Towards the Total Synthesis of Vinigrol: Synthesis of epi-C8-Dihydrovinigrol.

Lionel Gentric,[a] Xavier Le Goff[b] and Louis Ricard,[b] Issam Hanna[a]*,

a) Laboratoire de Synthèse Organique CNRS UMR 7652. b) Laboratoire Hétéroatomes et Coordination CNRS UMR 7653, Ecole Polytechnique, F-91128 Palaiseau Cedex, France.

hanna@poly.polytechnique.fr

Supporting Information

X-Ray Data

Crystal data for 17

Table 1.

Compound	**17**
Molecular formula	$C_{23}H_{40}O_4$
Molecular weight	380.55
Crystal habit	colorless block
Crystal dimensions(mm)	0.20x0.18x0.18
Crystal system	orthorhombic
Space group	$Pna2_1$
a(Å)	21.2980(10)
b(Å)	10.3260(10)
c(Å)	19.4070(10)
α(°)	90.00
β(°)	90.00
γ(°)	90.00
V(Å^3)	4268.0(5)
Z	8
d(g-cm^{-3})	1.184
F(000)	1680
μ(cm^{-1})	0.079
Absorption corrections	multi-scan ; 0.9844 min, 0.9860 max
Diffractometer	KappaCCD
X-ray source	MoKα
λ(Å)	0.71069
Monochromator	graphite
T (K)	150.0(1)
Scan mode	phi and omega scans
Maximum θ	30.02
HKL ranges	-29 29 ; -14 14 ; -27 27
Reflections measured	11869
Unique data	11869

Rint	0.0000
Reflections used	8975
Criterion	I > 2σI)
Refinement type	Fsqd
Hydrogen atoms	mixed
Parameters refined	503
Reflections / parameter	17
wR2	0.1191
R1	0.0469
Flack's parameter	0.0(6)
Weights a, b	0.0623 ; 0.0000
GoF	1.020
difference peak / hole (e $Å^{-3}$)	0.262(0.039) / -0.224(0.039)

Table 2. Atomic Coordinates (A x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for **17**

atom	x	y	z	U(eq)
O(1)	-6664(1)	4317(1)	-1623(1)	34(1)
O(2)	-6737(1)	6447(1)	-2317(1)	37(1)
O(3)	-6420(1)	342(1)	-1859(1)	41(1)
O(4)	-6749(1)	2220(1)	-2435(1)	33(1)
C(1)	-6079(1)	4642(2)	-1974(1)	28(1)
C(2)	-6103(1)	4027(1)	-2697(1)	27(1)
C(3)	-6119(1)	2547(1)	-2669(1)	29(1)
C(4)	-5639(1)	1943(1)	-2186(1)	30(1)
C(5)	-5616(1)	2606(1)	-1483(1)	31(1)
C(6)	-5565(1)	4097(1)	-1488(1)	29(1)
C(7)	-4883(1)	4624(1)	-1517(1)	30(1)
C(8)	-4432(1)	4050(2)	-2050(1)	35(1)
C(9)	-4460(1)	4568(2)	-2794(1)	39(1)
C(10)	-4960(1)	4089(2)	-3305(1)	35(1)
C(11)	-5647(1)	4578(2)	-3249(1)	31(1)
C(12)	-5720(1)	6065(2)	-3271(1)	36(1)
C(13)	-5673(1)	6712(2)	-2568(1)	36(1)
C(14)	-6129(1)	6152(2)	-2038(1)	33(1)
C(15)	-4588(1)	4601(2)	-777(1)	43(1)
C(16)	-4277(1)	3340(2)	-554(2)	91(1)
C(17)	-4108(1)	5682(2)	-690(1)	49(1)
C(18)	-4720(1)	4394(2)	-4030(1)	47(1)
C(19)	-6073(1)	6824(2)	-1342(1)	43(1)
C(20)	-5791(1)	504(2)	-2104(1)	37(1)
C(21)	-6877(1)	875(2)	-2301(1)	40(1)
C(22)	-7484(1)	868(2)	-1897(1)	57(1)
C(23)	-6930(1)	123(2)	-2973(1)	53(1)
O(5)	-8135(1)	-6678(1)	-3068(1)	34(1)
O(6)	-7219(1)	-6735(1)	-3934(1)	42(1)
O(7)	-8954(1)	-4293(1)	-1572(1)	43(1)
O(8)	-8021(1)	-4683(1)	-2173(1)	32(1)
C(24)	-8205(1)	-5725(1)	-3617(1)	29(1)
C(25)	-7877(1)	-4463(1)	-3379(1)	29(1)
C(26)	-8198(1)	-3857(2)	-2751(1)	30(1)
C(27)	-8910(1)	-3781(2)	-2791(1)	32(1)
C(28)	-9209(1)	-5034(2)	-3052(1)	32(1)
C(29)	-8926(1)	-5616(1)	-3717(1)	30(1)
C(30)	-9198(1)	-5064(2)	-4398(1)	33(1)
C(31)	-9266(1)	-3587(2)	-4469(1)	38(1)
C(32)	-8691(1)	-2820(2)	-4708(1)	45(1)
C(33)	-8176(1)	-2441(2)	-4193(1)	44(1)
C(34)	-7696(1)	-3466(2)	-3940(1)	36(1)
C(35)	-7330(1)	-4172(2)	-4514(1)	42(1)
C(36)	-7645(1)	-5396(2)	-4786(1)	39(1)
C(37)	-7813(1)	-6347(2)	-4221(1)	35(1)
C(38)	-9825(1)	-5763(2)	-4588(1)	57(1)
C(39)	-9934(1)	-5700(2)	-5365(1)	62(1)
C(40)	-10408(1)	-5303(4)	-4231(1)	113(2)
C(41)	-7816(1)	-1304(2)	-4517(1)	60(1)
C(42)	-8132(1)	-7558(2)	-4501(1)	42(1)
C(43)	-9166(1)	-3410(2)	-2087(1)	39(1)
C(44)	-8290(1)	-4363(2)	-1517(1)	43(1)
C(45)	-8008(1)	-3134(2)	-1230(1)	58(1)
C(46)	-8158(1)	-5520(3)	-1066(1)	65(1)

U(eq) is defined as 1/3 the trace of the Uij tensor.

Table 3. Bond lengths (A) and angles (deg) for **17**

O(1)-C(1)	1.459(2)	O(1)-H(1)	0.8400

O(2)-C(14)	1.436(2)	O(2)-H(2)	0.8400
O(3)-C(21)	1.409(2)	O(3)-C(20)	1.430(2)
O(4)-C(21)	1.438(2)	O(4)-C(3)	1.456(2)
C(1)-C(2)	1.539(2)	C(1)-C(6)	1.552(2)
C(1)-C(14)	1.568(2)	C(2)-C(3)	1.530(2)
C(2)-C(11)	1.553(2)	C(2)-H(2A)	1.0000
C(3)-C(4)	1.521(2)	C(3)-H(3)	1.0000
C(4)-C(5)	1.528(2)	C(4)-C(20)	1.529(2)
C(4)-H(4)	1.0000	C(5)-C(6)	1.544(2)
C(5)-H(5A)	0.9900	C(5)-H(5B)	0.9900
C(6)-C(7)	1.552(2)	C(6)-H(6)	1.0000
C(7)-C(8)	1.531(2)	C(7)-C(15)	1.568(2)
C(7)-H(7)	1.0000	C(8)-C(9)	1.540(2)
C(8)-H(8A)	0.9900	C(8)-H(8B)	0.9900
C(9)-C(10)	1.537(3)	C(9)-H(9A)	0.9900
C(9)-H(9B)	0.9900	C(10)-C(18)	1.530(2)
C(10)-C(11)	1.552(2)	C(10)-H(10)	1.0000
C(11)-C(12)	1.544(2)	C(11)-H(11)	1.0000
C(12)-C(13)	1.523(2)	C(12)-H(12A)	0.9900
C(12)-H(12B)	0.9900	C(13)-C(14)	1.528(2)
C(13)-H(13A)	0.9900	C(13)-H(13B)	0.9900
C(14)-C(19)	1.524(2)	C(15)-C(16)	1.523(3)
C(15)-C(17)	1.524(3)	C(15)-H(15)	1.0000
C(16)-H(16A)	0.9800	C(16)-H(16B)	0.9800
C(16)-H(16C)	0.9800	C(17)-H(17A)	0.9800
C(17)-H(17B)	0.9800	C(17)-H(17C)	0.9800
C(18)-H(18A)	0.9800	C(18)-H(18B)	0.9800
C(18)-H(18C)	0.9800	C(19)-H(19A)	0.9800
C(19)-H(19B)	0.9800	C(19)-H(19C)	0.9800
C(20)-H(20A)	0.9900	C(20)-H(20B)	0.9900
C(21)-C(22)	1.513(3)	C(21)-C(23)	1.522(3)
C(22)-H(22A)	0.9800	C(22)-H(22B)	0.9800
C(22)-H(22C)	0.9800	C(23)-H(23A)	0.9800
C(23)-H(23B)	0.9800	C(23)-H(23C)	0.9800
O(5)-C(24)	1.457(2)	O(5)-H(5A)	0.8400
O(6)-C(37)	1.440(2)	O(6)-H(6)	0.8400
O(7)-C(44)	1.421(2)	O(7)-C(43)	1.427(2)
O(8)-C(44)	1.434(2)	O(8)-C(26)	1.458(2)
C(24)-C(25)	1.548(2)	C(24)-C(29)	1.553(2)
C(24)-C(37)	1.576(2)	C(25)-C(26)	1.532(2)
C(25)-C(34)	1.548(2)	C(25)-H(25)	1.0000
C(26)-C(27)	1.520(2)	C(26)-H(26)	1.0000
C(27)-C(43)	1.519(2)	C(27)-C(28)	1.529(2)
C(27)-H(27)	1.0000	C(28)-C(29)	1.544(2)
C(28)-H(28A)	0.9900	C(28)-H(28B)	0.9900
C(29)-C(30)	1.553(2)	C(29)-H(29)	1.0000
C(30)-C(31)	1.538(2)	C(30)-C(38)	1.561(3)
C(30)-H(30)	1.0000	C(31)-C(32)	1.531(3)
C(31)-H(31A)	0.9900	C(31)-H(31B)	0.9900
C(32)-C(33)	1.534(3)	C(32)-H(32A)	0.9900
C(32)-H(32B)	0.9900	C(33)-C(41)	1.537(3)
C(33)-C(34)	1.551(3)	C(33)-H(33)	1.0000
C(34)-C(35)	1.542(2)	C(34)-H(34)	1.0000
C(35)-C(36)	1.525(2)	C(35)-H(35A)	0.9900
C(35)-H(35B)	0.9900	C(36)-C(37)	1.515(3)
C(36)-H(36A)	0.9900	C(36)-H(36B)	0.9900
C(37)-C(42)	1.523(2)	C(38)-C(40)	1.500(4)
C(38)-C(39)	1.528(3)	C(38)-H(38)	1.0000
C(39)-H(39A)	0.9800	C(39)-H(39B)	0.9800
C(39)-H(39C)	0.9800	C(40)-H(40A)	0.9800
C(40)-H(40B)	0.9800	C(40)-H(40C)	0.9800
C(41)-H(41A)	0.9800	C(41)-H(41B)	0.9800
C(41)-H(41C)	0.9800	C(42)-H(42A)	0.9800
C(42)-H(42B)	0.9800	C(42)-H(42C)	0.9800
C(43)-H(43A)	0.9900	C(43)-H(43B)	0.9900
C(44)-C(46)	1.508(3)	C(44)-C(45)	1.510(3)
C(45)-H(45A)	0.9800	C(45)-H(45B)	0.9800
C(45)-H(45C)	0.9800	C(46)-H(46A)	0.9800
C(46)-H(46B)	0.9800	C(46)-H(46C)	0.9800

