supplementary materials

Title

Computing details

Data collection: Bruker *APEX2*; cell refinement: Bruker *SAINT*; data reduction: Bruker *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *SHELXL97* (Sheldrick, 2008).

(A522B30)

Crystal data

$C_{11}H_{16}O_3$
$M_r = 196.24$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 7.1064\ (10)$ Å
$b = 10.1181\ (18)$ Å
$c = 14.701\ (2)$ Å
$\alpha = 92.151\ (12)°$
$\beta = 90.261\ (9)°$
$\gamma = 94.803\ (10)°$
$V = 1052.6\ (3)$ Å^3
$Z = 4$
$F(000) = 424$
$D_x = 1.238$ Mg m^{-3}
Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å
Cell parameters from 3606 reflections
$\theta = 4.4–66.8°$
$\mu = 0.73$ mm^{-1}
$T = 100$ K
Lath, colourless
0.40 × 0.09 × 0.01 mm

Data collection

Bruker Kappa APEX-II DUO diffractometer
Radiation source: IμS microfocus
QUAZAR multilayer optics monochromator
φ and ω scans
Absorption correction: multi-scan *SADABS* (Sheldrick, 2004)
$T_{min} = 0.760$, $T_{max} = 0.993$
12697 measured reflections
3628 independent reflections
2604 reflections with $I > 2\sigma(I)$
$R_{int} = 0.038$
$\theta_{max} = 67.7°$, $\theta_{min} = 4.4°$
$h = -8\rightarrow7$
$k = -12\rightarrow11$
$l = -16\rightarrow17$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.048$
$wR(F^2) = 0.137$
$S = 1.03$
3628 reflections
295 parameters
4 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0616P)^2 + 0.5257P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.51$ e Å^{-3}
$\Delta\rho_{min} = -0.29$ e Å^{-3}

Special details

Refinement

Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
O1	0.17616 (19)	0.19910 (16)	0.87649 (10)	0.0359 (4)	
O2	0.4542 (2)	0.11637 (18)	0.88113 (10)	0.0418 (4)	
O3	0.05843 (19)	0.11705 (16)	1.13956 (9)	0.0351 (4)	
C1	0.3128 (3)	0.1387 (2)	0.92386 (15)	0.0342 (5)	
C2	0.2741 (3)	0.1100 (2)	1.01536 (14)	0.0328 (5)	
H2	0.3636	0.0687	1.0505	0.039*	
C3	0.1081 (3)	0.1415 (2)	1.05346 (14)	0.0308 (5)	
C4	−0.0310 (3)	0.2048 (2)	1.00229 (14)	0.0314 (5)	
C5	0.0084 (3)	0.2300 (2)	0.91579 (15)	0.0334 (5)	
C6	−0.1083 (3)	0.2942 (3)	0.84814 (16)	0.0401 (6)	
H6A	−0.0731	0.3900	0.8498	0.060*	
H6B	−0.2423	0.2779	0.8632	0.060*	
H6C	−0.0862	0.2569	0.7870	0.060*	
C7	−0.2086 (3)	0.2433 (2)	1.04914 (15)	0.0366 (6)	
H7A	−0.2553	0.1717	1.0894	0.044*	
H7B	−0.3074	0.2529	1.0027	0.044*	
C8	−0.1742 (3)	0.3734 (3)	1.10548 (18)	0.0510 (7)	
H8A	−0.1500	0.4473	1.0637	0.061*	
H8B	−0.0597	0.3694	1.1436	0.061*	
C9	−0.3423 (5)	0.4031 (4)	1.1681 (2)	0.0791 (11)	
H9A	−0.3794	0.3231	1.2028	0.095*	
H9B	−0.2995	0.4754	1.2125	0.095*	
C10	−0.5093 (5)	0.4415 (4)	1.1195 (3)	0.0777 (10)	
H10A	−0.5492	0.3727	1.0731	0.117*	
H10B	−0.4779	0.5259	1.0903	0.117*	
H10C	−0.6120	0.4515	1.1628	0.117*	
C11	0.1963 (3)	0.0633 (3)	1.19711 (15)	0.0405 (6)	
H11A	0.3047	0.1290	1.2067	0.061*	
H11B	0.2386	−0.0174	1.1678	0.061*	
H11C	0.1393	0.0421	1.2559	0.061*	
O4	0.67524 (19)	0.19602 (15)	0.63291 (9)	0.0335 (4)	
O5	0.9538 (2)	0.11362 (18)	0.62534 (10)	0.0432 (4)	
O6	0.56163 (19)	0.11883 (16)	0.36371 (9)	0.0353 (4)	
C12	0.8125 (3)	0.1372 (2)	0.58281 (14)	0.0332 (5)	
C13	0.7761 (3)	0.1114 (2)	0.48899 (14)	0.0320 (5)	
H13	0.8677	0.0731	0.4518	0.038*	
C14	0.6098 (3)	0.1415 (2)	0.45174 (14)	0.0300 (5)	
C15	0.4695 (3)	0.2025 (2)	0.50602 (15)	0.0331 (5)	
C16	0.5089 (3)	0.2273 (2)	0.59468 (14)	0.0320 (5)	
C17	0.3913 (3)	0.2922 (3)	0.66436 (16)	0.0416 (6)	
H17A	0.4274	0.3879	0.6678	0.062*	
H17B	0.4117	0.2549	0.7238	0.062*	
H17C	0.2577	0.2765	0.6472	0.062*	
C18A	0.3043 (13)	0.2602 (11)	0.4554 (9)	0.029 (3)	0.469 (8)

