Appendix I: rate calculations

For the cycloadditions, the dienophile (BVE) is also the solvent so we can assume that the reaction is first order only in the starting pyrone. In the cycloreversions, the reaction is also first order only in the starting cycloadduct.

The rate of cycloaddition reaction is defined as the concentration of product (ie cycloadduct), formed per second. According to the rate equation, this rate equal the rate constant multiplied by the concentration of the starting material, ie the pyrone.

$$\text{rate} = [\text{cycloadduct}] \div \text{reaction time} = k\,[\text{pyrone}]$$

$$\ln(\text{rate}) = \ln(k) - \ln([\text{pyrone}])$$

According to the Arrhenius equation:

$$k = A \exp(-E_A/RT)$$

and

$$\ln(k) = \ln(A) - E_A/RT$$

Therefore,

$$\ln(\text{rate}) = \ln(A) - E_A/RT - \ln([\text{pyrone}])$$

or

$$\ln(\text{rate}) = \ln(A) - \ln([\text{pyrone}]) - E_A/RT$$

Therefore, a graph of ln(rate), (y-axis), versus 1/RT, (x-axis), gives a slope of $-E_A$ and intercept of ln(A)- ln([pyrone]).

It is important to note that as the concentration of the cycloadduct increases, the concentration of pyrone decreases. So the values for the rate are most accurate at the earlier part of the reaction. This means that there is an inherent error to the values which are read over longer periods.

E_A for the cycloaddition of ethyl coumalate and BVE

Scheme 107

Table 11

Temperature/^{o}C	**Time/h**	**Conversion/%**	**1/RT** (mol/KJ)	**ln(rate)**
45	1.5	27	0.3782	-11.603
60	1.5	56	0.3612	-11.428
75	1.5	89	0.3456	-10.729

Typical calculations:

At 45 ^{o}C,

At t = 0 sec: [product] = 0, [SM] = 25 mg/mL or 0.149 moldm^{-3}

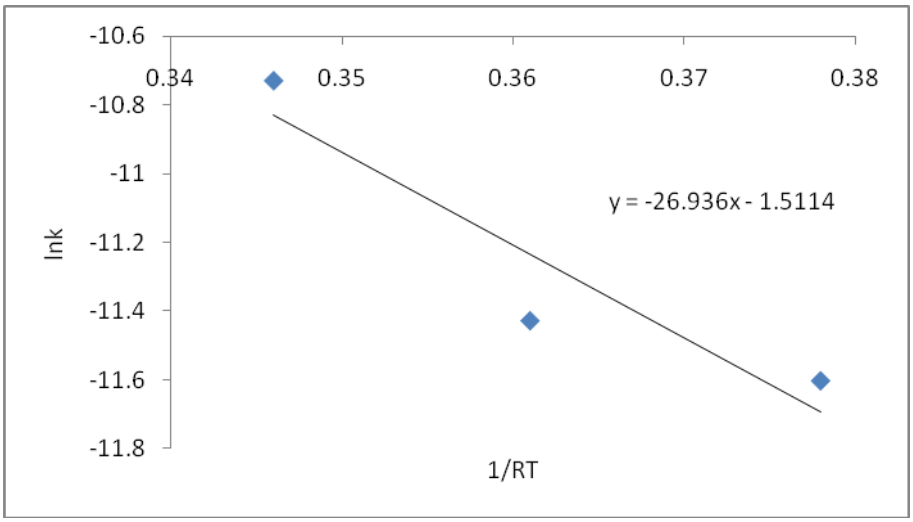
At t = 4400 sec (after 1.5 hours): [product] = 0.27 [reactant]

Rate = 0.149 moldm^{-3} x 0.27 in 4400 s

= 9.14 x 10^{-6} moldm^{-3}s^{-1}

ln (rate) = -11.603

1/RT = 1/(8.314 x10^{-3} x 318) = 0.37822



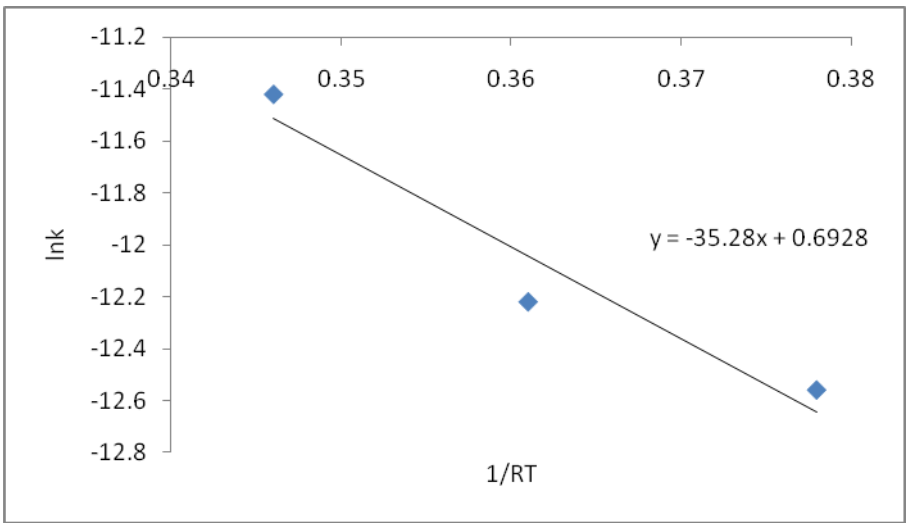
E_A = 26.9 KJmol^{-1} or 6.4 Kcalmol^{-1}

E_A for the cycloaddition of 3-carbomethoxypyran-2-one and BVE

Scheme 108

Table 12

Temperature/^{o}C	**Time/h**	**Conversion/%**	**1/RT** (mol/kJ)	**ln(rate)**
45	1.5	0.095	0.378	-12.56
60	1.5	16	0.361	-12.22
75	1.5	39	0.346	-11.42



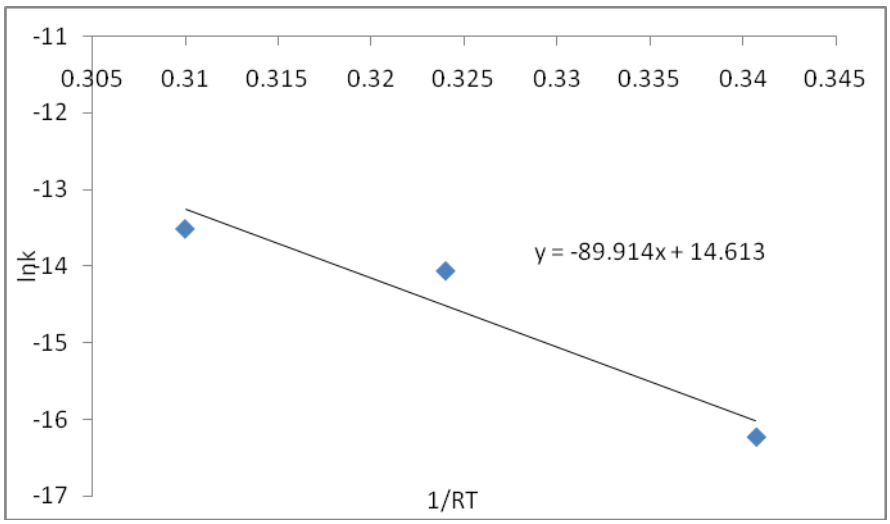
E_A = 35.3 KJmol^{-1} or 8.4 Kcalmol^{-1}

E_A for the loss of CO_2 from the cycloadduct of 3-carbomethoxypyran-2one and BVE

Scheme 109

Table 13

Temperature/^{o}C	**Time/h**	**Conversion/%**	**1/RT** (mol/KJ)	**ln(rate)**
80	11.5	4	0.341	-16.23
98	11.5	35	0.324	-14.06
115	11.5	61	0.310	-13.51



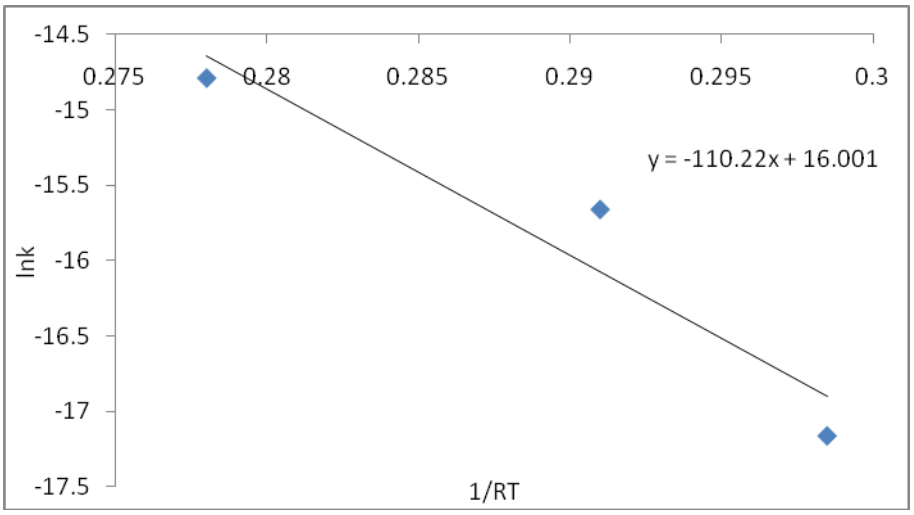
E_A = 89.9 KJmol^{-1} or 21.5 Kcalmol^{-1}

E_A for the loss of CO_2 from the cycloadduct of ethyl coumalate and BVE

Scheme 110

Table 14

Temperature/^{o}C	Time/h	Conversion/%	1/RT (mol/KJ)	ln(rate)
130	14	2.4	0.2985	-17.16
140	12h10min	9.3	0.291	-15.66
160	12h15min	22.5	0.278	-14.79



