Supporting Information

Total Synthesis of Spiromastilactone A

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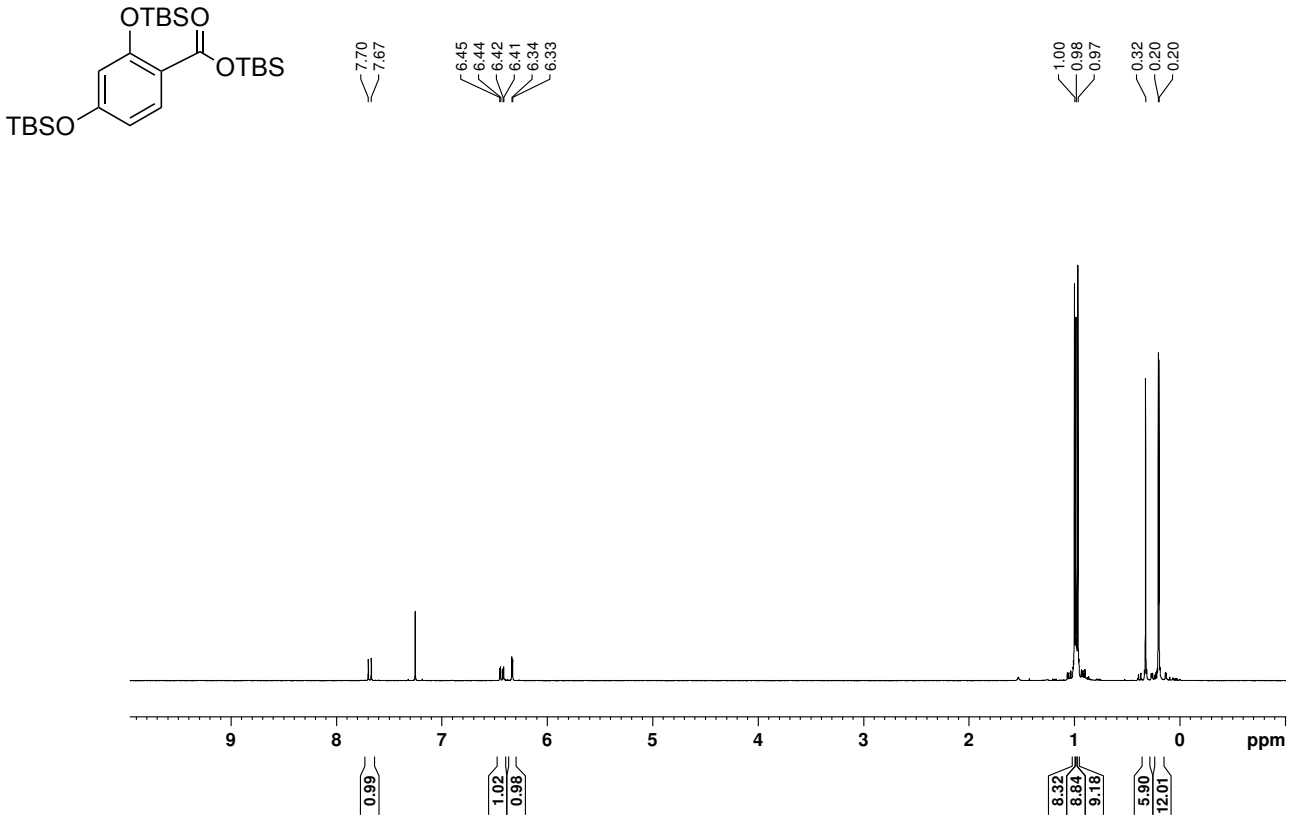
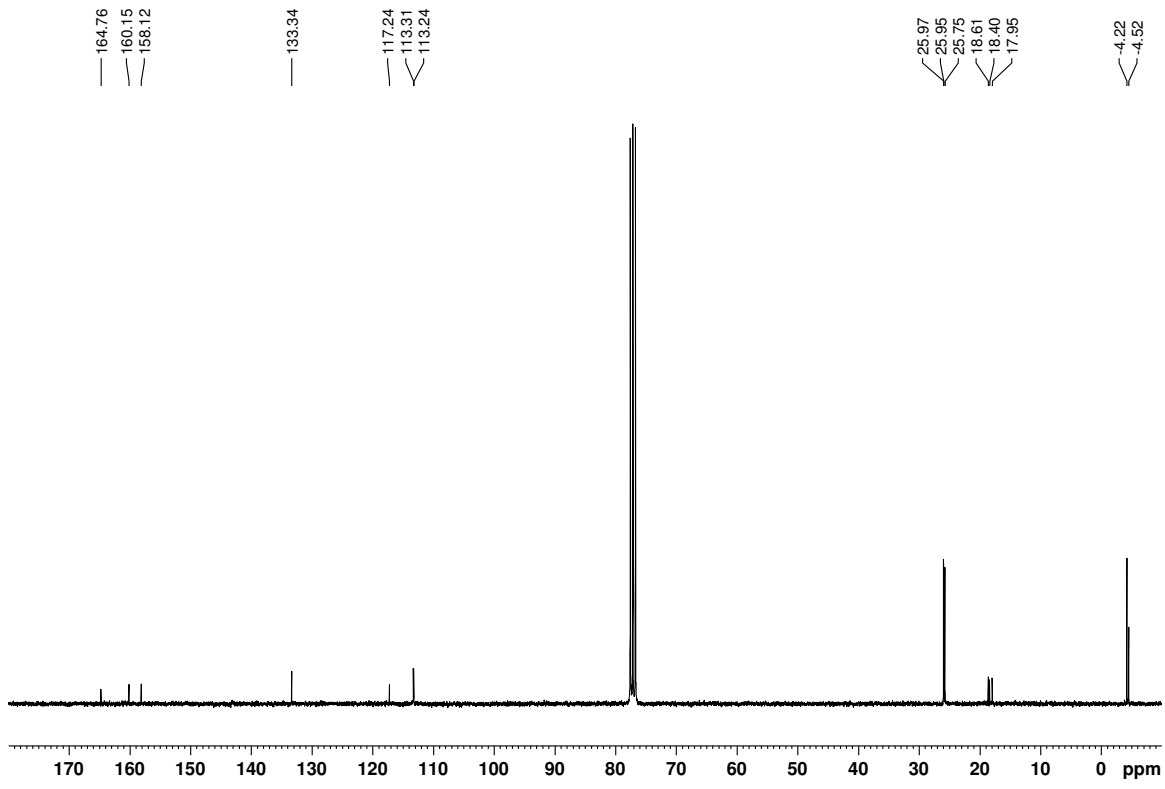
1. ^{1}H and ^{13}C spectra

tert-Butyldimethylsilyl 2,4-bis[(*tert*-butyldimethylsilyl)oxy]benzoate 2

^{1}H (300 MHz, $CDCl_3$)

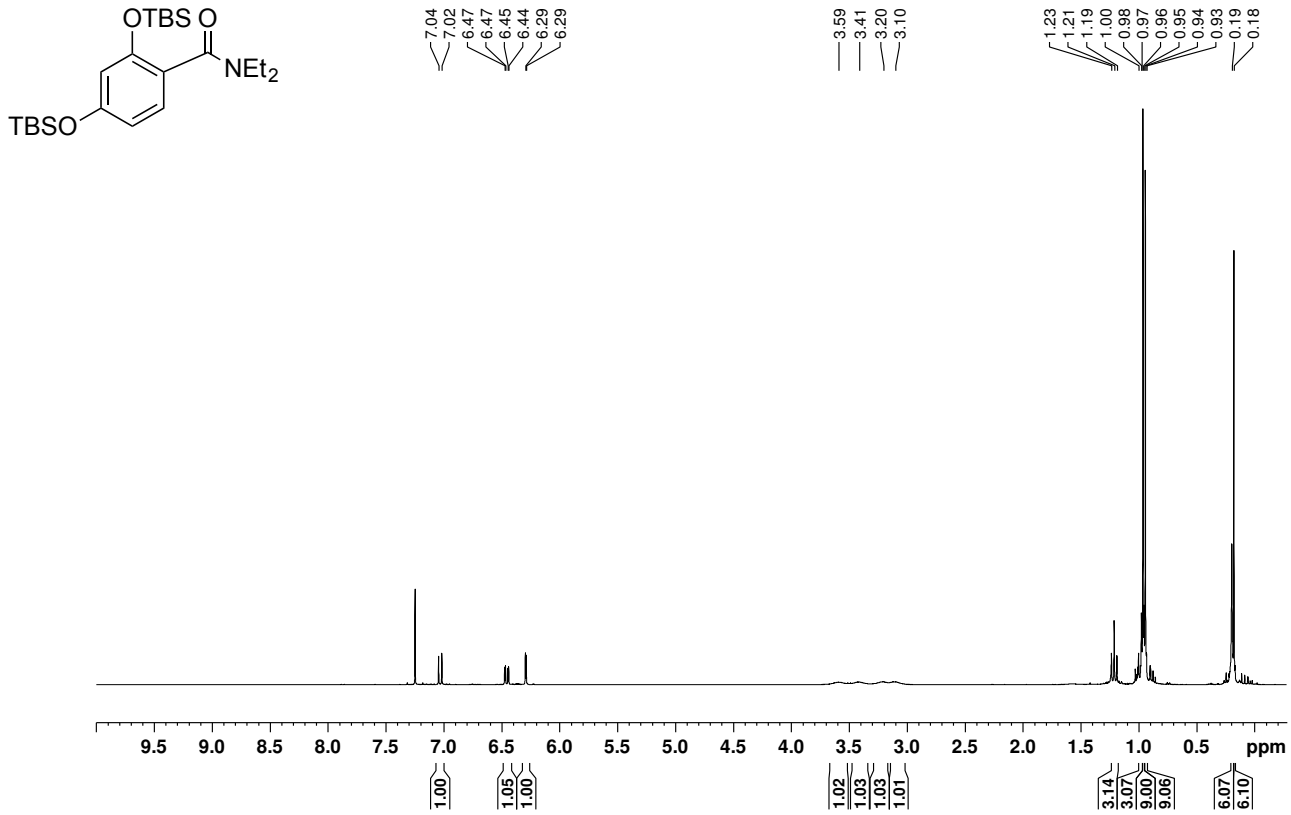


^{13}C (75 MHz, $CDCl_3$)



2,4-Bis[(*tert*-butyldimethylsilyl)oxy]-*N*,*N*-diethylbenzamide 3

^{1}H (300 MHz, $CDCl_3$)



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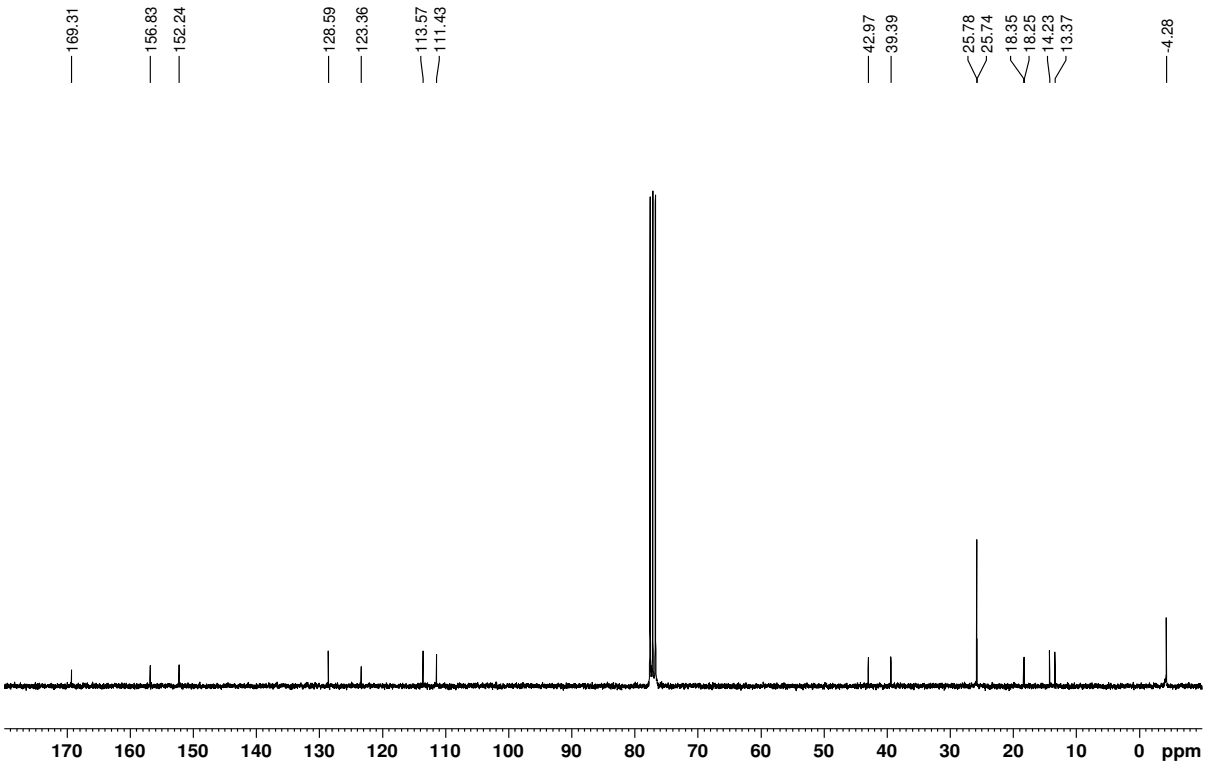
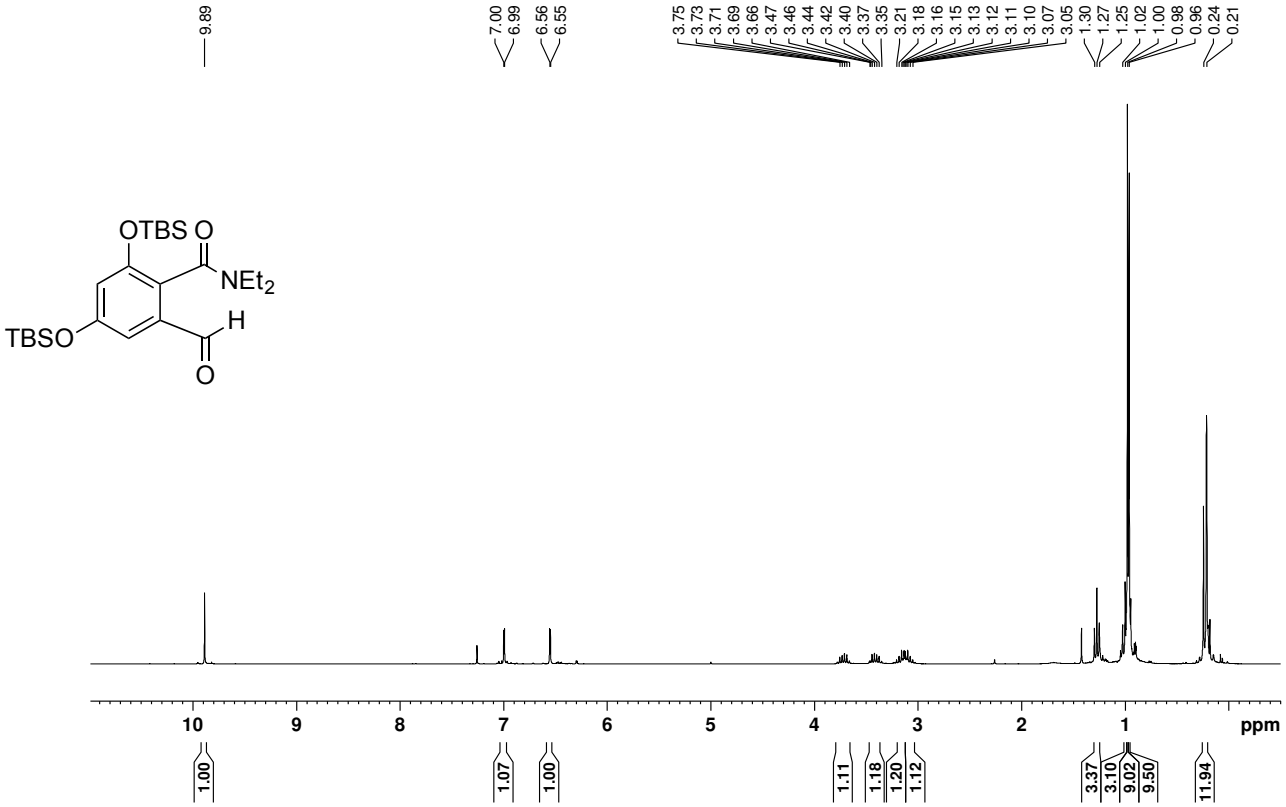
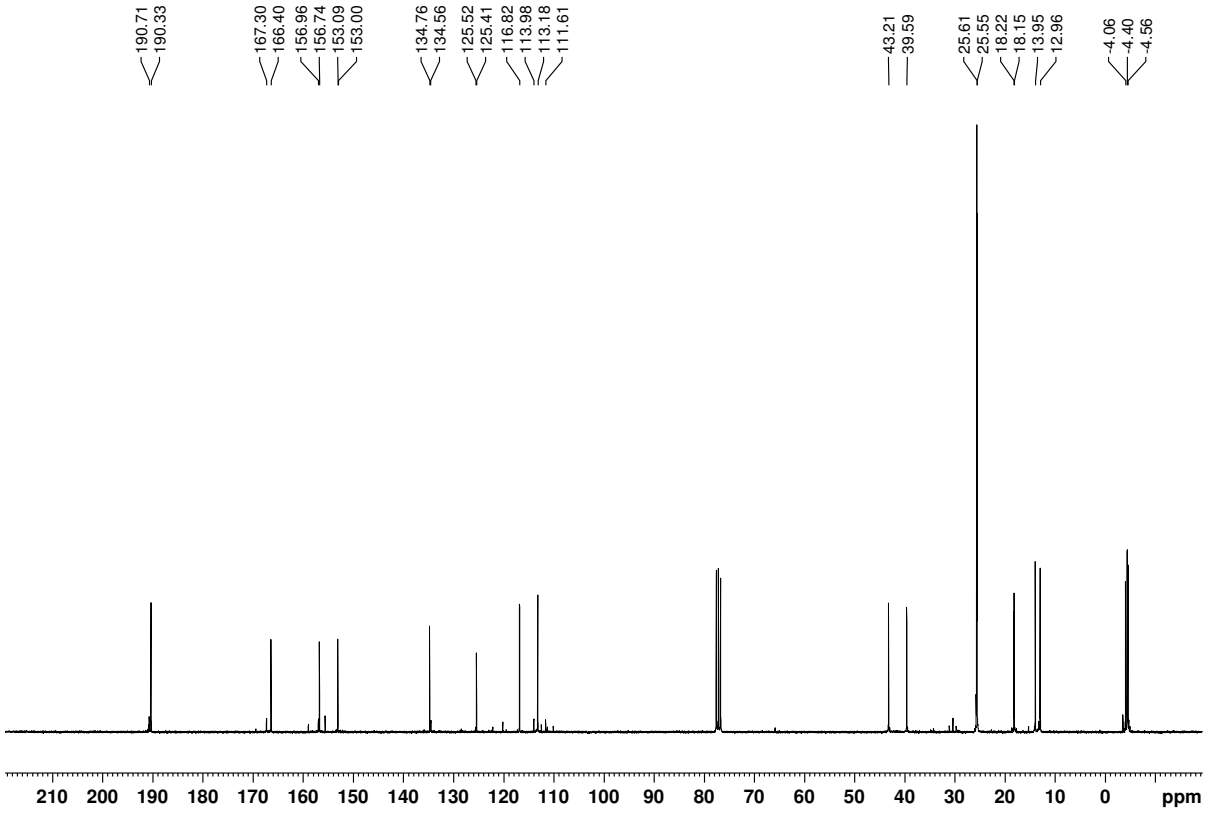


