

Supporting Information

Inhibition of cancer stem-like cells by curcumin and other polyphenol derivatives in TNBC cells

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1. Supplementary materials of the biological assays

1.1. Western blot antibodies

Table S1. Western Blot Antibodies

<i>Antibody</i>	<i>Molecular Weight (kDa)</i>	<i>Blocking solution</i>	<i>Dilution</i>	<i>Source</i>	<i>Commercial</i>
<i>CD24 (ab179821) (EPR3006(N))</i>	35-50	5% of Milk in TBS-T	1:1000	Rabbit	Abcam ©
<i>CD44 (ab243894)(BLR038F)</i>	81		1:1000		
<i>LC3B (ab48394)</i>	13-15		1:15000		
<i>ALDH1A1 (D9Q8E) XP®</i>	55	5% of BSA in TBS-T	1:1000	Rabbit	Cell Signaling Technology®
<i>Notch (Val11744) (D3B8)</i>	110		1:1000		
<i>Shh (C9C5)</i>	19		1:1000		
<i>β-Catenin (6B3)</i>	92	% of Milk in TBS-T	1:1000	Rabbit	Cell Signaling Technology®
<i>GAPDH (D16H11)</i>	37		1:2500		
<i>PARP</i>	89-116		1:1000		
<i>Phospho-β-Catenin (Ser552) (D8E11)</i>	92		1:1000		

1.2. Cytotoxicity of DMSO, TL3 and TL4

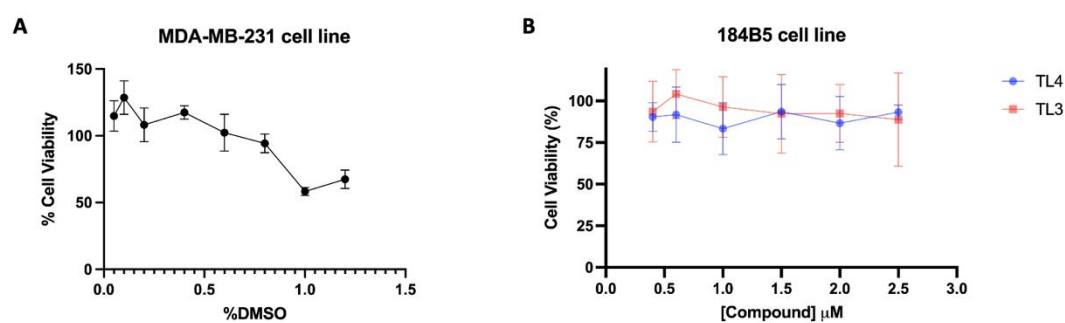


Figure S1. (A) Cell viability of MDA-MB-231 cells treated with 0.05 to 1.20% DMSO for 72 hours. **(B)** Cell viability of non-tumoral 184B5 breast epithelial cells treated with TL3 (from 0.4 to 2.5 μ M) and TL4 (from 0.4 to 2.5 μ M) for 72 hours.

1.3. Cell viability of MDA-MB-231 treated cells

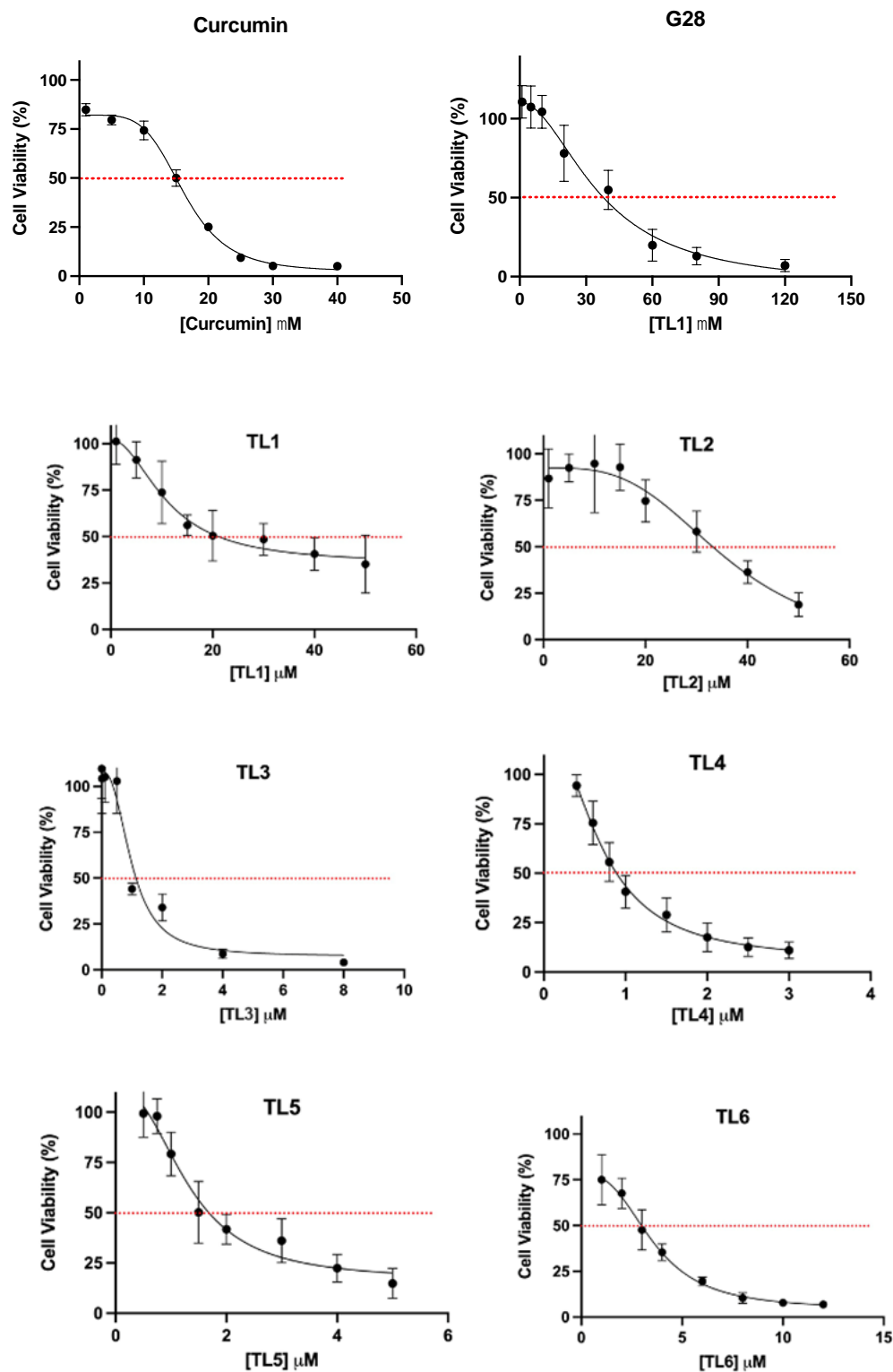


Figure S2. Cell viability of MDA-MB-231 cells treated with curcumin (from 1 to 40 μ M), G28 (from 1 to 120 μ M), TL1 (from 1 to 50 μ M), TL2 (from 1 to 50 μ M), TL3 (from 0.001 to 8 μ M), TL4 (from 0.4 to 3 μ M), TL5 (from 0.5 to 5 μ M), and TL6 (from 1 to 12 μ M) for 72 hours. Results are expressed as the percentage of surviving cells (mean \pm SE) compared to control (untreated cells) from at least three independent experiments.

1.4. Western blot results

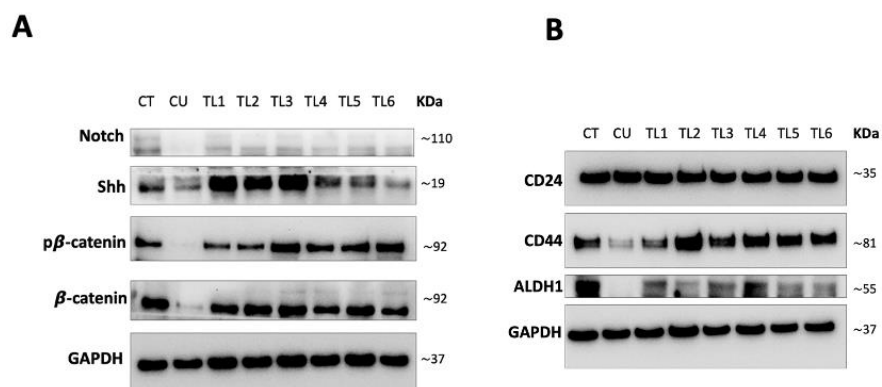
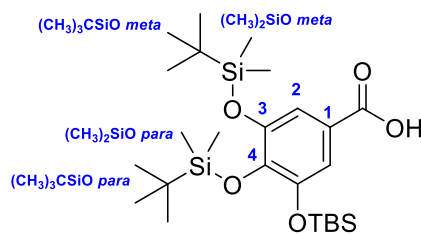


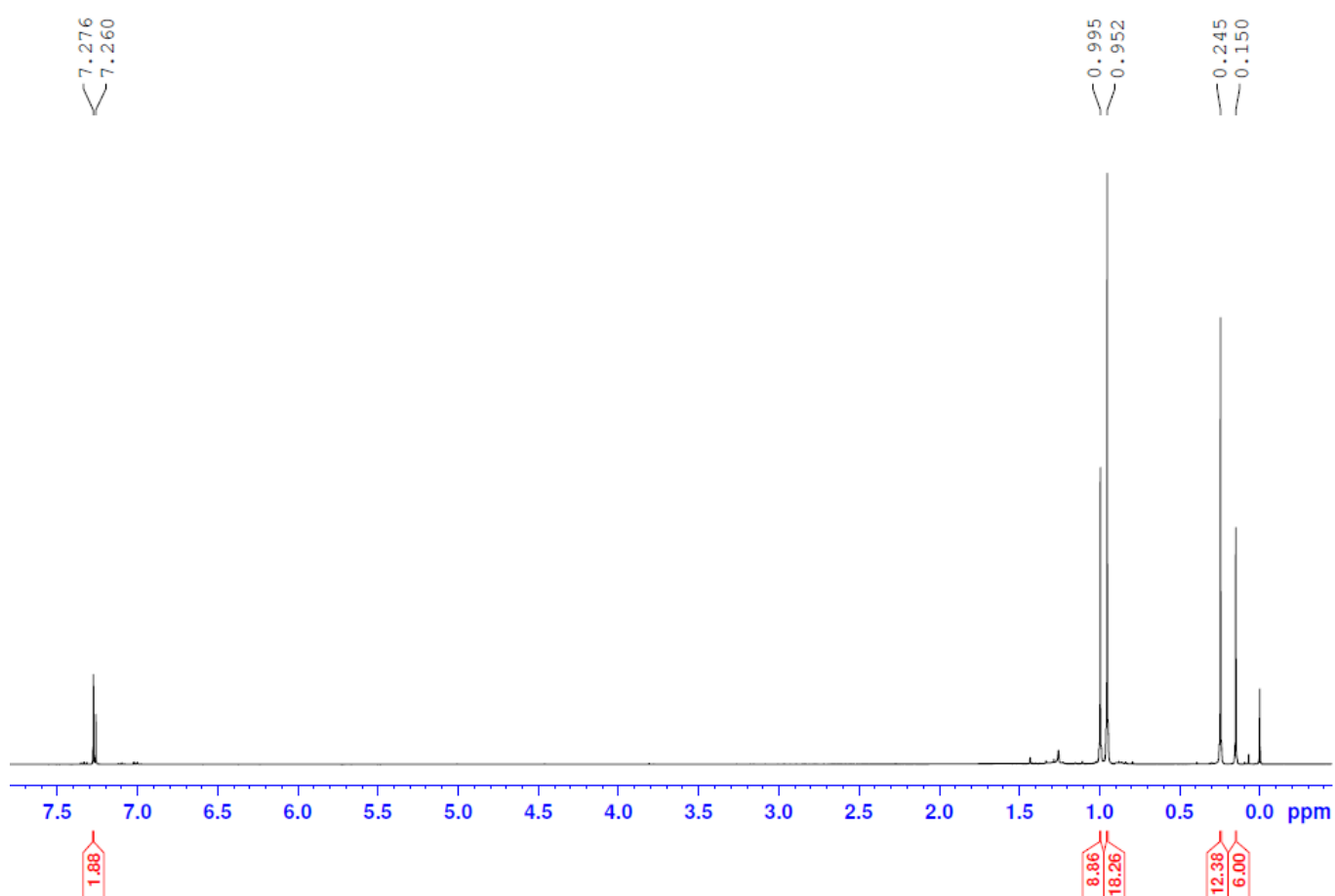
Figure S3. (A) Representative western blot images of proteins from embryonic signaling pathways are shown from a total of four replicates. **(B)** Representative western blot images of proteins from stemness markers are shown from a total of four replicates.

2. Spectroscopic data (NMR, IR, UV-VIS, HPLC, ESI-MS and HRMS) of the compounds synthesized

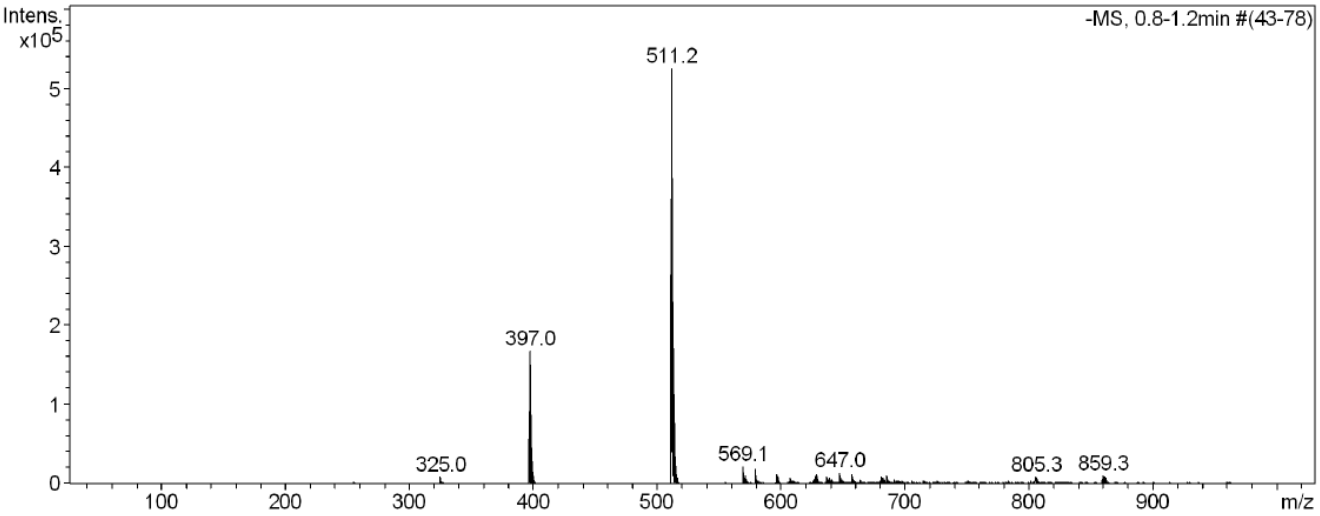
2.1. 3,4,5-Tris(*tert*-butyldimethylsilyloxy)benzoic acid



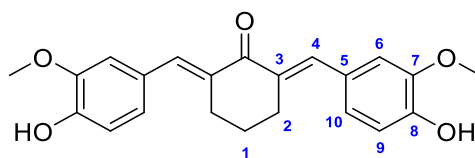
¹H-NMR



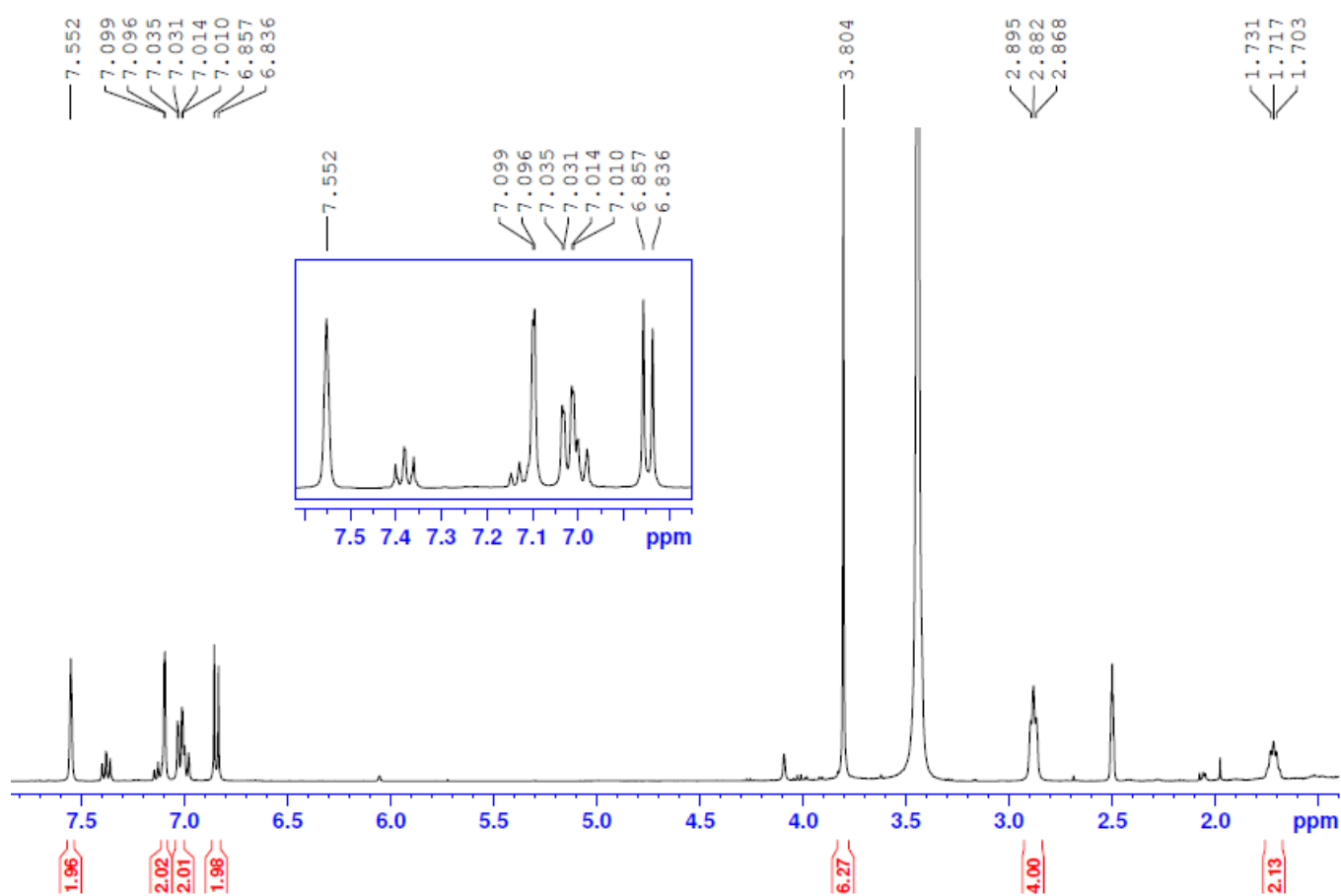
(-)-ESI-MS (*m/z*)



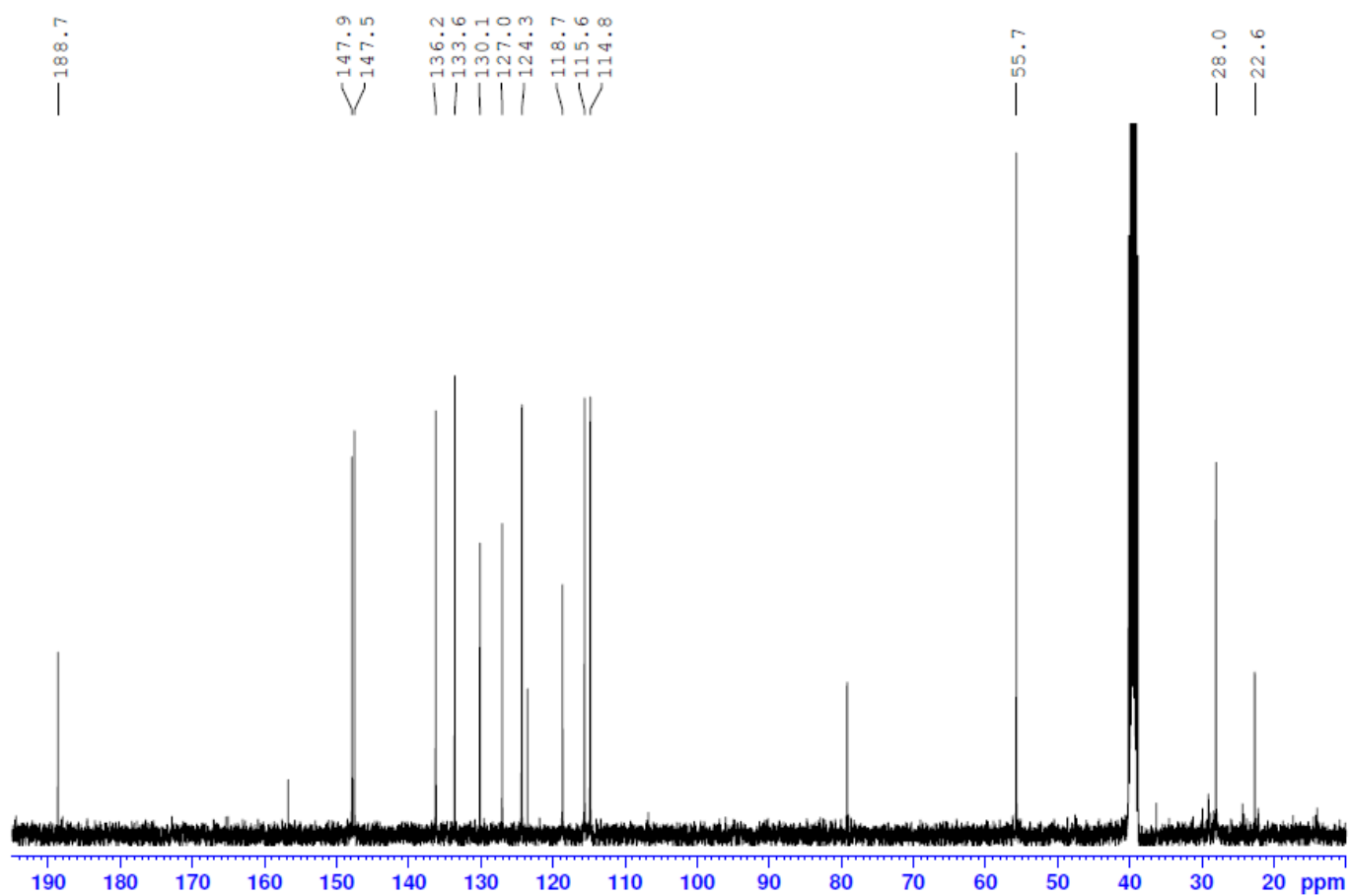
2.2. 2,6-Bis((*E*)-4-hydroxy-3-methoxybenzylidene)cyclohexan-1-one (1)



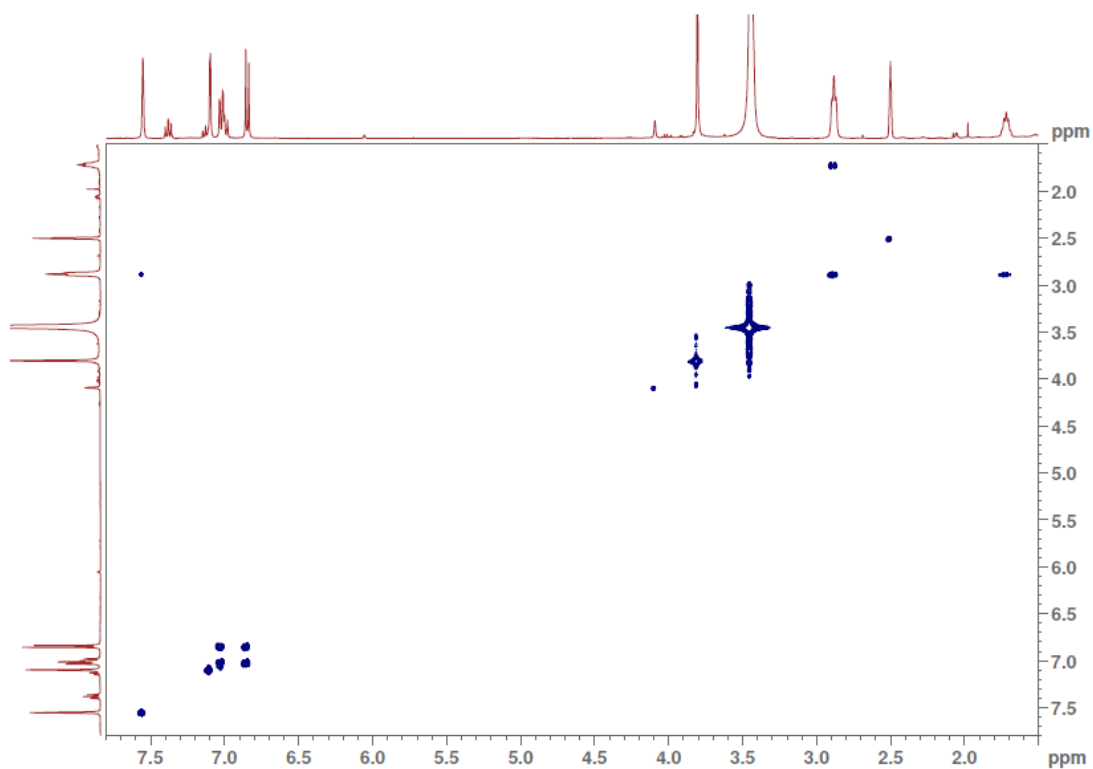
$^1\text{H-NMR}$ (400 MHz, $(\text{CD}_3)_2\text{SO}$), δ (ppm)



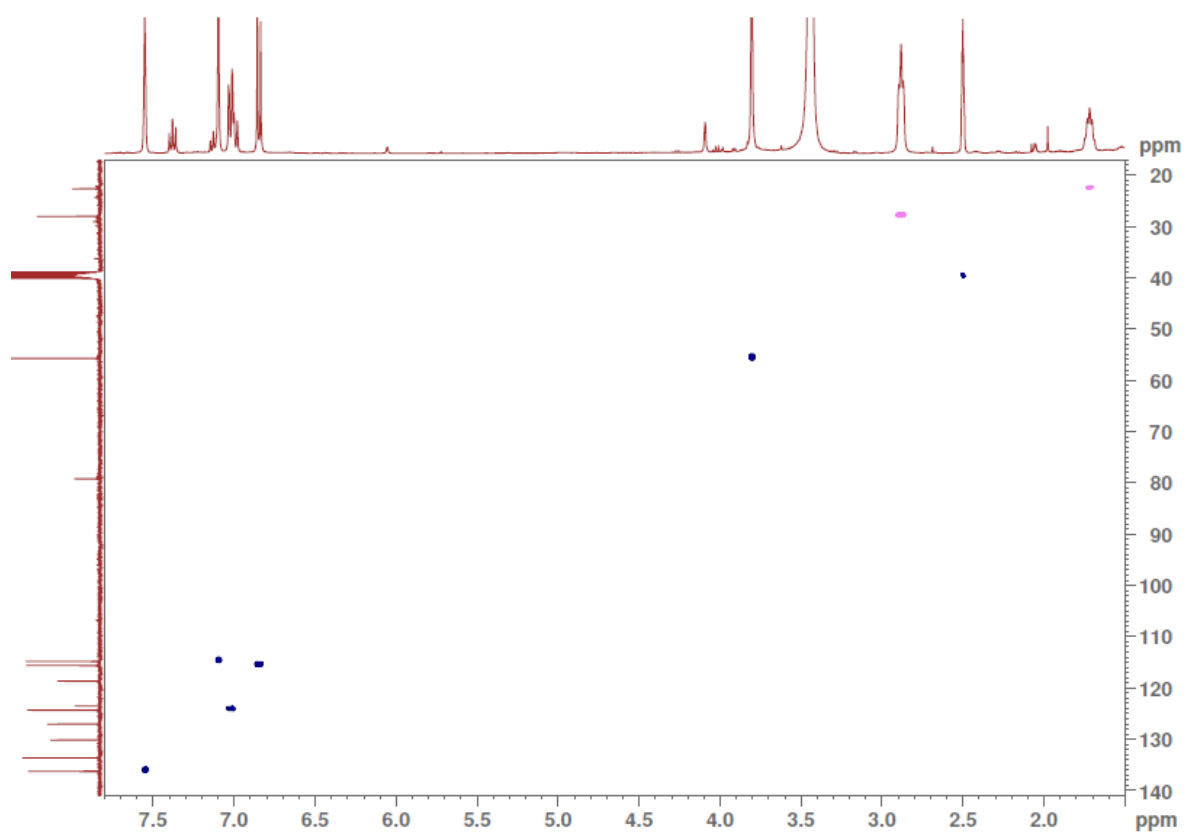
^{13}C -NMR (100 MHz, $(\text{CD}_3)_2\text{SO}$), δ (ppm)



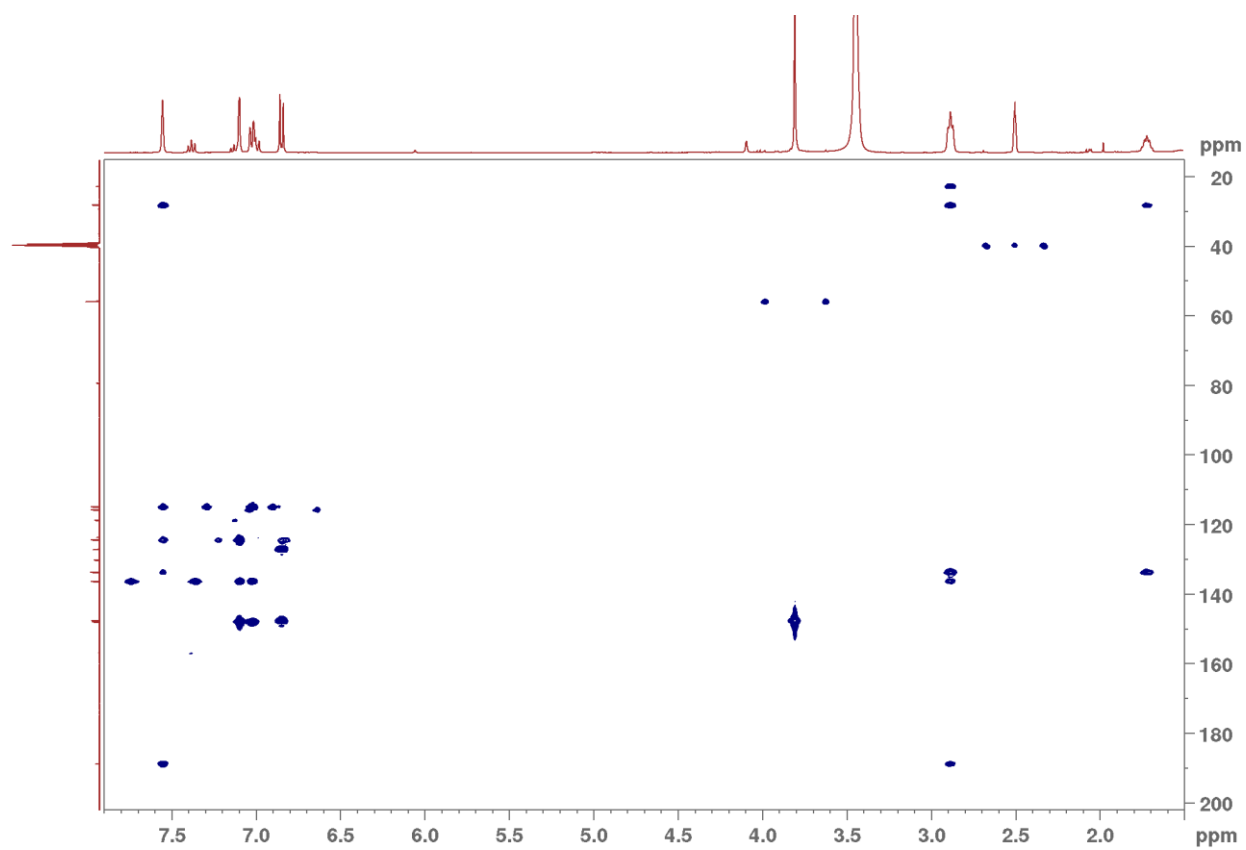
COSY ^1H - ^1H ($(\text{CD}_3)_2\text{SO}$), δ (ppm)



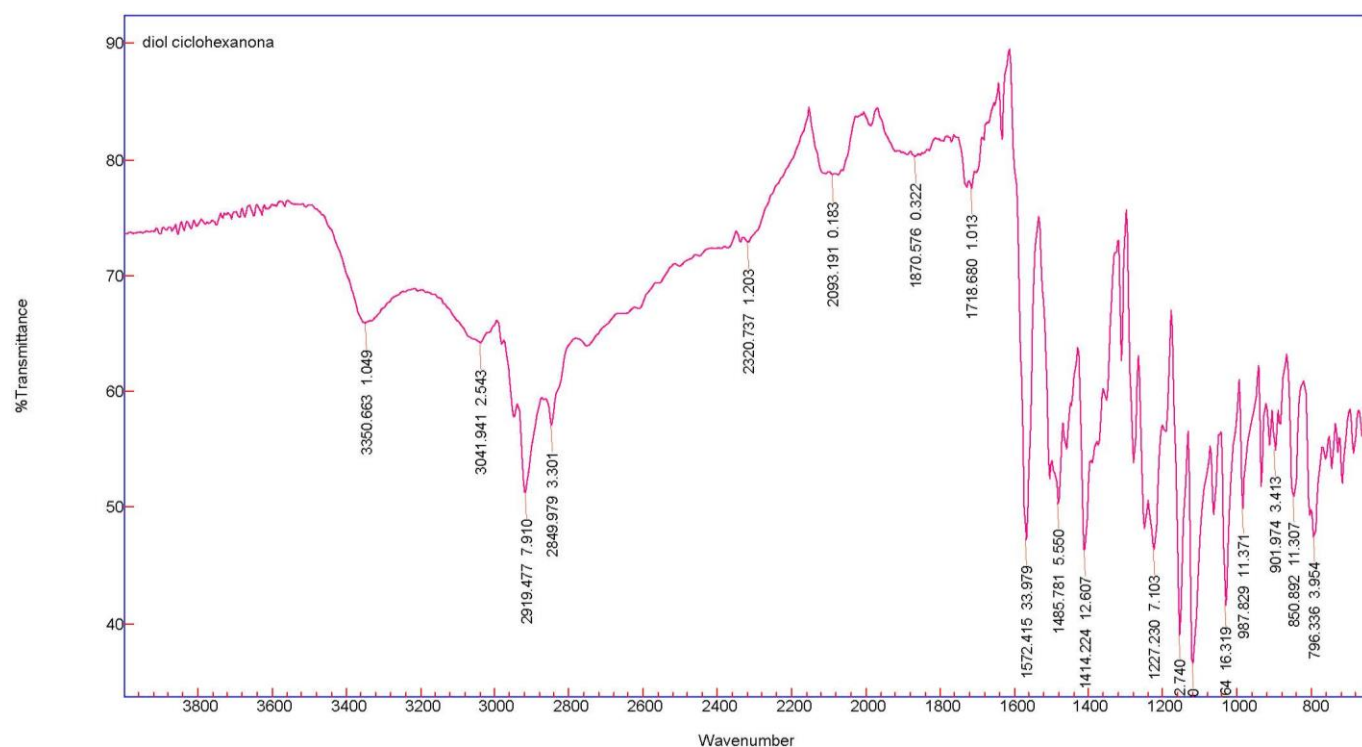
HSQC ^1H - ^{13}C ($(\text{CD}_3)_2\text{SO}$), δ (ppm)



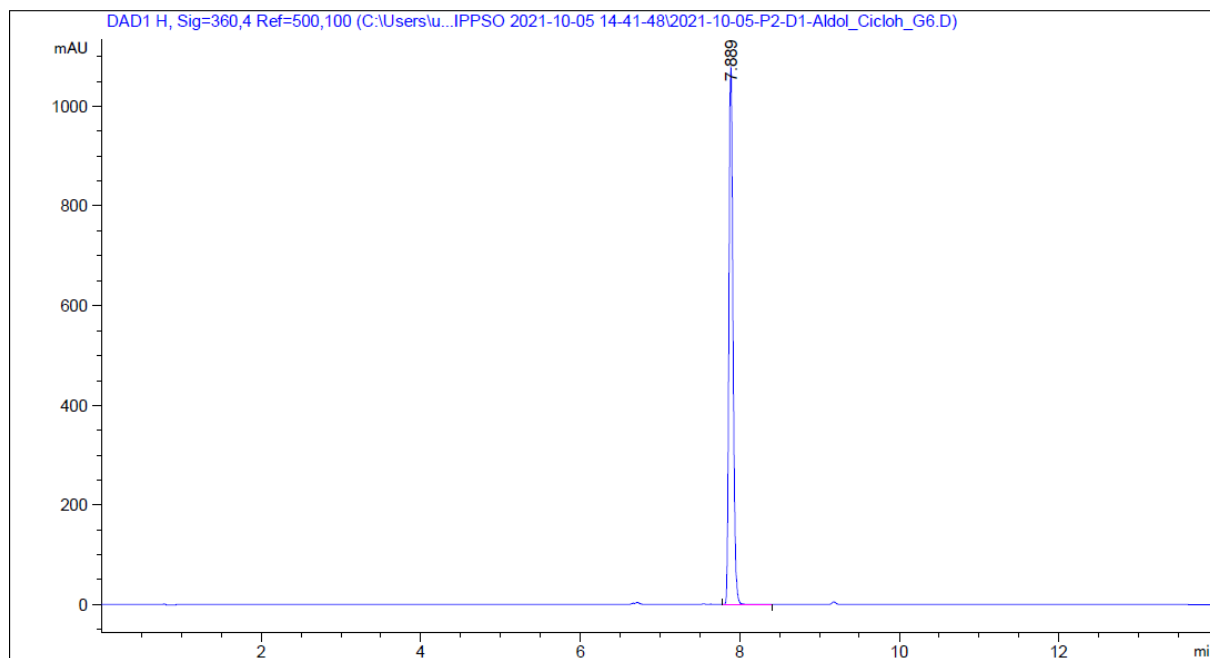
HMBC ^1H - ^{13}C ($(\text{CD}_3)_2\text{SO}$), δ (ppm)



FT-IR (neat), ν (cm⁻¹)



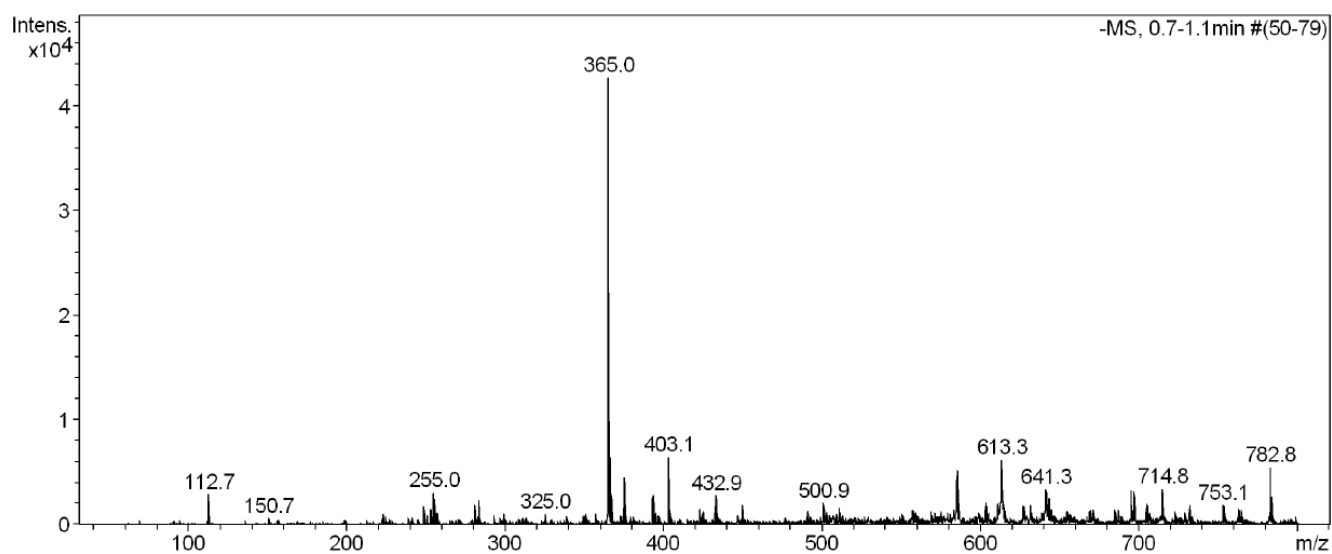
HPLC (λ = 360 nm)



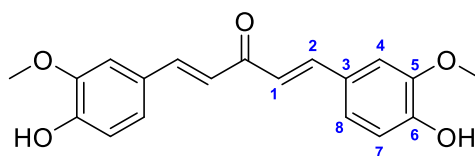
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
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Totals : 3834.10083 1082.30933

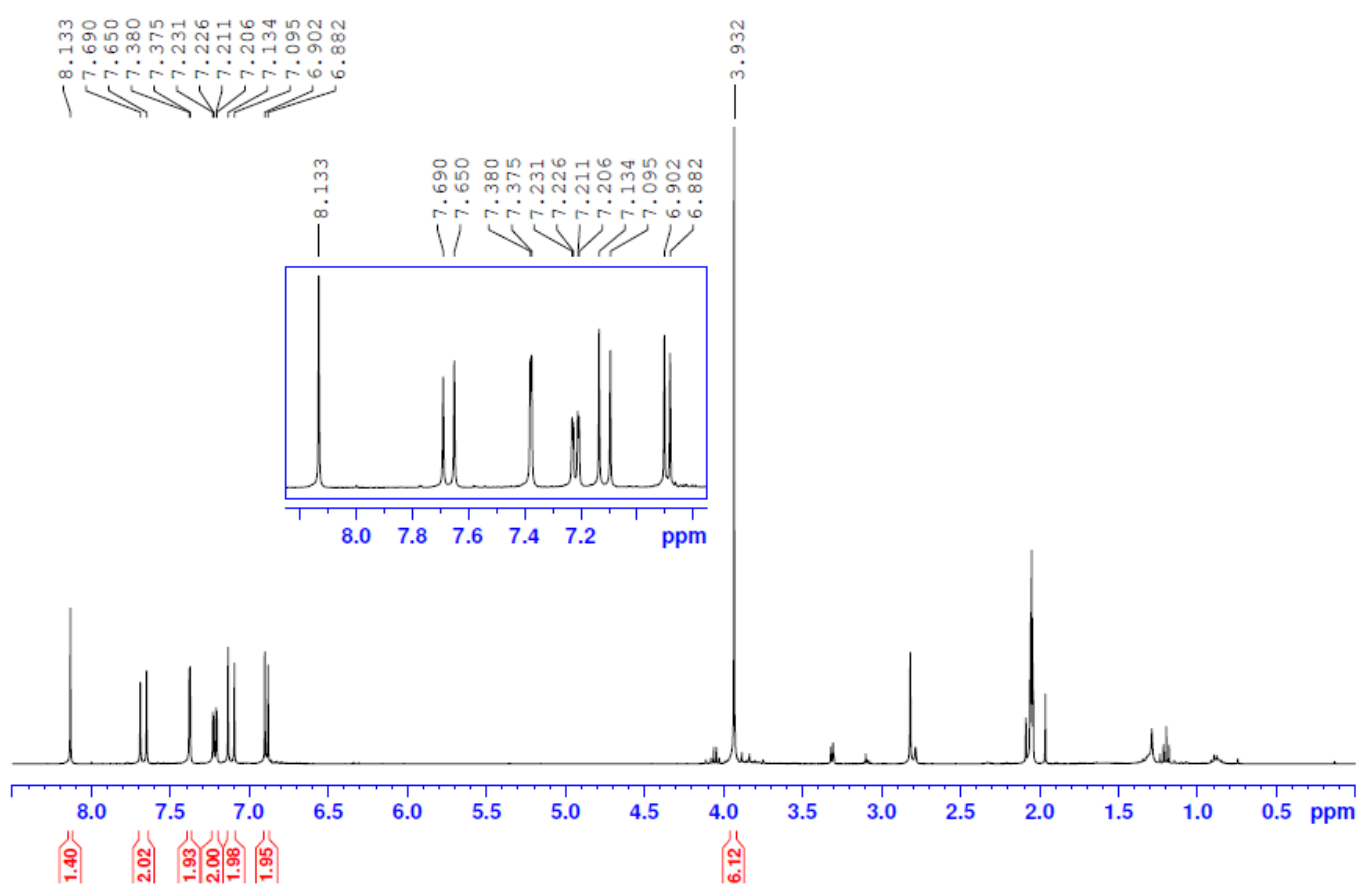
(-)-ESI-MS (m/z)

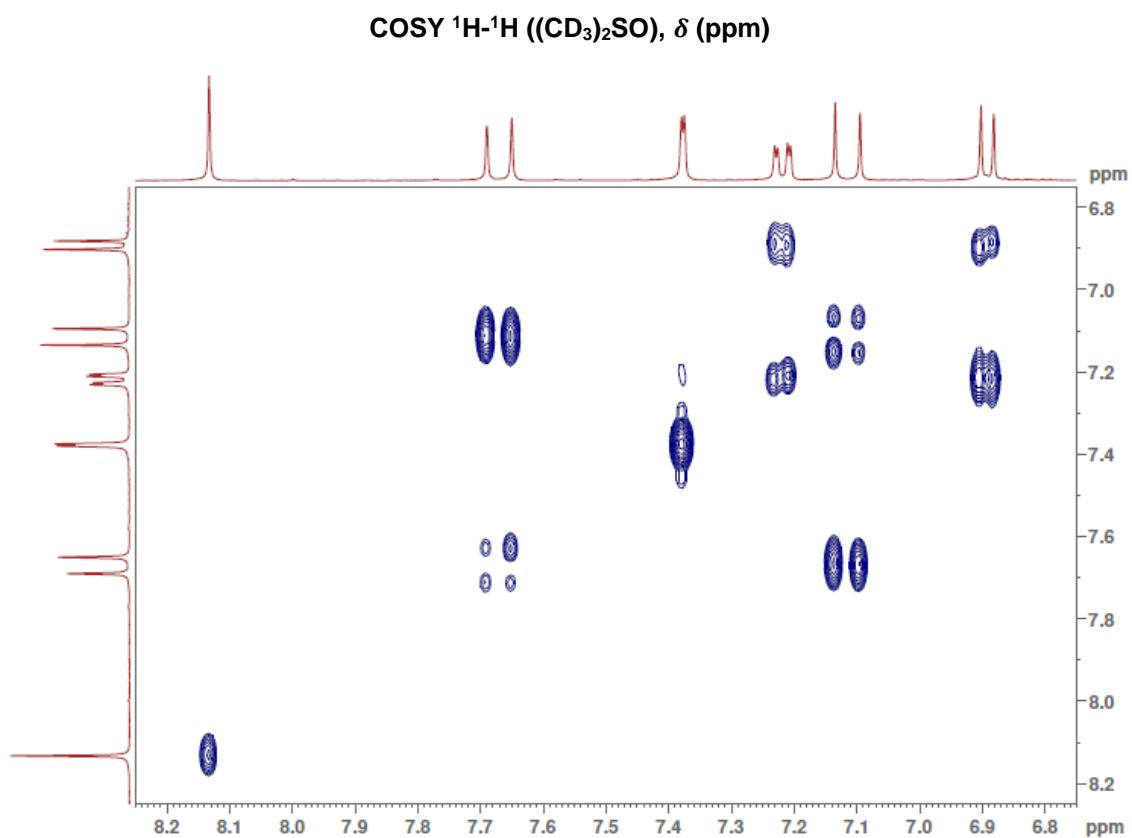
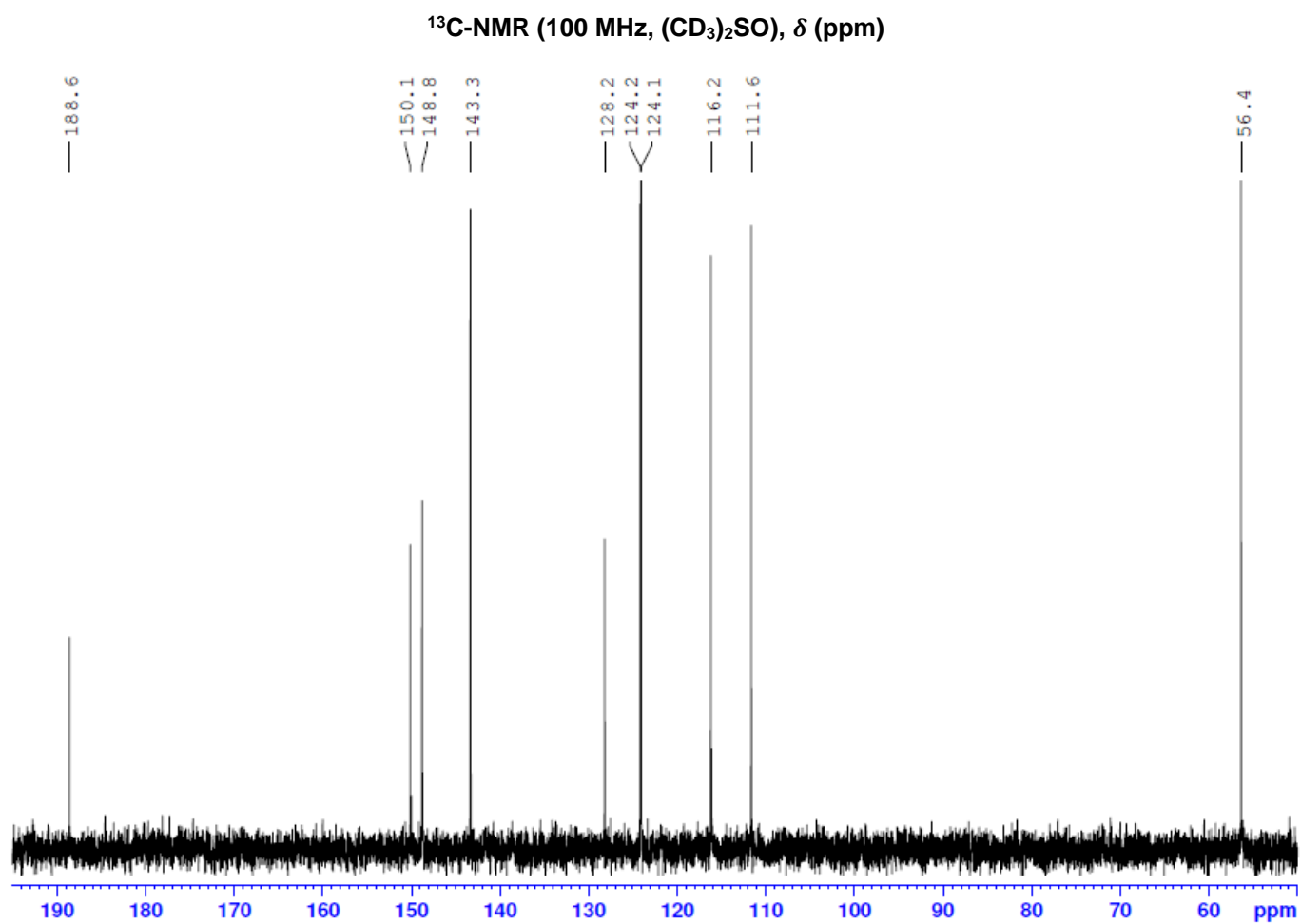


2.3. (1E,4E)-1,5-Bis(4-hydroxy-3-methoxyphenyl)penta-1,4-dien-3-one (2)

