Integrating Metal-Catalyzed C–H and C–O Functionalization to Achieve Sterically Controlled Regioselectivity in Arene Acylation

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General Experimental Procedures: All acylation reactions were prepared under inert atmosphere in an oxygen-regulated glovebox. Reaction progress was monitored using thin-layer chromatography (TLC) on 0.25 mm silica plates from SiliCycle. Eluted plates were visualized with UV light. Silica Gel Flash Column Chromatography was performed on 230–400 mesh (particle size 0.04–0.063 mm) silica gel purchased from SiliCycle.

Materials. Unless otherwise indicated, chemicals were obtained from commercial sources and used without further purification. All arenes were degassed by bubbling a stream of argon through the liquid in a Schlenk flask and stored over 3 Å molecular sieves in a nitrogen-filled glovebox.

Instrumentation: NMR characterization data were collected at 300 K on Bruker FT NMR instruments. ^{1}H NMR spectra were internally referenced to TMS (δ = 0 ppm). ^{13}C NMR spectra were internally referenced to the residual solvent signal (δ = 77.2 ppm). Data from ^{1}H NMR are reported as follows: chemical shift (δ ppm), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad), coupling constant (Hz), integration.

Melting point ranges for solid products were determined on a MEL-TEMP instrument and are reported uncorrected.

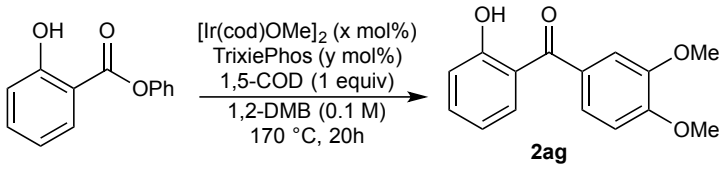
IR Spectra were obtained as films either neat or from CH_2Cl_2 on sodium chloride plates on a Thermo Scientific FT-IR or with an ATR source using a Nicolet iS5 FT-IR spectrometer. Data are presented as absorption frequency (cm^{-1}).

High-resolution mass spectrometry (HRMS) using GC-MS was performed on an Agilent 7200-QTOF GC/MS, GC column RTX-5MS 30 m length, 0.255 mm ID, 0.25 µm df. Method: inlet temperature 250

˚C, source temperature 280 ˚C. The initial column temperature of 120 ˚C was held for 4 minutes after injection. Column temperature was ramped to 325 ˚C over 10 minutes and then held for 31 minutes. High-resolution mass spectrometry (HRMS) using ESI experiments were performed on a Bruker BioTOF II instrument using sodium trifluoroacetate internal standard and ammonium bicarbonate additive.

REACTION OPTIMIZATION

Control Experiments

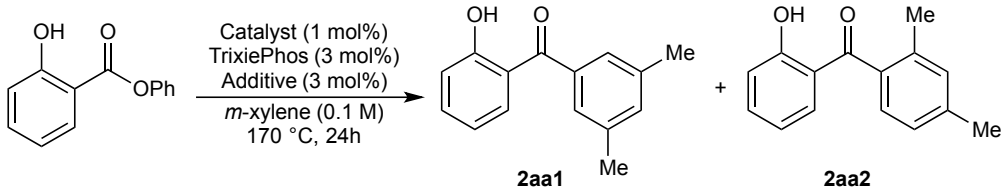


Reactions were prepared using phenyl salicylate (0.2 mmol, 1.0 equiv) *Rac*-2-(di-*t*-butyldiphosphino)-1,1'-binapthyl (TrixiePhos, y mol%), $[Ir(cod)OMe]_2$ (x mol%), 1,5-cyclooctadiene (1,5-COD, 1.0 equiv), and 1,2-dimethoxybenzene (1,2-DMB, 0.1 M) in PTFE-lined crimp-top vials. The vials sealed, removed from the glovebox, and heated to 170 °C in an oil bath for twenty hours. Product yields were determined by ^{1}H NMR using 1,3,5-trimethoxybenzene internal standard (0.8 mL, 0.1M in $CDCl_3$).

Table S1. Control Experiments

Entry	[Ir] (mol%)	TrixiePhos (mol%)	Yield **2ag** (%) (^{1}H NMR)
1	0	3	0
2	1	0	24
3	0	0	0

Catalyst Optimization

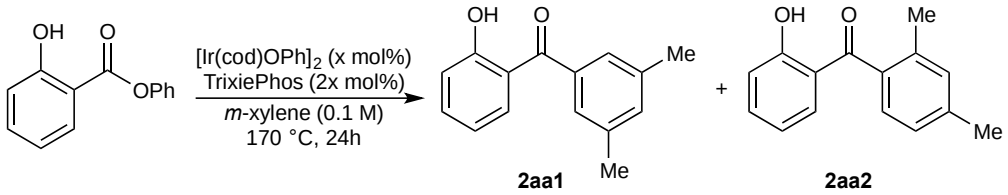


Reactions were prepared using phenyl salicylate (0.1 mmol, 1.0 equiv), TrixiePhos (0.003 mmol, 3 mol%), catalyst (0.001 mmol, 1 mol%), base (0.018 mmol, 3 mol%), and *m*-xylene (1.0 mL, 0.1 M) in PTFE-lined crimp-top vials. The vials were sealed, removed from the glovebox, and heated to 170 °C in an oil bath for 24 h. Product yields were determined by ^{1}H NMR using 1,3,5-trimethoxybenzene internal standard (0.7 mL, 0.143M in $CDCl_3$).

Table S2. Effect of catalyst choice on product yield

Entry	Catalyst	Additive	Yield **2aa1** (%) (^{1}H NMR)
1	$[Ir(cod)OPh]_2$	-	81
2	$[Ir(cod)Cl]_2$	KOPh	44
3	$[Ir(cod)Cl]_2$	K_2CO_3	3
4	$[Ir(coe)_2Cl]_2$	KOPh	49
5	$[Ir(coe)_2Cl]_2$	K_2CO_3	1

Catalyst Loading Optimization

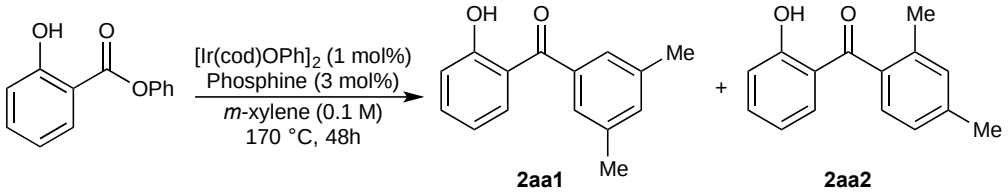


Reactions were prepared using phenyl salicylate (0.1 mmol, 1.0 equiv), $[Ir(cod)OPh]_2$ (x mol%), TrixiePhos (2x mol%) and *m*-xylene (1.0 mL, 0.1 M) in PTFE-lined crimp-top vials. The vials were sealed, removed from the glovebox, and heated to 170 °C in an oil bath for 24 h. Product yields were determined by ^{1}H NMR using 1,3,5-trimethoxybenzene internal standard (0.7 mL, 0.143M in $CDCl_3$).

Table S3. Effect of catalyst loading on product yield and distribution

Entry	[Ir] (mol%)	Yield **2aa1** (%) (^{1}H NMR)	Ratio **2aa1**:**2aa2** (^{1}H NMR)
1	0.5	49	>20:1
2	1	70	18:1
3	2.5	69	15:1
4	5	60	11:1
5	10	43	7:1

Phosphine/Ligand Screen



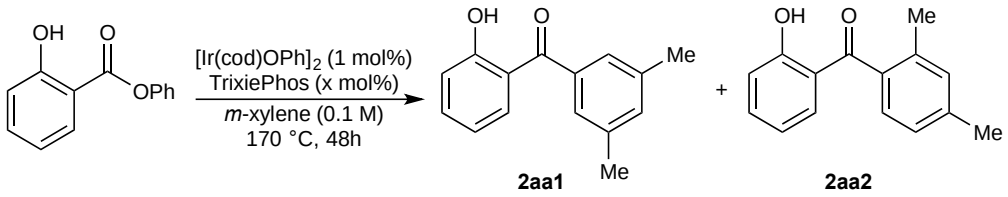
Reactions were prepared using phenyl salicylate (0.1 mmol, 1.0 equiv), $[Ir(cod)OPh]_2$ (0.001 mmol, 1 mol%), Phosphine (0.003 mmol, 3 mol%) and *m*-xylene (1.0 mL, 0.1 M) in PTFE-lined crimp-top vials. The vials were sealed, removed from the glovebox, and heated to 170 °C in an oil bath for 48 h. Product

yields were determined by ^{1}H NMR using 1,3,5-trimethoxybenzene internal standard (0.7 mL, 0.143M in $CDCl_3$).

Table S4. Effect of phosphine/ligand identity on product yield

Entry	Phosphine	Yield **2aa1** (%) (^{1}H NMR)
1	*t*-Bu Xphos	44
2	Xphos	6
3	*t*-Bu BrettPhos	58
4	BrettPhos	4
5	Cy-JohnPhos	8
6	JohnPhos	61
7	*t*-Bu MePhos	55
8	MePhos	7
9	*t*-Bu DavePhos	47
10	DavePhos	6
11	RuPhos	11
12	S-Phos	10
13	TrixiePhos	60
14	P(*o*-tol)$_3$	2
15	dppp	0
16	bpy	0
17	TMEDA	0

Phosphine Loading Optimization



Reactions were prepared using phenyl salicylate (0.1 mmol, 1.0 equiv), $[Ir(cod)OPh]_2$ (0.001 mmol, 1 mol%), TrixiePhos (x mol%) and *m*-xylene (1.0 mL, 0.1 M) in PTFE-lined crimp-top vials. The vials were sealed, removed from the glovebox, and heated to 170 °C in an oil bath for 48 h. Product yields were determined by ^{1}H NMR using 1,3,5-trimethoxybenzene internal standard (0.7 mL, 0.143M in $CDCl_3$).

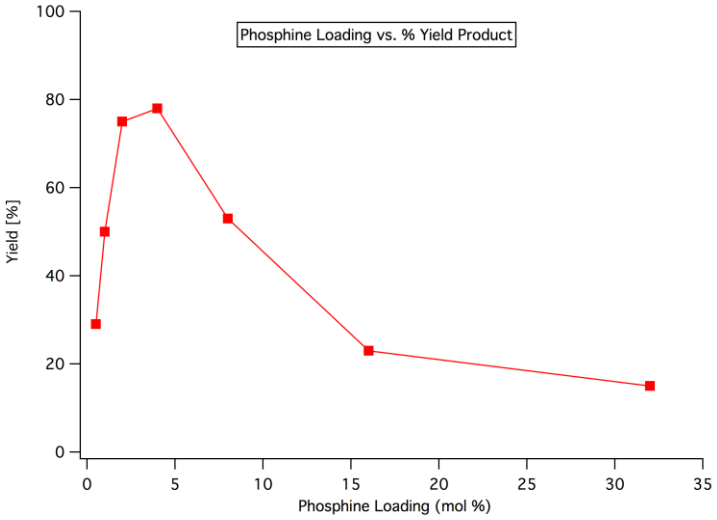
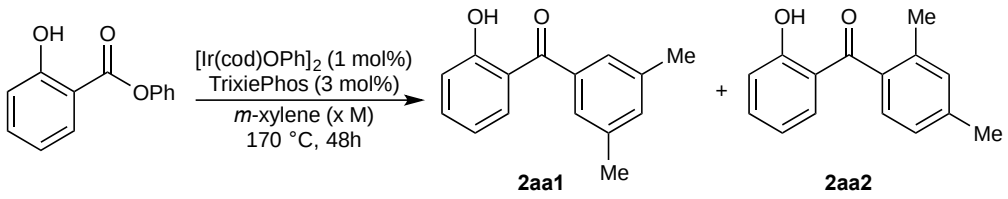


Table S5. Effect of phosphine loading on product yield

Entry	TrixiePhos (mol%)	Yield **2aa1** (%) (^{1}H NMR)
1	0.5	29
2	1	50
3	2	75
4	4	78
5	8	53
6	16	23
7	32	15

Substrate Concentration Optimization



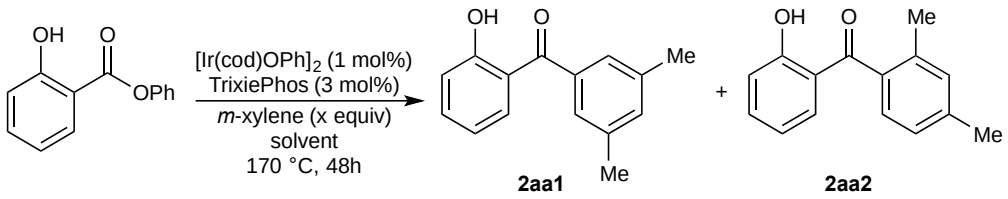
Reactions were prepared using phenyl salicylate (1.0 equiv), $[Ir(cod)OPh]_2$ (1 mol%), TrixiePhos (3 mol%) and *m*-xylene (volume required to obtain listed concentration of phenyl salicylate) in PTFE-lined crimp-top vials. The vials were sealed, removed from the glovebox, and heated to 170 °C in an oil bath for 48 h. Product yields were determined by ^{1}H NMR using 1,3,5-trimethoxybenzene internal standard (0.7 mL, 0.143M in $CDCl_3$).

Table S6. Effect of substrate concentration on product yield

Entry	Phenyl salicylate (mmol)	Phenyl Salicylate (M)	Yield **2aa1** (%) (^{1}H NMR)
1	0.05	0.05	76
2	0.05	0.07	77
3	0.1	0.10	80
4	0.1	0.14	80
5	0.2	0.20	69
6	0.2	0.29	61
7	0.2	0.40	51

Effect of Arene Dilution

We made a preliminary attempt use less than solvent quantities of arene in the acylation reaction. Decaline and mesitylene were examined as solvents in the acylation below. Lower concentration of arene led to significantly lower yield of acylation products.

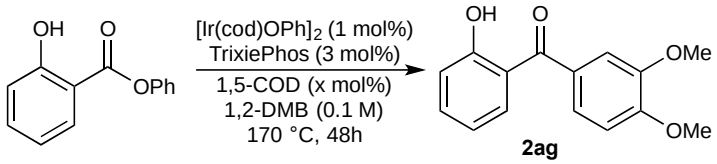


Reactions were prepared in PTFE-lined crimp-top vials using phenyl salicylate (0.1 mmol, 1.0 equiv), $[Ir(cod)OPh]_2$ (0.001 mmol, 1 mol%), TrixiePhos (0.003 mmol, 3 mol%), *m*-xylene (x equiv), and additional solvent to dilute to a final volume of 1.0 mL. The vials were sealed, removed from the glovebox, and heated to 170 °C in an oil bath for 48 h. Product yields were determined by ^{1}H NMR using 1,3,5-trimethoxybenzene internal standard (0.7 mL, 0.143M in $CDCl_3$).

Table S7. Effect of arene equivalencies on product yield

Entry	*m*-xylene (equiv)	Additional Solvent	Yield **2aa1** (%) (^{1}H NMR)
1	40.5	Decalin	51
2	20.3	Decalin	33
3	8.1	Decalin	13
4	4.0	Decalin	6
5	2.0	Decalin	3
6	40.5	Mesitylene	59
7	20.3	Mesitylene	39
8	8.1	Mesitylene	18
9	4.0	Mesitylene	9
10	2.0	Mesitylene	5

COD Equivalents Optimization

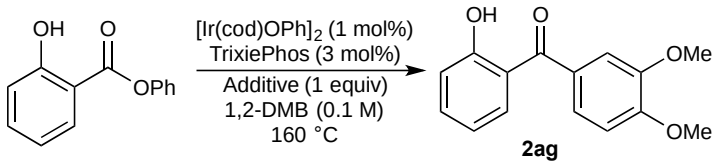


Reactions were prepared using phenyl salicylate (1.0 equiv), $[Ir(cod)OPh]_2$ (1 mol%), TrixiePhos (3 mol%), 1,5-COD (volume required to obtain listed concentrations) and 1,2-dimethoxybenzene (0.1 M final concentration relative to phenyl salicylate, 1 mL final volume) in PTFE-lined crimp top vials. The vials were sealed, removed from the glovebox, and heated to 170 °C in an oil bath for 4 hr. Product yield was determined by ^{1}H NMR using dibromomethane internal standard.

Table S8. Effect of 1,5-cyclooctadiene additive on product yield

Entry	1,5-COD (μL)	1,5-COD (mol%)	Yield **2ag** (%) (^{1}H NMR)
1	0	0	39
2	20	2	47
3	40	4	50
4	60	6	53
5	80	8	49
6	120	12	53
7	160	16	59
8	200	20	59
9[1]	12	20	20
10[1]	61	100	27
11[1]	310	500	52

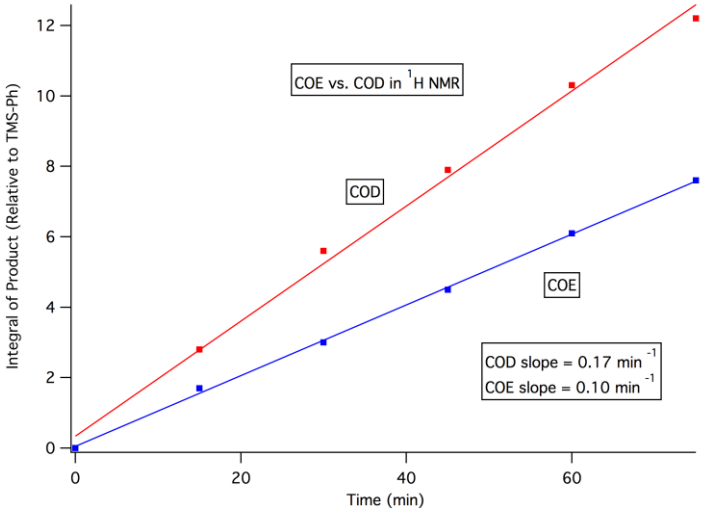
Alkene Additive Screen



Reactions were prepared using phenyl salicylate (1 equiv), $[Ir(cod)OMe]_2$ (1 mol%), TrixiePhos (3 mol%), alkene additive (1 equiv) and 1,2-dimethoxybenzene (0.8 mL) in a 5 mm J-young NMR tube. The tube was sealed, removed from the glovebox, and heated to 160 °C. Single-scan ^{1}H NMR spectra were

[1] Reactions were run on a 5 mL scale for 2 hours.

taken at regular intervals. The reaction progress was monitored until approximately 10% completion was observed, relative to trimethyl(phenyl)silane internal standard.



*Norbornadiene gives no significant conversion to product after 45 minutes at 160 °C.

Iridium Carbonyl from Catalyst Decomposition

TrixiePhos (10 mg, 0.024 mmol, 3 mol%), $[Ir(cod)OMe]_2$ (50 mg, 0.08 mmol, 10 mol%, phenyl salicylate (0.171 g, 0.8 mmol, 1 equiv), 1,5-COD (98 µL, 0.8 mmol, 1 equiv), and mesitylene (8 mL, 0.1 M) were added to a 10 mL PTFE-lined crimp-top vial. The vial was sealed, removed from the glovebox, and heated to 170 °C in an oil bath for 20 h. The solvent was removed *in vacuo.* An IR spectrum was obtained of the crude product mixture. Distinguishing peaks at 1981 and 2027 cm^{-1} indicated the probable presence of an Ir–CO complex.

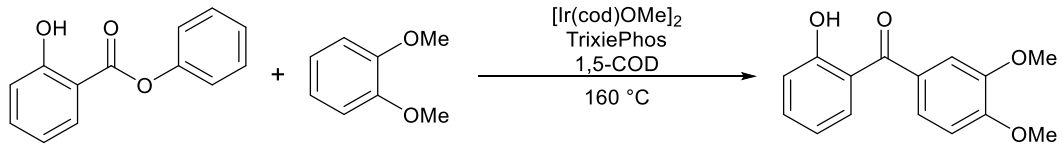
Phosphorus Degradation

TrixiePhos (5.0 mg, 0.013 mmol, 3 mol%), $[Ir(cod)OMe]_2$ (3.0 mg, 0.005 mmol, 1 mol%), phenyl salicylate (90.1 mg, 0.421 mmol, 1 equiv), 1.5-COD (51 µL, 0.416 mmol, 1 equiv) and 1,3-dimethoxybenzene (4.3 mL, 0.1 M) were mixed in a 20 mL scintillation vial. 0.6 mL of the solution was transferred to a J-Young NMR tube, the tube was sealed and removed from the glovebox. A ^{31}P NMR spectrum was taken (t=0), and the reaction mixture was heated to 170 °C in an oil bath. ^{31}P NMR spectra were taken at regular increments.

Reaction in CO Atmosphere

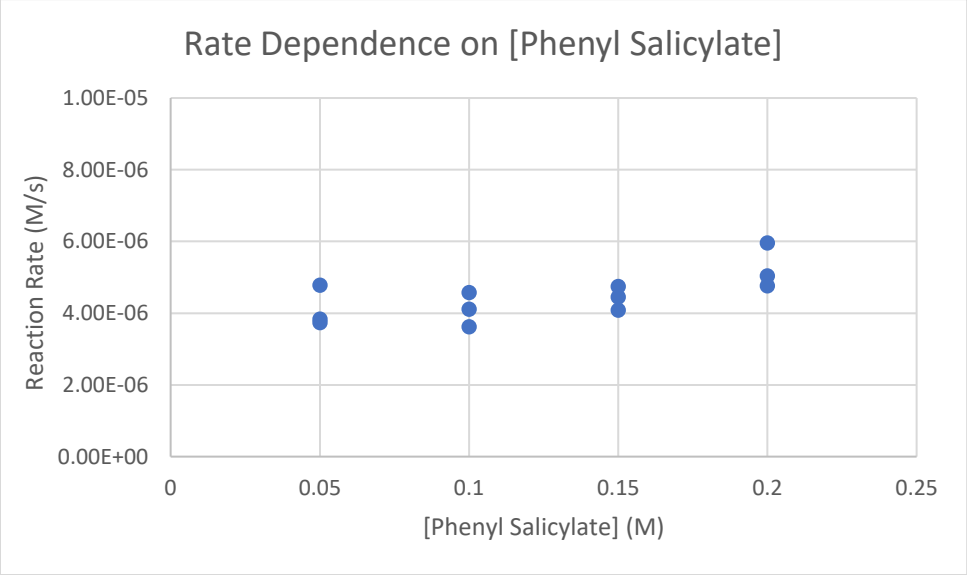
TrixiePhos (9.6 mg, 0.024 mmol, 3 mol%), $[Ir(cod)OMe]_2$ (5.3 mg, 0.008 mmol, 1 mol%), phenyl salicylate (171.4 mg, 0.800 mmol, 1 equiv), 1.5-COD (98 µL, 0.800 mmol, 1 equiv) and 1,3-dimethoxybenzene (8.0 mL, 0.1 M) were mixed in a 20 mL scintillation vial. The solution was transferred to a 50 mL Schlenk tube, the tube was sealed and removed from the glovebox. The Schlenk tube was evacuated and backfilled with CO. The tube was sealed and heated to 170 °C in an oil bath for 20 h. Solvent was removed *in vacuo* and the crude product mixture was analyzed by ^{1}H NMR.

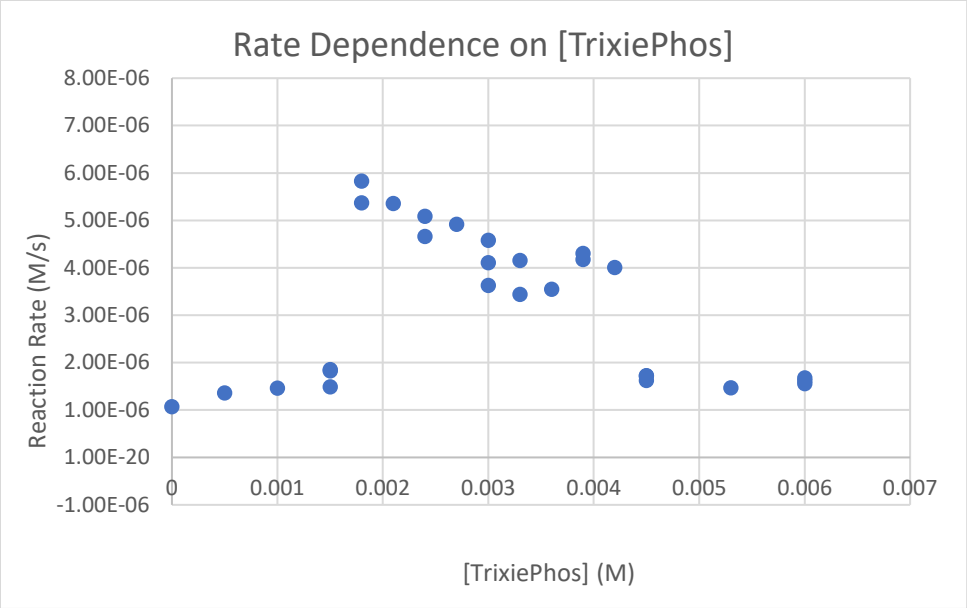
KINETIC STUDIES

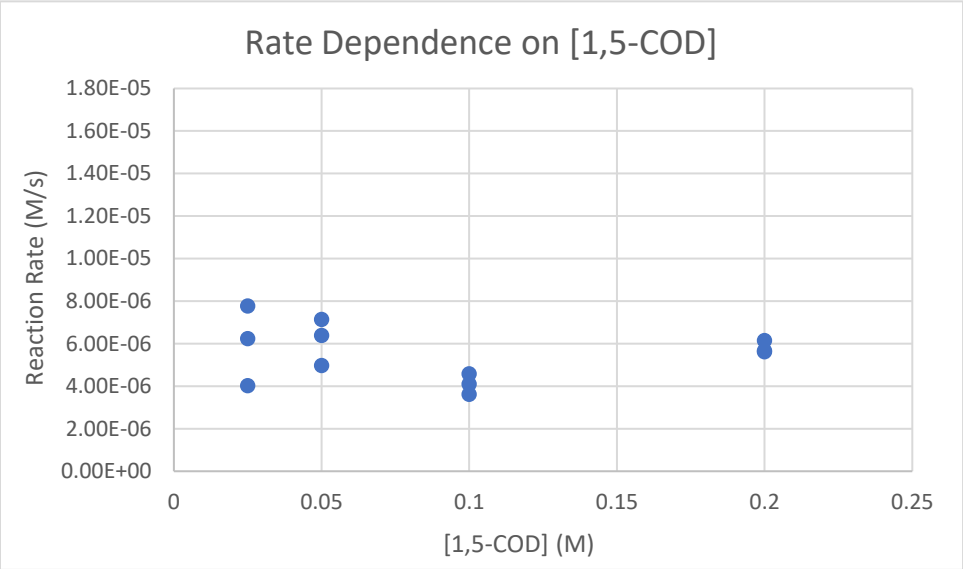


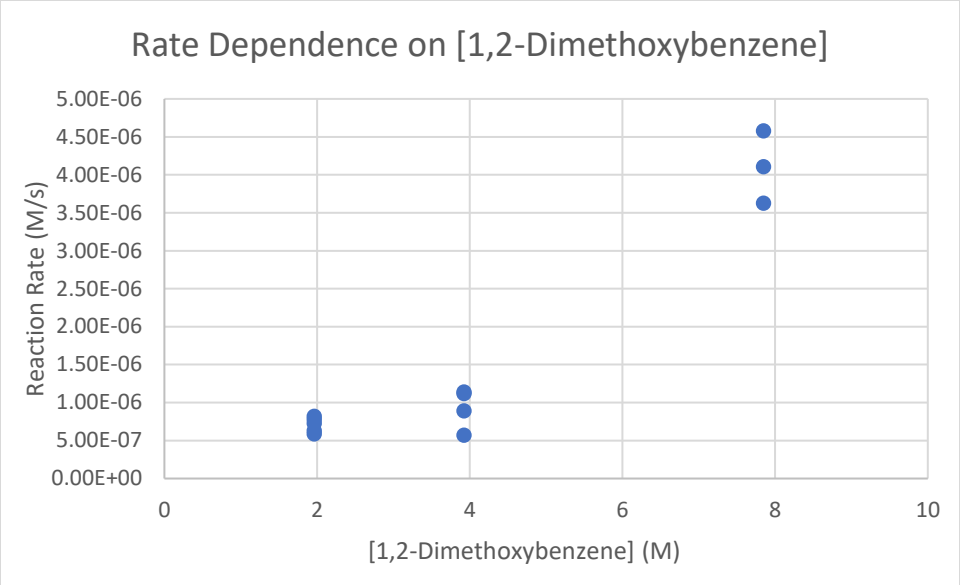
Initial Rates General. Reactions were prepared using phenyl salicylate, $[Ir(cod)OMe]_2$, TrixiePhos, 1,5-COD, in 1,2-dimethoxybenzene (0.8 mL) in a 5 mm J-young NMR tube. The tube was sealed, removed from the glovebox, and heated to 160 °C. Single-scan ^{1}H N MR spectra were taken at regular intervals. The reaction progress was monitored until approximately 10% completion was observed, relative to 1,3,5-trimethoxybenzene internal standard.

One reagent concentration was varied at a time to determine the reaction order of that component, all other reagent concentrations were held constant at the concentration used in standard reactions (phenyl salicylate (0.100 M), $[Ir(cod)OMe]_2$ (1.0 mM), TrixiePhos (3.0 mM), 1,5-COD (0.100 M)). When varying arene solvent concentration, 1,2-dimethoxybenzene was diluted with mesitylene.




Rate Dependence on [TrixiePhos]
Reaction Rate (M/s)
8.00E-06
7.00E-06
6.00E-06
5.00E-06
4.00E-06
3.00E-06
2.00E-06
1.00E-06
1.00E-20
-1.00E-06
0
0.001
0.002
0.003
0.004
0.005
0.006
0.007
[TrixiePhos] (M)

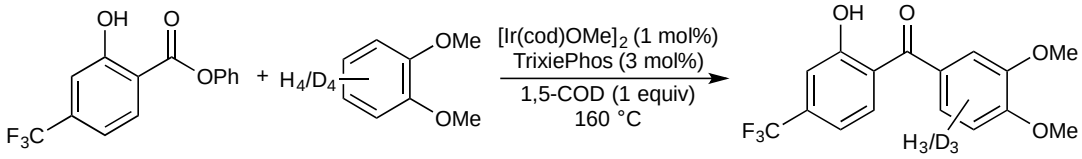

Rate Dependence on [1,5-COD]
Reaction Rate (M/s)
1.80E-05
1.60E-05
1.40E-05
1.20E-05
1.00E-05
8.00E-06
6.00E-06
4.00E-06
2.00E-06
0.00E+00
0
0.05
0.1
0.15
0.2
0.25
[1,5-COD] (M)



Kinetic Isotopes General. The kinetic isotope effects were determined by the following equation:

$$KIE = \frac{Prod_H}{Prod_D} = \frac{k_H}{k_D}$$

Initial Rates



Proteo - Reactions were performed in triplicate using 0.7 mL aliquots from a stock solution containing phenyl 4-(trifluoromethyl)salicylate (0.4 mmol, 1 equiv), $[Ir(cod)OMe]_2$ (0.004 mmol, 1 mol%), TrixiePhos (0.012 mmol, 3 mol%), 1,5-COD (0.4 mmol, 1 equiv), 1-methoxy-3-(trifluoromethyl)benzene (0.463 mmol, 1.16 equiv) and H_4-1,2-dimethoxybenzene (4 mL) in a J-Young NMR tube. Once sealed, the tubes were removed from the glovebox, and single-scan ^1H and ^{19}F NMR spectra were taken for time (t) = 0 min. The reaction mixtures were heated to 160 °C in an oil bath and monitored for 70 minutes. Product formation was monitored by ^{19}F using 1-methoxy-3-(trifluoromethyl)benzene internal standard. Linear fits were obtained, removing the t=0 data point due to an observed induction period.

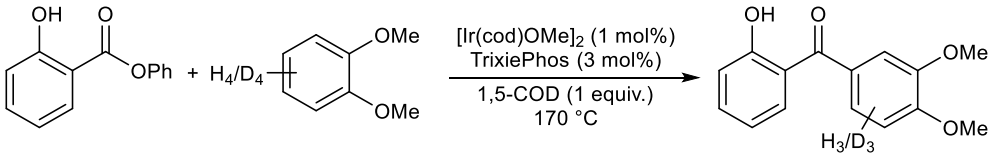
Deutero - Reactions were performed in triplicate using 0.7 mL aliquots from a stock solution containing phenyl 4-(trifluoromethyl)salicylate (0.4 mmol, 1 equiv), $[Ir(cod)OMe]_2$ (0.004 mmol, 1 mol%), TrixiePhos (0.012 mmol, 3 mol%), 1,5-COD (0.4 mmol, 1 equiv), 1-methoxy-3-(trifluoromethyl)benzene (0.407 mmol, 1.02 equiv) and D_4-1,2-dimethoxybenzene (4 mL) in a J-Young NMR tube. Once sealed, the tubes were removed from the glovebox, and single-scan ^1H and ^{19}F NMR spectra were taken for time (t) = 0 min. The reaction mixtures were heated to 160 °C in an oil bath and monitored for 90 minutes.

Product formation was monitored by ^{19}F using 1-methoxy-3-(trifluoromethyl)benzene internal standard. Linear fits were obtained, removing the t=0 data point due to an observed induction period.

Table S9. Initial rates kinetics results

Entry	k_H (%/min)	k_D (%/min)	KIE
1	0.360	0.198	–
2	0.380	0.220	–
3	0.348	0.201	–
Average	0.363 ± 0.012	0.206 ± 0.016	1.77 ± 0.13

Intermolecular Competition



Reactions were prepared in triplicate using phenyl salicylate (1 equiv), $[Ir(cod)OMe]_2$ (1 mol%), TrixiePhos (3 mol%), 1,5-COD (1 equiv), H_4-1,2-dimethoxybenzene (0.5 mL) and D_4-1,2-dimethoxybenzene (92.8% d_4 by ^1H NMR, 0.5 mL) in PTFE-lined crimp top vials. The vials were sealed, removed from the glovebox, and heated to 170 °C in an oil bath for 20 hr. Crude mixtures were analyzed by ESI-MS.

$$Prod_H = I_{257}$$

$$Prod_D = I_{260}$$

Intramolecular Competition

Reactions were prepared in triplicate using phenyl salicylate (1 equiv), $[Ir(cod)OMe]_2$ (1 mol%), TrixiePhos (3 mol%), 1,5-COD (1 equiv), 4-D_1-1,2-dimethoxybenzene (90.4% d_4 by ^1H NMR, 1 mL) in PTFE-lined crimp top vials. The vials were sealed, removed from the glovebox, and heated to 170 °C in an oil bath for 20 hr. Crude mixtures were analyzed by ESI-MS. Correction factors are used to account for ^{13}C of products.

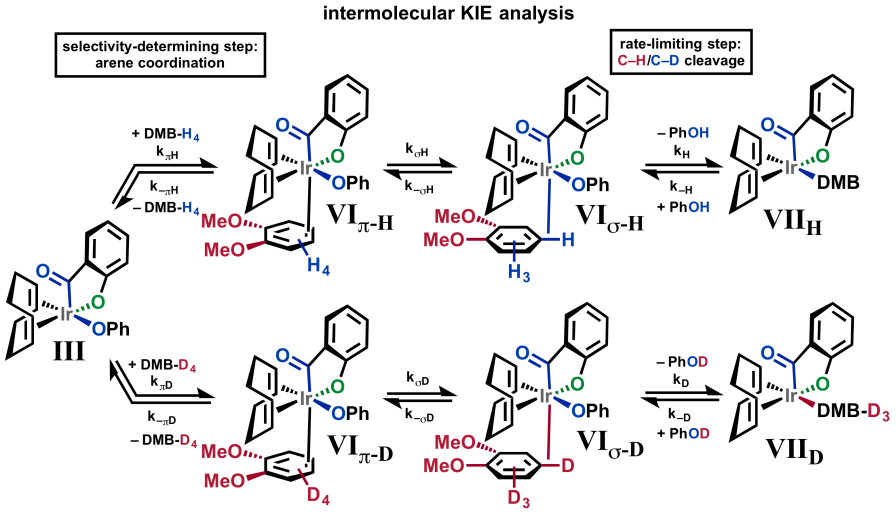
$$k_H = I_{259} + (I_{258} - (I_{257} \times 0.16))$$

$$k_D = I_{257} \times 1.16$$

Table S10. Product ratios and KIE values from inter- and intramolecular competition experiments

Entry	$Prod_H/k_H$	$Prod_D/k_D$	KIE
Inter1	3332	704	4.73
Inter2	4546	983	4.62
Inter3	5577	1259	4.43
Average			4.59 ± 0.15
Intra1	1057	687	1.54
Intra2	770	457	1.68
Intra3	1437	843	1.70
Average			1.64 ± 0.09

Cumulative effect of secondary isotope effects and differential reversibility on observed intermolecular KIE



Rate with respect to the formation of $[VII_H]$ is

$$\frac{\Delta[VII_H]}{\Delta t} = k_H[VI_{\sigma H}] - k_{-H}[VII_H] \tag{1}$$

Steady-state approximation on $[VI_{\sigma H}]$

$$0 = \frac{\Delta[VI_{\sigma H}]}{\Delta t} = k_{\sigma H}[VI_{\pi H}] + k_{-H}[VII_H] - k_{-\sigma H}[VI_{\sigma H}] - k_H[VI_{\sigma H}] \tag{2}$$

$$[VI_{\sigma H}] = \frac{k_{\sigma H}[VI_{\pi H}] + k_{-H}[VII_H]}{k_{-\sigma H} + k_H} \tag{3}$$

Substitute Equation 3 into Equation 1

$$\frac{\Delta[VII_H]}{\Delta t} = \frac{k_H k_{\sigma H}[VI_{\pi H}] - k_{-H}k_{-\sigma H}[VII_H]}{k_{-\sigma H}k_H} \tag{4}$$

Steady state approximation on $[VI_{\pi H}]$

$$0 = \frac{\Delta[VI_{\pi H}]}{\Delta t} = k_{\pi H}[III][DMB - H_4] + k_{-\sigma H}[VI_{\sigma H}] - k_{-\pi H}[VI_{\pi H}] - k_{\sigma H}[VI_{\pi H}] \tag{5}$$

Substitute Equation 3 into Equation 5

$$0 = \frac{k_{\sigma H}k_{-\sigma H}[VI_{\pi H}] + k_{-H}k_{-\sigma H}[VII_H]}{k_{-\sigma H} + k_H} + k_{\pi H}[III][DMB - H_4] - k_{-\pi H}[VI_{\pi H}] - k_{\sigma H}[VI_{\pi H}] \tag{6}$$

$$[VI_{\pi H}] = \frac{K_{-\sigma H}k_{\pi H}[III][DMB - H_4] + k_H k_{\pi H}[III][DMB - H_4] + k_{-H}k_{-\sigma H}[VII_H]}{k_{-\sigma H}k_{-\pi H} + k_H k_{-\pi H} + k_H k_{-\sigma H}} \tag{7}$$

Substitute Equation 7 into Equation 4

$$\frac{\Delta[VII_H]}{\Delta t} = \frac{k_H k_{\sigma H}k_{\pi H}[III][DMB - H_4](k_{-\sigma H} + k_H) + k_{-H}k_{-\sigma H}k_{-\pi H}[VII_H](k_{-\sigma H} - k_H)}{k_H^2 k_{\sigma H} + k_H^2 k_{-\pi H} + 2k_H k_{-\sigma H}k_{-\pi H} + k_H k_{\sigma H}k_{-\sigma H} + k_{-\sigma H}^2 k_{-\pi H}} \tag{8}$$

The second term in numerator should be negligible relative to the first term, so Equation 8 simplifies to

$$\frac{\Delta[VII_H]}{\Delta t} = \frac{k_H k_{\sigma H}k_{\pi H}[III][DMB - H_4](k_{-\sigma H} + k_H)}{k_H^2 k_{\sigma H} + k_H^2 k_{-\pi H} + 2k_H k_{-\sigma H}k_{-\pi H} + k_H k_{\sigma H}k_{-\sigma H} + k_{-\sigma H}^2 k_{-\pi H}} \tag{9}$$

Therefore

$$\frac{\Delta[VII_D]}{\Delta t} = \frac{k_D k_{\sigma D}k_{\pi D}[III][DMB - D_4](k_{-\sigma D} + k_D)}{k_D^2 k_{\sigma D} + k_D^2 k_{-\pi D} + 2k_D k_{-\sigma D}k_{-\pi D} + k_D k_{\sigma D}k_{-\sigma D} + k_{-\sigma D}^2 k_{-\pi D}} \tag{10}$$

$KIE_{intermolecular}$ can be described as $\frac{\frac{\Delta[VII_H]}{\Delta t}}{\frac{\Delta[VII_D]}{\Delta t}}$ so

$$\frac{\frac{\Delta[VII_H]}{\Delta t}}{\frac{\Delta[VII_D]}{\Delta t}} = \frac{\frac{k_H k_{\sigma H} k_{\pi H}[III][DMB-H_4](k_{-\sigma H}+k_H)}{k_D k_{\sigma D} k_{\pi D}[III][DMB-D_4](k_{-\sigma D}+k_D)}}{\frac{k_D^2 k_{\sigma D}+k_D^2 k_{-\pi D}+2k_D k_{-\sigma D} k_{-\pi D}+k_D k_{\sigma D} k_{-\sigma D}+k_{-\sigma D}^2 k_{-\pi D}}{k_H^2 k_{\sigma H}+k_H^2 k_{-\pi H}+2k_H k_{-\sigma H} k_{-\pi H}+k_H k_{\sigma H} k_{-\sigma H}+k_{-\sigma H}^2 k_{-\pi H}}} \tag{11}$$

Because $[DMB-H_4]_i = [DMB-D_4]_i$ and $\Delta[DMB] << [DMB]_i$, $[DMB-H_4] = [DMB-D_4]$

$$\frac{\frac{\Delta[VII_H]}{\Delta t}}{\frac{\Delta[VII_D]}{\Delta t}} = \left(\frac{k_H}{k_D}\right) \frac{k_{\sigma H} k_{\pi H}}{k_{\sigma D} k_{\pi D}} \frac{k_{-\sigma H}+k_H}{k_{-\sigma D}+k_D} \frac{k_D^2 k_{\sigma D}+k_D^2 k_{-\pi D}+2k_D k_{-\sigma D} k_{-\pi D}+k_D k_{\sigma D} k_{-\sigma D}+k_{-\sigma D}^2 k_{-\pi D}}{k_H^2 k_{\sigma H}+k_H^2 k_{-\pi H}+2k_H k_{-\sigma H} k_{-\pi H}+k_H k_{\sigma H} k_{-\sigma H}+k_{-\sigma H}^2 k_{-\pi H}} \tag{12}$$

ARENE SUBSTRATE SCOPE

Although we used [Ir(cod)OPh]$_2$ during our optimization study, we found that commercially available [Ir(cod)OMe]$_2$ was more convenient in our substrate scope study.

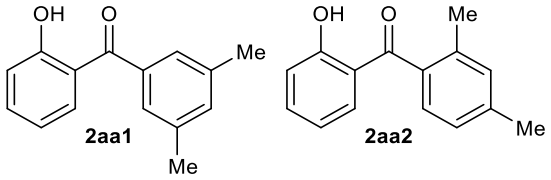
Procedure A. TrixiePhos (10 mg, 0.024 mmol, 3 mol%), [Ir(cod)OMe]$_2$ (5 mg, 0.008 mmol, 1 mol%), phenyl salicylate (0.171 g, 0.8 mmol, 1 equiv), 1,5-COD (98 μL, 0.8 mmol, 1 equiv), and arene (8 mL, 0.1 M) were added to a 10 mL PTFE-lined crimp-top vial. The vial was sealed, removed from the glovebox, and heated to 170 °C in an oil bath for 20 h. The solvent was removed *in vacuo*. Oligomer and residual starting material were hydrolyzed (see general procedure below), and the aqueous layer was extracted with ethyl acetate (3 × 12 mL). The organic extracts were dried over Na_2SO_4 and concentrated. The crude product was purified by column chromatography.

Procedure B. TrixiePhos (14 mg, 0.036 mmol, 3 mol%), [Ir(cod)OMe]$_2$ (8 mg, 0.012 mmol, 1 mol%), phenyl salicylate (0.257 g, 1.2 mmol, 1 equiv), 1,5-cyclooctadiene (1,5-COD, 147 μL, 1.2 mmol, 1 equiv), and arene (12 mL, 0.1 M) were added to a dry 15 mL pressure tube. The pressure tube was sealed, removed from the glovebox, and heated to 170 °C in an oil bath for 20 h. The solvent was removed *in vacuo*. Oligomer and residual starting material were hydrolyzed (see general procedure below), and the aqueous layer was extracted with ethyl acetate (3 × 15 mL). The organic extracts were dried over Na_2SO_4 and concentrated. The crude product was purified by column chromatography.

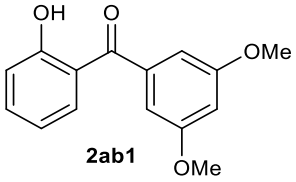
Procedure C. Followed procedure B, then after 20 hr, the reaction mixture was cooled to rt, brought into the glovebox, where TrixiePhos (14 mg, 0.036 mmol, 3 mol%) and [Ir(cod)OMe]$_2$ (8 mg, 0.012 mmol, 1 mol%) was added. The pressure tube was sealed, removed from the glovebox, and heated to 170 °C in an oil bath for 20 h. The solvent was removed *in vacuo*. Oligomer and residual starting material were hydrolyzed (see general procedure below), and the aqueous layer was extracted with ethyl acetate (3 × 15 mL). The organic extracts were dried over Na_2SO_4 and concentrated. The crude product was purified by column chromatography.

Procedure D. Followed procedure A, then after 20 h, the reaction mixture was cooled to rt, brought into the glovebox, and TrixiePhos (10 mg, 0.024 mmol, 3 mol%) and [Ir(cod)OMe]$_2$ (5 mg, 0.008 mmol, 1 mol%) was added. The vial was sealed, removed from the glovebox, and heated to 170 °C for 20 h. The solvent was removed *in vacuo*. Oligomer and residual starting material were hydrolyzed (see general procedure below), and the aqueous layer was extracted with ethyl acetate (3 × 12 mL). The organic extracts were dried over Na_2SO_4 and concentrated. The crude product was purified by column chromatography.

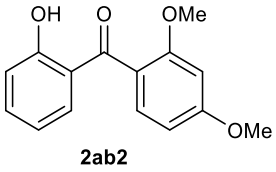
General procedure for hydrolysis. Crude material was dissolved in toluene (10 mL), NaOH (30%, 5 mL for procedure A/C, 6.5 mL for procedure B/D) was added, and the mixture was allowed to stir overnight. The mixture was cooled to 0 °C, and HCl (6 M, 7 mL for Procedure A/C, 8 mL for Procedure B or D) was added dropwise.



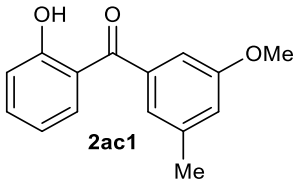
(3,5-dimethylphenyl)(2-hydroxyphenyl)methanone (**2aa1**), (2,4-dimethylphenyl)(2-hydroxyphenyl)methanone (**2aa2**). Prepared using procedure A. Brown oil (0.110 g, 0.487 mmol, 61%) as an inseparable mixture of regioisomers (11:1 **2aa1**:**2aa2**). R_f = 0.31 (5% ethyl acetate in hexanes). ^{1}H NMR (400 MHz, $CDCl_3$) **2aa1**: δ 12.07 (s, 1H), 7.60 (dd, *J* = 8.0, 1.6 Hz, 1H), 7.50 (ddd, *J* = 8.4, 6.8, 1.6 Hz, 1H), 7.27 (br s, 2H), 7.22 (br s, 1H), 7.06 (dd, *J* = 8.4, 0.8 Hz, 1H), 6.87 (dd, *J* = 8.0, 7.2, 1.2 Hz, 1H), 2.39 (s, 6H); **2aa2**: δ 12.28 (s, 1H), 7.33 (dd, *J* = 8.0, 1.6 Hz, 1H), 7.18 (d, *J* = 8.0 Hz, 1H), 7.13–7.08 (m, 2H), 6.80 (ddd, *J* = 8.4, 7.6, 1.6 Hz, 1H), 2.28 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$, only **2aa1** observed) δ 202.3, 163.3, 138.2, 138.1, 136.3, 133.8, 133.7, 127.0, 119.4, 118.7, 118.5, 21.4. IR (neat) 1626 cm^{-1}. HRMS (ESI) calcd for $C_{15}H_{13}O_2$ $[M-H]^-$ *m/z* 225.0921; found 225.0927.



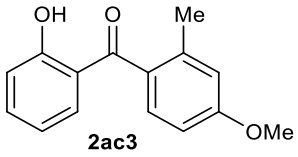
(3,5-dimethoxyphenyl)(2-hydroxyphenyl)methanone (**2ab1**). Prepared using procedure C with no hydrolysis in the same reaction as **2ab2**; isolated by column chromatography. Brown oil (0.099 g, 0.384 mmol, 48%). R_f = 0.33 (5% ethyl acetate in hexanes with 1% acetic acid additive). ^{1}H NMR (400 MHz, $CDCl_3$) δ 11.96 (s, 1H), 7.64 (dd, *J* = 8.0, 1.6 Hz, 1H), 7.49 (ddd, *J* = 8.8, 7.2, 2.0 Hz, 1H), 7.05 (dd, *J* = 8.4, 0.8 Hz), 6.86 (ddd, *J* = 8.0, 7.2, 1.2 Hz, 1H), 6.78 (d, *J* = 2.4 Hz, 2H), 6.65 (t, *J* = 2.4 Hz, 1H), 3.82 (s, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 201.5, 163.3, 160.7, 139.8, 136.6, 133.7, 119.2, 118.8, 118.5, 107.1, 104.1, 55.7. IR (neat) 1626 cm^{-1}. HRMS (ESI) calcd for $C_{15}H_{13}O_4$ $[M-H]^-$ *m/z* 257.0819; found 257.0826.



(2,4-dimethoxyphenyl)(2-hydroxyphenyl)methanone (**2ab2**). Prepared using procedure C with no hydrolysis in the same reaction as **2ab1**; isolated by column chromatography. Brown oil (0.066 g, 0.256 mmol, 32%). R_f = 0.22 (5% ethyl acetate in hexanes with 1% acetic acid additive). ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.22 (s, 1H), 7.45 (ddd, *J* = 8.7, 7.2, 1.6 Hz, 1H), 7.40 (dd, *J* = 8.0, 1.6 Hz, 1H), 7.28 (d, *J* = 8.4 H, 1H), 7.01 (dd, *J* = 8.4, 1.2 Hz, 1H), 6.80 (ddd, *J* = 8.0, 7.2, 1.2 Hz, 1H), 6.56 (dd, *J* = 8, 2 Hz, 1H), 6.54 (d, *J* = 2.4 Hz, 1H), 3.87 (s, 3H), 3.75 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 201.3, 163.3, 163.0, 158.7, 136.2, 133.9, 131.1, 120.9, 120.6, 118.6, 118.1, 104.7, 99.0, 55.8, 55.7. IR (thin film, CH_2Cl_2) 1606 cm^{-1}. HRMS (ESI) calcd for $C_{15}H_{13}O_4$ $[M-H]^-$ *m/z* 257.0819; found 227.0832.

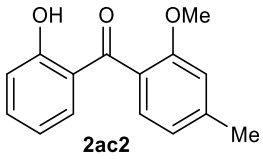


2ac1



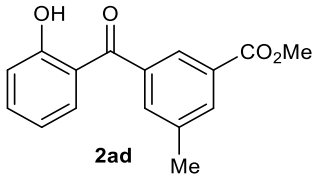
2ac3

(3-methoxy-5-methylphenyl)(2-hydroxyphenyl)methanone (**2ac1**), (4-methoxy-2-methylphenyl)(2-hydroxyphenyl)methanone (**2ac3**). Prepared using procedure A in the same reaction as **2ac2**; isolated by column chromatography as an inseparable mixture of isomers (9:1 **2ac1**: **2ac3**). Yellow oil (0.109 mg, 0.450 mmol, 57%) R_f = 0.42 (10% ethyl acetate in hexanes). ^{1}H NMR (400 MHz, $CDCl_3$) **2ac1**: δ 12.01 (s, 1H), 7.62 (dd, *J* = 8.0, 1.6 Hz, 1H), 7.51 (ddd, *J* = 8.4, 7.2, 1.6 Hz, 1H), 7.08–7.04 (m, 2H), 7.00–6.98 (m, 1H), 6.95–6.93 (m, 1H), 6.87 (ddd, *J* = 8.0, 7.2, 1.2 Hz, 1H), 3.84 (s, 3H), 2.40 (s, 3H); **2ac3**: δ 12.27 (s, 1H), 7.37 (dd, *J* = 8.0, 1.8 Hz, 1H), 6.84–6.76 (m, 3H), 3.86 (s, 3H), 2.34 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$, only **2ac1** observed) δ 201.8, 163.3, 159.6, 139.9, 139.2, 136.5, 133.8, 122.5, 119.3, 118.81, 118.76, 118.5, 111.3, 55.6, 21.7. IR (thin film, CH_2Cl_2) 1627 cm^{-1}. HRMS (ESI) calcd for $C_{15}H_{14}O_3$ $[M-H]^-$ *m/z* 241.0870; found 241.0870.



2ac2

(2-methoxy-4-methylphenyl)(2-hydroxyphenyl)methanone (**2ac2**). Prepared using procedure A in the same reaction as **2ac1** and **2ac3**; isolated by column chromatography. Yellow solid (0.027 g, 0.11 mmol, 14%). mp = 97–99 °C. R_f = 0.31 (10% ethyl acetate in hexanes). ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.21 (s, 1H), 7.45 (ddd, *J* = 8.8, 7.2, 1.6 Hz, 1H), 7.36 (dd, *J* = 8.0, 1.6 Hz, 1H), 7.19 (d, *J* = 7.6 Hz, 1H), 7.02 (dd, *J* = 8.4, 9.2 Hz, 1H), 6.86 (d, *J* = 7.6 Hz, 1H), 6.82 (s, 1H), 6.79 (t, *J* = 8.0 Hz, 1H), 3.76 (s, 3H), 2.43 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 202.3, 163.0, 156.9, 142.8, 136.4, 134.0, 129.2, 125.2, 121.3, 120.5, 118.7, 118.1, 112.4, 55.7, 22.0. IR (thin film, CH_2Cl_2) 1626 cm^{-1}. HRMS (ESI) calcd for $C_{15}H_{14}O_3$ $[M-H]^-$ *m/z* 241.0870; found 241.0875.

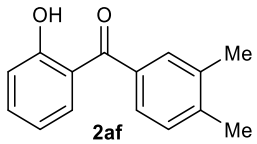


2ad

Methyl 5-(2-hydroxybenzoyl)-3-methylbenzoate (**2ad**)[2]. Prepared using procedure A with no hydrolysis. Brown oil (0.086 g, 0.321 mmol, 40%). R_f = 0.24 (10% diethyl ether in hexanes). ^{1}H NMR (400 MHz, $CDCl_3$) δ 11.94 (s, 1H), 8.12 (br s, 1H), 8.08 (br s, 1H), 7.68 (br s, 1H), 7.53 (ddd, *J* = 8, 6.4, 1.6 Hz, 2H), 7.09 (dd, *J* = 8.8, 1.2 Hz), 6.95–6.85 (m, 2H), 3.94 (s, 3H), 2.50 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 201.0, 166.5, 163.4, 139.1, 138.4, 136.8, 133.8, 133.5, 133.5, 130.4, 127.6, 119.1, 119.0, 118.7, 52.5,

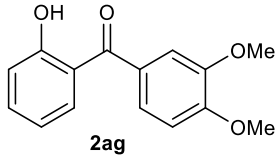
[2] Arene prepared following procedure detailed in *Bioorganic & Medicinal Chemistry* **2006**, 14, 6106-6119

21.4. IR (thin film, CH_2Cl_2) 1725, 1628 cm^{-1}. HRMS (ESI) calcd for $C_{16}H_{13}O_4$ $[M-H]^-$ *m/z* 269.0819; found 269.0830.



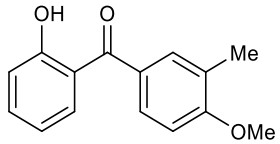
2af

(3,4-dimethylphenyl)(2-hydroxyphenyl)methanone (**2af**). Prepared using procedure C. Yellow oil (0.143 g, 0.630 mmol, 79%). mp = 74–77 °C. R_f = 0.27 (4% ethyl acetate in hexanes). ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.06 (s, 1H), 7.63 (dd, *J* = 8.0, 1.6 Hz, 1H), 7.52–7.47 (m, 2H), 7.42 (dd, *J* = 8.0, 2.0 Hz, 1H), 7.06 (dd, *J* = 8.4, 0.8 Hz, 1H), 6.87 (ddd, *J* = 8.4, 7.6, 2.0, 1H), 7.26 (d, *J* = 7.3 Hz, 1H), 2.36 (s, 3H), 2.34 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 201.5, 163.1, 141.4, 136.9, 136.0, 135.6, 133.6, 130.4, 129.5, 127.1, 119.4, 118.5, 118.3, 20.0, 19.8. IR (thin film, CH_2Cl_2) 1626 cm^{-1}. HRMS (ESI) calcd for $C_{15}H_{13}O_2$ $[M-H]^-$ *m/z* 225.0921; found 225.0921.



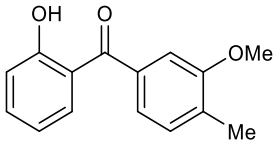
2ag

(3,4-dimethoxyphenyl)(2-hydroxyphenyl)methanone (**2ag**). Prepared using procedure A with no hydrolysis. Brown solid (0.192 g, 0.74 mmol, 93%). mp = 67–68 °C. R_f = 0.29 (20% ethyl acetate in hexanes). ^{1}H NMR (400 MHz, $CDCl_3$) δ 11.88 (s, 1H), 7.67 (dd, *J* = 8.0, 1.6 Hz, 1H), 7.50 (ddd, *J* = 8.4, 7.2, 1.6 Hz, 1H), 7.33 (dd, *J* = 8.4, 2.0 Hz, 1H), 7.31 (d, *J* = 2.0 Hz, 1H), 7.07 (dd, *J* = 8.4, 0.9 Hz, 1H), 6.95 (d, *J* = 8.2 Hz, 1H), 6.89 (ddd, *J* = 8.2, 7.3, 1.1 Hz, 1H), 3.98 (s, 3H), 3.94 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 200.1, 163.0, 152.8, 149.1, 136.0, 133.4, 130.5, 124.4, 119.5, 118.6, 118.5, 112.2, 110.1, 56.3, 56.2. IR (thin film, CH_2Cl_2) 1624 cm^{-1}. HRMS (ESI) calcd for $C_{15}H_{13}O_4$ $[M-H]^-$ *m/z* 257.0819; found 257.0829.



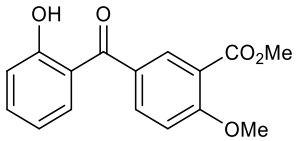
2ah1

2-hydroxyphenyl)(4-methoxy-3-methylphenyl)methanone (**2ah1**). Prepared using procedure A in the same reaction as **2ah2**; isolated by column chromatography. Yellow oil (0.098 g, 0.404 mmol, 50%). R_f = 0.21 (4% ethyl acetate in hexanes). ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.00 (s, 1H), 7.66 (dd, *J* = 8.0, 1.6 Hz, 1H), 7.50 (ddd, *J* = 8.8, 7.6, 1.6 Hz, 1H), 7.24 (dd, *J* = 8.0, 0.8 Hz, 1H), 7.19 – 7.16 (m, 2H), 7.07 (dd, *J* = 8.4, 0.8 Hz, 1H), 6.88 (ddd, *J* = 8.4, 7.2, 1.2 Hz, 1H), 3.89 (s, 3H), 2.31 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 201.4, 163.2, 157.9, 136.7, 136.2, 133.7, 131.9, 130.2, 122.2, 119.4, 118.7, 118.5, 110.3, 55.6, 16.6. IR (neat) 1629 cm^{-1}. HRMS (ESI) calcd for $C_{16}H_{15}O_5$ $[M-H]^-$ *m/z* 241.0870; found 241.0865.



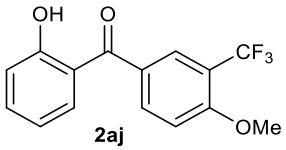
2ah2

(2-hydroxyphenyl)(3-methoxy-4-methylphenyl)methanone (**2ah2**). Prepared using procedure A in the same reaction as **2ah1**; isolated by column chromatography. Yellow solid (0.039 g, 0.16 mmol, 20%). mp = 75-76 °C. R_f = 0.16 (4% ethyl acetate in hexanes). ^{1}H NMR (400 MHz, $CDCl_3$) δ 11.99 (s, 1H), 7.65 (dd, *J* = 8, 1.6 Hz, 1H), 7.61 – 7.55 (m, 2H), 7.49 (ddd, *J* = 8.8, 7.2, 1.6 Hz, 1H), 7.06 (dd, *J* = 8.4, 0.8 Hz, 1H), 6.90 (d, *J* = 8.4 Hz, 1H), 6.88 (ddd, *J* = 8.4, 7.2, 1.2 Hz, 1H), 3.92 (s, 3H), 2.28 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 200.3, 162.9, 161.2, 135.7, 133.4, 132.1, 129.9, 129.7, 127.0, 119.5, 118.4, 118.3, 109.1, 55.6, 16.3. IR (thin film, CH_2Cl_2) 1625 cm^{-1}. HRMS (ESI) calcd for $C_{16}H_{15}O_5$ $[M-H]^-$ *m/z* 241.0870; found 241.0877.



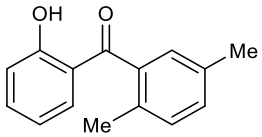
2ai

Methyl 5-(2-hydroxybenzoyl)-2-methoxybenzoate (**2ai**). Prepared using procedure A with no hydrolysis. Yellow solid (0.113 g, 0.394 mmol, 49%). mp = 117–119 °C. R_f = 0.07 (25% ethyl acetate in hexanes with 2% acetic acid additive). ^{1}H NMR (400 MHz, $CDCl_3$) δ 11.85 (s, 1H), 8.21 (d, *J* = 2.4 Hz), 7.89 (dd, *J* = 8.8, 2.4 Hz, 1H), 7.59 (dd, *J* = 8.0, 1.6 Hz, 1H), 7.52 (ddd, *J* = 8.4, 7.2, 1.6 Hz, 1H), 7.12–7.06 (m, 2H), 6.91 (ddd, *J* = 8.0, 7.2, 1.2 Hz, 1H), 4.01 (s, 3H), 3.91 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 199.3, 165.8, 163.1, 162.1, 136.3, 135.2, 133.7, 133.2, 129.9, 120.1, 119.2, 118.9, 118.6, 111.9, 56.5, 52.5. IR (thin film, CH_2Cl_2) 1731, 1626 cm^{-1}. HRMS (ESI) calcd for $C_{16}H_{13}O_5$ $[M-H]^-$ *m/z* 285.0768; found 285.0762.



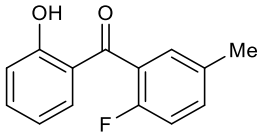
2aj

(2-hydroxyphenyl)(4-methoxy-3-(trifluoromethyl)phenyl)methanone (**2aj**). Prepared using procedure A light yellow solid (0.083 g, 0.280 mmol, 35%). mp = 68–70 °C. R_f = 0.34 (20% ethyl acetate in hexanes). ^{1}H NMR (400 MHz, $CDCl_3$) δ 11.79 (s, 1H), 7.99 (d, *J* = 1.9 Hz, 1H), 7.91 (dd, *J* = 8.6, 1.9 Hz, 1H), 7.59–7.49 (m, 2H), 7.12 (d, *J* = 8.7 Hz, 1H), 7.09 (d, *J* = 8.4 Hz, 1H), 6.91 (t, *J* = 7.6 Hz, 1H), 4.01 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 199.0, 163.2, 160.6, 136.5, 135.3, 133.0, 129.9, 129.2 (q, *J* = 5.3 Hz), 124.5, 121.8, 119.2 – 118.8 (m), 118.7, 111.7, 56.5. IR (thin film, CH_2Cl_2) 1632 cm^{-1}. HRMS (ESI) calcd for $C_{16}H_{16}O_4$ $[M-H]^-$ *m/z* 295.0588, found 295.0598.



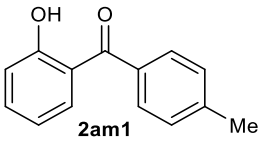
2ak

(2,5-dimethylphenyl)(2-hydroxyphenyl)methanone (**2ak**). Prepared using procedure C. Due to low yield, the product was unable to be fully purified, and is obtained with minor impurities. Brown oil (0.026 g, 0.114 mmol, 14%). R_f = 0.43 (10% ethyl acetate in hexanes). ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.26 (s, 1H), 7.49 (ddd, *J* = 8.8, 7.6, 1.6 Hz, 2H), 7.31 (dd, *J* = 8.0, 1.6 Hz, 1H), 7.21 (dd, *J* = 8.0, 2.0 Hz, 1H), 7.18 (d, *J* = 7.6 Hz, 1H), 7.07 (br s, 1H), 7.05 (dd, *J* = 8.0, 0.8 Hz, 1H), 2.35 (s, 3H), 2.24 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 204.8, 163.4, 137.9, 136.8, 135.1, 133.9, 132.4, 131.0, 130.9, 128.0, 120.1, 119.0, 118.4, 21.0, 19.2. IR (neat) 1629 cm^{-1}. HRMS (ESI) calcd for $C_{15}H_{13}O_2$ $[M-H]^-$ *m/z* 225.0921; found 225.0932.

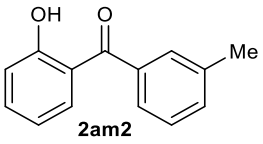


2al

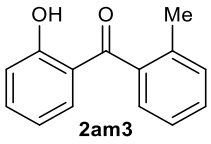
(2-fluoro-5-methylphenyl)(2-hydroxyphenyl)methanone (**2al**). Prepared using procedure C. Brown oil (0.024 g, 0.104 mmol, 9%). R_f = 0.47 (10% ethyl acetate in hexanes). ^{1}H NMR (400 MHz, $CDCl_3$) δ 11.98 (s, 1H), 7.51 (ddd, *J* = 8.8, 7.2, 1.6 Hz, 1H), 7.43 (ddd, *J* = 8.0, 2.8, 1.6 Hz, 1H), 7.32 (ddddd, *J* = 8.0, 7.2, 4.8, 2.4, 0.4 Hz, 1H), 7.25 (dd, *J* = 6.4, 2.0 Hz, 1H), 7.07 (t, *J* = 9.2 Hz, 1H), 7.05 (dd, *J* = 8.4, 0.8 Hz, 1H), 6.86 (ddd, *J* = 8.4, 7.2, 1.2 Hz, 1H), 2.38 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 199.0, 163.1, 157.2 (d, J_{C-F} = 247.3), 137.1, 134.2 (d, J_{C-F} = 3.7 Hz), 133.6 (d, J_{C-F} = 6.2 Hz), 133.5 (d, J_{C-F} = 12.0 Hz), 130.1 (d, J_{C-F} = 2.7 Hz), 126.1 (d, J_{C-F} = 15.9 Hz), 119.9, 119.2, 118.4, 116.1 (d, *J* = 21.5 Hz), 20.7. ^{19}F {^{1}H} NMR (377 MHz, $CDCl_3$) δ −117.56. IR (neat) 1629 cm^{-1}. HRMS (QTOF) calcd for $C_{14}H_{11}FO_2$ $[M-H]^-$ *m/z* 230.0738; found 230.0742. "Through-space" $^5J_{C-F}$ coupling has been reported in the literature.[3,4]



2am1



2am2

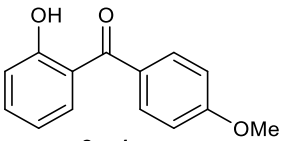


2am3

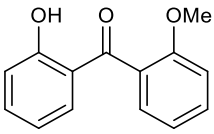
(2-hydroxyphenyl)(*o*-tolyl)methanone (**2am1**), (2-hydroxyphenyl)(*m*-tolyl)methanone (**2aa2**), (2-hydroxyphenyl)(*p*-tolyl)methanone (**2am3**). Prepared using procedure B. Yellow oil (0.128 g, 0.601 mmol, 50%) as an inseparable mixture of regioisomers (1:17:17 **2am1***:***2am2***:***2am3**). R_f = 0.44 (5% ethyl acetate in hexanes). ^{1}H NMR (500 MHz, $CDCl_3$) **2am1**: δ 12.03 (s, 1H), 2.45 (s, 3H); **2am2**: δ 12.05 (s, 1H), 7.31 (dd, *J* = 8.0, 0.5 Hz, 2H), 2.44 (s, 3H); **2am3**: δ 12.25 (s, 1H), 6.81 (ddd, *J* = 8.5, 8, 1 Hz, 1H),

[3] Jaime-Figueroa, S.; Kurz, L. J.; Liu, Y.; Cruz, R. *Spectrochim. Acta, Part A* **2000**, 56, 1167.
[4] Chen, J.; Reibenspies, J.; Derecskei-Kovacs, A.; Burgess, K. *Chem. Commun.* **1999**, 2501.

2.30 (s, 3H); Overlapping signals from **2am1** and **2am2**: δ 7.63–7.58 (m), 7.53–7.44 (m), 7.41–7.36 (m), 7.07 (m), 6.87 (m). ^{13}C NMR (126 MHz, $CDCl_3$, only **2am1** and **2am2** observed) δ 202.0, 201.5, 163.3, 163.2, 142.9, 138.4, 138.1, 136.4, 136.2, 135.3, 133.8, 133.7, 132.8, 129.7, 129.6, 129.2, 128.3, 126.5, 119.42, 119.36, 118.74, 118.68, 118.49, 118.48, 21.8, 21.5. IR (neat) 1626 cm^{-1}. HRMS (ESI) calcd for $C_{14}H_{11}O_2$ $[M-H]^-$ *m/z* 211.0765; found 211.0769.

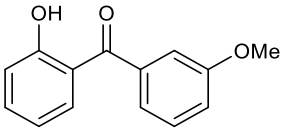


2an1



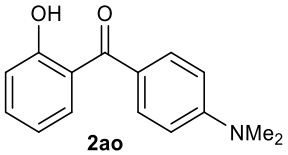
2an3

(2-hydroxyphenyl)(4-methoxyphenyl)methanone (**2an1**), (2-hydroxyphenyl)(2-methoxyphenyl)methanone (**2an3**). Prepared using procedure C in the same reaction as **2an2**; isolated by column chromatography as an inseparable mixture (10;1 **2an1**:**2an3**). Brown oil (0.164 g, 0.719 mmol, 60%) . R_f = 0.24 (8% diethyl ether in hexanes). ^{1}H NMR (400 MHz, $CDCl_3$) **2an1**: δ 11.96 (s, 1H), 7.74–7.70 (m, 2H), 7.63 (dd, *J* = 8.0, 2.0 Hz, 1H), 7.49 (ddd, *J* = 8.8, 7.2, 1.6 Hz, 1H), 7.06 (d, *J* = 8.4 Hz, 1H), 7.02–6.97 (m, 2H), 6.88 (ddd, *J* = 8.4, 7.2, 1.2 Hz), 3.90 (s, 3H); **2an3**: δ 12.17 (s, 1H), 7.33 (dd, *J* = 8.0, 1.7 Hz, 1H), 7.29 (dd, *J* = 7.5, 1.8 Hz, 1H), 6.80 (ddd, *J* = 8.1, 7.2, 1.1 Hz, 1H), 3.78 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$, only **2an1** observed) δ 200.2, 163.1, 136.0, 133.4, 132.0, 130.5, 119.5, 118.6, 118.5, 113.8, 55.7. IR (neat) 1625 cm^{-1}. HRMS (ESI) calcd for $C_{14}H_{11}O_3$ $[M-H]^-$ *m/z* 227.0717; found 227.0711.



2an2

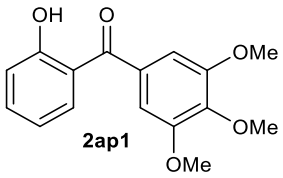
(2-hydroxyphenyl)(3-methoxyphenyl)methanone (**2an2**). Prepared using procedure C in the same reaction as **2an1**; isolated by column chromatography. Brown oil (0.038 g, 0.166 mmol, 14%). R_f = 0.32 (8% diethyl ether in hexanes). ^{1}H NMR (400 MHz, $CDCl_3$) δ 11.99 (s, 1H), 7.62 (dd, *J* = 8.0, 1.2 Hz, 1H), 7.51 (app t, *J* = 8.4 Hz, 1H), 7.41 (t, *J* = 8.0 Hz, 1H), 7.23 (d, *J* = 6.8 Hz, 1H), 7.20 (m, 1H) 7.12 (dd, *J* = 8.4, 2.4 Hz, 1H), 7.07 (d, *J* = 8.4 Hz, 1H), 6.87 (t, *J* = 8.0 Hz, 1H) 3.86 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 201.5, 163.4, 159.7, 139.3, 136.5, 133.7, 129.5, 121.8, 119.3, 118.8, 118.5, 118.1, 114.1, 55.6. IR (neat) 1626 cm^{-1}. HRMS (ESI) calcd for $C_{14}H_{11}O_3$ $[M-H]^-$ *m/z* 227.0717; found 227.0714.



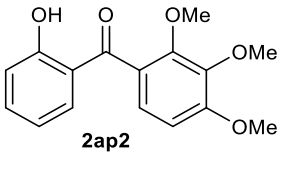
2ao

(4-(dimethylamino)phenyl)(2-hydroxyphenyl)methanone (**2ao**). Prepared using procedure C with no hydrolysis. Yellow solid (0.167 g, 0.692 mmol, 87%). mp = 58–60 °C. R_f = 0.33 (20% ethyl acetate in hexanes), 0.45 (3% ethyl acetate in benzene). ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.00 (s, 1H), 7.75–7.70 (m, 2H), 7.68 (dd, *J* = 8.0, 1.6 Hz, 1H), 7.45 (ddd, *J* = 8.4, 7.2, 1.6 Hz, 1H), 7.05 (dd, *J* = 8.4, 0.8 Hz, 1H), 6.87 (ddd, *J* = 8.0, 7.2, 1.2 Hz, 1H), 6.74–6.69 (m, 2H), 3.09 (s, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ

199.0, 162.7, 153.3, 135.1, 133.2, 132.5, 125.0, 120.1, 118.4, 118.2, 110.8, 40.2. IR (thin film, CH_2Cl_2) 1621 cm^{-1}. HRMS (ESI) calcd for $C_{15}H_{14}NO_2$ $[M-H]^-$ *m/z* 240.1030; found 240.1033.

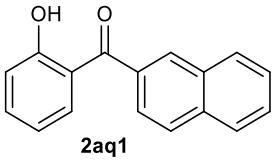


2ap1

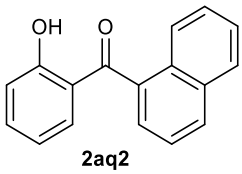


2ap2

(3,4,5-trimethoxyphenyl)(2-hydroxyphenyl)methanone (**2ap1**), (2,3,4-trimethoxy)(2-hydroxyphenyl)methanone (**2ap2**). Prepared using procedure A. Yellow oil (0.176 g, 0.610 mmol, 76%) as an inseparable mixture of isomers (5:1 **2ap1**:**2ap2**). R_f = 0.22 (15% ethyl acetate in hexanes). **2ap1**: ^{1}H NMR (500 MHz, $CDCl_3$) δ 11.87 (s, 1H), 7.67 (dd, *J* = 6.4, 1.2 Hz, 1H), 7.52 (ddd, *J* = 7.0, 5.7, 1.4 Hz, 1H), 7.09 (d, *J* = 6.7 Hz, 1H), 6.94 (s, 2H), 6.90 (t, *J* = 6.0 Hz, 1H), 3.95 (s, 3H), 3.90 (s, 6H); **2ap2**: δ 12.18 (s, 1H), 7.48 (ddd, *J* = 6.9, 5.7, 1.4 Hz, 1H), 7.41 (dd, *J* = 6.4, 1.2 Hz, 1H), 7.03 (d, *J* = 6.8 Hz, 2H), 6.83 (t, *J* = 5.9 Hz, 1H), 6.74 (d, *J* = 6.8 Hz, 1H), 3.93 (s, 3H), 3.92 (s, 3H), 3.83 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) **2ap1**: δ 200.7, 163.3, 153.1, 141.7, 136.4, 133.4, 133.1, 119.3, 118.6, 107.1, 61.2, 56.5; **2ap2**: δ 201.0, 163.1, 156.1, 151.9, 142.3, 136.6, 133.9, 125.7, 124.1, 120.4, 118.8, 118.2, 106.9, 62.1, 56.3. IR (neat) 1625 cm^{-1}. HRMS (ESI) calcd for $C_{16}H_{15}O_5$ $[M-H]^-$ *m/z* 287.0925; found 287.0931.

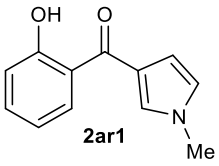


2aq1



2aq2

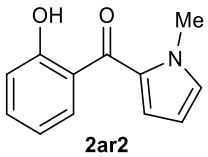
(2-hydroxyphenyl)(napthanlen-2-yl)methanone (**2aq1**), (2-hydroxyphenyl)(naphthalen-1-yl)methanone (**2aq2**). Prepared using procedure A. Yellow solid (0.150 g, 0.61 mmol, 78%) as an inseparable mixture of isomers (>20:1 ratio). mp = 80–82 °C. R_f = 0.19 (4% ethyl acetate in hexanes). ^{1}H NMR (400 MHz, $CDCl_3$) **2aq1**: δ 12.04 (s, 1H), 8.19 (br s, 1H), 7.99–7.91 (m, 3H), 7.78 (dd, *J* = 8.8, 2.0 Hz, 1H), 7.68 (dd, *J* = 8.0, 1.6 Hz, 1H), 7.66–7.56 (m, 2H), 7.54 (ddd, *J* = 8.8, 7.2, 1.6 Hz, 1H), 7.11 (dd, *J* = 8.4, 1.2 Hz, 1H), 6.90 (ddd, *J* = 8.0, 7.2, 1.2 Hz, 1H); **2aq2**: δ 12.34 (s, 1 H). ^{13}C NMR (101 MHz, $CDCl_3$, only **2aq1** observed) δ 201.6, 163.4, 136.5, 135.3, 135.0, 133.8, 132.3, 130.6, 129.3, 128.5, 128.4, 128.0, 127.2, 125.5, 119.5, 118.9, 118.6. IR (thin film, CH_2Cl_2) 1625 cm^{-1}. HRMS (ESI) calcd for $C_{15}H_{14}O_3$ $[M-H]^-$ *m/z* 247.0765; found 247.0767.



2ar1

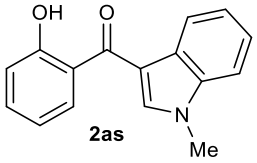
(2-hydroxyphenyl)(1-methyl-1*H*-pyrrol-3-yl)methanone (**2ar1**). Prepared using procedure B in the same reaction as **2ar2**; isolated by column chromatography. Brown oil (0.143 g, 0.71 mmol, 59%). R_f = 0.24 (20% ethyl acetate in hexanes). ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.22 (s, 1H), 7.94 (dd, *J* = 8.0, 1.6 Hz, 1H), 7.44 (ddd, *J* = 8.8, 7.2, 1.6 Hz, 1H), 7.28 (t, *J* = 2.0 Hz, 1H), 7.02 (dd, *J* = 8.0, 0.8 Hz, 1H), 6.90 (ddd, *J* = 8.0, 7.2, 1.2 Hz, 1H), 6.70–6.65 (m, 2H), 3.74 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 193.8,

162.5, 135.0, 131.9, 128.5, 123.8, 123.4, 120.7, 118.6, 118.2, 111.6, 36.9. IR (neat) 1621 cm^{-1}. HRMS (ESI) calcd for $C_{12}H_{11}NO_2$ $[M-H]^-$ *m/z* 200.0717; found 200.0720.



2ar2

(2-hydroxyphenyl)(1-methyl-1*H*-pyrrol-2-yl)methanone (**2ar2**).Prepared using procedure B in the same reaction as **2ar1**; isolated by column chromatography. Brown oil (0.018 g, 0.09 mmol, 7%). R_f = 0.48 (20% ethyl acetate in hexanes). ^{1}H NMR (400 MHz, $CDCl_3$) δ 11.76 (s, 1H), 7.88 (dd, *J* = 8.0, 1.6 Hz, 1H), 7.45 (ddd, *J* = 8.8, 7.6, 1.6 Hz, 1H), 7.01 (dd, *J* = 8.0, 0.8 Hz, 1H), 6.94 (t, *J* = 2.0 Hz, 1H), 6.89 (ddd, *J* = 8.0, 7.2, 1.2 Hz, 1H), 6.83 (dd, *J* = 4.0, 1.6 Hz, 1H), 6.20 (dd, *J* = 4.0, 2.4 Hz, 1H), 3.97 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 188.9, 162.2, 134.9, 132.3, 131.7, 129.7, 122.7, 120.7, 118.5, 118.0, 108.5, 37.1. IR (thin film, CH_2Cl_2) 1621 cm^{-1}. HRMS (ESI) calcd for $C_{12}H_{11}NO_2$ $[M-H]^-$ *m/z* 200.0717; found 200.0719.

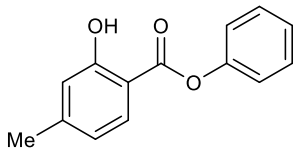


2as

(2-hydroxyphenyl)(1-methyl-1*H*-indol-3-yl)methanone (**2as**). Prepared using procedure A. Brown solid (0.146 g, 0.581 mmol, 73%). mp = 111–116 °C. R_f = 0.47 (50% ethyl acetate in hexanes). ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.03 (s, 1H), 8.33 – 8.26 (m, 1H), 7.85 (dd, *J* = 8.0, 9.6 Hz, 1H), 7.63 (s, 1H), 7.46 (ddd, *J* = 8.8, 7.6, 2.0 Hz), 7.40 – 7.31 (m, 3H), 7.05 (dd, *J* = 8.4, 1.2 Hz, 1H), 6.91 (ddd, *J* = 8.4, 7.6, 1.2 Hz, 1H), 3.86 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 193.6, 162.0, 137.5, 137.0, 134.7, 131.5, 127.4, 123.9, 122.9, 122.5, 121.5, 118.7, 118.2, 114.8, 109.9, 33.8. IR (thin film, CH_2Cl_2) 1679 cm^{-1}. HRMS (ESI) calcd for $C_{16}H_{13}NO_2$ $[M-H]^-$ *m/z* 250.0874; found 250.0873.

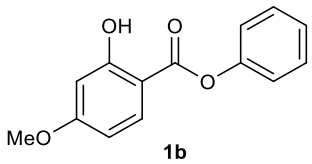
SALICYLATE ESTERS

General procedure for salicylate ester synthesis. Salicyclic acid (10 mmol, 1 equiv) and phenol (4.70 g, 50 mmol, 5 equiv) were massed in a 5 dram reaction vial, and then 10 mL of dry toluene was added. Phosphorus oxychloride (0.61g, 0.37 mL, 4 mmol, 0.4 equiv) was dripped in to the solution via syringe. A stir bar was then added and the reaction vial was sealed. The reaction mixture was then heated at 110 °C in an aluminum heating block on a hot plate fitted with a thermocouple for 16-18 h. The mixture was allowed to cool to room temperature, transferred to a separatory funnel, and quenched with saturated aqueous sodium carbonate solution (3 × 10 mL). The aqueous washes were then extracted with diethyl ether (3 × 10 mL). The organic layers were combined, dried with sodium sulfate, and concentrated *in vacuo*. The crude product was purified by column chromatography.



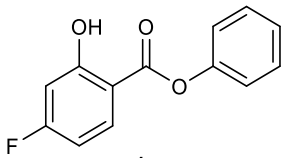
1a

Phenyl 2-hydroxy-4-methylbenzoate (**1a**). White solid (1.20 g, 5.30 mmol, 53%). mp = 43–44 °C. R_f = 0.33 (5% ethyl acetate in hexanes). ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.45 (s, 1H), 7.94 (d, *J* = 8.1 Hz, 1H), 7.49–7.39 (m, 2H), 7.34–7.25 (m, 1H), 7.21 (d, *J* = 1.3 Hz, 1H), 7.19 (dd, *J* = 2.1, 0.9 Hz, 1H), 2.38 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 169.1, 162.3, 150.3, 148.2, 130.3, 129.7, 126.4, 121.8, 120.9, 118.1, 109.4, 22.1. IR (neat) 1683 cm^{-1}. HRMS (ESI) calcd for $C_{14}H_{12}O_3$ $[M-H]^-$ *m/z* 227.0714, found 227.0724.



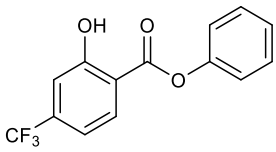
1b

Phenyl 2-hydroxy-4-methoxybenzoate (**1b**). Phenol (264 mg, 2.8 mmol, 1.4 equiv), 4-methoxysalicylic acid (336.3 mg, 2.0 mmol, 1 equiv), and DMAP (49 mg, 0.4 mmol, 0.2 equiv) were dissolved in methylene chloride (8 mL). DCC (743 mg, 3.6 mmol, 1.8 equiv) was added; the reaction mixture was heated to reflux and allowed to reflux overnight. Removal of the solvent and purification by column chromatography (2% ethyl acetate in hexanes to 3% ethyl acetate in hexanes) ultimately gave a white solid (0.358 g, 1.46 mmol, 73%). mp = 59–61 °C. R_f = 0.28 (5% ethyl acetate in hexanes). ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.72 (s, 1H), 7.97 (d, *J* = 8.8 Hz, 1H), 7.49–7.39 (m, 2H), 7.34–7.25 (m, 1H), 7.24–7.15 (m, 2H), 6.53 (dd, *J* = 8.8, 2.5 Hz, 1H), 6.50 (d, *J* = 2.4 Hz, 1H), 3.86 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.9, 166.4, 164.7, 150.4, 131.9, 129.7, 126.3, 121.9, 108.2, 105.0, 101.0, 55.7. IR (neat) 1671 cm^{-1}. HRMS (ESI) calcd for $C_{14}H_{12}O_4$ $[M-H]^-$ *m/z* 243.0663, found 243.0661.



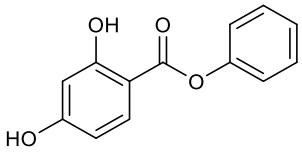
1c

Phenyl 4-fluoro-2-hydroxybenzoate (**1c**). White solid (1.20 g, 5.20 mmol 52%). mp = 58–60 °C. R_f = 0.32 (5% ethyl acetate in hexanes). ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.73 (d, *J* = 1.6 Hz, 1H), 8.09 (dd, *J* = 8.9, 6.5 Hz, 1H), 7.51–7.40 (m, 2H), 7.37–7.27 (m, 1H), 7.24–7.16 (m, 2H), 6.77–6.58 (m, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.4, 167.9 (d, J_{C-F} = 255.4 Hz), 164.5 (d, J_{C-F} = 14.3 Hz), 150.1, 132.8 (d, J_{C-F} = 11.5 Hz), 129.6, 126.6, 121.7, 108.8 (d, J_{C-F} = 2.4 Hz), 108.0 (d, J_{C-F} = 22.7 Hz), 104.8 (d, J_{C-F} = 24.4 Hz). ^{19}F {^{1}H} NMR (376 MHz, $CDCl_3$) δ −99.65. IR (neat) 1682 cm^{-1}. HRMS (ESI) calcd for $C_{13}H_9FO_3$ $[M-H]^-$ *m/z* 231.0463, found 231.0473.



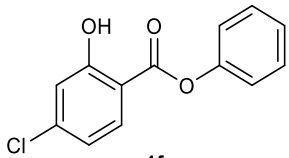
1d

Phenyl 2-hydroxy-4-(trifluoromethyl)benzoate (**1d**). White solid (1.24 g, 4.40 mmol, 44%). mp = 68–69 °C. R_f = 0.39 (5% ethyl acetate in hexanes). ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.63 (s, 1H), 8.20 (d, *J* = 8.3 Hz, 1H), 7.52–7.41 (m, 2H), 7.37–7.28 (m, 2H), 7.24–7.18 (m, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.2, 162.2, 150.0, 137.7 (q, $J_{C–F}$ = 32.9 Hz), 131.4, 129.9, 127.3, 123.2 (q, $J_{C–F}$ = 273.8 Hz), 121.5, 115.8 (q, $J_{C–F}$ = 3.6 Hz), 115.3 (q, $J_{C–F}$ = 4.0 Hz), 114.6. ^{19}F {^{1}H} NMR (376 MHz, $CDCl_3$) δ −63.85. IR (neat) 1686 cm^{-1}. HRMS (ESI) calcd for $C_{14}H_9F_3O_3$ $[M–H]^-$ *m/z* 281.0431, found 281.0436.



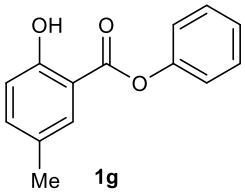
1e

Phenyl 2,4-dihydroxybenzoate (**1e**). White solid (0.65g, 2.80 mmol, 28%). mp = 144–145 °C. R_f = 0.28 (20% ethyl acetate in hexanes). ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.73 (s, 1H), 7.96 (d, *J* = 8.5 Hz, 1H), 7.44 (t, *J* = 7.9 Hz, 2H), 7.29 (t, *J* = 7.4 Hz, 1H), 7.19 (d, *J* = 7.4 Hz, 1H), 6.47–6.38 (m, 2H), 5.78 (s, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.9, 164.3, 162.8, 150.2, 132.6, 129.8, 126.5, 121.8, 108.5, 105.4, 103.5, 77.5, 77.2, 76.8. IR (neat) 1658 cm^{-1}. HRMS (ESI) calcd for $C_{13}H_{10}O_4$ $[M–H]^-$ *m/z* 229.0506, found 229.0510 (avg of 6).



1f

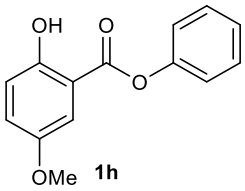
Phenyl 4-chloro-2-hydroxybenzoate (**1f**). White solid (1.47g, 5.9 mmol, 59%). mp = 52–53 °C. R_f = 0.40 (5% ethyl acetate in hexanes). ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.60 (s, 1H), 8.00 (d, *J* = 8.6 Hz, 1H), 7.50–7.40 (m, 2H), 7.36–7.27 (m, 1H), 7.24–7.16 (m, 2H), 7.06 (d, *J* = 2.0 Hz, 1H), 6.95 (dd, *J* = 8.6, 2.0 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.5, 162.8, 150.1, 142.5, 131.5, 129.8, 126.7, 121.7, 120.4, 118.2, 110.6. IR (neat) 1673 cm^{-1}. HRMS (ESI) calcd for $C_{13}H_9ClO_3$ $[M–H]^-$ *m/z* 247.0167, found 247.0161.



1g

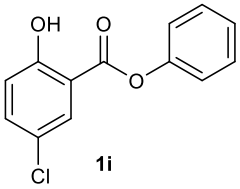
Phenyl 2-hydroxy-5-methylbenzoate (**1g**). White solid (1.61 g, 7.70 mmol, 77%). mp = 87–89 °C. R_f = 0.36 (5% ethyl acetate in hexanes). ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.31 (s, 1H), 7.86 (d, *J* = 2.2 Hz, 1H),

7.50–7.39 (m, 2H), 7.34 (dd, *J* = 8.5, 2.3 Hz, 1H), 7.35–7.26 (m, 1H), 7.24–7.16 (m, 3H), 6.94 (d, *J* = 8.5 Hz, 1H), 2.34 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 169.1, 160.3, 150.3, 137.6, 130.1, 129.8, 128.8, 126.5, 121.8, 117.7, 111.5, 20.6. IR (neat) 1682 cm^{-1}. HRMS (ESI) calcd for $C_{14}H_{12}O_3$ $[M-H]^-$ *m/z* 227.0714, found 227.0707.



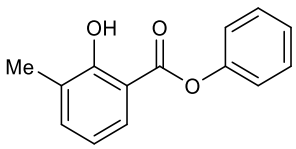
1h

Phenyl 2-hydroxy-5-methoxybenzoate (**1h**). White solid (0.75 g, 3.10 mmol, 31%). mp = 40–41 °C. R_f = 0.26 (5% ethyl acetate in hexanes). ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.13 (s, 1H), 7.51 (d, *J* = 3.1 Hz, 1H), 7.51–7.40 (m, 2H), 7.36–7.27 (m, 1H), 7.27–7.15 (m, 2H), 7.16 (dd, *J* = 9.1, 3.2 Hz, 1H), 6.98 (d, *J* = 9.1 Hz, 1H), 3.82 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.9, 156.9, 152.3, 150.2, 129.8, 126.6, 125.2, 121.8, 119.0, 112.1, 111.4, 77.5, 77.2, 76.8, 56.1. IR (neat) 1687 cm^{-1}. HRMS (ESI) calcd for $C_{14}H_{12}O_4$ $[M-H]^-$ *m/z* 243.0663, found 243.0653 (avg of 8).



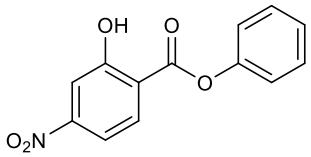
1i

Phenyl 5-chloro-2-hydroxybenzoate (**1i**). White solid (1.30 g, 5.20 mmol, 52%). mp = 94–96 °C. R_f = 0.39 (5% ethyl acetate in hexanes). ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.43 (s, 1H), 8.04 (d, *J* = 2.7 Hz, 1H), 7.52–7.41 (m, 3H), 7.37–7.27 (m, 1H), 7.22–7.18 (m, 2H), 6.99 (d, *J* = 8.9 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.1, 160.9, 150.0, 136.5, 129.8, 129.7, 126.7, 124.4, 121.6, 119.6, 112.9, 77.5, 77.2, 76.8. IR (neat) 1685 cm^{-1}. HRMS (ESI) calcd for $C_{13}H_9ClO_3$ $[M-H]^-$ *m/z* 247.0167, found 247.0179 (avg of 6).



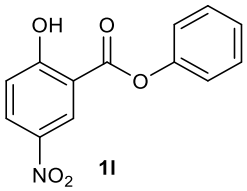
1j

Phenyl 2-hydroxy-3-methylbenzoate (**1j**). White solid (0.76 g, 3.30 mmol, 33%). mp = 145–145 °C. R_f = 0.45 (5% ethyl acetate in hexanes). ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.76 (s, 1H), 7.93 (dd, *J* = 8.0, 1.7 Hz, 1H), 7.49–7.40 (m, 2H), 7.39 (ddt, *J* = 7.4, 1.7, 0.8 Hz, 1H), 7.35–7.25 (m, 1H), 7.24–7.17 (m, 2H), 6.87 (t, *J* = 7.7 Hz, 1H), 2.30 (s, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 169.6, 160.8, 150.3, 137.4, 129.8, 128.0, 127.1, 126.5, 121.8, 118.9, 111.2, 15.8. IR (neat) 1685 cm^{-1}. HRMS (ESI) calcd for $C_{14}H_{12}O_3$ $[M-H]^-$ *m/z* 227.0714, found 227.0725 (avg of 6).



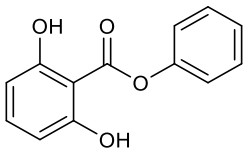
1k

Phenyl 2-hydroxy-4-nitrobenzoate (**1k**). White solid (3 mmol scale, 0.237 g, 0.90 mmol, 30 %). mp = 148–150 °C. R_f = 0.36 (5% ethyl acetate in hexanes). ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.73 (s, 1H), 8.27 (d, *J* = 8.7 Hz, 1H), 7.87 (s, 1H), 7.79 (d, *J* = 8.2 Hz, 1H), 7.49 (t, *J* = 7.8 Hz, 2H), 7.36 (t, *J* = 7.5 Hz, 1H), 7.23 (d, *J* = 7.9 Hz, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 167.8, 162.7, 152.7, 149.8, 131.9, 130.0, 127.1, 121.5, 116.8, 113.9, 113.4. IR (neat) 1686 cm^{-1}. HRMS (ESI) calcd for $C_{13}H_9NO_5$ $[M-H]^-$ *m/z* 258.0408, found 258.0420.



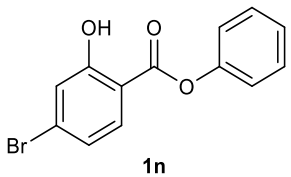
1l

Phenyl 2-hydroxy-5-nitrobenzoate (**1l**). White solid (1.558 g, 6.01 mmol 60%). mp = 148–150 °C. R_f = 0.18 (5% ethyl acetate in hexanes). ^{1}H NMR (400 MHz, $CDCl_3$) δ 11.17 (s, 1H), 9.04 (d, *J* = 2.8 Hz, 1H), 8.42 (dd, *J* = 9.2, 2.8 Hz, 1H), 7.54–7.44 (m, 2H), 7.41–7.31 (m, 1H), 7.28–7.20 (m, 2H), 7.16 (d, *J* = 9.2 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 167.9, 166.8, 149.8, 140.4, 131.3, 130.0, 127.2, 127.1, 121.5, 119.1, 111.9. IR (neat) 1693 cm^{-1}. HRMS (ESI) calcd for $C_{13}H_9NO_5$ $[M-H]^-$ *m/z* 258.0408, found 258.0418.



1m

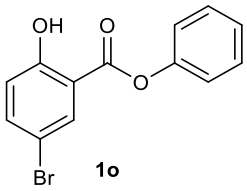
Phenyl 2,6-dihydroxybenzoate (**1m**). White solid (0.88 g, 3.80 mmol, 38%). mp = 91–94 °C. R_f = 0.36 (5% ethyl acetate in hexanes). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.57 (s, 2H), 7.54–7.44 (m, 2H), 7.44–7.33 (m, 2H), 7.27–7.18 (m, 2H), 6.56 (d, *J* = 8.3 Hz, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.6, 161.3, 148.6, 137.6, 130.1, 127.5, 122.1, 108.7, 99.8. IR (neat) 1683 cm^{-1}. HRMS (ESI) calcd for $C_{13}H_{10}O_4$ $[M-H]^-$ *m/z* 229.0506, found 229.0508 (avg of 11).



1n

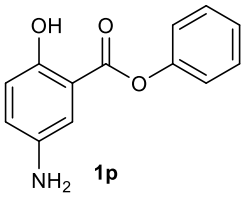
Phenyl 4-bromo-2-hydroxybenzoate (**1n**). Off-white solid (5 mmol scale, 1.16g, 3.95 mmol, 79%). mp = 69–72 °C. R_f = 0.38 (5% ethyl acetate in hexanes).^{1}H NMR (400 MHz, $CDCl_3$) δ 10.57 (s, 1H), 7.92 (d, *J*

= 8.5 Hz, 1H), 7.50–7.40 (m, 2H), 7.36–7.27 (m, 1H), 7.24 (d, *J* = 1.9 Hz, 1H), 7.24–7.16 (m, 2H), 7.11 (dd, *J* = 8.5, 1.9 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.7, 162.7, 150.0, 131.4, 131.0, 129.8, 126.7, 123.2, 121. 7, 121.3, 111.0. IR (neat) 1673 cm^{-1}. HRMS (ESI) calcd for $C_{13}H_9BrO_3$ $[M-H]^-$ *m/z* 290.9662, found 290.9667.



1o

Phenyl 5-bromo-2-hydroxybenzoate (**1o**). White solid (1.11 g, 3.80 mmol, 38%). mp = 94–96 °C. R_f = 0.36 (5% ethyl acetate in hexanes). ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.45 (s, 1H), 8.19 (d, *J* = 2.5 Hz, 1H), 7.61 (dd, *J* = 8.9, 2.6 Hz, 1H), 7.51–7.41 (m, 2H), 7.37–7.27 (m, 1H), 7.23–7.18 (m, 2H), 6.94 (d, *J* = 8.9 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.0, 161.3, 150.0, 139.3, 132.7, 129.9, 126.8, 121.6, 120.0, 113.5, 111.3, 77.5, 77.2, 76.8. IR (neat) 1694 cm^{-1}. HRMS (ESI) calcd for $C_{13}H_9BrO_3$ $[M-H]^-$ *m/z* 290.9662, found 290.9653.

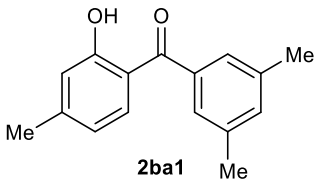


1p

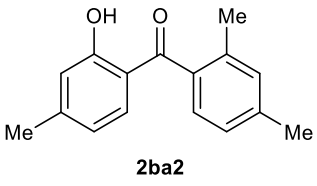
Phenyl 5-amino-2-hydroxybenzoate (**1p**). Brown solid (0.383 g, 1.70 mmol, 17%). mp = 72–73 °C. R_f = 0.28 (40% ethyl acetate in hexanes). ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.96 (s, 1H), 7.49–7.39 (m, 2H), 7.37 (d, *J* = 2.9 Hz, 1H), 7.34–7.25 (m, 1H), 7.24–7.15 (m, 2H), 6.94 (dd, *J* = 8.8, 2.8 Hz, 1H), 6.88 (d, *J* = 8.8 Hz, 1H), 3.52 (s, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.9, 155.6, 150.3, 138.7, 129.7, 126.4, 125.3, 121.8, 118.6, 114.9, 111.7, 77.5, 77.2, 76.8. IR (neat) 1678 cm^{-1}. HRMS (ESI) calcd for $C_{13}H_{11}NO_3$ $[M-H]^-$ *m/z* 228.0666, found 228.0679 (avg of 8).

SALICYLATE SUBSTRATE SCOPE

For all acylations of *m*-xylene, yield determined for the desired products based on relative integration in the ^{1}H NMR.



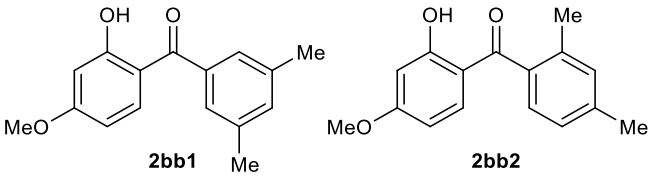
2ba1



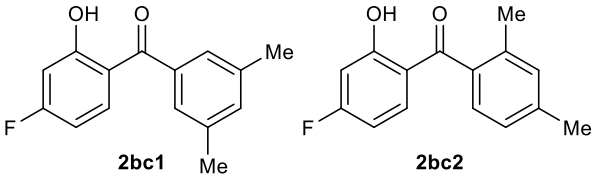
2ba2

(3,5-dimethylphenyl)(2-hydroxy-4-methylphenyl)methanone (**2ba1**), (2,4-dimethylphenyl)(2-hydroxy-4-methylphenyl)methanone (**2ba2**): Prepared using procedure C; isolated by column chromatography. Yellow solid (0.086g, 0.358 mmol, 45%), as an inseparable mixture (2.3:1 **2ba1**/**2ba2**:**2aa1**, 10:1

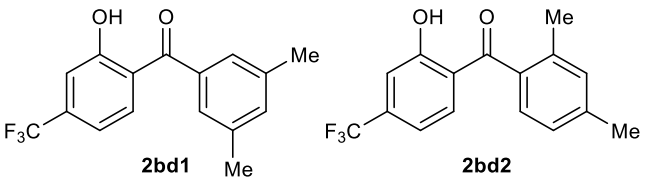
2ba1:**2ba2**). R_f = 0.36 (5% ethyl acetate in hexanes). ^{1}H NMR (500 MHz, $CDCl_3$) **2ba1**: δ 12.15 (s, 1H), 7.48 (d, *J* = 8.1 Hz, 1H), 7.26–7.22 (m, 2H), 7.20 (m, 1H), 6.88–6.85 (m, 1H), 6.68 (dd, *J* = 8.2, 1.6 Hz, 1H), 2.39 (s, 3H), 2.38 (s, 6H); **2ba2**: δ 11.95 (s, 1H). ^{13}C NMR (126 MHz, $CDCl_3$, only **2ba1** observed) δ 201.7, 163.5, 148.0, 138.3, 138.1, 133.7, 133.4, 126.9, 120.0, 118.5, 117.2, 22.1, 21.4. IR (neat) 1624 cm^{-1}. HRMS (ESI) calcd for $C_{16}H_{16}O_2$ $[M–H]^-$ *m/z* 239.1078, found 239.1086.



(3,5-dimethylphenyl)(2-hydroxy-4-methoxyphenyl)methanone (**2bb1**) , (2,4-dimethylphenyl)(2-hydroxy-4-methoxyphenyl)methanone (**2bb2**). Prepared using procedure C; isolated by column chromatography. Brown oil (0.0769g, 0.300 mmol, 38%), as an inseparable mixture (4:1 **2bb1**/**2bb2**:**2aa1**, 10:1 **2bb1**:**2bb2**). R_f = 0.28 (5% ethyl acetate in hexanes). ^{1}H NMR (500 MHz, $CDCl_3$) **2bb1**: δ 12.72 (s, 1H), 7.52 (d, *J* = 9.0 Hz), 7.22 (br s, 2H), 6.51 (d, *J* = 2.5 Hz, 1H), 6.41 (dd, *J* = 9.0, 2.5 Hz, 1H), 3.86 (s, 3H), 2.38 (s, 6H); **2bb2**: δ 12.83 (s, 1H), 3.85 (s, 3H), 2.38 (s, 6H). ^{13}C NMR (126 MHz, $CDCl_3$, only **2bb1** observed) δ 200.7, 166.4, 166.3, 138.5, 135.5, 133.9, 133.2, 126.7, 113.4, 107.4, 101.2, 55.8, 21.4. IR (neat) 1621 cm^{-1}. HRMS (ESI) calcd for $C_{16}H_{16}O_3$ $[M–H]^-$ *m/z* 256.1099, found 255.1036.

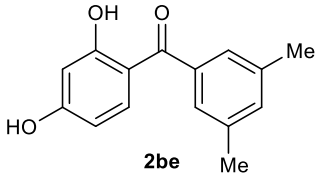


(3,5-dimethylphenyl)(4-fluoro-2-hydroxyphenyl)methanone (**2bc1**), (2,4-dimethylphenyl)(4-fluoro-2-hydroxyphenyl)methanone (**2bc2**). Prepared using procedure C; isolated by column chromatography. Yellow oil (0.102g, 0.416 mmol, 52%), as an inseparable mixture (4:1 **2bc1**/**2bc2**:**2aa1**, >20:1 **2bc1**:**2bc2**). R_f = 0.32 (5% Ethyl acetate in hexanes). ^{1}H NMR (500 MHz, $CDCl_3$) **2bc1**: δ 12.46 (d, $^5J_{H–F}$ = 1.5 Hz, 1H), 7.62 (dd, *J* = 9.0, 6.6 Hz, 1H), 7.24–7.23 (m, 2H), 7.23–7.21 (m, 1H), 6.74 (dd, *J* = 10.4, 2.5 Hz, 1H), 6.58 (ddd, *J* = 9.0, 8.0, 2.5 Hz, 1H) 2.39 (s, 6H); **2bc2**: δ 12.63 (d, $^5J_{H–F}$ = 1.4 Hz, 1H), 2.39 (s, 6H). ^{19}F NMR (471 MHz, $CDCl_3$) -98.79 (q, J = 8.6 Hz). ^{13}C NMR (126 MHz, $CDCl_3$, only **2bc1** observed) δ 201.2, 167.5 (d, $^1J_{C–F}$ = 257.1 Hz), 165.9 (d, $^3J_{C–F}$ = 14.2 Hz), 138.3, 138.0, 136.2 (d, $^3J_{C–F}$ = 11.7 Hz), 126.8, 116.5 (d, $^4J_{C–F}$ = 2.2 Hz), 107.0 (d, $^2J_{C–F}$ = 22.5 Hz), 105.1 (d, $^2J_{C-F}$ = 23.8 Hz), 21.4. ^{19}F NMR (471 MHz, $CDCl_3$) −99.43 (q, *J* = 8.3 Hz). IR (neat) 1623 cm^{-1}. HRMS (ESI) calcd for $C_{15}H_{13}FO_2$ $[M–H]^-$ *m/z* 243.0827, found 243.0833.



