A Novel Route to Preussomerins *via* 2-Aryl Acetal Anions

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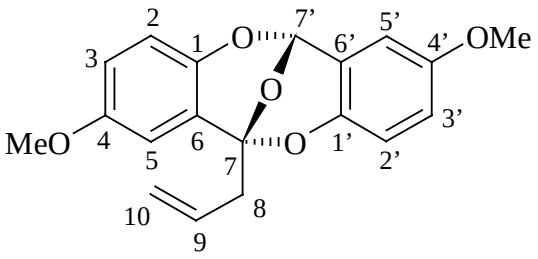
Experimental procedures and analytical data for compounds 12, 15 and 21, crystallographic data for compound 14 (referred in the following as rjkt99-5) and compound 8 (referred in the following as rjkt99-7)

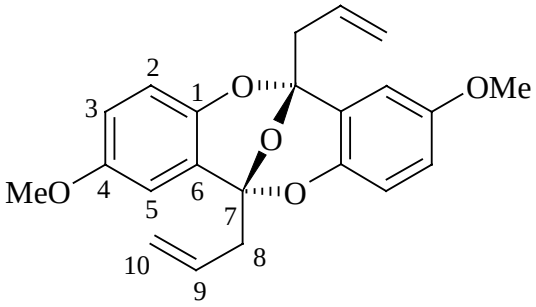
6*H*,12*H*-6-Allyl-2,8-dimethoxy-6,12-epoxydibenzo[*b,f*][1,5]dioxocin (12)



To a solution of methoxydimer **8** (3.0 g, 0.01 mol) in dry THF (50 mL) at -78 °C under N_2, was slowly added a solution of ^{n}BuLi (2.5*M* in hexanes, 4.8 mL, 0.012 mol). The solution became purple / red, and after 40 min at –78 °C an excess of allyl bromide (6.3 g, 4.3 mL, 0.05 mol), freshly passed through alumina, was added. After 40 min stirring at -78 °C, the reaction was quenched with sat. NH_4Cl solution (50 mL) and allowed to warm to rt. The reaction mixture was diluted with EtOAc (50 mL) and the two phases were separated. The aqueous phase was extracted twice with EtOAc (2 × 50 mL) and the organic phases were combined. Drying ($MgSO_4$), filtration and evaporation of the solvents afforded an orange oil which was chromatographed (EtOAc-PE, 3:7) to give the *title compound* **12** (3.1 g, 91 %) as a colorless oil; R_f 0.26 (EtOAc-PE, 1:4); ν_{max} (film)/ cm^{-1} 1497, 1466, 1429, 1275, 1249, 1225, 1205, 1037, 998 and 965; δ_H (270 MHz; $CDCl_3$) 6.82-6.77 (6 H, m, H-2,3,5,2',3',5'), 6.27 (1 H, s, H-7'), 5.85 (1 H, t_{app}dd, *J* 7.0, 10.0 and 17.0, H-9), 5.23 (1 H, br q_{app}d, *J ca.* 1.5 and 17.0, H-10$_{TRANS}$), 5.15 (1 H, br q_{app}d, *J ca.* 1.5 and 10.0, H-10$_{CIS}$), 3.74 (3 H, s, OMe), 3.73 (3 H, s, OMe), 3.16 (1 H, t_{app}dd, *J* 1.5, 7.0 and 15.0, H-8), 3.00 (1 H, t_{app}dd, *J* 1.5, 7.0 and 15.0, H-8); δ_C (67.5 MHz; $CDCl_3$) 154.2 (C-1 or C-1'), 154.0 (C-1 or C-1'), 145.8 (C-4 or C-4'), 144.7 (C-4 or C-4'), 131.0 (C-9), 122.7 (C-6 or C-6'), 120.0 (C-6 or C-6'), 119.1 (C-10), 117.51 (C-5 or C-5'), 117.47 (C-5 or C-5'), 117.1 (C-3 or C-3'), 116.8 (C-3 or C-3'), 110.9 (C-2 or C-2'), 110.3 (C-2 or C-2'), 96.1 (C-7),

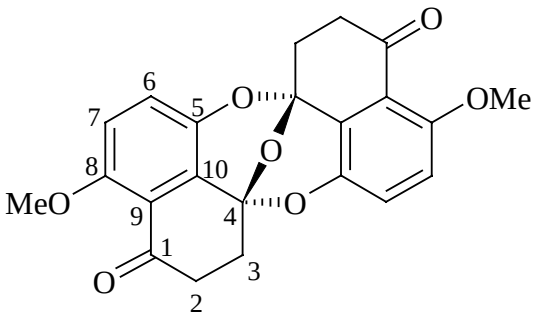
90.1 (C-7’), 55.6 (2 × OMe), 42.0 (C-8); *m /z* (EI) 326 (M^+, 100 %), 257 (92), 175 (53), 151 (40); [HRMS (EI): calc. for $C_{19}H_{18}O_5$, 326.11542. Found: M^+, 326.11544 (0.1 ppm error)].