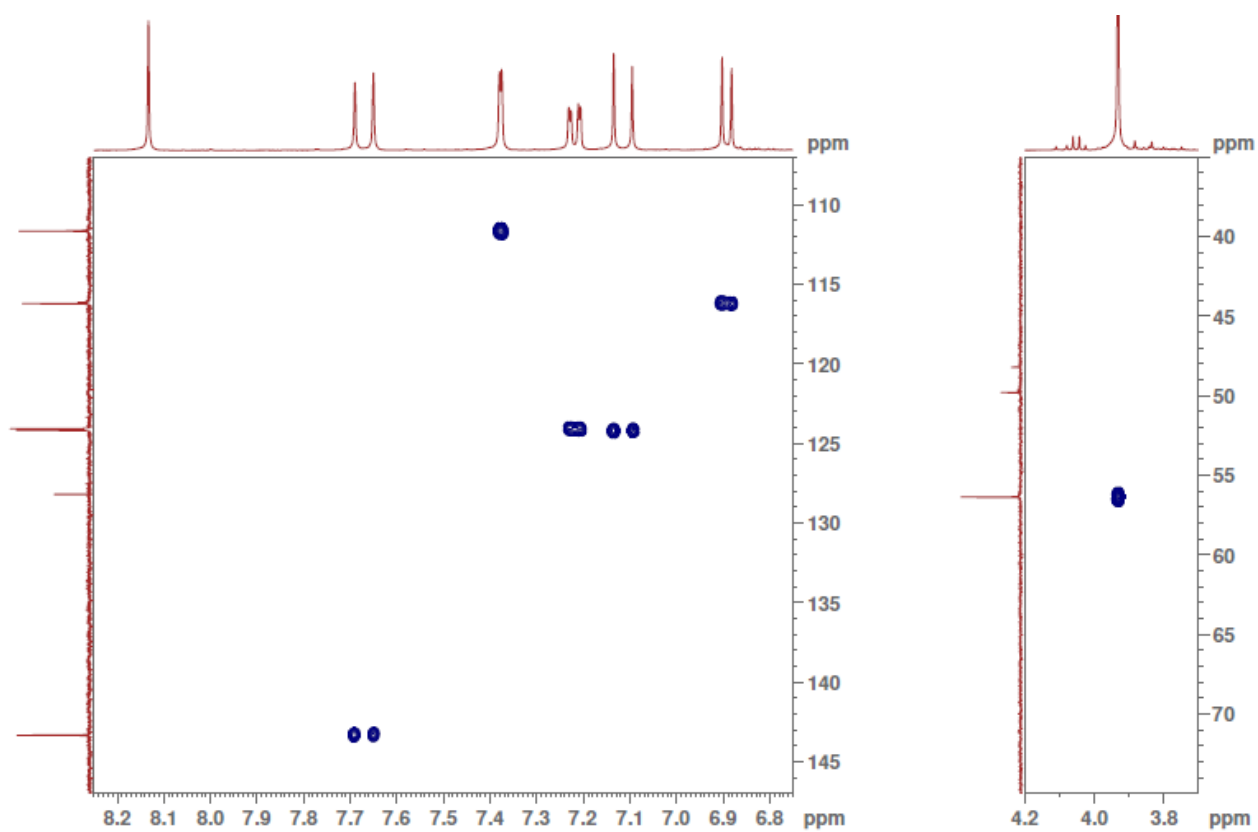


$^1\text{H-NMR}$ (400 MHz, $(\text{CD}_3)_2\text{CO}$), δ (ppm)

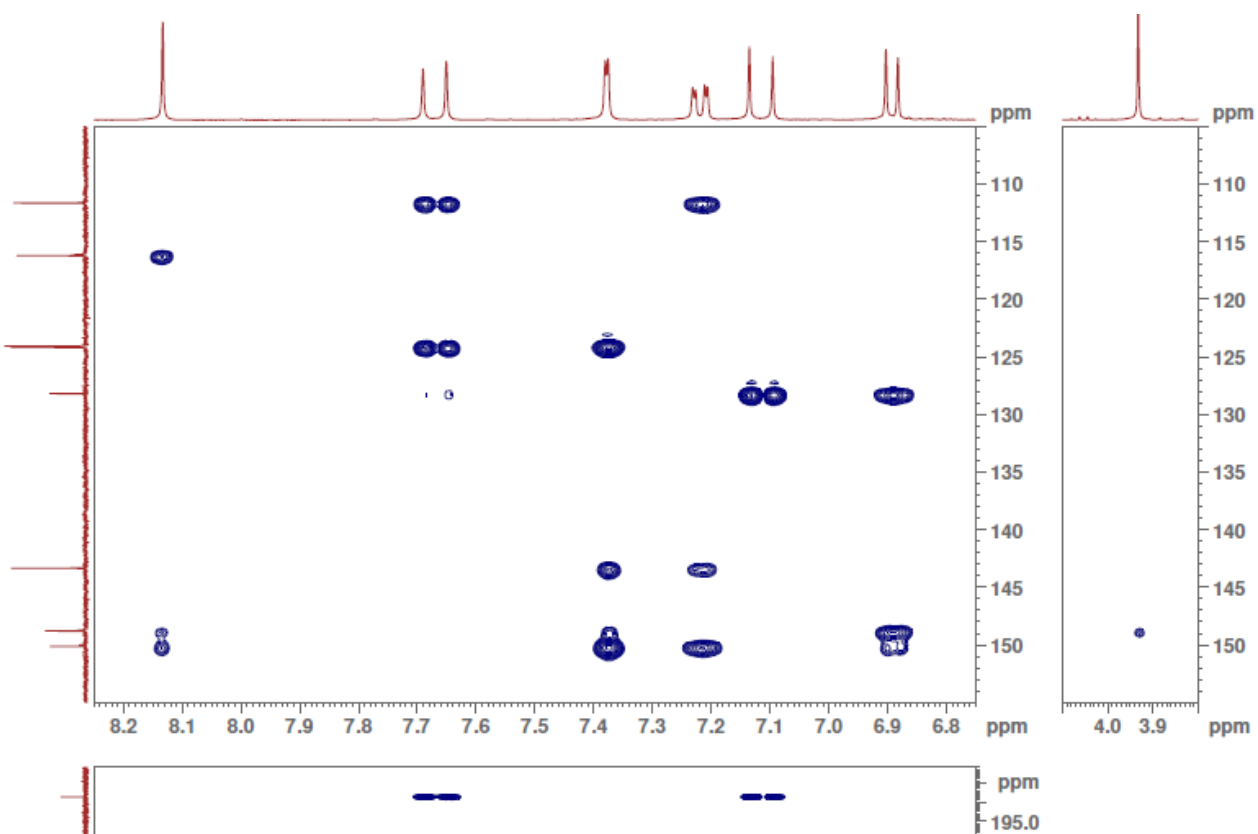




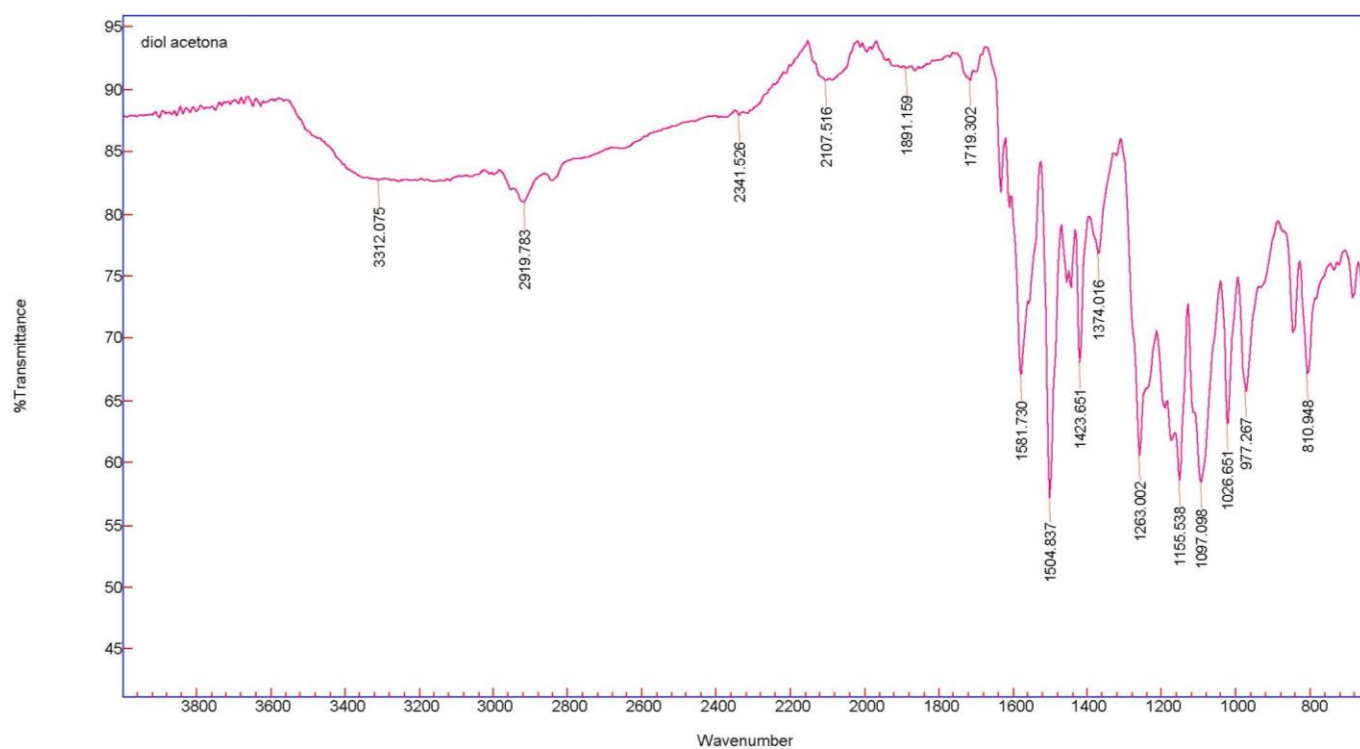
HSQC ^1H - ^{13}C ($(\text{CD}_3)_2\text{SO}$), δ (ppm)



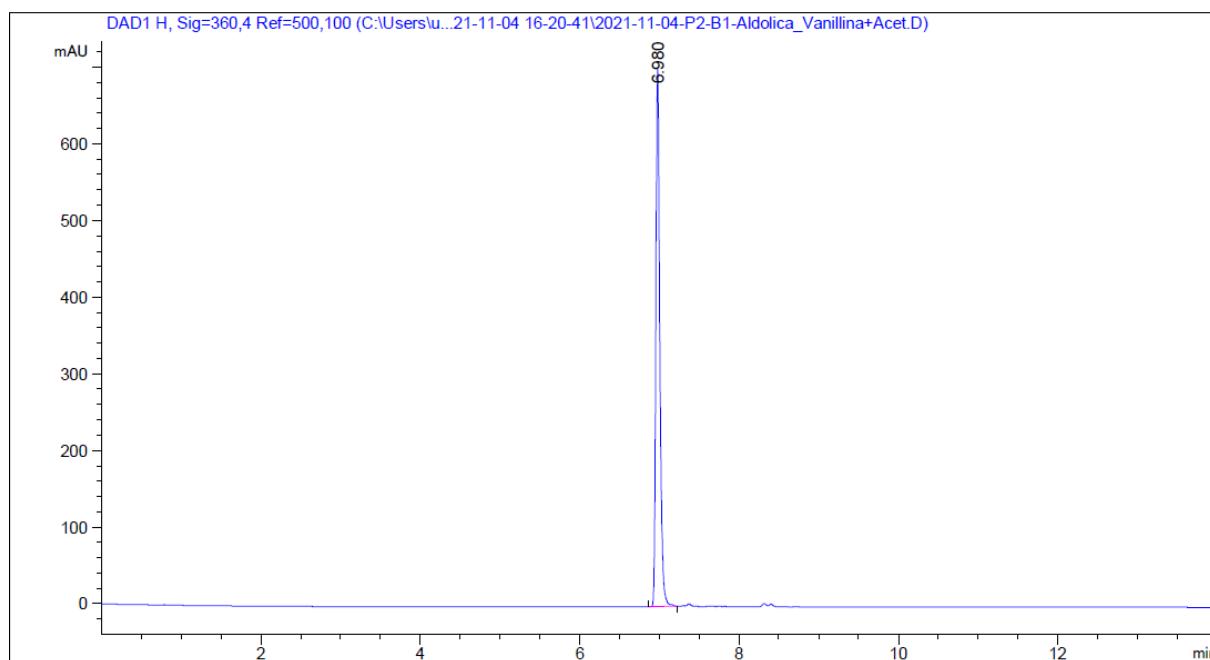
HMBC ^1H - ^{13}C ($(\text{CD}_3)_2\text{SO}$), δ (ppm)



FT-IR (neat), ν (cm⁻¹)



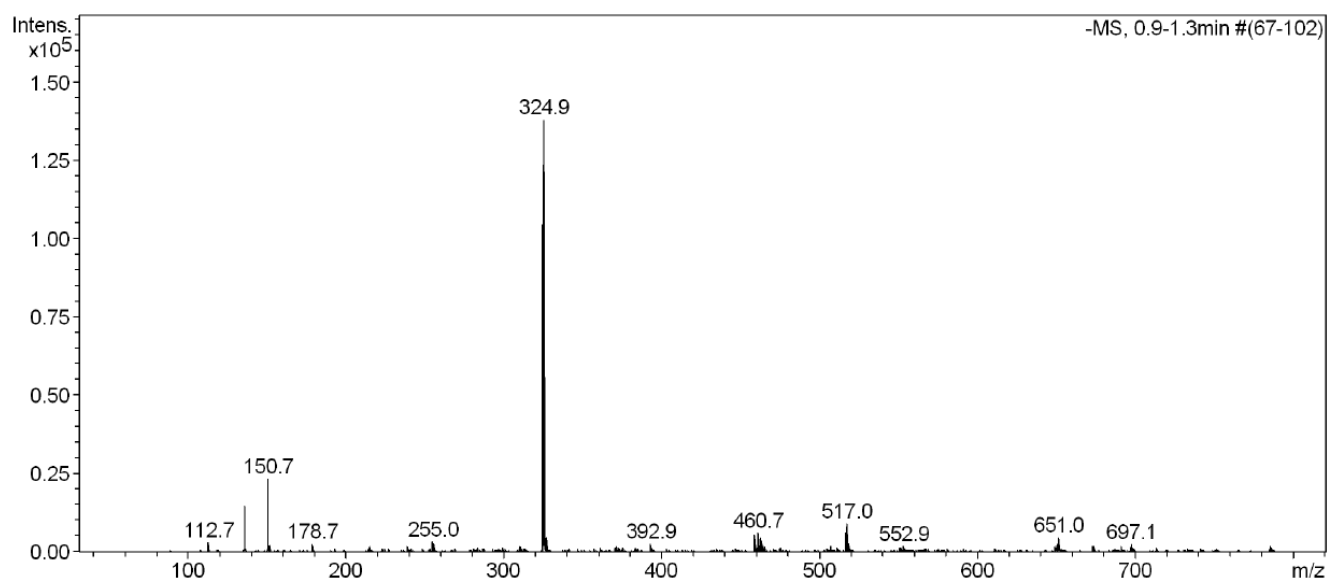
HPLC (λ = 360 nm)



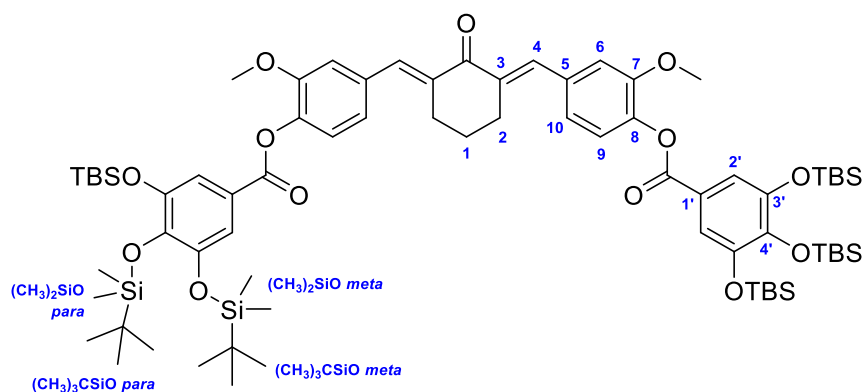
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
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Totals : 2411.89429 702.30542

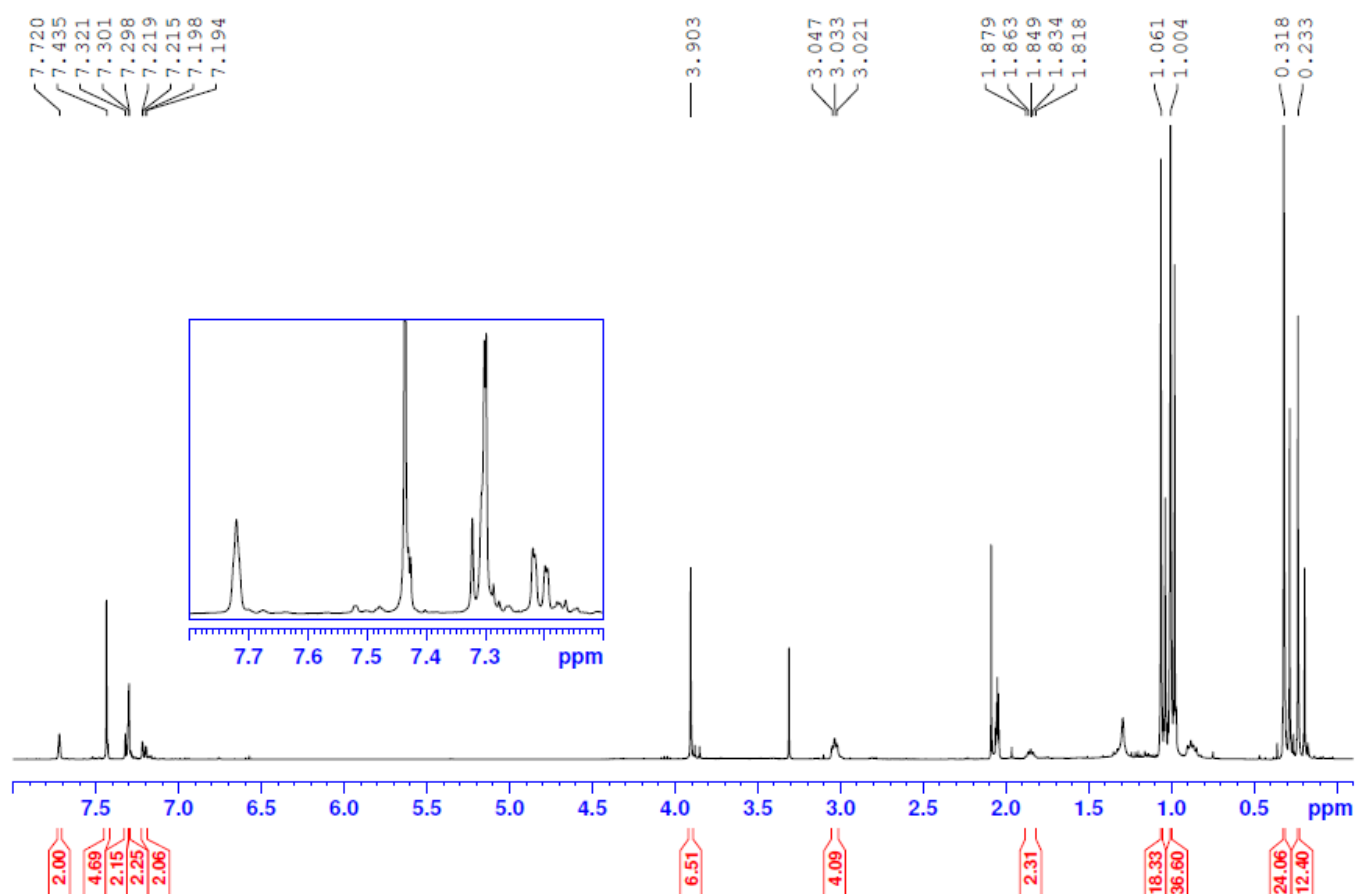
(-)-ESI-MS (m/z)



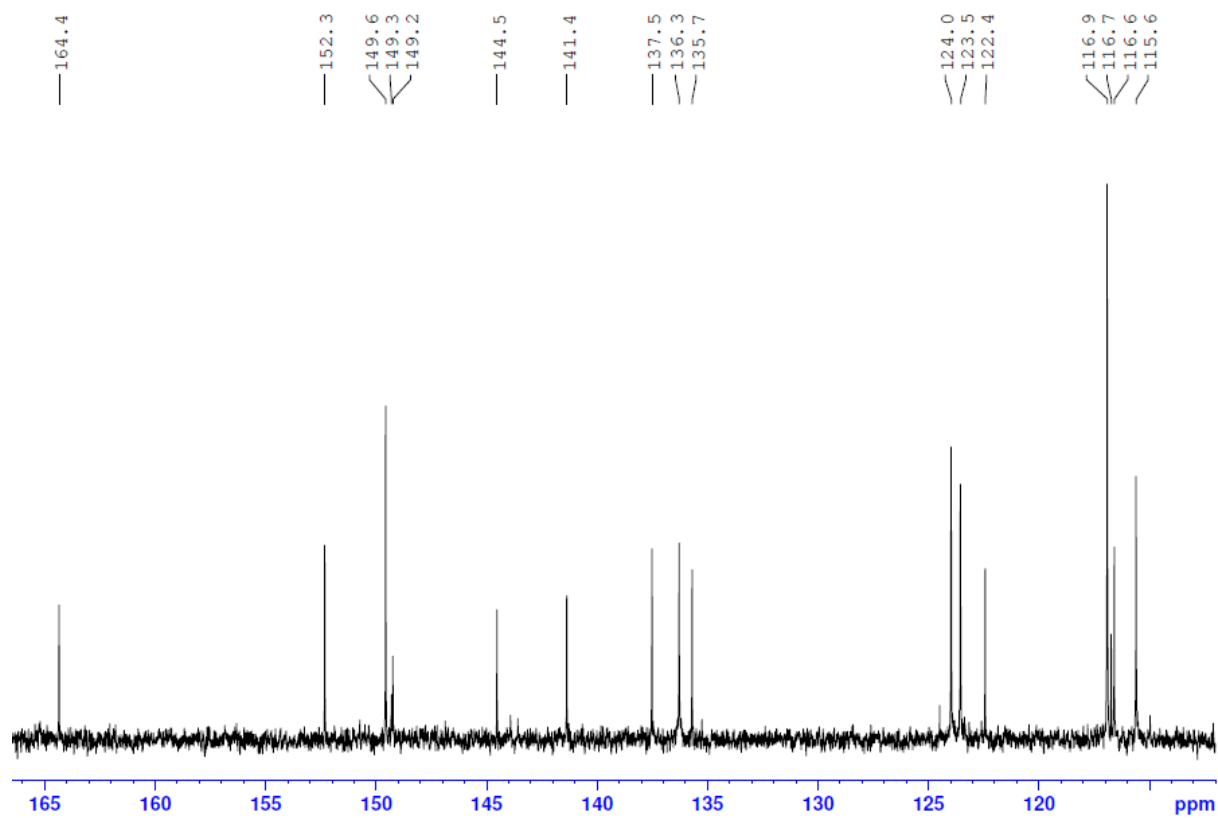
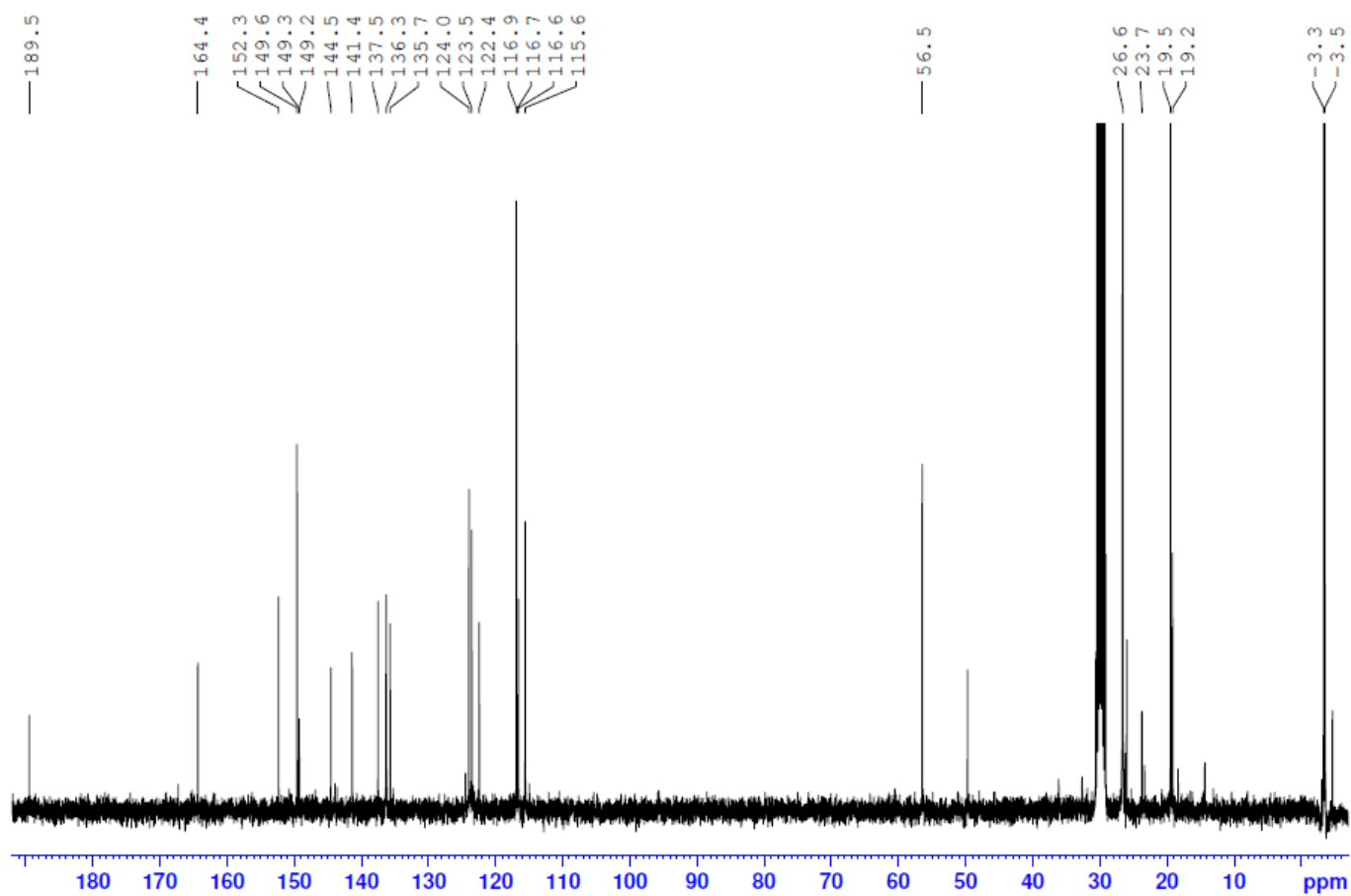
2.4. ((1*E*,1'*E*)-(2-Oxocyclohexane-1,3-diylidene)bis(methanylylidene))bis(2-methoxy-4,1-phenylene) bis[3,4,5-tris((*tert*-butyldimethylsilyl)oxy)benzoate]



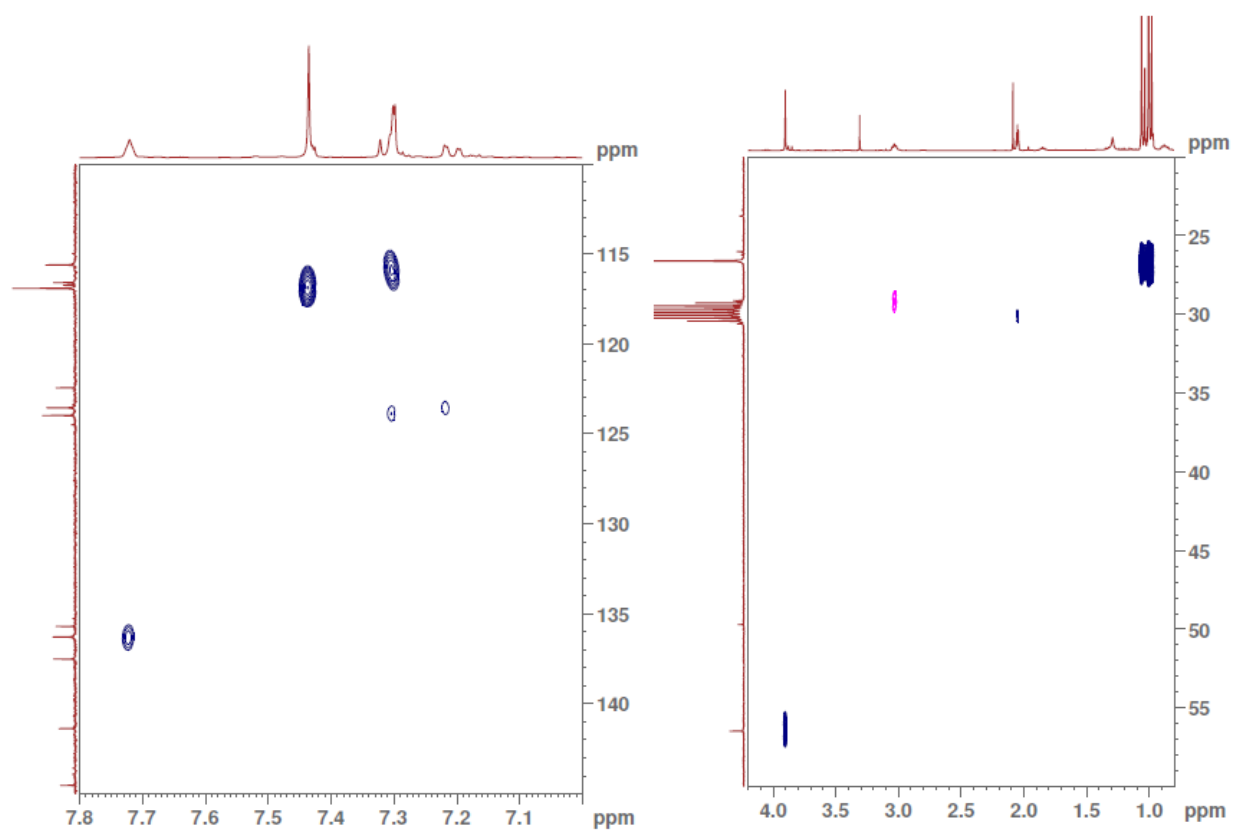
¹H-NMR (400 MHz, (CD₃)₂CO), δ (ppm)



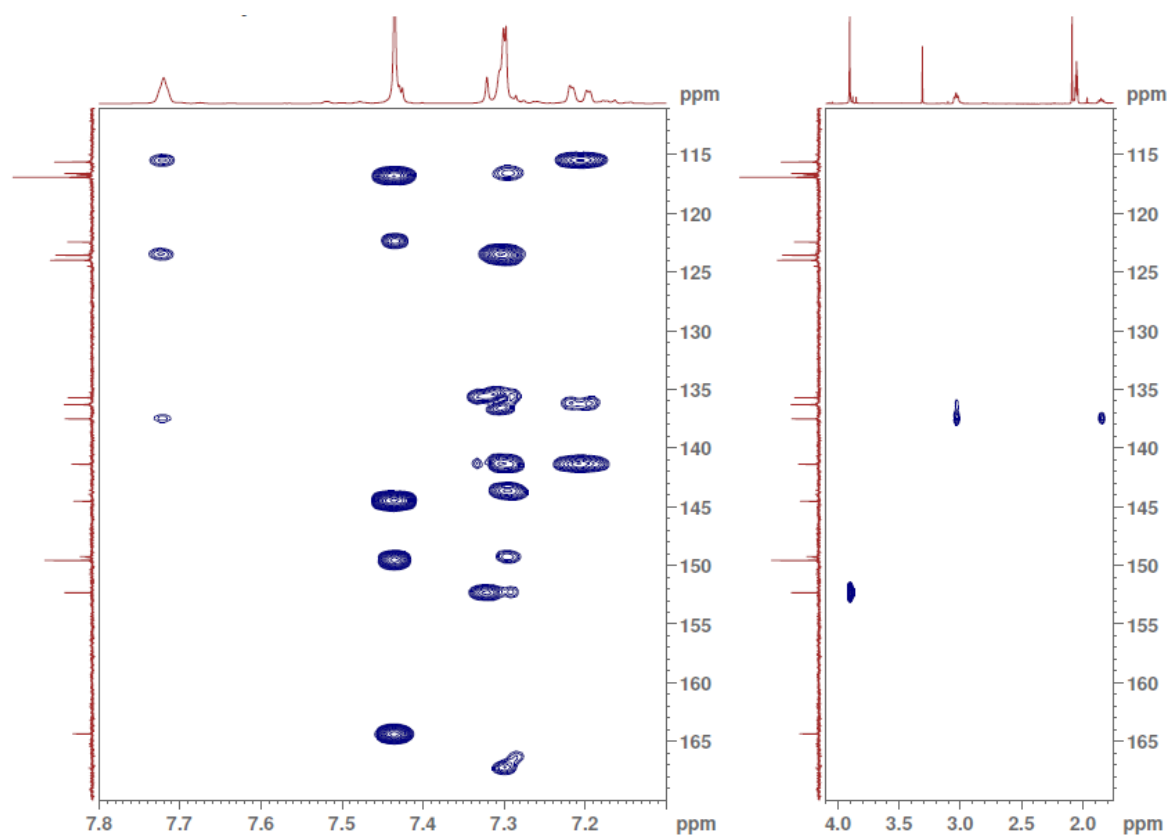
^{13}C -NMR (100 MHz, $(\text{CD}_3)_2\text{CO}$), δ (ppm)



HSQC ^1H - ^{13}C ($(\text{CD}_3)_2\text{CO}$), δ (ppm)



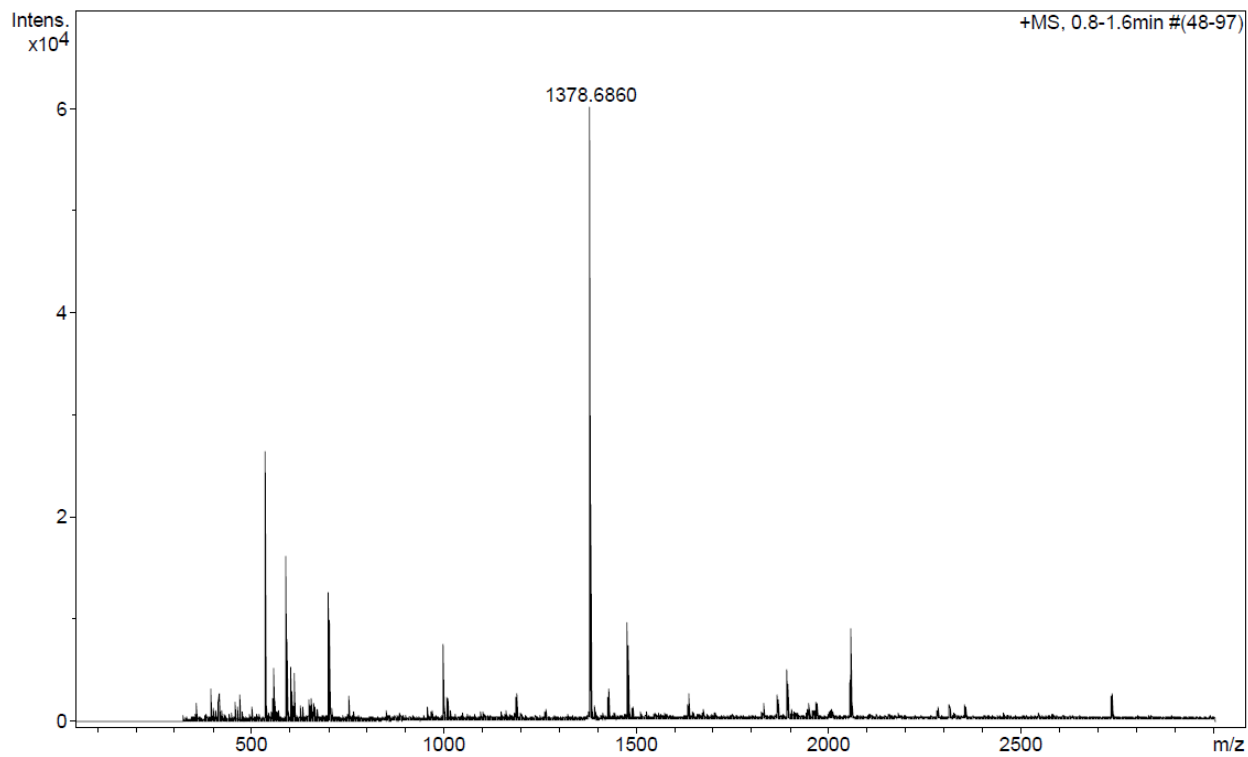
HMBC ^1H - ^{13}C ($(\text{CD}_3)_2\text{CO}$), δ (ppm)

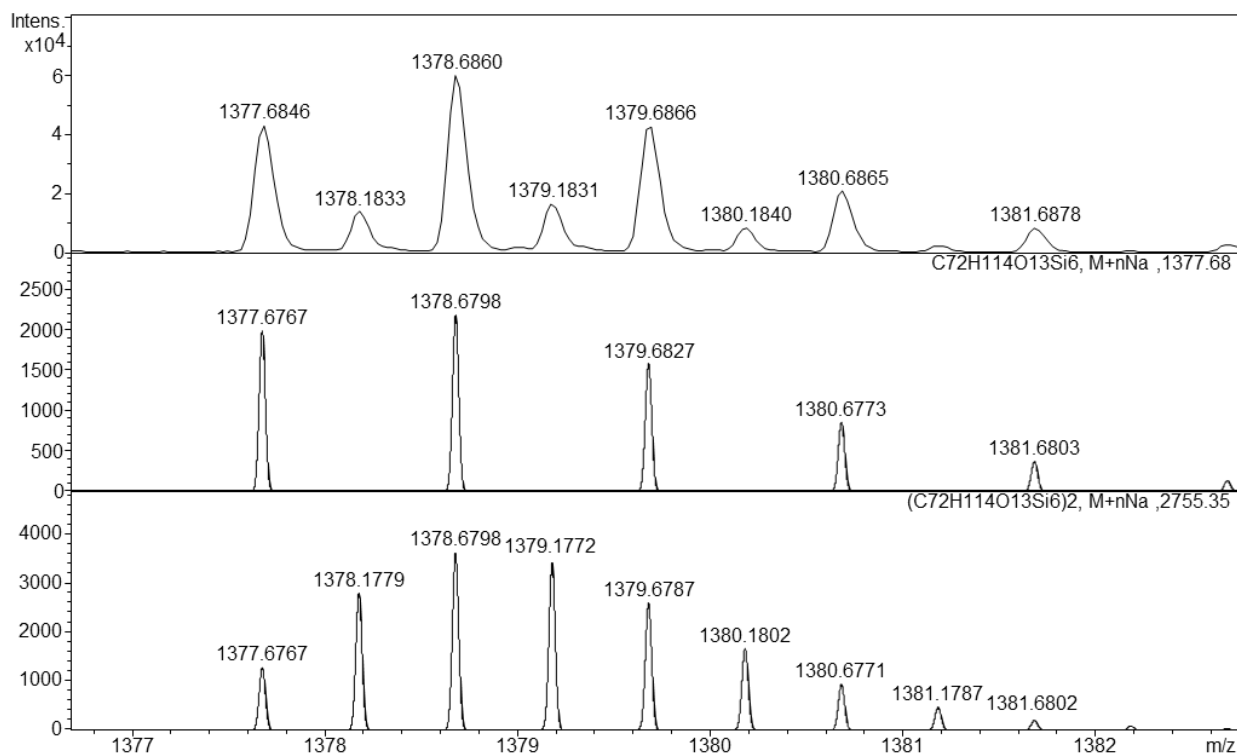


FT-IR (neat), ν (cm⁻¹)



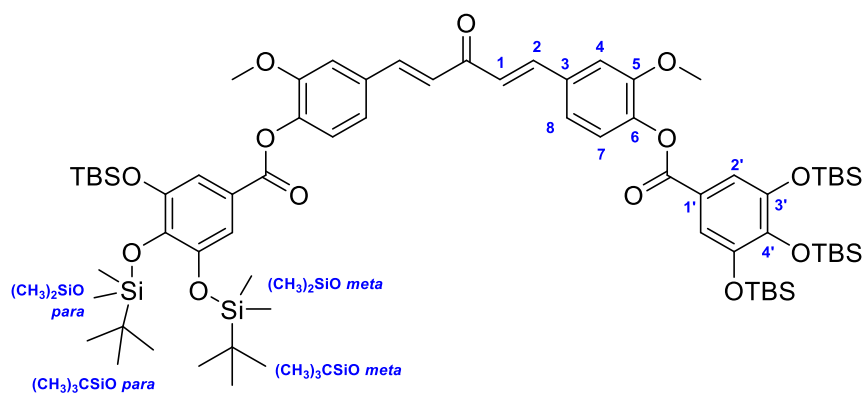
HRMS (m/z)



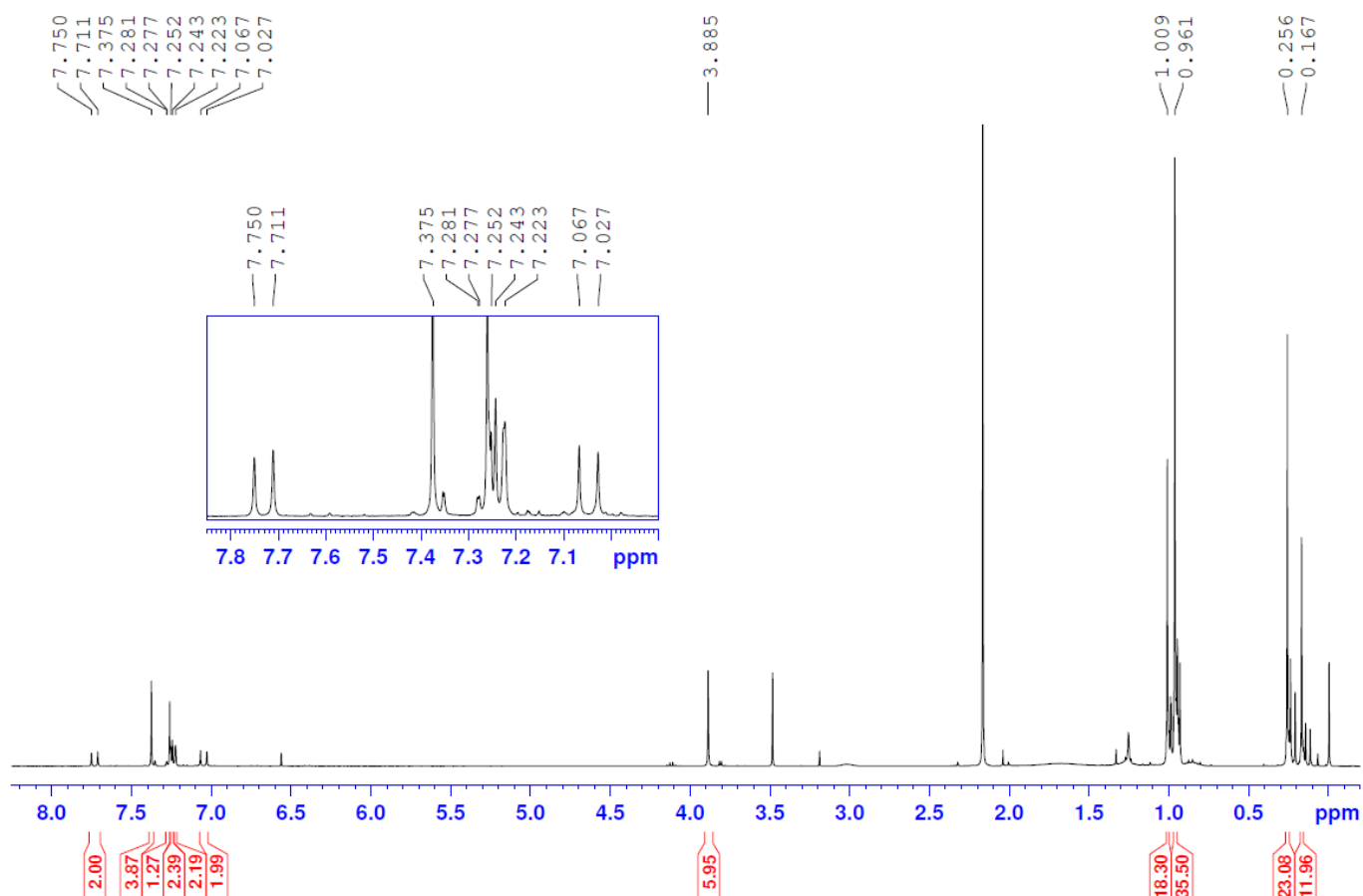


	Molecular formula	Calculated	Found
[M+Na]⁺	$C_{72}H_{114}O_{13}Si_6Na$	1377.6767	1377.6846
[2M+2Na]²⁺	$(C_{72}H_{114}O_{13}Si_6)_2Na_2$	1377.6767	1377.6846

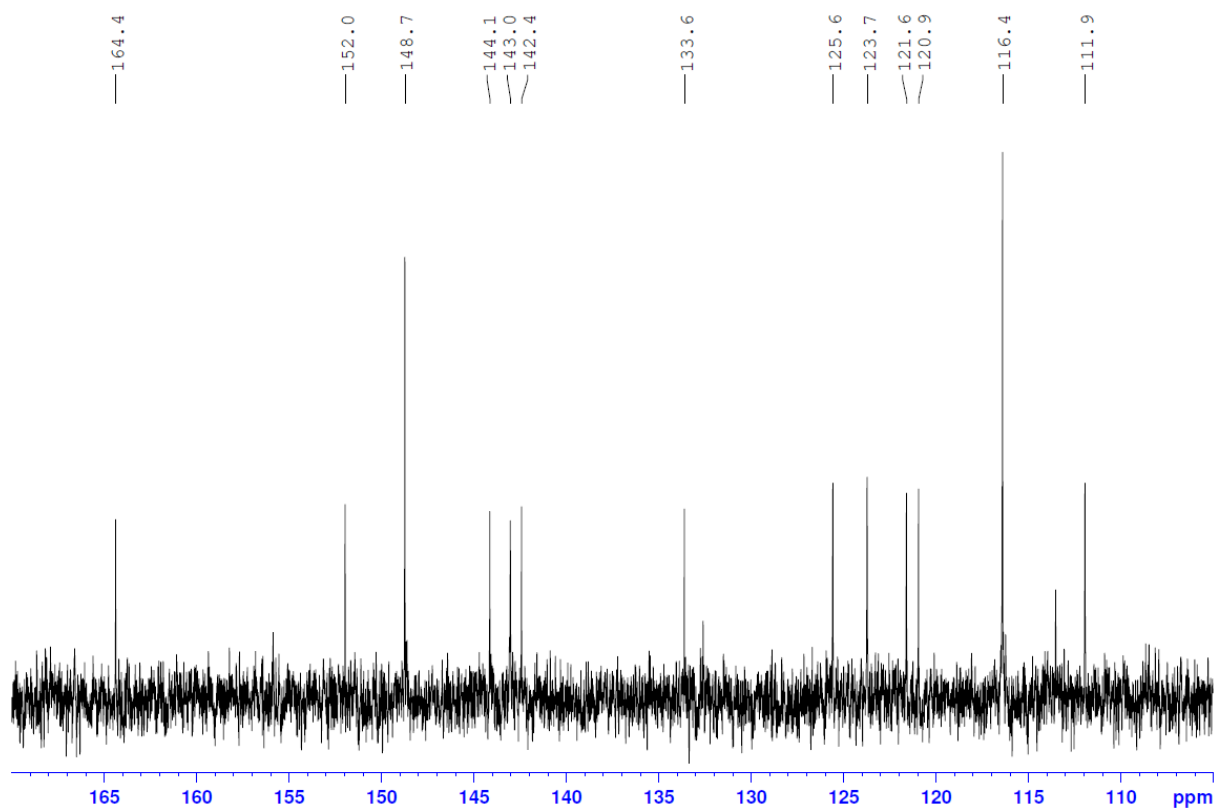
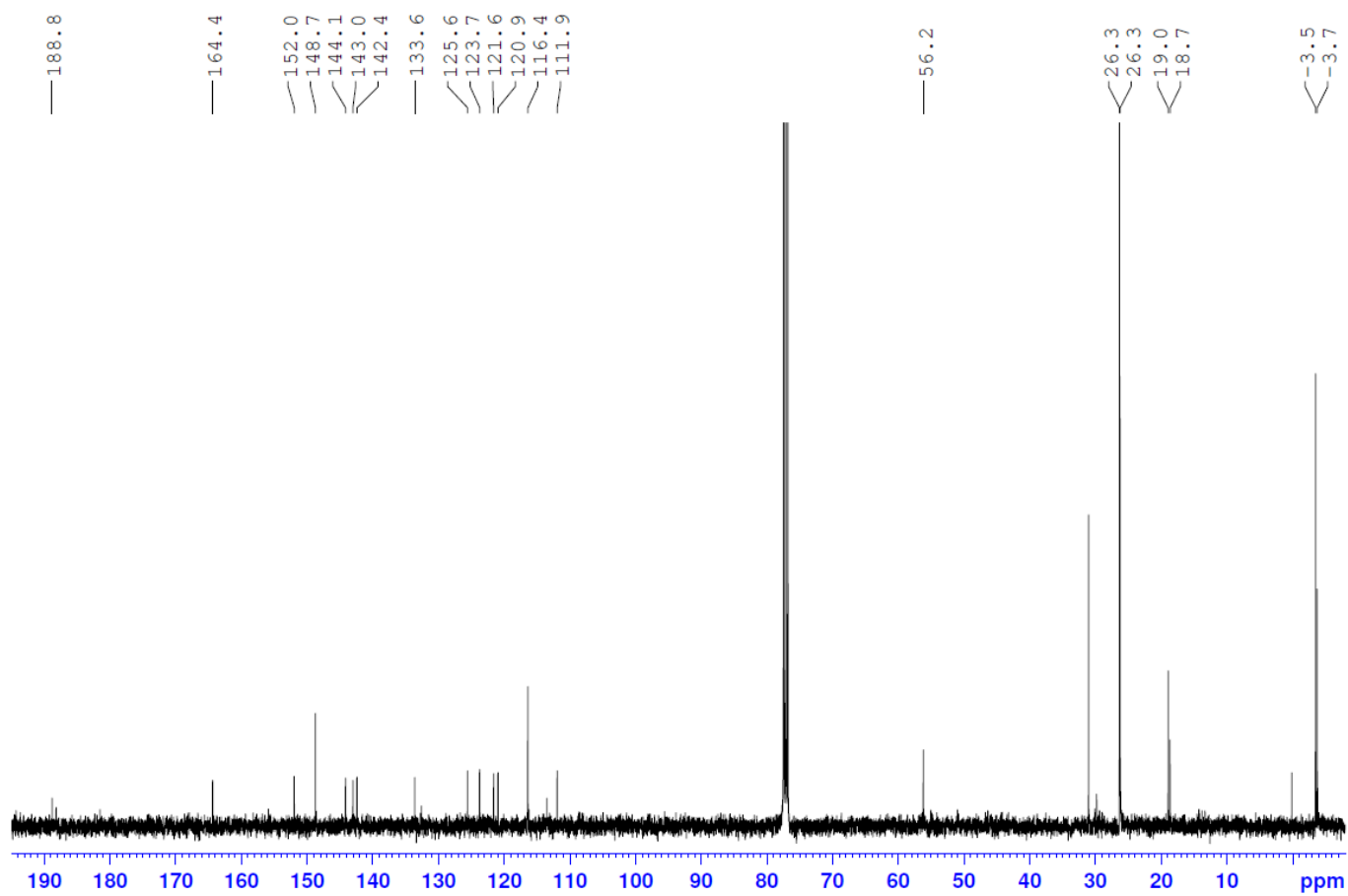
2.5. ((1*E*,4*E*)-3-Oxopenta-1,4-diene-1,5-diyl)bis(2-methoxy-4,1-phenylene) bis[3,4,5-tris((*tert*-butyldimethylsilyl)oxy)benzoate]