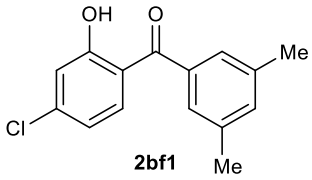
(3,5-dimethylphenyl)(2-hydroxy-4-(trifluoromethyl)phenyl)methanone (**2bd1**), (2,4-dimethylphenyl)(2-hydroxy-4-(trifluoromethyl)phenyl)methanone (**2bd2**). Prepared using procedure C; isolated by column

chromatography. Brown oil (0.105g, 0.357 mmol, 45%), as an inseparable mixture (7:1 **2bd1**/**2bd2**:**2aa1**, 13:1 **2bd1:2bd2**). R_f = 0.39 (5% ethyl acetate in hexanes). ^{1}H (500 MHz, CD_2Cl_2) **2bd1**: δ 11.99 (s, 1H), 7.77 (d, *J* = 8.3 Hz, 1H), 7.32 (d, *J* = 1.3 Hz, 1H), 7.29 (s, 3H), 7.14 (dd, *J* = 8.3, 1.4 Hz, 1H), 2.40 (s, 3H); **2bd2**: δ 12.26 (s, 1H), 7.18 (d, *J* = 7.8 Hz, 1H), 7.17 (s, 1H), 7.07 (dd, *J* = 8.4, 1.8 Hz, 1H), 2.40 (s, 6H). ^{13}C (126 MHz, CD_2Cl_2, only **2bd1** observed) δ 201.1, 163.4, 139.0, 137.8, 137.2 (q, $^2J_{C–F}$ = 32.8 Hz), 135.0, 134.8, 127.5, 127.4, 123.8 (q, $^1J_{C–F}$ = 273.4 Hz), 116.0 (q, $^3J_{C–F}$ = 3.9 Hz), 115.5 (q, $^3J_{C–F}$ = 3.6 Hz), 21.5. ^{19}F (471 MHz, CD_2Cl_2) **2bd1**: δ −64.13; **2bd2**: δ −64.18. IR (neat) 1639 cm^{-1}. HRMS (ESI) calcd for $C_{16}H_{13}F_3O_2$ $[M–H]^-$ *m/z* 293.0795, found 293.0789.

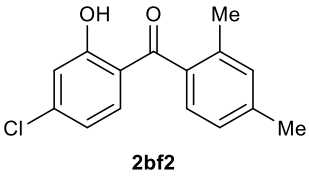


2be

(3,5-dimethylphenyl)(2,4-dihydroxyphenyl)methanone (**2be**). Prepared using procedure C; isolated by column chromatography. Yellow solid (0.0306g, 0.126 mmol, 16%). R_f = 0.16 (10% ethyl acetate in hexanes). ^{1}H NMR (500 MHz, Acetone-d_6) δ 12.70 (s, 1H), 9.64 (br s, 1H), 7.52–7.48 (m, 1H), 7.25 (s, 1H), 7.24 (s, 2H), 6.45–6.41 (m, 2H), 2.38 (s, 6H). ^{13}C NMR (126 MHz, Acetone-d_6) δ 201.2, 167.3, 165.8, 139.5, 138.9, 136.9, 133.7, 127.2, 113.5, 108.7, 103.8, 21.3. IR (neat) 1623 cm^{-1}. HRMS (ESI) calcd for $C_{15}H_{14}O_3$ $[M–H]^-$ *m/z* 241.0870, found 241.0874.

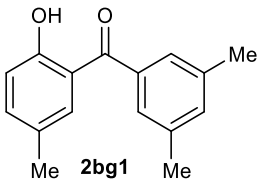


2bf1

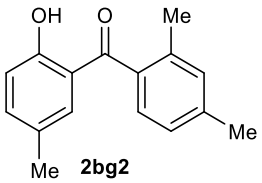


2bf2

(3,5-dimethylphenyl)(4-chloro-2-hydroxyphenyl)methanone (**2bf1**), (2,4-dimethylphenyl)(4-chloro-2-hydroxyphenyl)methanone (**2bf2**). Prepared using procedure C; isolated by column chromatography. Brown solid (0.0897g, 0.344 mmol, 43%), as an inseparable mixture (18:1 **2bf1**/**2bf2**:**2aa1**, 20:1 **2bf1**:**2bf2**). R_f = 0.40 (5% ethyl acetate in hexanes). ^{1}H NMR (500 MHz, $CDCl_3$) **2bf1**: δ 12.22 (s, 1H), 7.54 (d, *J* = 8.6 Hz, 1H), 7.24 (br s, 2H), 7.23 (br s, 1H), 7.08 (d, *J* = 2.0 Hz, 1H), 6.85 (dd, *J* = 8.6, 2.1 Hz, 1H), 2.39 (s, 6H); **2bf2**: δ 12.42 (s, 1H), 2.28 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$, only **2bf1** observed) δ 201.5, 164.0, 142.2, 138.4, 137.8, 134.7, 134.0, 126.9, 119.4, 118.6, 118.0, 21.4. IR (neat) 1618 cm^{-1}. HRMS (ESI) calcd for $C_{15}H_{13}ClO_2$ $[M–H]^-$ *m/z* 259.0531, found 259.0533.



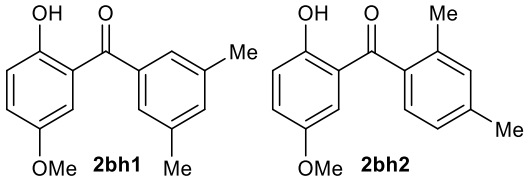
2bg1



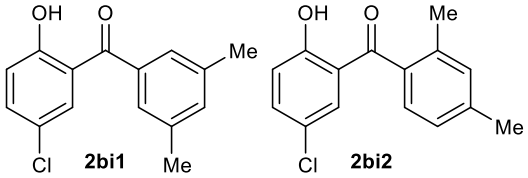
2bg2

(3,5-dimethylphenyl)(2-hydroxy-5-methylphenyl)methanone (**2bg1**), (2,4-dimethylphenyl)(2-hydroxy-5-methylphenyl)methanone (**2bg2**). Prepared using procedure C; isolated by column chromatography. Yellow solid (0.0574g, 0.239 mmol, 30%), as an inseparable mixture (1.8:1 **2bg1**/**2bg2**:**2aa1**, 18:1

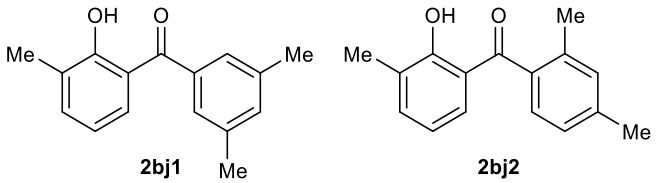
2bg1:**2bg2**). R_f = 0.37 (5% ethyl acetate in hexanes). ^{1}H NMR (500 MHz, $CDCl_3$) **2bg1**: δ 11.87 (s, 1H), 7.36 (d, *J* = 1.9 Hz, 1H), 7.31 (dd, *J* = 8.5, 2.3 Hz, 1H), 7.26 (m, 1H), 7.22 (br s, 1H), 6.97 (d, *J* = 8.4 Hz, 1H), 2.39 (s, 6H), 2.25 (s, 3H); **2bg1**: δ 12.12 (s 1H). ^{13}C NMR (126 MHz, $CDCl_3$, only **2bg1** observed) δ 202.3, 161.2, 138.3, 137.4, 133.5, 133.4, 127.8, 126.9, 119.1, 118.4, 118.2, 21.4, 20.6. IR (neat) 1630 cm^{-1}. HRMS (ESI) calcd for $C_{16}H_{16}O_2$ $[M–H]^-$ *m/z* 239.1078, found 239.1089.



(3,5-dimethylphenyl)(2-hydroxy-5-methoxyphenyl)methanone (**2bh1**), (2,4-dimethylphenyl)(2-hydroxy-5-methoxyphenyl)methanone (**2bh2**). Prepared using procedure C; isolated by column chromatography. Orange oil (0.085g, 0.331 mmol, 41%), as an inseparable mixture (2.6:1 **2bh1**/**2bh2**:**2aa1**, >20:1 **2bh1**:**2bh2**). R_f = 0.26(5% ethyl acetate in hexanes). ^{1}H NMR (500 MHz, Chloroform-*d*) δ 11.61 (s, 1H), 7.29 (br s, 2H), 7.22 (br s, 1H), 7.14 (dd, *J* = 9.1, 3.1 Hz, 1H), 7.08 (d, *J* = 3.1 Hz, 1H), 7.01 (d, *J* = 9.1 Hz, 1H), 3.70 (s, 3H), 2.39 (s, 6H) **2bh2**: δ 11.89 (s, 1H), 3.64 (s, 3H), 2.30 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$, only **2bh1** observed) δ 201.8, 157.6, 151.5, 138.2, 138.1, 133.7, 126.9, 124.1, 119.3, 119. 0, 116.6, 56.1, 21.4. IR (neat) 1626 cm^{-1}. HRMS (ESI) calcd for $C_{16}H_{16}O_3$ $[M–H]^-$ *m/z* 255.1027, found 255.1034.

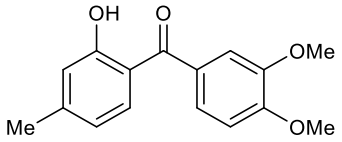


(3,5-dimethylphenyl)(5-chloro-2-hydroxyphenyl)methanone (**2bi1**), (2,4-dimethylphenyl)(5-chloro-2-hydroxyphenyl)methanone (**2bi2**). Prepared using procedure C; isolated by column chromatography. Yellow solid (0.092g, 0.351 mmol, 44%), as an inseparable mixture (5.5:1 **2bi1**/**2bi2**:**2aa1**, 19:1 **2bi1**:**2bi2**). R_f = 0.39 (5% ethyl acetate in hexanes). ^{1}H NMR (500 MHz, $CDCl_3$) **2bi1**: δ 11.94 (s, 1H), 7.56 (d, *J* = 2.6 Hz, 1H), 7.44 (dd, *J* = 8.9, 2.7 Hz, 1H), 7.25 (m, 3H), 7.02 (d, *J* = 8.9 Hz, 1H), 2.40 (s, 6H); **2bi2**:z δ 12.18 (s, 1H), 2.29 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$, only **2bi1** observed) δ 201.3, 161.8, 138.5, 137.5, 136.2, 134.1, 132.6, 126.8, 123.4, 120.10, 120.08, 21.4. IR (neat) 1621 cm^{-1}. HRMS (ESI) calcd for $C_{15}H_{13}ClO_2$ $[M–H]^-$ *m/z* 259.0531, found 259.0535.



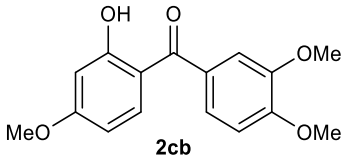
(3,5-dimethylphenyl)(2-hydroxy-3-methylphenyl)methanone (**2bj1**), (2,4-dimethylphenyl)(2-hydroxy-3-methylphenyl)methanone (**2bj2**). Prepared using procedure C; isolated by column chromatography. Brown oil (0.082g, 0.343 mmol, 43%), as an inseparable mixture (3:1 **2bj1**/**2bj2**:**2aa1**, 10:1 **2bj1**:**2bj2**).

R_f = 0.45 (5% ethyl acetate in hexanes). ^{1}H NMR (500 MHz, $CDCl_3$) δ 12.15 (s, 1H), 7.48 (d, *J* = 8.2 Hz, 1H), 7.25 (br s, 2H), 7.20 (br s, 1H) 6.87 (m, 1H), 6.68 (dd, *J* = 8.0, 0.9 Hz, 1H), 2.38 (s, 6H), 2.37 (s, 3H); **2bj2**: δ 11.95 (s, 1H), 7.55 (s, 1H), 6.95 (s, 2H), 2.40 (s, 9H). ^{13}C NMR (126 MHz, $CDCl_3$, only **2bj1** observed) δ 201.8, 163.5, 148.0, 138.3, 138.1, 133.7, 133.4, 126.9, 126.8, 120.0, 118.4, 117.2, 22.1. IR (neat) 1618 cm^{-1}. HRMS (ESI) calcd for $C_{16}H_{16}O_2$ $[M–H]^-$ *m/z* 239.1078, found 239.1073.



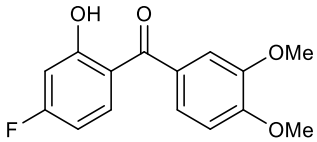
2ca

(3,4-dimethoxyphenyl)(2-hydroxy-4-methylphenyl)methanone (**2ca**). Prepared using procedure A; isolated by column chromatography. Light yellow solid (0.169g, 0.616 mmol, 77%). mp = 135–138 °C. R_f = 0.39 (30% ethyl acetate in hexanes). ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.00 (s, 1H), 7.55 (d, *J* = 8.1 Hz, 1H), 7.36–7.23 (m, 2H), 6.94 (d, *J* = 8.1 Hz, 1H), 6.88 (s, 1H), 6.70 (d, *J* = 8.2 Hz, 1H), 3.97 (s, 3H), 3.94 (s, 4H), 2.38 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 199.6, 163.2, 152.5, 149.0, 147.6, 133.3, 130.7, 124.1, 119.9, 118.5, 117.2, 112.1, 110.1, 56.2, 56.1, 22.0. IR (thin film, CH_2Cl_2) 1625 cm^{-1}. HRMS (ESI) calcd for $C_{16}H_{16}O_4$ $[M–H]^-$ *m/z* 271.0976, found 271.0977.



2cb

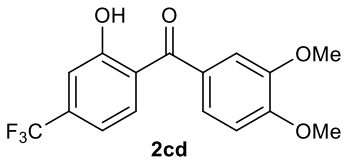
(3,4-dimethoxyphenyl)(2-hydroxy-4-methoxyphenyl)methanone (**2cb**). Prepared using procedure A; isolated by column chromatography. Light yellow solid (0.183 g, 0.640 mmol, 80%). mp = 135–137 °C. R_f = 0.29 (30% ethyl acetate in hexanes). ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.62 (s, 1H), 7.59 (d, *J* = 9.0 Hz, 1H), 7.31–7.23 (m, 1H), 6.94 (d, *J* = 8.1 Hz, 1H), 6.52 (d, *J* = 2.5 Hz, 1H), 6.43 (dd, *J* = 8.9, 2.5 Hz, 1H), 3.97 (s, 3H), 3.94 (s, 3H), 3.87 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 198.7, 166.2, 166.0, 152.3, 149.1, 135.1, 130.9, 123.7, 113.4, 112.0, 110.1, 107.3, 101.3, 56.2, 56.2, 55.7. IR (thin film, CH_2Cl_2) 1623 cm^{-1}. HRMS (ESI) calcd for $C_{16}H_{16}O_5$ $[M–H]^-$ *m/z* 287.0925, found 287.0922.



2cc

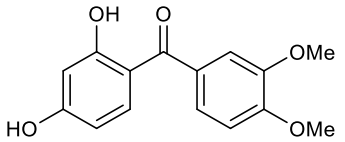
(3,4-dimethoxyphenyl)(4-fluoro-2-hydroxyphenyl)methanone (**2cc**). Prepared using procedure A; isolated by column chromatography. Light yellow solid (0.656 mmol, 0.182 g, 82%). mp = 103–105 °C. R_f = 0.37 (30% ethyl acetate in hexanes). ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.31 (s, 1H), 7.69 (dd, *J* = 8.9, 6.6 Hz, 1H), 7.31 – 7.25 (m, 2H), 6.95 (d, *J* = 8.2 Hz, 1H), 6.75 (dd, *J* = 10.4, 2.5 Hz, 1H), 6.60 (td, *J* = 8.6, 2.6 Hz, 1H), 3.98 (s, 3H), 3.94 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 198.9, 168.5, 165.9, 165.5 (d, *J* = 14.3 Hz), 152.8, 149.2, 135.6 (d, *J* = 11.6 Hz), 130.3, 124.1, 116.4 (d, *J* = 2.3 Hz), 111.0 (d, *J* = 189.5 Hz), 106.7 (d, *J* = 22.5 Hz), 105.1 (d, *J* = 23.8 Hz), 56.2, 56.1. ^{19}F {^{1}H} NMR (376 MHz, $CDCl_3$) δ −100.00.

IR (thin film, CH_2Cl_2) 1628 cm^{-1}. HRMS (ESI) calcd for $C_{15}H_{13}FO_4$ $[M-H]^-$ *m/z* 275.0725, found 275.0724.



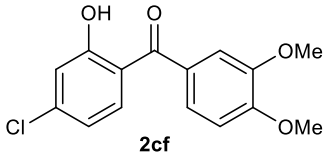
2cd

(3,4-dimethoxyphenyl)(2-hydroxy-4-(trifluoromethyl)phenyl)methanone (**2cd**). Prepared using procedure A; isolated by column chromatography. Yellow solid (0.230 g, 0.705 mmol, 88%). mp = 74–76 °C. R_f = 0.37 (30% ethyl acetate in hexanes). ^{1}H NMR (400 MHz, $CDCl_3$) δ 11.78 (s, 1H), 7.79 (dd, *J* = 8.3, 1.0 Hz, 1H), 7.36–7.31 (m, 3H), 7.13 (dd, *J* = 8.3, 1.8 Hz, 1H), 6.96 (d, *J* = 8.8 Hz, 1H), 3.99 (s, 3H), 3.95 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 199.0, 162.7, 153.4, 149.4, 136.7 (q, *J* = 33.0 Hz), 133.8, 129.8, 124.8, 123.3 (q, *J* = 273.5 Hz), 121.7, 115.8 (q, *J* = 3.9 Hz), 114.9 (q, *J* = 3.6 Hz), 112.1, 110.2, 56.3, 56.2. ^{19}F {^{1}H} NMR (376 MHz, $CDCl_3$) δ −63.78. IR (thin film, CH_2Cl_2) 1638 cm^{-1}. HRMS (ESI) calcd for $C_{16}H_{13}F_3O_4$ $[M-H]^-$ *m/z* 325.0693, found 325.0689.



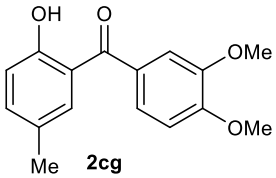
2ce

(3,4-dimethoxyphenyl) (2,4-dihydroxyphenyl)methanone (**2ce**). Prepared using procedure A; isolated by column chromatography. Light yellow solid (0.146 g, 0.536 mmol, 67%). mp = 174–172 °C. R_f = 0.36 (40% hexanes in ethyl acetate). ^{1}H NMR (400 MHz, Acetone-d_6) δ 12.51 (s, 1H), 7.47 (d, *J* = 9.0 Hz, 1H), 7.18–7.11 (m, 2H), 6.95 (d, *J* = 8.9 Hz, 1H), 6.34–6.27 (m, 2H), 3.78 (s, 3H), 3.75 (s, 3H). ^{13}C NMR (101 MHz, Acetone-d_6) δ 199.4, 167.0, 165.5, 153.5, 150.1, 136.6, 131.6, 124.2, 113.5, 113.2, 111.5, 108.5, 103.9, 56.3, 56.2. IR (thin film, CH_2Cl_2) 1626 cm^{-1}. HRMS (ESI) calcd for $C_{15}H_{14}O_5$ $[M-H]^-$ *m/z* 273.0768, found 273.0766.



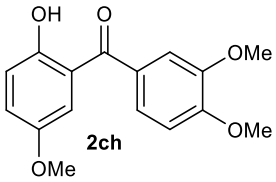
2cf

(3,4-dimethoxyphenyl)(4-chloro-2-hydroxyphenyl)methanone (**2cf**). Prepared using procedure A; isolated by column chromatography. Light yellow solid (0.108 g, 0.368 mmol, 46%). mp = 114–116 °C. R_f = 0.37 (30% ethyl acetate in hexanes). ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.06 (s, 1H), 7.60 (d, *J* = 8.6 Hz, 1H), 7.32–7.28 (m, 2H), 7.09 (d, *J* = 2.1 Hz, 1H), 6.95 (d, *J* = 8.1 Hz, 1H), 6.87 (dd, *J* = 8.6, 2.1 Hz, 1H), 3.98 (s, 3H), 3.94 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 199.2, 163.7, 153.0, 149.3, 141.8, 134.3, 130.2, 124.3, 119.3, 118.6, 118.1, 112.1, 110.2, 56.3, 56.2. IR (thin film, CH_2Cl_2) 1622 cm^{-1}. HRMS (ESI) calcd for $C_{15}H_{13}ClO_4$ $[M-H]^-$ *m/z* 291.0430, found 291.0432.



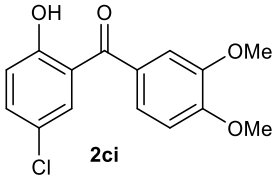
2cg

(3,4-dimethoxyphenyl)(2-hydroxy-5-methylphenyl)methanone (**2cg**). Prepared using procedure A; isolated by column chromatography. Light yellow solid (0.211 g, 0.776 mmol, 97%). mp = 113–116 °C. R_f = 0.34 (30% ethyl acetate in hexanes). ^{1}H NMR (400 MHz, $CDCl_3$) δ 11.68 (s, 1H), 7.44 (d, *J* = 1.9 Hz, 1H), 7.35–7.29 (m, 3H), 6.98 (d, *J* = 8.4 Hz, 1H), 6.95 (d, *J* = 8.2 Hz, 1H), 3.98 (s, 3H), 3.94 (s, 3H), 2.28 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 200.0, 160.9, 152.7, 149.1, 137.0, 133.1, 130.7, 127.7, 124.3, 119.2, 118.2, 112.2, 110.1, 56.2, 56.2, 20.7. IR (thin film, CH_2Cl_2) 1630 cm^{-1}. HRMS (ESI) calcd for $C_{16}H_{16}O_4$ $[M-H]^-$ *m/z* 271.0976, found 271.0982.



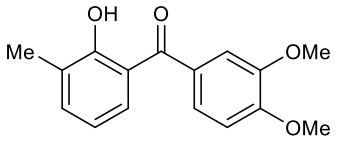
2ch

(3,4-dimethoxyphenyl)(2-hydroxy-5-methoxyphenyl)methanone (**2ch**). Prepared using procedure A; isolated by column chromatography. Yellow-orange solid (0.192 g, 0.664 mmol, 83%). mp = 86–87 °C. R_f = 0.28 (30% ethyl acetate in hexanes). ^{1}H NMR (400 MHz, $CDCl_3$) δ 11.41 (s, 1H), 7.37 (dd, *J* = 8.3, 2.0 Hz, 1H), 7.33 (d, *J* = 2.0 Hz, 1H), 7.17–7.09 (m, 2H), 7.06–6.98 (m, 1H), 6.95 (d, *J* = 8.3 Hz, 1H), 3.98 (s, 3H), 3.95 (s, 3H), 3.73 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 199.4, 157.1, 152.7, 151.4, 149.0, 130.4, 124.2, 123.5, 119.2, 119.0, 116.2, 112.1, 110.0, 56.1, 56.1, 56.0. IR (thin film, CH_2Cl_2) 1632 cm^{-1}. HRMS (ESI) calcd for $C_{16}H_{16}O_5$ $[M-H]^-$ *m/z* 287.0925, found 287.0931.



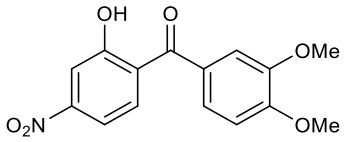
2ci

(3,4-dimethoxyphenyl)(5-chloro-2-hydroxyphenyl)methanone (**2ci**). Prepared using procedure A; isolated by column chromatography. Light yellow solid (0.187 g, 0.640 mmol, 80%). mp = 127–130 °C. R_f = 0.29 (20% ethyl acetate in hexanes). ^{1}H NMR (400 MHz, $CDCl_3$) δ 11.73 (s, 1H), 7.64 (d, *J* = 2.7 Hz, 1H), 7.44 (dd, *J* = 8.9, 2.7 Hz, 1H), 7.36–7.27 (m, 2H), 7.02 (d, *J* = 8.9 Hz, 1H), 6.96 (d, *J* = 8.3 Hz, 1H), 3.98 (s, 3H), 3.95 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 198.8, 161.4, 153.2, 149.3, 135.7, 132.3, 129.8, 124.5, 123.4, 120.2, 120.1, 112.1, 110.2, 56.3, 56.2. IR (thin film, CH_2Cl_2) 1623 cm^{-1}. HRMS (ESI) calcd for $C_{15}H_{13}ClO_4$ $[M-H]^-$ *m/z* 291.0430, found 291.0424.



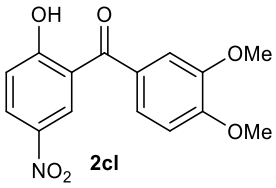
2cj

(3,4-dimethoxyphenyl)(2-hydroxy-3-methylphenyl)methanone (**2cj**). Prepared using procedure A; isolated by column chromatography. Yellow solid (0.192 g, 0.704 mmol, 88%). mp = 81–84 °C. R_f = 0.26 (20% ethyl acetate in hexanes). ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.17 (s, 1H), 7.50 (d, *J* = 7.9 Hz, 1H), 7.36 (d, *J* = 7.2 Hz, 1H), 7.34–7.28 (m, 2H), 6.93 (d, *J* = 8.0 Hz, 1H), 6.78 (t, *J* = 7.7 Hz, 1H), 3.96 (s, 3H), 3.93 (s, 3H), 2.32 (s, 3H).^{13}C NMR (101 MHz, $CDCl_3$) δ 200.4, 161.4, 152.7, 149.0, 136.8, 131.1, 130.8, 127.5, 124.4, 118.8, 117.9, 112.2, 110.0, 56.2, 56.2, 15.8. IR (neat) 1597 cm^{-1}. HRMS (ESI) calcd for $C_{16}H_{16}O_4$ $[M-H]^-$ *m/z* 279.0976, found 279.0976.



2ck

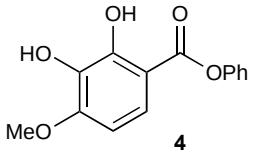
(4-nitro-2-hydroxyphenyl)(3,4-dimethoxyphenyl)methanone (**2ck**). Prepared using procedure A; isolated by column chromatography. Orange solid (0.118 g, 0.392mmol, 49%). mp = 128–130 °C. R_f = 0.25 (5% acetic acid 15% ethyl acetate in hexanes). ^{1}H NMR (400 MHz, $CDCl_3$) δ 11.76 (s, 1H), 7.89 (d, *J* = 2.1 Hz, 1H), 7.85 (d, *J* = 8.7 Hz, 1H), 7.73 (d, *J* = 2.2 Hz, 1H), 7.38–7.30 (m, 2H), 6.97 (d, *J* = 8.9 Hz, 1H), 4.00 (s, 3H), 3.96 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 198.6, 163.0, 153.8, 151.7, 149.5, 134.1, 129.5, 125.0, 123.6, 113.8, 113.0, 112.1, 110.2, 56.4, 56.3. IR (thin film, CH_2Cl_2) 1623 cm^{-1}. HRMS (ESI) calcd for $C_{15}H_{13}NO_6$ $[M-H]^-$ *m/z* 302.0670, found 302.0666.



2cl

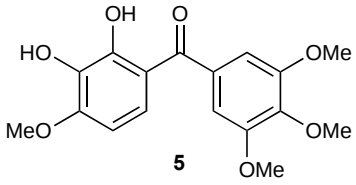
(5-nitro-2-hydroxyphenyl)(3,4-dimethoxyphenyl)methanone (**2cl**). Prepared using procedure A; isolated by column chromatography. Light brown solid (0.114 g, 0.376mmol, 47%). mp = 179–182 °C. R_f = 0.25 (5% acetic acid 15% ethyl acetate in hexanes). ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.59 (s, 1H), 8.69 (d, *J* = 2.7 Hz, 1H), 8.38 (dd, *J* = 9.2, 2.7 Hz, 1H), 7.37 (dd, *J* = 8.3, 1.8 Hz, 1H), 7.34 (d, *J* = 1.7 Hz, 1H), 7.17 (d, *J* = 9.2 Hz, 1H), 7.01 (d, *J* = 8.3 Hz, 1H), 4.01 (s, 3H), 3.97 (s, 3H).^{13}C NMR (101 MHz, $CDCl_3$) δ ^{13}C NMR (101 MHz, $CDCl_3$) δ 198.5, 167.9, 153.9, 149.6, 139.5, 130.5, 129.5, 128.9, 124.8, 119.5, 118.4, 112.0, 110.5, 56.4, 56.3. IR (thin film, CH_2Cl_2) 1622 cm^{-1}. HRMS (ESI) calcd for $C_{15}H_{13}NO_6$ $[M-H]^-$ *m/z* 302.0670, found 302.0670.

HYDROXYPHENSTATIN



4

Phenyl 2,3-dihydroxy-4-methoxybenzoate (**4**). In a nitrogen-filled glovebox, 2,3-dihydroxy-4-methoxybenzoic acid[5] (500 mg, 2.72 mmol, 1 equiv), phenol (5.11 g, 54.3 mmol, 20 equiv), and phosphorus oxychloride (631.5 mg, 4.07 mmol, 1.5 equiv) were added to a 25 mL screw top pressure vessel with a stir bar. The vessel was sealed, removed from the glovebox, and heated to 100 °C for 20 hours. The vessel was cooled to room temperature and the solvent and remaining phenol removed *in vacuo*. The product was purified by flash chromatography (20%→30% ethyl acetate in hexanes). Off-white/yellow powder (0.532 g, 2.04 mmol, 75% yield). mpt = 145–147 °C. R_f = 0.16 (20% ethyl acetate in hexanes). ^{1}H NMR (500 MHz, $CDCl_3$) δ 10.56 (s, 1H), 7.66 (d, *J* = 9.0 Hz, 1H), 7.45 (t, *J* = 7.5 Hz. 2H), 7.30 (t, *J* = 7.3 Hz, 1H), 7.21 (d, *J* = 8.1 Hz, 2H), 6.59 (d, *J* = 9.0 Hz, 1H), 4.97 (br s, 1H), 3.98 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 168.9, 152.3, 150.3, 149.9, 133.6, 129.7, 126.4, 122.1, 121.8, 106.2, 103.4, 56.4. IR (thin film, CH_2Cl_2) 1667 cm^{-1}. HRMS (ESI) calcd for $C_{14}H_{12}O_5$ $[M-H]^-$ *m/z* 259.0612, found 259.0599.



5

(2,3-dihydroxy-4-methoxyphenyl)(3,4,5-trimethoxyphenyl)methanone (Hydroxyphenstatin, **5**). TrixiePhos (18 mg, 0.045 mmol, 3 mol%), $[Ir(cod)OMe]_2$ (10 mg, 0.015 mmol, 1 mol%), phenyl salicylate (0.390 g, 1.5 mmol, 1 equiv), 1,5-COD (0.18 mL, 1.5 mmol, 1 equiv), and 1,2,3-trimethoxybenzene (19.8 g, 78.5 equiv) were added to a 75 mL PTFE-sealed screw top pressure vessel. The vessel was sealed, removed from the glovebox, and heated to 170 °C in an oil bath for 20 h. The vessel was then cooled to room temperature and brought into the glovebox and TrixiePhos (18 mg, 0.045 mmol, 3 mol%) and $[Ir(cod)OMe]_2$ (10 mg, 0.015 mmol, 1 mol%) was added. The vessel was sealed, brought out of box, and heated to 170 °C for an additional 20 hours. After 20 hours, the vessel was then cooled and a third charge of iridium and phosphine was added as before. The vessel was heated to 170 °C for an additional 20 hours. The vessel was then cooled to room temperature and contents were transferred to a 50 mL pear-shaped flask. The solvent was removed *in vacuo*. Product was purified by column chromatography (20:80:2 EtOAc:Hex:AcOH for ~10 column volumes, 40:60:2 for ~4 column volumes, 50:50:2 for ~4 column volumes). Yellow/brown solid obtained (0.240 g, 0.718 mmol, 48% yield). R_f = 0.22 (50% ethyl acetate in hexanes). ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.22 (s, 1H), 7.26 (d, *J* = 9.0 Hz,

[5] Prepared using method reported in *Org. Process Res. Dev.* **2011**, *15*, 376–381.

1H), 6.92 (s, 2H), 6.51 (d, J = 9.0 Hz, 1H), 5.56 (bs, 1H), 3.98 (s, 3H), 3.94 (s, 3H), 3.90 (s, 6H). IR (neat) 1635 cm^{-1}. All data are consistent with prior synthesis.[6]

[6] *J. Med. Chem.* **2000**, *43*, 2731–2737.

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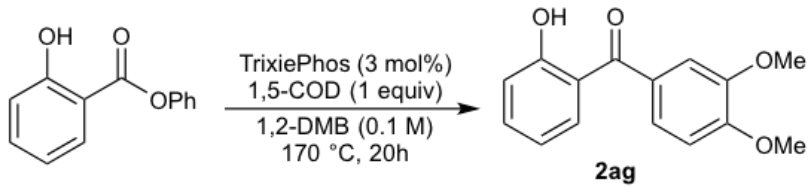

2ag

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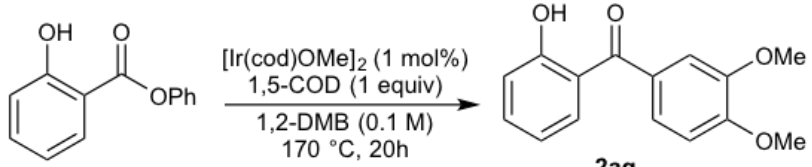

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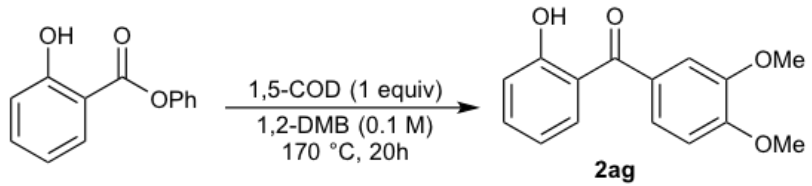

2ag

NAS1-89/10
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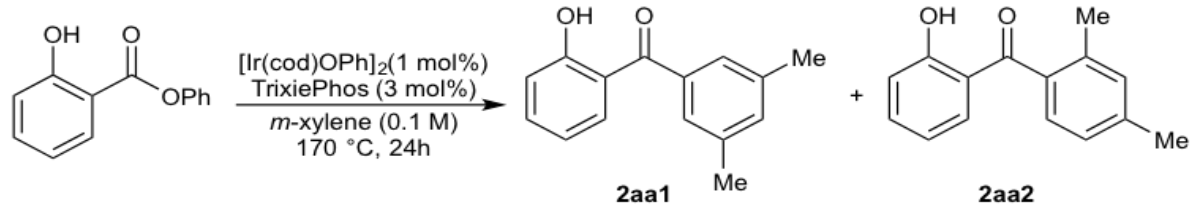
C6D6



NAS1-89/20
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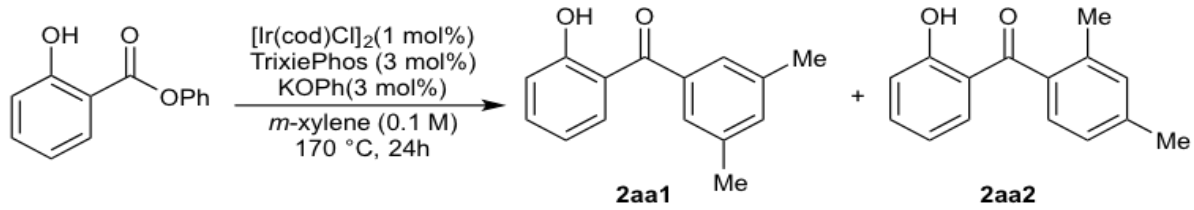
C6D6



NAS1-89/30
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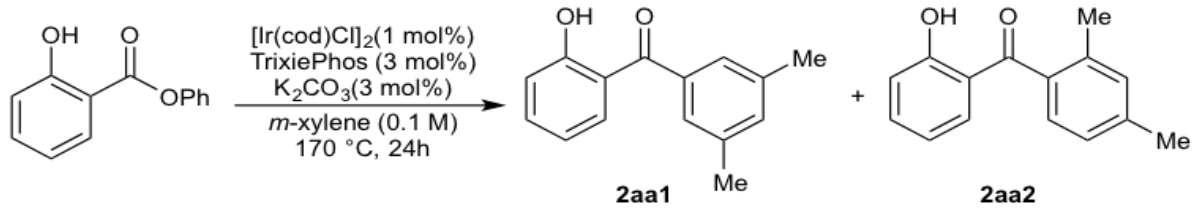
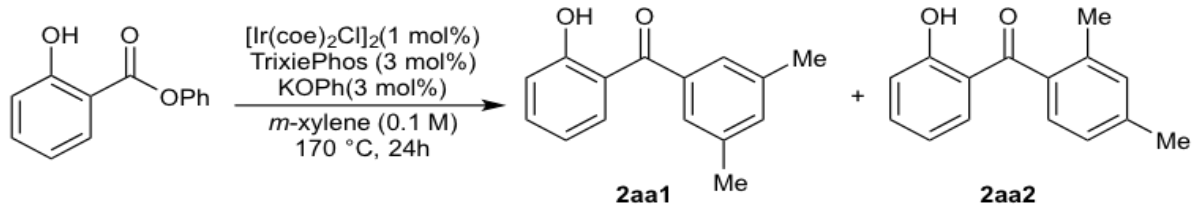
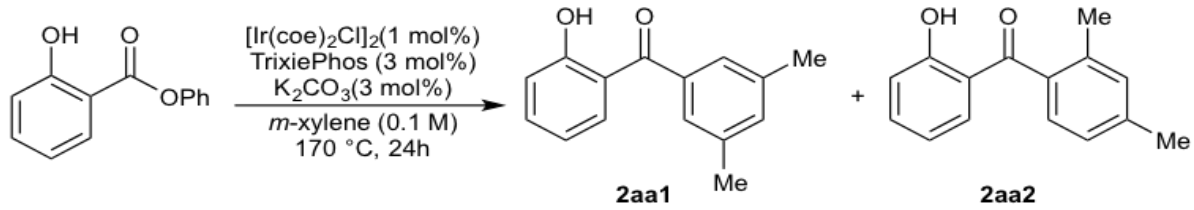
294.0
C6D6



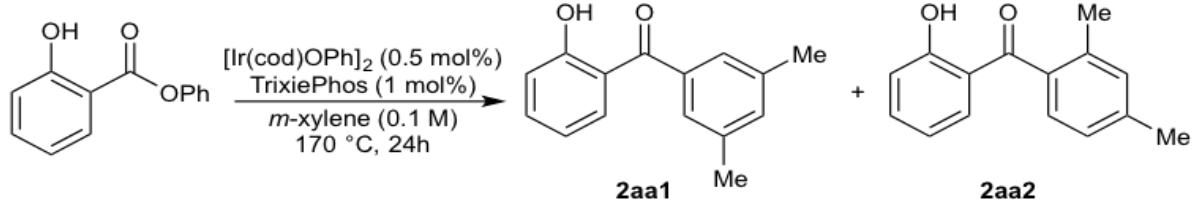
NAS1-89/40
[Ir(coe)2Cl] Dimer, with KOPh
500.13
294.0
C6D6



NAS1-89/50
[Ir(coe)2Cl] Dimer, with K2CO3
500.13
294.0
C6D6

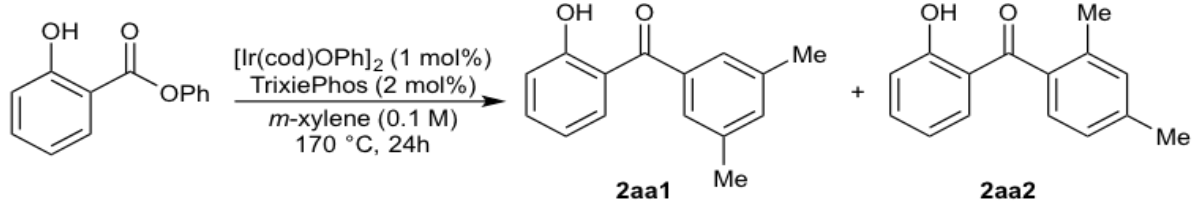


NAS1-71/1
1% Catalyst
500.33
300.0
C6D6

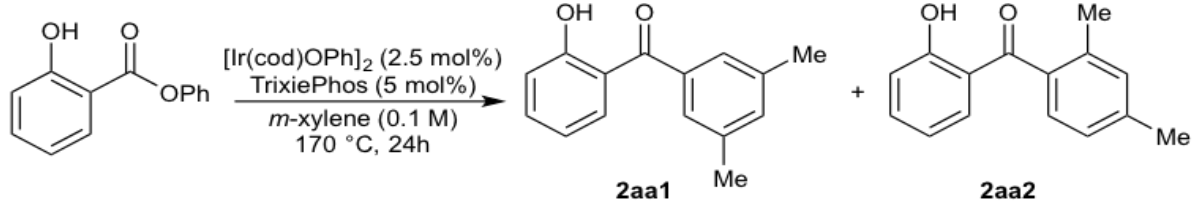


2aa1 **2aa2**

NAS1-71/2
2% Catalyst
500.33
300.0
C6D6

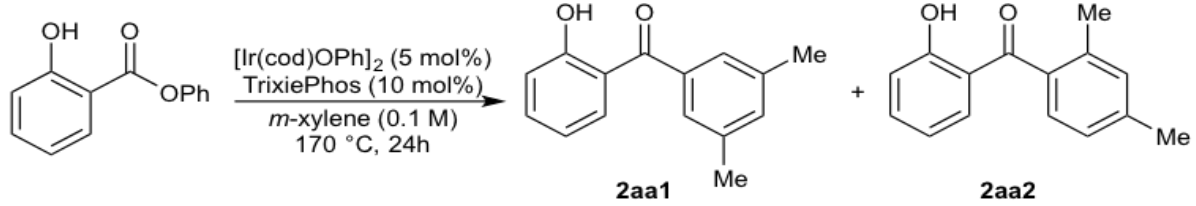


NAS1-71/3
5% Catalyst
500.33
300.1
C6D6



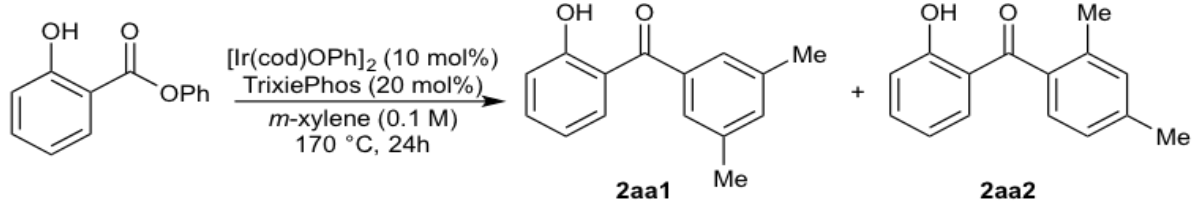
2aa1 **2aa2**

NAS1-71/4
10% Catalyst
500.33
300.0
C6D6



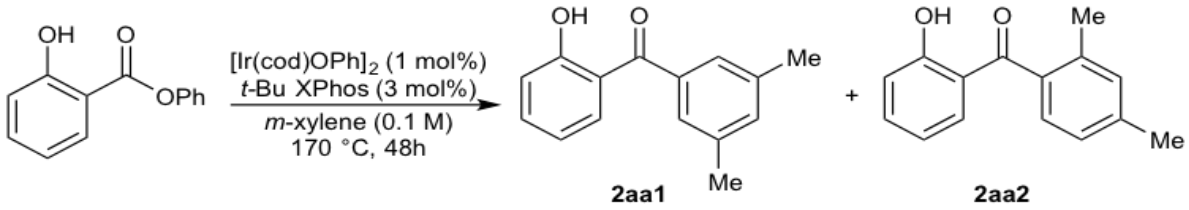
2aa1 **2aa2**

NAS1-71/5
20% Catalyst
500.33
300.0
C6D6

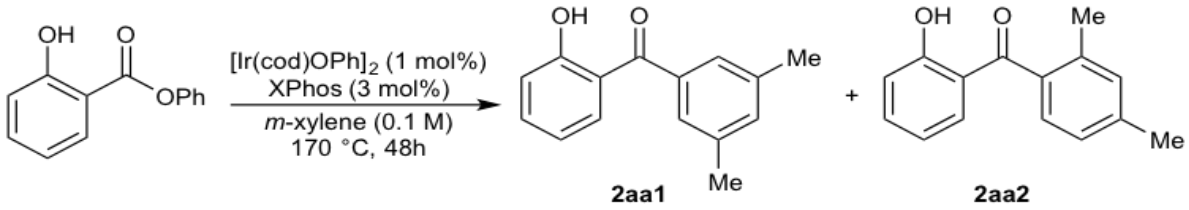


2aa1 **2aa2**

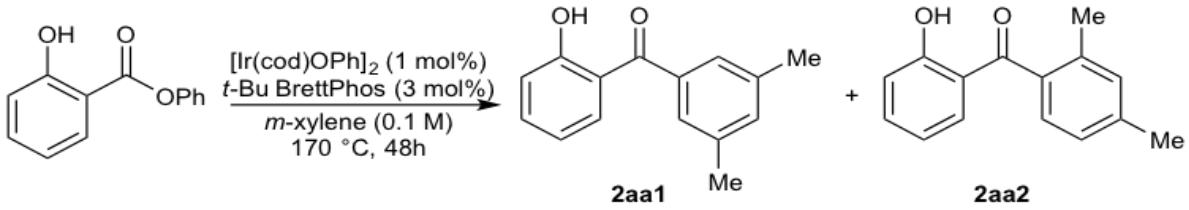
NAS1-103/10
t-Bu XPhos
500.13
297.0
C6D6



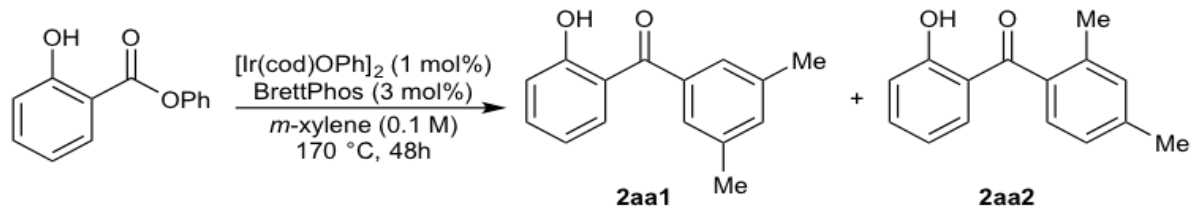
NAS1-103/20
XPhos
500.13
297.0
C6D6



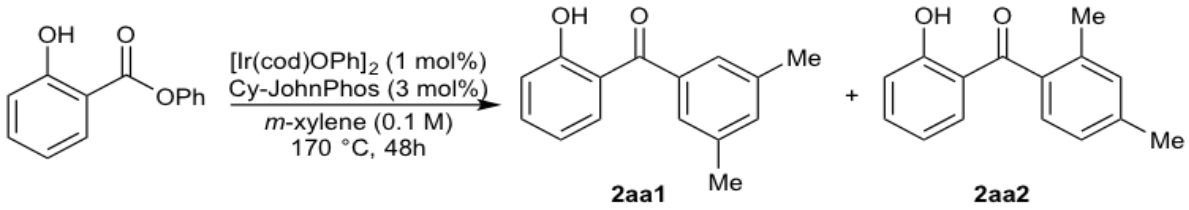
NAS1-103/30
t-Bu BrettPhos
500.13
297.0
C6D6



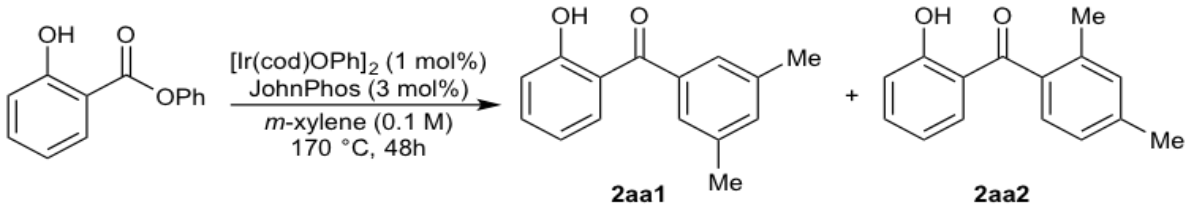
NAS1-103/40
BrettPhos
500.13
297.0
C6D6



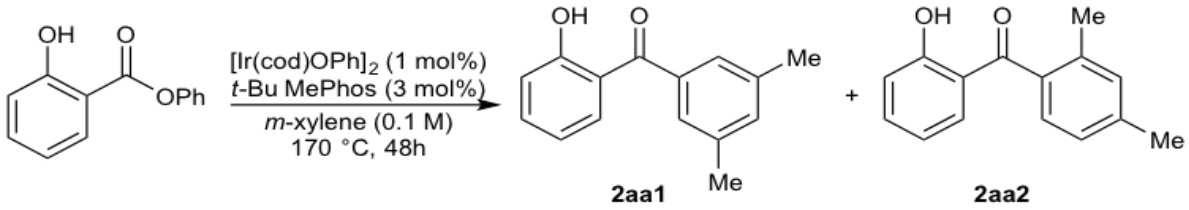
NAS1-103/50
Cy-JohnPhos
500.13
297.0
C6D6



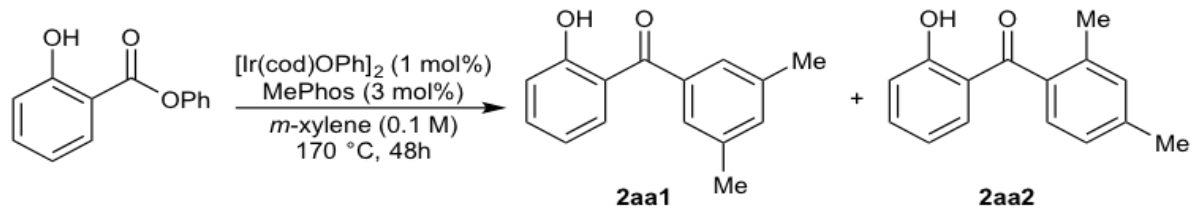
NAS1-103/100
JohnPhos
500.13
297.0
C6D6



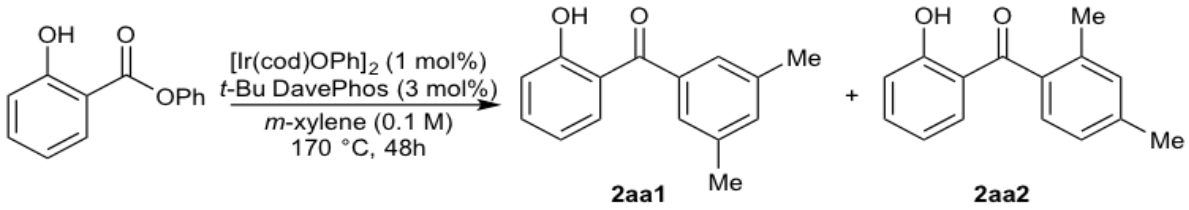
NAS1-103/110
t-Bu MePhos
500.13
297.0
C6D6



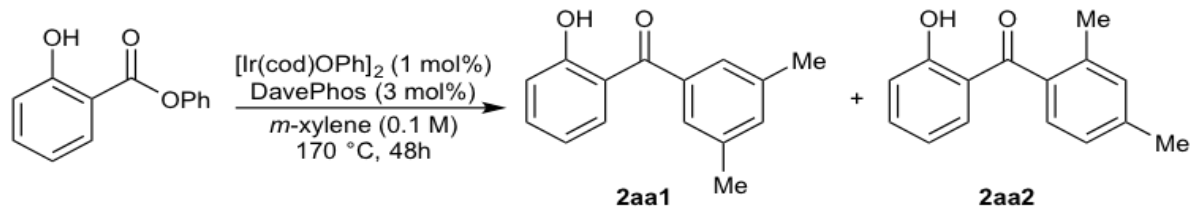
NAS1-103/60
MePhos
500.13
297.0
C6D6



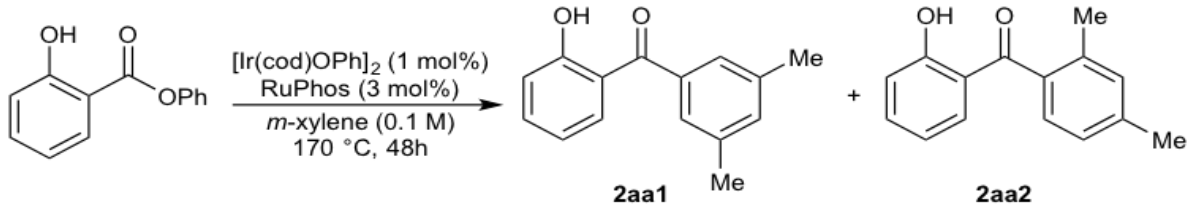
NAS1-103/120
t-Bu DavePhos
500.13
297.0
C6D6



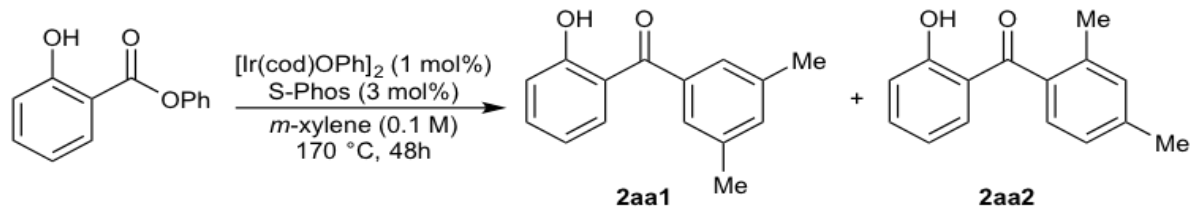
NAS1-103/70
DavePhos
500.13
297.0
C6D6



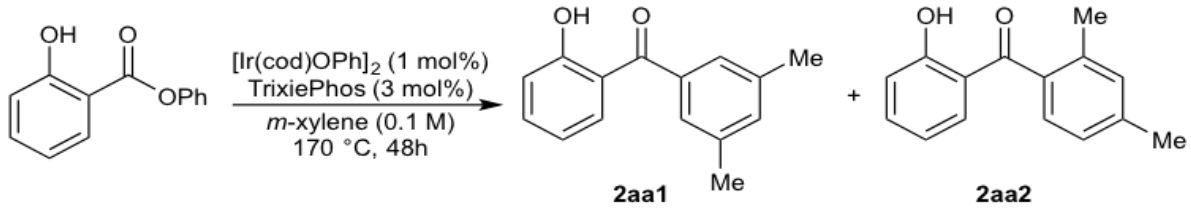
NAS1-103/80
RuPhos
500.13
297.0
C6D6



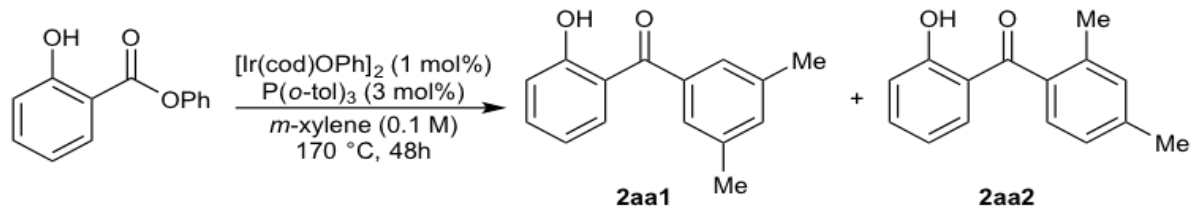
NAS1-103/90
S-Phos
500.13
297.0
C6D6



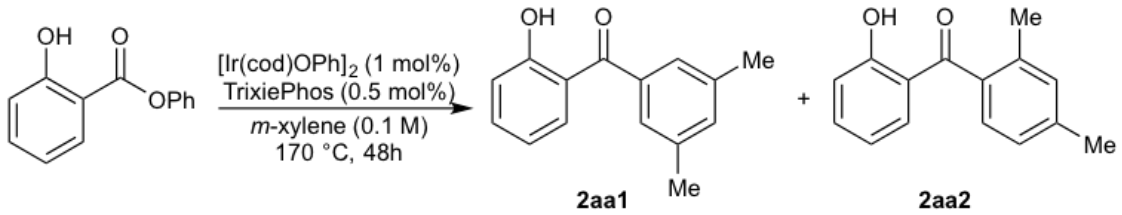
NAS1-103/130
TrixiePhos
500.13
297.0
C6D6



NAS1-103/140
P(o-tol)3
500.13
297.0
C6D6

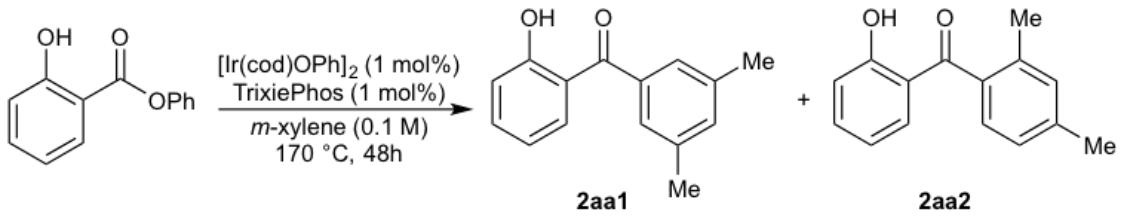


NAS1-73.1.fid
0.5 mol% TrixiePhos
500.33
300.0
C6D6



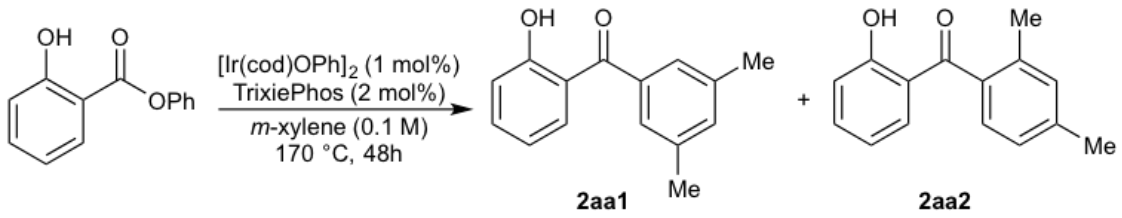
2aa1 **2aa2**

NAS1-73.2.fid
1 mol% TrixiePhos
500.33
300.1
C6D6



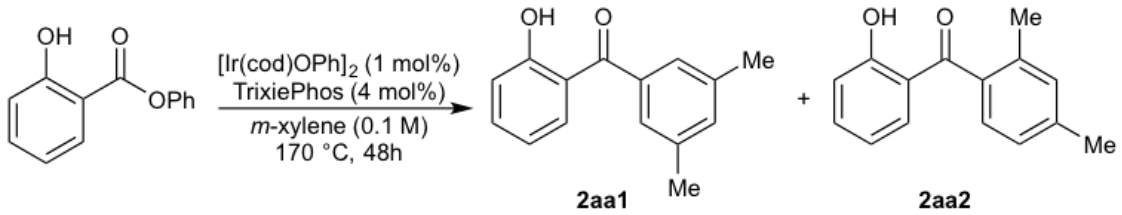
2aa1 **2aa2**

NAS1-73.3.fid
2 mol% TrixiePhos
500.33
300.0
C6D6



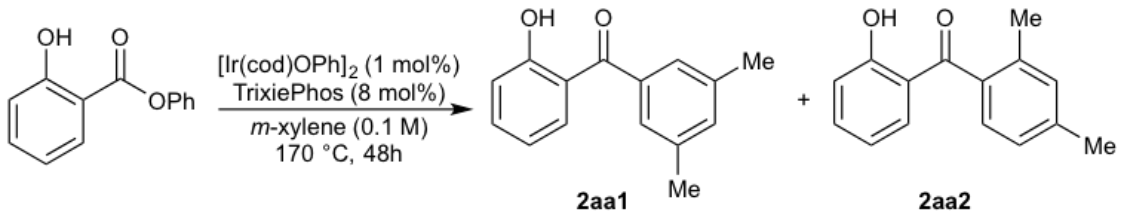
2aa1 **2aa2**

NAS1-73.4.fid
4 mol% TrixiePhos
500.33
299.9
C6D6



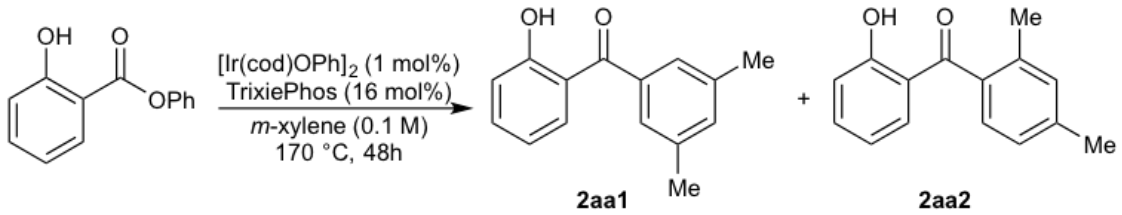
2aa1 **2aa2**

NAS1-73.5.fid
8 mol% TrixiePhos
500.33
300.0
C6D6



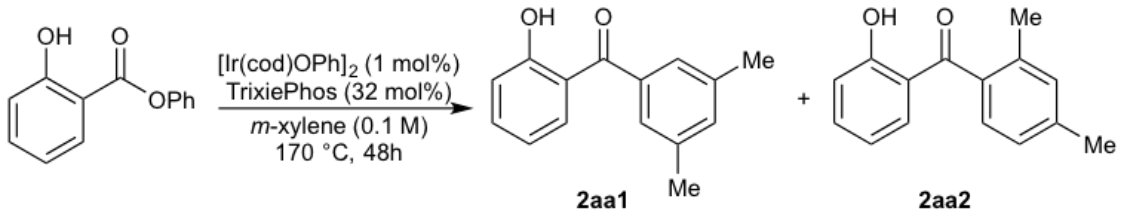
2aa1 **2aa2**

NAS1-73.6.fid
16 mol% TrixiePhos
500.33
300.0
C6D6



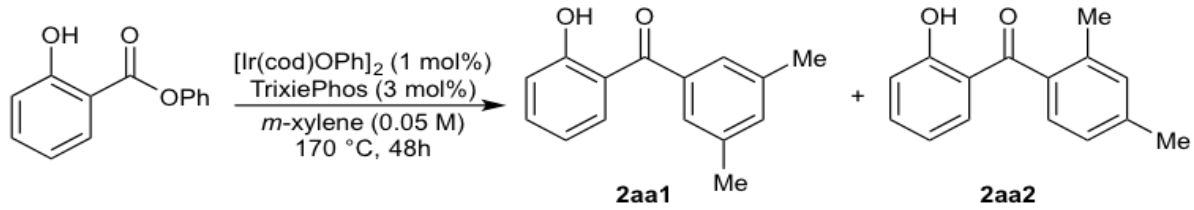
2aa1 **2aa2**

NAS1-73.7.fid
32 mol% TrixiePhos
500.33
300.0
C6D6



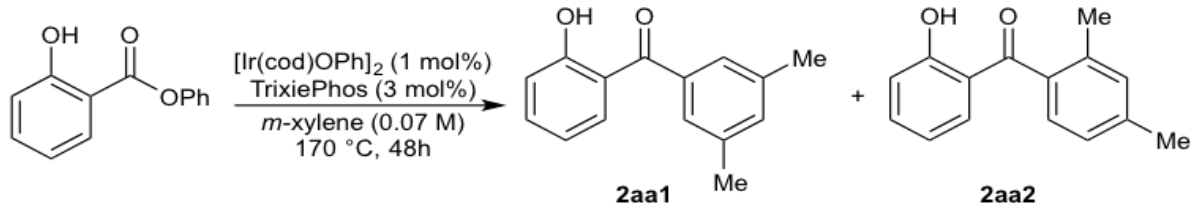
2aa1 **2aa2**

NAS1-85/10
0.05M Phenyl Salicylate
500.13
294.0
C6D6



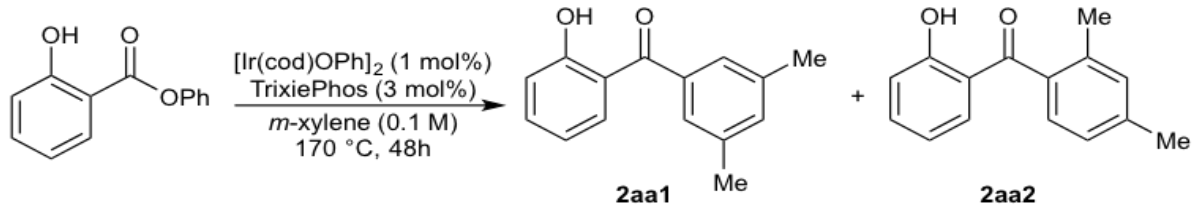
2aa1 **2aa2**

NAS1-85/20
0.07M Phenyl Salicylate
500.13
294.0
C6D6

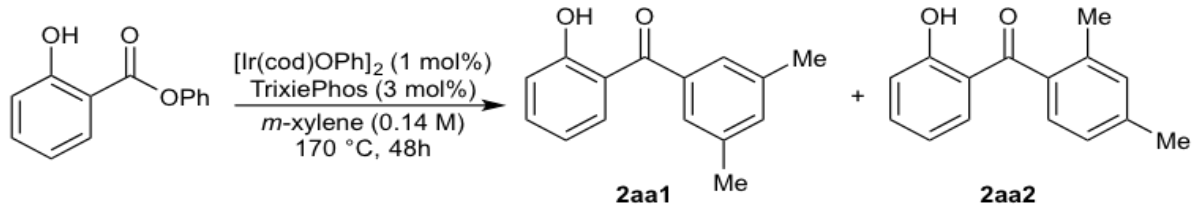


2aa1 **2aa2**

NAS1-85/30
0.1M Phenyl Salicylate
500.13
294.0
C6D6

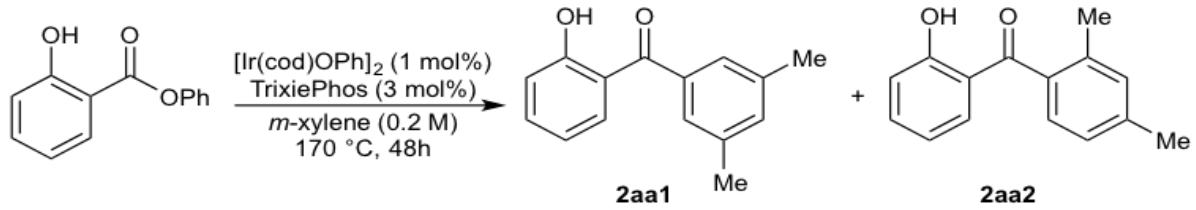


NAS1-85/40
0.14M Phenyl Salicylate
500.13
294.0
C6D6

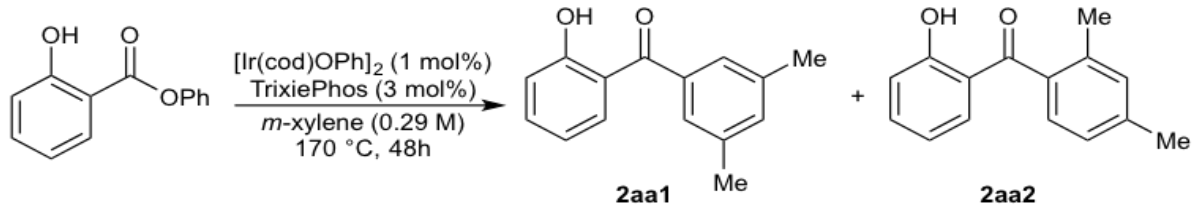


2aa1 **2aa2**

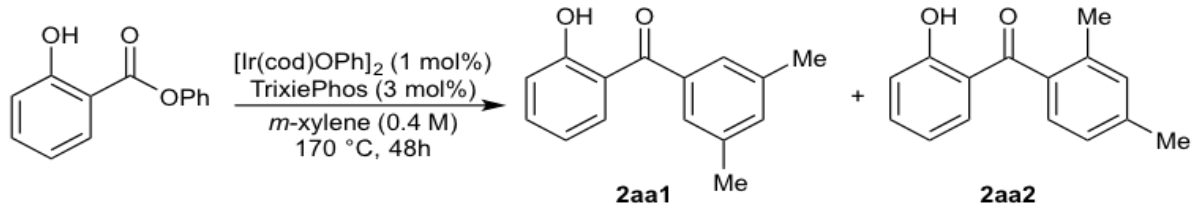
NAS1-85/50
0.2M Phenyl Salicylate
500.13
294.0
C6D6



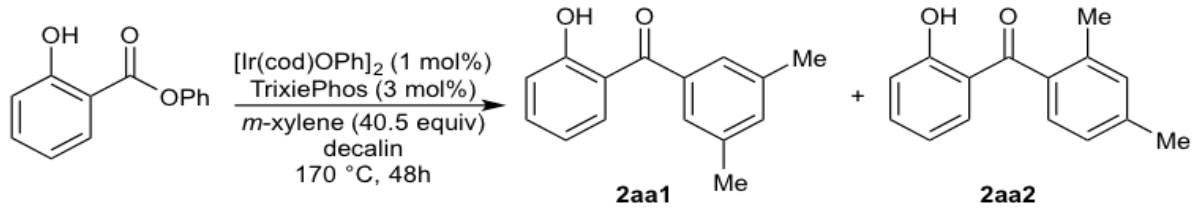
NAS1-85/60
0.29M Phenyl Salicylate
500.13
294.0
C6D6



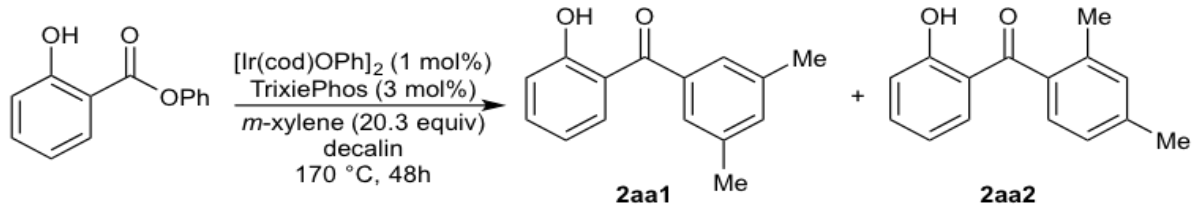
NAS1-85/70
0.4M Phenyl Salicylate
500.13
294.0
C6D6



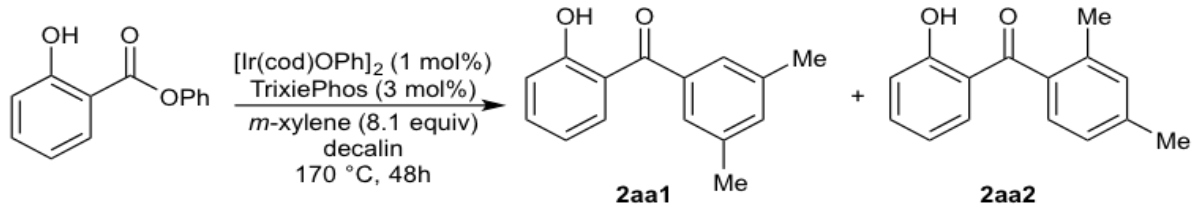
NAS1-75/1
40.5 equiv m-xylene in decalin
500.33
300.0
C6D6



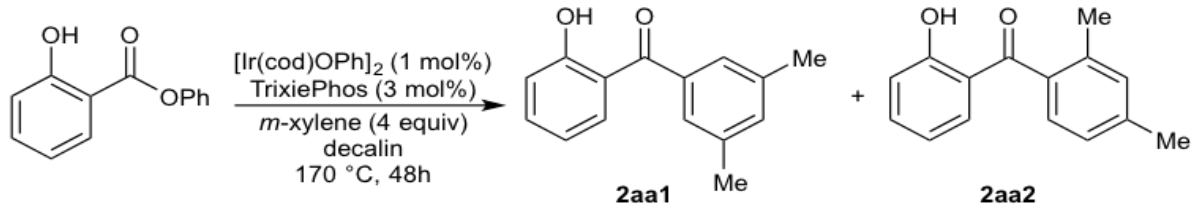
NAS1-75/2
20.3 equiv m-xylene in decalin
500.33
300.0
C6D6



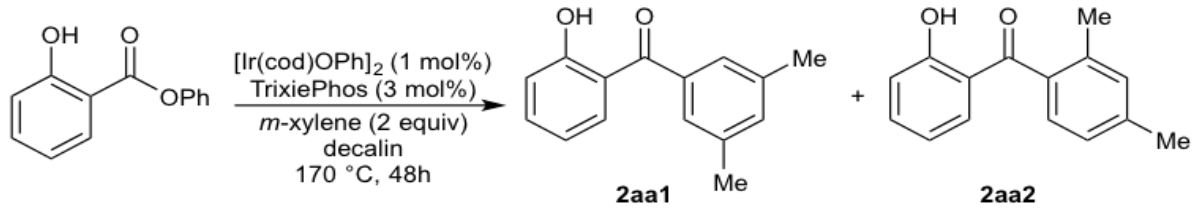
NAS1-75/3
8.1 equiv m-xylene in decalin
500.33
300.0
C6D6



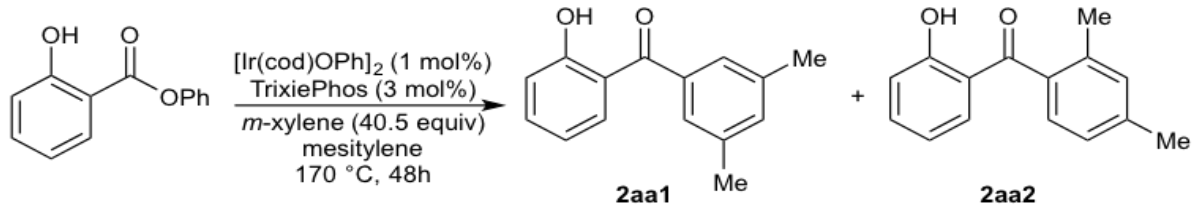
NAS1-75/4
4 equiv m-xylene in decalin
500.33
300.1
C6D6



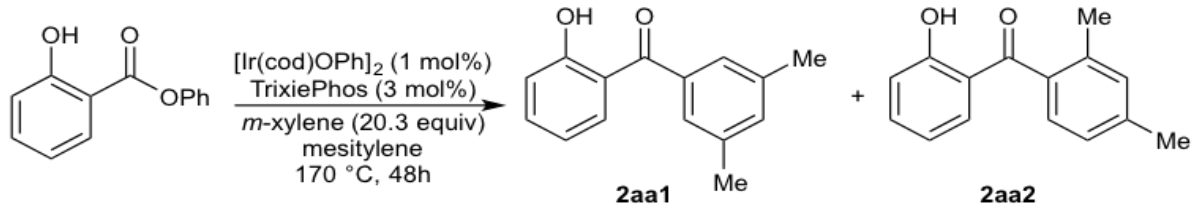
NAS1-75/5
2 equiv m-xylene in decalin
500.33
300.0
C6D6



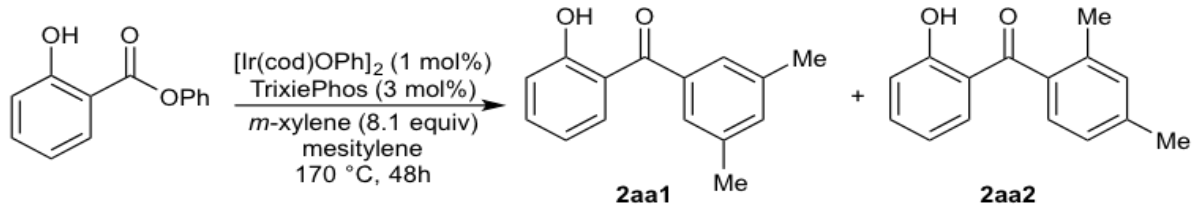
NAS1-75/6
40.5 equiv m-xylene in mesitylene
500.33
300.0
C6D6



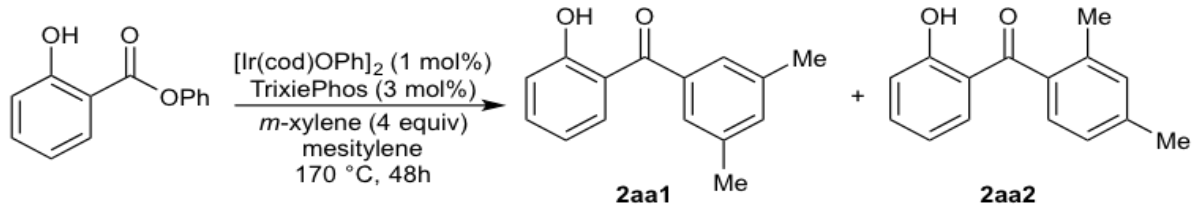
NAS1-75/7
20.3 equiv m-xylene in mesitylene
500.33
300.1
C6D6



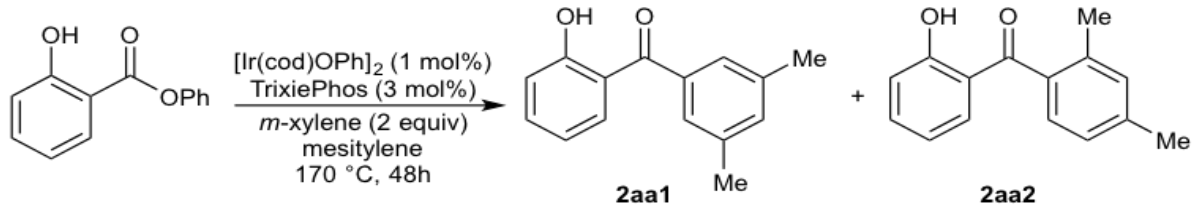
NAS1-75/8
8.1 equiv m-xylene in mesitylene
500.33
300.1
C6D6



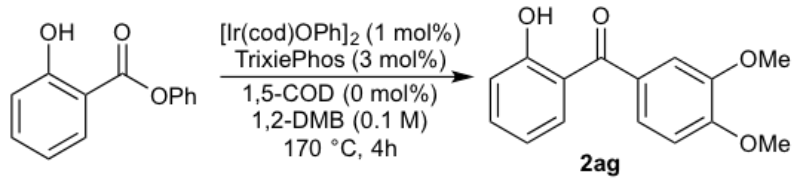
NAS1-75/9
4 equiv m-xylene in mesitylene
500.33
300.1
C6D6



NAS1-75/10
2 equiv m-xylene in mesitylene
500.33
300.1
C6D6

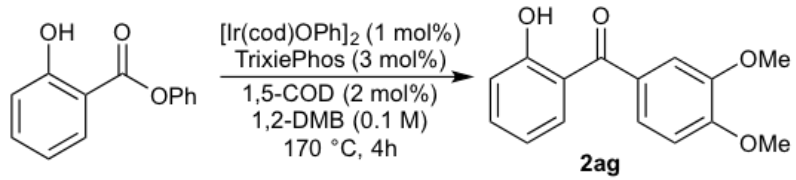


NAS1-178.10.fid
0% COD
500.13
296.0
CDCl3



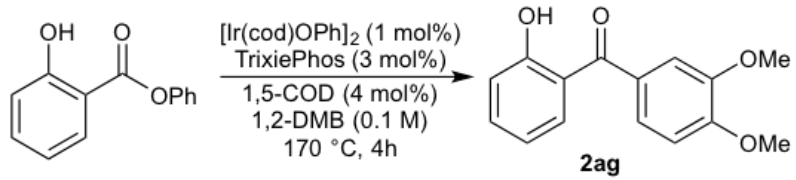
2ag

NAS1-178.20.fid
2% COD
500.13
296.0
CDCl3



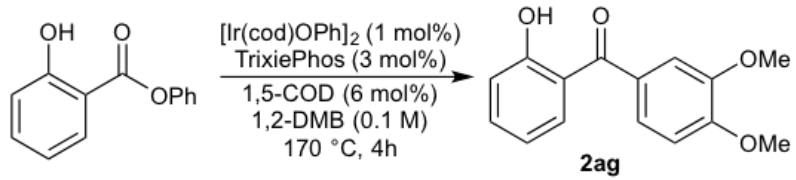
2ag

NAS1-178.30.fid
4% COD
500.13
296.0
CDCl3



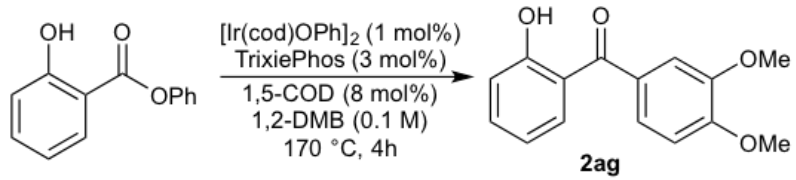
2ag

NAS1-178.40.fid
6% COD
500.13
296.0
CDCl3



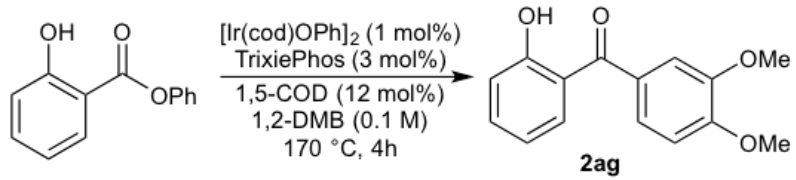
2ag

NAS1-178.50.fid
8% COD
500.13
296.0
CDCl3



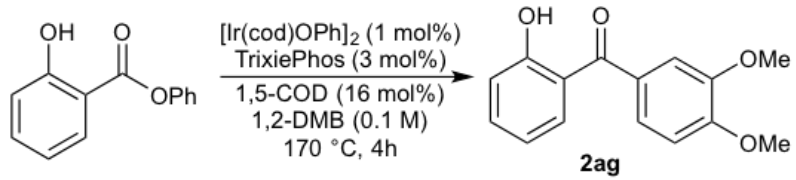
2ag

NAS1-178.60.fid
12% COD
500.13
296.0
CDCl3



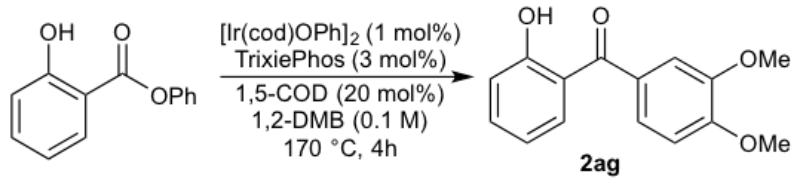
2ag

NAS1-178.70.fid
16% COD
500.13
296.0
CDCl3



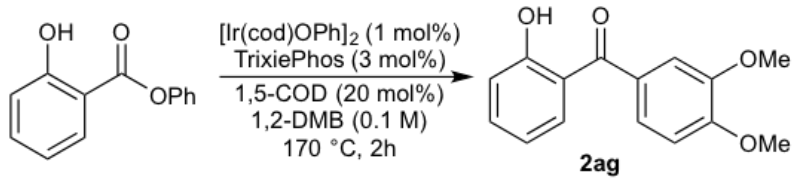
2ag

NAS1-178.80.fid
20% COD
500.13
296.0
CDCl3



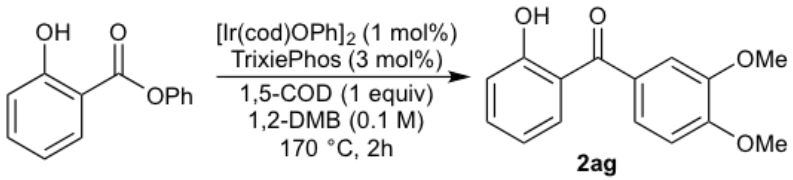
2ag

cdocba-160726-2.10.fid
20% COD for 2 hours
400.13
298.0
CDCl3
Yield in mmol rel. to CH2Br2



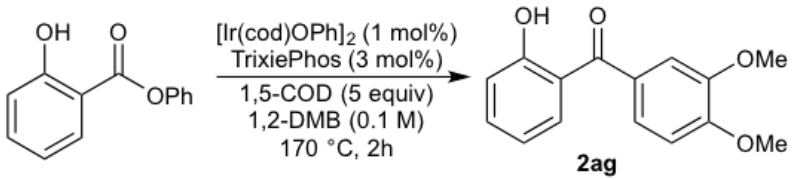
2ag

cdocba-160726-13.10.fid
1 equiv COD for 2 hours
400.13
298.0
CDCl3
Yield in mmol rel. to CH2Br2



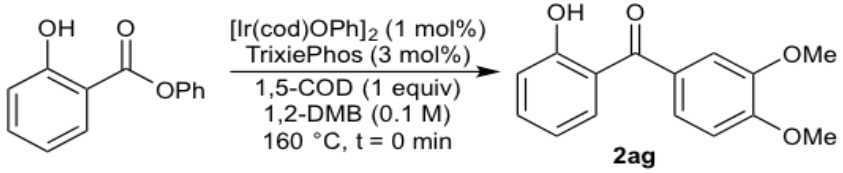
2ag

cdocba-160726-14.10.fid
5 equiv COD for 2 hours
400.13
298.0
CDCl3
Yield in mmol rel. to CH2Br2



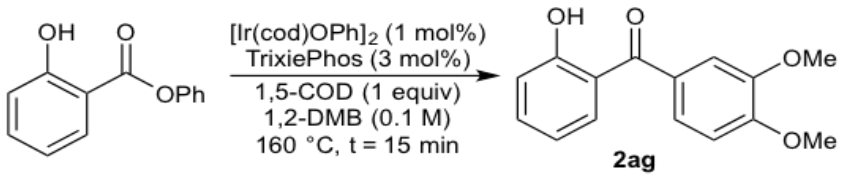
2ag

NAS1-236_A-t0/1
t=0 COD
399.87
298.0
1,2-DMB



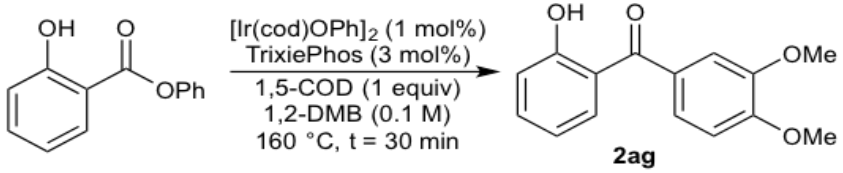
2ag

NAS1-236_A-15m/1
t=15min COD
399.87
298.0
1,2-DMB



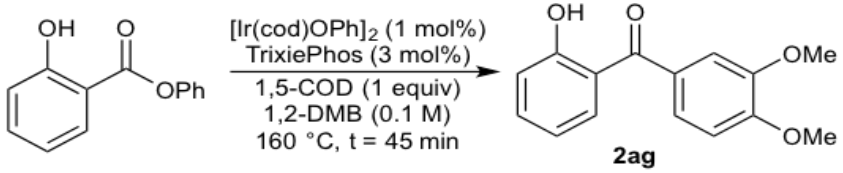
2ag

NAS1-236_A-30m/1
t=30min COD
399.87
298.0
1,2-DMB



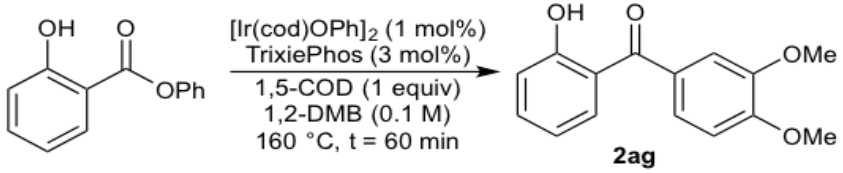
2ag

NAS1-236_A-45m/1
t=45min COD
399.87
298.0
1,2-DMB



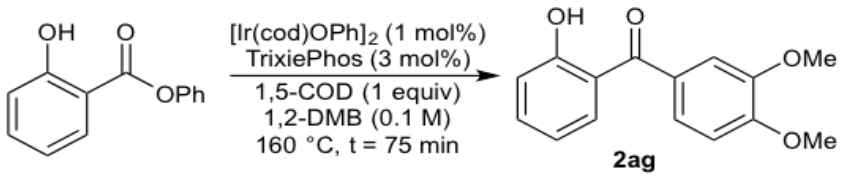
2ag

NAS1-236_A-60m/1
t=60min COD
399.87
298.0
1,2-DMB

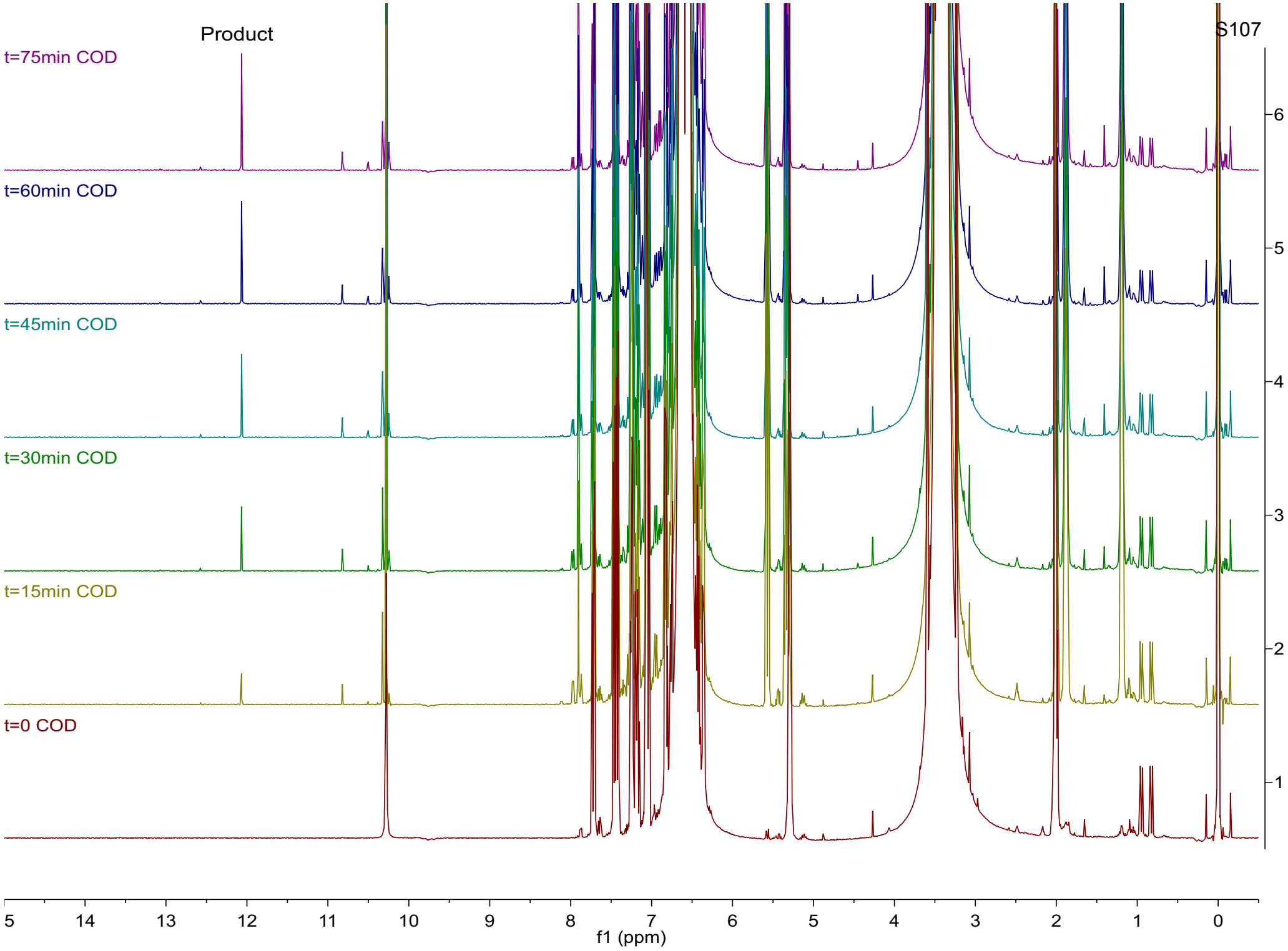


2ag

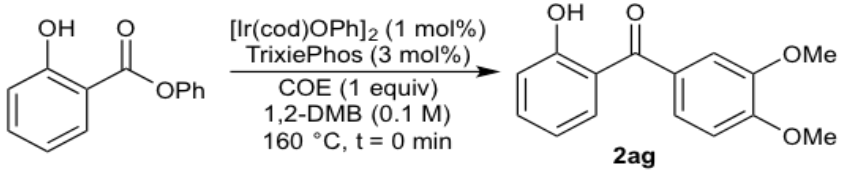
NAS1-236_A-75m/1
t=75min COD
399.87
298.0
1,2-DMB



2ag

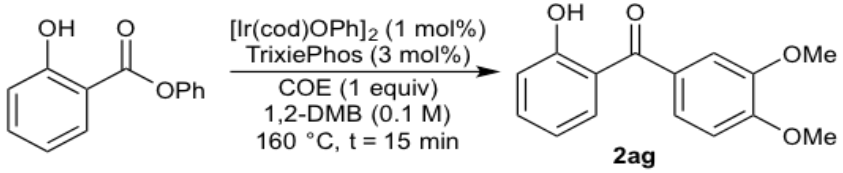

Product
S107
t=75min COD
t=60min COD
t=45min COD
t=30min COD
t=15min COD
t=0 COD
6
5
4
3
2
1
5
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
f1 (ppm)

NAS1-236_C-t0/1
t=0 COE
399.87
298.0
1,2-DMB



2ag

NAS1-236_C-15m/1
t=15min coe
399.87
298.0
1,2-DMB



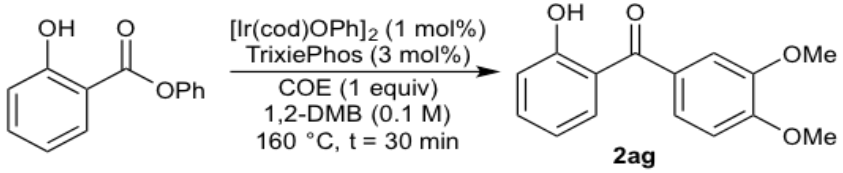
2ag

1.74

900.00

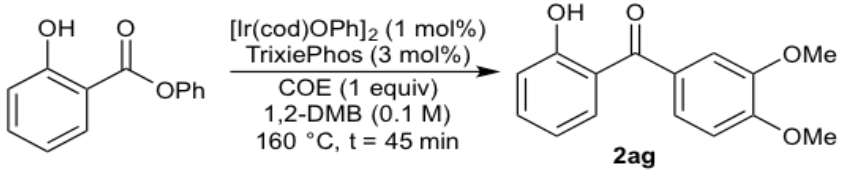
f1 (ppm)

NAS1-236_C-30m/1
t=30min COE
399.87
298.0
1,2-DMB



2ag

NAS1-236_C-45m/1
t=45min COE
399.87
298.0
1,2-DMB



2ag

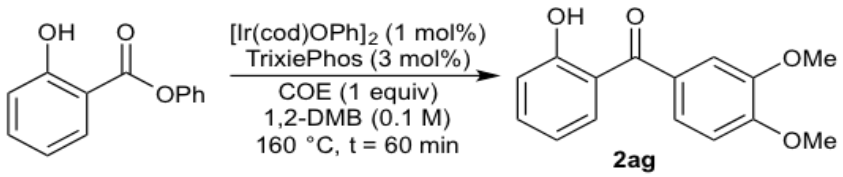
S111

4.50

900.00

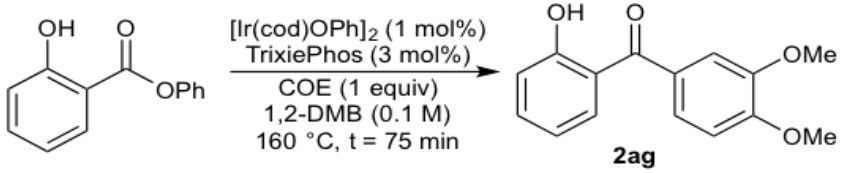
f1 (ppm)

NAS1-236_C-60m/1
t=60min COE
399.87
298.0
1,2-DMB



2ag

NAS1-236_C-75m/1
t=75min COE
399.87
298.0
1,2-DMB

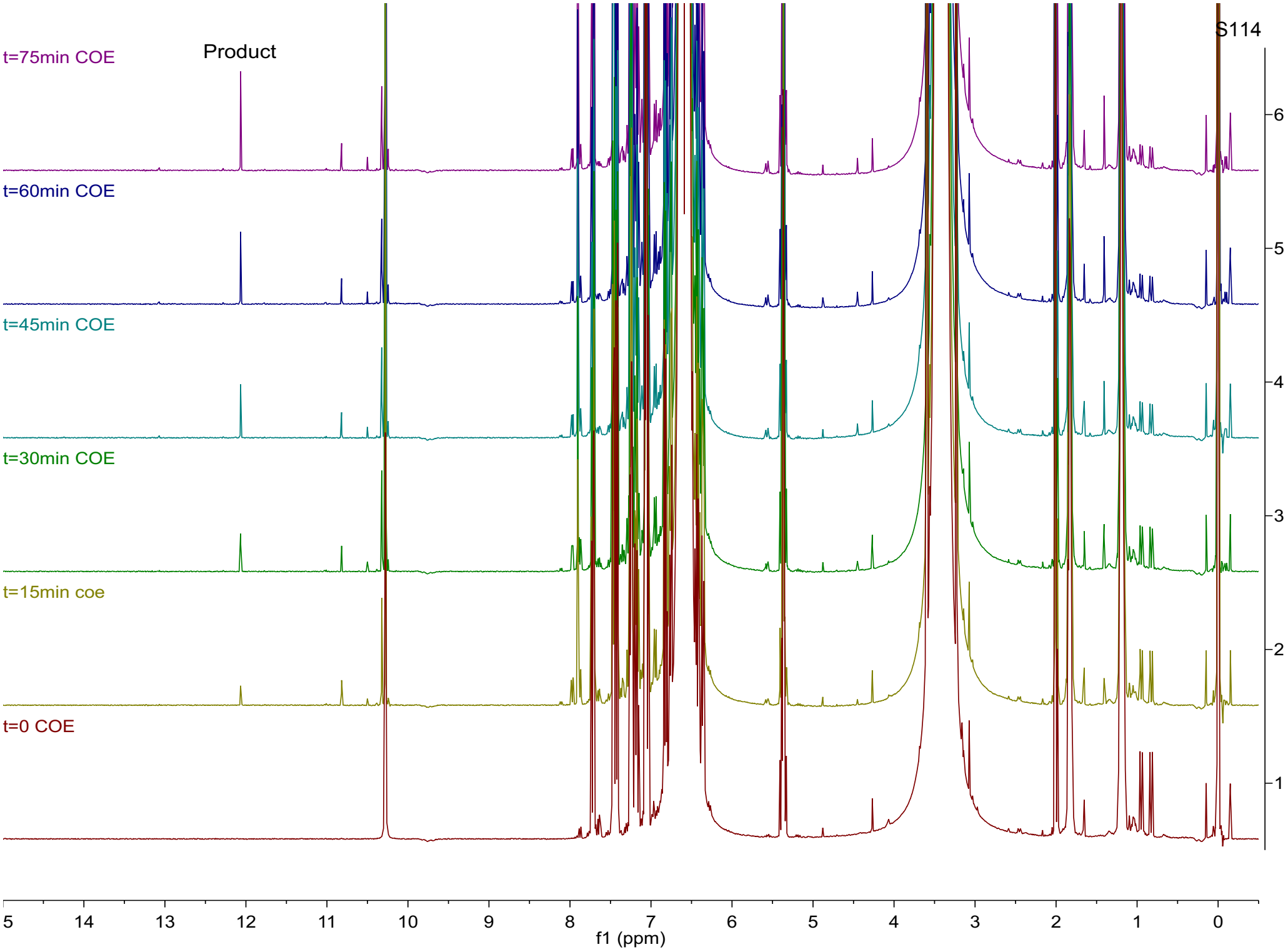


2ag

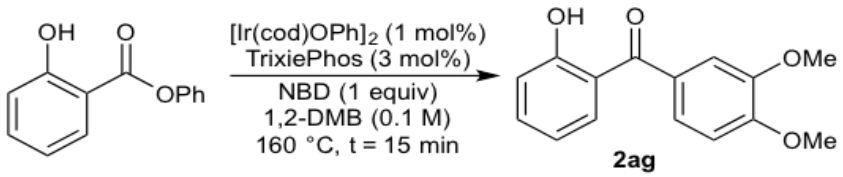
7.60

900.00

f1 (ppm)

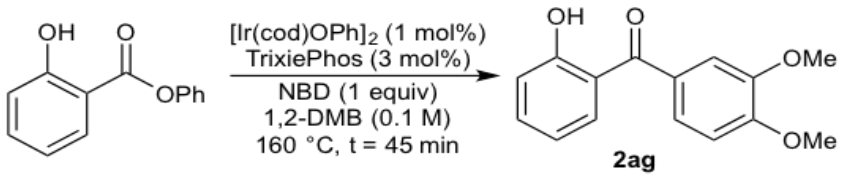

t=75min COE
Product
t=60min COE
t=45min COE
t=30min COE
t=15min coe
t=0 COE
6
5
4
3
2
1
5
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
f1 (ppm)