H18A	0.2510	0.1912	0.4109	0.035*	0.469 (8)
H18B	0.2046	0.2749	0.5006	0.035*	0.469 (8)
C19A	0.3432 (11)	0.3883 (9)	0.4050 (5)	0.0351 (18)	0.469 (8)
H19A	0.4586	0.3814	0.3682	0.042*	0.469 (8)
H19B	0.3696	0.4623	0.4504	0.042*	0.469 (8)
C20A	0.1832 (9)	0.4231 (7)	0.3419 (4)	0.0465 (19)	0.469 (8)
H20A	0.2300	0.4993	0.3056	0.056*	0.469 (8)
H20B	0.1509	0.3467	0.2990	0.056*	0.469 (8)
C21A	0.0059 (9)	0.4574 (8)	0.3904 (5)	0.061 (2)	0.469 (8)
H21A	−0.0893	0.4769	0.3455	0.092*	0.469 (8)
H21B	0.0348	0.5354	0.4312	0.092*	0.469 (8)
H21C	−0.0430	0.3822	0.4260	0.092*	0.469 (8)
C18B	0.2745 (11)	0.2181 (10)	0.4664 (8)	0.0252 (18)	0.531 (8)
H18C	0.2424	0.1485	0.4184	0.030*	0.531 (8)
H18D	0.1779	0.2098	0.5146	0.030*	0.531 (8)
C19B	0.2796 (9)	0.3539 (8)	0.4267 (5)	0.0343 (15)	0.531 (8)
H19C	0.3873	0.3652	0.3844	0.041*	0.531 (8)
H19D	0.2990	0.4227	0.4762	0.041*	0.531 (8)
C20B	0.0966 (7)	0.3723 (5)	0.3757 (4)	0.0382 (15)	0.531 (8)
H20C	0.0876	0.3125	0.3207	0.046*	0.531 (8)
H20D	−0.0120	0.3467	0.4152	0.046*	0.531 (8)
C21B	0.0847 (8)	0.5145 (6)	0.3476 (4)	0.0552 (18)	0.531 (8)
H21D	−0.0346	0.5213	0.3151	0.083*	0.531 (8)
H21E	0.1905	0.5399	0.3076	0.083*	0.531 (8)
H21F	0.0904	0.5739	0.4019	0.083*	0.531 (8)
C22	0.6998 (3)	0.0659 (3)	0.30421 (15)	0.0410 (6)	
H22A	0.8084	0.1315	0.2992	0.062*	
H22B	0.6434	0.0455	0.2438	0.062*	
H22C	0.7414	−0.0154	0.3292	0.062*	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0258 (7)	0.0541 (10)	0.0288 (8)	0.0129 (7)	−0.0015 (6)	−0.0047 (7)
O2	0.0265 (8)	0.0708 (12)	0.0295 (8)	0.0159 (7)	0.0014 (6)	−0.0082 (8)
O3	0.0259 (7)	0.0554 (10)	0.0257 (8)	0.0161 (7)	−0.0015 (6)	−0.0044 (7)
C1	0.0225 (10)	0.0490 (14)	0.0314 (12)	0.0111 (9)	−0.0055 (9)	−0.0097 (10)
C2	0.0227 (10)	0.0487 (14)	0.0277 (11)	0.0118 (9)	−0.0046 (8)	−0.0074 (10)
C3	0.0252 (10)	0.0423 (13)	0.0250 (11)	0.0089 (9)	−0.0028 (8)	−0.0094 (10)
C4	0.0241 (10)	0.0415 (13)	0.0293 (12)	0.0109 (9)	−0.0043 (8)	−0.0099 (10)
C5	0.0236 (10)	0.0428 (14)	0.0343 (12)	0.0098 (9)	−0.0030 (9)	−0.0090 (10)
C6	0.0353 (12)	0.0507 (15)	0.0362 (13)	0.0142 (11)	−0.0048 (10)	0.0014 (11)
C7	0.0236 (10)	0.0542 (15)	0.0332 (12)	0.0156 (10)	−0.0039 (9)	−0.0086 (11)
C8	0.0408 (14)	0.0626 (18)	0.0508 (16)	0.0221 (12)	−0.0064 (11)	−0.0206 (14)
C9	0.078 (2)	0.083 (2)	0.081 (2)	0.0482 (19)	−0.0105 (18)	−0.0382 (19)
C10	0.067 (2)	0.078 (2)	0.092 (3)	0.0345 (18)	0.0070 (18)	−0.0028 (19)
C11	0.0338 (12)	0.0634 (17)	0.0268 (12)	0.0223 (11)	−0.0035 (9)	−0.0035 (11)
O4	0.0265 (7)	0.0492 (10)	0.0262 (8)	0.0150 (7)	−0.0032 (6)	−0.0065 (7)
O5	0.0343 (8)	0.0691 (12)	0.0292 (8)	0.0268 (8)	−0.0078 (7)	−0.0060 (8)
O6	0.0295 (8)	0.0557 (10)	0.0229 (8)	0.0195 (7)	−0.0046 (6)	−0.0068 (7)
C12	0.0270 (11)	0.0456 (14)	0.0288 (12)	0.0159 (9)	−0.0024 (9)	−0.0029 (10)
C13	0.0276 (11)	0.0440 (14)	0.0262 (11)	0.0168 (9)	−0.0012 (8)	−0.0042 (10)
C14	0.0284 (11)	0.0389 (13)	0.0236 (11)	0.0092 (9)	−0.0026 (8)	−0.0022 (9)
C15	0.0225 (10)	0.0458 (14)	0.0318 (12)	0.0106 (9)	−0.0026 (9)	−0.0056 (10)