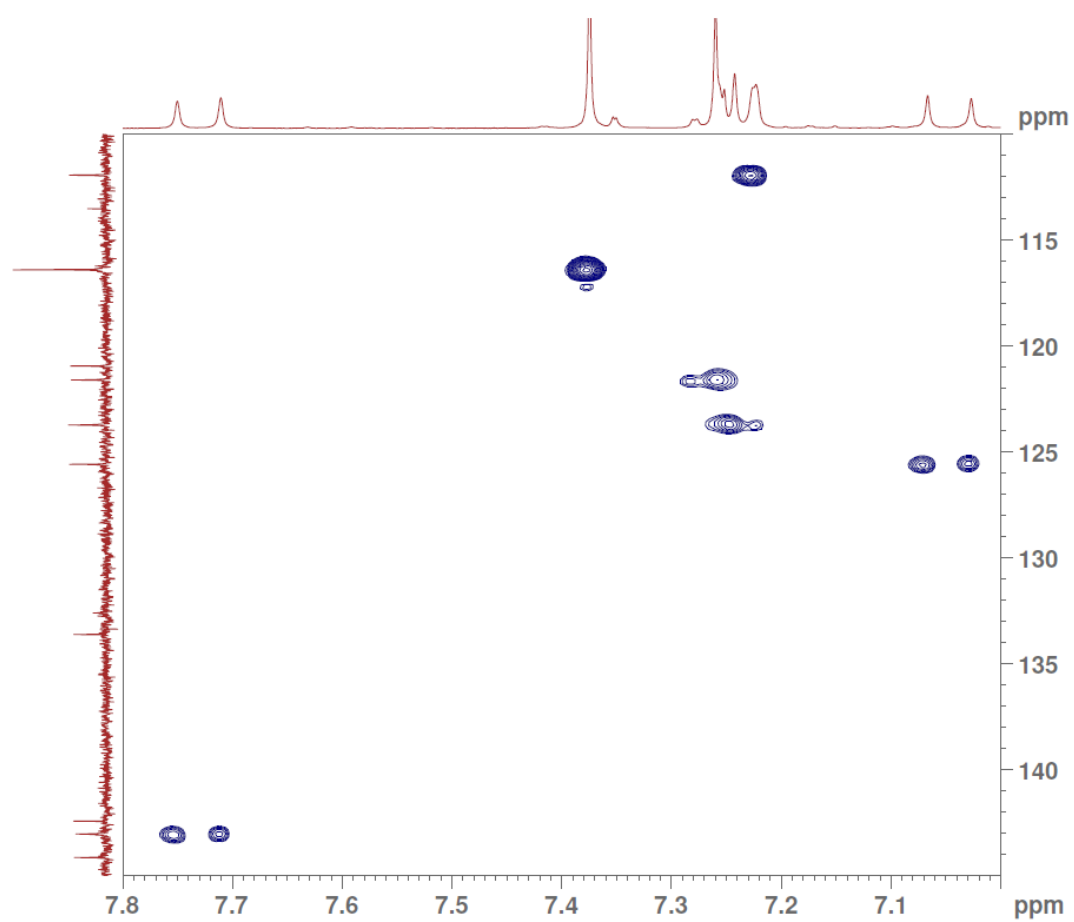
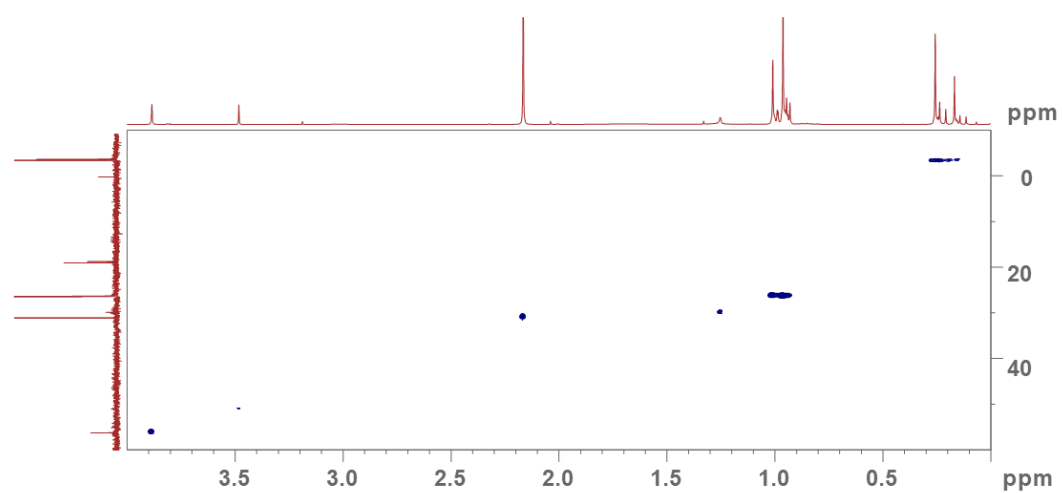
¹H-NMR (400 MHz, CDCl₃), δ (ppm)



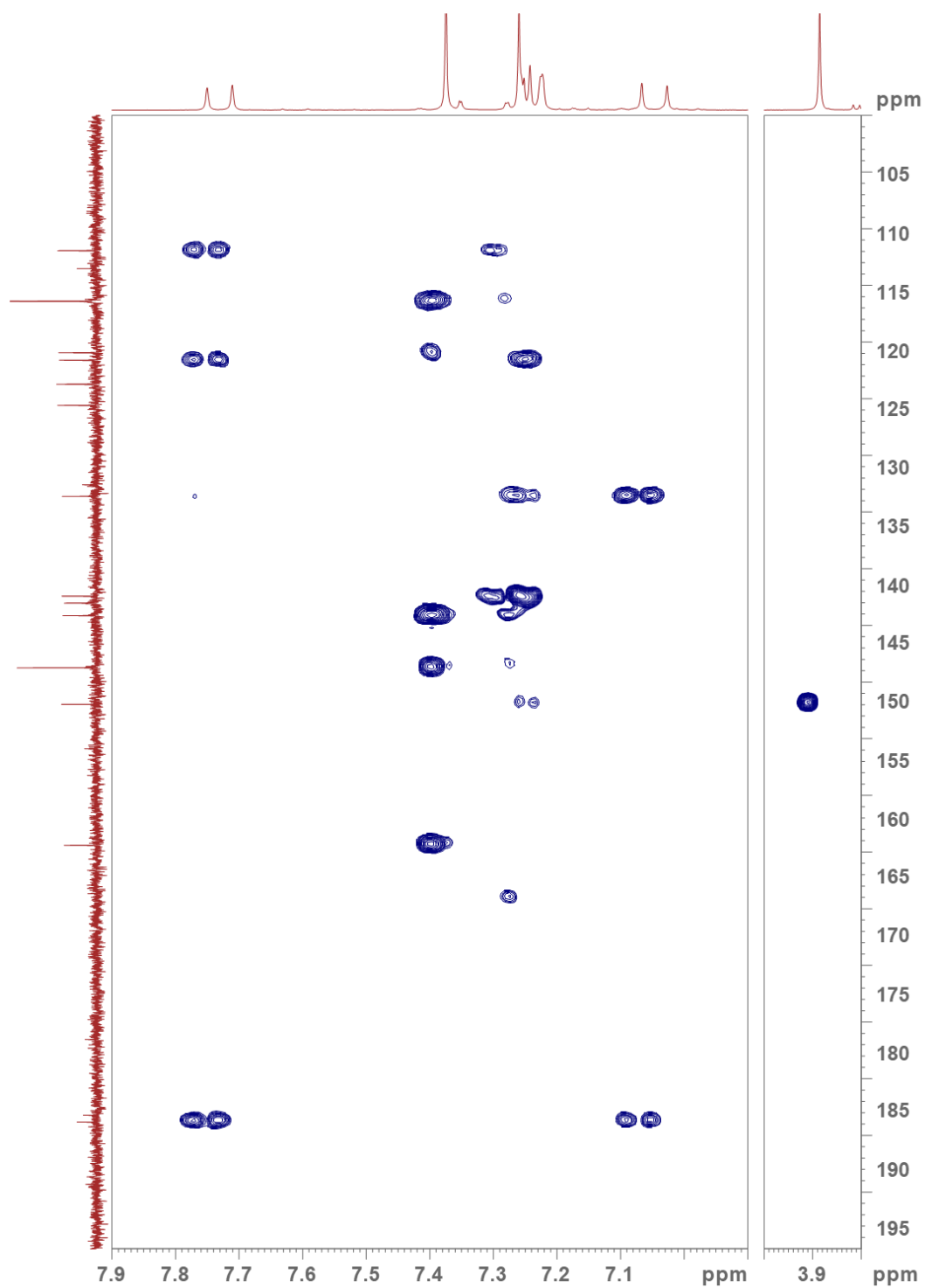
^{13}C -NMR (100 MHz, CDCl_3), δ (ppm)



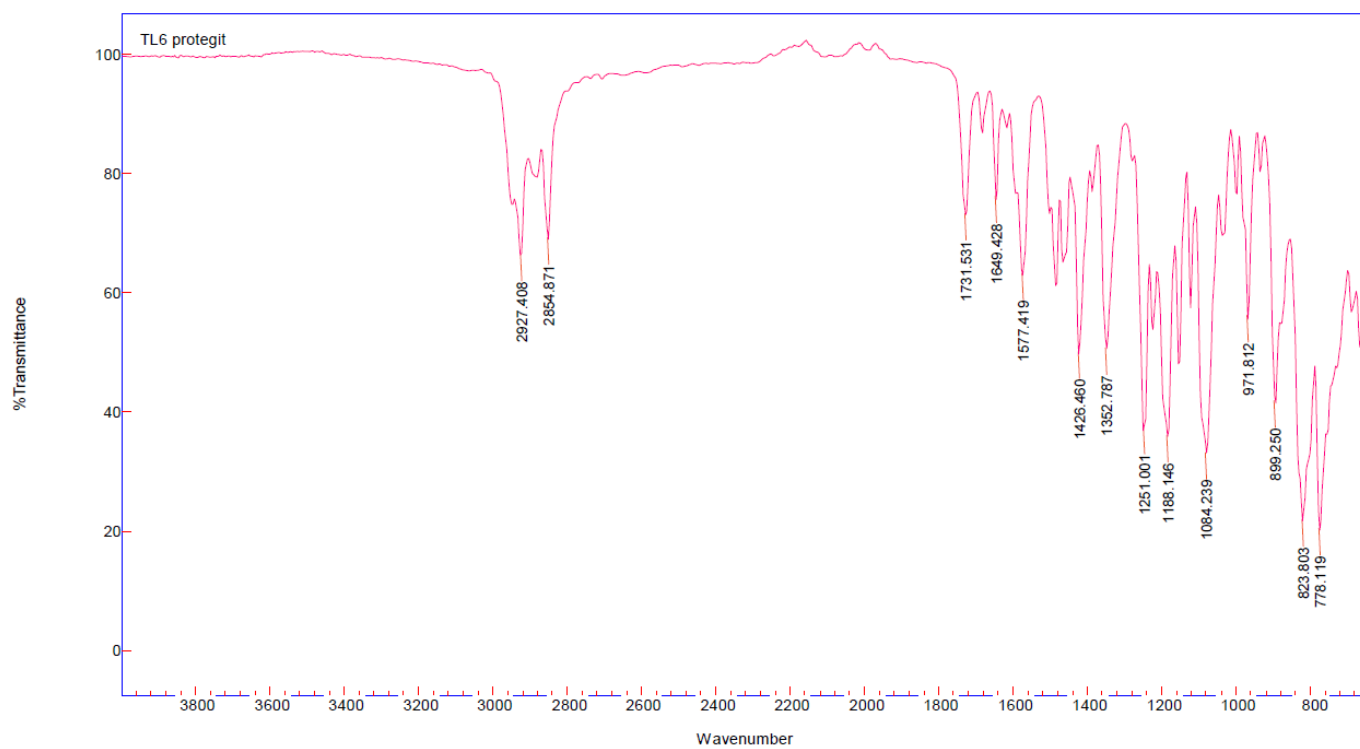
HSQC ^1H - ^{13}C (CDCl_3), δ (ppm)



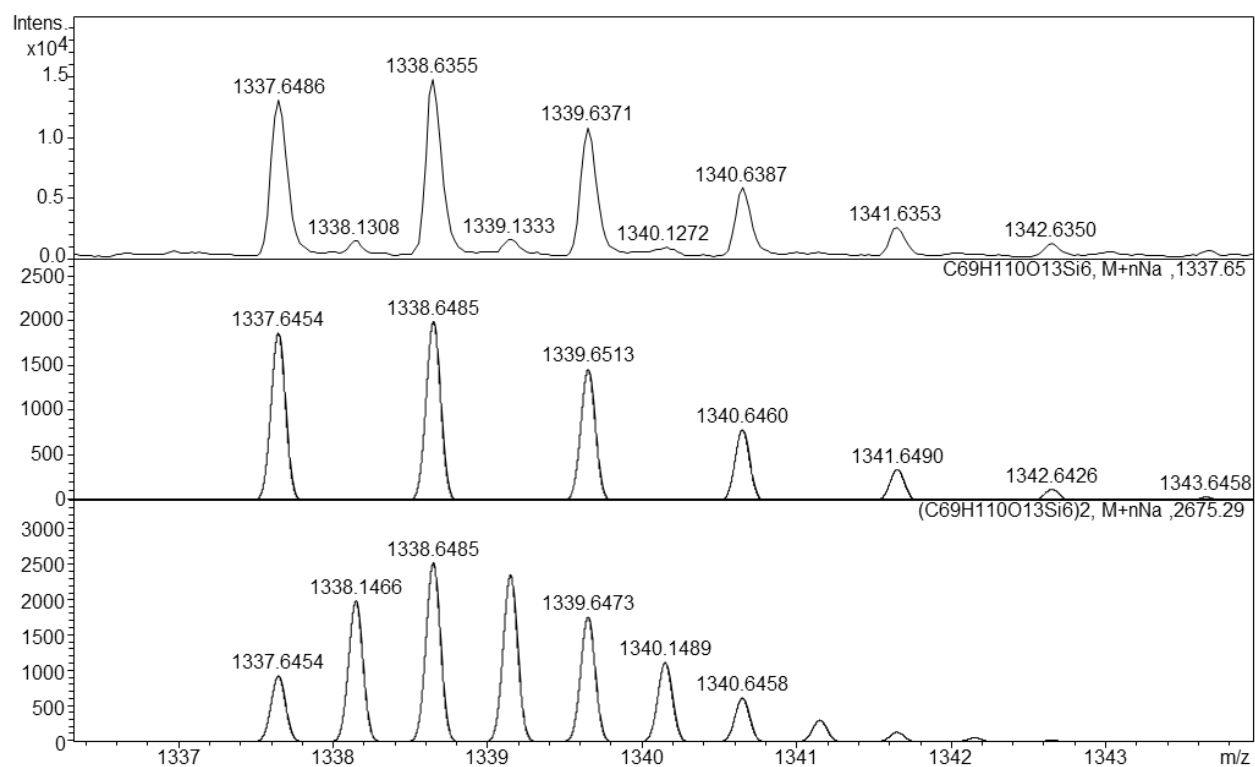
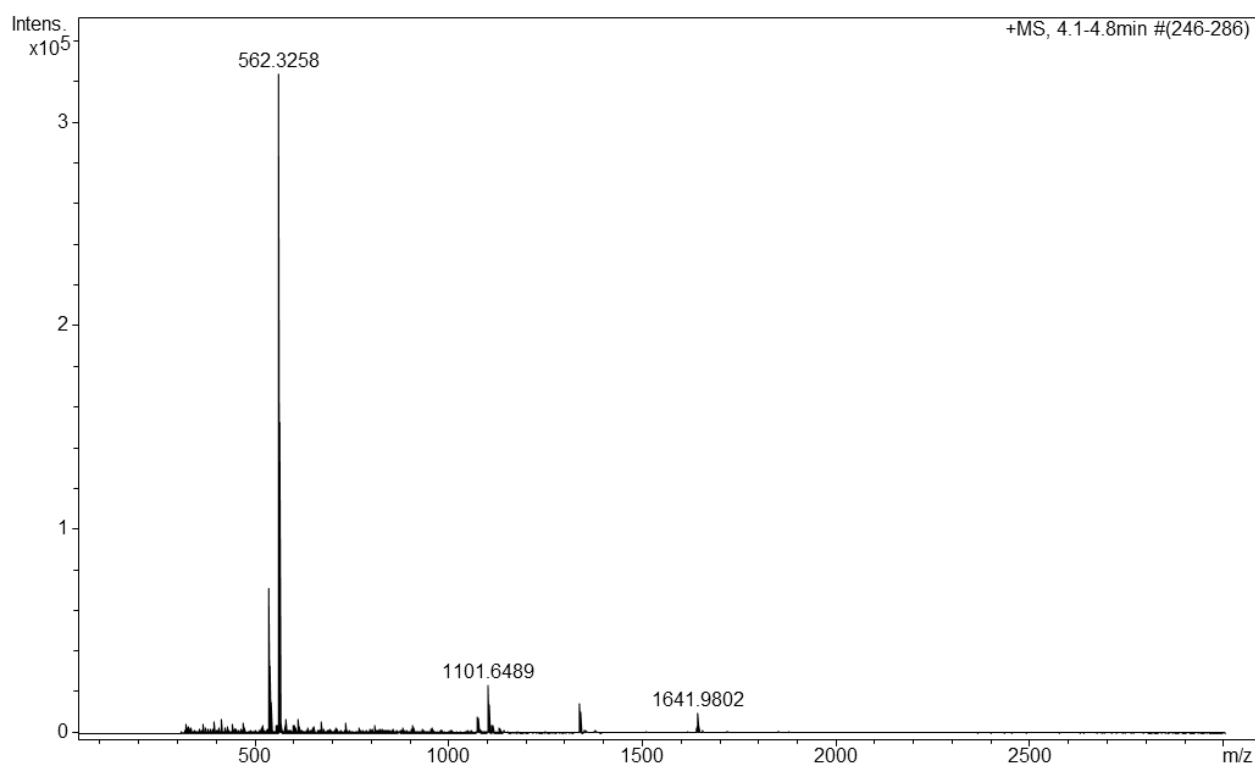
HMBC ^1H - ^{13}C (CDCl_3), δ (ppm)



FT-IR (neat), ν (cm⁻¹)

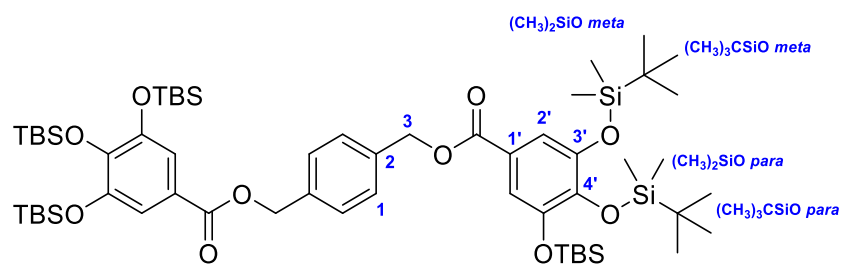


HRMS (m/z)

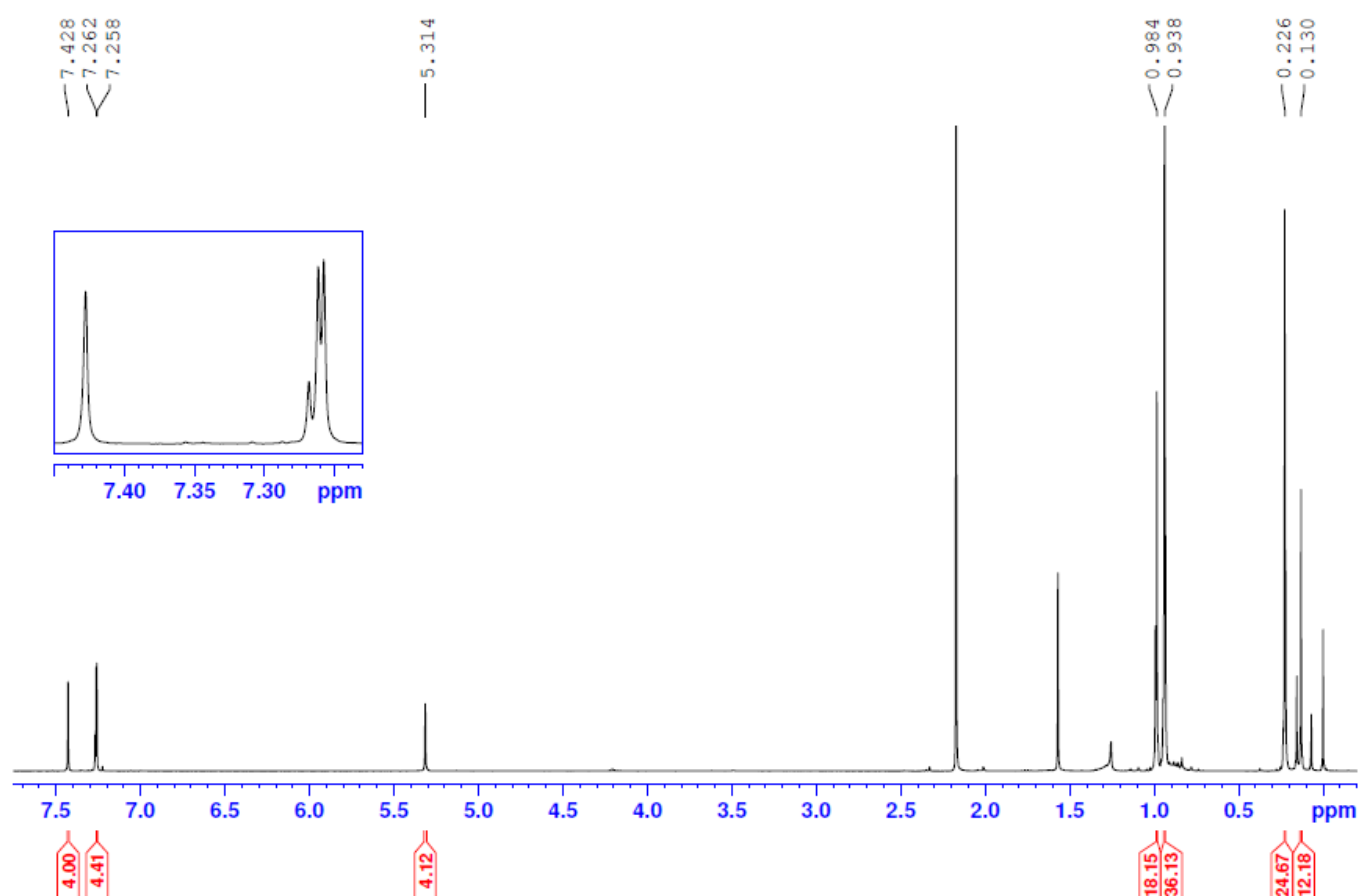


	Molecular formula	Calculated	Found
[M+Na] ⁺	C ₆₉ H ₁₁₀ O ₁₃ Si ₆ Na	1337.6454	1337.6486
[2M+2Na] ²⁺	(C ₆₉ H ₁₁₀ O ₁₃ Si ₆) ₂ Na ₂	1337.6454	1337.6486

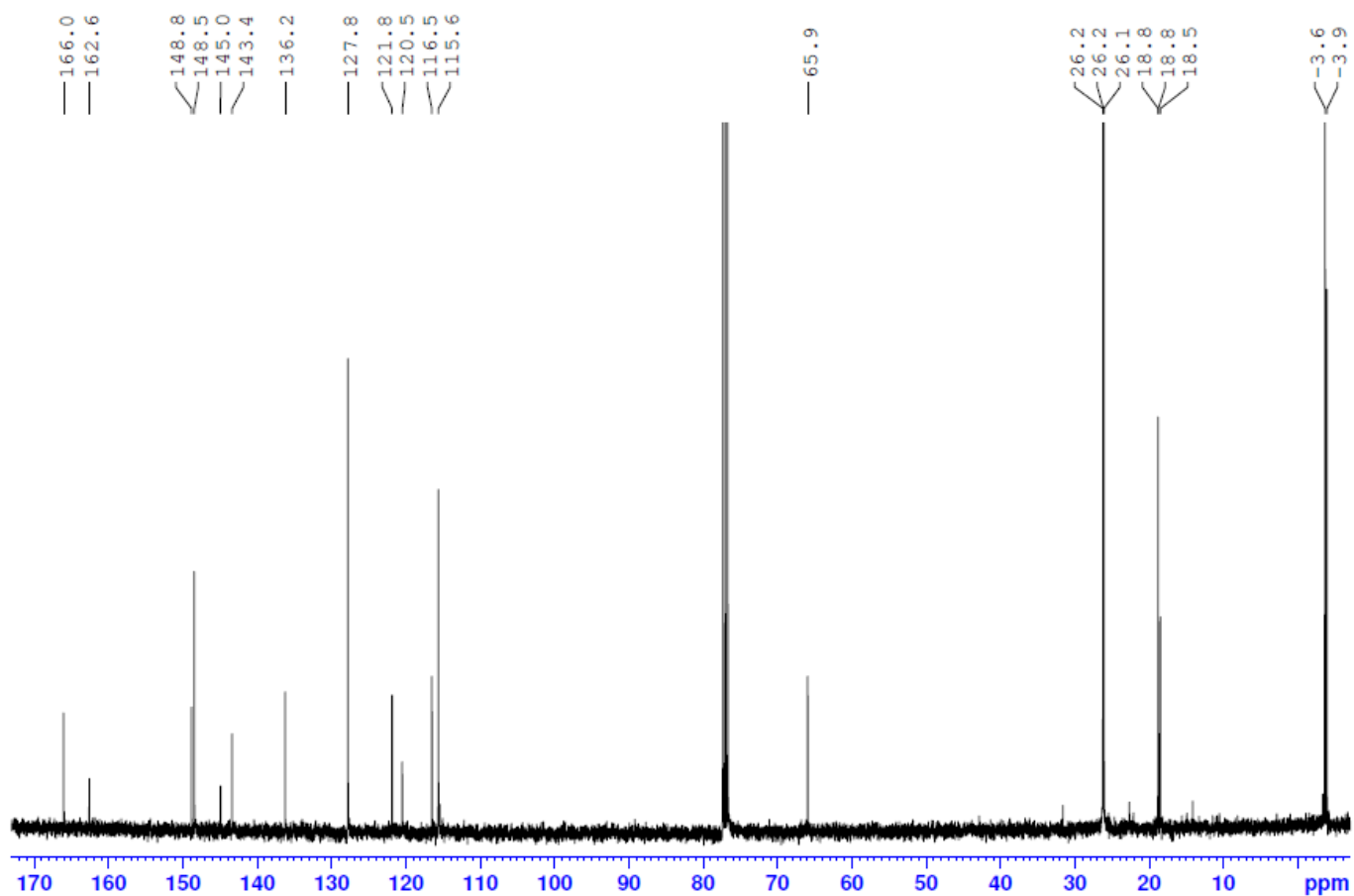
2.6. 1,4-Phenylenebis(methylene) bis[3,4,5-tris((*tert*-butyldimethylsilyl)oxy)benzoate]



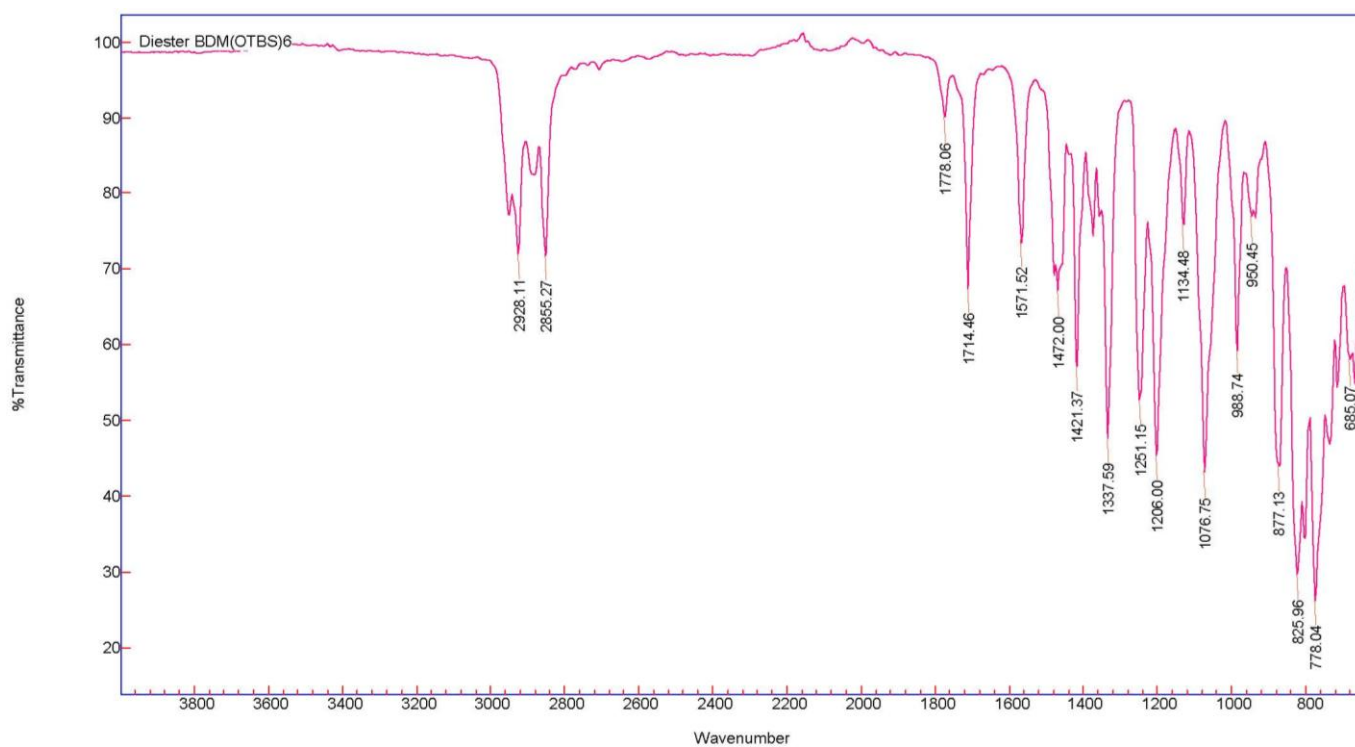
¹H-NMR (400 MHz, CDCl₃), δ (ppm)



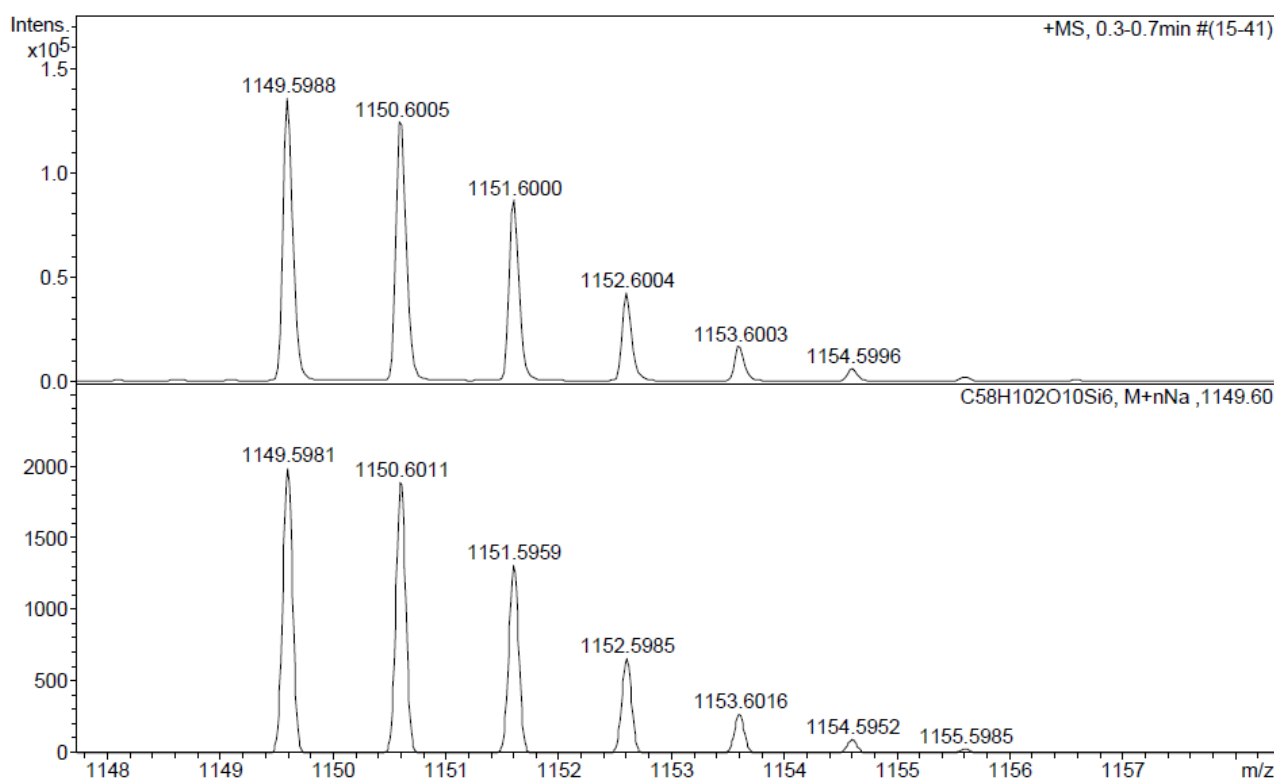
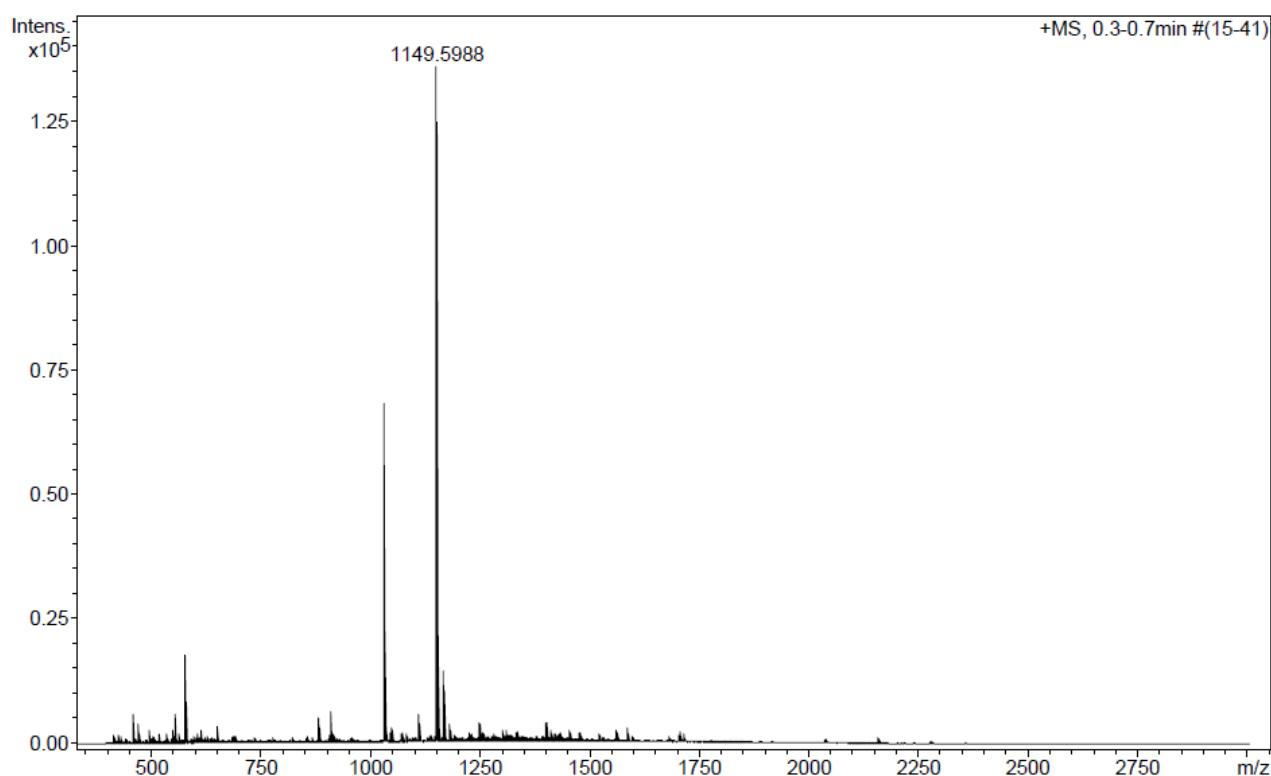
^{13}C -NMR (100 MHz, CDCl_3), δ (ppm)



FT-IR (neat), ν (cm^{-1})

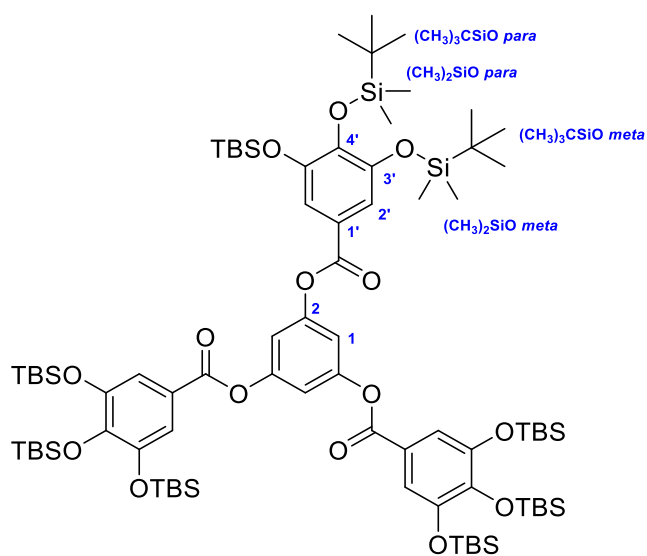


HRMS (m/z)

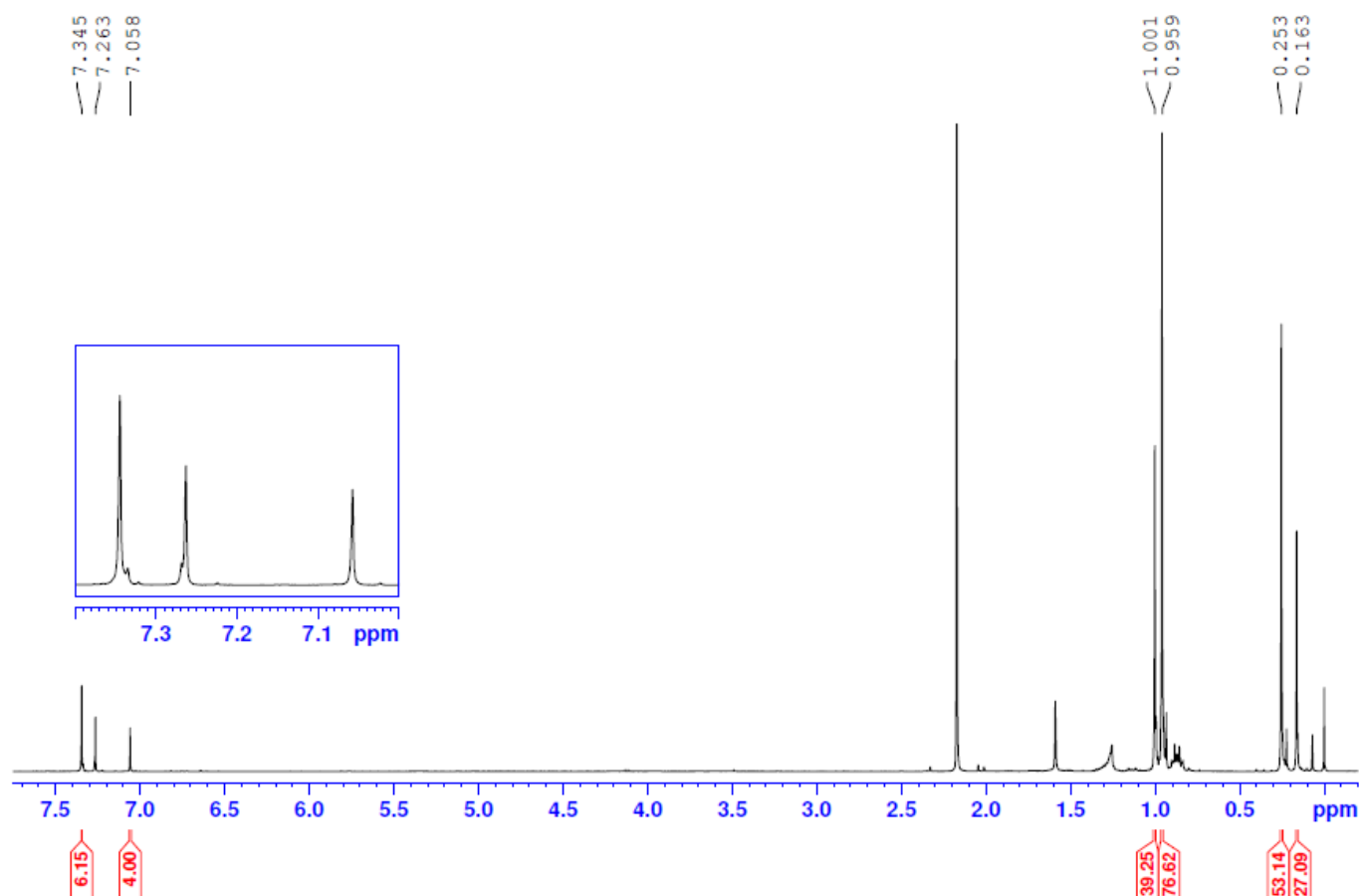


	Molecular formula	Calculated	Found
[M+Na] ⁺	C ₅₈ H ₁₀₂ O ₁₀ Si ₆ Na	1149.5981	1149.5988

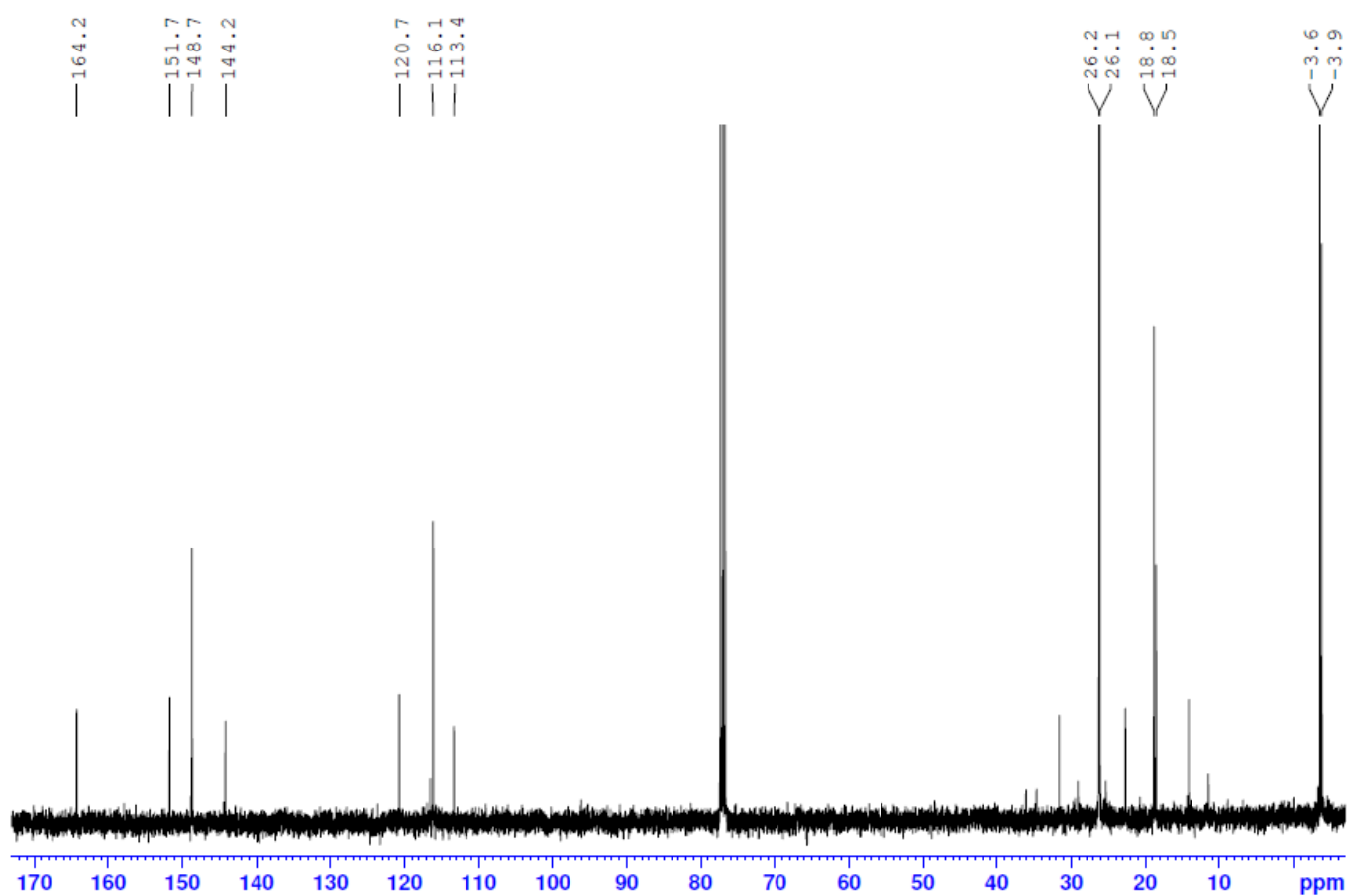
2.7. Benzene-1,3,5-triyl tris[3,4,5-tris((*tert*-butyldimethylsilyl)oxy)benzoate]



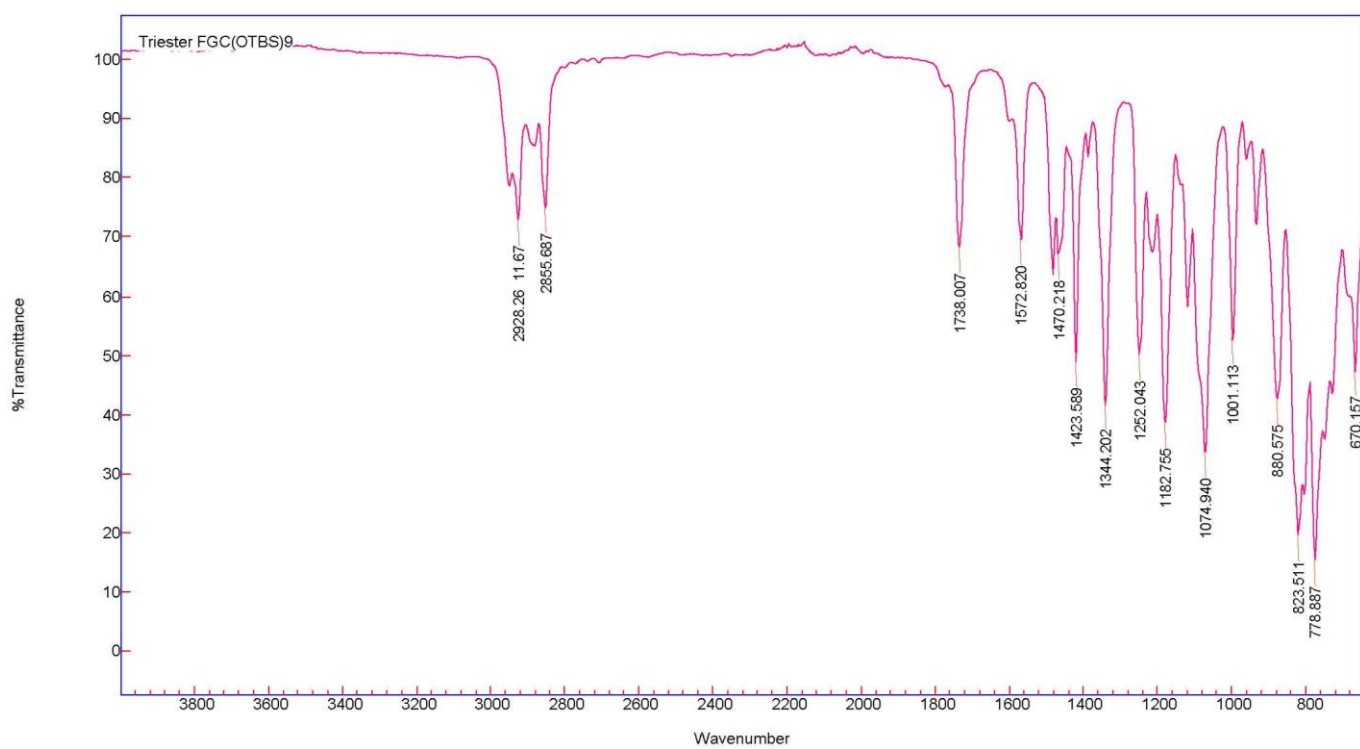
$^1\text{H-NMR}$ (400 MHz, CDCl_3), δ (ppm)



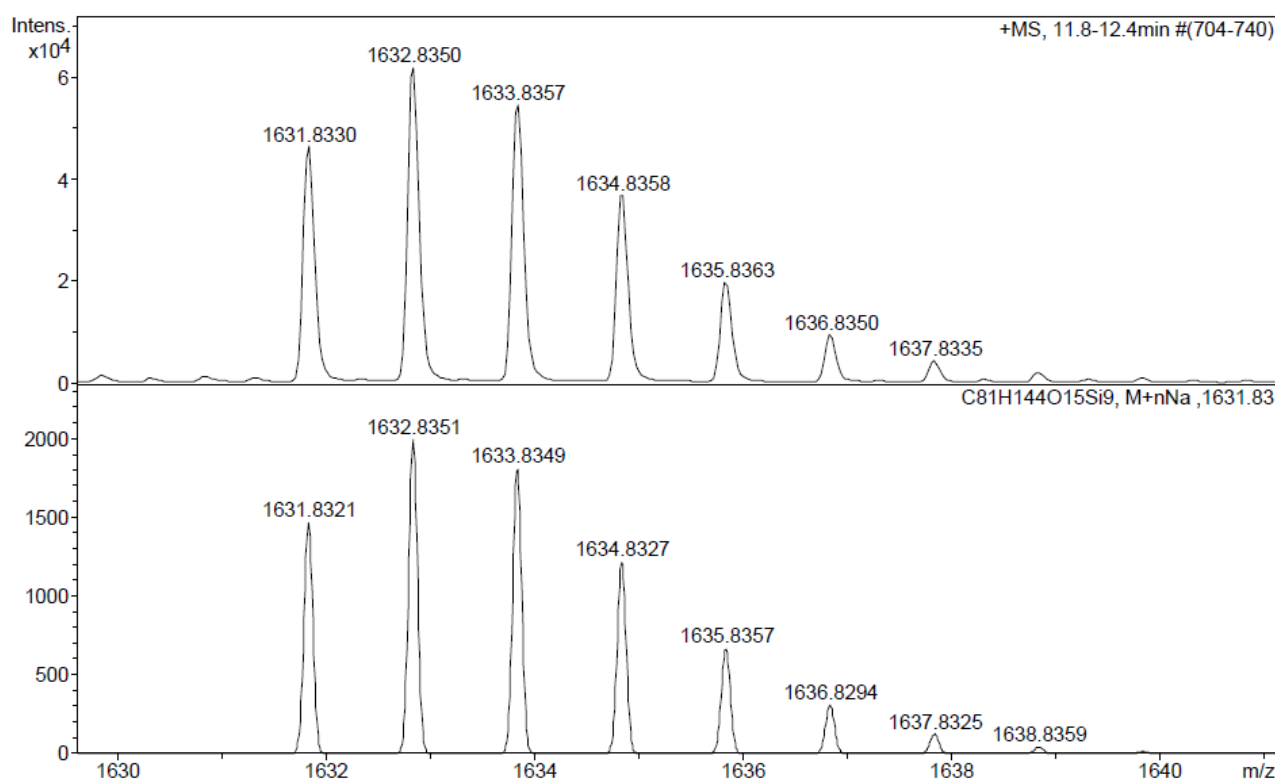
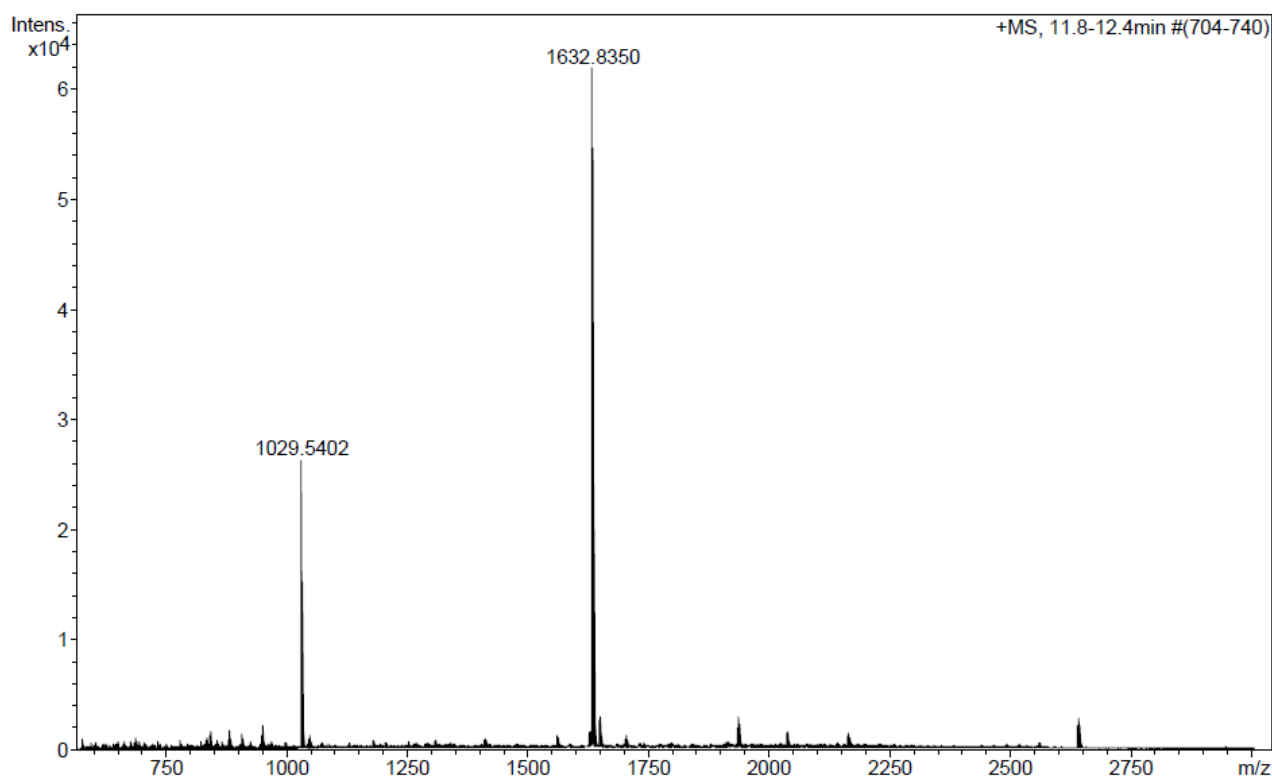
^{13}C -NMR (100 MHz, CDCl_3), δ (ppm)