6*H*,12*H*-6,12-Diallyl-2,8-dimethoxy-6,12-epoxydibenzo[*b,f*][1,5]dioxocin (15)



To a solution of allyl-dimer **12** (3.5 g, 0.011 mol) in dry THF (50 mL) at -78 °C under N_2, was slowly added a solution of ^{s}BuLi (1.3*M*, 9.3 mL, 0.012 mol). The solution became dark red, and after 45 min at –78 °C an excess of allyl bromide (2.6 g, 1.8 mL, 0.02 mol), freshly passed through alumina, was added. After 45 min stirring at –78 °C, the reaction was quenched with a sat. NH_4Cl solution (50 mL) and allowed to warm to rt. The reaction mixture was diluted with EtOAc (50 mL) and the two phases were separated. The aqueous phase was extracted twice with EtOAc (2 × 50 mL) and the organic phases were combined. Drying ($MgSO_4$), filtration and evaporation of the solvents afforded an orange oil which was chromatographed (EtOAc-PE, 1:9) to give the *title compound* **15** (2.04 g, 52 %, 99 % based on RSM) as a bright yellow oil; R_f 0.52 (EtOAc-PE, 1:1); ν_{max} (film)/ cm^{-1} 2931, 1494, 1466, 1432, 1276, 1210, 1159, 1039, 991 and 814; δ_H (270 MHz; acetone-d_6) 6.98 (2 H, d, *J* 2.5, H-5), 6.79 (2 H, dd, *J* 2.5 and 9.0, H-3), 6.73 (2 H, d, *J* 9.0, H-2), 5.78 (2 H, dddd, *J* 6.5, 7.0, 10.0 and 17.0, H-9), 5.20 (2 H, q_{app}d, *J* 1.5 and 17.0, H-10_{TRANS}), 5.05 (2 H, $quin_{app}$d, *J ca.* 1.5 and 10.0, H-10_{CIS}), 3.71 (6 H, s, OMe), 3.14 (2 H, t_{app}dd, *J* 1.5, 7.0 and 15.0, H-8), 3.06 (2 H, t_{app}dd, *J* 1.5, 7.0 and 15.0, H-8); δ_C (67.5 MHz; acetone-d_6) 155.1 (C-1), 147.0 (C-4), 132.3 (C-9), 123.4 (C-6), 119.1 (C-10), 117.8 (C-5), 117.5 (C-3), 111.2 (C-2), 97.6 (C-7), 55.9 (OMe), 42.4 (C-8); *m /z* (CI) 367 (MH^+, 100 %), 327 (25), 193 (23); [HRMS (CI): calc. for $C_{22}H_{23}O_5$, 367.15455. Found: MH^+, 367.15450 (0.1 ppm error)].

4*H*,11*H*-5,6,12,13-Tetrahydro-3,10-dimethoxy-6a,13a-epoxydinaphtho[1,8-*bc*: 1',8'-*fg*][1,5]dioxocin-4,11-dione (21)



(a) Oxalyl chloride (39 mg, 27 μL, 0.31 mmol) was added slowly to a stirred solution of diacid **20** (60 mg, 0.14 mmol) at 0 °C (ice bath) under N_2, in dry DCM (1.5 mL) containing 1 drop of dimethylformamide. The reaction was then stirred for 30 min at rt. Removal of the solvent under vacuum gave the acid chloride which was used in the next step without further purification.