E_A = 110.2 KJmol^{-1} or 26.3 Kcalmol^{-1}

Appendix II: X-ray crystallography

Compound 246b

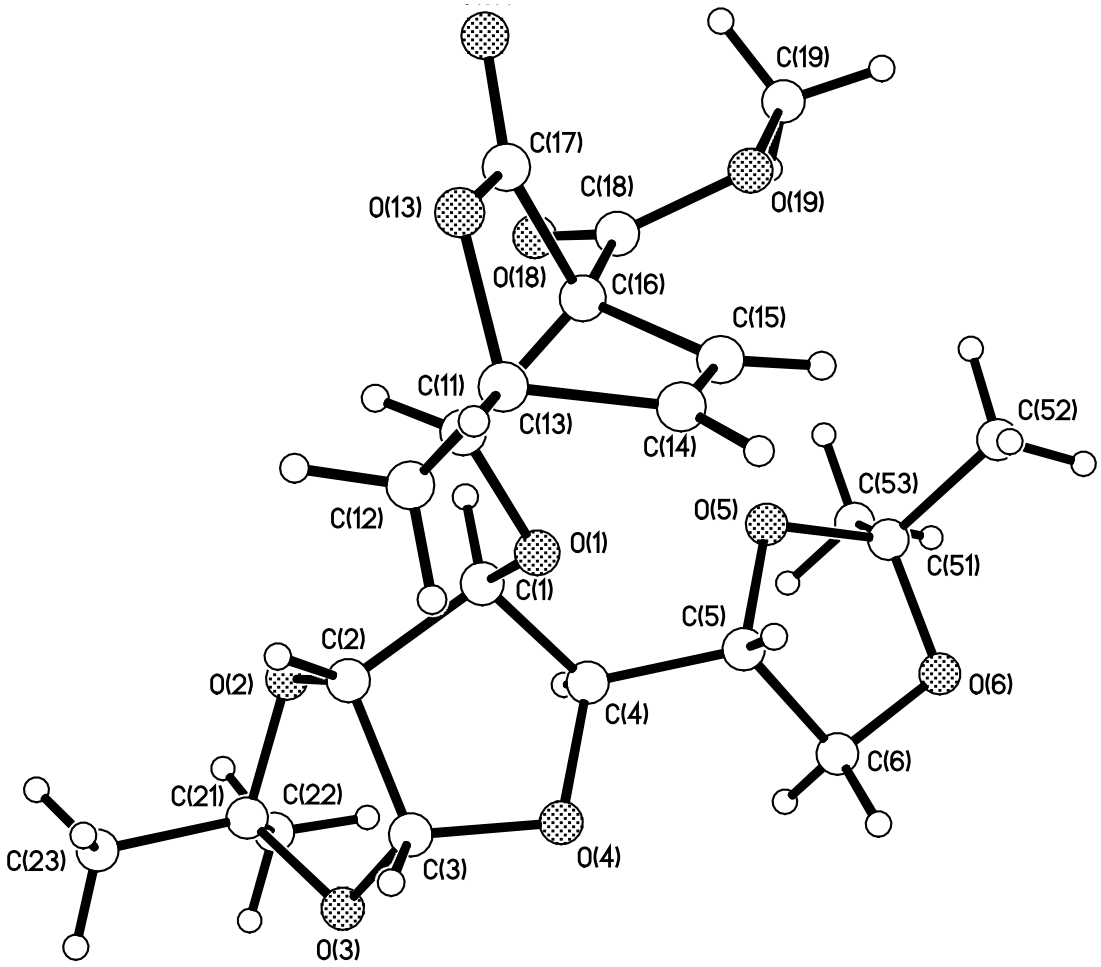


Table 16. Crystal data and structure refinement for compound **246b**.

Empirical formula	C21 H28 O10	
Formula weight	440.43	
Temperature	296(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	P2(1)2(1)2(1)	
Unit cell dimensions	a = 10.0245(6) Å	α= 90°.
	b = 12.5364(8) Å	β= 90°.
	c = 17.6378(11) Å	γ = 90°.
Volume	2216.6(2) $Å^3$	
Z	4	
Density (calculated)	1.320 Mg/m^3	
Absorption coefficient	0.105 mm^{-1}	
F(000)	936	
Crystal size	0.55 x 0.39 x 0.28 mm^3	

Theta range for data collection	1.99 to 28.02°.
Index ranges	-12<=h<=13, -16<=k<=16, -22<=l<=23
Reflections collected	17484
Independent reflections	5134 [R(int) = 0.0239]
Completeness to theta = 28.02°	97.4 %
Absorption correction	None
Max. and min. transmission	0.9707 and 0.9448
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	5134 / 0 / 285
Goodness-of-fit on F^2	1.032
Final R indices [I>2sigma(I)]	R1 = 0.0437, wR2 = 0.1004
R indices (all data)	R1 = 0.0626, wR2 = 0.1095
Absolute structure parameter	-0.5(9)
Largest diff. peak and hole	0.251 and -0.204 e.Å^{-3}

Table 17. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å^{2}x 10^3) for compound **246b**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O(1)	4512(1)	-727(1)	1137(1)	47(1)
O(2)	7692(1)	576(1)	1457(1)	56(1)
O(3)	7304(2)	659(1)	2718(1)	64(1)
O(4)	5067(1)	634(1)	2392(1)	52(1)
O(5)	3259(2)	1744(2)	807(1)	85(1)
O(6)	1984(2)	2674(2)	1638(1)	98(1)
O(13)	3776(2)	-3427(1)	-108(1)	77(1)
O(17)	3874(2)	-2487(1)	-1165(1)	74(1)
O(18)	4598(2)	-83(1)	-633(1)	69(1)
O(19)	2399(2)	-336(1)	-739(1)	70(1)
C(1)	5443(2)	126(1)	1142(1)	41(1)
C(2)	6672(2)	-180(2)	1598(1)	45(1)
C(3)	6270(2)	51(2)	2417(1)	46(1)
C(4)	4865(2)	1005(1)	1632(1)	45(1)
C(5)	3410(2)	1255(2)	1529(1)	55(1)
C(6)	2875(3)	2086(2)	2067(1)	64(1)
C(11)	4746(2)	-1558(1)	604(1)	43(1)
C(12)	4739(2)	-2641(2)	1002(1)	57(1)
C(13)	3571(3)	-3288(2)	712(1)	69(1)
C(14)	2334(3)	-2677(2)	824(1)	72(1)
C(15)	2310(2)	-1746(2)	468(1)	59(1)

C(16)	3574(2)	-1539(2)	21(1)	44(1)
C(17)	3755(2)	-2510(2)	-490(1)	56(1)
C(18)	3607(2)	-563(2)	-476(1)	50(1)
C(19)	2330(3)	540(2)	-1273(2)	95(1)
C(21)	8322(2)	823(2)	2161(1)	63(1)
C(22)	8691(3)	1990(3)	2154(2)	97(1)
C(23)	9457(3)	77(3)	2311(2)	101(1)
C(51)	2353(3)	2619(2)	863(1)	67(1)
C(52)	1113(3)	2368(3)	426(2)	99(1)
C(53)	2995(4)	3624(3)	602(2)	106(1)

Table 18. Bond lengths [Å] and angles [°] for compound **246b**.

O(1)-C(1)	1.419(2)	C(15)-C(16)	1.515(3)
O(1)-C(11)	1.423(2)	C(16)-C(18)	1.505(3)
O(2)-C(2)	1.416(2)	C(16)-C(17)	1.525(3)
O(2)-C(21)	1.428(2)	C(21)-C(23)	1.497(4)
O(3)-C(3)	1.392(2)	C(21)-C(22)	1.509(4)
O(3)-C(21)	1.432(3)	C(51)-C(53)	1.488(4)
O(4)-C(3)	1.411(2)	C(51)-C(52)	1.496(4)
O(4)-C(4)	1.434(2)	C(1)-O(1)-C(11)	116.54(13)
O(5)-C(5)	1.422(3)	C(2)-O(2)-C(21)	108.18(14)
O(5)-C(51)	1.428(3)	C(3)-O(3)-C(21)	110.31(15)
O(6)-C(6)	1.383(3)	C(3)-O(4)-C(4)	108.51(13)
O(6)-C(51)	1.418(3)	C(5)-O(5)-C(51)	109.68(16)
O(13)-C(17)	1.332(3)	C(6)-O(6)-C(51)	109.41(18)
O(13)-C(13)	1.470(3)	C(17)-O(13)-C(13)	113.07(16)
O(17)-C(17)	1.198(2)	C(18)-O(19)-C(19)	115.67(19)
O(18)-C(18)	1.193(2)	O(1)-C(1)-C(4)	107.43(14)
O(19)-C(18)	1.327(3)	O(1)-C(1)-C(2)	110.22(14)
O(19)-C(19)	1.448(3)	C(4)-C(1)-C(2)	101.00(14)
C(1)-C(4)	1.516(3)	O(2)-C(2)-C(1)	108.84(14)
C(1)-C(2)	1.521(2)	O(2)-C(2)-C(3)	103.29(14)
C(2)-C(3)	1.527(3)	C(1)-C(2)-C(3)	103.85(15)
C(4)-C(5)	1.503(3)	O(3)-C(3)-O(4)	111.38(15)
C(5)-C(6)	1.508(3)	O(3)-C(3)-C(2)	105.59(16)
C(11)-C(12)	1.528(3)	O(4)-C(3)-C(2)	107.13(14)
C(11)-C(16)	1.562(2)	O(4)-C(4)-C(5)	108.49(16)
C(12)-C(13)	1.514(3)	O(4)-C(4)-C(1)	104.10(14)
C(13)-C(14)	1.471(4)	C(5)-C(4)-C(1)	116.98(16)
C(14)-C(15)	1.326(3)	O(5)-C(5)-C(4)	107.55(18)

O(5)-C(5)-C(6)	103.16(16)	O(17)-C(17)-O(13)	121.5(2)
C(4)-C(5)-C(6)	114.43(18)	O(17)-C(17)-C(16)	125.4(2)
O(6)-C(6)-C(5)	104.75(18)	O(13)-C(17)-C(16)	113.14(17)
O(1)-C(11)-C(12)	110.25(15)	O(18)-C(18)-O(19)	124.76(19)
O(1)-C(11)-C(16)	107.49(14)	O(18)-C(18)-C(16)	124.29(19)
C(12)-C(11)-C(16)	108.23(15)	O(19)-C(18)-C(16)	110.91(17)
C(13)-C(12)-C(11)	108.92(17)	O(2)-C(21)-O(3)	104.50(16)
O(13)-C(13)-C(14)	108.2(2)	O(2)-C(21)-C(23)	110.7(2)
O(13)-C(13)-C(12)	106.7(2)	O(3)-C(21)-C(23)	109.3(2)
C(14)-C(13)-C(12)	109.10(18)	O(2)-C(21)-C(22)	108.2(2)
C(15)-C(14)-C(13)	114.2(2)	O(3)-C(21)-C(22)	108.6(2)
C(14)-C(15)-C(16)	112.5(2)	C(23)-C(21)-C(22)	114.9(2)
C(18)-C(16)-C(15)	117.49(18)	O(6)-C(51)-O(5)	105.71(18)
C(18)-C(16)-C(17)	107.66(15)	O(6)-C(51)-C(53)	111.7(2)
C(15)-C(16)-C(17)	105.68(16)	O(5)-C(51)-C(53)	110.7(2)
C(18)-C(16)-C(11)	112.27(15)	O(6)-C(51)-C(52)	106.9(2)
C(15)-C(16)-C(11)	106.43(15)	O(5)-C(51)-C(52)	109.3(2)
C(17)-C(16)-C(11)	106.64(16)	C(53)-C(51)-C(52)	112.2(3)