2,4-Bis[(*tert*-butyldimethylsilyl)oxy]-*N*,*N*-diethyl-6-formylbenzamide 4

^{1}H (300 MHz, $CDCl_3$)

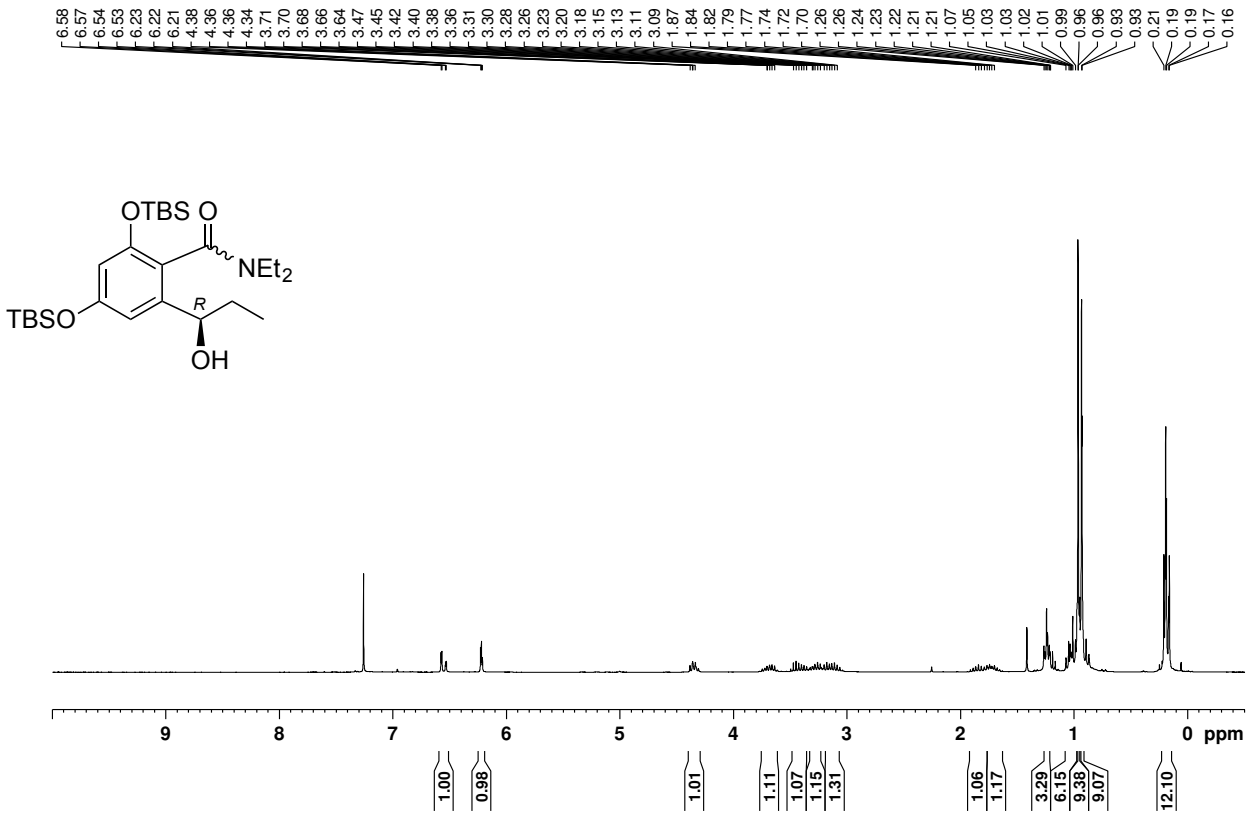


^{13}C (75 MHz, $CDCl_3$)

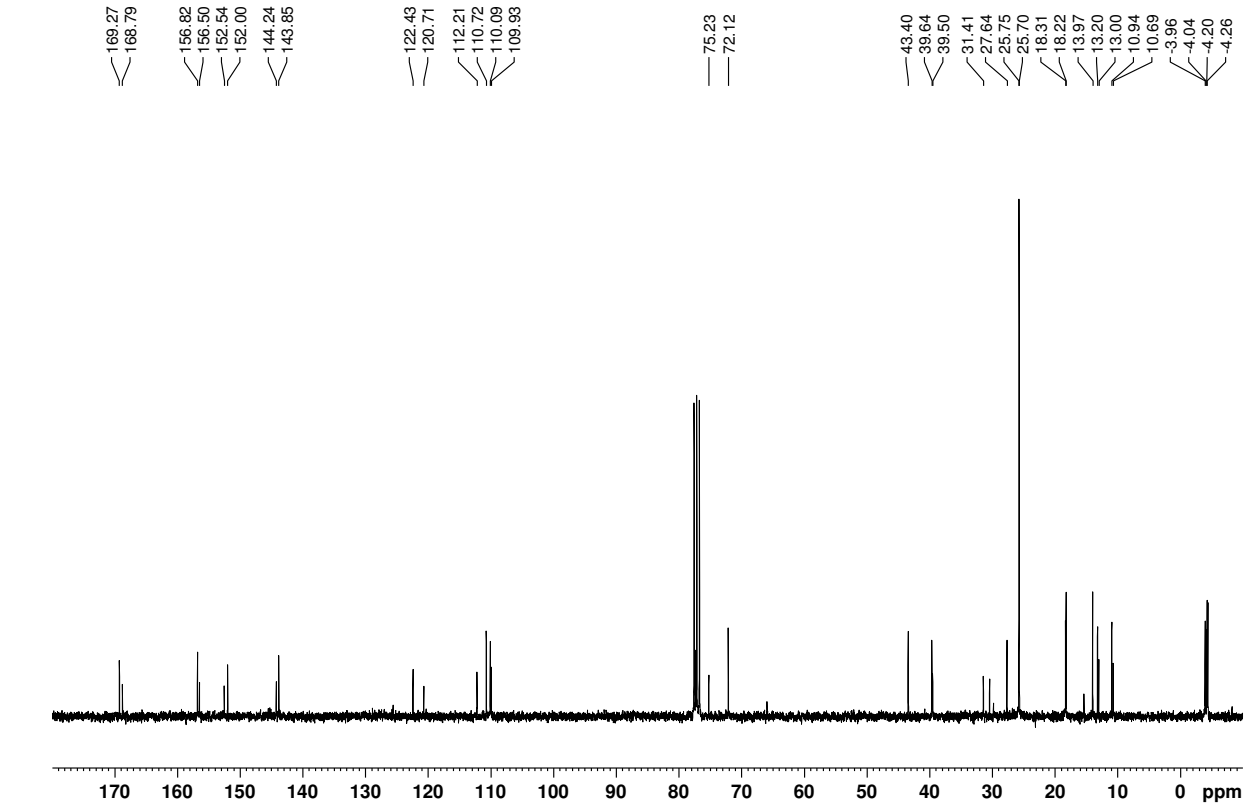


2,4-Bis[(*tert*-butyldimethylsilyl)oxy]-*N*,*N*-diethyl-6-(1-hydroxypropyl)benzamide 6

^{1}H (300 MHz, $CDCl_3$)

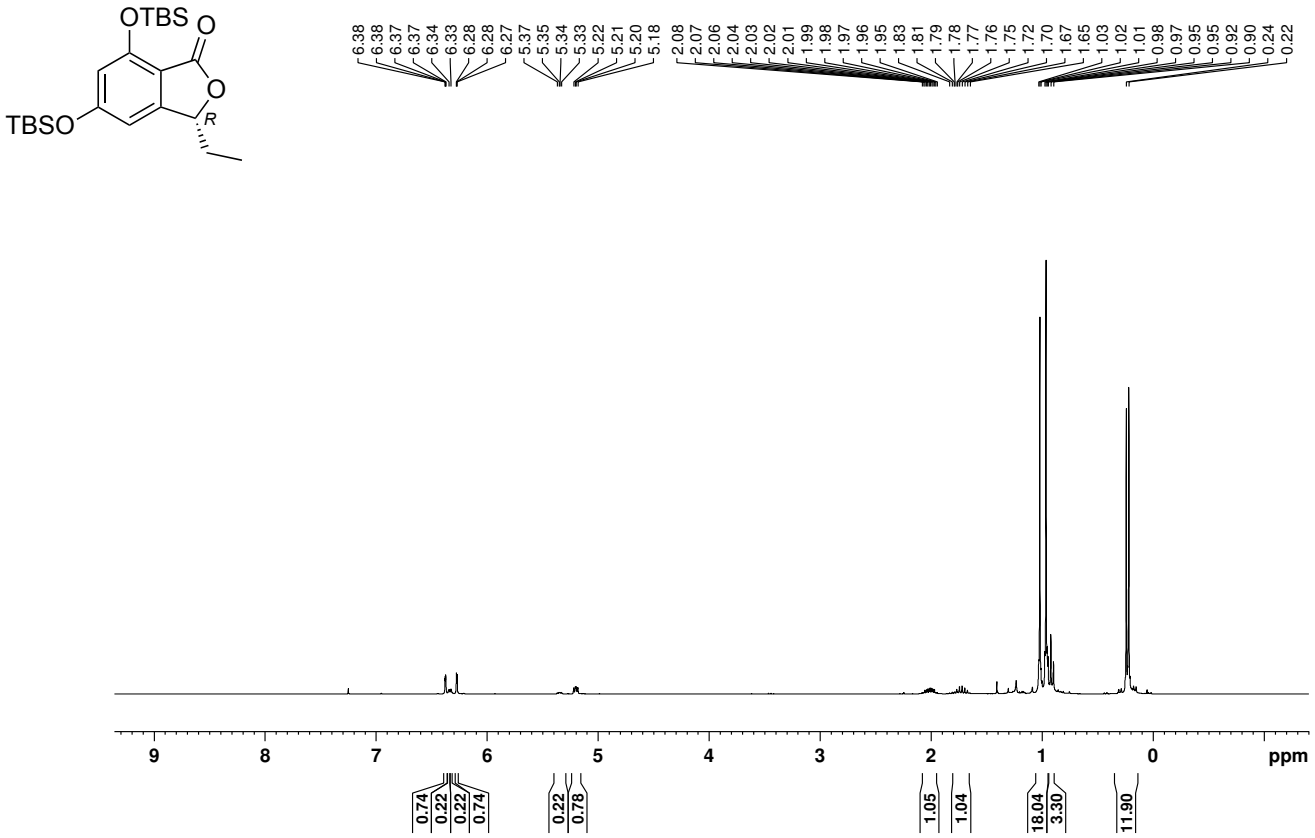


^{13}C (75 MHz, $CDCl_3$)

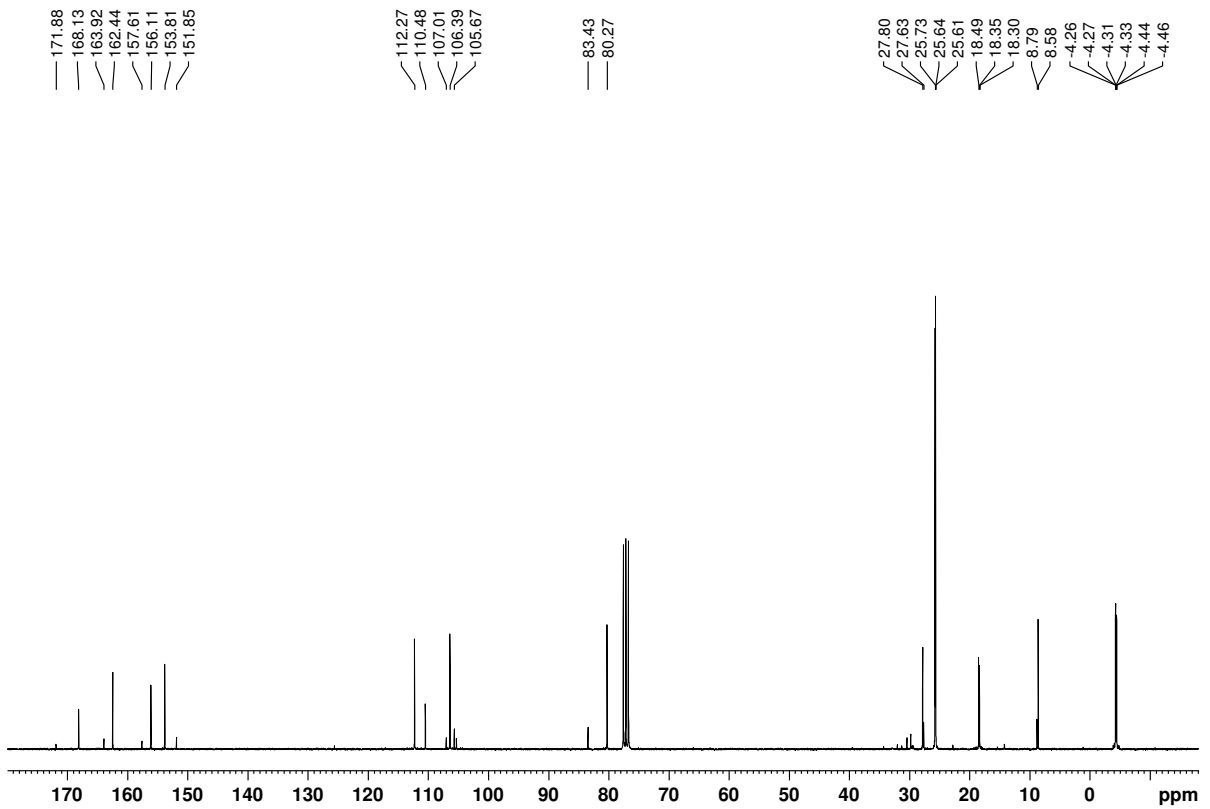


5,7-Bis[(*tert*-butyldimethylsilyl)oxy]-3-ethyl-*iso*-benzofuran-1(3*H*)-one 7

^{1}H (300 MHz, $CDCl_3$)