(b) The crude acid chloride prepared in step (a) was dissolved in dry nitrobenzene (10 mL) under N_2 and cooled down to 10 °C (ice bath). $AlCl_3$ (41 mg, 0.31 mmol) was added to the stirred reaction mixture which was warmed to rt. The reaction was quenched after 20 min with Et_3N (0.2 mL). Silica (1 g) was added and the solvent evaporated under high vacuum at 60 °C. Purification by column chromatography (EtOAc-PE, 1:1 then 4:1) afforded the *title compound* **21** (40 mg, 73 %) as a colorless oil; R_f 0.23 (EtOAc-PE, 7:3); ν_{max} (film)/ cm^{-1} 2937, 1685, 1590, 1481, 1438, 185, 1238, 1184, 1048, 1006, 928, 904, 869, 821 and 751; δ_H (500 MHz; acetone-d_6) 7.13 (2 H, d part of an AB system, *J* 9.0, H-7), 7.10 (2 H, d part of an AB system, *J* 9.0, H-6), 3.78 (6 H, s, 2 × OMe), 3.00 (2 H, ddd, *J* 6.5, 13.0 and 19.0, H-2), 2.78 (2 H, ddd, *J* 1.5, 7.0 and 19.0, H-2), 2.63 (2 H, ddd, *J* 1.5, 6.5 and 13.0, H-3), 2.49 (2 H, dt, *J* 7.0 and 13.0, H-3); δ_C (125 MHz; acetone-d_6) 193.3 (C-1), 155.2 (C-5), 143.5 (C-8), 124.4 (C^{IV}), 122.8 (C-H), 120.0 (C^{IV}), 117.0 (C-H), 94.2 (C-4), 56.7 (OMe), 34.7 (C-2), 32.3 (C-3); *m/z* (CI) 395 (MH^+, 100 %); [HRMS (CI): calc. for $C_{22}H_{19}O_7$, 395.11308. Found: MH^+, 395.11389 (2.1 ppm error)].

Crystallographic data for compound 14 (referred in the following as rjkt99-5).

Table 1. Crystal data and structure refinement for rjkt99-5.

Identification code	rjkt99-5
Empirical formula	C20 H18 O3
Formula weight	306.34
Temperature	150(2) K
Wavelength	0.71069 A
Crystal system, space group	Triclinic, P-1
Unit cell dimensions	a = 8.6702(16) A alpha = 91.81(2) deg. b = 12.416(2) A beta = 111.099(17) deg. c = 8.311(2) A gamma = 104.026(15) deg.
Volume	802.8(3) A^3
Z, Calculated density	2, 1.267 Mg/m^3
Absorption coefficient	0.084 mm^-1
F(000)	324
Crystal size	0.50 x 0.30 x 0.20 mm

Theta range for data collection	1.71 to 24.99 deg.
Limiting indices	0<=h<=10, -14<=k<=14, -9<=l<=9
Reflections collected / unique	3033 / 2828 [R(int) = 0.0216]
Completeness to theta = 24.99	100.0 %
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2828 / 0 / 209
Goodness-of-fit on F^2	1.039
Final R indices [I>2sigma(I)]	R1 = 0.0368, wR2 = 0.0962
R indices (all data)	R1 = 0.0653, wR2 = 0.1101
Extinction coefficient	0.030(4)
Largest diff. peak and hole	0.216 and -0.215 e.A^-3

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for shelxl.
U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
O(1)	9422(1)	1181(1)	3178(2)	27(1)
O(2)	11412(1)	1999(1)	2002(1)	24(1)
O(3)	13013(1)	3683(1)	3775(1)	26(1)
C(1)	8901(2)	2147(1)	3083(2)	25(1)
C(2)	7343(2)	2094(2)	3244(2)	35(1)
C(3)	6808(2)	3054(2)	3237(2)	42(1)
C(4)	7826(3)	4068(2)	3096(2)	41(1)
C(5)	9343(2)	4106(2)	2877(2)	33(1)
C(6)	9883(2)	3141(1)	2830(2)	24(1)
C(7)	11428(2)	3125(1)	2369(2)	23(1)
C(8)	11462(2)	3703(1)	789(2)	30(1)
C(9)	9873(2)	3238(2)	-797(2)	36(1)
C(10)	9854(3)	2892(2)	-2305(3)	54(1)
C(11)	13318(2)	3120(1)	5214(2)	23(1)
C(12)	14552(2)	3708(1)	6776(2)	30(1)
C(13)	14861(2)	3189(2)	8262(2)	35(1)
C(14)	13944(2)	2091(2)	8195(2)	35(1)
C(15)	12751(2)	1506(1)	6621(2)	30(1)

C(16)	12422(2)	2005(1)	5093(2)	23(1)
C(17)	11184(2)	1368(1)	3325(2)	24(1)
C(18)	11355(2)	210(1)	2925(2)	31(1)
C(19)	13066(2)	221(2)	2871(2)	35(1)
C(20)	13265(3)	-241(2)	1544(3)	49(1)

Table 3. Bond lengths [A] and angles [deg] for shelxl.

