Synthesis of Trisubstituted Alkenes via Direct Oxidative Arene-alkene Coupling

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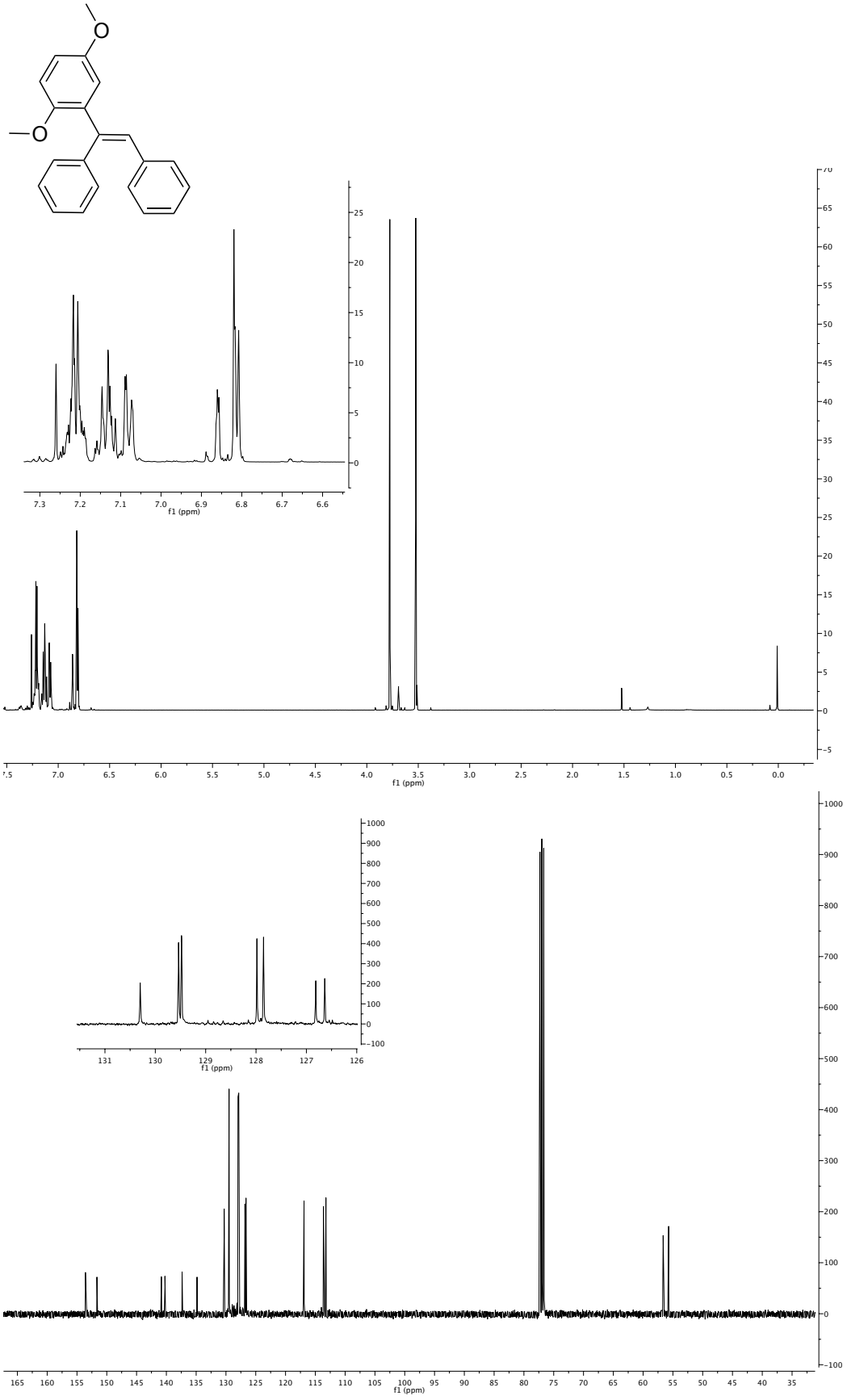
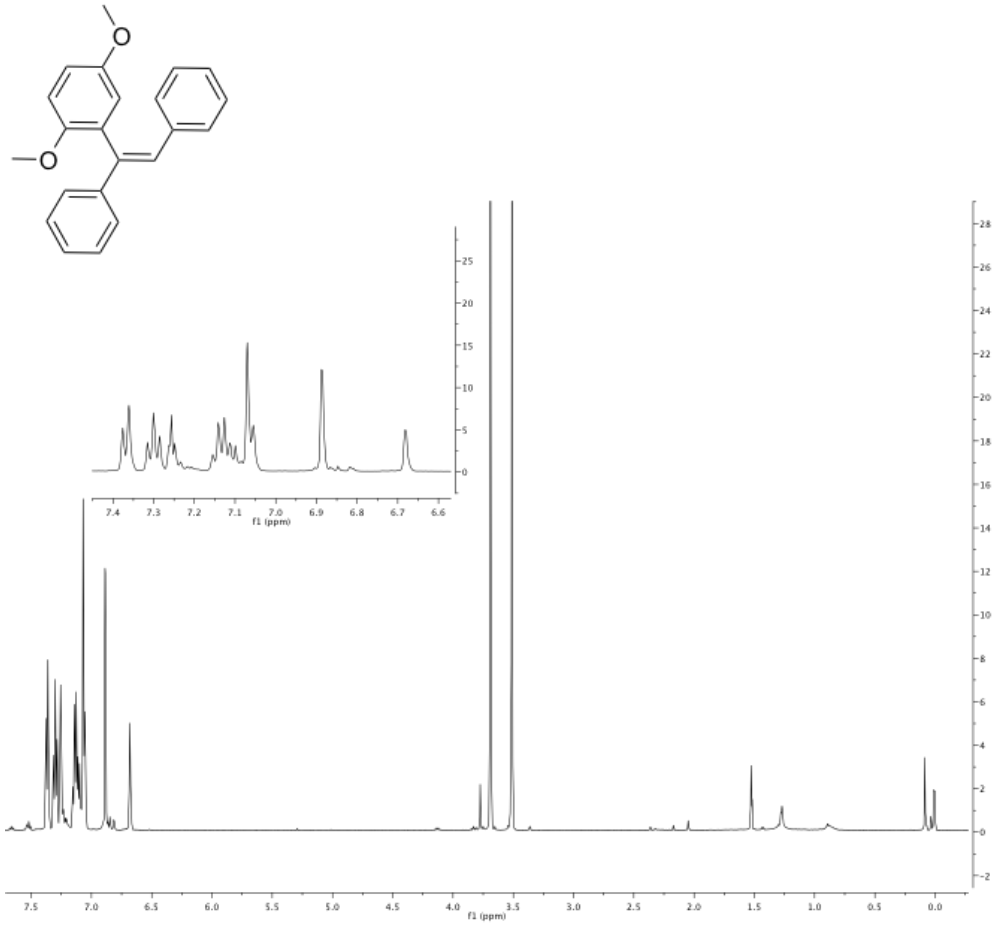