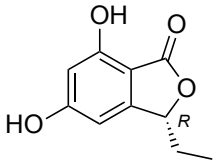


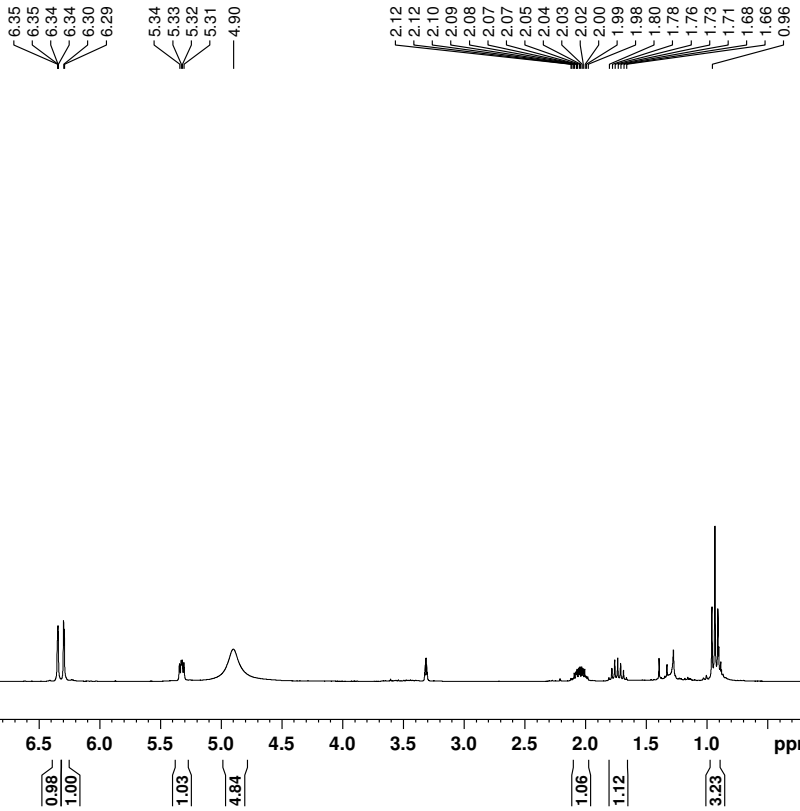
^{13}C (75 MHz, $CDCl_3$)



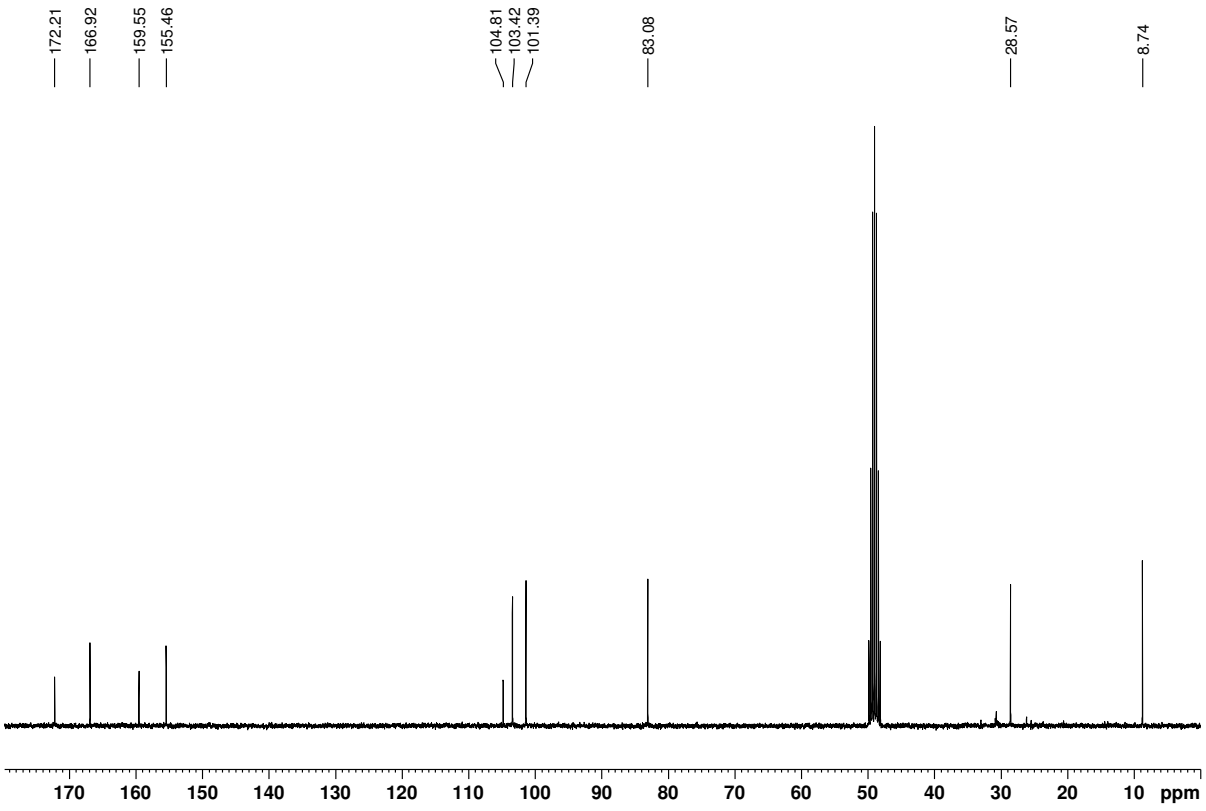
3-Ethyl-5,7-dihydroxy-*iso*-benzofuran-1(3*H*)-one 8

^{1}H (300 MHz, CD_3OD)



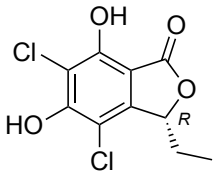


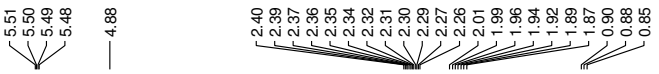
^{13}C (75 MHz, CD_3OD)

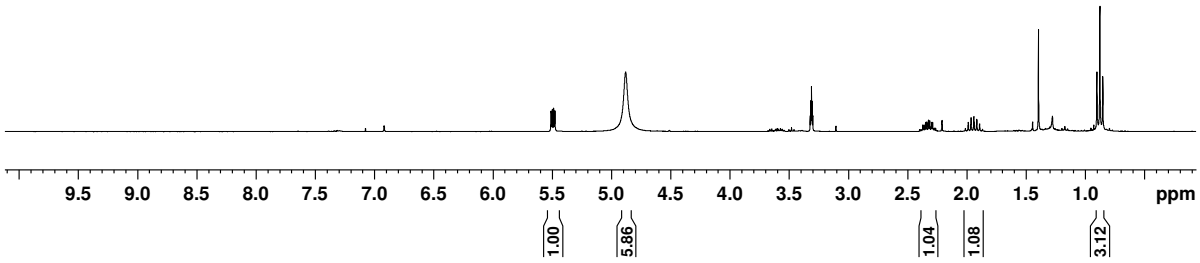


4,6-Dichloro-3-ethyl-5,7-dihydroxy-*iso*-benzofuran-1(3*H*)-one 1 – Spiromastilactone A

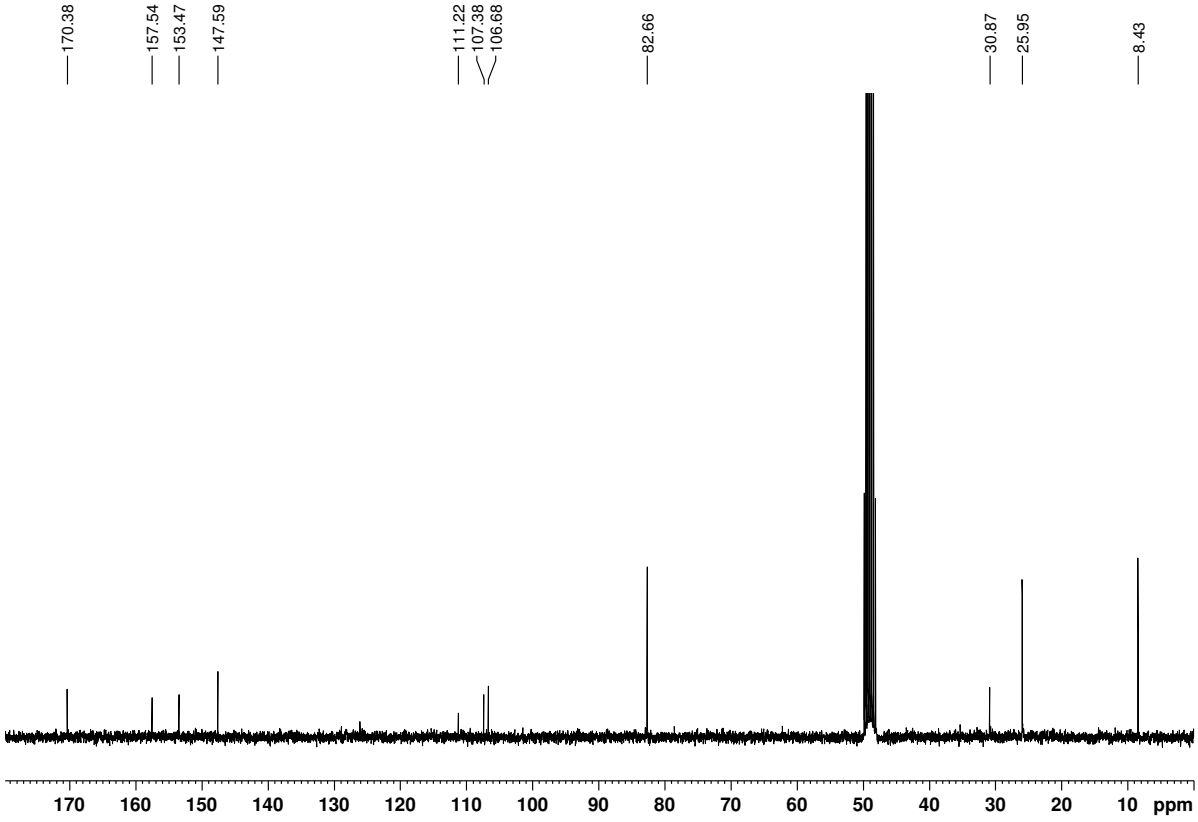
^{1}H (300 MHz, CD_3OD)





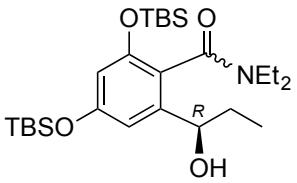


^{13}C (75 MHz, CD_3OD)



2. HPLC Traces

2,4-Bis[(*tert*-butyldimethylsilyl)oxy]-*N*,*N*-diethyl-6-(1-hydroxypropyl)benzamide 6



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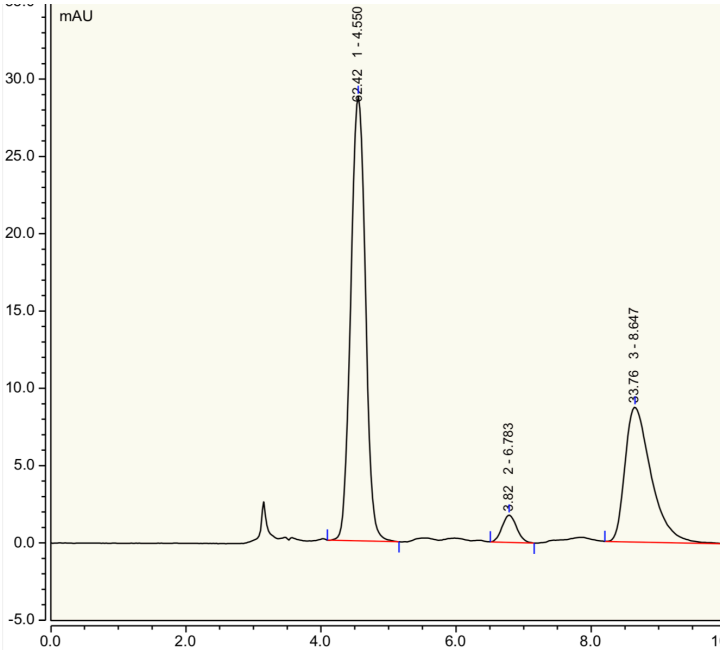

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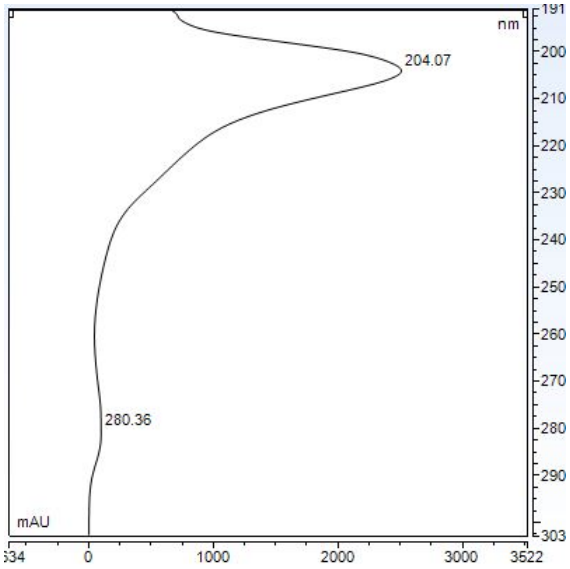

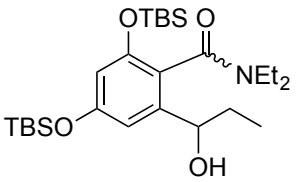

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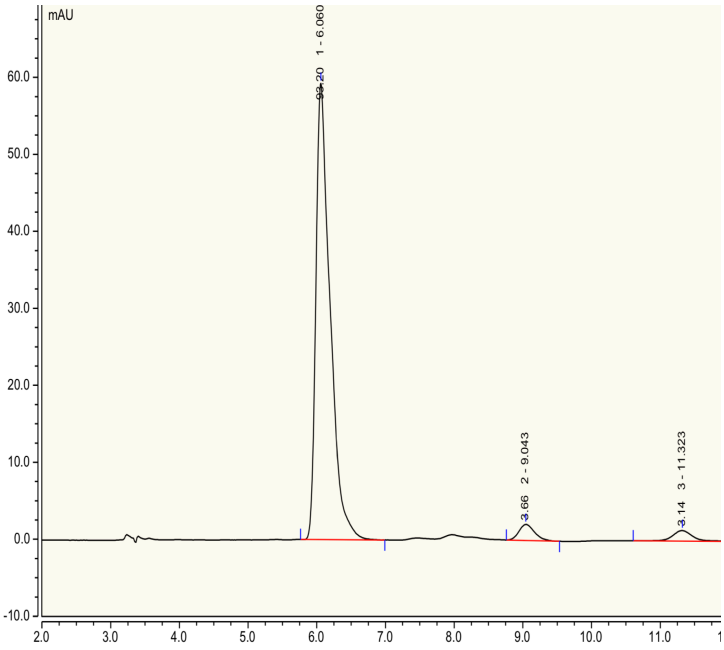

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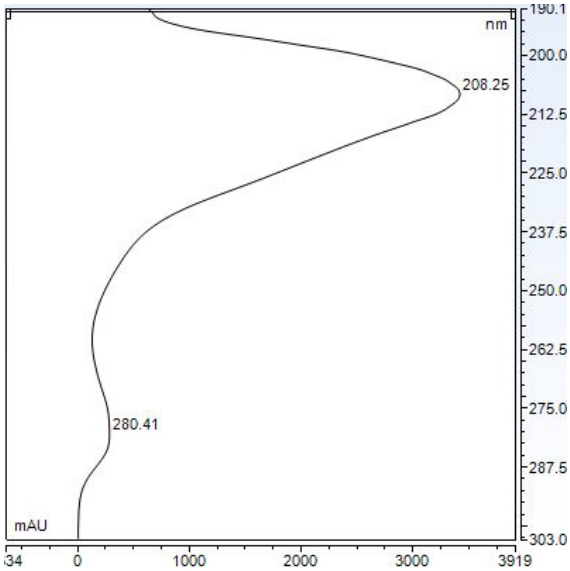
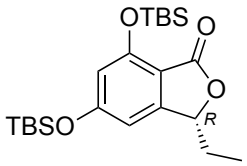


5,7-Bis[(*tert*-butyldimethylsilyl)oxy]-3-ethyl-*iso*-benzofuran-1(3*H*)-one 7



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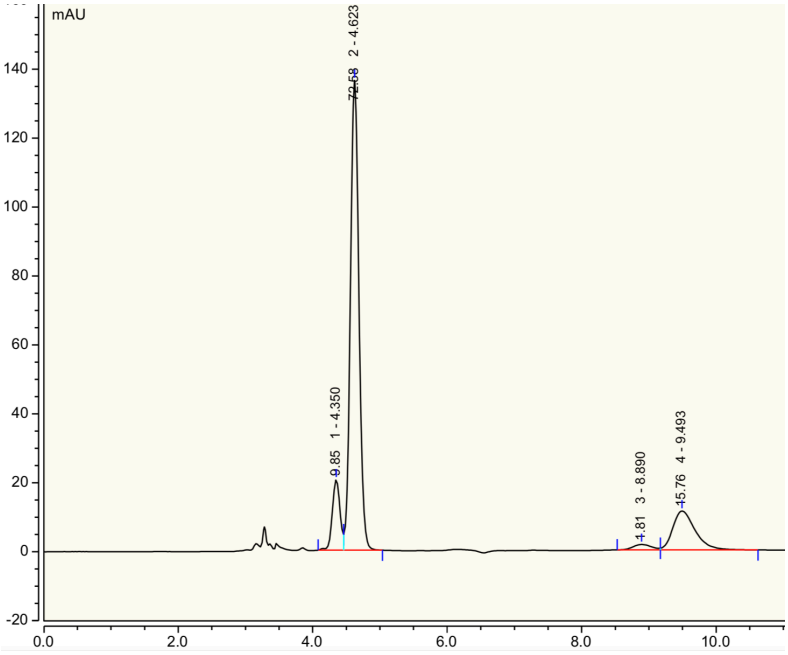