O(1)-C(1)	1.375(2)
O(1)-C(17)	1.4469(18)
O(2)-C(17)	1.4141(19)
O(2)-C(7)	1.4165(19)
O(3)-C(11)	1.3794(19)
O(3)-C(7)	1.4371(19)
C(1)-C(6)	1.389(2)
C(1)-C(2)	1.391(2)
C(2)-C(3)	1.379(3)
C(3)-C(4)	1.387(3)
C(4)-C(5)	1.381(3)
C(5)-C(6)	1.392(2)
C(6)-C(7)	1.525(2)
C(7)-C(8)	1.523(2)
C(8)-C(9)	1.492(2)
C(9)-C(10)	1.305(3)
C(11)-C(12)	1.387(2)
C(11)-C(16)	1.395(2)
C(12)-C(13)	1.381(2)
C(13)-C(14)	1.390(3)
C(14)-C(15)	1.382(3)
C(15)-C(16)	1.397(2)
C(16)-C(17)	1.522(2)
C(17)-C(18)	1.519(2)
C(18)-C(19)	1.497(2)

C(19)-C(20)	1.312(3)

C(1)-O(1)-C(17)	113.10(12)
C(17)-O(2)-C(7)	111.01(12)
C(11)-O(3)-C(7)	112.57(12)
O(1)-C(1)-C(6)	121.62(14)
O(1)-C(1)-C(2)	117.54(15)
C(6)-C(1)-C(2)	120.84(16)
C(3)-C(2)-C(1)	119.48(18)
C(2)-C(3)-C(4)	120.36(17)
C(5)-C(4)-C(3)	119.73(17)
C(4)-C(5)-C(6)	120.85(18)
C(1)-C(6)-C(5)	118.53(16)
C(1)-C(6)-C(7)	119.18(14)
C(5)-C(6)-C(7)	122.11(15)
O(2)-C(7)-O(3)	108.98(12)
O(2)-C(7)-C(8)	107.41(13)
O(3)-C(7)-C(8)	105.93(13)
O(2)-C(7)-C(6)	109.15(12)
O(3)-C(7)-C(6)	111.31(12)
C(8)-C(7)-C(6)	113.87(13)
C(9)-C(8)-C(7)	112.99(14)
C(10)-C(9)-C(8)	124.6(2)
O(3)-C(11)-C(12)	117.28(14)
O(3)-C(11)-C(16)	121.12(14)
C(12)-C(11)-C(16)	121.60(15)
C(13)-C(12)-C(11)	119.26(16)
C(12)-C(13)-C(14)	120.44(17)
C(15)-C(14)-C(13)	119.66(16)

C(14)-C(15)-C(16)	121.21(16)
C(11)-C(16)-C(15)	117.76(15)
C(11)-C(16)-C(17)	119.67(14)
C(15)-C(16)-C(17)	122.54(14)
O(2)-C(17)-O(1)	108.51(12)
O(2)-C(17)-C(18)	107.60(13)
O(1)-C(17)-C(18)	104.80(12)
O(2)-C(17)-C(16)	109.36(13)
O(1)-C(17)-C(16)	110.78(13)
C(18)-C(17)-C(16)	115.49(13)
C(19)-C(18)-C(17)	113.62(14)
C(20)-C(19)-C(18)	123.91(18)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for shelxl. The anisotropic displacement factor exponent takes the form:

-2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
O(1)	20(1)	23(1)	36(1)	5(1)	11(1)	2(1)
O(2)	26(1)	20(1)	28(1)	4(1)	11(1)	6(1)
O(3)	23(1)	21(1)	28(1)	6(1)	5(1)	0(1)
C(1)	21(1)	30(1)	20(1)	1(1)	4(1)	6(1)
C(2)	23(1)	49(1)	31(1)	4(1)	9(1)	8(1)
C(3)	30(1)	69(2)	33(1)	3(1)	11(1)	24(1)
C(4)	43(1)	53(1)	32(1)	-2(1)	8(1)	32(1)
C(5)	37(1)	31(1)	27(1)	1(1)	5(1)	15(1)
C(6)	23(1)	25(1)	19(1)	0(1)	4(1)	6(1)
C(7)	22(1)	19(1)	25(1)	2(1)	6(1)	3(1)
C(8)	32(1)	25(1)	32(1)	9(1)	13(1)	6(1)
C(9)	42(1)	34(1)	29(1)	10(1)	10(1)	10(1)
C(10)	73(2)	50(1)	36(1)	2(1)	11(1)	27(1)
C(11)	19(1)	23(1)	27(1)	6(1)	9(1)	6(1)
C(12)	24(1)	25(1)	35(1)	2(1)	7(1)	4(1)
C(13)	28(1)	42(1)	27(1)	2(1)	2(1)	10(1)
C(14)	37(1)	40(1)	30(1)	13(1)	12(1)	16(1)
C(15)	29(1)	27(1)	35(1)	11(1)	14(1)	7(1)
C(16)	18(1)	21(1)	30(1)	5(1)	10(1)	5(1)