FT-IR (neat), ν (cm^{-1})

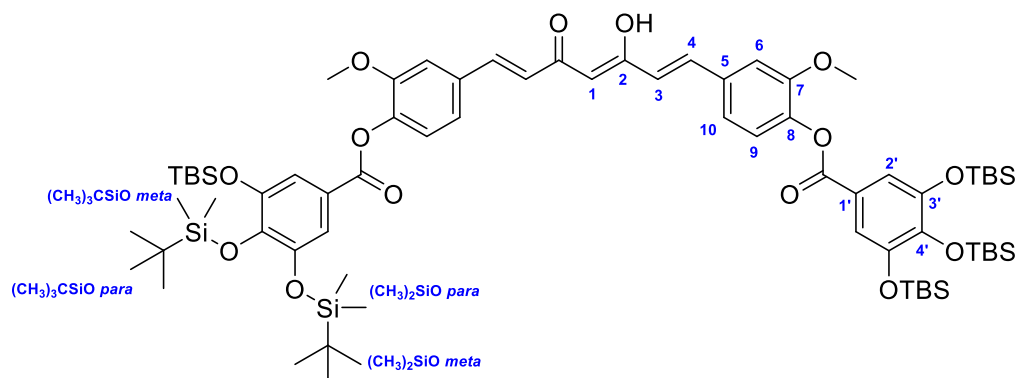


HRMS (m/z)

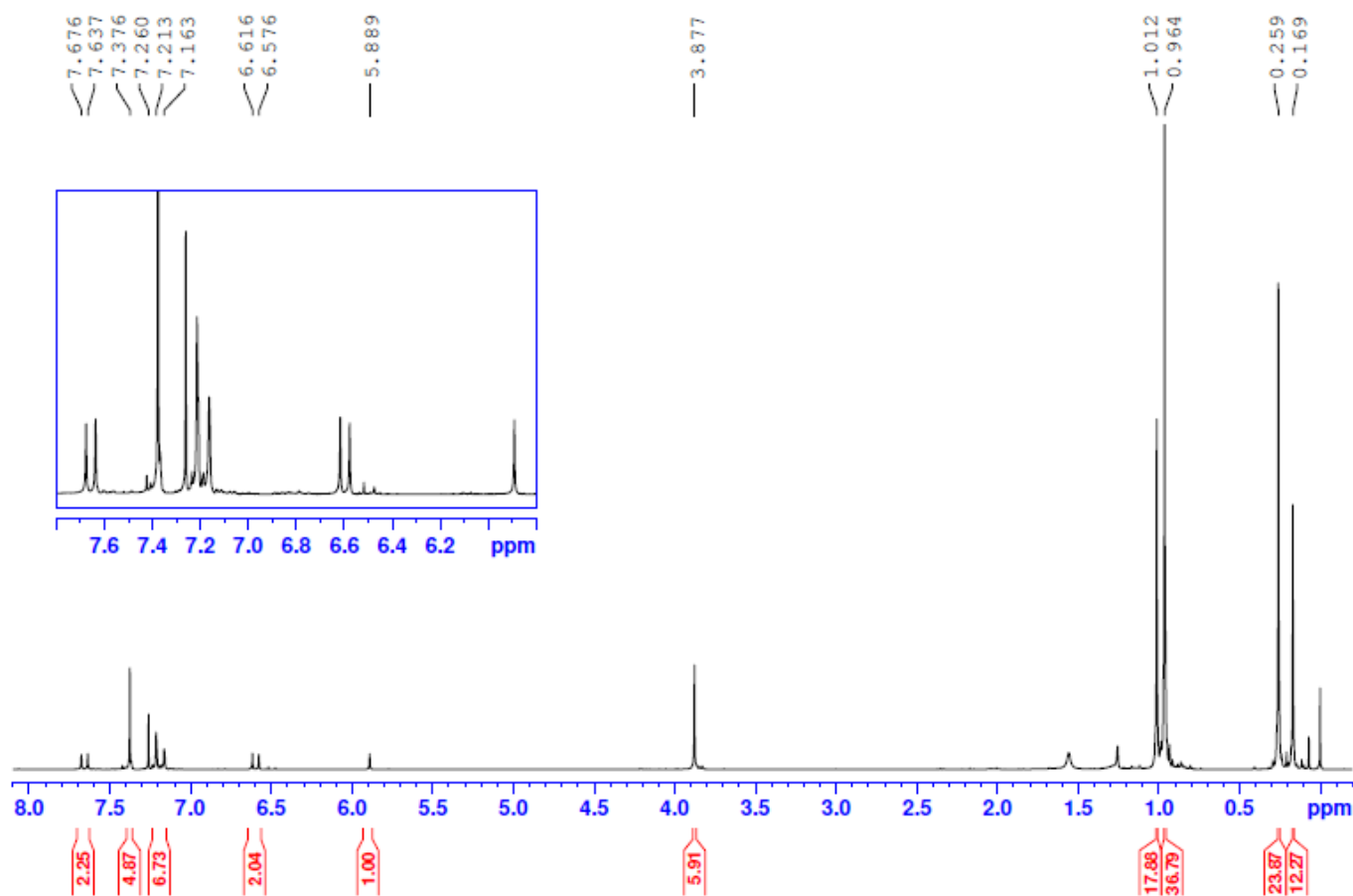


	Molecular formula	Calculated	Found
[M+Na] ⁺	C ₈₁ H ₁₄₄ O ₁₅ Si ₉ Na	1631.8321	1631.8330

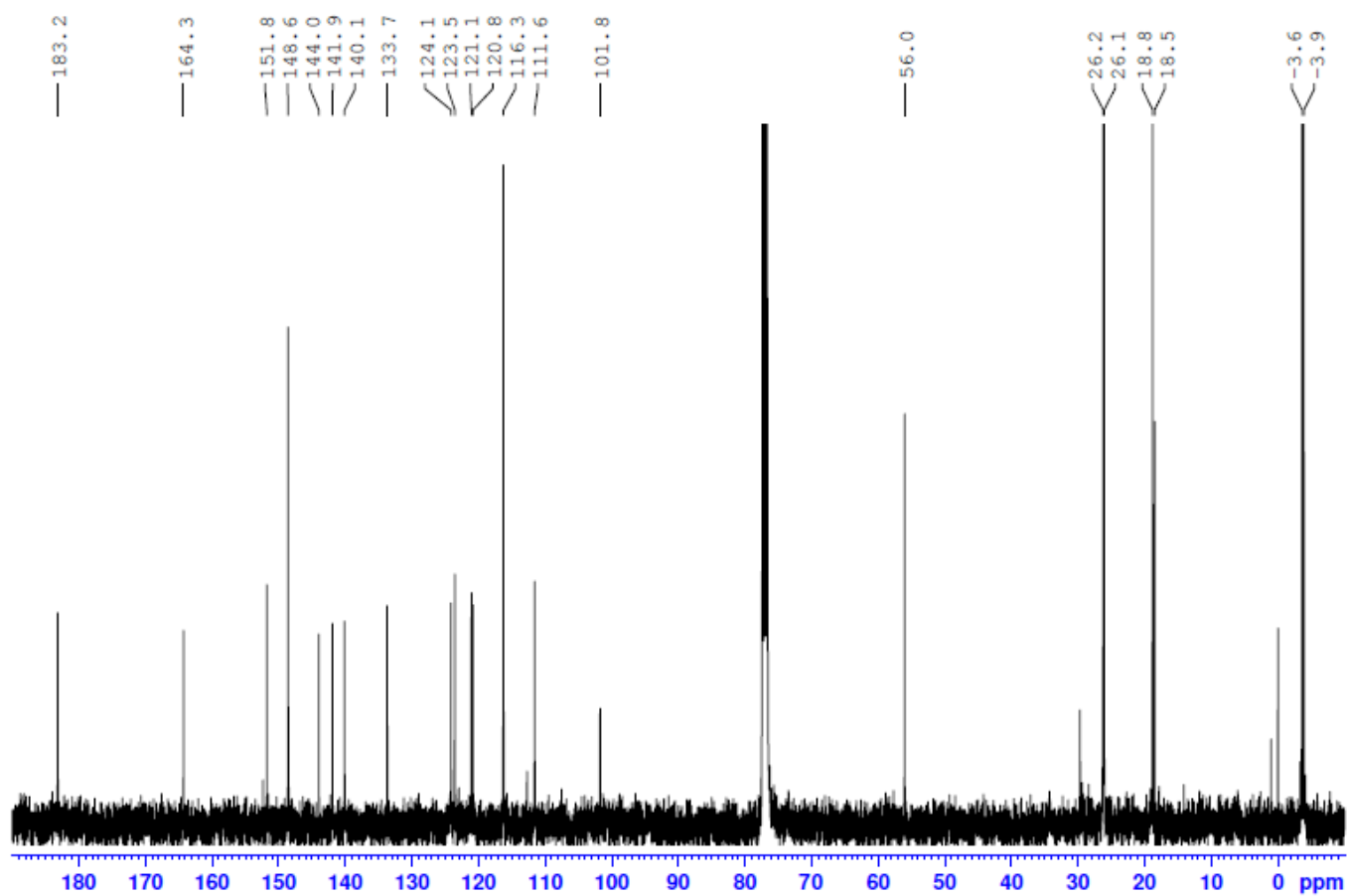
2.8. ((1*E*,3*Z*,6*E*)-3-Hydroxy-5-oxohepta-1,3,6-triene-1,7-diyliden)bis(2-methoxy-4,1-phenylene) bis[3,4,5-tris((*tert*-butyldimethylsilyl)oxy)benzoate]



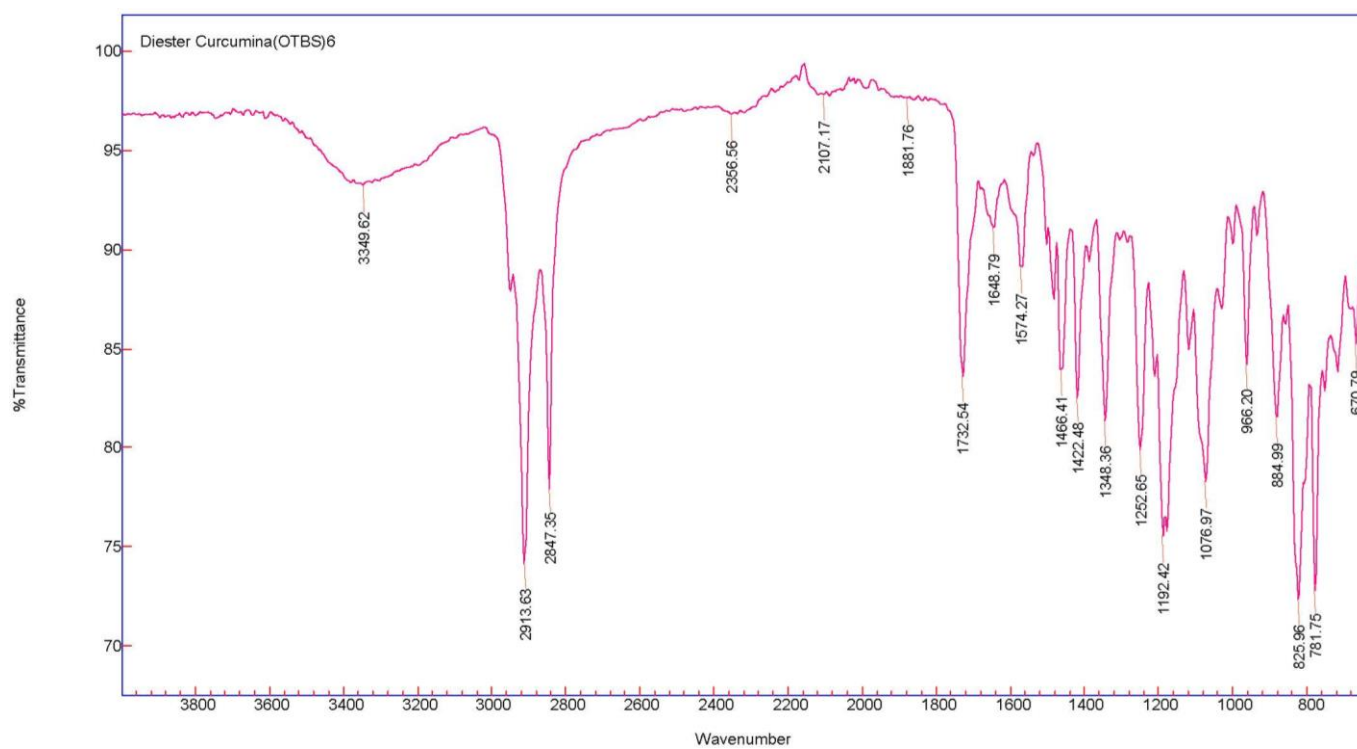
¹H-NMR (400 MHz, CDCl₃), δ (ppm)



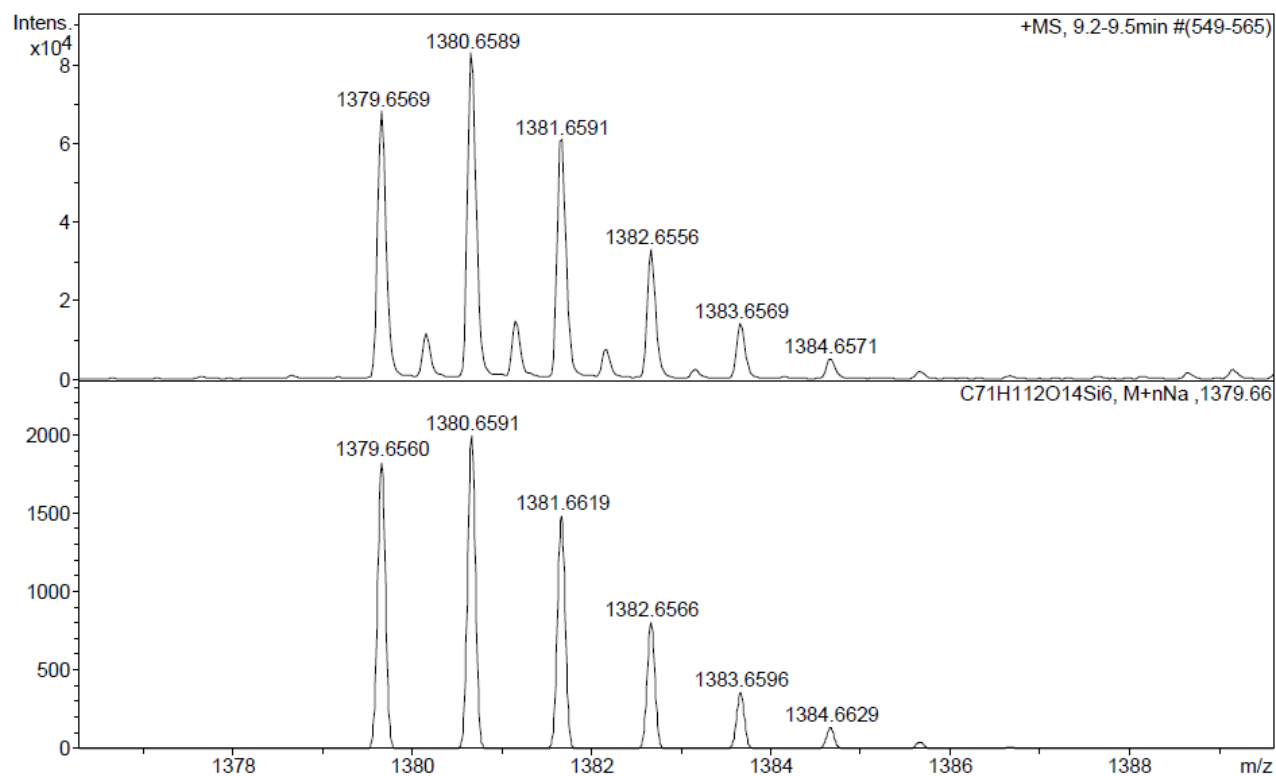
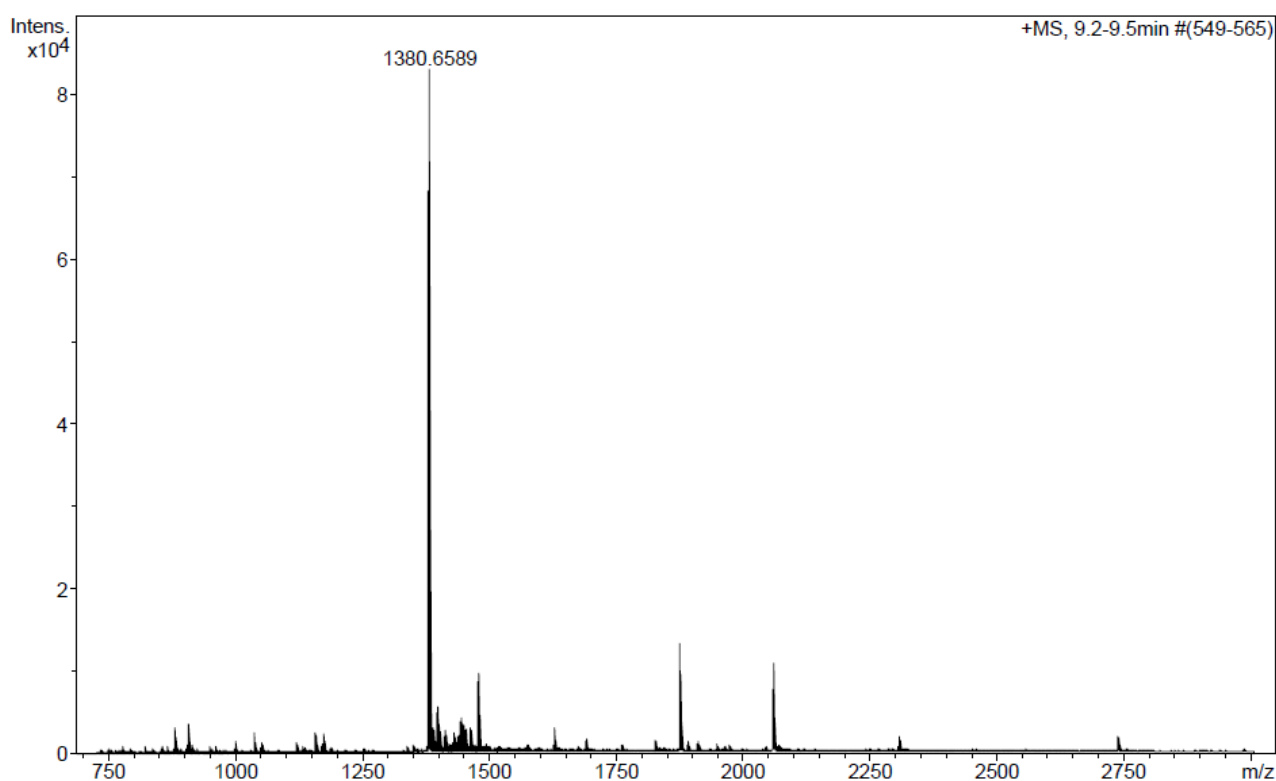
^{13}C -NMR (100 MHz, CDCl_3), δ (ppm)



FT-IR (neat), ν (cm^{-1})

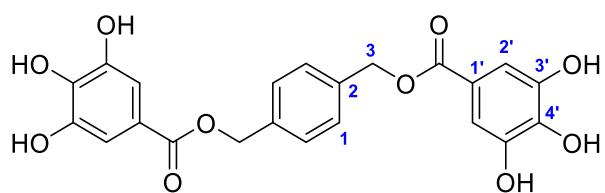


HRMS (m/z)

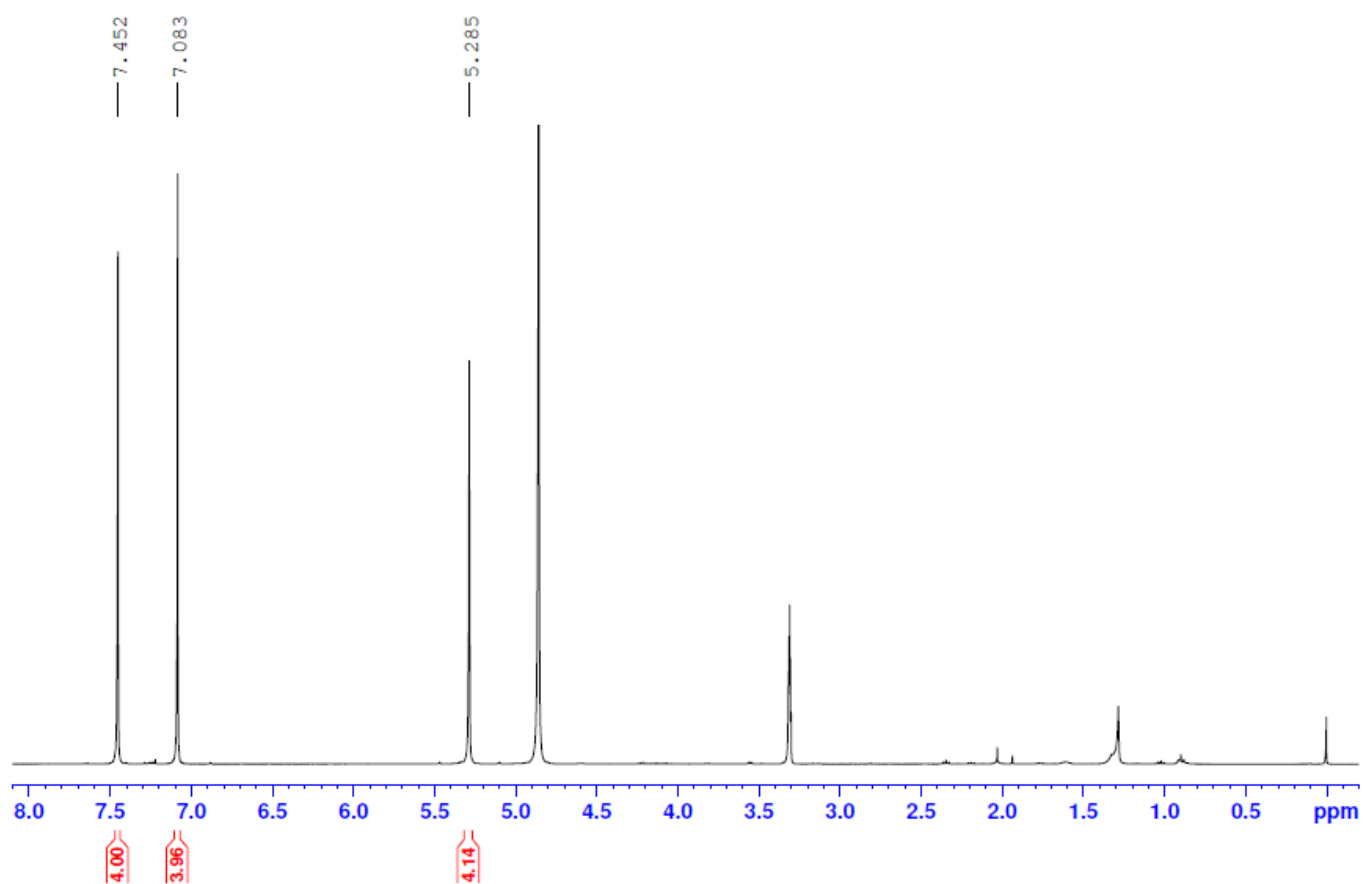


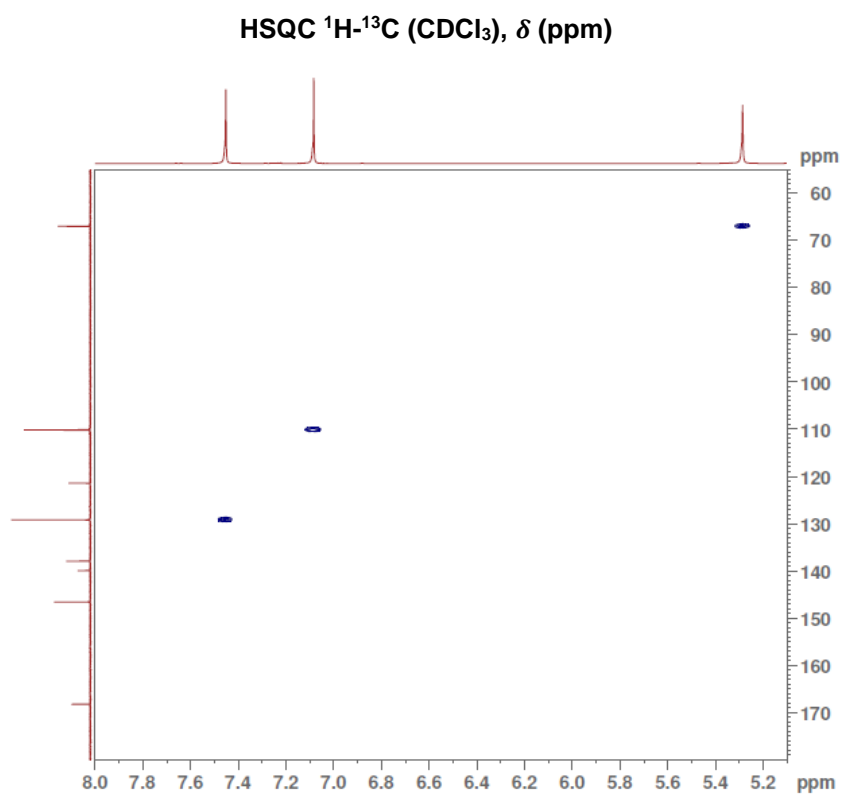
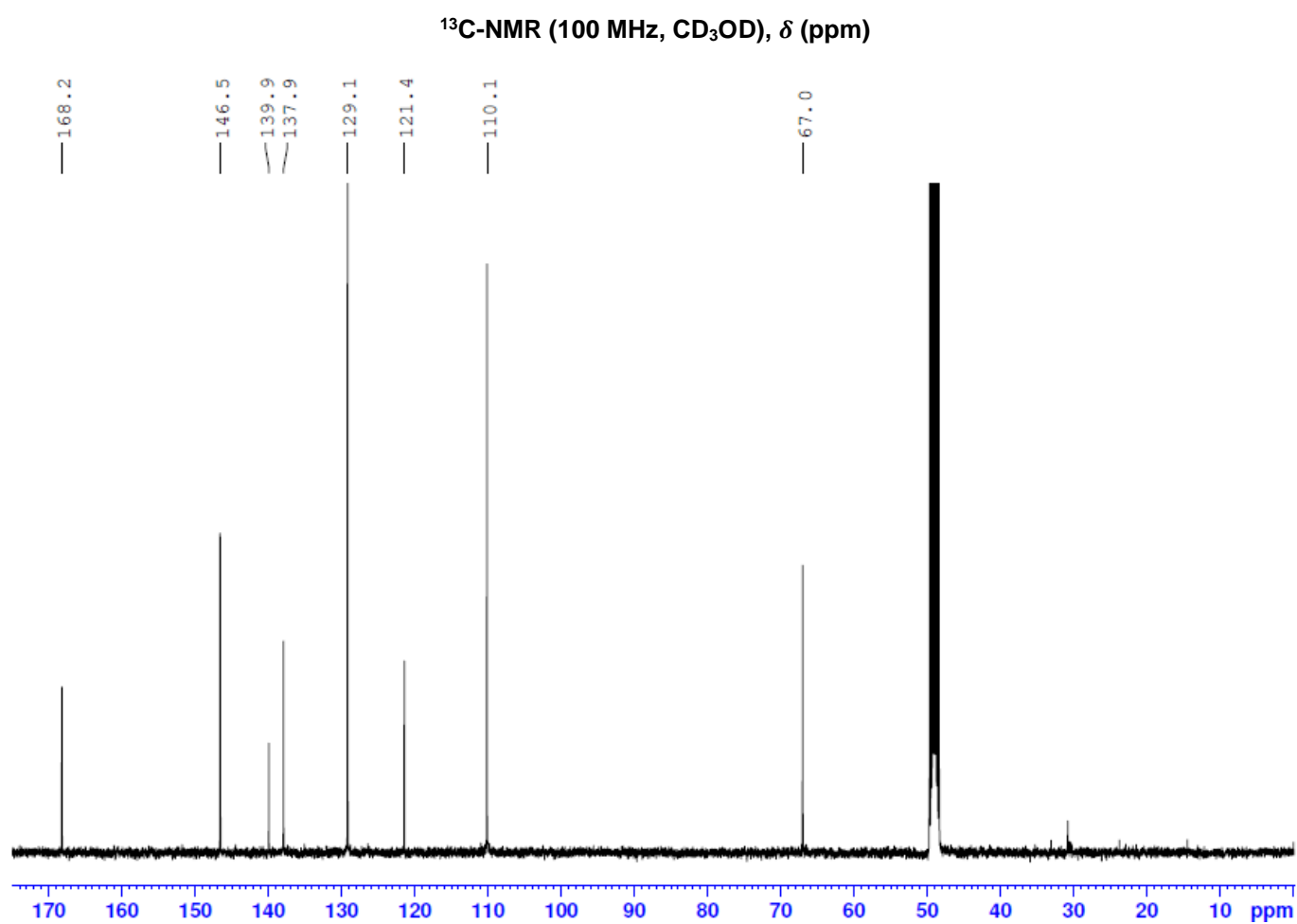
	Molecular formula	Calculated	Found
[M+Na] ⁺	C ₇₁ H ₁₁₂ O ₁₄ Si ₆ Na	1379.6560	1379.6569

2.9. 1,4-Phenylenebis(methylene) bis(3,4,5-trihydroxybenzoate) (TL1)

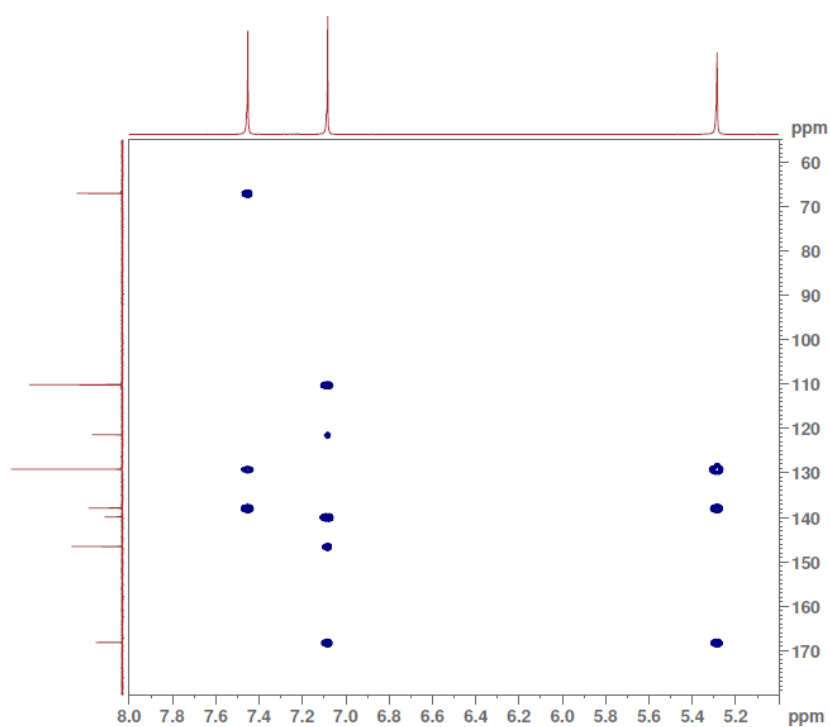


¹H-NMR (400 MHz, CD₃OD), δ (ppm)

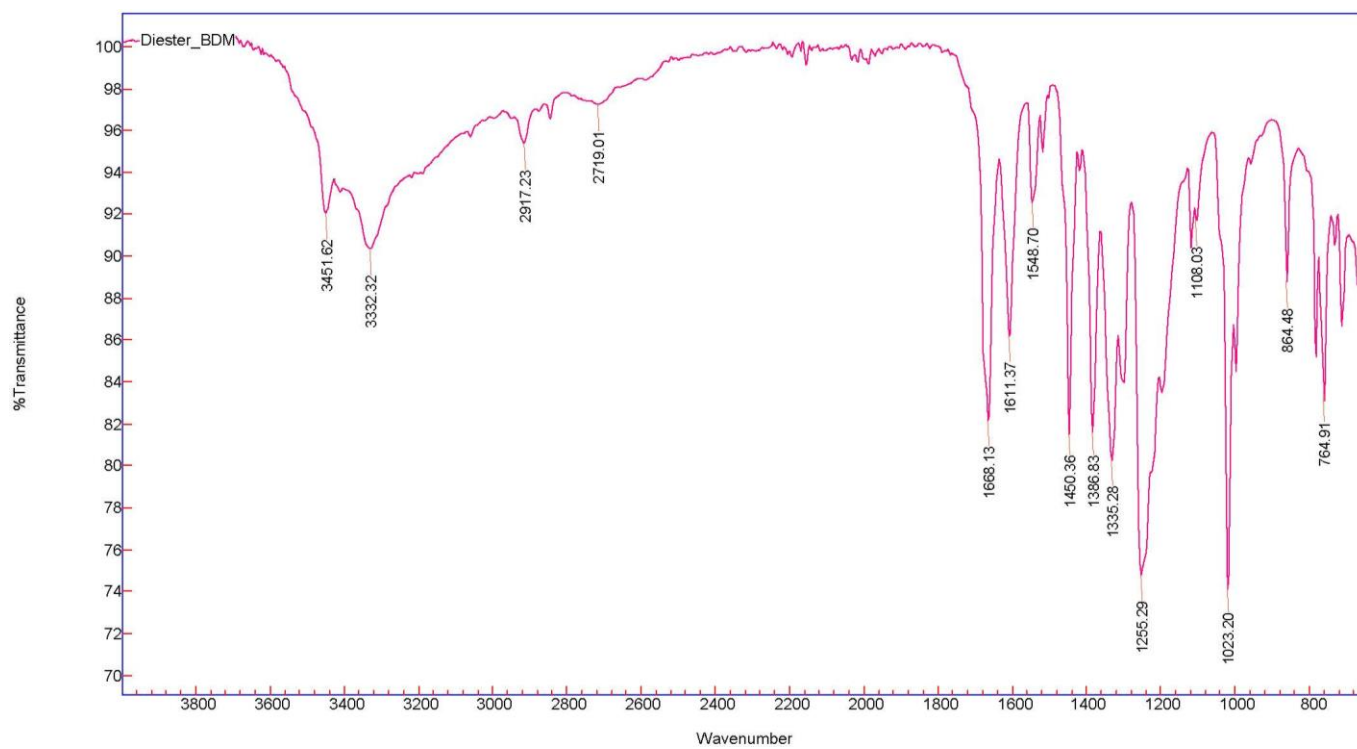




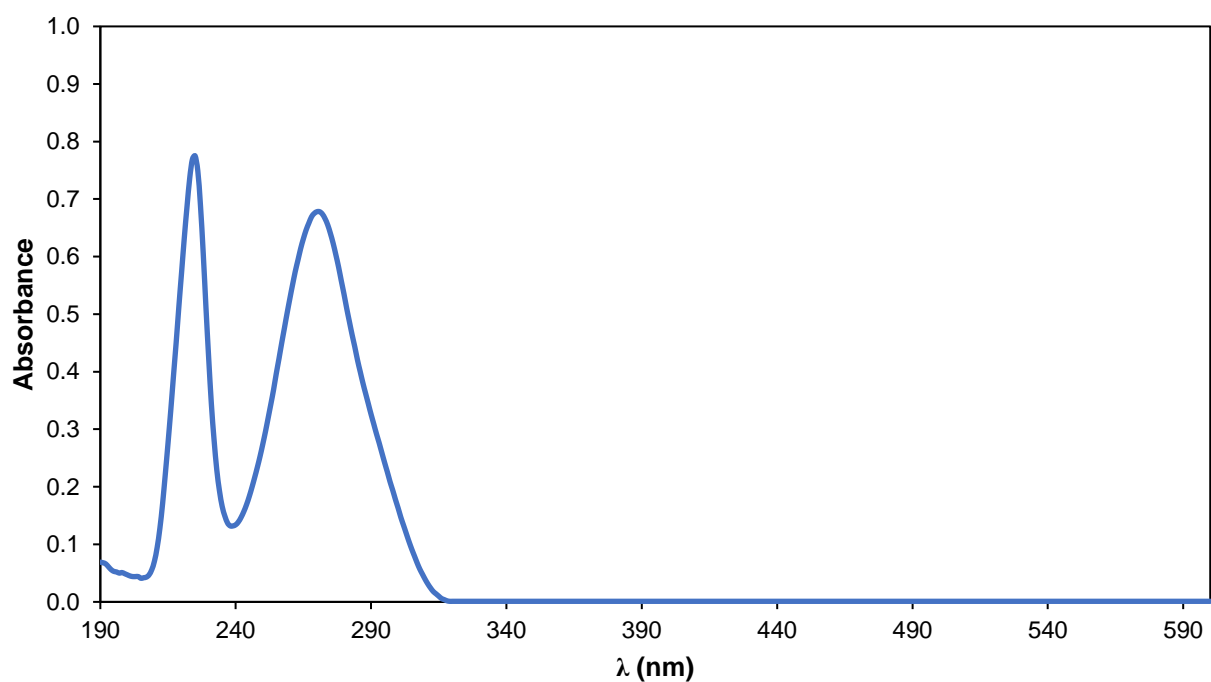
HMBC ^1H - ^{13}C (CDCl_3), δ (ppm)



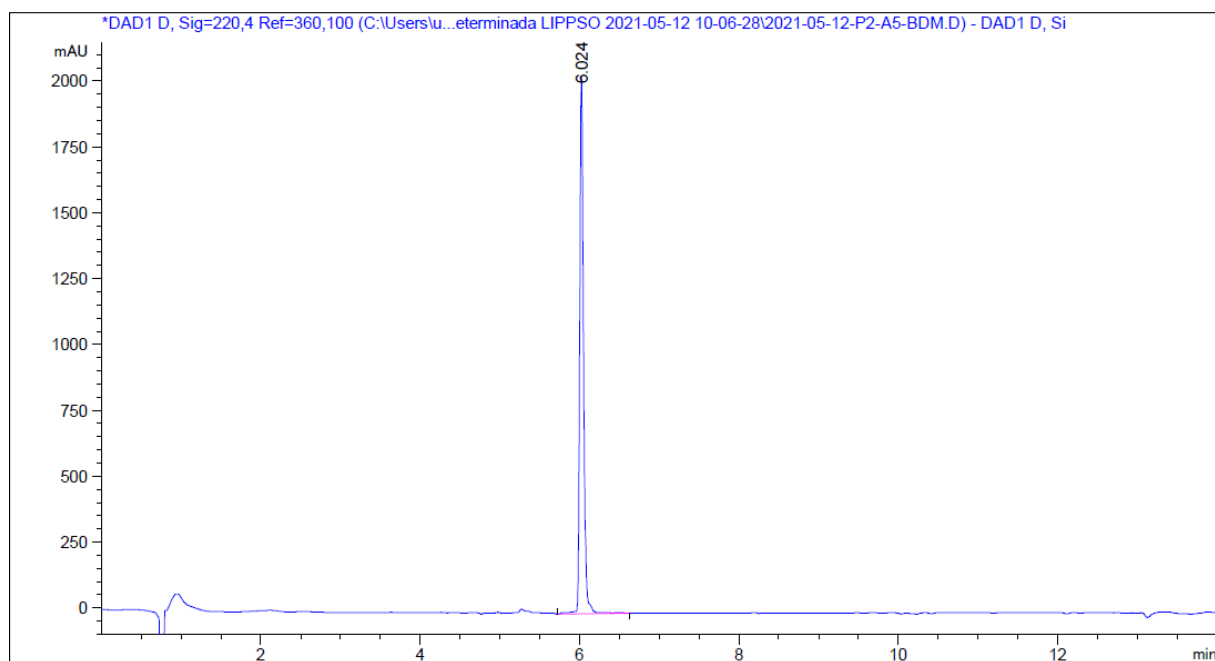
FT-IR (neat), ν (cm^{-1})



UV-Vis (CH₃CN), λ (nm)

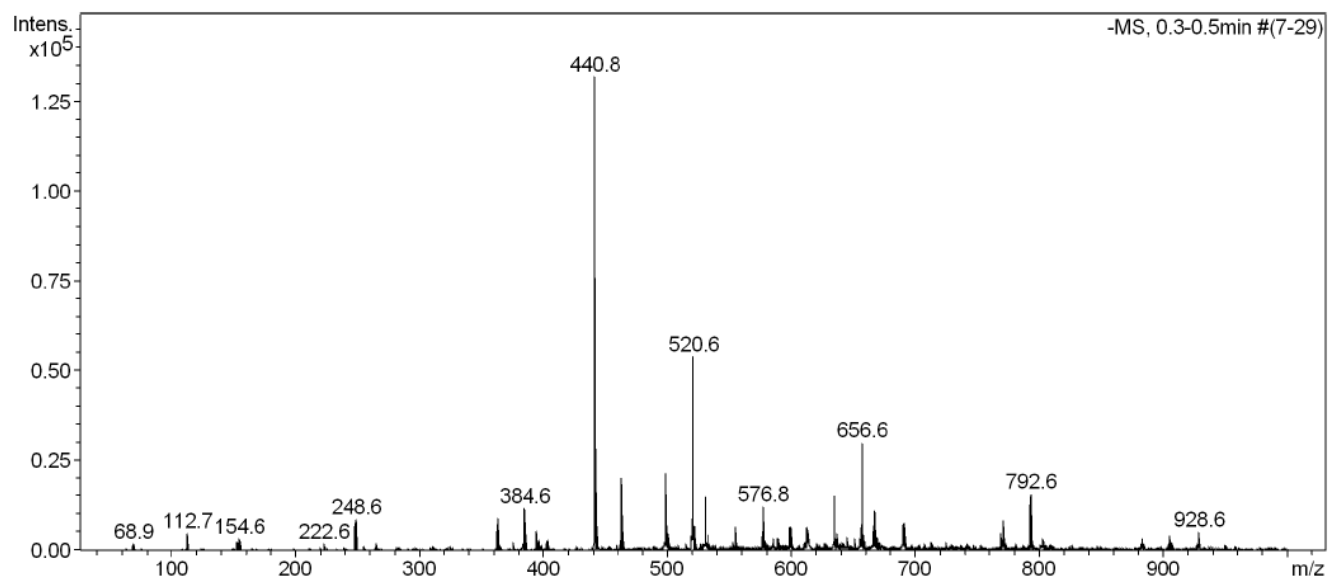


HPLC ($\lambda = 220$ nm)

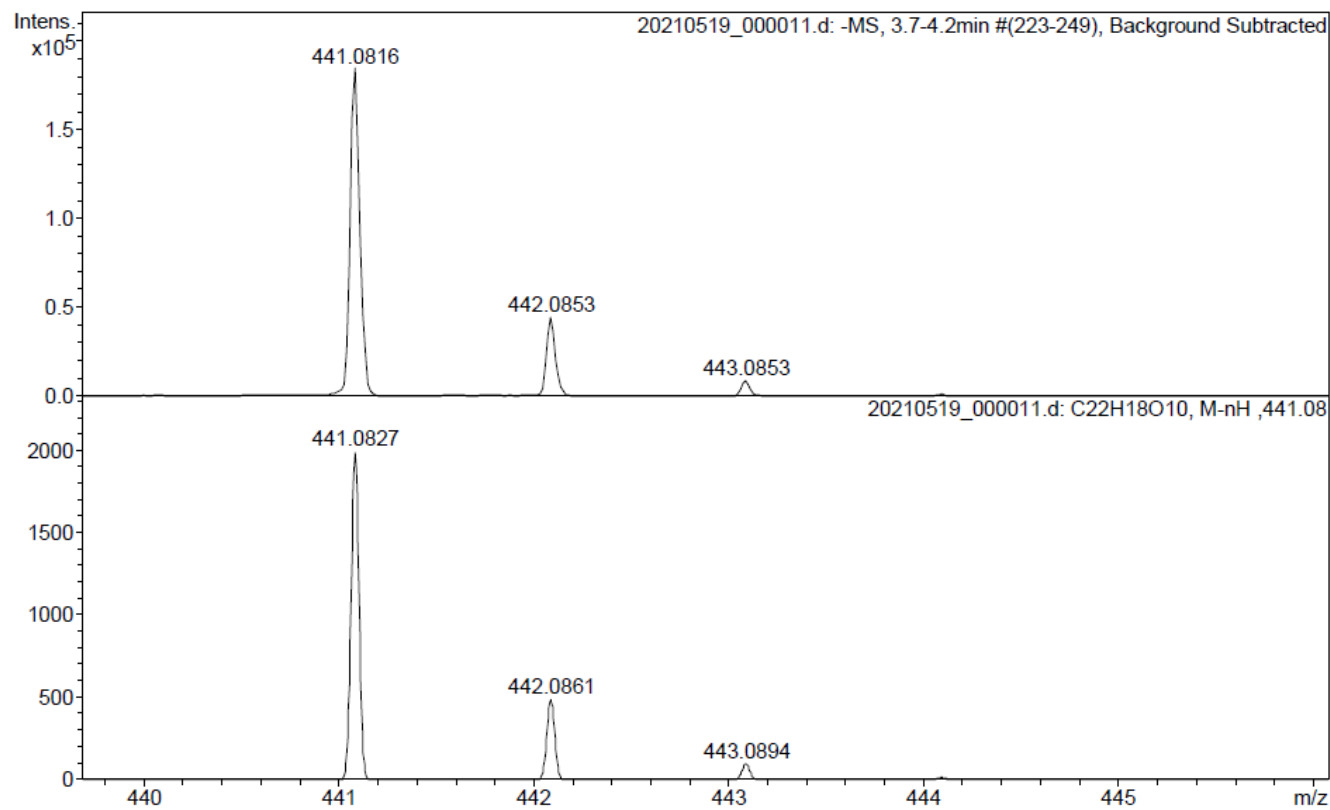
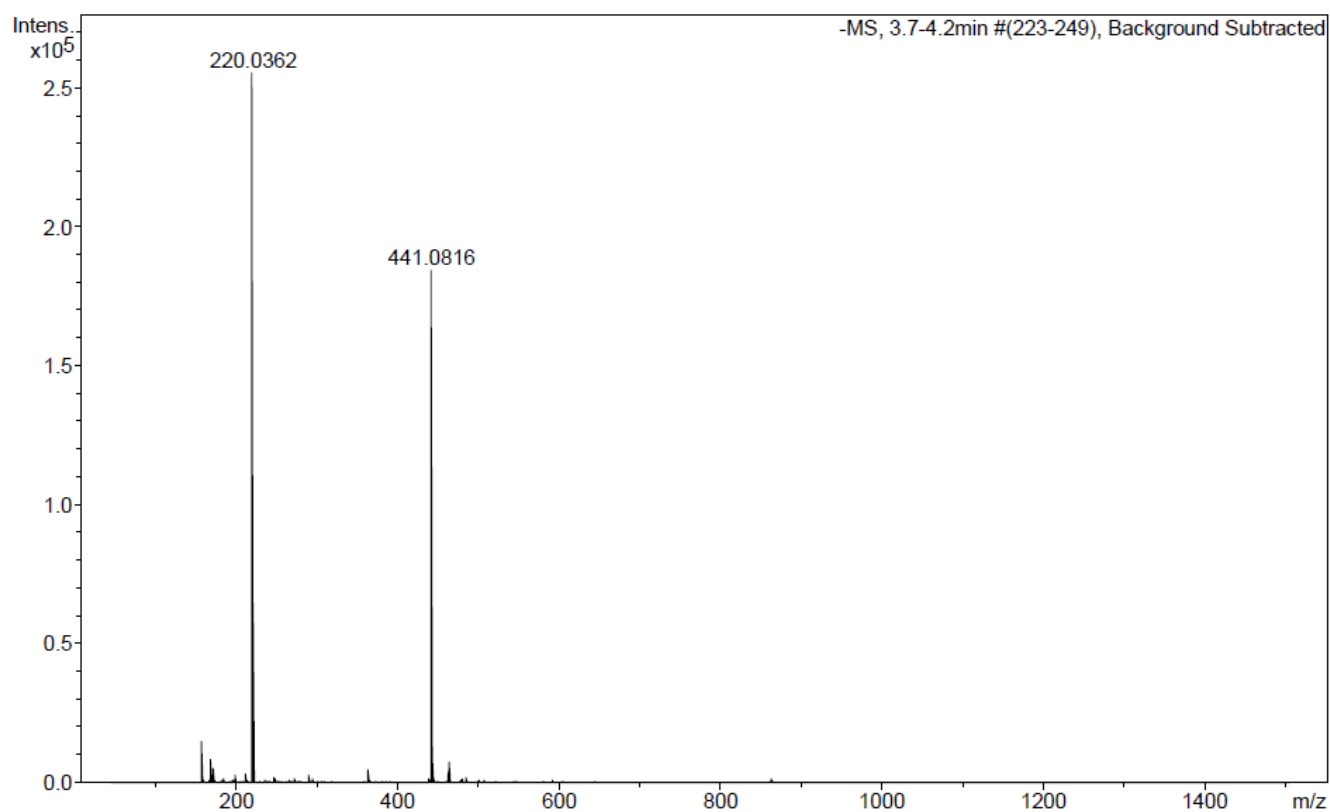