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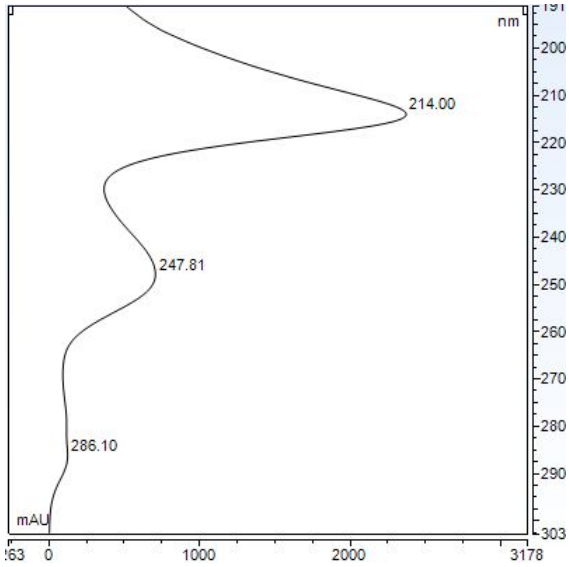

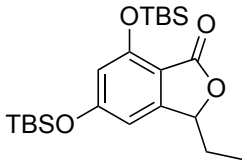

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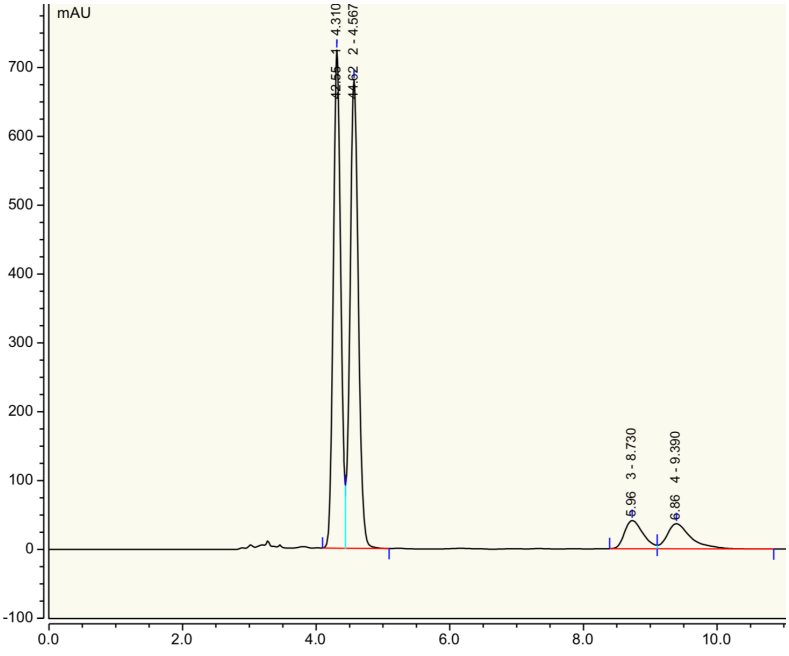

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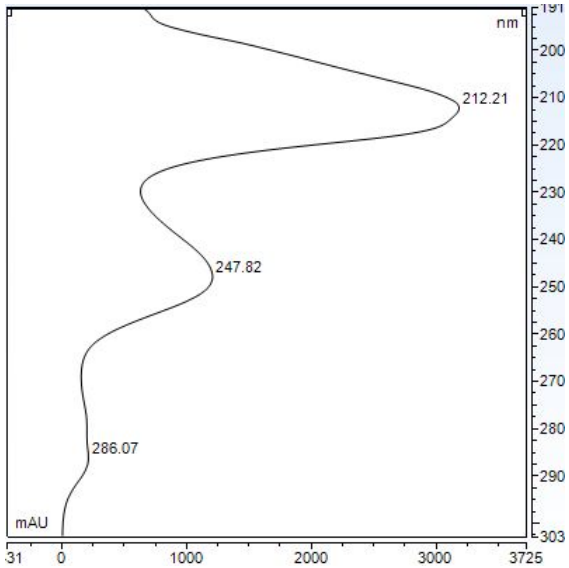
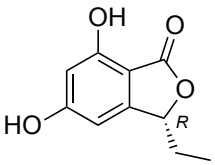


3-Ethyl-5,7-dihydroxy-*iso*-benzofuran-1(3*H*)-one 8



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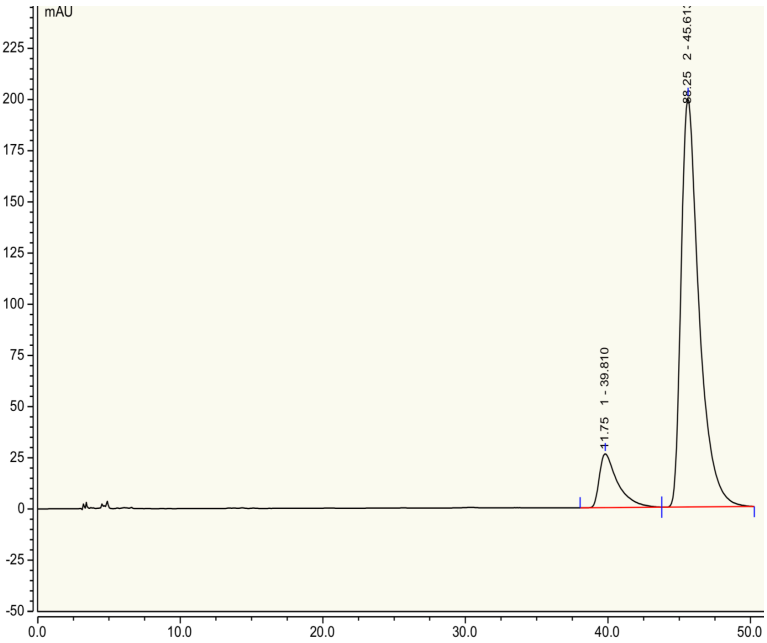

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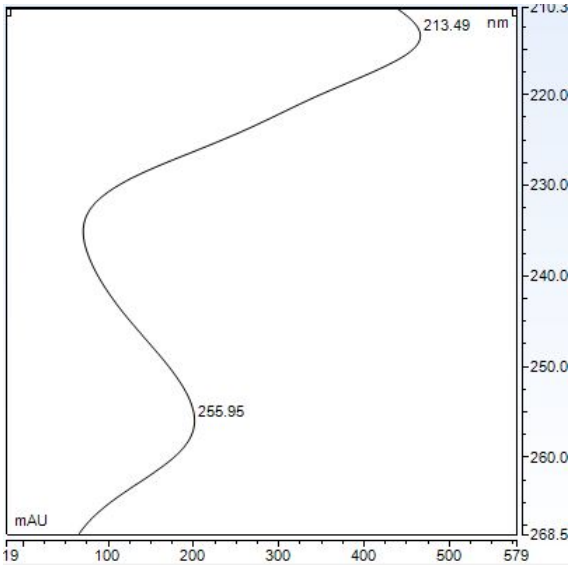

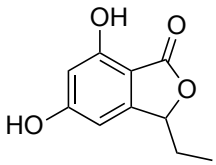

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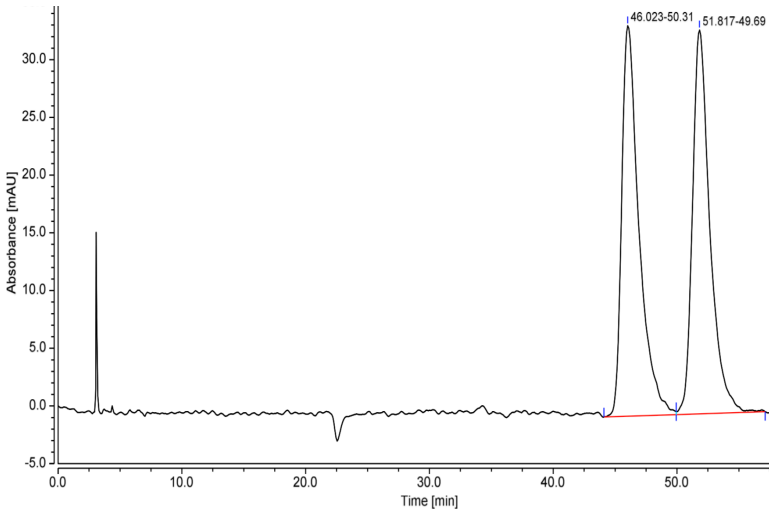

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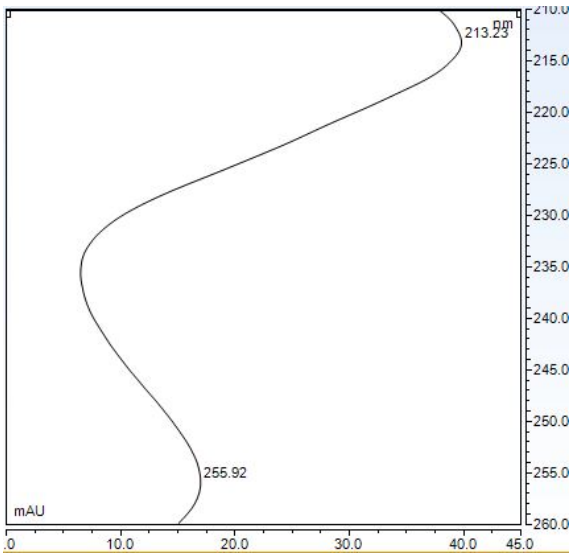
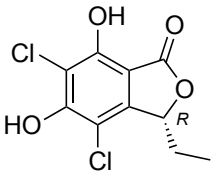


4,6-Dichloro-3-ethyl-5,7-dihydroxy-*iso*-benzofuran-1(3*H*)-one 1 – Spiromastilactone A



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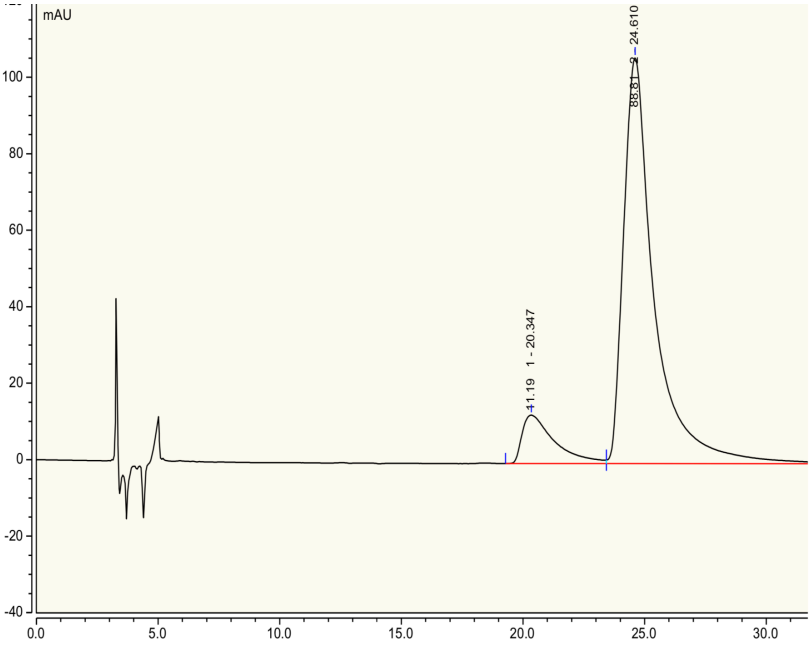

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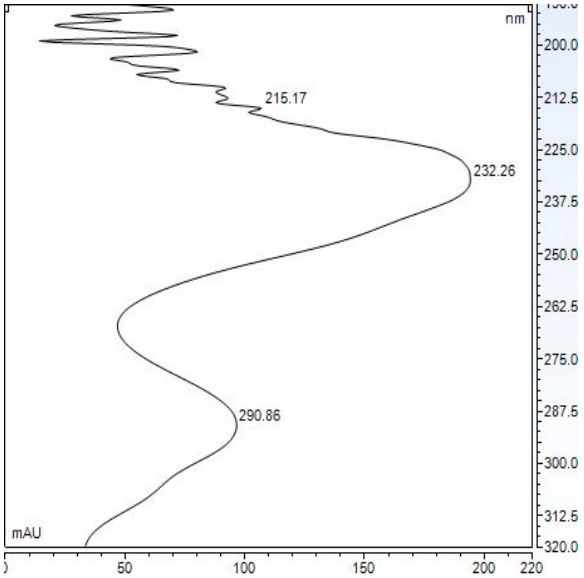

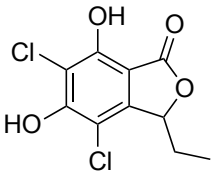

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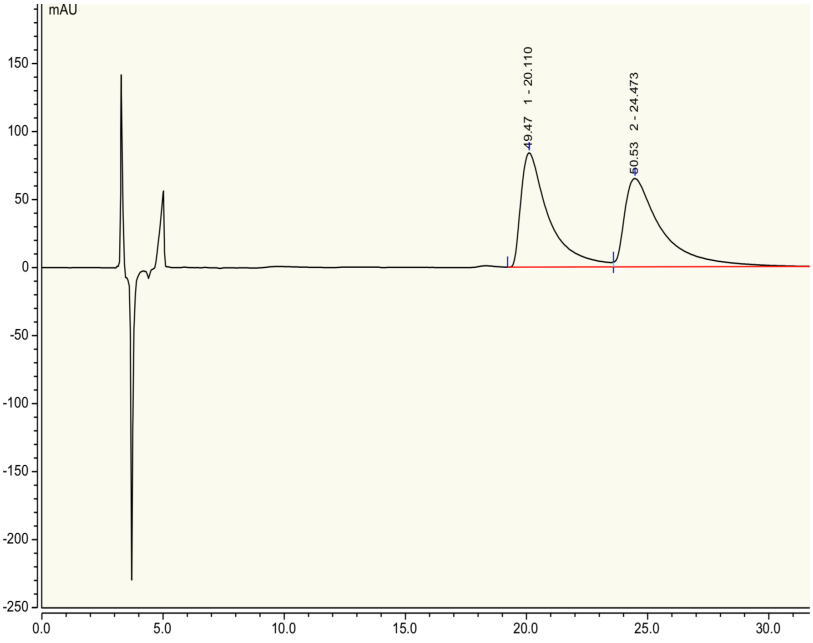

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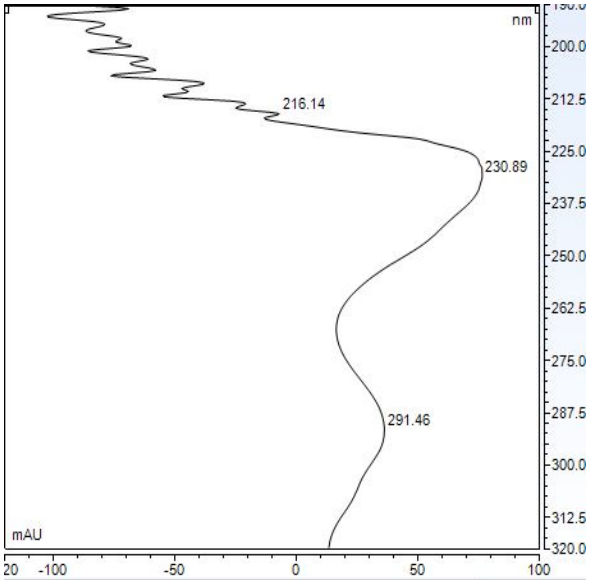


3. X-Ray crystallography data

2,4-Bis[(*tert*-butyldimethylsilyl)oxy]-*N*,*N*-diethyl-6-(1-hydroxypropyl)benzamide 6

SAMPLE PREPARATION

In a little flask, about 50 mg of pure 6 was poured in pentane and solubilized by heating. A cap with holes was added to the flask to permit a slow evaporation of the solvent. After few days crystals grew up and were analyzed by X-ray diffraction.

DATA COLLECTION

The crystal structure of 6 [$C_{26}H_{49}NO_4Si_2$] has been determined from single crystal X-Ray diffraction. The chosen crystal was stuck on a glass fibre and mounted on the full three-circle goniometer of a Bruker SMART APEX diffractometer with a CCD area detector. Three sets of exposures (a total of 1800 frames) were recorded, corresponding to three ω scans (steps of 0.3°), for three different values of ϕ. The details of data collection are given in annexe 1.