NAS1-237_D-15m/1
15 min NBD
399.87
298.0
1,2-DMB

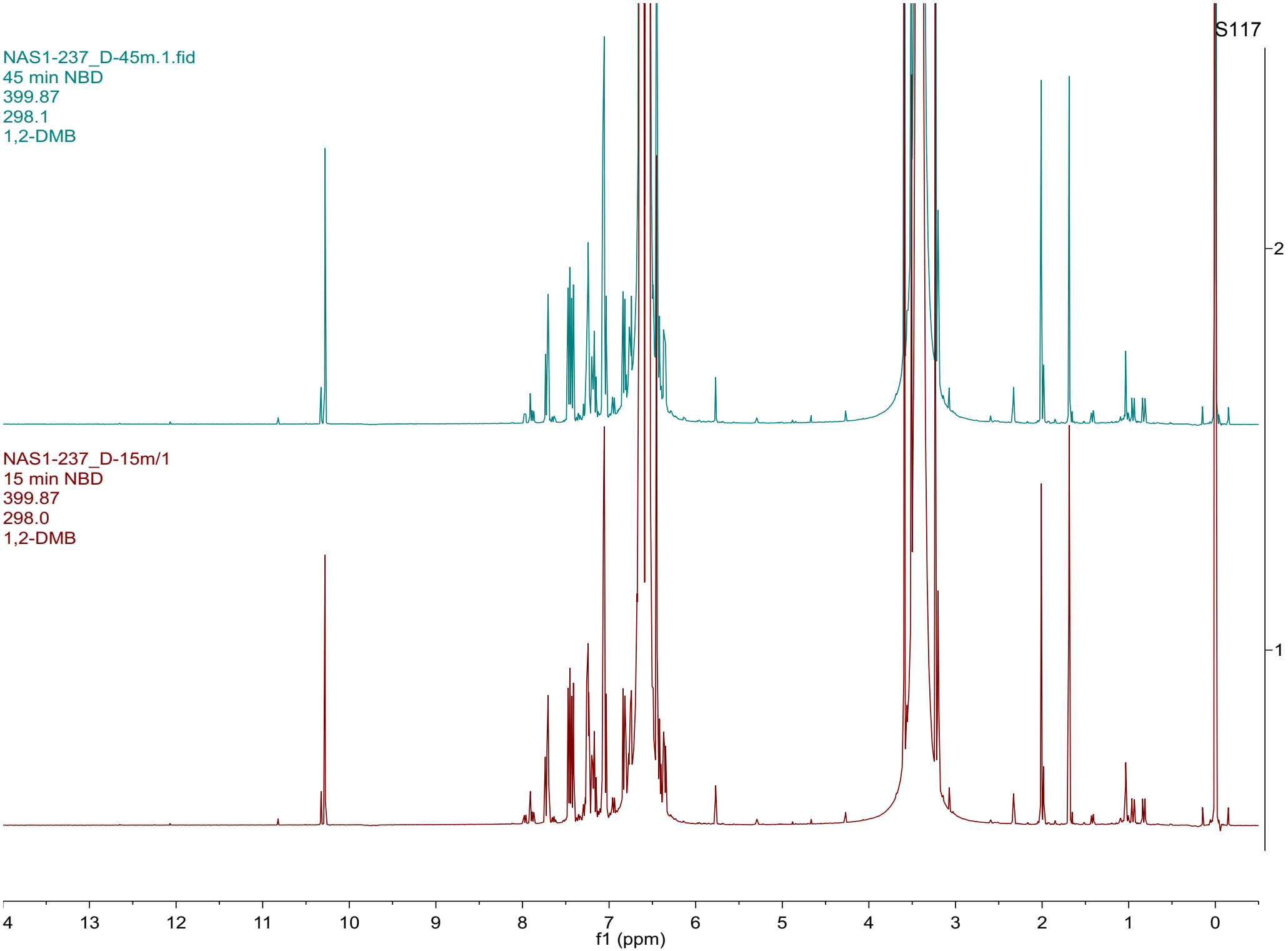


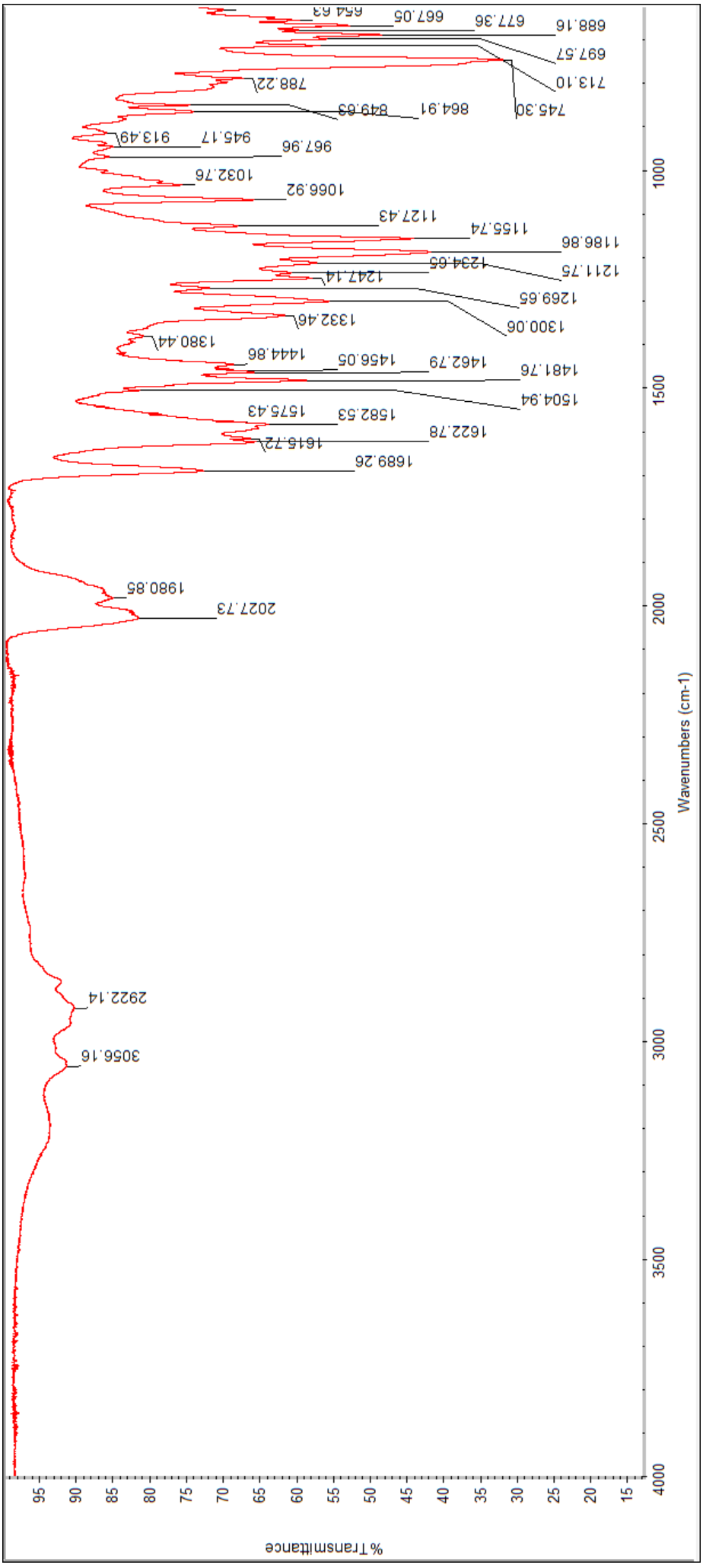
2ag

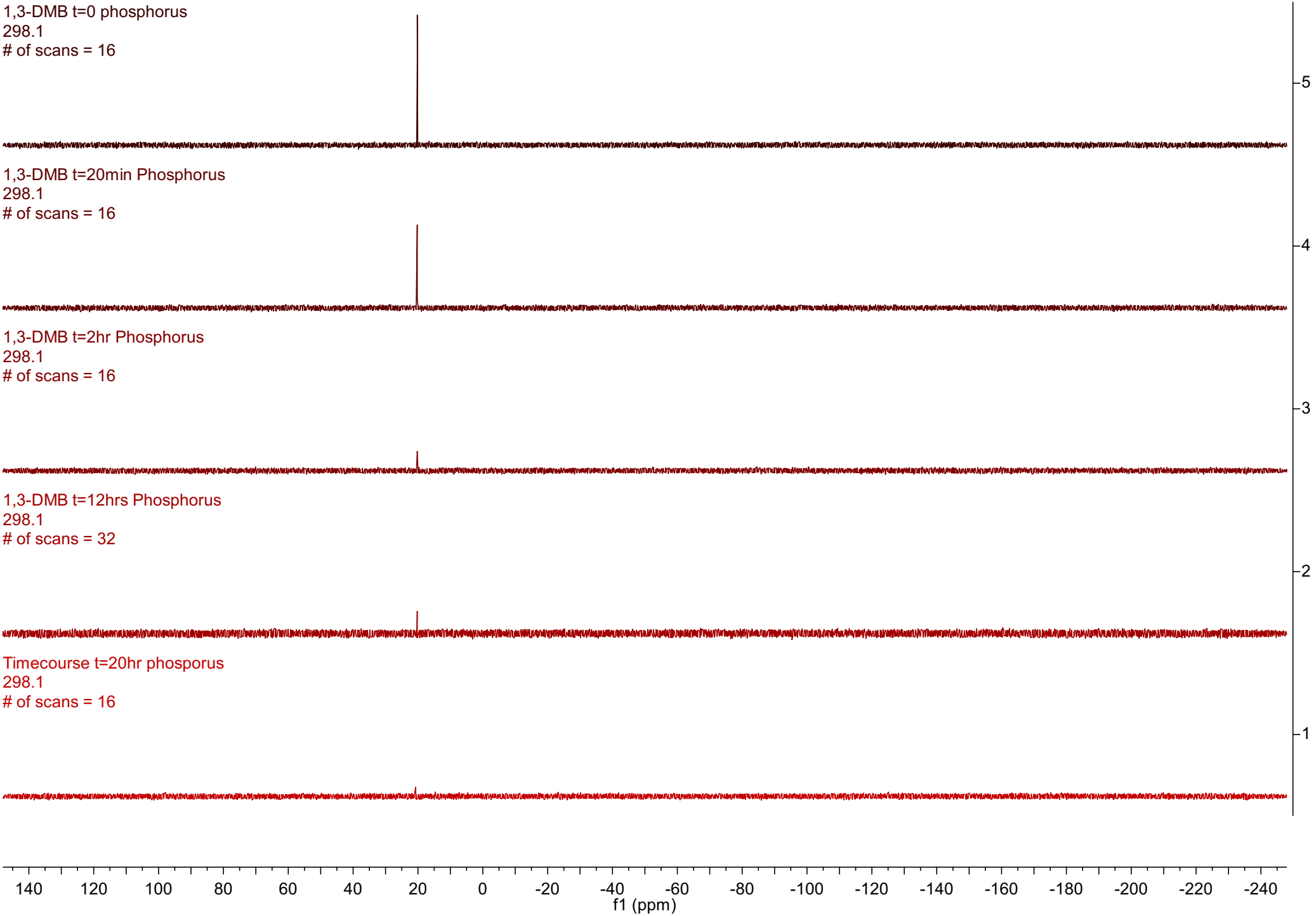
NAS1-237_D-45m.1.fid
45 min NBD
399.87
298.1
1,2-DMB



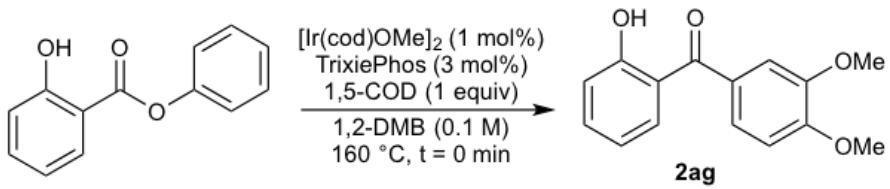
2ag


NAS1-237_D-45m.1.fid
45 min NBD
399.87
298.1
1,2-DMB
NAS1-237_D-15m/1
15 min NBD
399.87
298.0
1,2-DMB
2
1
4
13
12
11
10
9
8
7
6
5
4
3
2
1
0
f1 (ppm)


%Transmittance
Wavenumbers (cm-1)
4000
3500
3000
2500
2000
1500
1000
95
90
85
80
75
70
65
60
55
50
45
40
35
30
25
20
15
2922.14
3056.16
1980.85
2027.73
1689.26
1622.78
1615.72
1582.53
1575.43
1504.94
1481.76
1462.79
1456.05
1444.86
1380.44
1332.46
1300.06
1269.65
1247.14
1234.65
1211.75
1186.86
1155.74
1127.43
1066.92
1032.76
967.96
945.17
913.49
864.91
849.63
788.22
745.30
713.10
697.57
688.16
677.36
667.05
654.63

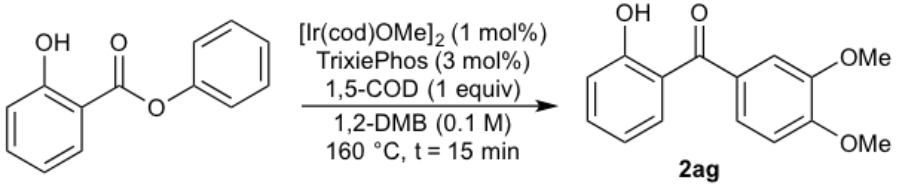

1,3-DMB t=0 phosphorus
298.1
# of scans = 16
1,3-DMB t=20min Phosphorus
298.1
# of scans = 16
1,3-DMB t=2hr Phosphorus
298.1
# of scans = 16
1,3-DMB t=12hrs Phosphorus
298.1
# of scans = 32
Timecourse t=20hr phosporus
298.1
# of scans = 16
5
4
3
2
1
140 120 100 80 60 40 20 0 -20 -40 -60 -80 -100 -120 -140 -160 -180 -200 -220 -240
f1 (ppm)

PhSali t=0 min
399.87
298.0
1,2-DMB



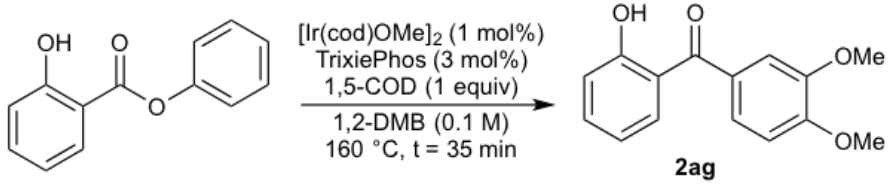
2ag

PhSali t=15 min
399.87
298.0
1,2-DMB



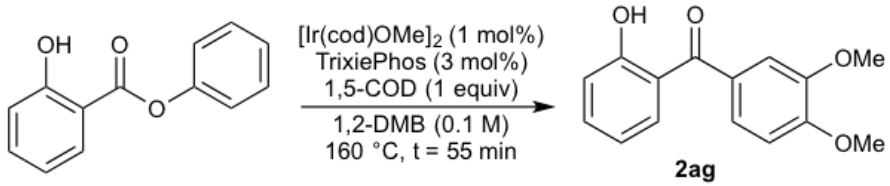
2ag

PhSali t=35 min
399.87
298.0
1,2-DMB



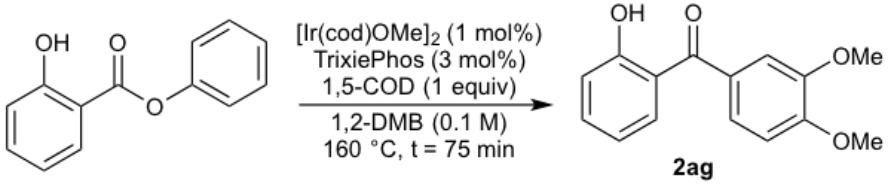
2ag

PhSali t=55 min
399.87
298.0
1,2-DMB



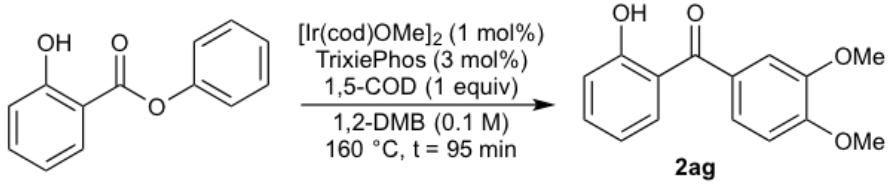
2ag

PhSali t=75 min
399.87
298.0
1,2-DMB



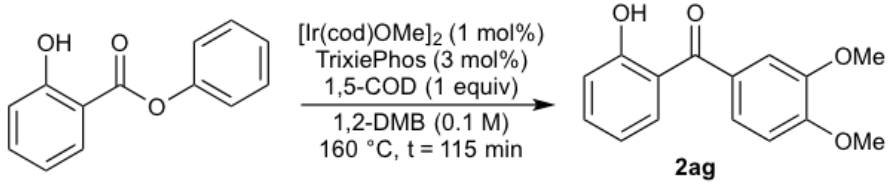
2ag

PhSali t=95 min
399.87
298.0
1,2-DMB



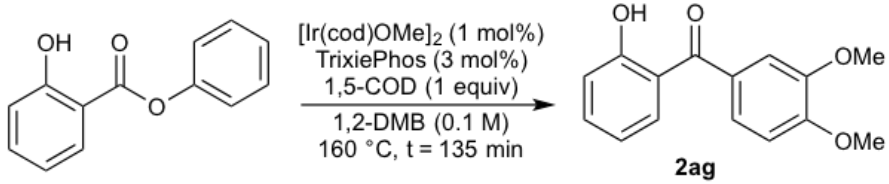
2ag

PhSali t=115 min
399.87
298.0
1,2-DMB

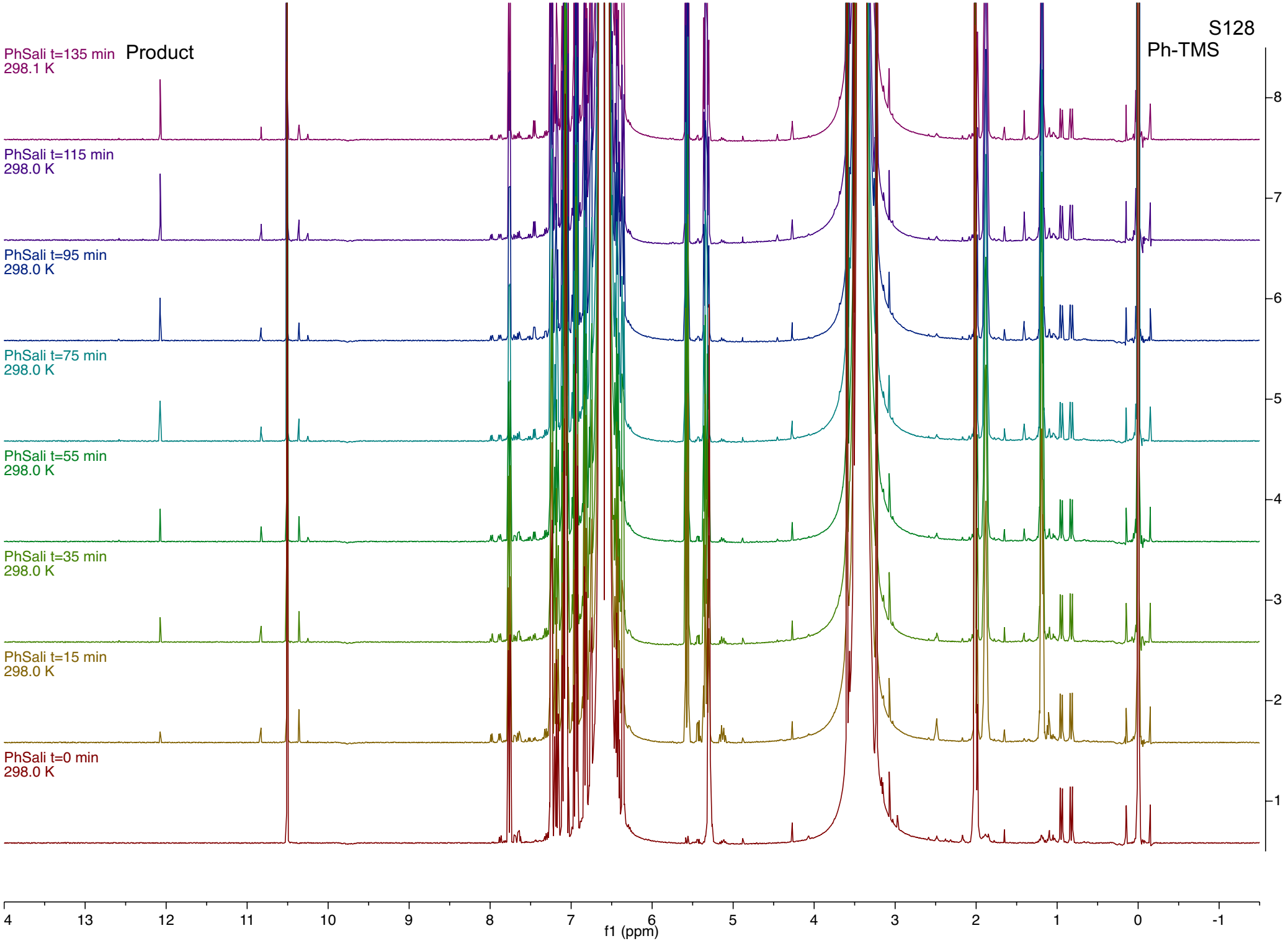


2ag

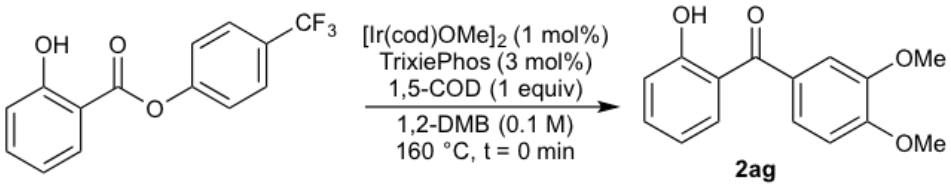
PhSali t=135 min
399.87
298.1
1,2-DMB



2ag

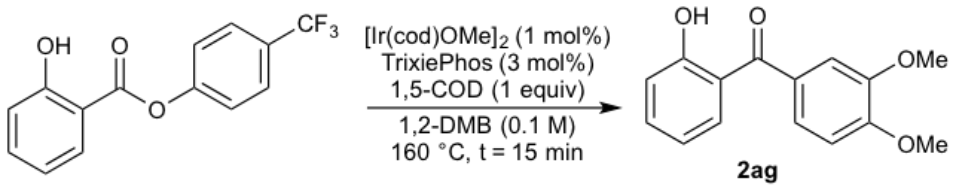

PhSali t=135 min
298.1 K
Product
Ph-TMS
PhSali t=115 min
298.0 K
PhSali t=95 min
298.0 K
PhSali t=75 min
298.0 K
PhSali t=55 min
298.0 K
PhSali t=35 min
298.0 K
PhSali t=15 min
298.0 K
PhSali t=0 min
298.0 K
8
7
6
5
4
3
2
1
4
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
f1 (ppm)

CF3 PhSali t=0 min
399.87
298.0
1,2-DMB



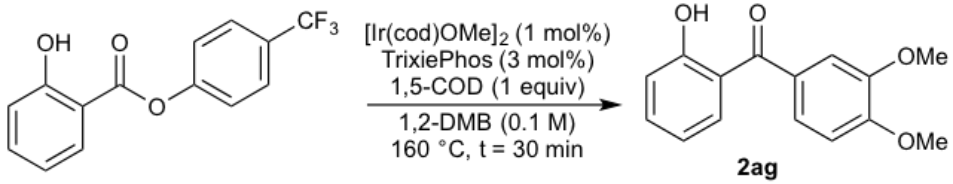
2ag

CF3 PhSali t=15 min
399.87
298.0
1,2-DMB



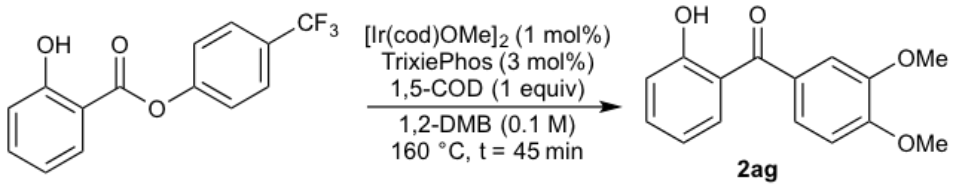
2ag

CF3 PhSali t=30 min
399.87
298.0
1,2-DMB



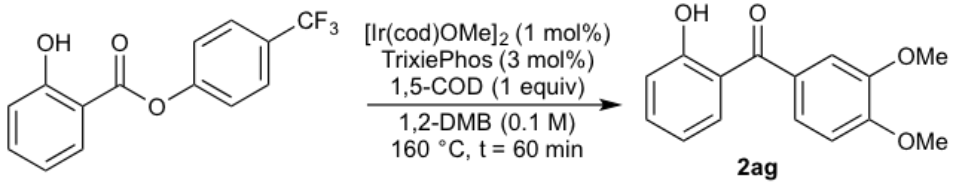
2ag

CF3 PhSali t=45 min
399.87
298.0
1,2-DMB



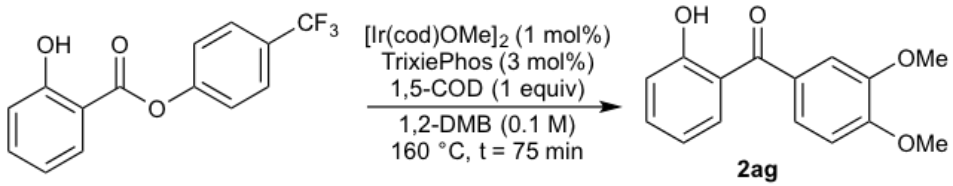
2ag

CF3 PhSali t=60 min
399.87
298.0
1,2-DMB

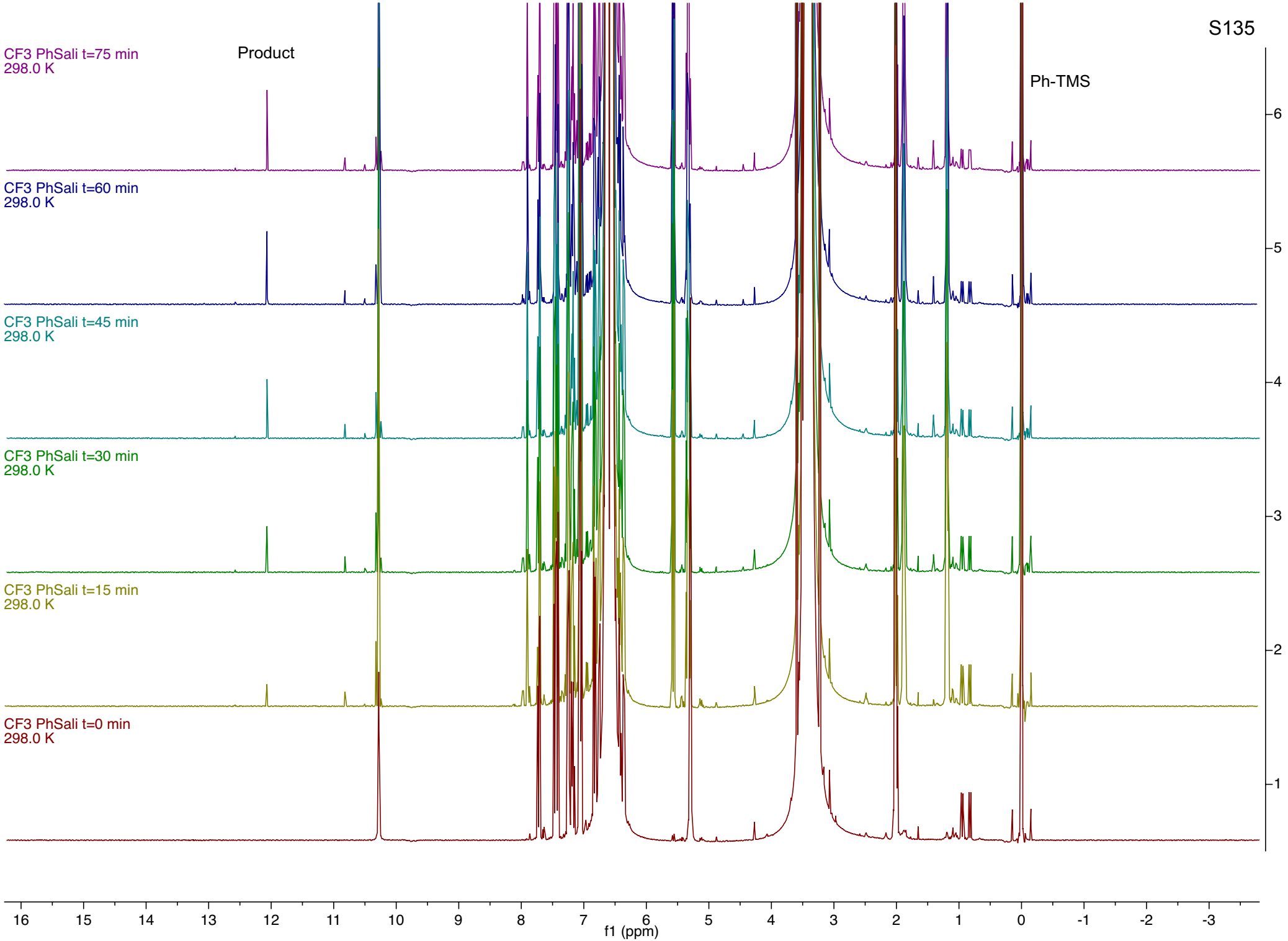


2ag

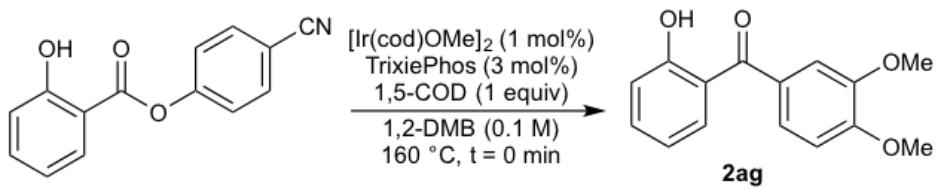
CF3 PhSali t=75 min
399.87
298.0
1,2-DMB



2ag

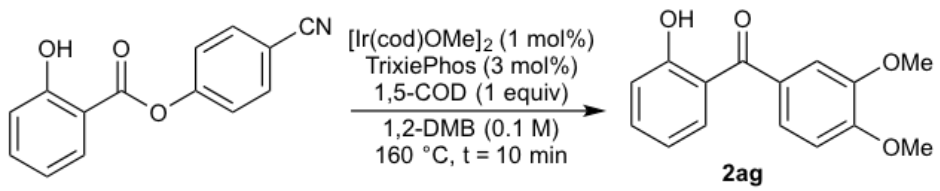

CF3 PhSali t=75 min
298.0 K
Product
Ph-TMS
CF3 PhSali t=60 min
298.0 K
CF3 PhSali t=45 min
298.0 K
CF3 PhSali t=30 min
298.0 K
CF3 PhSali t=15 min
298.0 K
CF3 PhSali t=0 min
298.0 K
6
5
4
3
2
1
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
f1 (ppm)

4-CN t=0
399.87
300.6
1,2-DMB



2ag

4-CN, t=10min
399.87
298.0
1,2-DMB



2ag

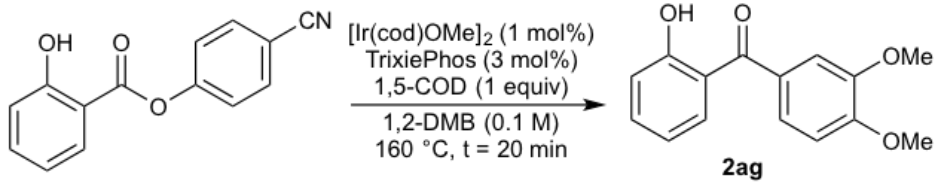
5.48

900.00

13.5 13.0 12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1

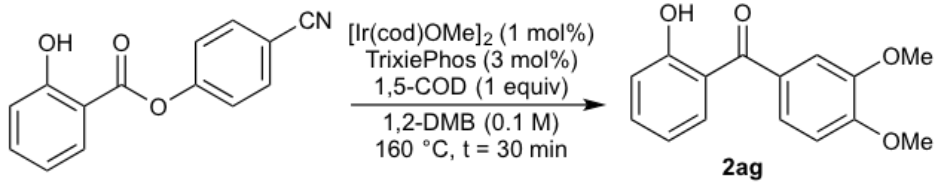
f1 (ppm)

4-CN, t=20min
399.87
298.0
1,2-DMB



2ag

4-CN, t=30min
399.87
298.1
1,2-DMB



2ag

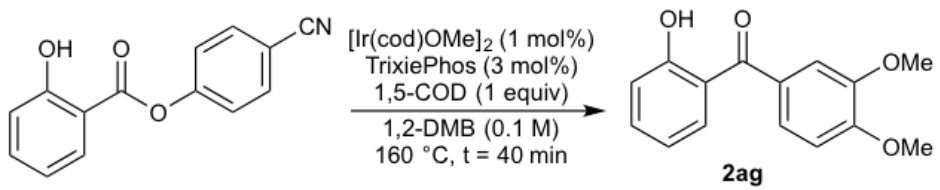
16.30

900.00

13.5 13.0 12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1

f1 (ppm)

4-CN, t=40min
399.87
298.0
1,2-DMB



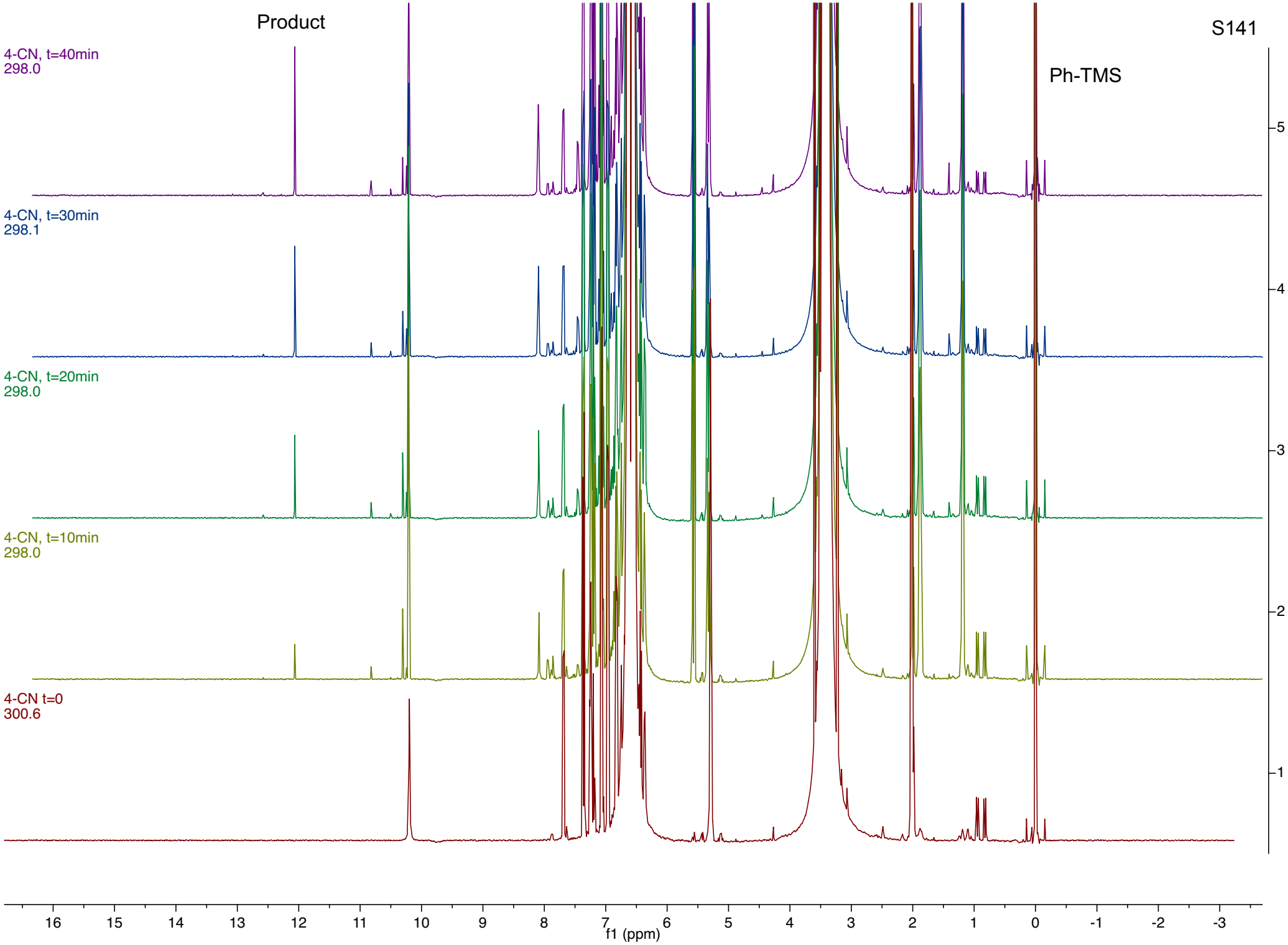
2ag

22.67

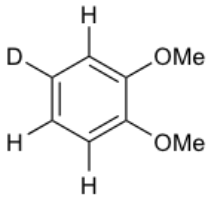
900.00

13.5 13.0 12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1

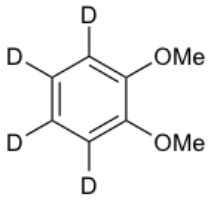
f1 (ppm)

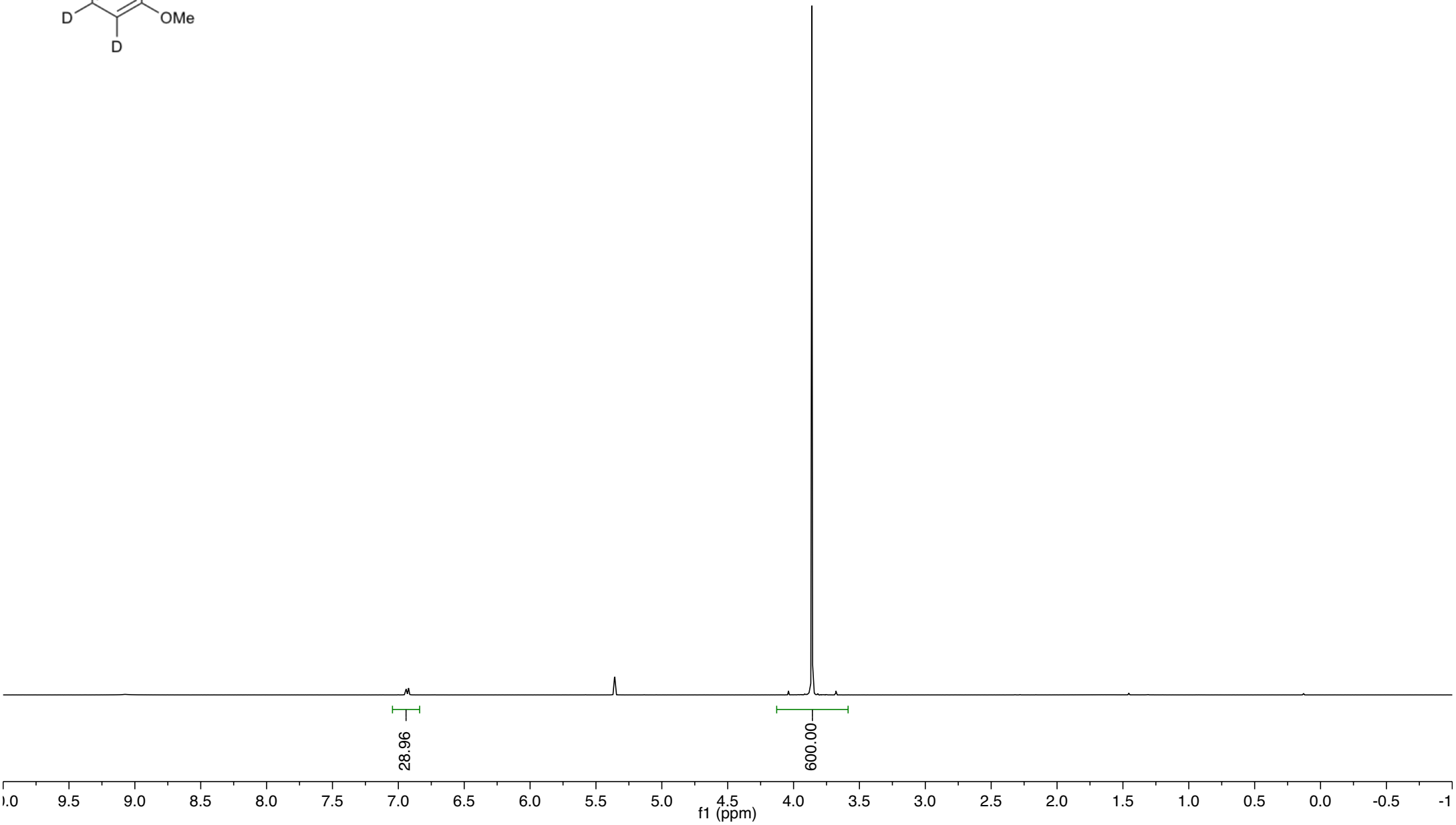

Product
S141
Ph-TMS
4-CN, t=40min
298.0
4-CN, t=30min
298.1
4-CN, t=20min
298.0
4-CN, t=10min
298.0
4-CN t=0
300.6
5
4
3
2
1
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
-3
f1 (ppm)

Purified d1-veratrole
298.0

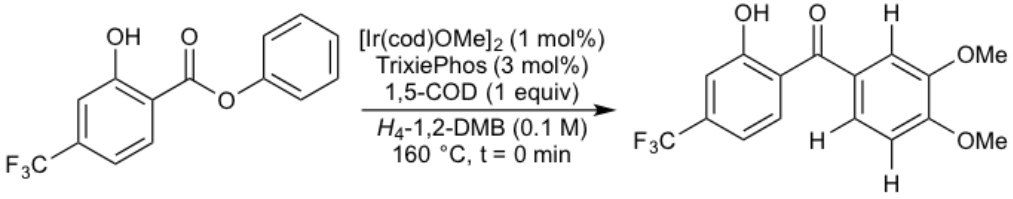


d4-veratrole
298.0 K

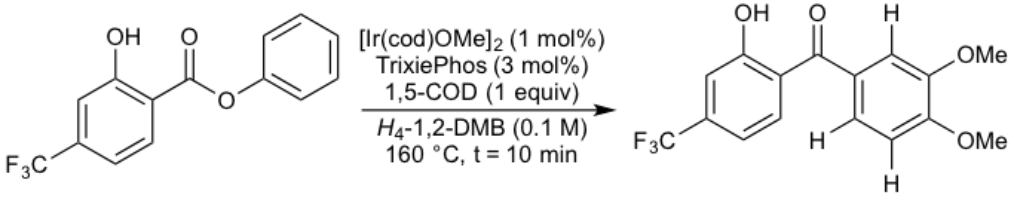




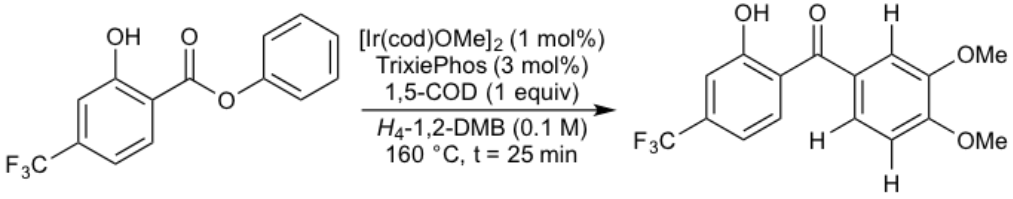
Proteo Experiment 1, t = 0 min
19F
298.1



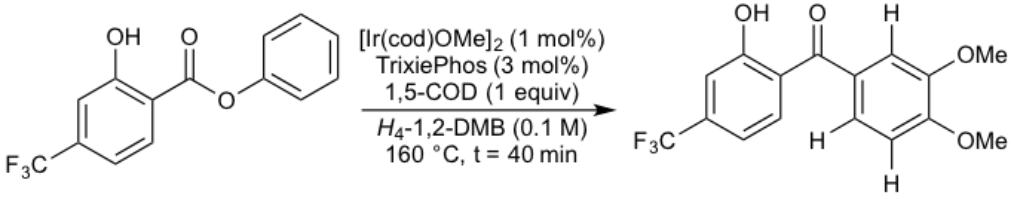
Proteo Experiment 1, t = 10 min
19F
298.0



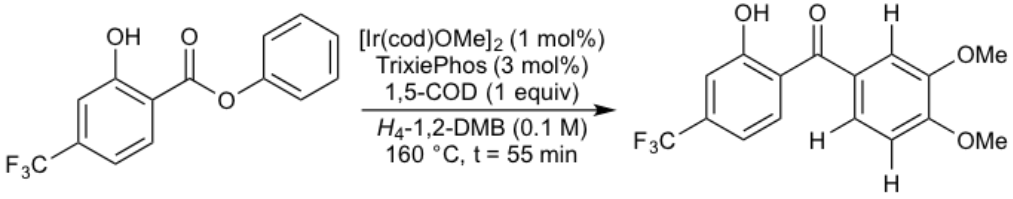
Proteo Experiment 1, t = 25 min
19F
298.0



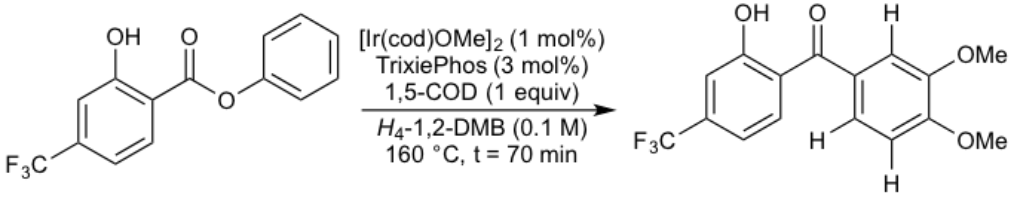
Proteo Experiment 1, t = 40 min
19F
298.0

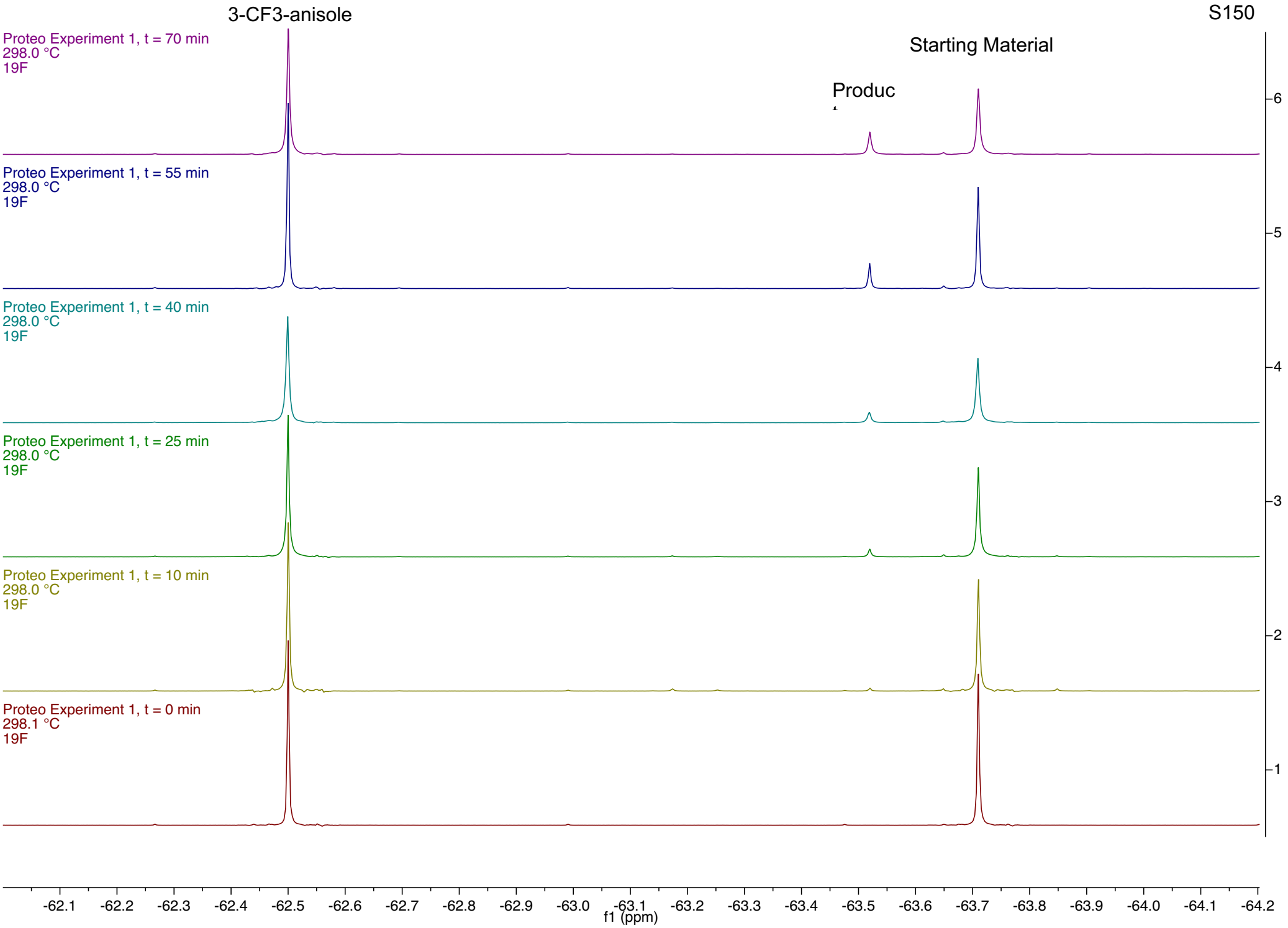


Proteo Experiment 1, t = 55 min
19F
298.0

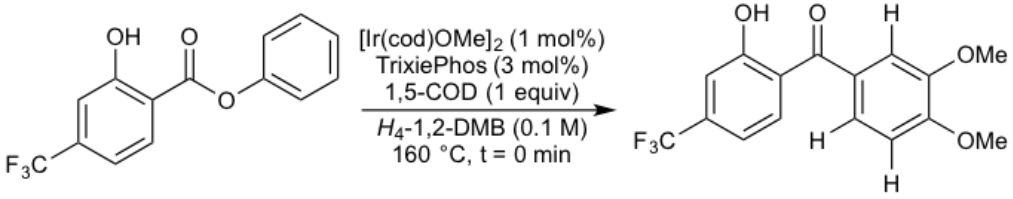


Proteo Experiment 1, t = 70 min
19F
298.0

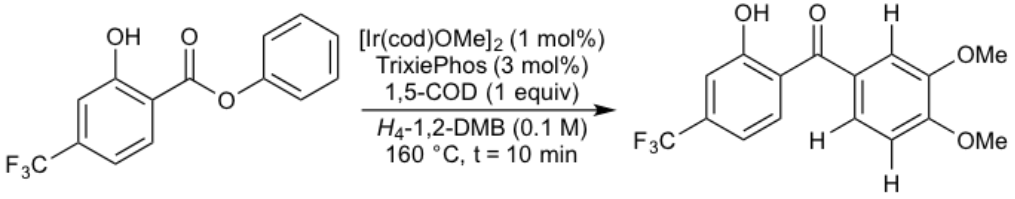



3-CF3-anisole
Starting Material
Produc
Proteo Experiment 1, t = 70 min
298.0 °C
19F
Proteo Experiment 1, t = 55 min
298.0 °C
19F
Proteo Experiment 1, t = 40 min
298.0 °C
19F
Proteo Experiment 1, t = 25 min
298.0 °C
19F
Proteo Experiment 1, t = 10 min
298.0 °C
19F
Proteo Experiment 1, t = 0 min
298.1 °C
19F
6
5
4
3
2
1
-62.1
-62.2
-62.3
-62.4
-62.5
-62.6
-62.7
-62.8
-62.9
-63.0
-63.1
-63.2
-63.3
-63.4
-63.5
-63.6
-63.7
-63.8
-63.9
-64.0
-64.1
-64.2
f1 (ppm)

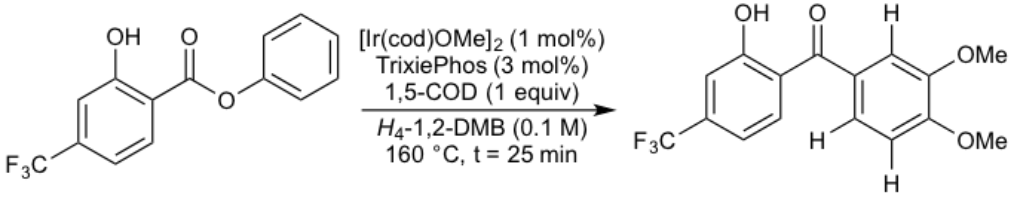
Proteo Experiment 2, t = 0 min
19F
298.0



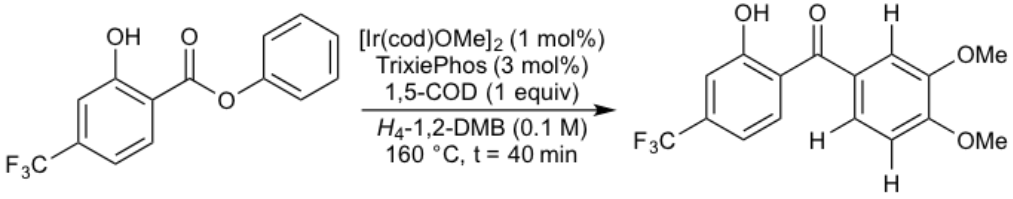
Proteo Experiment 2, t = 10 min
19F
298.0



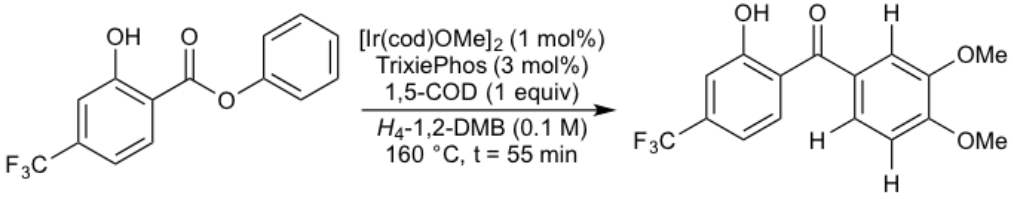
Proteo Experiment 2, t = 25 min
19F
298.0



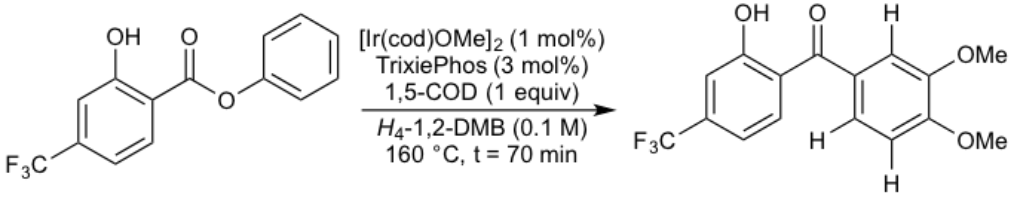
Proteo Experiment 2, t = 40 min
19F
298.0

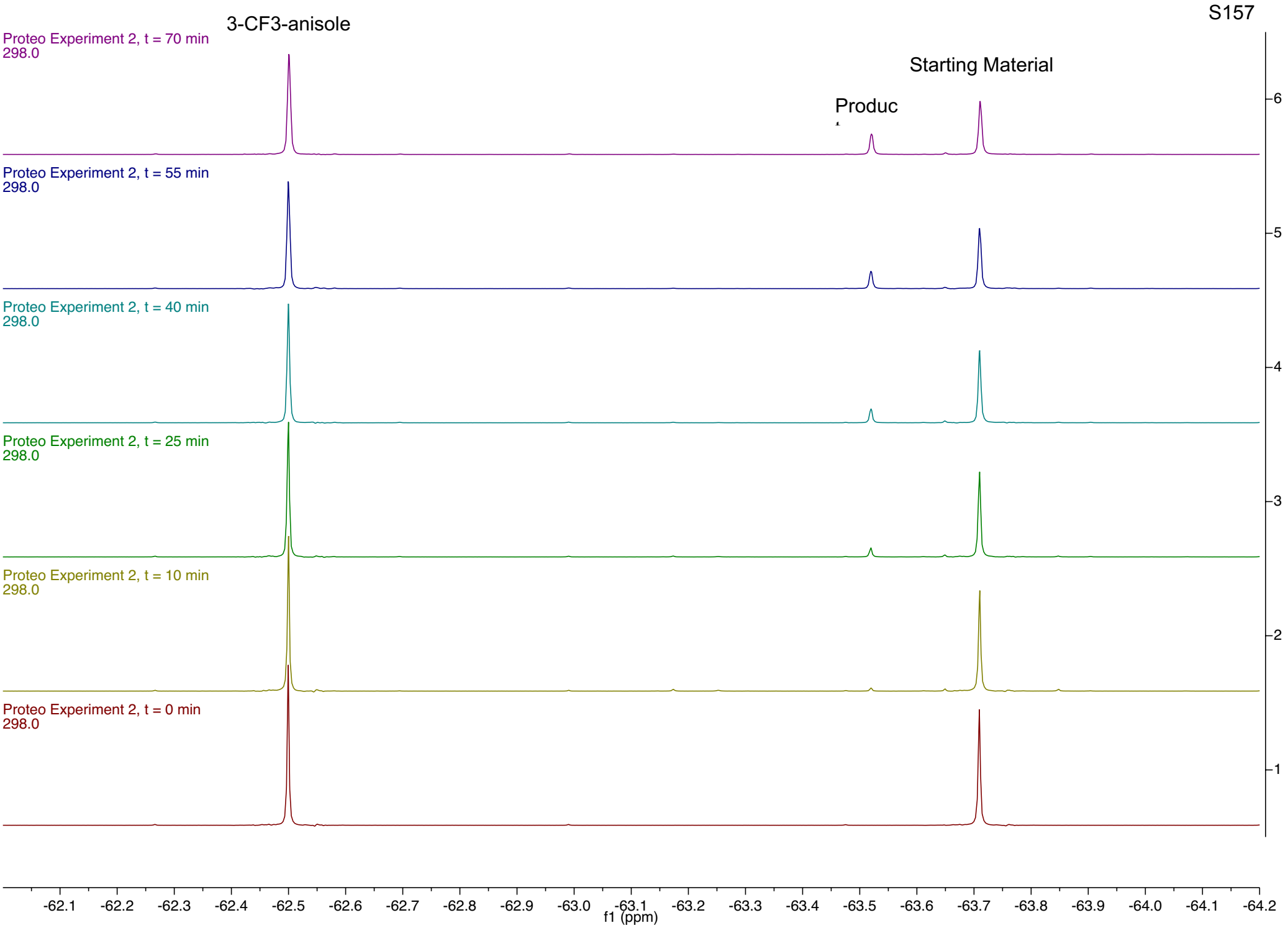


Proteo Experiment 2, t = 55 min
19F
298.0

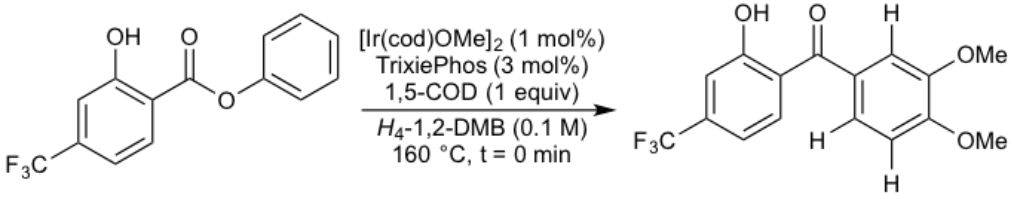


Proteo Experiment 2, t = 70 min
19F
298.0

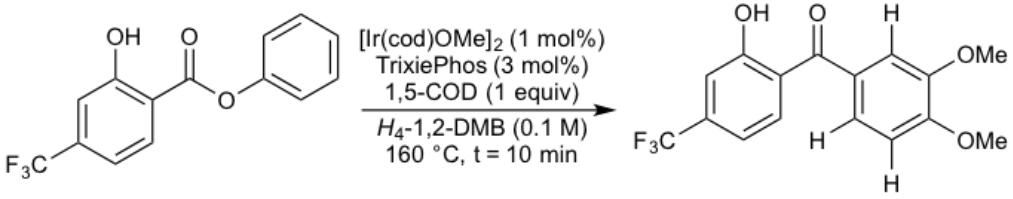



3-CF3-anisole
Starting Material
Produc
Proteo Experiment 2, t = 70 min
298.0
Proteo Experiment 2, t = 55 min
298.0
Proteo Experiment 2, t = 40 min
298.0
Proteo Experiment 2, t = 25 min
298.0
Proteo Experiment 2, t = 10 min
298.0
Proteo Experiment 2, t = 0 min
298.0
6
5
4
3
2
1
-62.1
-62.2
-62.3
-62.4
-62.5
-62.6
-62.7
-62.8
-62.9
-63.0
-63.1
-63.2
-63.3
-63.4
-63.5
-63.6
-63.7
-63.8
-63.9
-64.0
-64.1
-64.2
f1 (ppm)

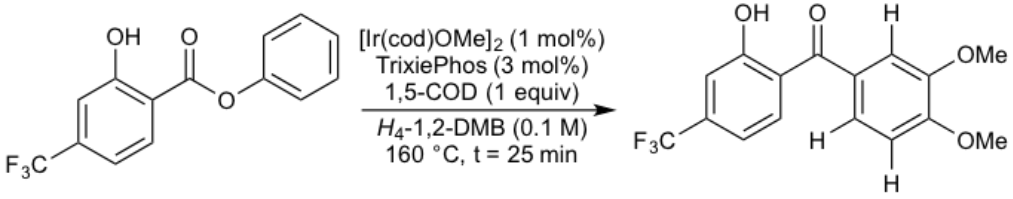
Proteo Experiment 3, t = 0 min
19F
298.1



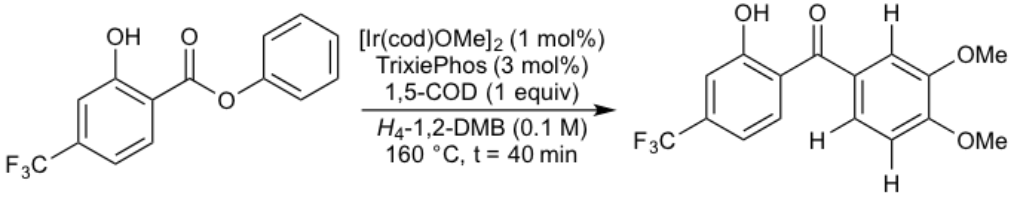
Proteo Experiment 3, t = 10 min
19F
298.0



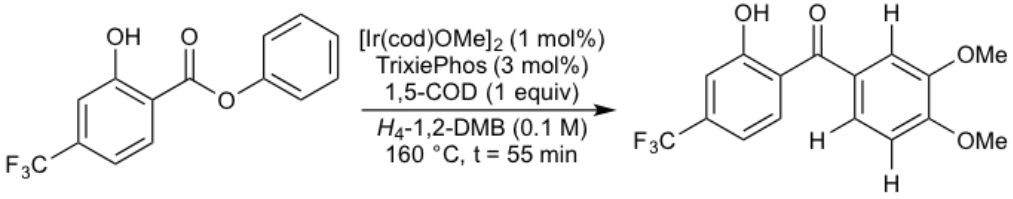
Proteo Experiment 3, t = 25 min
19F
298.0



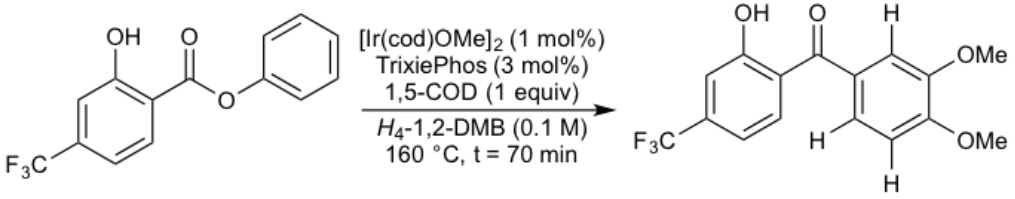
Proteo Experiment 3, t = 40 min
19F
298.0

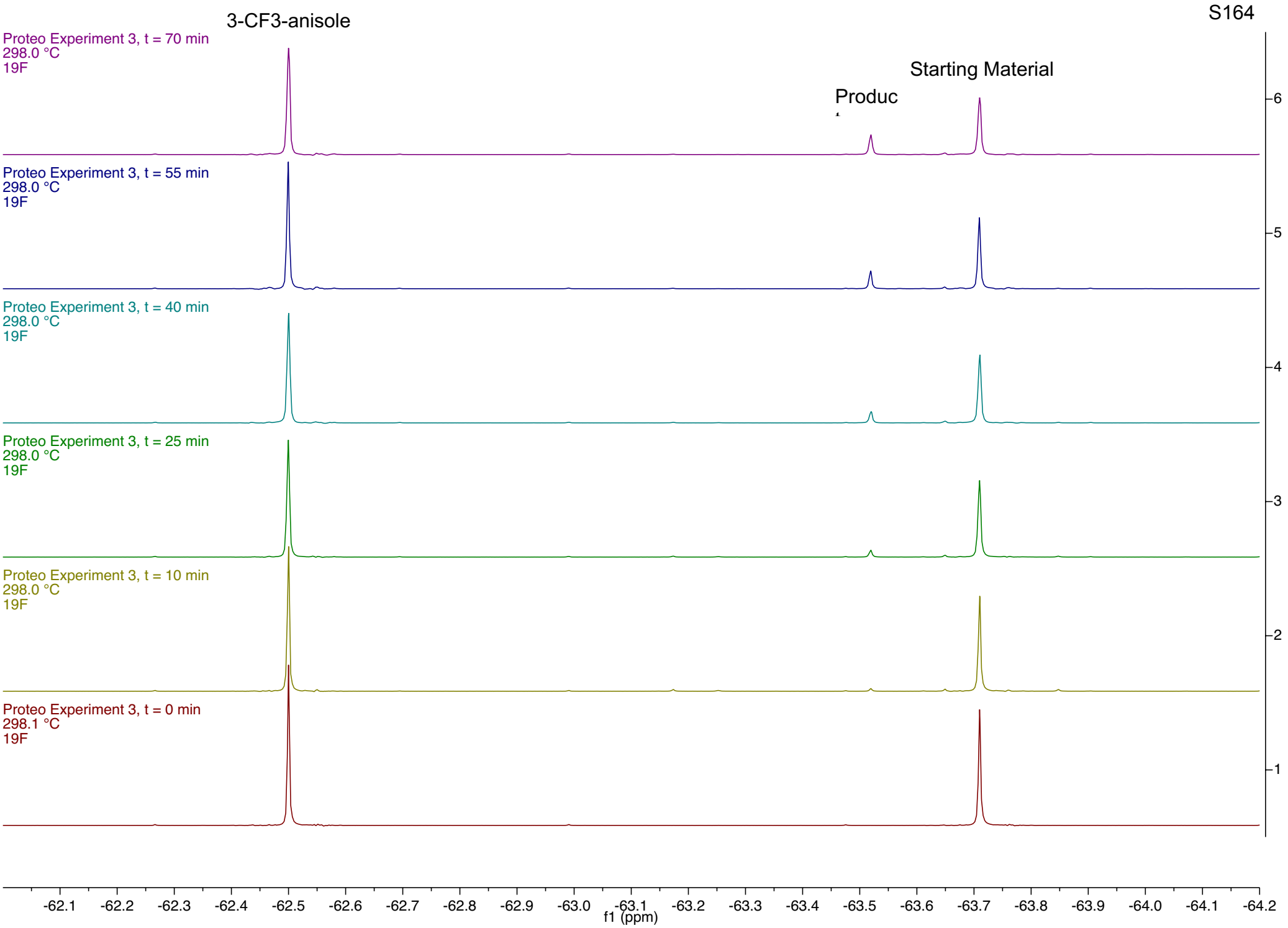


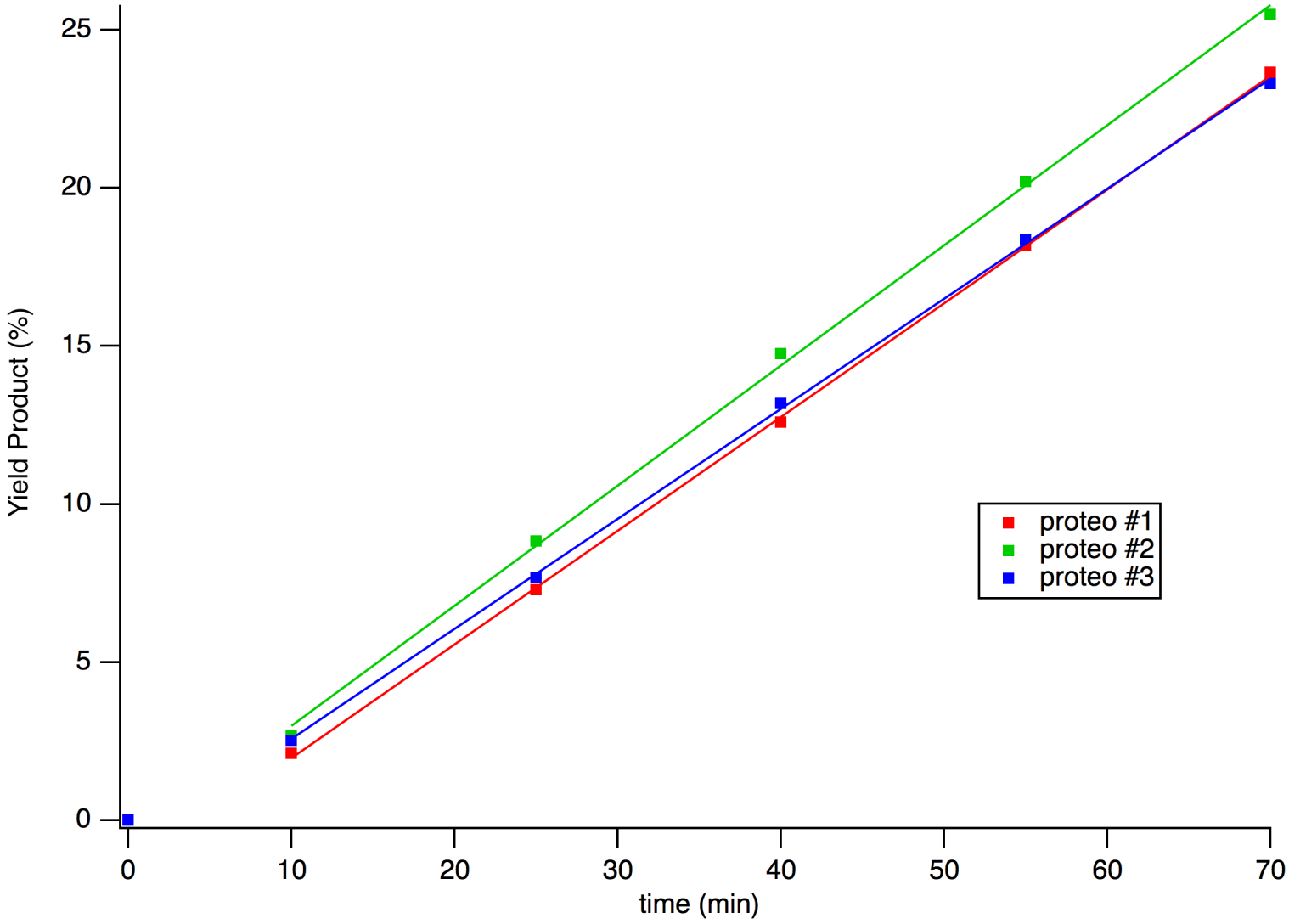
Proteo Experiment 3, t = 55 min
19F
298.0



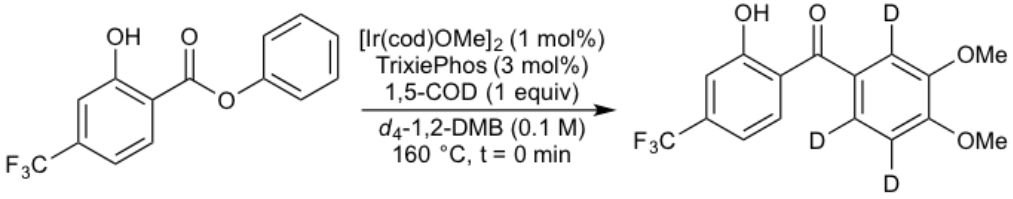
Proteo Experiment 3, t = 70 min
19F
298.0



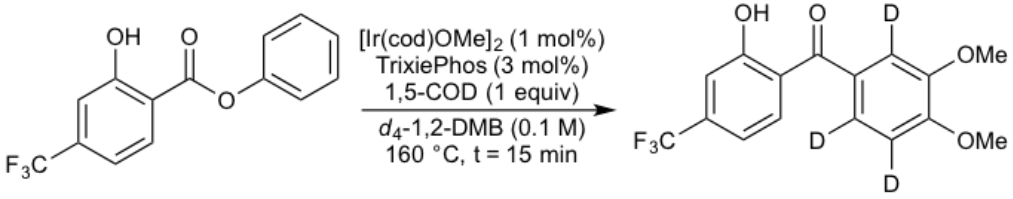

3-CF3-anisole
Starting Material
Produc
Proteo Experiment 3, t = 70 min
298.0 °C
19F
Proteo Experiment 3, t = 55 min
298.0 °C
19F
Proteo Experiment 3, t = 40 min
298.0 °C
19F
Proteo Experiment 3, t = 25 min
298.0 °C
19F
Proteo Experiment 3, t = 10 min
298.0 °C
19F
Proteo Experiment 3, t = 0 min
298.1 °C
19F
6
5
4
3
2
1
-62.1
-62.2
-62.3
-62.4
-62.5
-62.6
-62.7
-62.8
-62.9
-63.0
-63.1
-63.2
-63.3
-63.4
-63.5
-63.6
-63.7
-63.8
-63.9
-64.0
-64.1
-64.2
f1 (ppm)


25
20
15
10
5
0
Yield Product (%)
0
10
20
30
40
50
60
70
time (min)
proteo #1
proteo #2
proteo #3

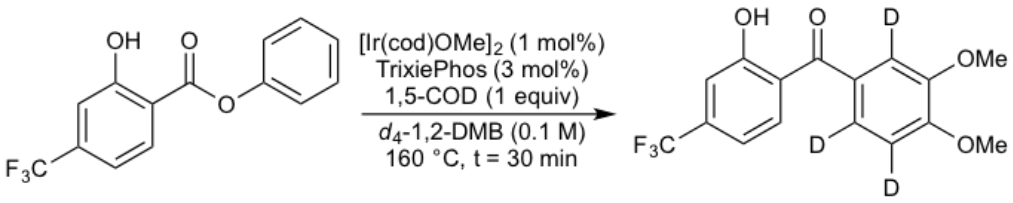
Deutero Experiment 1, t = 0 min
19F
298.0



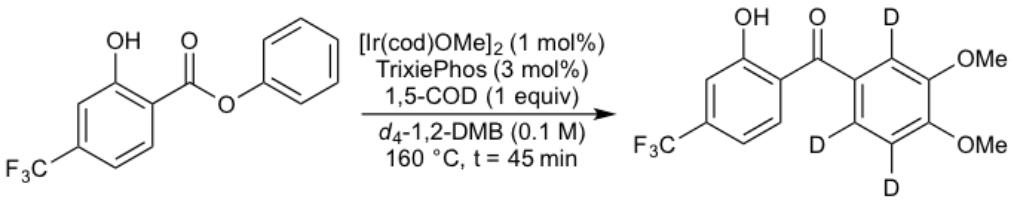
Deutero Experiment 1, t = 15 min
19F
298.0



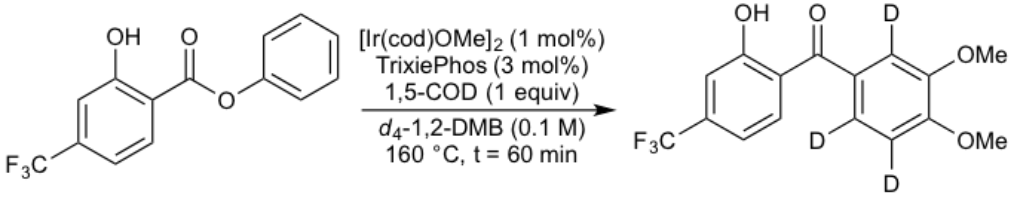
Deutero Experiment 1, t = 30 min
19F
298.0



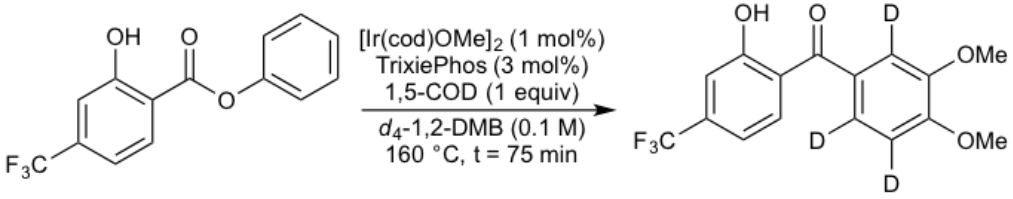
Deutero Experiment 1, t = 45 min
19F
298.0



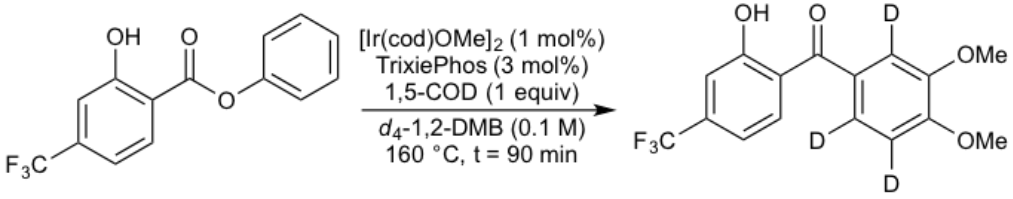
Deutero Experiment 1, t = 60 min
19F
298.0

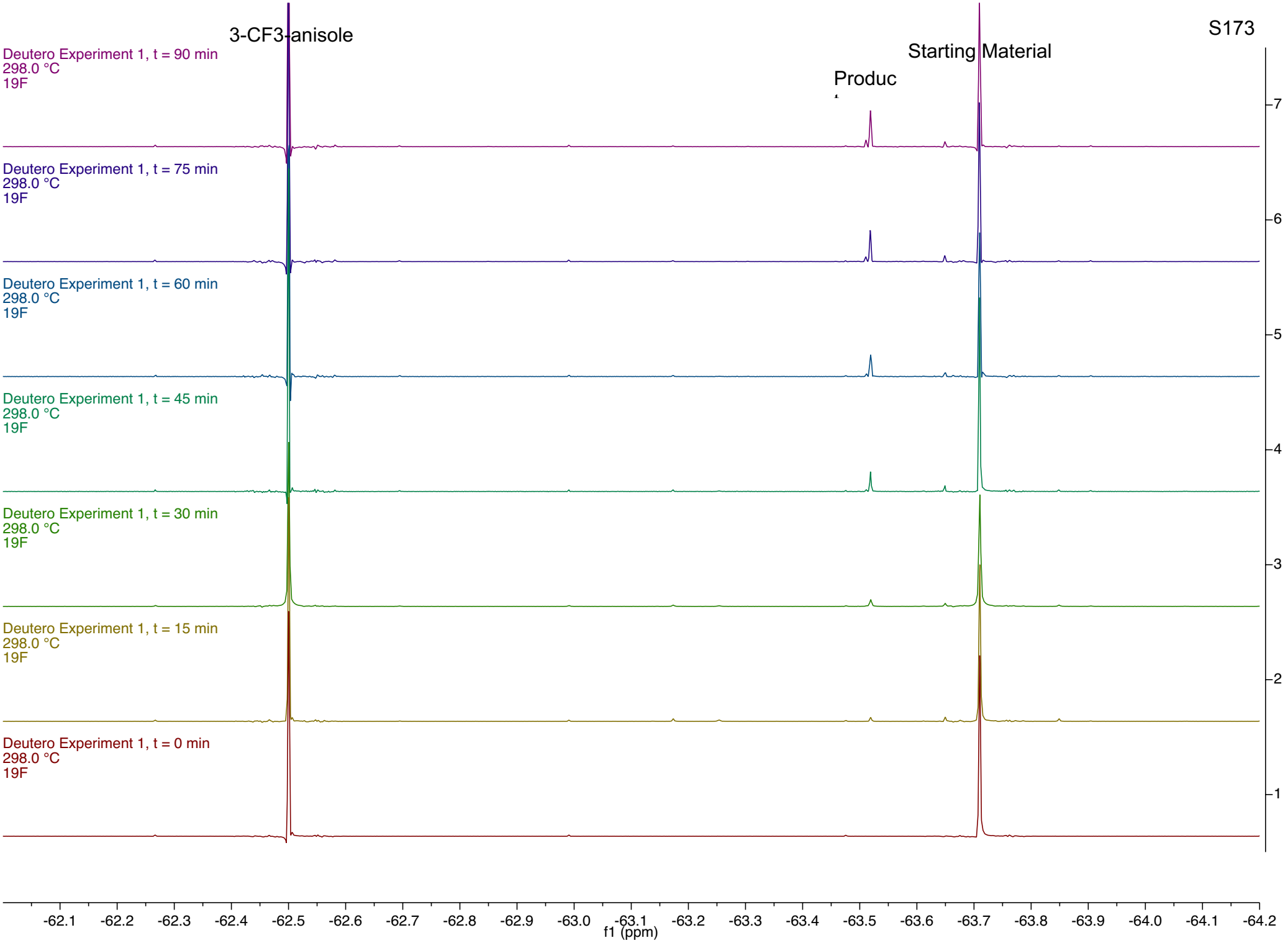


Deutero Experiment 1, t = 75 min
19F
298.0

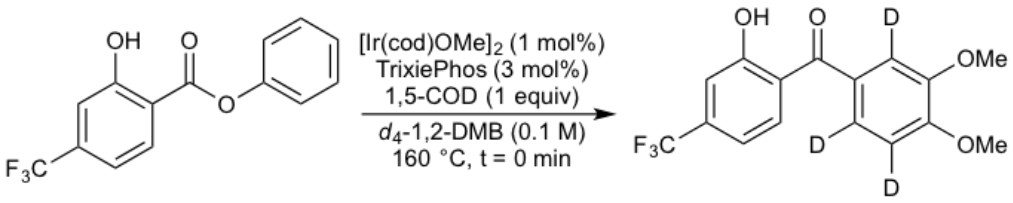


Deutero Experiment 1, t = 90 min
19F
298.0

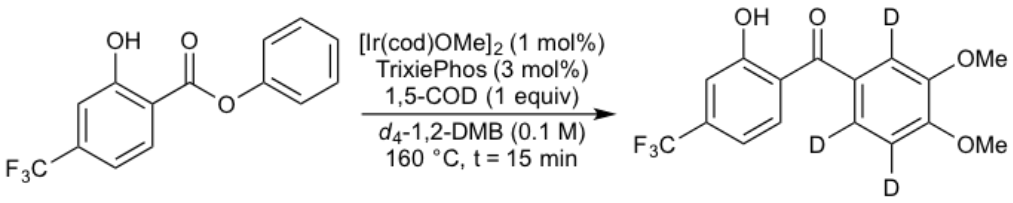



3-CF3-anisole
Starting Material
Produc
Deutero Experiment 1, t = 90 min
298.0 °C
19F
Deutero Experiment 1, t = 75 min
298.0 °C
19F
Deutero Experiment 1, t = 60 min
298.0 °C
19F
Deutero Experiment 1, t = 45 min
298.0 °C
19F
Deutero Experiment 1, t = 30 min
298.0 °C
19F
Deutero Experiment 1, t = 15 min
298.0 °C
19F
Deutero Experiment 1, t = 0 min
298.0 °C
19F
7
6
5
4
3
2
1
-62.1
-62.2
-62.3
-62.4
-62.5
-62.6
-62.7
-62.8
-62.9
-63.0
-63.1
-63.2
-63.3
-63.4
-63.5
-63.6
-63.7
-63.8
-63.9
-64.0
-64.1
-64.2
f1 (ppm)

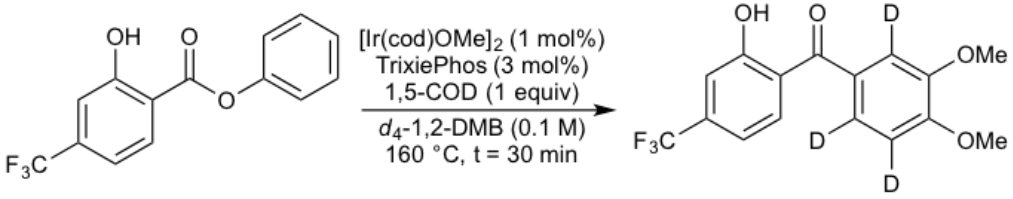
Deutero Experiment 2, t = 0 min
19F
298.0



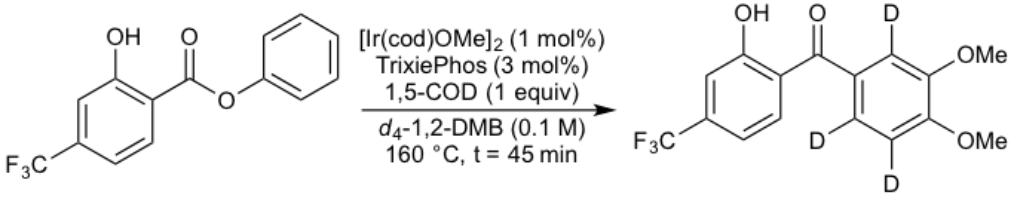
Deutero Experiment 2, t = 15 min
19F
298.0



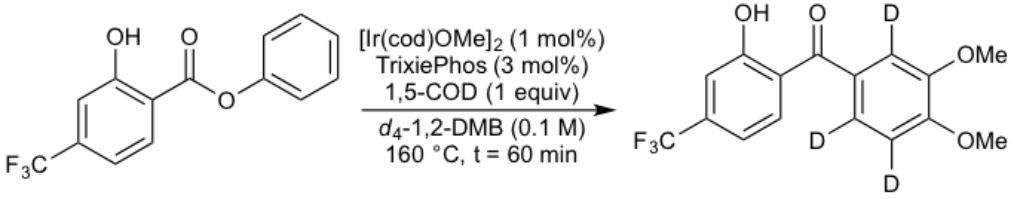
Deutero Experiment 2, t = 30 min
19F
298.0



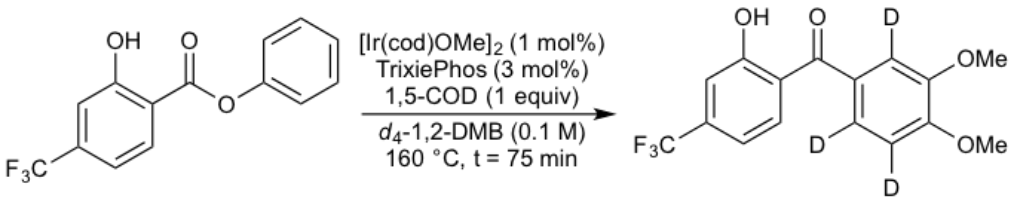
Deutero Experiment 2, t = 45 min
19F
298.0



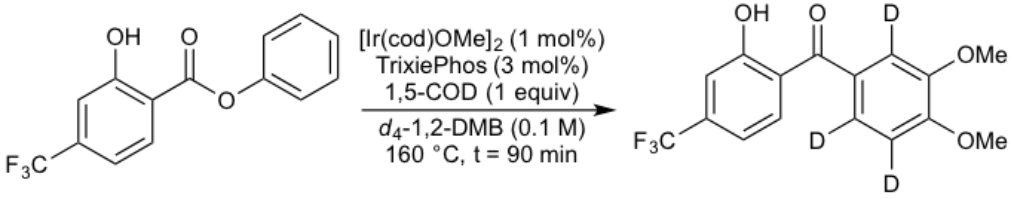
Deutero Experiment 2, t = 60 min
19F
298.0

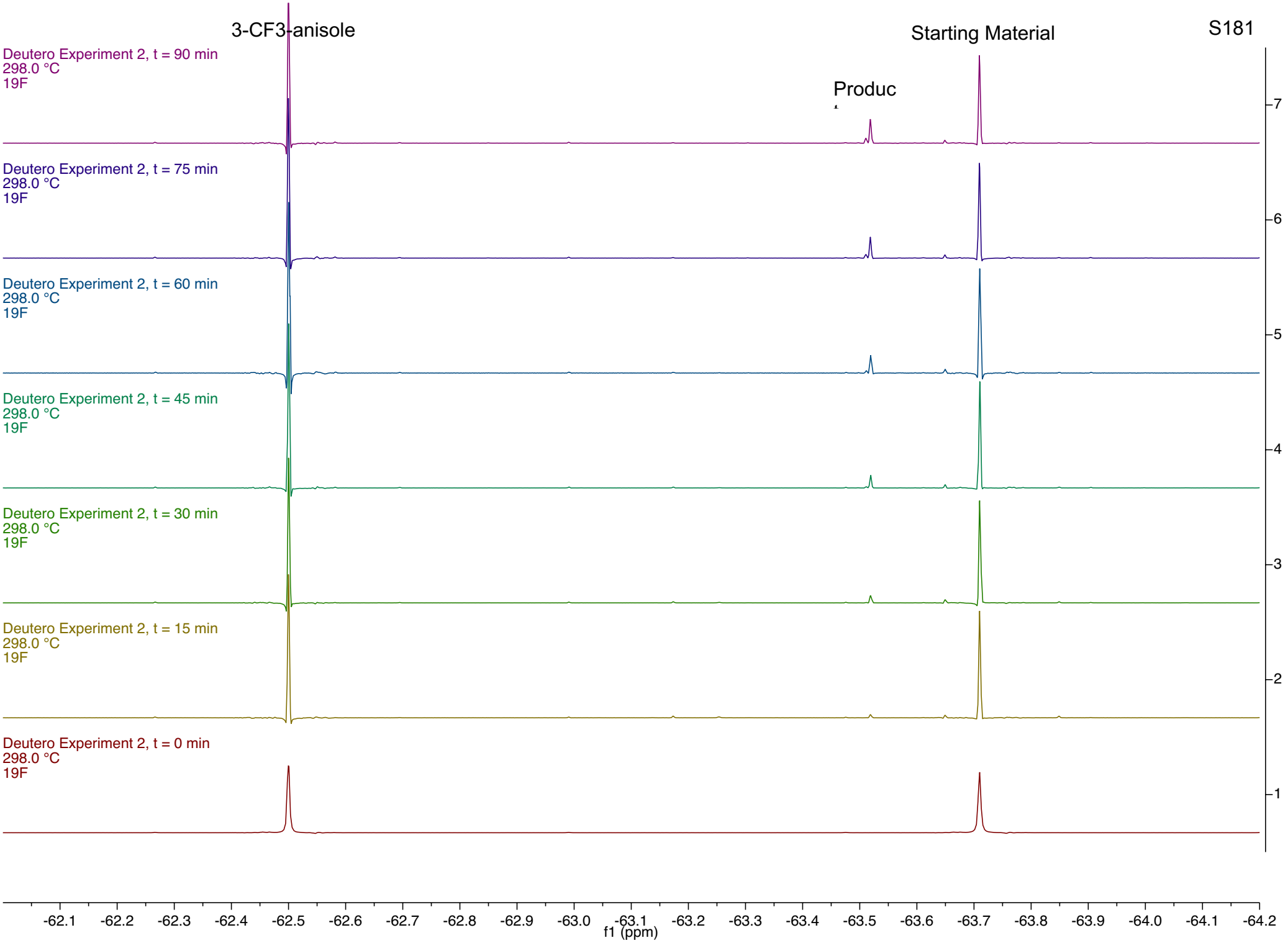


Deutero Experiment 2, t = 75 min
19F
298.0

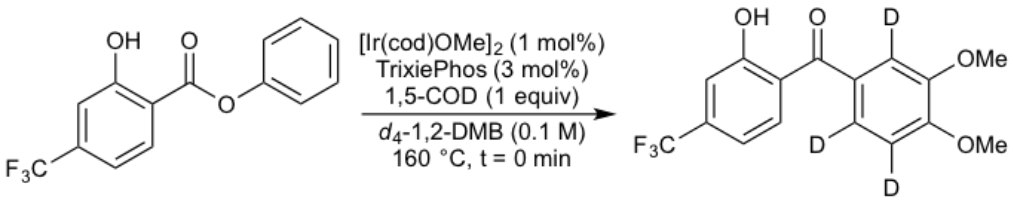


Deutero Experiment 2, t = 90 min
19F
298.0

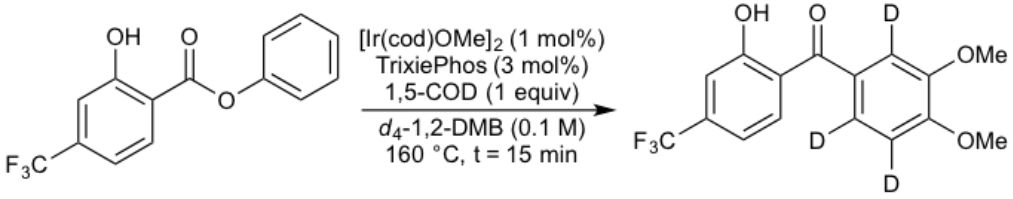



3-CF3-anisole
Starting Material
Produc
Deutero Experiment 2, t = 90 min
298.0 °C
19F
Deutero Experiment 2, t = 75 min
298.0 °C
19F
Deutero Experiment 2, t = 60 min
298.0 °C
19F
Deutero Experiment 2, t = 45 min
298.0 °C
19F
Deutero Experiment 2, t = 30 min
298.0 °C
19F
Deutero Experiment 2, t = 15 min
298.0 °C
19F
Deutero Experiment 2, t = 0 min
298.0 °C
19F
7
6
5
4
3
2
1
-62.1
-62.2
-62.3
-62.4
-62.5
-62.6
-62.7
-62.8
-62.9
-63.0
-63.1
-63.2
-63.3
-63.4
-63.5
-63.6
-63.7
-63.8
-63.9
-64.0
-64.1
-64.2
f1 (ppm)

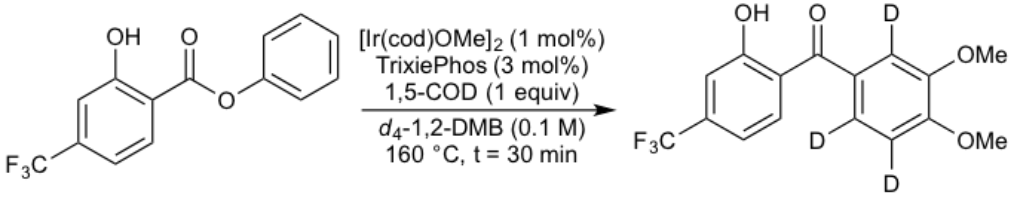
Deutero Experiment 3, t = 0 min
19F
298.0



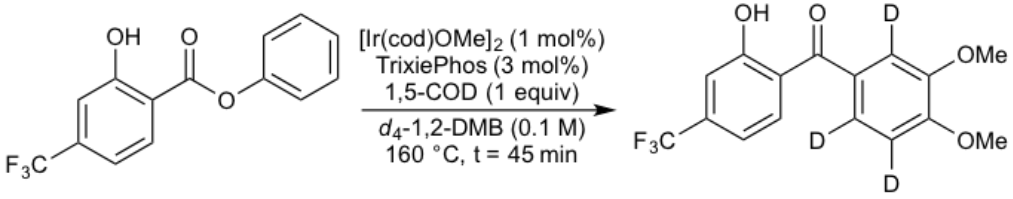
Deutero Experiment 3, t = 15 min
19F
298.0



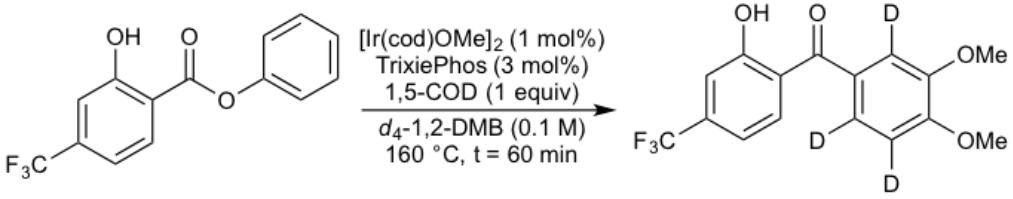
Deutero Experiment 3, t = 30 min
19F
298.0



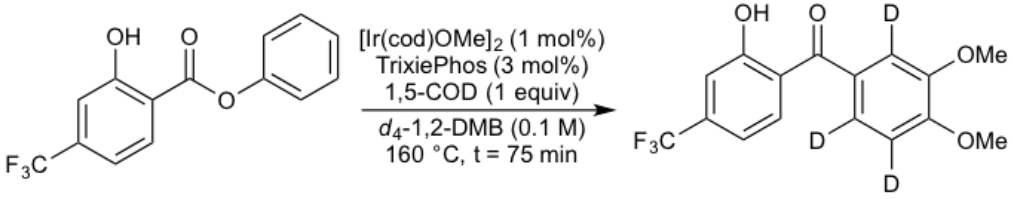
Deutero Experiment 3, t = 45 min
19F
298.0



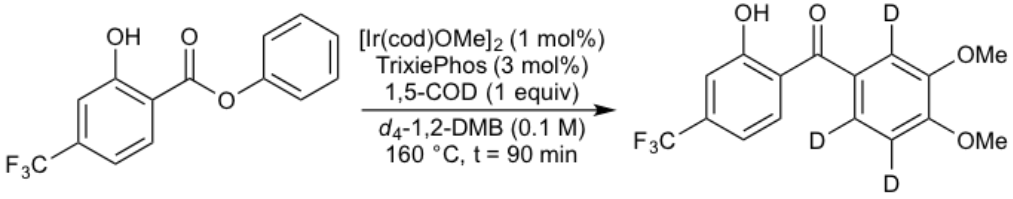
Deutero Experiment 3, t = 60 min
19F
298.0

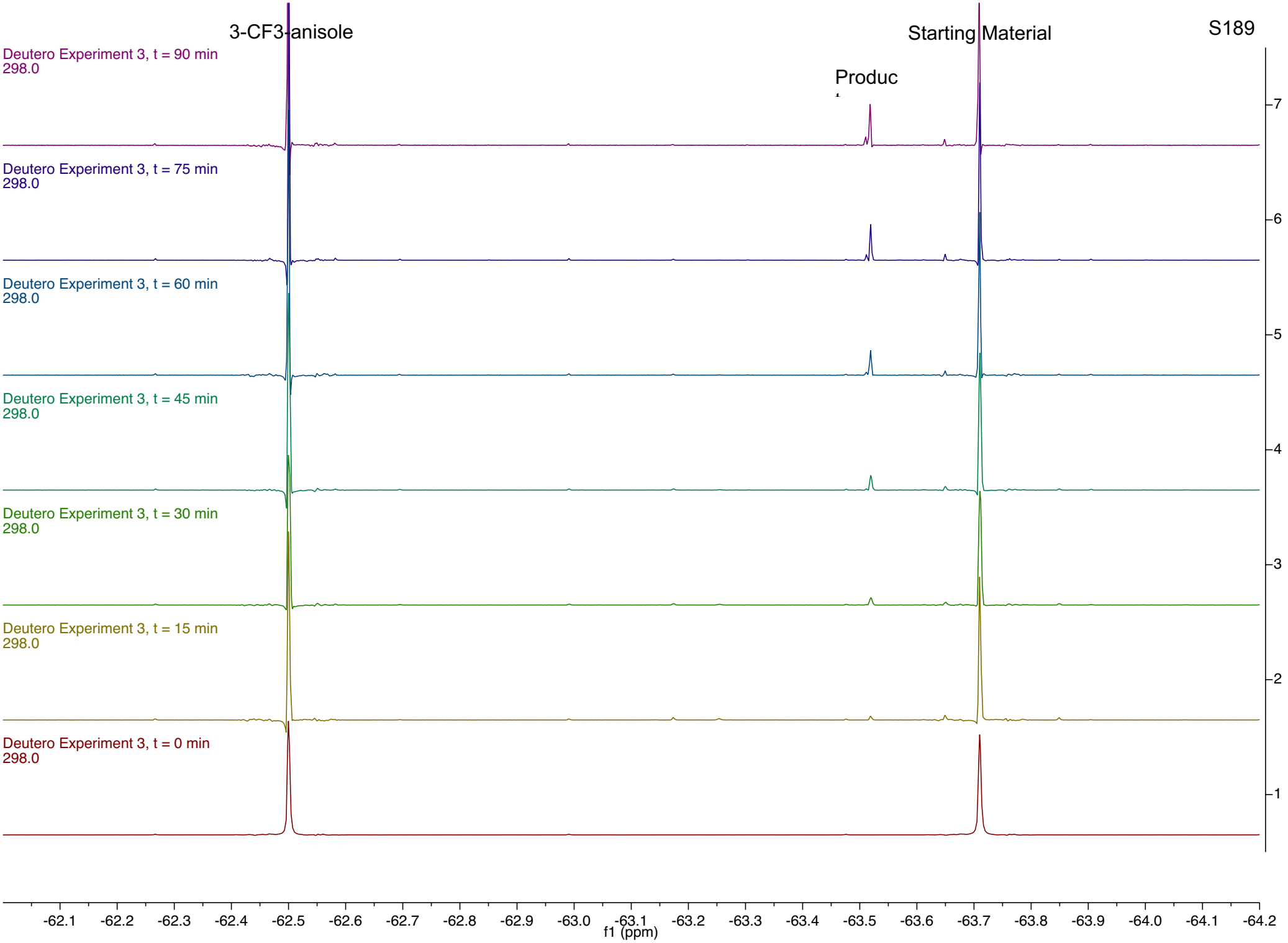


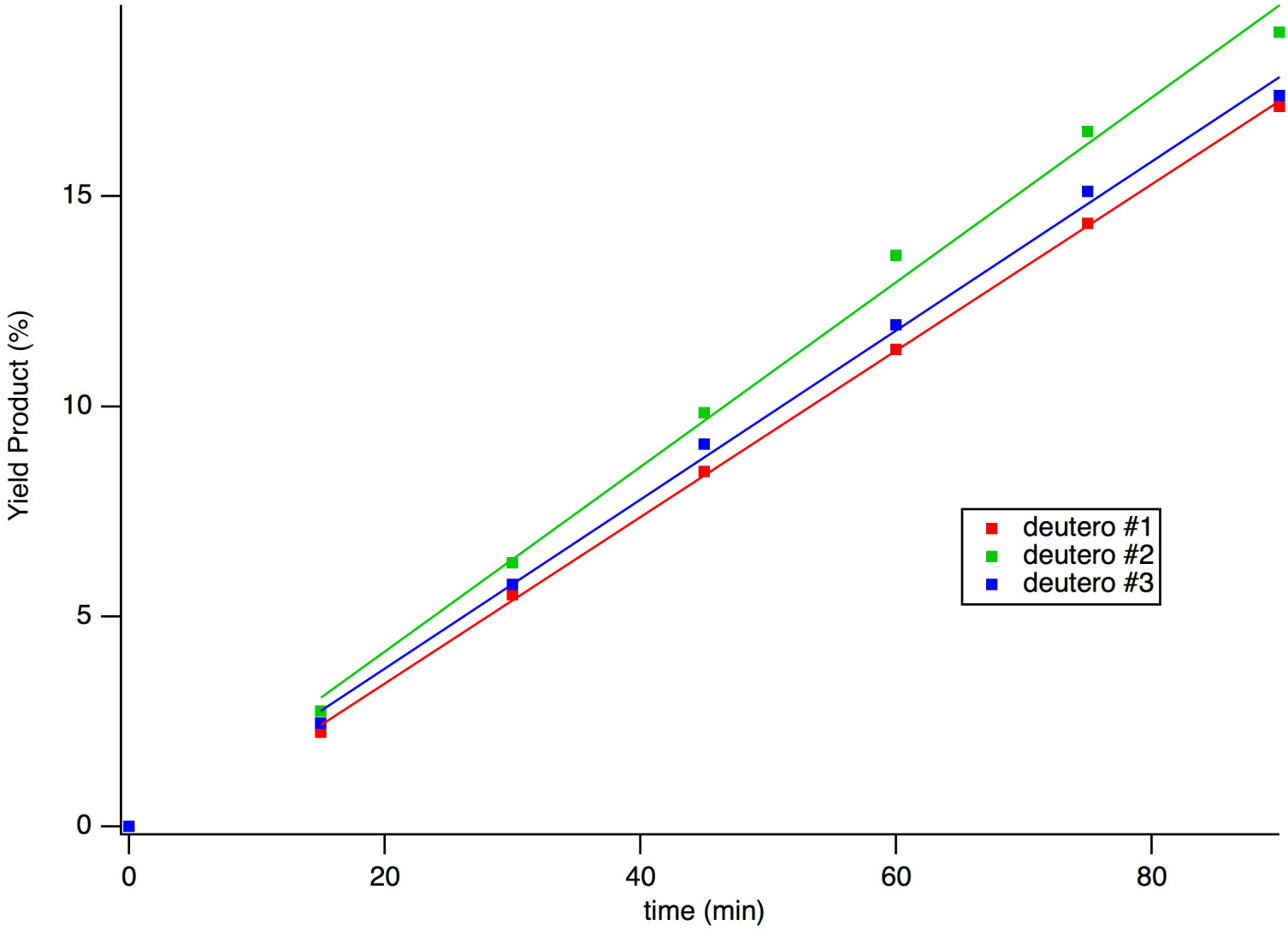
Deutero Experiment 3, t = 75 min
19F
298.0



Deutero Experiment 3, t = 90 min
19F
298.0




3-CF3-anisole
Starting Material
S189
Produc
Deutero Experiment 3, t = 90 min
298.0
Deutero Experiment 3, t = 75 min
298.0
Deutero Experiment 3, t = 60 min
298.0
Deutero Experiment 3, t = 45 min
298.0
Deutero Experiment 3, t = 30 min
298.0
Deutero Experiment 3, t = 15 min
298.0
Deutero Experiment 3, t = 0 min
298.0
7
6
5
4
3
2
1
-62.1
-62.2
-62.3
-62.4
-62.5
-62.6
-62.7
-62.8
-62.9
-63.0
-63.1
-63.2
-63.3
-63.4
-63.5
-63.6
-63.7
-63.8
-63.9
-64.0
-64.1
-64.2
f1 (ppm)


Yield Product (%)
0
5
10
15
0
20
40
60
80
time (min)
deutero #1
deutero #2
deutero #3

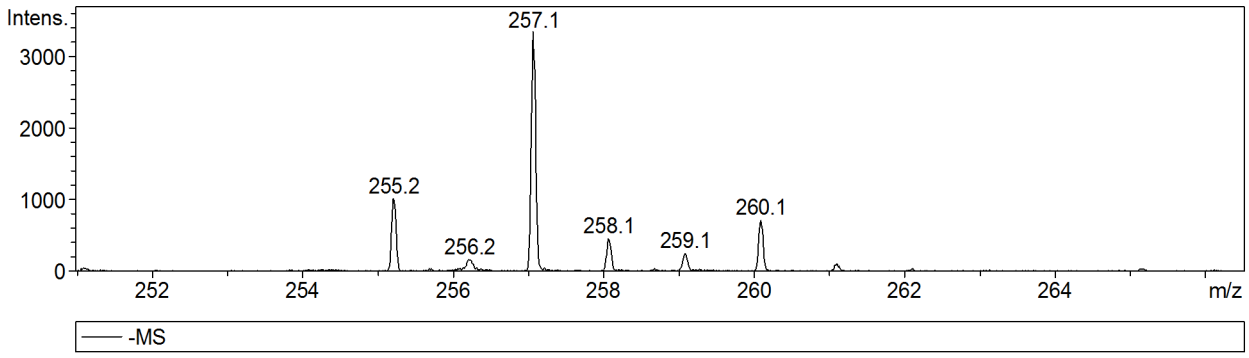
Mass Spectrum List Report

Analysis Info

Analysis Name	2LOW RES	Acquisition Date	4/20/2017 9:08:35 AM
Method	negative_010616.tofpar	Operator	operator name
Sample Name	NAS-2-63_4-1	Instrument	BioTOF II
	ESI NEGFree format commentsFree format comments		

Acquisition Parameter

n/a	n/a	n/a	n/a	detbias	1800 V
EndP	3000 V	n/a	n/a	n/a	n/a



#	m/z	I
1	145.0	1485
2	169.1	144
3	212.1	268
4	213.0	4652
5	214.1	811
6	215.1	112
7	241.0	141
8	255.2	1002
9	256.2	156
10	257.1	3332
11	258.1	450
12	259.1	249
13	260.1	704
14	283.2	1502
15	284.2	237

Intermolecular Competition 1

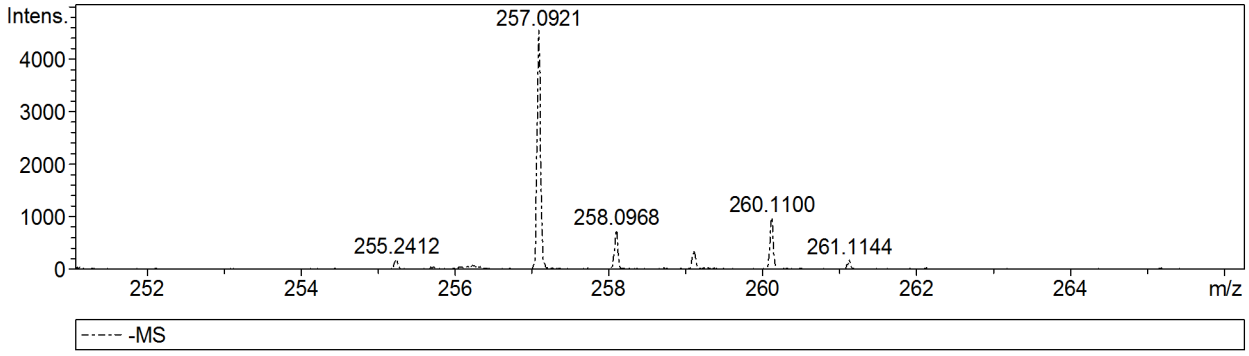
Mass Spectrum List Report

Analysis Info

Analysis Name	14-2 low res	Acquisition Date	4/21/2017 10:39:37 AM
Method	negative_010616.tofpar	Operator	operator name
Sample Name	NAS-2-63_4-2	Instrument	BioTOF II
	ESI NEGFree format commentsFree format comments		

Acquisition Parameter

n/a	n/a	n/a	n/a	detbias	1800 V
EndP	3000 V	n/a	n/a	n/a	n/a



#	m/z	I
1	137.0352	1272
2	138.0418	524
3	144.9769	273
4	169.0767	292
5	212.0851	1128
6	213.0673	7994
7	214.0718	1491
8	215.0769	204
9	229.0971	529
10	230.1012	218
11	232.1133	145
12	241.0612	174
13	242.0710	109
14	255.2412	185
15	257.0921	4546
16	258.0968	745
17	259.1042	358
18	260.1100	983
19	261.1144	176
20	283.0535	327
21	283.2723	175
22	284.0571	251
23	333.0851	110
24	339.0162	173
25	377.1115	675
26	378.1166	168
27	380.1266	165

Intermolecular Competition 2

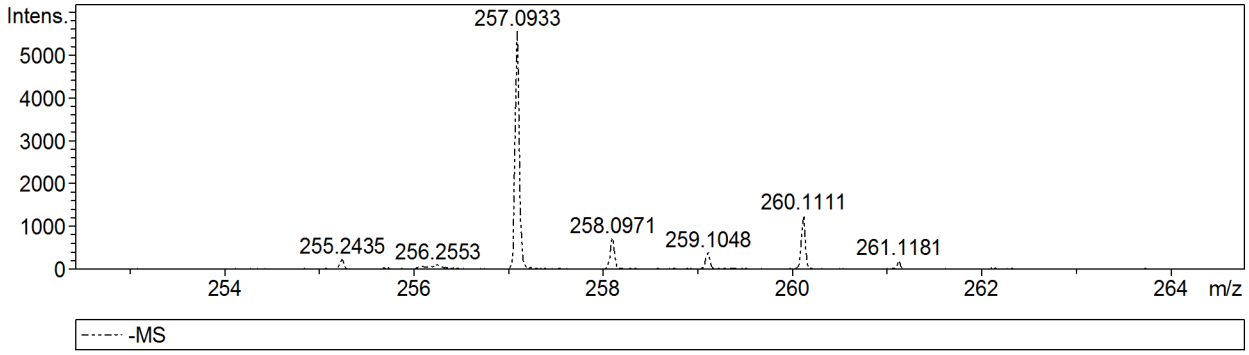
Mass Spectrum List Report

Analysis Info

Analysis Name	14-3 low res	Acquisition Date	4/21/2017 10:41:55 AM
Method	negative_010616.tofpar	Operator	operator name
Sample Name	NAS-2-63_4-3	Instrument	BioTOF II
	ESI NEGFree format commentsFree format comments		