C(1)-O(1)-H(1)	109.5	C(14)-O(2)-H(2)	109.5
C(21)-O(3)-C(20)	113.5(1)	C(21)-O(4)-C(3)	117.0(1)
O(1)-C(1)-C(2)	107.7(1)	O(1)-C(1)-C(6)	103.6(1)
C(2)-C(1)-C(6)	115.3(1)	O(1)-C(1)-C(14)	102.0(1)
C(2)-C(1)-C(14)	109.6(1)	C(6)-C(1)-C(14)	117.1(1)
C(3)-C(2)-C(1)	112.4(1)	C(3)-C(2)-C(11)	113.8(1)
C(1)-C(2)-C(11)	117.2(1)	C(3)-C(2)-H(2A)	103.8
C(1)-C(2)-H(2A)	103.8	C(11)-C(2)-H(2A)	103.8
O(4)-C(3)-C(4)	109.4(1)	O(4)-C(3)-C(2)	105.3(1)
C(4)-C(3)-C(2)	114.5(1)	O(4)-C(3)-H(3)	109.2
C(4)-C(3)-H(3)	109.2	C(2)-C(3)-H(3)	109.2
C(3)-C(4)-C(5)	112.9(1)	C(3)-C(4)-C(20)	108.7(1)
C(5)-C(4)-C(20)	110.4(1)	C(3)-C(4)-H(4)	108.3
C(5)-C(4)-H(4)	108.3	C(20)-C(4)-H(4)	108.3
C(4)-C(5)-C(6)	116.3(1)	C(4)-C(5)-H(5A)	108.2
C(6)-C(5)-H(5A)	108.2	C(4)-C(5)-H(5B)	108.2
C(6)-C(5)-H(5B)	108.2	H(5A)-C(5)-H(5B)	107.4
C(5)-C(6)-C(1)	108.3(1)	C(5)-C(6)-C(7)	114.6(1)
C(1)-C(6)-C(7)	120.8(1)	C(5)-C(6)-H(6)	103.7
C(1)-C(6)-H(6)	103.7	C(7)-C(6)-H(6)	103.7
C(8)-C(7)-C(6)	118.4(1)	C(8)-C(7)-C(15)	111.3(1)
C(6)-C(7)-C(15)	109.6(1)	C(8)-C(7)-H(7)	105.5
C(6)-C(7)-H(7)	105.5	C(15)-C(7)-H(7)	105.5
C(7)-C(8)-C(9)	118.3(1)	C(7)-C(8)-H(8A)	107.7
C(9)-C(8)-H(8A)	107.7	C(7)-C(8)-H(8B)	107.7
C(9)-C(8)-H(8B)	107.7	H(8A)-C(8)-H(8B)	107.1
C(10)-C(9)-C(8)	121.3(1)	C(10)-C(9)-H(9A)	107.0
C(8)-C(9)-H(9A)	107.0	C(10)-C(9)-H(9B)	107.0
C(8)-C(9)-H(9B)	107.0	H(9A)-C(9)-H(9B)	106.7
C(18)-C(10)-C(9)	107.2(2)	C(18)-C(10)-C(11)	108.2(1)
C(9)-C(10)-C(11)	120.2(1)	C(18)-C(10)-H(10)	106.8
C(9)-C(10)-H(10)	106.8	C(11)-C(10)-H(10)	106.8
C(12)-C(11)-C(10)	114.6(1)	C(12)-C(11)-C(2)	108.7(1)
C(10)-C(11)-C(2)	121.2(1)	C(12)-C(11)-H(11)	103.3
C(10)-C(11)-H(11)	103.3	C(2)-C(11)-H(11)	103.3
C(13)-C(12)-C(11)	113.8(1)	C(13)-C(12)-H(12A)	108.8
C(11)-C(12)-H(12A)	108.8	C(13)-C(12)-H(12B)	108.8
C(11)-C(12)-H(12B)	108.8	H(12A)-C(12)-H(12B)	107.7
C(12)-C(13)-C(14)	113.3(1)	C(12)-C(13)-H(13A)	108.9
C(14)-C(13)-H(13A)	108.9	C(12)-C(13)-H(13B)	108.9
C(14)-C(13)-H(13B)	108.9	H(13A)-C(13)-H(13B)	107.7
O(2)-C(14)-C(19)	107.9(1)	O(2)-C(14)-C(13)	103.8(1)
C(19)-C(14)-C(13)	112.0(1)	O(2)-C(14)-C(1)	107.6(1)
C(19)-C(14)-C(1)	112.2(1)	C(13)-C(14)-C(1)	112.8(1)
C(16)-C(15)-C(17)	107.7(2)	C(16)-C(15)-C(7)	116.5(2)
C(17)-C(15)-C(7)	111.0(2)	C(16)-C(15)-H(15)	107.1
C(17)-C(15)-H(15)	107.1	C(7)-C(15)-H(15)	107.1
C(15)-C(16)-H(16A)	109.5	C(15)-C(16)-H(16B)	109.5
H(16A)-C(16)-H(16B)	109.5	C(15)-C(16)-H(16C)	109.5
H(16A)-C(16)-H(16C)	109.5	H(16B)-C(16)-H(16C)	109.5
C(15)-C(17)-H(17A)	109.5	C(15)-C(17)-H(17B)	109.5
H(17A)-C(17)-H(17B)	109.5	C(15)-C(17)-H(17C)	109.5
H(17A)-C(17)-H(17C)	109.5	H(17B)-C(17)-H(17C)	109.5
C(10)-C(18)-H(18A)	109.5	C(10)-C(18)-H(18B)	109.5

H(18A)-C(18)-H(18B)	109.5	C(10)-C(18)-H(18C)	109.5
H(18A)-C(18)-H(18C)	109.5	H(18B)-C(18)-H(18C)	109.5
C(14)-C(19)-H(19A)	109.5	C(14)-C(19)-H(19B)	109.5
H(19A)-C(19)-H(19B)	109.5	C(14)-C(19)-H(19C)	109.5
H(19A)-C(19)-H(19C)	109.5	H(19B)-C(19)-H(19C)	109.5
O(3)-C(20)-C(4)	110.3(1)	O(3)-C(20)-H(20A)	109.6
C(4)-C(20)-H(20A)	109.6	O(3)-C(20)-H(20B)	109.6
C(4)-C(20)-H(20B)	109.6	H(20A)-C(20)-H(20B)	108.1
O(3)-C(21)-O(4)	110.9(1)	O(3)-C(21)-C(22)	
105.8(2)			
O(4)-C(21)-C(22)	105.0(1)	O(3)-C(21)-C(23)	
112.0(2)			
O(4)-C(21)-C(23)	110.6(2)	C(22)-C(21)-C(23)	
112.2(2)			
C(21)-C(22)-H(22A)	109.5	C(21)-C(22)-H(22B)	109.5
H(22A)-C(22)-H(22B)	109.5	C(21)-C(22)-H(22C)	109.5
H(22A)-C(22)-H(22C)	109.5	H(22B)-C(22)-H(22C)	109.5
C(21)-C(23)-H(23A)	109.5	C(21)-C(23)-H(23B)	109.5
H(23A)-C(23)-H(23B)	109.5	C(21)-C(23)-H(23C)	109.5
H(23A)-C(23)-H(23C)	109.5	H(23B)-C(23)-H(23C)	109.5
C(24)-O(5)-H(5A)	109.5	C(37)-O(6)-H(6)	109.5
C(44)-O(7)-C(43)	113.6(1)	C(44)-O(8)-C(26)	
116.4(1)			
O(5)-C(24)-C(25)	107.8(1)	O(5)-C(24)-C(29)	
104.0(1)			
C(25)-C(24)-C(29)	114.9(1)	O(5)-C(24)-C(37)	
102.4(1)			
C(25)-C(24)-C(37)	109.0(1)	C(29)-C(24)-C(37)	
117.4(1)			
C(26)-C(25)-C(34)	113.6(1)	C(26)-C(25)-C(24)	
112.4(1)			
C(34)-C(25)-C(24)	117.6(1)	C(26)-C(25)-H(25)	103.8
C(34)-C(25)-H(25)	103.8	C(24)-C(25)-H(25)	103.8
O(8)-C(26)-C(27)	109.0(1)	O(8)-C(26)-C(25)	
105.0(1)			
C(27)-C(26)-C(25)	115.1(1)	O(8)-C(26)-H(26)	109.2
C(27)-C(26)-H(26)	109.2	C(25)-C(26)-H(26)	109.2
C(43)-C(27)-C(26)	109.0(1)	C(43)-C(27)-C(28)	
111.2(1)			
C(26)-C(27)-C(28)	112.9(1)	C(43)-C(27)-H(27)	107.9
C(26)-C(27)-H(27)	107.9	C(28)-C(27)-H(27)	107.9
C(27)-C(28)-C(29)	116.4(1)	C(27)-C(28)-H(28A)	108.2
C(29)-C(28)-H(28A)	108.2	C(27)-C(28)-H(28B)	108.2
C(29)-C(28)-H(28B)	108.2	H(28A)-C(28)-H(28B)	107.4
C(28)-C(29)-C(24)	108.0(1)	C(28)-C(29)-C(30)	
115.1(1)			
C(24)-C(29)-C(30)	120.2(1)	C(28)-C(29)-H(29)	103.9
C(24)-C(29)-H(29)	103.9	C(30)-C(29)-H(29)	103.9
C(31)-C(30)-C(29)	118.4(1)	C(31)-C(30)-C(38)	
110.9(1)			
C(29)-C(30)-C(38)	110.4(1)	C(31)-C(30)-H(30)	105.3
C(29)-C(30)-H(30)	105.3	C(38)-C(30)-H(30)	105.3
C(32)-C(31)-C(30)	117.7(2)	C(32)-C(31)-H(31A)	107.9
C(30)-C(31)-H(31A)	107.9	C(32)-C(31)-H(31B)	107.9
C(30)-C(31)-H(31B)	107.9	H(31A)-C(31)-H(31B)	107.2
C(31)-C(32)-C(33)	120.5(2)	C(31)-C(32)-H(32A)	107.2
C(33)-C(32)-H(32A)	107.2	C(31)-C(32)-H(32B)	107.2
C(33)-C(32)-H(32B)	107.2	H(32A)-C(32)-H(32B)	106.8
C(32)-C(33)-C(41)	106.6(2)	C(32)-C(33)-C(34)	
120.2(2)			
C(41)-C(33)-C(34)	108.8(2)	C(32)-C(33)-H(33)	106.8
C(41)-C(33)-H(33)	106.8	C(34)-C(33)-H(33)	106.8
C(35)-C(34)-C(25)	108.6(1)	C(35)-C(34)-C(33)	
115.3(1)			
C(25)-C(34)-C(33)	120.8(1)	C(35)-C(34)-H(34)	103.2
C(25)-C(34)-H(34)	103.2	C(33)-C(34)-H(34)	103.2
C(36)-C(35)-C(34)	114.8(1)	C(36)-C(35)-H(35A)	108.6
C(34)-C(35)-H(35A)	108.6	C(36)-C(35)-H(35B)	108.6
C(34)-C(35)-H(35B)	108.6	H(35A)-C(35)-H(35B)	107.5
C(37)-C(36)-C(35)	113.0(1)	C(37)-C(36)-H(36A)	109.0

C(35)-C(36)-H(36A)	109.0	C(37)-C(36)-H(36B)	109.0
C(35)-C(36)-H(36B)	109.0	H(36A)-C(36)-H(36B)	107.8
O(6)-C(37)-C(36)	104.6(1)	O(6)-C(37)-C(42)	107.6(1)
C(36)-C(37)-C(42)	112.3(1)	O(6)-C(37)-C(24)	106.9(1)
C(36)-C(37)-C(24)	113.6(1)	C(42)-C(37)-C(24)	111.3(1)
C(40)-C(38)-C(39)	108.4(2)	C(40)-C(38)-C(30)	117.0(2)
C(39)-C(38)-C(30)	110.0(2)	C(40)-C(38)-H(38)	107.0
C(39)-C(38)-H(38)	107.0	C(30)-C(38)-H(38)	107.0
C(38)-C(39)-H(39A)	109.5	C(38)-C(39)-H(39B)	109.5
H(39A)-C(39)-H(39B)	109.5	C(38)-C(39)-H(39C)	109.5
H(39A)-C(39)-H(39C)	109.5	H(39B)-C(39)-H(39C)	109.5
C(38)-C(40)-H(40A)	109.5	C(38)-C(40)-H(40B)	109.5
H(40A)-C(40)-H(40B)	109.5	C(38)-C(40)-H(40C)	109.5
H(40A)-C(40)-H(40C)	109.5	H(40B)-C(40)-H(40C)	109.5
C(33)-C(41)-H(41A)	109.5	C(33)-C(41)-H(41B)	109.5
H(41A)-C(41)-H(41B)	109.5	C(33)-C(41)-H(41C)	109.5
H(41A)-C(41)-H(41C)	109.5	H(41B)-C(41)-H(41C)	109.5
C(37)-C(42)-H(42A)	109.5	C(37)-C(42)-H(42B)	109.5
H(42A)-C(42)-H(42B)	109.5	C(37)-C(42)-H(42C)	109.5
H(42A)-C(42)-H(42C)	109.5	H(42B)-C(42)-H(42C)	109.5
O(7)-C(43)-C(27)	110.8(1)	O(7)-C(43)-H(43A)	109.5
C(27)-C(43)-H(43A)	109.5	O(7)-C(43)-H(43B)	109.5
C(27)-C(43)-H(43B)	109.5	H(43A)-C(43)-H(43B)	108.1
O(7)-C(44)-O(8)	110.0(1)	O(7)-C(44)-C(46)	105.6(2)
O(8)-C(44)-C(46)	105.0(1)	O(7)-C(44)-C(45)	112.4(2)
O(8)-C(44)-C(45)	111.3(2)	C(46)-C(44)-C(45)	112.2(2)
C(44)-C(45)-H(45A)	109.5	C(44)-C(45)-H(45B)	109.5
H(45A)-C(45)-H(45B)	109.5	C(44)-C(45)-H(45C)	109.5
H(45A)-C(45)-H(45C)	109.5	H(45B)-C(45)-H(45C)	109.5
C(44)-C(46)-H(46A)	109.5	C(44)-C(46)-H(46B)	109.5
H(46A)-C(46)-H(46B)	109.5	C(44)-C(46)-H(46C)	109.5
H(46A)-C(46)-H(46C)	109.5	H(46B)-C(46)-H(46C)	109.5