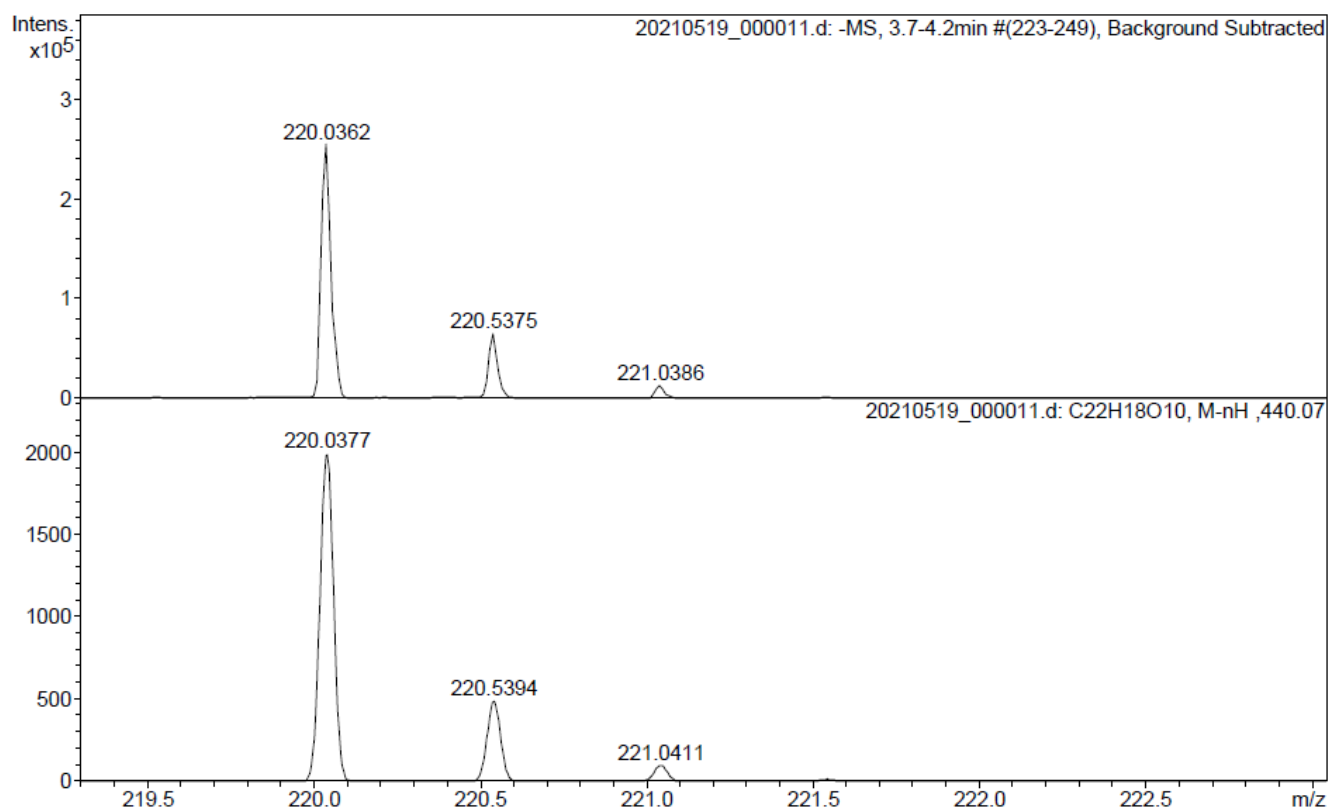
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.024	BV R	0.0510	6825.30957	2041.29993	100.0000
Totals :				6825.30957	2041.29993	

ESI-MS (m/z)



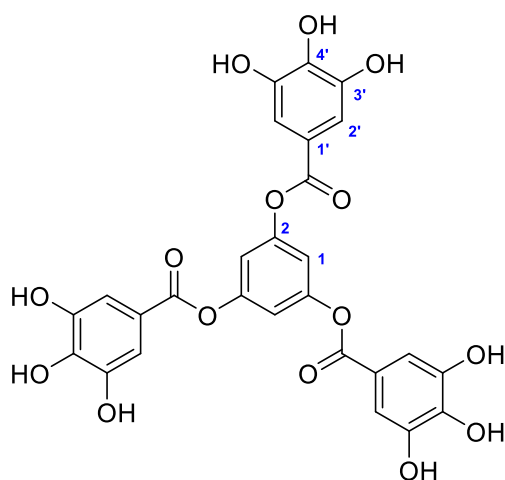
HRMS (m/z)



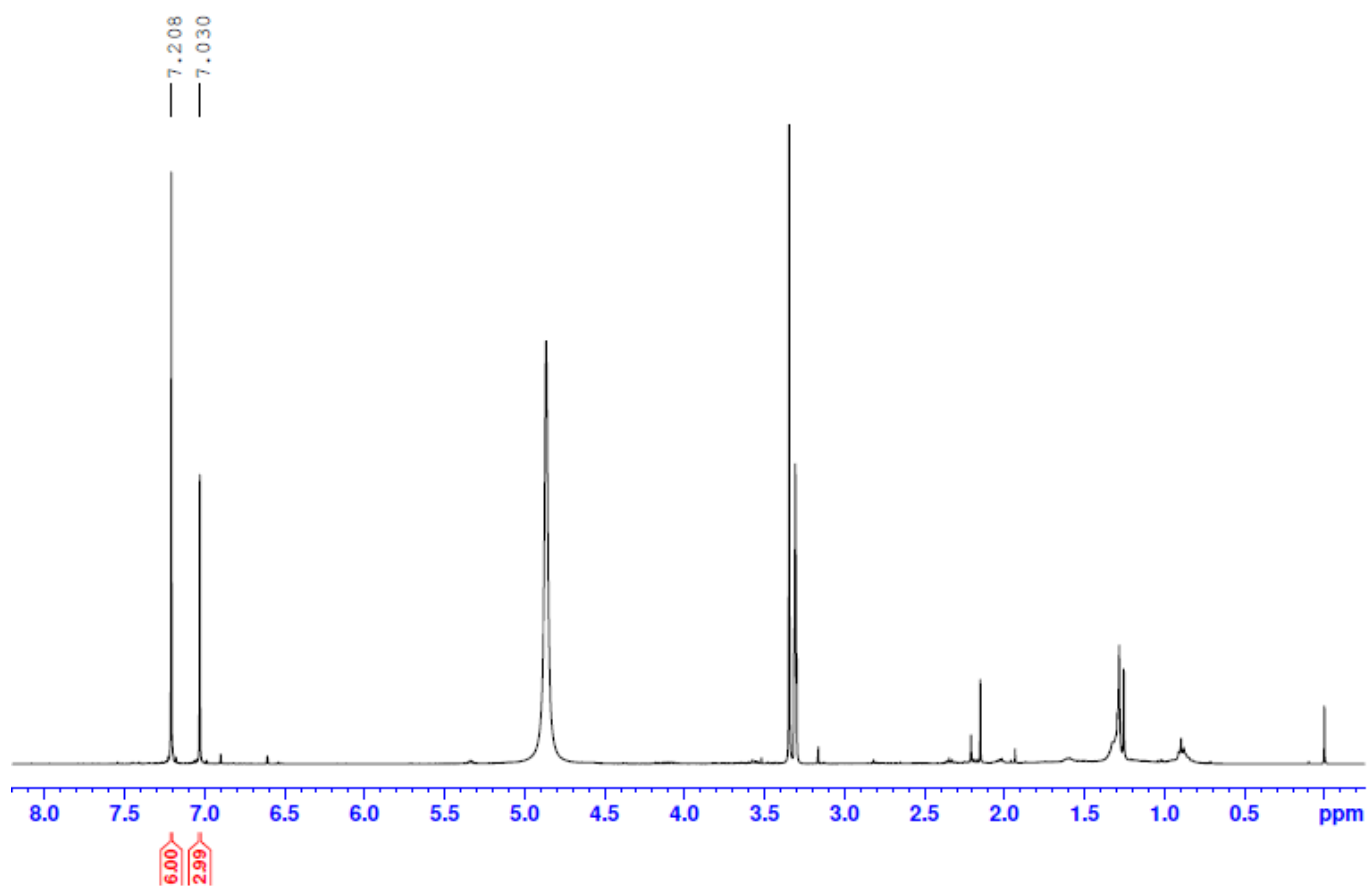


	Molecular formula	Calculated	Found
[M-H] ⁻	C ₂₂ H ₁₉ O ₁₀	441.0827	441.0816
[M-2H] ²⁻	C ₂₂ H ₁₈ O ₁₀	220.0377	220.0362

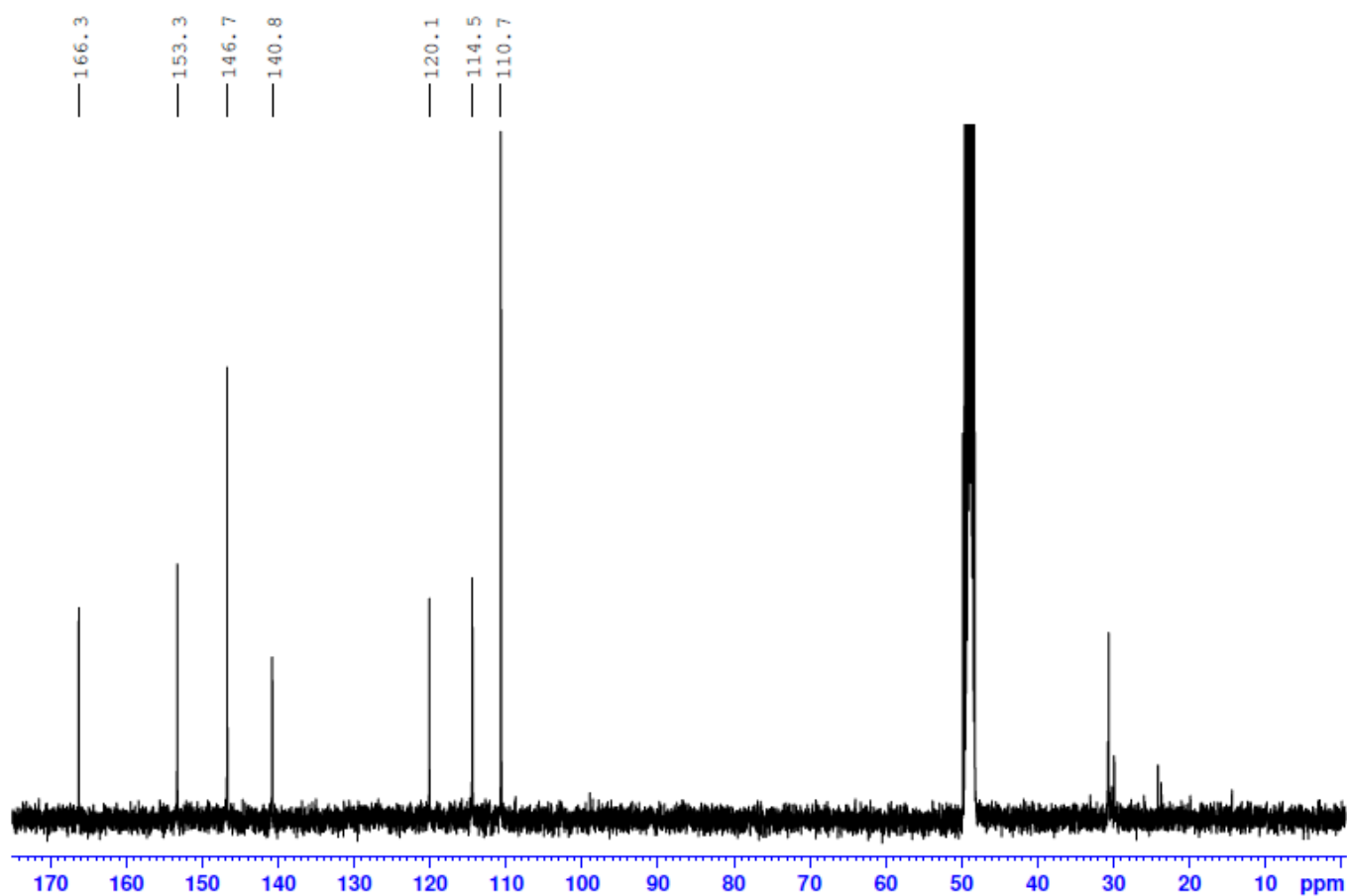
2.10. Benzene-1,3,5-triyl tris(3,4,5-trihydroxybenzoate) (TL2)



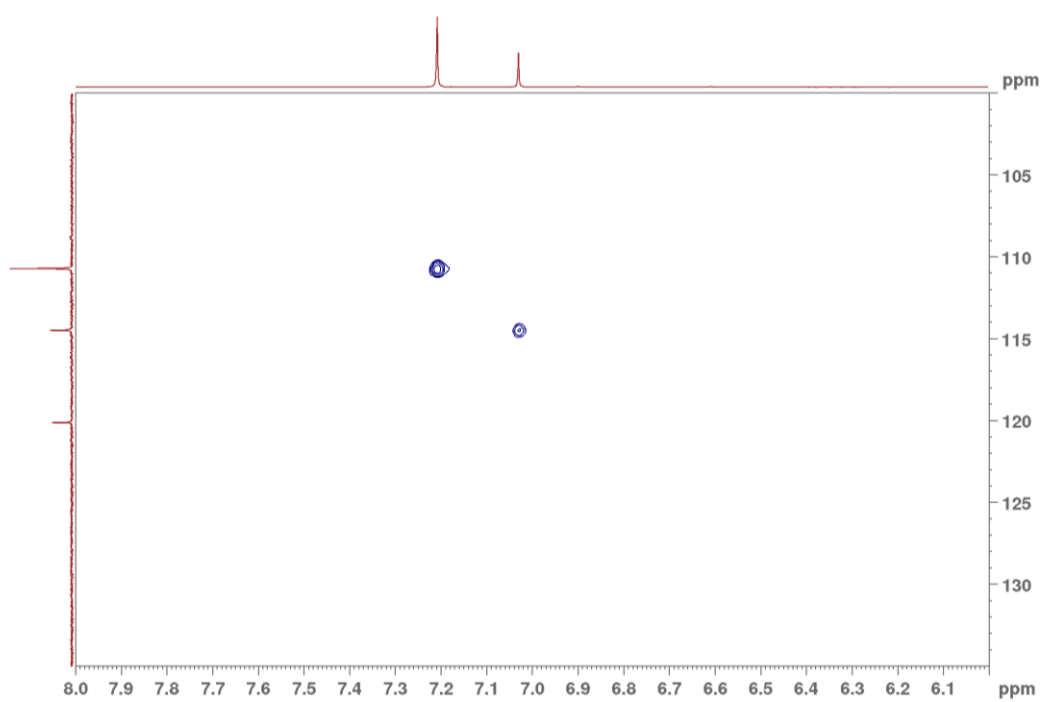
$^1\text{H-NMR}$ (400 MHz, CD_3OD), δ (ppm)



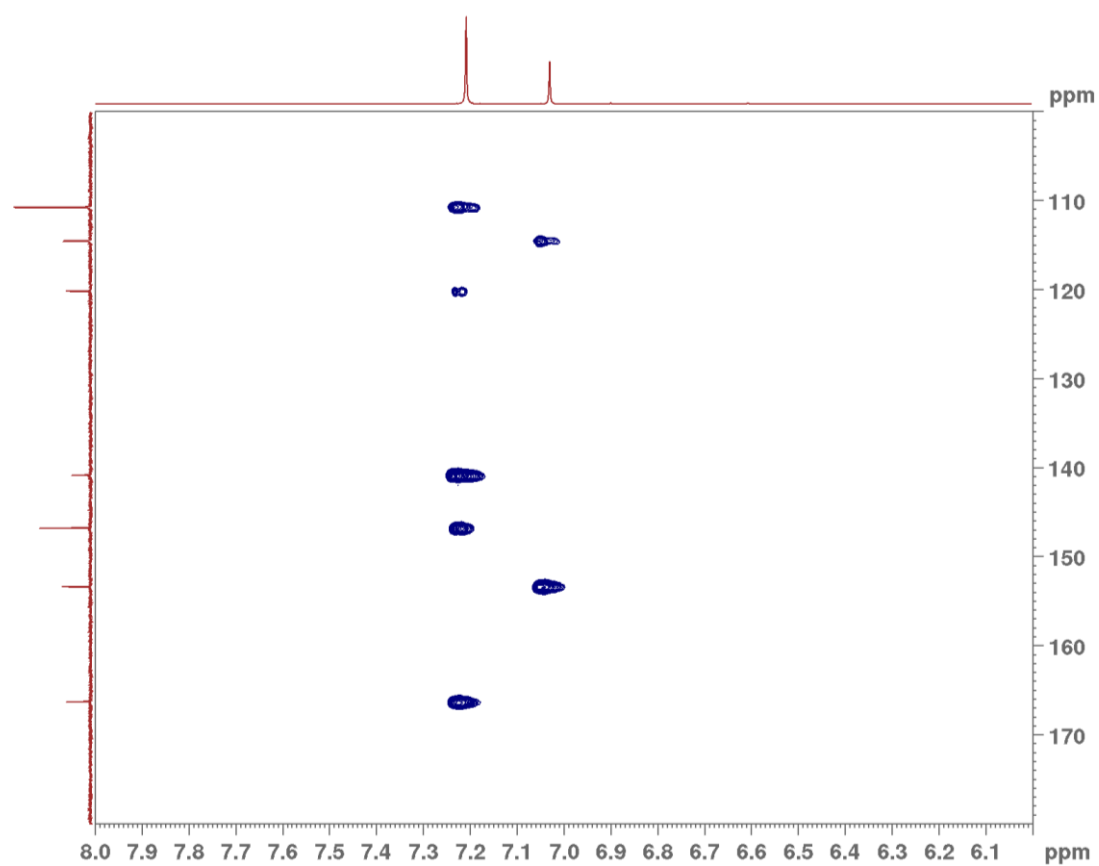
^{13}C -NMR (100 MHz, CD_3OD), δ (ppm)



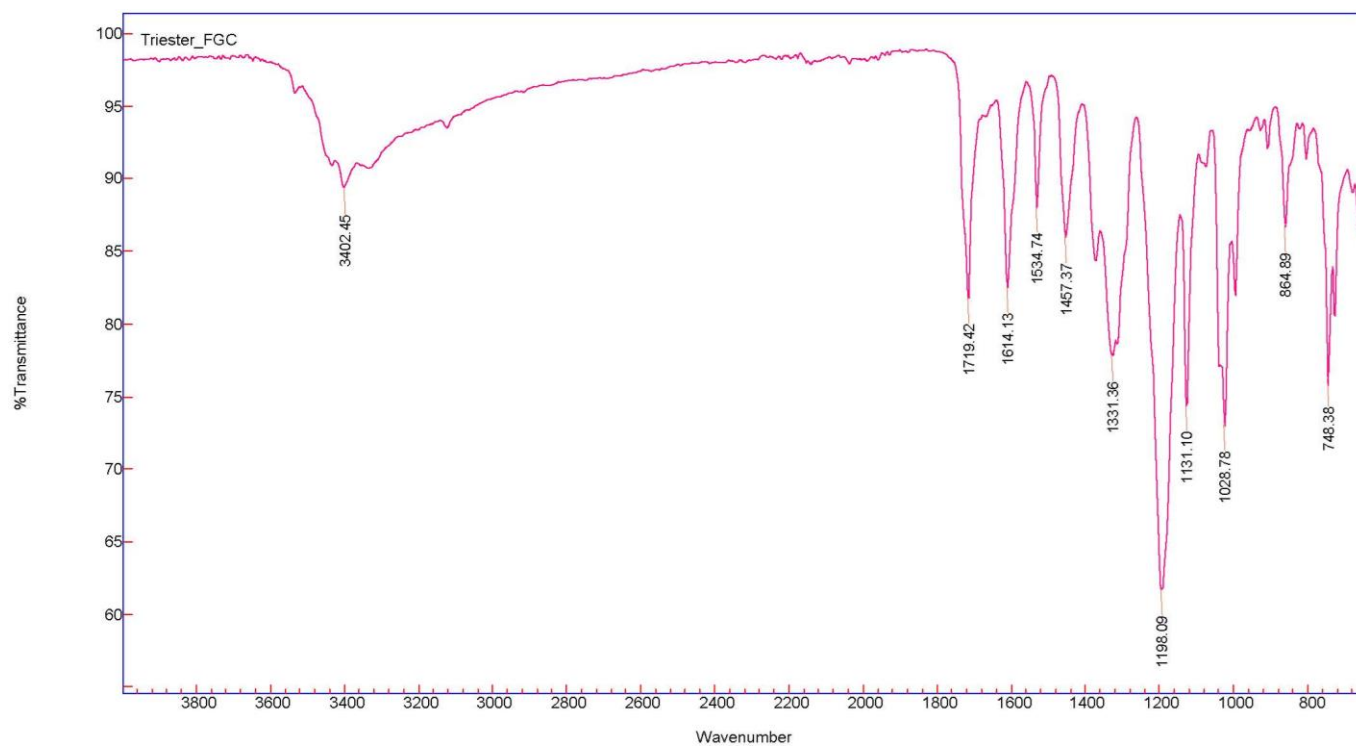
HSQC ^1H - ^{13}C (CD_3OD), δ (ppm)



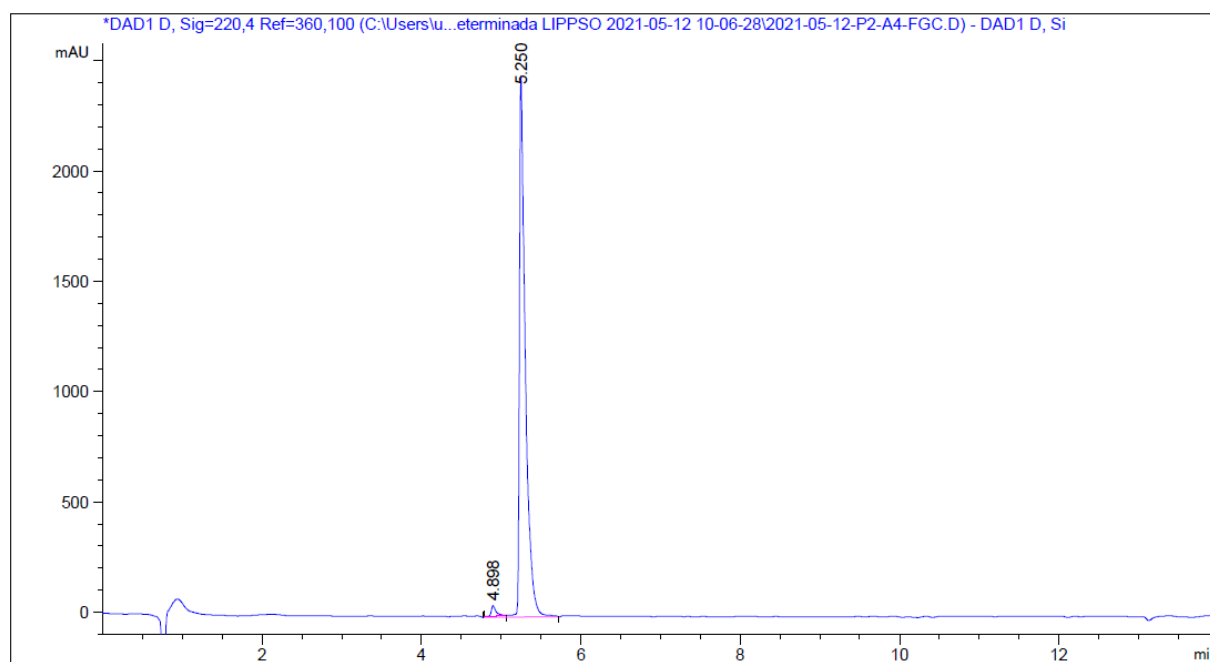
HMBC ^1H - ^{13}C (CD_3OD), δ (ppm)



FT-IR (neat), ν (cm^{-1})



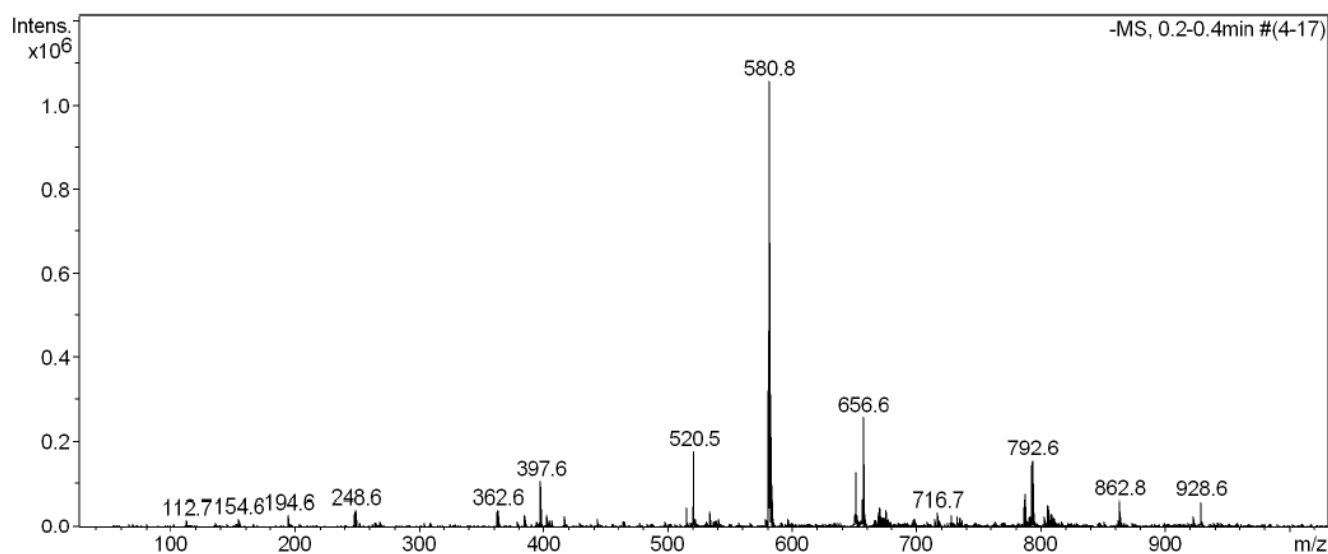
HPLC ($\lambda = 220$ nm)



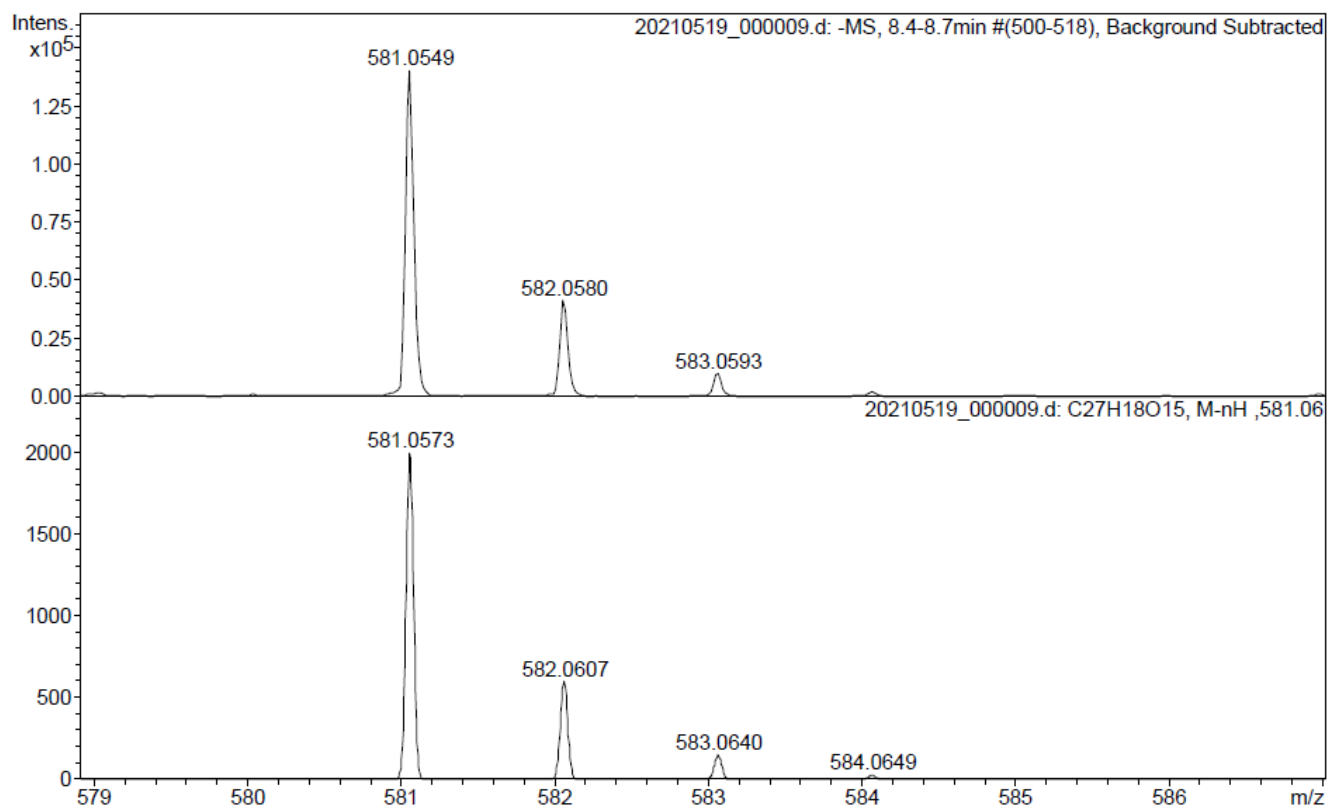
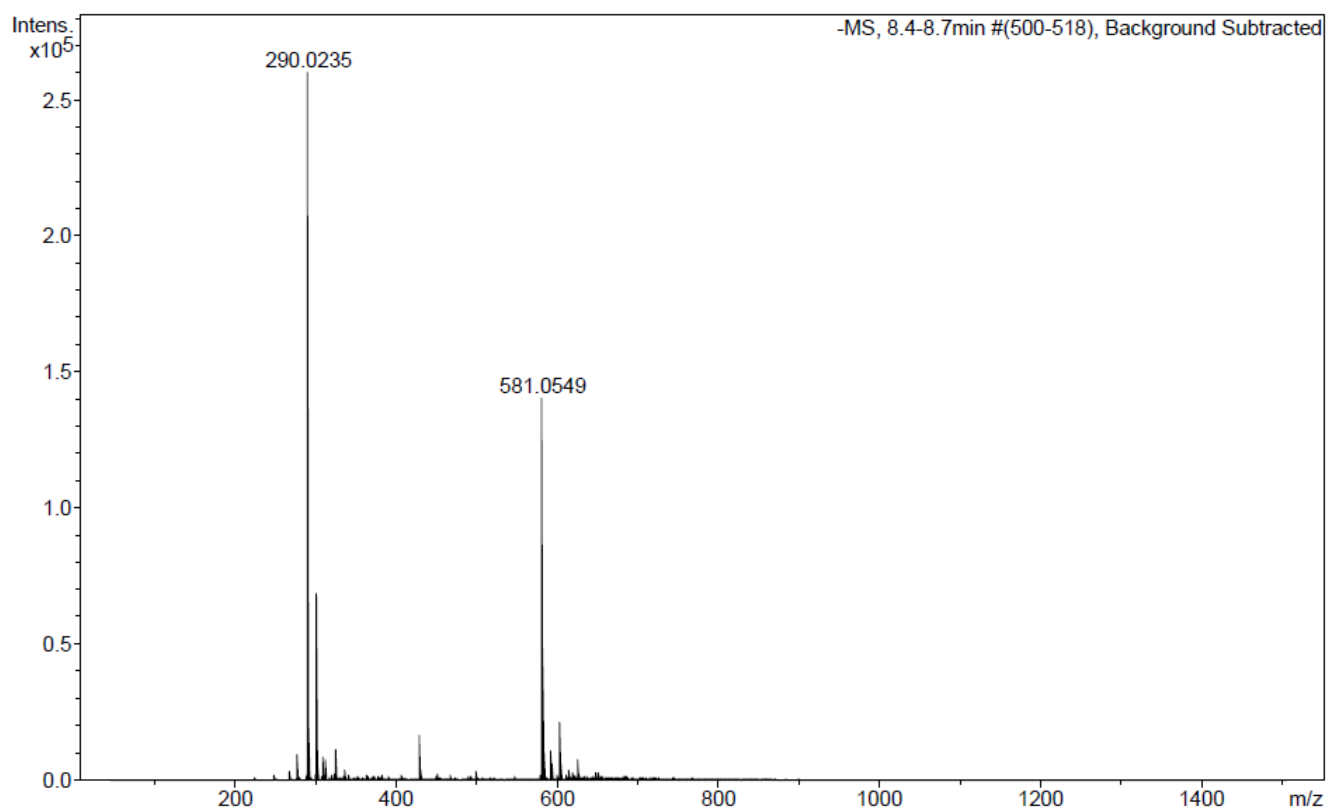
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.898	BV E	0.0589	195.00165	47.47273	1.4974
2	5.250	VB R	0.0721	1.28280e4	2451.05640	98.5026

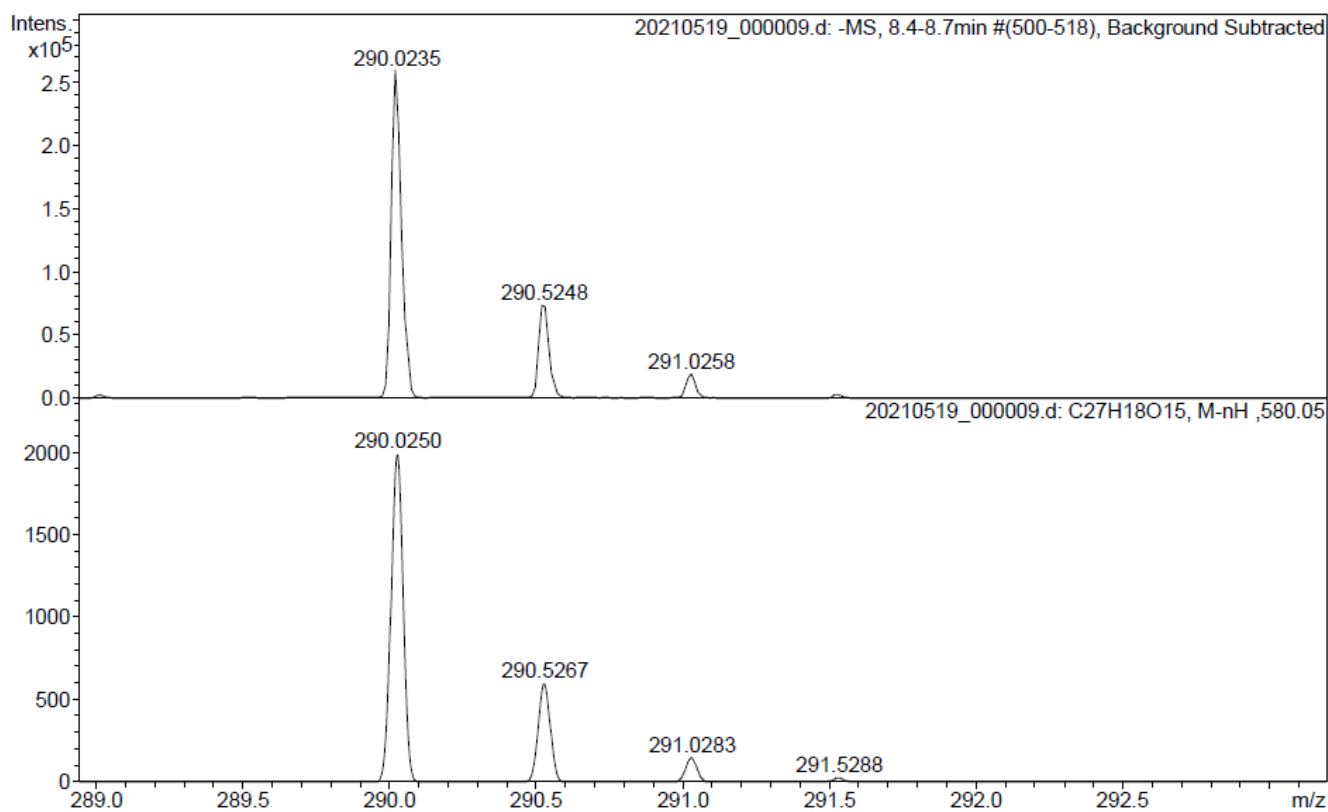
Totals : 1.30230e4 2498.52913

ESI-MS (m/z)



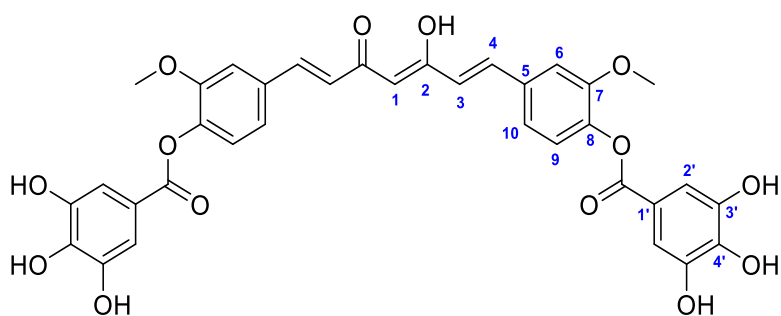
HRMS (m/z)



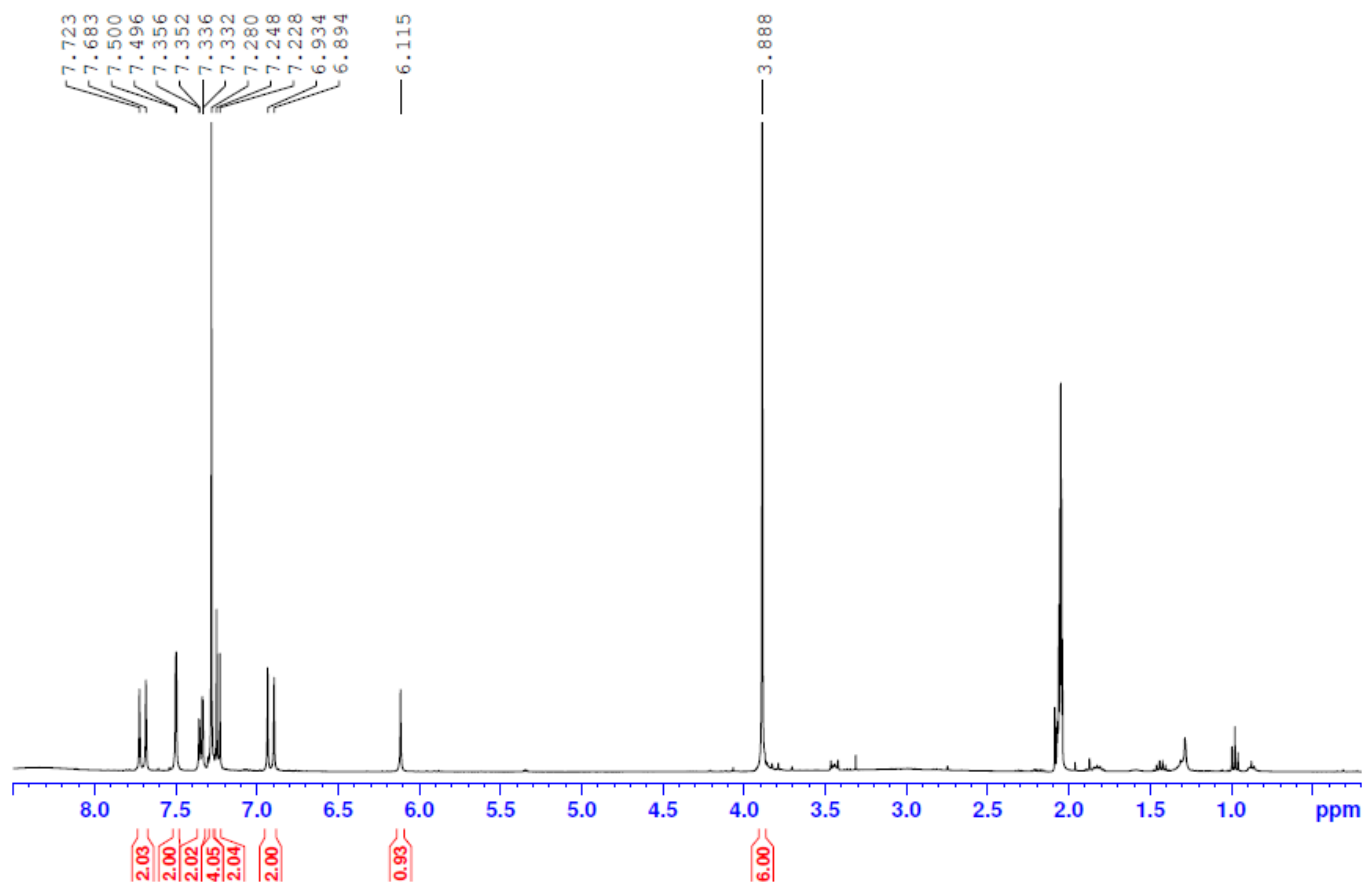


	Molecular formula	Calculated	Found
[M-H] ⁻	C ₂₇ H ₁₇ O ₁₅	518.0573	581.0549
[M-2H] ²⁻	C ₂₇ H ₁₆ O ₁₀	290.0250	290.0239

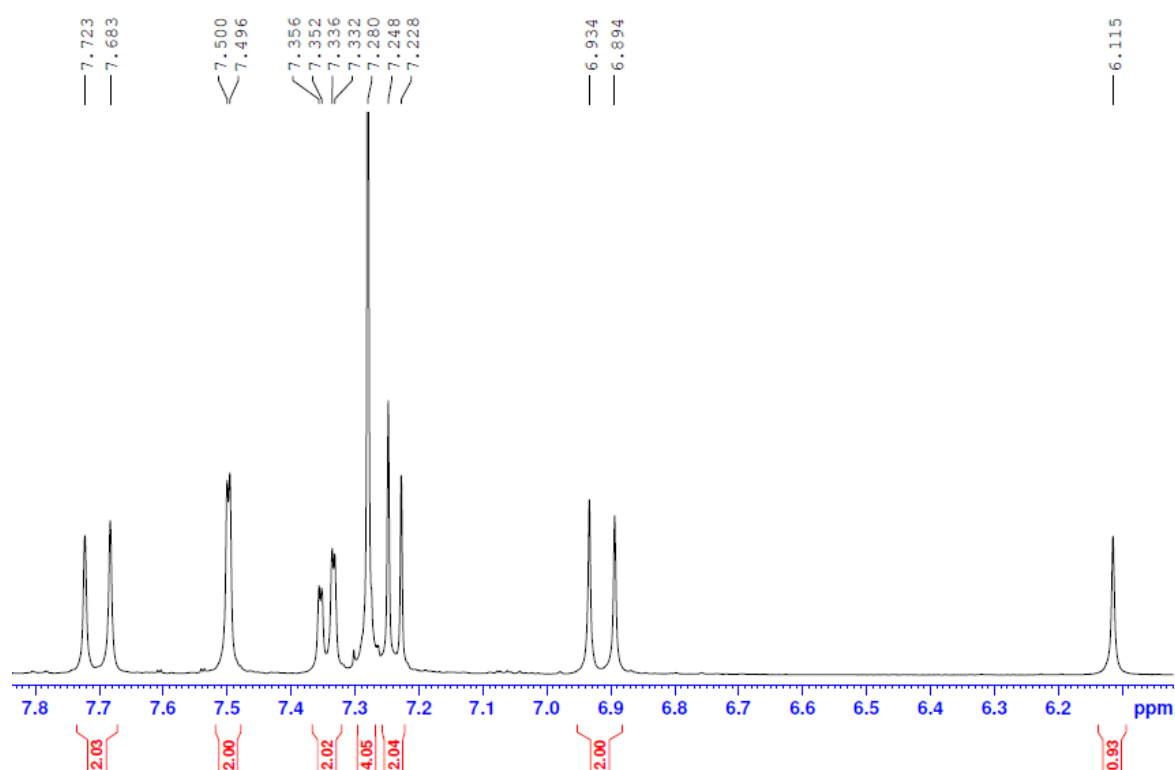
2.11. 1,4-Phenylenebis(methylene) bis(3,4,5-trihydroxybenzoate) (TL3)



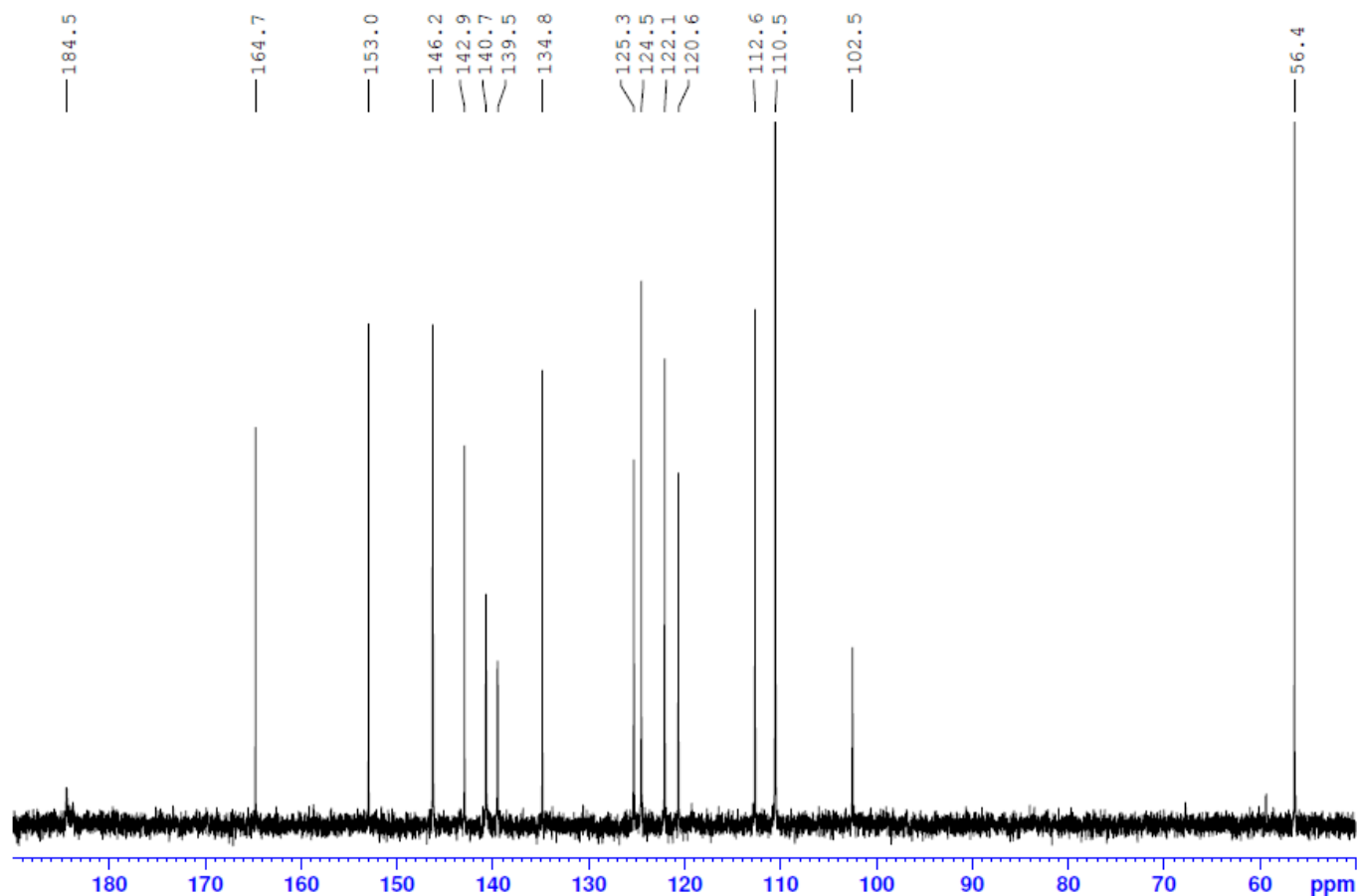
$^1\text{H-NMR}$ (400 MHz, $(\text{CD}_3)_2\text{CO}$), δ (ppm)



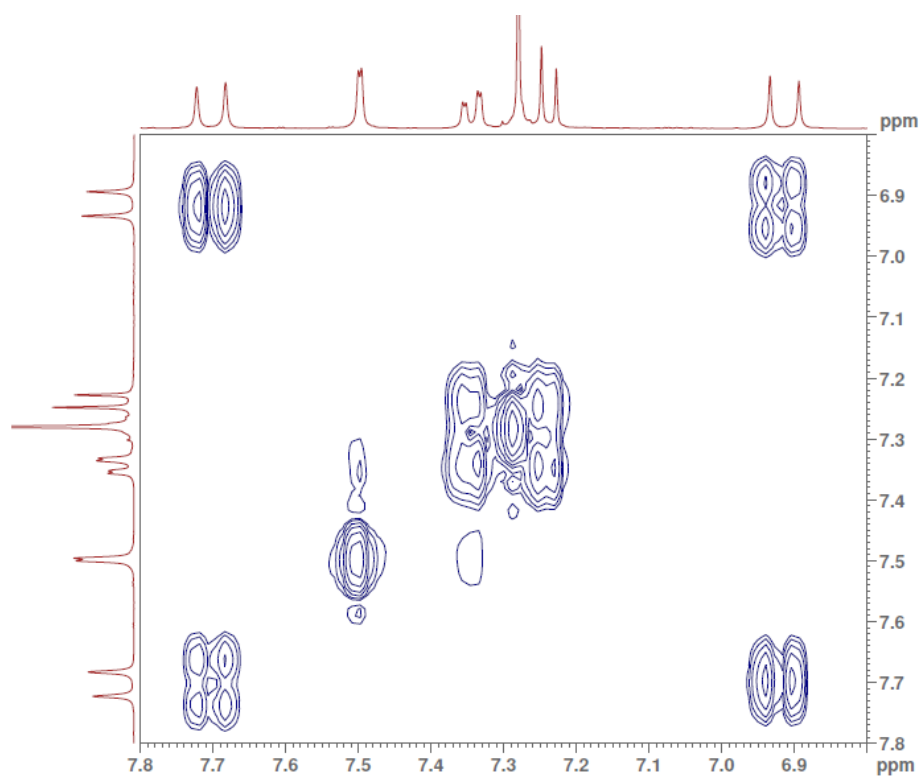
^1H -NMR (400 MHz, $(\text{CD}_3)_2\text{CO}$), δ (ppm) – Aromatic zoom



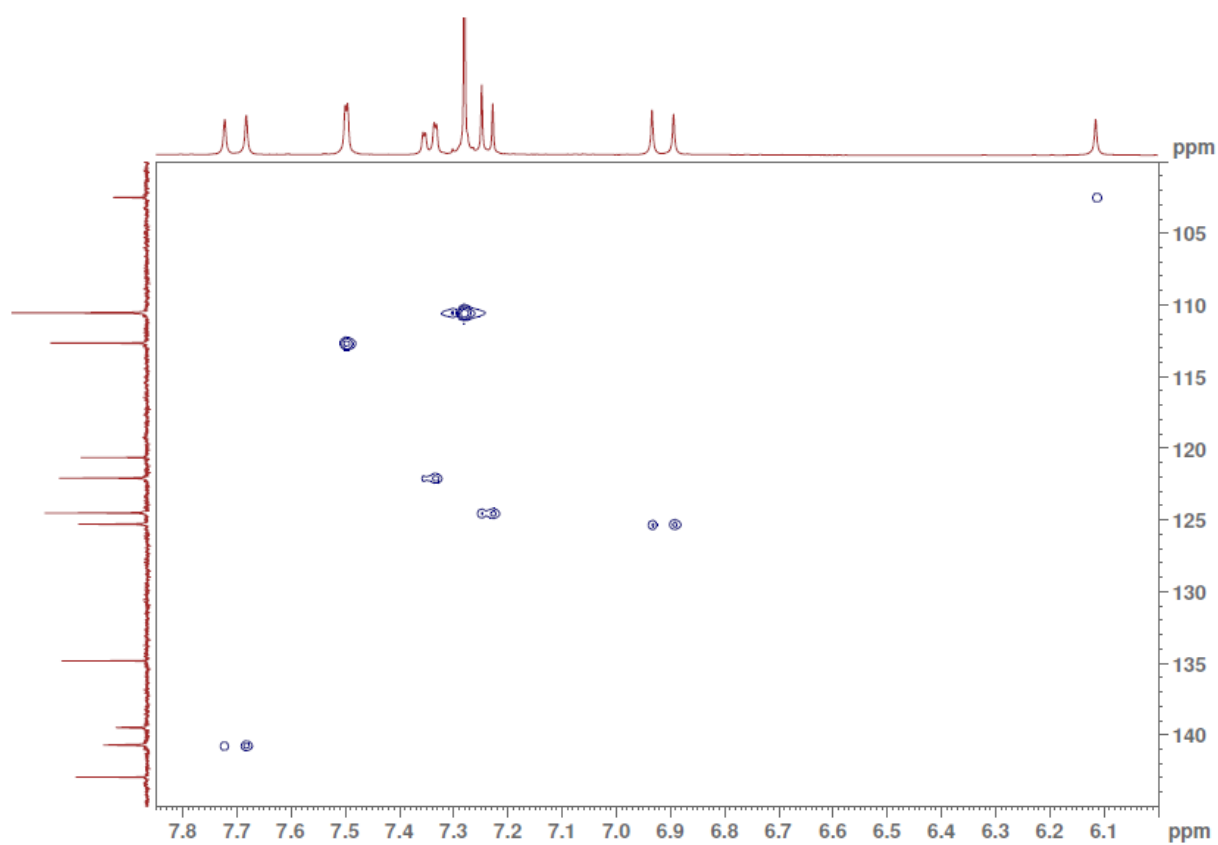
^{13}C -NMR (100 MHz, $(\text{CD}_3)_2\text{CO}$), δ (ppm)



