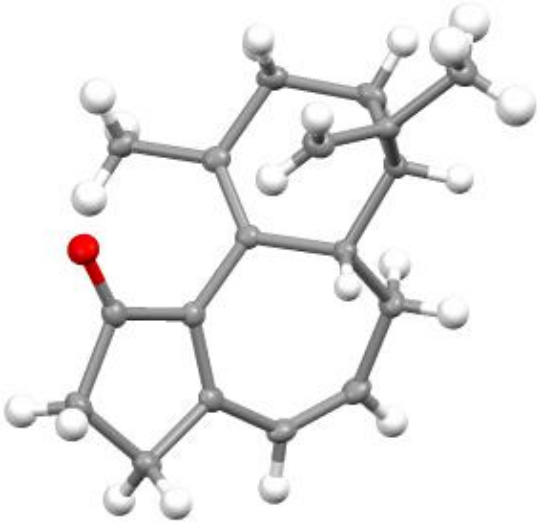
APPENDIX 3

X-Ray Crystallography Data

Relevant to Chapter 1

A3.1 CRYSTAL STRUCTURE ANALYSIS OF 73



73

Low-temperature diffraction data (f- and w-scans) were collected on a Bruker AXS D8 VENTURE KAPPA diffractometer coupled to a PHOTON II CPAD detector with Cu K_a radiation (l = 1.54178 Å) from an IμS micro-source for the structure of compound **73**. The structure was solved by direct methods using SHELX[1] and refined against F^2 on all data by full-matrix least squares with SHELXL-2017[2] using established refinement techniques.[3] All non-hydrogen atoms were refined anisotropically. All hydrogen atoms were included into the model at geometrically calculated positions and refined using a riding model. The isotropic displacement parameters of all hydrogen atoms were fixed to 1.2 times the *U* value of the atoms they are linked to (1.5 times for methyl groups). Compound **73** crystallizes in the orthorhombic space group $P2_12_12_1$ with one molecule in the asymmetric unit.

*Table A3.1.1. Crystal data and structure refinement for **73**.*

Identification code	**73**	
Empirical formula	C18 H22 O	
Formula weight	254.35	
Temperature	100(2) K	
Wavelength	1.54178 Å	
Crystal system	Orthorhombic	
Space group	$P2_12_12_1$	
Unit cell dimensions	a = 6.7708(6) Å	a= 90°.
	b = 10.8979(10) Å	b= 90°.
	c = 19.414(2) Å	g = 90°.
Volume	1432.5(2) Å^3	
Z	4	

Density (calculated)	1.179 Mg/m^3
Absorption coefficient	0.541 mm^{-1}
F(000)	552
Crystal size	0.350 x 0.300 x 0.150 mm^3
Theta range for data collection	4.555 to 74.559°.
Index ranges	-8<=h<=8, -13<=k<=13, -24<=l<=24
Reflections collected	48838
Independent reflections	2938 [R(int) = 0.0345]
Completeness to theta = 67.679°	99.9 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.7538 and 0.6977
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2938 / 0 / 174
Goodness-of-fit on F^2	1.059
Final R indices [I>2sigma(I)]	R1 = 0.0282, wR2 = 0.0699
R indices (all data)	R1 = 0.0283, wR2 = 0.0700
Absolute structure parameter	0.10(4)
Extinction coefficient	n/a
Largest diff. peak and hole	0.172 and -0.158 $e.Å^{-3}$

Table A3.1.2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å^{2}x 10^3) for ***73****. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.*

	x	y	z	U(eq)
C(1)	7111(2)	2852(1)	6676(1)	16(1)
C(2)	5291(2)	2699(1)	7094(1)	18(1)
O(1)	4865(2)	1814(1)	7449(1)	25(1)
C(3)	4012(2)	3832(1)	6999(1)	23(1)
C(4)	5034(2)	4576(1)	6436(1)	22(1)
C(5)	6946(2)	3901(1)	6303(1)	17(1)
C(6)	8389(2)	4420(1)	5831(1)	20(1)
C(7)	9894(2)	3860(1)	5516(1)	21(1)
C(8)	10489(2)	2534(1)	5518(1)	22(1)
C(9)	9146(2)	1577(1)	5867(1)	16(1)
C(10)	10099(2)	302(1)	5786(1)	18(1)
C(15)	8664(2)	-766(1)	5788(1)	18(1)
C(16)	6898(2)	-743(1)	6084(1)	22(1)
C(17)	9404(3)	-1896(1)	5419(1)	27(1)
C(11)	11718(2)	133(1)	6336(1)	21(1)
C(12)	10898(2)	276(1)	7063(1)	20(1)
C(13)	9533(2)	1357(1)	7152(1)	17(1)