C16	0.0221 (10)	0.0434 (14)	0.0311 (12)	0.0094 (9)	−0.0020 (8)	−0.0043 (10)
C17	0.0309 (12)	0.0620 (17)	0.0327 (13)	0.0139 (11)	0.0009 (9)	−0.0101 (12)
C18A	0.015 (3)	0.045 (8)	0.027 (4)	−0.002 (4)	0.005 (3)	−0.002 (5)
C19A	0.030 (4)	0.041 (5)	0.037 (4)	0.016 (3)	−0.002 (3)	0.008 (3)
C20A	0.049 (4)	0.047 (4)	0.046 (4)	0.014 (3)	−0.012 (3)	0.014 (3)
C21A	0.042 (4)	0.063 (5)	0.083 (5)	0.026 (4)	−0.009 (3)	0.016 (4)
C18B	0.015 (3)	0.034 (5)	0.027 (3)	0.001 (3)	0.001 (3)	0.005 (3)
C19B	0.029 (3)	0.028 (4)	0.047 (4)	0.005 (3)	−0.005 (3)	0.010 (3)
C20B	0.028 (2)	0.039 (3)	0.049 (3)	0.011 (2)	−0.005 (2)	0.015 (2)
C21B	0.042 (3)	0.053 (4)	0.075 (4)	0.019 (3)	−0.002 (3)	0.033 (3)
C22	0.0369 (12)	0.0633 (17)	0.0259 (12)	0.0267 (11)	−0.0023 (9)	−0.0081 (11)