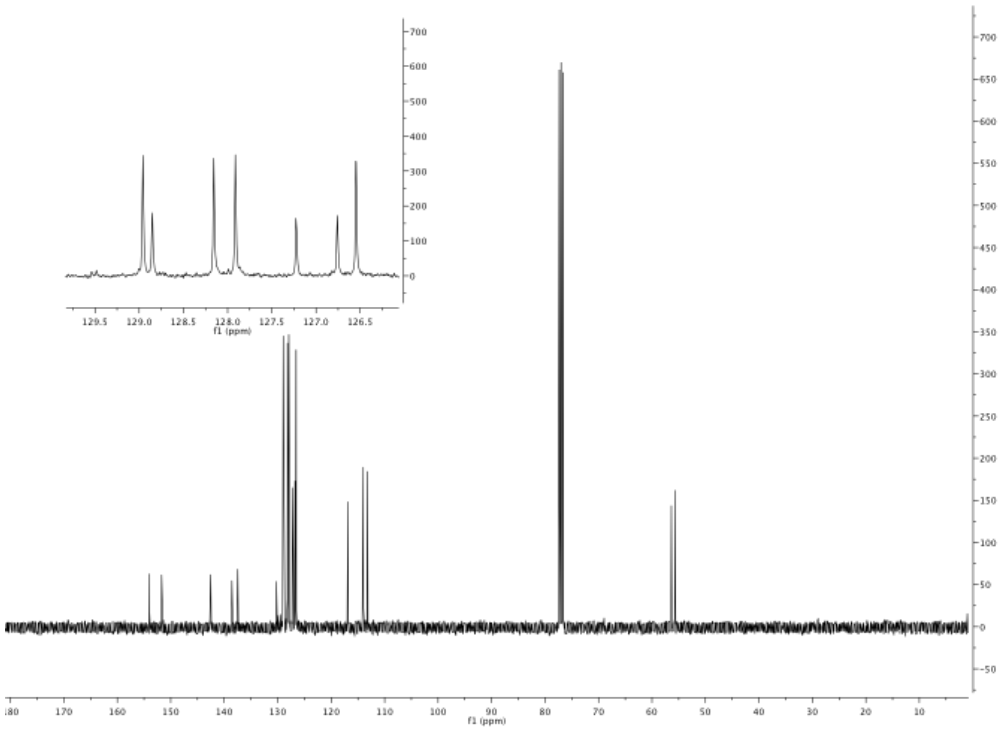
^{1}H and ^{13}C NMR (500 and 100 MHz, respectively in $CDCl_3$) of (*E*)-(1-(2,5-dimethoxyphenyl)ethene-1,2-diyl)dibenzene, **(*E*)-3a**