Symmetry transformations used to generate equivalent atoms:

Table 19. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for compound **246b**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O(1)	47(1)	48(1)	47(1)	-14(1)	7(1)	-8(1)
O(2)	47(1)	80(1)	41(1)	-1(1)	-3(1)	-12(1)
O(3)	62(1)	87(1)	42(1)	-6(1)	-8(1)	-14(1)
O(4)	57(1)	61(1)	39(1)	-8(1)	0(1)	11(1)
O(5)	97(1)	108(1)	51(1)	-13(1)	-11(1)	61(1)
O(6)	126(2)	106(1)	61(1)	-1(1)	8(1)	71(1)
O(13)	126(2)	48(1)	57(1)	-7(1)	-9(1)	-19(1)
O(17)	101(1)	79(1)	42(1)	-13(1)	-5(1)	-12(1)
O(18)	73(1)	76(1)	57(1)	18(1)	-5(1)	-23(1)
O(19)	65(1)	74(1)	72(1)	19(1)	-14(1)	3(1)
C(1)	45(1)	43(1)	36(1)	-2(1)	0(1)	-5(1)
C(2)	45(1)	45(1)	43(1)	-3(1)	-2(1)	3(1)
C(3)	54(1)	45(1)	39(1)	3(1)	-6(1)	0(1)
C(4)	49(1)	39(1)	46(1)	-3(1)	-7(1)	-2(1)
C(5)	53(1)	55(1)	58(1)	-10(1)	-8(1)	9(1)
C(6)	74(2)	66(1)	53(1)	-7(1)	-4(1)	22(1)

C(11)	46(1)	45(1)	38(1)	-7(1)	-3(1)	1(1)
C(12)	74(1)	49(1)	50(1)	1(1)	-6(1)	8(1)
C(13)	104(2)	51(1)	51(1)	3(1)	-8(1)	-16(1)
C(14)	74(2)	90(2)	52(1)	12(1)	-2(1)	-37(1)
C(15)	47(1)	82(2)	47(1)	0(1)	-2(1)	-11(1)
C(16)	47(1)	49(1)	38(1)	-2(1)	-3(1)	-10(1)
C(17)	66(1)	56(1)	45(1)	-8(1)	-7(1)	-14(1)
C(18)	58(1)	53(1)	37(1)	0(1)	-5(1)	-6(1)
C(19)	110(2)	87(2)	88(2)	33(2)	-13(2)	21(2)
C(21)	56(1)	85(2)	47(1)	-3(1)	-8(1)	-12(1)
C(22)	105(2)	102(2)	83(2)	-5(2)	0(2)	-48(2)
C(23)	61(2)	153(3)	90(2)	-9(2)	-24(1)	18(2)
C(51)	70(2)	77(2)	55(1)	-8(1)	-3(1)	31(1)
C(52)	77(2)	119(2)	101(2)	-6(2)	-16(2)	27(2)
C(53)	111(3)	107(2)	101(2)	0(2)	8(2)	-4(2)

Table 20. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^{2}x 10^3) for compound **246b**.

	x	y	z	U(eq)
H(1)	5666	372	630	49
H(2)	6957	-919	1516	53
H(3)	6156	-613	2703	55
H(4)	5385	1658	1555	53
H(5)	2876	602	1558	66
H(6A)	2428	1753	2493	77
H(6B)	3589	2536	2256	77
H(11)	5599	-1445	345	51
H(12A)	5567	-3015	900	69
H(12B)	4660	-2542	1545	69
H(13)	3522	-3980	968	82
H(14)	1630	-2923	1120	86
H(15)	1597	-1273	490	71
H(19A)	2731	1162	-1052	142
H(19B)	1414	687	-1392	142
H(19C)	2800	351	-1728	142
H(22A)	9292	2127	1741	145
H(22B)	9116	2173	2624	145
H(22C)	7900	2413	2091	145

H(23A)	9143	-646	2289	152
H(23B)	9819	217	2805	152
H(23C)	10138	181	1935	152
H(52A)	814	1663	552	148
H(52B)	1300	2406	-108	148
H(52C)	431	2875	552	148
H(53A)	2391	4209	674	159
H(53B)	3215	3564	74	159
H(53C)	3794	3747	889	159

Compound 260

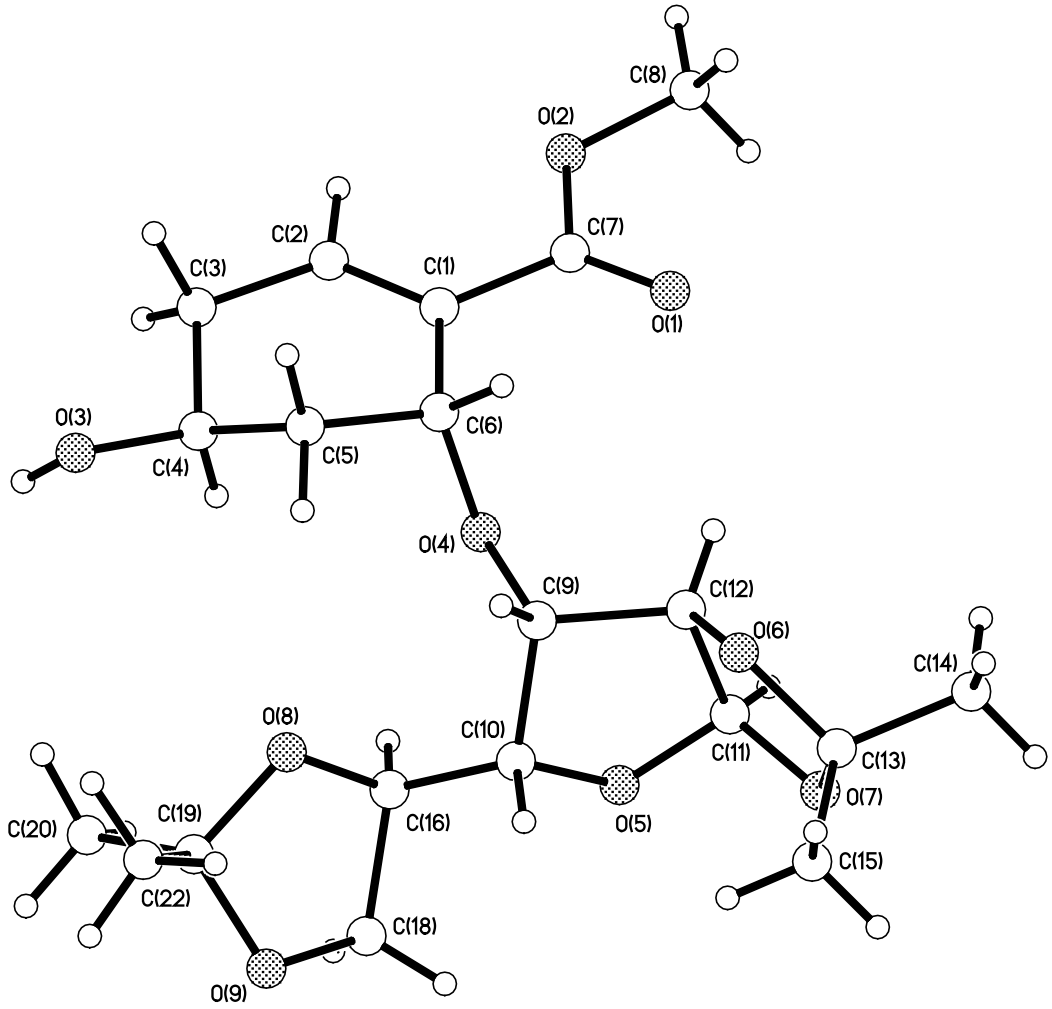


Table 21. Crystal data and structure refinement for compound **260**.

Empirical formula	C20 H30 O9	
Formula weight	414.44	
Temperature	296(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	C222(1)	
Unit cell dimensions	a = 15.1798(2) Å	α= 90°.
	b = 20.4990(3) Å	β= 90°.
	c = 13.4697(2) Å	γ = 90°.
Volume	4191.38(10) $Å^3$	
Z	8	
Density (calculated)	1.314 Mg/m^3	
Absorption coefficient	0.103 mm^{-1}	
F(000)	1776	
Crystal size	0.42 x 0.38 x 0.26 mm^3	

Theta range for data collection	2.68 to 30.00°.
Index ranges	-21<=h<=21, -28<=k<=28, -17<=l<=18
Reflections collected	42933
Independent reflections	6110 [R(int) = 0.0279]
Completeness to theta = 30.00°	99.9 %
Absorption correction	Empirical
Max. and min. transmission	0.9736 and 0.9577
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	6110 / 0 / 319
Goodness-of-fit on F^2	1.004
Final R indices [I>2sigma(I)]	R1 = 0.0440, wR2 = 0.1107
R indices (all data)	R1 = 0.0565, wR2 = 0.1175
Absolute structure parameter	0.0(8)
Largest diff. peak and hole	0.298 and -0.241 e.$Å^{-3}$

Table 22. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for compound **260**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O(1)	1063(1)	1299(1)	2400(1)	55(1)
O(2)	1055(1)	389(1)	3308(1)	64(1)
O(3)	-376(1)	2401(1)	6550(1)	85(1)
O(4)	1296(1)	2435(1)	3960(1)	38(1)
O(5)	2726(1)	3363(1)	3371(1)	64(1)
O(6)	1201(1)	3441(1)	1749(1)	54(1)
O(7)	2677(1)	3612(1)	1700(1)	62(1)
O(8)	1259(1)	3572(1)	5443(1)	96(1)
O(9)	2051(1)	4447(1)	5883(1)	67(1)
C(1)	657(1)	1376(1)	4100(1)	41(1)
C(2)	513(1)	1065(1)	4959(2)	54(1)
C(3)	167(1)	1387(1)	5867(1)	60(1)
C(4)	178(1)	2115(1)	5806(1)	51(1)
C(5)	-129(1)	2340(1)	4792(1)	47(1)
C(6)	468(1)	2090(1)	3971(1)	36(1)
C(7)	951(1)	1033(1)	3185(1)	44(1)
C(8)	1264(2)	29(1)	2414(2)	70(1)
C(9)	1246(1)	3037(1)	3438(1)	36(1)
C(10)	1910(1)	3523(1)	3857(1)	46(1)

C(11)	2554(1)	3133(1)	2416(1)	42(1)
C(12)	1575(1)	2953(1)	2372(1)	38(1)
C(13)	1875(1)	3769(1)	1214(1)	48(1)
C(14)	1922(2)	3539(2)	157(2)	79(1)
C(15)	1722(2)	4493(1)	1300(3)	96(1)
C(16)	2078(1)	3489(1)	4959(1)	59(1)
C(18)	2642(2)	4058(1)	5345(2)	66(1)
C(19)	1349(1)	4045(1)	6203(1)	58(1)
C(20)	1562(2)	3725(1)	7186(2)	67(1)
C(22)	518(2)	4438(2)	6236(3)	102(1)

Table 23. Bond lengths [Å] and angles [°] for compound **260**.