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for **17**

atom	U11	U22	U33	U23	U13	U12
O(1)	26(1)	38(1)	38(1)	-6(1)	4(1)	-2(1)
O(2)	30(1)	33(1)	50(1)	-5(1)	-7(1)	4(1)
O(3)	40(1)	34(1)	49(1)	8(1)	1(1)	-6(1)
O(4)	27(1)	29(1)	42(1)	-1(1)	0(1)	-3(1)
C(1)	24(1)	29(1)	32(1)	-3(1)	1(1)	-1(1)
C(2)	26(1)	26(1)	29(1)	-1(1)	-1(1)	-1(1)
C(3)	25(1)	29(1)	32(1)	-2(1)	-1(1)	-2(1)
C(4)	29(1)	29(1)	32(1)	1(1)	1(1)	1(1)
C(5)	30(1)	32(1)	30(1)	4(1)	-2(1)	-3(1)
C(6)	28(1)	32(1)	26(1)	-2(1)	1(1)	-1(1)
C(7)	27(1)	34(1)	30(1)	3(1)	-3(1)	-2(1)
C(8)	25(1)	41(1)	39(1)	0(1)	-1(1)	-1(1)
C(9)	32(1)	43(1)	41(1)	2(1)	6(1)	-2(1)
C(10)	37(1)	38(1)	31(1)	3(1)	5(1)	1(1)
C(11)	33(1)	32(1)	28(1)	2(1)	-1(1)	-1(1)
C(12)	35(1)	32(1)	40(1)	6(1)	-5(1)	-3(1)
C(13)	35(1)	25(1)	48(1)	3(1)	-7(1)	-2(1)
C(14)	29(1)	29(1)	42(1)	-5(1)	-7(1)	2(1)
C(15)	33(1)	64(1)	31(1)	6(1)	-5(1)	-10(1)
C(16)	108(2)	68(2)	98(2)	44(2)	-75(2)	-42(2)
C(17)	53(1)	47(1)	48(1)	-3(1)	-20(1)	-4(1)
C(18)	52(1)	53(1)	36(1)	8(1)	11(1)	4(1)
C(19)	41(1)	38(1)	50(1)	-14(1)	-8(1)	4(1)
C(20)	38(1)	31(1)	42(1)	2(1)	0(1)	0(1)

C(21)	36(1)	29(1)	53(1)	2(1)	-4(1)	-7(1)
C(22)	44(1)	47(1)	81(2)	12(1)	9(1)	-12(1)
C(23)	59(1)	33(1)	66(1)	-6(1)	-18(1)	-7(1)
O(5)	34(1)	33(1)	33(1)	4(1)	-3(1)	3(1)
O(6)	32(1)	51(1)	42(1)	-7(1)	0(1)	5(1)
O(7)	31(1)	67(1)	30(1)	6(1)	4(1)	14(1)
O(8)	29(1)	45(1)	24(1)	2(1)	0(1)	7(1)
C(24)	28(1)	31(1)	26(1)	3(1)	-1(1)	0(1)
C(25)	28(1)	33(1)	26(1)	-3(1)	0(1)	-2(1)
C(26)	32(1)	33(1)	25(1)	3(1)	-1(1)	1(1)
C(27)	32(1)	35(1)	28(1)	1(1)	-1(1)	6(1)
C(28)	26(1)	37(1)	31(1)	5(1)	-2(1)	1(1)
C(29)	28(1)	33(1)	30(1)	3(1)	-2(1)	-2(1)
C(30)	36(1)	32(1)	31(1)	3(1)	-4(1)	-1(1)
C(31)	45(1)	35(1)	34(1)	2(1)	-9(1)	1(1)
C(32)	60(1)	35(1)	40(1)	7(1)	-6(1)	-8(1)
C(33)	57(1)	39(1)	35(1)	7(1)	0(1)	-12(1)
C(34)	36(1)	41(1)	30(1)	-1(1)	1(1)	-11(1)
C(35)	41(1)	53(1)	32(1)	-1(1)	7(1)	-15(1)
C(36)	35(1)	51(1)	30(1)	-7(1)	4(1)	-5(1)
C(37)	28(1)	45(1)	33(1)	-8(1)	0(1)	-1(1)
C(38)	60(1)	49(1)	60(1)	23(1)	-32(1)	-18(1)
C(39)	62(2)	62(1)	62(1)	-25(1)	-32(1)	16(1)
C(40)	50(2)	249(4)	40(1)	14(2)	-6(1)	-60(2)
C(41)	82(2)	51(1)	46(1)	11(1)	-1(1)	-24(1)
C(42)	42(1)	40(1)	43(1)	-10(1)	-1(1)	1(1)
C(43)	34(1)	51(1)	32(1)	-1(1)	-1(1)	13(1)
C(44)	34(1)	71(1)	23(1)	3(1)	3(1)	16(1)
C(45)	41(1)	94(2)	38(1)	-22(1)	-5(1)	13(1)
C(46)	52(1)	107(2)	35(1)	27(1)	12(1)	32(1)

The anisotropic displacement factor exponent takes the form
2 pi^2 [h^2a*^2U(11) +...+ 2hka*b*U(12)]

Table 5. Hydrogen Coordinates (A x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for **25**

atom	x	y	z	U(eq)
H(1)	-6827	3667	-1811	51
H(2)	-7007	5965	-2134	56
H(2A)	-6528	4268	-2873	32
H(3)	-6056	2195	-3143.9998	35
H(4)	-5215	2020	-2404	36
H(5A)	-5252	2256	-1225	37
H(5B)	-6000	2365	-1225	37
H(6)	-5713.0005	4354	-1018	34
H(7)	-4923	5561	-1640	36
H(8A)	-4506	3104	-2068	42
H(8B)	-3999	4180	-1879.0001	42
H(9A)	-4505	5521	-2763.0002	47
H(9B)	-4045	4398	-3005	47
H(10)	-4974	3125	-3263	42
H(11)	-5840	4290	-3693	37
H(12A)	-5392	6427	-3576	43
H(12B)	-6133.0005	6279	-3477	43
H(13A)	-5758	7649	-2621	43
H(13B)	-5238.9995	6614	-2393	43
H(15)	-4936	4776	-443	51
H(16A)	-3912	3169	-848	137
H(16B)	-4579	2627	-598	137
H(16C)	-4142	3412	-73	137
H(17A)	-3964	5707	-210	74
H(17B)	-4301	6513	-810	74
H(17C)	-3749.0002	5520	-994	74
H(18A)	-5030	4107	-4371	71
H(18B)	-4322	3940	-4108	71
H(18C)	-4654	5329	-4075.9998	71

H(19A)	-6197	7734	-1388	64
H(19B)	-5637	6777	-1181	64
H(19C)	-6348	6394	-1008	64
H(20A)	-5743	61	-2552.9998	44
H(20B)	-5493.9995	107	-1773.9999	44
H(22A)	-7598	-26	-1782.9999	86
H(22B)	-7817.9995	1261	-2174	86
H(22C)	-7429	1365	-1471	86
H(23A)	-6548	247	-3246	79
H(23B)	-7293	438	-3234	79
H(23C)	-6984	-800	-2872	79
H(5A)	-8089	-6294.9995	-2690	50
H(6)	-7279.0005	-7125	-3559	62
H(25)	-7464	-4767	-3197	35
H(26)	-8024	-2967.9998	-2674	36
H(27)	-9021	-3074	-3121	38
H(28A)	-9661	-4870	-3134	38
H(28B)	-9178	-5692	-2683	38
H(29)	-9073	-6535	-3712	36
H(30)	-8893	-5324	-4765	40
H(31A)	-9612	-3413	-4798	46
H(31B)	-9398	-3239.0002	-4017	46
H(32A)	-8846	-2010	-4920	54
H(32B)	-8488	-3327.0002	-5079	54
H(33)	-8392	-2097	-3774	53
H(34)	-7365.0005	-2931	-3710	43
H(35A)	-7269	-3565	-4903	50
H(35B)	-6910	-4404	-4335	50
H(36A)	-8031	-5152	-5037.9995	46
H(36B)	-7359	-5826	-5116.9995	46
H(38)	-9771	-6697.0005	-4466	68
H(39A)	-10267	-6307	-5493	93
H(39B)	-9545	-5932.0005	-5606	93
H(39C)	-10058	-4819	-5493.9995	93
H(40A)	-10391.0010	-5546	-3743	169
H(40B)	-10777	-5704	-4446	169
H(40C)	-10439	-4359	-4271	169
H(41A)	-7473.0005	-1043	-4210	90
H(41B)	-8103	-572	-4584	90
H(41C)	-7643	-1571.0001	-4963	90
H(42A)	-7846	-8005	-4817	63
H(42B)	-8515	-7312	-4748	63
H(42C)	-8240	-8136	-4118	63
H(43A)	-9027	-2523	-1969	47
H(43B)	-9631	-3414	-2102	47
H(45A)	-8075	-2423	-1556	87
H(45B)	-7556	-3257.0002	-1158	87
H(45C)	-8209	-2922.9998	-790.0001	87
H(46A)	-8340	-5378	-608	97
H(46B)	-7702.9995	-5641.0005	-1023.0001	97
H(46C)	-8345	-6294.9995	-1273	97

Crystal data for 23.

Table 1.