C(18)	9244(2)	1734(1)	7891(1)	21(1)
C(14)	8673(2)	1915(1)	6613(1)	15(1)

Table A3.1.3. Bond lengths [Å] and angles [°] for ***73****.*

C(1)-C(5)	1.3575(18)
C(1)-C(14)	1.4751(18)
C(1)-C(2)	1.4843(18)
C(2)-O(1)	1.2201(18)
C(2)-C(3)	1.5198(19)
C(3)-C(4)	1.526(2)
C(3)-H(3A)	0.9900
C(3)-H(3B)	0.9900
C(4)-C(5)	1.5119(19)
C(4)-H(4A)	0.9900
C(4)-H(4B)	0.9900
C(5)-C(6)	1.454(2)
C(6)-C(7)	1.335(2)
C(6)-H(6)	0.9500
C(7)-C(8)	1.500(2)
C(7)-H(7)	0.9500
C(8)-C(9)	1.5408(18)
C(8)-H(8A)	0.9900
C(8)-H(8B)	0.9900
C(9)-C(14)	1.5278(18)
C(9)-C(10)	1.5411(17)

C(9)-H(9)	1.0000
C(10)-C(15)	1.5166(19)
C(10)-C(11)	1.5414(19)
C(10)-H(10)	1.0000
C(15)-C(16)	1.327(2)
C(15)-C(17)	1.5105(19)
C(16)-H(16A)	0.9500
C(16)-H(16B)	0.9500
C(17)-H(17A)	0.9800
C(17)-H(17B)	0.9800
C(17)-H(17C)	0.9800
C(11)-C(12)	1.524(2)
C(11)-H(11A)	0.9900
C(11)-H(11B)	0.9900
C(12)-C(13)	1.5073(19)
C(12)-H(12A)	0.9900
C(12)-H(12B)	0.9900
C(13)-C(14)	1.3436(18)
C(13)-C(18)	1.5048(19)
C(18)-H(18A)	0.9800
C(18)-H(18B)	0.9800
C(18)-H(18C)	0.9800
C(5)-C(1)-C(14)	126.68(12)

C(5)-C(1)-C(2)	108.52(12)
C(14)-C(1)-C(2)	124.24(11)
O(1)-C(2)-C(1)	126.43(13)
O(1)-C(2)-C(3)	125.18(13)
C(1)-C(2)-C(3)	108.39(11)
C(2)-C(3)-C(4)	105.08(12)
C(2)-C(3)-H(3A)	110.7
C(4)-C(3)-H(3A)	110.7
C(2)-C(3)-H(3B)	110.7
C(4)-C(3)-H(3B)	110.7
H(3A)-C(3)-H(3B)	108.8
C(5)-C(4)-C(3)	104.58(11)
C(5)-C(4)-H(4A)	110.8
C(3)-C(4)-H(4A)	110.8
C(5)-C(4)-H(4B)	110.8
C(3)-C(4)-H(4B)	110.8
H(4A)-C(4)-H(4B)	108.9
C(1)-C(5)-C(6)	127.50(13)
C(1)-C(5)-C(4)	112.91(12)
C(6)-C(5)-C(4)	119.56(12)
C(7)-C(6)-C(5)	128.54(13)
C(7)-C(6)-H(6)	115.7
C(5)-C(6)-H(6)	115.7