Acquisition Parameter

n/a	n/a	n/a	n/a	detbias	1800 V
EndP	3000 V	n/a	n/a	n/a	n/a



#	m/z	I
1	137.0359	381
2	138.0422	134
3	144.9772	344
4	169.0771	335
5	211.9322	108
6	212.0862	1104
7	213.0690	9619
8	214.0733	2021
9	215.0787	235
10	227.0564	104
11	229.0974	429
12	230.1013	212
13	232.1144	152
14	241.0630	278
15	242.0707	111
16	255.2435	230
17	256.2553	103
18	257.0933	5577
19	258.0971	717
20	259.1048	376
21	260.1111	1259
22	261.1181	195
23	283.0545	187
24	283.2726	322
25	284.0579	154
26	333.0878	117
27	377.1130	747
28	378.1167	159
29	380.1296	193

Intermolecular Competition 3

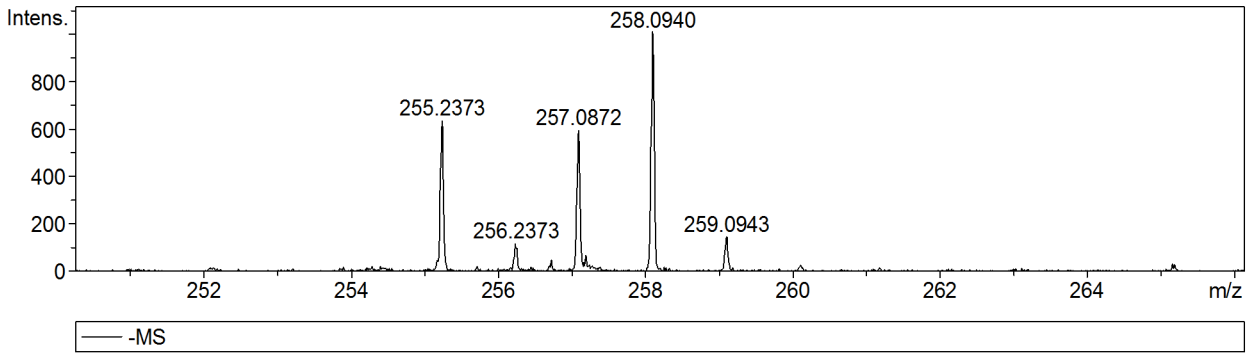
Mass Spectrum List Report

Analysis Info

Analysis Name	11-1 low res	Acquisition Date	4/21/2017 10:30:58 AM
Method	negative_010616.tofpar	Operator	operator name
Sample Name	NAS-2-63_1-1	Instrument	BioTOF II
	ESI NEGFree format commentsFree format comments		

Acquisition Parameter

n/a	n/a	n/a	n/a	detbias	1800 V
EndP	3000 V	n/a	n/a	n/a	n/a



#	m/z	I
1	137.0321	2104
2	138.0389	456
3	144.9732	3873
4	212.0814	693
5	213.0637	1525
6	214.0684	254
7	229.0890	131
8	230.0972	270
9	231.1001	101
10	243.0776	132
11	244.0814	189
12	255.2373	632
13	256.2373	114
14	257.0872	593
15	258.0940	1009
16	259.0943	143
17	282.0462	150
18	283.0469	811
19	283.2674	806
20	284.0531	351
21	284.2673	121
22	378.1092	105

Intramolecular Competition 1

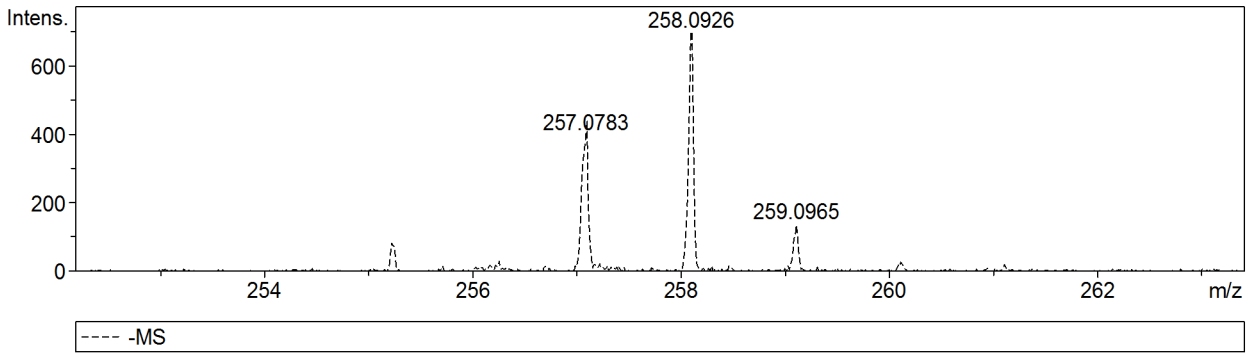
Mass Spectrum List Report

Analysis Info

Analysis Name	11-2 low res	Acquisition Date	4/21/2017 10:34:27 AM
Method	negative_010616.tofpar	Operator	operator name
Sample Name	NAS-2-63_1-2	Instrument	BioTOF II
	ESI NEGFree format commentsFree format comments		

Acquisition Parameter

n/a	n/a	n/a	n/a	detbias	1800 V
EndP	3000 V	n/a	n/a	n/a	n/a



#	m/z	I
1	137.0335	7775
2	138.0391	1620
3	139.0411	120
4	144.9737	1990
5	212.0817	436
6	213.0667	551
7	214.0708	177
8	230.1000	204
9	257.0783	394
10	258.0926	699
11	259.0965	134
12	273.0839	131
13	274.0908	207
14	282.0472	196
15	283.0490	999
16	283.2700	129
17	284.0519	404
18	297.0425	306
19	298.0456	181

Intramolecular Competition 2

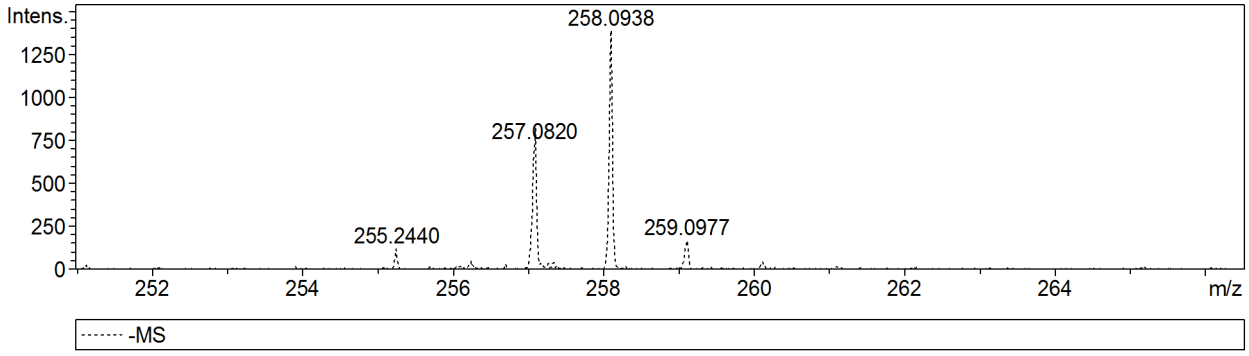
Mass Spectrum List Report

Analysis Info

Analysis Name	11-3 low res	Acquisition Date	4/21/2017 10:36:41 AM
Method	negative_010616.tofpar	Operator	operator name
Sample Name	NAS-2-63_1-3	Instrument	BioTOF II
	ESI NEGFree format commentsFree format comments		

Acquisition Parameter

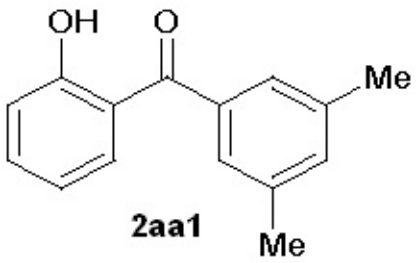
n/a	n/a	n/a	n/a	detbias	1800 V
EndP	3000 V	n/a	n/a	n/a	n/a



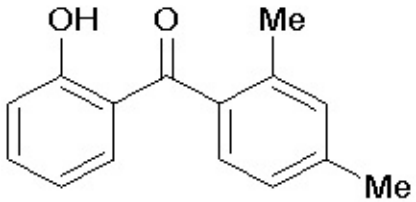
#	m/z	I
1	137.0338	6045
2	138.0388	1360
3	139.0431	104
4	144.9735	530
5	212.0826	655
6	213.0675	1163
7	214.0728	269
8	229.0926	126
9	230.0996	258
10	243.0836	111
11	244.0828	167
12	255.2440	119
13	257.0820	727
14	258.0938	1387
15	259.0977	166
16	273.0821	165
17	274.0870	316
18	282.0524	129
19	283.0495	662
20	283.2674	131
21	284.0532	321
22	297.0419	171
23	298.0507	122
24	378.1095	118

Intramolecular Competition 3

2aa1/2aa2 proton
399.87
298.0
CDCl3

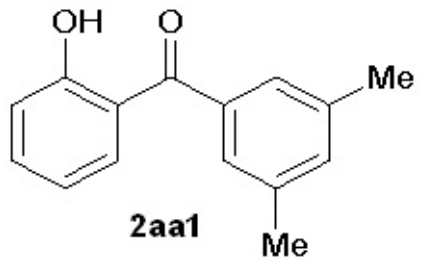


2aa1



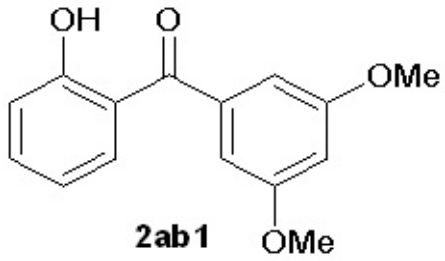
2aa2

2aa1/2aa2 carbon
100.56
298.2
CDCl3



2aa1

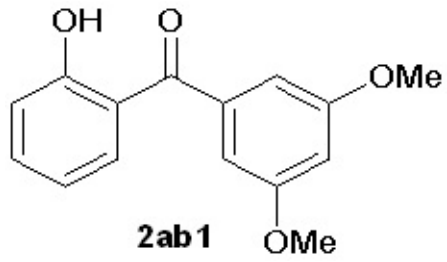
2ab1 proton
400.13
298.0
CDCl3



2ab1

2ab1 carbon
100.56
298.1
CDCl3

S200

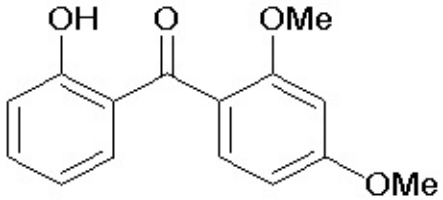


2ab1

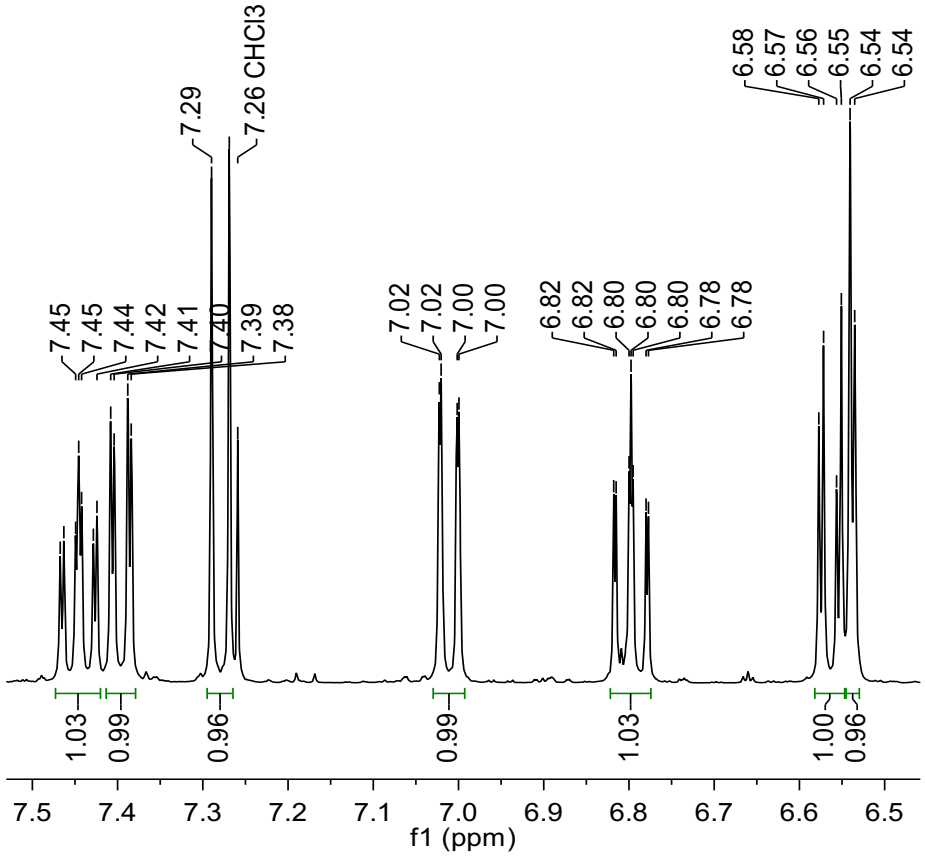
201.46
163.34
160.72
139.78
136.57
133.70
119.16
118.80
118.49
107.11
104.10
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
55.74

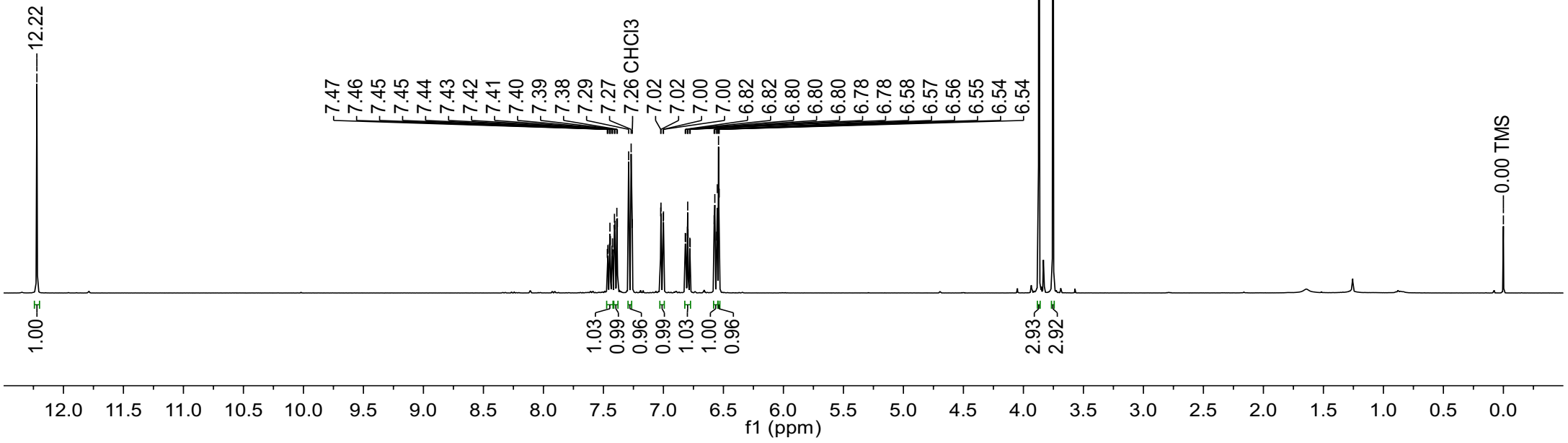
f1 (ppm)

2ab2 proton
400.13
298.0
CDCl3

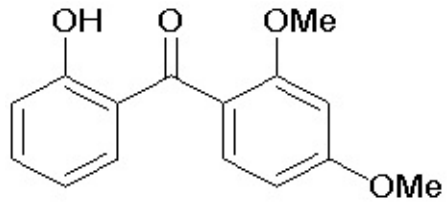


2ab2



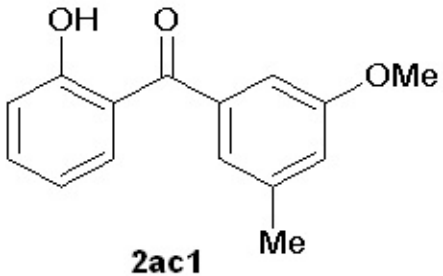


2ab2 carbon
100.56
298.1
CDCl3

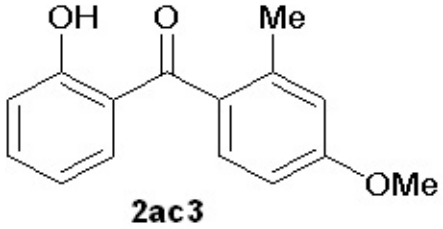


2ab2

2ac1/2ac3 proton
399.87
298.0
CDCl3

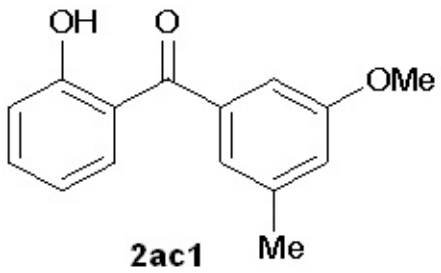


2ac1



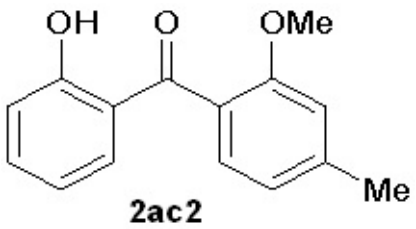
2ac3

2ac1/2ac3 carbon
100.56
298.1
CDCl3



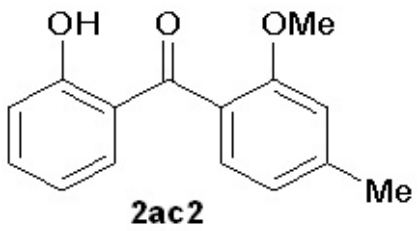
2ac1

2ac2 proton
399.87
298.0
CDCl3



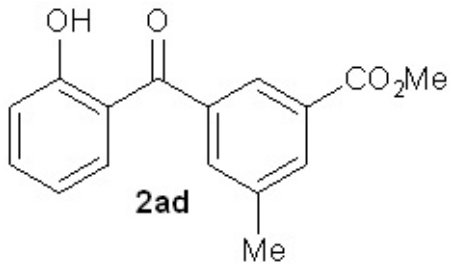
2ac2

2ac2 carbon
100.56
298.1
CDCl3



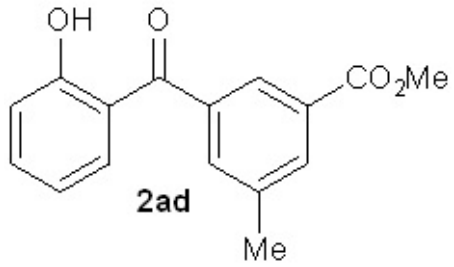
2ac2

2ad proton
400.13
298.0
CDCl3



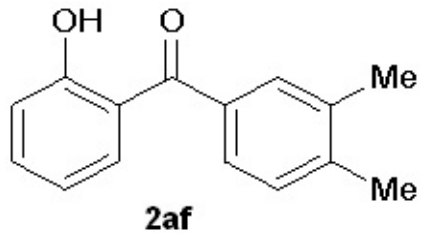
2ad

2ad carbon
100.56
298.0
CDCl3



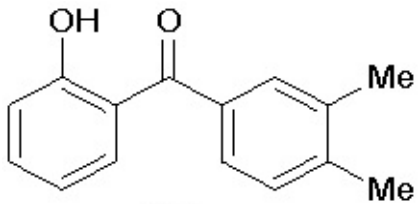
2ad

2af proton
399.87
298.0
CDCl3



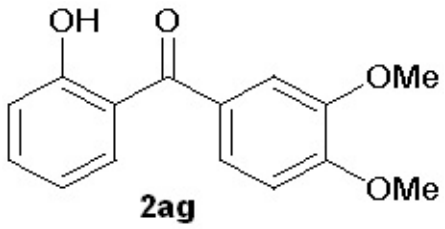
2af

2af carbon
100.62
298.0
CDCl3

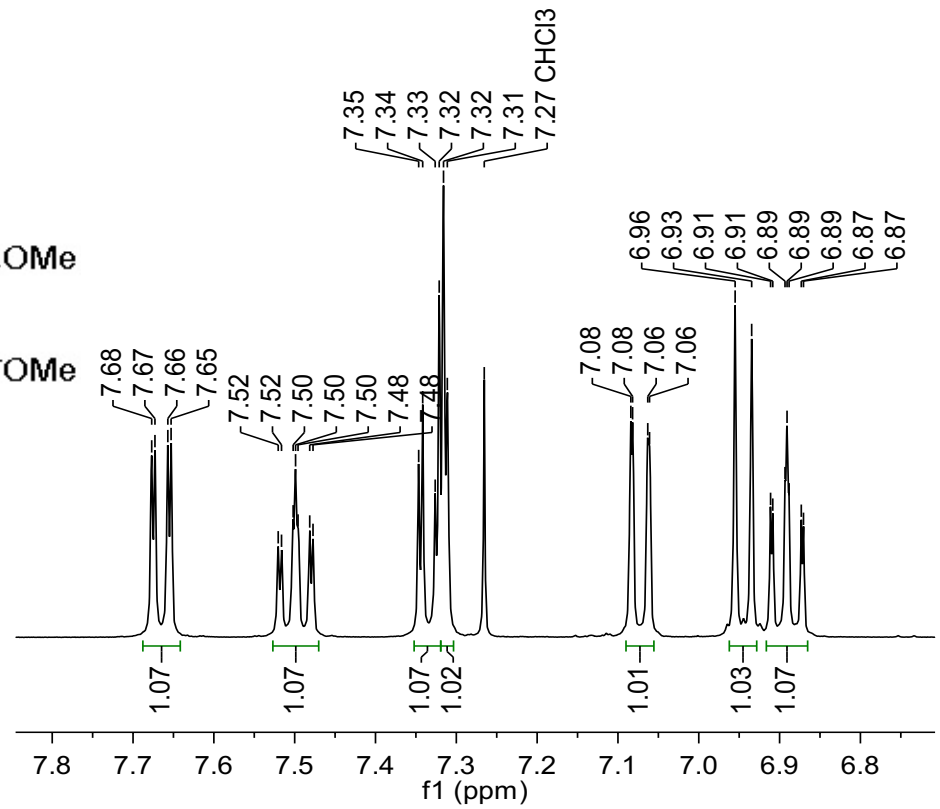


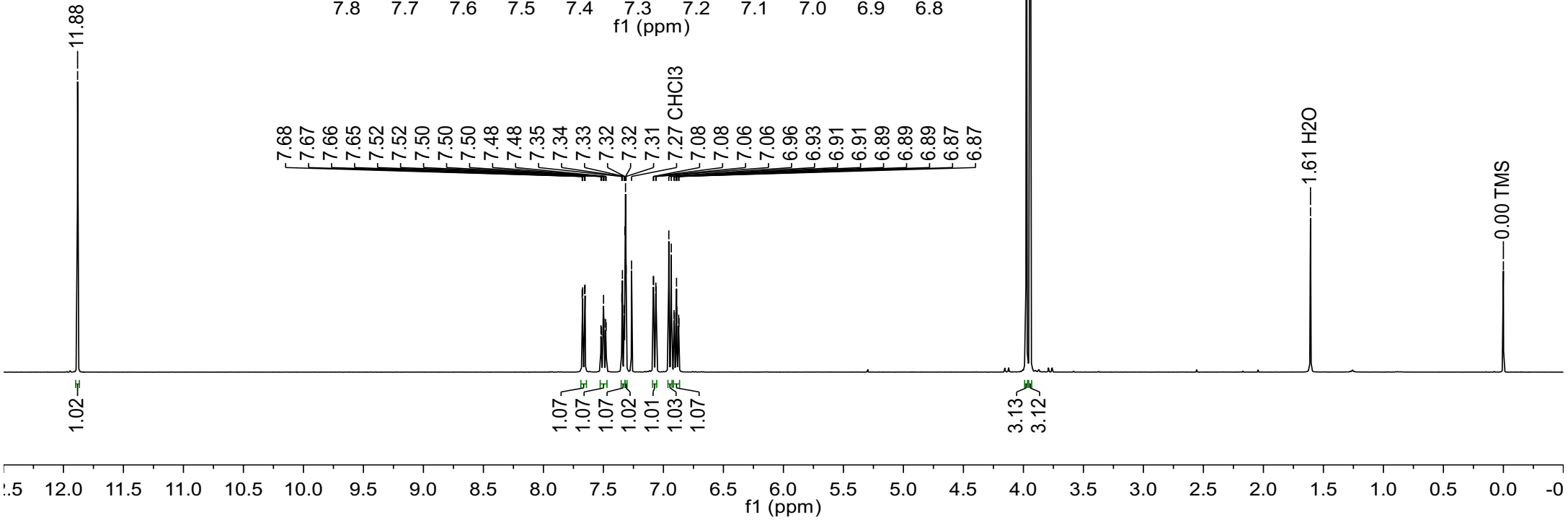
2af

2ag proton
399.87
298.0
CDCl3



2ag

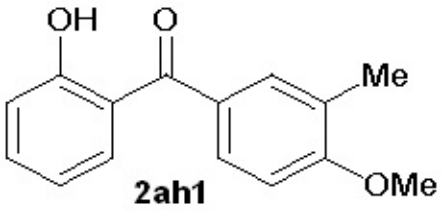




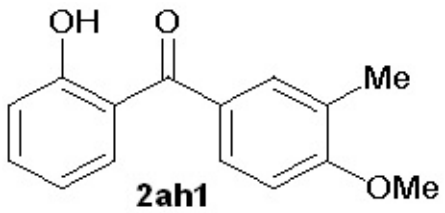
2ag carbon
100.56
298.0
CDCl3

2ag

2ah1 proton
399.87
294.1
CDCl3

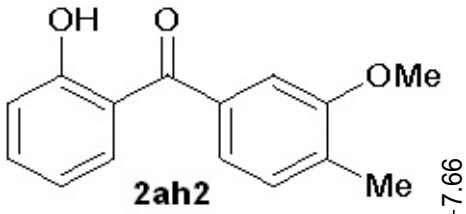


2ah1 carbon
100.56
294.7
CDCl3

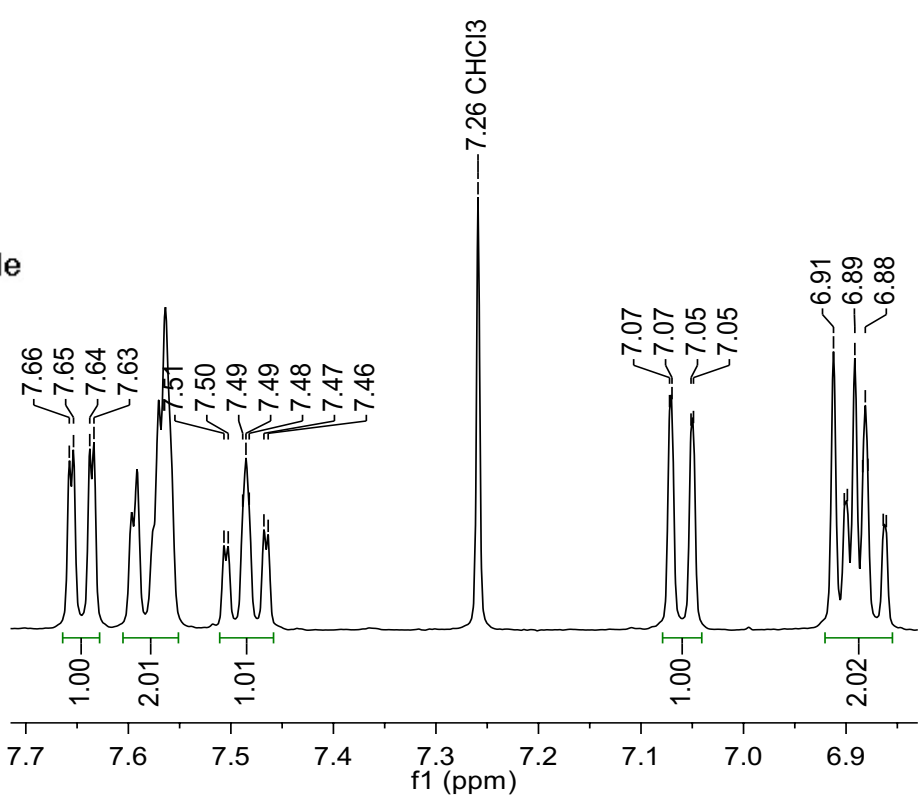


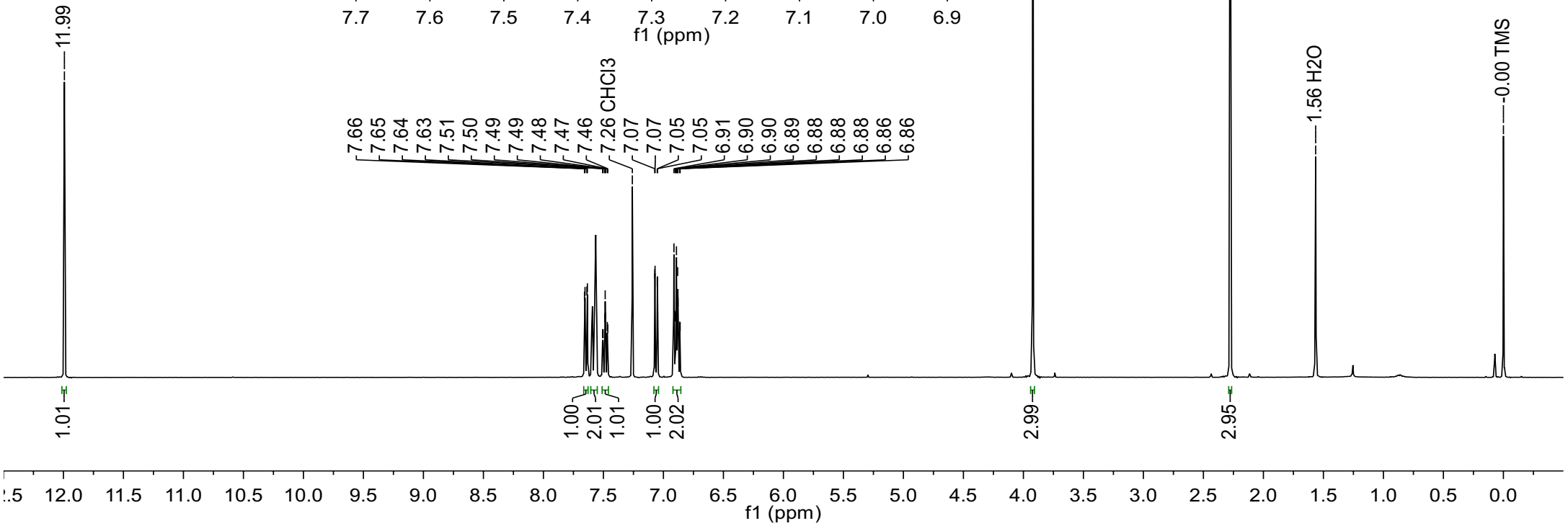
2ah1

2ah2 proton
399.87
298.0
CDCl3

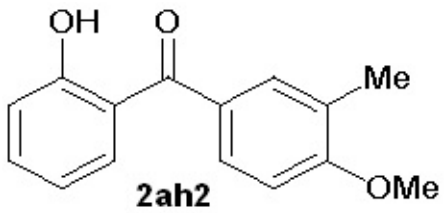


2ah2





2ah2 carbon
100.56
298.0
CDCl3

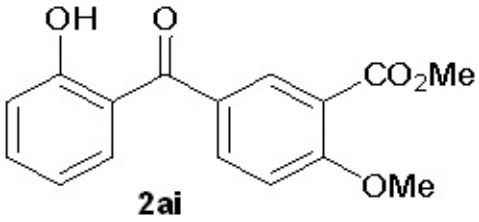


2ah2

77.34 CDCl3
77.02 CDCl3
76.70 CDCl3
200.33
162.90
161.20
135.69
133.38
132.11
129.92
129.72
126.93
119.52
118.44
118.27
109.10
55.58
16.28

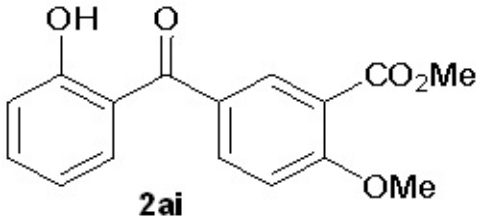
f1 (ppm)

2ai proton
399.87
298.0
CDCl3



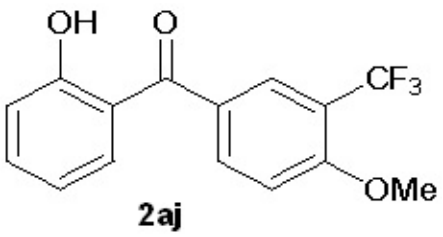
2ai

2ai carbon
100.56
298.0
CDCl3



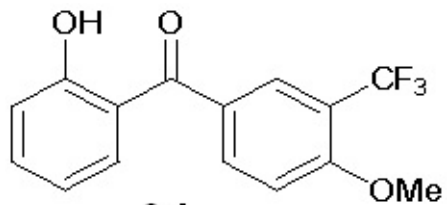
2ai

2aj proton
399.87
298.0
CDCl3



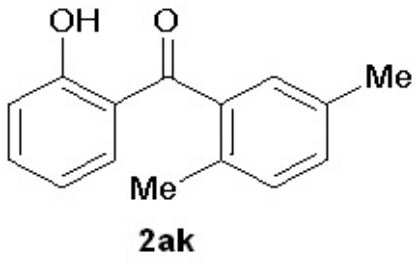
2aj

2aj carbon
100.56
298.1
CDCl3



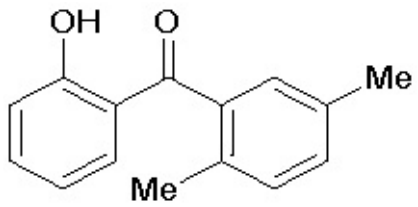
2aj

2ak proton
399.87
298.0
CDCl3



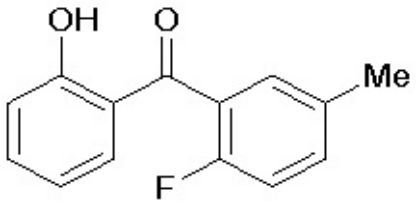
2ak

2ak carbon
100.56
298.0
CDCl3



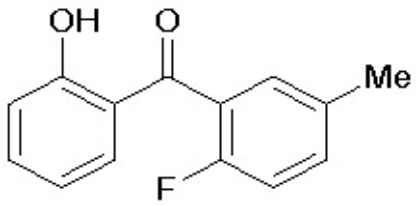
2ak

2al proton
399.87
298.0
CDCl3



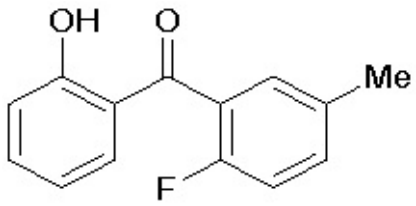
2al

2al carbon
100.56
298.1
CDCl3



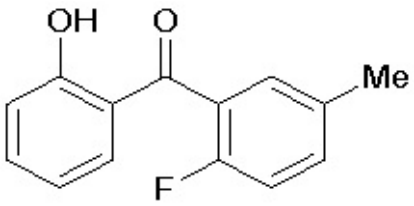
2al

2al with {1H} decoupling 19F
376.22
298.1
CDCl3



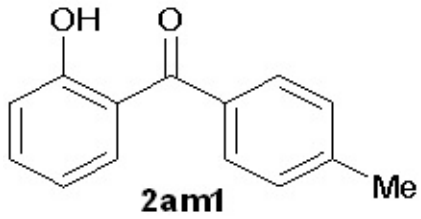
2al

2al no decoupling 19F
376.22
298.0
CDCl3

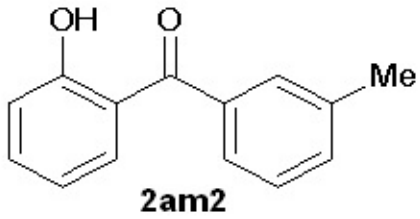


2al

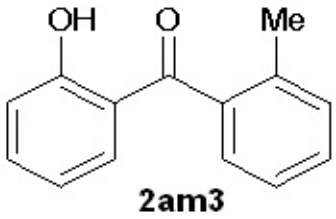
2am1/2am2/2am3 proton
500.13
298.0
CDCl3



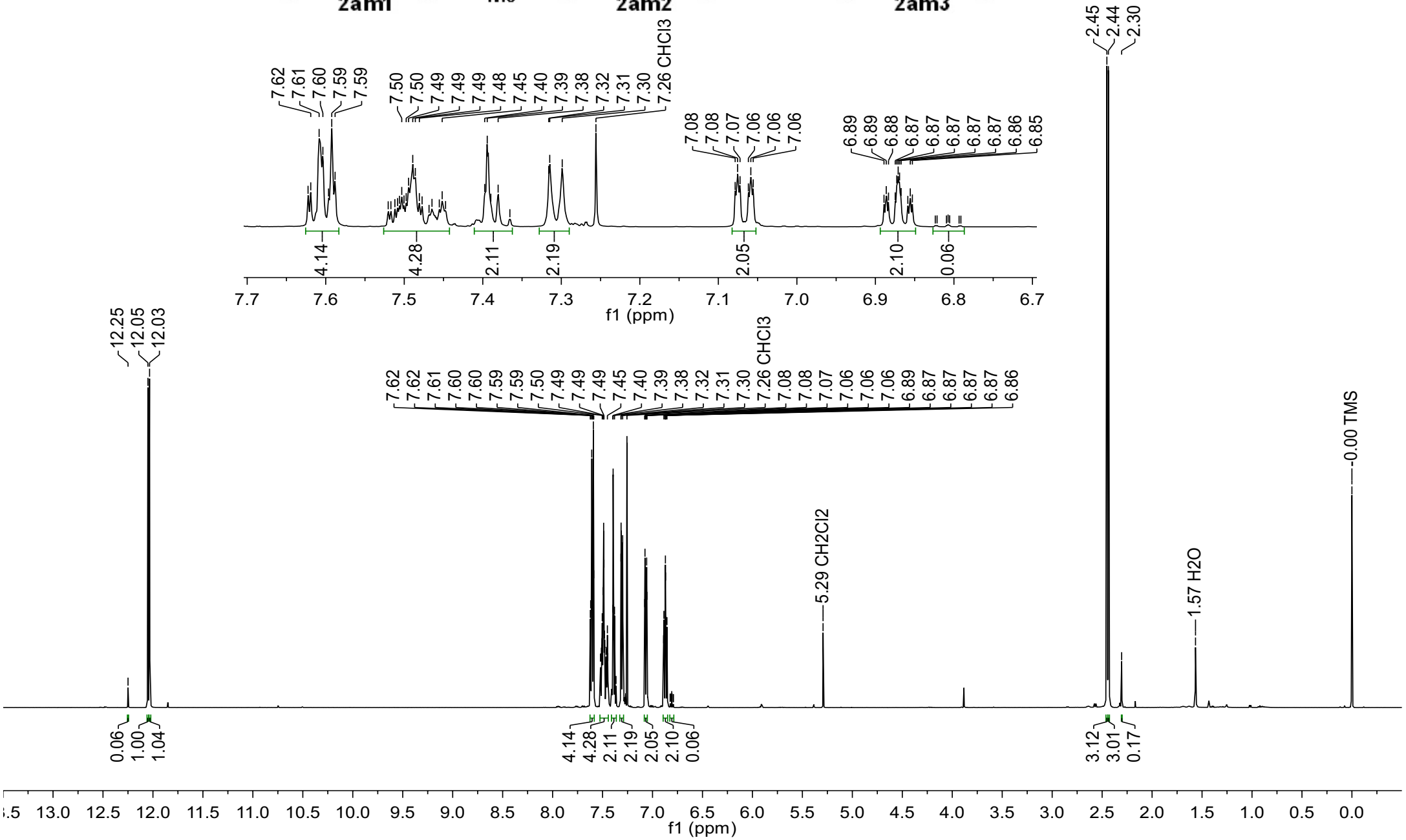
2am1



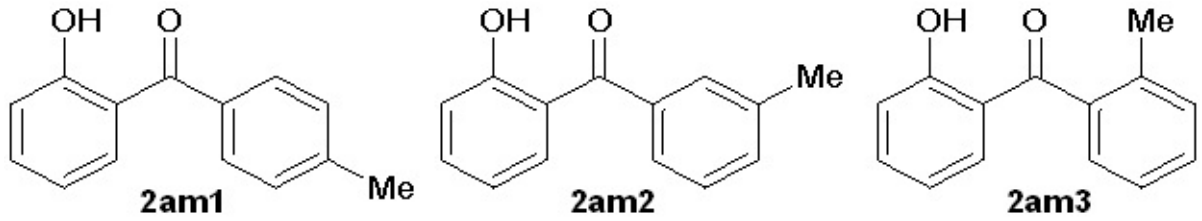
2am2

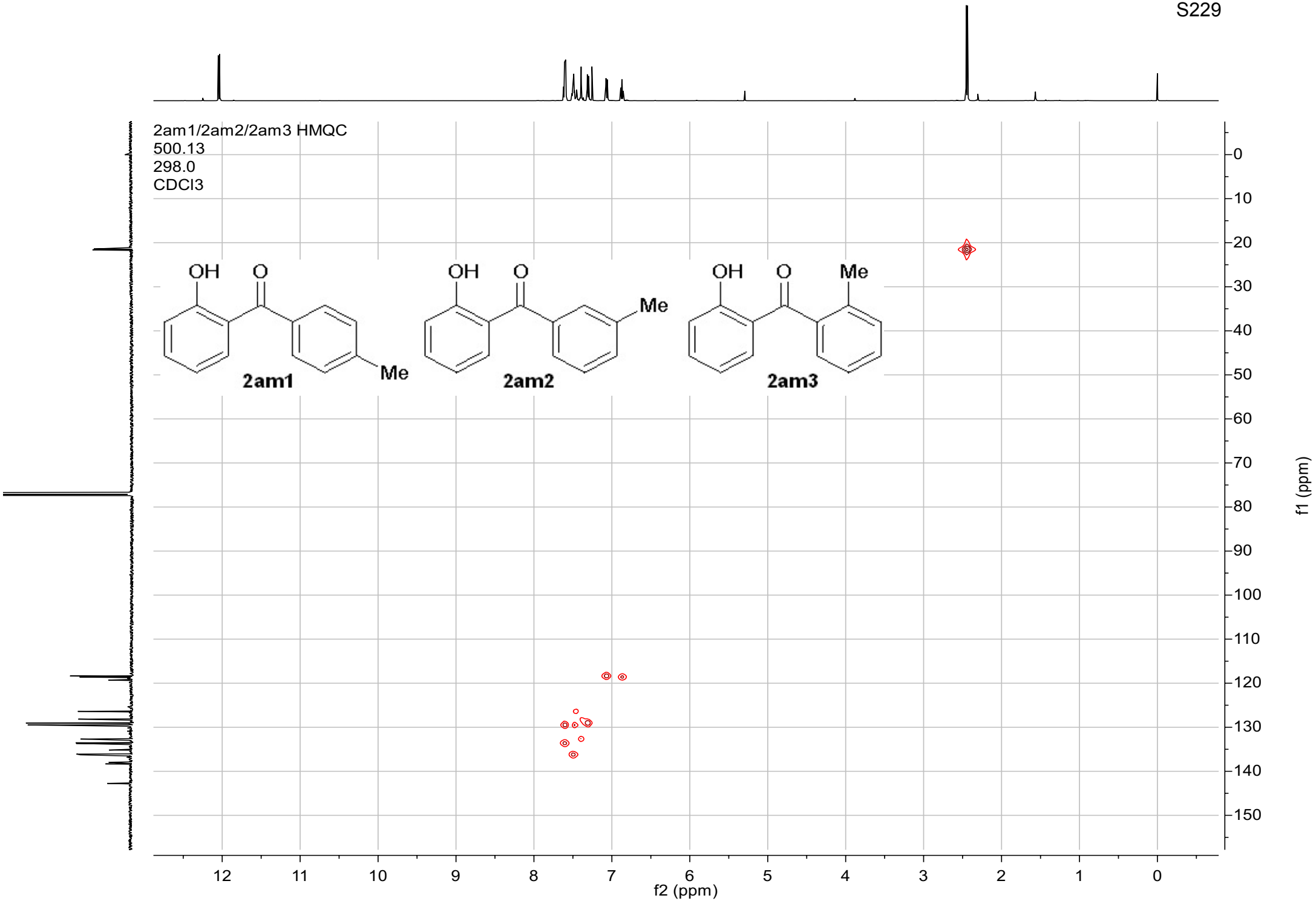


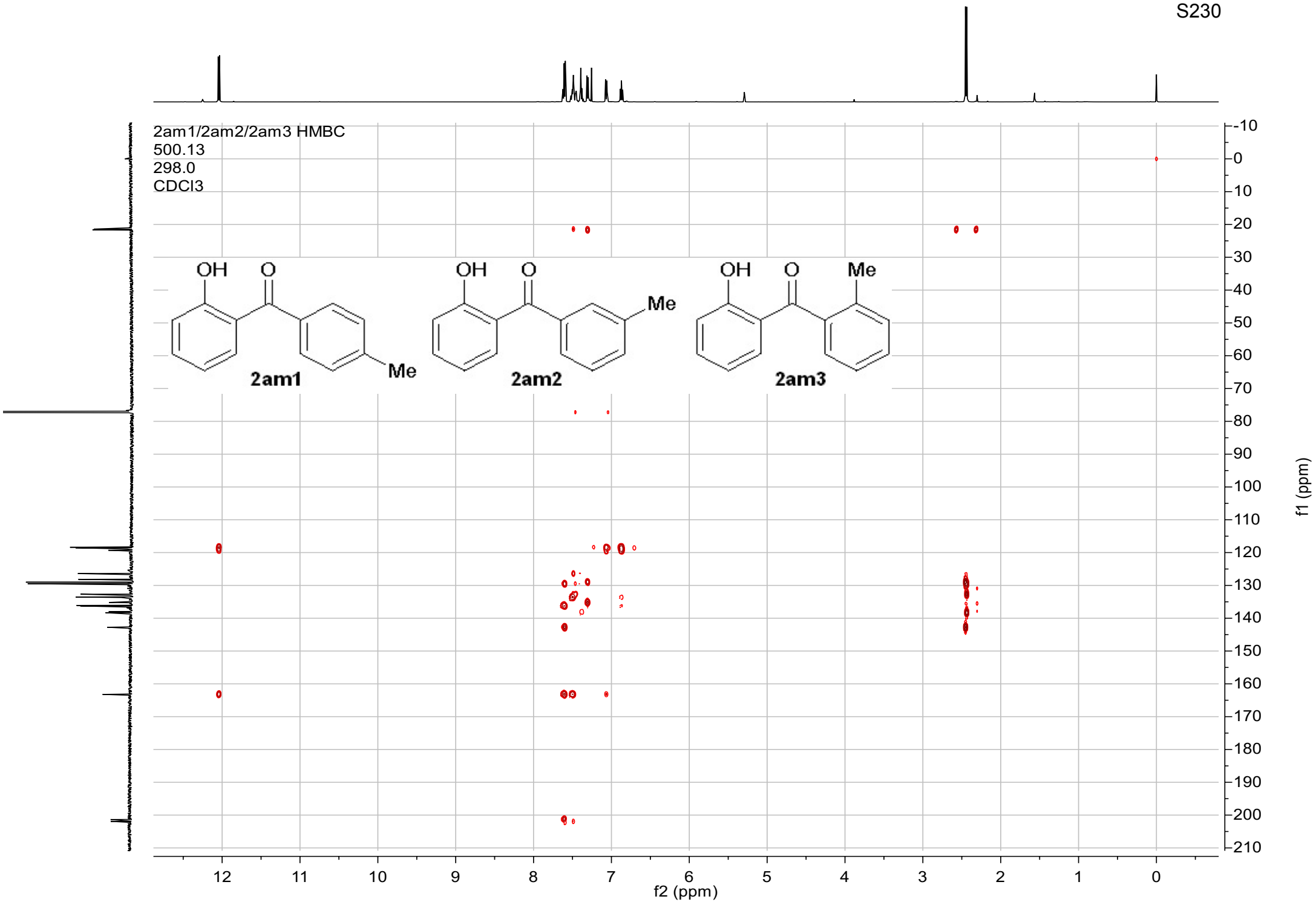
2am3



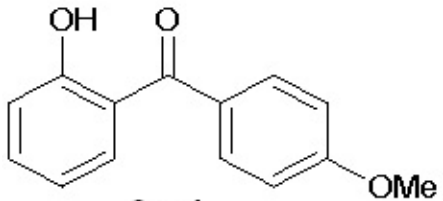
2am1/2am2/2am3 carbon
125.77
298.0
CDCl3



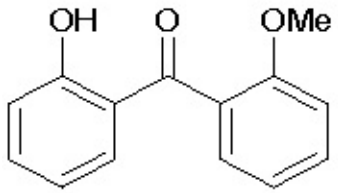

2am1/2am2/2am3 HMQC
500.13
298.0
CDCl3
OH
O
Me
2am1
2am2
2am3
f2 (ppm)
f1 (ppm)


2am1/2am2/2am3 HMBC
500.13
298.0
CDCl3
OH
O
Me
2am1
2am2
2am3
f2 (ppm)
f1 (ppm)

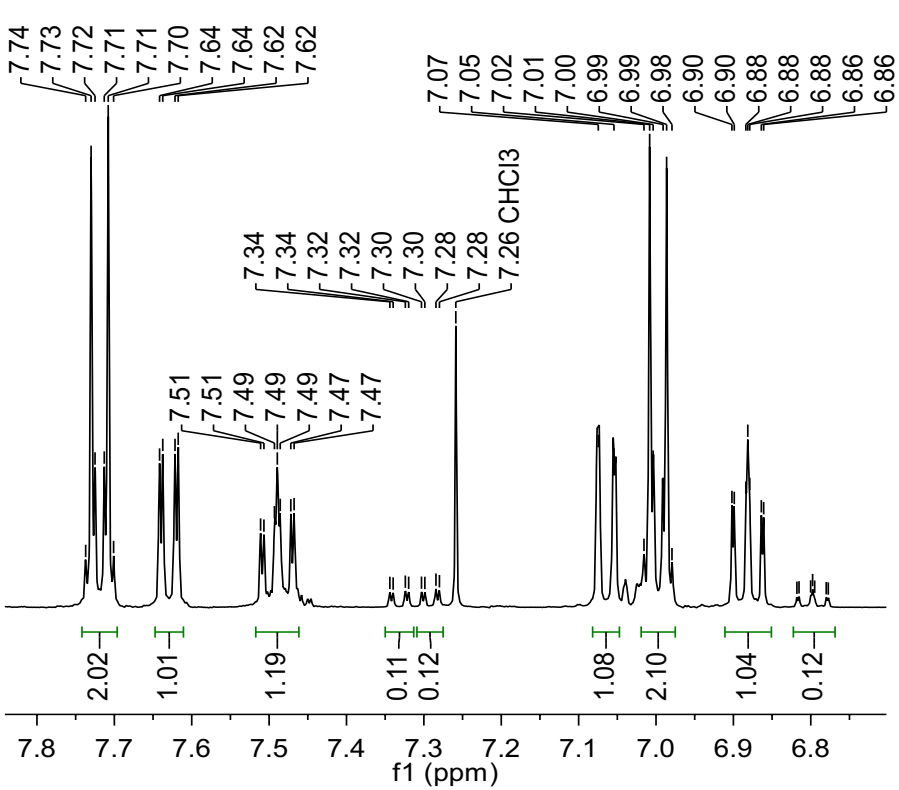
2an1/2an3 proton
399.87
298.0
CDCl3



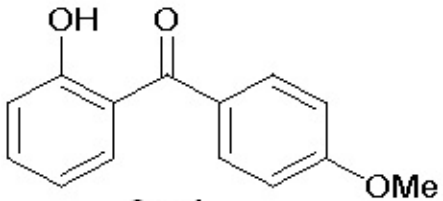
2an1



2an3

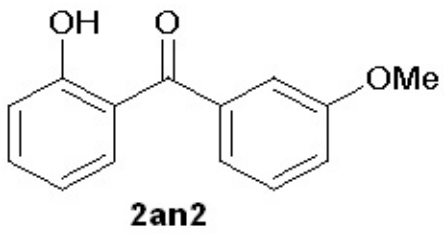


2an1/2an3 carbon
100.62
298.0
CDCl3

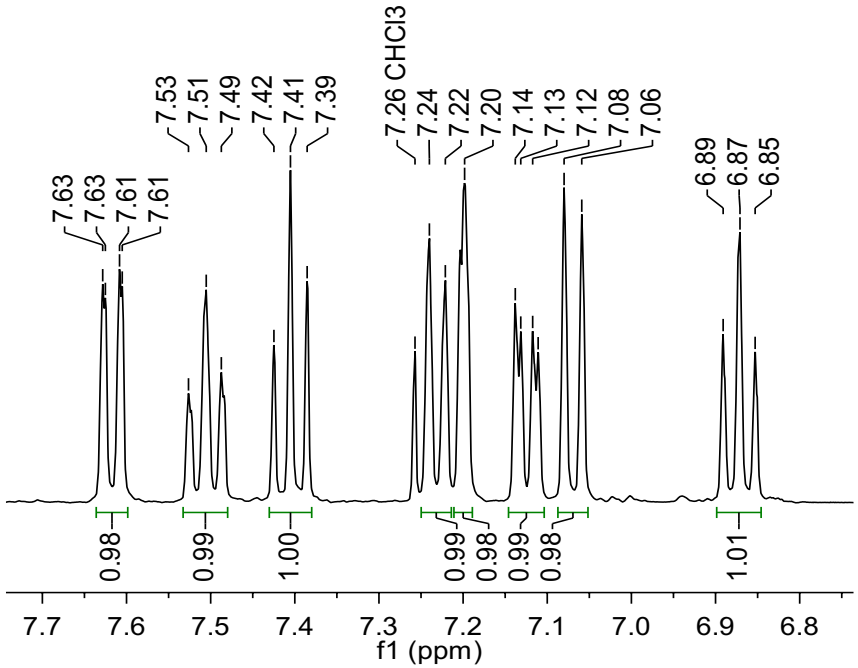


2an1

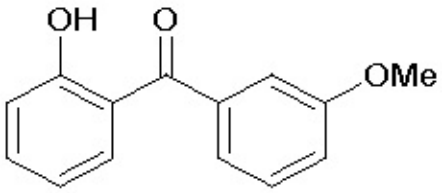
2an2 proton
399.87
298.0
CDCl3



2an2



2an2 carbon
100.62
298.0
CDCl3

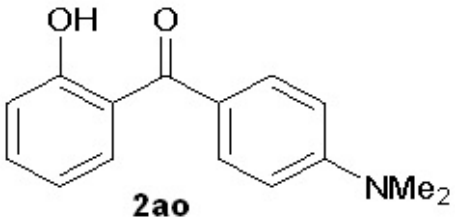


2an2

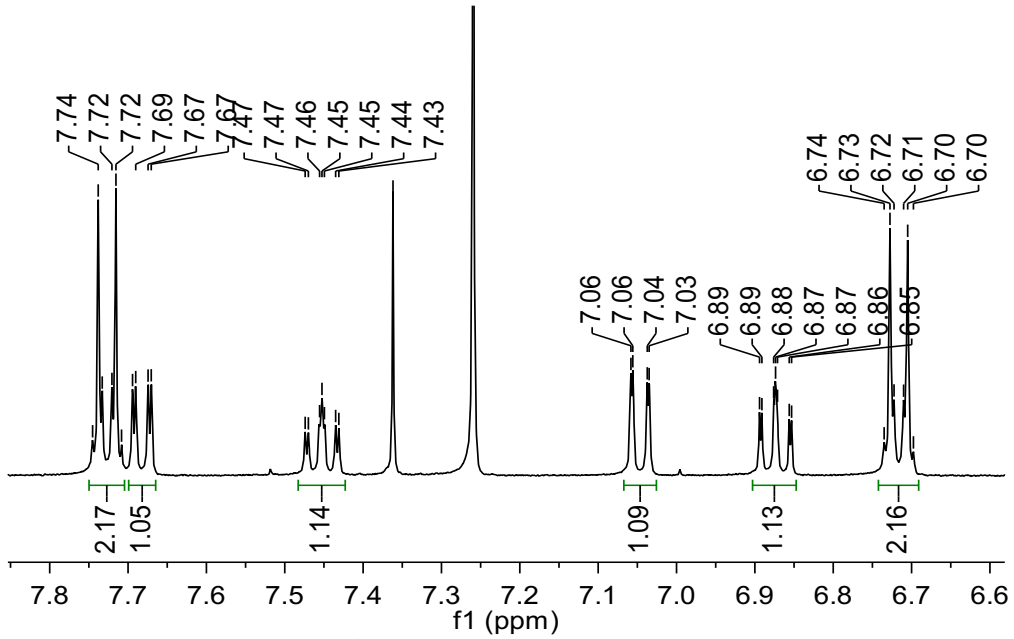
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
139.27
136.51
133.74
129.48
121.78
119.25
118.78
118.51
118.14
114.06
55.61
201.53
163.35
159.65

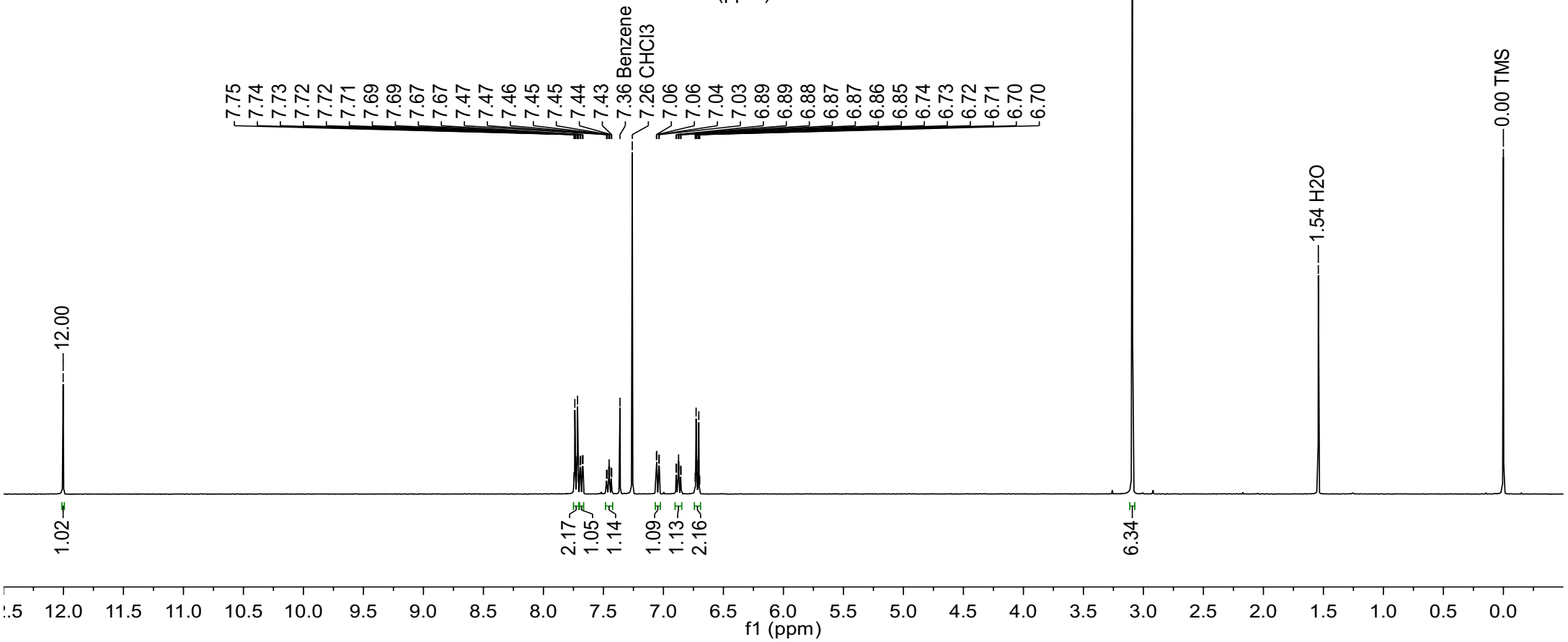
f1 (ppm)

2ao proton
400.13
298.0
CDCl3

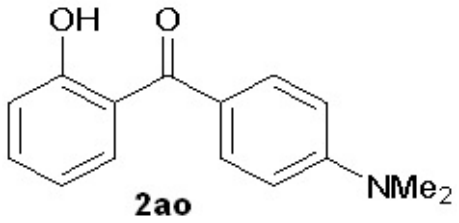


2ao



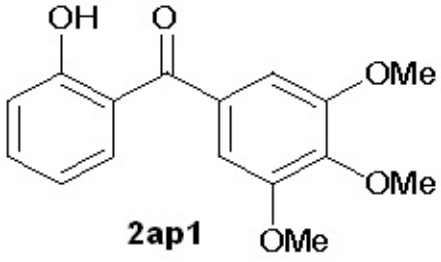


2ao carbon
100.56
298.6
CDCl3

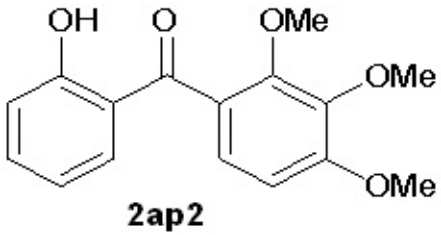


2ao

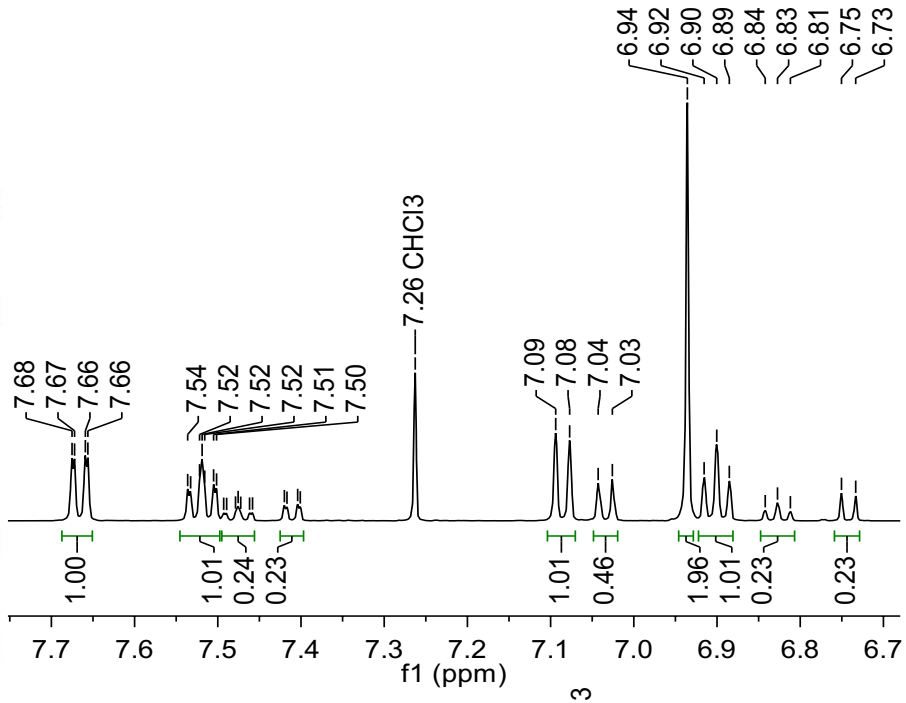
2ap1/2ap2 proton
500.13
298.0
CDCl3

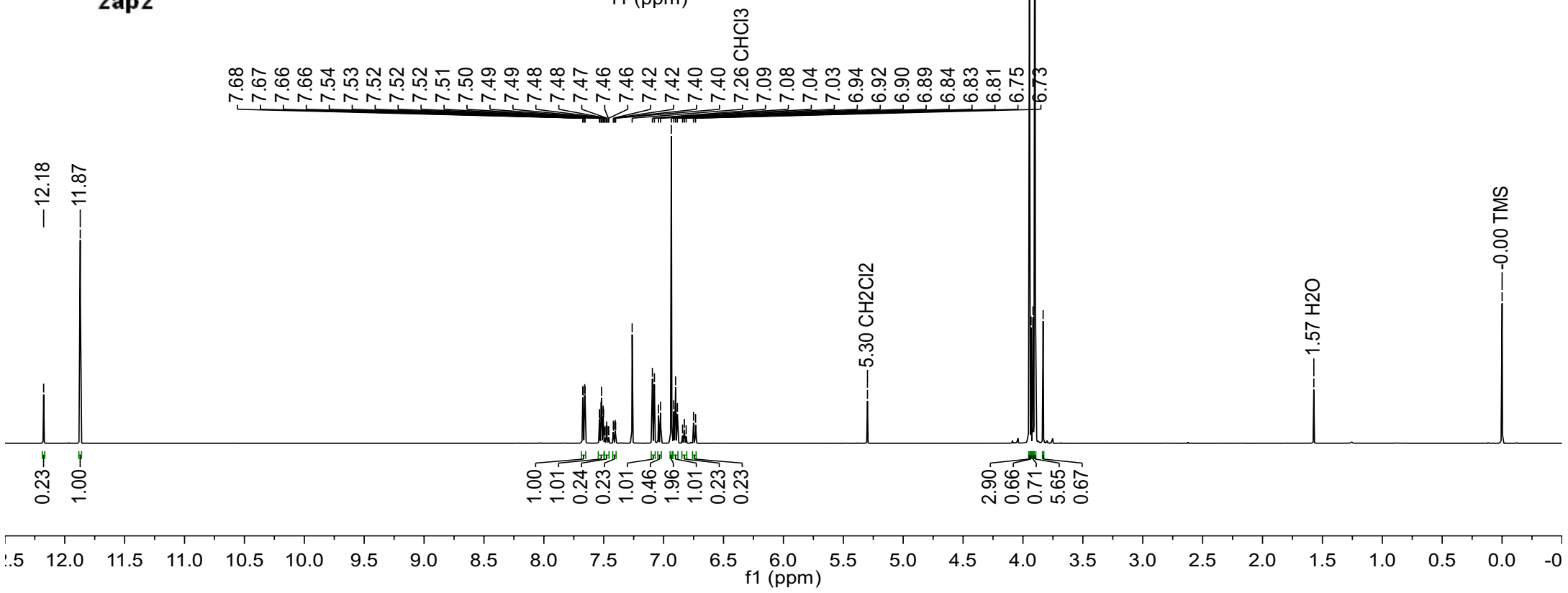


2ap1

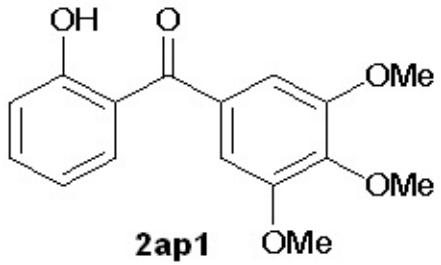


2ap2

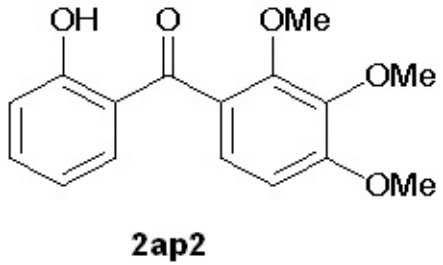




2ap1/2ap2 carbon
125.77
298.0
CDCl3

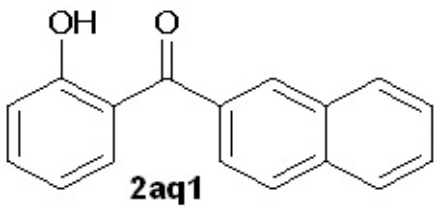


2ap1

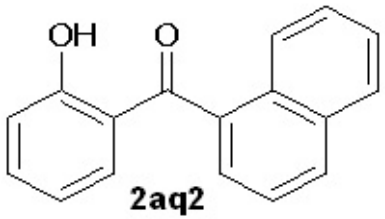


2ap2

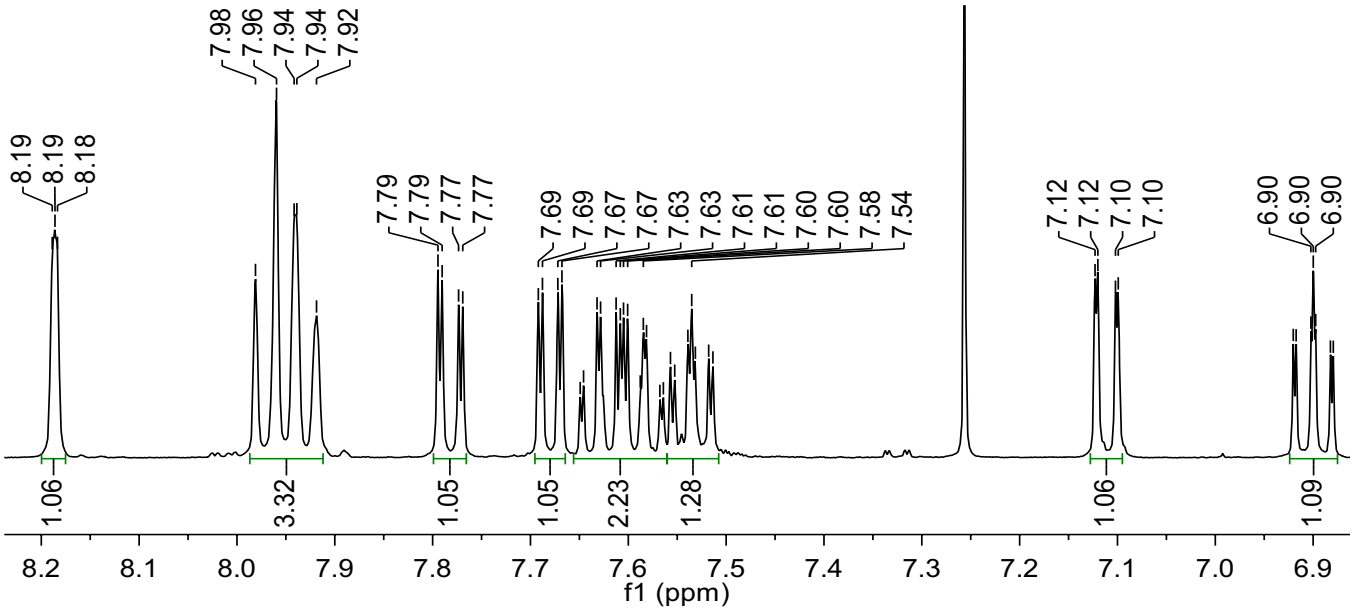
2aq1/2aq2 proton
400.13
298.0
CDCl3

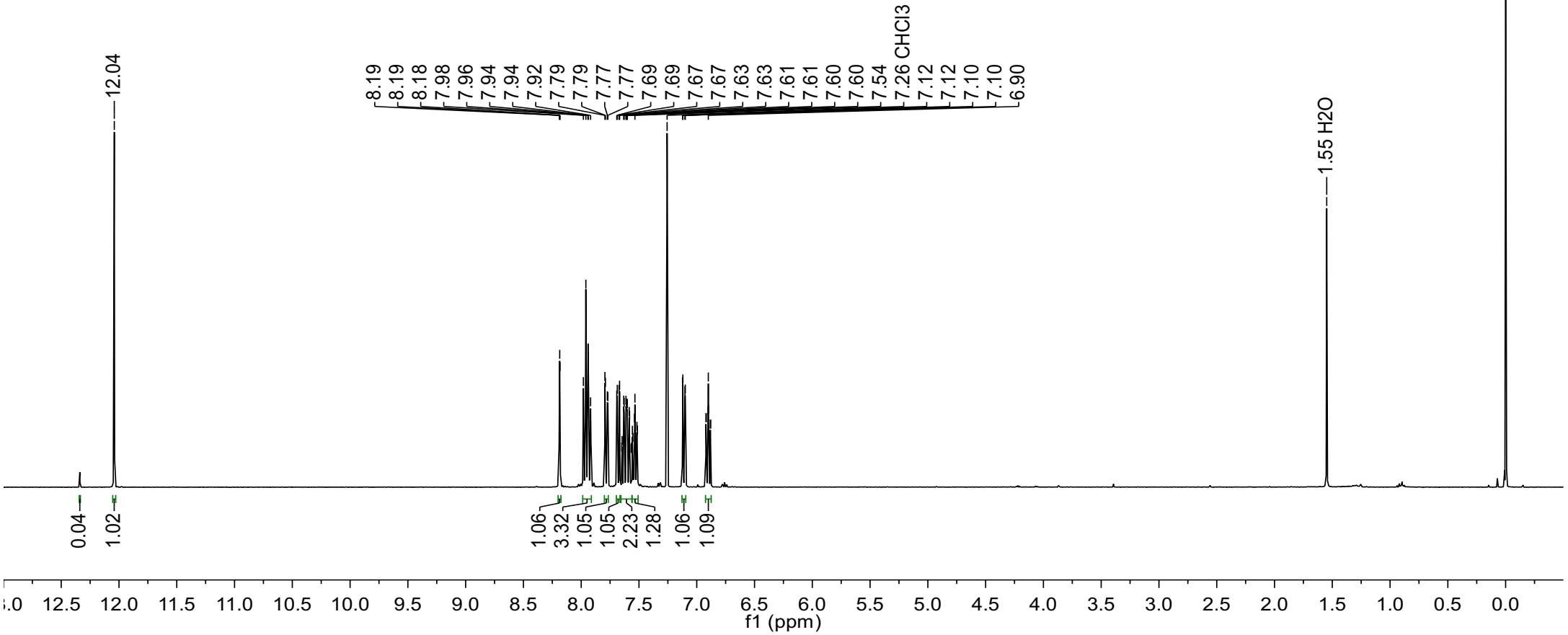


2aq1

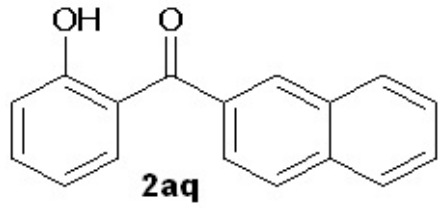


2aq2



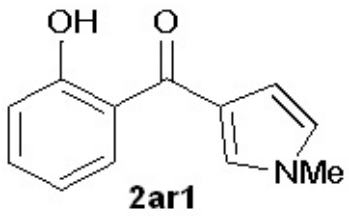


2aq1/2aq2 carbon
100.62
298.0
CDCl3

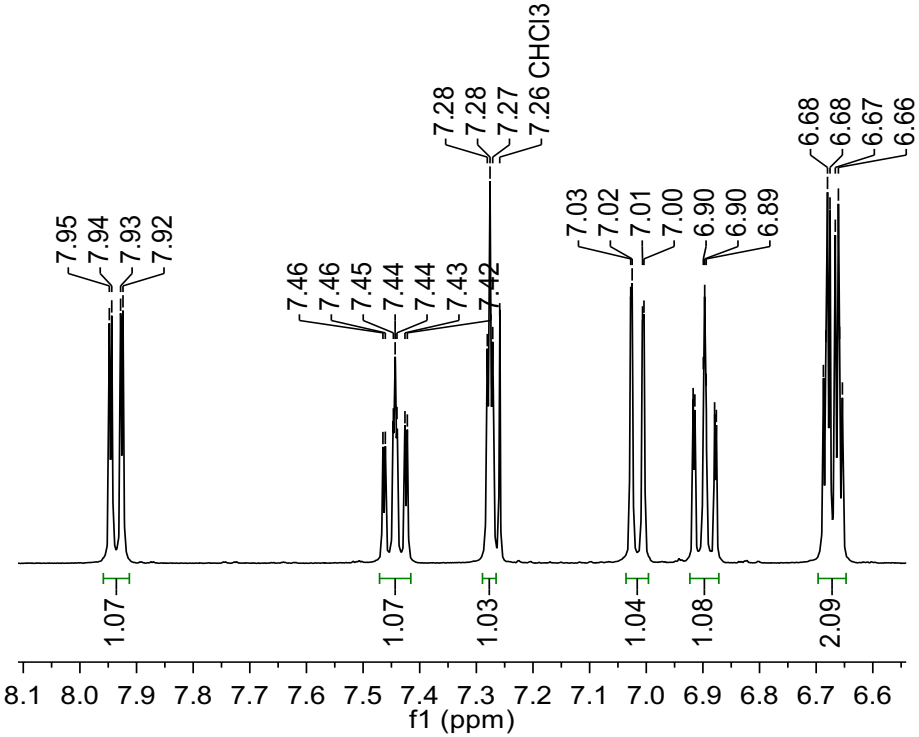


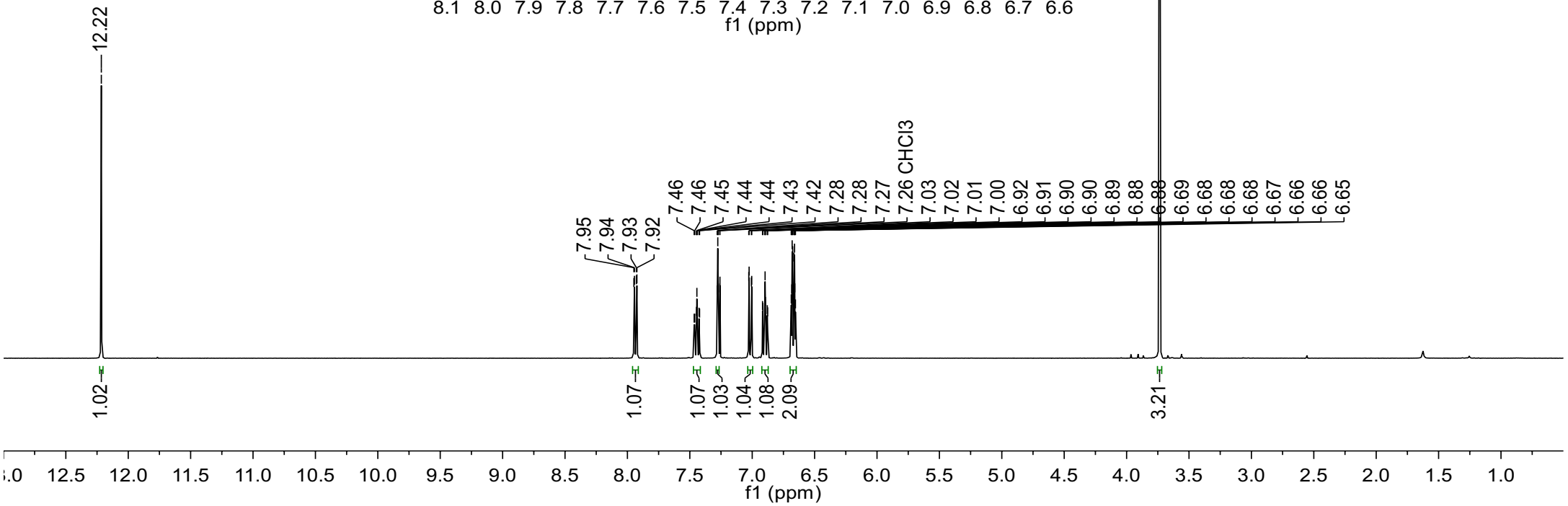
2aq

2ar1 proton
400.13
295.6
CDCl3

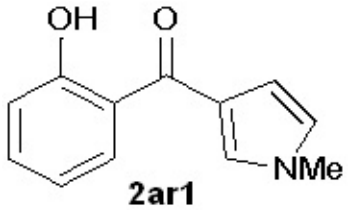


2ar1

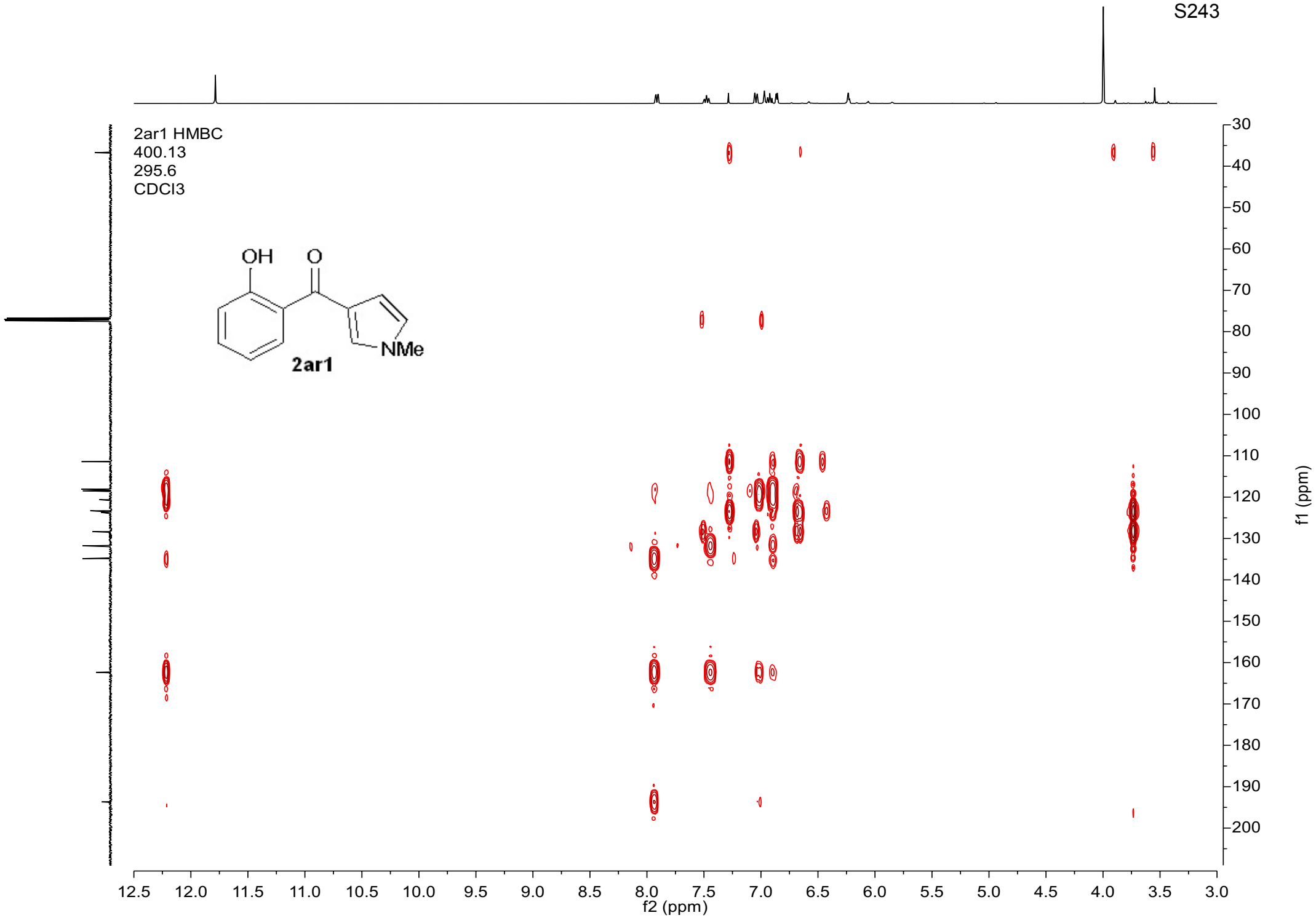




2ar1 carbon
100.62
298.0
CDCl3

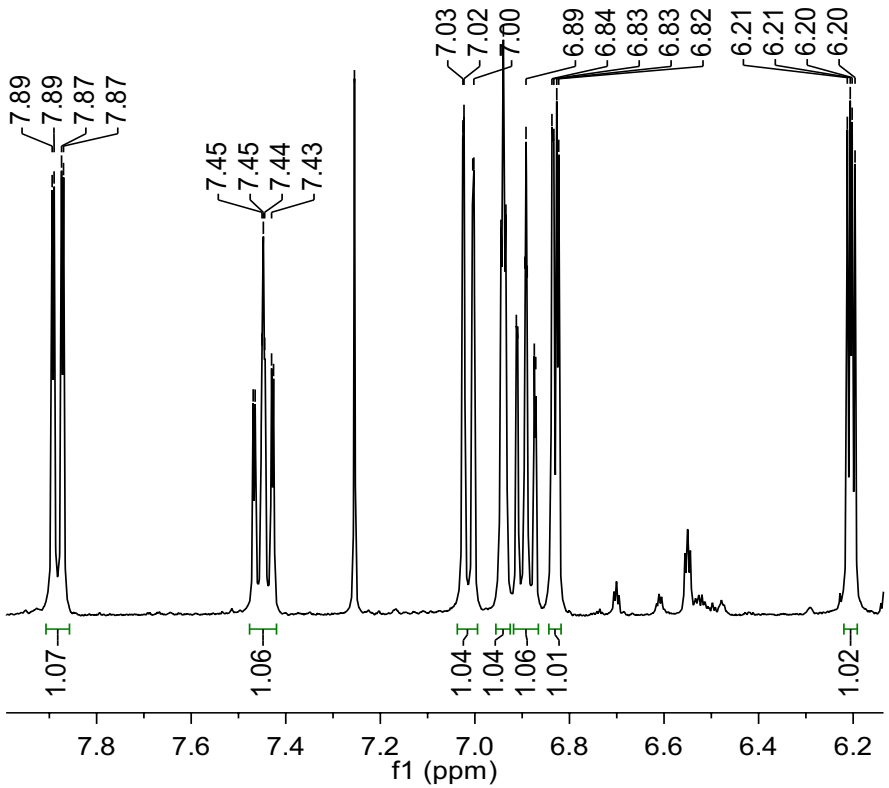


2ar1

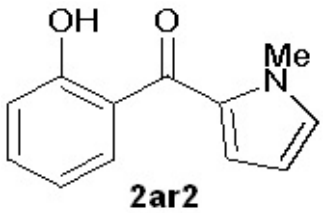

2ar1 HMBC
400.13
295.6
CDCl3
OH
O
NMe
2ar1
f2 (ppm)
f1 (ppm)

2ar2 proton
399.87
298.0
CDCl3

2ar2



2ar2 carbon
100.56
298.0
CDCl3

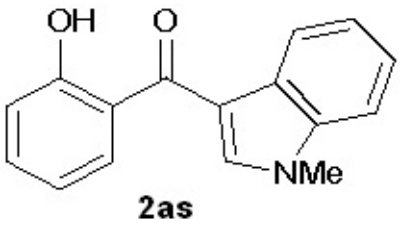


2ar2

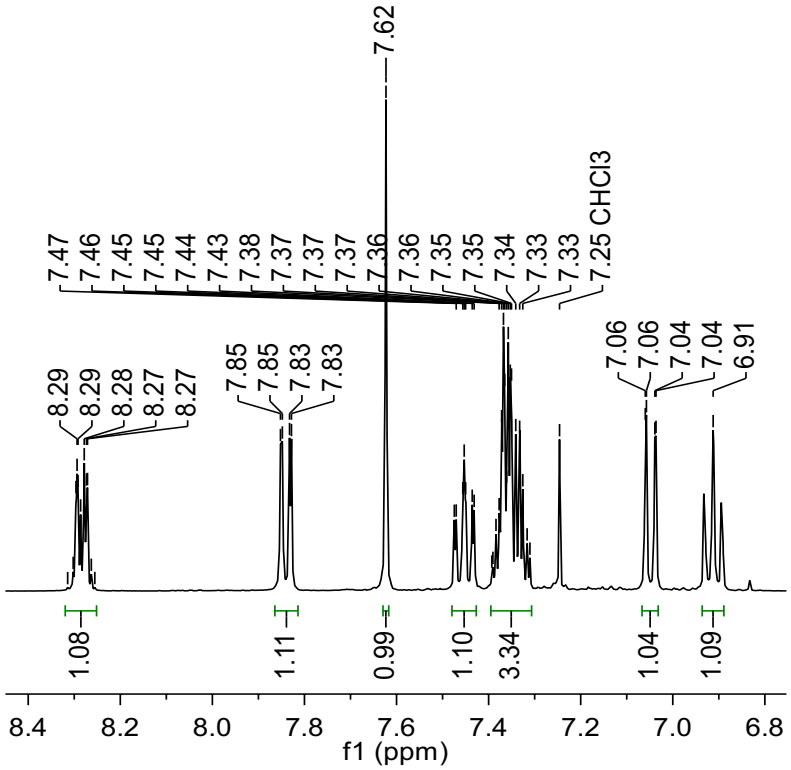
77.48 CHCl3
77.16 CHCl3
76.84 CHCl3
134.99
132.46
131.80
129.85
122.78
120.81
118.60
118.09
108.65
188.97
162.34
37.21

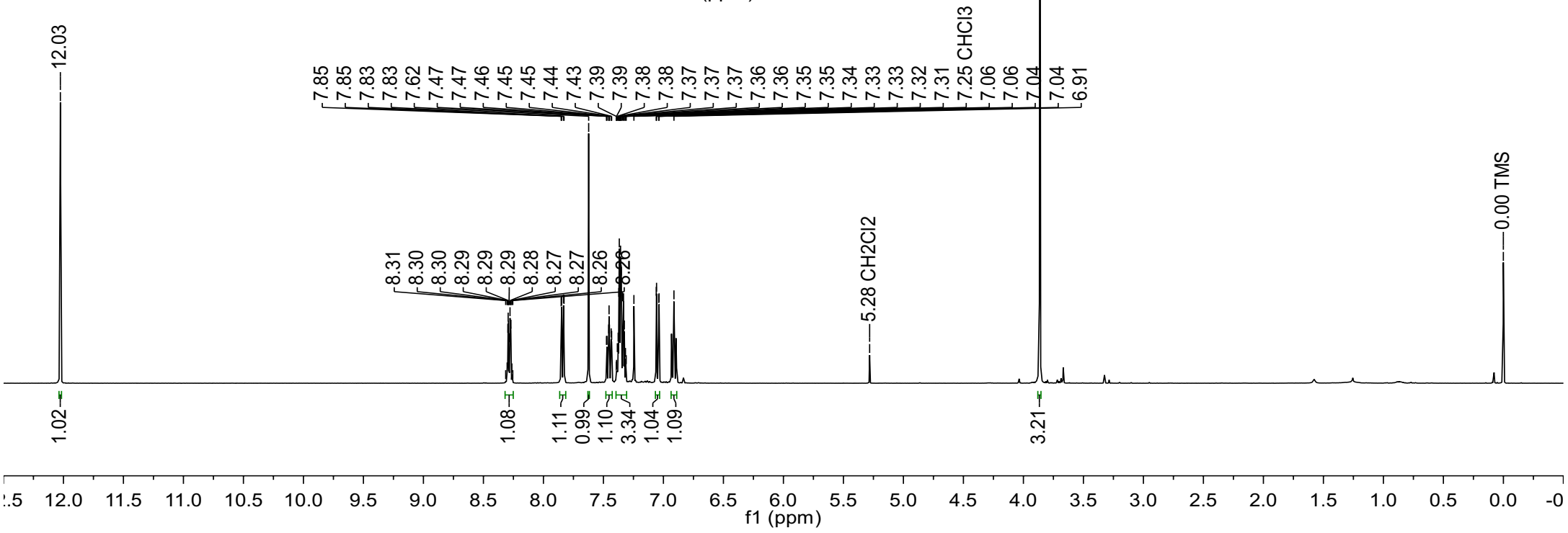
f1 (ppm)

2as proton
399.87
298.0
CDCl3

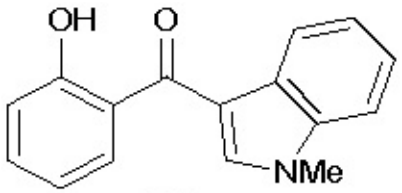


2as



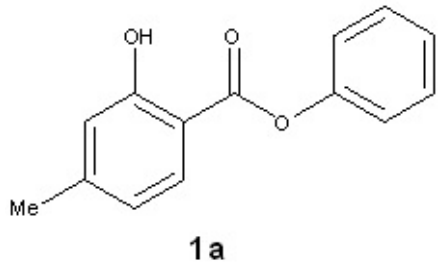


2as carbon
100.56
298.0
CDCl3



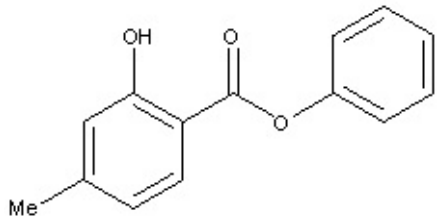
2as

1a proton
399.87
298.0
CDCl3



1a

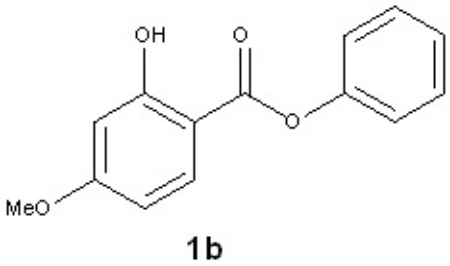
1a 13C
100.56
298.1
CDCl3



1a

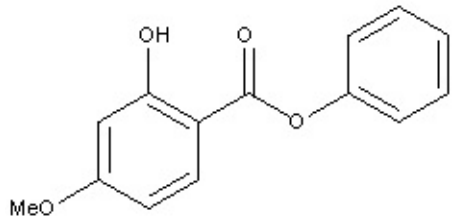
1b proton
399.87
298.0
CDCl3

S250



1b

1b carbon13
100.56
298.0
CDCl3

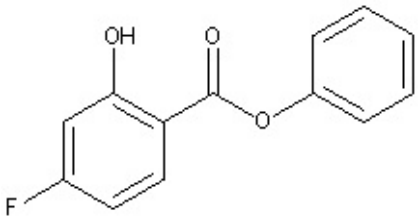


1b

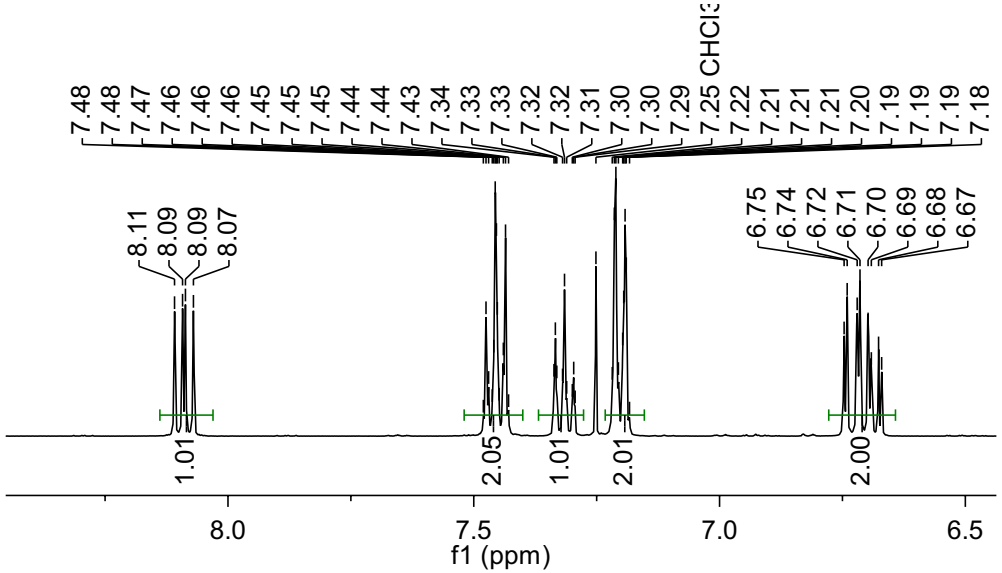
77.5 CDCl3
77.2 CDCl3
76.9 CDCl3
131.9
129.7
126.4
121.9
108.2
105.0
101.0
55.8
168.9
166.5
164.7
150.4

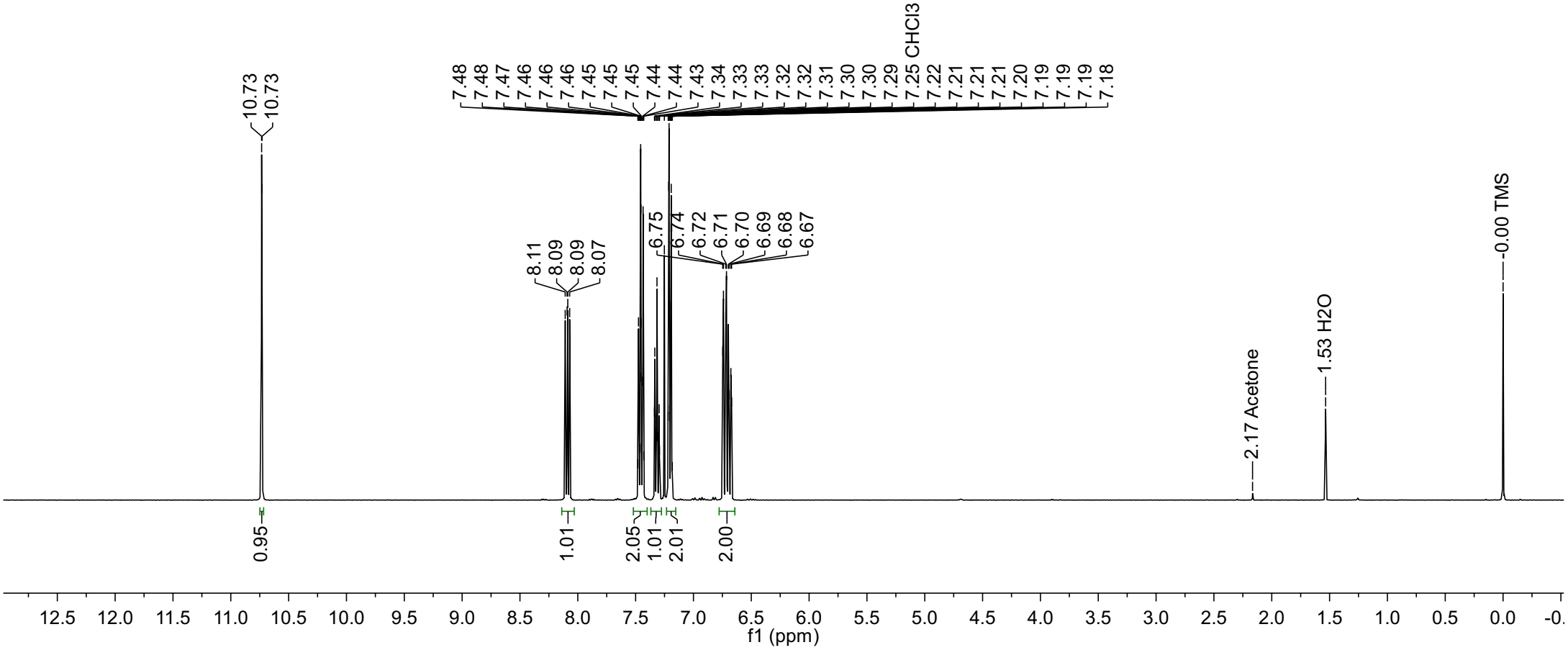
f1 (ppm)

1c proton
399.87
298.0
CDCl3

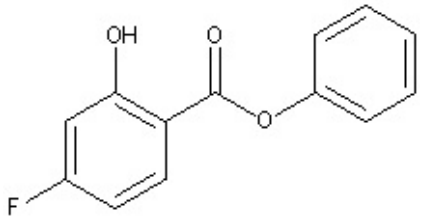


1c



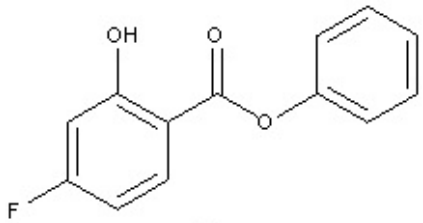


1c 13C
100.56
298.1
CDCl3



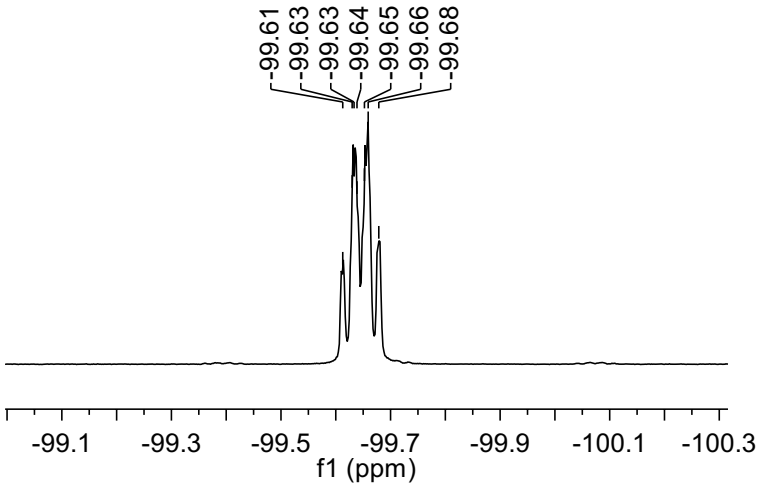
1c

1c no decoupling 19F
376.46
298.0
CDCl3



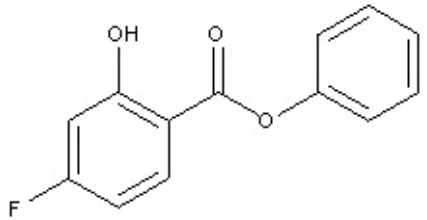
1c

-99.61
-99.63
-99.63
-99.64
-99.65
-99.66
-99.68



10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

1c with {1H} decoupling 19F
376.46
298.0
CDCl3

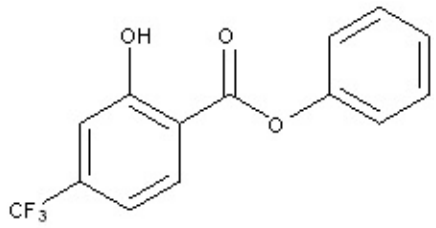


1c

-99.65

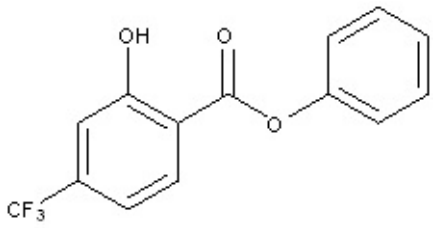
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

1d proton
399.87
298.0
CDCl3



1d

1d carbon
100.56
298.0
CDCl3



1d

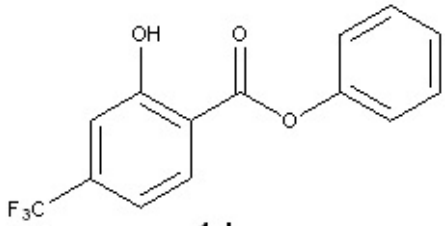
131.43
129.94
127.35
126.91
124.63
121.91
121.65
119.19
116.08
116.04
116.00
115.97
115.54
115.51
115.47
115.43
114.78

77.52 CDCl3
77.20 CDCl3
76.88 CDCl3

168.28
162.28
150.02
138.30
137.97
137.64
137.31

200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

1d no decoupling 19F
376.46
298.0
CDCl3



1d

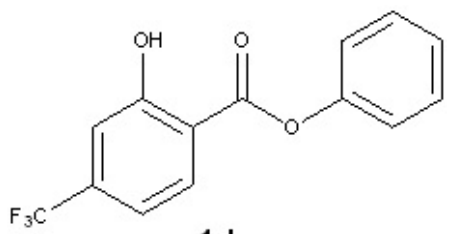
-63.82
-63.85

-63.85

-63.5 -63.6 -63.7 -63.8 -63.9 -64.0 -64.1 -64.2
f1 (ppm)

10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

1d with {1H} decoupling 19F
376.46
298.1
CDCl3

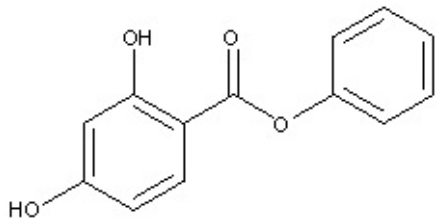


1d

-63.85

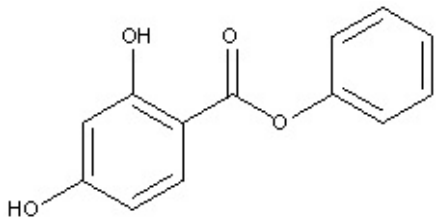
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