Compound	**23**
Molecular formula	$C_{24}H_{38}O_5$
Molecular weight	406.54
Crystal habit	colorless block
Crystal dimensions(mm)	0.22x0.20x0.16
Crystal system	monoclinic
Space group	$P2_1/n$
a(Å)	11.4050(10)
b(Å)	14.6640(10)
c(Å)	13.7140(10)
α(°)	90.00
β(°)	107.2310(10)
γ(°)	90.00
V(Å^3)	2190.6(3)
Z	4
d(g-cm^{-3})	1.233
F(000)	888
μ(cm^{-1})	0.084
Absorption corrections	multi-scan ; 0.9817 min, 0.9866 max
Diffractometer	KappaCCD
X-ray source	MoKα
λ(Å)	0.71069
Monochromator	graphite
T (K)	150.0(1)
Scan mode	phi and omega scans
Maximum θ	30.03
HKL ranges	-16 16 ; -20 17 ; -19 19
Reflections measured	10183
Unique data	6309
Rint	0.0217
Reflections used	4911
Criterion	I > 2σI)
Refinement type	Fsqd
Hydrogen atoms	mixed
Parameters refined	269
Reflections / parameter	18
wR2	0.1173
R1	0.0411
Weights a, b	0.0546 ; 0.2546
GoF	1.064
difference peak / hole (e Å^{-3})	0.288(0.039) / -0.208(0.039)

Table 2. Atomic Coordinates (A x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for **23**

atom	x	y	z	U(eq)
O(1)	428(1)	2522(1)	7347(1)	31(1)
O(2)	885(1)	3441(1)	6226(1)	24(1)
O(3)	719(1)	1931(1)	5924(1)	40(1)
O(4)	3451(1)	3802(1)	6558(1)	22(1)
O(5)	3777(1)	4992(1)	5537(1)	27(1)
C(1)	2668(1)	4054(1)	8961(1)	24(1)
C(2)	1829(1)	3425(1)	9355(1)	31(1)
C(3)	465(1)	3498(1)	8779(1)	33(1)
C(4)	158(1)	3455(1)	7625(1)	27(1)
C(5)	1017(1)	4002(1)	7153(1)	21(1)
C(6)	2377(1)	3907(1)	7794(1)	21(1)
C(7)	3276(1)	4402(1)	7331(1)	22(1)
C(8)	2853(1)	5344(1)	6885(1)	24(1)
C(9)	1515(1)	5347(1)	6233(1)	25(1)
C(10)	591(1)	4958(1)	6743(1)	22(1)
C(11)	169(1)	5596(1)	7483(1)	24(1)
C(12)	1134(1)	6146(1)	8287(1)	27(1)
C(13)	1769(1)	5673(1)	9304(1)	32(1)
C(14)	2852(1)	5034(1)	9403(1)	27(1)
C(15)	-1210(1)	3592(1)	7098(1)	36(1)
C(16)	682(1)	2575(1)	6447(1)	29(1)
C(17)	3691(1)	5661(1)	6269(1)	31(1)
C(18)	3587(1)	4982(1)	10538(1)	36(1)
C(19)	-904(1)	6220(1)	6879(1)	33(1)
C(20)	-521(1)	7090(1)	6445(1)	44(1)
C(21)	-1738(1)	6456(1)	7534(1)	45(1)
C(22)	4228(1)	4141(1)	5996(1)	23(1)
C(23)	5566(1)	4212(1)	6646(1)	32(1)
C(24)	4083(1)	3481(1)	5124(1)	29(1)

U(eq) is defined as 1/3 the trace of the Uij tensor.

Table 3. Bond lengths (A) and angles (deg) for **23**

O(1)-C(16)	1.350(1)	O(1)-C(4)	1.477(1)
O(2)-C(16)	1.342(1)	O(2)-C(5)	1.483(1)
O(3)-C(16)	1.194(1)	O(4)-C(22)	1.425(1)
O(4)-C(7)	1.437(1)	O(5)-C(22)	1.425(1)
O(5)-C(17)	1.427(1)	C(1)-C(2)	1.538(1)
C(1)-C(14)	1.549(1)	C(1)-C(6)	1.551(1)
C(2)-C(3)	1.526(2)	C(3)-C(4)	1.517(2)
C(4)-C(15)	1.526(2)	C(4)-C(5)	1.548(1)
C(5)-C(10)	1.535(1)	C(5)-C(6)	1.546(1)
C(6)-C(7)	1.538(1)	C(7)-C(8)	1.530(1)
C(8)-C(9)	1.523(2)	C(8)-C(17)	1.524(1)
C(9)-C(10)	1.537(1)	C(10)-C(11)	1.558(1)
C(11)-C(12)	1.536(1)	C(11)-C(19)	1.556(1)
C(12)-C(13)	1.533(2)	C(13)-C(14)	1.524(2)
C(14)-C(18)	1.536(2)	C(19)-C(20)	1.524(2)
C(19)-C(21)	1.529(2)	C(22)-C(24)	1.508(1)
C(22)-C(23)	1.526(2)		

C(16)-O(1)-C(4)	107.52(7)	C(16)-O(2)-C(5)	108.15(8)
C(22)-O(4)-C(7)	115.47(7)	C(22)-O(5)-C(17)	112.52(8)
C(2)-C(1)-C(14)	116.4(1)	C(2)-C(1)-C(6)	108.28(8)
C(14)-C(1)-C(6)	119.69(8)	C(3)-C(2)-C(1)	114.9(1)
C(4)-C(3)-C(2)	114.9(1)	O(1)-C(4)-C(3)	107.8(1)

O(1)-C(4)-C(15)	105.18(8)	C(3)-C(4)-C(15)	112.1(1)
O(1)-C(4)-C(5)	99.49(8)	C(3)-C(4)-C(5)	115.8(1)
C(15)-C(4)-C(5)	114.8(1)	O(2)-C(5)-C(10)	104.58(7)
O(2)-C(5)-C(6)	105.52(7)	C(10)-C(5)-C(6)	116.43(8)
O(2)-C(5)-C(4)	98.95(7)	C(10)-C(5)-C(4)	117.08(8)
C(6)-C(5)-C(4)	111.63(8)	C(7)-C(6)-C(5)	113.72(8)
C(7)-C(6)-C(1)	113.29(8)	C(5)-C(6)-C(1)	116.54(8)
O(4)-C(7)-C(8)	110.74(8)	O(4)-C(7)-C(6)	105.73(7)
C(8)-C(7)-C(6)	114.91(8)	C(9)-C(8)-C(17)	110.9(1)
C(9)-C(8)-C(7)	112.32(8)	C(17)-C(8)-C(7)	108.79(8)
C(8)-C(9)-C(10)	115.73(8)	C(5)-C(10)-C(9)	108.72(8)
C(5)-C(10)-C(11)	115.81(8)	C(9)-C(10)-C(11)	117.37(8)
C(12)-C(11)-C(19)	110.88(8)	C(12)-C(11)-C(10)	119.25(8)
C(19)-C(11)-C(10)	110.64(8)	C(13)-C(12)-C(11)	117.4(1)
C(14)-C(13)-C(12)	120.8(1)	C(13)-C(14)-C(18)	107.8(1)
C(13)-C(14)-C(1)	121.6(1)	C(18)-C(14)-C(1)	108.7(1)
O(3)-C(16)-O(2)	125.0(1)	O(3)-C(16)-O(1)	124.0(1)
O(2)-C(16)-O(1)	111.00(8)	O(5)-C(17)-C(8)	111.42(8)
C(20)-C(19)-C(21)	109.9(1)	C(20)-C(19)-C(11)	115.4(1)
C(21)-C(19)-C(11)	110.3(1)	O(5)-C(22)-O(4)	110.09(8)
O(5)-C(22)-C(24)	105.73(8)	O(4)-C(22)-C(24)	105.40(8)
O(5)-C(22)-C(23)	111.48(8)	O(4)-C(22)-C(23)	112.51(8)
C(24)-C(22)-C(23)	111.2(1)		

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for **23**

atom	U11	U22	U33	U23	U13	U12
O(1)	34(1)	20(1)	41(1)	1(1)	12(1)	-4(1)
O(2)	27(1)	19(1)	26(1)	-3(1)	6(1)	-1(1)
O(3)	49(1)	22(1)	46(1)	-7(1)	9(1)	0(1)
O(4)	24(1)	21(1)	24(1)	-1(1)	11(1)	-1(1)
O(5)	33(1)	23(1)	29(1)	3(1)	16(1)	2(1)
C(1)	26(1)	26(1)	21(1)	3(1)	7(1)	2(1)
C(2)	38(1)	31(1)	26(1)	6(1)	13(1)	-2(1)
C(3)	36(1)	32(1)	37(1)	6(1)	20(1)	-5(1)
C(4)	25(1)	22(1)	35(1)	1(1)	12(1)	-4(1)
C(5)	22(1)	19(1)	22(1)	-2(1)	7(1)	-2(1)

C(6)	22(1)	20(1)	21(1)	2(1)	7(1)	1(1)
C(7)	22(1)	22(1)	22(1)	-2(1)	8(1)	-2(1)
C(8)	30(1)	20(1)	27(1)	-2(1)	14(1)	-3(1)
C(9)	31(1)	21(1)	25(1)	4(1)	12(1)	4(1)
C(10)	23(1)	20(1)	22(1)	0(1)	6(1)	1(1)
C(11)	24(1)	22(1)	24(1)	-1(1)	8(1)	2(1)
C(12)	29(1)	24(1)	27(1)	-5(1)	7(1)	2(1)
C(13)	37(1)	33(1)	25(1)	-7(1)	6(1)	2(1)
C(14)	29(1)	30(1)	21(1)	-2(1)	5(1)	-1(1)
C(15)	23(1)	34(1)	53(1)	-1(1)	12(1)	-6(1)
C(16)	27(1)	21(1)	35(1)	-1(1)	4(1)	-1(1)
C(17)	39(1)	22(1)	39(1)	-1(1)	23(1)	-3(1)
C(18)	36(1)	43(1)	24(1)	-2(1)	1(1)	1(1)
C(19)	30(1)	33(1)	33(1)	-7(1)	3(1)	10(1)
C(20)	55(1)	38(1)	38(1)	8(1)	14(1)	23(1)
C(21)	33(1)	42(1)	60(1)	-10(1)	17(1)	9(1)
C(22)	23(1)	24(1)	24(1)	2(1)	11(1)	1(1)
C(23)	23(1)	44(1)	31(1)	1(1)	9(1)	-2(1)
C(24)	33(1)	29(1)	29(1)	-2(1)	14(1)	1(1)

The anisotropic displacement factor exponent takes the form
2 pi^2 [h^2a*^2U(11) +...+ 2hka*b*U(12)]

Table 5. Hydrogen Coordinates (A x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for **23**

atom	x	y	z	U(eq)
H(1)	3498	3771	9244	29
H(2A)	2092	2786	9316	37
H(2B)	1944	3565	10082	37
H(3A)	26	2997	9008	39
H(3B)	150	4080	8967	39
H(6)	2561	3247	7730	25
H(7)	4079	4472	7874	26
H(8)	2942	5779	7464	29
H(9A)	1448	4992	5605	30
H(9B)	1276	5983	6024	30
H(10)	-170.0000	4849	6165	26
H(11)	-205	5179	7886	28
H(12A)	1777	6340	7979	32
H(12B)	734	6706	8437	32
H(13A)	2052	6158	9823	39
H(13B)	1132	5321	9498	39
H(14)	3397	5349	9057	33
H(15A)	-1368	3564	6355	55
H(15B)	-1464	4189	7286	55
H(15C)	-1676.9999	3112	7313	55
H(17A)	3368	6236	5912	37
H(17B)	4520	5784	6736	37
H(18A)	3884	5592	10785	54
H(18B)	4288	4571	10624	54
H(18C)	3059	4750	10930	54
H(19)	-1407	5856	6286	40
H(20A)	-1252	7451	6110	66
H(20B)	-91	6931	5947	66
H(20C)	26	7446	7000	66
H(21A)	-1275	6811	8129	67
H(21B)	-2036	5892	7763	67
H(21C)	-2438	6815	7130	67
H(23A)	5630	4633	7213	48
H(23B)	6064	4439	6225	48
H(23C)	5863	3608	6916	48
H(24A)	4415	2885	5393	43
H(24B)	4530	3712	4665	43
H(24C)	3211	3419	4747	43

Crystal data for 25

Table 1.