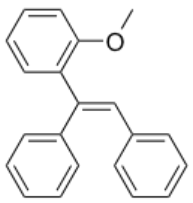


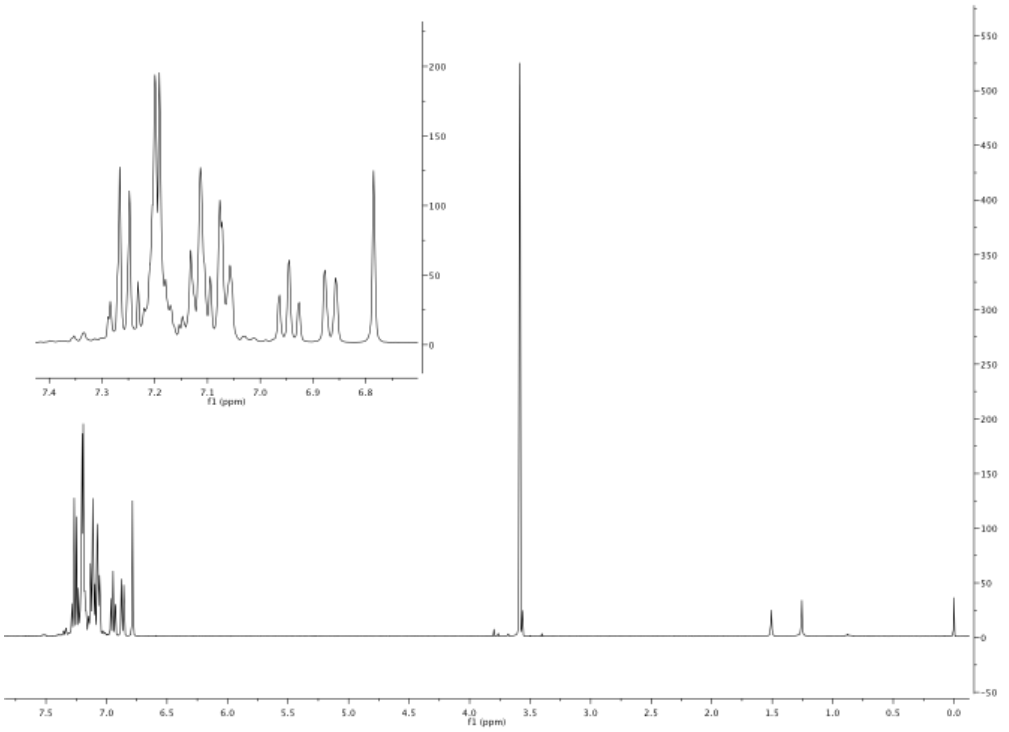
^{1}H and ^{13}C NMR (500 and 100 MHz, respectively in $CDCl_3$) of (*Z*)-(1-(2,5-dimethoxyphenyl)ethene-1,2-diyl)dibenzene, **(*Z*)-3a**.