The cell parameters and the orientation matrix of the crystal were preliminary determined by using SMART Software[1]. Data integration and global cell refinement were performed with SAINT Software[2]. Intensities were corrected for Lorentz, polarisation, decay and absorption effects (SAINT and SADABS Softwares) and reduced to F_O^2. The program package WinGX[3] was used for space group determination, structure solution and refinement.

DATA REFINEMENT

The non-standard space group $P2_1/a$ (n°14) was determined from systematic extinctions and relative F_O^2 of equivalent reflections. The structure was solved by direct methods[4]. Anisotropic displacement parameters were refined for all non-hydrogen atoms. Every Hydrogen atoms were located from subsequent difference Fourier syntheses and placed with geometrical constraints (SHELXL[5]). The final cycle of full-matrix least-square refinement on F^2 was based on 6430 observed reflections and 312 variable parameters and converged with unweighted and weighted agreement factors of:
R1 = 0.0786, wR2 = 0.1887 for 2549 reflections with $I>2\sigma I$ and R1 = 0.1895, wR2 = 0.2425 for all data.

The refinement data are given in annexe 1 table 2

CRYSTALLOGRAPHIC DATA AND STRUCTURAL DESCRIPTION

Crystallographic data

The crystal data are collected in Table 1. The full crystallographic parameters (atomic coordinates, bond length, angles and anisotropic displacements) are reported in annexe 2.

Table 1: Crystal data

Chemical Formula	$C_{26}H_{49}NO_4Si_2$
Molecular Weight / *g.mol*$^{-1}$	495.84
Crystal System	Monoclinic
Space Group	$P2_1/a$
Z , Z' (asymmetric units per unit cell)	4,1
a / *Å*	12.427(2)
b / *Å*	19.931(4)
c / *Å*	12.720(2)
α / °	90
β / °	96.168(4)
γ / °	90
V / *Å*^{3}	3132.4(1)
d_{calc} / *g.cm*$^{-3}$	1.051
F(000) / e^-	1088
Absorption coefficient μ (Mo$K\alpha_1$) / *mm*$^{-1}$	0.140

Structural description

The asymmetric unit is composed of one molecule of $C_{26}H_{49}NO_4Si_2$ (figures 1&2). The hydroxyl and the carbonyl moieties from two consecutive molecules along b axis establish Hydrogen bonds back and forth, so that it generates dimers (figure 3, table 2). The cohesion of the dimers within the packing is ensured by van der Waals interactions (figures 4, 5 , 6).

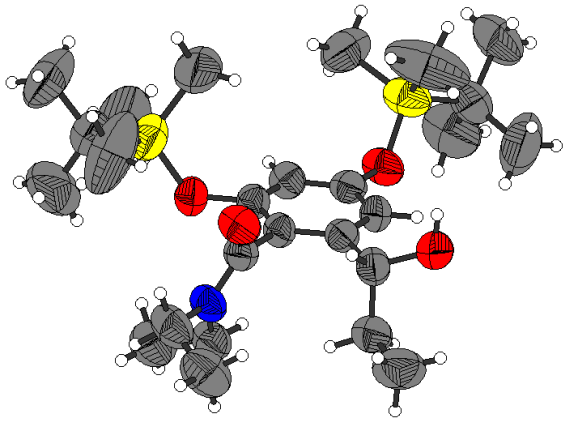


Figure1: Asymmetric unit of 6 in thermal ellipsoidal representation (50% probability of presence)

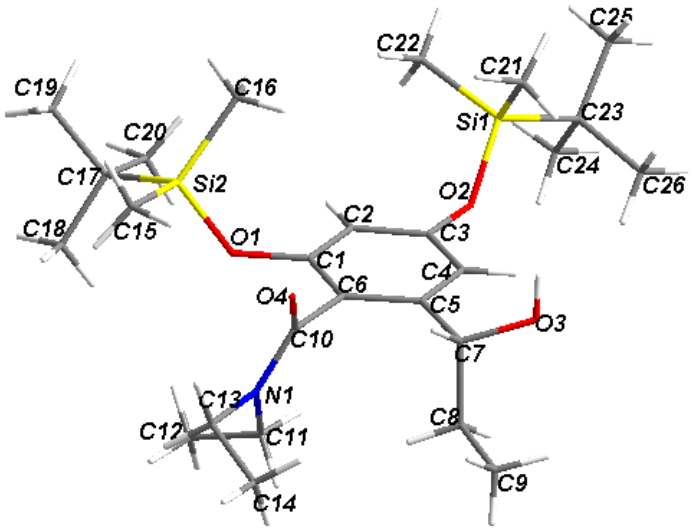


Figure 2: asymmetric unit with atom labels

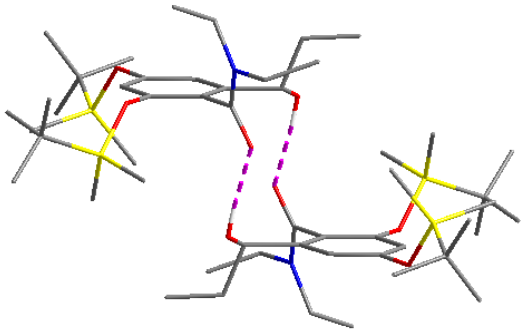


Figure 3: Dimers built from the Hydrogen bond interaction (dashed pink lines)

Table 2: hydrogen bond table

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
O(3)-H(3)...O(4)#1	0.82	1.98	2.779(4)	164

Symmetry transformation used to generate equivalent atoms
#1 -x+1,-y,-z+1

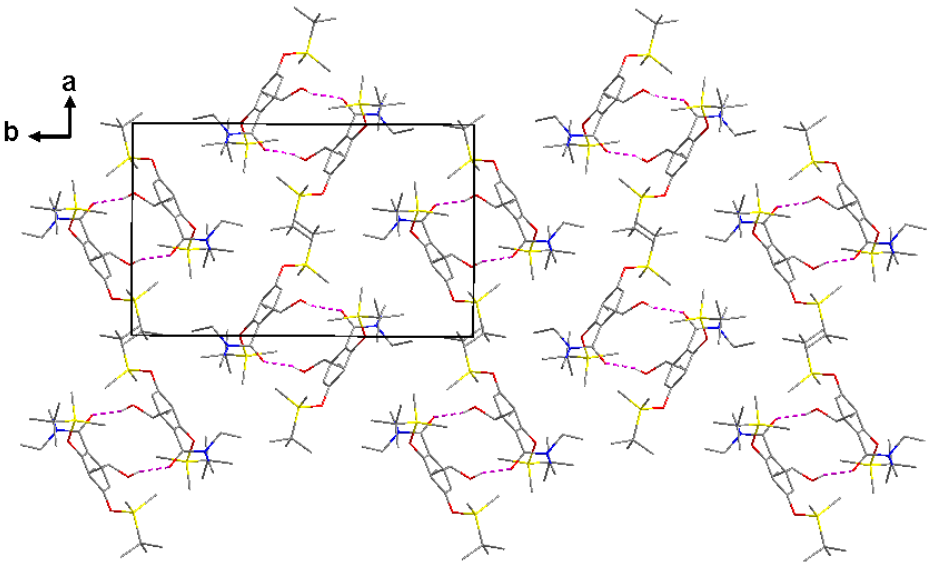


Figure 4: Projection along c

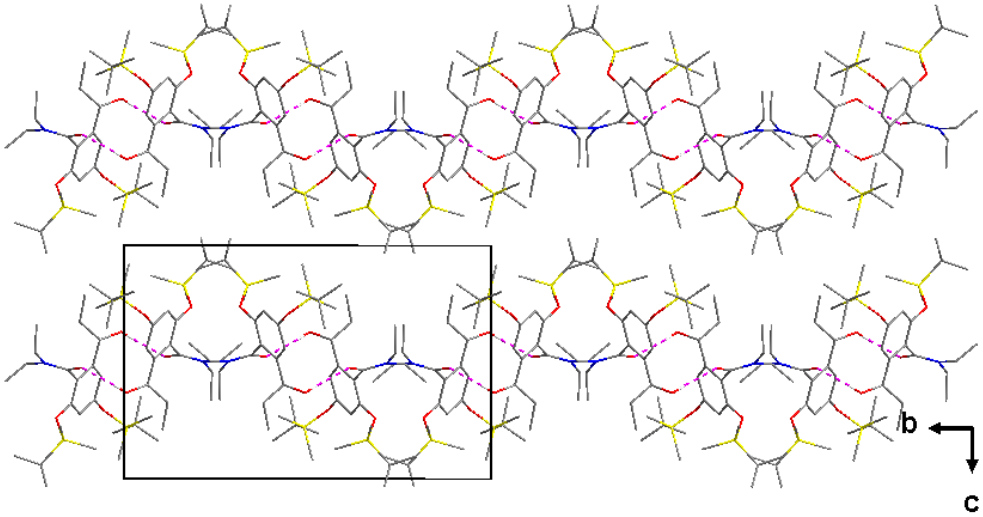


Figure 5a: Projection along a

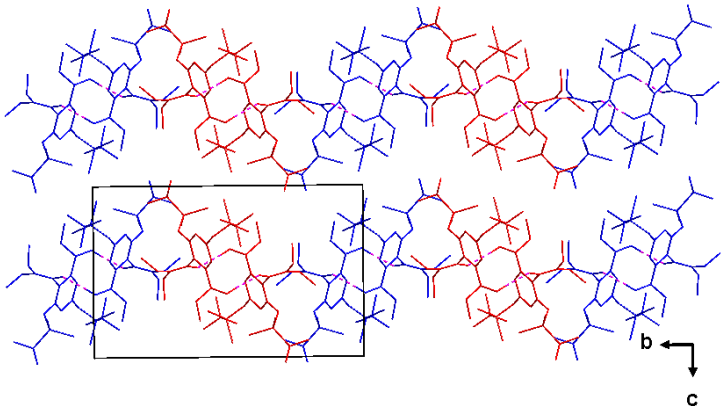


Figure 5b: The dimers are displayed in different colour for clarity

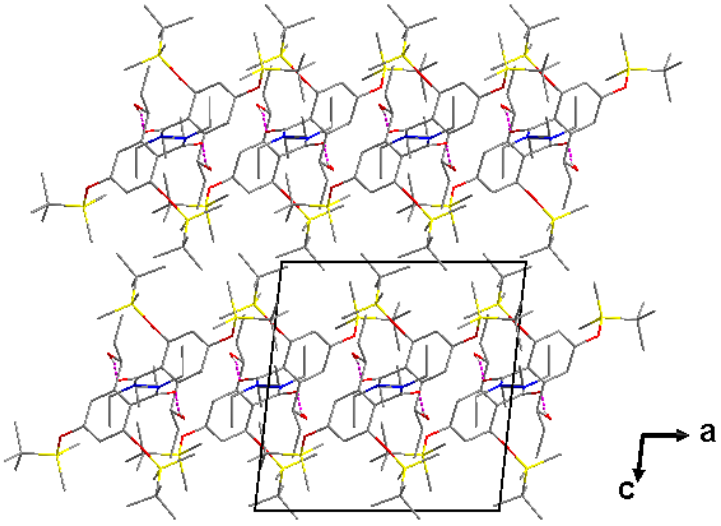


Figure 6a: Projection along b

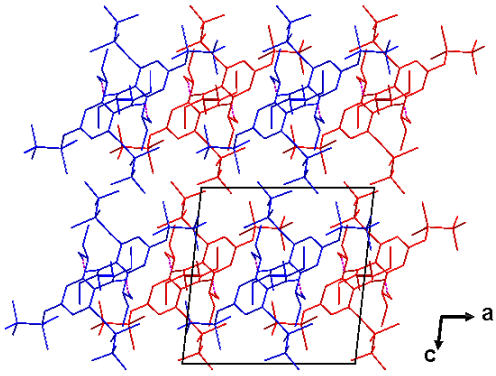


Figure 6 b: the dimers are displayed in different colour for clarity

ANNEXE 1 :

- Table 1 : DATA COLLECTION FOR 6

Date	23/10/18
Temperature / *K*	RT
Radiation	Mo-Kα_1 (λ = 0.71073 Å)
Monochromator	Graphite
Collimator / *mm*	0.5
Generator set	50 kV 40mA
Crystal-detector distance / mm	60
Detector 2θ angle / °	-28
ω oscillations / °	-0.3
ω scan 1	χ = 54.7°, ϕ = 0°, -28° ≤ ω ≤ -208°
ω scan 2	χ = 54.7°, ϕ = 120°, -28° ≤ ω ≤ -208°
ω scan 3	χ = 54.7°, ϕ = 240°, -28° ≤ ω ≤ -208°
Time exposure / *s*	10
Total number of reflections	24651
Unique reflections [F_o > 4.0 $\sigma(F_o)$]	6430 / 2549
θ range / °	1.91 to 26.43
hkl range	-15≤h≤15, -24≤k≤24, -15≤l≤15
R_{int} = $\Sigma[\lvert F_O^2 - F_O^2(mean)\rvert]$ / $\Sigma[F_O^2]$	0.0912
Completeness to θ = 26.40 / %	99.7

- Table 2 : REFINEMENT DATA FOR 6

Number of reflections (n) (with F_O > 4.0 $\sigma(F_O)$)	2549
Number of refined parameters (p) / restraints	312 /0
Final R indices [I>2sigma(I)]	R1 = 0.0786, wR2 = 0.1887
R indices (all data)	R1 = 0.1895, wR2 = 0.2425
Goodness of Fit indicator (Restrained GooF)	1.014
Maximum peak in Final Difference Map / $e^-Å^{-3}$	0.572
Maximum hole in Final Difference Map / $e^-Å^{-3}$	-0.227

$R_1 = \Sigma\ (||F_O|-|F_C||) / \Sigma\ |F_O|$

$wR_2 = [\ \Sigma\ [\ w\ (F_O^2 - F_C^2)^{\ 2}] / \Sigma\ [\ w\ (F_O^2)^2\ ]\ ]^{1/2}$

$GooF = [\Sigma\ [\ w\ (F_O^2 - F_C^2)^{\ 2}] / (n - p)\ ]^{1/2}$

ANNEXE 2 : CRYSTALLOGRAPHIC DATA FOR 6

Table 1a: Atomic coordinates ($x10^4$) and equivalent isotropic displacement parameters ($Å^2$ x 10^3). U(eq) is defined as one third of the trace of the orthogonalized Uij tensor

	x	y	z	U(eq)
C(1)	5735(3)	-1425(2)	3217(3)	66(1)
C(2)	6654(3)	-1200(2)	2789(3)	70(1)
C(3)	7373(3)	-796(2)	3390(3)	67(1)
C(4)	7184(3)	-616(2)	4402(3)	68(1)
C(5)	6280(3)	-839(2)	4841(3)	62(1)
C(6)	5550(3)	-1262(2)	4243(3)	61(1)
C(7)	6110(3)	-647(2)	5964(3)	72(1)
C(8)	6502(4)	-1196(2)	6731(3)	89(1)
C(9)	6304(5)	-1058(3)	7865(4)	129(2)
C(10)	4552(3)	-1517(2)	4678(3)	67(1)
C(11)	5418(3)	-2634(2)	4948(3)	83(1)
C(12)	5206(4)	-3211(2)	4189(4)	121(2)
C(13)	3547(4)	-2406(2)	5423(4)	98(1)
C(14)	3686(4)	-2375(3)	6608(4)	121(2)
C(15)	2729(4)	-1735(4)	2271(5)	198(4)
C(16)	4260(6)	-759(3)	1288(6)	197(4)
C(17)	4115(4)	-2253(3)	610(3)	95(1)
C(18)	3972(7)	-2951(3)	978(5)	197(4)
C(19)	3242(5)	-2101(4)	-311(4)	173(3)
C(20)	5239(5)	-2183(4)	186(5)	187(3)
C(21)	7508(6)	757(3)	2681(6)	203(4)
C(22)	7885(5)	-139(3)	819(4)	153(2)
C(23)	9819(4)	305(3)	2320(4)	105(2)
C(24)	10476(4)	-275(3)	1975(5)	147(2)
C(25)	9992(5)	924(3)	1655(5)	146(2)
C(26)	10202(6)	466(4)	3528(5)	194(3)
N(1)	4512(2)	-2167(2)	4962(2)	73(1)
O(1)	5025(2)	-1840(1)	2620(2)	77(1)
O(2)	8291(2)	-577(1)	2979(2)	79(1)
O(3)	6697(2)	-65(1)	6305(2)	86(1)
O(4)	3786(2)	-1132(1)	4764(2)	81(1)
Si(1)	8365(1)	90(1)	2228(1)	97(1)
Si(2)	4042(1)	-1636(1)	1690(1)	99(1)