Compound	**25**
Molecular formula	$C_{23}H_{40}O_4$
Molecular weight	380.55
Crystal habit	colorless block
Crystal dimensions(mm)	0.30x0.22x0.16
Crystal system	monoclinic
Space group	C2/c
a(Å)	32.5660(10)
b(Å)	8.2150(10)
c(Å)	18.2200(10)
α(°)	90.00
β(°)	118.6550(10)
γ(°)	90.00
V(Å^3)	4277.4(6)
Z	8
d(g-cm^{-3})	1.182
F(000)	1680
μ(cm^{-1})	0.078
Absorption corrections	multi-scan ; 0.9768 min, 0.9876 max
Diffractometer	KappaCCD
X-ray source	MoKα
λ(Å)	0.71069
Monochromator	graphite
T (K)	150.0(1)
Scan mode	phi and omega scans
Maximum θ	30.03
HKL ranges	-45 45 ; -10 11 ; -25 25
Reflections measured	10763
Unique data	6219
Rint	0.0259
Reflections used	4467
Criterion	I > 2σI)
Refinement type	Fsqd
Hydrogen atoms	mixed
Parameters refined	253
Reflections / parameter	17
wR2	0.1374
R1	0.0454
Weights a, b	0.0706 ; 0.2996
GoF	1.087
difference peak / hole (e Å^{-3})	0.368(0.043) / -0.225(0.043)

Table 2. Atomic Coordinates (A x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for **25**

atom	x	y	z	U(eq)
O(1)	4481(1)	1758(1)	1309(1)	29(1)
O(2)	4904(1)	4324(1)	1210(1)	33(1)
O(3)	3872(1)	-313(1)	2634(1)	32(1)
O(4)	4385(1)	1739(1)	2691(1)	27(1)
C(1)	4172(1)	3171(1)	1015(1)	25(1)
C(2)	4193(1)	4030(1)	1785(1)	26(1)
C(3)	4012(1)	2925(1)	2249(1)	26(1)
C(4)	3557(1)	2024(1)	1697(1)	28(1)
C(5)	3541(1)	1280(1)	914(1)	29(1)
C(6)	3693(1)	2403(1)	408(1)	27(1)
C(7)	3285(1)	3423(2)	-262(1)	33(1)
C(8)	2996(1)	4482(2)	23(1)	37(1)
C(9)	3176(1)	6183(2)	374(1)	42(1)
C(10)	3520(1)	6441(1)	1304(1)	38(1)
C(11)	4033(1)	5837(1)	1687(1)	31(1)
C(12)	4301(1)	6755(1)	1311(1)	35(1)
C(13)	4256(1)	6015(1)	509(1)	34(1)
C(14)	4413(1)	4249(1)	619(1)	29(1)
C(15)	2933(1)	2321(2)	-992(1)	45(1)
C(16)	3131(1)	785(2)	-1180(1)	61(1)
C(17)	2690(1)	3328(2)	-1797(1)	65(1)
C(18)	3287(1)	6110(2)	1852(1)	44(1)
C(19)	4365(1)	3529(2)	-190(1)	37(1)
C(20)	3479(1)	739(1)	2223(1)	32(1)
C(21)	4297(1)	512(1)	3160(1)	29(1)
C(22)	4680(1)	-734(1)	3393(1)	36(1)
C(23)	4295(1)	1259(2)	3924(1)	37(1)

U(eq) is defined as 1/3 the trace of the Uij tensor.

Table 3. Bond lengths (A) and angles (deg) for **25**

O(1)-C(1)	1.459(1)	O(1)-H(1)	0.8400
O(2)-C(14)	1.439(1)	O(2)-H(2)	0.8400
O(3)-C(21)	1.421(1)	O(3)-C(20)	1.423(1)
O(4)-C(21)	1.437(1)	O(4)-C(3)	1.461(1)
C(1)-C(2)	1.542(1)	C(1)-C(6)	1.549(1)
C(1)-C(14)	1.570(1)	C(2)-C(3)	1.537(1)
C(2)-C(11)	1.554(2)	C(2)-H(2A)	1.0000
C(3)-C(4)	1.525(2)	C(3)-H(3)	1.0000
C(4)-C(20)	1.527(2)	C(4)-C(5)	1.529(2)
C(4)-H(4)	1.0000	C(5)-C(6)	1.544(2)
C(5)-H(5A)	0.9900	C(5)-H(5B)	0.9900
C(6)-C(7)	1.549(2)	C(6)-H(6)	1.0000
C(7)-C(8)	1.544(2)	C(7)-C(15)	1.563(2)
C(7)-H(7)	1.0000	C(8)-C(9)	1.531(2)
C(8)-H(8A)	0.9900	C(8)-H(8B)	0.9900
C(9)-C(10)	1.533(2)	C(9)-H(9A)	0.9900
C(9)-H(9B)	0.9900	C(10)-C(18)	1.538(2)
C(10)-C(11)	1.553(2)	C(10)-H(10)	1.0000
C(11)-C(12)	1.541(2)	C(11)-H(11)	1.0000
C(12)-C(13)	1.523(2)	C(12)-H(12A)	0.9900
C(12)-H(12B)	0.9900	C(13)-C(14)	1.519(2)
C(13)-H(13A)	0.9900	C(13)-H(13B)	0.9900
C(14)-C(19)	1.526(2)	C(15)-C(16)	1.529(2)
C(15)-C(17)	1.533(2)	C(15)-H(15)	1.0000
C(16)-H(16A)	0.9800	C(16)-H(16B)	0.9800
C(16)-H(16C)	0.9800	C(17)-H(17A)	0.9800
C(17)-H(17B)	0.9800	C(17)-H(17C)	0.9800
C(18)-H(18A)	0.9800	C(18)-H(18B)	0.9800
C(18)-H(18C)	0.9800	C(19)-H(19A)	0.9800
C(19)-H(19B)	0.9800	C(19)-H(19C)	0.9800
C(20)-H(20A)	0.9900	C(20)-H(20B)	0.9900
C(21)-C(22)	1.508(2)	C(21)-C(23)	1.524(2)
C(22)-H(22A)	0.9800	C(22)-H(22B)	0.9800
C(22)-H(22C)	0.9800	C(23)-H(23A)	0.9800
C(23)-H(23B)	0.9800	C(23)-H(23C)	0.9800

C(1)-O(1)-H(1)	109.5	C(14)-O(2)-H(2)	109.5
C(21)-O(3)-C(20)	113.84(8)	C(21)-O(4)-C(3)	116.49(8)
O(1)-C(1)-C(2)	107.98(8)	O(1)-C(1)-C(6)	102.67(8)
C(2)-C(1)-C(6)	115.34(8)	O(1)-C(1)-C(14)	101.97(8)
C(2)-C(1)-C(14)	110.21(8)	C(6)-C(1)-C(14)	117.04(8)
C(3)-C(2)-C(1)	111.57(8)	C(3)-C(2)-C(11)	115.54(8)
C(1)-C(2)-C(11)	117.8(1)	C(3)-C(2)-H(2A)	103.2
C(1)-C(2)-H(2A)	103.2	C(11)-C(2)-H(2A)	103.2
O(4)-C(3)-C(4)	109.09(8)	O(4)-C(3)-C(2)	105.10(8)
C(4)-C(3)-C(2)	115.6(1)	O(4)-C(3)-H(3)	109.0
C(4)-C(3)-H(3)	109.0	C(2)-C(3)-H(3)	109.0
C(3)-C(4)-C(20)	108.8(1)	C(3)-C(4)-C(5)	113.44(8)
C(20)-C(4)-C(5)	111.6(1)	C(3)-C(4)-H(4)	107.6
C(20)-C(4)-H(4)	107.6	C(5)-C(4)-H(4)	107.6
C(4)-C(5)-C(6)	116.2(1)	C(4)-C(5)-H(5A)	108.2
C(6)-C(5)-H(5A)	108.2	C(4)-C(5)-H(5B)	108.2
C(6)-C(5)-H(5B)	108.2	H(5A)-C(5)-H(5B)	107.4
C(5)-C(6)-C(1)	108.50(8)	C(5)-C(6)-C(7)	113.2(1)
C(1)-C(6)-C(7)	122.1(1)	C(5)-C(6)-H(6)	103.6
C(1)-C(6)-H(6)	103.6	C(7)-C(6)-H(6)	103.6
C(8)-C(7)-C(6)	118.1(1)	C(8)-C(7)-C(15)	

107.2(1)

C(6)-C(7)-C(15)	111.1(1)	C(8)-C(7)-H(7)	106.6
C(6)-C(7)-H(7)	106.6	C(15)-C(7)-H(7)	106.6
C(9)-C(8)-C(7)	118.8(1)	C(9)-C(8)-H(8A)	107.6
C(7)-C(8)-H(8A)	107.6	C(9)-C(8)-H(8B)	107.6
C(7)-C(8)-H(8B)	107.6	H(8A)-C(8)-H(8B)	107.1
C(8)-C(9)-C(10)	121.3(1)	C(8)-C(9)-H(9A)	107.0
C(10)-C(9)-H(9A)	107.0	C(8)-C(9)-H(9B)	107.0
C(10)-C(9)-H(9B)	107.0	H(9A)-C(9)-H(9B)	106.8
C(9)-C(10)-C(18)	111.6(1)	C(9)-C(10)-C(11)	

120.7(1)

C(18)-C(10)-C(11)	114.3(1)	C(9)-C(10)-H(10)	102.4
C(18)-C(10)-H(10)	102.4	C(11)-C(10)-H(10)	102.4
C(12)-C(11)-C(10)	111.2(1)	C(12)-C(11)-C(2)	

106.7(1)

C(10)-C(11)-C(2)	125.9(1)	C(12)-C(11)-H(11)	103.5
C(10)-C(11)-H(11)	103.5	C(2)-C(11)-H(11)	103.5
C(13)-C(12)-C(11)	114.6(1)	C(13)-C(12)-H(12A)	108.6
C(11)-C(12)-H(12A)	108.6	C(13)-C(12)-H(12B)	108.6
C(11)-C(12)-H(12B)	108.6	H(12A)-C(12)-H(12B)	107.6
C(14)-C(13)-C(12)	112.5(1)	C(14)-C(13)-H(13A)	109.1
C(12)-C(13)-H(13A)	109.1	C(14)-C(13)-H(13B)	109.1
C(12)-C(13)-H(13B)	109.1	H(13A)-C(13)-H(13B)	107.8
O(2)-C(14)-C(13)	103.9(1)	O(2)-C(14)-C(19)	

107.4(1)

C(13)-C(14)-C(19)	111.8(1)	O(2)-C(14)-C(1)	

107.59(8)

C(13)-C(14)-C(1)	112.9(1)	C(19)-C(14)-C(1)	

112.6(1)

C(16)-C(15)-C(17)	108.1(1)	C(16)-C(15)-C(7)	

116.9(1)

C(17)-C(15)-C(7)	109.4(1)	C(16)-C(15)-H(15)	107.3
C(17)-C(15)-H(15)	107.3	C(7)-C(15)-H(15)	107.3
C(15)-C(16)-H(16A)	109.5	C(15)-C(16)-H(16B)	109.5
H(16A)-C(16)-H(16B)	109.5	C(15)-C(16)-H(16C)	109.5
H(16A)-C(16)-H(16C)	109.5	H(16B)-C(16)-H(16C)	109.5
C(15)-C(17)-H(17A)	109.5	C(15)-C(17)-H(17B)	109.5
H(17A)-C(17)-H(17B)	109.5	C(15)-C(17)-H(17C)	109.5
H(17A)-C(17)-H(17C)	109.5	H(17B)-C(17)-H(17C)	109.5
C(10)-C(18)-H(18A)	109.5	C(10)-C(18)-H(18B)	109.5
H(18A)-C(18)-H(18B)	109.5	C(10)-C(18)-H(18C)	109.5
H(18A)-C(18)-H(18C)	109.5	H(18B)-C(18)-H(18C)	109.5
C(14)-C(19)-H(19A)	109.5	C(14)-C(19)-H(19B)	109.5
H(19A)-C(19)-H(19B)	109.5	C(14)-C(19)-H(19C)	109.5
H(19A)-C(19)-H(19C)	109.5	H(19B)-C(19)-H(19C)	109.5
O(3)-C(20)-C(4)	111.8(1)	O(3)-C(20)-H(20A)	109.2
C(4)-C(20)-H(20A)	109.2	O(3)-C(20)-H(20B)	109.2
C(4)-C(20)-H(20B)	109.2	H(20A)-C(20)-H(20B)	107.9
O(3)-C(21)-O(4)	109.90(8)	O(3)-C(21)-C(22)	

105.8(1)

O(4)-C(21)-C(22)	105.6(1)	O(3)-C(21)-C(23)	

112.3(1)

O(4)-C(21)-C(23)	110.5(1)	C(22)-C(21)-C(23)	

112.5(1)

C(21)-C(22)-H(22A)	109.5	C(21)-C(22)-H(22B)	109.5
H(22A)-C(22)-H(22B)	109.5	C(21)-C(22)-H(22C)	109.5
H(22A)-C(22)-H(22C)	109.5	H(22B)-C(22)-H(22C)	109.5
C(21)-C(23)-H(23A)	109.5	C(21)-C(23)-H(23B)	109.5
H(23A)-C(23)-H(23B)	109.5	C(21)-C(23)-H(23C)	109.5
H(23A)-C(23)-H(23C)	109.5	H(23B)-C(23)-H(23C)	109.5

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for **25**

atom	U11	U22	U33	U23	U13	U12
O(1)	30(1)	28(1)	30(1)	3(1)	15(1)	6(1)
O(2)	27(1)	37(1)	34(1)	3(1)	14(1)	-1(1)
O(3)	32(1)	28(1)	34(1)	4(1)	15(1)	-3(1)
O(4)	29(1)	27(1)	26(1)	4(1)	13(1)	-1(1)