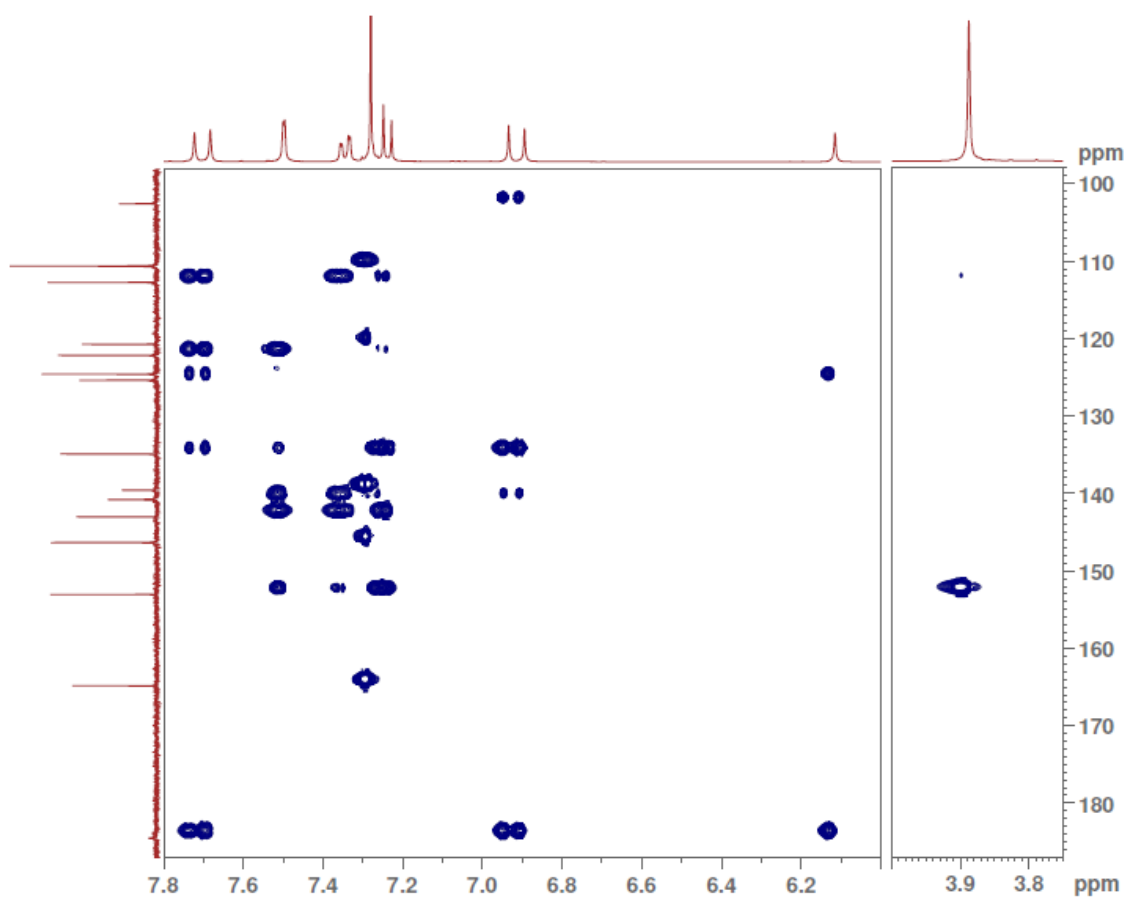
COSY ^1H - ^1H ($(\text{CD}_3)_2\text{CO}$), δ (ppm)



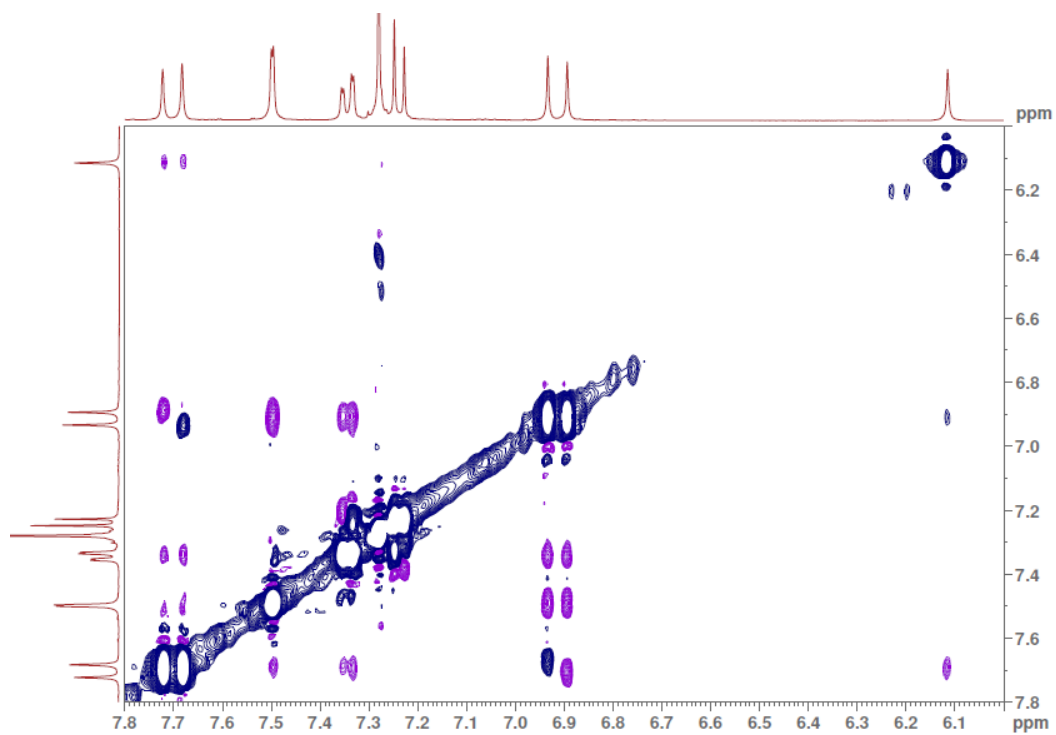
HSQC ^1H - ^{13}C ($(\text{CD}_3)_2\text{CO}$), δ (ppm)



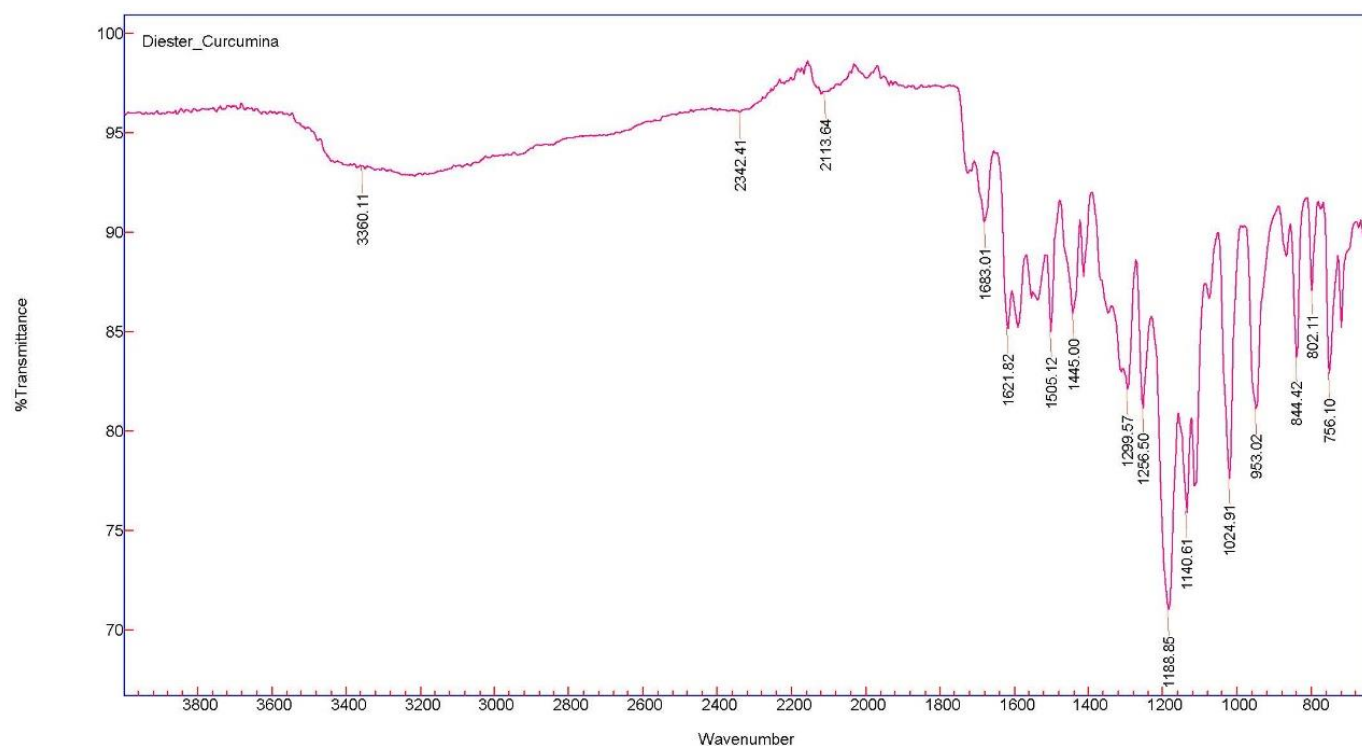
HMBC ^1H - ^{13}C ($(\text{CD}_3)_2\text{CO}$), δ (ppm)



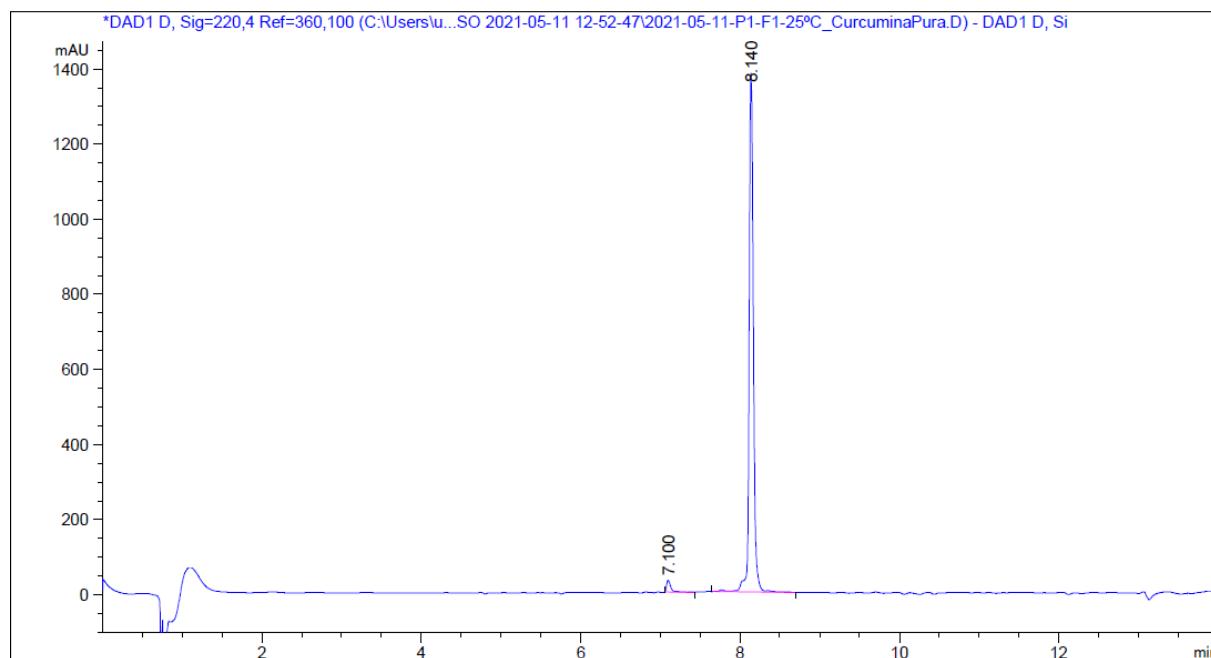
NOESY ^1H - ^1H ($(\text{CD}_3)_2\text{CO}$), δ (ppm)



FT-IR (neat), ν (cm⁻¹)



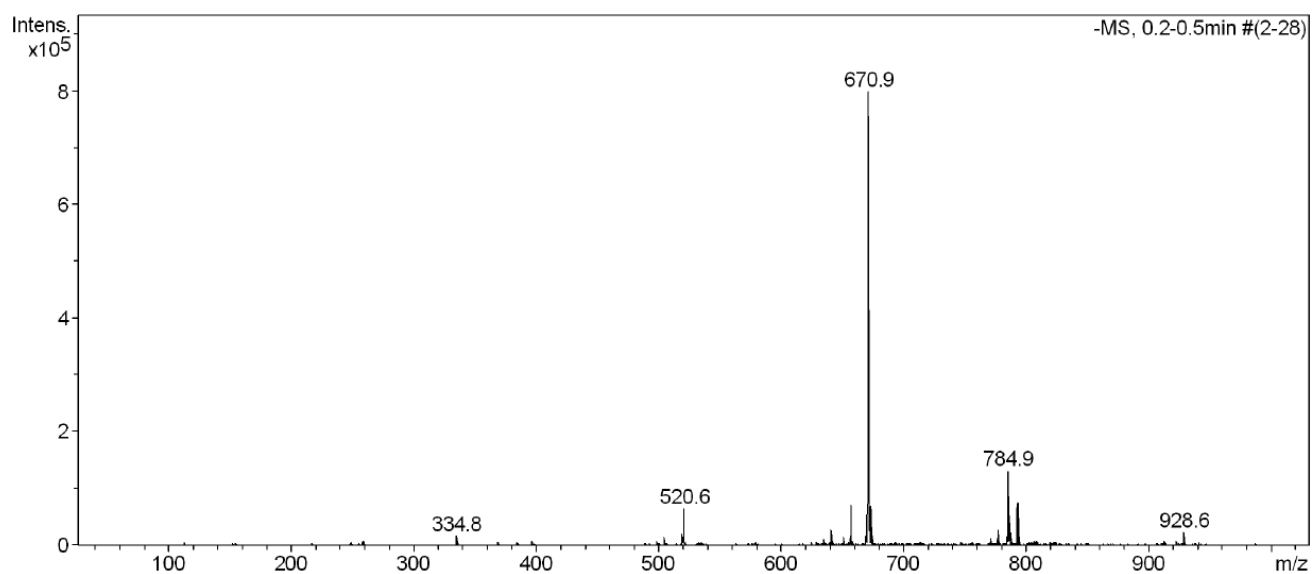
HPLC (λ = 220 nm)



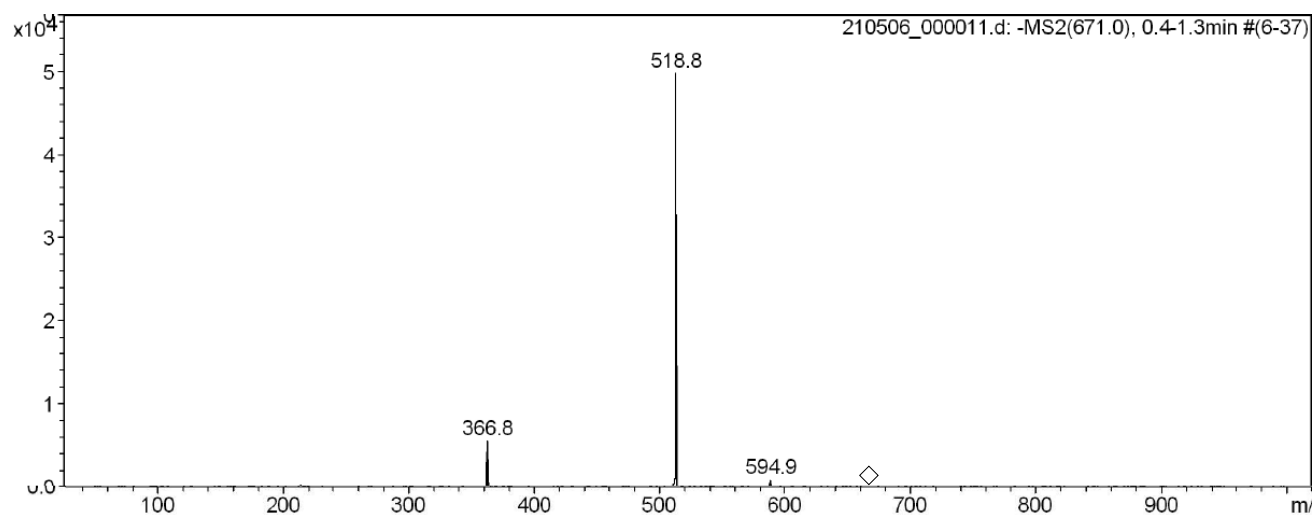
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.100	BV R	0.0556	121.17000	32.40459	2.3111
2	8.140	VV R	0.0572	5121.74561	1382.56921	97.6889

Totals : 5242.91560 1414.97380

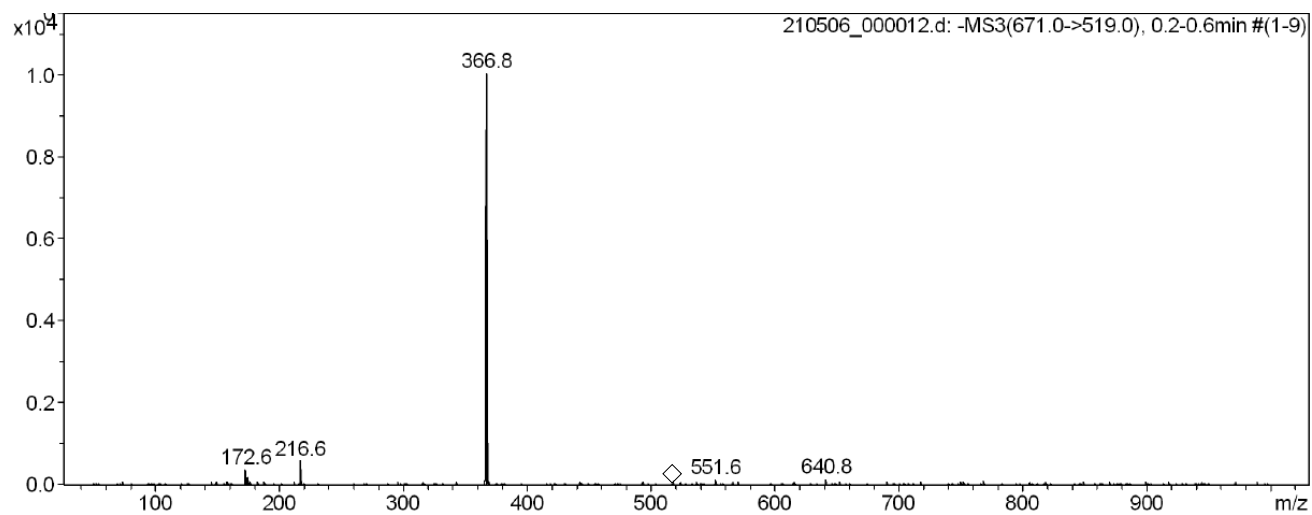
(-)-ESI-MS (m/z)



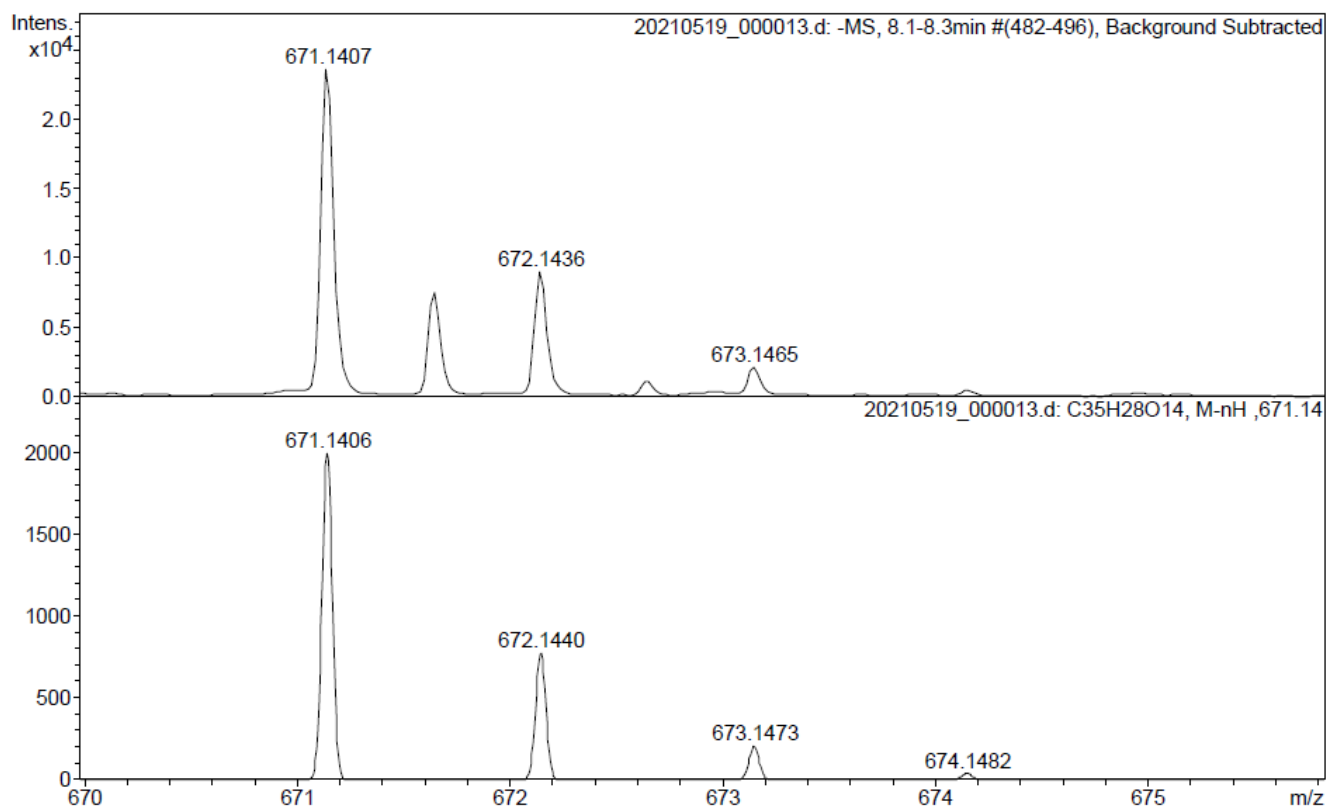
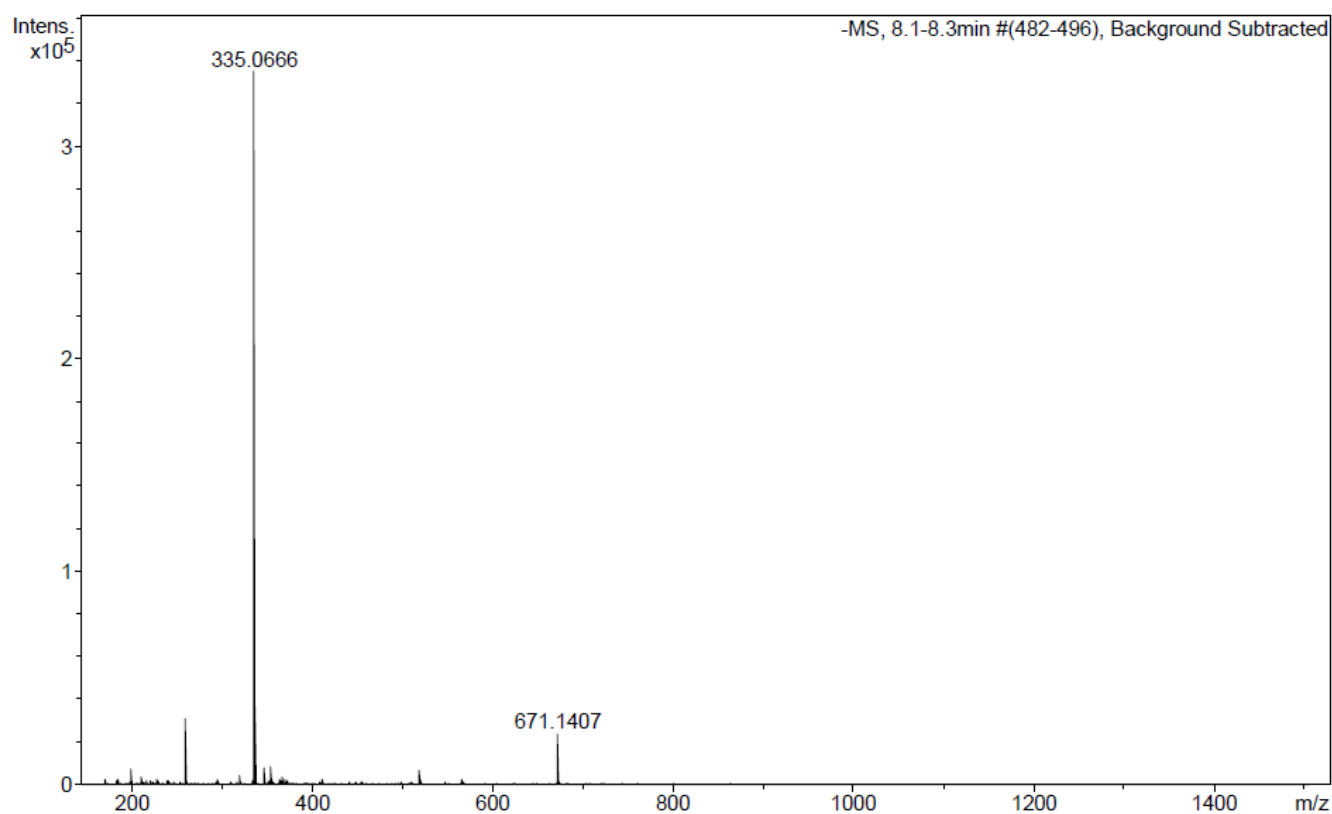
(-)-ESI-MS² ($m/z = 670.9$)

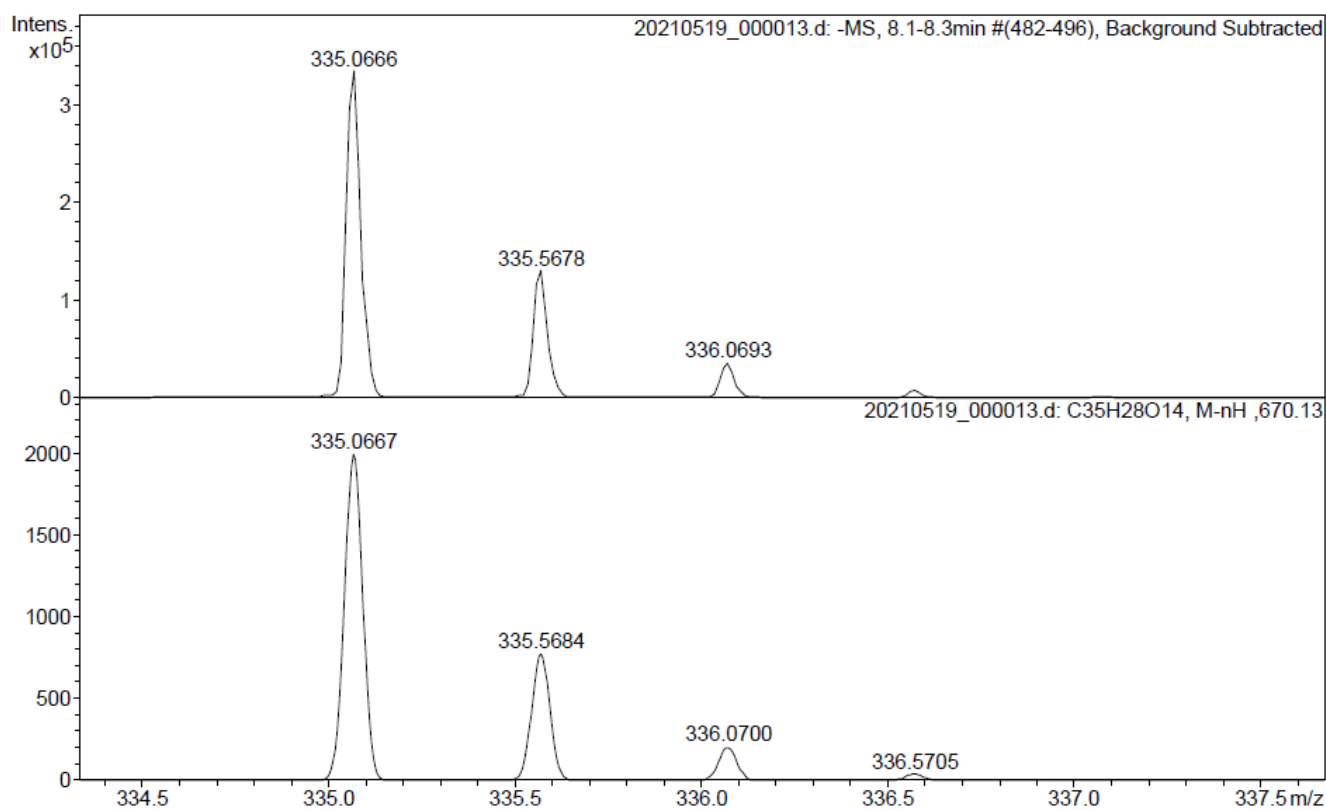


(-)-ESI-MS³ ($m/z = 518.8$)



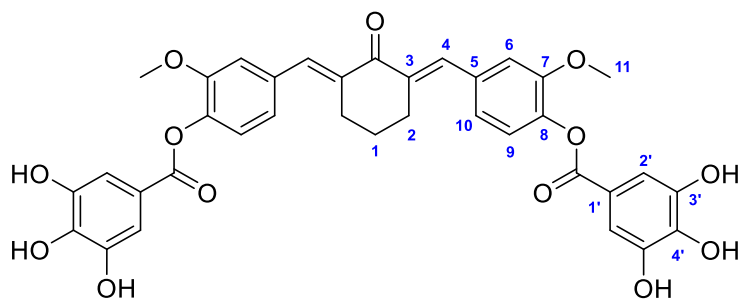
HRMS (*m/z*)



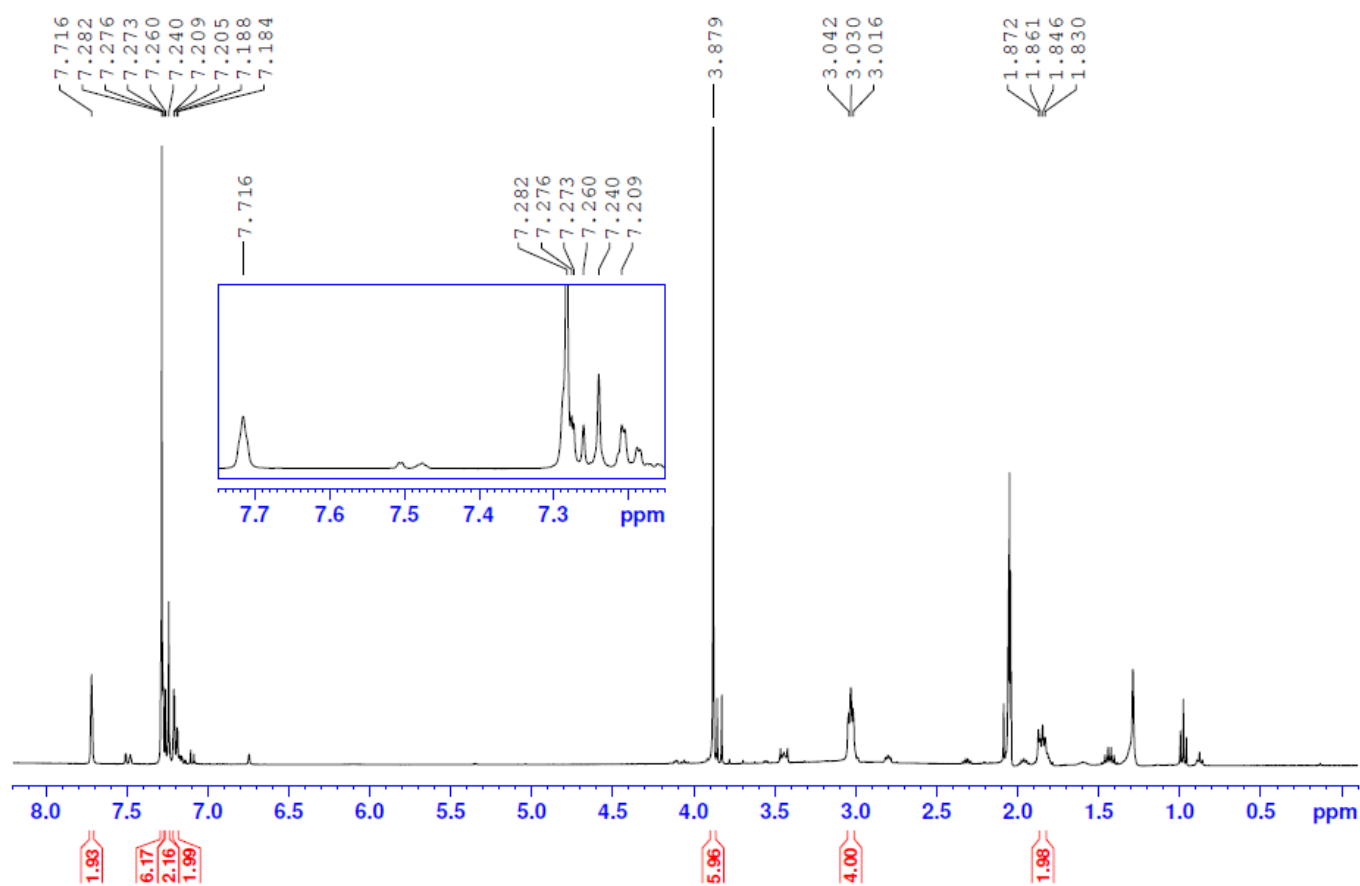


	Molecular formula	Calculated	Found
[M-H] ⁻	C ₃₅ H ₂₇ O ₁₄	671.1406	671.1407
[M-2H] ²⁻	C ₃₅ H ₂₆ O ₁₄	335.0667	335.0666

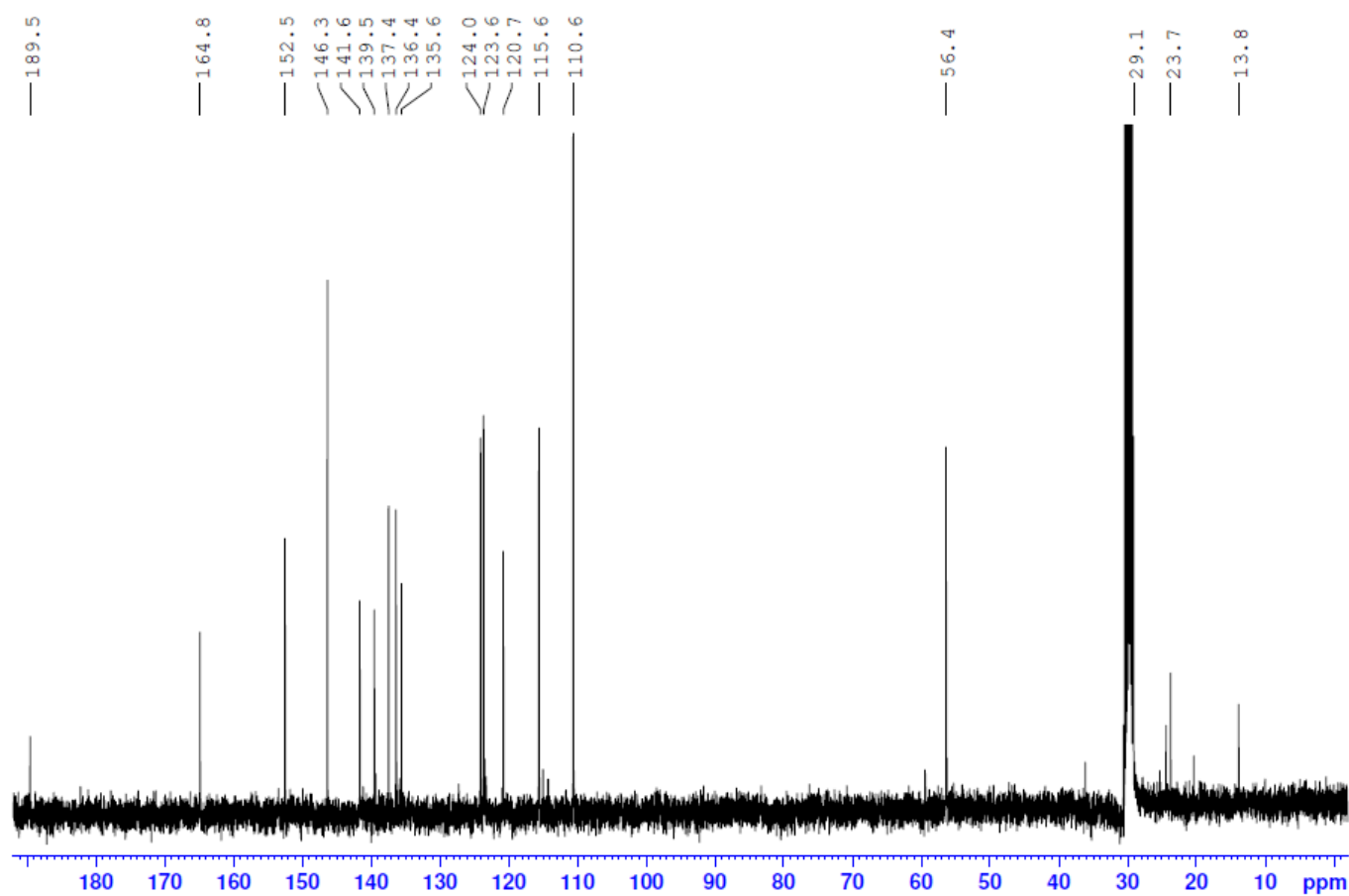
2.12. ((1*E*,1'*E*)-(2-Oxocyclohexane-1,3-diylidene)bis(methanylylidene))bis(2-methoxy-4,1-phenylene) bis[3,4,5-tris((*tert*-butyldimethylsilyl)oxy)benzoate] (TL4)



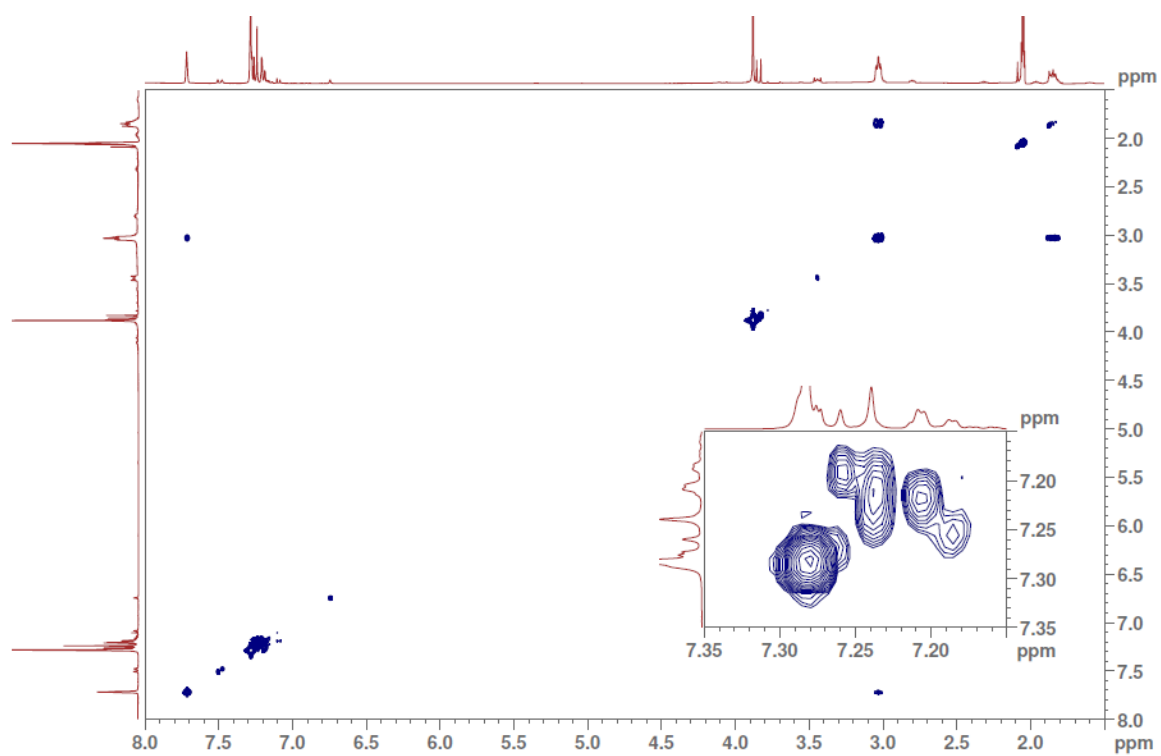
¹H-NMR (400 MHz, (CD₃)₂CO), δ (ppm)



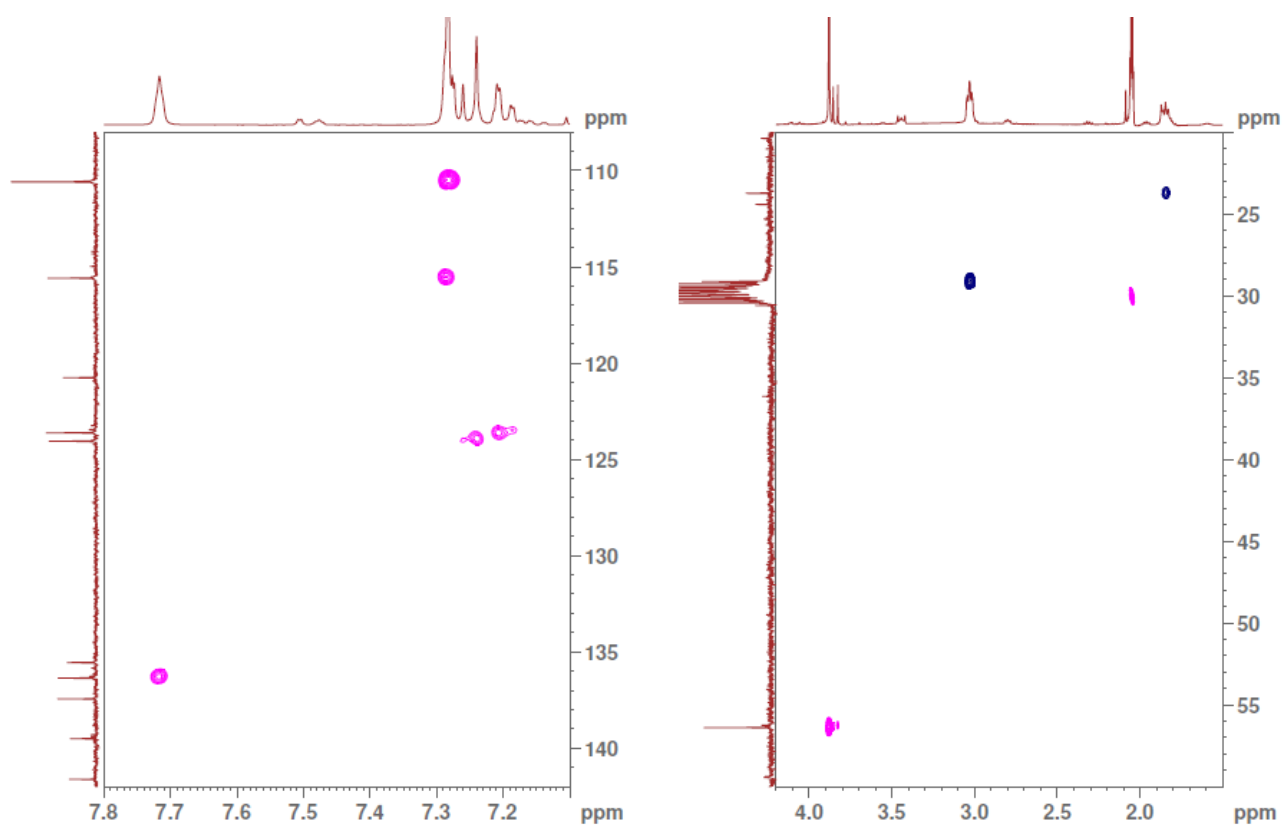
^{13}C -NMR (100 MHz, $(\text{CD}_3)_2\text{CO}$), δ (ppm)



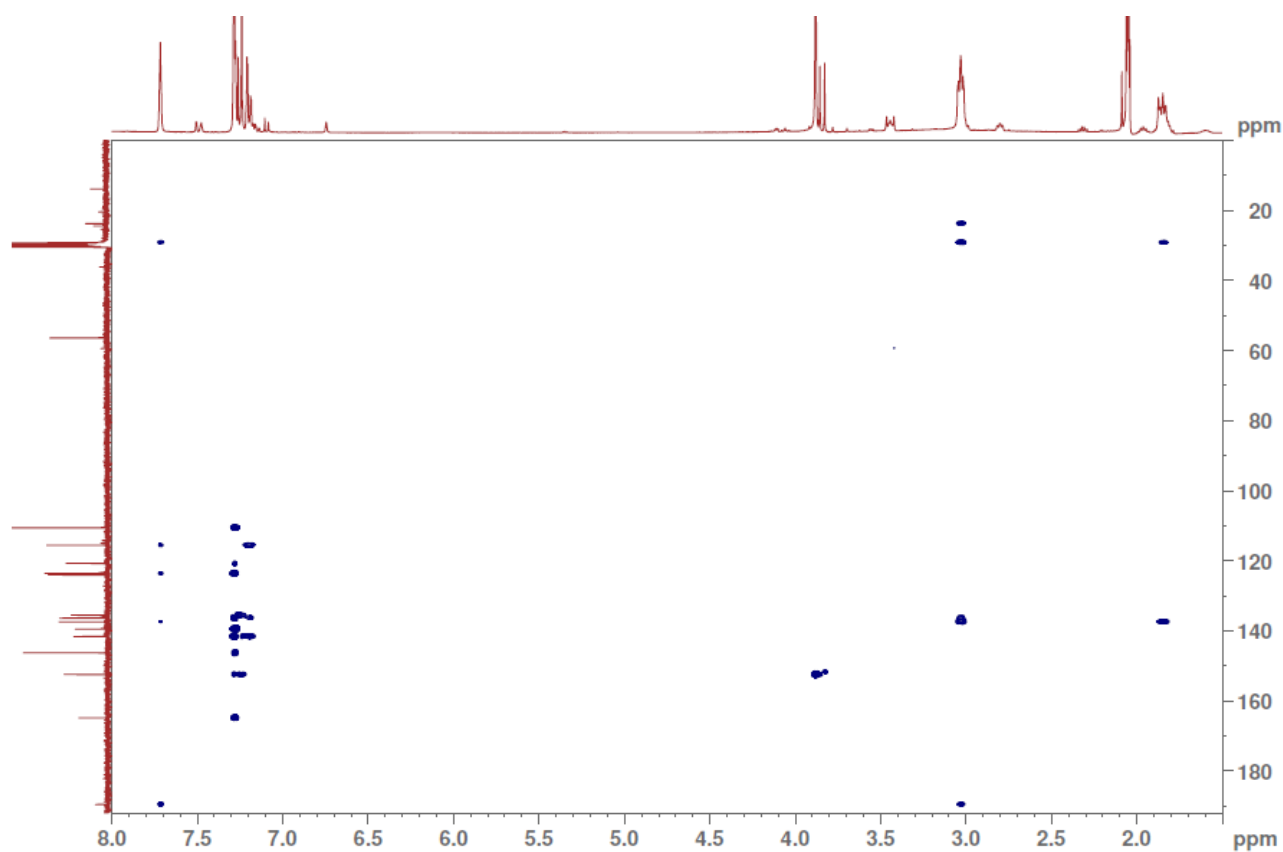
COSY ^1H - ^1H ($(\text{CD}_3)_2\text{CO}$), δ (ppm)



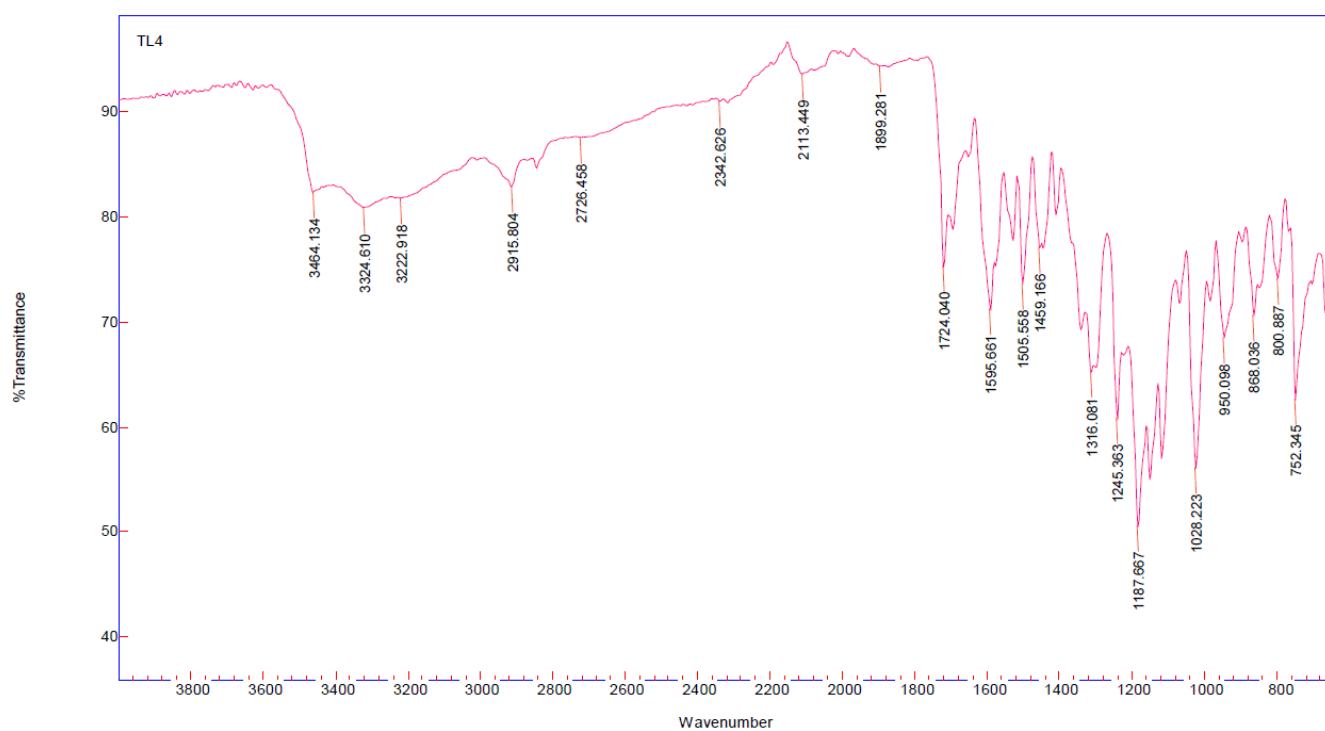
HSQC ^1H - ^{13}C ($(\text{CD}_3)_2\text{CO}$), δ (ppm)