Table 1b: Hydrogen coordinates ($x10^4$) and equivalent isotropic displacement parameters ($Å^2$ x 10^3). U(eq) is defined as one third of the trace of the orthogonalized Uij tensor

	x	y	z	U(eq)
H(2)	6779	-1321	2107	84
H(4)	7676	-339	4797	82
H(7)	5338	-569	6006	86
H(8A)	6142	-1612	6505	106
H(8B)	7272	-1259	6703	106
H(9A)	6696	-664	8113	193
H(9B)	6545	-1434	8299	193
H(9C)	5545	-988	7901	193
H(11A)	5594	-2812	5655	99
H(11B)	6044	-2388	4763	99
H(12A)	4600	-3466	4376	181
H(12B)	5834	-3494	4223	181
H(12C)	5048	-3041	3483	181
H(13A)	3399	-2866	5202	118
H(13B)	2930	-2135	5157	118
H(14A)	4277	-2659	6876	182
H(14B)	3034	-2525	6876	182
H(14C)	3837	-1921	6831	182
H(15A)	2587	-2203	2368	298
H(15B)	2155	-1543	1801	298
H(15C)	2772	-1510	2941	298
H(16A)	4473	-493	1904	296
H(16B)	3601	-584	929	296
H(16C)	4818	-746	823	296
H(18A)	3888	-3247	381	296
H(18B)	3339	-2974	1348	296
H(18C)	4595	-3082	1444	296
H(19A)	3150	-2485	-768	260
H(19B)	3461	-1723	-703	260
H(19C)	2570	-2002	-36	260
H(20A)	5790	-2355	702	280
H(20B)	5379	-1719	55	280
H(20C)	5239	-2432	-459	280
H(21A)	6761	636	2520	305
H(21B)	7647	1169	2330	305
H(21C)	7667	813	3431	305
H(22A)	8249	-538	626	230
H(22B)	8041	222	359	230
H(22C)	7119	-218	752	230
H(24A)	10217	-397	1263	220
H(24B)	10405	-650	2435	220
H(24C)	11223	-146	2008	220
H(25A)	10750	1025	1701	219
H(25B)	9610	1297	1914	219
H(25C)	9724	838	932	219
H(26A)	10133	70	3945	291
H(26B)	9760	816	3770	291
H(26C)	10944	608	3598	291
H(3)	6430	262	5984	129

Table 2: Bond lengths (Å)

C(9)-C(8)	1.514(6)	C(16)-Si(2)	1.849(6)
C(9)-H(9A)	0.96	C(16)-H(16A)	0.96
C(9)-H(9B)	0.96	C(16)-H(16B)	0.96
C(9)-H(9C)	0.96	C(16)-H(16C)	0.96
C(1)-O(1)	1.378(4)	C(17)-C(18)	1.486(7)
C(1)-C(2)	1.392(5)	C(17)-C(19)	1.539(6)
C(1)-C(6)	1.388(5)	C(17)-C(20)	1.557(7)
C(2)-C(3)	1.372(5)	C(17)-Si(2)	1.854(5)
C(2)-H(2)	0.93	C(18)-H(18A)	0.96
C(3)-O(2)	1.377(4)	C(18)-H(18B)	0.96
C(3)-C(4)	1.381(5)	C(18)-H(18C)	0.96
C(4)-C(5)	1.381(5)	C(19)-H(19A)	0.96
C(4)-H(4)	0.93	C(19)-H(19B)	0.96
C(5)-C(6)	1.403(5)	C(19)-H(19C)	0.96
C(5)-C(7)	1.515(5)	C(20)-H(20A)	0.96
C(6)-C(10)	1.500(5)	C(20)-H(20B)	0.96
C(7)-O(3)	1.415(4)	C(20)-H(20C)	0.96
C(7)-C(8)	1.512(5)	C(21)-Si(1)	1.836(6)
C(7)-H(7)	0.98	C(21)-H(21A)	0.96
C(8)-H(8A)	0.97	C(21)-H(21B)	0.96
C(8)-H(8B)	0.97	C(21)-H(21C)	0.96
C(10)-O(4)	1.237(4)	C(22)-Si(1)	1.884(5)
C(10)-N(1)	1.348(4)	C(22)-H(22A)	0.96
C(11)-N(1)	1.464(5)	C(22)-H(22B)	0.96
C(11)-C(12)	1.508(6)	C(22)-H(22C)	0.96
C(11)-H(11A)	0.97	C(23)-C(24)	1.508(7)
C(11)-H(11B)	0.97	C(23)-C(25)	1.523(6)
C(12)-H(12A)	0.96	C(23)-C(26)	1.591(7)
C(12)-H(12B)	0.96	C(23)-Si(1)	1.850(5)
C(12)-H(12C)	0.96	C(24)-H(24A)	0.96
C(13)-N(1)	1.471(5)	C(24)-H(24B)	0.96
C(13)-C(14)	1.501(6)	C(24)-H(24C)	0.96
C(13)-H(13A)	0.97	C(25)-H(25A)	0.96
C(13)-H(13B)	0.97	C(25)-H(25B)	0.96
C(14)-H(14A)	0.96	C(25)-H(25C)	0.96
C(14)-H(14B)	0.96	C(26)-H(26A)	0.96
C(14)-H(14C)	0.96	C(26)-H(26B)	0.96
C(15)-Si(2)	1.874(6)	C(26)-H(26C)	0.96
C(15)-H(15A)	0.96	O(1)-Si(2)	1.658(3)
C(15)-H(15B)	0.96	O(2)-Si(1)	1.646(3)
C(15)-H(15C)	0.96	O(3)-H(3)	0.82

Table 3: Angles (°)

C(8)-C(9)-H(9A)	109.5	N(1)-C(13)-C(14)	112.1(4)	Si(1)-C(21)-H(21C)	109.5
C(8)-C(9)-H(9B)	109.5	N(1)-C(13)-H(13A)	109.2	H(21A)-C(21)-H(21C)	109.5
H(9A)-C(9)-H(9B)	109.5	C(14)-C(13)-H(13A)	109.2	H(21B)-C(21)-H(21C)	109.5
C(8)-C(9)-H(9C)	109.5	N(1)-C(13)-H(13B)	109.2	Si(1)-C(22)-H(22A)	109.5
H(9A)-C(9)-H(9C)	109.5	C(14)-C(13)-H(13B)	109.2	Si(1)-C(22)-H(22B)	109.5
H(9B)-C(9)-H(9C)	109.5	H(13A)-C(13)-H(13B)	107.9	H(22A)-C(22)-H(22B)	109.5
O(1)-C(1)-C(2)	118.9(4)	C(13)-C(14)-H(14A)	109.5	Si(1)-C(22)-H(22C)	109.5
O(1)-C(1)-C(6)	119.8(3)	C(13)-C(14)-H(14B)	109.5	H(22A)-C(22)-H(22C)	109.5
C(2)-C(1)-C(6)	121.3(4)	H(14A)-C(14)-H(14B)	109.5	H(22B)-C(22)-H(22C)	109.5
C(3)-C(2)-C(1)	119.0(4)	C(13)-C(14)-H(14C)	109.5	C(24)-C(23)-C(25)	110.3(4)
C(3)-C(2)-H(2)	120.5	H(14A)-C(14)-H(14C)	109.5	C(24)-C(23)-C(26)	108.7(5)
C(1)-C(2)-H(2)	120.5	H(14B)-C(14)-H(14C)	109.5	C(25)-C(23)-C(26)	109.2(5)
O(2)-C(3)-C(2)	119.4(4)	Si(2)-C(15)-H(15A)	109.5	C(24)-C(23)-Si(1)	111.1(4)
O(2)-C(3)-C(4)	120.2(4)	Si(2)-C(15)-H(15B)	109.5	C(25)-C(23)-Si(1)	110.3(4)
C(2)-C(3)-C(4)	120.4(4)	H(15A)-C(15)-H(15B)	109.5	C(26)-C(23)-Si(1)	107.3(4)
C(5)-C(4)-C(3)	121.4(4)	Si(2)-C(15)-H(15C)	109.5	C(23)-C(24)-H(24A)	109.5
C(5)-C(4)-H(4)	119.3	H(15A)-C(15)-H(15C)	109.5	C(23)-C(24)-H(24B)	109.5
C(3)-C(4)-H(4)	119.3	H(15B)-C(15)-H(15C)	109.5	H(24A)-C(24)-H(24B)	109.5
C(4)-C(5)-C(6)	118.8(3)	Si(2)-C(16)-H(16A)	109.5	C(23)-C(24)-H(24C)	109.5
C(4)-C(5)-C(7)	120.1(3)	Si(2)-C(16)-H(16B)	109.5	H(24A)-C(24)-H(24C)	109.5
C(6)-C(5)-C(7)	121.0(3)	H(16A)-C(16)-H(16B)	109.5	H(24B)-C(24)-H(24C)	109.5
C(1)-C(6)-C(5)	119.2(3)	Si(2)-C(16)-H(16C)	109.5	C(23)-C(25)-H(25A)	109.5
C(1)-C(6)-C(10)	119.7(3)	H(16A)-C(16)-H(16C)	109.5	C(23)-C(25)-H(25B)	109.5
C(5)-C(6)-C(10)	121.1(3)	H(16B)-C(16)-H(16C)	109.5	H(25A)-C(25)-H(25B)	109.5
O(3)-C(7)-C(8)	106.1(3)	C(18)-C(17)-C(19)	109.0(5)	C(23)-C(25)-H(25C)	109.5
O(3)-C(7)-C(5)	112.2(3)	C(18)-C(17)-C(20)	109.6(5)	H(25A)-C(25)-H(25C)	109.5
C(8)-C(7)-C(5)	111.0(3)	C(19)-C(17)-C(20)	107.8(5)	H(25B)-C(25)-H(25C)	109.5
O(3)-C(7)-H(7)	109.2	C(18)-C(17)-Si(2)	111.8(4)	C(23)-C(26)-H(26A)	109.5
C(8)-C(7)-H(7)	109.2	C(19)-C(17)-Si(2)	110.3(4)	C(23)-C(26)-H(26B)	109.5
C(5)-C(7)-H(7)	109.2	C(20)-C(17)-Si(2)	108.3(3)	H(26A)-C(26)-H(26B)	109.5
C(9)-C(8)-C(7)	114.2(4)	C(17)-C(18)-H(18A)	109.5	C(23)-C(26)-H(26C)	109.5
C(9)-C(8)-H(8A)	108.7	C(17)-C(18)-H(18B)	109.5	H(26A)-C(26)-H(26C)	109.5
C(7)-C(8)-H(8A)	108.7	H(18A)-C(18)-H(18B)	109.5	H(26B)-C(26)-H(26C)	109.5
C(9)-C(8)-H(8B)	108.7	C(17)-C(18)-H(18C)	109.5	C(10)-N(1)-C(11)	124.0(3)
C(7)-C(8)-H(8B)	108.7	H(18A)-C(18)-H(18C)	109.5	C(10)-N(1)-C(13)	118.3(3)
H(8A)-C(8)-H(8B)	107.6	H(18B)-C(18)-H(18C)	109.5	C(11)-N(1)-C(13)	117.4(3)
O(4)-C(10)-N(1)	121.5(4)	C(17)-C(19)-H(19A)	109.5	C(1)-O(1)-Si(2)	128.7(2)
O(4)-C(10)-C(6)	119.9(3)	C(17)-C(19)-H(19B)	109.5	C(3)-O(2)-Si(1)	125.2(2)
N(1)-C(10)-C(6)	118.6(3)	H(19A)-C(19)-H(19B)	109.5	C(7)-O(3)-H(3)	109.5
N(1)-C(11)-C(12)	114.3(3)	C(17)-C(19)-H(19C)	109.5	O(2)-Si(1)-C(21)	109.4(2)
N(1)-C(11)-H(11A)	108.7	H(19A)-C(19)-H(19C)	109.5	O(2)-Si(1)-C(23)	105.43(19)
C(12)-C(11)-H(11A)	108.7	H(19B)-C(19)-H(19C)	109.5	C(21)-Si(1)-C(23)	114.1(3)
N(1)-C(11)-H(11B)	108.7	C(17)-C(20)-H(20A)	109.5	O(2)-Si(1)-C(22)	108.9(2)
C(12)-C(11)-H(11B)	108.7	C(17)-C(20)-H(20B)	109.5	C(21)-Si(1)-C(22)	109.8(4)
H(11A)-C(11)-H(11B)	107.6	H(20A)-C(20)-H(20B)	109.5	C(23)-Si(1)-C(22)	109.0(3)
C(11)-C(12)-H(12A)	109.5	C(17)-C(20)-H(20C)	109.5	O(1)-Si(2)-C(16)	108.1(2)
C(11)-C(12)-H(12B)	109.5	H(20A)-C(20)-H(20C)	109.5	O(1)-Si(2)-C(17)	105.94(18)
H(12A)-C(12)-H(12B)	109.5	H(20B)-C(20)-H(20C)	109.5	C(16)-Si(2)-C(17)	113.8(3)
C(11)-C(12)-H(12C)	109.5	Si(1)-C(21)-H(21A)	109.5	O(1)-Si(2)-C(15)	107.4(3)
H(12A)-C(12)-H(12C)	109.5	Si(1)-C(21)-H(21B)	109.5	C(16)-Si(2)-C(15)	111.6(4)
H(12B)-C(12)-H(12C)	109.5	H(21A)-C(21)-H(21B)	109.5	C(17)-Si(2)-C(15)	109.6(3)

Table 4: Anisotropic displacement parameters (Å² x 10^3) The anisotropic displacement factor exponent takes the form: -2 π^2 [h² a*² U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
C(1)	54(2)	64(2)	79(3)	0(2)	0(2)	-4(2)
C(2)	62(3)	74(3)	75(3)	0(2)	12(2)	0(2)
C(3)	52(2)	69(3)	79(3)	14(2)	5(2)	1(2)
C(4)	57(2)	68(2)	78(3)	4(2)	-4(2)	-12(2)
C(5)	53(2)	58(2)	73(3)	7(2)	3(2)	0(2)
C(6)	51(2)	60(2)	71(3)	2(2)	5(2)	-2(2)
C(7)	68(3)	67(3)	79(3)	-5(2)	3(2)	-7(2)
C(8)	109(4)	78(3)	78(3)	9(2)	6(2)	-8(3)
C(9)	180(6)	119(4)	87(4)	21(3)	17(3)	-1(4)
C(10)	59(3)	66(3)	74(3)	5(2)	4(2)	-4(2)
C(11)	74(3)	69(3)	106(3)	7(2)	10(2)	1(2)
C(12)	154(5)	79(3)	126(4)	-13(3)	2(4)	14(3)
C(13)	78(3)	84(3)	135(4)	15(3)	28(3)	-17(2)
C(14)	137(5)	104(4)	130(5)	6(3)	48(4)	-10(3)
C(15)	66(3)	343(11)	185(6)	-121(6)	8(4)	18(5)
C(16)	240(8)	84(4)	234(7)	7(4)	-132(6)	24(4)
C(17)	90(3)	112(4)	81(3)	-9(3)	1(3)	-6(3)
C(18)	360(12)	96(5)	132(5)	-22(4)	11(6)	-47(6)
C(19)	166(6)	217(7)	121(4)	-37(5)	-56(4)	12(5)
C(20)	135(6)	285(10)	149(5)	-69(6)	52(4)	-17(6)
C(21)	225(8)	138(5)	275(9)	82(6)	152(7)	100(5)
C(22)	149(5)	186(6)	114(4)	45(4)	-36(4)	-35(5)
C(23)	99(4)	101(4)	118(4)	20(3)	21(3)	-30(3)
C(24)	81(4)	149(5)	218(7)	36(5)	52(4)	9(4)
C(25)	154(5)	121(4)	167(5)	33(4)	41(4)	-52(4)
C(26)	183(7)	254(9)	133(5)	-4(5)	-39(5)	-103(6)
N(1)	56(2)	66(2)	98(2)	13(2)	10(2)	-8(2)
O(1)	69(2)	77(2)	82(2)	-6(1)	-2(1)	-11(1)
O(2)	52(2)	82(2)	105(2)	24(2)	21(1)	-2(1)
O(3)	99(2)	73(2)	83(2)	-1(2)	-8(2)	-4(2)
O(4)	59(2)	77(2)	110(2)	10(2)	20(2)	8(2)
Si(1)	79(1)	92(1)	123(1)	29(1)	26(1)	9(1)
Si(2)	80(1)	99(1)	111(1)	-22(1)	-17(1)	10(1)

Table 5: Torsion angles (°)