C(1)	26(1)	24(1)	25(1)	1(1)	13(1)	3(1)
C(2)	30(1)	24(1)	26(1)	0(1)	15(1)	-1(1)
C(3)	31(1)	23(1)	27(1)	1(1)	15(1)	1(1)
C(4)	27(1)	28(1)	30(1)	2(1)	15(1)	1(1)
C(5)	29(1)	28(1)	30(1)	-3(1)	14(1)	-3(1)
C(6)	27(1)	29(1)	25(1)	-2(1)	12(1)	2(1)
C(7)	27(1)	43(1)	28(1)	5(1)	13(1)	4(1)
C(8)	29(1)	48(1)	34(1)	9(1)	15(1)	10(1)
C(9)	41(1)	42(1)	51(1)	12(1)	28(1)	15(1)
C(10)	48(1)	27(1)	52(1)	3(1)	34(1)	6(1)
C(11)	41(1)	23(1)	35(1)	-1(1)	23(1)	-1(1)
C(12)	43(1)	24(1)	44(1)	3(1)	25(1)	-1(1)
C(13)	37(1)	32(1)	37(1)	9(1)	22(1)	1(1)
C(14)	27(1)	35(1)	27(1)	3(1)	14(1)	1(1)
C(15)	30(1)	73(1)	29(1)	-5(1)	12(1)	-2(1)
C(16)	47(1)	83(1)	40(1)	-27(1)	12(1)	-8(1)
C(17)	40(1)	122(2)	29(1)	8(1)	13(1)	16(1)
C(18)	55(1)	36(1)	59(1)	-3(1)	41(1)	4(1)
C(19)	37(1)	50(1)	30(1)	-1(1)	21(1)	0(1)
C(20)	29(1)	34(1)	35(1)	3(1)	17(1)	-1(1)
C(21)	32(1)	28(1)	27(1)	4(1)	14(1)	-3(1)
C(22)	37(1)	32(1)	37(1)	8(1)	16(1)	2(1)
C(23)	45(1)	42(1)	29(1)	1(1)	21(1)	-5(1)

The anisotropic displacement factor exponent takes the form
2 pi^2 [h^2a*^2U(11) +...+ 2hka*b*U(12)]

Table 5. Hydrogen Coordinates (A x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for **25**

atom	x	y	z	U(eq)
H(1)	4507	1442	1767	43
H(2)	4999	3396	1412	50
H(2A)	4536	4097	2180	31
H(3)	3970	3591	2667	31
H(4)	3296	2829	1509	33
H(5A)	3217	912	540	35
H(5B)	3744	304	1083	35
H(6)	3771	1626	70	33
H(7)	3426	4181	-509	39
H(8A)	2679	4610	-462	45
H(8B)	2960	3865	455	45
H(9A)	2899	6867	239	51
H(9B)	3326	6632	55	51
H(10)	3558	7650	1342	45
H(11)	4180	6242	2277	37
H(12A)	4187	7891	1196	42
H(12B)	4637	6789	1733	42
H(13A)	3925	6084	66	41
H(13B)	4447	6657	324	41
H(15)	2685	1967	-848	54
H(16A)	2881	220	-1661	91
H(16B)	3257	65	-691	91
H(16C)	3380	1085	-1307.0001	91
H(17A)	2916	3598	-1989	98
H(17B)	2567	4333	-1688	98
H(17C)	2432	2696	-2230	98
H(18A)	3001	6769	1652	67
H(18B)	3504	6398	2433	67
H(18C)	3206	4954	1819	67
H(19A)	4559	4153	-368	56
H(19B)	4037	3579	-627.0001	56
H(19C)	4470	2392	-96.0000	56
H(20A)	3419	1286	2647	38
H(20B)	3200	85	1858	38
H(22A)	4626	-1646	3683	54

H(22B)	4983	-232	3762	54
H(22C)	4679	-1132	2885	54
H(23A)	4041	2061	3741	56
H(23B)	4595	1797	4273	56
H(23C)	4246	402	4248	56

Crystal data for 28

Table 1.

Compound	**28**
Molecular formula	$C_{23}H_{40}O_3$
Molecular weight	364.55
Crystal habit	Colorless Plate
Crystal dimensions(mm)	0.24x0.10x0.05
Crystal system	monoclinic
Space group	$P2_1/a$
a(Å)	17.8800(10)
b(Å)	9.2480(10)
c(Å)	13.0310(10)
α(°)	90.00
β(°)	98.4930(10)
γ(°)	90.00
V(Å^3)	2131.1(3)
Z	4
d(g-cm^{-3})	1.136
F(000)	808
μ(cm^{-1})	0.073
Absorption corrections	multi-scan ; 0.9828 min, 0.9964 max
Diffractometer	KappaCCD
X-ray source	MoKα
λ(Å)	0.71069
Monochromator	graphite
T (K)	150.0(1)
Scan mode	phi and omega scans
Maximum θ	23.81
HKL ranges	-20 20 ; -10 10 ; -14 14
Reflections measured	5894
Unique data	3281
Rint	0.0266
Reflections used	2359
Criterion	I > 2σI)
Refinement type	Fsqd
Hydrogen atoms	mixed
Parameters refined	241
Reflections / parameter	9
wR2	0.1098

R1	0.0415
Weights a, b	0.0599 ; 0.0089
GoF	1.009
difference peak / hole (e $Å^{-3}$)	0.168(0.036) / -0.160(0.036)

Table 2. Atomic Coordinates (A x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for **28**

atom	x	y	z	U(eq)
O(1)	7813(1)	16623(1)	8032(1)	35(1)
O(2)	9046(1)	17400(1)	7142(1)	32(1)
O(3)	9029(1)	19820(1)	6599(1)	40(1)
C(1)	7500(1)	15987(2)	7035(1)	27(1)
C(2)	8174(1)	15568(2)	6488(1)	26(1)
C(3)	8608(1)	16888(2)	6181(1)	28(1)
C(4)	8123(1)	18128(2)	5669(1)	28(1)
C(5)	7452(1)	18482(2)	6236(1)	30(1)
C(6)	6972(1)	17182(2)	6493(1)	29(1)
C(7)	6321(1)	16788(2)	5615(1)	32(1)
C(8)	6526(1)	16523(2)	4524(1)	35(1)
C(9)	6871(1)	15090(2)	4238(2)	38(1)
C(10)	7723(1)	14836(2)	4506(1)	32(1)
C(11)	8045(1)	14405(2)	5627(1)	29(1)
C(12)	7655(1)	13086(2)	6036(2)	35(1)
C(13)	6980(1)	13452(2)	6568(2)	39(1)
C(14)	7114(1)	14613(2)	7354(2)	38(1)
C(15)	5685(1)	17968(2)	5493(2)	37(1)
C(16)	5563(1)	18775(2)	6474(2)	51(1)
C(17)	4929(1)	17308(2)	5022(2)	57(1)
C(18)	7950(1)	13680(2)	3770(2)	44(1)
C(19)	6453(1)	14857(2)	7946(2)	44(1)
C(20)	8628(1)	19433(2)	5602(2)	37(1)
C(21)	9491(1)	18684(2)	7077(2)	37(1)
C(22)	10165(1)	18378(2)	6521(2)	47(1)
C(23)	9739(1)	19135(2)	8182(2)	49(1)

U(eq) is defined as 1/3 the trace of the Uij tensor.

Table 3. Bond lengths (A) and angles (deg) for **28**

O(1)-C(1)	1.461(2)	O(1)-H(1)	0.8199
O(2)-C(21)	1.438(2)	O(2)-C(3)	1.456(2)
O(3)-C(21)	1.422(2)	O(3)-C(20)	1.434(2)
C(1)-C(14)	1.532(2)	C(1)-C(2)	1.538(2)
C(1)-C(6)	1.554(2)	C(2)-C(3)	1.530(2)
C(2)-C(11)	1.547(2)	C(2)-H(2)	1.0000
C(3)-C(4)	1.530(2)	C(3)-H(3)	1.0000
C(4)-C(20)	1.518(2)	C(4)-C(5)	1.534(2)
C(4)-H(4)	1.0000	C(5)-C(6)	1.543(2)
C(5)-H(5A)	0.9900	C(5)-H(5B)	0.9900
C(6)-C(7)	1.550(2)	C(6)-H(6)	1.0000
C(7)-C(8)	1.540(3)	C(7)-C(15)	1.567(2)
C(7)-H(7)	1.0000	C(8)-C(9)	1.531(2)
C(8)-H(8A)	0.9900	C(8)-H(8B)	0.9900
C(9)-C(10)	1.530(2)	C(9)-H(9A)	0.9900
C(9)-H(9B)	0.9900	C(10)-C(18)	1.531(2)
C(10)-C(11)	1.543(2)	C(10)-H(10)	1.0000
C(11)-C(12)	1.538(2)	C(11)-H(11)	1.0000
C(12)-C(13)	1.516(3)	C(12)-H(12A)	0.9900
C(12)-H(12B)	0.9900	C(13)-C(14)	1.479(2)
C(13)-H(13A)	0.9900	C(13)-H(13B)	0.9900
C(14)-C(19)	1.520(3)	C(14)-H(14)	1.0000
C(15)-C(16)	1.524(3)	C(15)-C(17)	1.528(2)
C(15)-H(15)	1.0000	C(16)-H(16A)	0.9800
C(16)-H(16B)	0.9800	C(16)-H(16C)	0.9800
C(17)-H(17A)	0.9800	C(17)-H(17B)	0.9800
C(17)-H(17C)	0.9800	C(18)-H(18A)	0.9800
C(18)-H(18B)	0.9800	C(18)-H(18C)	0.9800
C(19)-H(19A)	0.9800	C(19)-H(19B)	0.9800

C(19)-H(19C)	0.9800	C(20)-H(20A)	0.9900
C(20)-H(20B)	0.9900	C(21)-C(23)	1.502(3)
C(21)-C(22)	1.522(3)	C(22)-H(22A)	0.9800
C(22)-H(22B)	0.9800	C(22)-H(22C)	0.9800
C(23)-H(23A)	0.9800	C(23)-H(23B)	0.9800
C(23)-H(23C)	0.9800		

C(1)-O(1)-H(1)	109.4	C(21)-O(2)-C(3)	116.7(1)
C(21)-O(3)-C(20)	113.1(1)	O(1)-C(1)-C(14)	102.8(1)
O(1)-C(1)-C(2)	106.8(1)	C(14)-C(1)-C(2)	109.4(1)
O(1)-C(1)-C(6)	104.2(1)	C(14)-C(1)-C(6)	116.4(1)
C(2)-C(1)-C(6)	115.7(1)	C(3)-C(2)-C(1)	112.5(1)
C(3)-C(2)-C(11)	113.1(1)	C(1)-C(2)-C(11)	117.9(1)
C(3)-C(2)-H(2)	103.8	C(1)-C(2)-H(2)	103.8
C(11)-C(2)-H(2)	103.8	O(2)-C(3)-C(2)	105.3(1)
O(2)-C(3)-C(4)	109.2(1)	C(2)-C(3)-C(4)	115.8(1)
O(2)-C(3)-H(3)	108.8	C(2)-C(3)-H(3)	108.8
C(4)-C(3)-H(3)	108.8	C(20)-C(4)-C(3)	108.5(1)
C(20)-C(4)-C(5)	111.8(1)	C(3)-C(4)-C(5)	112.5(1)
C(20)-C(4)-H(4)	107.9	C(3)-C(4)-H(4)	107.9
C(5)-C(4)-H(4)	107.9	C(4)-C(5)-C(6)	116.0(1)
C(4)-C(5)-H(5A)	108.3	C(6)-C(5)-H(5A)	108.3
C(4)-C(5)-H(5B)	108.3	C(6)-C(5)-H(5B)	108.3
H(5A)-C(5)-H(5B)	107.4	C(5)-C(6)-C(7)	113.8(1)
C(5)-C(6)-C(1)	109.4(1)	C(7)-C(6)-C(1)	120.5(1)
C(5)-C(6)-H(6)	103.6	C(7)-C(6)-H(6)	103.6
C(1)-C(6)-H(6)	103.6	C(8)-C(7)-C(6)	117.7(1)
C(8)-C(7)-C(15)	106.6(1)	C(6)-C(7)-C(15)	111.4(1)
C(8)-C(7)-H(7)	106.9	C(6)-C(7)-H(7)	106.9
C(15)-C(7)-H(7)	106.9	C(9)-C(8)-C(7)	121.5(2)
C(9)-C(8)-H(8A)	107.0	C(7)-C(8)-H(8A)	107.0
C(9)-C(8)-H(8B)	107.0	C(7)-C(8)-H(8B)	107.0
H(8A)-C(8)-H(8B)	106.7	C(10)-C(9)-C(8)	120.0(2)
C(10)-C(9)-H(9A)	107.3	C(8)-C(9)-H(9A)	107.3
C(10)-C(9)-H(9B)	107.3	C(8)-C(9)-H(9B)	107.3
H(9A)-C(9)-H(9B)	106.9	C(9)-C(10)-C(18)	108.1(2)
C(9)-C(10)-C(11)	118.4(2)	C(18)-C(10)-C(11)	108.5(1)
C(9)-C(10)-H(10)	107.1	C(18)-C(10)-H(10)	107.1
C(11)-C(10)-H(10)	107.1	C(12)-C(11)-C(10)	114.1(1)
C(12)-C(11)-C(2)	109.1(1)	C(10)-C(11)-C(2)	120.2(1)
C(12)-C(11)-H(11)	103.8	C(10)-C(11)-H(11)	103.8
C(2)-C(11)-H(11)	103.8	C(13)-C(12)-C(11)	114.4(1)
C(13)-C(12)-H(12A)	108.7	C(11)-C(12)-H(12A)	108.7
C(13)-C(12)-H(12B)	108.7	C(11)-C(12)-H(12B)	108.7
H(12A)-C(12)-H(12B)	107.6	C(14)-C(13)-C(12)	114.8(2)
C(14)-C(13)-H(13A)	108.6	C(12)-C(13)-H(13A)	108.6