C(17)	20(1)	23(1)	31(1)	7(1)	12(1)	3(1)
C(18)	31(1)	20(1)	38(1)	4(1)	11(1)	5(1)
C(19)	28(1)	27(1)	45(1)	0(1)	8(1)	9(1)
C(20)	40(1)	43(1)	63(1)	-9(1)	17(1)	16(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for shelxl.

	x	y	z	U(eq)
H(2)	6653	1402	3359	41
H(3)	5737	3020	3328	51
H(4)	7481	4734	3149	50
H(5)	10026	4799	2758	39
H(8A)	11606	4512	1062	36
H(8B)	12467	3625	541	36
H(9)	8800	3190	-701	43
H(10A)	10903	2927	-2448	65
H(10B)	8791	2605	-3260	65
H(12)	15178	4459	6824	36
H(13)	15705	3585	9337	42
H(14)	14136	1744	9226	42
H(15)	12144	751	6577	35
H(18A)	11166	-253	3820	37
H(18B)	10446	-147	1787	37
H(19)	14067	585	3856	42
H(20A)	12289	-611	540	59
H(20B)	14386	-207	1587	59

Table 6. Torsion angles [deg] for shelxl.

C(17)-O(1)-C(1)-C(6)	11.1(2)
C(17)-O(1)-C(1)-C(2)	-169.03(14)
O(1)-C(1)-C(2)-C(3)	177.03(15)
C(6)-C(1)-C(2)-C(3)	-3.1(3)
C(1)-C(2)-C(3)-C(4)	-1.0(3)
C(2)-C(3)-C(4)-C(5)	3.1(3)
C(3)-C(4)-C(5)-C(6)	-1.2(3)
O(1)-C(1)-C(6)-C(5)	-175.21(14)
C(2)-C(1)-C(6)-C(5)	5.0(2)
O(1)-C(1)-C(6)-C(7)	9.6(2)
C(2)-C(1)-C(6)-C(7)	-170.25(14)
C(4)-C(5)-C(6)-C(1)	-2.8(2)
C(4)-C(5)-C(6)-C(7)	172.30(15)
C(17)-O(2)-C(7)-O(3)	72.33(15)
C(17)-O(2)-C(7)-C(8)	-173.33(12)
C(17)-O(2)-C(7)-C(6)	-49.42(16)
C(11)-O(3)-C(7)-O(2)	-53.26(16)
C(11)-O(3)-C(7)-C(8)	-168.55(13)
C(11)-O(3)-C(7)-C(6)	67.18(16)
C(1)-C(6)-C(7)-O(2)	9.50(19)
C(5)-C(6)-C(7)-O(2)	-165.54(14)
C(1)-C(6)-C(7)-O(3)	-110.84(15)
C(5)-C(6)-C(7)-O(3)	74.12(18)
C(1)-C(6)-C(7)-C(8)	129.51(16)
C(5)-C(6)-C(7)-C(8)	-45.5(2)

O(2)-C(7)-C(8)-C(9)	64.99(18)
O(3)-C(7)-C(8)-C(9)	-178.65(14)
C(6)-C(7)-C(8)-C(9)	-55.99(19)
C(7)-C(8)-C(9)-C(10)	-127.6(2)
C(7)-O(3)-C(11)-C(12)	-163.88(13)
C(7)-O(3)-C(11)-C(16)	16.28(19)
O(3)-C(11)-C(12)-C(13)	177.92(15)
C(16)-C(11)-C(12)-C(13)	-2.2(2)
C(11)-C(12)-C(13)-C(14)	-0.1(3)
C(12)-C(13)-C(14)-C(15)	1.8(3)
C(13)-C(14)-C(15)-C(16)	-1.2(3)
O(3)-C(11)-C(16)-C(15)	-177.38(14)
C(12)-C(11)-C(16)-C(15)	2.8(2)
O(3)-C(11)-C(16)-C(17)	4.6(2)
C(12)-C(11)-C(16)-C(17)	-175.19(14)
C(14)-C(15)-C(16)-C(11)	-1.0(2)
C(14)-C(15)-C(16)-C(17)	176.89(15)
C(7)-O(2)-C(17)-O(1)	72.55(15)
C(7)-O(2)-C(17)-C(18)	-174.57(12)
C(7)-O(2)-C(17)-C(16)	-48.41(15)
C(1)-O(1)-C(17)-O(2)	-50.50(17)
C(1)-O(1)-C(17)-C(18)	-165.23(13)
C(1)-O(1)-C(17)-C(16)	69.57(16)
C(11)-C(16)-C(17)-O(2)	11.29(19)
C(15)-C(16)-C(17)-O(2)	-166.58(14)
C(11)-C(16)-C(17)-O(1)	-108.27(15)
C(15)-C(16)-C(17)-O(1)	73.85(18)
C(11)-C(16)-C(17)-C(18)	132.80(16)
C(15)-C(16)-C(17)-C(18)	-45.1(2)