O(1)-C(1)-C(2)-C(3)	-178.8(3)	C(12)-C(11)-N(1)-C(13)	70.1(5)
C(6)-C(1)-C(2)-C(3)	-1.6(5)	C(14)-C(13)-N(1)-C(10)	-93.3(5)
C(1)-C(2)-C(3)-O(2)	179.2(3)	C(14)-C(13)-N(1)-C(11)	80.2(5)
C(1)-C(2)-C(3)-C(4)	-0.1(5)	C(2)-C(1)-O(1)-Si(2)	-82.7(4)
O(2)-C(3)-C(4)-C(5)	-178.8(3)	C(6)-C(1)-O(1)-Si(2)	100.1(4)
C(2)-C(3)-C(4)-C(5)	0.5(5)	C(2)-C(3)-O(2)-Si(1)	85.2(4)
C(3)-C(4)-C(5)-C(6)	0.7(5)	C(4)-C(3)-O(2)-Si(1)	-95.4(4)
C(3)-C(4)-C(5)-C(7)	178.4(3)	C(3)-O(2)-Si(1)-C(21)	37.0(5)
O(1)-C(1)-C(6)-C(5)	-180.0(3)	C(3)-O(2)-Si(1)-C(23)	160.2(3)
C(2)-C(1)-C(6)-C(5)	2.8(5)	C(3)-O(2)-Si(1)-C(22)	-83.0(4)
O(1)-C(1)-C(6)-C(10)	-2.6(5)	C(24)-C(23)-Si(1)-O(2)	57.7(4)
C(2)-C(1)-C(6)-C(10)	-179.8(3)	C(25)-C(23)-Si(1)-O(2)	-179.7(4)
C(4)-C(5)-C(6)-C(1)	-2.4(5)	C(26)-C(23)-Si(1)-O(2)	-60.9(4)
C(7)-C(5)-C(6)-C(1)	180.0(3)	C(24)-C(23)-Si(1)-C(21)	177.8(4)
C(4)-C(5)-C(6)-C(10)	-179.7(3)	C(25)-C(23)-Si(1)-C(21)	-59.7(5)
C(7)-C(5)-C(6)-C(10)	2.6(5)	C(26)-C(23)-Si(1)-C(21)	59.2(5)
C(4)-C(5)-C(7)-O(3)	21.7(5)	C(24)-C(23)-Si(1)-C(22)	-59.1(4)
C(6)-C(5)-C(7)-O(3)	-160.6(3)	C(25)-C(23)-Si(1)-C(22)	63.5(5)
C(4)-C(5)-C(7)-C(8)	-96.7(4)	C(26)-C(23)-Si(1)-C(22)	-177.7(4)
C(6)-C(5)-C(7)-C(8)	81.0(4)	C(1)-O(1)-Si(2)-C(16)	15.6(5)
O(3)-C(7)-C(8)-C(9)	61.1(5)	C(1)-O(1)-Si(2)-C(17)	137.9(3)
C(5)-C(7)-C(8)-C(9)	-176.9(4)	C(1)-O(1)-Si(2)-C(15)	-105.0(4)
C(1)-C(6)-C(10)-O(4)	-104.5(4)	C(18)-C(17)-Si(2)-O(1)	59.5(5)
C(5)-C(6)-C(10)-O(4)	72.8(5)	C(19)-C(17)-Si(2)-O(1)	-179.1(4)
C(1)-C(6)-C(10)-N(1)	75.1(4)	C(20)-C(17)-Si(2)-O(1)	-61.4(4)
C(5)-C(6)-C(10)-N(1)	-107.6(4)	C(18)-C(17)-Si(2)-C(16)	178.1(5)
O(4)-C(10)-N(1)-C(11)	-175.5(4)	C(19)-C(17)-Si(2)-C(16)	-60.5(5)
C(6)-C(10)-N(1)-C(11)	4.9(5)	C(20)-C(17)-Si(2)-C(16)	57.3(5)
O(4)-C(10)-N(1)-C(13)	-2.6(6)	C(18)-C(17)-Si(2)-C(15)	-56.1(6)
C(6)-C(10)-N(1)-C(13)	177.9(3)	C(19)-C(17)-Si(2)-C(15)	65.3(5)
C(12)-C(11)-N(1)-C(10)	-116.9(4)	C(20)-C(17)-Si(2)-C(15)	-177.0(4)

Table 6: Calculated reflections from PowderCell*

h	k	l	2θ/°	d/Å	I/rel.	|F(hkl)|	h	k	l	2θ/°	d/Å	I/rel.	|F(hkl)|
0	0	1	6.98	12.65	94.58	130.82	2	3	0	19.61	4.52	21.64	126.04
0	1	1	8.27	10.68	75.71	98.12	-1	3	2	20.13	4.41	21.92	130.36
1	1	0	8.41	10.50	20.98	52.53	1	4	1	20.71	4.29	5.99	70.19
0	2	0	8.87	9.97	2.53	27.19	0	0	3	21.06	4.22	2.61	66.64
1	2	0	11.40	7.76	4.13	31.65	2	0	2	21.14	4.20	11.93	143.07
1	1	1	11.42	7.74	57.00	117.90	2	3	1	21.36	4.16	6.28	74.19
-1	2	1	12.97	6.82	62.50	140.38	2	1	2	21.61	4.11	3.32	54.61
0	0	2	13.99	6.32	3.26	48.97	-1	1	3	21.97	4.04	2.24	45.67
2	0	0	14.33	6.18	11.40	93.80	3	1	0	22.02	4.03	6.93	80.45
0	1	2	14.69	6.03	5.41	46.84	0	4	2	22.70	3.91	2.87	53.41
2	1	0	15.00	5.90	17.39	85.85	2	2	2	22.97	3.87	13.17	115.87
1	3	0	15.13	5.85	100.00	207.68	-1	2	3	23.31	3.81	5.04	72.81
-2	0	1	15.26	5.80	25.58	149.85	0	5	1	23.38	3.80	8.51	94.88
-1	1	2	15.67	5.65	13.68	79.64	-3	2	1	23.72	3.75	2.95	56.72
-2	1	1	15.90	5.57	16.41	88.49	3	1	1	23.83	3.73	16.99	136.80
-1	3	1	16.35	5.42	7.70	62.40	-2	0	3	24.23	3.67	3.23	85.81
0	2	2	16.59	5.34	51.55	163.85	1	2	3	24.69	3.60	4.22	70.73
2	0	1	16.62	5.33	9.91	101.76	1	5	1	24.70	3.60	2.04	49.22
2	2	0	16.87	5.25	24.91	115.86	3	2	1	25.07	3.55	2.37	53.87
1	1	2	17.00	5.21	10.89	77.23	0	5	2	26.41	3.37	2.12	53.84
1	3	1	17.00	5.21	10.80	76.89	1	3	3	26.66	3.34	4.09	75.51
-1	2	2	17.47	5.07	3.68	46.18	2	0	3	26.83	3.32	2.32	80.97
-2	2	1	17.67	5.01	2.15	35.70	-1	5	2	26.99	3.30	2.94	64.89
2	2	1	18.87	4.70	21.87	121.77	2	1	3	27.21	3.27	4.17	77.97
0	4	1	19.13	4.64	3.69	50.71	0	6	1	27.74	3.21	2.54	62.03
1	4	0	19.19	4.62	21.48	122.84	0	0	4	28.20	3.16	2.15	82.22
0	3	2	19.36	4.58	5.66	63.65	-3	4	1	28.39	3.14	2.01	56.62
-2	1	2	19.48	4.55	2.55	42.97							

Source: Cu-$K_{\alpha 1}$ (λ = 1.540598 Å)
Condition on reflections: I ≥ 2
Range (2θ): From 3° to 30°
*PowderCell for Windows (version 2.4) by Kraus W. & Nolze G., Federal institute for materials Research and testing, Rudower Chausse 5, 12489 Berlin Germany.

4,6-Dichloro-3-ethyl-5,7-dihydroxy-*iso*-benzofuran-1(3*H*)-one 1 – Spiromastilactone A

SAMPLE PREPARATION

In a little flask, about 10 mg of pure Spiromastilactone A (1) was solubilized in chloroform. A cap with holes was added to the flask to permit a slow evaporation of the solvent. After few days crystals grew up and were analyzed by X-ray diffraction.

DATA COLLECTION

The crystal structure of Spiromastilactone A (**1**) [$C_{10}H_8Cl_2O_4$] has been determined from single crystal X-Ray diffraction. The chosen crystal was stuck on a glass fibre and mounted on the full three-circle goniometer of a Bruker SMART APEX diffractometer with a CCD area detector. Three sets of exposures (a total of 1800 frames) were recorded, corresponding to three ω scans (steps of 0.3°), for three different values of ϕ. The details of data collection are given in annexe 1.

The cell parameters and the orientation matrix of the crystal were preliminary determined by using SMART Software[1]. Data integration and global cell refinement were performed with SAINT Software[2]. Intensities were corrected for Lorentz, polarisation, decay and absorption effects (SAINT and SADABS Softwares) and reduced to F_O^2. The program package WinGX[3] was used for space group determination, structure solution and refinement.

DATA REFINEMENT

The non-standard space group $P2_1/a$(n°14) was determined from systematic extinctions and relative F_O^2 of equivalent reflections. The structure was solved by direct methods[4]. Anisotropic displacement parameters were refined for all non-hydrogen atoms. Every Hydrogen atoms were located from subsequent difference Fourier syntheses and placed with geometrical constraints (SHELXL[5]). The final cycle of full-matrix least-square refinement on F^2 was based on 2153 observed reflections and 148 variable parameters and converged with unweighted and weighted agreement factors of:
R1 = 0.0388, wR2 = 0.0986 for 1714 reflections with $I>2\sigma I$ and R1 = 0.0501, wR2 = 0.1066 for all data.

The refinement data are given in annexe 3 table 2

CRYSTALLOGRAPHIC DATA AND STRUCTURAL DESCRIPTION

Crystallographic data

The crystal data are collected in Table 1. The full crystallographic parameters (atomic coordinates, bond length, angles and anisotropic displacements) are reported in annexe 4.

Table 1: Crystal data

Chemical Formula	$C_{10}H_8Cl_2O_4$
Molecular Weight / *g.mol*$^{-1}$	263.06
Crystal System	Monoclinic
Space Group	$P2_1/a$
Z , Z' (asymmetric units per unit cell)	4, 1
a / *Å*	7.191(2)
b / *Å*	17.064(4)
c / *Å*	8.726(2)
α / °	90
β / °	100.039(4)
γ / °	90
V / *Å*^{3}	1054.3(4)
d_{calc} / *g.cm*$^{-3}$	1.657
F(000) / *e*$^-$	536
Absorption coefficient μ (MoKα_1) / *mm*$^{-1}$	0.609

Structural description

The asymmetric unit is composed of one molecule of Spiromastilactone A (**1**), [$C_{10}H_8Cl_2O_4$] (Figure 1). The molecules establish hydrogen bonds between adjacent molecules that give rise to molecular bond chains spreading along b (Figures 2, Table 2). These chains are interacting along a by means of ππ interactions (d~3.6Å) (Figures 3). These lead to molecular layers in ab, of d_{001} thickness (Figures 4).

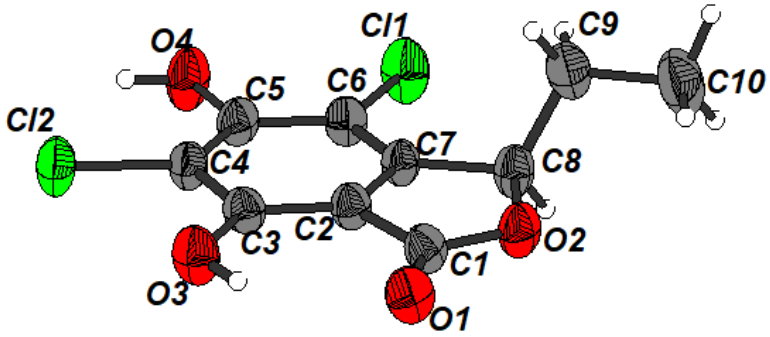
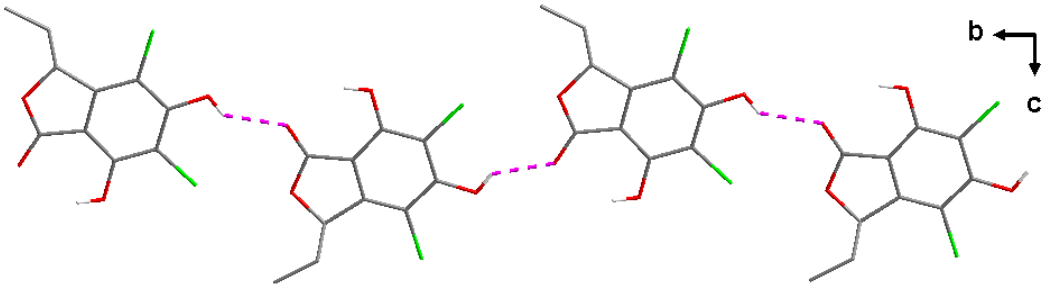
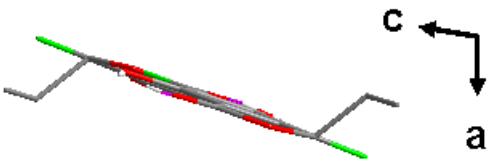


Figure 1: Asymmetric unit in thermal ellipsoidal representation



2a-Molecular chain formed by the hydrogen bond interaction (dashed pink lines) with adjacent molecules along b.

2b- Projection along c of one molecular chain

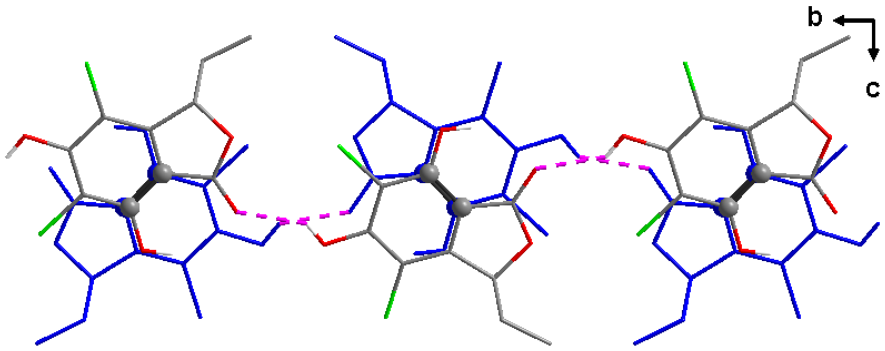


2c- Projection along b of one molecular chain
Figure 2: Projections of one molecular chain

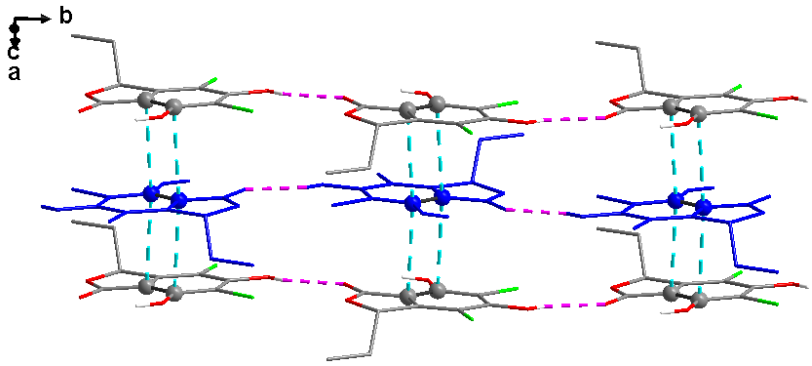
Table 2: Hydrogen bond table

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)	
O(3)-H(3)...O(1)	0.82	2.27	2.951(2)	141.4	Intramolecular
O(4)-H(4)...O(1)#1	0.82	2.03	2.690(2)	136.8	Intermolecular

Symmetry transformations used to generate equivalent atoms: #1 -x+1/2,y-1/2,-z+1

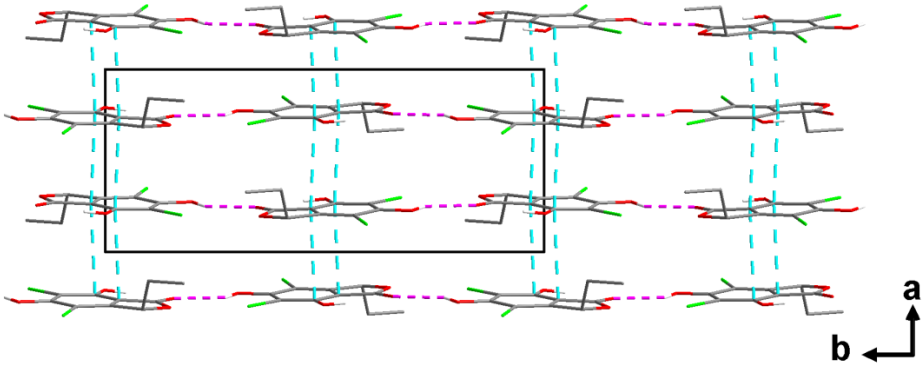


3a- Consecutive molecular chains along a, one molecular chain is displayed in blue. Ball atoms highlight the shortest distance between molecular chains (d~3.6Å).

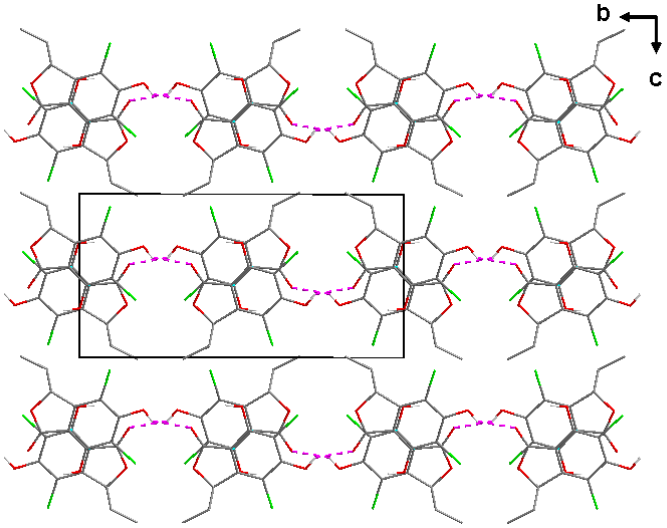


3b- The ππ interaction is displayed in dashed pink lines. Ball atoms highlight the shortest atomic distance (d~3.6Å).