O(1)-C(7)	1.202(2)	C(11)-C(12)	1.533(2)
O(2)-C(7)	1.340(2)	C(13)-C(14)	1.502(3)
O(2)-C(8)	1.447(3)	C(13)-C(15)	1.507(3)
O(3)-C(4)	1.433(2)	C(16)-C(18)	1.538(3)
O(4)-C(9)	1.4241(17)	C(19)-C(22)	1.498(4)
O(4)-C(6)	1.4423(16)	C(19)-C(20)	1.513(3)
O(5)-C(11)	1.394(2)	C(7)-O(2)-C(8)	115.17(16)
O(5)-C(10)	1.439(2)	C(9)-O(4)-C(6)	112.52(10)
O(6)-C(13)	1.421(2)	C(11)-O(5)-C(10)	109.60(12)
O(6)-C(12)	1.4239(19)	C(13)-O(6)-C(12)	110.16(12)
O(7)-C(11)	1.389(2)	C(11)-O(7)-C(13)	111.41(12)
O(7)-C(13)	1.420(2)	C(16)-O(8)-C(19)	109.40(15)
O(8)-C(16)	1.413(3)	C(18)-O(9)-C(19)	107.98(16)
O(8)-C(19)	1.416(3)	C(2)-C(1)-C(7)	122.65(15)
O(9)-C(18)	1.401(3)	C(2)-C(1)-C(6)	122.16(16)
O(9)-C(19)	1.414(3)	C(7)-C(1)-C(6)	115.05(13)
C(1)-C(2)	1.340(2)	C(1)-C(2)-C(3)	123.91(17)
C(1)-C(7)	1.487(2)	C(2)-C(3)-C(4)	113.24(14)
C(1)-C(6)	1.502(2)	O(3)-C(4)-C(3)	111.30(16)
C(2)-C(3)	1.485(3)	O(3)-C(4)-C(5)	109.01(14)
C(3)-C(4)	1.495(3)	C(3)-C(4)-C(5)	110.43(16)
C(4)-C(5)	1.516(2)	C(4)-C(5)-C(6)	111.77(13)
C(5)-C(6)	1.517(2)	O(4)-C(6)-C(1)	108.14(11)
C(9)-C(10)	1.526(2)	O(4)-C(6)-C(5)	111.27(13)
C(9)-C(12)	1.530(2)	C(1)-C(6)-C(5)	111.03(13)
C(10)-C(16)	1.507(2)	O(1)-C(7)-O(2)	122.60(17)

O(1)-C(7)-C(1)	123.82(14)	O(7)-C(13)-C(14)	108.94(18)
O(2)-C(7)-C(1)	113.54(15)	O(6)-C(13)-C(14)	111.40(16)
O(4)-C(9)-C(10)	110.37(12)	O(7)-C(13)-C(15)	108.7(2)
O(4)-C(9)-C(12)	110.35(12)	O(6)-C(13)-C(15)	108.54(19)
C(10)-C(9)-C(12)	101.90(12)	C(14)-C(13)-C(15)	112.9(2)
O(5)-C(10)-C(16)	106.97(15)	O(8)-C(16)-C(10)	107.47(18)
O(5)-C(10)-C(9)	104.52(13)	O(8)-C(16)-C(18)	103.98(17)
C(16)-C(10)-C(9)	116.47(14)	C(10)-C(16)-C(18)	113.06(17)
O(7)-C(11)-O(5)	112.15(14)	O(9)-C(18)-C(16)	104.43(17)
O(7)-C(11)-C(12)	105.92(12)	O(9)-C(19)-O(8)	104.49(15)
O(5)-C(11)-C(12)	107.43(12)	O(9)-C(19)-C(22)	109.2(2)
O(6)-C(12)-C(9)	110.07(13)	O(8)-C(19)-C(22)	108.0(2)
O(6)-C(12)-C(11)	103.90(12)	O(9)-C(19)-C(20)	111.03(18)
C(9)-C(12)-C(11)	104.60(12)	O(8)-C(19)-C(20)	110.95(19)
O(7)-C(13)-O(6)	106.05(12)	C(22)-C(19)-C(20)	112.8(2)

Symmetry transformations used to generate equivalent atoms:

Table 24. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for compound **260**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O(1)	66(1)	48(1)	50(1)	6(1)	9(1)	5(1)
O(2)	79(1)	40(1)	73(1)	9(1)	14(1)	3(1)
O(3)	92(1)	127(2)	36(1)	9(1)	15(1)	38(1)
O(4)	35(1)	39(1)	39(1)	8(1)	-4(1)	-2(1)
O(5)	43(1)	102(1)	49(1)	-5(1)	-4(1)	-19(1)
O(6)	40(1)	77(1)	44(1)	23(1)	-2(1)	-2(1)
O(7)	47(1)	74(1)	65(1)	26(1)	-4(1)	-18(1)
O(8)	92(1)	137(2)	58(1)	-43(1)	23(1)	-64(1)
O(9)	87(1)	58(1)	56(1)	3(1)	2(1)	-18(1)
C(1)	37(1)	43(1)	43(1)	9(1)	-5(1)	-4(1)
C(2)	53(1)	51(1)	57(1)	19(1)	-3(1)	-2(1)
C(3)	58(1)	79(1)	43(1)	28(1)	0(1)	-3(1)
C(4)	47(1)	75(1)	33(1)	12(1)	4(1)	8(1)
C(5)	39(1)	60(1)	40(1)	15(1)	3(1)	7(1)
C(6)	34(1)	42(1)	31(1)	9(1)	-4(1)	-2(1)
C(7)	38(1)	40(1)	55(1)	8(1)	1(1)	-3(1)

C(8)	76(1)	45(1)	89(2)	2(1)	18(1)	8(1)
C(9)	36(1)	37(1)	35(1)	4(1)	0(1)	-1(1)
C(10)	53(1)	46(1)	40(1)	-2(1)	-2(1)	-9(1)
C(11)	36(1)	42(1)	47(1)	2(1)	2(1)	3(1)
C(12)	42(1)	37(1)	36(1)	2(1)	-1(1)	-5(1)
C(13)	51(1)	50(1)	43(1)	12(1)	0(1)	-5(1)
C(14)	70(1)	117(2)	48(1)	-4(1)	12(1)	-25(1)
C(15)	119(2)	54(1)	115(2)	17(1)	2(2)	8(1)
C(16)	72(1)	65(1)	41(1)	-4(1)	-4(1)	-22(1)
C(18)	70(1)	80(1)	49(1)	-14(1)	1(1)	-27(1)
C(19)	67(1)	65(1)	42(1)	-1(1)	0(1)	-10(1)
C(20)	87(1)	63(1)	52(1)	8(1)	4(1)	-3(1)

Table 25. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^{3}) for compound **260**.

	x	y	z	U(eq)
H(3)	-121	2396	7087	127
H(8A)	817	107	1924	105
H(8B)	1289	-429	2563	105
H(8C)	1824	170	2163	105
H(14A)	2002	3074	146	118
H(14B)	2409	3746	-171	118
H(14C)	1384	3649	-179	118
H(15A)	1156	4600	1027	144
H(15B)	2173	4722	941	144
H(15C)	1740	4619	1986	144
H(20A)	1091	3436	7369	101
H(20B)	1629	4054	7687	101
H(20C)	2099	3482	7126	101
H(22A)	383	4596	5583	153
H(22B)	595	4801	6678	153
H(22C)	43	4170	6470	153
H(2A)	624(14)	591(11)	4963(17)	59(6)
H(3A)	-452(19)	1212(12)	5960(20)	78(7)
H(4A)	730(13)	2246(9)	5883(14)	43(5)
H(5A)	-124(15)	2834(11)	4781(17)	61(6)

H(6A)	189(12)	2131(9)	3341(14)	42(5)
H(9A)	648(11)	3209(7)	3465(12)	28(4)
H(10A)	1705(16)	3991(13)	3672(18)	73(7)
H(11A)	2964(12)	2803(9)	2297(14)	43(5)
H(12A)	1461(12)	2565(10)	2128(14)	43(5)
H(16A)	2367(15)	3059(12)	5124(18)	65(6)
H(18A)	3148(17)	3866(13)	5840(20)	76(7)
H(3B)	480(19)	1251(14)	6440(20)	84(8)
H(5B)	-734(14)	2200(10)	4692(15)	53(5)
H(18B)	2915(18)	4282(14)	4810(20)	79(7)

Compound 270a.