1e proton
399.87
298.0
CDCl3



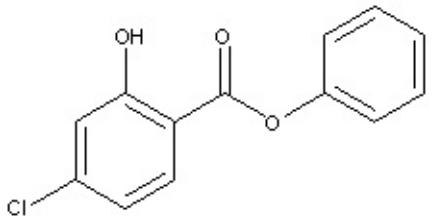
1e

1e carbon
100.56
298.1
CDCl3



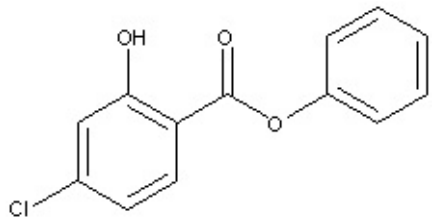
1e

1f proton
399.87
298.0
CDCl3



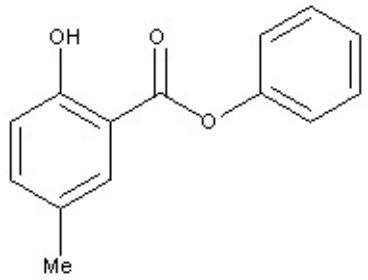
1f

1f carbon
100.56
298.1
CDCl3



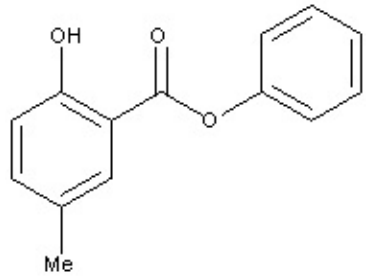
1f

1g proton
399.87
298.0
CDCl3



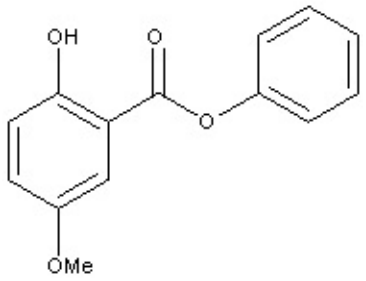
1g

1g carbon
100.56
298.1
CDCl3



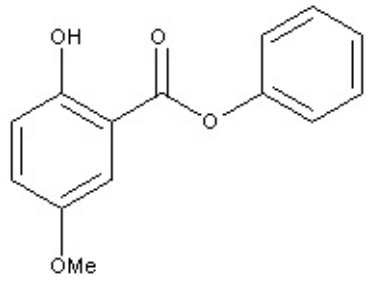
1g

1h proton
399.87
298.0
CDCl3



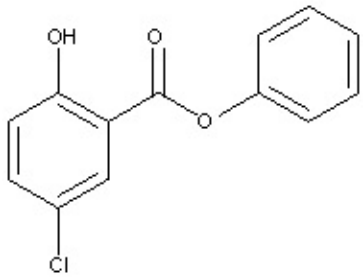
1h

1h carbon
100.56
298.1
CDCl3

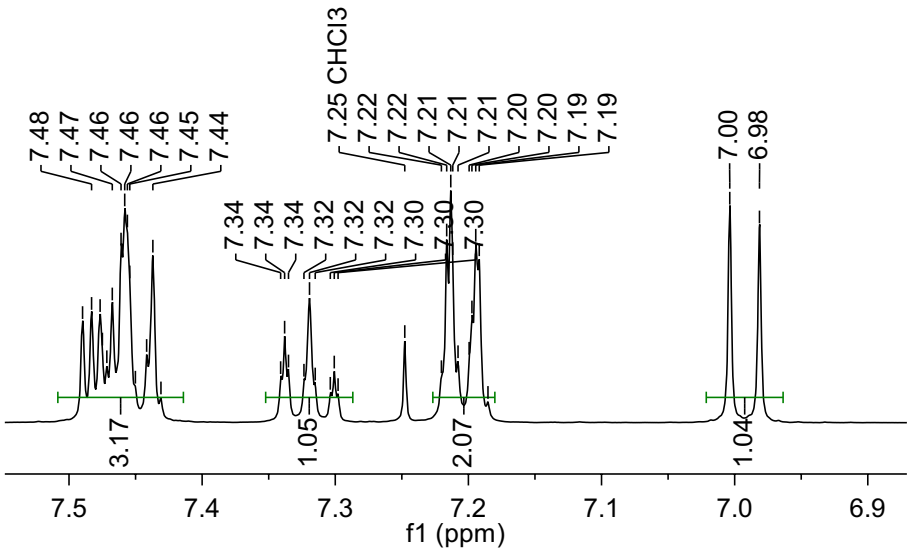


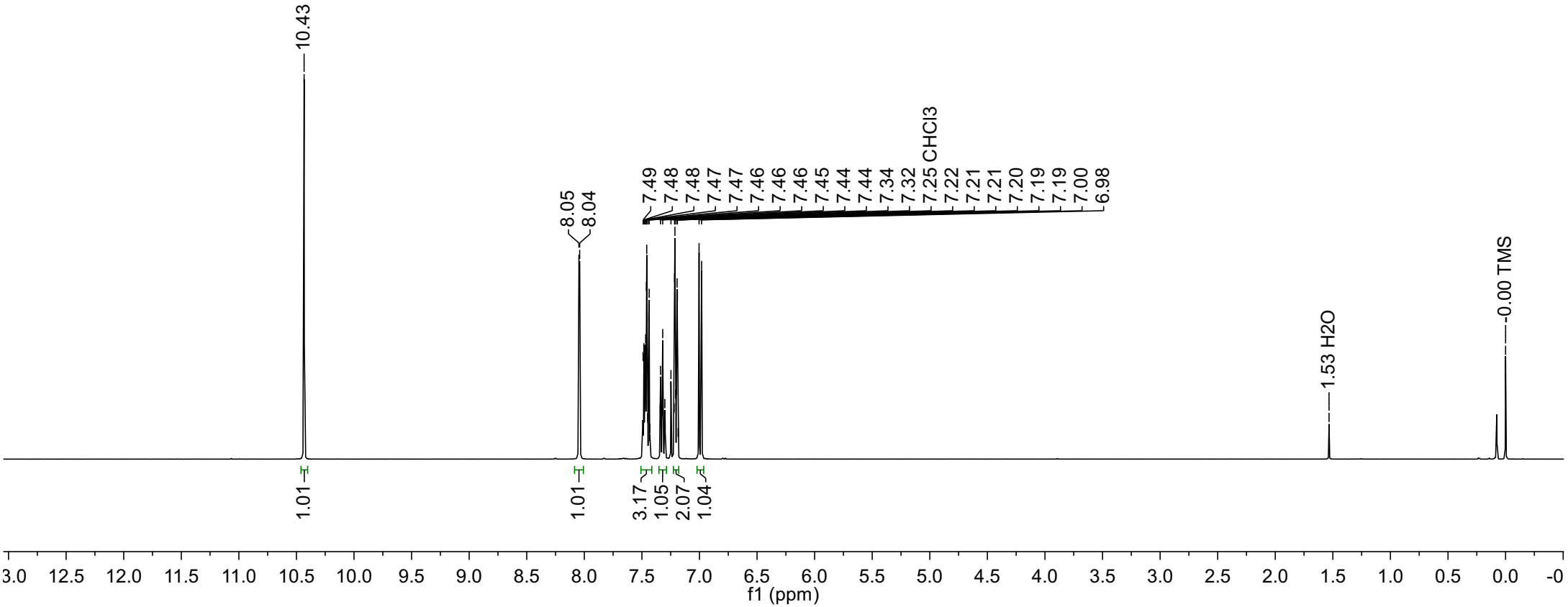
1h

1i proton
399.87
298.0
CDCl3

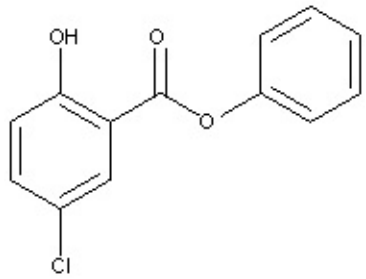


1i



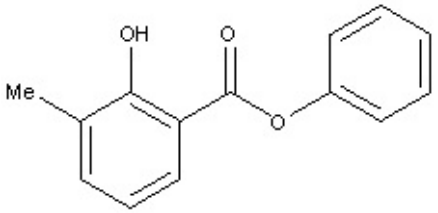


1i carbon
100.56
298.1
CDCl3

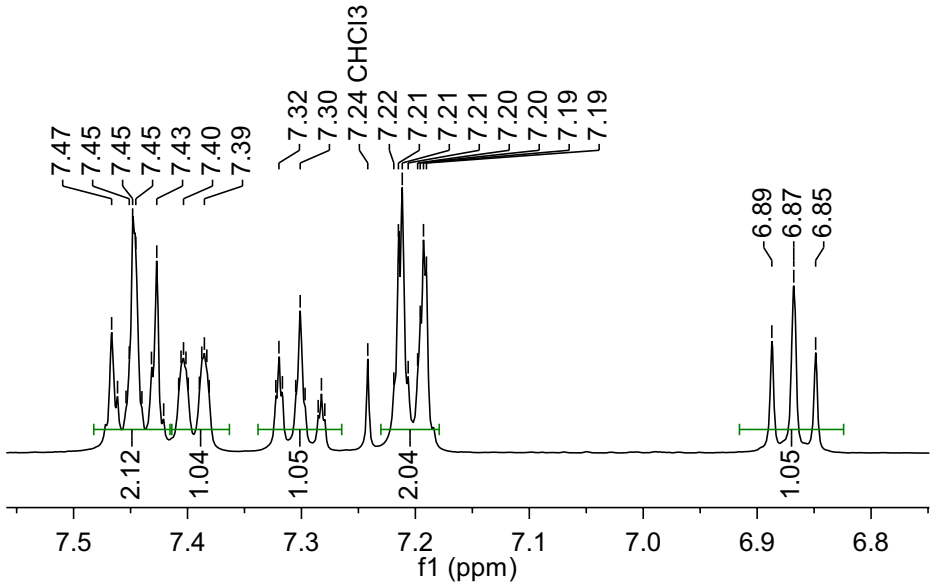


1i

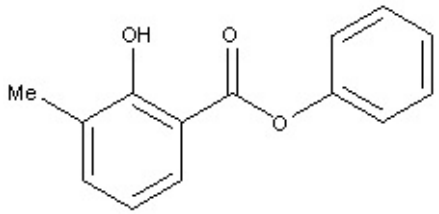
1j proton
399.87
298.0
CDCl3



1j

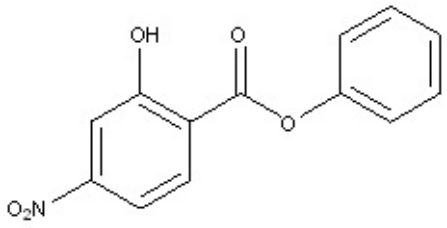


1j carbon
100.56
298.1
CDCl3

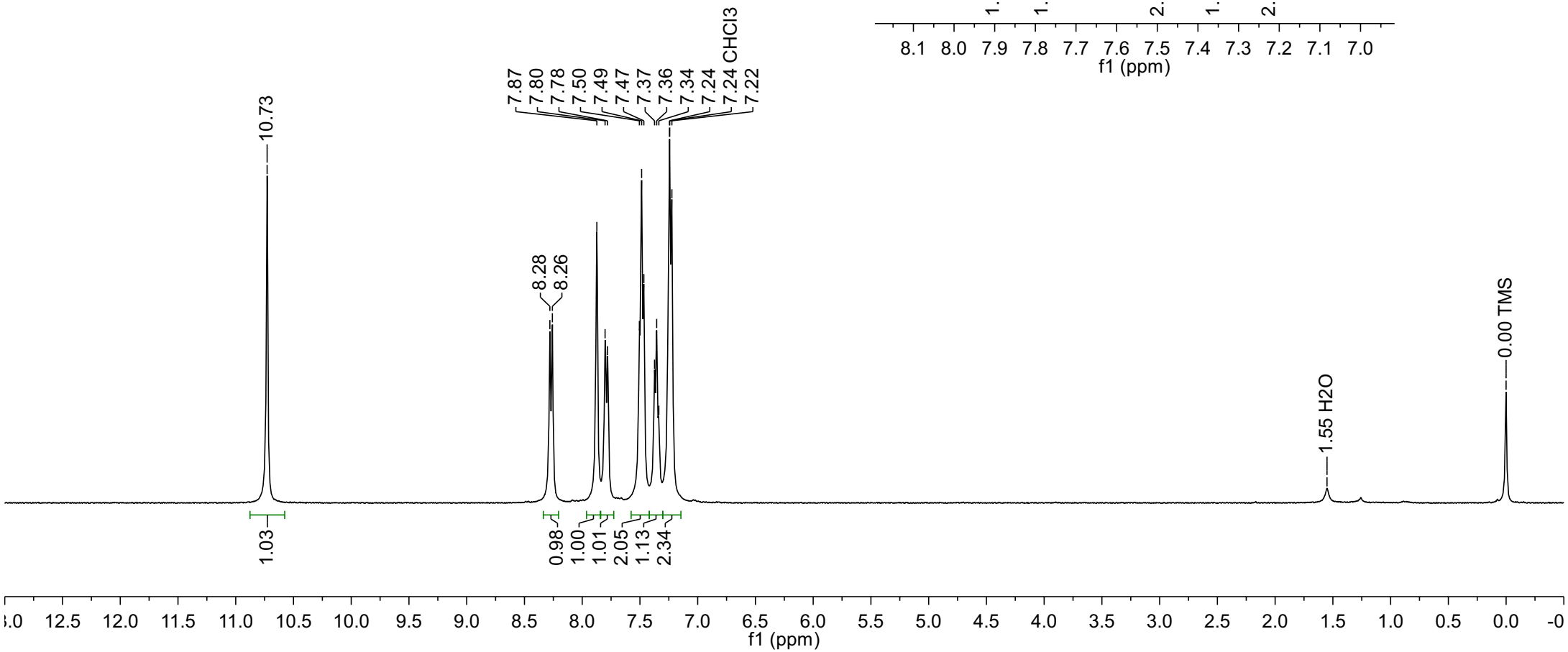


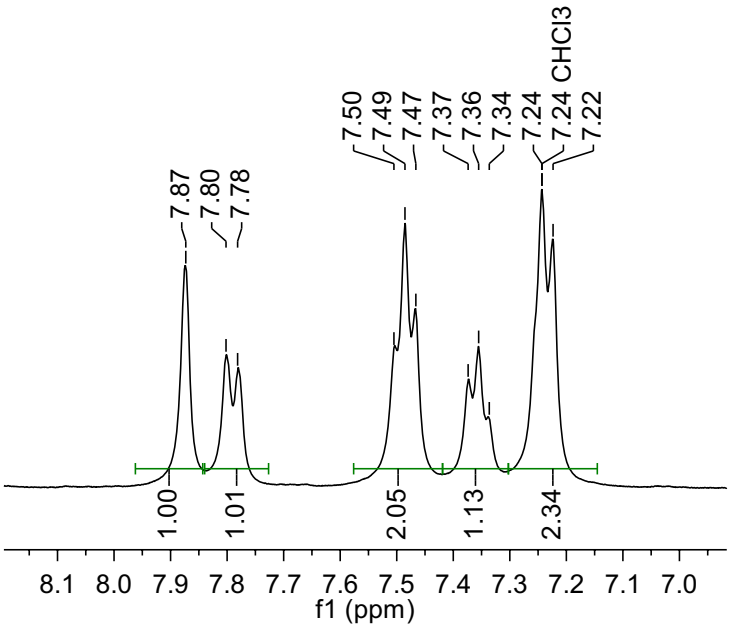
1j

1k proton
399.87
298.0
CDCl3

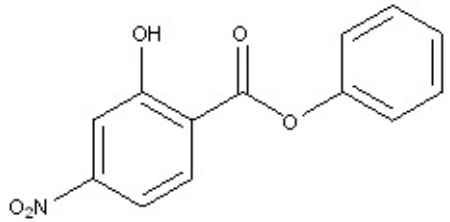


1k



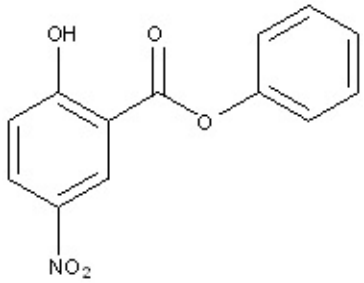


1k carbon
100.56
298.1
CDCl3

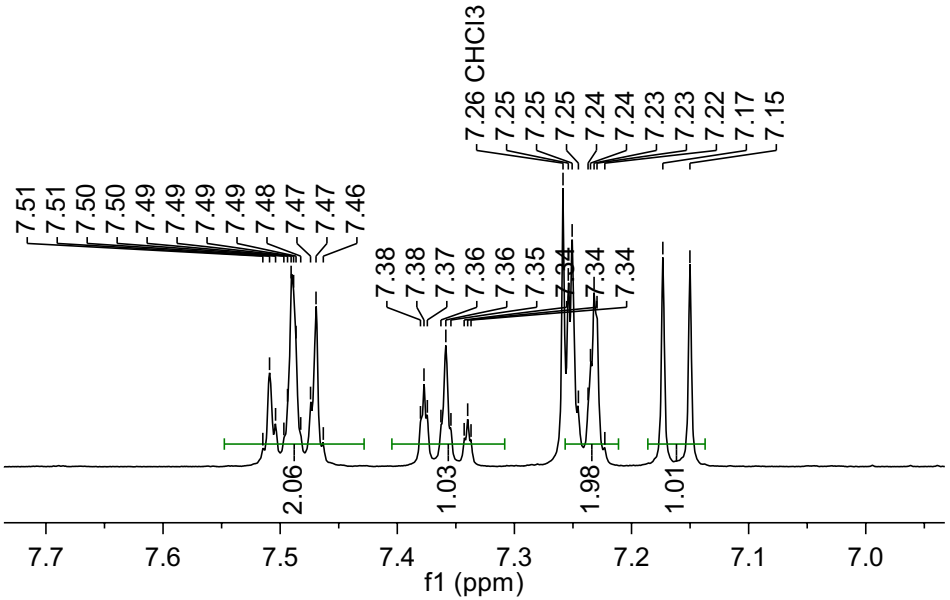


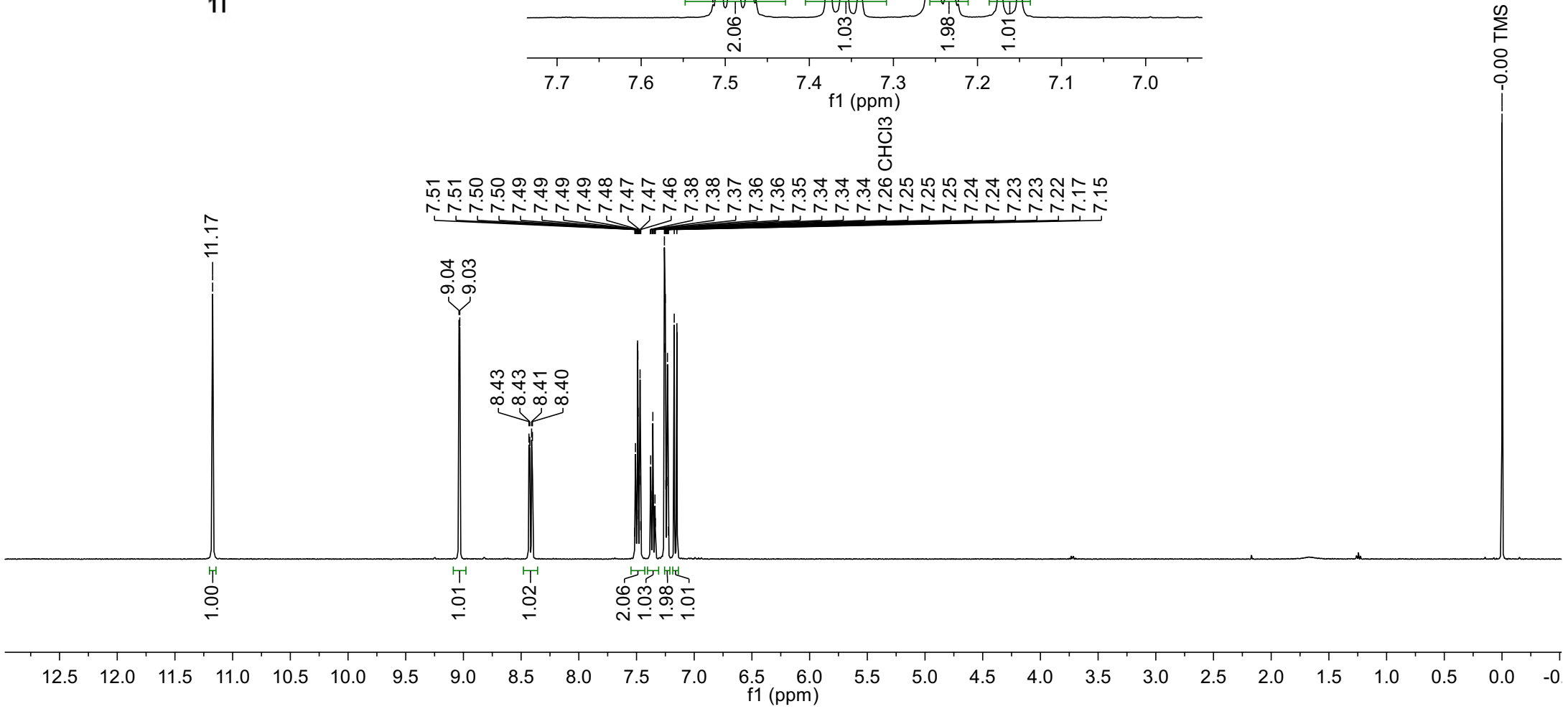
1k

1l proton
399.87
298.0
CDCl3

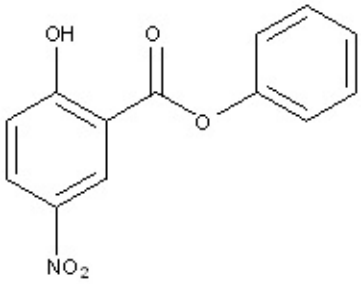


1l



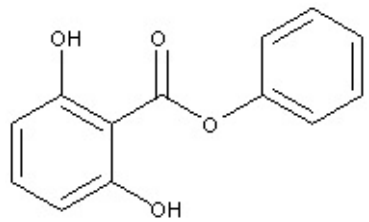


1l carbon
100.56
298.0
CDCl3



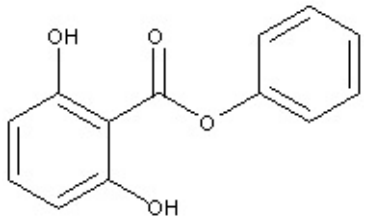
1l

1m proton
399.87
298.0
CDCl3



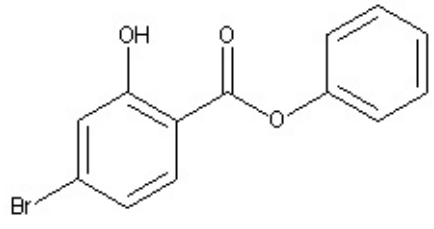
1m

1m carbon
100.56
298.1
CDCl3



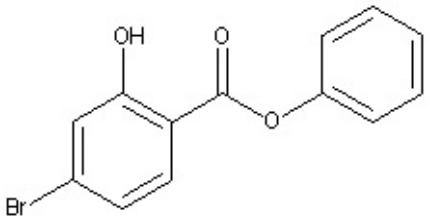
1m

1n proton
399.87
298.0
CDCl3



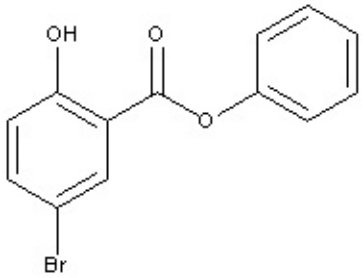
1n

1n carbon
100.56
298.1
CDCl3



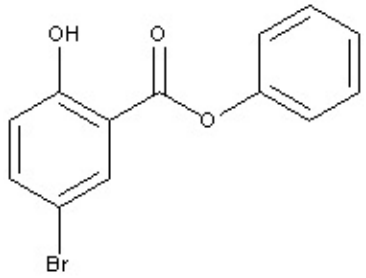
1n

1o proton
399.87
298.0
CDCl3



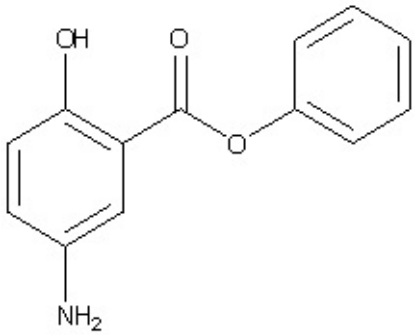
1o

1o carbon
100.56
298.1
CDCl3

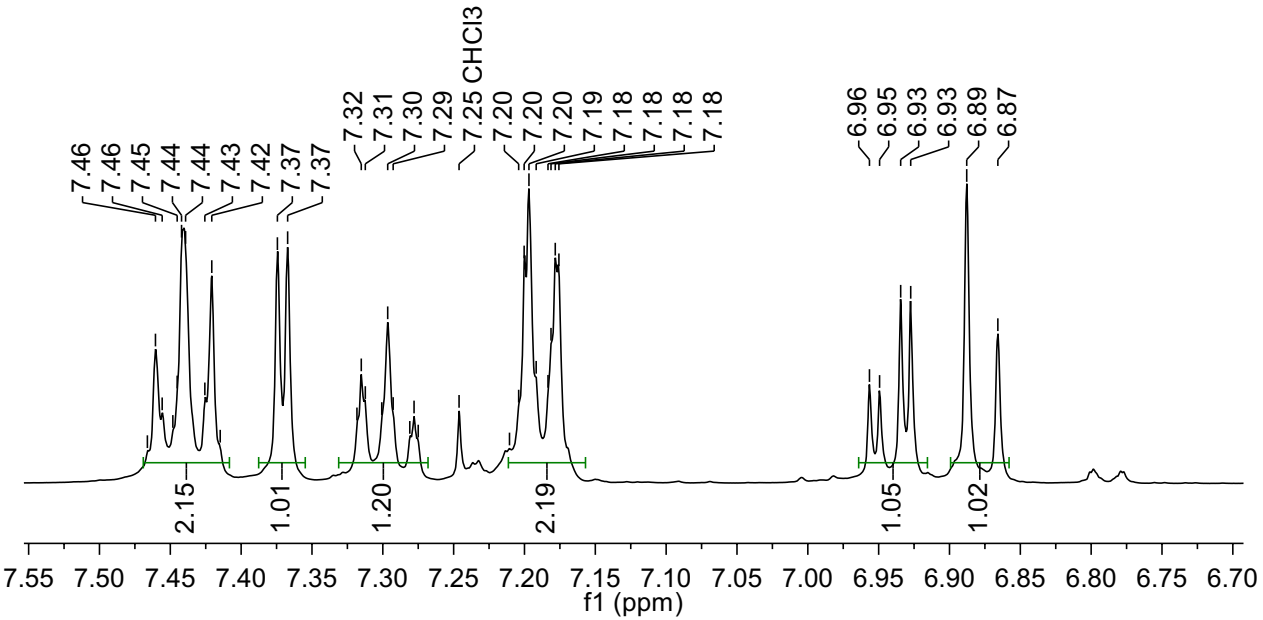


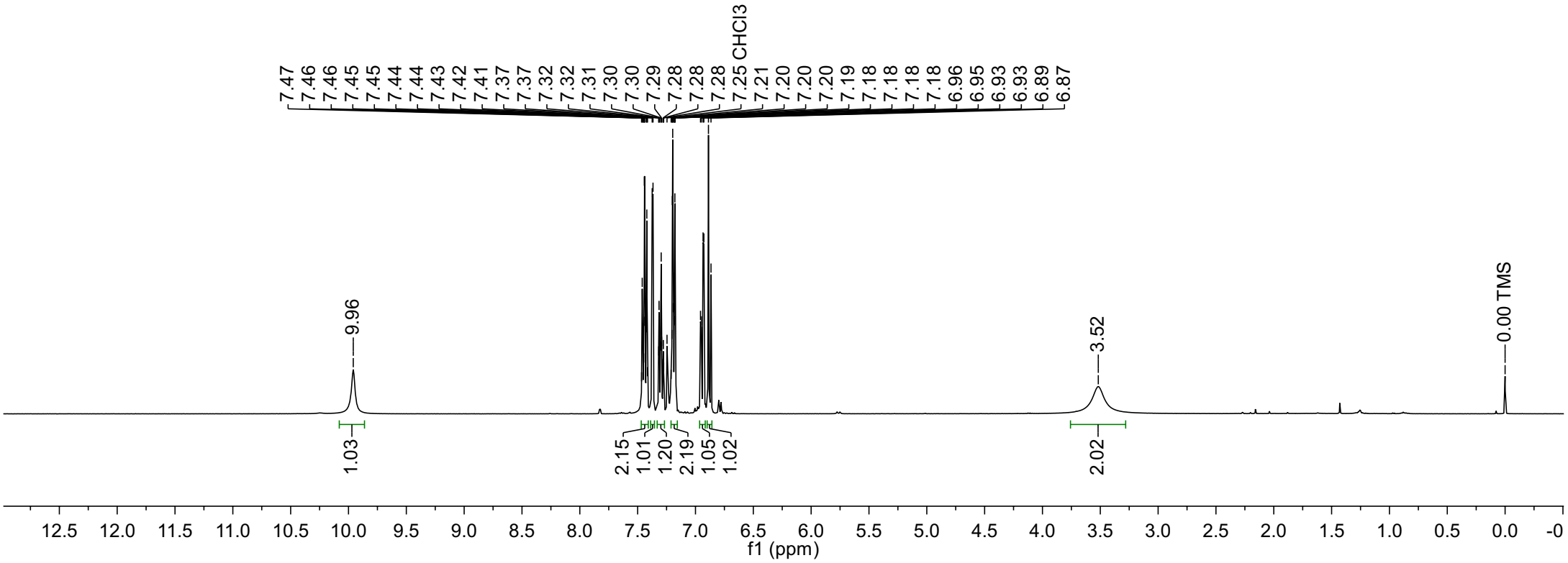
1o

1p proton
399.87
298.0
CDCl3

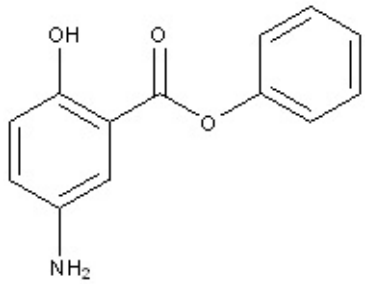


1p



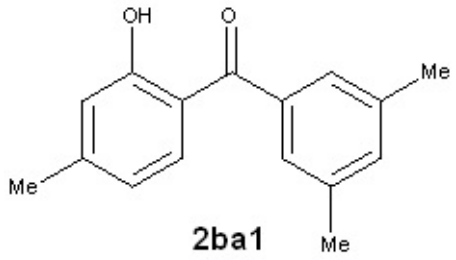


1p carbon
100.56
298.1
CDCl3

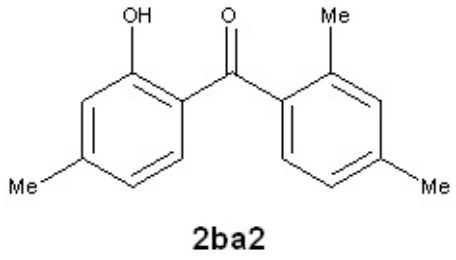


1p

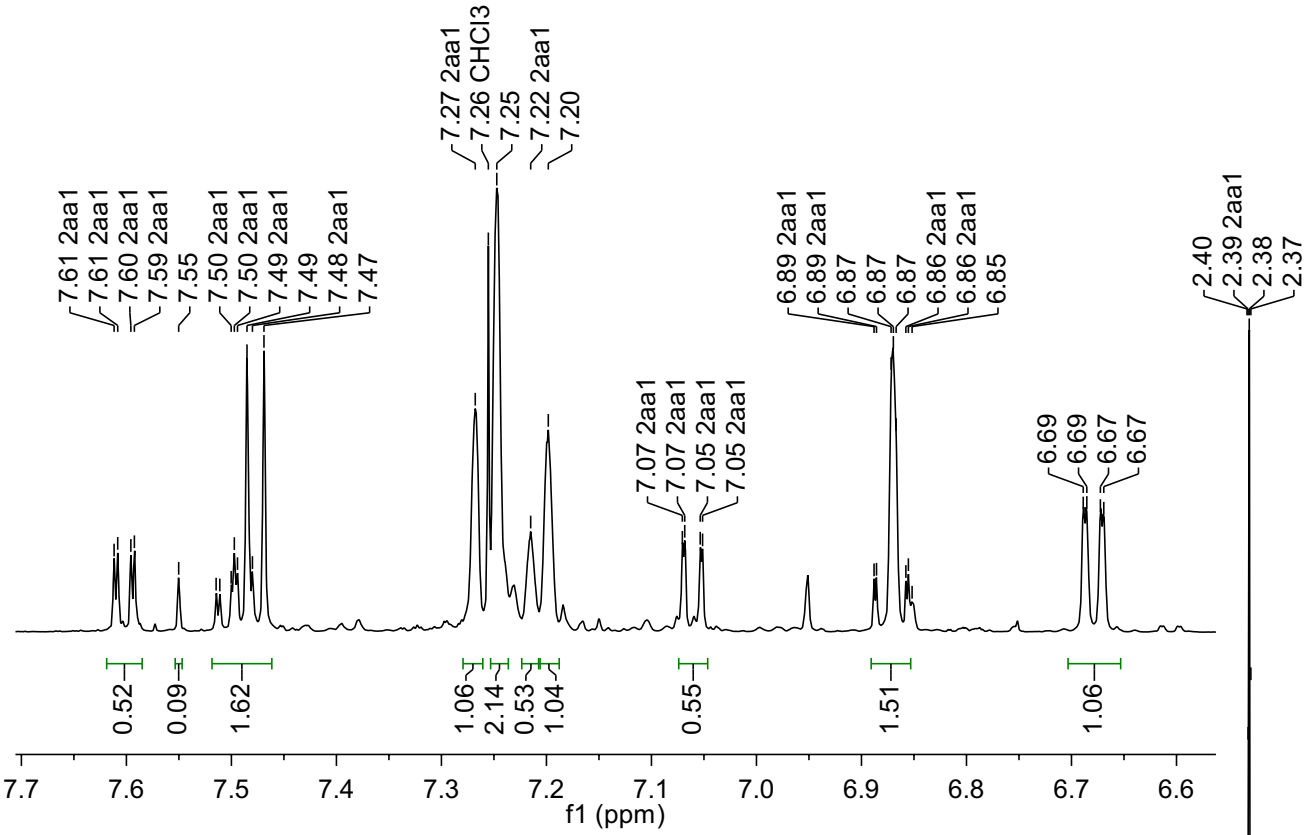
2ba1/2ba2 proton
500.15
298.0
CDCl3

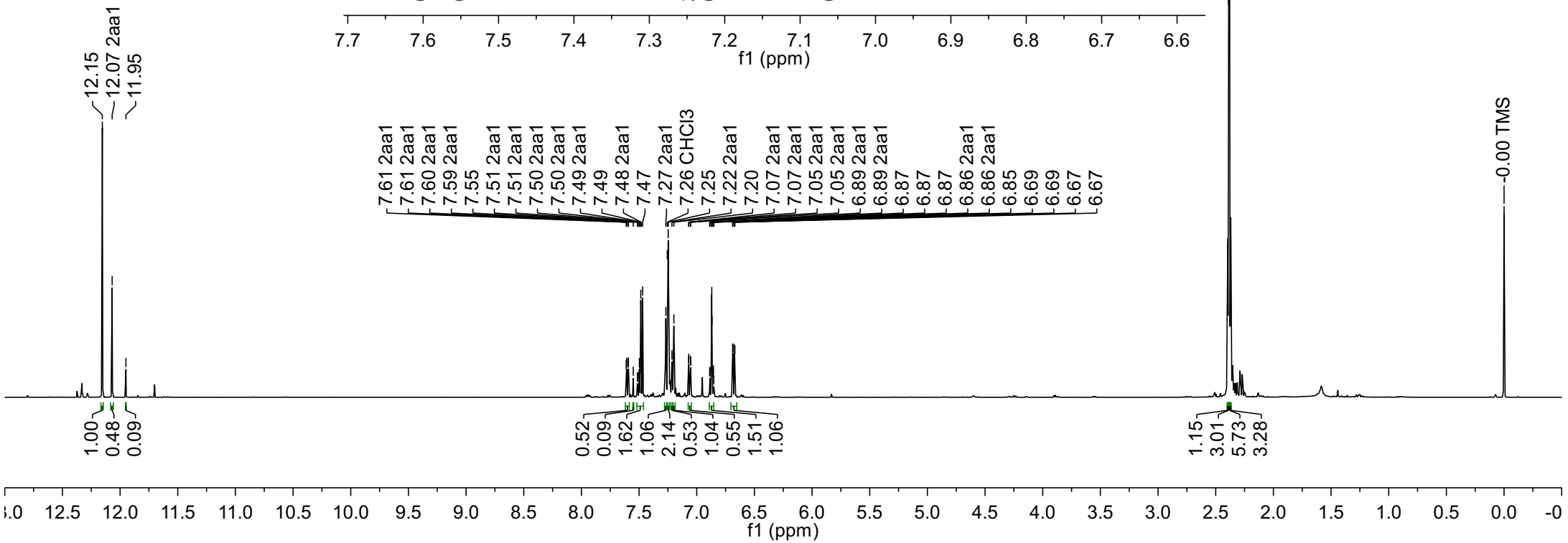


2ba1

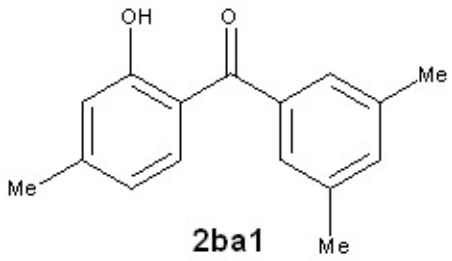


2ba2



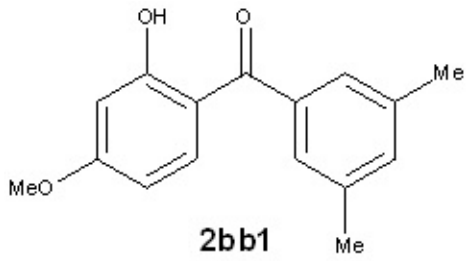


2ba1/2ba2 carbon
125.78
298.0
CDCl3

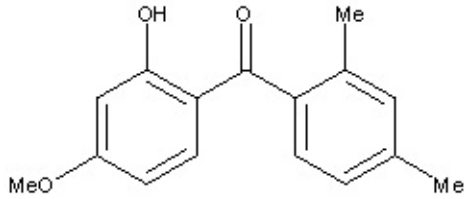


2ba1

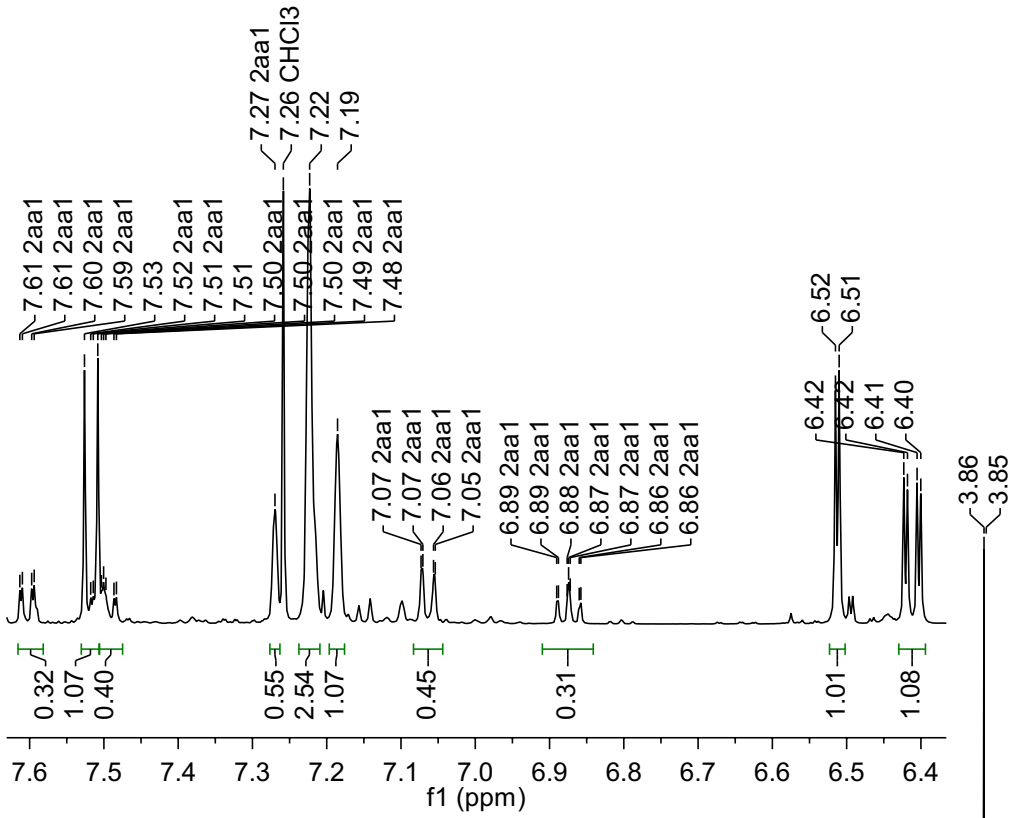
2bb1/2bb2 proton
500.15
298.0
CDCl3



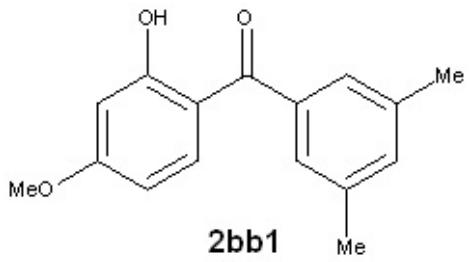
2bb1



2bb2

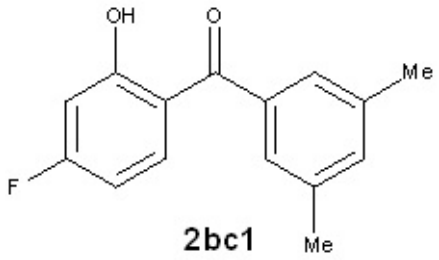


2bb1/2bb2 carbon
125.78
298.0
CDCl3

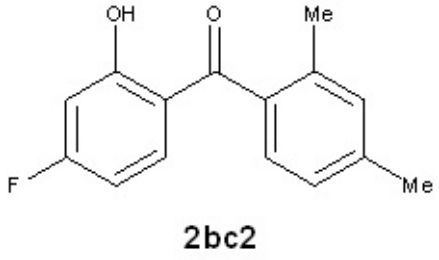


2bb1

2bc1/2bc2 proton
500.15
298.0
CDCl3

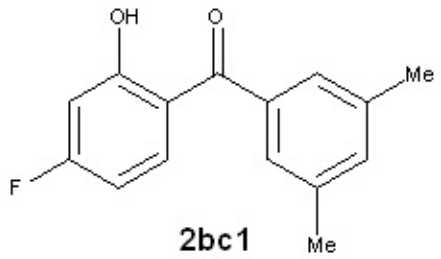


2bc1



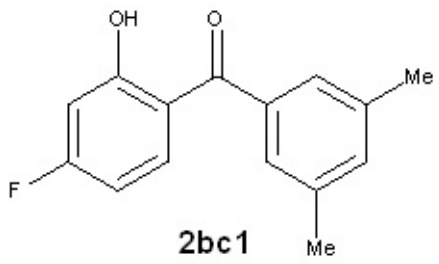
2bc2

2bc1/2bc2 carbon
125.78
298.0
CDCl3

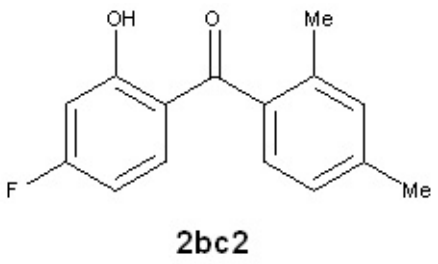


2bc1

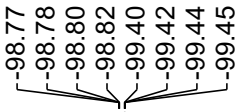
2bc1/2bc2 fluorine
470.56
298.0
CDCl3

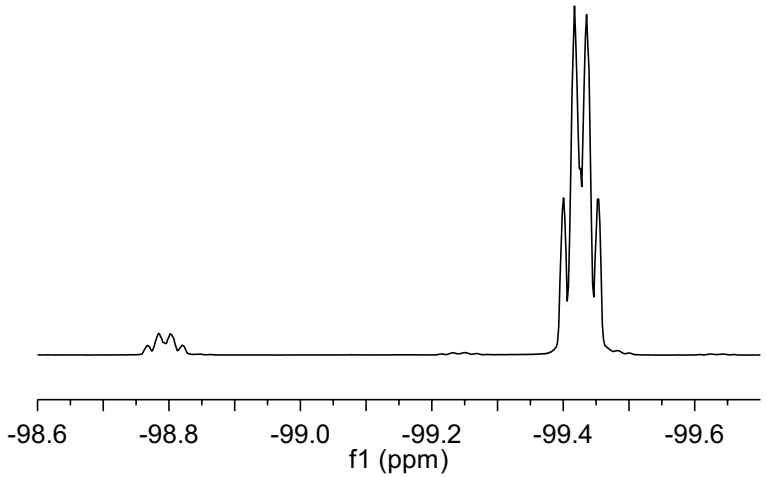


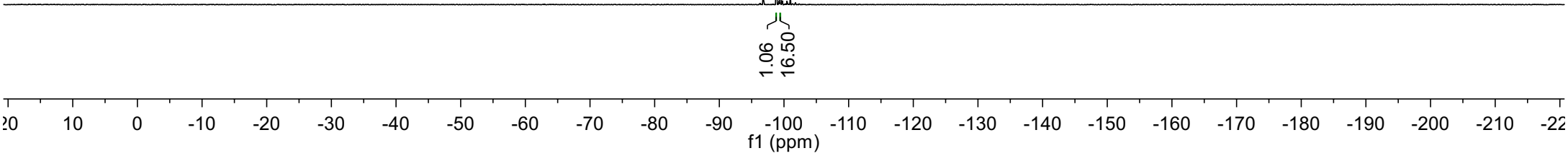
2bc1



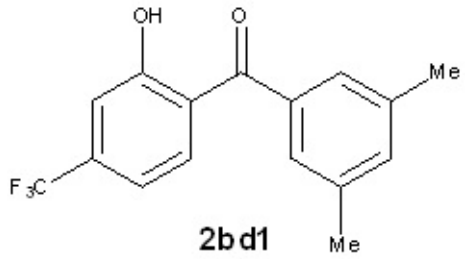
2bc2



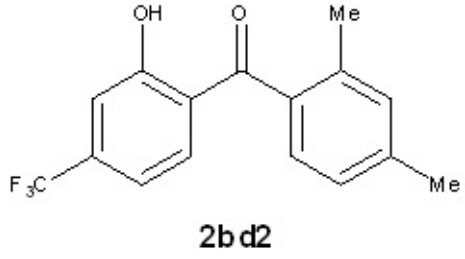




2bd1/2bd2 proton
500.15
298.0
CD2Cl2

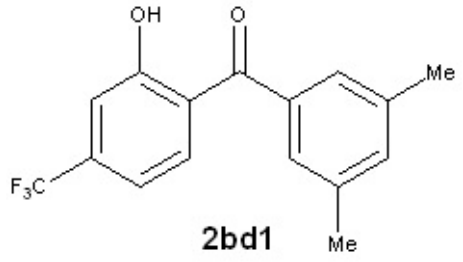


2bd1

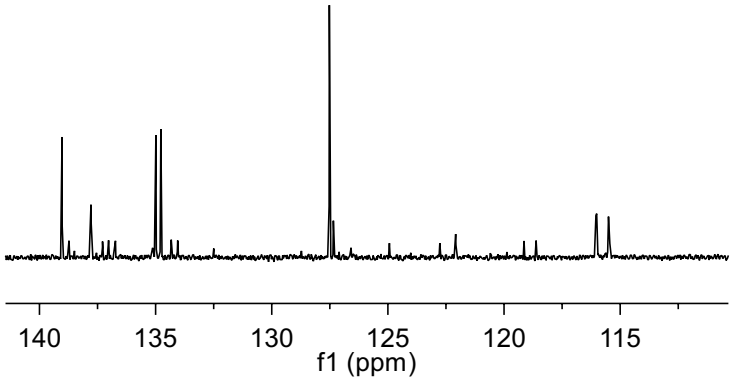


2bd2

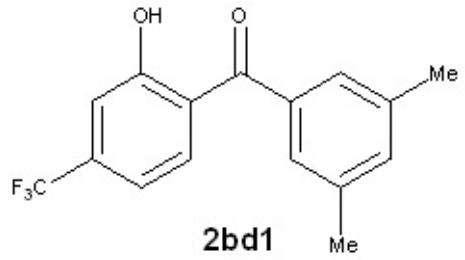
2bd1/2bd2 carbon
125.78
298.0
CD2Cl2



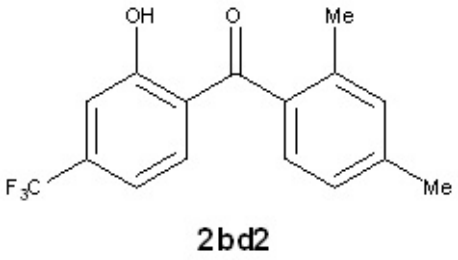
2bd1



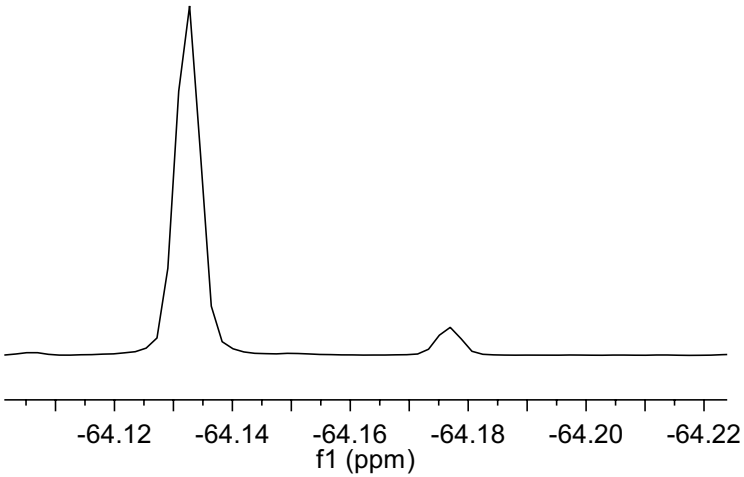
2bd1/2bd2 fluorine
470.56
298.0
CD2Cl2

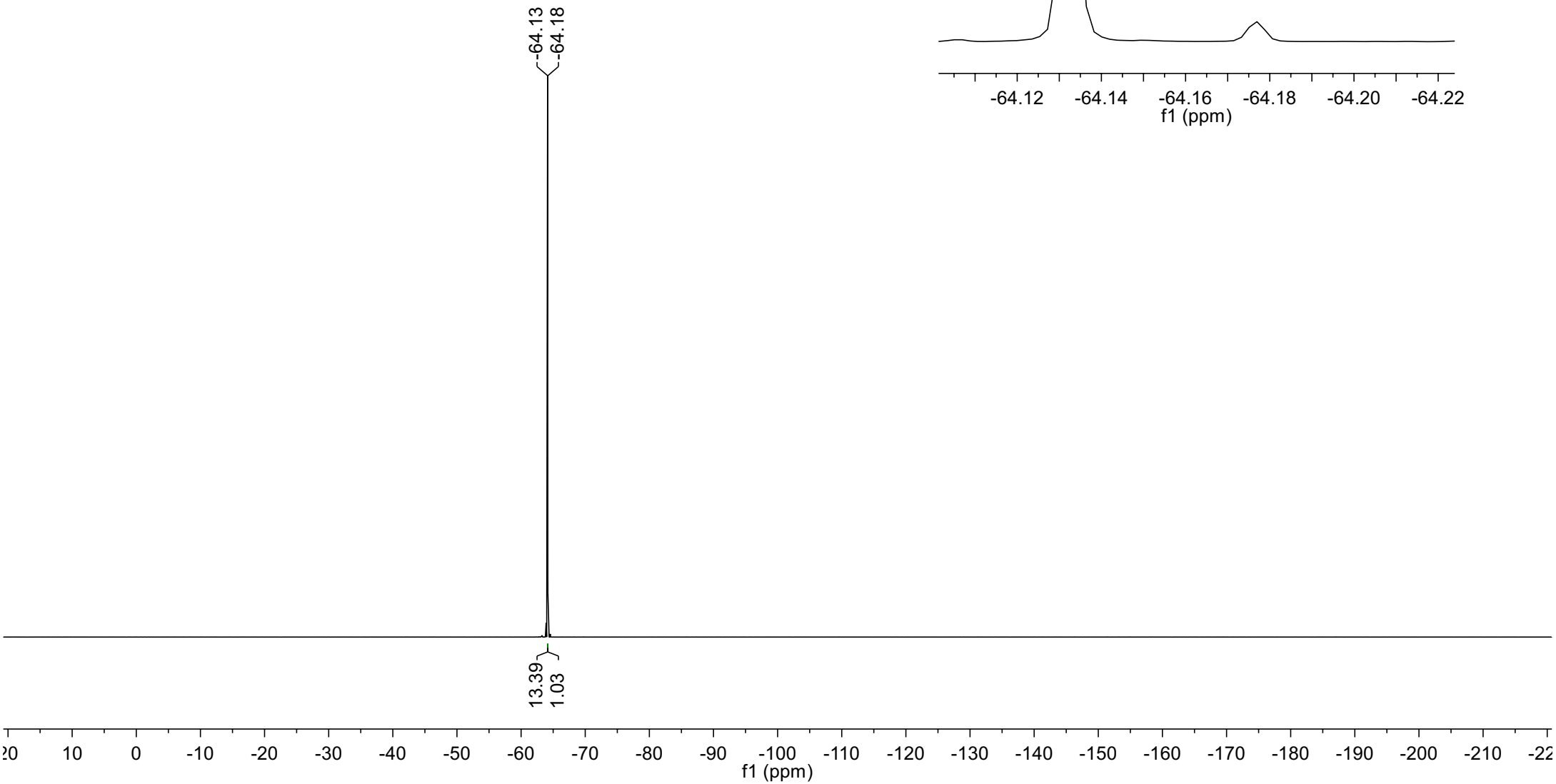


2bd1

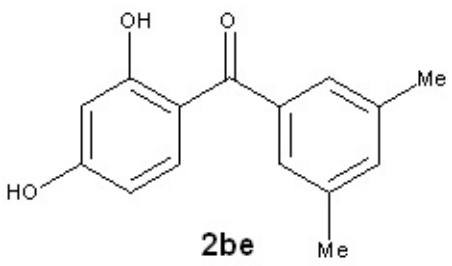


2bd2

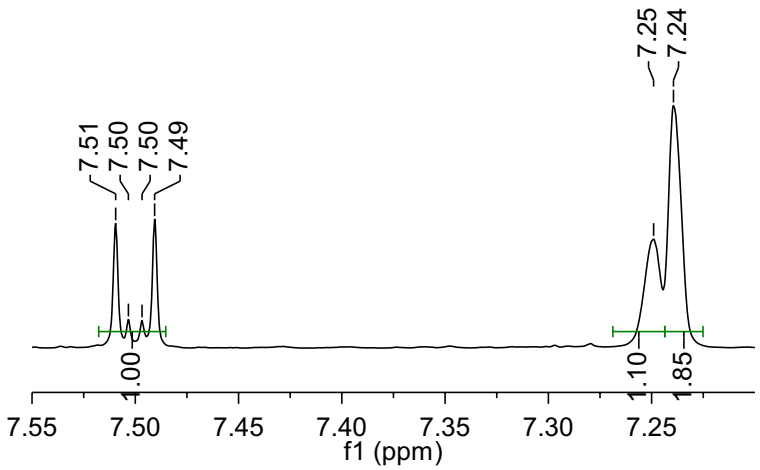


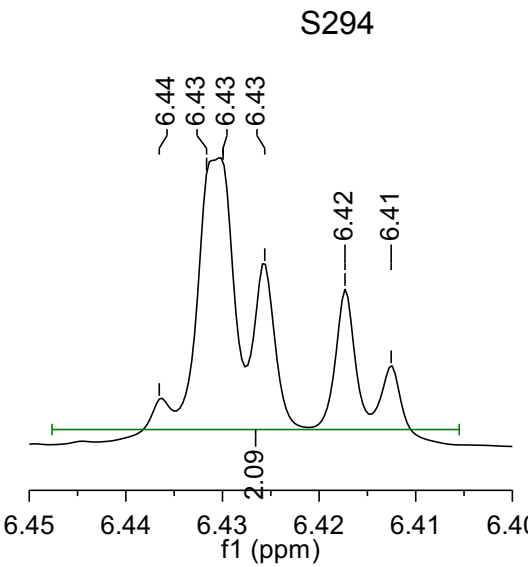


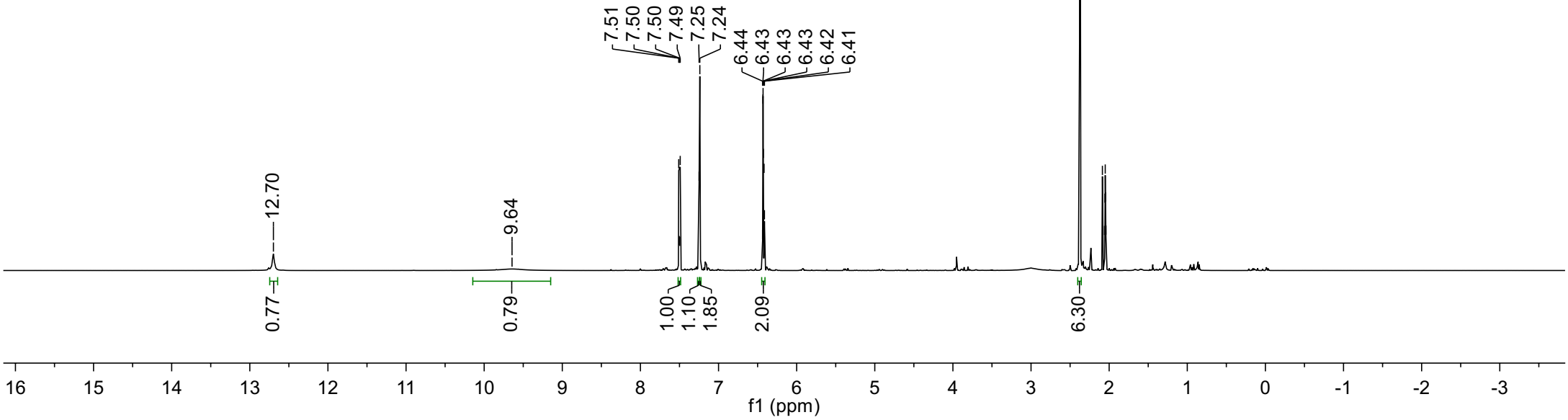
2be proton
500.15
298.0
Acetone-d6



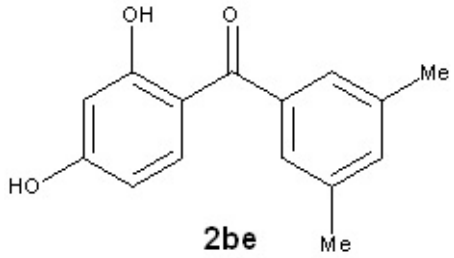
2be







2be carbon
125.78
298.0
Acetone

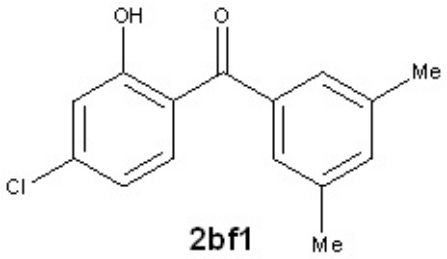


2be

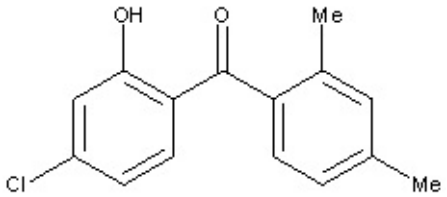
206.20 Acetone
201.11
30.30 Acetone
30.15 Acetone
29.99 Acetone
29.84 Acetone
29.69 Acetone
29.53 Acetone
29.38 Acetone
167.21
165.80
139.41
138.82
136.83
133.63
127.19
113.47
108.68
103.80
21.21

f1 (ppm)

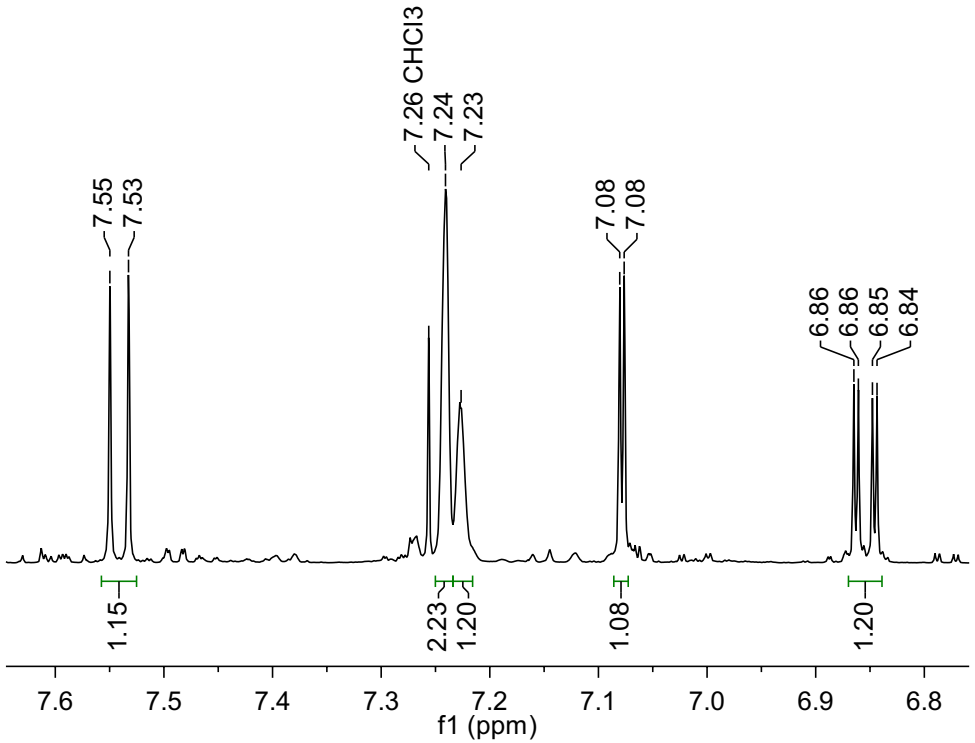
2bf1/2bf2 proton
500.15
298.0
CDCl3

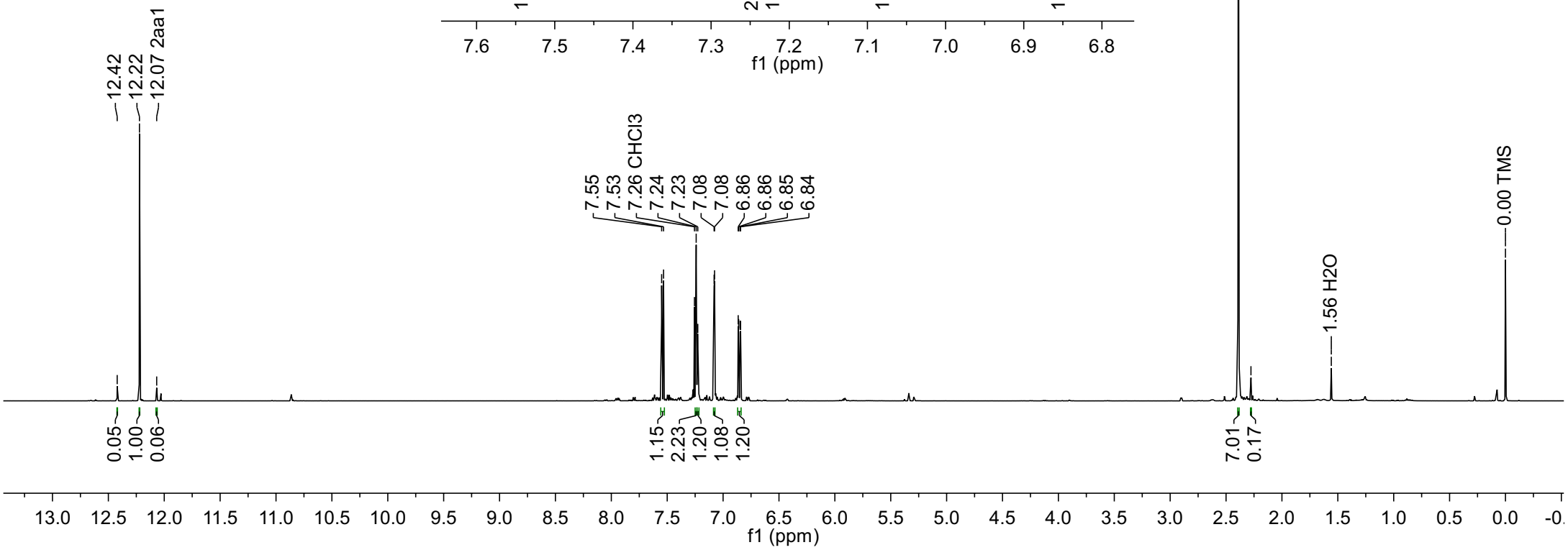


2bf1

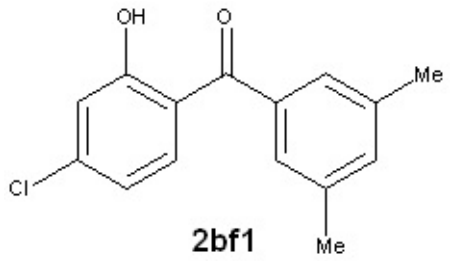


2bf2



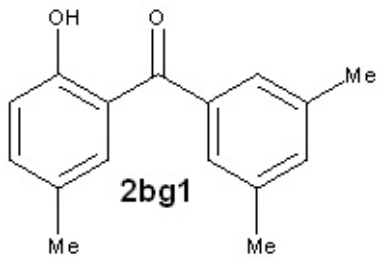


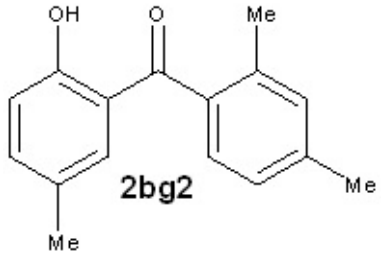
2bf1/2bf2 carbon
125.78
298.0
CDCl3



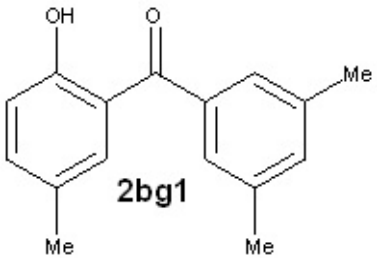
2bf1

2bg1/2bg2 proton
500.15
298.0
CDCl3



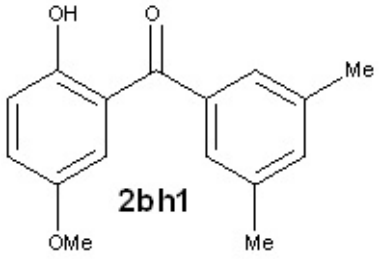


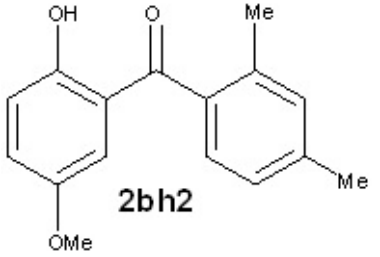
2bg1/2bg2 carbon
125.78
298.0
CDCl3

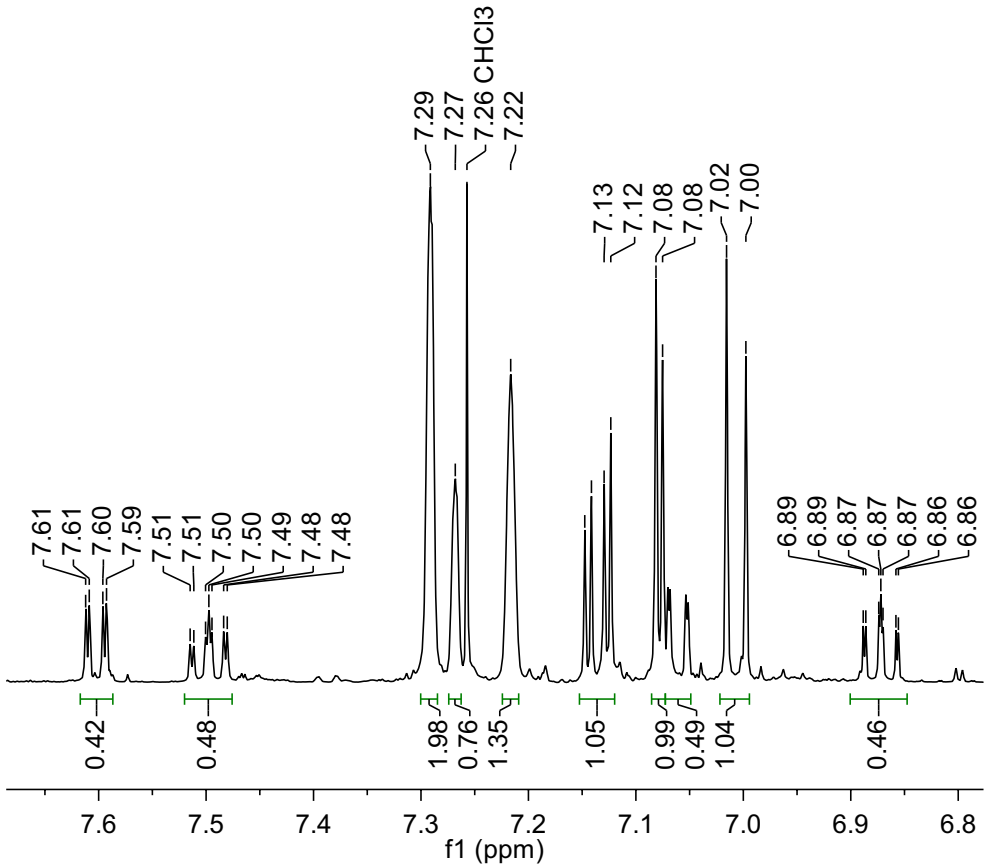


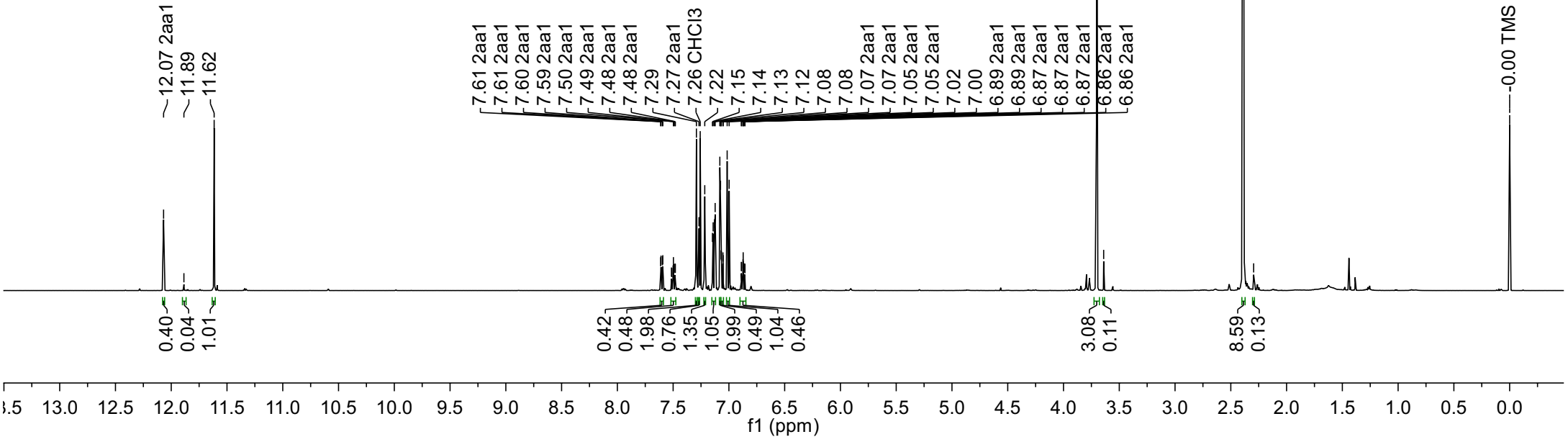
2bh1/2bh2 proton
500.15
298.0
CDCl3

S300

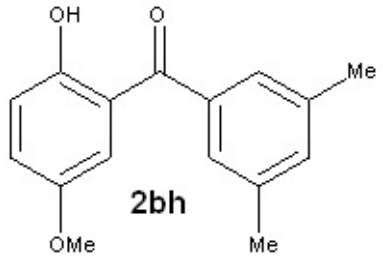




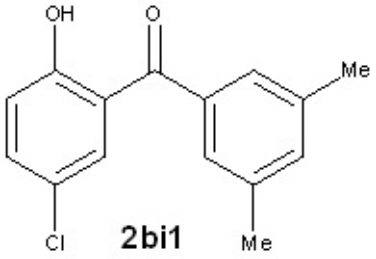




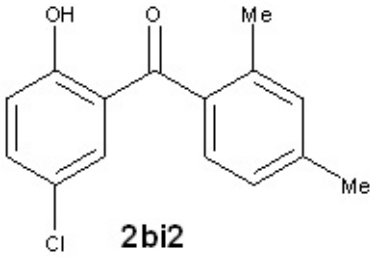
2bh1/2bh2 carbon
125.78
298.0
CDCl3



2bi1/2bi2 proton
500.15
298.0
CDCl3

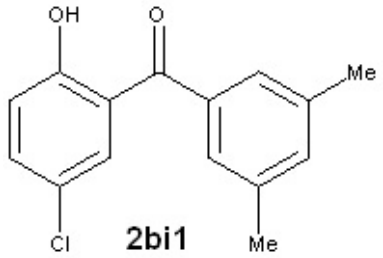


2bi1



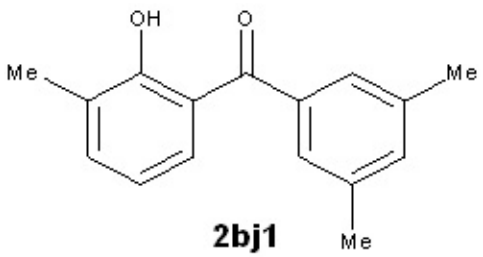
2bi2

2bi1/2bi2 carbon
125.78
298.0
CDCl3

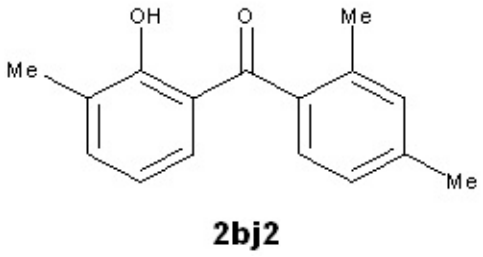


2bi1

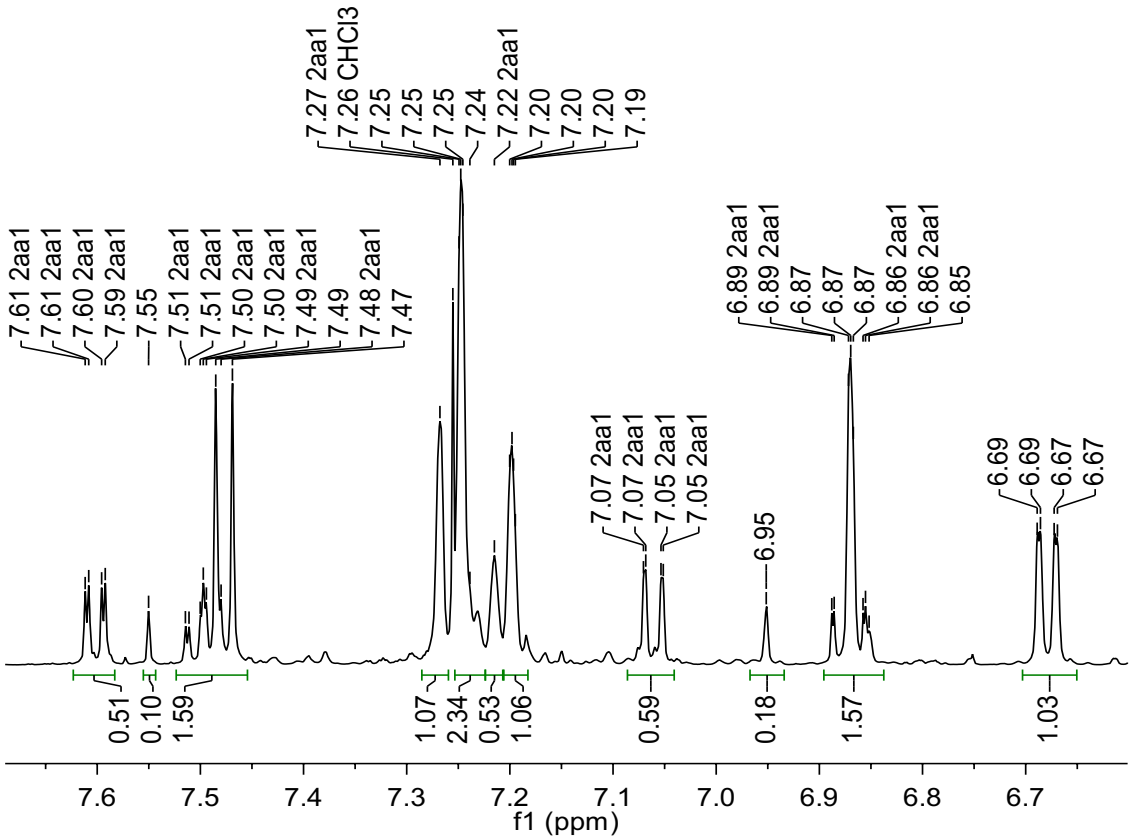
2bj1/2bj2 proton
500.15
298.0
CDCl3

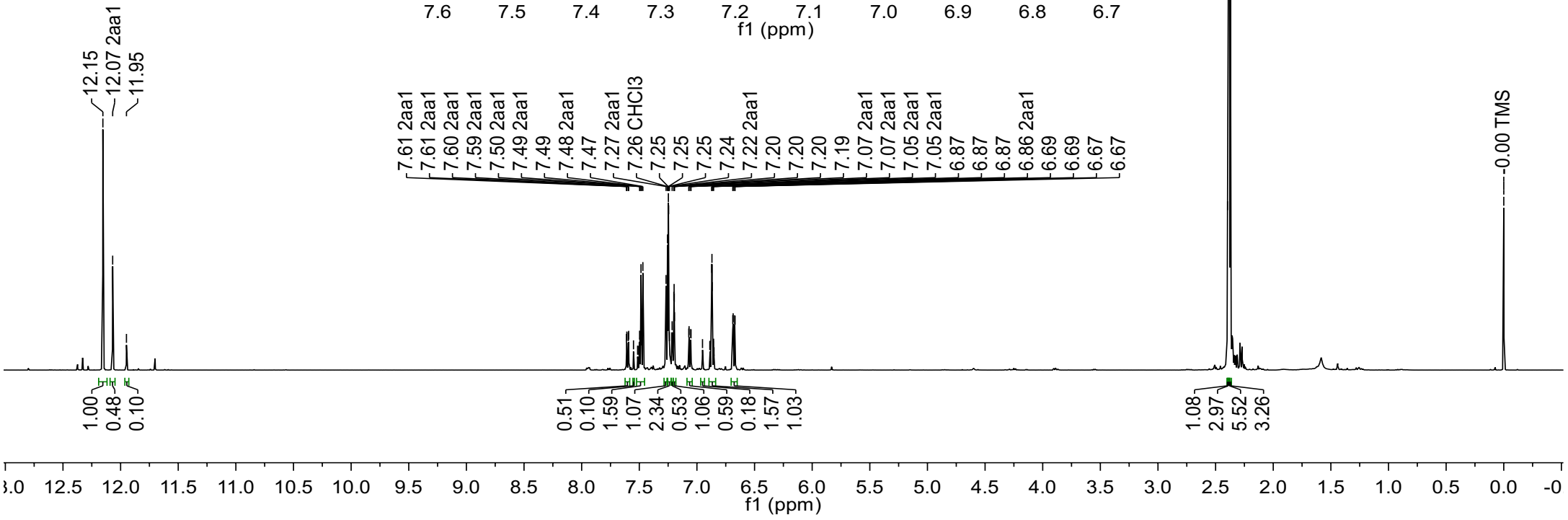


2bj1

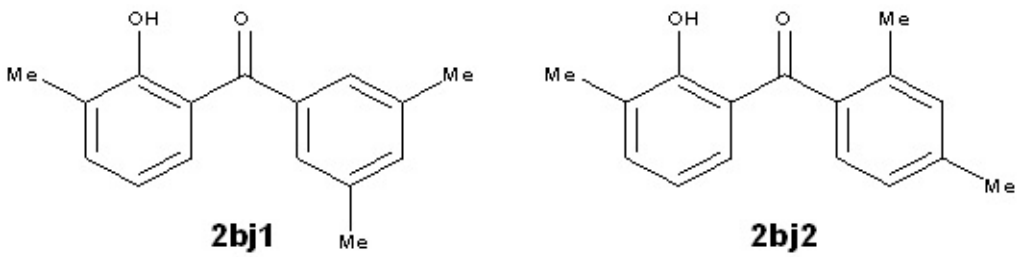


2bj2

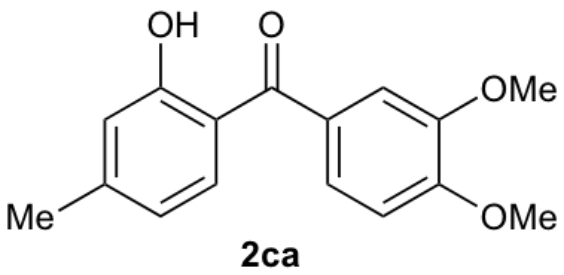




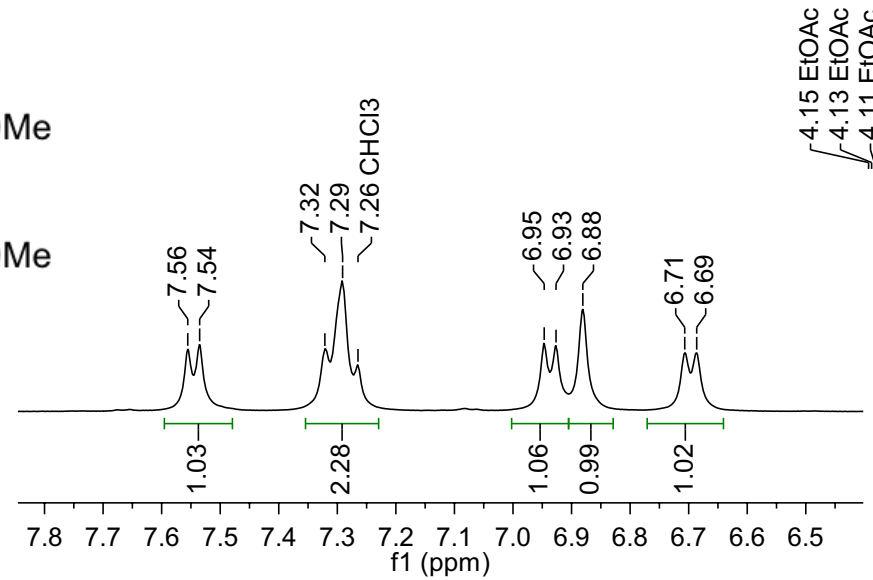
2bj1/2bj2 carbon
125.78
298.0
CDCl3



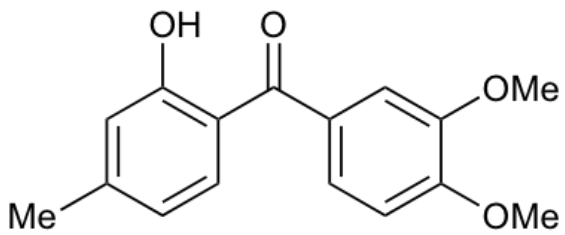
2ca proton
399.87
298.0
CDCl3



2ca

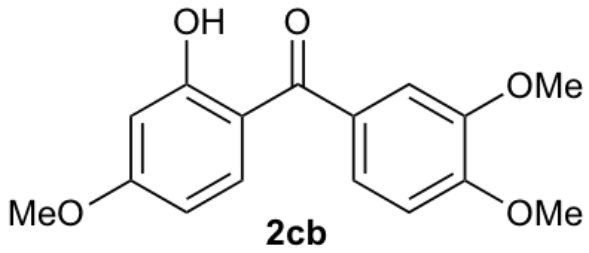


2ca carbon
100.56
298.2
CDCl3

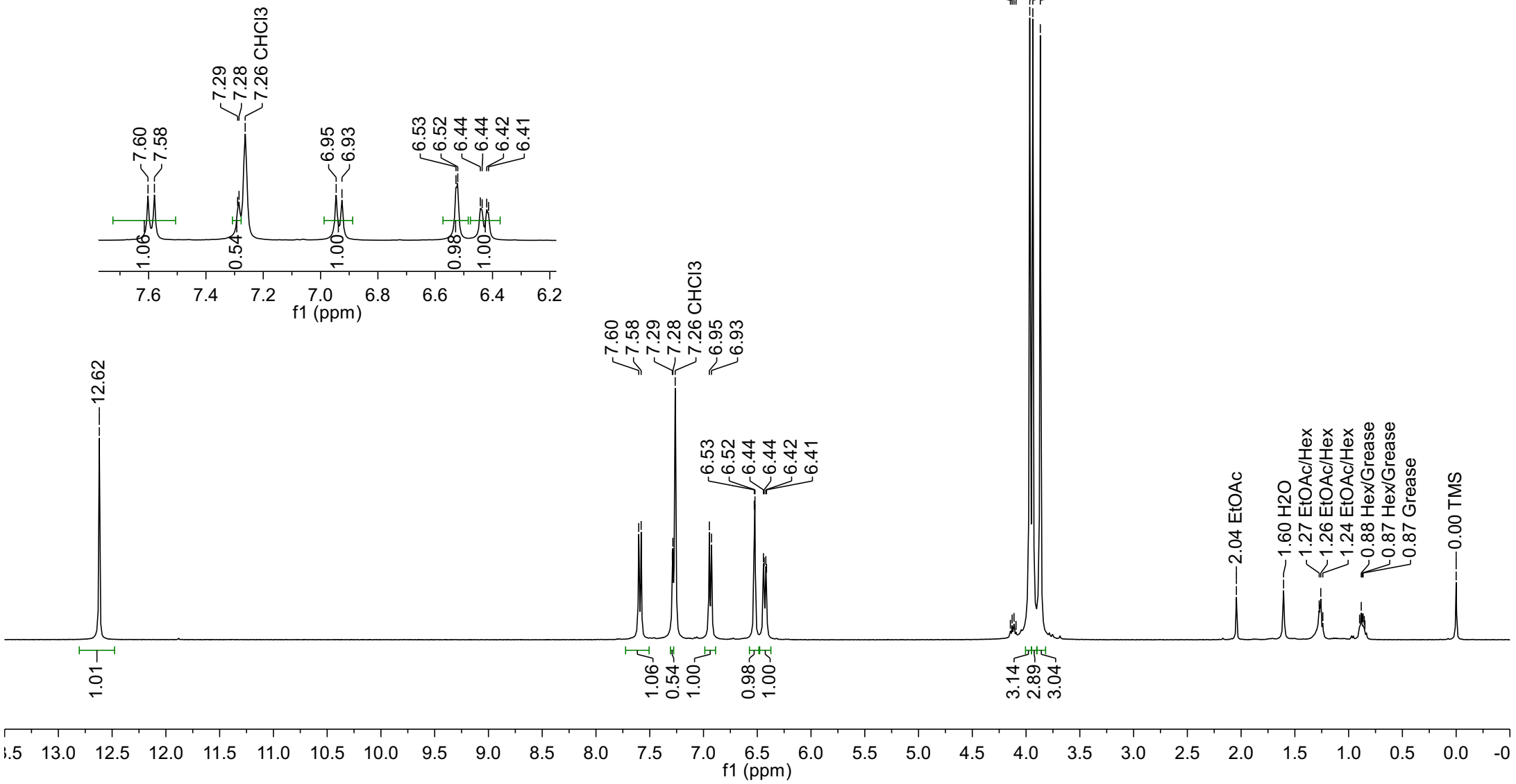


2ca

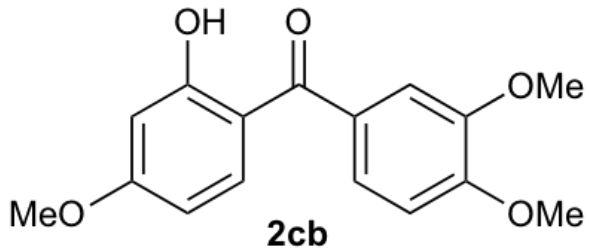
2cb proton
399.87
298.0
CDCl3



2cb

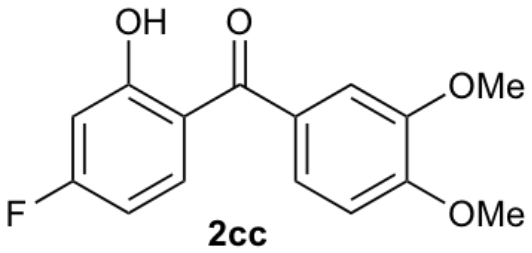


2cb carbon
100.56
298.1
CDCl3

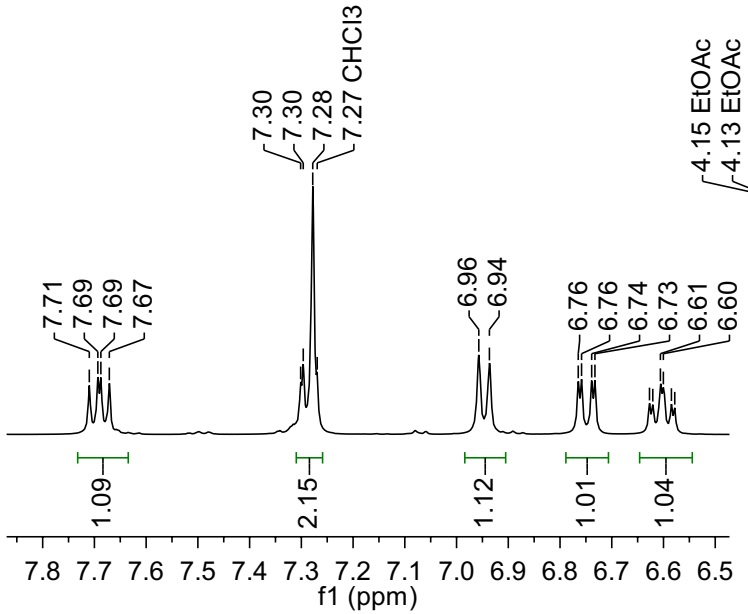


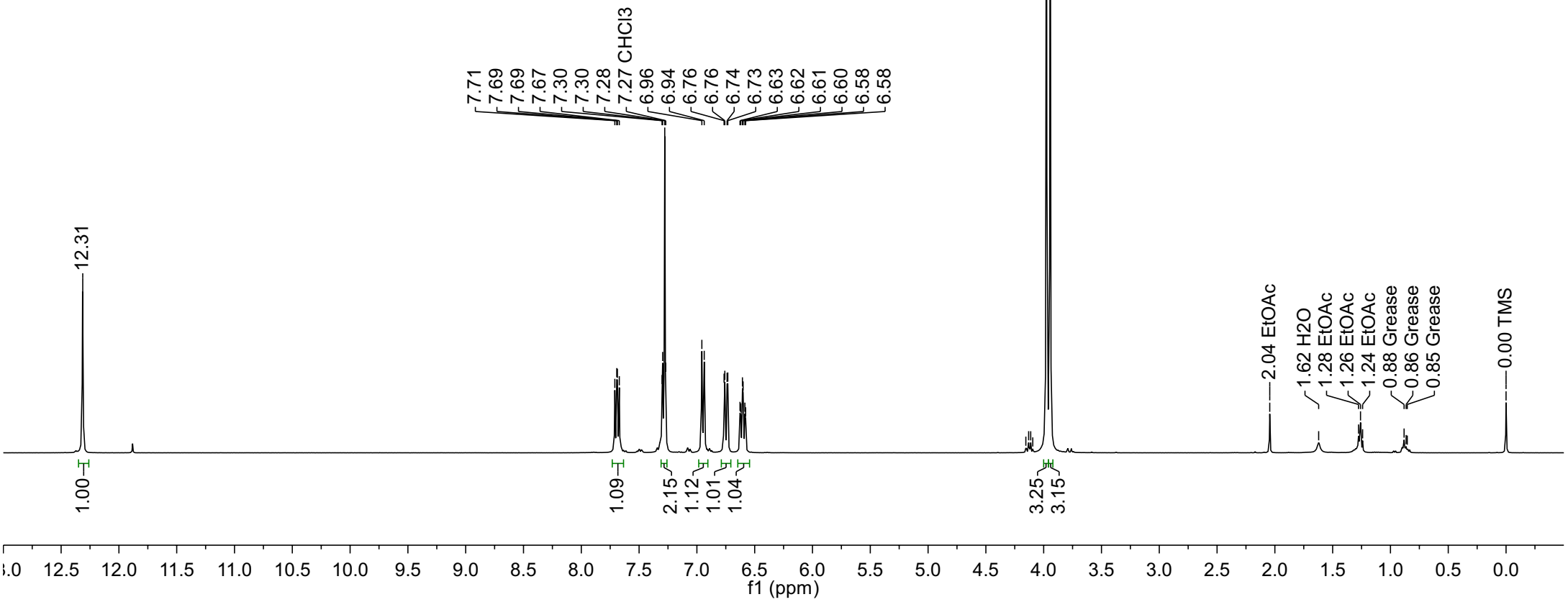
2cb

2cc proton
399.87
298.0
CDCl3



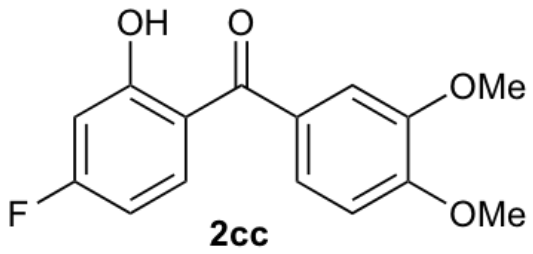
2cc





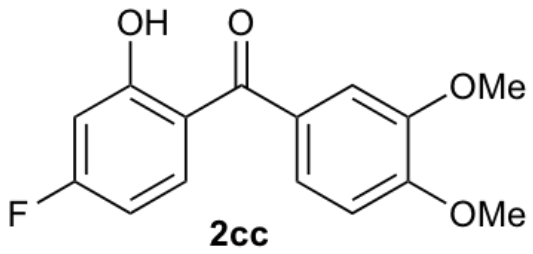
2cc carbon
100.56
298.2
CDCl3

S311



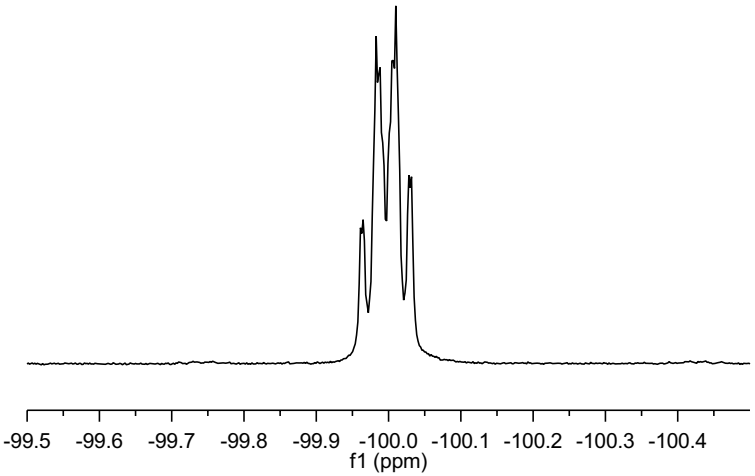
2cc

2cc no decoupling 19F
376.46
298.0
CDCl3



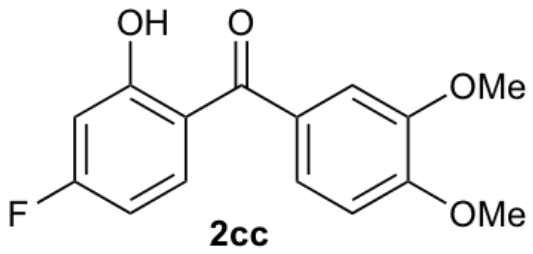
2cc

-99.96
-99.98
-99.99
-100.00
-100.01
-100.03



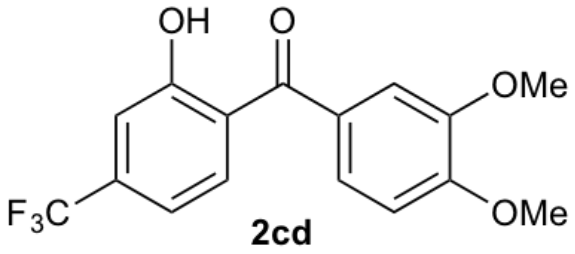
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

2cc with {1H} decoupling 19F
376.46
298.0
CDCl3

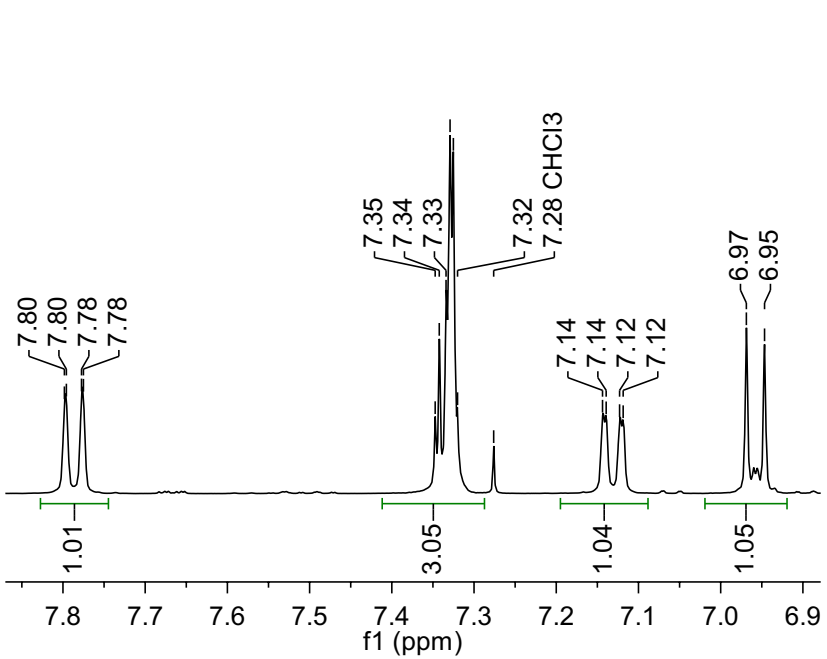


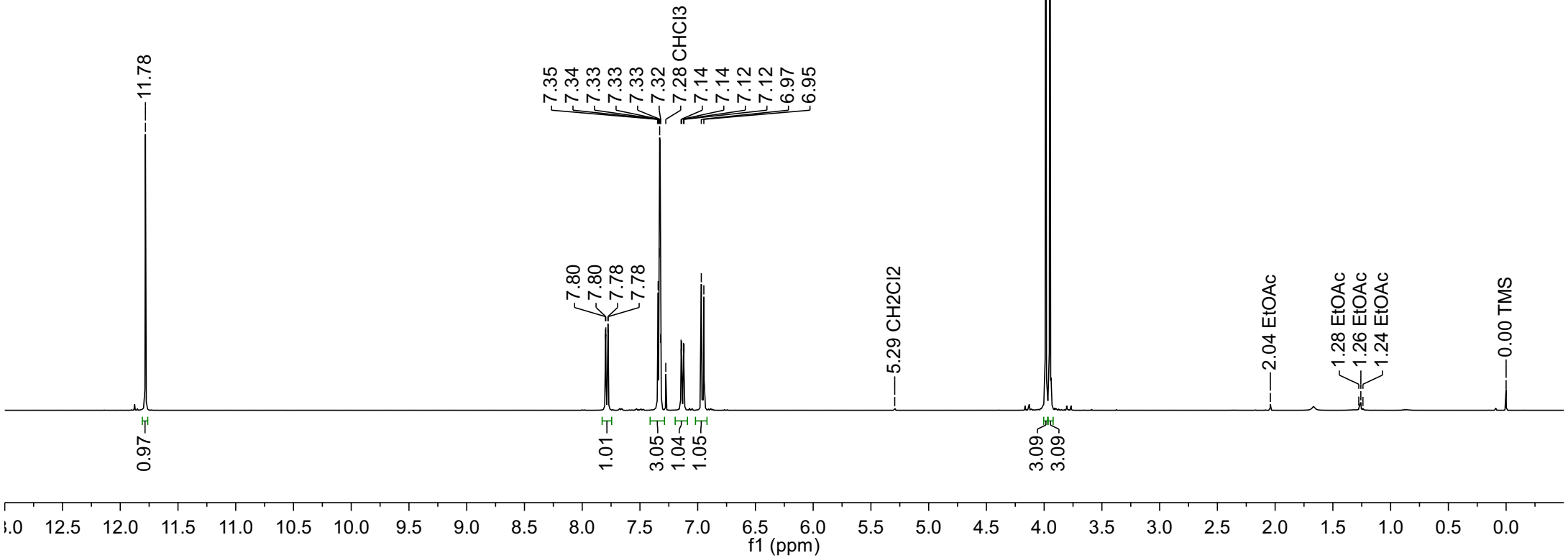
2cc

2cd proton
399.87
300.0
CDCl3

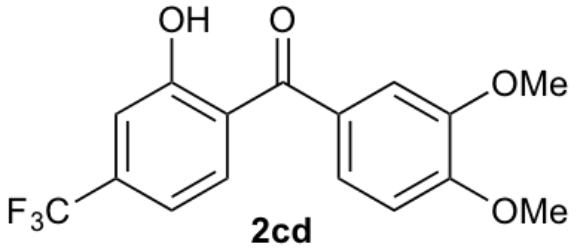


2cd



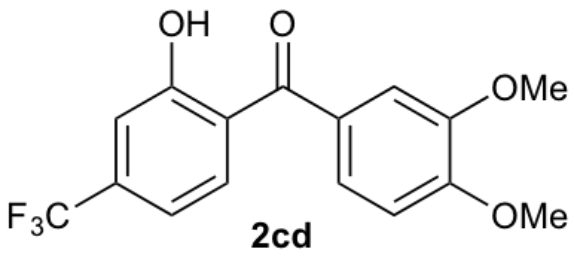


2cd carbon
100.56
300.0
CDCl3



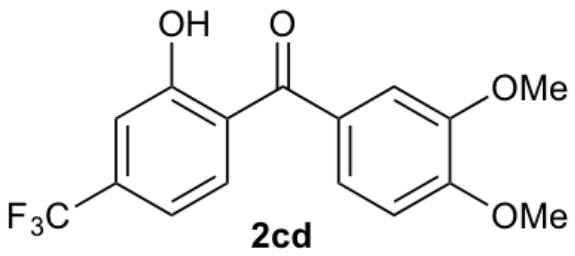
2cd

2cd no decoupling 19F
376.46
298.0
CDCl3



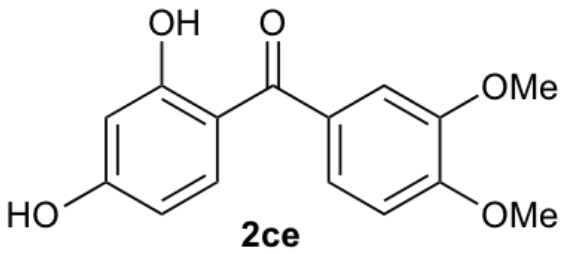
2cd

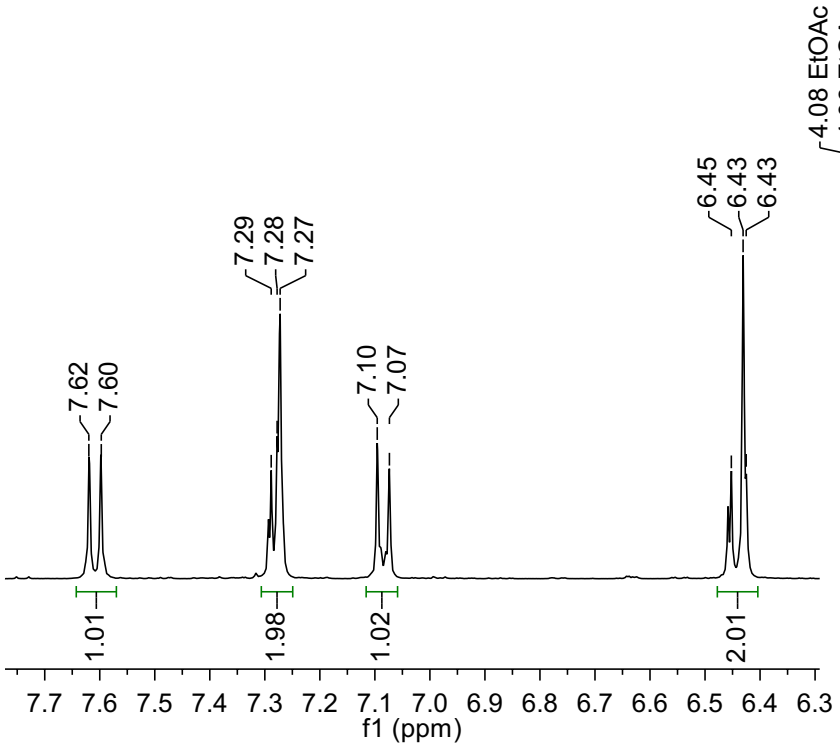
2cd with {1H} decoupling 19F
376.46
298.1
CDCl3

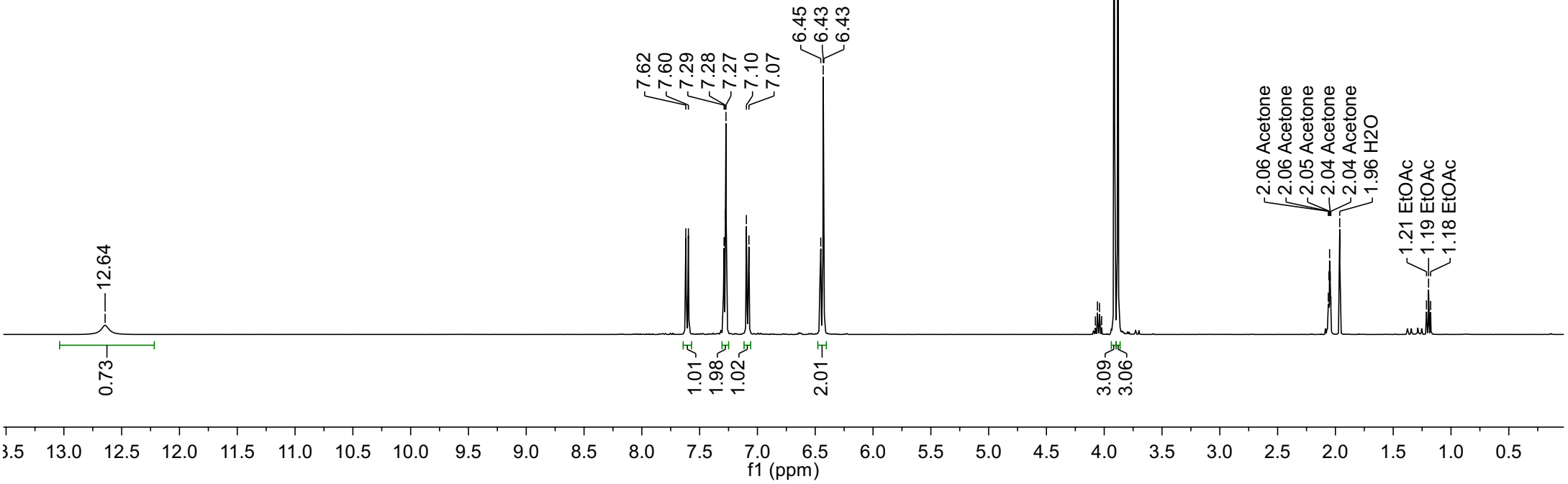


2cd

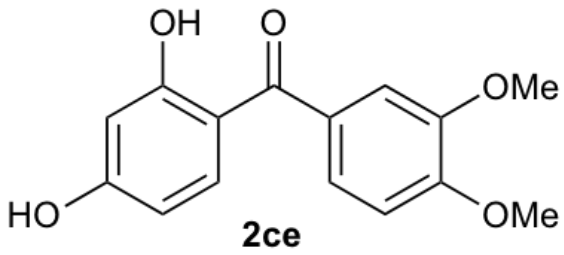
2ce proton
399.87
298.0
Acetone







2ce carbon
100.56
298.1
Acetone

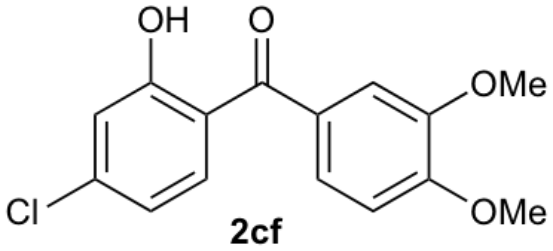


2ce

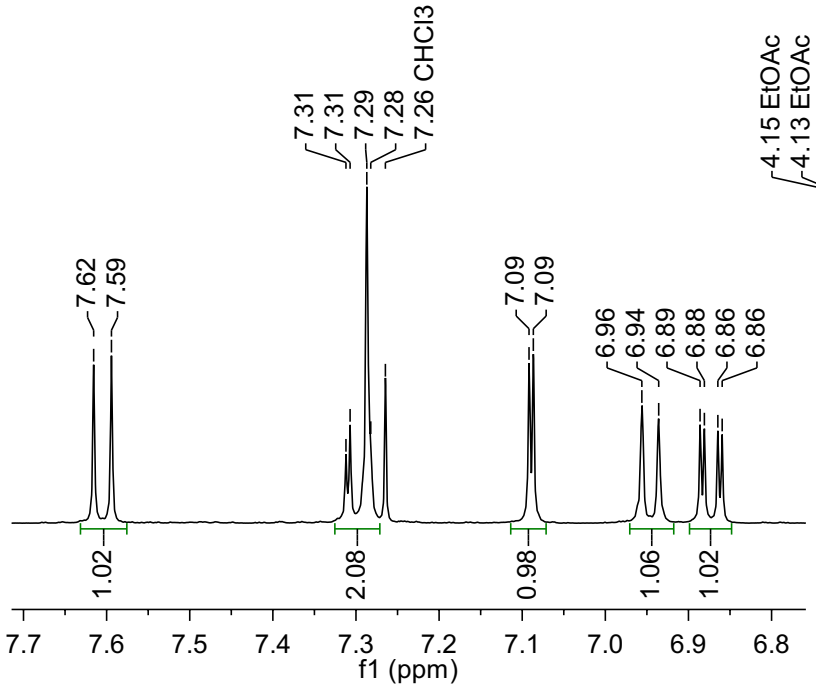
206.3 Acetone
199.4
167.0
165.5
153.5
150.1
136.6
131.6
124.1
113.5
113.2
111.5
108.5
103.9
56.3
56.2
30.5 Acetone
30.3 Acetone
30.1 Acetone
29.9 Acetone
29.7 Acetone
29.5 Acetone
29.3 Acetone