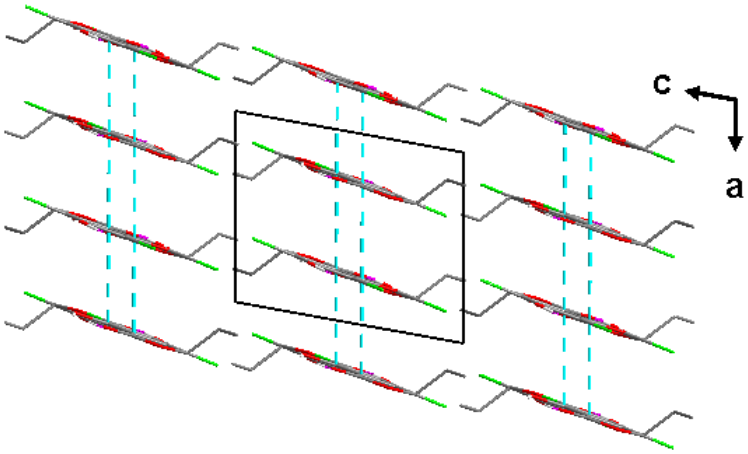
Figure 3: Projections of one molecular layer



4a- Projection along c



4b- Projection along a



4c-Projection along b

Figure 4: Projections of the crystal packing

ANNEXE 3 :

- Table 1 : DATA COLLECTION FOR SPIROMASTILACTONE A (1)

Date	05/11/18
Temperature / *K*	RT
Radiation	Mo-Kα_1 (λ = 0.71073 Å)
Monochromator	Graphite
Collimator / mm	0.5
Generator set	50 kV 40mA
Crystal-detector distance / *mm*	60
Detector 2θ angle / °	-28
ω oscillations / °	-0.3
ω scan 1	χ = 54.7°, ϕ = 0°, -28° ≤ ω ≤ -208°
ω scan 2	χ = 54.7°, ϕ = 120°, -28° ≤ ω ≤ -208°
ω scan 3	χ = 54.7°, ϕ = 240°, -28° ≤ ω ≤ -208°
Time exposure / s	10
Total number of reflections	8325
Unique reflections [F_o > 4.0 $\sigma(F_o)$]	2153 / 1714
θ range / °	2.37 to 26.41
hkl range	-8≤h≤8, -21≤k≤21, -10≤l≤10
$R_{int} = \Sigma[\|F_O^2 - F_O^2(mean)\|] / \Sigma[F_O^2]$	0.0344
Completeness to θ = 26.40 / %	100

- Table 2 : REFINEMENT DATA FOR Spiromastilactone A (1)

Number of reflections (n) (with $F_O > 4.0\ \sigma(F_O)$)	1714
Number of refined parameters (p) / restraints	148 / 0
Final R indices [I>2sigma(I)]	R1 = 0.0388, wR2 = 0.0986
R indices (all data)	R1 = 0.0501, wR2 = 0.1066
Goodness of Fit indicator (Restrained GooF)	1.039
Maximum peak in Final Difference Map / $e^{-}Å^{-3}$	0.304
Maximum hole in Final Difference Map / $e^{-}Å^{-3}$	-0.241

$R_1 = \Sigma\ (||F_O|-|F_C||) / \Sigma\ |F_O|$

$wR_2 = [\ \Sigma\ [\ w\ (F_O^2 - F_C^2)^{\ 2}] / \Sigma\ [\ w\ (F_O^2)^2\ ]\ ]^{1/2}$

$GooF = [\Sigma\ [\ w\ (F_O^2 - F_C^2)^{\ 2}] / (n - p)\ ]^{1/2}$

ANNEXE 4 : CRYSTALLOGRAPHIC DATA FOR SPIROMASTILACTONE A (1)

Table 1a: Atomic coordinates ($x10^4$) and equivalent isotropic displacement parameters ($Å^2$ x 10^3). U(eq) is defined as one third of the trace of the orthogonalized Uij tensor

	x	y	z	U(eq)
C(1)	2278(3)	6164(1)	5410(2)	39(1)
C(2)	2369(3)	5319(1)	5558(2)	34(1)
C(3)	2647(3)	4773(1)	4437(2)	35(1)
C(4)	2637(3)	3991(1)	4860(2)	36(1)
C(5)	2313(3)	3762(1)	6341(2)	36(1)
C(6)	2009(3)	4336(1)	7424(2)	36(1)
C(7)	2065(3)	5114(1)	7021(2)	34(1)
C(8)	1862(3)	5855(1)	7915(2)	39(1)
C(9)	3408(4)	5991(1)	9295(3)	49(1)
C(10)	3276(4)	6781(2)	10069(3)	67(1)
Cl(1)	1515(1)	4050(1)	9208(1)	54(1)
Cl(2)	2979(1)	3273(1)	3546(1)	51(1)
O(1)	2446(2)	6555(1)	4283(2)	49(1)
O(2)	1953(2)	6471(1)	6755(2)	43(1)
O(3)	2903(3)	4981(1)	3002(2)	49(1)
O(4)	2262(3)	3018(1)	6798(2)	51(1)

Table 1b: Hydrogen coordinates ($x10^4$) and equivalent isotropic displacement parameters ($Å^2$ x 10^3). U(eq) is defined as one third of the trace of the orthogonalized Uij tensor

	x	y	z	U(eq)
H(8)	628	5865	8246	47
H(9A)	4621	5954	8956	59
H(9B)	3356	5581	10055	59
H(10A)	3404	7191	9342	100
H(10B)	4268	6825	10957	100
H(10C)	2074	6826	10397	100
H(3)	2793	5457	2902	73
H(4)	2496	2729	6106	76

Table 2: Bond lengths (Å)

C(1)-O(1)	1.212(2)
C(1)-O(2)	1.342(2)
C(1)-C(2)	1.448(3)
C(2)-C(7)	1.378(3)
C(2)-C(3)	1.389(3)
C(3)-O(3)	1.344(2)
C(3)-C(4)	1.386(3)
C(4)-C(5)	1.408(3)
C(4)-Cl(2)	1.724(2)
C(5)-O(4)	1.333(2)
C(5)-C(6)	1.405(3)
C(6)-C(7)	1.376(3)
C(6)-Cl(1)	1.726(2)
C(7)-C(8)	1.506(3)
C(8)-O(2)	1.469(2)
C(8)-C(9)	1.508(3)
C(8)-H(8)	0.98
C(9)-C(10)	1.517(3)
C(9)-H(9A)	0.97
C(9)-H(9B)	0.97
C(10)-H(10A)	0.96
C(10)-H(10B)	0.96
C(10)-H(10C)	0.96
O(3)-H(3)	0.82
O(4)-H(4)	0.82

Table 3: Angles (°)

O(1)-C(1)-O(2)	123.45(19)
O(1)-C(1)-C(2)	127.70(19)
O(2)-C(1)-C(2)	108.84(17)
C(7)-C(2)-C(3)	123.24(18)
C(7)-C(2)-C(1)	108.84(17)
C(3)-C(2)-C(1)	127.89(19)
O(3)-C(3)-C(4)	120.62(18)
O(3)-C(3)-C(2)	122.63(18)
C(4)-C(3)-C(2)	116.74(18)
C(3)-C(4)-C(5)	121.39(19)
C(3)-C(4)-Cl(2)	119.97(16)
C(5)-C(4)-Cl(2)	118.63(15)
O(4)-C(5)-C(6)	116.53(18)
O(4)-C(5)-C(4)	123.80(18)
C(6)-C(5)-C(4)	119.67(18)
C(7)-C(6)-C(5)	119.05(18)
C(7)-C(6)-Cl(1)	121.54(16)
C(5)-C(6)-Cl(1)	119.39(15)
C(6)-C(7)-C(2)	119.89(18)
C(6)-C(7)-C(8)	131.87(18)
C(2)-C(7)-C(8)	108.24(17)
O(2)-C(8)-C(7)	102.91(15)
O(2)-C(8)-C(9)	108.93(17)
C(7)-C(8)-C(9)	114.77(18)
O(2)-C(8)-H(8)	110
C(7)-C(8)-H(8)	110
C(9)-C(8)-H(8)	110
C(8)-C(9)-C(10)	113.5(2)
C(8)-C(9)-H(9A)	108.9
C(10)-C(9)-H(9A)	108.9
C(8)-C(9)-H(9B)	108.9
C(10)-C(9)-H(9B)	108.9
H(9A)-C(9)-H(9B)	107.7
C(9)-C(10)-H(10A)	109.5
C(9)-C(10)-H(10B)	109.5
H(10A)-C(10)-H(10B)	109.5
C(9)-C(10)-H(10C)	109.5
H(10A)-C(10)-H(10C)	109.5
H(10B)-C(10)-H(10C)	109.5
C(1)-O(2)-C(8)	111.02(15)
C(3)-O(3)-H(3)	109.5
C(5)-O(4)-H(4)	109.5

Table 4: Anisotropic displacement parameters (Å² x 10^3) The anisotropic displacement factor exponent takes the form: $-2\pi^2$ [h² a*² U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
C(1)	48(1)	29(1)	37(1)	0(1)	6(1)	-4(1)
C(2)	43(1)	26(1)	34(1)	-1(1)	8(1)	-2(1)
C(3)	41(1)	34(1)	30(1)	0(1)	8(1)	-1(1)
C(4)	48(1)	27(1)	34(1)	-3(1)	10(1)	0(1)
C(5)	47(1)	23(1)	38(1)	1(1)	8(1)	0(1)
C(6)	48(1)	29(1)	32(1)	2(1)	12(1)	-1(1)
C(7)	42(1)	27(1)	33(1)	-2(1)	10(1)	-1(1)
C(8)	56(1)	26(1)	37(1)	-1(1)	16(1)	-1(1)
C(9)	66(2)	41(1)	39(1)	-6(1)	9(1)	-2(1)
C(10)	88(2)	51(2)	61(2)	-23(1)	7(2)	-9(1)
Cl(1)	86(1)	41(1)	40(1)	6(1)	27(1)	-2(1)
Cl(2)	74(1)	36(1)	45(1)	-11(1)	18(1)	4(1)
O(1)	74(1)	31(1)	42(1)	8(1)	11(1)	-3(1)
O(2)	64(1)	25(1)	43(1)	0(1)	13(1)	1(1)
O(3)	76(1)	40(1)	35(1)	4(1)	18(1)	0(1)
O(4)	87(1)	24(1)	46(1)	2(1)	22(1)	2(1)

Table 5: Torsion angles (°)

O(1)-C(1)-C(2)-C(7)	-178.7(2)
O(2)-C(1)-C(2)-C(7)	0.9(2)
O(1)-C(1)-C(2)-C(3)	-0.8(4)
O(2)-C(1)-C(2)-C(3)	178.7(2)
C(7)-C(2)-C(3)-O(3)	178.57(19)
C(1)-C(2)-C(3)-O(3)	1.0(3)
C(7)-C(2)-C(3)-C(4)	-1.0(3)
C(1)-C(2)-C(3)-C(4)	-178.6(2)
O(3)-C(3)-C(4)-C(5)	-178.0(2)
C(2)-C(3)-C(4)-C(5)	1.6(3)
O(3)-C(3)-C(4)-Cl(2)	0.6(3)
C(2)-C(3)-C(4)-Cl(2)	-179.75(16)
C(3)-C(4)-C(5)-O(4)	179.0(2)
Cl(2)-C(4)-C(5)-O(4)	0.3(3)
C(3)-C(4)-C(5)-C(6)	-0.6(3)
Cl(2)-C(4)-C(5)-C(6)	-179.26(17)
O(4)-C(5)-C(6)-C(7)	179.3(2)
C(4)-C(5)-C(6)-C(7)	-1.1(3)
O(4)-C(5)-C(6)-Cl(1)	-2.3(3)
C(4)-C(5)-C(6)-Cl(1)	177.32(16)
C(5)-C(6)-C(7)-C(2)	1.6(3)
Cl(1)-C(6)-C(7)-C(2)	-176.74(16)
C(5)-C(6)-C(7)-C(8)	-177.8(2)
Cl(1)-C(6)-C(7)-C(8)	3.8(3)
C(3)-C(2)-C(7)-C(6)	-0.6(3)
C(1)-C(2)-C(7)-C(6)	177.44(19)
C(3)-C(2)-C(7)-C(8)	179.0(2)
C(1)-C(2)-C(7)-C(8)	-3.0(2)
C(6)-C(7)-C(8)-O(2)	-176.7(2)
C(2)-C(7)-C(8)-O(2)	3.9(2)
C(6)-C(7)-C(8)-C(9)	65.1(3)
C(2)-C(7)-C(8)-C(9)	-114.3(2)
O(2)-C(8)-C(9)-C(10)	59.2(3)
C(7)-C(8)-C(9)-C(10)	174.0(2)
O(1)-C(1)-O(2)-C(8)	-178.6(2)
C(2)-C(1)-O(2)-C(8)	1.8(2)
C(7)-C(8)-O(2)-C(1)	-3.4(2)
C(9)-C(8)-O(2)-C(1)	118.79(19)

Table 6: Calculated reflections from PowderCell*

h	k	l	2θ/°	d/Å	I/rel.	|F(hkl)|
1	2	1	20.42	4.35	3.52	30.83
0	4	0	20.81	4.27	19.20	103.80
0	1	2	21.31	4.17	4.77	37.52
-1	1	2	22.85	3.89	10.03	58.49
2	0	0	25.14	3.54	100.00	288.61
-1	4	1	25.60	3.48	4.47	43.97
0	3	2	25.97	3.43	2.79	35.29
-2	1	1	26.00	3.42	12.16	73.77
1	1	2	26.57	3.35	3.50	40.49
1	4	1	27.35	3.26	3.62	42.45
1	2	2	28.10	3.17	2.37	35.37
2	0	1	28.88	3.09	40.20	211.99
1	5	0	29.02	3.07	3.37	43.61
2	1	1	29.36	3.04	5.34	55.60
0	4	2	29.48	3.03	4.88	53.39
2	3	0	29.70	3.01	2.25	36.57
-2	3	1	29.99	2.98	5.73	58.90
-1	5	1	30.10	2.97	4.35	51.56
-1	4	2	30.64	2.92	10.43	81.34
0	0	3	31.20	2.86	7.05	96.42
1	5	1	31.62	2.83	9.72	81.22
2	4	0	32.85	2.72	2.36	41.72
0	2	3	32.96	2.72	2.37	41.95
2	3	1	32.97	2.71	2.50	43.08
0	6	1	33.15	2.70	12.50	96.97
-1	2	3	33.31	2.69	6.75	71.61
2	1	2	35.93	2.50	2.13	43.73
-2	0	3	36.73	2.44	3.66	83.02
2	6	0	40.66	2.22	3.04	59.80
2	6	1	43.21	2.09	2.87	62.20
-2	6	2	43.82	2.06	3.65	71.32
0	6	3	44.88	2.02	4.14	78.00
-3	4	2	45.71	1.98	2.12	57.04
-3	2	3	46.59	1.95	3.04	69.72
3	5	0	46.76	1.94	2.06	57.71
1	2	4	47.50	1.91	2.03	58.22
4	0	1	54.69	1.68	5.55	159.87

Source: Cu-$K_{\alpha 1}$ (λ = 1.540598 Å)
Condition on reflections: I ≥ 2
Range (2θ): From 3° to 30°
*PowderCell for Windows (version 2.4) by Kraus W. & Nolze G., Federal institute for materials Research and testing, Rudower Chausse 5, 12489 Berlin Germany.

Sofwares :

(1)- SMART for WNT/2000 V5.622 (2001), Smart software reference manual, Bruker Advanced X Ray Solutions, Inc., Madison, Wisconsin, USA.

(2)- SAINT+ V6.02 (1999), Saint software reference manual, Bruker Advanced X Ray Solutions, Inc., Madison, Wisconsin, USA.

(3)-WinGX: Version 1.70.01: An integrated system of Windows Programs for the solution, refinement and analysis of Single Crystal X-Ray Diffraction Data, By LouisJ. Farrugia, Dept. of chemistry, University of Glasgow. L. J. Farrugia (1999) J. Appl. Cryst. 32, 837-838.

(4)-include in WinGX suite : SIR 92: A. Altomare, G. Cascarano, & A. Gualardi (1993) J. Appl. Cryst. 26, 343-350; SHELXS-97: Sheldrick, G. M., (1990) Acta cryst, A46, 467.

(5)-include in WinGX suite: SHELXL-97 – a program for crystal structure refinement, G. M. .Sheldrick, University of Goettingen, Germany, 1997, release 97-2.

(6)-PowderCell for Windows (version 2.4) by Kraus W. & Nolze G., Federal institute for materials Research and testing, Rudower Chausse 5, 12489 Berlin Germany.