O(2)-C(17)-C(18)-C(19)	60.16(18)
O(1)-C(17)-C(18)-C(19)	175.52(14)
C(16)-C(17)-C(18)-C(19)	-62.30(19)
C(17)-C(18)-C(19)-C(20)	-125.4(2)

Symmetry transformations used to generate equivalent atoms:

Table 7. Hydrogen bonds for shelxl [A and deg.].

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

Crystallographic data for compound 8 (referred in the following as rjkt99-7)

Table 1. Crystal data and structure refinement for rjkt99-7.

Identification code	rjkt99-7
Empirical formula	C16 H14 O5
Formula weight	286.27
Temperature	150(2) K
Wavelength	0.71069 A
Crystal system, space group	Monoclinic, P21/a
Unit cell dimensions	a = 7.358(5) A alpha = 90 deg. b = 21.363(4) A beta = 91.51(3) deg. c = 8.3379(17) A gamma = 90 deg.

Volume	1310.1(10) A^3
Z, Calculated density	4, 1.451 Mg/m^3
Absorption coefficient	0.108 mm^-1
F(000)	600
Crystal size	0.60 x 0.50 x 0.15 mm
Theta range for data collection	1.91 to 25.00 deg.
Limiting indices	0<=h<=8, 0<=k<=25, -9<=l<=9
Reflections collected / unique	2493 / 2306 [R(int) = 0.0515]
Completeness to theta = 25.00	100.0 %
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2305 / 0 / 191
Goodness-of-fit on F^2	1.010
Final R indices [I>2sigma(I)]	R1 = 0.0553, wR2 = 0.1477
R indices (all data)	R1 = 0.1089, wR2 = 0.1801
Extinction coefficient	0.025(4)

Largest diff. peak and hole	0.404 and -0.380 e.A^-3

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for shelxl.
U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
O(1)	2302(3)	2014(1)	627(2)	24(1)
O(2)	-3098(3)	217(1)	710(3)	32(1)
O(3)	4370(3)	1333(1)	1890(3)	29(1)
O(4)	2905(3)	1025(1)	4218(3)	30(1)
O(5)	2003(3)	3488(1)	6069(3)	35(1)
C(1)	1000(4)	1552(1)	709(4)	22(1)
C(2)	-597(4)	1619(2)	-225(4)	25(1)
C(3)	-1925(4)	1168(2)	-190(4)	24(1)
C(4)	-1701(4)	636(2)	779(4)	25(1)
C(5)	-101(4)	563(2)	1682(4)	24(1)
C(6)	1252(4)	1025(1)	1647(4)	24(1)
C(7)	3058(5)	929(2)	2522(4)	28(1)
C(8)	-2970(5)	-315(2)	1740(4)	34(1)
C(9)	2669(4)	1647(2)	4600(4)	25(1)
C(14)	3015(4)	2123(2)	3504(3)	22(1)
C(15)	3688(4)	1952(2)	1872(4)	24(1)
C(13)	2787(4)	2751(2)	3944(4)	24(1)
C(12)	2254(4)	2896(2)	5478(4)	26(1)

C(11)	1913(5)	2418(2)	6573(4)	30(1)
C(10)	2130(4)	1802(2)	6142(4)	29(1)
C(16)	2558(5)	3998(2)	5093(4)	35(1)

Table 3. Bond lengths [A] and angles [deg] for shelxl.