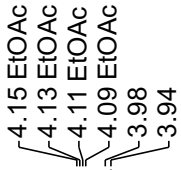
f1 (ppm)

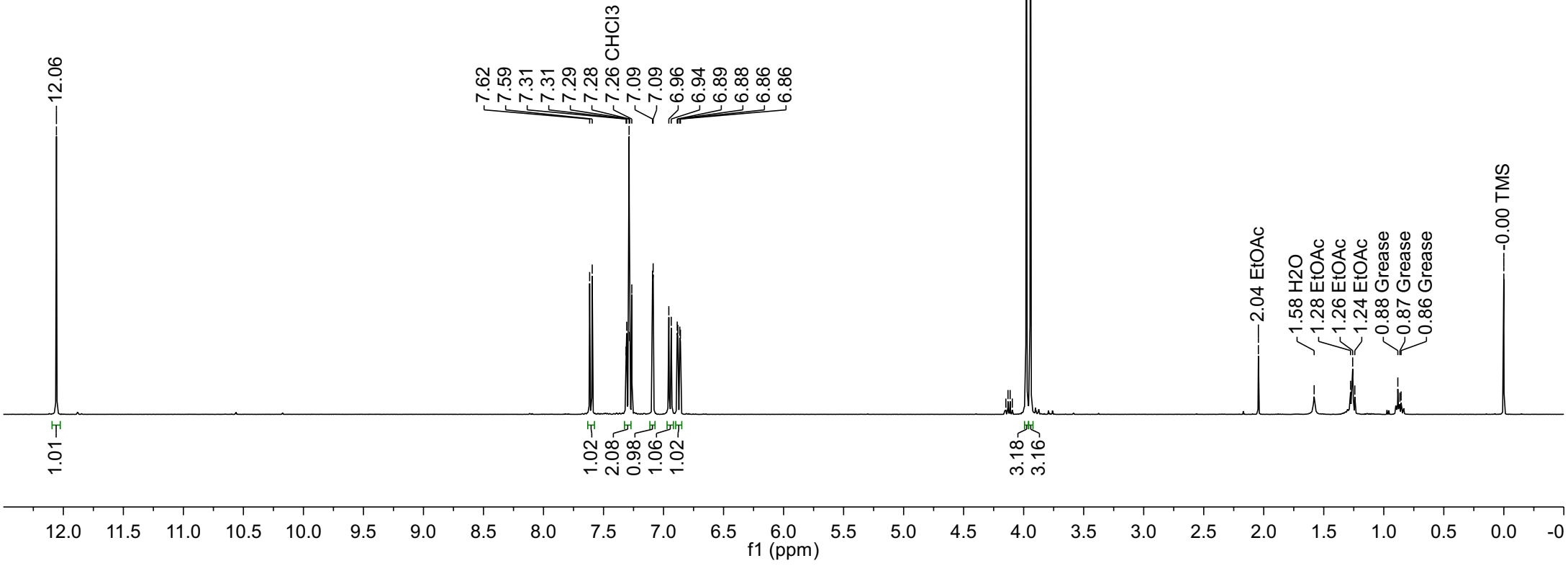
2cf proton
399.87
298.0
CDCl3



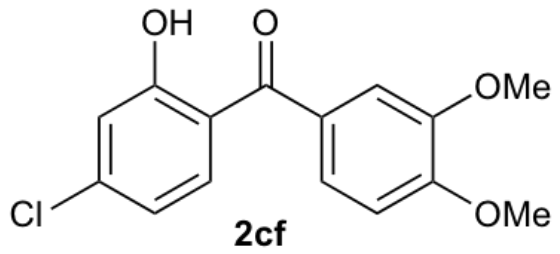
2cf





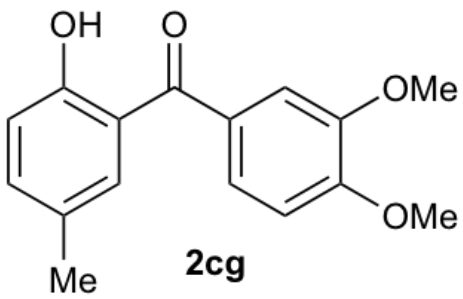


2cf carbon
100.56
298.1
CDCl3



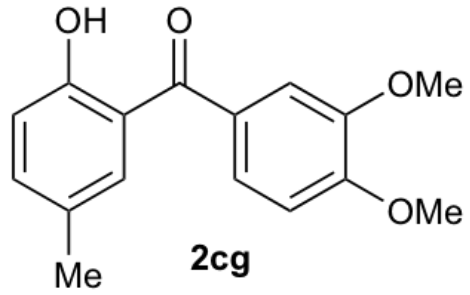
2cf

2cg proton
399.87
298.0
CDCl3



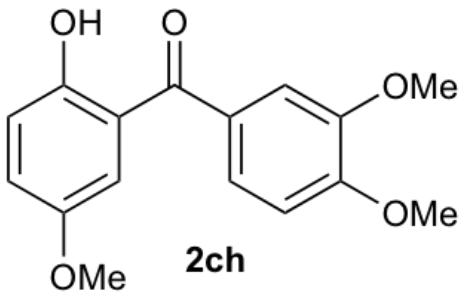
2cg

2cg carbon
100.56
298.1
CDCl3

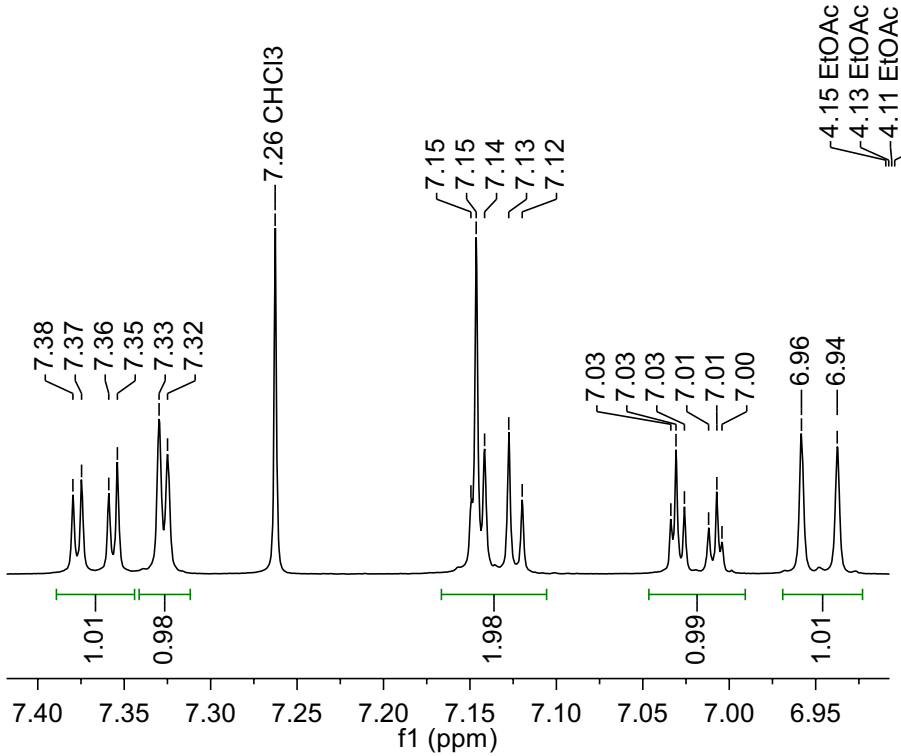


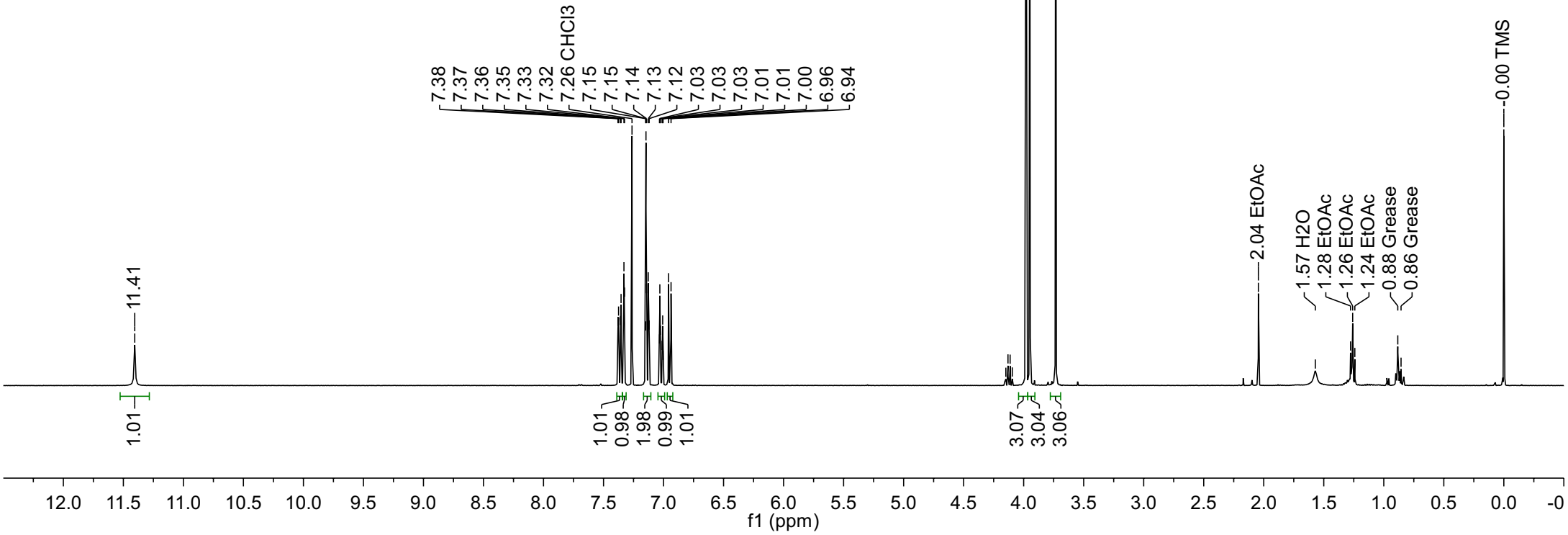
2cg

2ch proton
399.87
298.0
CDCl3

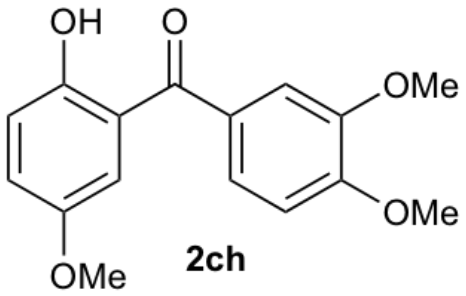


2ch





2ch carbon
100.56
298.1
CDCl3



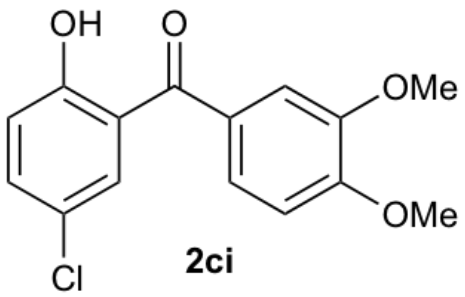
2ch

199.4
157.1
152.7
151.4
149.0
130.4
124.2
123.5
119.2
119.0
116.1
112.1
110.0
77.5 CDCl3
77.2 CDCl3
76.8 CDCl3
56.1
56.1
56.0

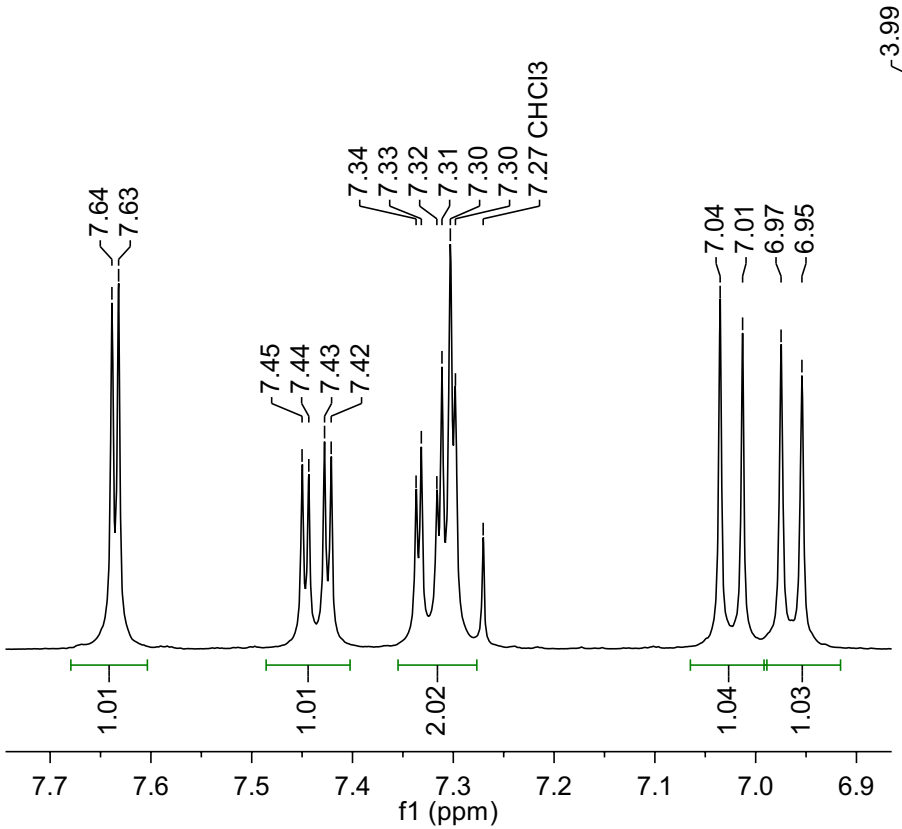
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 2

f1 (ppm)

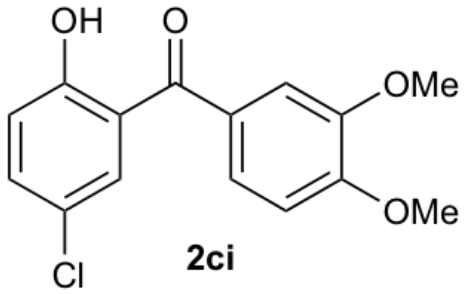
2ci proton
399.87
300.0
CDCl3



2ci

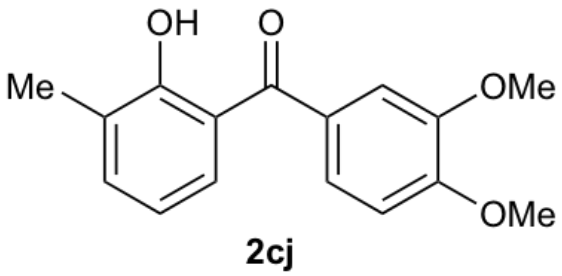


2ci carbon
100.56
300.0
CDCl3

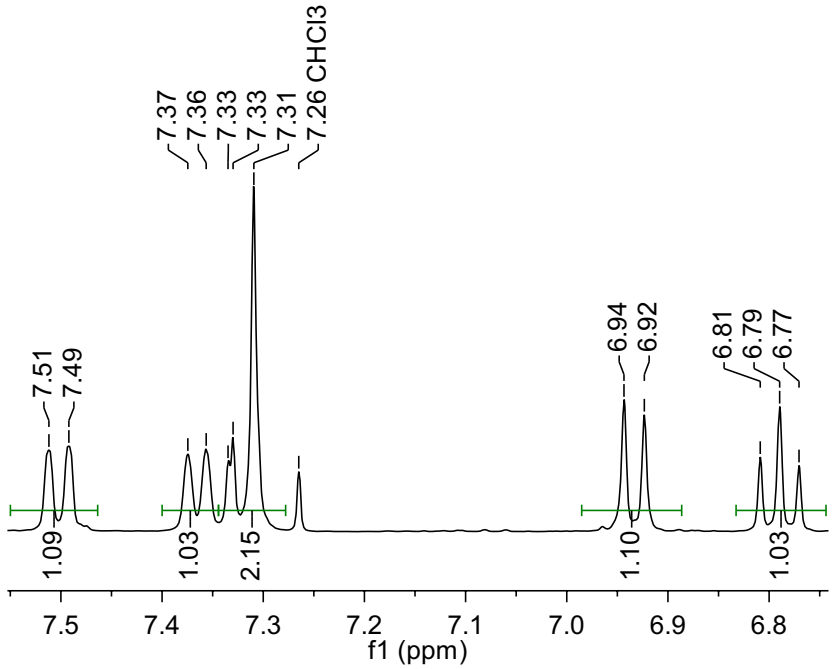


2ci

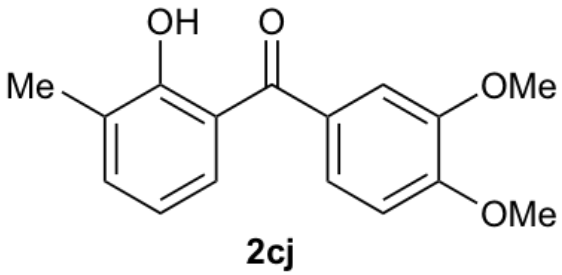
2cj proton
399.87
298.0
CDCl3



2cj

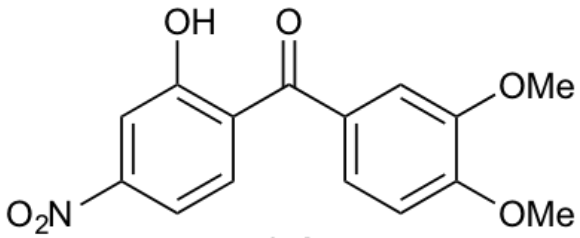


2cj carbon
100.56
298.1
CDCl3

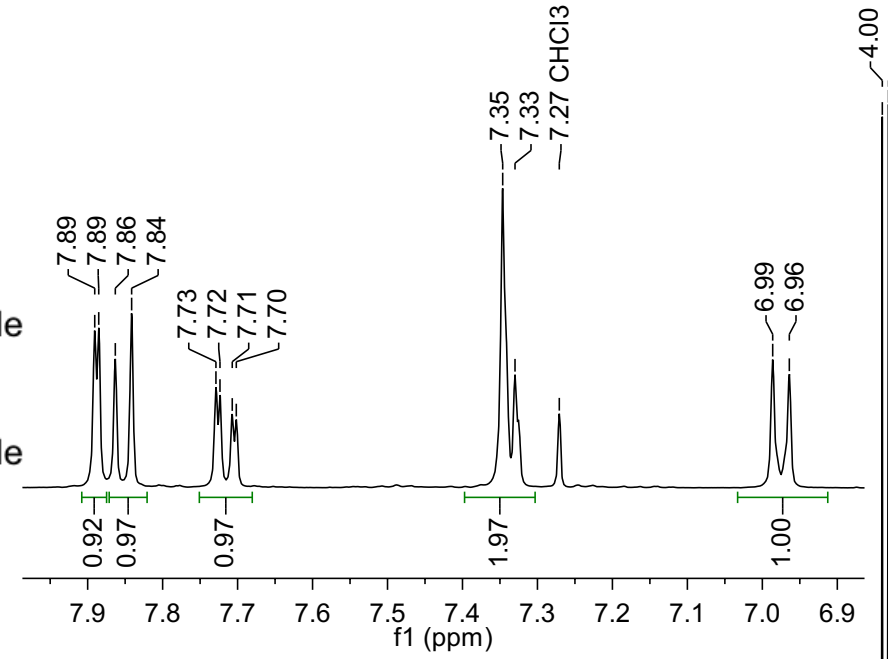


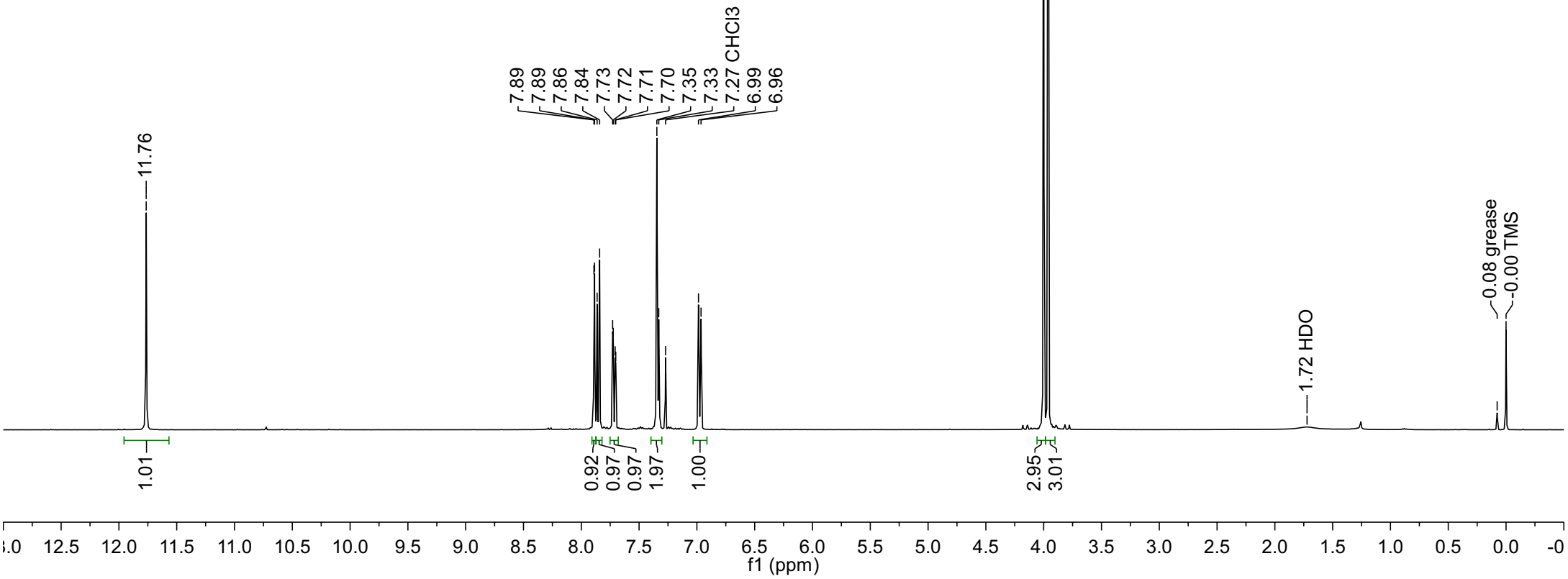
2cj

2ck proton
399.87
298.0
CDCl3

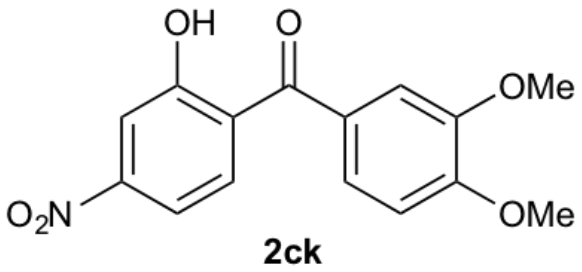


2ck



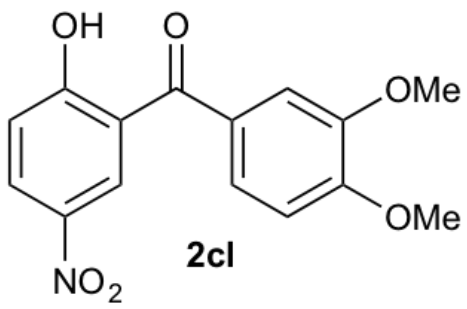


2ck carbon
100.56
298.1
CDCl3

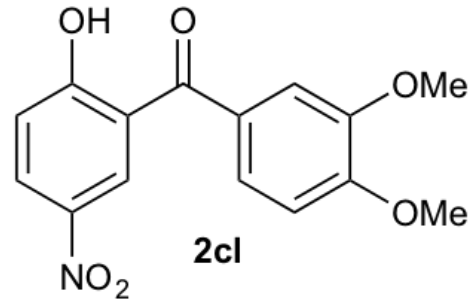


2ck

2cl proton
399.87
298.0
CDCl3

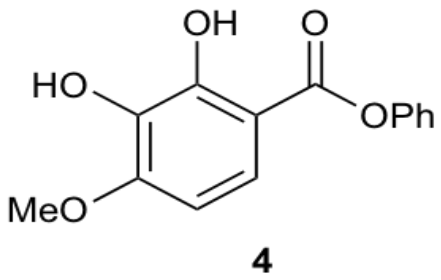


2cl carbon
100.56
298.1
CDCl3

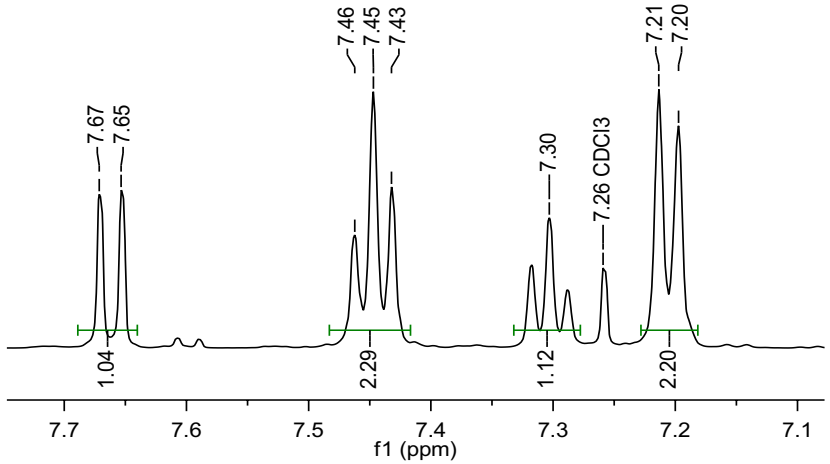


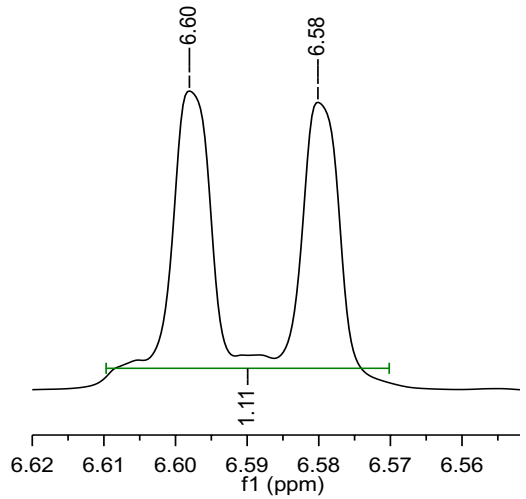
2cl

4 Proton
500.15
298.0
CDCl3

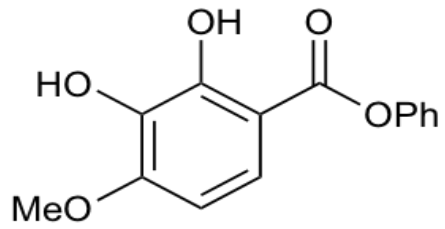


4



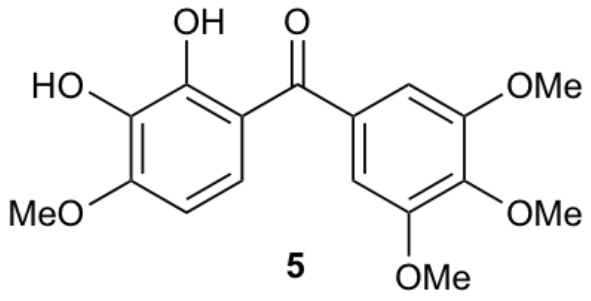


4 Carbon
125.78
298.0
CDCl3

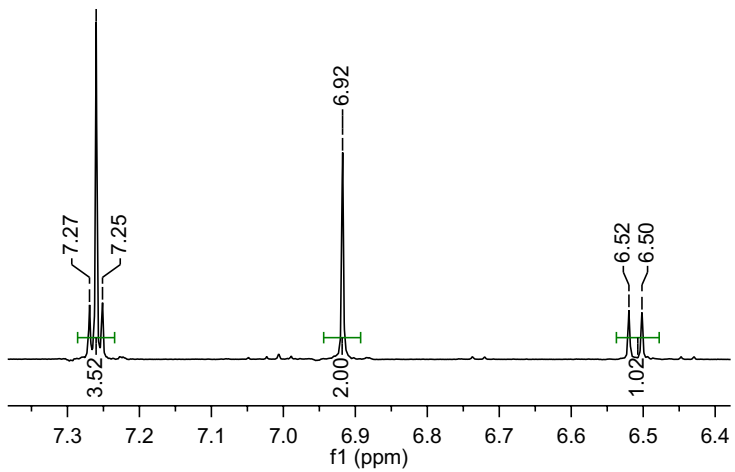


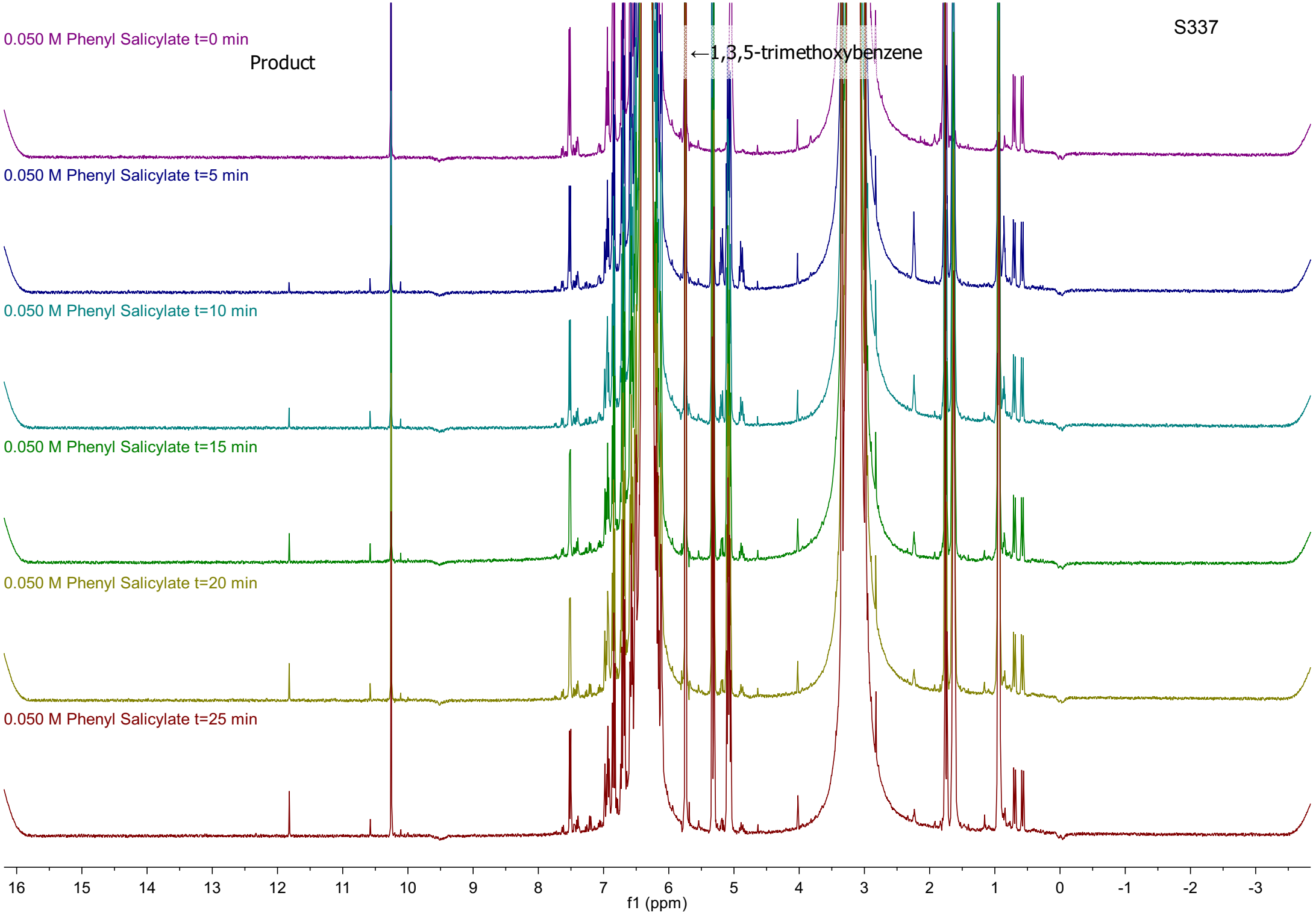
4

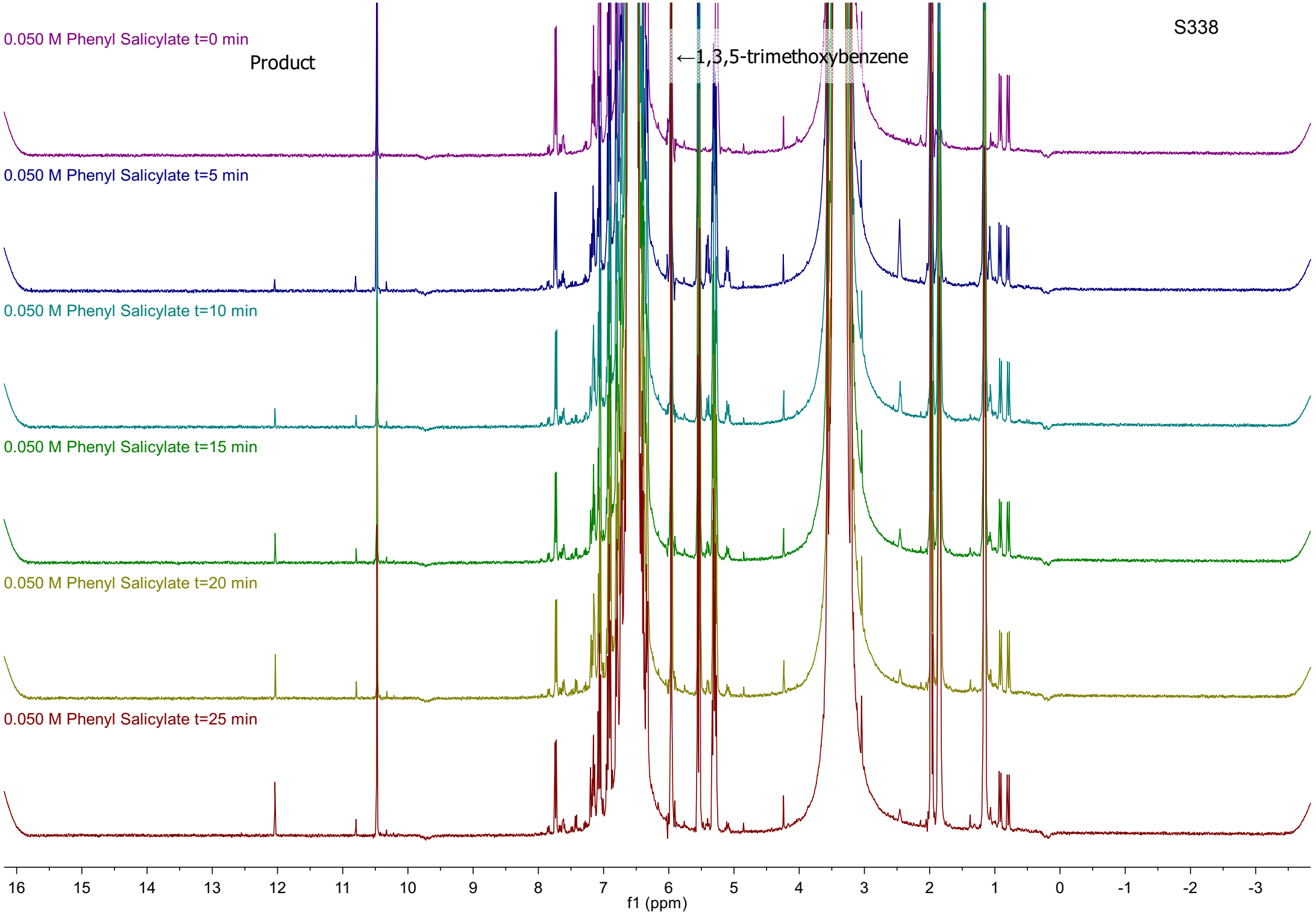
Purified Hydroxyphenstatin
500.33
300.1
CDCl3

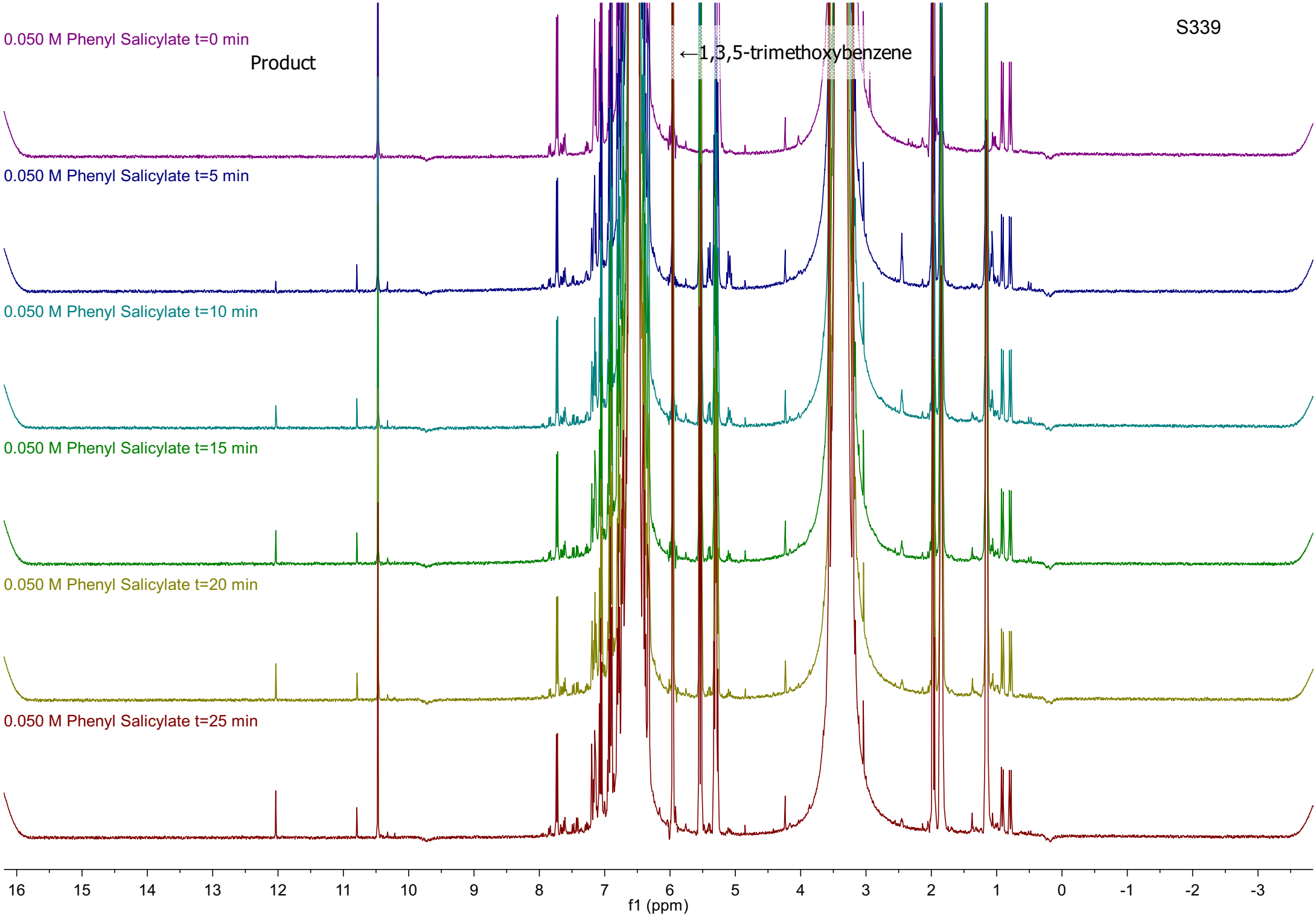


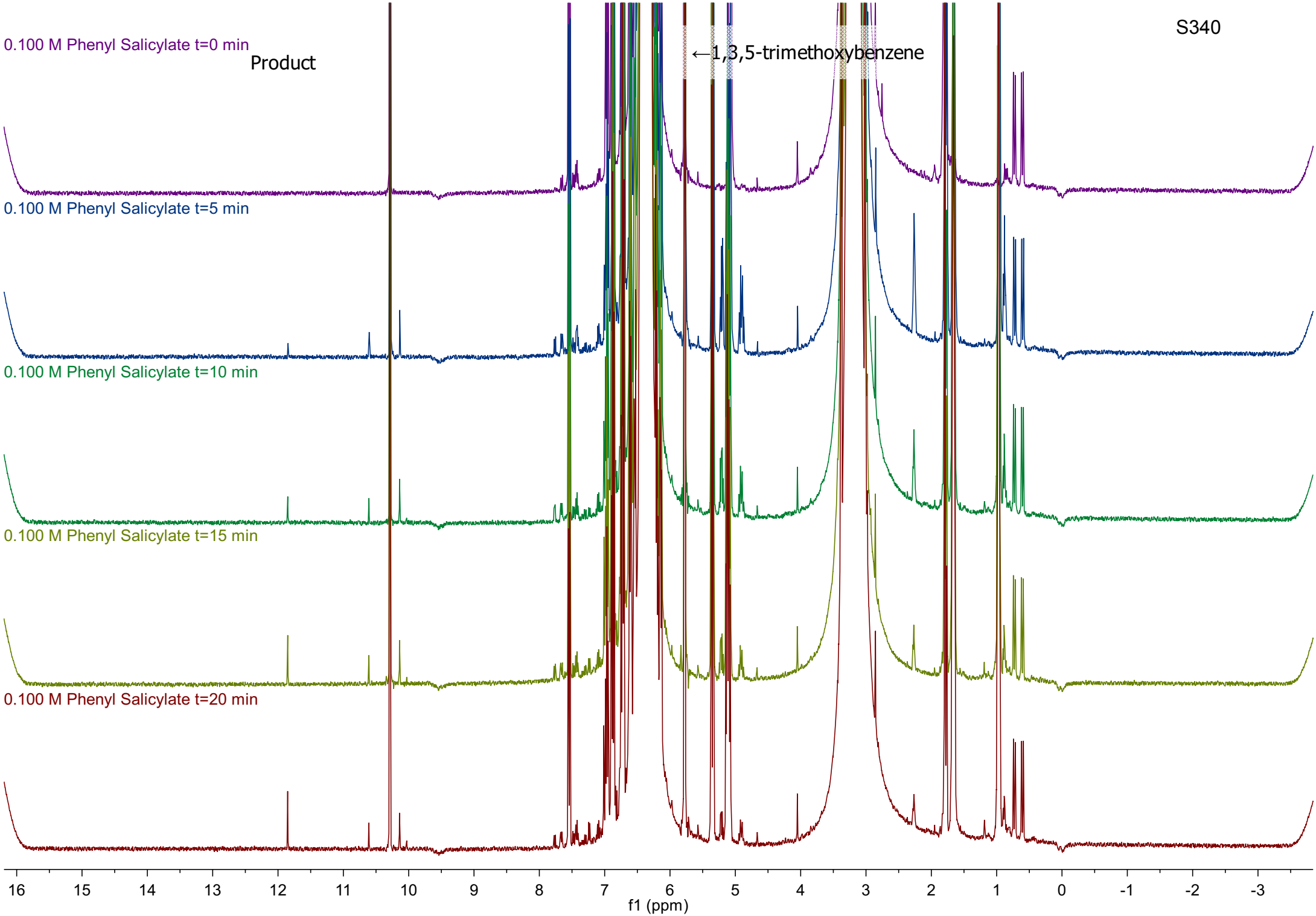
5

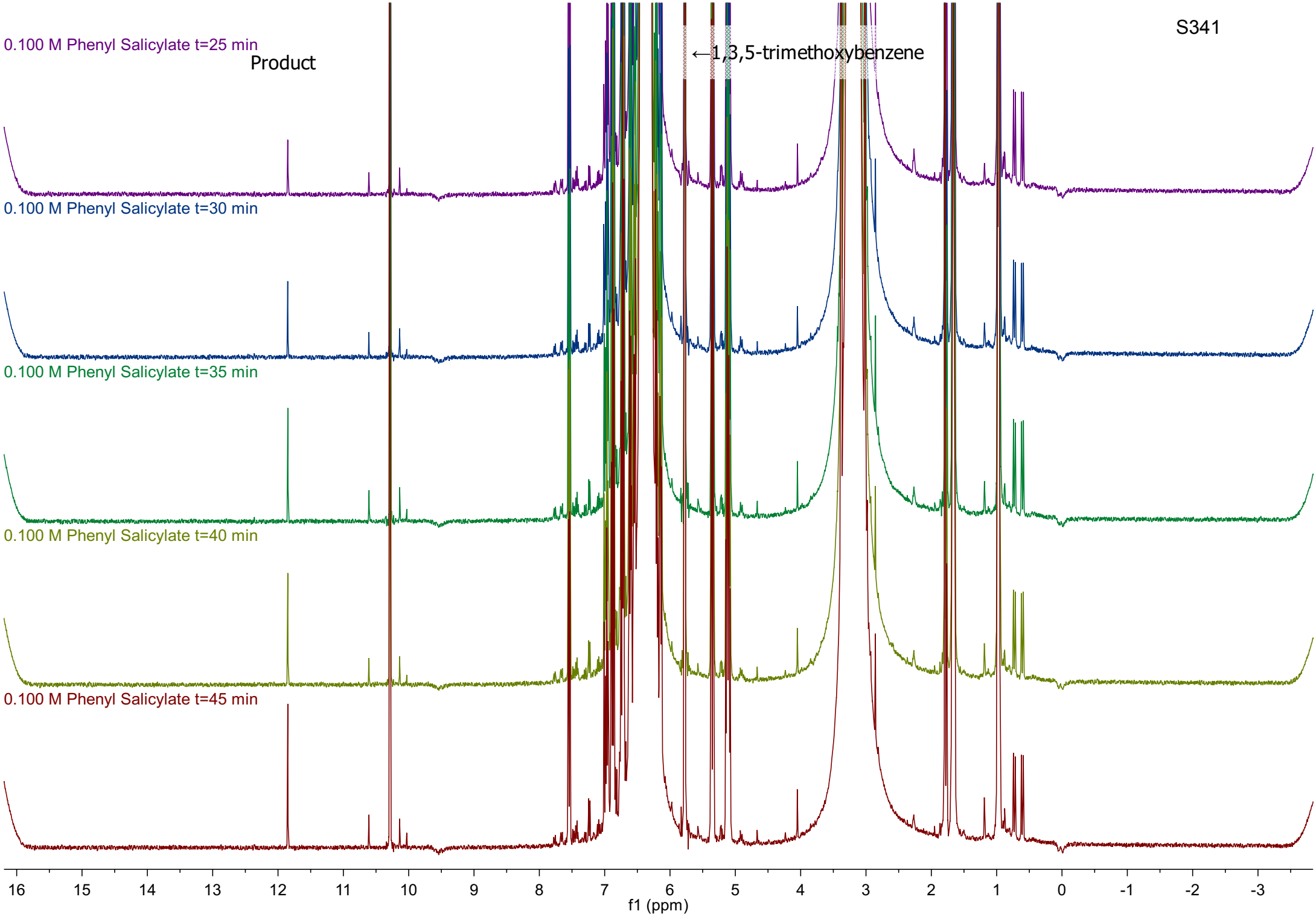


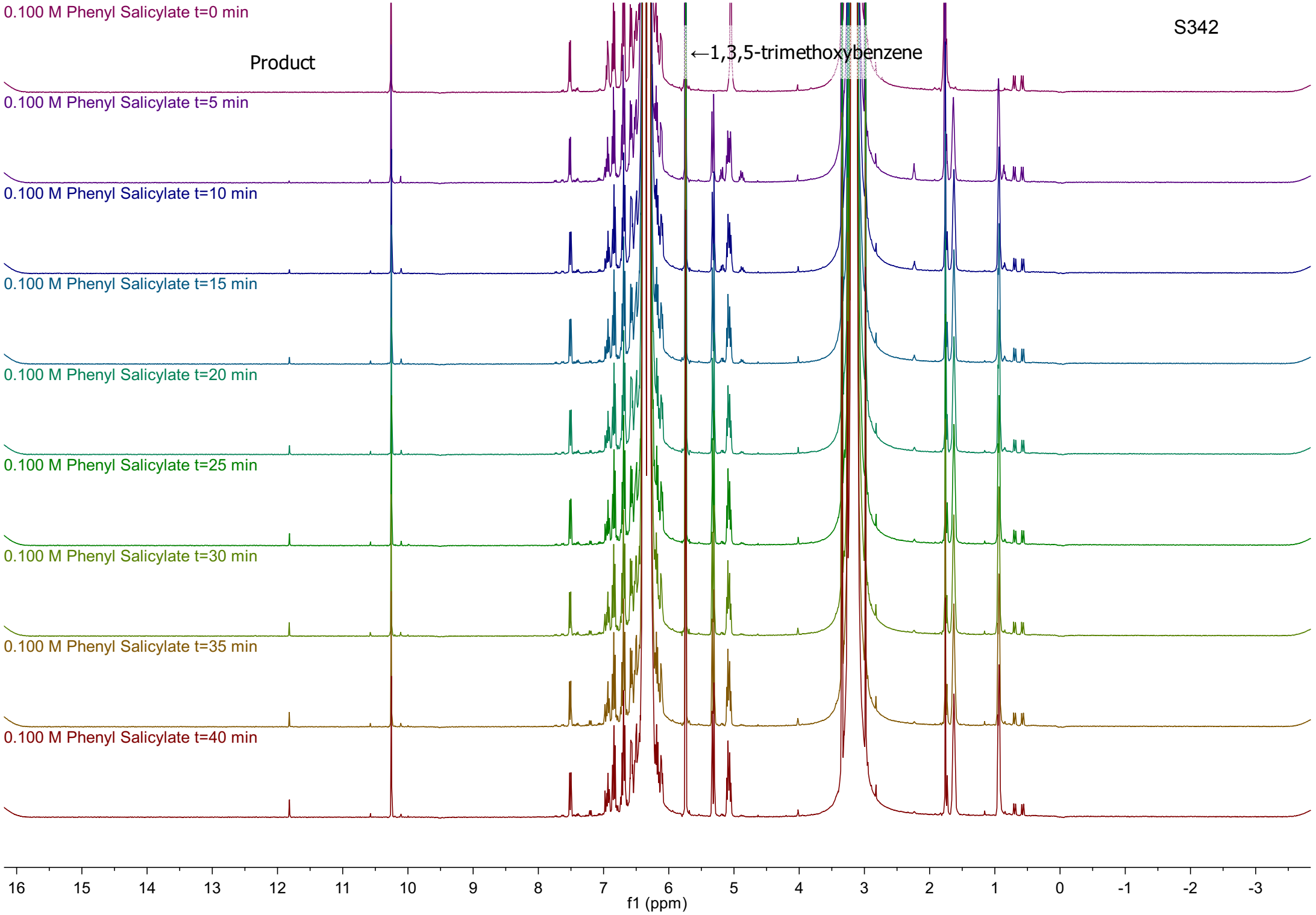

S337
0.050 M Phenyl Salicylate t=0 min
Product
←1,3,5-trimethoxybenzene
0.050 M Phenyl Salicylate t=5 min
0.050 M Phenyl Salicylate t=10 min
0.050 M Phenyl Salicylate t=15 min
0.050 M Phenyl Salicylate t=20 min
0.050 M Phenyl Salicylate t=25 min
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
f1 (ppm)

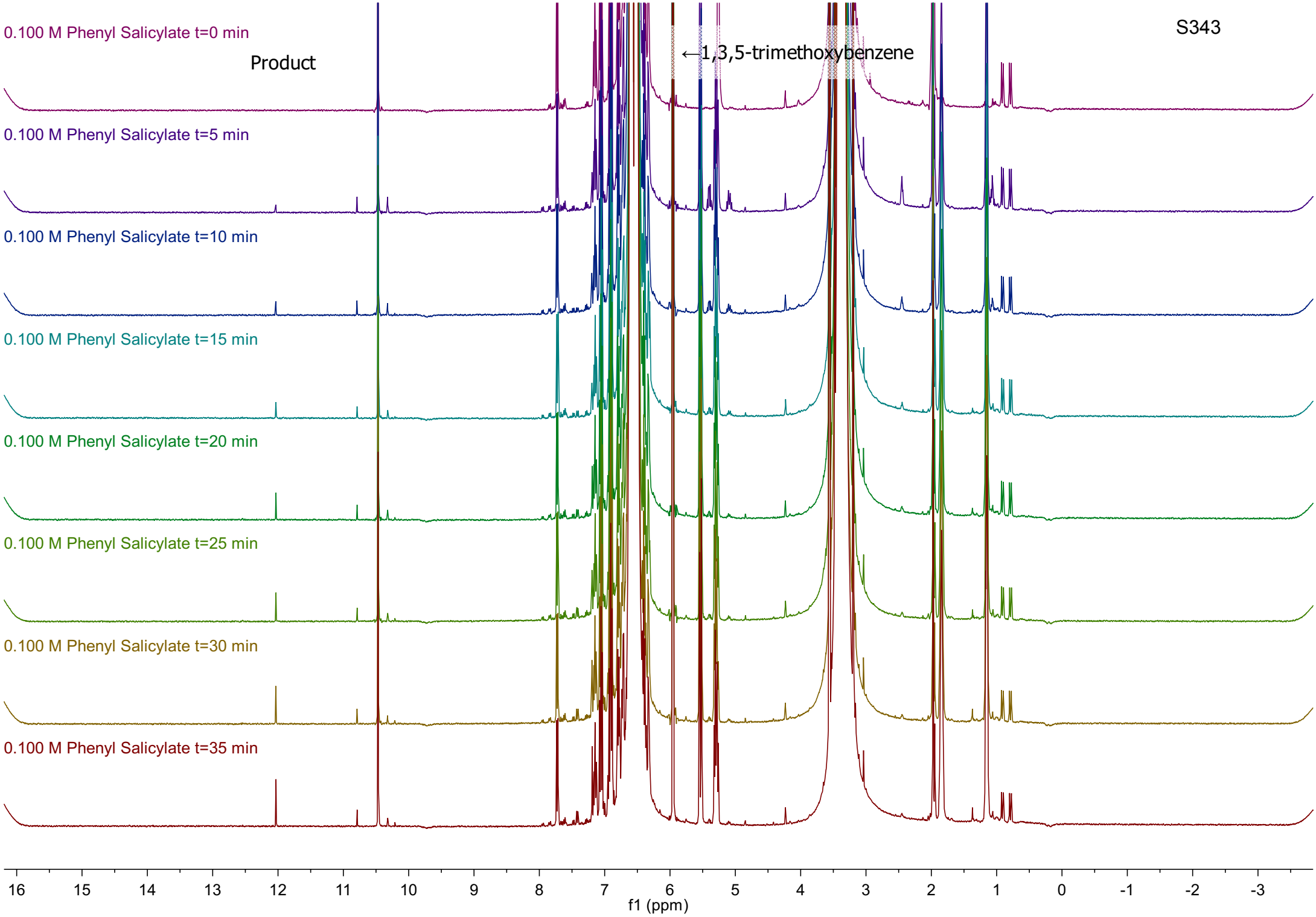

S338
0.050 M Phenyl Salicylate t=0 min
Product
←1,3,5-trimethoxybenzene
0.050 M Phenyl Salicylate t=5 min
0.050 M Phenyl Salicylate t=10 min
0.050 M Phenyl Salicylate t=15 min
0.050 M Phenyl Salicylate t=20 min
0.050 M Phenyl Salicylate t=25 min
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
f1 (ppm)

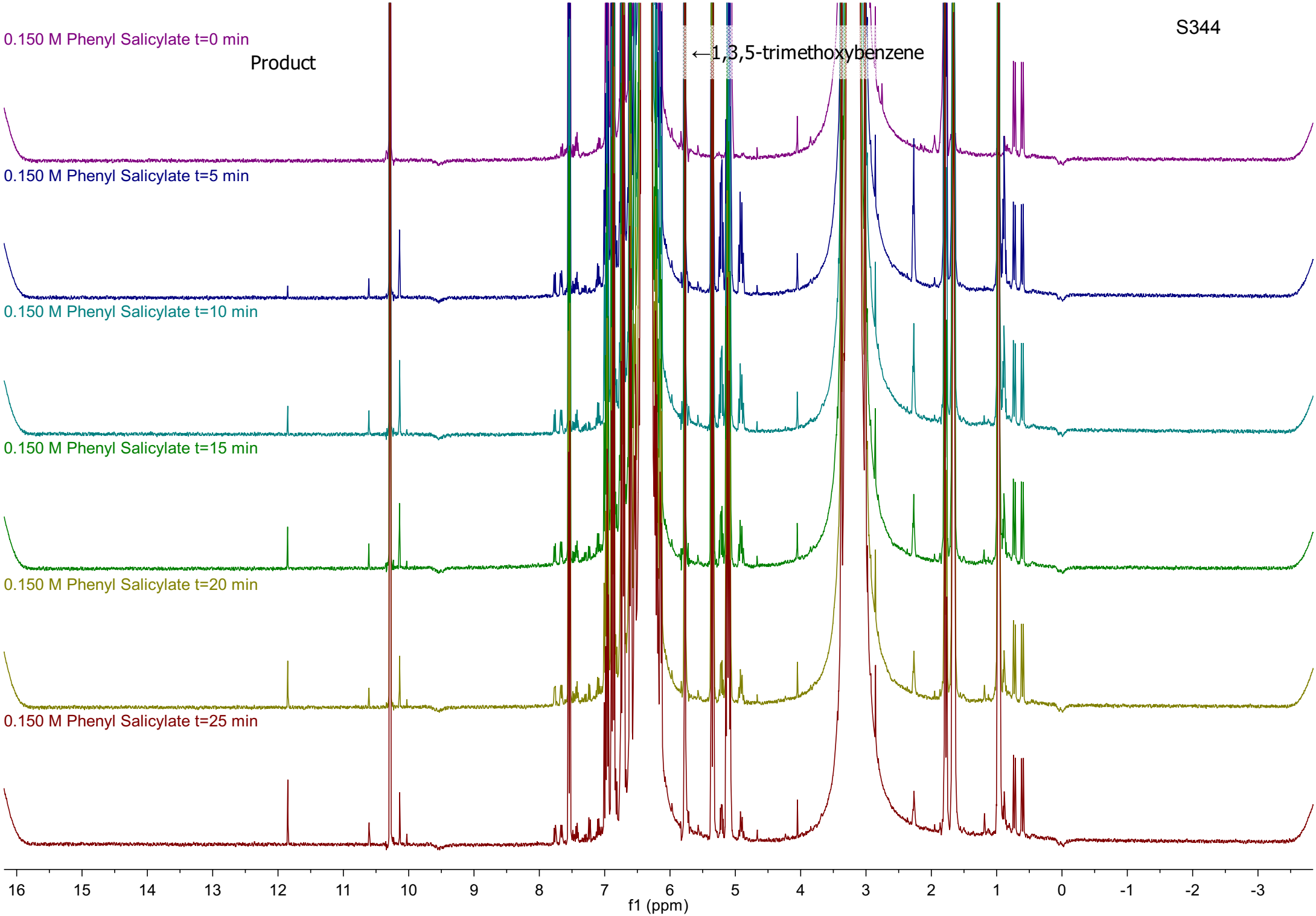

S339
0.050 M Phenyl Salicylate t=0 min
Product
←1,3,5-trimethoxybenzene
0.050 M Phenyl Salicylate t=5 min
0.050 M Phenyl Salicylate t=10 min
0.050 M Phenyl Salicylate t=15 min
0.050 M Phenyl Salicylate t=20 min
0.050 M Phenyl Salicylate t=25 min
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
f1 (ppm)

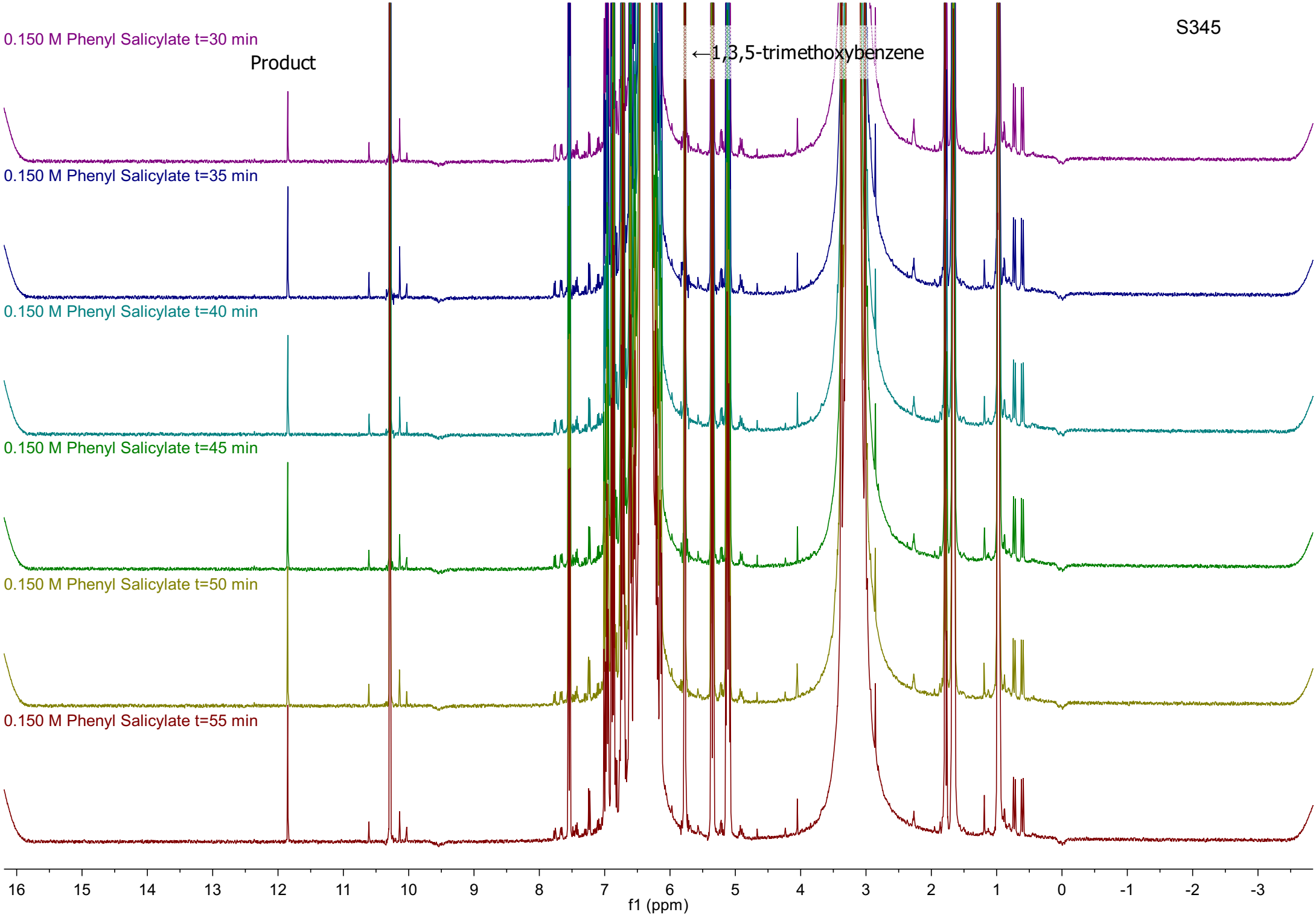

S340
0.100 M Phenyl Salicylate t=0 min
Product
←1,3,5-trimethoxybenzene
0.100 M Phenyl Salicylate t=5 min
0.100 M Phenyl Salicylate t=10 min
0.100 M Phenyl Salicylate t=15 min
0.100 M Phenyl Salicylate t=20 min
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
-3
f1 (ppm)

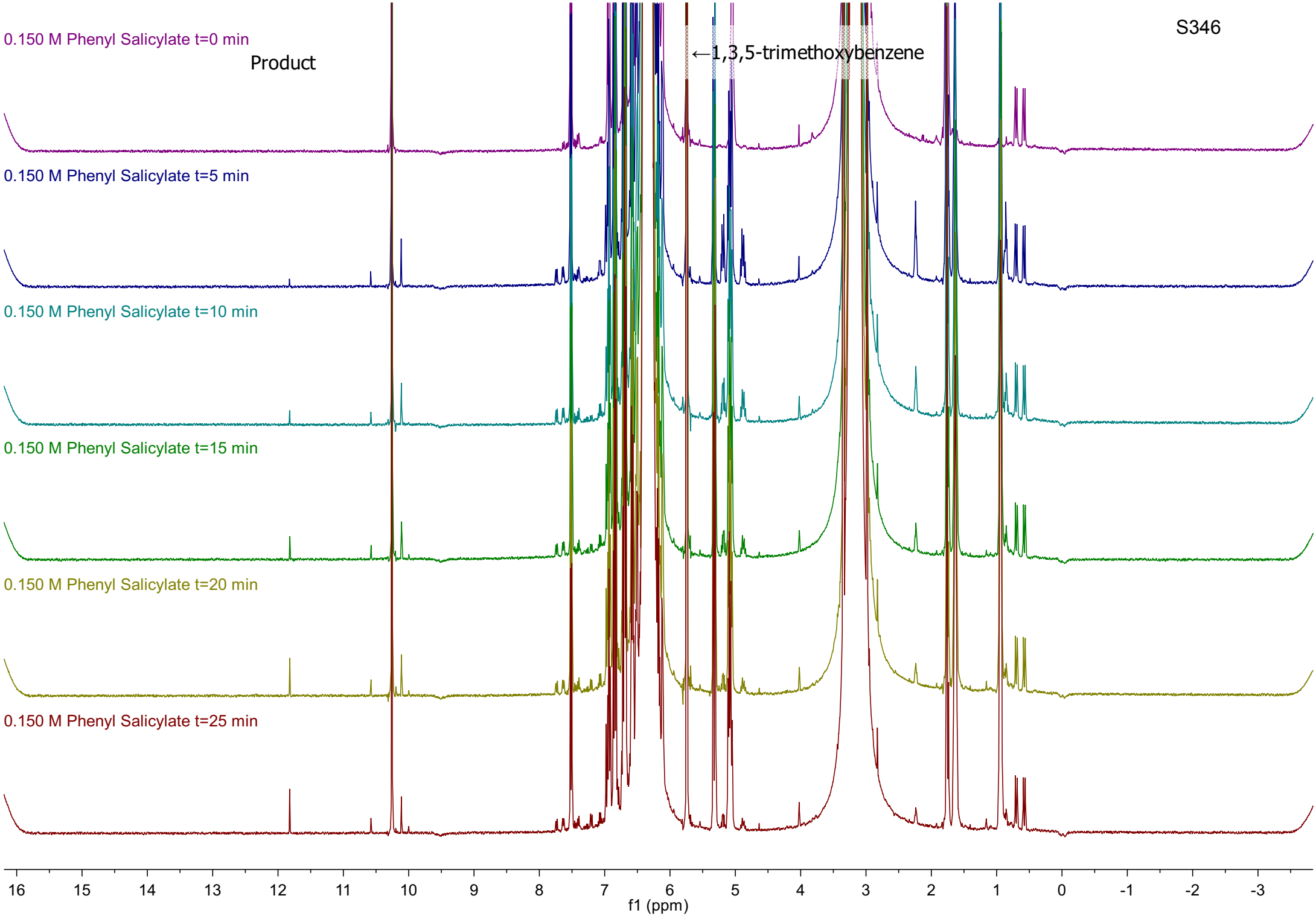

S341
0.100 M Phenyl Salicylate t=25 min
Product
←1,3,5-trimethoxybenzene
0.100 M Phenyl Salicylate t=30 min
0.100 M Phenyl Salicylate t=35 min
0.100 M Phenyl Salicylate t=40 min
0.100 M Phenyl Salicylate t=45 min
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
-3
f1 (ppm)

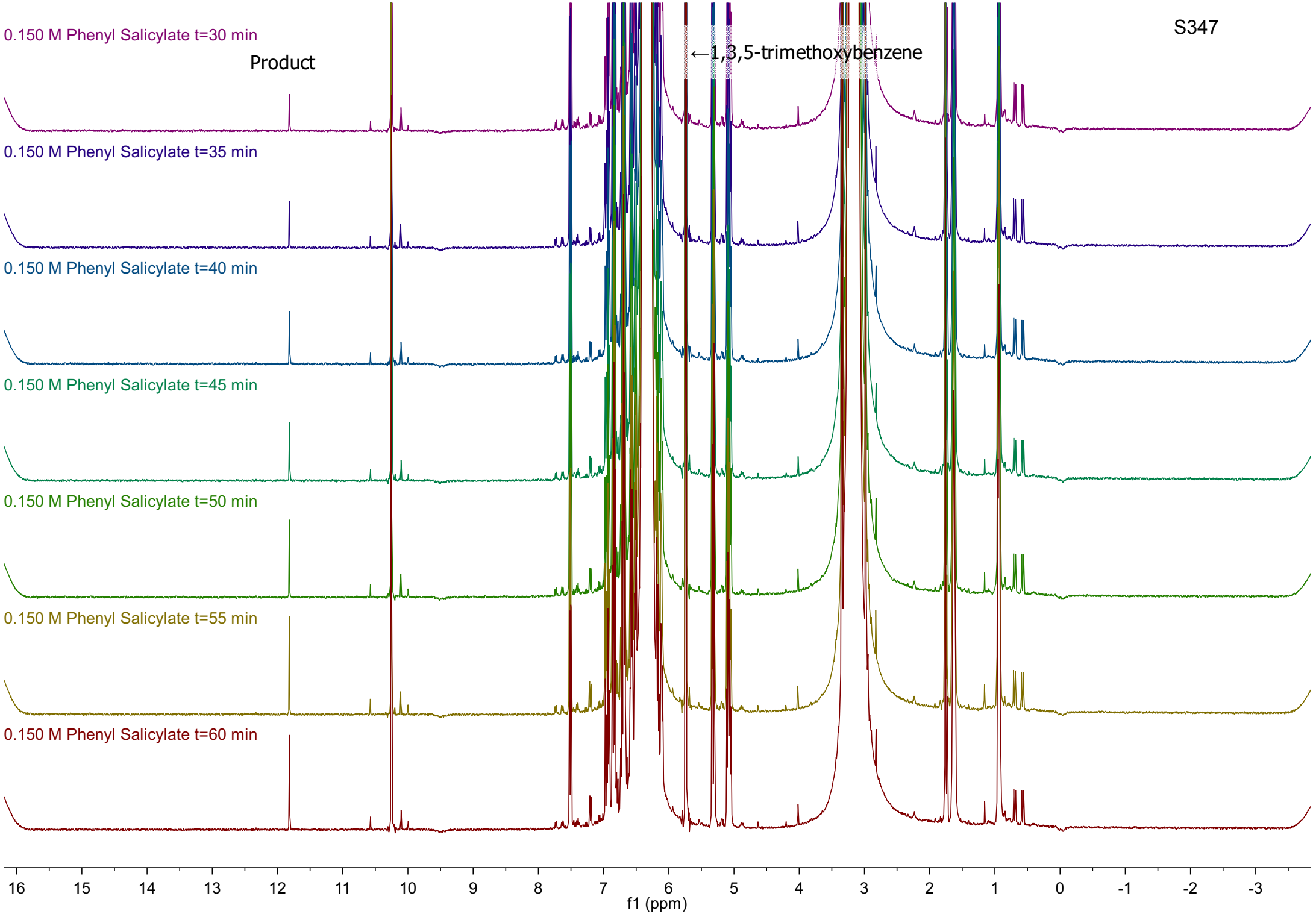

0.100 M Phenyl Salicylate t=0 min
Product
←1,3,5-trimethoxybenzene
S342
0.100 M Phenyl Salicylate t=5 min
0.100 M Phenyl Salicylate t=10 min
0.100 M Phenyl Salicylate t=15 min
0.100 M Phenyl Salicylate t=20 min
0.100 M Phenyl Salicylate t=25 min
0.100 M Phenyl Salicylate t=30 min
0.100 M Phenyl Salicylate t=35 min
0.100 M Phenyl Salicylate t=40 min
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
f1 (ppm)

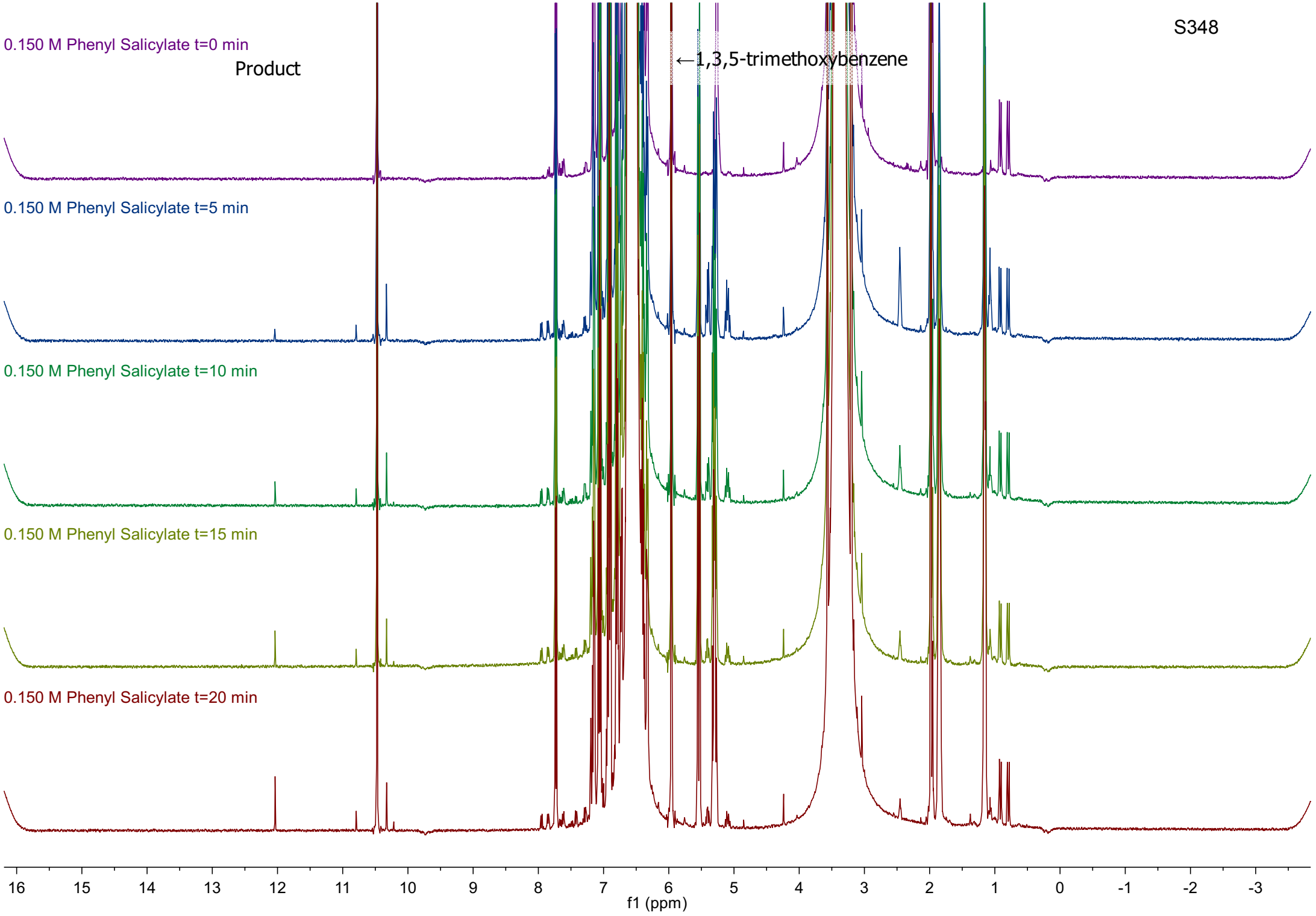

0.100 M Phenyl Salicylate t=0 min
Product
←1,3,5-trimethoxybenzene
S343
0.100 M Phenyl Salicylate t=5 min
0.100 M Phenyl Salicylate t=10 min
0.100 M Phenyl Salicylate t=15 min
0.100 M Phenyl Salicylate t=20 min
0.100 M Phenyl Salicylate t=25 min
0.100 M Phenyl Salicylate t=30 min
0.100 M Phenyl Salicylate t=35 min
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
f1 (ppm)

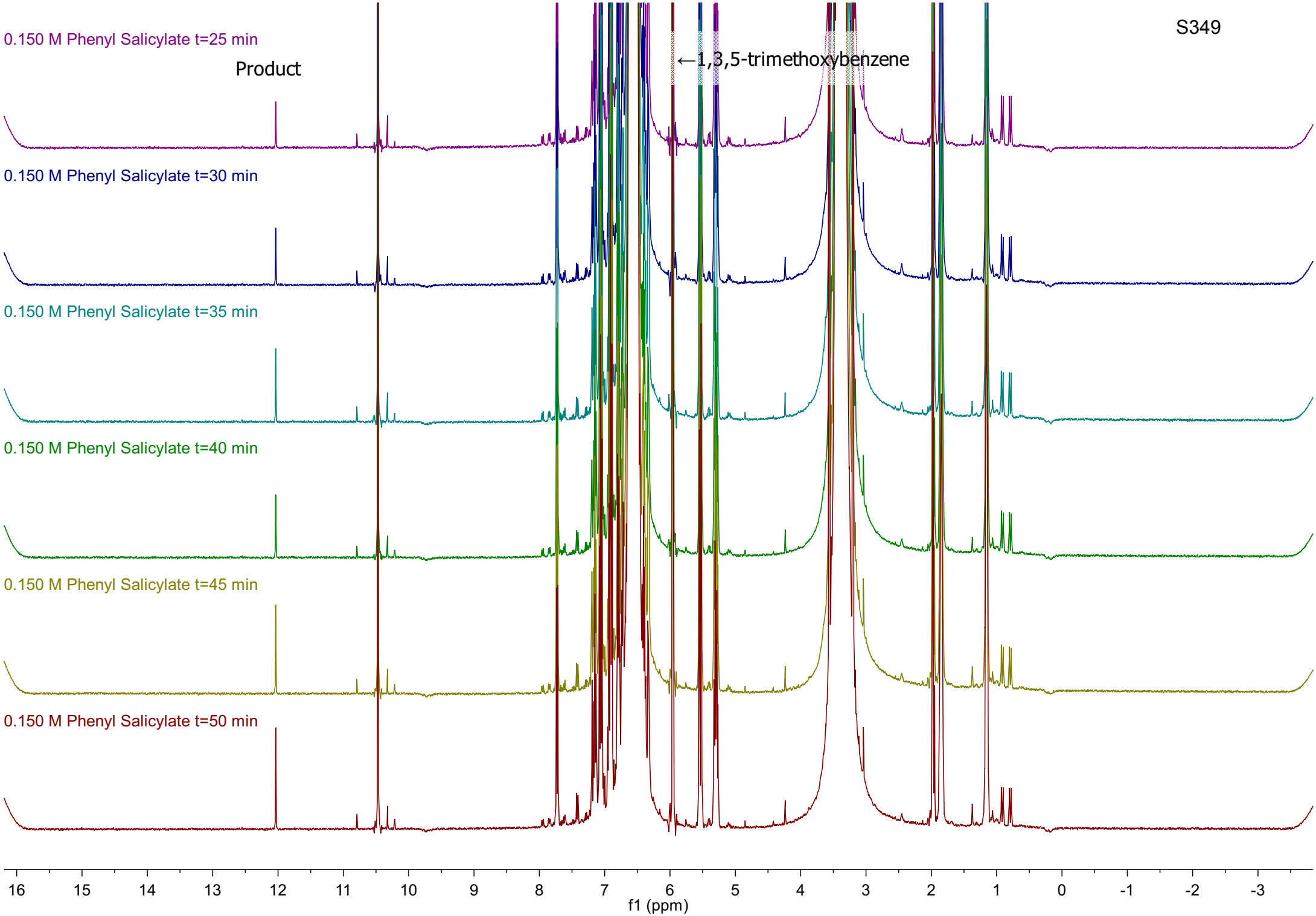

S344
0.150 M Phenyl Salicylate t=0 min
Product
←1,3,5-trimethoxybenzene
0.150 M Phenyl Salicylate t=5 min
0.150 M Phenyl Salicylate t=10 min
0.150 M Phenyl Salicylate t=15 min
0.150 M Phenyl Salicylate t=20 min
0.150 M Phenyl Salicylate t=25 min
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
f1 (ppm)

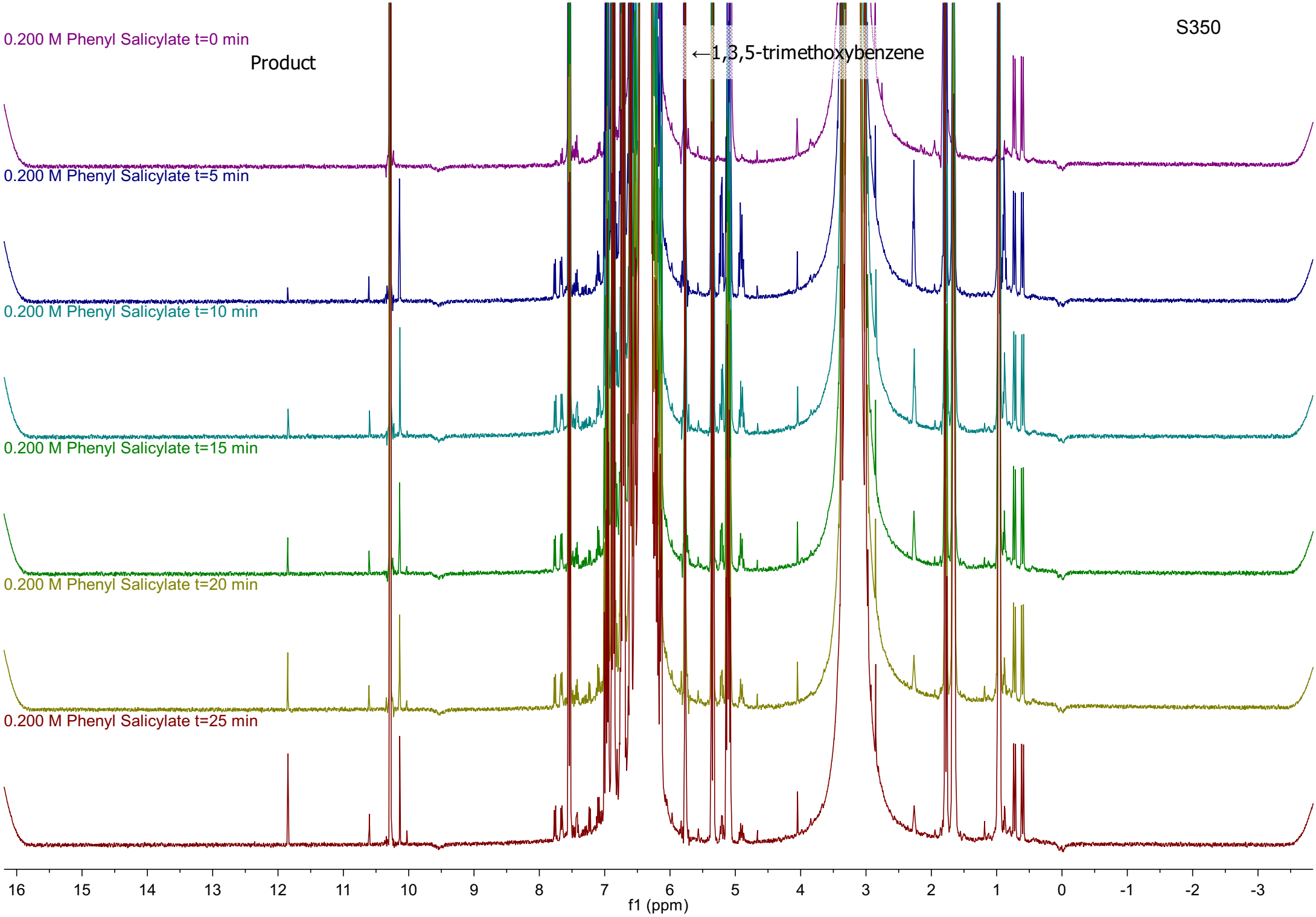

S345
0.150 M Phenyl Salicylate t=30 min
Product
←1,3,5-trimethoxybenzene
0.150 M Phenyl Salicylate t=35 min
0.150 M Phenyl Salicylate t=40 min
0.150 M Phenyl Salicylate t=45 min
0.150 M Phenyl Salicylate t=50 min
0.150 M Phenyl Salicylate t=55 min
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
f1 (ppm)

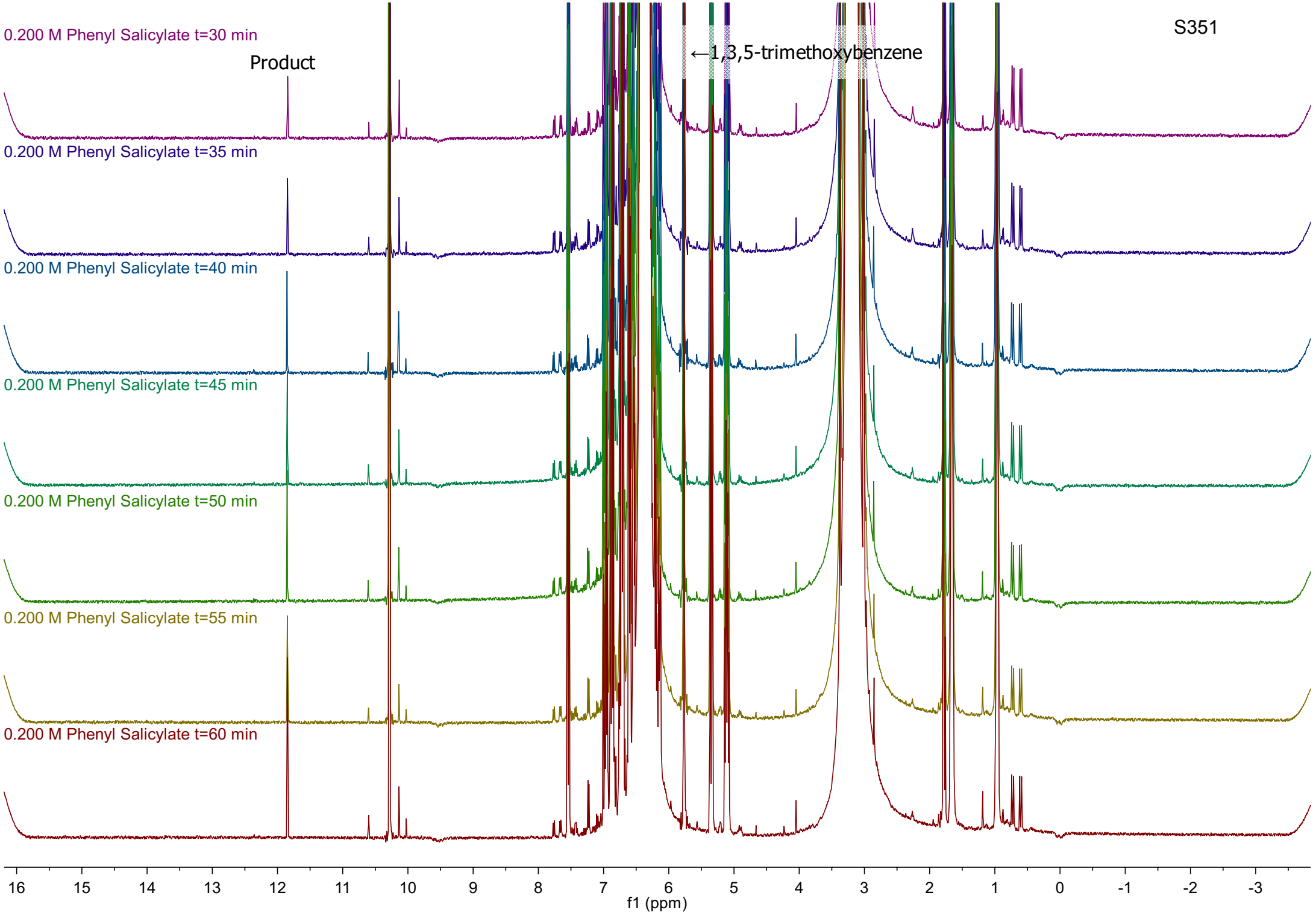

S346
0.150 M Phenyl Salicylate t=0 min
Product
←1,3,5-trimethoxybenzene
0.150 M Phenyl Salicylate t=5 min
0.150 M Phenyl Salicylate t=10 min
0.150 M Phenyl Salicylate t=15 min
0.150 M Phenyl Salicylate t=20 min
0.150 M Phenyl Salicylate t=25 min
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
f1 (ppm)

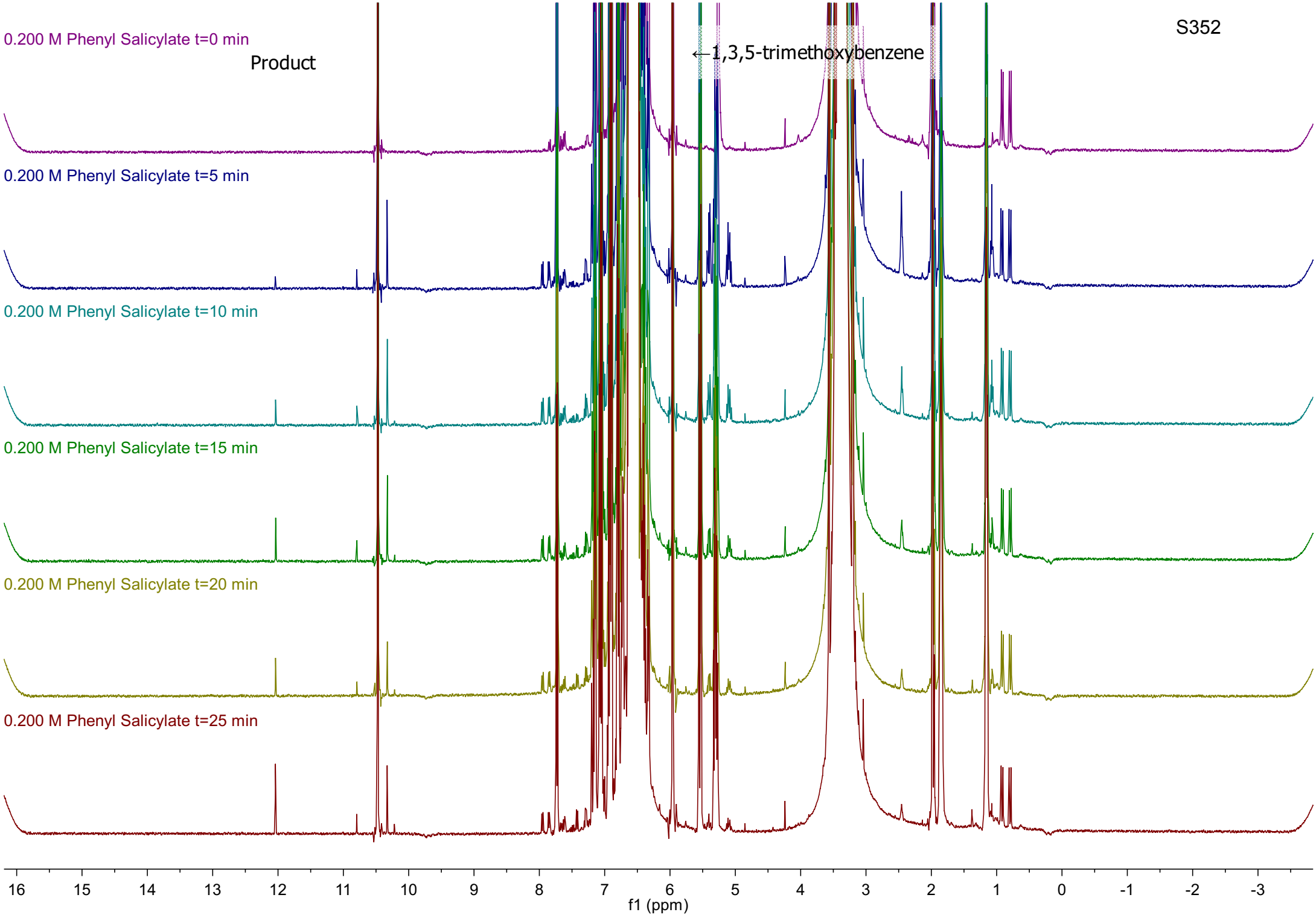

0.150 M Phenyl Salicylate t=30 min
Product
←1,3,5-trimethoxybenzene
S347
0.150 M Phenyl Salicylate t=35 min
0.150 M Phenyl Salicylate t=40 min
0.150 M Phenyl Salicylate t=45 min
0.150 M Phenyl Salicylate t=50 min
0.150 M Phenyl Salicylate t=55 min
0.150 M Phenyl Salicylate t=60 min
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
f1 (ppm)

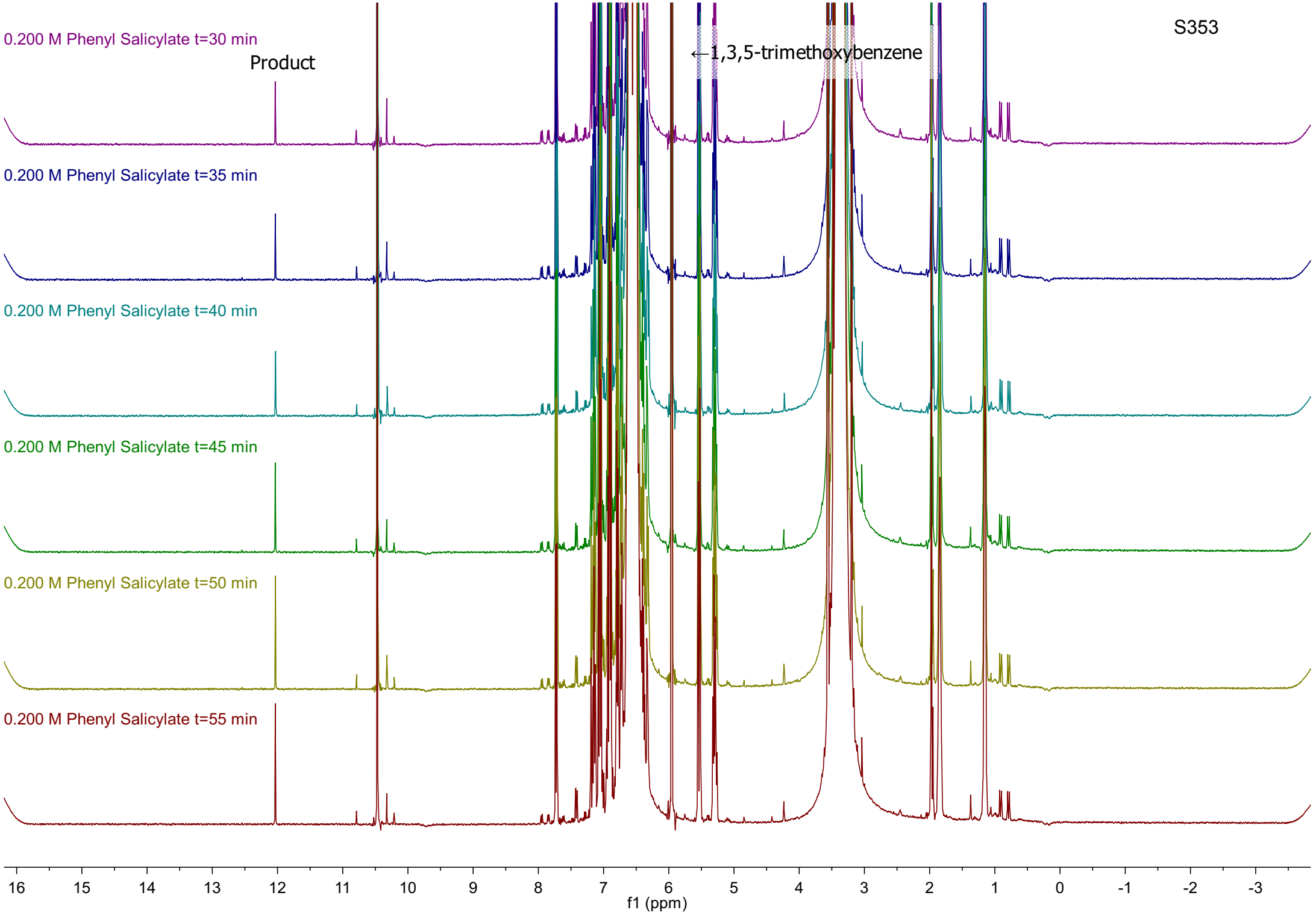

S348
0.150 M Phenyl Salicylate t=0 min
Product
←1,3,5-trimethoxybenzene
0.150 M Phenyl Salicylate t=5 min
0.150 M Phenyl Salicylate t=10 min
0.150 M Phenyl Salicylate t=15 min
0.150 M Phenyl Salicylate t=20 min
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
-3
f1 (ppm)

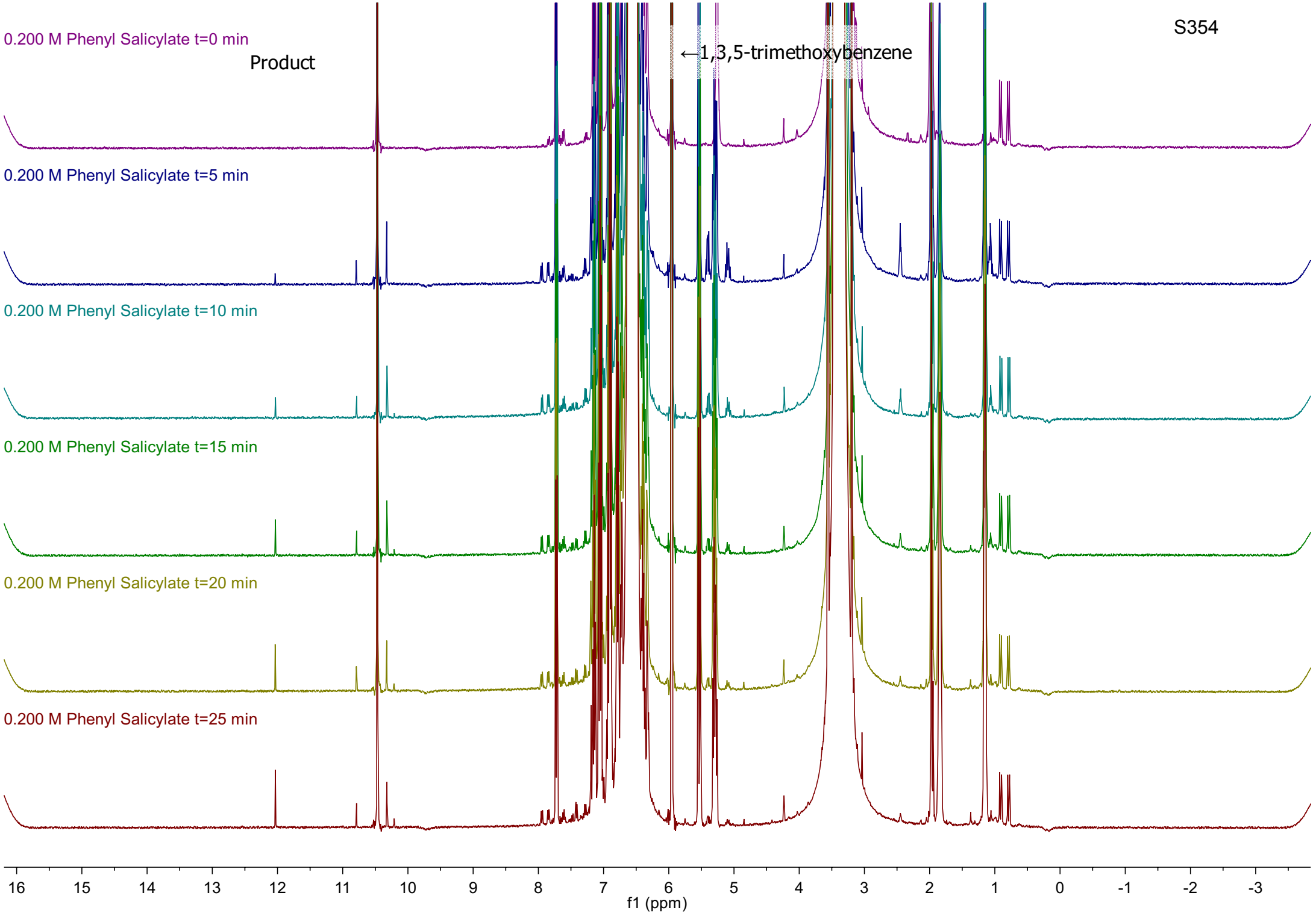

S349
0.150 M Phenyl Salicylate t=25 min
Product
←1,3,5-trimethoxybenzene
0.150 M Phenyl Salicylate t=30 min
0.150 M Phenyl Salicylate t=35 min
0.150 M Phenyl Salicylate t=40 min
0.150 M Phenyl Salicylate t=45 min
0.150 M Phenyl Salicylate t=50 min
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
f1 (ppm)

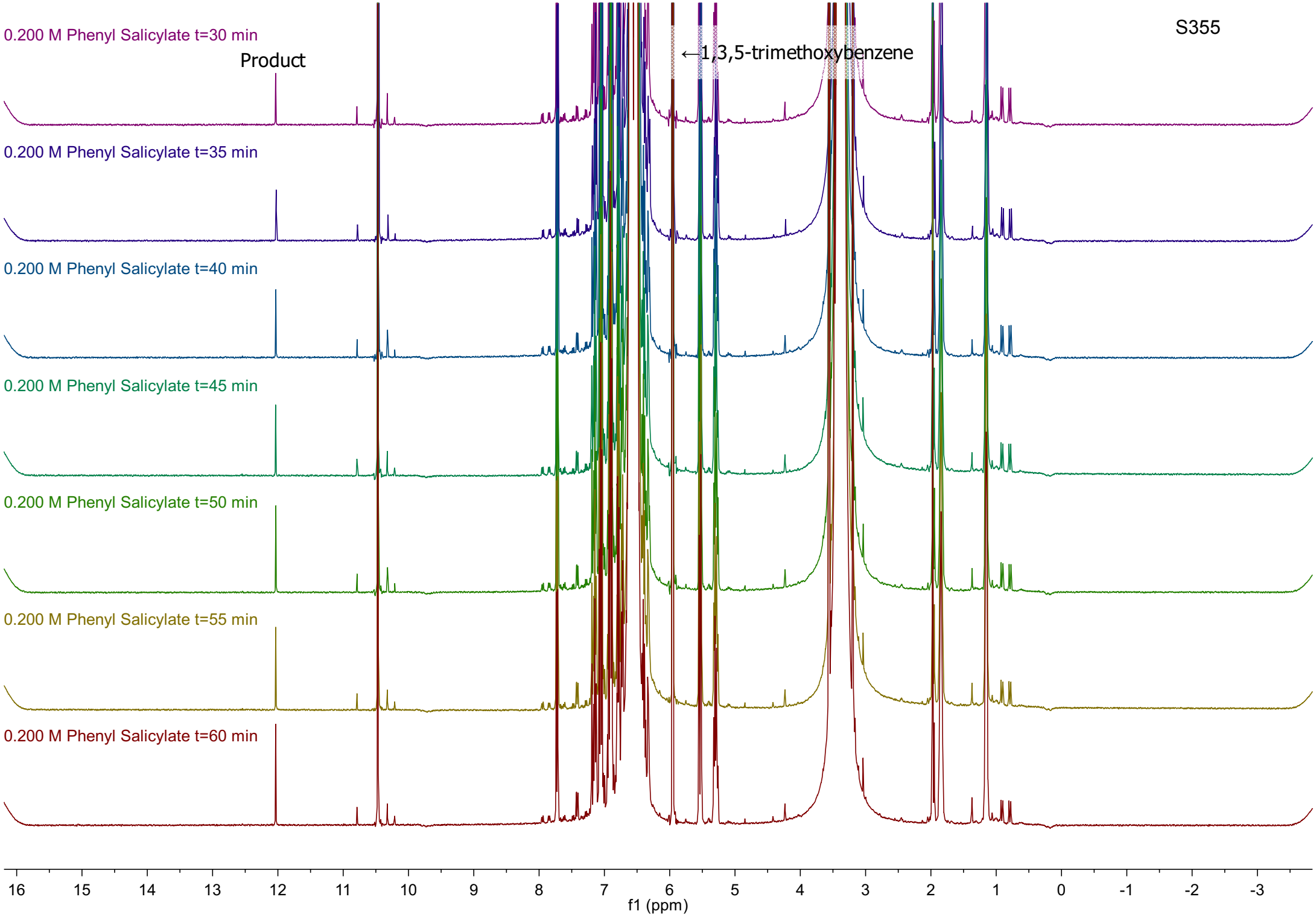

S350
0.200 M Phenyl Salicylate t=0 min
Product
←1,3,5-trimethoxybenzene
0.200 M Phenyl Salicylate t=5 min
0.200 M Phenyl Salicylate t=10 min
0.200 M Phenyl Salicylate t=15 min
0.200 M Phenyl Salicylate t=20 min
0.200 M Phenyl Salicylate t=25 min
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
f1 (ppm)