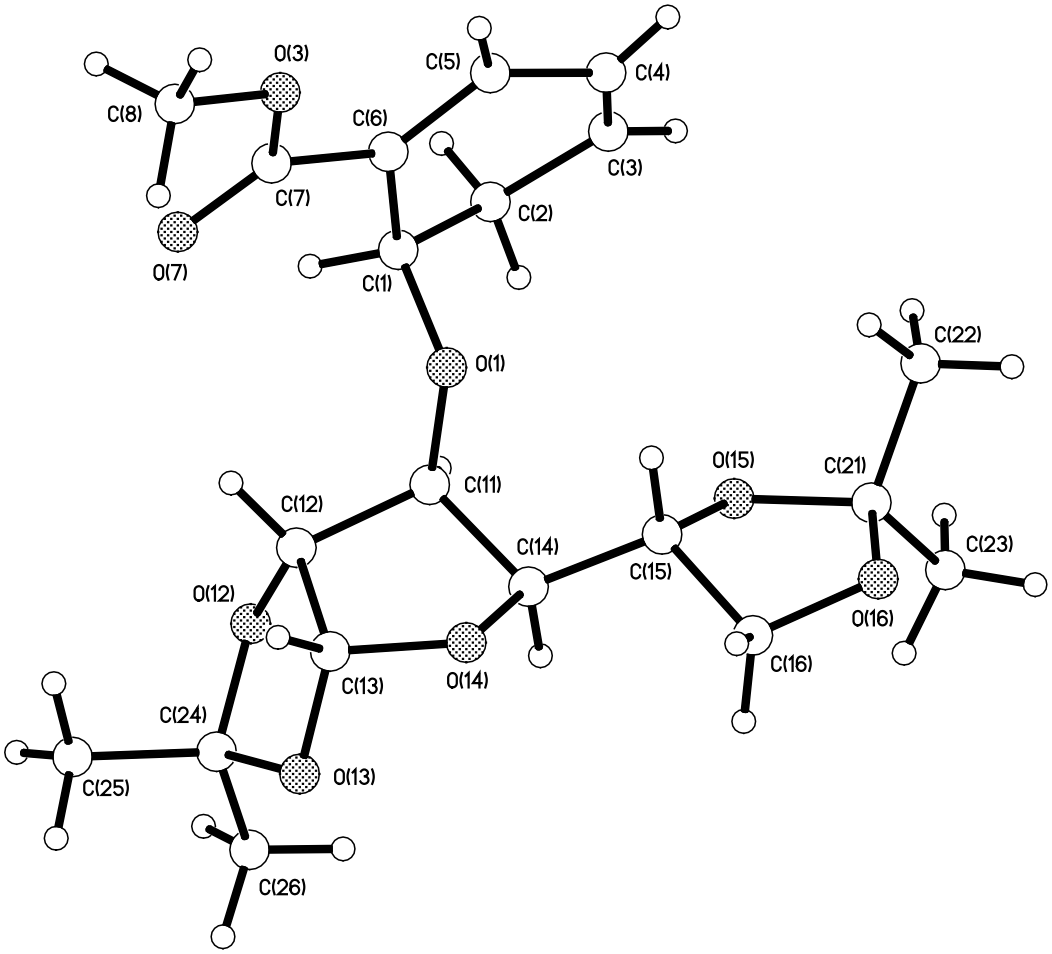


Table 26. Crystal data and structure refinement for compound **270a**.

Empirical formula	C20 H28 O8	
Formula weight	396.42	
Temperature	296(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	P2(1)2(1)2(1)	
Unit cell dimensions	a = 5.5207(4) Å	α= 90°.
	b = 10.1453(8) Å	β= 90°.
	c = 38.011(3) Å	γ = 90°.
Volume	2129.0(3) $Å^3$	
Z	4	
Density (calculated)	1.237 Mg/m^3	
Absorption coefficient	0.095 mm^{-1}	
F(000)	848	
Crystal size	0.42 x 0.06 x 0.05 mm^3	
Theta range for data collection	2.94 to 25.28°.	
Index ranges	-6<=h<=6, -12<=k<=12, -45<=l<=45	
Reflections collected	30460	
Independent reflections	3852 [R(int) = 0.1060]	
Completeness to theta = 25.28°	99.7 %	
Absorption correction	None	
Max. and min. transmission	0.9950 and 0.9607	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3852 / 0 / 270	
Goodness-of-fit on F^2	0.900	
Final R indices [I>2sigma(I)]	R1 = 0.0513, wR2 = 0.1046	
R indices (all data)	R1 = 0.1531, wR2 = 0.1264	
Absolute structure parameter	0.6(17)	
Largest diff. peak and hole	0.140 and -0.191 e.$Å^{-3}$	

Table 27. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for compound **270a**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O(1)	6540(4)	7025(2)	8801(1)	54(1)
O(3)	3859(6)	3273(3)	8884(1)	96(1)

O(7)	5512(6)	4498(3)	9304(1)	108(1)
O(12)	7906(4)	8967(2)	9570(1)	58(1)
O(13)	4320(4)	10014(2)	9564(1)	64(1)
O(14)	3453(4)	9248(2)	9006(1)	57(1)
O(15)	7358(4)	9545(2)	8246(1)	70(1)
O(16)	4174(5)	10355(5)	7938(1)	151(2)
C(1)	8299(7)	5989(4)	8818(1)	67(1)
C(2)	10400(8)	6234(5)	8567(1)	96(2)
C(3)	9859(12)	5922(7)	8197(2)	124(2)
C(4)	8161(15)	5063(7)	8119(2)	140(3)
C(5)	6786(10)	4388(5)	8386(1)	100(2)
C(6)	6894(8)	4771(4)	8721(1)	67(1)
C(7)	5401(8)	4180(5)	9000(1)	76(1)
C(8)	2210(9)	2700(4)	9134(1)	110(2)
C(11)	7234(6)	8208(3)	8976(1)	49(1)
C(12)	6390(6)	8157(3)	9359(1)	50(1)
C(13)	3957(6)	8888(4)	9355(1)	51(1)
C(14)	5738(6)	9329(3)	8823(1)	50(1)
C(15)	5201(6)	9254(4)	8433(1)	62(1)
C(16)	3494(7)	10300(5)	8296(1)	90(2)
C(21)	6609(8)	9994(5)	7904(1)	86(1)
C(22)	6861(13)	8881(6)	7639(1)	154(3)
C(23)	8096(9)	11180(5)	7813(1)	108(2)
C(24)	6446(6)	9829(3)	9771(1)	52(1)
C(25)	5764(7)	9206(4)	10121(1)	82(1)
C(26)	7754(9)	11123(4)	9811(1)	85(1)

Table 28. Bond lengths [Å] and angles [°] for compound **270a**.

O(1)-C(11)	1.423(4)	O(14)-C(13)	1.405(3)
O(1)-C(1)	1.432(4)	O(14)-C(14)	1.443(4)
O(3)-C(7)	1.328(5)	O(15)-C(15)	1.419(4)
O(3)-C(8)	1.437(5)	O(15)-C(21)	1.436(4)
O(7)-C(7)	1.202(4)	O(16)-C(21)	1.399(5)
O(12)-C(24)	1.413(4)	O(16)-C(16)	1.415(4)
O(12)-C(12)	1.422(4)	C(1)-C(6)	1.505(5)
O(13)-C(13)	1.407(4)	C(1)-C(2)	1.521(5)
O(13)-C(24)	1.425(4)	C(2)-C(3)	1.474(6)

C(3)-C(4)	1.314(8)	O(7)-C(7)-C(6)	123.8(5)
C(4)-C(5)	1.440(7)	O(3)-C(7)-C(6)	113.8(4)
C(5)-C(6)	1.333(5)	O(1)-C(11)-C(14)	107.8(3)
C(6)-C(7)	1.472(5)	O(1)-C(11)-C(12)	109.5(3)
C(11)-C(14)	1.522(4)	C(14)-C(11)-C(12)	102.9(3)
C(11)-C(12)	1.529(4)	O(12)-C(12)-C(11)	109.9(3)
C(12)-C(13)	1.534(5)	O(12)-C(12)-C(13)	104.0(3)
C(14)-C(15)	1.512(4)	C(11)-C(12)-C(13)	104.0(3)
C(15)-C(16)	1.512(5)	O(14)-C(13)-O(13)	110.6(3)
C(21)-C(23)	1.497(6)	O(14)-C(13)-C(12)	107.9(2)
C(21)-C(22)	1.520(6)	O(13)-C(13)-C(12)	105.2(3)
C(24)-C(26)	1.506(5)	O(14)-C(14)-C(15)	107.4(3)
C(24)-C(25)	1.520(4)	O(14)-C(14)-C(11)	104.3(2)
C(11)-O(1)-C(1)	114.6(3)	C(15)-C(14)-C(11)	116.3(3)
C(7)-O(3)-C(8)	117.9(4)	O(15)-C(15)-C(16)	101.8(3)
C(24)-O(12)-C(12)	109.1(2)	O(15)-C(15)-C(14)	108.5(3)
C(13)-O(13)-C(24)	108.8(3)	C(16)-C(15)-C(14)	115.1(3)
C(13)-O(14)-C(14)	107.3(2)	O(16)-C(16)-C(15)	101.2(3)
C(15)-O(15)-C(21)	106.2(3)	O(16)-C(21)-O(15)	106.1(3)
C(21)-O(16)-C(16)	109.4(3)	O(16)-C(21)-C(23)	109.7(4)
O(1)-C(1)-C(6)	104.0(3)	O(15)-C(21)-C(23)	107.9(4)
O(1)-C(1)-C(2)	111.7(3)	O(16)-C(21)-C(22)	110.0(5)
C(6)-C(1)-C(2)	111.9(3)	O(15)-C(21)-C(22)	109.7(4)
C(3)-C(2)-C(1)	114.1(4)	C(23)-C(21)-C(22)	113.1(4)
C(4)-C(3)-C(2)	120.2(5)	O(12)-C(24)-O(13)	104.7(2)
C(3)-C(4)-C(5)	122.2(6)	O(12)-C(24)-C(26)	108.6(3)
C(6)-C(5)-C(4)	120.7(5)	O(13)-C(24)-C(26)	109.5(3)
C(5)-C(6)-C(7)	123.0(5)	O(12)-C(24)-C(25)	110.9(3)
C(5)-C(6)-C(1)	119.8(4)	O(13)-C(24)-C(25)	109.5(3)
C(7)-C(6)-C(1)	116.5(4)	C(26)-C(24)-C(25)	113.2(3)
O(7)-C(7)-O(3)	122.4(4)		

Symmetry transformations used to generate equivalent atoms:

Table 29. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for compound **270a**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O(1)	44(1)	62(2)	57(1)	-8(1)	-4(1)	1(1)
O(3)	113(3)	73(2)	100(2)	-1(2)	25(2)	-12(2)
O(7)	133(3)	124(3)	68(2)	1(2)	14(2)	-18(2)
O(12)	41(2)	73(2)	60(1)	-13(1)	-9(1)	3(1)
O(13)	57(2)	71(2)	64(2)	-6(1)	-8(1)	16(1)
O(14)	39(1)	80(2)	53(1)	13(1)	-1(1)	1(1)
O(15)	46(2)	114(2)	51(1)	25(1)	1(1)	2(1)
O(16)	53(2)	308(6)	91(2)	103(3)	10(2)	34(3)
C(1)	56(3)	74(3)	71(2)	-2(2)	-6(2)	5(3)
C(2)	60(3)	99(4)	128(4)	-23(3)	30(3)	1(3)
C(3)	135(5)	136(5)	101(5)	-44(4)	56(4)	-44(4)
C(4)	193(7)	143(5)	83(4)	-45(4)	64(5)	-43(5)
C(5)	124(5)	91(4)	84(3)	-30(3)	40(3)	-25(4)
C(6)	75(3)	64(3)	61(3)	-3(2)	13(2)	12(2)
C(7)	91(3)	63(3)	73(3)	1(3)	14(3)	11(3)
C(8)	112(4)	75(3)	143(4)	30(3)	33(4)	-11(3)
C(11)	33(2)	63(2)	50(2)	-3(2)	-5(2)	-4(2)
C(12)	41(2)	54(2)	55(2)	10(2)	-5(2)	2(2)
C(13)	44(2)	63(2)	46(2)	11(2)	5(2)	-3(2)
C(14)	41(2)	63(2)	47(2)	6(2)	-4(2)	-7(2)
C(15)	42(2)	93(3)	52(2)	14(2)	-6(2)	-13(2)
C(16)	48(2)	158(4)	65(3)	47(3)	-1(2)	5(3)
C(21)	61(3)	136(4)	61(3)	35(3)	-4(2)	6(3)
C(22)	198(7)	197(6)	69(3)	-13(4)	-28(4)	-11(6)
C(23)	87(4)	144(4)	94(3)	52(3)	13(3)	17(4)
C(24)	44(2)	59(2)	53(2)	-3(2)	-4(2)	0(2)
C(25)	96(3)	93(3)	58(2)	11(2)	7(2)	-8(3)
C(26)	102(4)	68(3)	86(3)	-9(2)	0(3)	-16(3)