Geometric parameters (Å, º)

O1—C5	1.381 (2)	C13—H13	0.9500
O1—C1	1.388 (3)	C14—C15	1.444 (3)
O2—C1	1.220 (2)	C15—C16	1.342 (3)
O3—C3	1.342 (3)	C15—C18B	1.524 (8)
O3—C11	1.445 (3)	C15—C18A	1.553 (10)
C1—C2	1.410 (3)	C16—C17	1.492 (3)
C2—C3	1.365 (3)	C17—H17A	0.9800
C2—H2	0.9500	C17—H17B	0.9800
C3—C4	1.445 (3)	C17—H17C	0.9800
C4—C5	1.333 (3)	C18A—C19A	1.523 (9)
C4—C7	1.513 (3)	C18A—H18A	0.9900
C5—C6	1.493 (3)	C18A—H18B	0.9900
C6—H6A	0.9800	C19A—C20A	1.535 (8)
C6—H6B	0.9800	C19A—H19A	0.9900
C6—H6C	0.9800	C19A—H19B	0.9900
C7—C8	1.530 (3)	C20A—C21A	1.510 (9)
C7—H7A	0.9900	C20A—H20A	0.9900
C7—H7B	0.9900	C20A—H20B	0.9900
C8—C9	1.554 (4)	C21A—H21A	0.9800
C8—H8A	0.9900	C21A—H21B	0.9800
C8—H8B	0.9900	C21A—H21C	0.9800
C9—C10	1.469 (4)	C18B—C19B	1.510 (8)
C9—H9A	0.9900	C18B—H18C	0.9900
C9—H9B	0.9900	C18B—H18D	0.9900
C10—H10A	0.9800	C19B—C20B	1.526 (7)
C10—H10B	0.9800	C19B—H19C	0.9900
C10—H10C	0.9800	C19B—H19D	0.9900
C11—H11A	0.9800	C20B—C21B	1.521 (8)
C11—H11B	0.9800	C20B—H20C	0.9900
C11—H11C	0.9800	C20B—H20D	0.9900
O4—C16	1.373 (2)	C21B—H21D	0.9800
O4—C12	1.386 (2)	C21B—H21E	0.9800
O5—C12	1.225 (2)	C21B—H21F	0.9800
O6—C14	1.343 (2)	C22—H22A	0.9800
O6—C22	1.441 (2)	C22—H22B	0.9800
C12—C13	1.413 (3)	C22—H22C	0.9800
C13—C14	1.362 (3)		
C5—O1—C1	122.37 (17)	C16—C15—C18A	123.6 (5)
C3—O3—C11	117.44 (15)	C14—C15—C18A	117.8 (5)