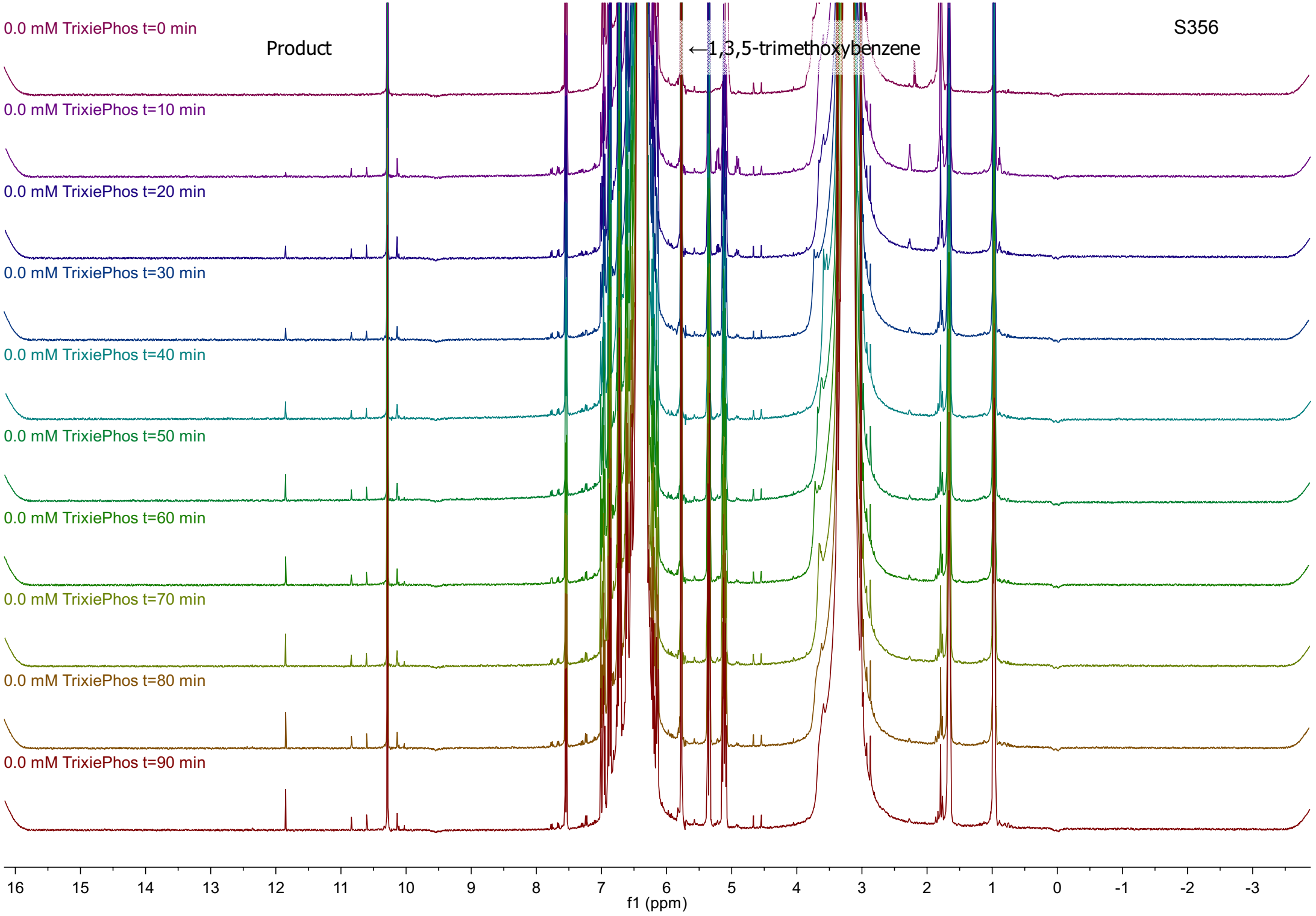

S351
0.200 M Phenyl Salicylate t=30 min
Product
←1,3,5-trimethoxybenzene
0.200 M Phenyl Salicylate t=35 min
0.200 M Phenyl Salicylate t=40 min
0.200 M Phenyl Salicylate t=45 min
0.200 M Phenyl Salicylate t=50 min
0.200 M Phenyl Salicylate t=55 min
0.200 M Phenyl Salicylate t=60 min
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
f1 (ppm)

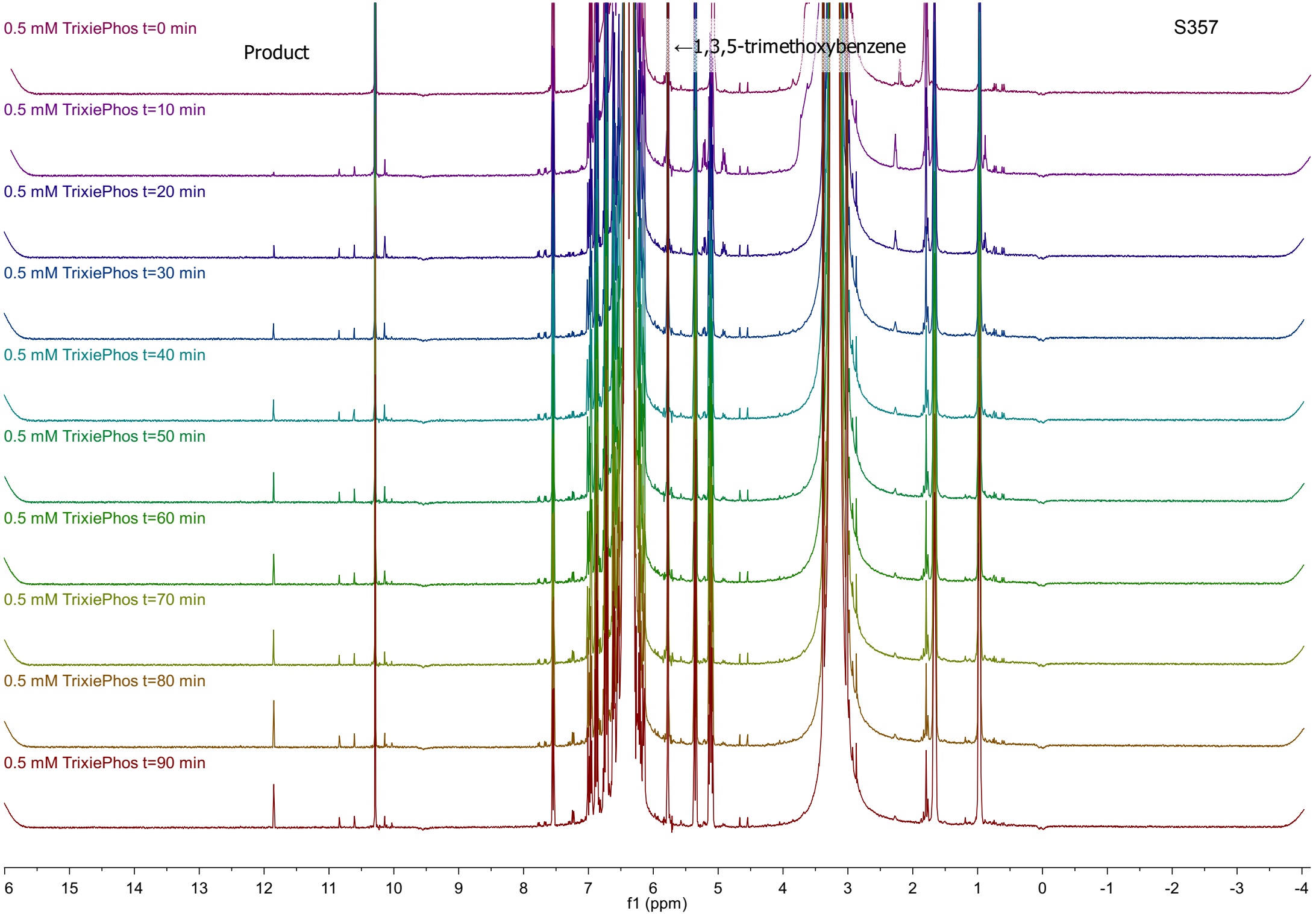

S352
0.200 M Phenyl Salicylate t=0 min
Product
←1,3,5-trimethoxybenzene
0.200 M Phenyl Salicylate t=5 min
0.200 M Phenyl Salicylate t=10 min
0.200 M Phenyl Salicylate t=15 min
0.200 M Phenyl Salicylate t=20 min
0.200 M Phenyl Salicylate t=25 min
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
f1 (ppm)

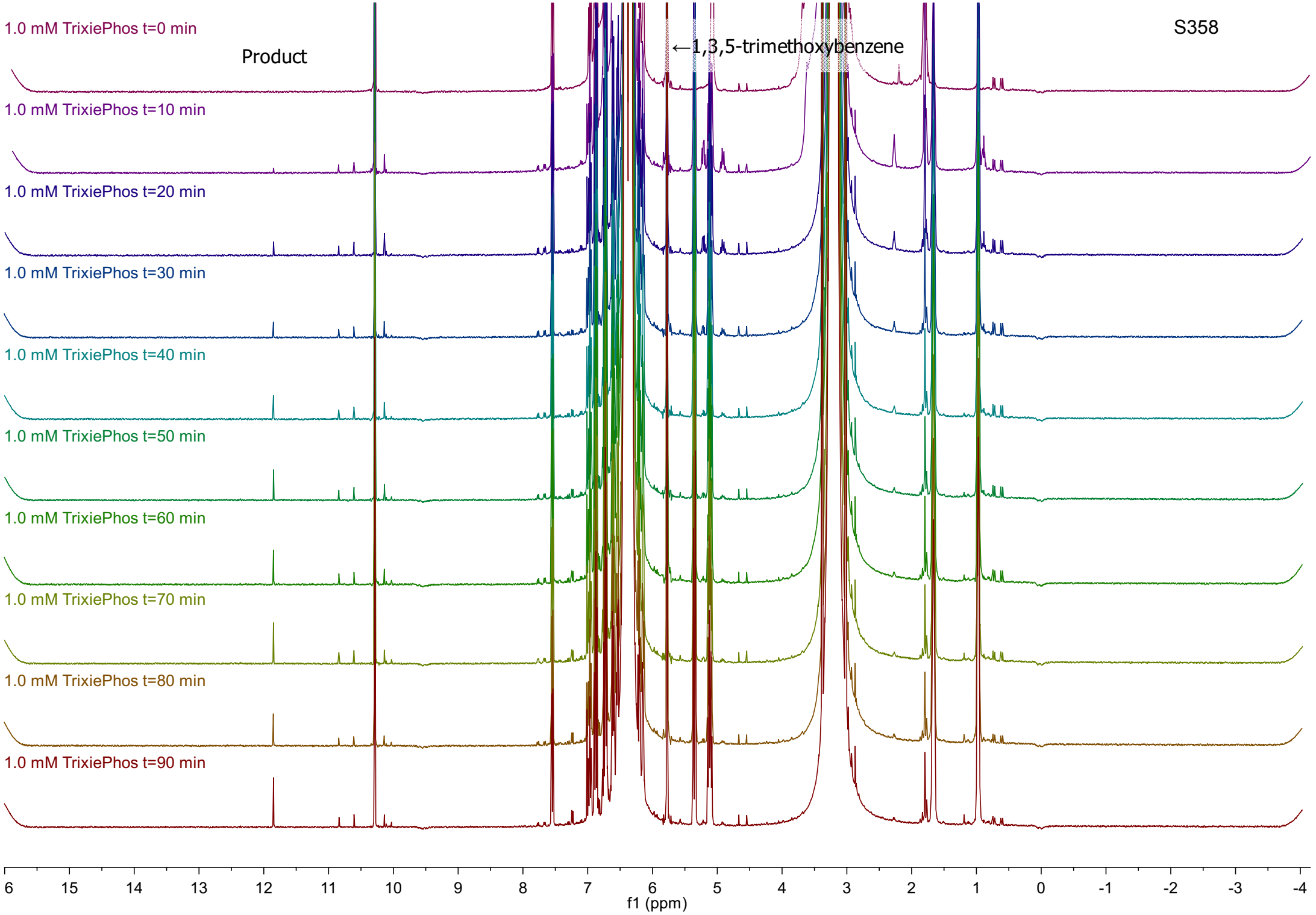

S353
0.200 M Phenyl Salicylate t=30 min
Product
←1,3,5-trimethoxybenzene
0.200 M Phenyl Salicylate t=35 min
0.200 M Phenyl Salicylate t=40 min
0.200 M Phenyl Salicylate t=45 min
0.200 M Phenyl Salicylate t=50 min
0.200 M Phenyl Salicylate t=55 min
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
f1 (ppm)

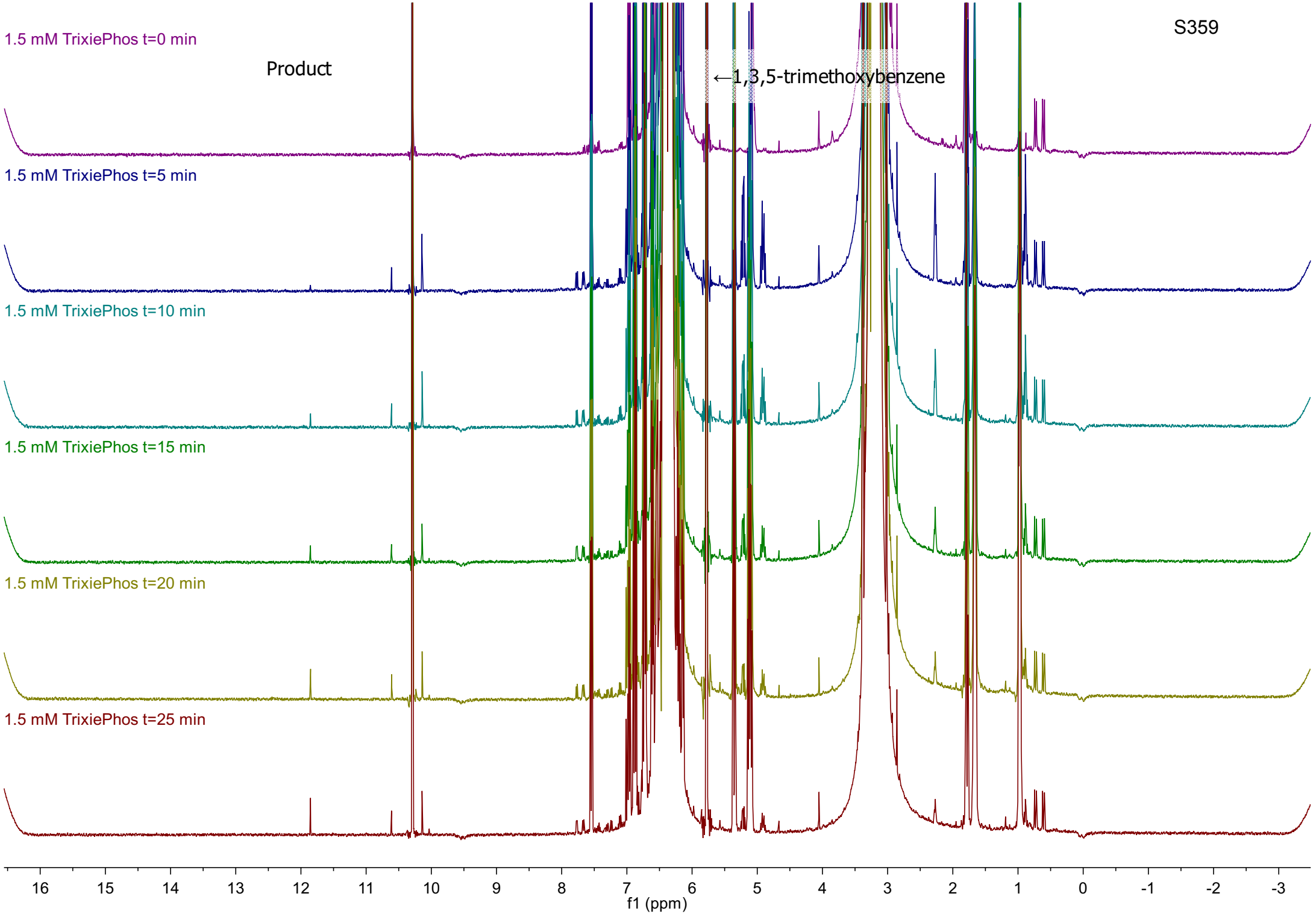

0.200 M Phenyl Salicylate t=0 min
Product
←1,3,5-trimethoxybenzene
S354
0.200 M Phenyl Salicylate t=5 min
0.200 M Phenyl Salicylate t=10 min
0.200 M Phenyl Salicylate t=15 min
0.200 M Phenyl Salicylate t=20 min
0.200 M Phenyl Salicylate t=25 min
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
f1 (ppm)

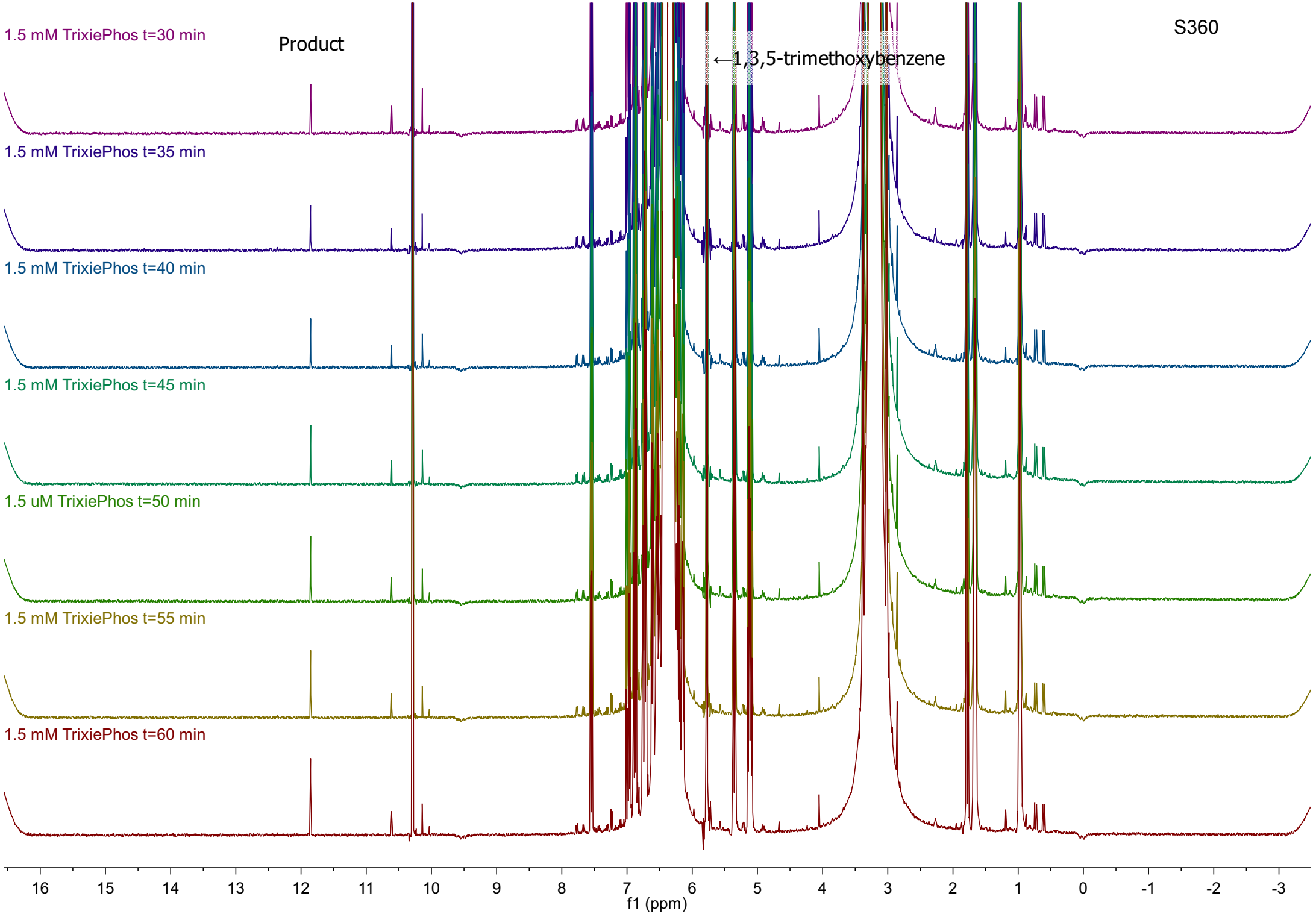

S355
0.200 M Phenyl Salicylate t=30 min
Product
←1,3,5-trimethoxybenzene
0.200 M Phenyl Salicylate t=35 min
0.200 M Phenyl Salicylate t=40 min
0.200 M Phenyl Salicylate t=45 min
0.200 M Phenyl Salicylate t=50 min
0.200 M Phenyl Salicylate t=55 min
0.200 M Phenyl Salicylate t=60 min
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
f1 (ppm)

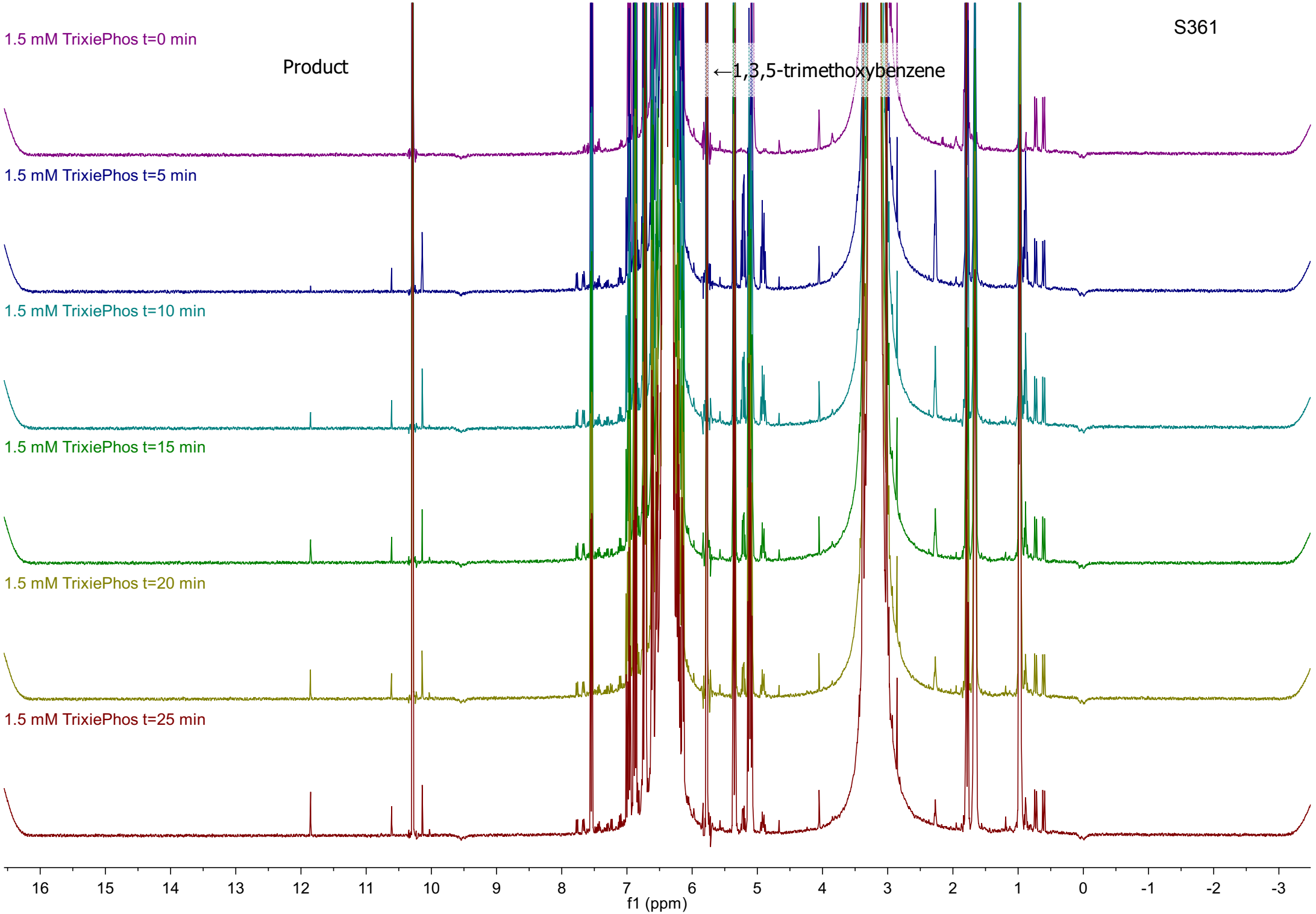

0.0 mM TrixiePhos t=0 min
Product
←1,3,5-trimethoxybenzene
S356
0.0 mM TrixiePhos t=10 min
0.0 mM TrixiePhos t=20 min
0.0 mM TrixiePhos t=30 min
0.0 mM TrixiePhos t=40 min
0.0 mM TrixiePhos t=50 min
0.0 mM TrixiePhos t=60 min
0.0 mM TrixiePhos t=70 min
0.0 mM TrixiePhos t=80 min
0.0 mM TrixiePhos t=90 min
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
f1 (ppm)

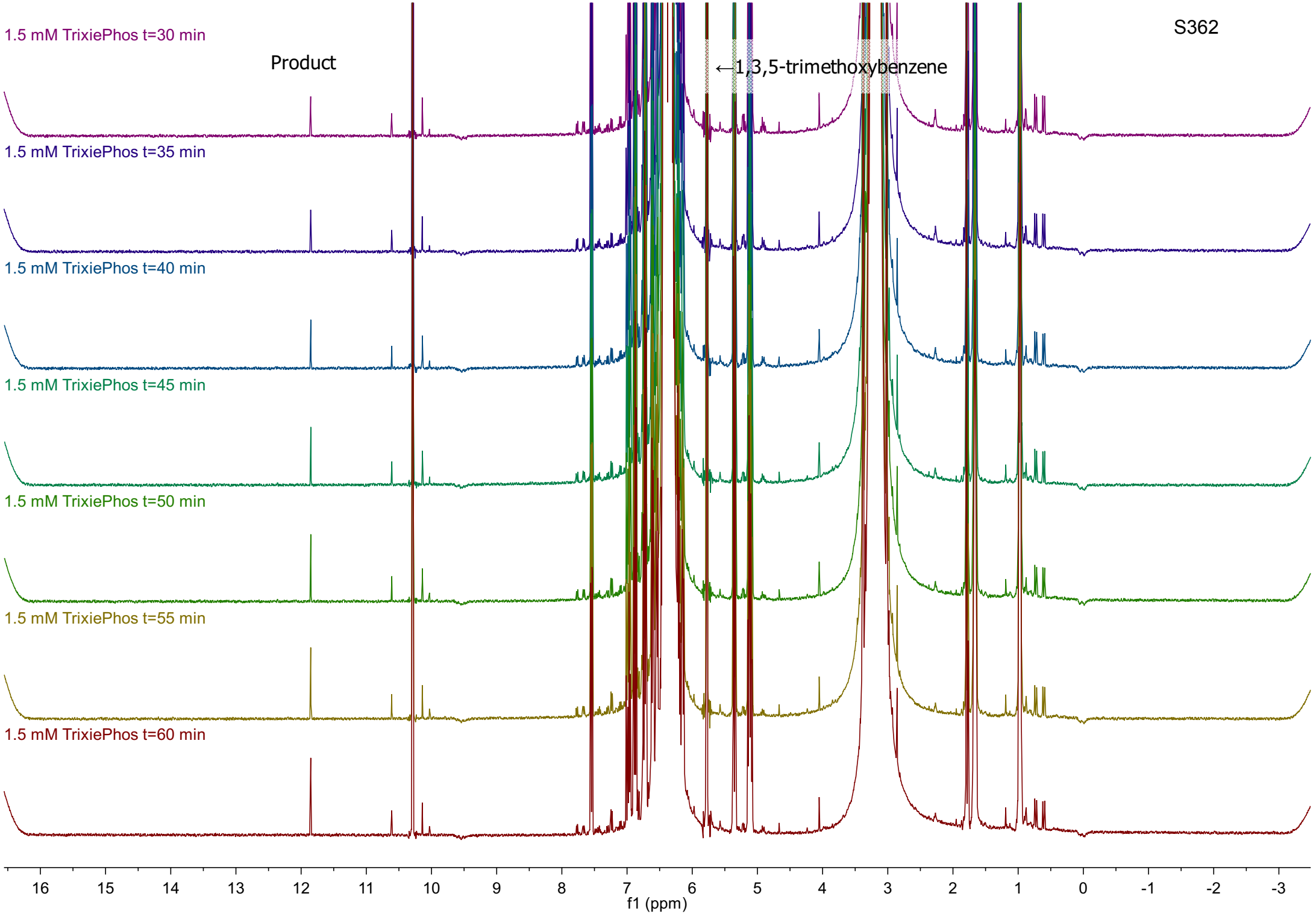

0.5 mM TrixiePhos t=0 min
Product
←1,3,5-trimethoxybenzene
S357
0.5 mM TrixiePhos t=10 min
0.5 mM TrixiePhos t=20 min
0.5 mM TrixiePhos t=30 min
0.5 mM TrixiePhos t=40 min
0.5 mM TrixiePhos t=50 min
0.5 mM TrixiePhos t=60 min
0.5 mM TrixiePhos t=70 min
0.5 mM TrixiePhos t=80 min
0.5 mM TrixiePhos t=90 min
6 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3 -4
f1 (ppm)

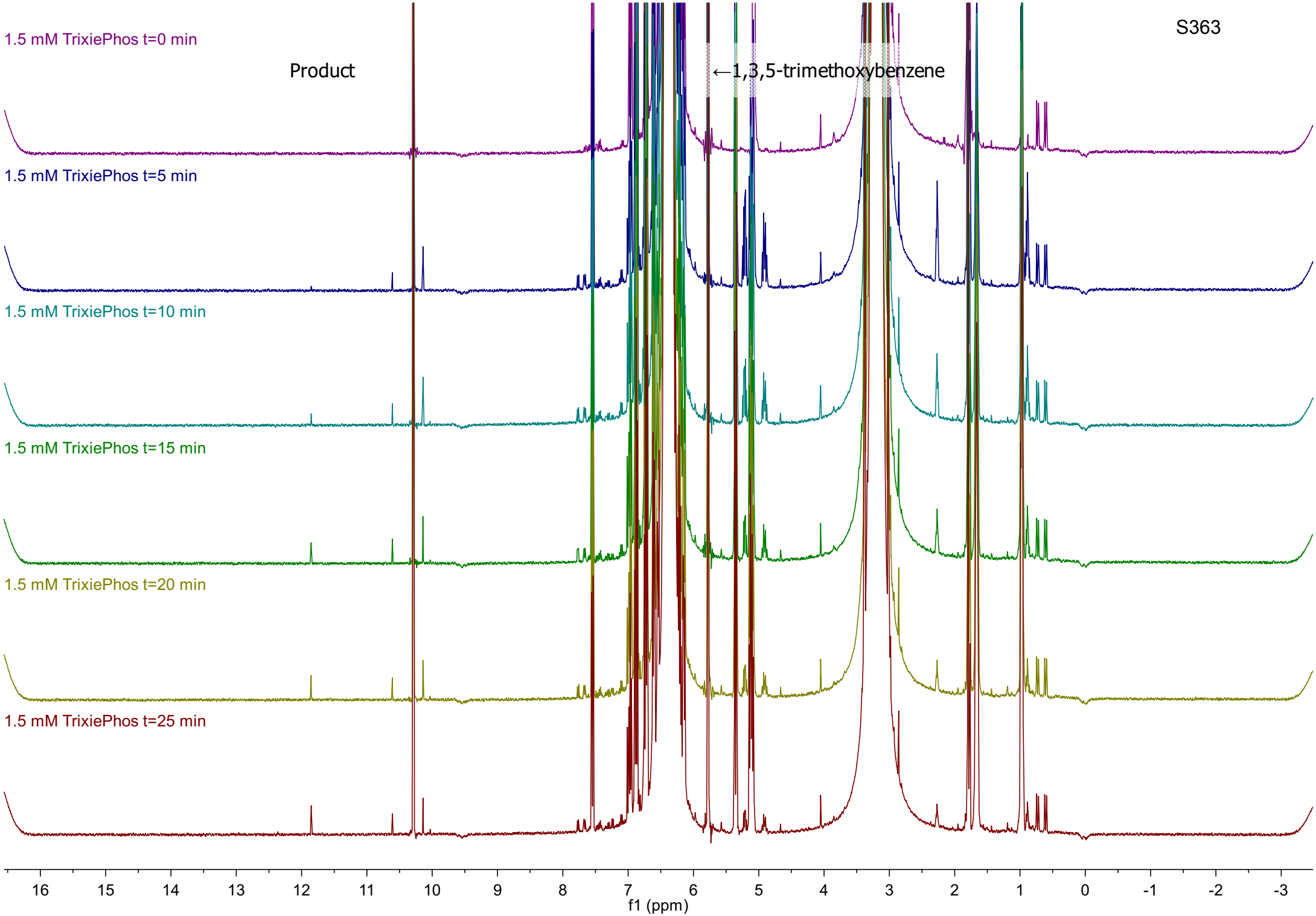

1.0 mM TrixiePhos t=0 min
Product
←1,3,5-trimethoxybenzene
S358
1.0 mM TrixiePhos t=10 min
1.0 mM TrixiePhos t=20 min
1.0 mM TrixiePhos t=30 min
1.0 mM TrixiePhos t=40 min
1.0 mM TrixiePhos t=50 min
1.0 mM TrixiePhos t=60 min
1.0 mM TrixiePhos t=70 min
1.0 mM TrixiePhos t=80 min
1.0 mM TrixiePhos t=90 min
6 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3 -4
f1 (ppm)

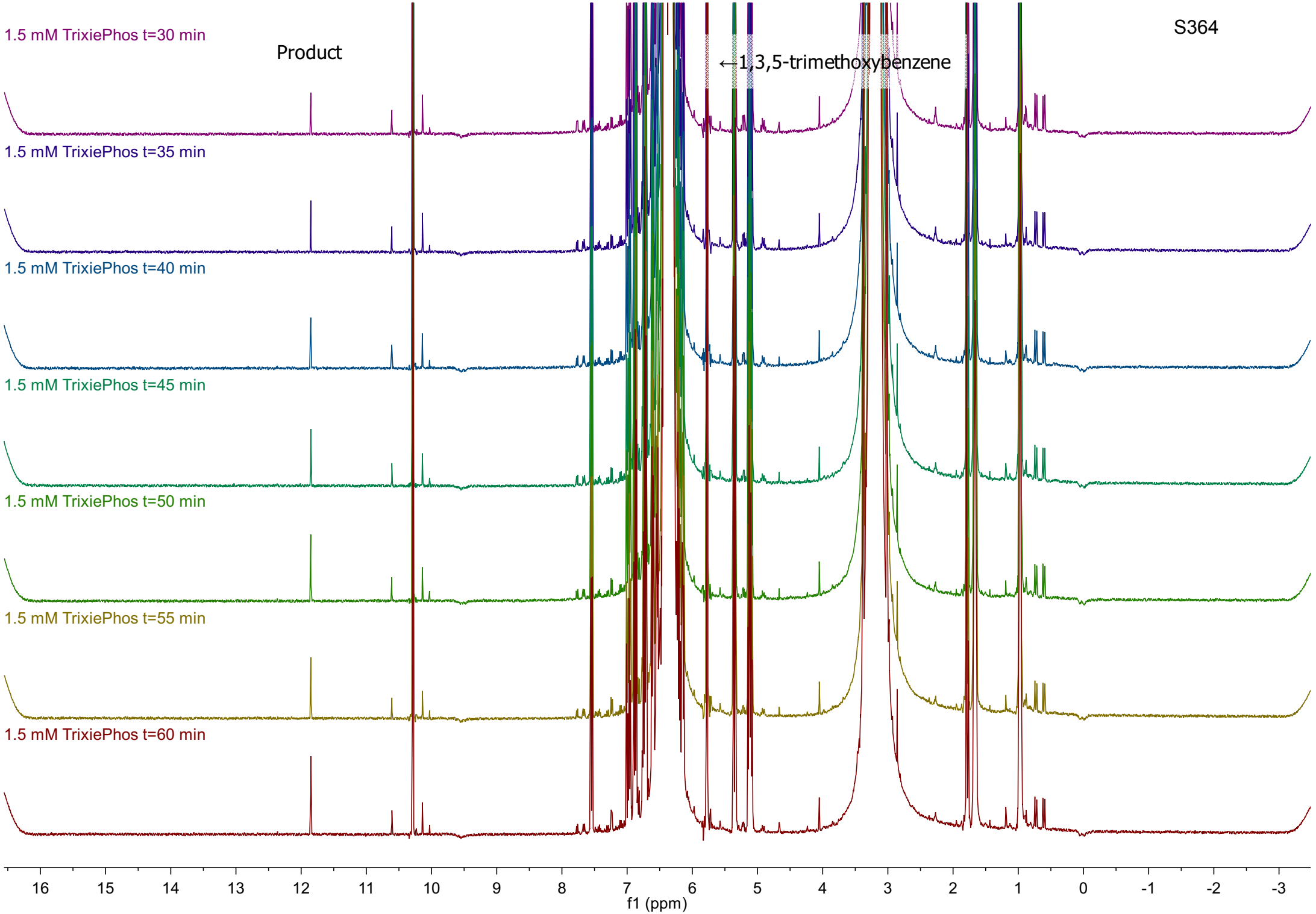

S359
1.5 mM TrixiePhos t=0 min
Product
←1,3,5-trimethoxybenzene
1.5 mM TrixiePhos t=5 min
1.5 mM TrixiePhos t=10 min
1.5 mM TrixiePhos t=15 min
1.5 mM TrixiePhos t=20 min
1.5 mM TrixiePhos t=25 min
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
-3
f1 (ppm)

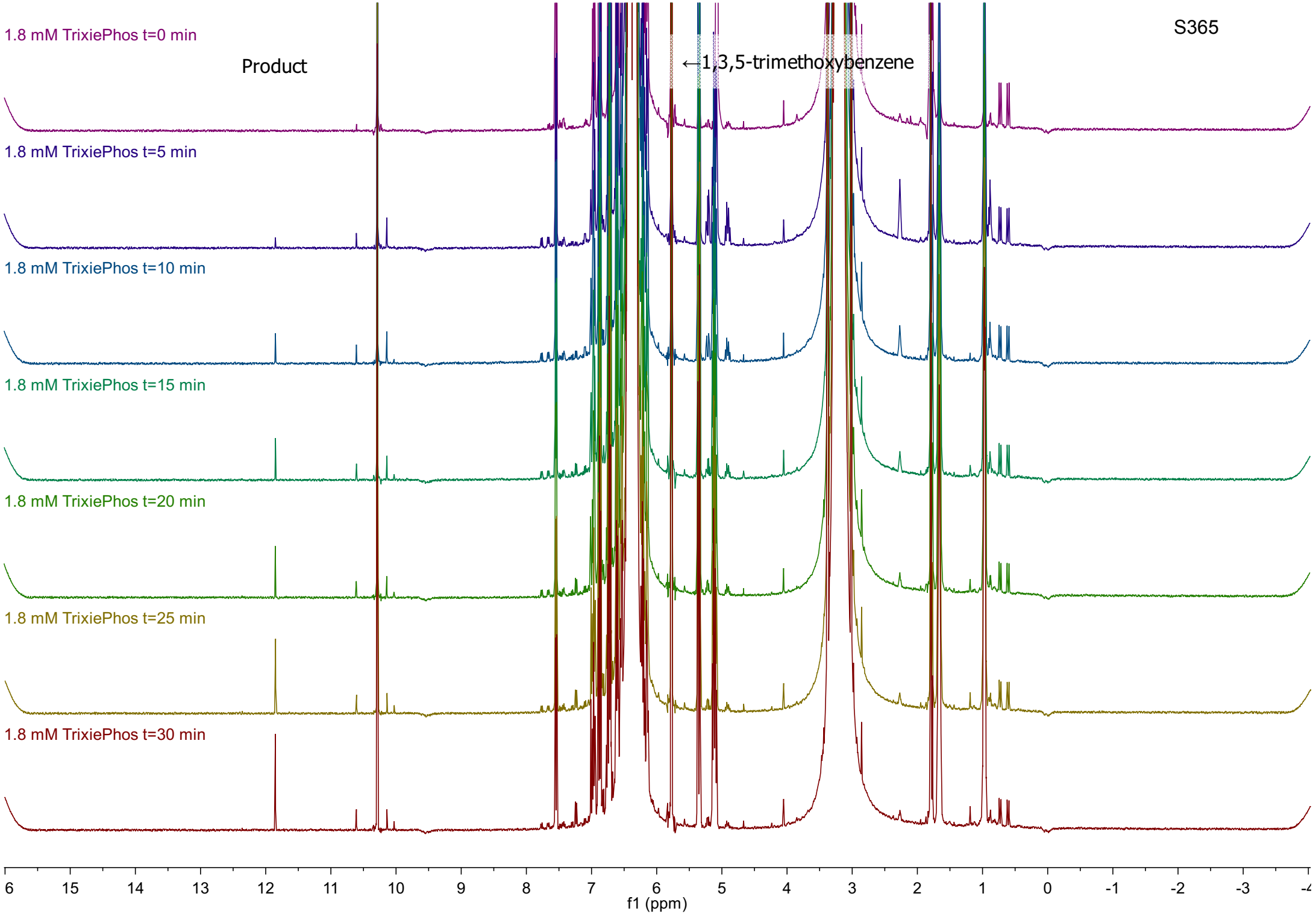

1.5 mM TrixiePhos t=30 min
Product
←1,3,5-trimethoxybenzene
S360
1.5 mM TrixiePhos t=35 min
1.5 mM TrixiePhos t=40 min
1.5 mM TrixiePhos t=45 min
1.5 uM TrixiePhos t=50 min
1.5 mM TrixiePhos t=55 min
1.5 mM TrixiePhos t=60 min
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
f1 (ppm)

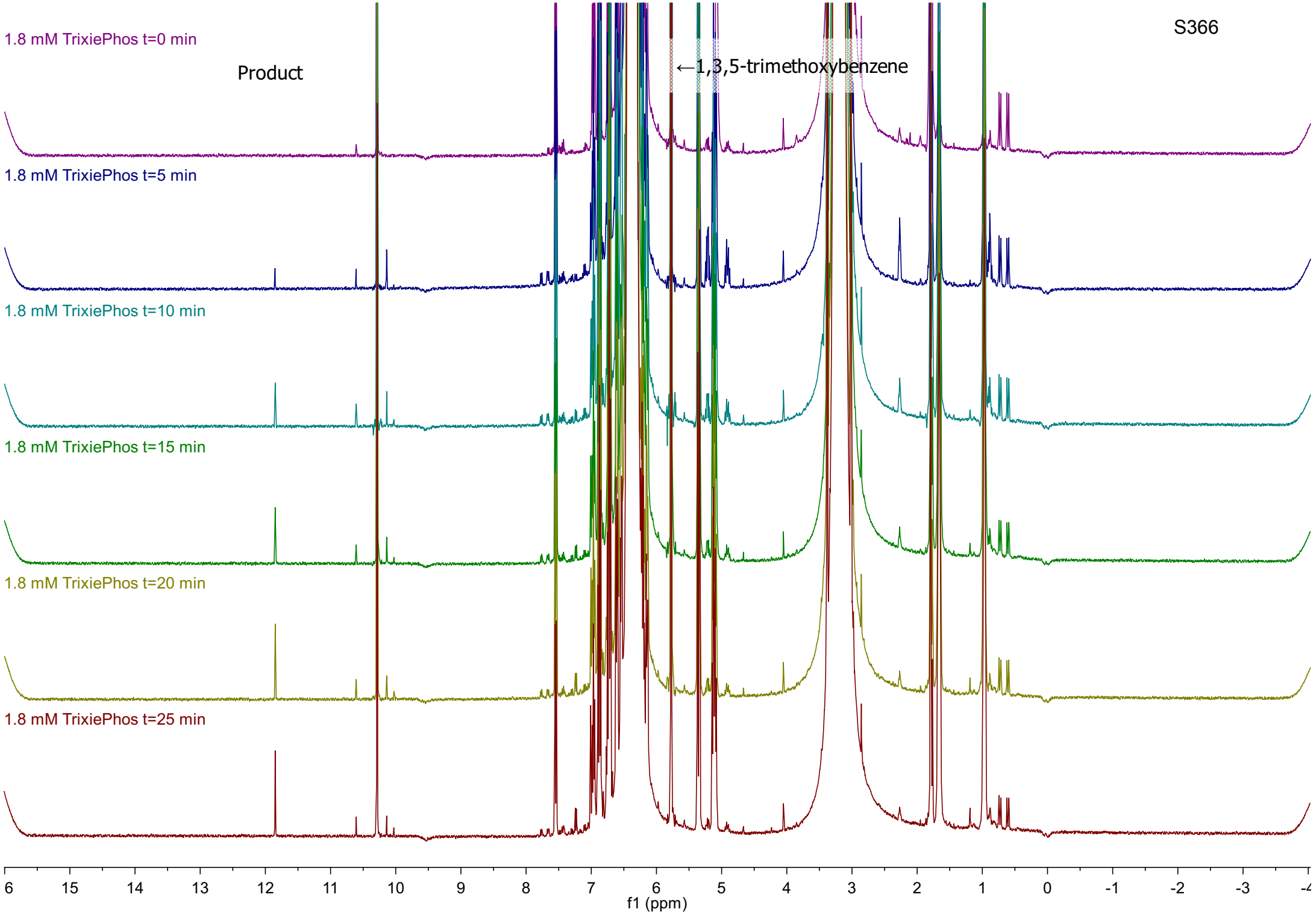

S361
1.5 mM TrixiePhos t=0 min
Product
←1,3,5-trimethoxybenzene
1.5 mM TrixiePhos t=5 min
1.5 mM TrixiePhos t=10 min
1.5 mM TrixiePhos t=15 min
1.5 mM TrixiePhos t=20 min
1.5 mM TrixiePhos t=25 min
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
-3
f1 (ppm)

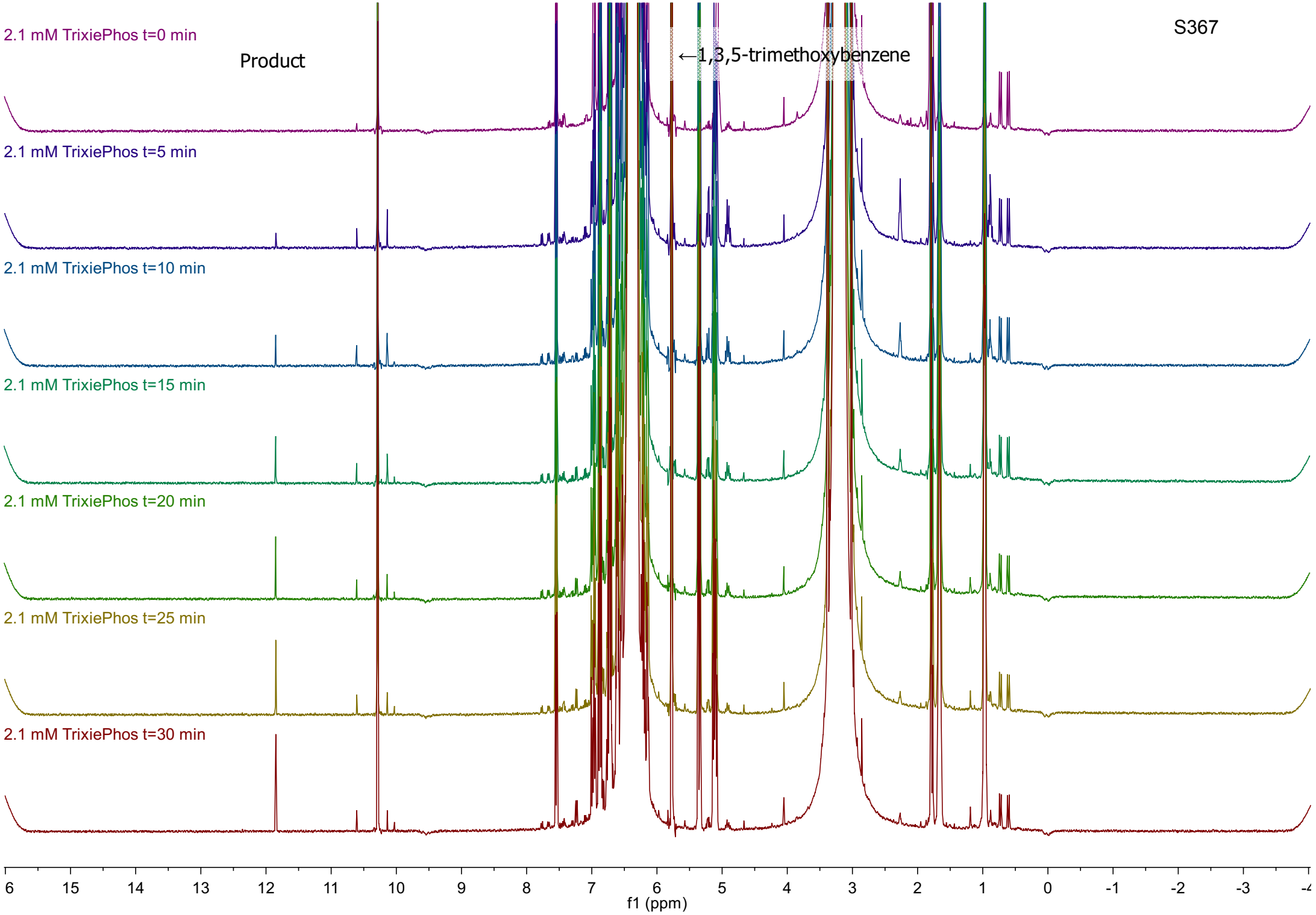

S362
1.5 mM TrixiePhos t=30 min
Product
←1,3,5-trimethoxybenzene
1.5 mM TrixiePhos t=35 min
1.5 mM TrixiePhos t=40 min
1.5 mM TrixiePhos t=45 min
1.5 mM TrixiePhos t=50 min
1.5 mM TrixiePhos t=55 min
1.5 mM TrixiePhos t=60 min
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
f1 (ppm)

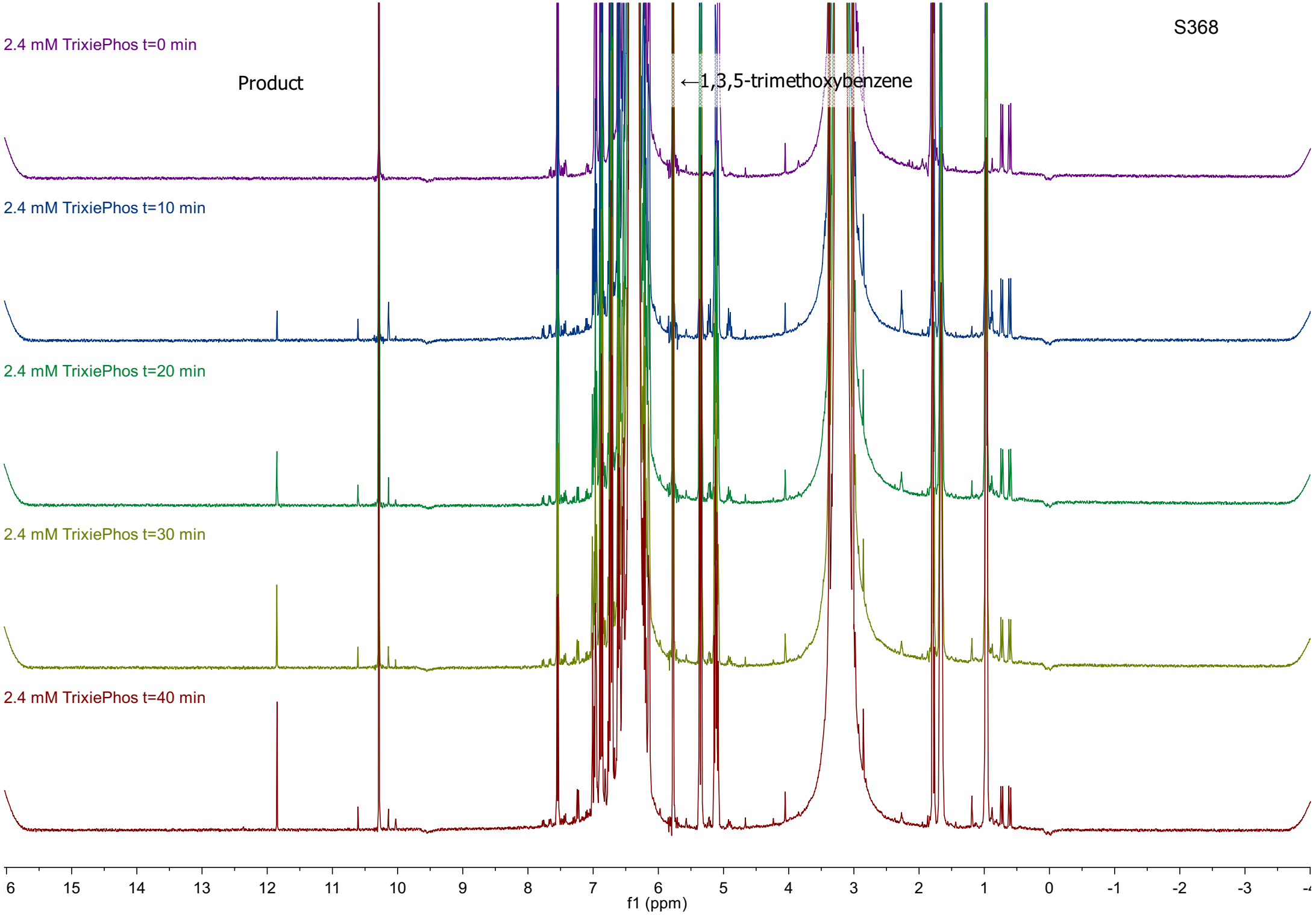

S363
1.5 mM TrixiePhos t=0 min
Product
←1,3,5-trimethoxybenzene
1.5 mM TrixiePhos t=5 min
1.5 mM TrixiePhos t=10 min
1.5 mM TrixiePhos t=15 min
1.5 mM TrixiePhos t=20 min
1.5 mM TrixiePhos t=25 min
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
-3
f1 (ppm)

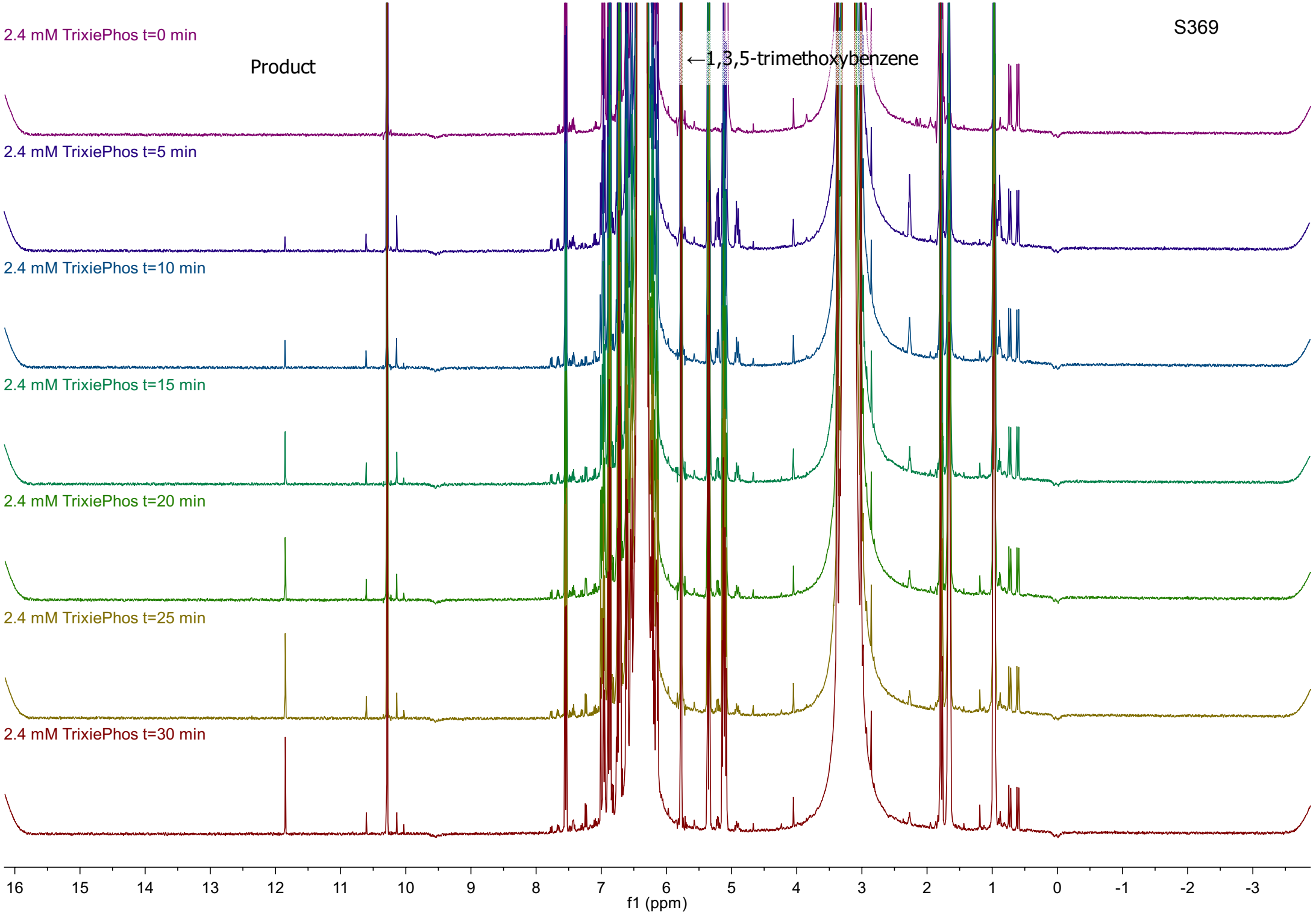

1.5 mM TrixiePhos t=30 min
Product
←1,3,5-trimethoxybenzene
S364
1.5 mM TrixiePhos t=35 min
1.5 mM TrixiePhos t=40 min
1.5 mM TrixiePhos t=45 min
1.5 mM TrixiePhos t=50 min
1.5 mM TrixiePhos t=55 min
1.5 mM TrixiePhos t=60 min
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
-3
f1 (ppm)

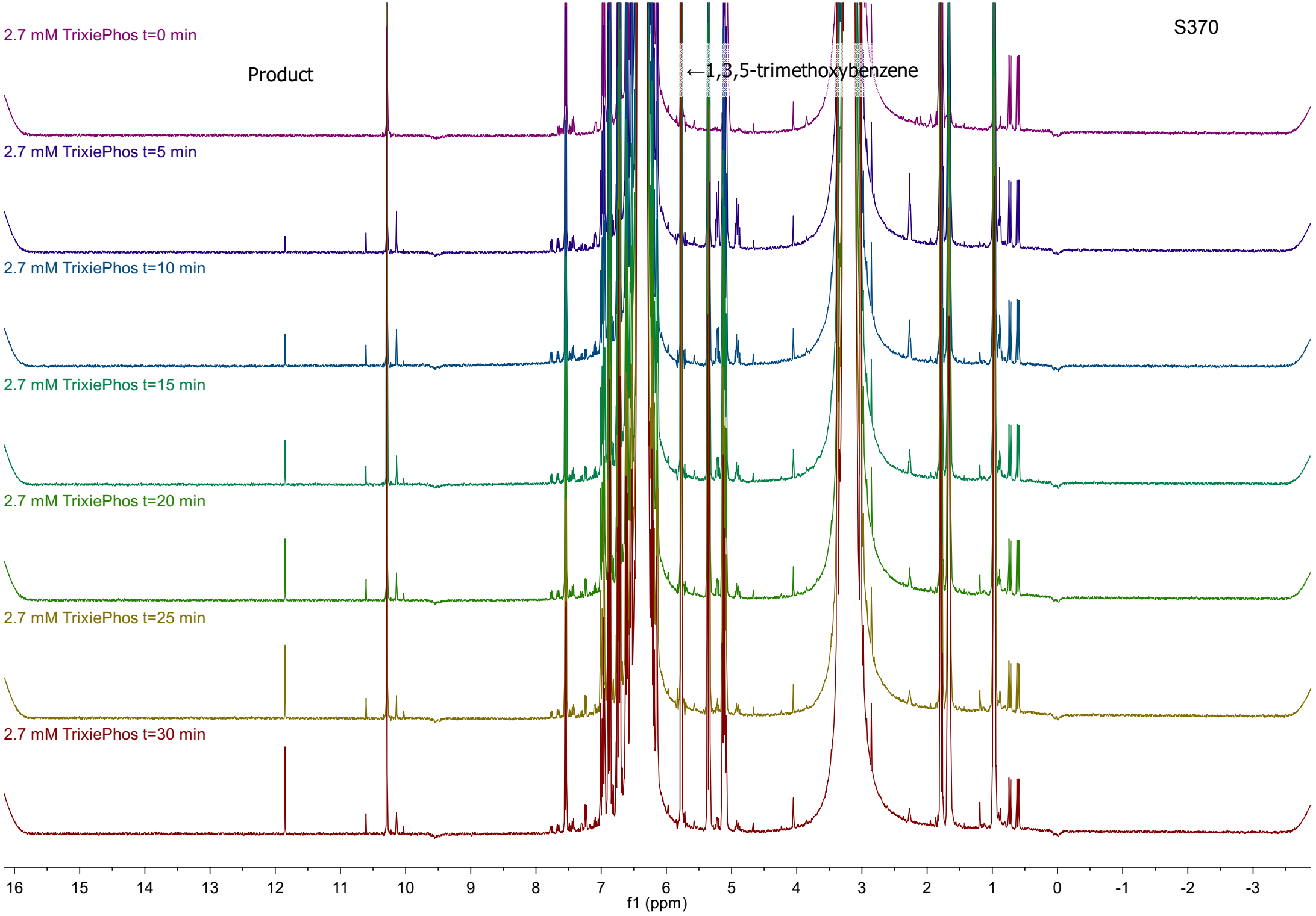

S365
1.8 mM TrixiePhos t=0 min
Product
←1,3,5-trimethoxybenzene
1.8 mM TrixiePhos t=5 min
1.8 mM TrixiePhos t=10 min
1.8 mM TrixiePhos t=15 min
1.8 mM TrixiePhos t=20 min
1.8 mM TrixiePhos t=25 min
1.8 mM TrixiePhos t=30 min
6 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3 -4
f1 (ppm)

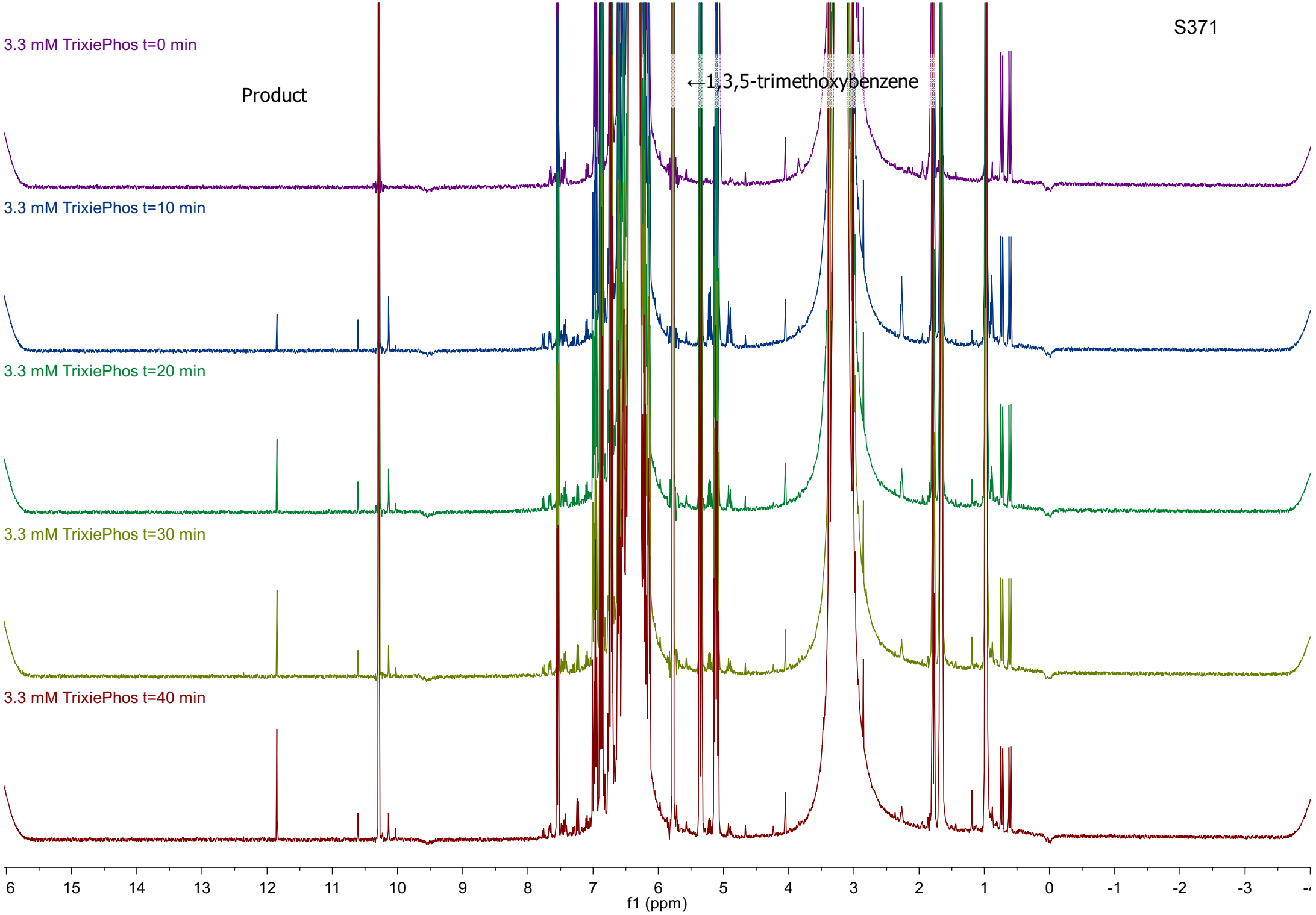

S366
1.8 mM TrixiePhos t=0 min
Product
←1,3,5-trimethoxybenzene
1.8 mM TrixiePhos t=5 min
1.8 mM TrixiePhos t=10 min
1.8 mM TrixiePhos t=15 min
1.8 mM TrixiePhos t=20 min
1.8 mM TrixiePhos t=25 min
6 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3 -4
f1 (ppm)

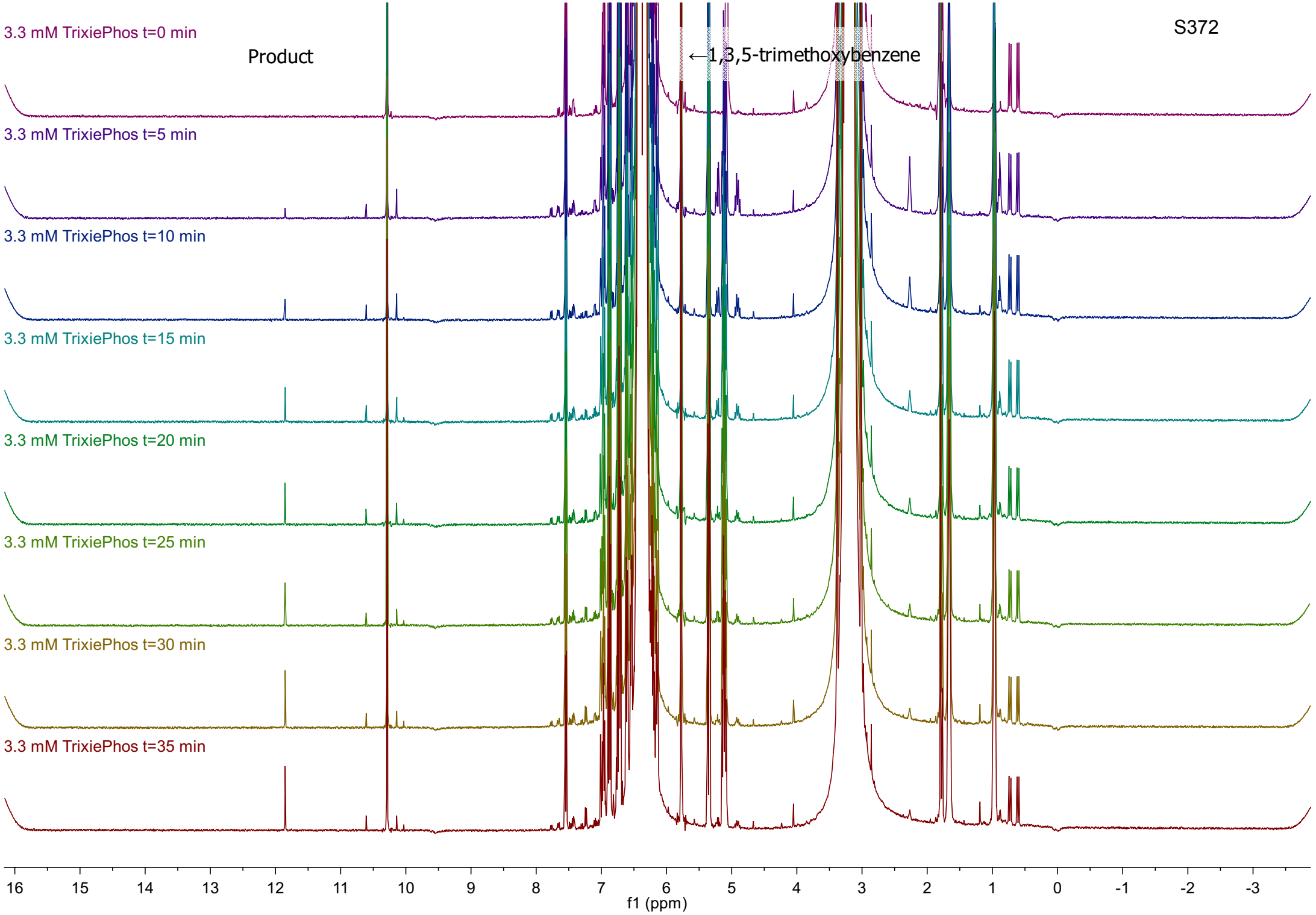

S367
2.1 mM TrixiePhos t=0 min
Product
←1,3,5-trimethoxybenzene
2.1 mM TrixiePhos t=5 min
2.1 mM TrixiePhos t=10 min
2.1 mM TrixiePhos t=15 min
2.1 mM TrixiePhos t=20 min
2.1 mM TrixiePhos t=25 min
2.1 mM TrixiePhos t=30 min
6 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
f1 (ppm)

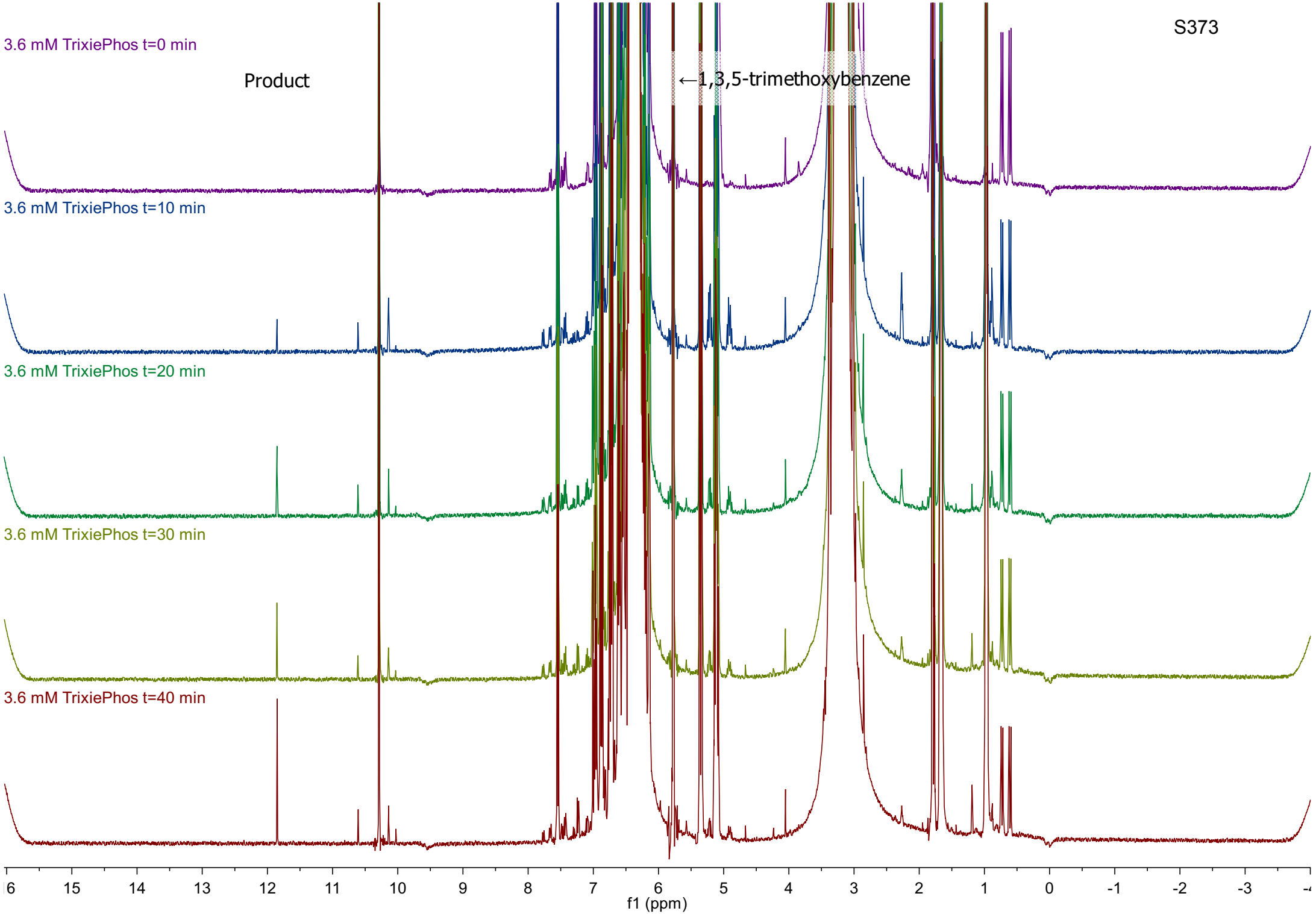

S368
2.4 mM TrixiePhos t=0 min
Product
←1,3,5-trimethoxybenzene
2.4 mM TrixiePhos t=10 min
2.4 mM TrixiePhos t=20 min
2.4 mM TrixiePhos t=30 min
2.4 mM TrixiePhos t=40 min
6 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
f1 (ppm)

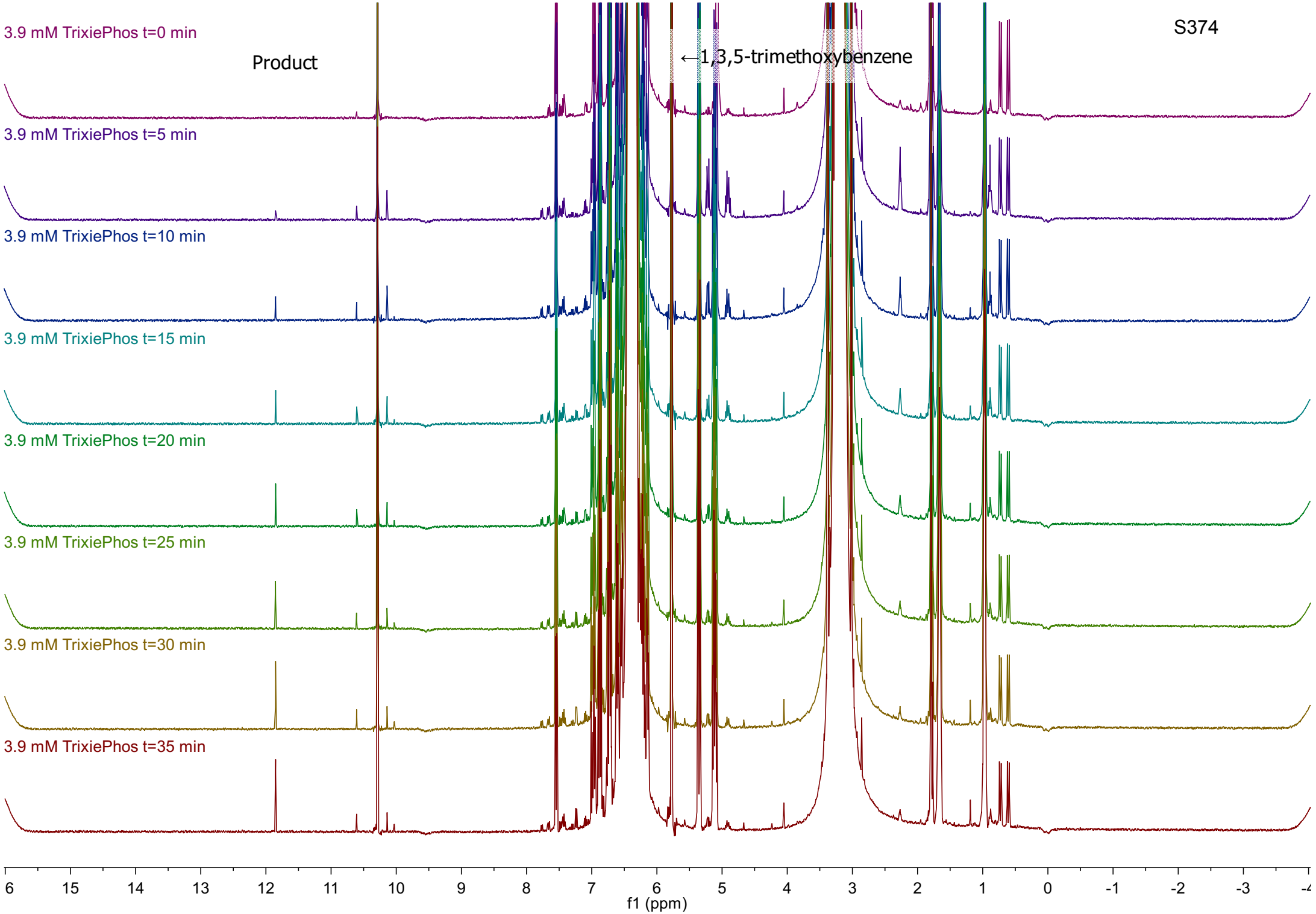

2.4 mM TrixiePhos t=0 min
Product
←1,3,5-trimethoxybenzene
S369
2.4 mM TrixiePhos t=5 min
2.4 mM TrixiePhos t=10 min
2.4 mM TrixiePhos t=15 min
2.4 mM TrixiePhos t=20 min
2.4 mM TrixiePhos t=25 min
2.4 mM TrixiePhos t=30 min
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
f1 (ppm)

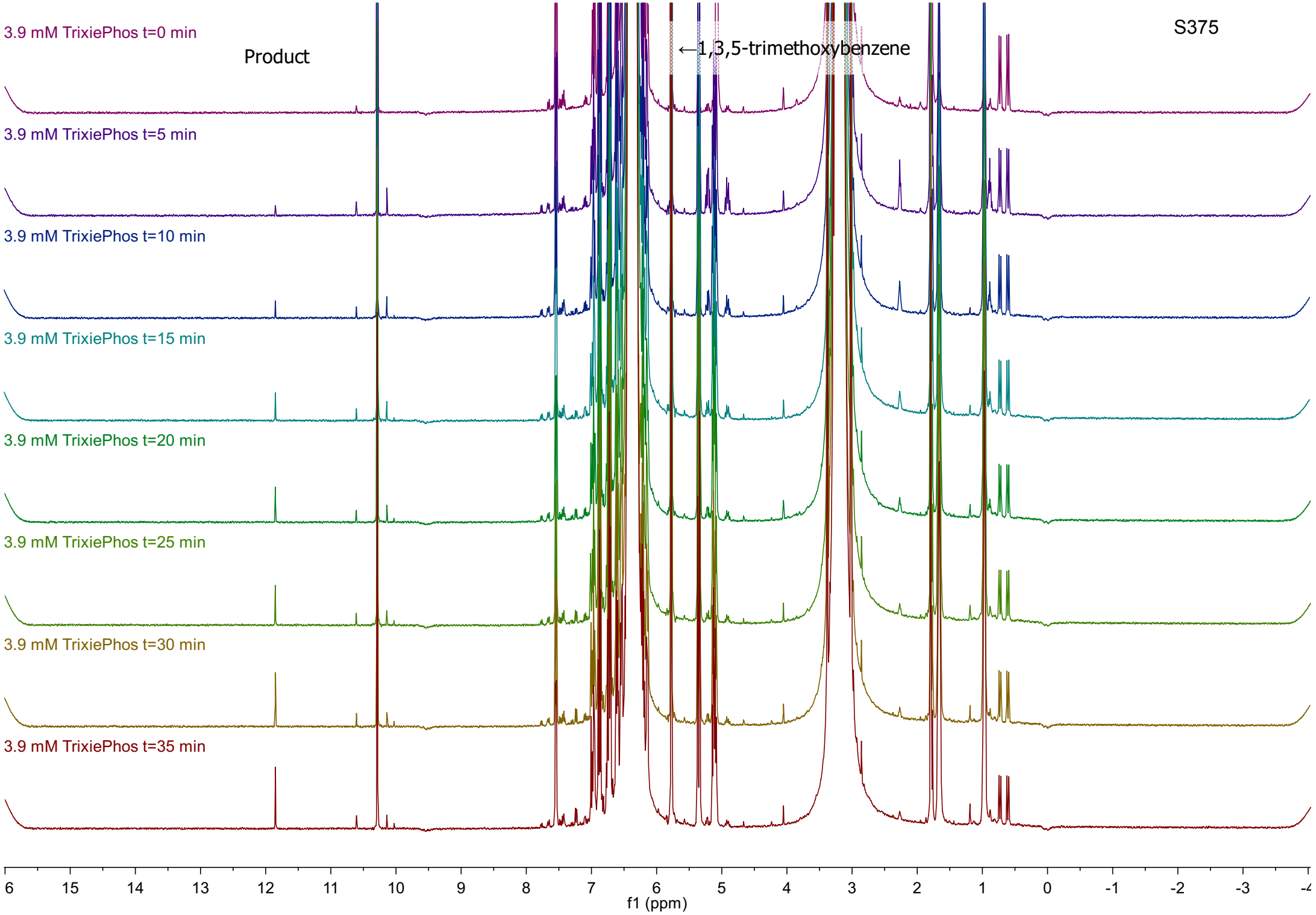

S370
2.7 mM TrixiePhos t=0 min
Product
←1,3,5-trimethoxybenzene
2.7 mM TrixiePhos t=5 min
2.7 mM TrixiePhos t=10 min
2.7 mM TrixiePhos t=15 min
2.7 mM TrixiePhos t=20 min
2.7 mM TrixiePhos t=25 min
2.7 mM TrixiePhos t=30 min
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
f1 (ppm)

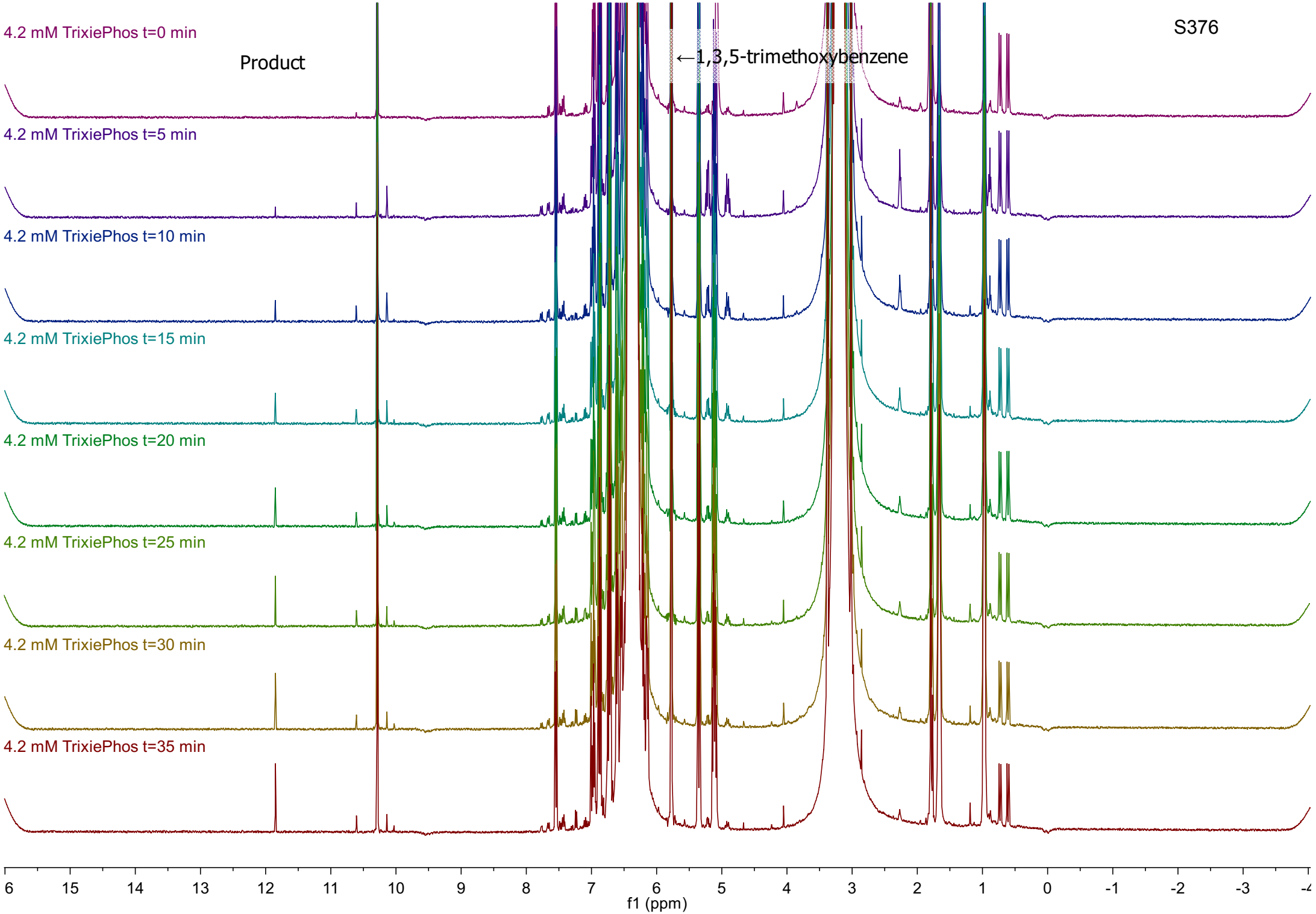

S371
3.3 mM TrixiePhos t=0 min
Product
←1,3,5-trimethoxybenzene
3.3 mM TrixiePhos t=10 min
3.3 mM TrixiePhos t=20 min
3.3 mM TrixiePhos t=30 min
3.3 mM TrixiePhos t=40 min
6 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
f1 (ppm)

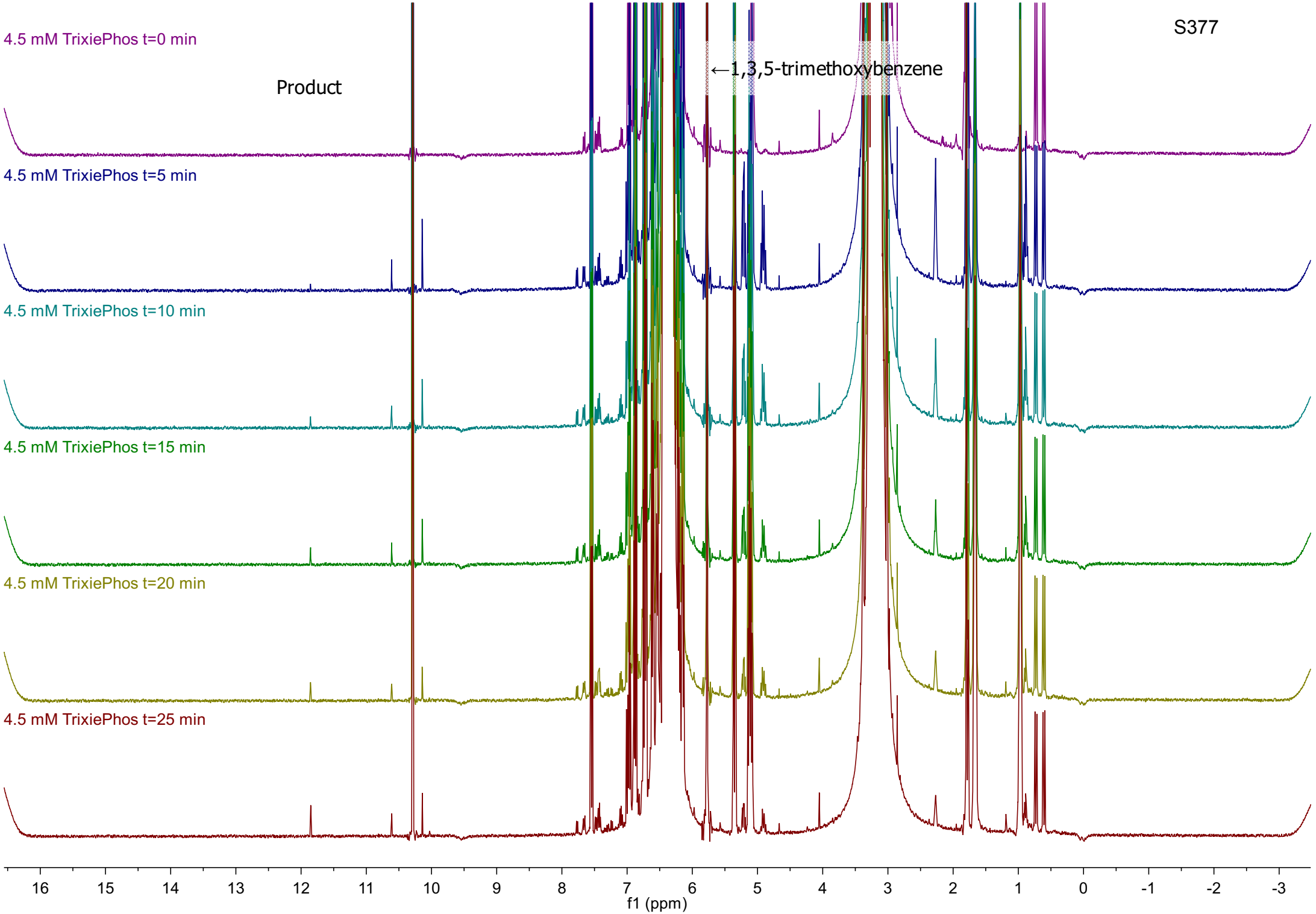

3.3 mM TrixiePhos t=0 min
Product
←1,3,5-trimethoxybenzene
S372
3.3 mM TrixiePhos t=5 min
3.3 mM TrixiePhos t=10 min
3.3 mM TrixiePhos t=15 min
3.3 mM TrixiePhos t=20 min
3.3 mM TrixiePhos t=25 min
3.3 mM TrixiePhos t=30 min
3.3 mM TrixiePhos t=35 min
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
f1 (ppm)

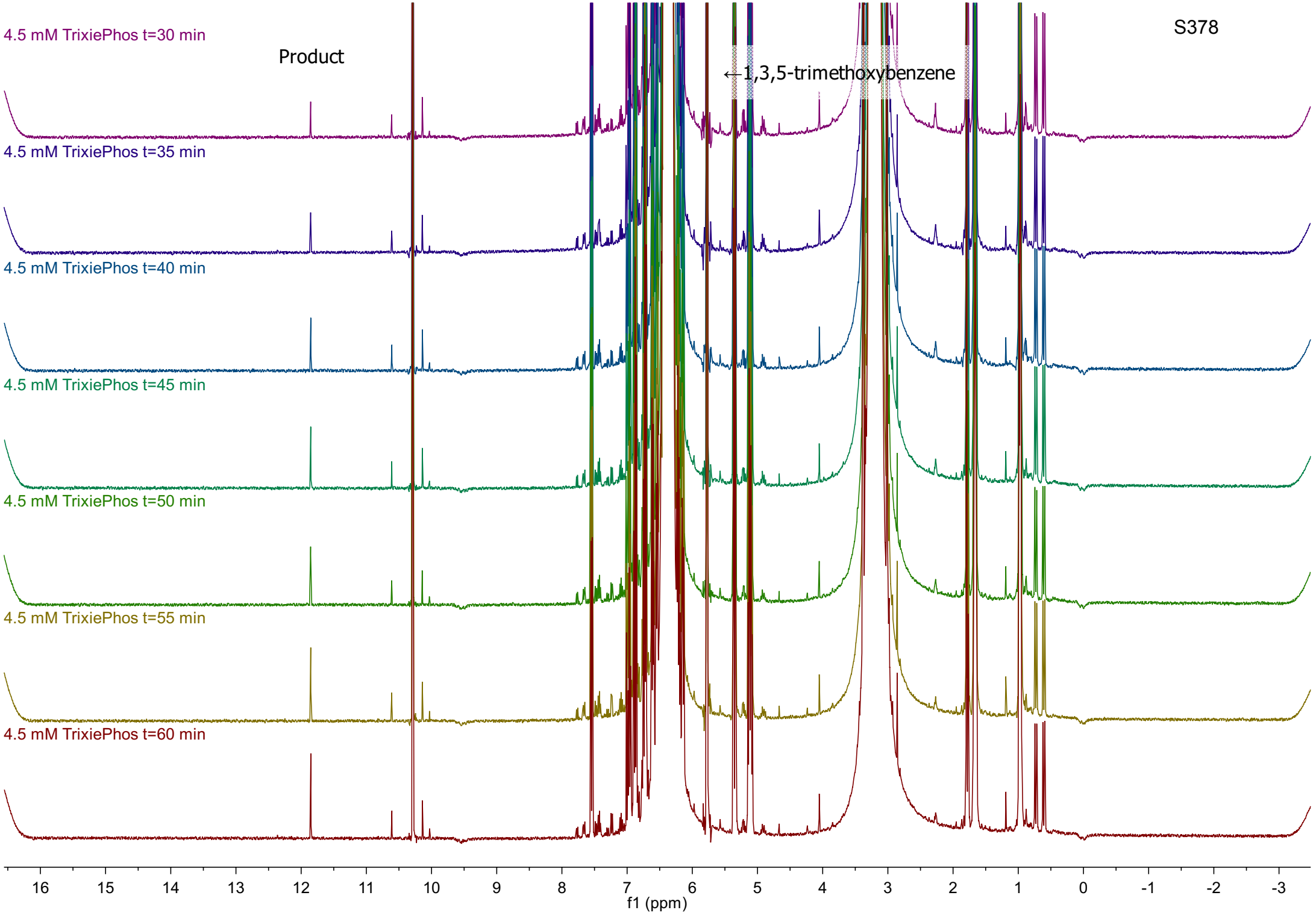

S373
3.6 mM TrixiePhos t=0 min
Product
←1,3,5-trimethoxybenzene
3.6 mM TrixiePhos t=10 min
3.6 mM TrixiePhos t=20 min
3.6 mM TrixiePhos t=30 min
3.6 mM TrixiePhos t=40 min
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
f1 (ppm)

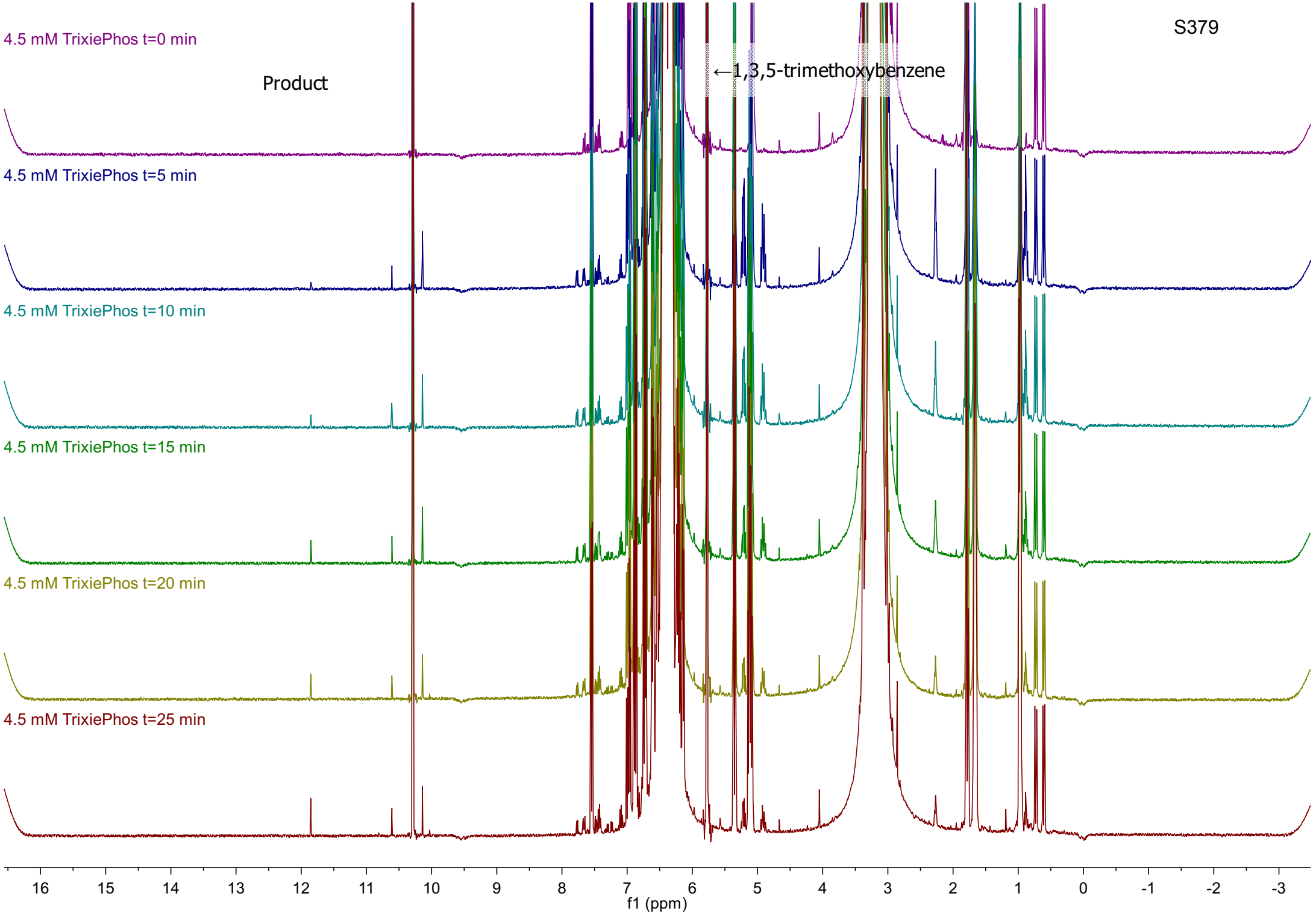

3.9 mM TrixiePhos t=0 min
Product
←1,3,5-trimethoxybenzene
S374
3.9 mM TrixiePhos t=5 min
3.9 mM TrixiePhos t=10 min
3.9 mM TrixiePhos t=15 min
3.9 mM TrixiePhos t=20 min
3.9 mM TrixiePhos t=25 min
3.9 mM TrixiePhos t=30 min
3.9 mM TrixiePhos t=35 min
6 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3 -4
f1 (ppm)

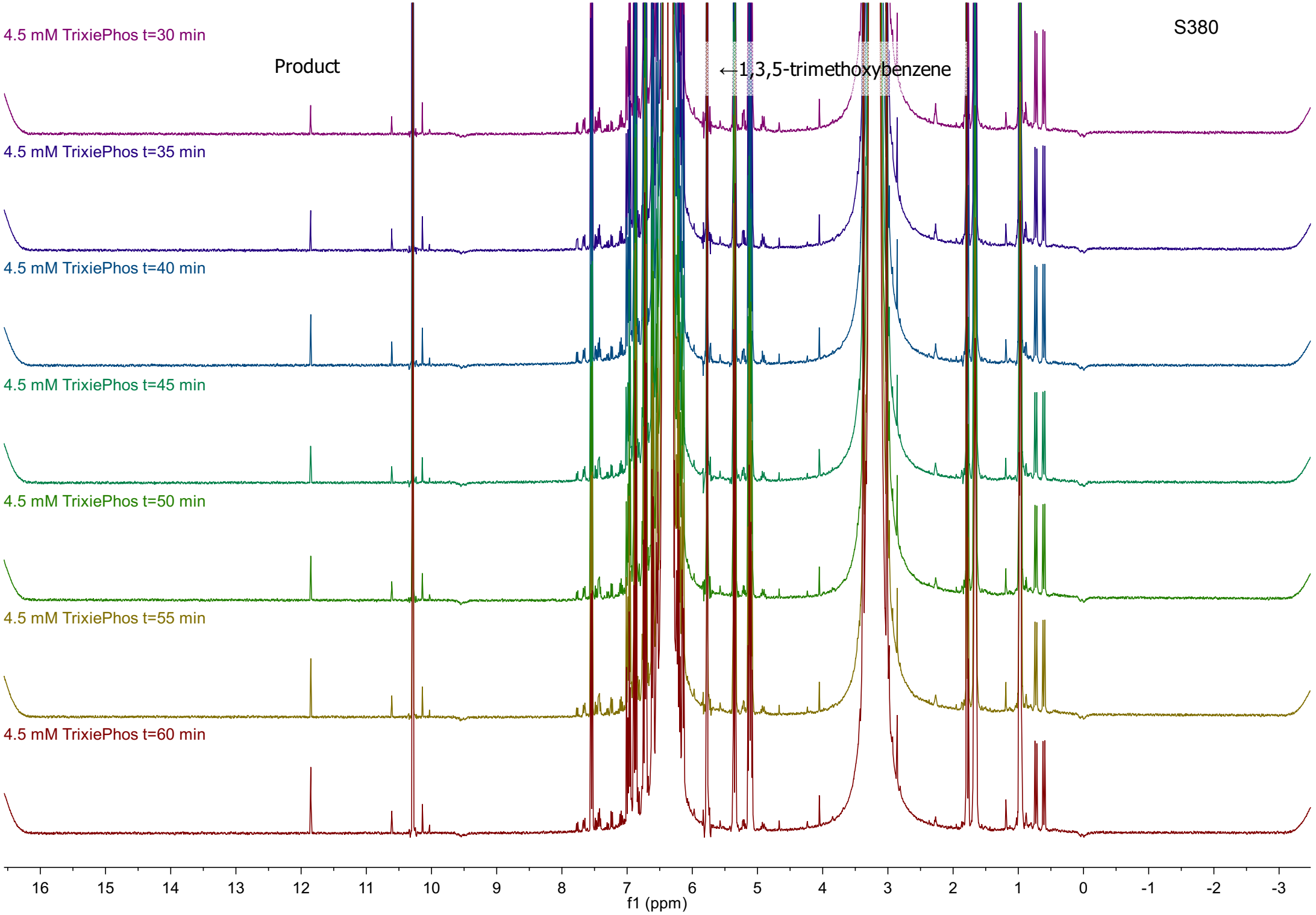

3.9 mM TrixiePhos t=0 min
Product
←1,3,5-trimethoxybenzene
S375
3.9 mM TrixiePhos t=5 min
3.9 mM TrixiePhos t=10 min
3.9 mM TrixiePhos t=15 min
3.9 mM TrixiePhos t=20 min
3.9 mM TrixiePhos t=25 min
3.9 mM TrixiePhos t=30 min
3.9 mM TrixiePhos t=35 min
6 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3 -4
f1 (ppm)

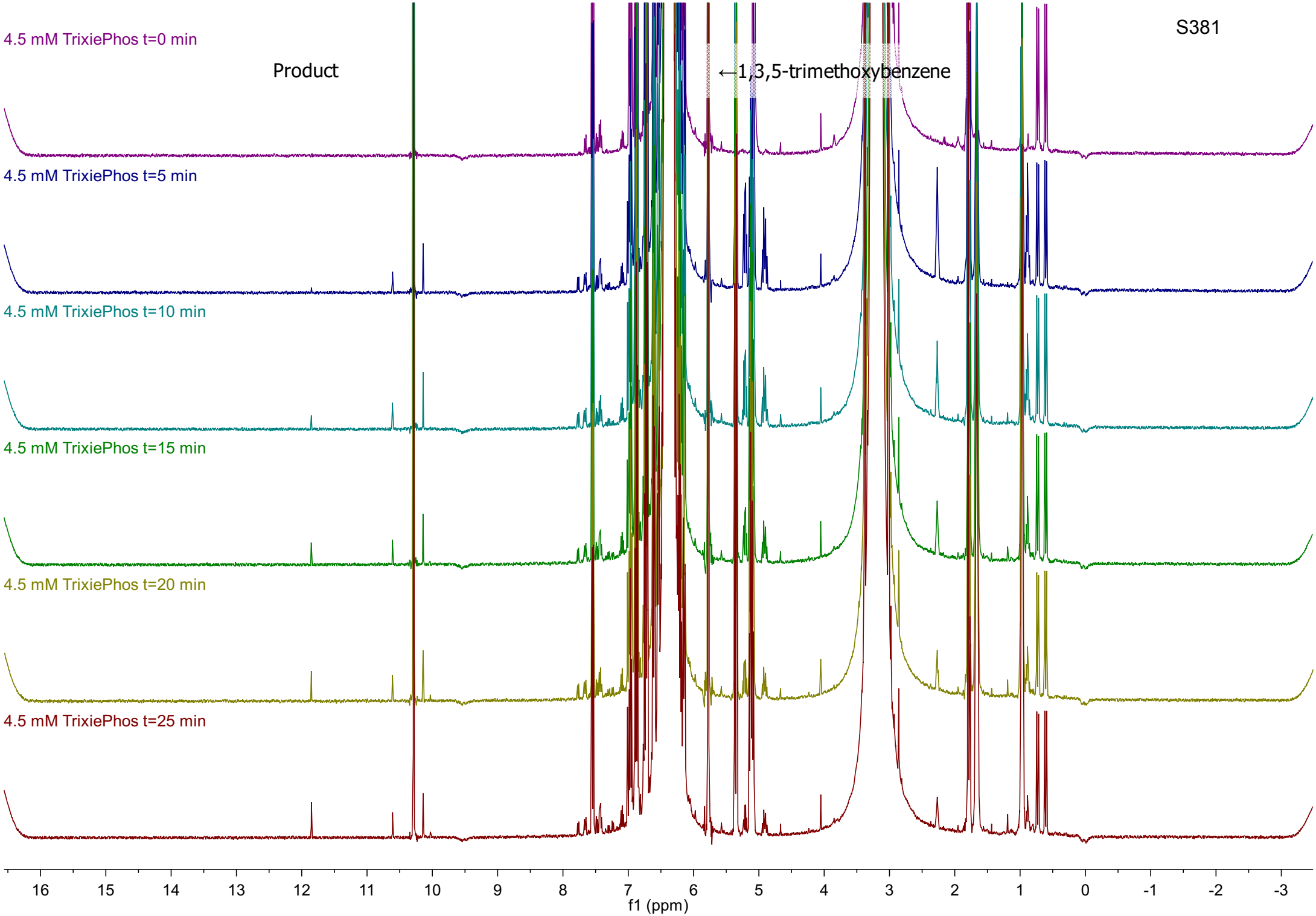

4.2 mM TrixiePhos t=0 min
Product
←1,3,5-trimethoxybenzene
S376
4.2 mM TrixiePhos t=5 min
4.2 mM TrixiePhos t=10 min
4.2 mM TrixiePhos t=15 min
4.2 mM TrixiePhos t=20 min
4.2 mM TrixiePhos t=25 min
4.2 mM TrixiePhos t=30 min
4.2 mM TrixiePhos t=35 min
6 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3 -4
f1 (ppm)