C(6)-C(7)-C(8)	130.09(13)
C(6)-C(7)-H(7)	115.0
C(8)-C(7)-H(7)	115.0
C(7)-C(8)-C(9)	119.62(12)
C(7)-C(8)-H(8A)	107.4
C(9)-C(8)-H(8A)	107.4
C(7)-C(8)-H(8B)	107.4
C(9)-C(8)-H(8B)	107.4
H(8A)-C(8)-H(8B)	106.9
C(14)-C(9)-C(8)	112.21(11)
C(14)-C(9)-C(10)	113.70(11)
C(8)-C(9)-C(10)	108.55(11)
C(14)-C(9)-H(9)	107.4
C(8)-C(9)-H(9)	107.4
C(10)-C(9)-H(9)	107.4
C(15)-C(10)-C(9)	115.06(11)
C(15)-C(10)-C(11)	111.22(11)
C(9)-C(10)-C(11)	109.56(11)
C(15)-C(10)-H(10)	106.9
C(9)-C(10)-H(10)	106.9
C(11)-C(10)-H(10)	106.9
C(16)-C(15)-C(17)	121.35(13)
C(16)-C(15)-C(10)	124.36(13)

C(17)-C(15)-C(10)	114.29(12)
C(15)-C(16)-H(16A)	120.0
C(15)-C(16)-H(16B)	120.0
H(16A)-C(16)-H(16B)	120.0
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(12)-C(11)-C(10)	111.74(11)
C(12)-C(11)-H(11A)	109.3
C(10)-C(11)-H(11A)	109.3
C(12)-C(11)-H(11B)	109.3
C(10)-C(11)-H(11B)	109.3
H(11A)-C(11)-H(11B)	107.9
C(13)-C(12)-C(11)	114.21(11)
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(18)	124.23(12)

C(14)-C(13)-C(12)	122.00(12)
C(18)-C(13)-C(12)	113.77(12)
C(13)-C(18)-H(18A)	109.5
C(13)-C(18)-H(18B)	109.5
H(18A)-C(18)-H(18B)	109.5
C(13)-C(18)-H(18C)	109.5
H(18A)-C(18)-H(18C)	109.5
H(18B)-C(18)-H(18C)	109.5
C(13)-C(14)-C(1)	124.00(12)
C(13)-C(14)-C(9)	122.59(12)
C(1)-C(14)-C(9)	113.32(11)

Table A3.1.4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for ***73****. The anisotropic displacement factor exponent takes the form:* $-2p^2[\ h^2\ a^{*2}U^{11} + \ldots + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	19(1)	14(1)	14(1)	-3(1)	-2(1)	-2(1)
C(2)	20(1)	18(1)	17(1)	-3(1)	-1(1)	0(1)
O(1)	25(1)	23(1)	28(1)	5(1)	6(1)	-2(1)
C(3)	24(1)	21(1)	24(1)	-3(1)	2(1)	5(1)
C(4)	26(1)	18(1)	22(1)	-2(1)	-2(1)	5(1)
C(5)	22(1)	14(1)	15(1)	-4(1)	-3(1)	0(1)
C(6)	28(1)	14(1)	18(1)	2(1)	-4(1)	-3(1)
C(7)	26(1)	18(1)	19(1)	4(1)	1(1)	-6(1)
C(8)	26(1)	18(1)	21(1)	2(1)	7(1)	-1(1)
C(9)	17(1)	14(1)	16(1)	2(1)	1(1)	0(1)
C(10)	18(1)	16(1)	18(1)	0(1)	4(1)	2(1)
C(15)	23(1)	16(1)	17(1)	1(1)	-2(1)	2(1)
C(16)	21(1)	19(1)	27(1)	-1(1)	-2(1)	-3(1)
C(17)	34(1)	18(1)	30(1)	-4(1)	2(1)	2(1)
C(11)	14(1)	19(1)	30(1)	1(1)	0(1)	2(1)
C(12)	19(1)	18(1)	24(1)	4(1)	-5(1)	0(1)
C(13)	16(1)	16(1)	19(1)	1(1)	-2(1)	-4(1)
C(18)	28(1)	19(1)	17(1)	2(1)	-5(1)	-3(1)

C(14)	15(1)	13(1)	16(1)	1(1)	0(1)	-3(1)