O2—C1—O1	115.8 (2)	C18B—C15—C18A	18.4 (4)
O2—C1—C2	127.1 (2)	C15—C16—O4	121.73 (18)
O1—C1—C2	117.05 (17)	C15—C16—C17	127.92 (19)
C3—C2—C1	120.0 (2)	O4—C16—C17	110.33 (17)
C3—C2—H2	120.0	C16—C17—H17A	109.5
C1—C2—H2	120.0	C16—C17—H17B	109.5
O3—C3—C2	124.18 (19)	H17A—C17—H17B	109.5
O3—C3—C4	114.26 (17)	C16—C17—H17C	109.5
C2—C3—C4	121.57 (19)	H17A—C17—H17C	109.5
C5—C4—C3	117.27 (18)	H17B—C17—H17C	109.5
C5—C4—C7	123.34 (19)	C19A—C18A—C15	119.0 (6)
C3—C4—C7	119.35 (19)	C19A—C18A—H18A	107.6
C4—C5—O1	121.75 (19)	C15—C18A—H18A	107.6
C4—C5—C6	128.45 (19)	C19A—C18A—H18B	107.6
O1—C5—C6	109.78 (18)	C15—C18A—H18B	107.6
C5—C6—H6A	109.5	H18A—C18A—H18B	107.0
C5—C6—H6B	109.5	C18A—C19A—C20A	115.2 (7)
H6A—C6—H6B	109.5	C18A—C19A—H19A	108.5
C5—C6—H6C	109.5	C20A—C19A—H19A	108.5
H6A—C6—H6C	109.5	C18A—C19A—H19B	108.5
H6B—C6—H6C	109.5	C20A—C19A—H19B	108.5
C4—C7—C8	111.90 (19)	H19A—C19A—H19B	107.5
C4—C7—H7A	109.2	C21A—C20A—C19A	114.6 (5)
C8—C7—H7A	109.2	C21A—C20A—H20A	108.6
C4—C7—H7B	109.2	C19A—C20A—H20A	108.6
C8—C7—H7B	109.2	C21A—C20A—H20B	108.6
H7A—C7—H7B	107.9	C19A—C20A—H20B	108.6
C7—C8—C9	113.3 (2)	H20A—C20A—H20B	107.6
C7—C8—H8A	108.9	C20A—C21A—H21A	109.5
C9—C8—H8A	108.9	C20A—C21A—H21B	109.5
C7—C8—H8B	108.9	H21A—C21A—H21B	109.5
C9—C8—H8B	108.9	C20A—C21A—H21C	109.5
H8A—C8—H8B	107.7	H21A—C21A—H21C	109.5
C10—C9—C8	114.4 (3)	H21B—C21A—H21C	109.5
C10—C9—H9A	108.7	C19B—C18B—C15	107.6 (6)
C8—C9—H9A	108.7	C19B—C18B—H18C	110.2
C10—C9—H9B	108.7	C15—C18B—H18C	110.2
C8—C9—H9B	108.7	C19B—C18B—H18D	110.2
H9A—C9—H9B	107.6	C15—C18B—H18D	110.2
C9—C10—H10A	109.5	H18C—C18B—H18D	108.5
C9—C10—H10B	109.5	C18B—C19B—C20B	111.2 (6)
H10A—C10—H10B	109.5	C18B—C19B—H19C	109.4
C9—C10—H10C	109.5	C20B—C19B—H19C	109.4
H10A—C10—H10C	109.5	C18B—C19B—H19D	109.4
H10B—C10—H10C	109.5	C20B—C19B—H19D	109.4
O3—C11—H11A	109.5	H19C—C19B—H19D	108.0
O3—C11—H11B	109.5	C21B—C20B—C19B	112.5 (5)
H11A—C11—H11B	109.5	C21B—C20B—H20C	109.1
O3—C11—H11C	109.5	C19B—C20B—H20C	109.1
H11A—C11—H11C	109.5	C21B—C20B—H20D	109.1
H11B—C11—H11C	109.5	C19B—C20B—H20D	109.1
C16—O4—C12	122.36 (16)	H20C—C20B—H20D	107.8
C14—O6—C22	117.48 (16)	C20B—C21B—H21D	109.5
O5—C12—O4	115.77 (18)	C20B—C21B—H21E	109.5