C(14)-C(13)-H(13B)	108.6	C(12)-C(13)-H(13B)	108.6
H(13A)-C(13)-H(13B)	107.5	C(13)-C(14)-C(19)	113.6(2)
C(13)-C(14)-C(1)	116.6(2)	C(19)-C(14)-C(1)	115.4(2)
C(13)-C(14)-H(14)	102.8	C(19)-C(14)-H(14)	102.8
C(1)-C(14)-H(14)	102.8	C(16)-C(15)-C(17)	107.7(2)
C(16)-C(15)-C(7)	116.6(2)	C(17)-C(15)-C(7)	110.4(2)
C(16)-C(15)-H(15)	107.2	C(17)-C(15)-H(15)	107.2
C(7)-C(15)-H(15)	107.2	C(15)-C(16)-H(16A)	109.5
C(15)-C(16)-H(16B)	109.5	H(16A)-C(16)-H(16B)	109.5
C(15)-C(16)-H(16C)	109.5	H(16A)-C(16)-H(16C)	109.5
H(16B)-C(16)-H(16C)	109.5	C(15)-C(17)-H(17A)	109.5
C(15)-C(17)-H(17B)	109.5	H(17A)-C(17)-H(17B)	109.5
C(15)-C(17)-H(17C)	109.5	H(17A)-C(17)-H(17C)	109.5
H(17B)-C(17)-H(17C)	109.5	C(10)-C(18)-H(18A)	109.5
C(10)-C(18)-H(18B)	109.5	H(18A)-C(18)-H(18B)	109.5
C(10)-C(18)-H(18C)	109.5	H(18A)-C(18)-H(18C)	109.5
H(18B)-C(18)-H(18C)	109.5	C(14)-C(19)-H(19A)	109.5
C(14)-C(19)-H(19B)	109.5	H(19A)-C(19)-H(19B)	109.5
C(14)-C(19)-H(19C)	109.5	H(19A)-C(19)-H(19C)	109.5
H(19B)-C(19)-H(19C)	109.5	O(3)-C(20)-C(4)	111.6(1)
O(3)-C(20)-H(20A)	109.3	C(4)-C(20)-H(20A)	109.3
O(3)-C(20)-H(20B)	109.3	C(4)-C(20)-H(20B)	109.3
H(20A)-C(20)-H(20B)	108.0	O(3)-C(21)-O(2)	110.2(1)
O(3)-C(21)-C(23)	106.6(1)	O(2)-C(21)-C(23)	105.1(2)
O(3)-C(21)-C(22)	112.2(2)	O(2)-C(21)-C(22)	111.0(1)
C(23)-C(21)-C(22)	111.4(2)	C(21)-C(22)-H(22A)	109.5
C(21)-C(22)-H(22B)	109.5	H(22A)-C(22)-H(22B)	109.5
C(21)-C(22)-H(22C)	109.5	H(22A)-C(22)-H(22C)	109.5
H(22B)-C(22)-H(22C)	109.5	C(21)-C(23)-H(23A)	109.5
C(21)-C(23)-H(23B)	109.5	H(23A)-C(23)-H(23B)	109.5
C(21)-C(23)-H(23C)	109.5	H(23A)-C(23)-H(23C)	109.5
H(23B)-C(23)-H(23C)	109.5		

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for **28**

atom	U11	U22	U33	U23	U13	U12
O(1)	34(1)	47(1)	25(1)	-4(1)	2(1)	1(1)
O(2)	25(1)	35(1)	35(1)	-5(1)	0(1)	-2(1)
O(3)	32(1)	33(1)	53(1)	-6(1)	3(1)	-3(1)
C(1)	26(1)	33(1)	22(1)	-2(1)	3(1)	0(1)
C(2)	23(1)	30(1)	24(1)	0(1)	0(1)	2(1)
C(3)	25(1)	32(1)	26(1)	-6(1)	3(1)	0(1)
C(4)	25(1)	29(1)	30(1)	-1(1)	3(1)	-1(1)
C(5)	26(1)	31(1)	32(1)	0(1)	4(1)	3(1)
C(6)	25(1)	32(1)	29(1)	-1(1)	6(1)	2(1)
C(7)	25(1)	34(1)	34(1)	1(1)	3(1)	1(1)
C(8)	28(1)	43(1)	31(1)	1(1)	-3(1)	3(1)
C(9)	39(1)	44(1)	28(1)	-5(1)	-2(1)	-1(1)
C(10)	36(1)	31(1)	31(1)	-4(1)	6(1)	0(1)
C(11)	27(1)	30(1)	31(1)	-2(1)	5(1)	2(1)
C(12)	38(1)	31(1)	36(1)	-2(1)	2(1)	0(1)
C(13)	41(1)	33(1)	45(1)	1(1)	10(1)	-5(1)
C(14)	40(1)	35(1)	40(1)	5(1)	13(1)	-1(1)
C(15)	25(1)	45(1)	41(1)	3(1)	2(1)	4(1)
C(16)	38(1)	60(1)	56(1)	-1(1)	7(1)	17(1)
C(17)	29(1)	65(1)	73(2)	-4(1)	-4(1)	5(1)
C(18)	54(1)	42(1)	38(1)	-7(1)	10(1)	2(1)
C(19)	48(1)	45(1)	44(1)	1(1)	18(1)	-7(1)
C(20)	33(1)	35(1)	44(1)	2(1)	7(1)	0(1)
C(21)	25(1)	36(1)	49(1)	-6(1)	2(1)	-4(1)

C(22)	30(1)	50(1)	62(2)	-6(1)	9(1)	-5(1)
C(23)	36(1)	52(1)	56(1)	-16(1)	-1(1)	-6(1)

The anisotropic displacement factor exponent takes the form
2 pi^2 [h^2a*^2U(11) +...+ 2hka*b*U(12)]

Table 5. Hydrogen Coordinates (A x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for **28**

atom	x	y	z	U(eq)
H(1)	8218	17014	7981	42
H(2)	8536	15099	7048	31
H(3)	8964	16568	5702	34
H(4)	7917	17826	4946	34
H(5A)	7117	19169	5802	36
H(5B)	7648	18983	6892	36
H(6)	6703	17554	7059	34
H(7)	6088	15875	5830	38
H(8A)	6059	16672	4023	42
H(8B)	6882	17298	4392	42
H(9A)	6614	14306	4569	45
H(9B)	6736	14964	3477	45
H(10)	7979	15754	4350	39
H(11)	8566	14050	5572	35
H(12A)	7488	12429	5448	42
H(12B)	8031	12558	6534	42
H(13A)	6556	13746	6032	47
H(13B)	6822	12568	6906	47
H(14)	7508	14183	7892	45
H(15)	5822	18704	4990	44
H(16A)	5128	19423	6318	76
H(16B)	6016	19342	6729	76
H(16C)	5468	18078	7006	76
H(17A)	4761	16624	5515	85
H(17B)	4988	16800	4379	85
H(17C)	4551	18077	4870	85
H(18A)	7657	12797	3835	66
H(18B)	8490	13469	3949	66
H(18C)	7846	14033	3054	66
H(19A)	6028	15274	7479	67
H(19B)	6607	15523	8524	67
H(19C)	6300	13932	8217	67
H(20A)	8996	19215	5125	45
H(20B)	8315	20262	5313	45
H(22A)	9986	17995	5827	71
H(22B)	10496	17666	6918	71
H(22C)	10446	19275	6460	71
H(23A)	10037	20026	8195	73
H(23B)	10048	18368	8550	73
H(23C)	9293	19305	8522	73

Crystal data for 32

Table 1.

Compound	**32**
Molecular formula	$C_{20}H_{36}O_3$
Molecular weight	324.49
Crystal habit	Colorless Plate
Crystal dimensions(mm)	0.26x0.20x0.02
Crystal system	orthorhombic
Space group	Pbca
a(Å)	9.457(1)
b(Å)	13.107(1)
c(Å)	28.175(1)
α(°)	90.00
β(°)	90.00
γ(°)	90.00
V(Å^3)	3492.4(5)
Z	8
d(g-cm^{-3})	1.234
F(000)	1440
μ(cm^{-1})	0.080
Absorption corrections	multi-scan ; 0.9795 min, 0.9984 max
Diffractometer	KappaCCD
X-ray source	MoKα
λ(Å)	0.71069
Monochromator	graphite
T (K)	150.0(1)
Scan mode	phi and omega scans
Maximum θ	27.48
HKL ranges	-12 9 ; -17 13 ; -22 36
Reflections measured	20672
Unique data	3983
Rint	0.0596
Reflections used	2480
Criterion	I > 2σI)
Refinement type	Fsqd
Hydrogen atoms	constr
Parameters refined	212
Reflections / parameter	11
wR2	0.1134
R1	0.0449
Weights a, b	0.0534 ; 0.1581
GoF	1.006
difference peak / hole (e Å^{-3})	0.221(0.044) / -0.181(0.044)

Table 2. Atomic Coordinates (A x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for **32**

atom	x	y	z	U(eq)
O(1)	2289(1)	3020(1)	1992(1)	26(1)
O(2)	3332(1)	4896(1)	1872(1)	24(1)
O(3)	5838(1)	1534(1)	2539(1)	28(1)
C(1)	3395(1)	2791(1)	1650(1)	22(1)
C(2)	3371(2)	3649(1)	1271(1)	21(1)
C(3)	4137(2)	4615(1)	1447(1)	20(1)
C(4)	5663(1)	4389(1)	1621(1)	20(1)
C(5)	5607(2)	3591(1)	2024(1)	21(1)
C(6)	4791(2)	2606(1)	1914(1)	21(1)
C(7)	4548(2)	1954(1)	2356(1)	25(1)
C(8)	3569(2)	3360(1)	738(1)	23(1)
C(9)	4970(2)	2958(1)	509(1)	26(1)
C(10)	5987(2)	2396(1)	837(1)	26(1)
C(11)	7085(2)	3043(1)	1107(1)	24(1)
C(12)	6770(2)	4169(1)	1221(1)	21(1)
C(13)	8169(2)	4760(1)	1318(1)	25(1)
C(14)	8965(2)	4434(1)	1766(1)	34(1)
C(15)	9182(2)	4723(1)	893(1)	34(1)
C(16)	3063(2)	4283(1)	447(1)	26(1)
C(17)	3934(2)	5235(1)	568(1)	27(1)
C(18)	3909(2)	5504(1)	1092(1)	23(1)
C(19)	4825(2)	6441(1)	1190(1)	28(1)
C(20)	4563(2)	2258(2)	95(1)	36(1)

U(eq) is defined as 1/3 the trace of the Uij tensor.