Table A3.1.5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^{2}x 10^3) for ***73****.*

	x	y	z	U(eq)
H(3A)	3934	4309	7432	28
H(3B)	2659	3601	6856	28
H(4A)	4214	4607	6015	26
H(4B)	5293	5425	6594	26
H(6)	8241	5270	5734	24
H(7)	10716	4384	5250	25
H(8A)	11803	2480	5739	26
H(8B)	10660	2280	5032	26
H(9)	7869	1565	5608	19
H(10)	10775	294	5328	21
H(16A)	6071	-1447	6066	27
H(16B)	6461	-23	6314	27
H(17A)	8433	-2558	5465	41
H(17B)	10662	-2156	5622	41
H(17C)	9599	-1708	4930	41
H(11A)	12311	-693	6287	25
H(11B)	12773	747	6261	25
H(12A)	10176	-482	7187	24
H(12B)	12017	364	7387	24
H(18A)	8262	2394	7915	32
H(18B)	10501	2027	8080	32

H(18C)	8782	1029	8159	32

*Table A3.1.6. Torsion angles [°] for **73**.*

C(5)-C(1)-C(2)-O(1)	173.96(14)
C(14)-C(1)-C(2)-O(1)	2.0(2)
C(5)-C(1)-C(2)-C(3)	-5.26(15)
C(14)-C(1)-C(2)-C(3)	-177.21(12)
O(1)-C(2)-C(3)-C(4)	-172.03(14)
C(1)-C(2)-C(3)-C(4)	7.20(14)
C(2)-C(3)-C(4)-C(5)	-6.34(14)
C(14)-C(1)-C(5)-C(6)	-9.2(2)
C(2)-C(1)-C(5)-C(6)	179.09(13)
C(14)-C(1)-C(5)-C(4)	172.73(12)
C(2)-C(1)-C(5)-C(4)	1.03(15)
C(3)-C(4)-C(5)-C(1)	3.51(15)
C(3)-C(4)-C(5)-C(6)	-174.72(12)
C(1)-C(5)-C(6)-C(7)	20.2(2)
C(4)-C(5)-C(6)-C(7)	-161.89(14)
C(5)-C(6)-C(7)-C(8)	4.0(3)
C(6)-C(7)-C(8)-C(9)	6.5(2)
C(7)-C(8)-C(9)-C(14)	-53.97(17)
C(7)-C(8)-C(9)-C(10)	179.52(12)
C(14)-C(9)-C(10)-C(15)	81.49(14)
C(8)-C(9)-C(10)-C(15)	-152.87(11)

C(14)-C(9)-C(10)-C(11)	-44.68(15)
C(8)-C(9)-C(10)-C(11)	80.97(14)
C(9)-C(10)-C(15)-C(16)	-24.94(19)
C(11)-C(10)-C(15)-C(16)	100.38(16)
C(9)-C(10)-C(15)-C(17)	155.08(12)
C(11)-C(10)-C(15)-C(17)	-79.61(15)
C(15)-C(10)-C(11)-C(12)	-70.33(14)
C(9)-C(10)-C(11)-C(12)	58.00(14)
C(10)-C(11)-C(12)-C(13)	-44.76(16)
C(11)-C(12)-C(13)-C(14)	18.31(18)
C(11)-C(12)-C(13)-C(18)	-161.66(12)
C(18)-C(13)-C(14)-C(1)	-9.3(2)
C(12)-C(13)-C(14)-C(1)	170.73(12)
C(18)-C(13)-C(14)-C(9)	174.39(13)
C(12)-C(13)-C(14)-C(9)	-5.6(2)
C(5)-C(1)-C(14)-C(13)	138.53(14)
C(2)-C(1)-C(14)-C(13)	-51.01(19)
C(5)-C(1)-C(14)-C(9)	-44.87(18)
C(2)-C(1)-C(14)-C(9)	125.60(13)
C(8)-C(9)-C(14)-C(13)	-104.09(14)
C(10)-C(9)-C(14)-C(13)	19.59(18)
C(8)-C(9)-C(14)-C(1)	79.25(14)
C(10)-C(9)-C(14)-C(1)	-157.07(11)

(1) Sheldrick, G. M. *Acta Cryst.* **1990**, A46, 467–473.
(2) Sheldrick, G. M. *Acta Cryst.* **2015**, C71, 3–8.
(3) Müller, P. *Crystallography Reviews* **2009**, *15*, 57–83.