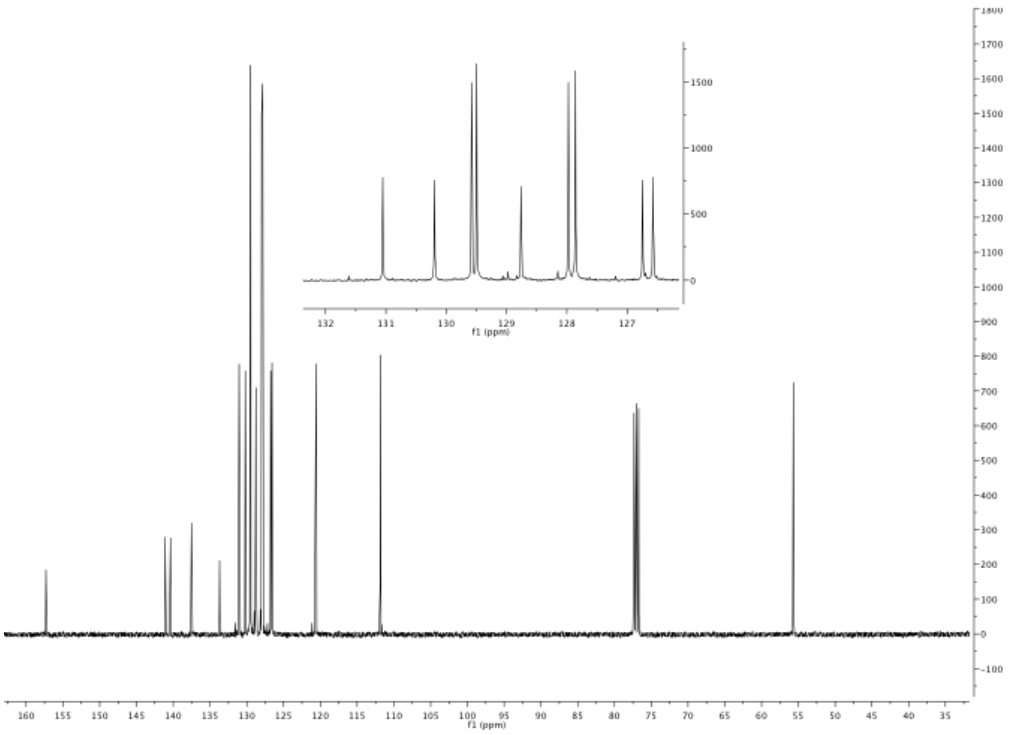




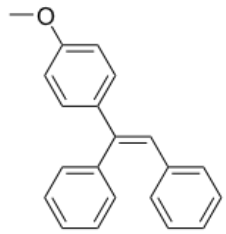
^{1}H and ^{13}C NMR (500 and 100 MHz, respectively in $CDCl_3$) of (*E*)-(1-(2-methoxyphenyl)ethene-1,2-diyl)dibenzene, **(*E*)-*ortho*-3b**.

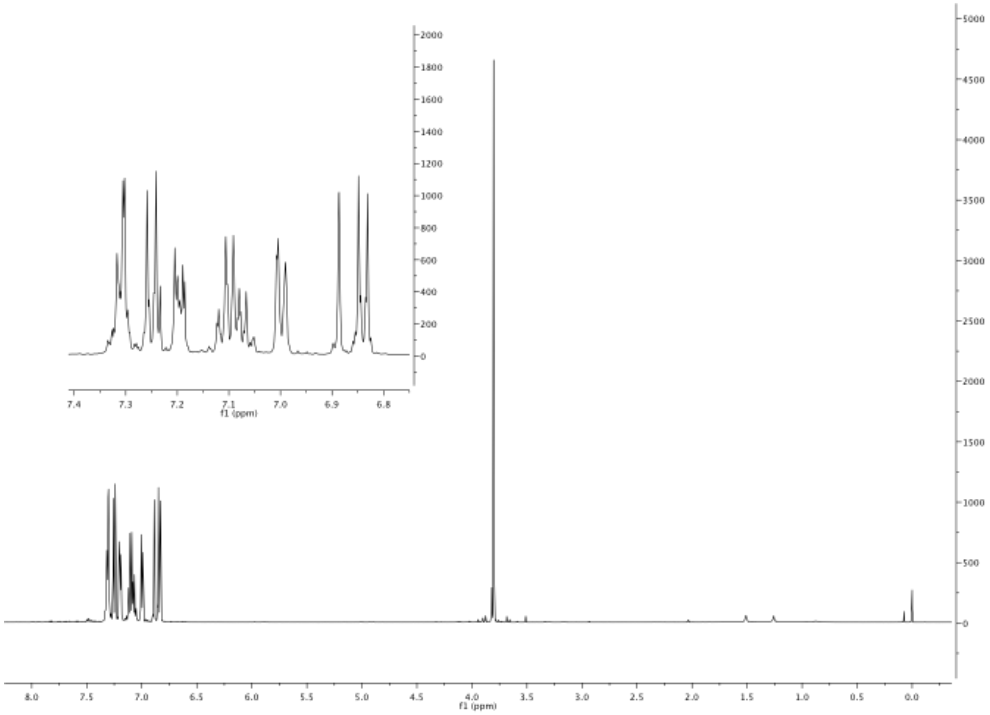


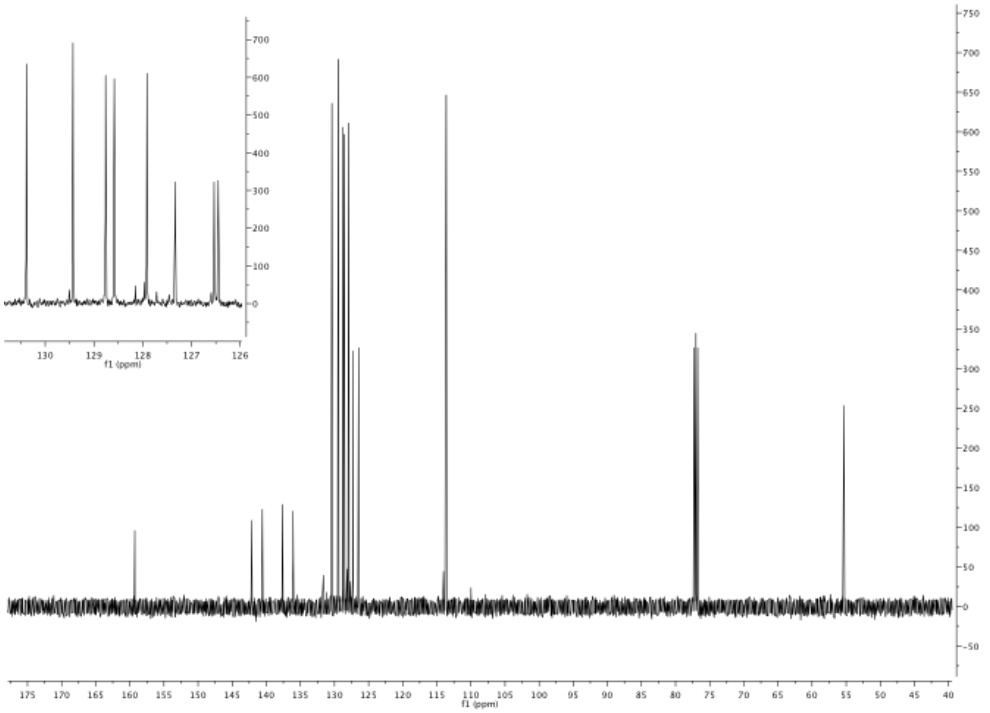