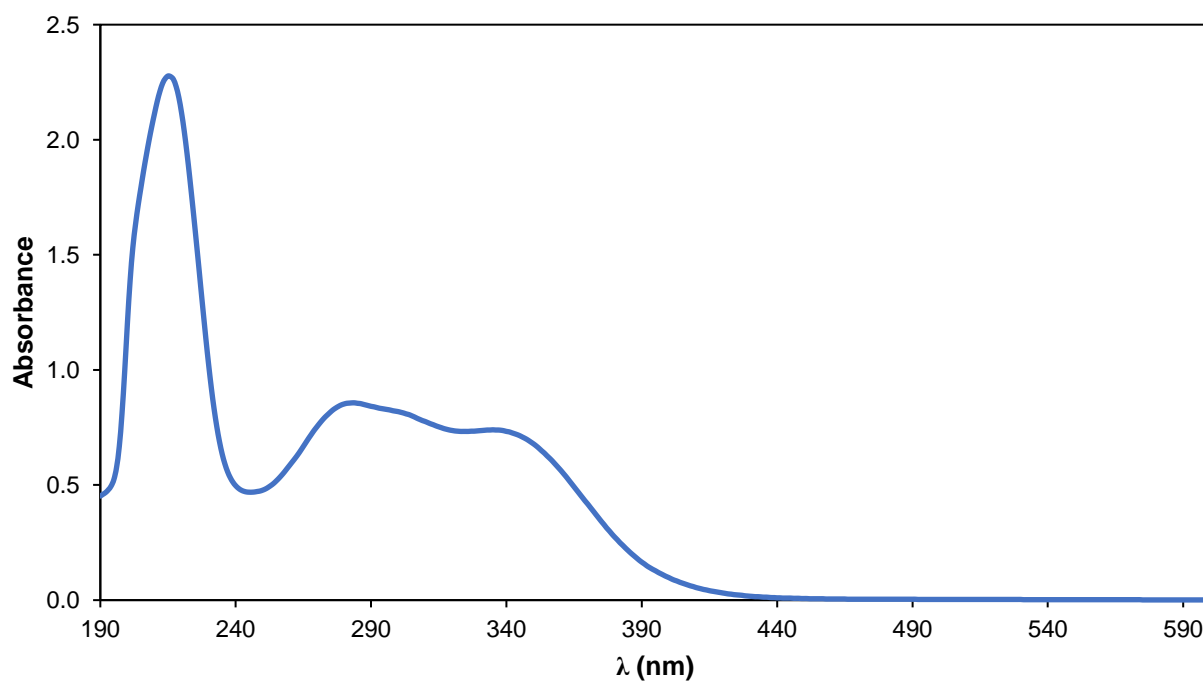
HMBC ^1H - ^{13}C ($(\text{CD}_3)_2\text{CO}$), δ (ppm)



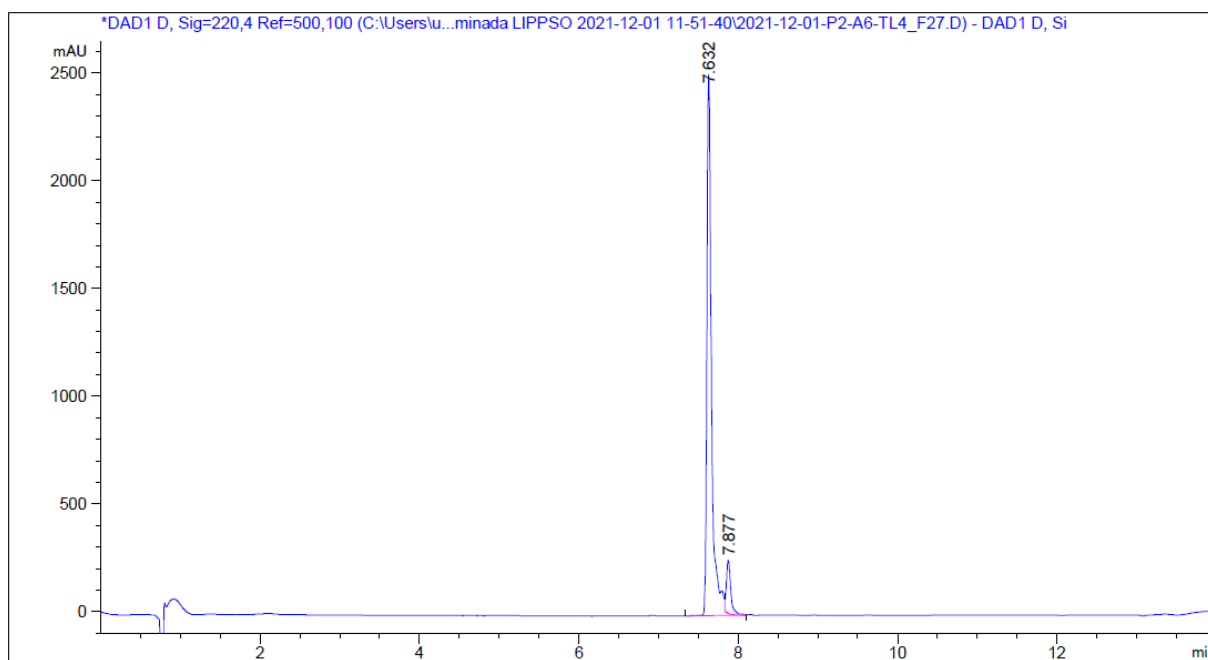
FT-IR (neat), ν (cm⁻¹)



UV-Vis (CH₃CN), λ (nm)



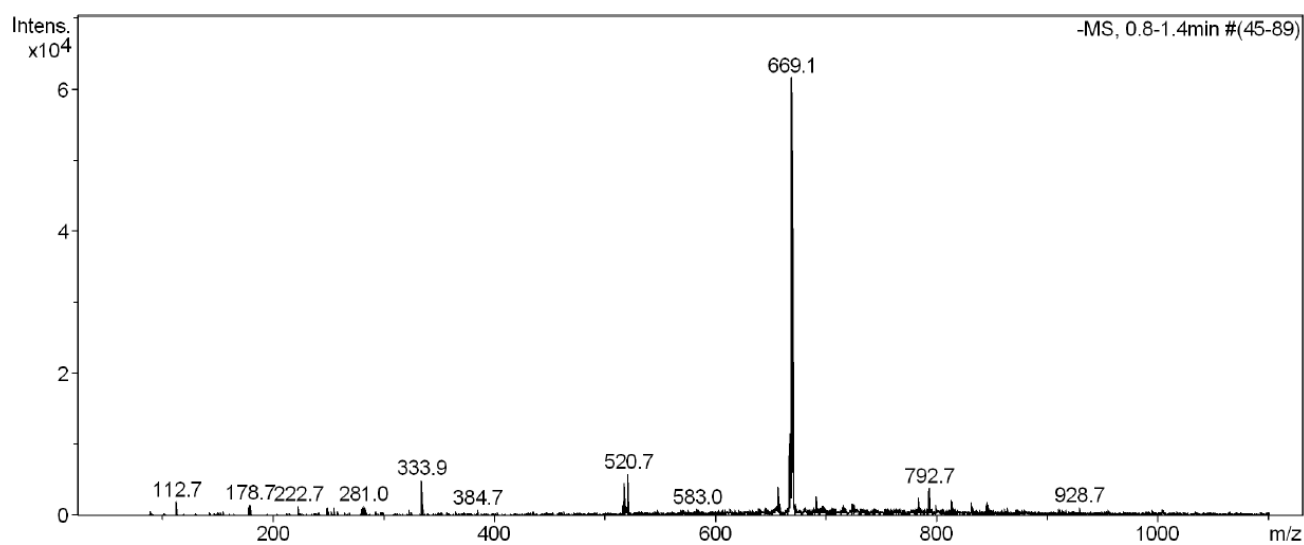
HPLC ($\lambda = 220 \text{ nm}$)



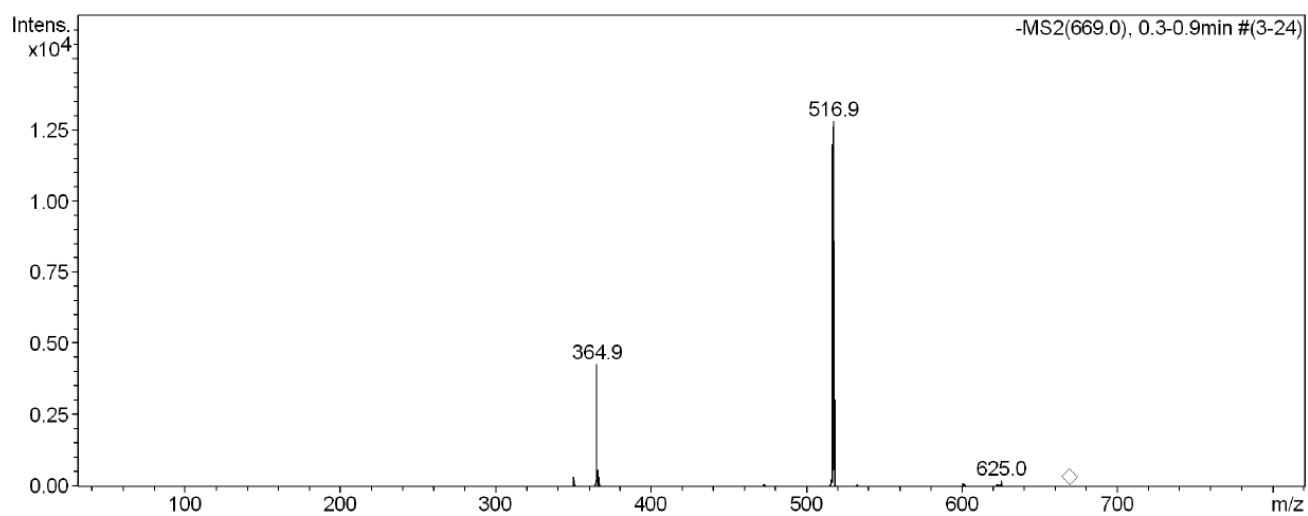
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.632	BV R	0.0618	1.02783e4	2507.92700	91.5710
2	7.877	VB E	0.0577	946.10846	246.68805	8.4290

Totals : 1.12244e4 2754.61505

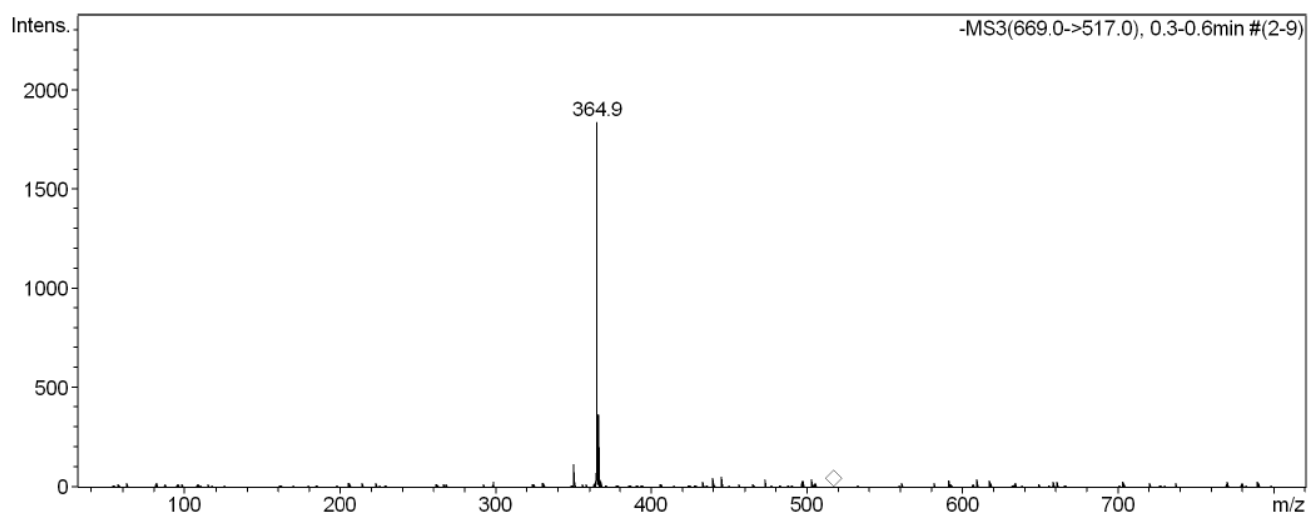
(-)-ESI-MS (m/z)



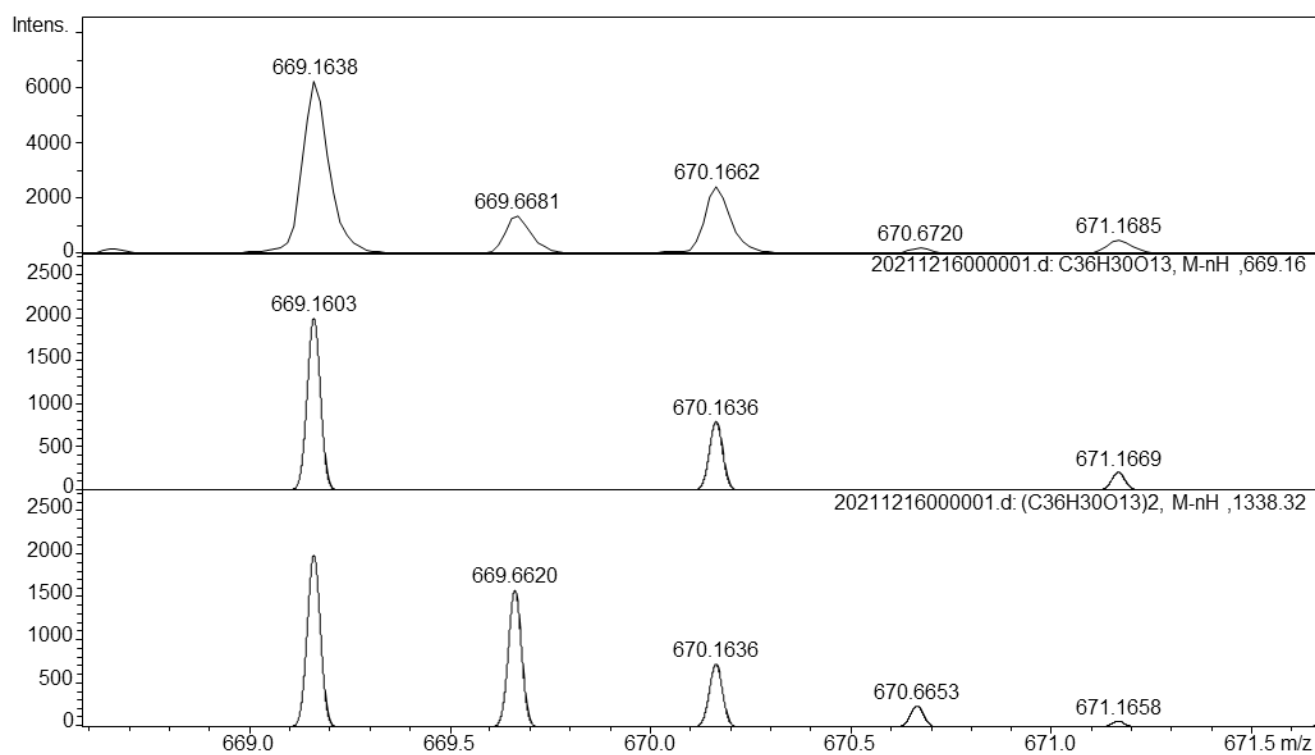
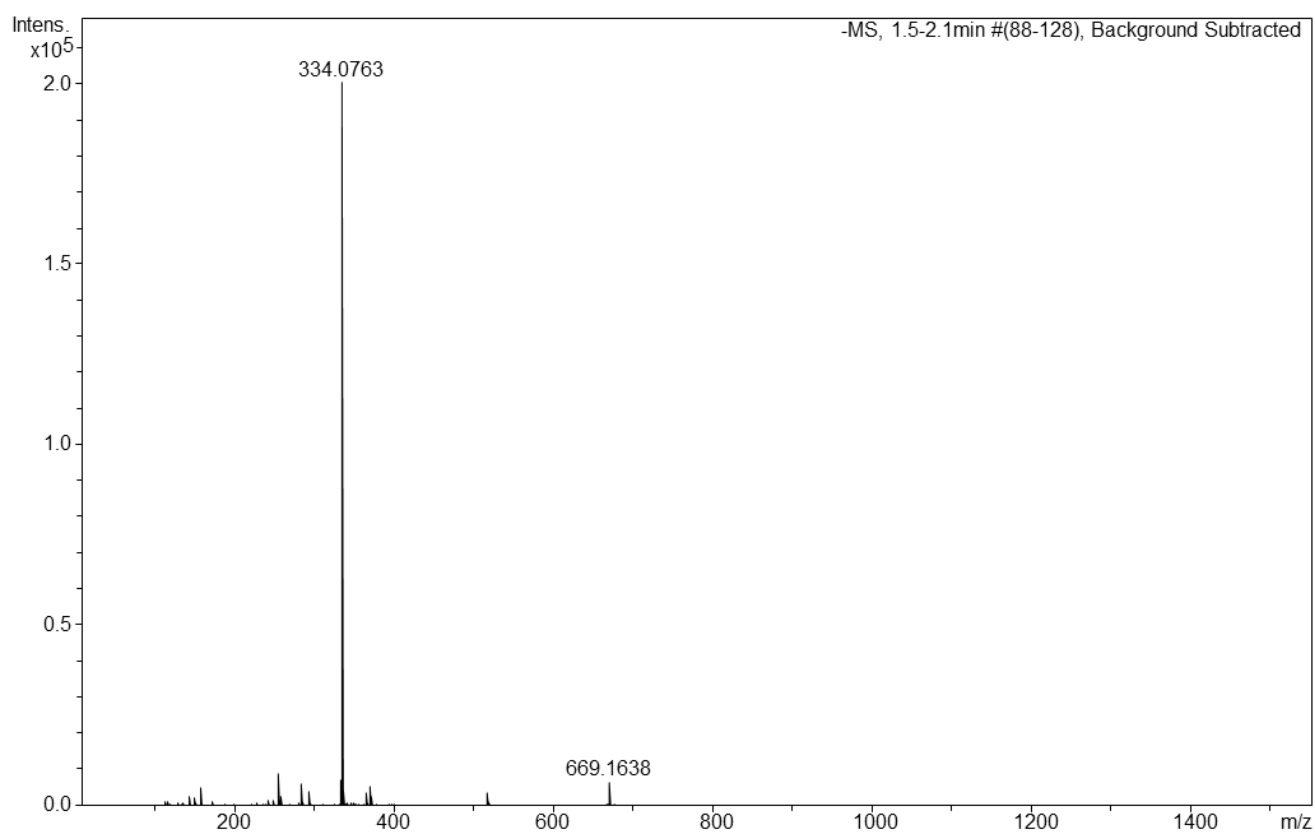
(-)-ESI-MS² (m/z = 669.1)

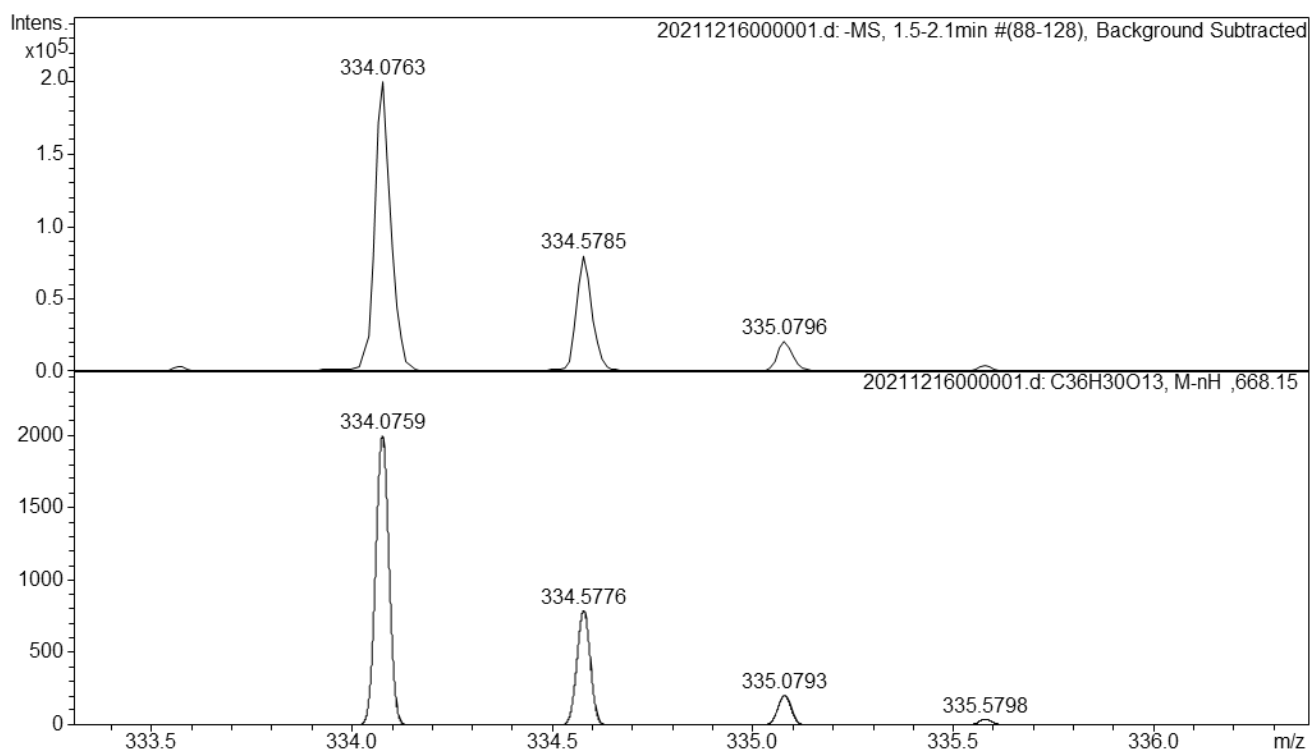


(-)-ESI-MS³ (m/z = 516.9)



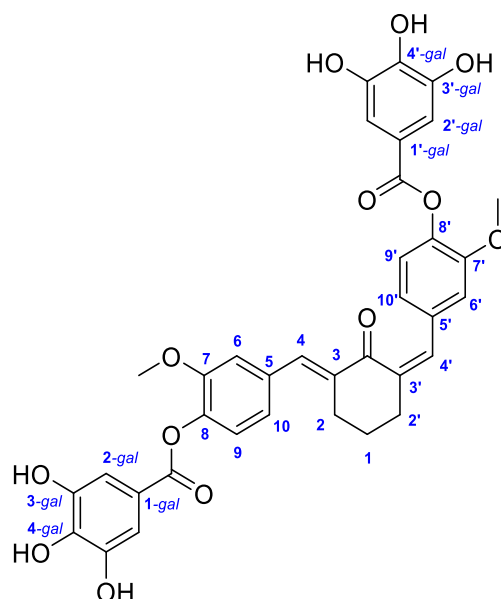
HRMS (*m/z*)



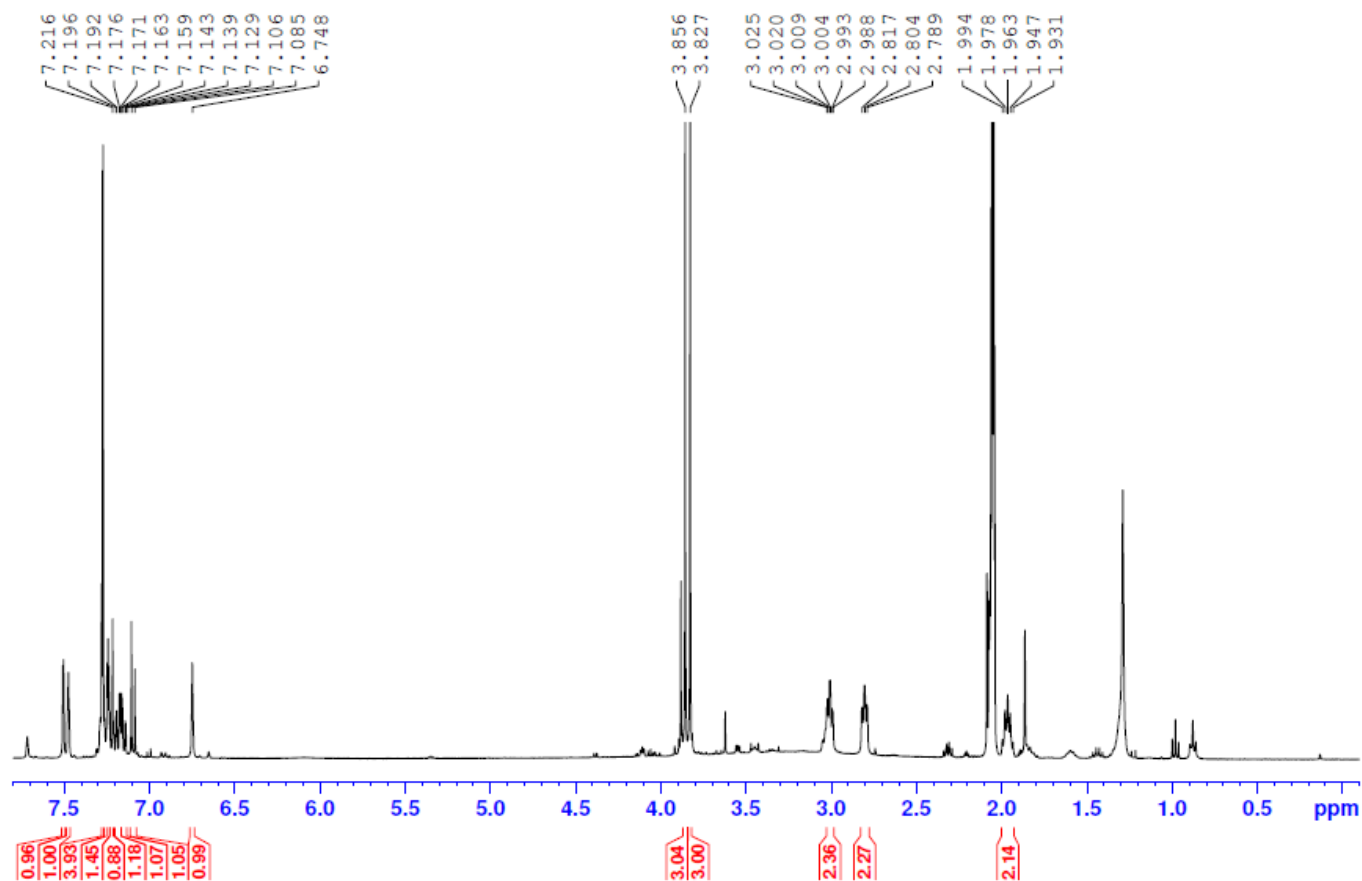


	Molecular formula	Calculated	Found
[M-H] ⁻	C ₃₆ H ₂₉ O ₁₃	669.1603	669.1638
[2M-2H] ⁻	(C ₃₆ H ₂₉ O ₁₃) ₂	669.1603	669.1638
[M-2H] ²⁻	C ₃₆ H ₂₈ O ₁₃	334.0759	334.0763

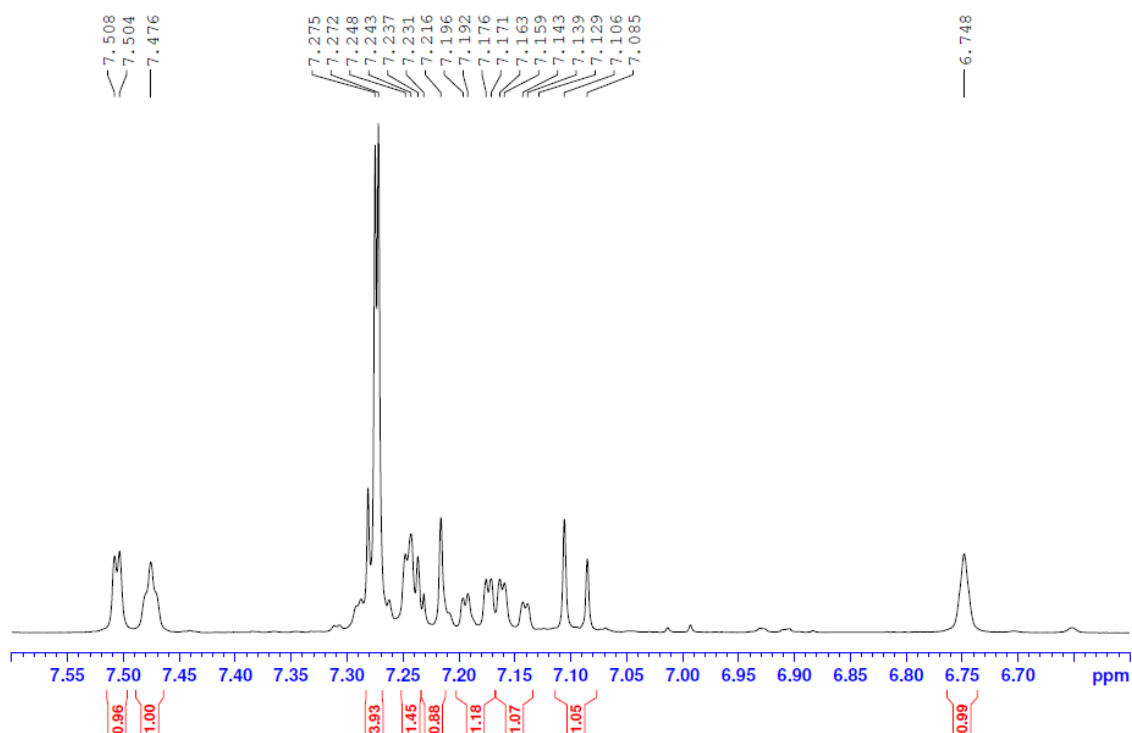
2.13. ((1*E*,1'*Z*)-(2-Oxocyclohexane-1,3-diylidene)bis(methanylylidene))bis(2-methoxy-4,1-phenylene) bis[3,4,5-tris((*tert*-butyldimethylsilyl)oxy)benzoate] (TL5)



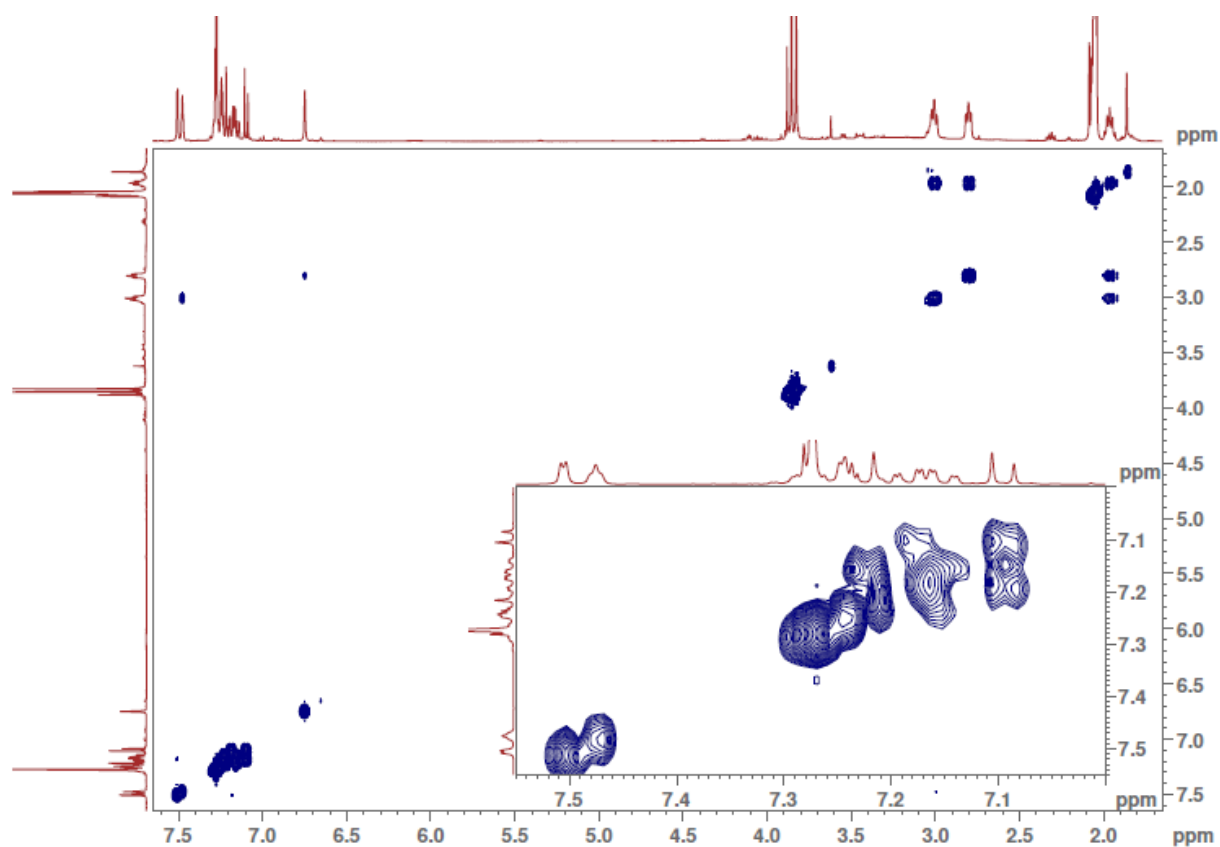
¹H-NMR (400 MHz, (CD₃)₂CO), δ (ppm)



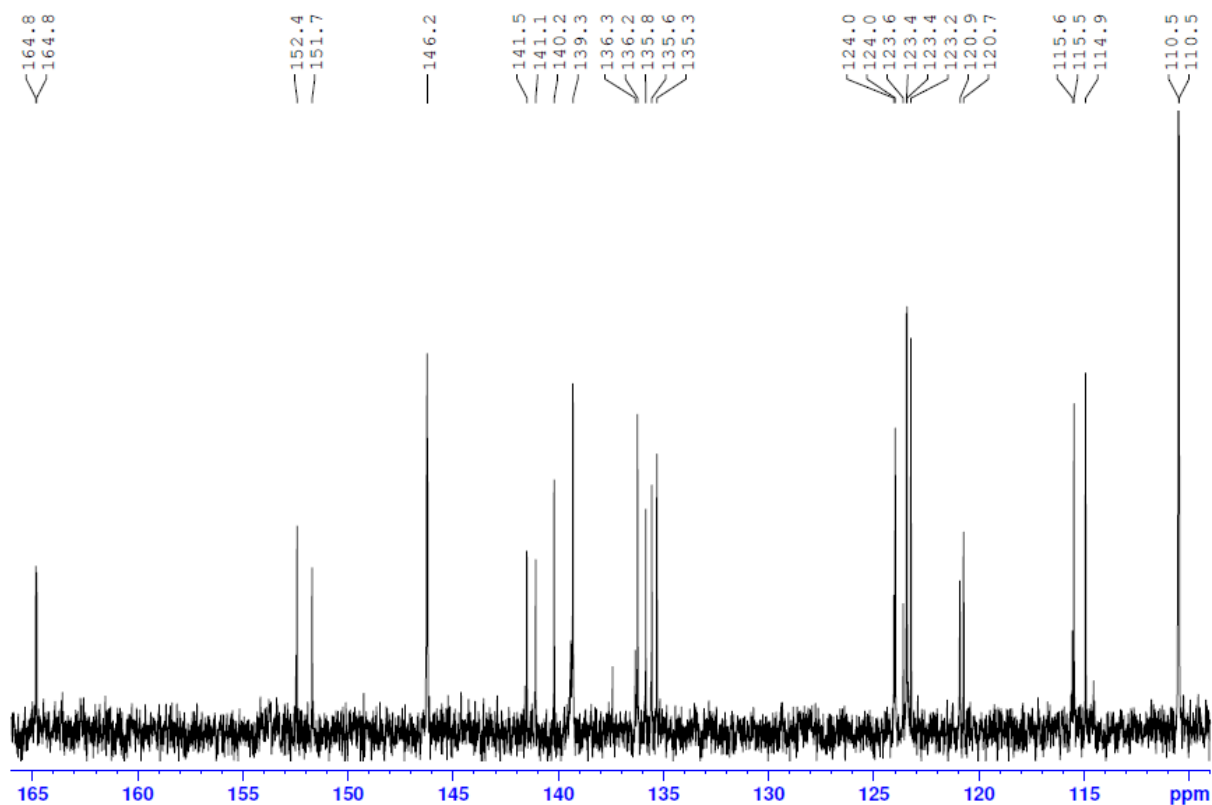
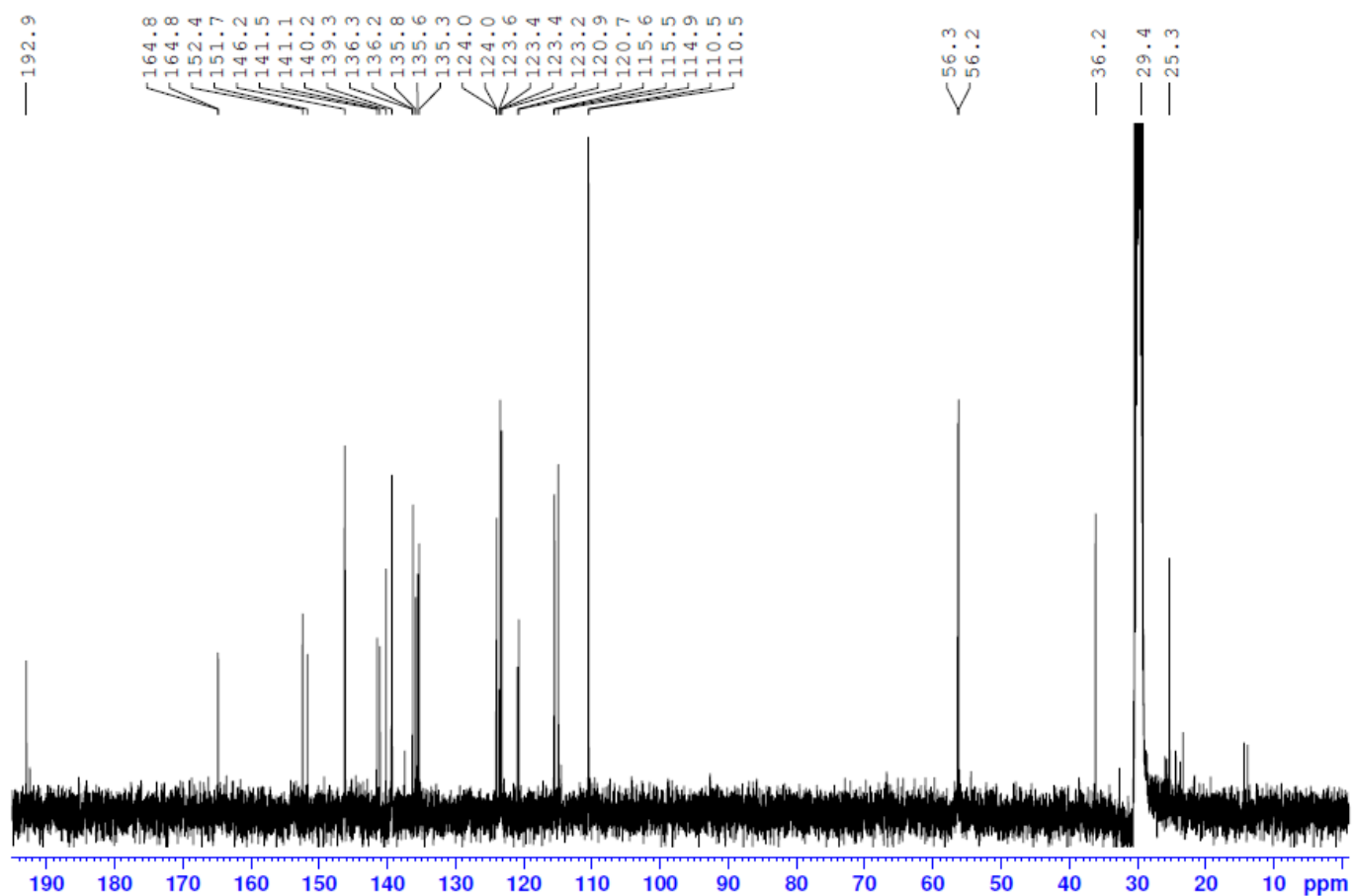
^1H -NMR (400 MHz, $(\text{CD}_3)_2\text{CO}$), δ (ppm) – Aromatic zoom



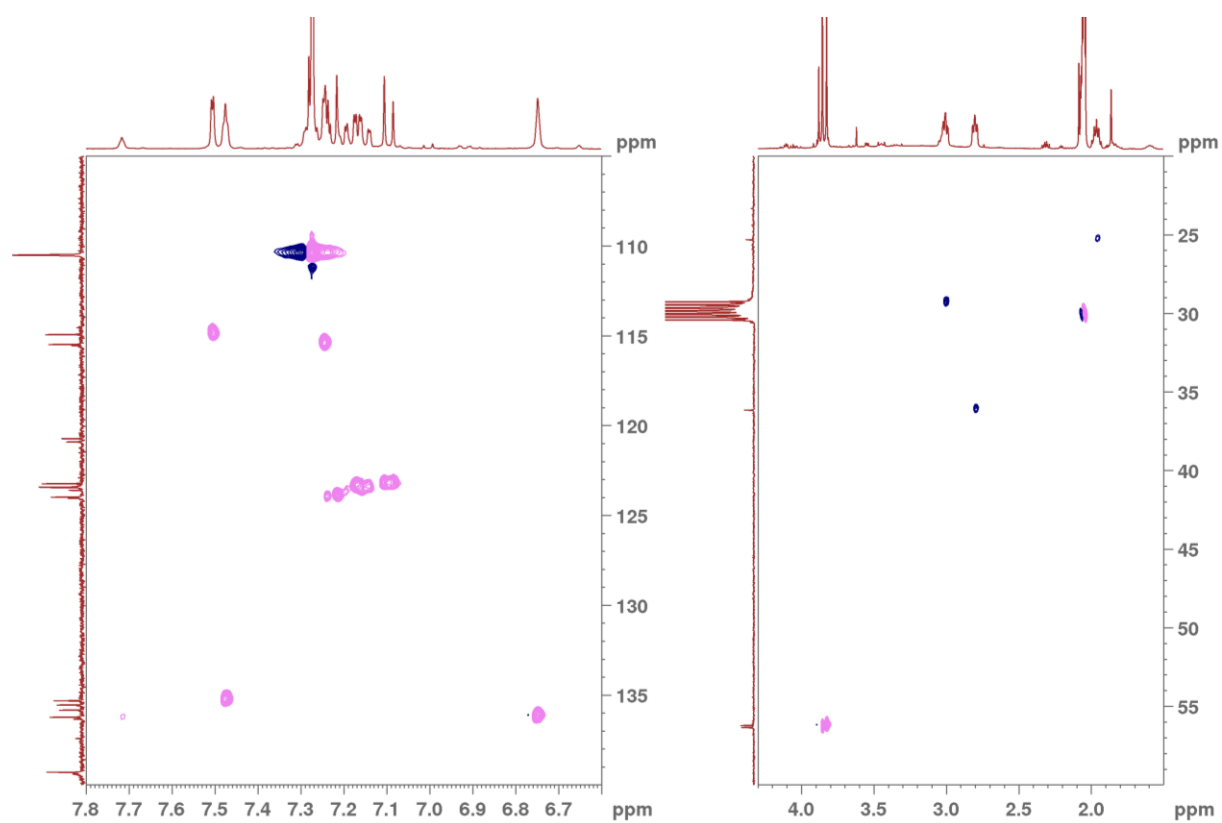
COSY ^1H - ^1H ($(\text{CD}_3)_2\text{CO}$), δ (ppm)



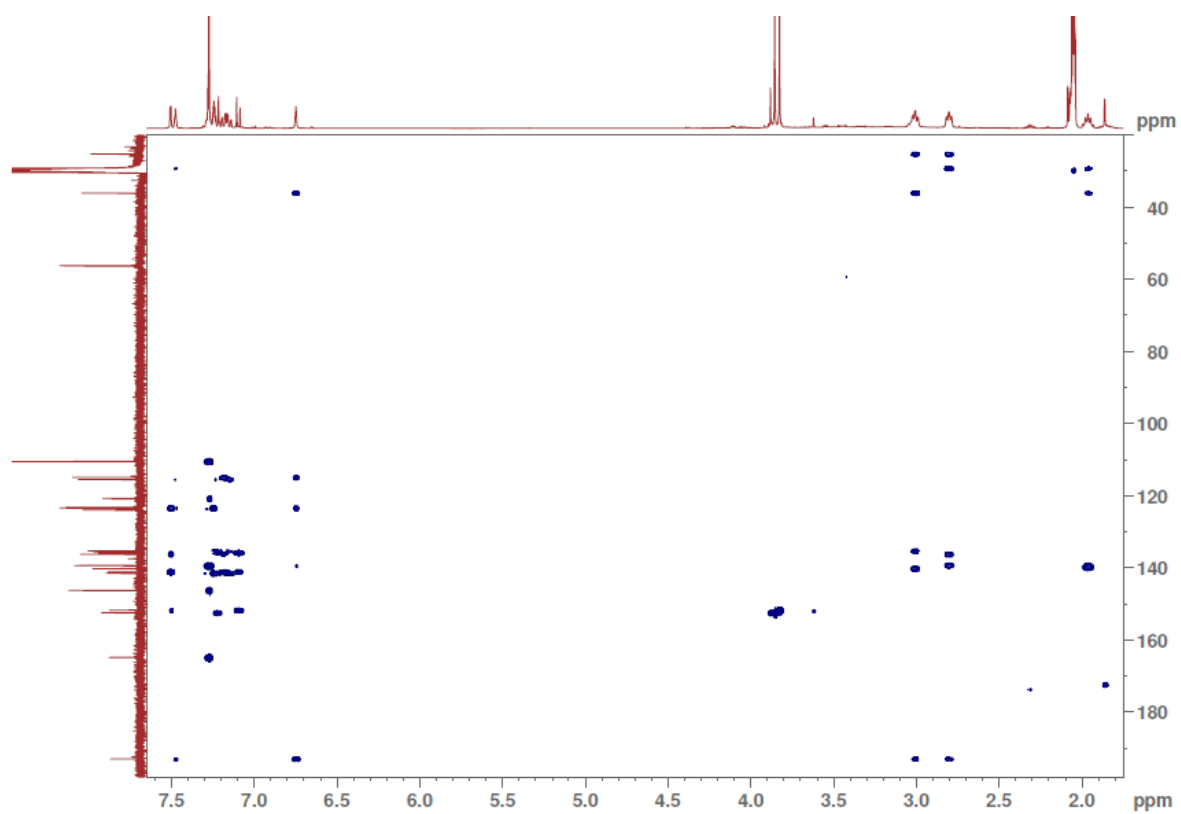
^{13}C -NMR (100 MHz, $(\text{CD}_3)_2\text{CO}$), δ (ppm)



HSQC ^1H - ^{13}C ($(\text{CD}_3)_2\text{CO}$), δ (ppm)

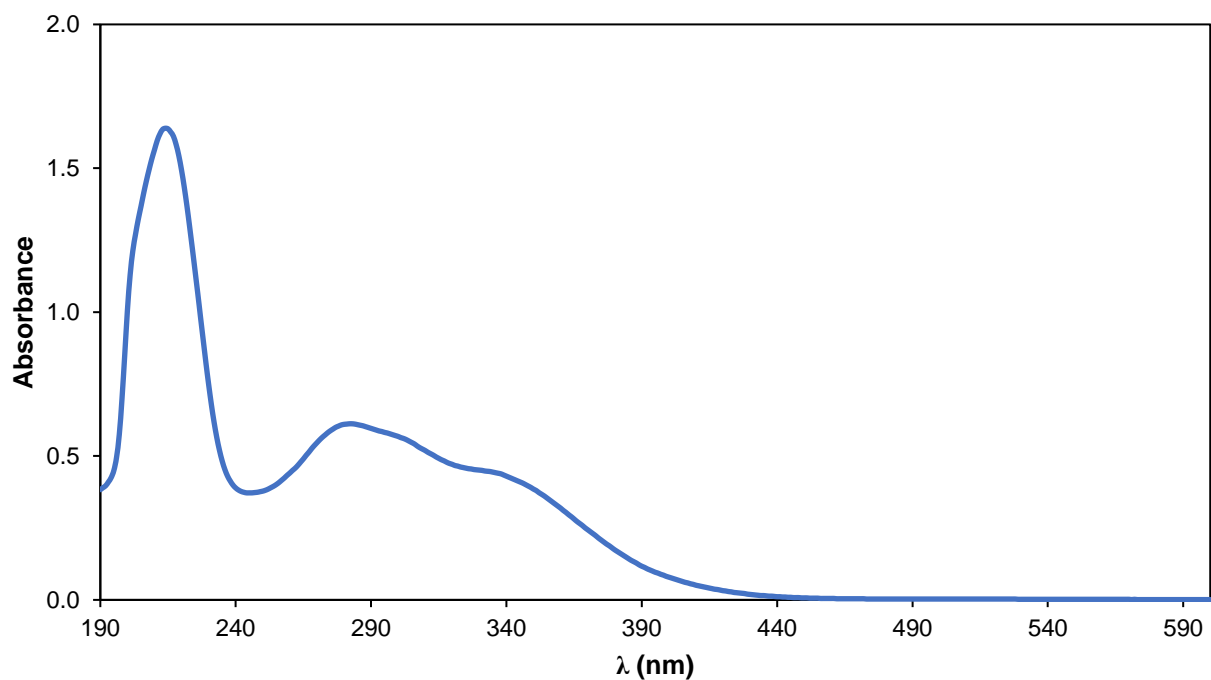


HMBC ^1H - ^{13}C ($(\text{CD}_3)_2\text{CO}$), δ (ppm)

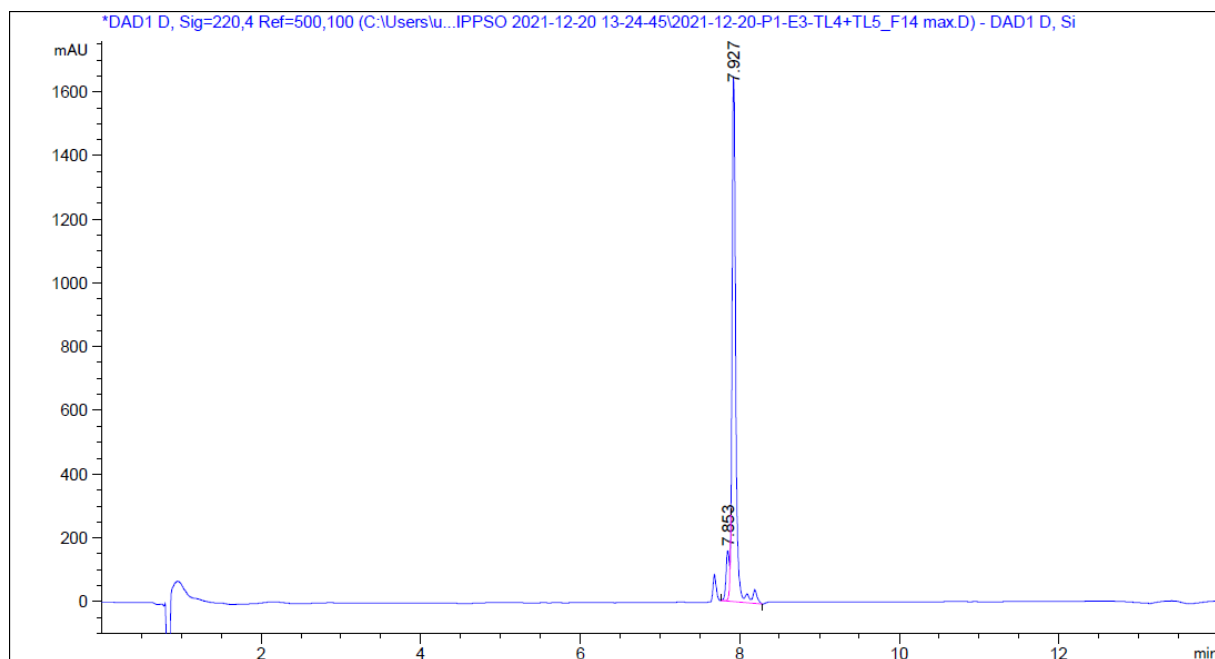


IR spectrum of compound 10 (TL5) showing % Transmittance vs Wavenumber (cm⁻¹). The spectrum displays characteristic absorption bands for an aldehyde, including a strong C=O stretch at 1717.913 cm⁻¹ and C-H stretches around 2873.306 and 2894.065 cm⁻¹. Other notable peaks are at 1595.321, 1506.551, 1445.419, 1306.854, 1186.114, 1028.613, 949.361, 853.421, 796.040, and 753.944 cm⁻¹.