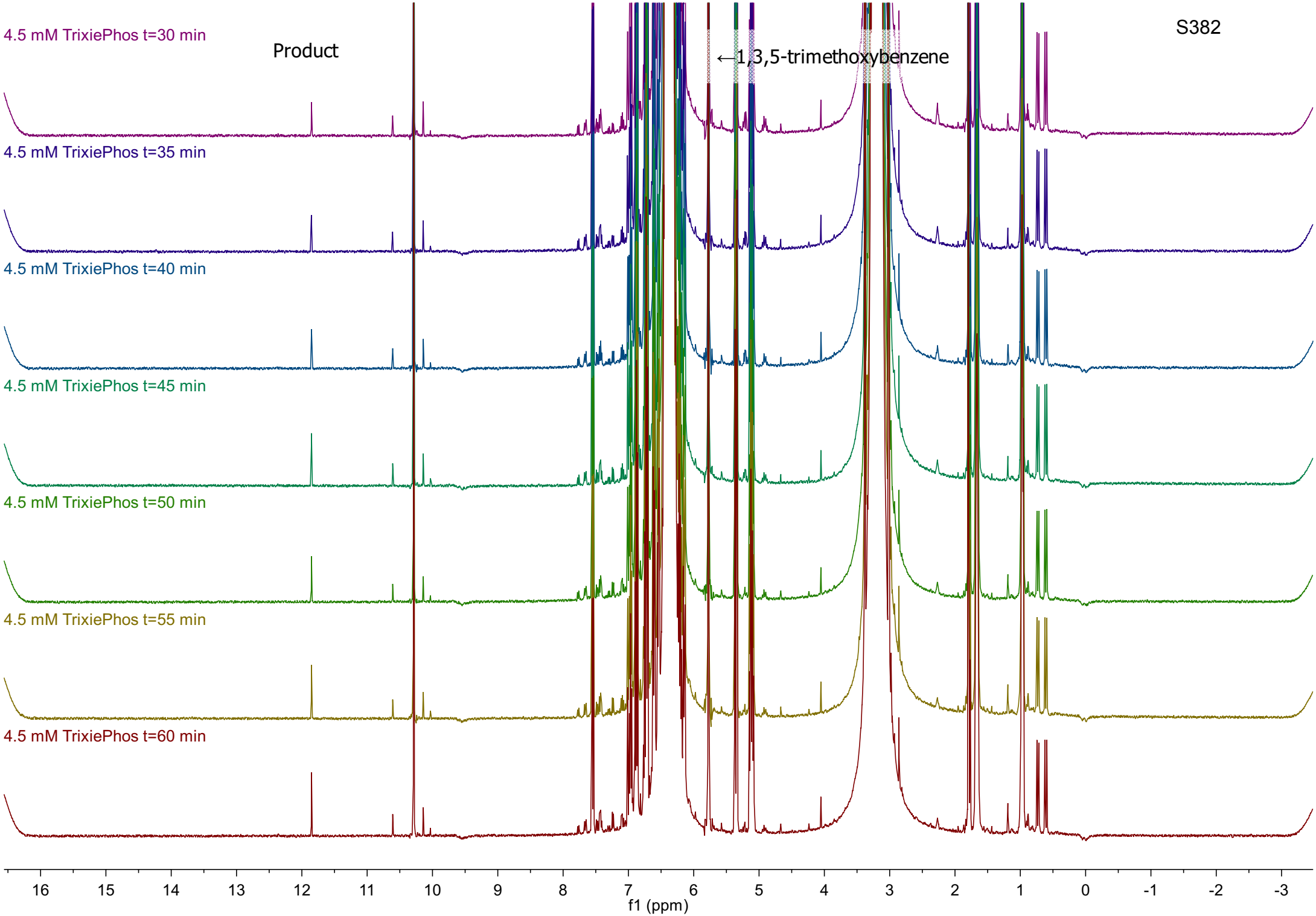

S377
4.5 mM TrixiePhos t=0 min
Product
←1,3,5-trimethoxybenzene
4.5 mM TrixiePhos t=5 min
4.5 mM TrixiePhos t=10 min
4.5 mM TrixiePhos t=15 min
4.5 mM TrixiePhos t=20 min
4.5 mM TrixiePhos t=25 min
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
f1 (ppm)

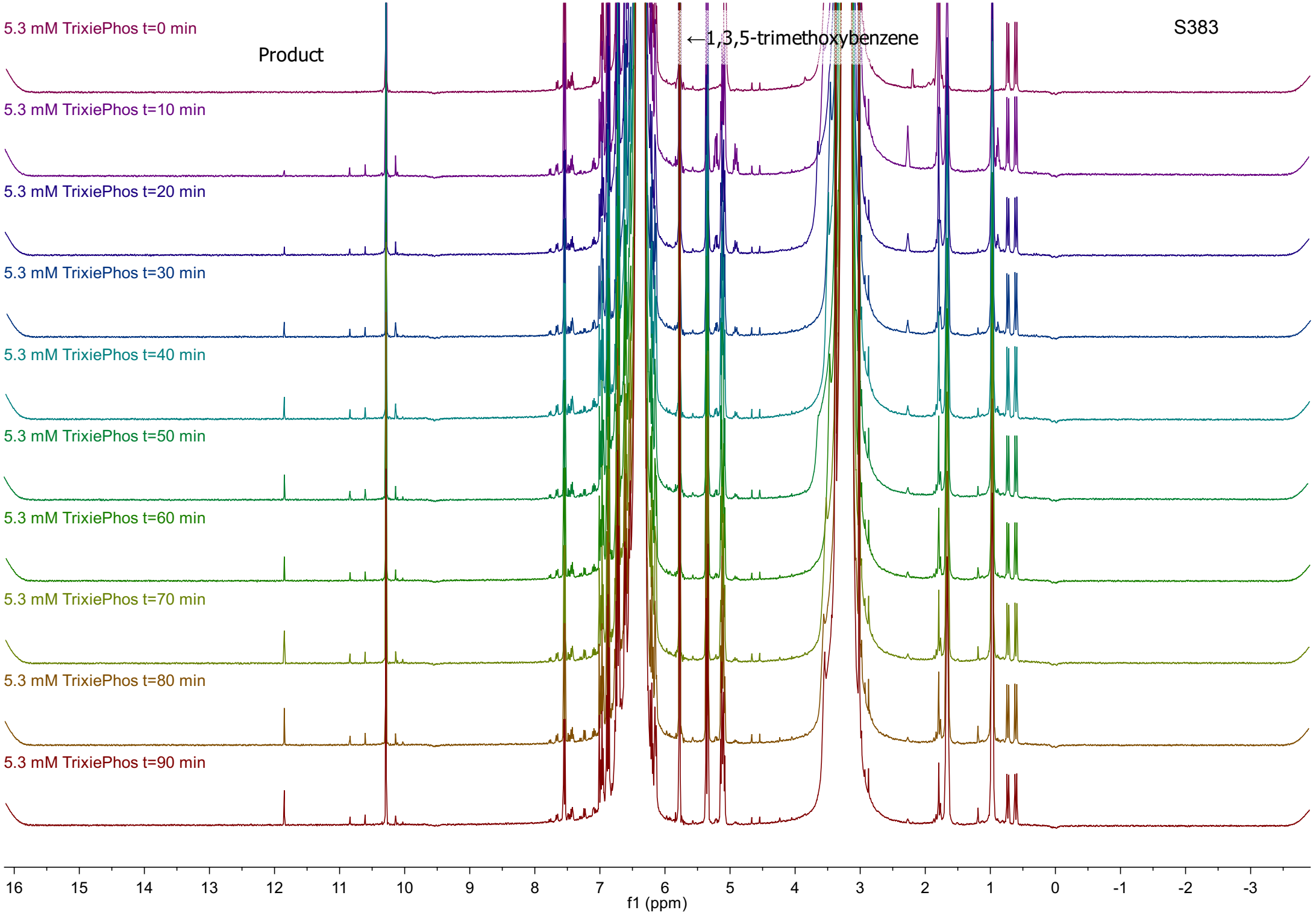

4.5 mM TrixiePhos t=30 min
Product
←1,3,5-trimethoxybenzene
S378
4.5 mM TrixiePhos t=35 min
4.5 mM TrixiePhos t=40 min
4.5 mM TrixiePhos t=45 min
4.5 mM TrixiePhos t=50 min
4.5 mM TrixiePhos t=55 min
4.5 mM TrixiePhos t=60 min
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
f1 (ppm)

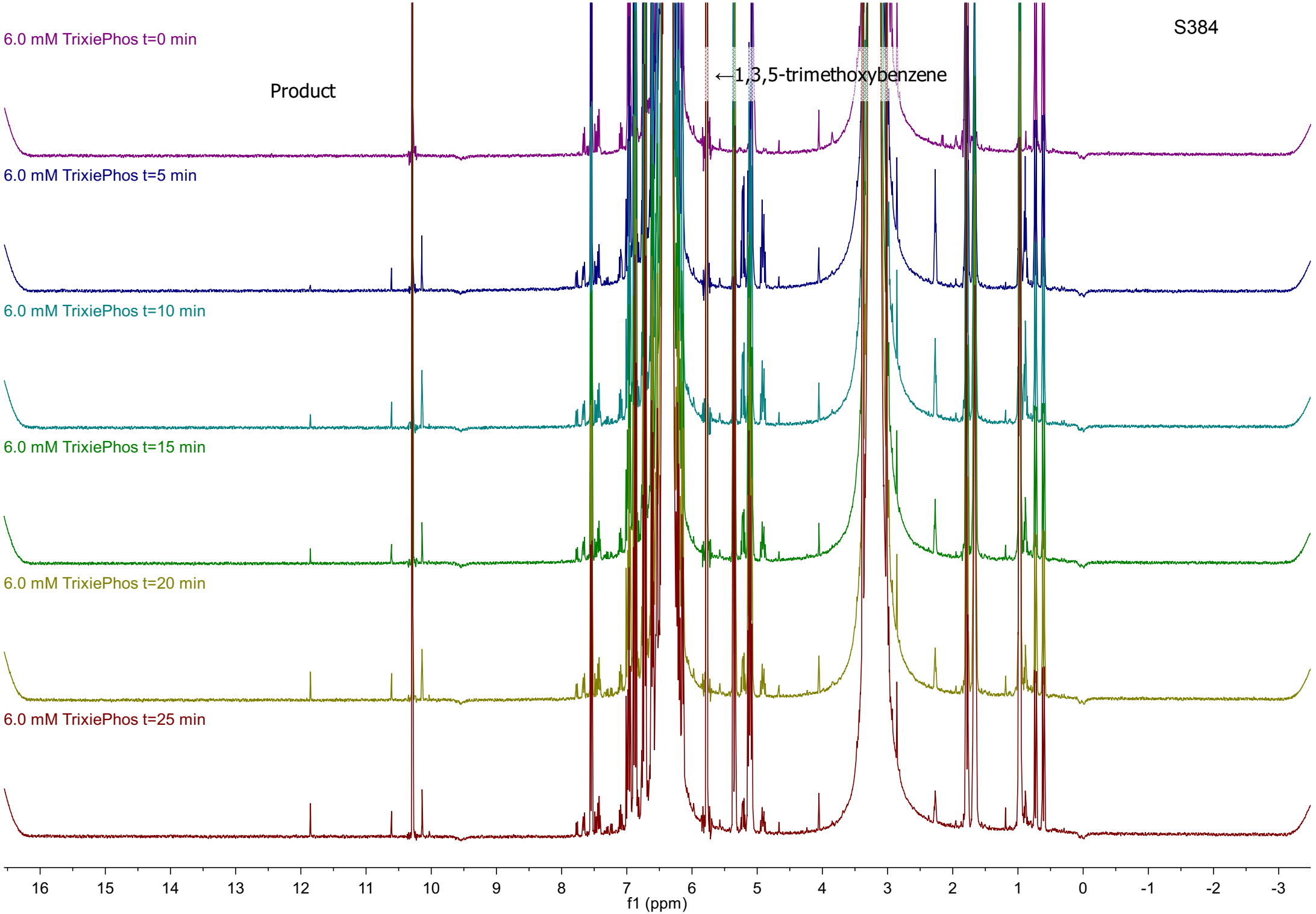

S379
4.5 mM TrixiePhos t=0 min
Product
←1,3,5-trimethoxybenzene
4.5 mM TrixiePhos t=5 min
4.5 mM TrixiePhos t=10 min
4.5 mM TrixiePhos t=15 min
4.5 mM TrixiePhos t=20 min
4.5 mM TrixiePhos t=25 min
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
f1 (ppm)

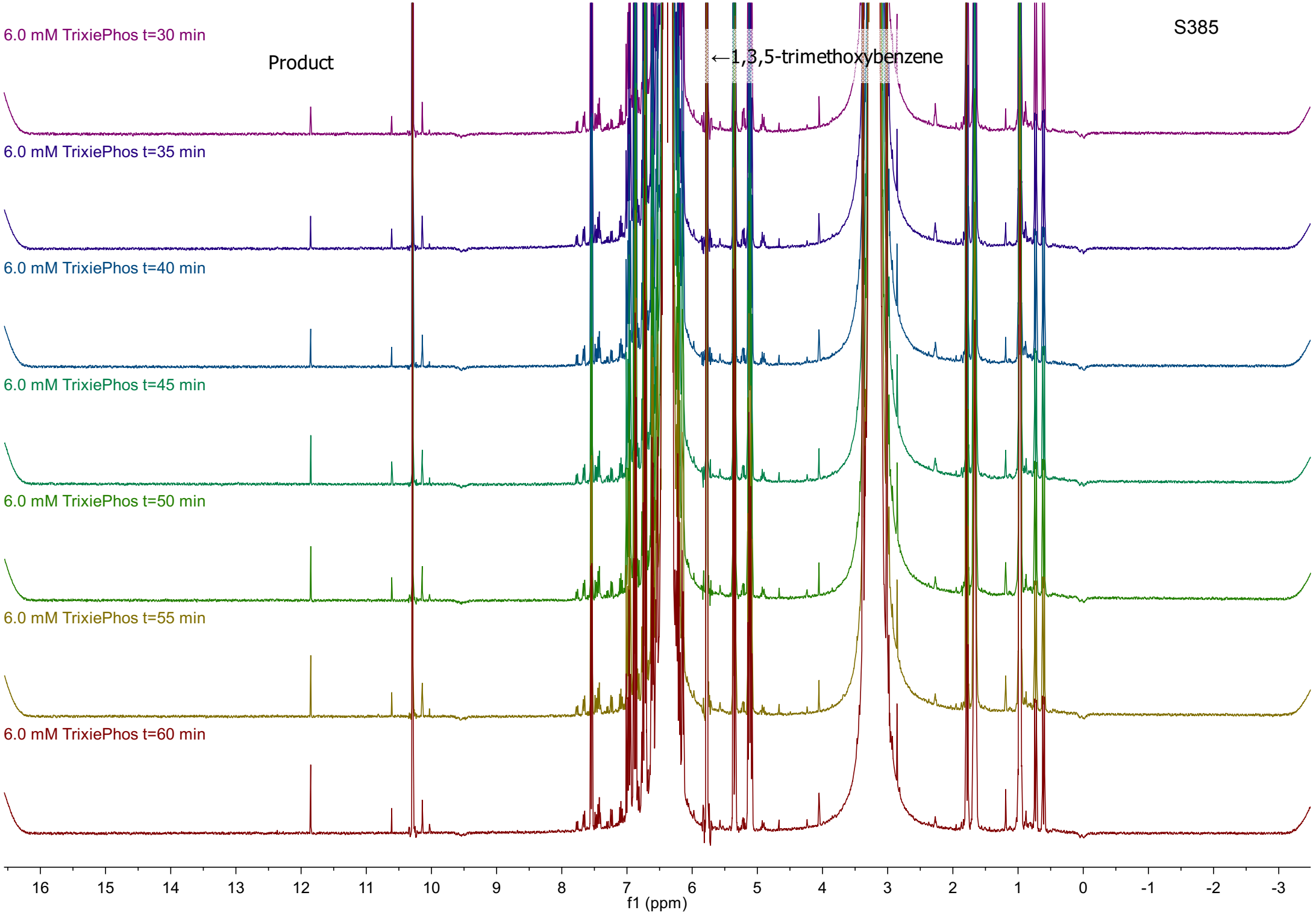

4.5 mM TrixiePhos t=30 min
Product
←1,3,5-trimethoxybenzene
S380
4.5 mM TrixiePhos t=35 min
4.5 mM TrixiePhos t=40 min
4.5 mM TrixiePhos t=45 min
4.5 mM TrixiePhos t=50 min
4.5 mM TrixiePhos t=55 min
4.5 mM TrixiePhos t=60 min
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
f1 (ppm)

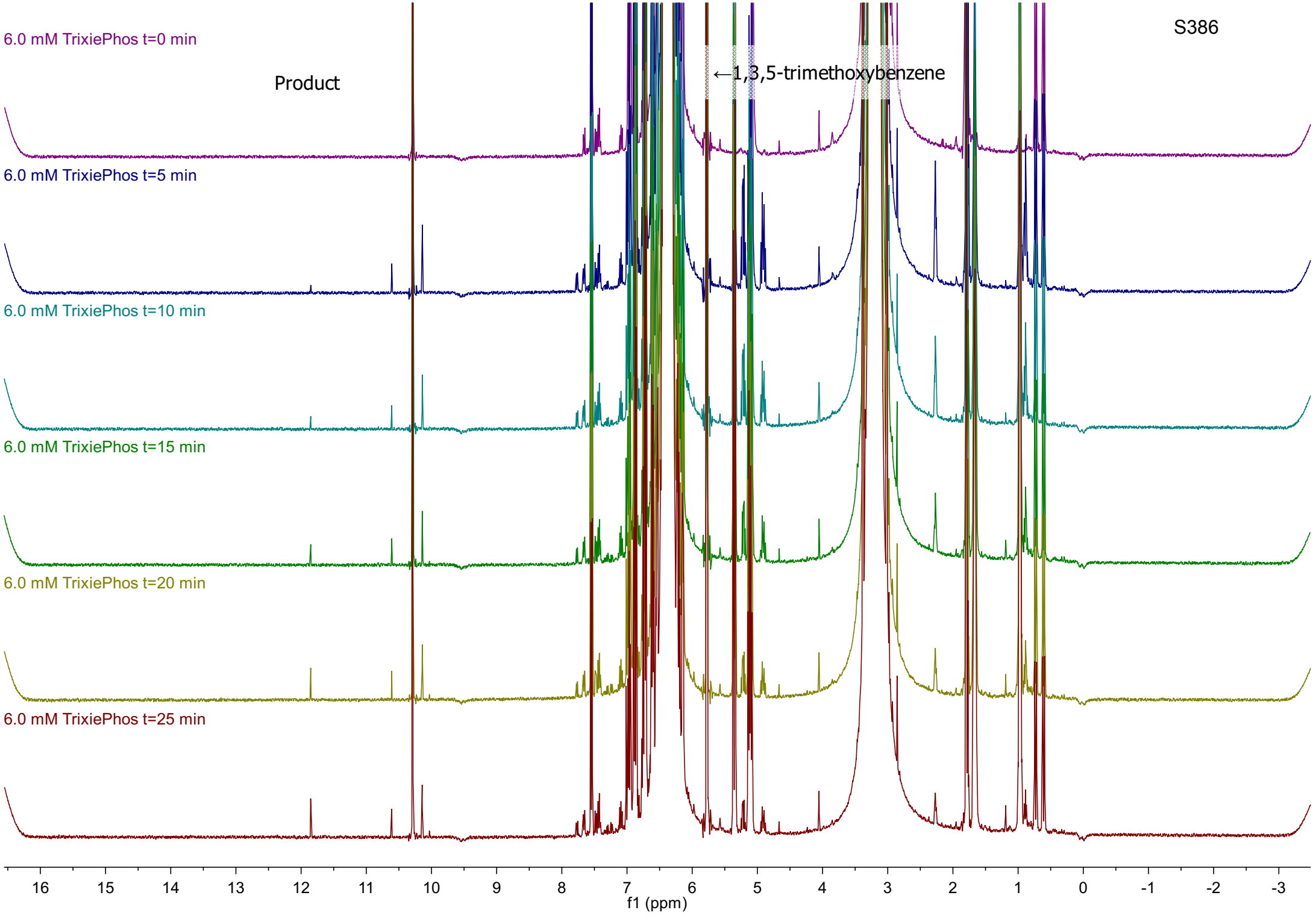

S381
4.5 mM TrixiePhos t=0 min
Product
←1,3,5-trimethoxybenzene
4.5 mM TrixiePhos t=5 min
4.5 mM TrixiePhos t=10 min
4.5 mM TrixiePhos t=15 min
4.5 mM TrixiePhos t=20 min
4.5 mM TrixiePhos t=25 min
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
f1 (ppm)

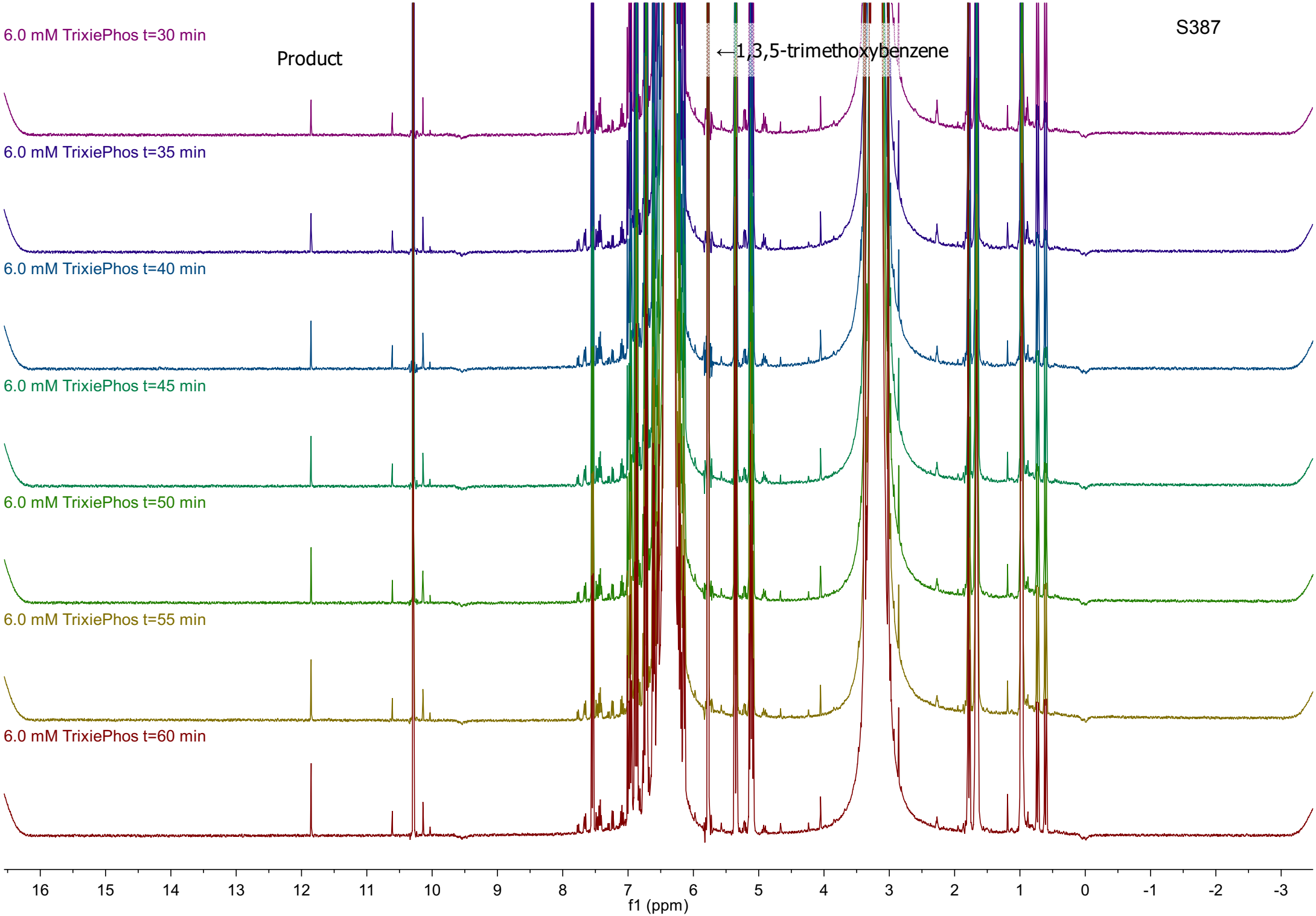

4.5 mM TrixiePhos t=30 min
Product
←1,3,5-trimethoxybenzene
S382
4.5 mM TrixiePhos t=35 min
4.5 mM TrixiePhos t=40 min
4.5 mM TrixiePhos t=45 min
4.5 mM TrixiePhos t=50 min
4.5 mM TrixiePhos t=55 min
4.5 mM TrixiePhos t=60 min
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
f1 (ppm)

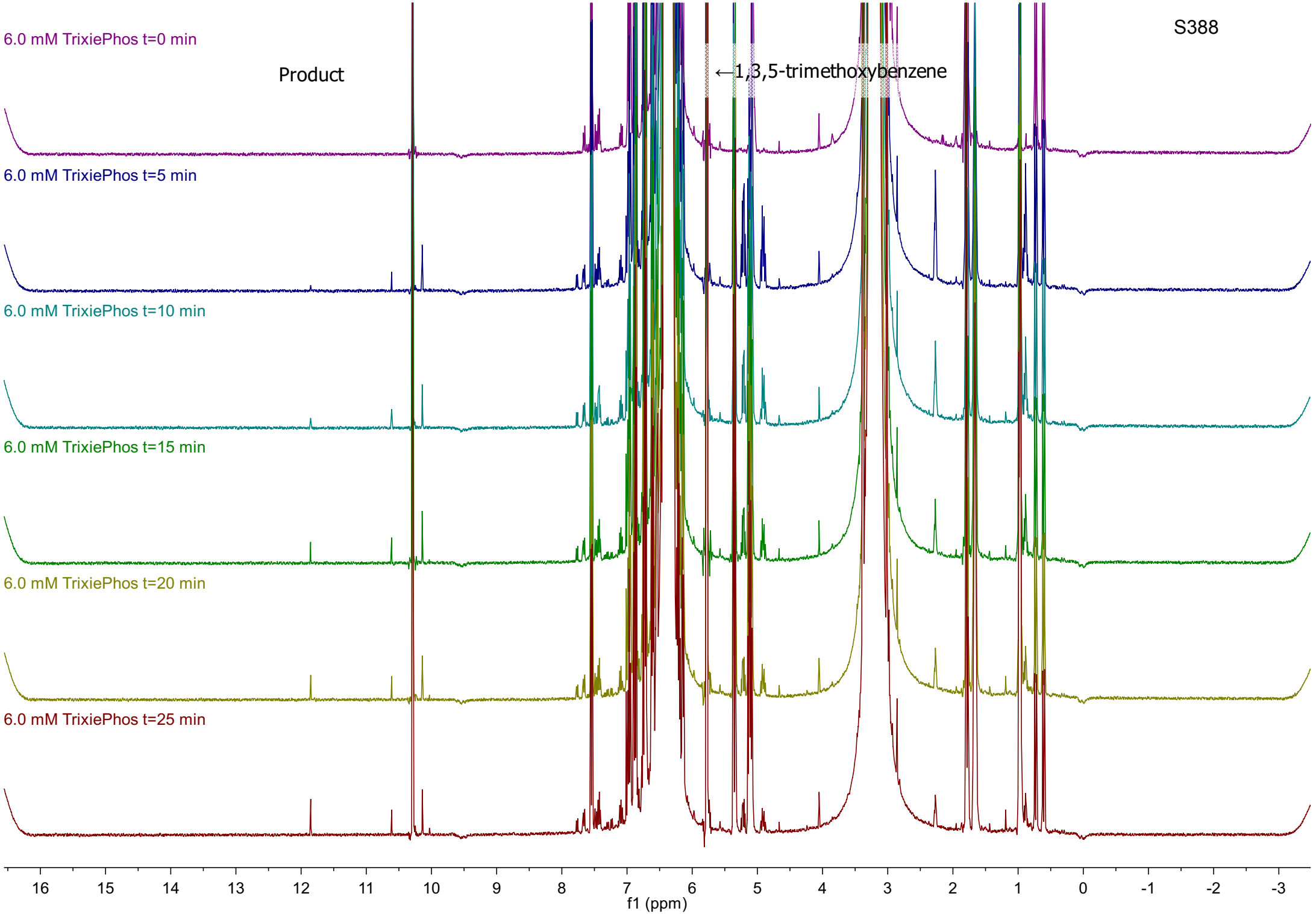

5.3 mM TrixiePhos t=0 min
Product
←1,3,5-trimethoxybenzene
S383
5.3 mM TrixiePhos t=10 min
5.3 mM TrixiePhos t=20 min
5.3 mM TrixiePhos t=30 min
5.3 mM TrixiePhos t=40 min
5.3 mM TrixiePhos t=50 min
5.3 mM TrixiePhos t=60 min
5.3 mM TrixiePhos t=70 min
5.3 mM TrixiePhos t=80 min
5.3 mM TrixiePhos t=90 min
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
f1 (ppm)

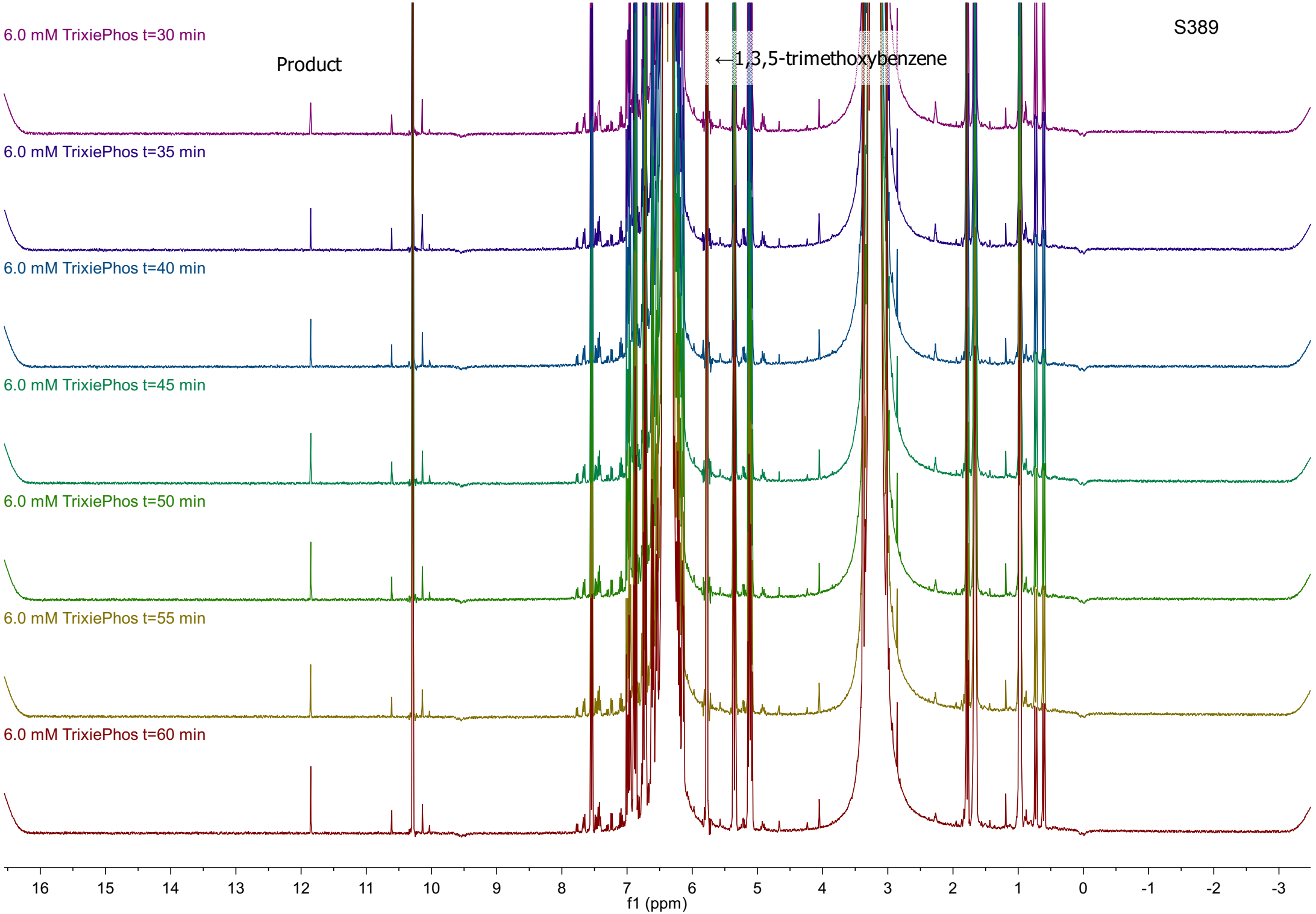

6.0 mM TrixiePhos t=0 min
Product
←1,3,5-trimethoxybenzene
S384
6.0 mM TrixiePhos t=5 min
6.0 mM TrixiePhos t=10 min
6.0 mM TrixiePhos t=15 min
6.0 mM TrixiePhos t=20 min
6.0 mM TrixiePhos t=25 min
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
f1 (ppm)

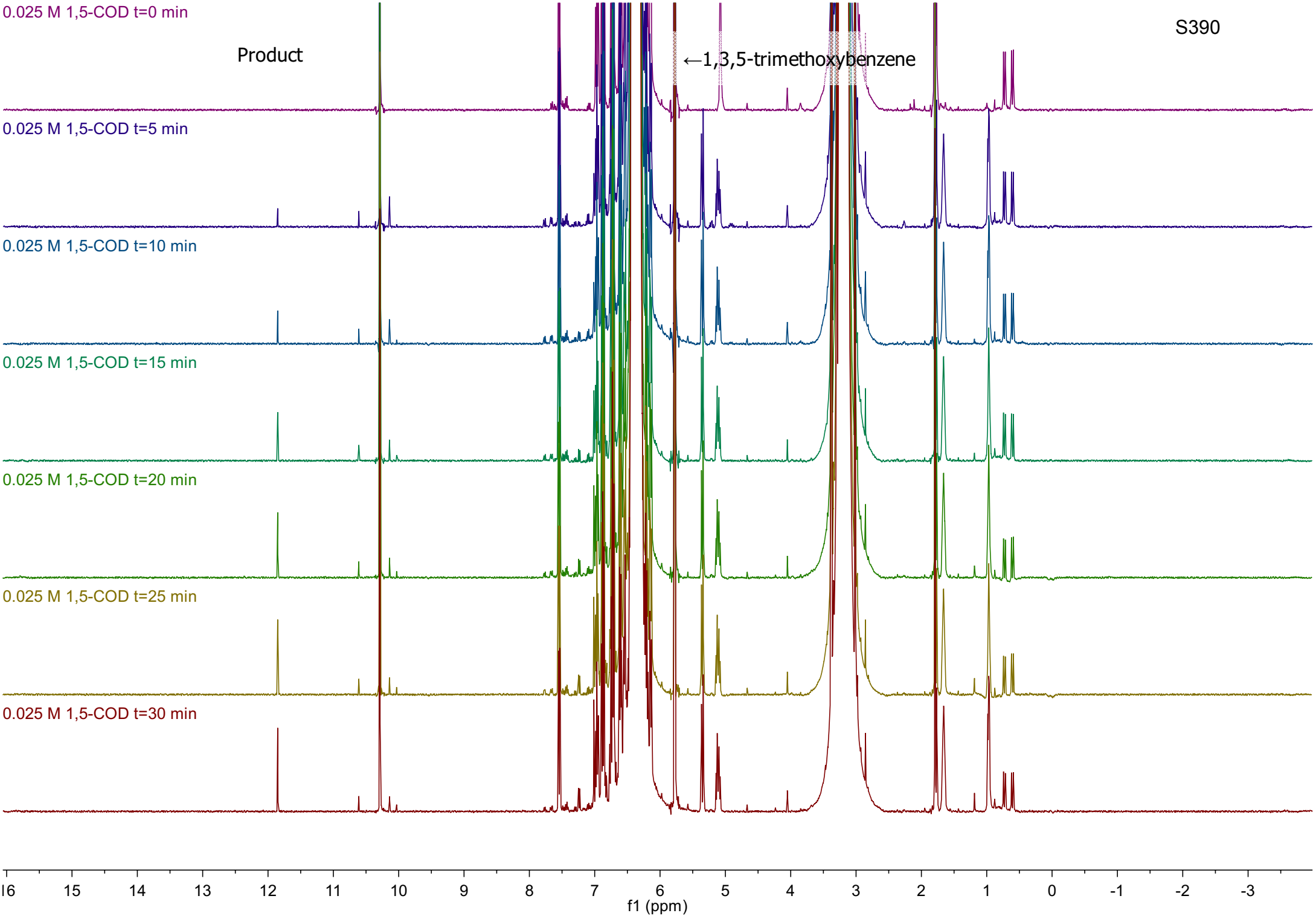

6.0 mM TrixiePhos t=30 min
Product
←1,3,5-trimethoxybenzene
S385
6.0 mM TrixiePhos t=35 min
6.0 mM TrixiePhos t=40 min
6.0 mM TrixiePhos t=45 min
6.0 mM TrixiePhos t=50 min
6.0 mM TrixiePhos t=55 min
6.0 mM TrixiePhos t=60 min
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
f1 (ppm)

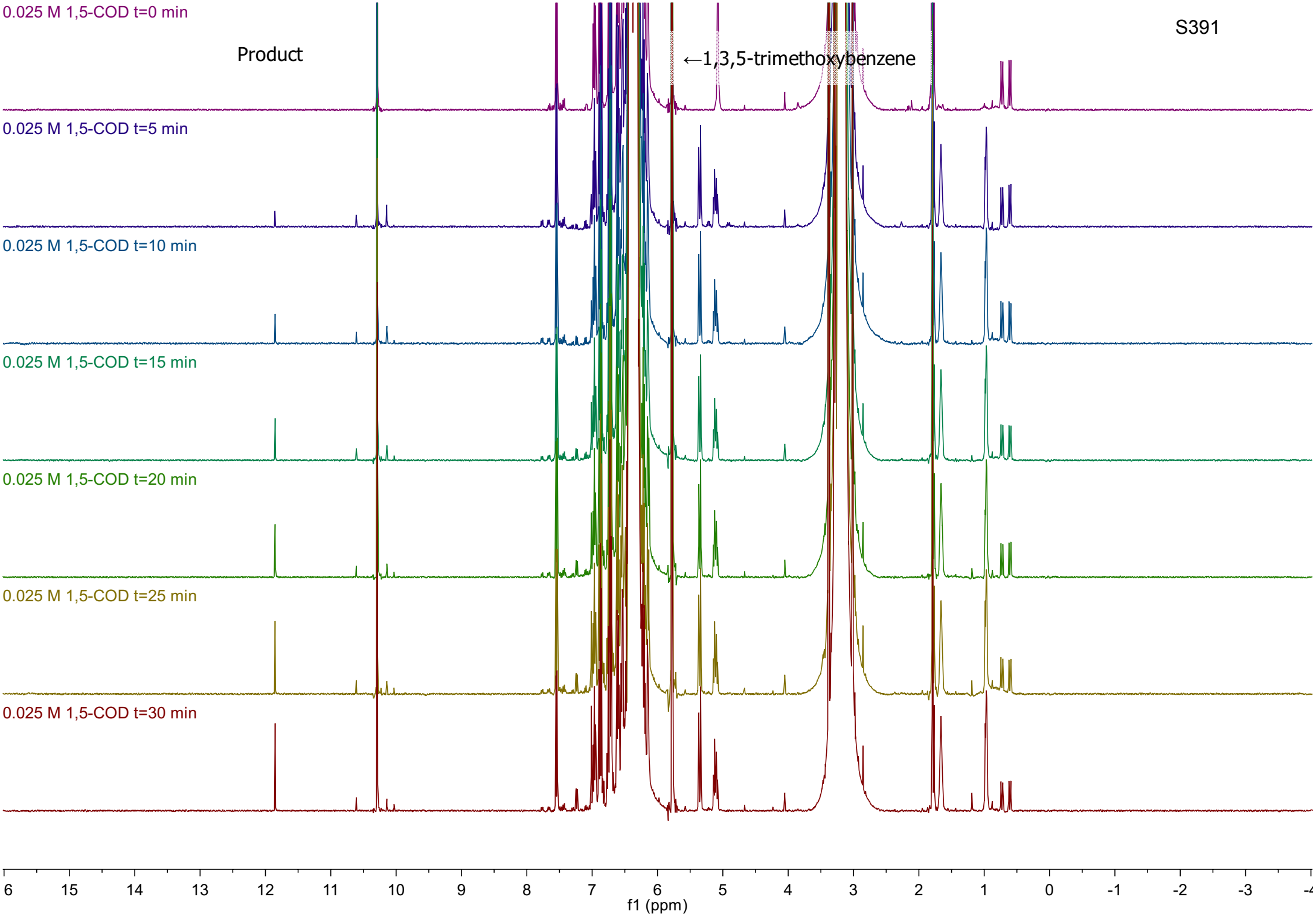

S386
6.0 mM TrixiePhos t=0 min
Product
←1,3,5-trimethoxybenzene
6.0 mM TrixiePhos t=5 min
6.0 mM TrixiePhos t=10 min
6.0 mM TrixiePhos t=15 min
6.0 mM TrixiePhos t=20 min
6.0 mM TrixiePhos t=25 min
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
f1 (ppm)

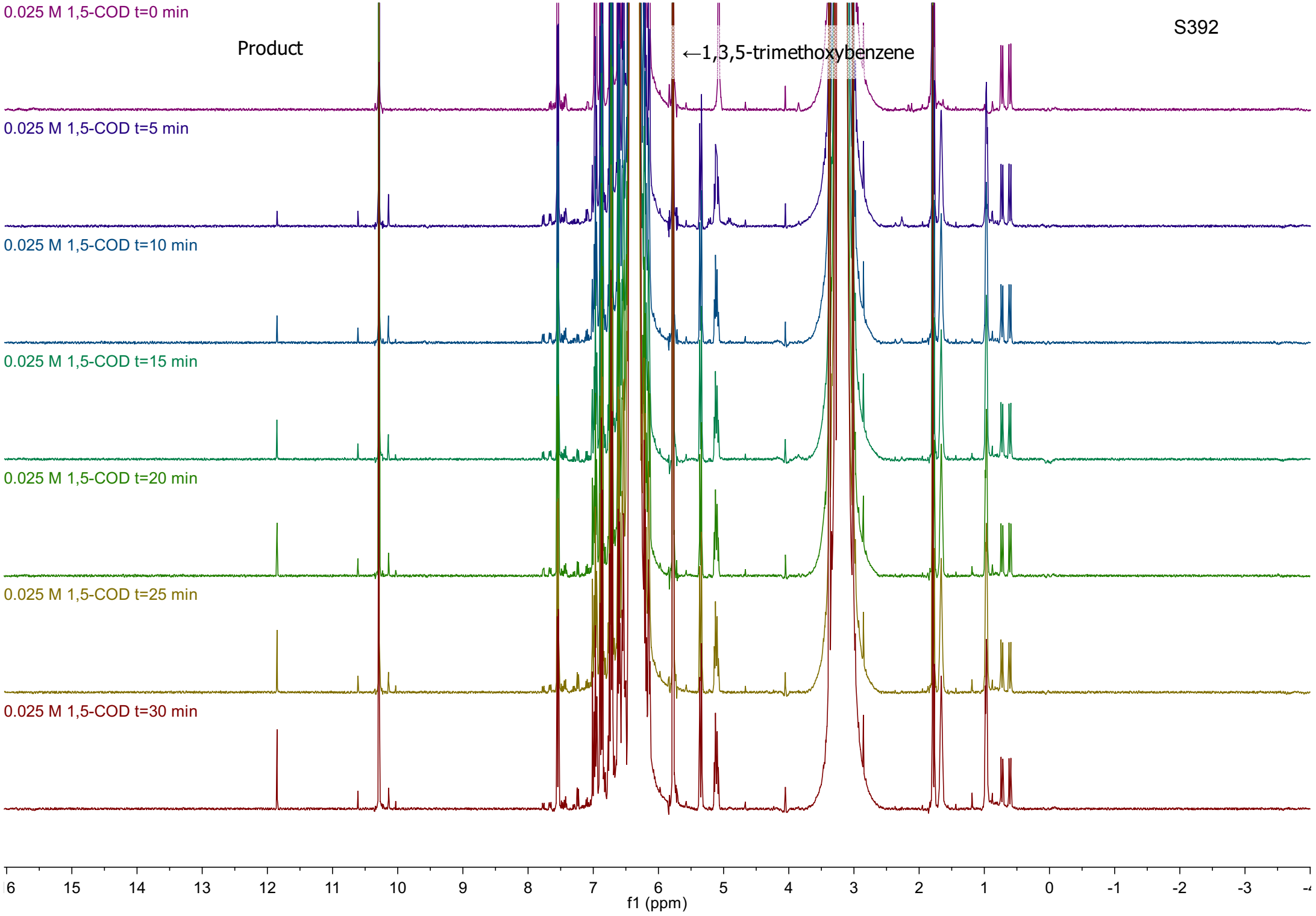

6.0 mM TrixiePhos t=30 min
Product
←1,3,5-trimethoxybenzene
S387
6.0 mM TrixiePhos t=35 min
6.0 mM TrixiePhos t=40 min
6.0 mM TrixiePhos t=45 min
6.0 mM TrixiePhos t=50 min
6.0 mM TrixiePhos t=55 min
6.0 mM TrixiePhos t=60 min
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
-3
f1 (ppm)

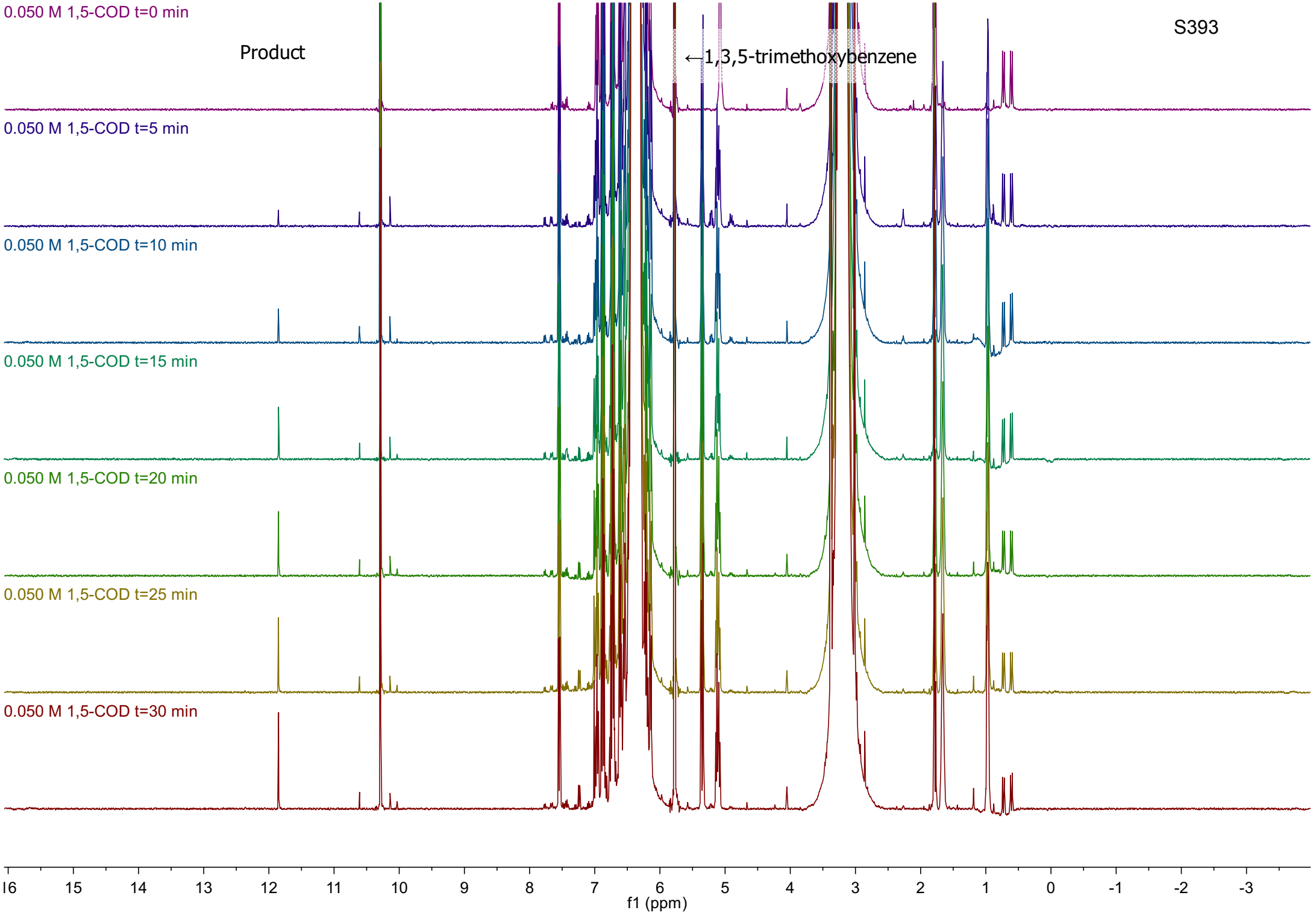

6.0 mM TrixiePhos t=0 min
Product
←1,3,5-trimethoxybenzene
S388
6.0 mM TrixiePhos t=5 min
6.0 mM TrixiePhos t=10 min
6.0 mM TrixiePhos t=15 min
6.0 mM TrixiePhos t=20 min
6.0 mM TrixiePhos t=25 min
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
f1 (ppm)

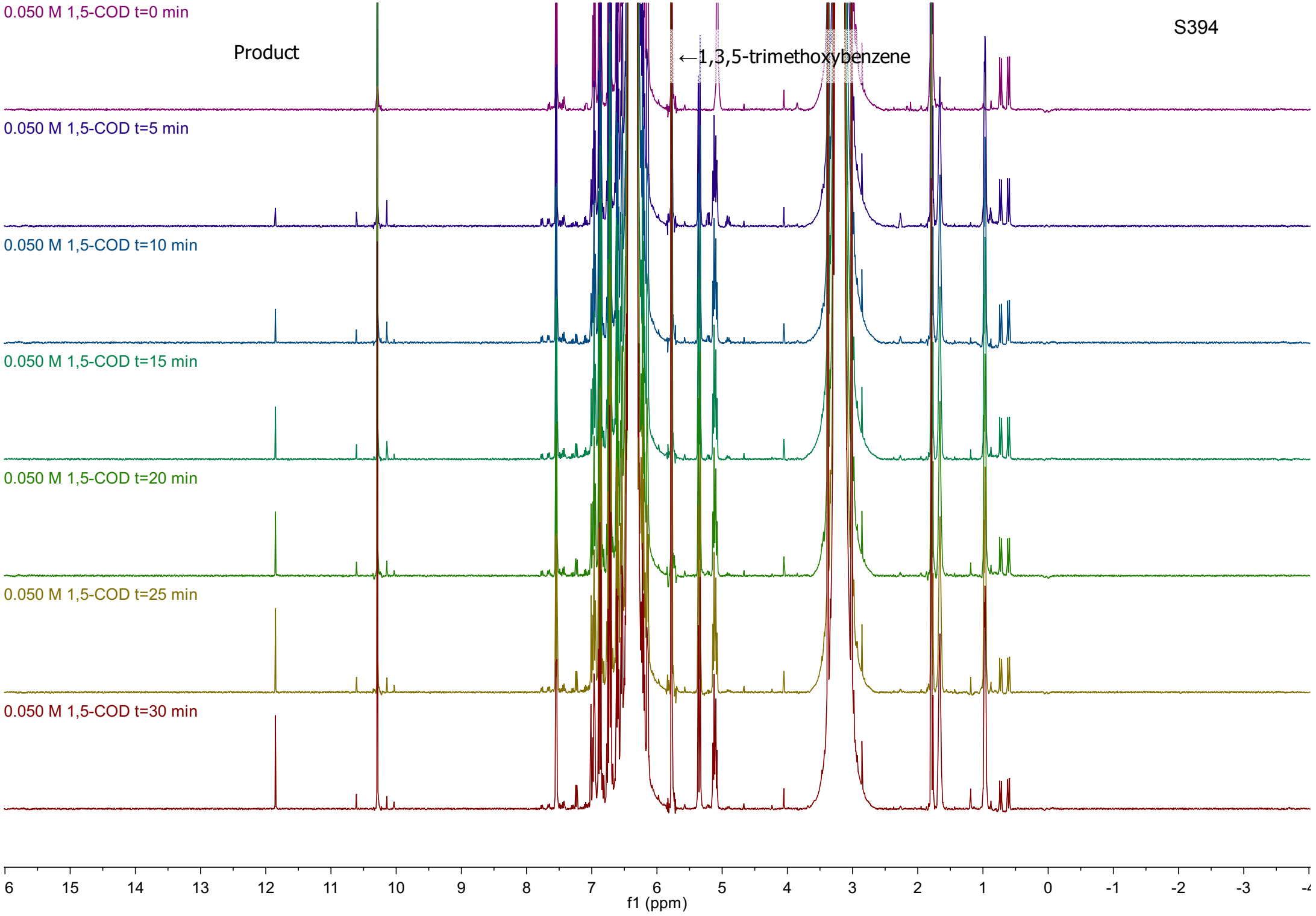

6.0 mM TrixiePhos t=30 min
Product
←1,3,5-trimethoxybenzene
S389
6.0 mM TrixiePhos t=35 min
6.0 mM TrixiePhos t=40 min
6.0 mM TrixiePhos t=45 min
6.0 mM TrixiePhos t=50 min
6.0 mM TrixiePhos t=55 min
6.0 mM TrixiePhos t=60 min
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
f1 (ppm)

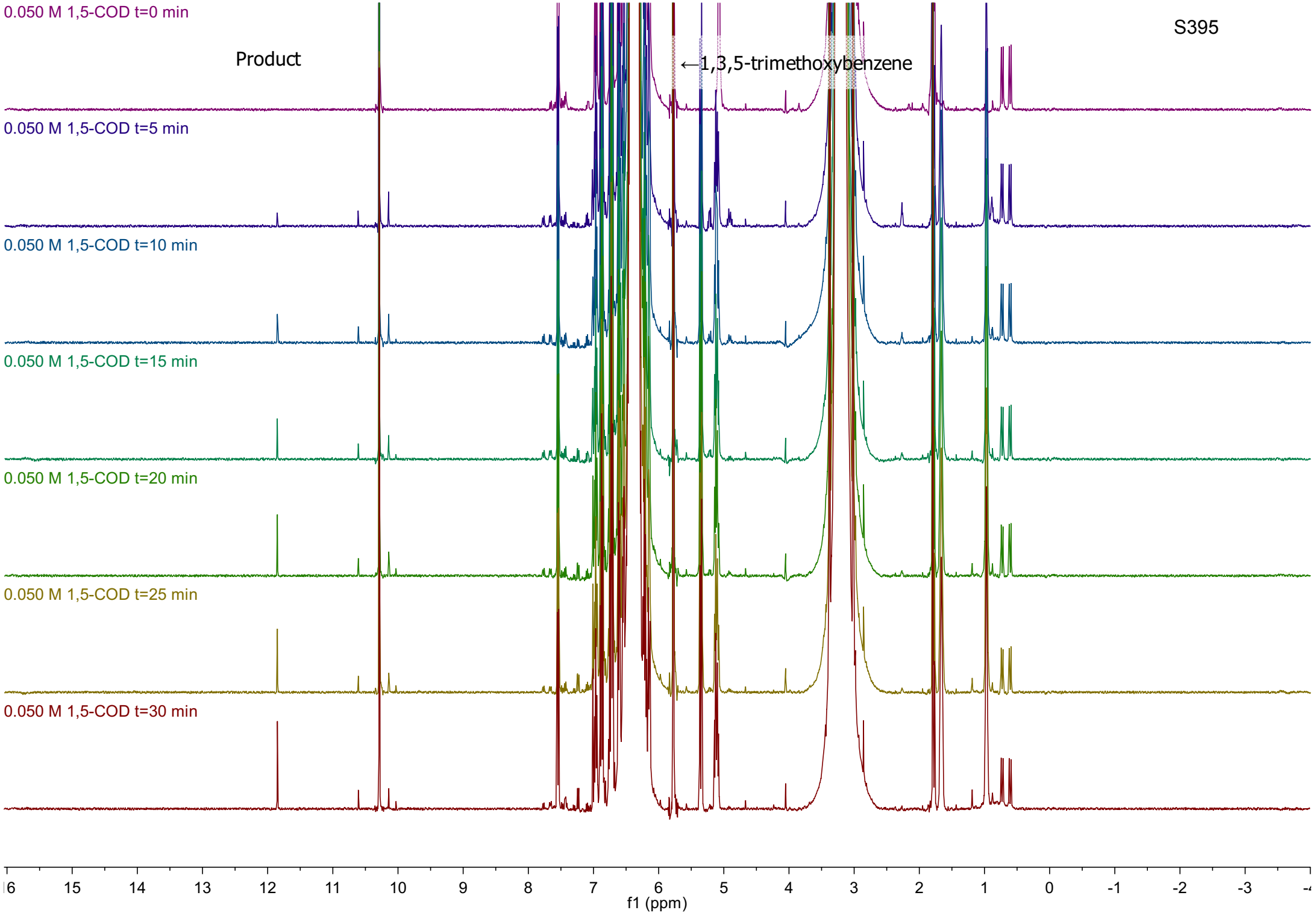

0.025 M 1,5-COD t=0 min
Product
←1,3,5-trimethoxybenzene
S390
0.025 M 1,5-COD t=5 min
0.025 M 1,5-COD t=10 min
0.025 M 1,5-COD t=15 min
0.025 M 1,5-COD t=20 min
0.025 M 1,5-COD t=25 min
0.025 M 1,5-COD t=30 min
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
f1 (ppm)

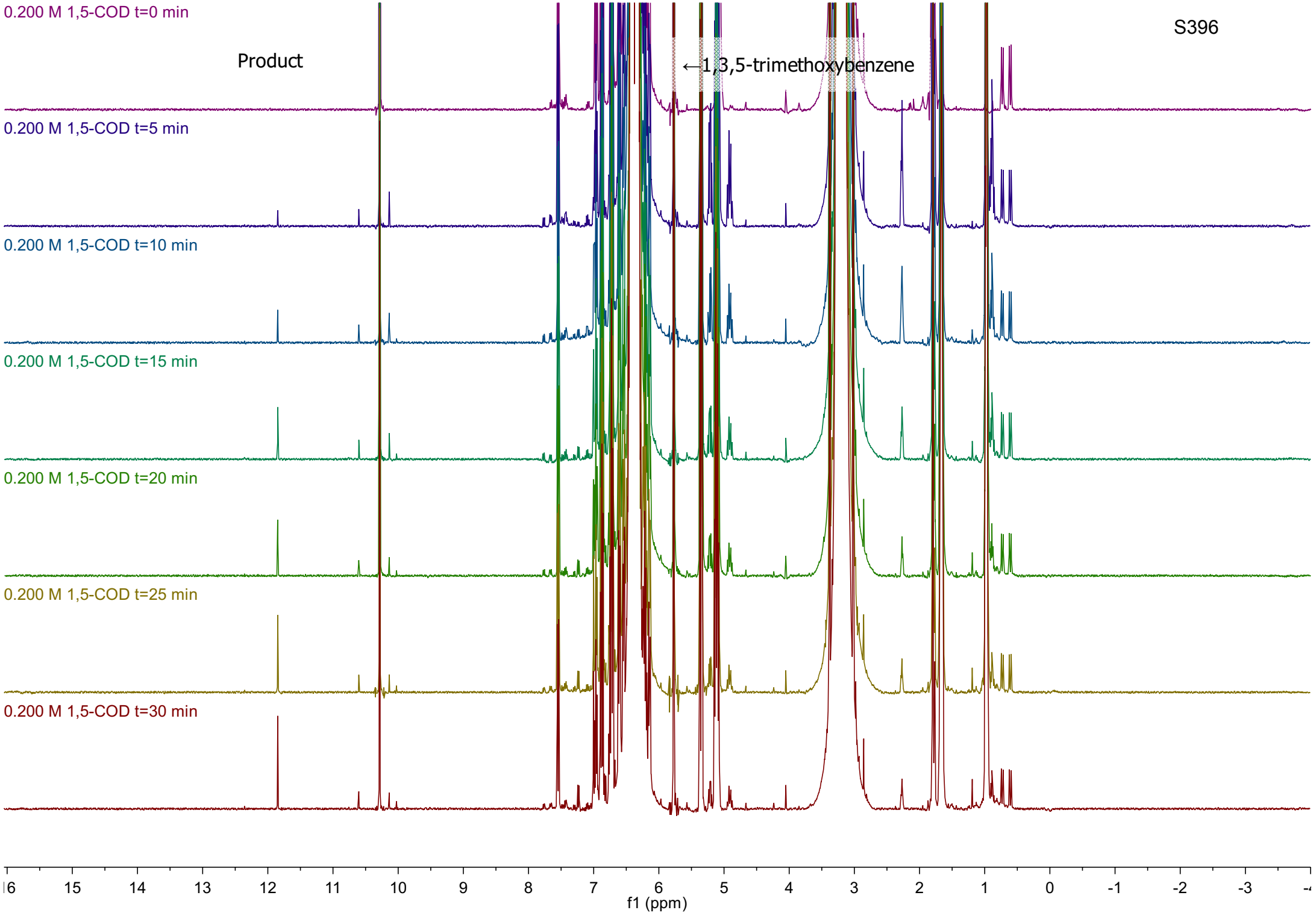

0.025 M 1,5-COD t=0 min
Product
←1,3,5-trimethoxybenzene
S391
0.025 M 1,5-COD t=5 min
0.025 M 1,5-COD t=10 min
0.025 M 1,5-COD t=15 min
0.025 M 1,5-COD t=20 min
0.025 M 1,5-COD t=25 min
0.025 M 1,5-COD t=30 min
6 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3 -4
f1 (ppm)

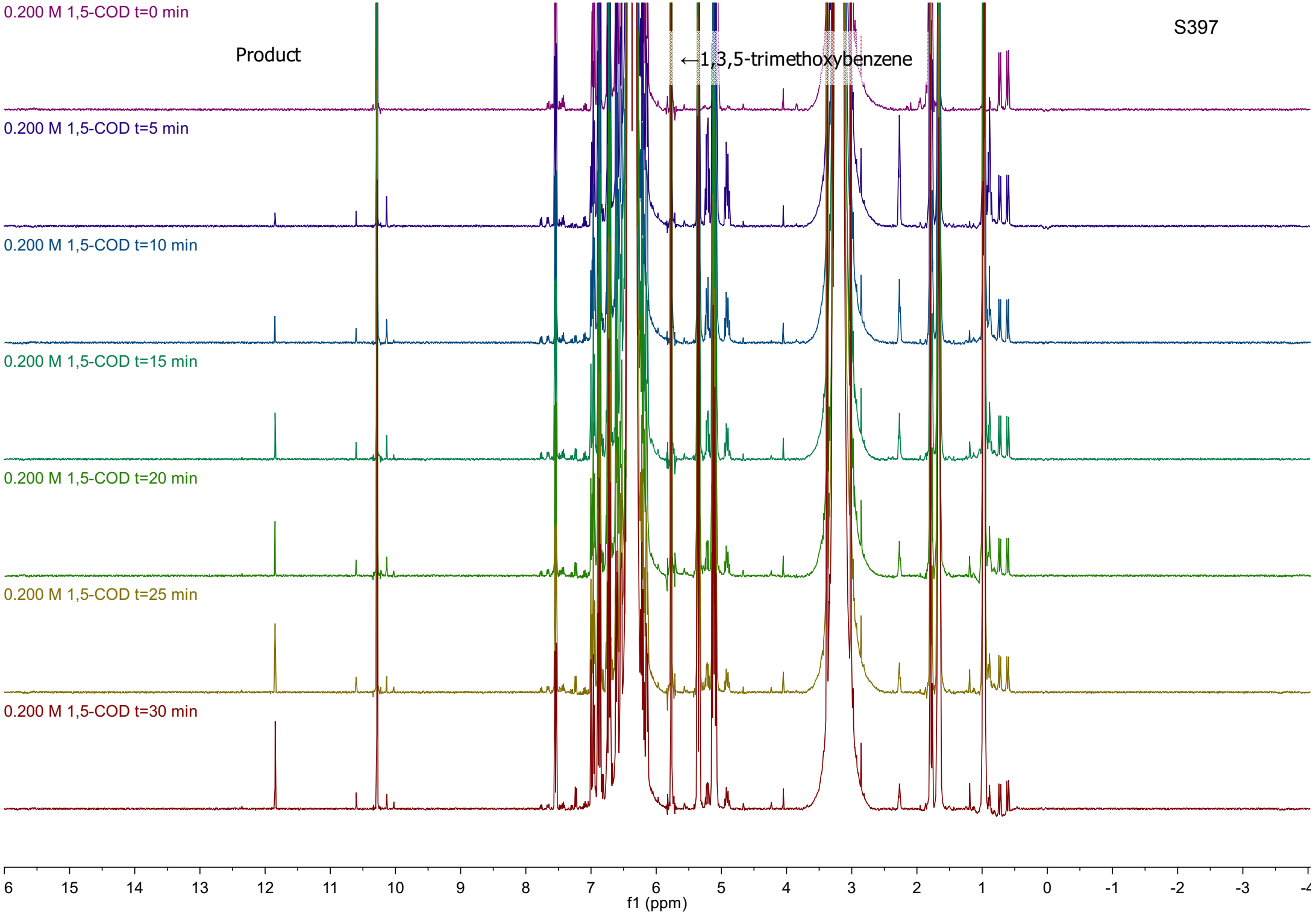

0.025 M 1,5-COD t=0 min
Product
←1,3,5-trimethoxybenzene
S392
0.025 M 1,5-COD t=5 min
0.025 M 1,5-COD t=10 min
0.025 M 1,5-COD t=15 min
0.025 M 1,5-COD t=20 min
0.025 M 1,5-COD t=25 min
0.025 M 1,5-COD t=30 min
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3 -4
f1 (ppm)

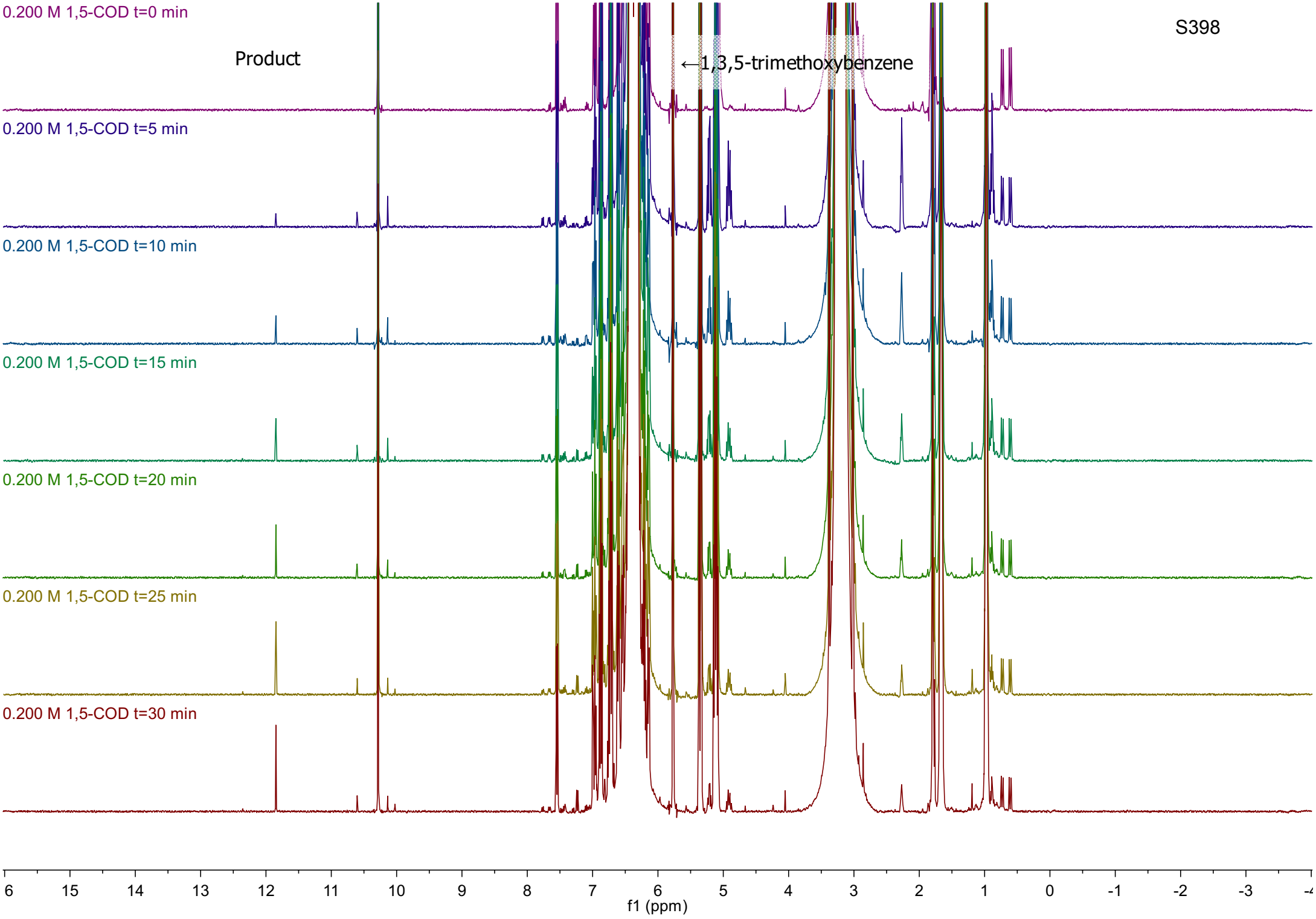

0.050 M 1,5-COD t=0 min
Product
←1,3,5-trimethoxybenzene
S393
0.050 M 1,5-COD t=5 min
0.050 M 1,5-COD t=10 min
0.050 M 1,5-COD t=15 min
0.050 M 1,5-COD t=20 min
0.050 M 1,5-COD t=25 min
0.050 M 1,5-COD t=30 min
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
f1 (ppm)

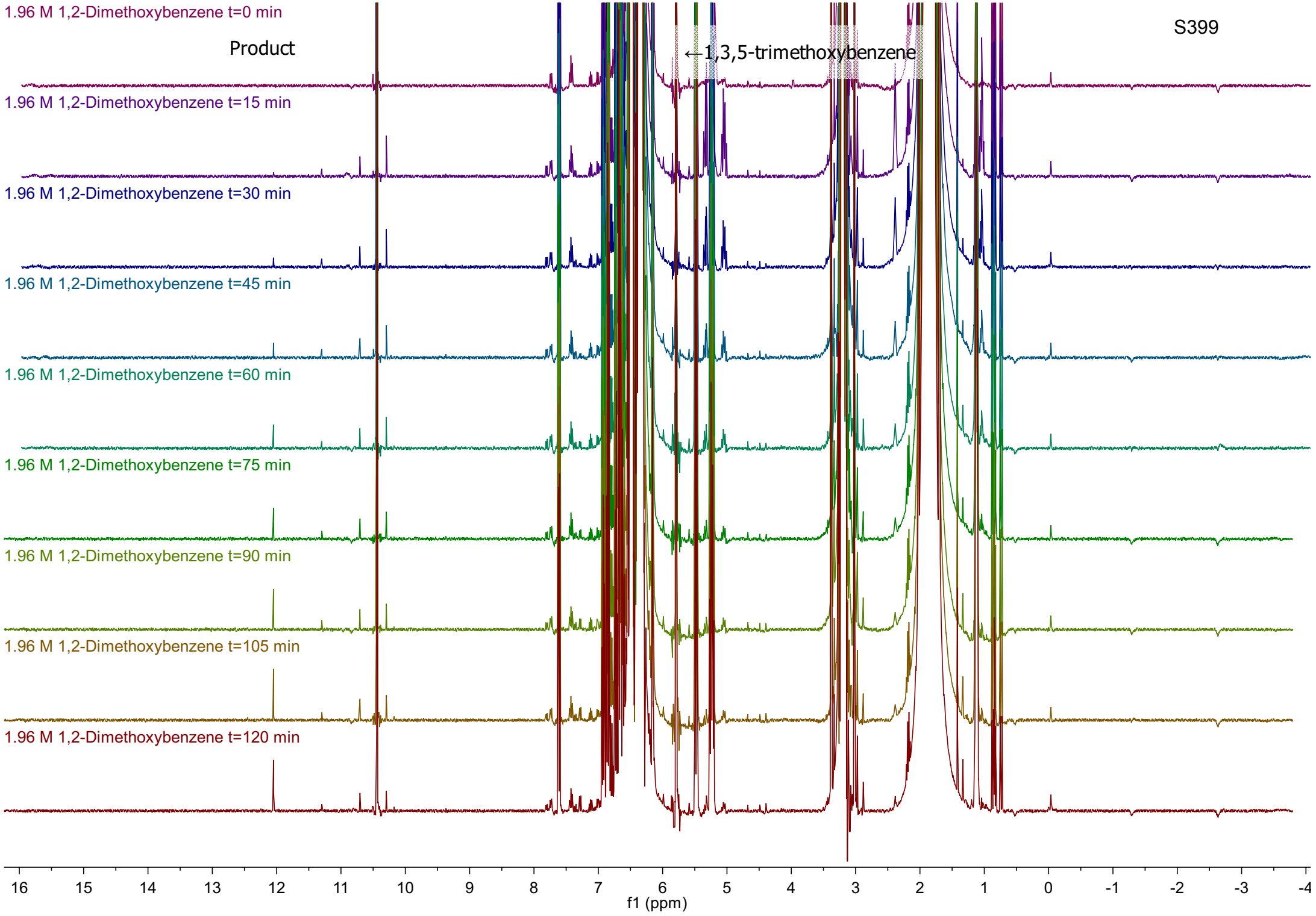

0.050 M 1,5-COD t=0 min
Product
←1,3,5-trimethoxybenzene
S394
0.050 M 1,5-COD t=5 min
0.050 M 1,5-COD t=10 min
0.050 M 1,5-COD t=15 min
0.050 M 1,5-COD t=20 min
0.050 M 1,5-COD t=25 min
0.050 M 1,5-COD t=30 min
6 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3 -4
f1 (ppm)

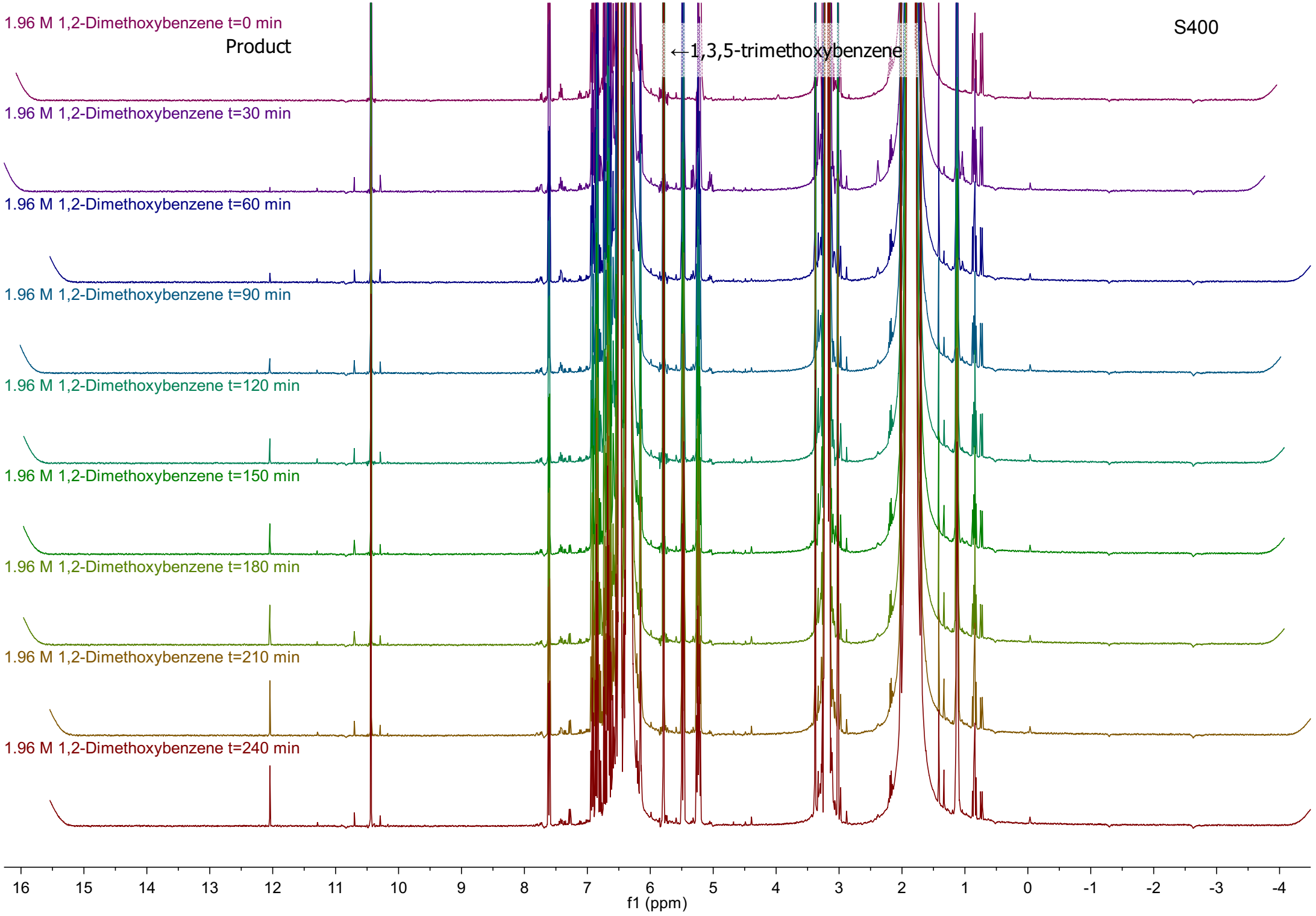

0.050 M 1,5-COD t=0 min
Product
←1,3,5-trimethoxybenzene
S395
0.050 M 1,5-COD t=5 min
0.050 M 1,5-COD t=10 min
0.050 M 1,5-COD t=15 min
0.050 M 1,5-COD t=20 min
0.050 M 1,5-COD t=25 min
0.050 M 1,5-COD t=30 min
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
f1 (ppm)

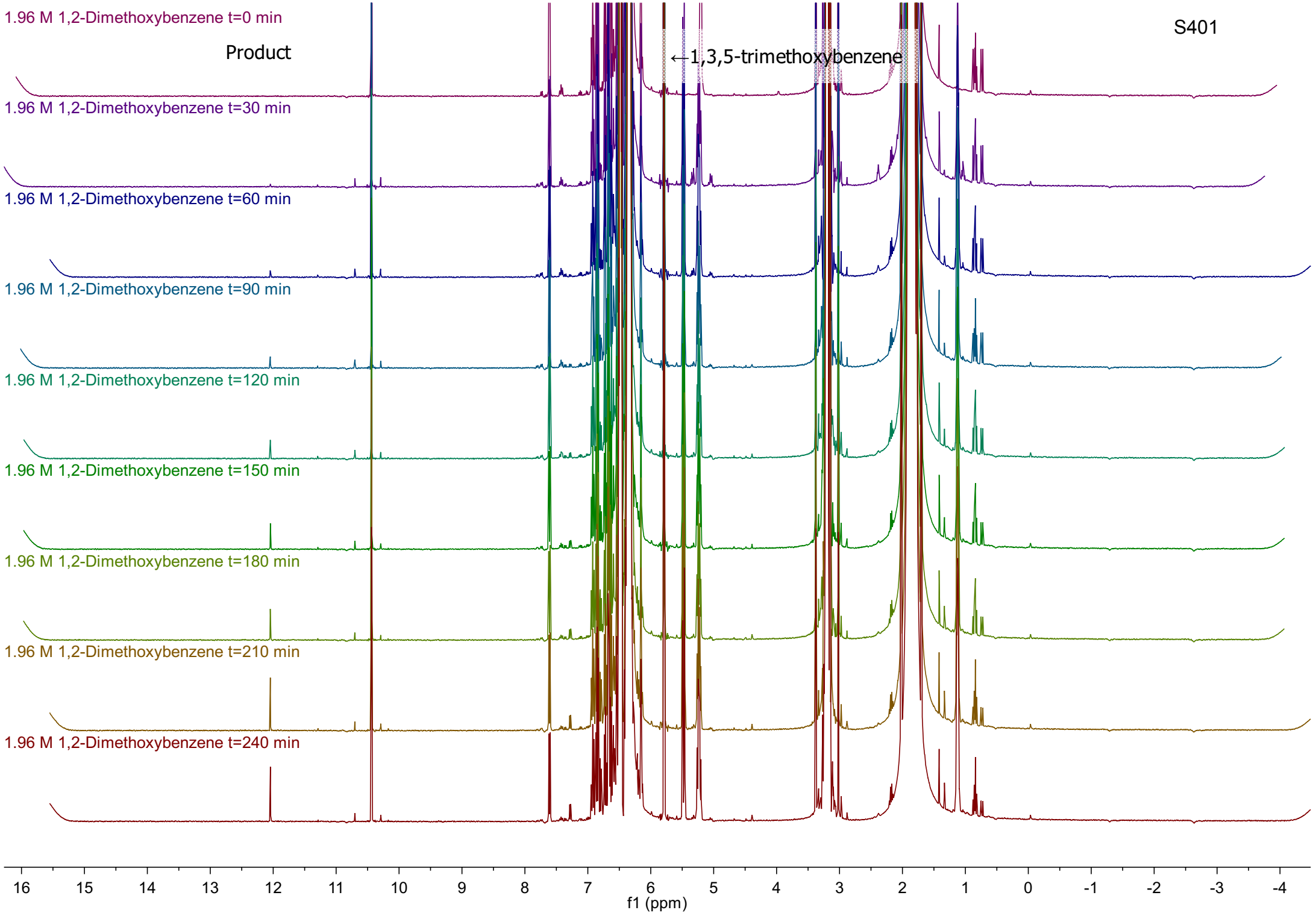

0.200 M 1,5-COD t=0 min
Product
←1,3,5-trimethoxybenzene
S396
0.200 M 1,5-COD t=5 min
0.200 M 1,5-COD t=10 min
0.200 M 1,5-COD t=15 min
0.200 M 1,5-COD t=20 min
0.200 M 1,5-COD t=25 min
0.200 M 1,5-COD t=30 min
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
f1 (ppm)

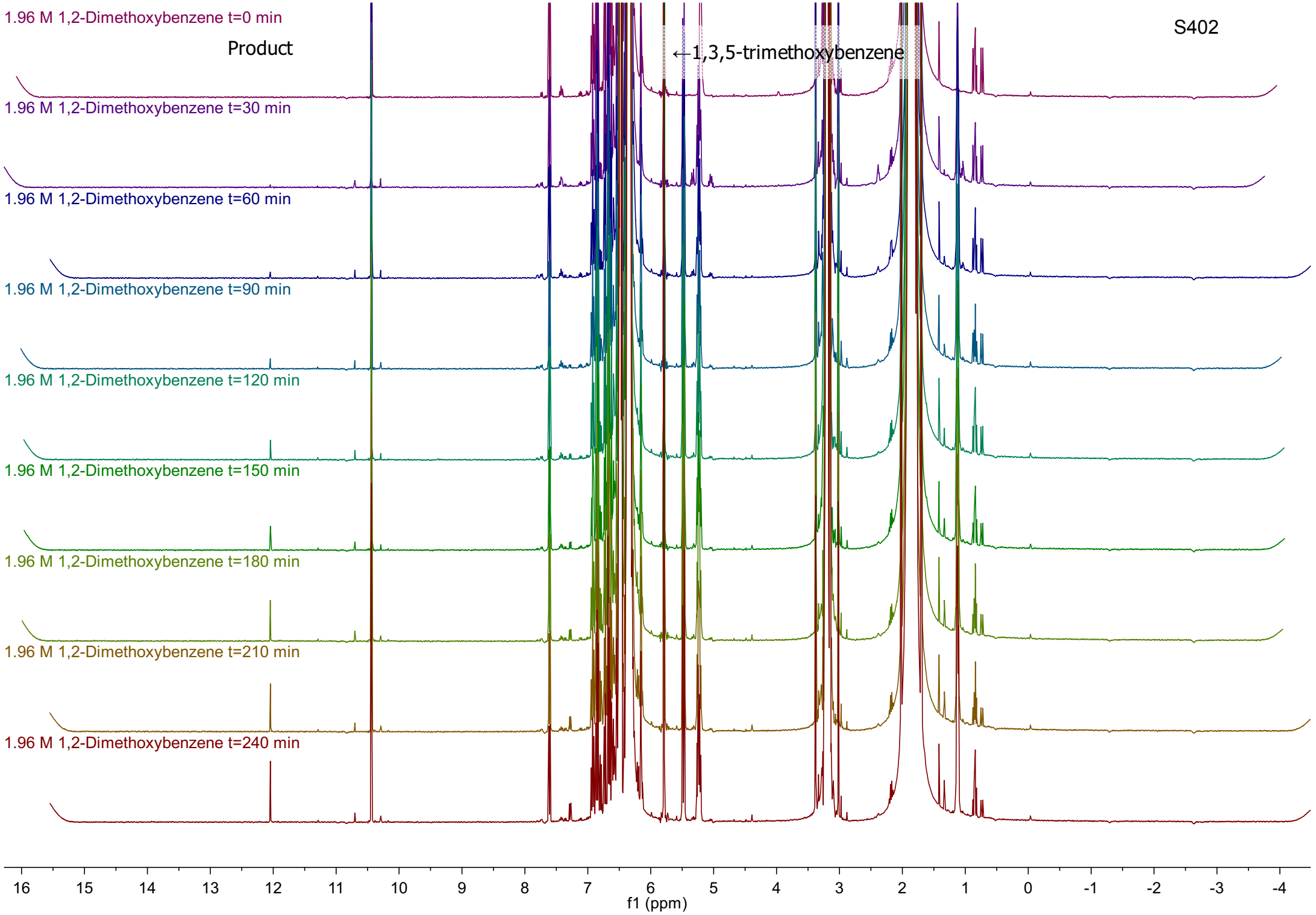

0.200 M 1,5-COD t=0 min
Product
←1,3,5-trimethoxybenzene
S397
0.200 M 1,5-COD t=5 min
0.200 M 1,5-COD t=10 min
0.200 M 1,5-COD t=15 min
0.200 M 1,5-COD t=20 min
0.200 M 1,5-COD t=25 min
0.200 M 1,5-COD t=30 min
6 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3 -4
f1 (ppm)

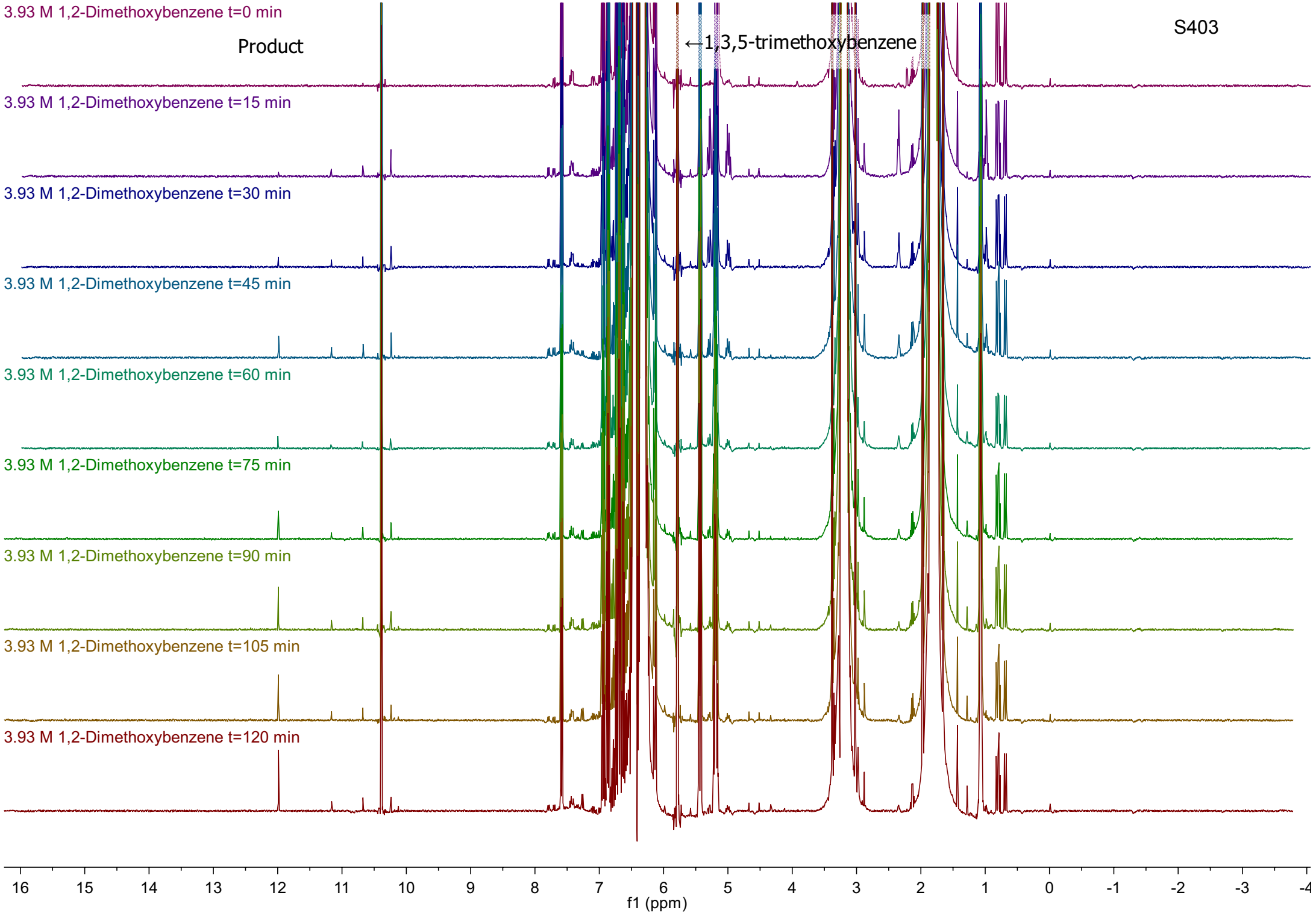

0.200 M 1,5-COD t=0 min
Product
←1,3,5-trimethoxybenzene
S398
0.200 M 1,5-COD t=5 min
0.200 M 1,5-COD t=10 min
0.200 M 1,5-COD t=15 min
0.200 M 1,5-COD t=20 min
0.200 M 1,5-COD t=25 min
0.200 M 1,5-COD t=30 min
6 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3 -4
f1 (ppm)

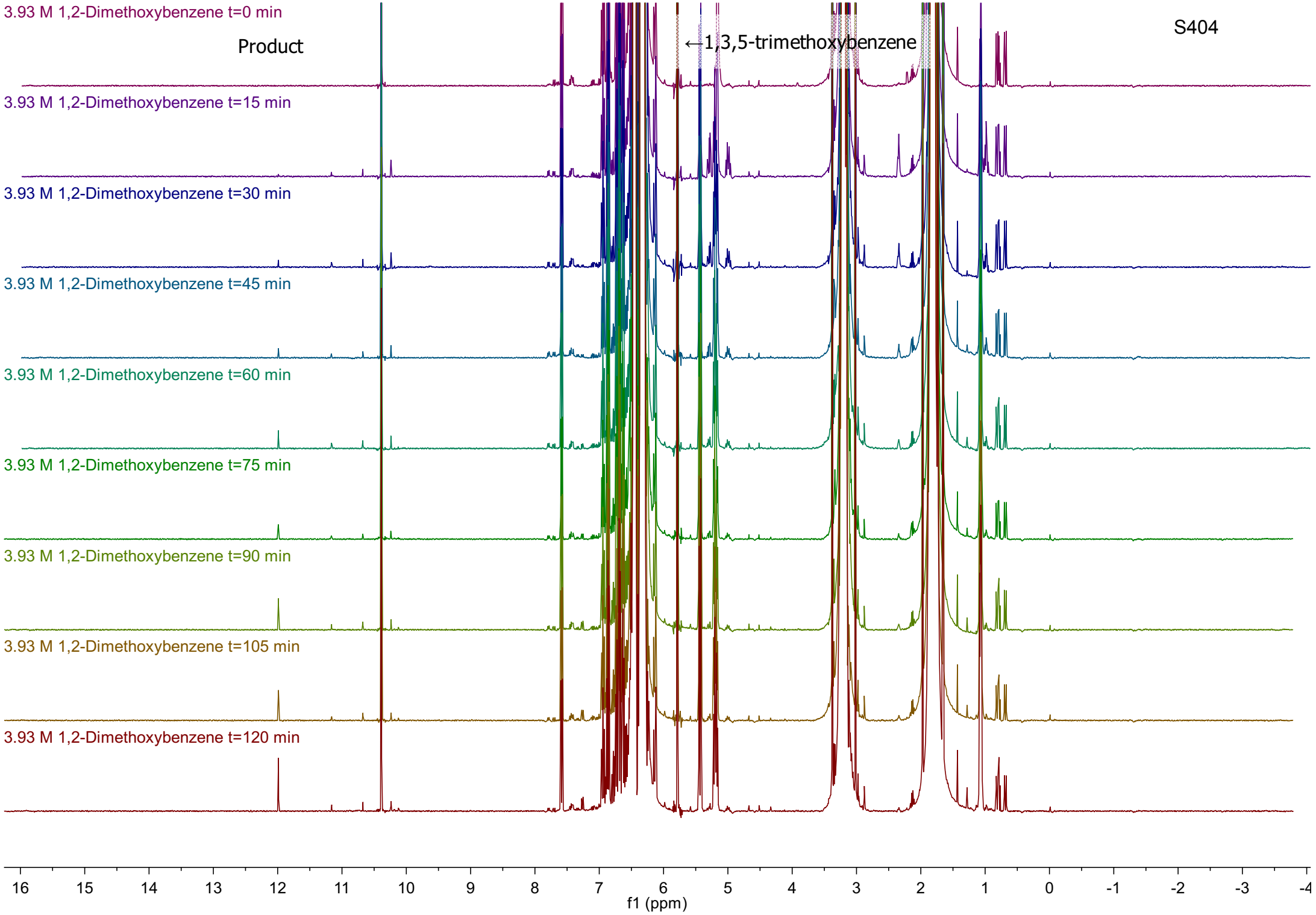

1.96 M 1,2-Dimethoxybenzene t=0 min
Product
←1,3,5-trimethoxybenzene
S399
1.96 M 1,2-Dimethoxybenzene t=15 min
1.96 M 1,2-Dimethoxybenzene t=30 min
1.96 M 1,2-Dimethoxybenzene t=45 min
1.96 M 1,2-Dimethoxybenzene t=60 min
1.96 M 1,2-Dimethoxybenzene t=75 min
1.96 M 1,2-Dimethoxybenzene t=90 min
1.96 M 1,2-Dimethoxybenzene t=105 min
1.96 M 1,2-Dimethoxybenzene t=120 min
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3 -4
f1 (ppm)

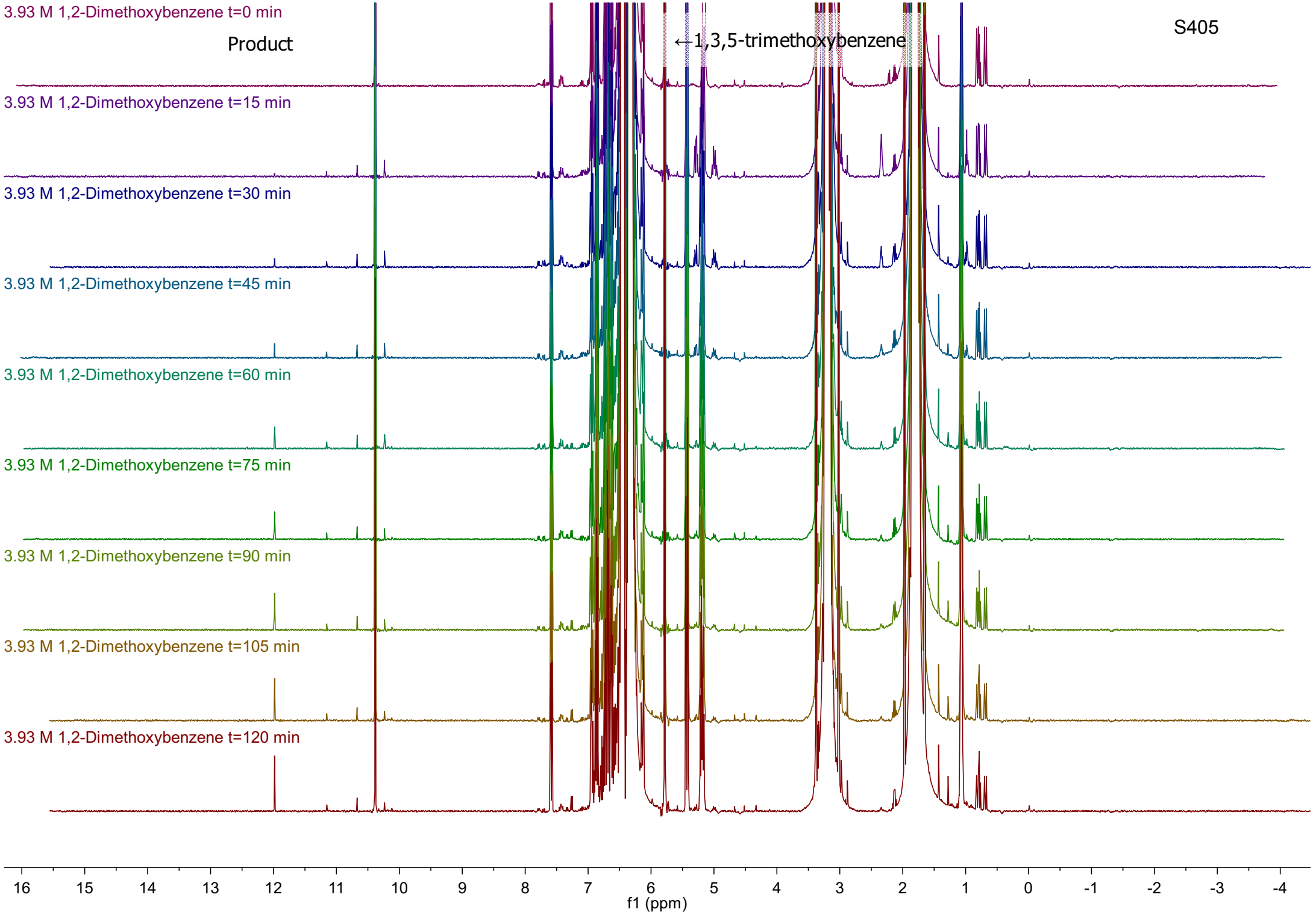

1.96 M 1,2-Dimethoxybenzene t=0 min
Product
←1,3,5-trimethoxybenzene
S400
1.96 M 1,2-Dimethoxybenzene t=30 min
1.96 M 1,2-Dimethoxybenzene t=60 min
1.96 M 1,2-Dimethoxybenzene t=90 min
1.96 M 1,2-Dimethoxybenzene t=120 min
1.96 M 1,2-Dimethoxybenzene t=150 min
1.96 M 1,2-Dimethoxybenzene t=180 min
1.96 M 1,2-Dimethoxybenzene t=210 min
1.96 M 1,2-Dimethoxybenzene t=240 min
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3 -4
f1 (ppm)

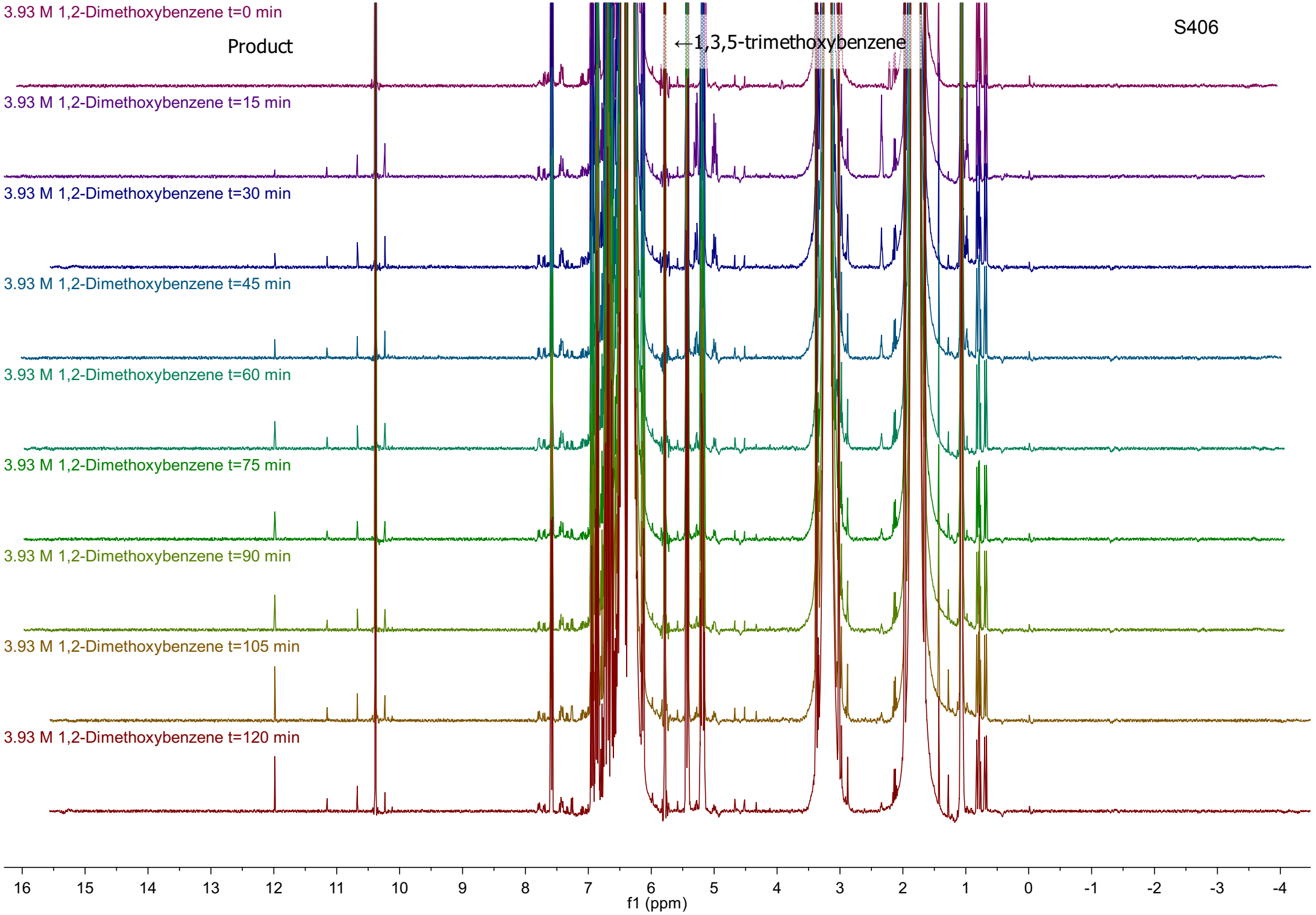

1.96 M 1,2-Dimethoxybenzene t=0 min
Product
←1,3,5-trimethoxybenzene
S401
1.96 M 1,2-Dimethoxybenzene t=30 min
1.96 M 1,2-Dimethoxybenzene t=60 min
1.96 M 1,2-Dimethoxybenzene t=90 min
1.96 M 1,2-Dimethoxybenzene t=120 min
1.96 M 1,2-Dimethoxybenzene t=150 min
1.96 M 1,2-Dimethoxybenzene t=180 min
1.96 M 1,2-Dimethoxybenzene t=210 min
1.96 M 1,2-Dimethoxybenzene t=240 min
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3 -4
f1 (ppm)


1.96 M 1,2-Dimethoxybenzene t=0 min
Product
←1,3,5-trimethoxybenzene
S402
1.96 M 1,2-Dimethoxybenzene t=30 min
1.96 M 1,2-Dimethoxybenzene t=60 min
1.96 M 1,2-Dimethoxybenzene t=90 min
1.96 M 1,2-Dimethoxybenzene t=120 min
1.96 M 1,2-Dimethoxybenzene t=150 min
1.96 M 1,2-Dimethoxybenzene t=180 min
1.96 M 1,2-Dimethoxybenzene t=210 min
1.96 M 1,2-Dimethoxybenzene t=240 min
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3 -4
f1 (ppm)


3.93 M 1,2-Dimethoxybenzene t=0 min
Product
←1,3,5-trimethoxybenzene
S403
3.93 M 1,2-Dimethoxybenzene t=15 min
3.93 M 1,2-Dimethoxybenzene t=30 min
3.93 M 1,2-Dimethoxybenzene t=45 min
3.93 M 1,2-Dimethoxybenzene t=60 min
3.93 M 1,2-Dimethoxybenzene t=75 min
3.93 M 1,2-Dimethoxybenzene t=90 min
3.93 M 1,2-Dimethoxybenzene t=105 min
3.93 M 1,2-Dimethoxybenzene t=120 min
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3 -4
f1 (ppm)


3.93 M 1,2-Dimethoxybenzene t=0 min
Product
←1,3,5-trimethoxybenzene
S404
3.93 M 1,2-Dimethoxybenzene t=15 min
3.93 M 1,2-Dimethoxybenzene t=30 min
3.93 M 1,2-Dimethoxybenzene t=45 min
3.93 M 1,2-Dimethoxybenzene t=60 min
3.93 M 1,2-Dimethoxybenzene t=75 min
3.93 M 1,2-Dimethoxybenzene t=90 min
3.93 M 1,2-Dimethoxybenzene t=105 min
3.93 M 1,2-Dimethoxybenzene t=120 min
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3 -4
f1 (ppm)


3.93 M 1,2-Dimethoxybenzene t=0 min
Product
←1,3,5-trimethoxybenzene
S405
3.93 M 1,2-Dimethoxybenzene t=15 min
3.93 M 1,2-Dimethoxybenzene t=30 min
3.93 M 1,2-Dimethoxybenzene t=45 min
3.93 M 1,2-Dimethoxybenzene t=60 min
3.93 M 1,2-Dimethoxybenzene t=75 min
3.93 M 1,2-Dimethoxybenzene t=90 min
3.93 M 1,2-Dimethoxybenzene t=105 min
3.93 M 1,2-Dimethoxybenzene t=120 min
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3 -4
f1 (ppm)


3.93 M 1,2-Dimethoxybenzene t=0 min
Product
←1,3,5-trimethoxybenzene
S406
3.93 M 1,2-Dimethoxybenzene t=15 min
3.93 M 1,2-Dimethoxybenzene t=30 min
3.93 M 1,2-Dimethoxybenzene t=45 min
3.93 M 1,2-Dimethoxybenzene t=60 min
3.93 M 1,2-Dimethoxybenzene t=75 min
3.93 M 1,2-Dimethoxybenzene t=90 min
3.93 M 1,2-Dimethoxybenzene t=105 min
3.93 M 1,2-Dimethoxybenzene t=120 min
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3 -4
f1 (ppm)