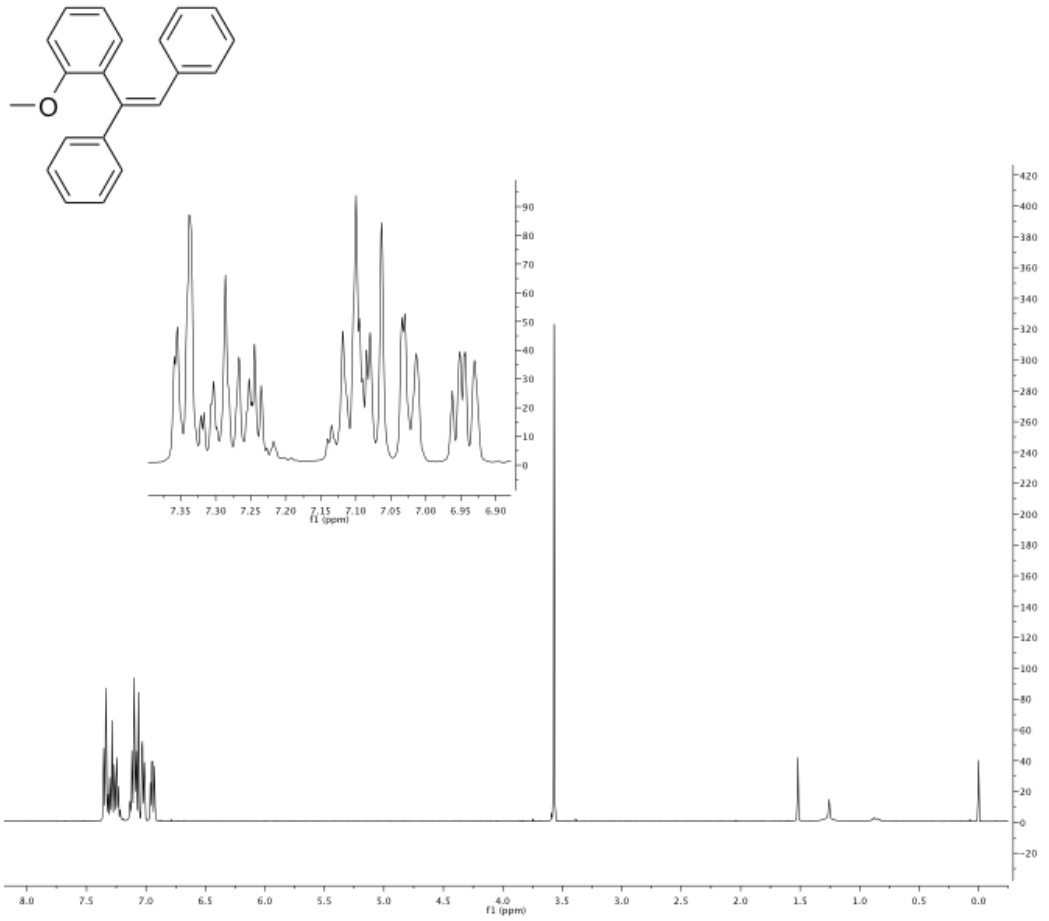
^{1}H and ^{13}C NMR (500 and 100 MHz, respectively in $CDCl_3$) of (*E*)-(1-(4-Methoxyphenyl)ethene-1,2-diyl)dibenzene, **(*E*)-*para*-3b**.