Table 30. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for compound **270a**.

	x	y	z	U(eq)
H(1)	8910	5907	9059	81
H(2A)	10869	7153	8583	115
H(2B)	11771	5707	8642	115
H(8A)	3095	2155	9296	165
H(8B)	1034	2175	9012	165
H(8C)	1401	3388	9261	165
H(11)	8978	8374	8957	58
H(12)	6254	7255	9449	60
H(13)	2662	8334	9451	61
H(14)	6519	10173	8876	60
H(15)	4609	8374	8371	75
H(16A)	1814	10039	8323	109
H(16B)	3752	11139	8413	109
H(22A)	5887	8147	7712	232
H(22B)	8527	8615	7625	232
H(22C)	6328	9180	7412	232
H(23A)	7484	11570	7600	163
H(23B)	9752	10921	7778	163
H(23C)	8006	11810	8001	163
H(25A)	5178	8327	10082	123
H(25B)	4520	9722	10232	123
H(25C)	7162	9176	10271	123
H(26A)	9305	10975	9918	128
H(26B)	6815	11704	9956	128
H(26C)	7978	11515	9583	128
H(4)	7980(90)	4740(50)	7906(11)	130(19)
H(3)	11360(100)	6430(50)	8050(13)	160(20)
H(5)	5560(80)	3710(40)	8350(10)	91(15)

Compound 280a.

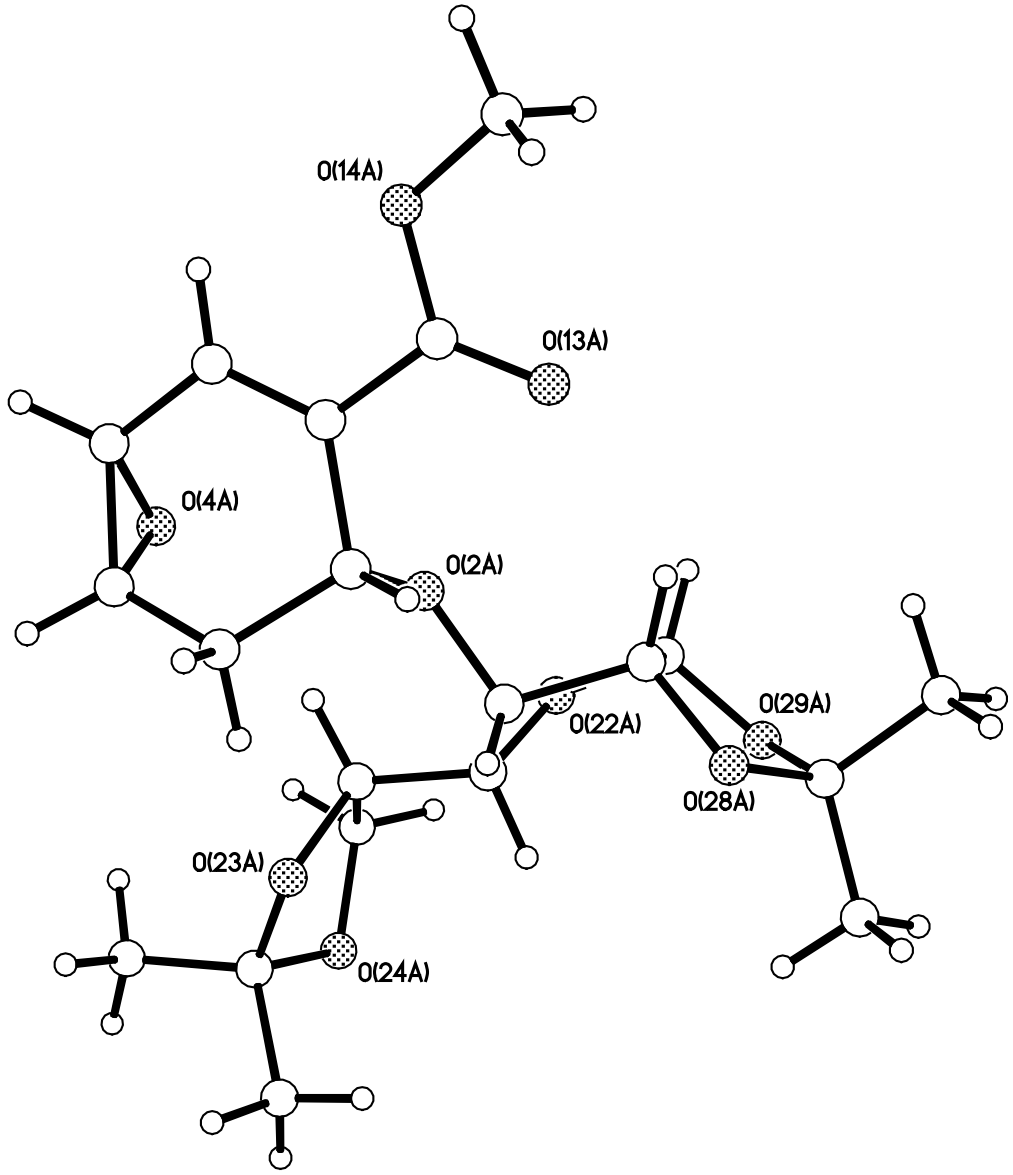


Table 31. Crystal data and structure refinement for compound **280a**.

Empirical formula	C23 H35 O9	
Formula weight	455.51	
Temperature	173(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)	
Unit cell dimensions	a = 13.0190(3) Å	α= 90°.
	b = 8.5252(2) Å	β= 101.8070(10)°.
	c = 21.5504(6) Å	γ = 90°.
Volume	2341.26(10) $Å^3$	
Z	4	

Density (calculated)	1.292 Mg/m^3
Absorption coefficient	0.099 mm^{-1}
F(000)	980
Crystal size	0.55 x 0.50 x 0.36 mm^3
Theta range for data collection	0.97 to 29.00°.
Index ranges	-17<=h<=17, -11<=k<=11, -29<=l<=29
Reflections collected	109082
Independent reflections	12258 [R(int) = 0.0555]
Completeness to theta = 29.00°	99.9 %
Absorption correction	None
Max. and min. transmission	0.9654 and 0.9478
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	12258 / 1 / 585
Goodness-of-fit on F^2	1.100
Final R indices [I>2sigma(I)]	R1 = 0.0531, wR2 = 0.1477
R indices (all data)	R1 = 0.0625, wR2 = 0.1536
Absolute structure parameter	-0.3(5)
Largest diff. peak and hole	0.693 and -0.257 $e.Å^{-3}$

Table 32. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for compound **280a**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O(2A)	6854(1)	6498(1)	500(1)	24(1)
O(4A)	5025(1)	8057(2)	898(1)	36(1)
O(13A)	7691(1)	8418(2)	-599(1)	50(1)
O(14A)	6385(1)	10112(2)	-961(1)	41(1)
O(22A)	6735(1)	3137(2)	610(1)	29(1)
O(23A)	6980(1)	5329(2)	2082(1)	40(1)
O(24A)	6546(2)	2913(2)	2354(1)	56(1)
O(28A)	9090(1)	4002(2)	477(1)	34(1)
O(29A)	8081(1)	1828(2)	270(1)	39(1)
C(1A)	6469(1)	9039(2)	50(1)	25(1)
C(2A)	7109(1)	8132(2)	592(1)	25(1)
C(3A)	6935(2)	8725(2)	1233(1)	32(1)
C(4A)	5807(2)	9075(2)	1244(1)	34(1)
C(5A)	5127(2)	9641(2)	663(1)	32(1)
C(6A)	5577(1)	9763(2)	89(1)	29(1)

C(13A)	6924(2)	9129(2)	-526(1)	32(1)
C(14A)	6816(2)	10377(3)	-1512(1)	56(1)
C(21A)	7684(1)	5470(2)	775(1)	24(1)
C(22A)	7249(1)	4128(2)	1119(1)	27(1)
C(23A)	6448(2)	4580(2)	1509(1)	29(1)
C(24A)	5938(2)	3149(3)	1747(1)	42(1)
C(25A)	6822(2)	4452(3)	2605(1)	45(1)
C(26A)	7863(3)	4313(6)	3067(2)	100(2)
C(27A)	5984(3)	5075(5)	2915(2)	83(1)
C(28A)	8060(1)	4580(2)	246(1)	28(1)
C(29A)	7362(1)	3088(2)	152(1)	29(1)
C(30A)	9114(2)	2397(2)	289(1)	32(1)
C(31A)	9872(2)	1536(3)	793(1)	48(1)
C(32A)	9406(2)	2263(3)	-353(1)	47(1)
O(2B)	3107(1)	909(1)	4590(1)	23(1)
O(4B)	4826(1)	2661(2)	4149(1)	35(1)
O(13B)	2336(1)	2693(2)	5781(1)	50(1)
O(14B)	3634(1)	4379(2)	6138(1)	50(1)
O(22B)	3223(1)	-2455(1)	4405(1)	27(1)
O(23B)	2746(1)	-104(2)	2972(1)	28(1)
O(24B)	3964(1)	-1582(2)	2607(1)	42(1)
O(28B)	943(1)	-1690(2)	4669(1)	31(1)
O(29B)	1982(1)	-3843(2)	4817(1)	36(1)
C(1B)	3494(1)	3400(2)	5105(1)	25(1)
C(2B)	2819(1)	2550(2)	4559(1)	22(1)
C(3B)	2899(1)	3256(2)	3920(1)	27(1)
C(4B)	3994(2)	3703(2)	3867(1)	31(1)
C(5B)	4737(2)	4192(2)	4441(1)	33(1)
C(6B)	4361(1)	4159(2)	5045(1)	30(1)
C(13B)	3085(2)	3421(2)	5699(1)	32(1)
C(14B)	3236(3)	4584(4)	6710(1)	67(1)
C(21B)	2269(1)	-121(2)	4336(1)	22(1)
C(22B)	2642(1)	-1408(2)	3937(1)	23(1)
C(23B)	3361(1)	-897(2)	3510(1)	24(1)
C(24B)	3835(2)	-2228(2)	3193(1)	32(1)
C(25B)	3339(2)	-198(2)	2482(1)	34(1)
C(26B)	4064(2)	1200(3)	2521(1)	55(1)
C(27B)	2591(2)	-354(4)	1848(1)	60(1)
C(28B)	1972(1)	-1094(2)	4869(1)	25(1)
C(29B)	2685(1)	-2557(2)	4909(1)	26(1)