Table 3. Bond lengths (A) and angles (deg) for **32**

O(1)-C(1)	1.453(2)	O(1)-H(1)	0.8400
O(2)-C(3)	1.465(2)	O(2)-H(2)	0.8400
O(3)-C(7)	1.434(2)	O(3)-H(3)	0.8400
C(1)-C(6)	1.534(2)	C(1)-C(2)	1.552(2)
C(1)-H(1A)	1.0000	C(2)-C(3)	1.541(2)
C(2)-C(8)	1.559(2)	C(2)-H(2A)	1.0000
C(3)-C(18)	1.552(2)	C(3)-C(4)	1.552(2)
C(4)-C(5)	1.545(2)	C(4)-C(12)	1.565(2)
C(4)-H(4)	1.0000	C(5)-C(6)	1.536(2)
C(5)-H(5A)	0.9900	C(5)-H(5B)	0.9900
C(6)-C(7)	1.528(2)	C(6)-H(6)	1.0000
C(7)-H(7A)	0.9900	C(7)-H(7B)	0.9900
C(8)-C(16)	1.538(2)	C(8)-C(9)	1.566(2)
C(8)-H(8)	1.0000	C(9)-C(10)	1.524(2)
C(9)-C(20)	1.533(2)	C(9)-H(9)	1.0000
C(10)-C(11)	1.541(2)	C(10)-H(10A)	0.9900
C(10)-H(10B)	0.9900	C(11)-C(12)	1.540(2)
C(11)-H(11A)	0.9900	C(11)-H(11B)	0.9900
C(12)-C(13)	1.558(2)	C(12)-H(12)	1.0000
C(13)-C(14)	1.530(2)	C(13)-C(15)	1.534(2)
C(13)-H(13)	1.0000	C(14)-H(14A)	0.9800
C(14)-H(14B)	0.9800	C(14)-H(14C)	0.9800
C(15)-H(15A)	0.9800	C(15)-H(15B)	0.9800
C(15)-H(15C)	0.9800	C(16)-C(17)	1.533(2)
C(16)-H(16A)	0.9900	C(16)-H(16B)	0.9900
C(17)-C(18)	1.519(2)	C(17)-H(17A)	0.9900
C(17)-H(17B)	0.9900	C(18)-C(19)	1.527(2)
C(18)-H(18)	1.0000	C(19)-H(19A)	0.9800
C(19)-H(19B)	0.9800	C(19)-H(19C)	0.9800
C(20)-H(20A)	0.9800	C(20)-H(20B)	0.9800
C(20)-H(20C)	0.9800		

C(1)-O(1)-H(1)	109.5	C(3)-O(2)-H(2)	109.5

C(7)-O(3)-H(3)	109.5	O(1)-C(1)-C(6)	109.4(1)
O(1)-C(1)-C(2)	107.2(1)	C(6)-C(1)-C(2)	117.4(1)
O(1)-C(1)-H(1A)	107.5	C(6)-C(1)-H(1A)	107.5
C(2)-C(1)-H(1A)	107.5	C(3)-C(2)-C(1)	111.5(1)
C(3)-C(2)-C(8)	117.0(1)	C(1)-C(2)-C(8)	119.0(1)
C(3)-C(2)-H(2A)	101.9	C(1)-C(2)-H(2A)	101.9
C(8)-C(2)-H(2A)	101.9	O(2)-C(3)-C(2)	103.0(1)
O(2)-C(3)-C(18)	105.4(1)	C(2)-C(3)-C(18)	110.1(1)
O(2)-C(3)-C(4)	105.9(1)	C(2)-C(3)-C(4)	112.4(1)
C(18)-C(3)-C(4)	118.4(1)	C(5)-C(4)-C(3)	109.2(1)
C(5)-C(4)-C(12)	115.4(1)	C(3)-C(4)-C(12)	115.4(1)
C(5)-C(4)-H(4)	105.2	C(3)-C(4)-H(4)	105.2
C(12)-C(4)-H(4)	105.2	C(6)-C(5)-C(4)	115.9(1)
C(6)-C(5)-H(5A)	108.3	C(4)-C(5)-H(5A)	108.3
C(6)-C(5)-H(5B)	108.3	C(4)-C(5)-H(5B)	108.3
H(5A)-C(5)-H(5B)	107.4	C(7)-C(6)-C(1)	110.7(1)
C(7)-C(6)-C(5)	112.3(1)	C(1)-C(6)-C(5)	113.4(1)
C(7)-C(6)-H(6)	106.6	C(1)-C(6)-H(6)	106.6
C(5)-C(6)-H(6)	106.6	O(3)-C(7)-C(6)	112.4(1)
O(3)-C(7)-H(7A)	109.1	C(6)-C(7)-H(7A)	109.1
O(3)-C(7)-H(7B)	109.1	C(6)-C(7)-H(7B)	109.1
H(7A)-C(7)-H(7B)	107.9	C(16)-C(8)-C(2)	106.6(1)
C(16)-C(8)-C(9)	107.9(1)	C(2)-C(8)-C(9)	125.5(1)
C(16)-C(8)-H(8)	105.1	C(2)-C(8)-H(8)	105.1
C(9)-C(8)-H(8)	105.1	C(10)-C(9)-C(20)	109.3(1)
C(10)-C(9)-C(8)	116.5(1)	C(20)-C(9)-C(8)	107.6(1)
C(10)-C(9)-H(9)	107.7	C(20)-C(9)-H(9)	107.7
C(8)-C(9)-H(9)	107.7	C(9)-C(10)-C(11)	117.3(1)
C(9)-C(10)-H(10A)	108.0	C(11)-C(10)-H(10A)	108.0
C(9)-C(10)-H(10B)	108.0	C(11)-C(10)-H(10B)	108.0
H(10A)-C(10)-H(10B)	107.2	C(12)-C(11)-C(10)	120.0(1)
C(12)-C(11)-H(11A)	107.3	C(10)-C(11)-H(11A)	107.3
C(12)-C(11)-H(11B)	107.3	C(10)-C(11)-H(11B)	107.3
H(11A)-C(11)-H(11B)	106.9	C(11)-C(12)-C(13)	110.4(1)
C(11)-C(12)-C(4)	117.1(1)	C(13)-C(12)-C(4)	110.4(1)
C(11)-C(12)-H(12)	106.0	C(13)-C(12)-H(12)	106.0
C(4)-C(12)-H(12)	106.0	C(14)-C(13)-C(15)	109.1(1)
C(14)-C(13)-C(12)	115.1(1)	C(15)-C(13)-C(12)	112.1(1)
C(14)-C(13)-H(13)	106.7	C(15)-C(13)-H(13)	106.7
C(12)-C(13)-H(13)	106.7	C(13)-C(14)-H(14A)	109.5
C(13)-C(14)-H(14B)	109.5	H(14A)-C(14)-H(14B)	109.5
C(13)-C(14)-H(14C)	109.5	H(14A)-C(14)-H(14C)	109.5
H(14B)-C(14)-H(14C)	109.5	C(13)-C(15)-H(15A)	109.5
C(13)-C(15)-H(15B)	109.5	H(15A)-C(15)-H(15B)	109.5
C(13)-C(15)-H(15C)	109.5	H(15A)-C(15)-H(15C)	109.5
H(15B)-C(15)-H(15C)	109.5	C(17)-C(16)-C(8)	110.8(1)

C(17)-C(16)-H(16A)	109.5	C(8)-C(16)-H(16A)	109.5
C(17)-C(16)-H(16B)	109.5	C(8)-C(16)-H(16B)	109.5
H(16A)-C(16)-H(16B)	108.1	C(18)-C(17)-C(16)	113.3(1)
C(18)-C(17)-H(17A)	108.9	C(16)-C(17)-H(17A)	108.9
C(18)-C(17)-H(17B)	108.9	C(16)-C(17)-H(17B)	108.9
H(17A)-C(17)-H(17B)	107.7	C(17)-C(18)-C(19)	110.8(1)
C(17)-C(18)-C(3)	116.8(1)	C(19)-C(18)-C(3)	114.1(1)
C(17)-C(18)-H(18)	104.6	C(19)-C(18)-H(18)	104.6
C(3)-C(18)-H(18)	104.6	C(18)-C(19)-H(19A)	109.5
C(18)-C(19)-H(19B)	109.5	H(19A)-C(19)-H(19B)	109.5
C(18)-C(19)-H(19C)	109.5	H(19A)-C(19)-H(19C)	109.5
H(19B)-C(19)-H(19C)	109.5	C(9)-C(20)-H(20A)	109.5
C(9)-C(20)-H(20B)	109.5	H(20A)-C(20)-H(20B)	109.5
C(9)-C(20)-H(20C)	109.5	H(20A)-C(20)-H(20C)	109.5
H(20B)-C(20)-H(20C)	109.5		

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for **32**

atom	U11	U22	U33	U23	U13	U12
O(1)	24(1)	26(1)	27(1)	0(1)	7(1)	-2(1)
O(2)	26(1)	21(1)	25(1)	-6(1)	5(1)	-2(1)
O(3)	29(1)	23(1)	31(1)	5(1)	-8(1)	-4(1)
C(1)	23(1)	22(1)	21(1)	-3(1)	5(1)	-1(1)
C(2)	19(1)	21(1)	22(1)	-1(1)	0(1)	1(1)
C(3)	20(1)	20(1)	20(1)	-3(1)	4(1)	1(1)
C(4)	22(1)	18(1)	20(1)	-3(1)	-1(1)	-1(1)
C(5)	22(1)	22(1)	19(1)	0(1)	0(1)	0(1)
C(6)	22(1)	21(1)	20(1)	1(1)	2(1)	0(1)
C(7)	25(1)	24(1)	25(1)	1(1)	-2(1)	-1(1)
C(8)	24(1)	25(1)	20(1)	-2(1)	-1(1)	-3(1)
C(9)	28(1)	25(1)	24(1)	-3(1)	2(1)	0(1)
C(10)	28(1)	24(1)	25(1)	-5(1)	3(1)	1(1)
C(11)	23(1)	22(1)	25(1)	-2(1)	2(1)	0(1)
C(12)	20(1)	20(1)	22(1)	0(1)	1(1)	1(1)
C(13)	22(1)	20(1)	32(1)	0(1)	2(1)	-1(1)
C(14)	26(1)	34(1)	40(1)	-1(1)	-7(1)	-5(1)
C(15)	27(1)	29(1)	47(1)	-3(1)	11(1)	-3(1)
C(16)	26(1)	31(1)	22(1)	1(1)	0(1)	1(1)
C(17)	27(1)	27(1)	26(1)	6(1)	0(1)	3(1)
C(18)	22(1)	22(1)	24(1)	2(1)	0(1)	2(1)
C(19)	31(1)	21(1)	32(1)	4(1)	1(1)	1(1)
C(20)	40(1)	40(1)	29(1)	-10(1)	-1(1)	4(1)

The anisotropic displacement factor exponent takes the form
2 pi^2 [h^2a*^2U(11) +...+ 2hka*b*U(12)]

Table 5. Hydrogen Coordinates (A x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for **32**

atom	x	y	z	U(eq)
H(1)	2391	3617	2093	39
H(2)	3639	5450	1980	36
H(3)	6391	2010	2613	41
H(1A)	3132	2139	1488	26
H(2A)	2358	3868	1279	25
H(4)	5990	5034	1775	24
H(5A)	6588	3405	2110	25
H(5B)	5171	3915	2306	25
H(6)	5403	2195	1696	26
H(7A)	4097	2378	2604	29
H(7B)	3892	1391	2277	29
H(8)	2854	2814	677	28

H(9)	5484	3559	374	31
H(10A)	6505	1885	644	31
H(10B)	5419	2018	1074	31
H(11A)	7277	2694	1412	28
H(11B)	7974	3024	921	28
H(12)	6366	4469	923	25
H(13)	7904	5493	1363	30
H(14A)	9224	3712	1740	51
H(14B)	8357	4533	2044	51
H(14C)	9822	4847	1800	51
H(15A)	9593	4038	868	52
H(15B)	9939	5224	938	52
H(15C)	8661	4882	602	52
H(16A)	3155	4130	104	31
H(16B)	2053	4415	515	31
H(17A)	4926	5121	469	32
H(17B)	3564	5820	383	32
H(18)	2919	5740	1153	27
H(19A)	5807	6293	1103	42
H(19B)	4776	6612	1529	42
H(19C)	4479	7018	1002	42
H(20A)	5411	2086	−88	54
H(20B)	3882	2608	−110	54
H(20C)	4136	1631	220	54