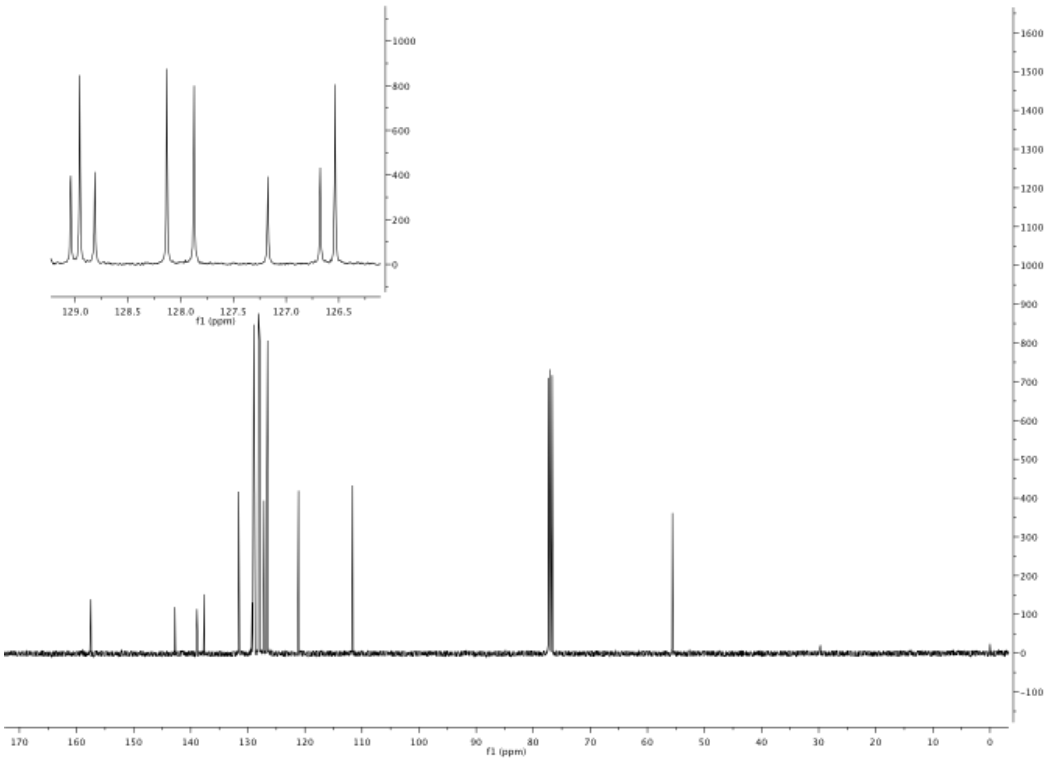




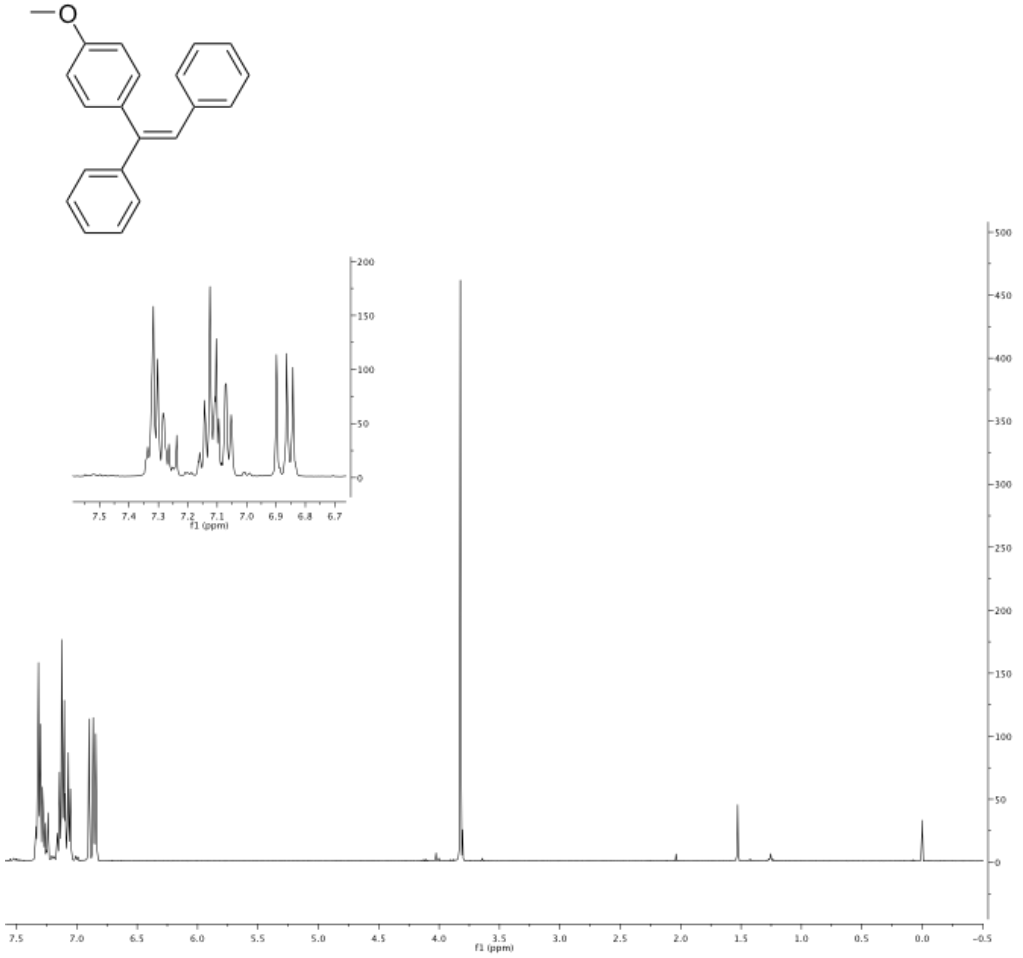


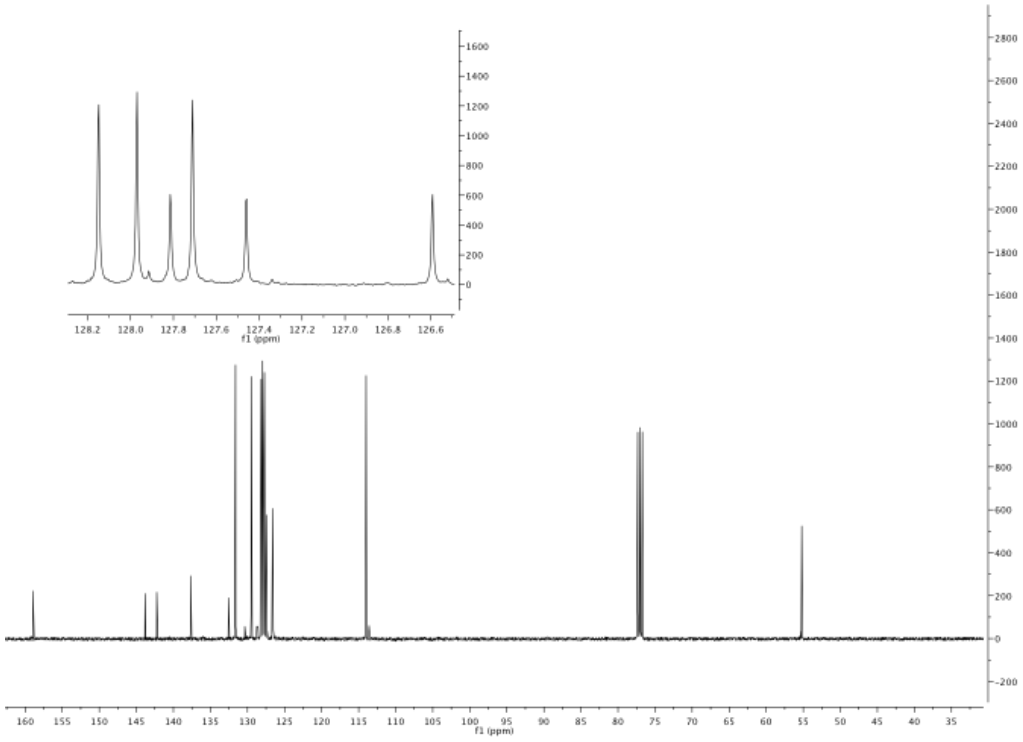
^{1}H and ^{13}C NMR (500 