O(1)-C(1)	1.379(4)
O(1)-C(15)	1.443(3)
O(2)-C(4)	1.363(4)
O(2)-C(8)	1.426(4)
O(3)-C(7)	1.408(4)
O(3)-C(15)	1.414(4)
O(4)-C(9)	1.380(4)
O(4)-C(7)	1.436(4)
O(5)-C(12)	1.371(4)
O(5)-C(16)	1.427(4)
C(1)-C(6)	1.380(4)
C(1)-C(2)	1.400(4)
C(2)-C(3)	1.373(4)
C(3)-C(4)	1.401(4)
C(4)-C(5)	1.389(4)
C(5)-C(6)	1.403(4)
C(6)-C(7)	1.513(5)
C(9)-C(14)	1.394(4)
C(9)-C(10)	1.395(5)
C(14)-C(13)	1.401(4)
C(14)-C(15)	1.506(4)
C(13)-C(12)	1.383(5)
C(12)-C(11)	1.397(5)
C(11)-C(10)	1.375(5)

C(1)-O(1)-C(15)	112.1(2)
C(4)-O(2)-C(8)	117.4(3)
C(7)-O(3)-C(15)	109.3(2)
C(9)-O(4)-C(7)	112.2(2)
C(12)-O(5)-C(16)	117.2(3)
O(1)-C(1)-C(6)	122.0(3)
O(1)-C(1)-C(2)	118.2(3)
C(6)-C(1)-C(2)	119.8(3)
C(3)-C(2)-C(1)	120.2(3)
C(2)-C(3)-C(4)	120.6(3)
O(2)-C(4)-C(5)	125.1(3)
O(2)-C(4)-C(3)	115.5(3)
C(5)-C(4)-C(3)	119.3(3)
C(4)-C(5)-C(6)	120.0(3)
C(1)-C(6)-C(5)	120.1(3)
C(1)-C(6)-C(7)	119.1(3)
C(5)-C(6)-C(7)	120.6(3)
O(3)-C(7)-O(4)	110.7(3)
O(3)-C(7)-C(6)	109.8(3)
O(4)-C(7)-C(6)	111.4(3)
O(4)-C(9)-C(14)	121.7(3)
O(4)-C(9)-C(10)	118.7(3)
C(14)-C(9)-C(10)	119.5(3)
C(9)-C(14)-C(13)	120.1(3)
C(9)-C(14)-C(15)	119.0(3)
C(13)-C(14)-C(15)	120.9(3)
O(3)-C(15)-O(1)	109.8(2)
O(3)-C(15)-C(14)	110.1(3)
O(1)-C(15)-C(14)	112.6(2)

C(12)-C(13)-C(14)	119.7(3)
O(5)-C(12)-C(13)	125.7(3)
O(5)-C(12)-C(11)	114.3(3)
C(13)-C(12)-C(11)	120.1(3)
C(10)-C(11)-C(12)	120.3(3)
C(11)-C(10)-C(9)	120.3(3)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for shelxl. The anisotropic displacement factor exponent takes the form:

-2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
O(1)	25(1)	28(1)	19(1)	4(1)	-3(1)	-6(1)
O(2)	32(1)	26(1)	39(1)	4(1)	-6(1)	-6(1)
O(3)	22(1)	33(1)	32(1)	1(1)	-1(1)	1(1)
O(4)	35(1)	28(1)	25(1)	8(1)	-9(1)	0(1)
O(5)	46(2)	37(2)	22(1)	-4(1)	2(1)	-9(1)
C(1)	26(2)	26(2)	15(2)	-2(1)	3(1)	0(1)
C(2)	33(2)	28(2)	14(2)	0(1)	2(1)	4(2)
C(3)	25(2)	29(2)	19(2)	-4(1)	-4(1)	2(1)
C(4)	29(2)	22(2)	24(2)	-4(1)	2(1)	-3(1)
C(5)	30(2)	22(2)	20(2)	3(1)	0(1)	5(1)
C(6)	27(2)	23(2)	21(2)	-2(1)	0(1)	3(1)
C(7)	29(2)	29(2)	25(2)	1(1)	0(1)	7(2)
C(8)	43(2)	25(2)	35(2)	1(2)	2(2)	-4(2)
C(9)	21(2)	29(2)	23(2)	5(1)	-8(1)	-1(1)
C(14)	17(2)	33(2)	17(2)	4(1)	-6(1)	-4(1)
C(15)	14(2)	31(2)	26(2)	4(1)	-4(1)	1(1)
C(13)	23(2)	30(2)	18(2)	3(1)	-3(1)	-3(1)
C(12)	24(2)	33(2)	21(2)	-2(1)	-4(1)	-7(1)