UV-Vis (CH₃CN), λ (nm)



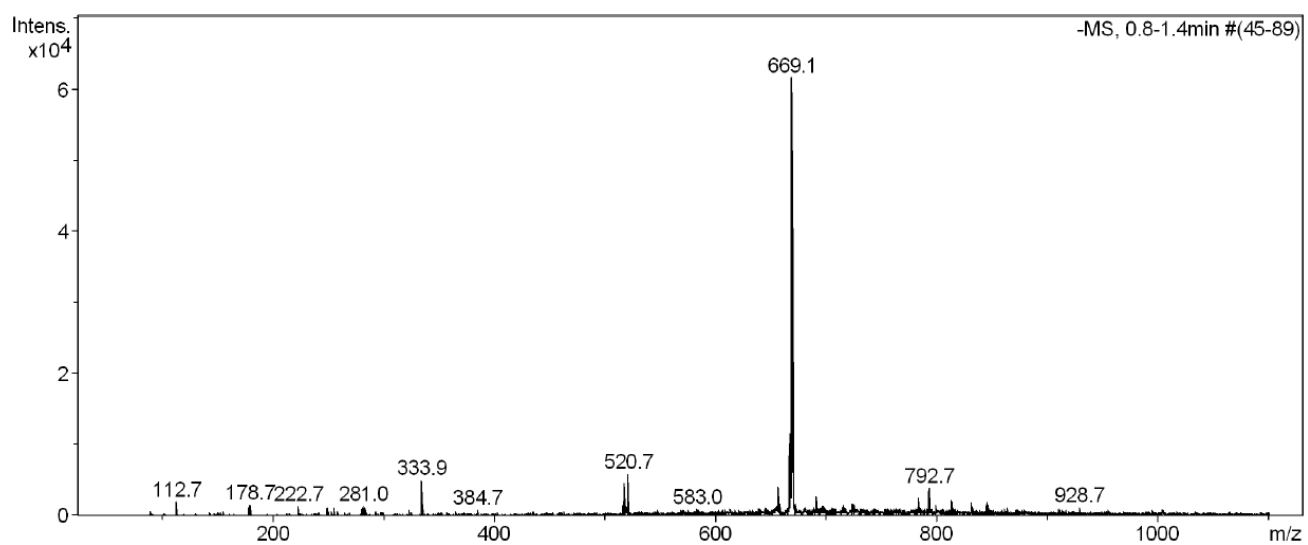
HPLC (λ = 220 nm)



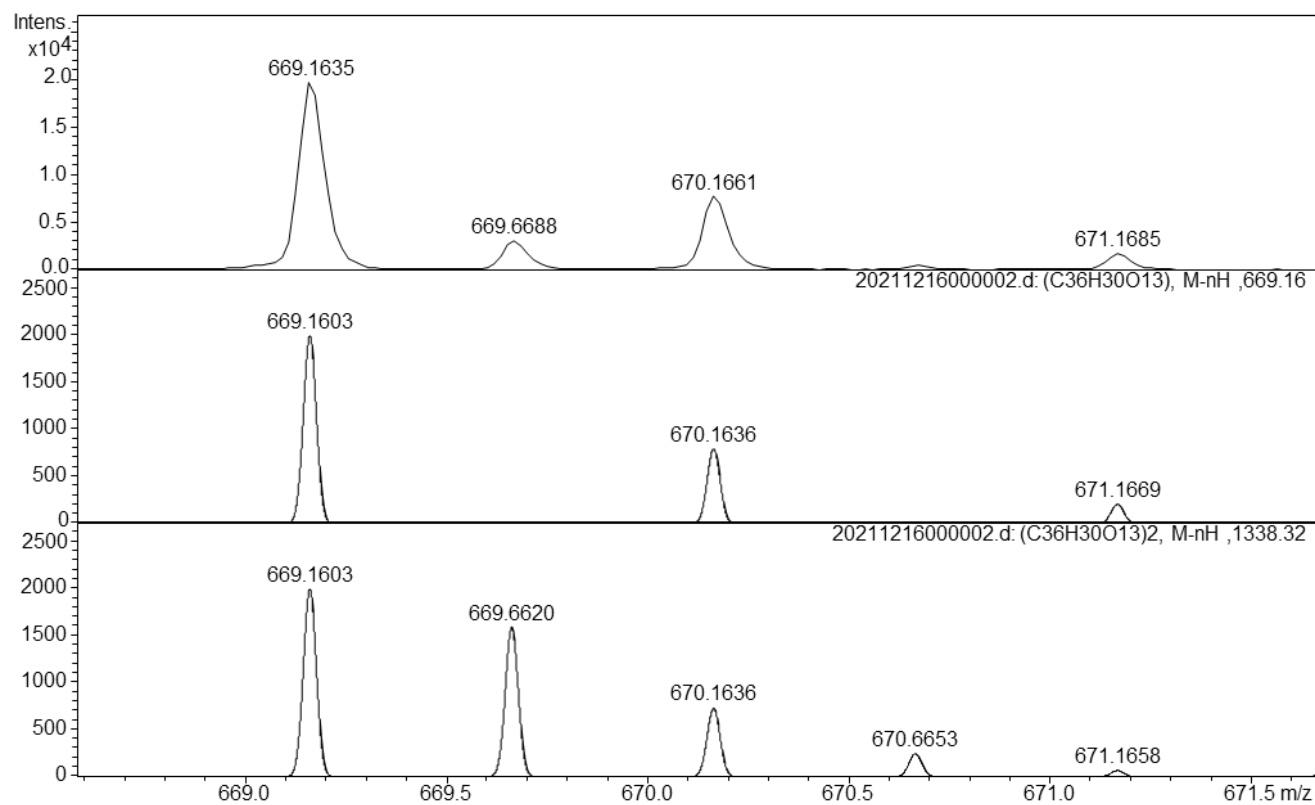
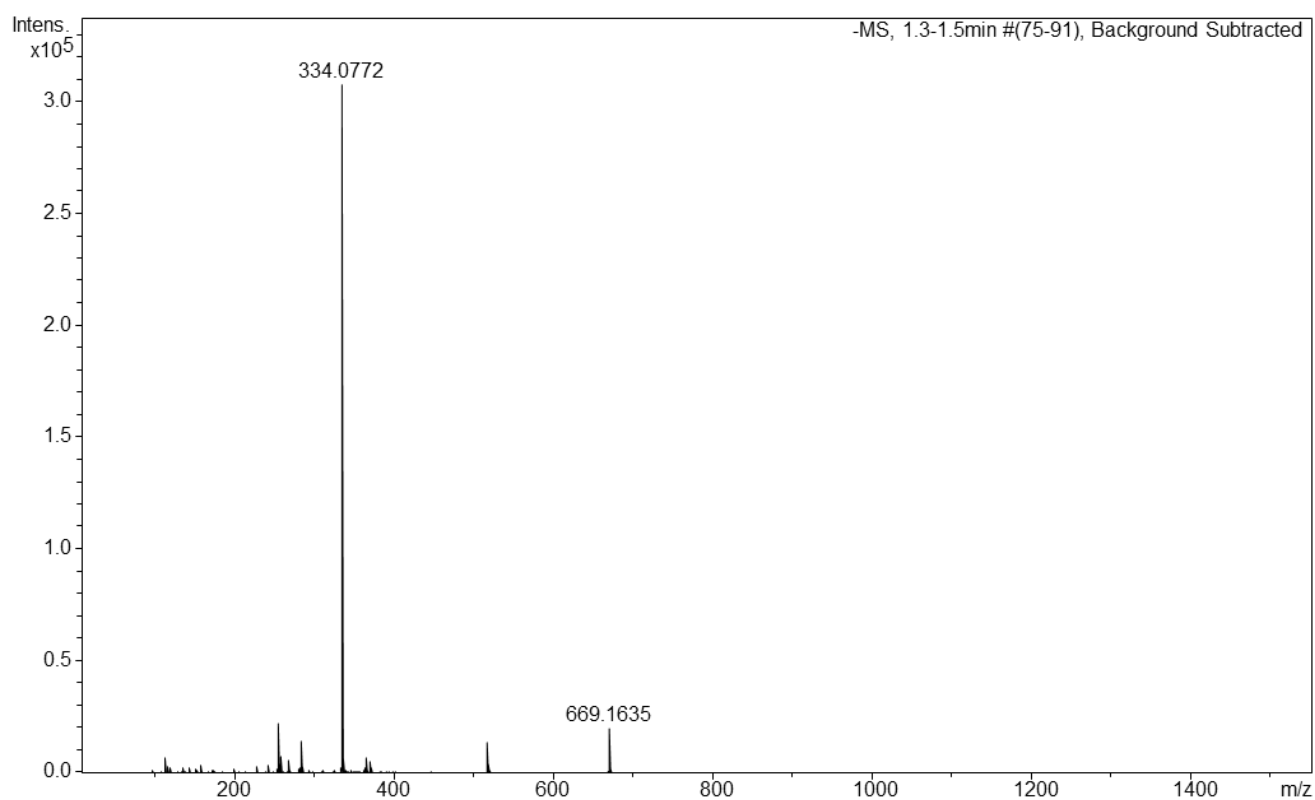
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.853	BV E	0.0401	399.56467	149.81126	6.9752
2	7.927	VV R	0.0487	5328.81641	1647.83044	93.0248

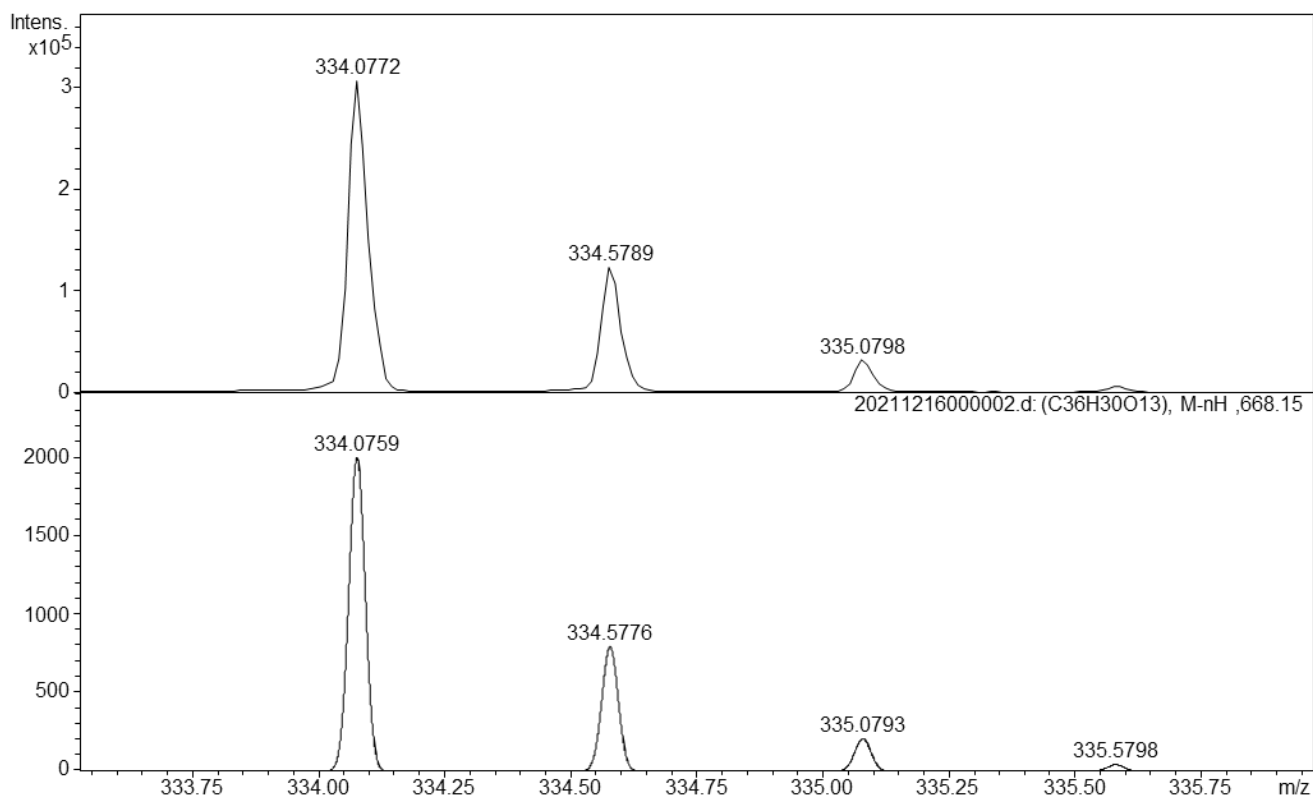
Totals : 5728.38107 1797.64171

(-)-ESI-MS (m/z)



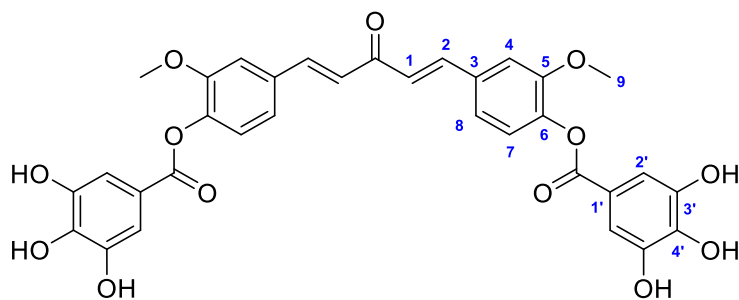
HRMS (*m/z*)



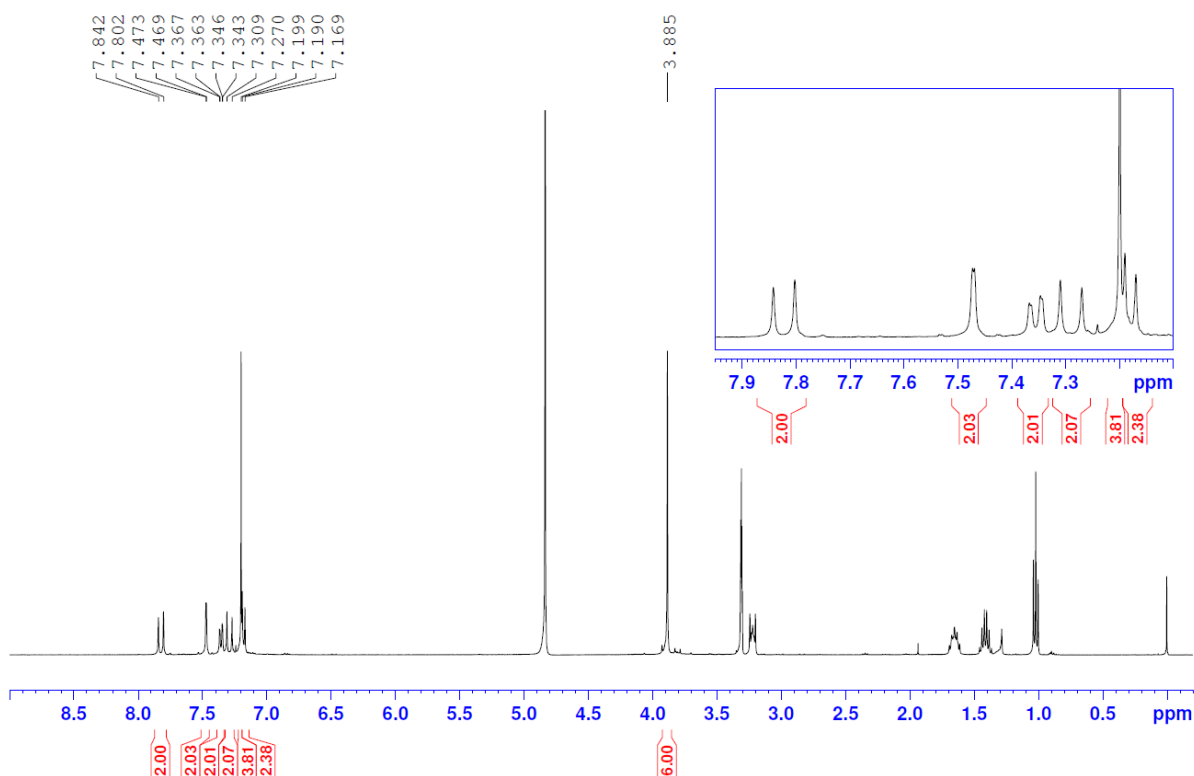


	Molecular formula	Calculated	Found
[M-H] ⁻	C ₃₆ H ₂₉ O ₁₃	669.1603	669.1635
[2M-2H] ⁻	(C ₃₆ H ₂₉ O ₁₃) ₂	669.1603	669.1635
[M-2H] ²⁻	C ₃₆ H ₂₈ O ₁₃	334.0759	334.0772

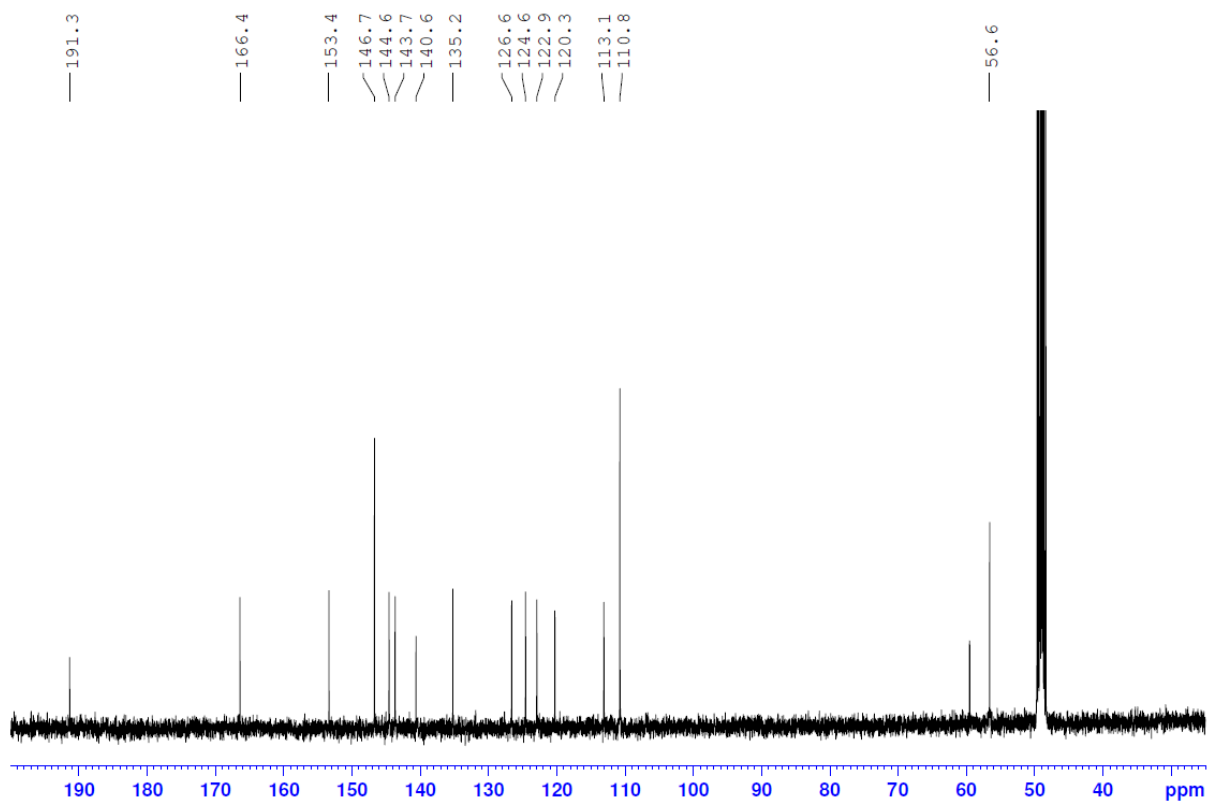
2.14. ((1*E*,4*E*)-3-Oxopenta-1,4-diene-1,5-diyl)bis(2-methoxy-4,1-phenylene) bis[3,4,5-tris((*tert*-butyldimethylsilyl)oxy)benzoate] (TL6)



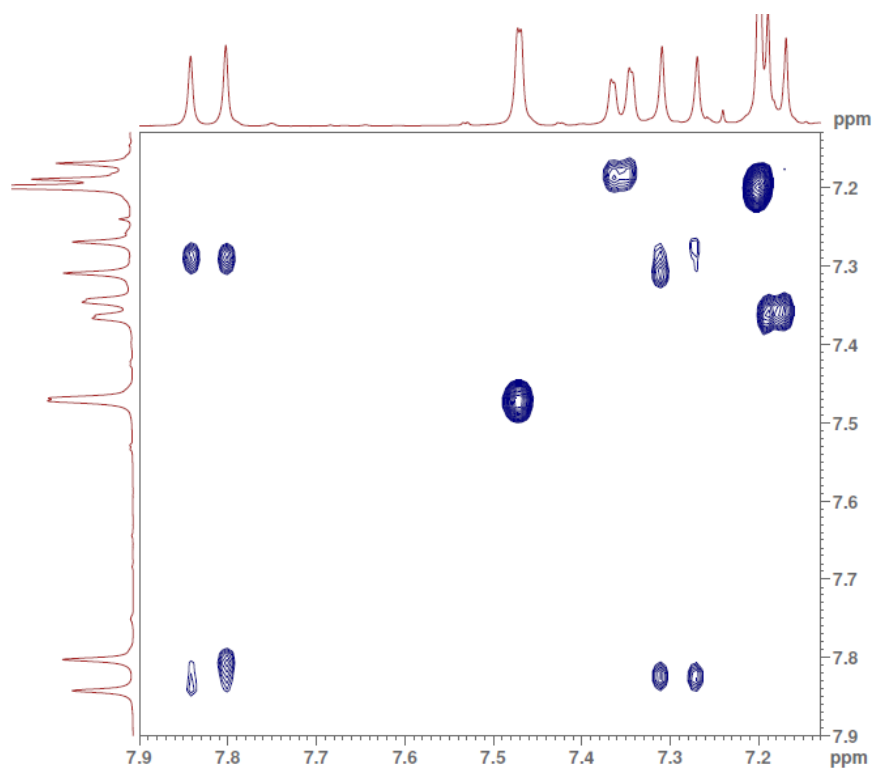
¹H-NMR (400 MHz, CD₃OD), δ (ppm)



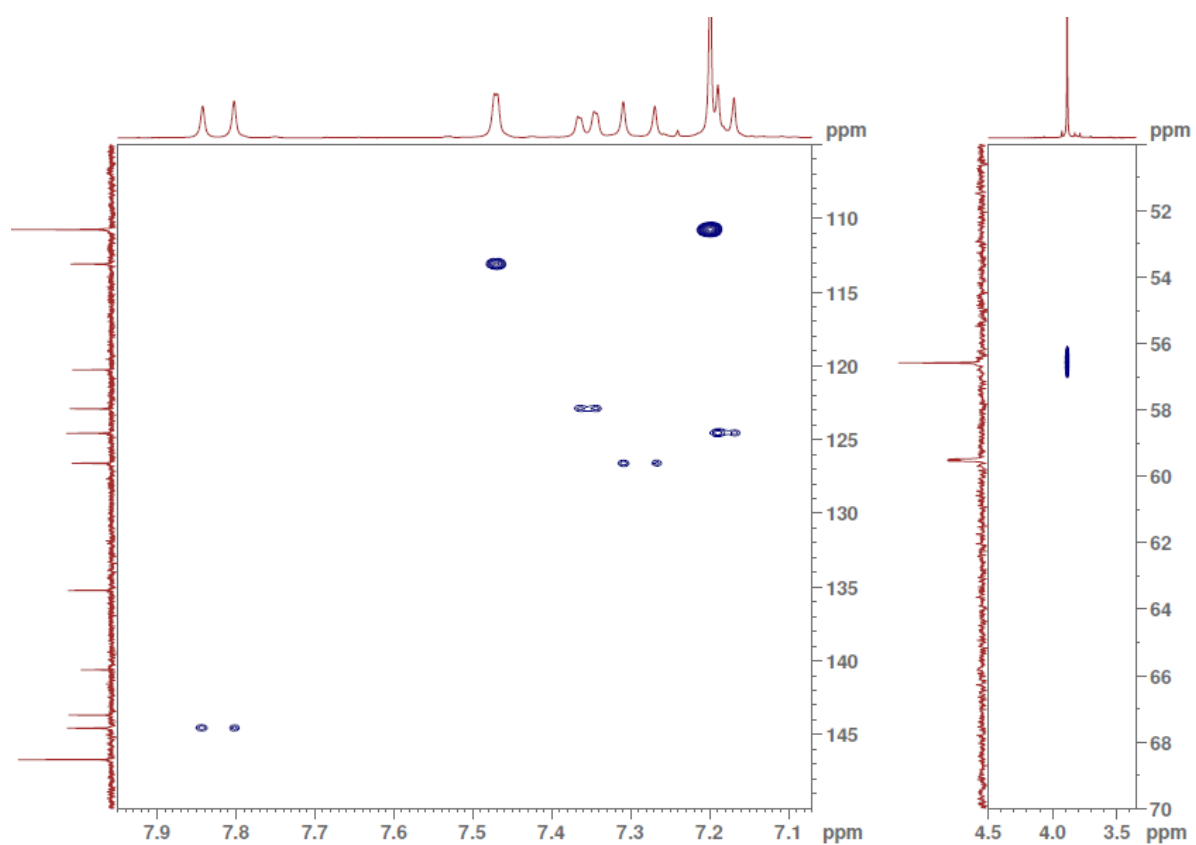
^{13}C -NMR (100 MHz, CD_3OD), δ (ppm)



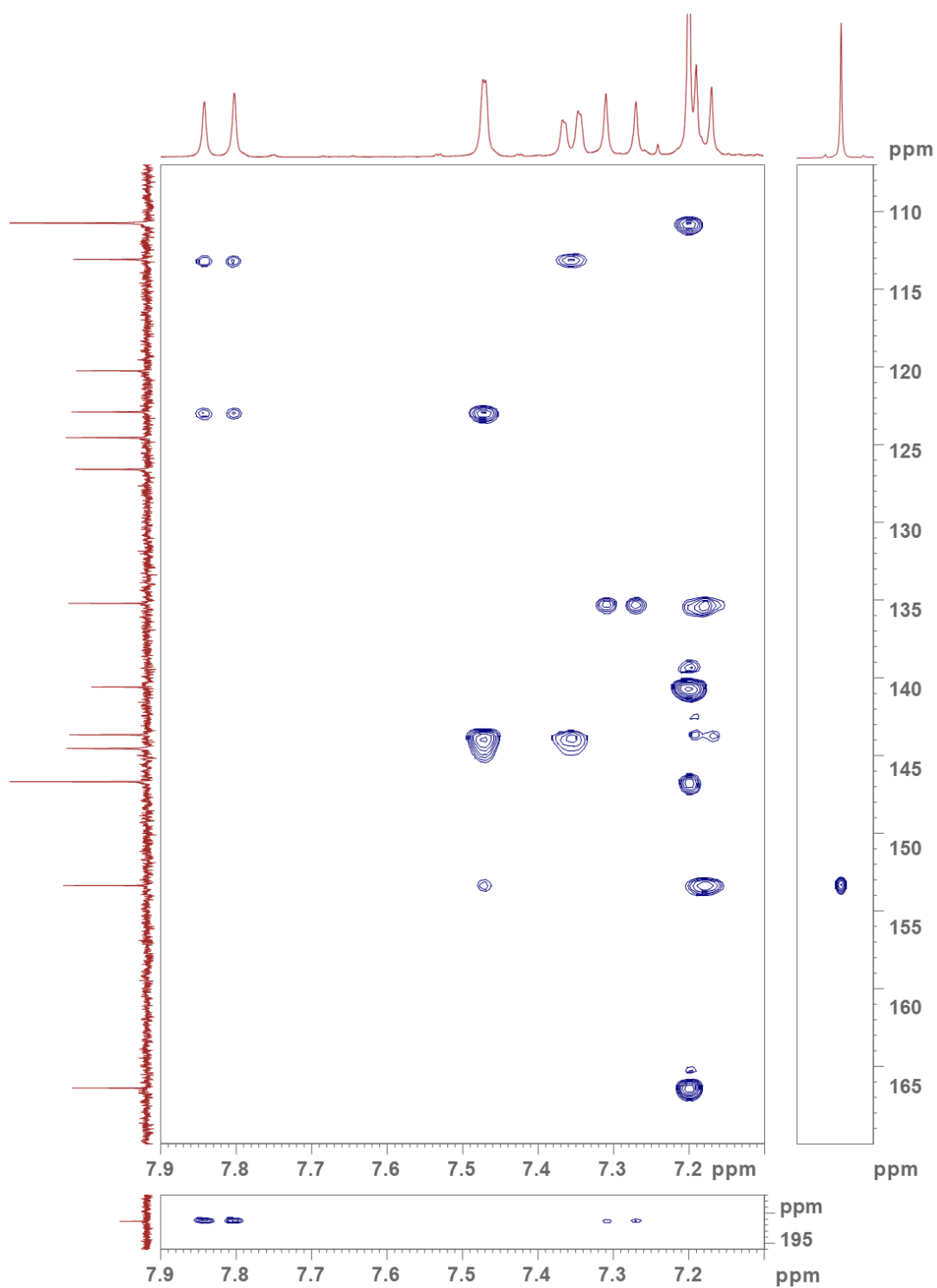
COSY ^1H - ^1H (CD_3OD), δ (ppm)



HSQC ^1H - ^{13}C (CD_3OD), δ (ppm)



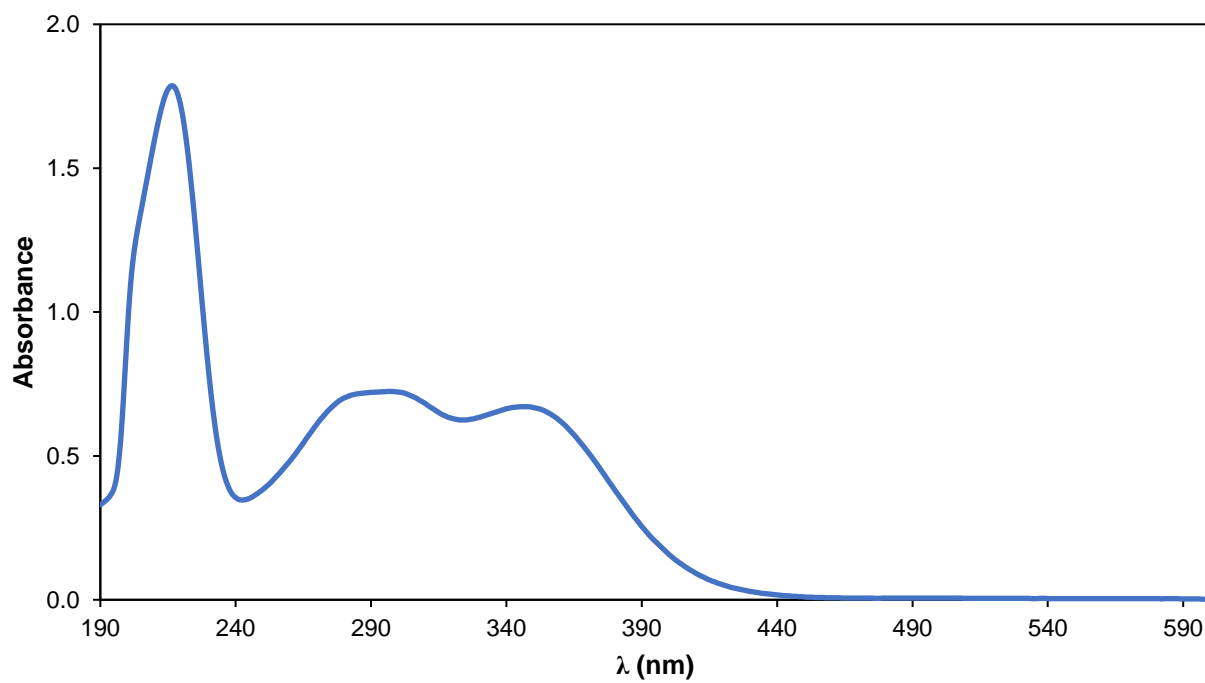
HMBC ^1H - ^{13}C (CD_3OD), δ (ppm)



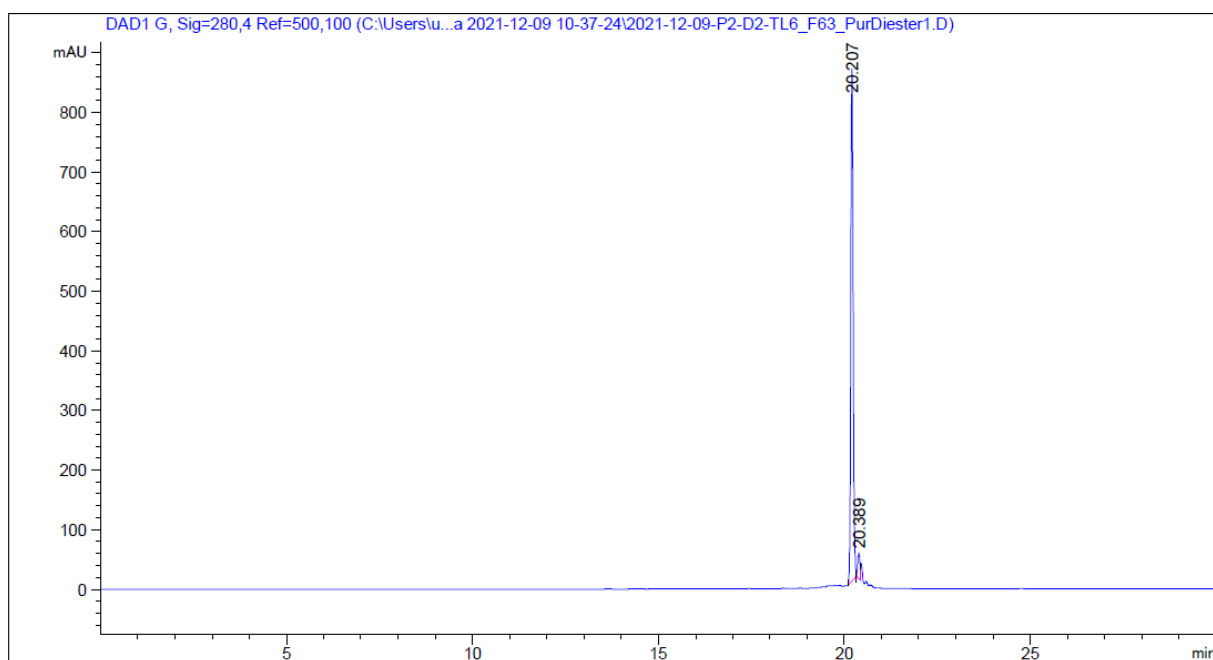
FT-IR (neat), ν (cm⁻¹)



UV-Vis (CH₃CN), λ (nm)



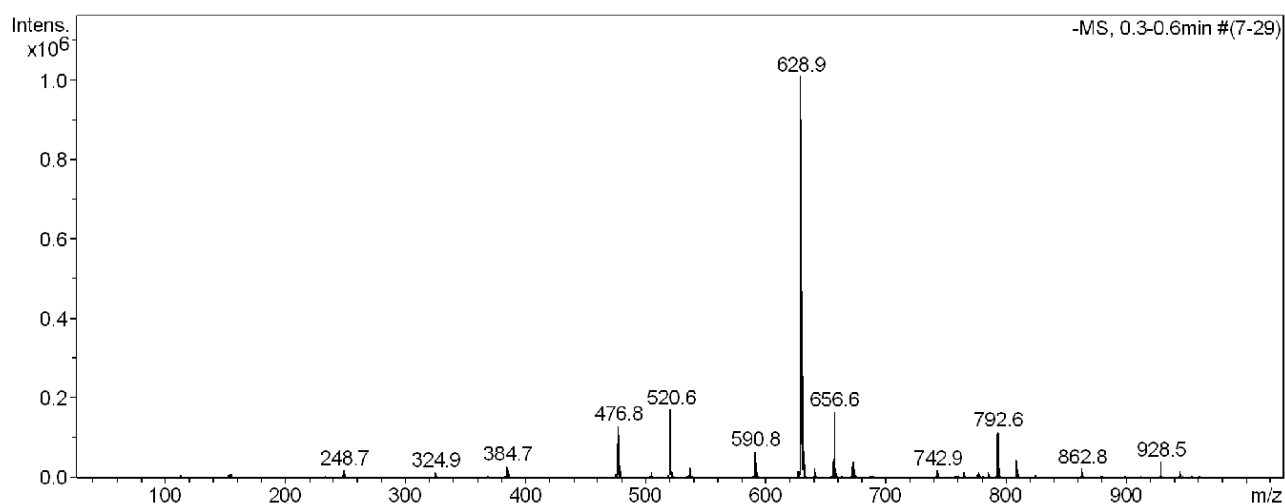
HPLC ($\lambda = 220 \text{ nm}$)



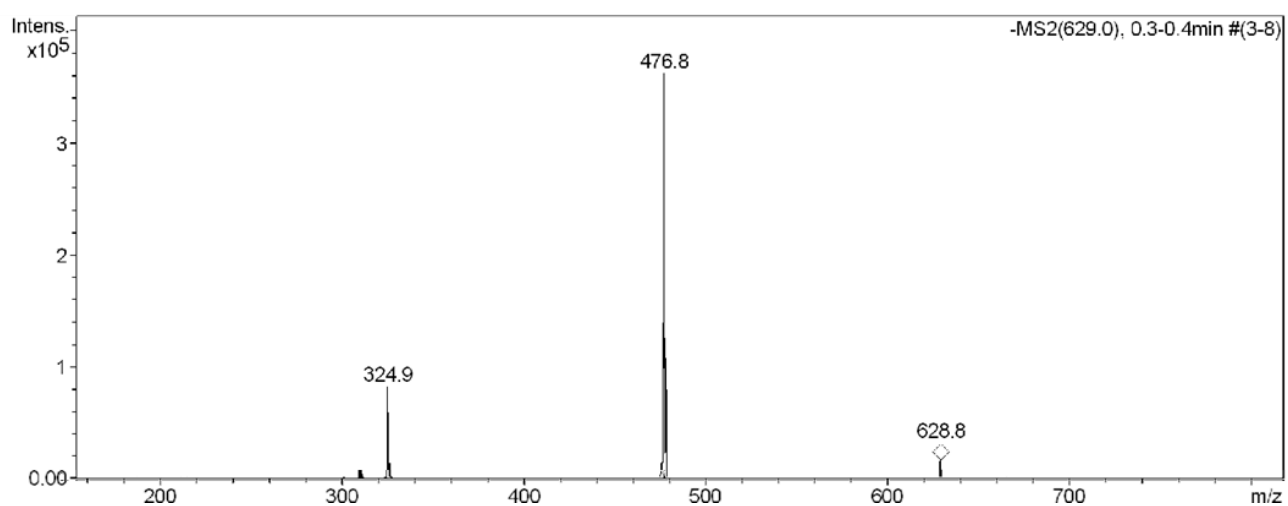
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	20.207	BB	0.0681	3769.46753	861.06512	95.6510
2	20.389	BV	0.0615	171.38843	42.06042	4.3490

Totals : 3940.85596 903.12555

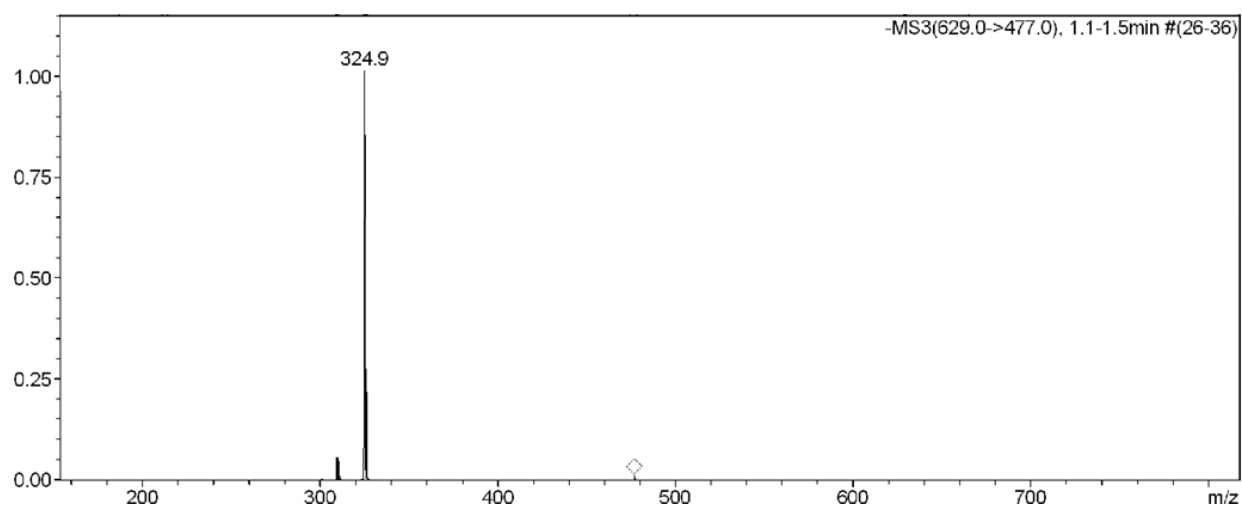
(-)-ESI-MS (m/z)



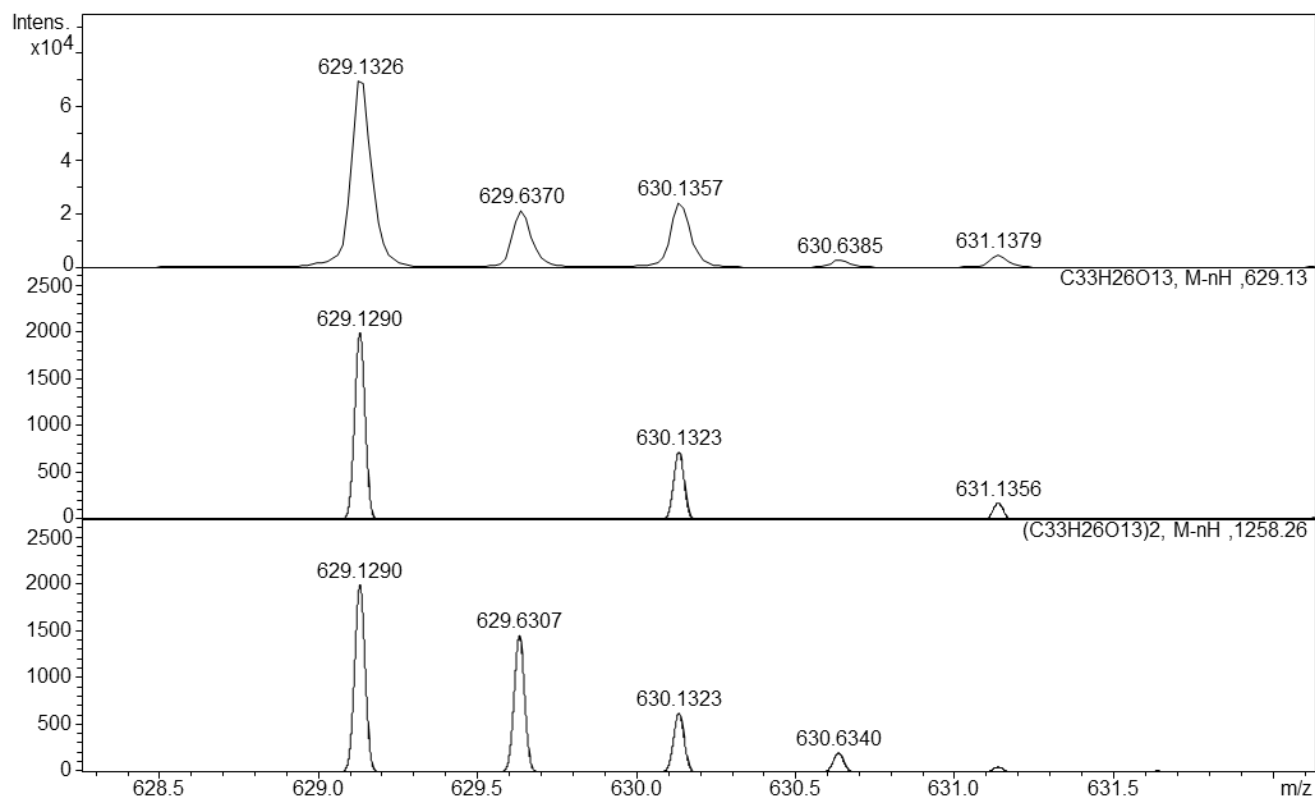
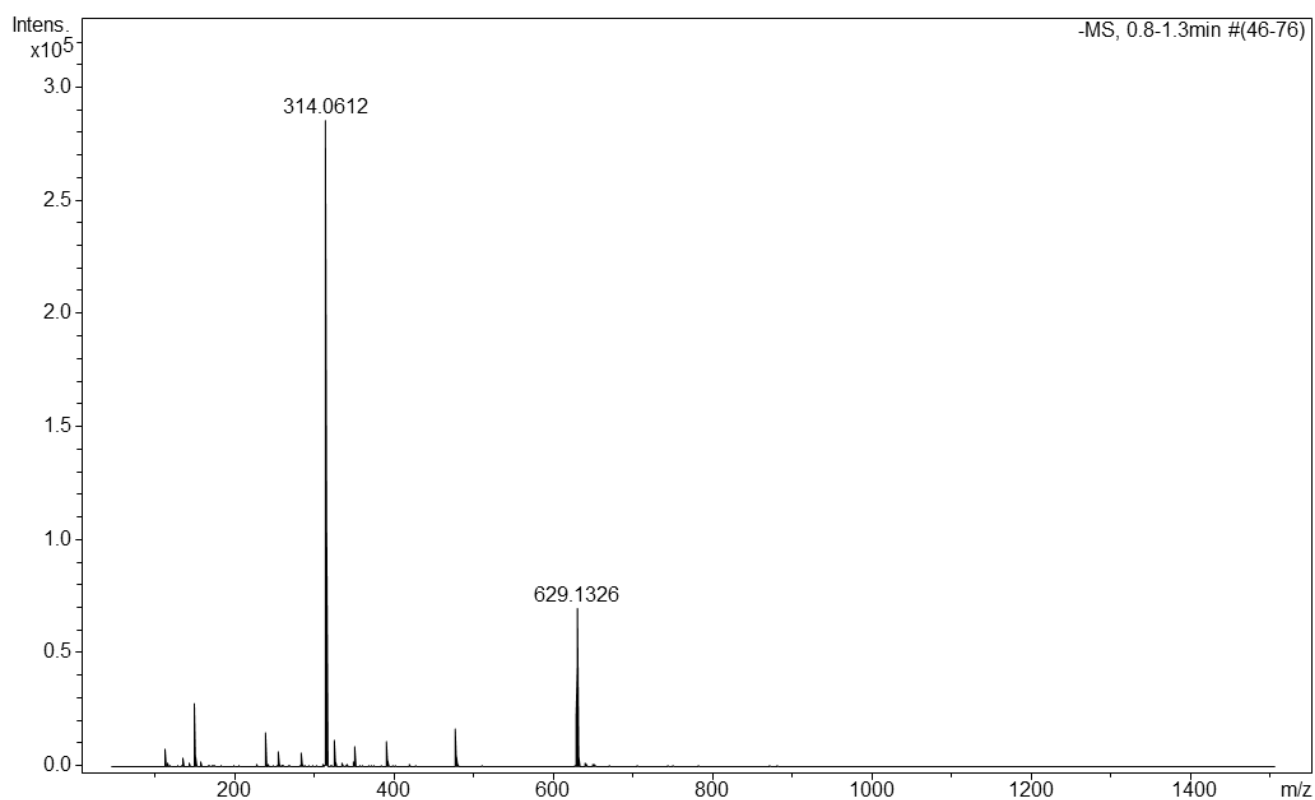
(-)-ESI-MS² (m/z = 628.9)

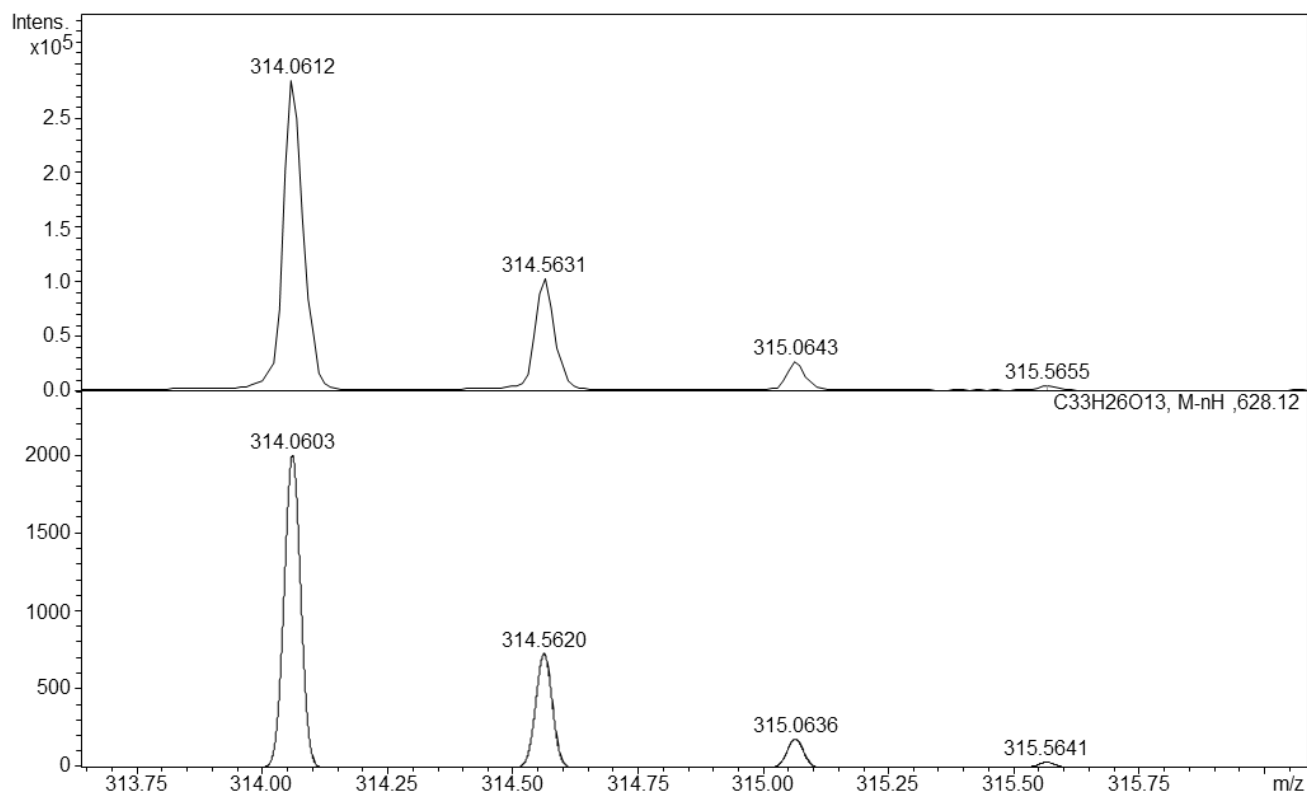


(-)-ESI-MS³ (m/z = 476.8)



HRMS (m/z)





	Molecular formula	Calculated	Found
[M-H] ⁻	<chem>C33H25O13</chem>	629.1290	629.1326
[2M-2H] ⁻	(<chem>C33H25O13</chem>) ₂	629.1290	629.1326
[M-2H] ²⁻	<chem>C33H24O13</chem>	314.0612	314.0603