O5—C12—C13	127.06 (18)	H21D—C21B—H21E	109.5
O4—C12—C13	117.17 (17)	C20B—C21B—H21F	109.5
C14—C13—C12	120.10 (18)	H21D—C21B—H21F	109.5
C14—C13—H13	119.9	H21E—C21B—H21F	109.5
C12—C13—H13	119.9	O6—C22—H22A	109.5
O6—C14—C13	124.30 (18)	O6—C22—H22B	109.5
O6—C14—C15	114.44 (17)	H22A—C22—H22B	109.5
C13—C14—C15	121.26 (19)	O6—C22—H22C	109.5
C16—C15—C14	117.36 (18)	H22A—C22—H22C	109.5
C16—C15—C18B	121.7 (5)	H22B—C22—H22C	109.5
C14—C15—C18B	120.3 (5)		

C5—O1—C1—O2	179.7 (2)	C22—O6—C14—C15	176.2 (2)
C5—O1—C1—C2	−0.3 (3)	C12—C13—C14—O6	−179.0 (2)
O2—C1—C2—C3	180.0 (2)	C12—C13—C14—C15	1.4 (4)
O1—C1—C2—C3	0.0 (3)	O6—C14—C15—C16	179.7 (2)
C11—O3—C3—C2	5.0 (3)	C13—C14—C15—C16	−0.6 (3)
C11—O3—C3—C4	−175.30 (19)	O6—C14—C15—C18B	8.6 (5)
C1—C2—C3—O3	179.4 (2)	C13—C14—C15—C18B	−171.7 (4)
C1—C2—C3—C4	−0.3 (3)	O6—C14—C15—C18A	−12.3 (5)
O3—C3—C4—C5	−179.0 (2)	C13—C14—C15—C18A	167.4 (5)
C2—C3—C4—C5	0.8 (3)	C14—C15—C16—O4	−0.1 (3)
O3—C3—C4—C7	3.1 (3)	C18B—C15—C16—O4	170.9 (5)
C2—C3—C4—C7	−177.2 (2)	C18A—C15—C16—O4	−167.4 (5)
C3—C4—C5—O1	−1.0 (3)	C14—C15—C16—C17	178.2 (2)
C7—C4—C5—O1	176.8 (2)	C18B—C15—C16—C17	−10.8 (6)
C3—C4—C5—C6	−179.8 (2)	C18A—C15—C16—C17	11.0 (6)
C7—C4—C5—C6	−1.9 (4)	C12—O4—C16—C15	0.0 (3)
C1—O1—C5—C4	0.9 (3)	C12—O4—C16—C17	−178.7 (2)
C1—O1—C5—C6	179.80 (19)	C16—C15—C18A—C19A	94.2 (10)
C5—C4—C7—C8	−97.7 (3)	C14—C15—C18A—C19A	−73.0 (11)
C3—C4—C7—C8	80.1 (3)	C18B—C15—C18A—C19A	−176 (4)
C4—C7—C8—C9	−169.6 (2)	C15—C18A—C19A—C20A	168.5 (7)
C7—C8—C9—C10	−71.9 (4)	C18A—C19A—C20A—C21A	67.1 (10)
C16—O4—C12—O5	−179.5 (2)	C16—C15—C18B—C19B	96.2 (7)
C16—O4—C12—C13	0.8 (3)	C14—C15—C18B—C19B	−93.0 (8)
O5—C12—C13—C14	178.9 (2)	C18A—C15—C18B—C19B	−6 (2)
O4—C12—C13—C14	−1.5 (3)	C15—C18B—C19B—C20B	173.5 (6)
C22—O6—C14—C13	−3.5 (3)	C18B—C19B—C20B—C21B	171.1 (8)