C(11)	30(2)	40(2)	19(2)	0(2)	-4(1)	-8(2)
C(10)	27(2)	41(2)	20(2)	10(2)	-6(1)	-9(2)
C(16)	36(2)	36(2)	33(2)	-1(2)	-2(2)	-4(2)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for shelxl.

	x	y	z	U(eq)
H(2)	-763	1978	-885	30
H(3)	-3005	1217	-828	29
H(5)	76	199	2324	29
H(7)	3458	488	2342	34
H(8A)	-4050	-578	1577	52
H(8B)	-2890	-176	2860	52
H(8C)	-1880	-556	1491	52
H(15)	4710	2240	1616	29
H(13)	2999	3075	3192	28
H(11)	1530	2519	7621	36
H(10)	1911	1479	6897	35
H(16A)	2310	4393	5642	53
H(16B)	1882	3987	4065	53
H(16C)	3863	3965	4903	53

Table 6. Torsion angles [deg] for shelxl.

C(15)-O(1)-C(1)-C(6)	-13.3(4)
C(15)-O(1)-C(1)-C(2)	168.2(3)
O(1)-C(1)-C(2)-C(3)	179.6(3)
C(6)-C(1)-C(2)-C(3)	1.0(4)
C(1)-C(2)-C(3)-C(4)	0.1(5)
C(8)-O(2)-C(4)-C(5)	5.1(5)
C(8)-O(2)-C(4)-C(3)	-176.8(3)
C(2)-C(3)-C(4)-O(2)	-179.5(3)
C(2)-C(3)-C(4)-C(5)	-1.3(5)
O(2)-C(4)-C(5)-C(6)	179.4(3)
C(3)-C(4)-C(5)-C(6)	1.4(5)
O(1)-C(1)-C(6)-C(5)	-179.4(3)
C(2)-C(1)-C(6)-C(5)	-0.9(4)
O(1)-C(1)-C(6)-C(7)	-4.3(4)
C(2)-C(1)-C(6)-C(7)	174.1(3)
C(4)-C(5)-C(6)-C(1)	-0.3(5)
C(4)-C(5)-C(6)-C(7)	-175.3(3)
C(15)-O(3)-C(7)-O(4)	-71.7(3)
C(15)-O(3)-C(7)-C(6)	51.7(3)
C(9)-O(4)-C(7)-O(3)	50.8(3)
C(9)-O(4)-C(7)-C(6)	-71.6(3)
C(1)-C(6)-C(7)-O(3)	-15.0(4)
C(5)-C(6)-C(7)-O(3)	160.0(3)
C(1)-C(6)-C(7)-O(4)	108.0(3)
C(5)-C(6)-C(7)-O(4)	-77.0(4)

C(7)-O(4)-C(9)-C(14)	-15.2(4)
C(7)-O(4)-C(9)-C(10)	167.0(3)
O(4)-C(9)-C(14)-C(13)	-179.0(3)
C(10)-C(9)-C(14)-C(13)	-1.2(4)
O(4)-C(9)-C(14)-C(15)	-0.9(4)
C(10)-C(9)-C(14)-C(15)	176.9(3)
C(7)-O(3)-C(15)-O(1)	-72.7(3)
C(7)-O(3)-C(15)-C(14)	51.8(3)
C(1)-O(1)-C(15)-O(3)	50.7(3)
C(1)-O(1)-C(15)-C(14)	-72.3(3)
C(9)-C(14)-C(15)-O(3)	-17.4(4)
C(13)-C(14)-C(15)-O(3)	160.7(3)
C(9)-C(14)-C(15)-O(1)	105.5(3)
C(13)-C(14)-C(15)-O(1)	-76.5(4)
C(9)-C(14)-C(13)-C(12)	1.3(5)
C(15)-C(14)-C(13)-C(12)	-176.8(3)
C(16)-O(5)-C(12)-C(13)	-8.4(5)
C(16)-O(5)-C(12)-C(11)	171.9(3)
C(14)-C(13)-C(12)-O(5)	179.2(3)
C(14)-C(13)-C(12)-C(11)	-1.1(5)
O(5)-C(12)-C(11)-C(10)	-179.4(3)
C(13)-C(12)-C(11)-C(10)	0.9(5)
C(12)-C(11)-C(10)-C(9)	-0.8(5)
O(4)-C(9)-C(10)-C(11)	178.8(3)
C(14)-C(9)-C(10)-C(11)	1.0(5)

Symmetry transformations used to generate equivalent atoms:

Table 7. Hydrogen bonds for shelxl [A and deg.].

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