C(30B)	945(1)	-3308(2)	4832(1)	27(1)
C(31B)	162(2)	-4161(3)	4336(1)	43(1)
C(32B)	730(2)	-3540(3)	5494(1)	43(1)
C(1S)	-807(5)	10446(8)	2609(3)	123(2)
C(2S)	-172(10)	9142(17)	2313(6)	118(3)
C(3S)	-256(4)	7762(9)	2351(4)	154(3)
C(4S)	448(5)	6502(8)	2416(3)	129(2)
C(5S)	409(9)	4726(16)	2450(6)	115(3)
C(6S)	1145(4)	3848(8)	2309(2)	107(2)
C(2S2)	-233(9)	9187(15)	2800(5)	110(3)
C(5S2)	409(7)	5200(12)	2049(4)	89(2)

Table 33. Bond lengths [Å] and angles [°]compound **280a**.

O(2A)-C(21A)	1.423(2)	C(21A)-C(28A)	1.531(2)
O(2A)-C(2A)	1.4358(19)	C(21A)-C(22A)	1.533(2)
O(4A)-C(4A)	1.426(2)	C(22A)-C(23A)	1.518(3)
O(4A)-C(5A)	1.458(2)	C(23A)-C(24A)	1.527(3)
O(13A)-C(13A)	1.205(3)	C(25A)-C(27A)	1.488(4)
O(14A)-C(13A)	1.343(2)	C(25A)-C(26A)	1.513(4)
O(14A)-C(14A)	1.432(3)	C(28A)-C(29A)	1.552(2)
O(22A)-C(29A)	1.404(2)	C(30A)-C(31A)	1.502(3)
O(22A)-C(22A)	1.437(2)	C(30A)-C(32A)	1.511(3)
O(23A)-C(25A)	1.401(2)	O(2B)-C(21B)	1.420(2)
O(23A)-C(23A)	1.436(2)	O(2B)-C(2B)	1.4462(18)
O(24A)-C(24A)	1.398(3)	O(4B)-C(4B)	1.436(2)
O(24A)-C(25A)	1.437(3)	O(4B)-C(5B)	1.463(2)
O(28A)-C(28A)	1.420(2)	O(13B)-C(13B)	1.198(3)
O(28A)-C(30A)	1.429(2)	O(14B)-C(13B)	1.340(2)
O(29A)-C(29A)	1.414(2)	O(14B)-C(14B)	1.441(3)
O(29A)-C(30A)	1.423(2)	O(22B)-C(29B)	1.410(2)
C(1A)-C(6A)	1.333(3)	O(22B)-C(22B)	1.440(2)
C(1A)-C(13A)	1.483(2)	O(23B)-C(25B)	1.432(2)
C(1A)-C(2A)	1.502(2)	O(23B)-C(23B)	1.435(2)
C(2A)-C(3A)	1.531(3)	O(24B)-C(24B)	1.419(2)
C(3A)-C(4A)	1.503(3)	O(24B)-C(25B)	1.427(2)
C(4A)-C(5A)	1.460(3)	O(28B)-C(28B)	1.416(2)
C(5A)-C(6A)	1.475(3)	O(28B)-C(30B)	1.423(2)

O(29B)-C(29B)	1.417(2)	O(2A)-C(2A)-C(1A)	108.44(13)
O(29B)-C(30B)	1.432(2)	O(2A)-C(2A)-C(3A)	111.86(14)
C(1B)-C(6B)	1.331(3)	C(1A)-C(2A)-C(3A)	111.76(15)
C(1B)-C(13B)	1.485(3)	C(4A)-C(3A)-C(2A)	113.84(15)
C(1B)-C(2B)	1.503(2)	O(4A)-C(4A)-C(5A)	60.69(12)
C(2B)-C(3B)	1.525(2)	O(4A)-C(4A)-C(3A)	117.82(16)
C(3B)-C(4B)	1.503(3)	C(5A)-C(4A)-C(3A)	118.43(16)
C(4B)-C(5B)	1.466(3)	O(4A)-C(5A)-C(4A)	58.51(12)
C(5B)-C(6B)	1.482(3)	O(4A)-C(5A)-C(6A)	115.90(15)
C(21B)-C(28B)	1.530(2)	C(4A)-C(5A)-C(6A)	117.80(17)
C(21B)-C(22B)	1.533(2)	C(1A)-C(6A)-C(5A)	121.39(16)
C(22B)-C(23B)	1.506(2)	O(13A)-C(13A)-O(14A)	123.42(18)
C(23B)-C(24B)	1.520(2)	O(13A)-C(13A)-C(1A)	124.56(17)
C(25B)-C(27B)	1.513(3)	O(14A)-C(13A)-C(1A)	112.01(16)
C(25B)-C(26B)	1.512(3)	O(2A)-C(21A)-C(28A)	109.02(14)
C(28B)-C(29B)	1.546(2)	O(2A)-C(21A)-C(22A)	109.75(14)
C(30B)-C(31B)	1.504(3)	C(28A)-C(21A)-C(22A)	101.28(14)
C(30B)-C(32B)	1.521(3)	O(22A)-C(22A)-C(23A)	107.87(14)
C(1S)-C(2S2)	1.324(13)	O(22A)-C(22A)-C(21A)	103.34(13)
C(1S)-C(2S)	1.595(15)	C(23A)-C(22A)-C(21A)	116.20(15)
C(2S)-C(2S2)	1.069(14)	O(23A)-C(23A)-C(22A)	108.97(15)
C(2S)-C(3S)	1.186(15)	O(23A)-C(23A)-C(24A)	103.52(14)
C(3S)-C(4S)	1.400(10)	C(22A)-C(23A)-C(24A)	112.24(16)
C(3S)-C(2S2)	1.549(14)	O(24A)-C(24A)-C(23A)	103.07(17)
C(4S)-C(5S2)	1.357(12)	O(23A)-C(25A)-O(24A)	104.53(16)
C(4S)-C(5S)	1.518(15)	O(23A)-C(25A)-C(27A)	114.3(3)
C(5S)-C(5S2)	0.953(13)	O(24A)-C(25A)-C(27A)	110.2(2)
C(5S)-C(6S)	1.300(13)	O(23A)-C(25A)-C(26A)	108.2(2)
C(6S)-C(5S2)	1.530(11)	O(24A)-C(25A)-C(26A)	107.3(3)
C(21A)-O(2A)-C(2A)	113.96(12)	C(27A)-C(25A)-C(26A)	111.9(3)
C(4A)-O(4A)-C(5A)	60.80(12)	O(28A)-C(28A)-C(21A)	109.49(15)
C(13A)-O(14A)-C(14A)	116.11(19)	O(28A)-C(28A)-C(29A)	104.33(13)
C(29A)-O(22A)-C(22A)	107.85(13)	C(21A)-C(28A)-C(29A)	103.63(14)
C(25A)-O(23A)-C(23A)	109.25(15)	O(22A)-C(29A)-O(29A)	110.75(15)
C(24A)-O(24A)-C(25A)	105.77(17)	O(22A)-C(29A)-C(28A)	107.04(14)
C(28A)-O(28A)-C(30A)	107.88(14)	O(29A)-C(29A)-C(28A)	104.50(14)
C(29A)-O(29A)-C(30A)	109.49(14)	O(29A)-C(30A)-O(28A)	105.00(15)
C(6A)-C(1A)-C(13A)	122.13(16)	O(29A)-C(30A)-C(31A)	109.34(18)
C(6A)-C(1A)-C(2A)	122.73(16)	O(28A)-C(30A)-C(31A)	108.18(17)
C(13A)-C(1A)-C(2A)	115.10(15)	O(29A)-C(30A)-C(32A)	111.16(18)

