A—C11—H11	120.0
C1—C1A—C11A	118.26 (17)	C6—C5—H5A	109.2
C10—C11—C11A	122.00 (19)	C4A—C5—H5A	110.0
C1A—C4A—C4	120.27 (19)	C6—C5—H5B	108.1
C1A—C4A—C5	120.56 (19)	C4A—C5—H5B	109.1
C4—C4A—C5	119.17 (19)	H5A—C5—H5B	109.5
O9—C9—C8	122.53 (19)	C2—C1—H1	118.7
O9—C9—C10	118.5 (2)	C1A—C1—H1	119.7
C8—C9—C10	118.93 (19)	C3—C4—H4	119.2
C6—C5—C4A	111.02 (19)	C4A—C4—H4	120.3
O8—C8—C9	120.43 (19)	C6—C7—H7	115.4
O8—C8—C7A	117.22 (19)	C7A—C7—H7	119.9
C9—C8—C7A	122.15 (18)	C7—C6—H6	118.2
C2—C1—C1A	121.54 (19)	C5—C6—H6	119.7
C11—C11A—C7A	118.38 (18)	O9—C14—H14A	109.9
C11—C11A—C1A	117.51 (17)	O9—C14—H14B	110.1
C7A—C11A—C1A	124.03 (18)	H14A—C14—H14B	109.5
O3—C3—C4	126.11 (19)	O9—C14—H14C	108.4
O3—C3—C2	114.54 (19)	H14A—C14—H14C	109.5
C4—C3—C2	119.34 (19)	H14B—C14—H14C	109.5
C3—O3—C12	118.60 (19)	O8—C13—H13A	109.4
C3—C4—C4A	120.54 (19)	O8—C13—H13B	110.0
C8—C7A—C11A	118.46 (19)	H13A—C13—H13B	109.5
C8—C7A—C7	117.80 (18)	O8—C13—H13C	109.0
C11A—C7A—C7	123.64 (19)	H13A—C13—H13C	109.5
C1—C2—O2	119.40 (18)	H13B—C13—H13C	109.5
C1—C2—C3	120.39 (19)	O3—C12—H12A	109.6
O2—C2—C3	120.20 (19)	O3—C12—H12B	109.3
C6—C7—C7A	124.7 (2)	H12A—C12—H12B	109.5
C7—C6—C5	122.1 (2)	O3—C12—H12C	109.5
O10—C10—C11	118.67 (19)	H12A—C12—H12C	109.5
O10—C10—C9	121.30 (19)	H12B—C12—H12C	109.5