O(28A)-C(30A)-C(32A)	110.88(17)	O(24B)-C(25B)-C(27B)	109.49(19)
C(31A)-C(30A)-C(32A)	112.01(18)	O(23B)-C(25B)-C(27B)	109.10(18)
C(21B)-O(2B)-C(2B)	114.07(12)	O(24B)-C(25B)-C(26B)	108.38(18)
C(4B)-O(4B)-C(5B)	60.74(12)	O(23B)-C(25B)-C(26B)	109.91(17)
C(13B)-O(14B)-C(14B)	116.4(2)	C(27B)-C(25B)-C(26B)	113.4(2)
C(29B)-O(22B)-C(22B)	107.71(12)	O(28B)-C(28B)-C(21B)	109.51(14)
C(25B)-O(23B)-C(23B)	105.88(13)	O(28B)-C(28B)-C(29B)	104.31(13)
C(24B)-O(24B)-C(25B)	108.72(14)	C(21B)-C(28B)-C(29B)	103.79(13)
C(28B)-O(28B)-C(30B)	108.61(13)	O(22B)-C(29B)-O(29B)	110.42(14)
C(29B)-O(29B)-C(30B)	109.64(13)	O(22B)-C(29B)-C(28B)	107.15(13)
C(6B)-C(1B)-C(13B)	122.59(16)	O(29B)-C(29B)-C(28B)	104.65(13)
C(6B)-C(1B)-C(2B)	122.03(16)	O(28B)-C(30B)-O(29B)	104.88(13)
C(13B)-C(1B)-C(2B)	115.25(15)	O(28B)-C(30B)-C(31B)	109.00(16)
O(2B)-C(2B)-C(1B)	108.98(13)	O(29B)-C(30B)-C(31B)	109.56(16)
O(2B)-C(2B)-C(3B)	111.05(13)	O(28B)-C(30B)-C(32B)	111.51(15)
C(1B)-C(2B)-C(3B)	112.29(14)	O(29B)-C(30B)-C(32B)	109.56(17)
C(4B)-C(3B)-C(2B)	114.24(15)	C(31B)-C(30B)-C(32B)	112.08(16)
O(4B)-C(4B)-C(5B)	60.55(12)	C(2S2)-C(1S)-C(2S)	41.7(6)
O(4B)-C(4B)-C(3B)	117.06(15)	C(2S2)-C(2S)-C(3S)	86.6(13)
C(5B)-C(4B)-C(3B)	118.64(16)	C(2S2)-C(2S)-C(1S)	55.5(9)
O(4B)-C(5B)-C(4B)	58.71(12)	C(3S)-C(2S)-C(1S)	126.9(11)
O(4B)-C(5B)-C(6B)	115.45(15)	C(2S)-C(3S)-C(4S)	134.4(9)
C(4B)-C(5B)-C(6B)	117.20(16)	C(2S)-C(3S)-C(2S2)	43.5(7)
C(1B)-C(6B)-C(5B)	122.25(16)	C(4S)-C(3S)-C(2S2)	127.6(8)
O(13B)-C(13B)-O(14B)	122.97(18)	C(5S2)-C(4S)-C(3S)	128.6(7)
O(13B)-C(13B)-C(1B)	124.45(17)	C(5S2)-C(4S)-C(5S)	38.2(5)
O(14B)-C(13B)-C(1B)	112.57(17)	C(3S)-C(4S)-C(5S)	138.0(7)
O(2B)-C(21B)-C(28B)	109.73(13)	C(5S2)-C(5S)-C(6S)	84.0(11)
O(2B)-C(21B)-C(22B)	110.59(13)	C(5S2)-C(5S)-C(4S)	61.7(10)
C(28B)-C(21B)-C(22B)	100.97(13)	C(6S)-C(5S)-C(4S)	121.9(10)
O(22B)-C(22B)-C(23B)	107.71(13)	C(5S)-C(6S)-C(5S2)	38.3(6)
O(22B)-C(22B)-C(21B)	103.49(13)	C(2S)-C(2S2)-C(1S)	82.9(11)
C(23B)-C(22B)-C(21B)	116.32(14)	C(2S)-C(2S2)-C(3S)	49.8(10)
O(23B)-C(23B)-C(22B)	108.44(14)	C(1S)-C(2S2)-C(3S)	120.3(9)
O(23B)-C(23B)-C(24B)	101.48(13)	C(5S)-C(5S2)-C(4S)	80.1(11)
C(22B)-C(23B)-C(24B)	114.80(14)	C(5S)-C(5S2)-C(6S)	57.7(10)
O(24B)-C(24B)-C(23B)	104.10(14)	C(4S)-C(5S2)-C(6S)	117.2(7)
O(24B)-C(25B)-O(23B)	106.28(14)		

Symmetry transformations used to generate equivalent atoms:

Table 34. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for compound **280a**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O(2A)	25(1)	14(1)	32(1)	2(1)	1(1)	-1(1)
O(4A)	38(1)	27(1)	45(1)	-1(1)	13(1)	-7(1)
O(13A)	57(1)	41(1)	60(1)	12(1)	32(1)	14(1)
O(14A)	52(1)	35(1)	35(1)	11(1)	8(1)	2(1)
O(22A)	28(1)	25(1)	36(1)	-8(1)	9(1)	-7(1)
O(23A)	63(1)	32(1)	27(1)	-2(1)	10(1)	-18(1)
O(24A)	76(1)	39(1)	56(1)	20(1)	19(1)	-2(1)
O(28A)	22(1)	22(1)	58(1)	-1(1)	8(1)	-1(1)
O(29A)	28(1)	21(1)	69(1)	-4(1)	13(1)	0(1)
C(1A)	28(1)	16(1)	31(1)	3(1)	2(1)	-2(1)
C(2A)	24(1)	15(1)	35(1)	3(1)	3(1)	-1(1)
C(3A)	38(1)	22(1)	32(1)	-1(1)	-2(1)	-3(1)
C(4A)	48(1)	23(1)	34(1)	-3(1)	13(1)	-4(1)
C(5A)	30(1)	22(1)	44(1)	-3(1)	10(1)	-2(1)
C(6A)	28(1)	23(1)	32(1)	2(1)	1(1)	0(1)
C(13A)	39(1)	21(1)	38(1)	2(1)	10(1)	-3(1)
C(14A)	79(2)	52(1)	39(1)	12(1)	17(1)	-14(1)
C(21A)	24(1)	18(1)	30(1)	3(1)	3(1)	0(1)
C(22A)	29(1)	20(1)	29(1)	2(1)	3(1)	-2(1)
C(23A)	37(1)	24(1)	27(1)	-1(1)	7(1)	-8(1)
C(24A)	59(1)	28(1)	43(1)	-2(1)	23(1)	-16(1)
C(25A)	55(1)	52(1)	29(1)	6(1)	11(1)	-3(1)
C(26A)	88(3)	124(4)	70(2)	35(2)	-27(2)	-1(3)
C(27A)	86(2)	86(2)	92(2)	-37(2)	51(2)	-23(2)
C(28A)	28(1)	22(1)	36(1)	4(1)	8(1)	2(1)
C(29A)	28(1)	24(1)	36(1)	-4(1)	8(1)	2(1)
C(30A)	27(1)	24(1)	45(1)	2(1)	10(1)	3(1)
C(31A)	48(1)	37(1)	58(1)	8(1)	5(1)	12(1)
C(32A)	53(1)	39(1)	54(1)	4(1)	24(1)	9(1)
O(2B)	22(1)	14(1)	33(1)	-2(1)	2(1)	-1(1)
O(4B)	31(1)	23(1)	51(1)	-3(1)	11(1)	4(1)
O(13B)	58(1)	52(1)	43(1)	-11(1)	20(1)	-15(1)
O(14B)	49(1)	57(1)	43(1)	-27(1)	7(1)	-5(1)
O(22B)	28(1)	19(1)	37(1)	3(1)	12(1)	4(1)

O(23B)	31(1)	24(1)	31(1)	0(1)	9(1)	4(1)
O(24B)	61(1)	33(1)	39(1)	0(1)	25(1)	12(1)
O(28B)	24(1)	19(1)	52(1)	4(1)	15(1)	0(1)
O(29B)	25(1)	18(1)	68(1)	4(1)	16(1)	0(1)
C(1B)	23(1)	18(1)	33(1)	-5(1)	1(1)	4(1)
C(2B)	20(1)	14(1)	33(1)	-5(1)	4(1)	3(1)
C(3B)	32(1)	14(1)	33(1)	0(1)	3(1)	3(1)
C(4B)	37(1)	17(1)	41(1)	0(1)	12(1)	0(1)
C(5B)	28(1)	22(1)	52(1)	-2(1)	13(1)	-1(1)
C(6B)	26(1)	21(1)	41(1)	-8(1)	1(1)	3(1)
C(13B)	35(1)	26(1)	33(1)	-6(1)	1(1)	4(1)
C(14B)	73(2)	79(2)	48(1)	-35(1)	12(1)	3(2)
C(21B)	22(1)	15(1)	30(1)	-3(1)	6(1)	-2(1)
C(22B)	24(1)	17(1)	29(1)	-3(1)	7(1)	1(1)
C(23B)	27(1)	21(1)	26(1)	-2(1)	8(1)	0(1)
C(24B)	38(1)	22(1)	39(1)	0(1)	16(1)	5(1)
C(25B)	44(1)	30(1)	30(1)	0(1)	13(1)	4(1)
C(26B)	69(2)	34(1)	74(2)	-1(1)	43(1)	-7(1)
C(27B)	76(2)	70(2)	32(1)	-2(1)	3(1)	13(2)
C(28B)	25(1)	19(1)	31(1)	-3(1)	10(1)	-2(1)
C(29B)	24(1)	21(1)	34(1)	3(1)	9(1)	0(1)
C(30B)	26(1)	17(1)	40(1)	-1(1)	13(1)	-2(1)
C(31B)	44(1)	36(1)	47(1)	-1(1)	6(1)	-13(1)
C(32B)	58(1)	34(1)	43(1)	-1(1)	24(1)	-13(1)
C(1S)	110(4)	125(5)	142(4)	7(4)	46(3)	-3(4)
C(3S)	92(3)	121(5)	244(8)	-60(5)	25(4)	-2(4)
C(4S)	119(4)	101(4)	161(5)	38(4)	11(4)	-24(3)
C(6S)	101(3)	122(4)	92(3)	10(3)	3(2)	-8(3)

Table 35. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for compound **280a**.

	x	y	z	U(eq)
H(2A)	7867	8271	580	30
H(3A1)	7197	7925	1560	39
H(3A2)	7354	9690	1347	39
H(4A)	5666	9507	1650	41
H(5A)	4571	10416	709	38
H(6A)	5223	10370	-259	34
H(14A)	7547	10718	-1383	83
H(14B)	6408	11191	-1775	83
H(14C)	6786	9403	-1757	83
H(21A)	8269	6037	1060	29
H(22A)	7842	3544	1390	32
H(23A)	5903	5291	1262	35
H(24A)	5196	3362	1764	50
H(24B)	5968	2227	1472	50
H(26D)	8417	4077	2834	150
H(26E)	7823	3467	3369	150
H(26F)	8022	5304	3296	150
H(27D)	6154	6155	3055	125
H(27E)	5935	4422	3281	125
H(27F)	5311	5060	2611	125
H(28A)	8000	5212	-151	34
H(29A)	6924	3037	-287	35
H(31A)	10578	1968	824	72
H(31B)	9873	420	684	72
H(31C)	9659	1658	1201	72
H(32A)	8939	2928	-658	70
H(32B)	9335	1170	-495	70
H(32C)	10134	2606	-321	70
H(2B)	2072	2632	4606	27
H(3B1)	2621	2489	3582	32
H(3B2)	2449	4201	3845	32
H(4B)	4070	4223	3462	37
H(5B)	5266	5009	4393	40
H(6B)	4753	4698	5402	36
H(14D)	2475	4418	6617	101

H(14E)	3391	5651	6872	101
H(14F)	3571	3824	7029	101
H(21B)	1651	461	4089	27
H(22B)	2022	-1969	3683	27
H(23B)	3924	-190	3742	29
H(24C)	4519	-2556	3453	38
H(24D)	3359	-3145	3124	38
H(26A)	4538	1220	2937	82
H(26B)	3648	2166	2461	82
H(26C)	4476	1119	2189	82
H(27A)	2993	-438	1511	90
H(27B)	2136	571	1773	90
H(27C)	2161	-1297	1849	90
H(28B)	2068	-509	5279	30
H(29B)	3184	-2621	5328	31
H(31D)	-535	-3697	4308	64
H(31E)	143	-5270	4452	64
H(31F)	368	-4070	3925	64
H(32D)	1242	-2941	5800	65
H(32E)	790	-4656	5604	65
H(32F)	20	-3173	5504	65