and 100 MHz, respectively in $CDCl_3$) of (*Z*)-(1-(2-methoxyphenyl)ethene-1,2-diyl)dibenzene, **(*Z*)-*ortho*-3b**.



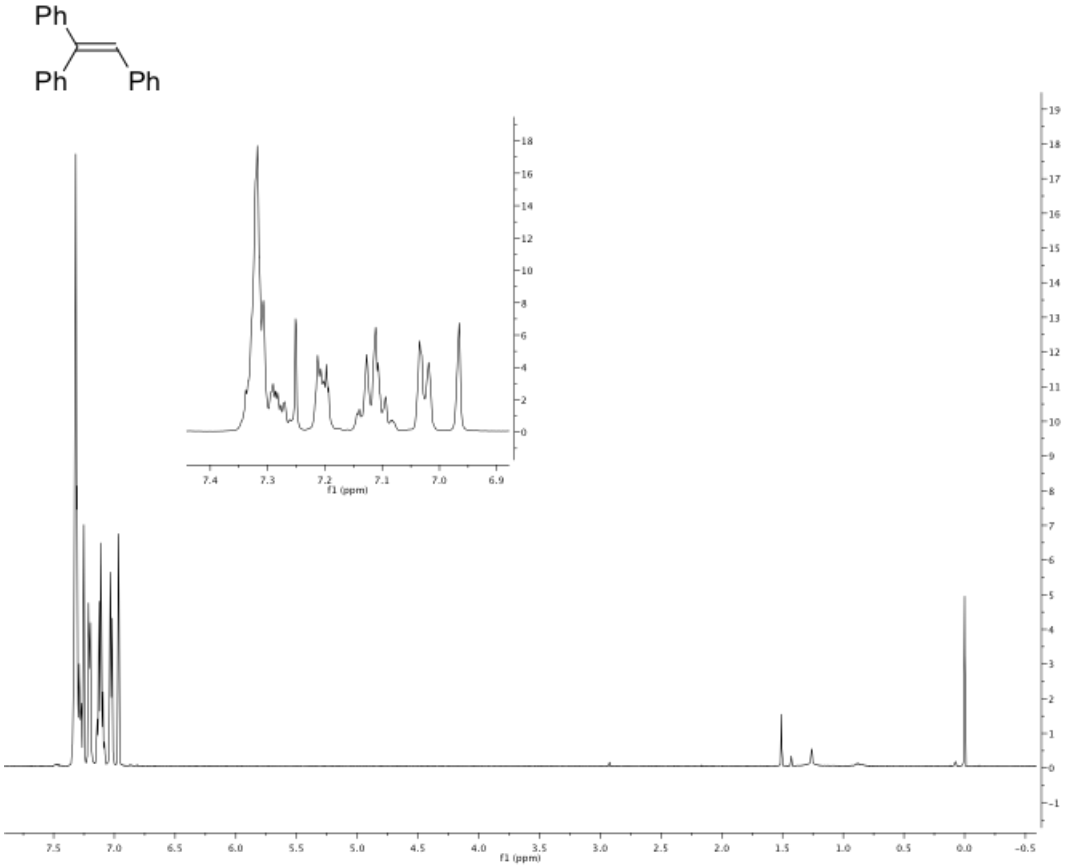


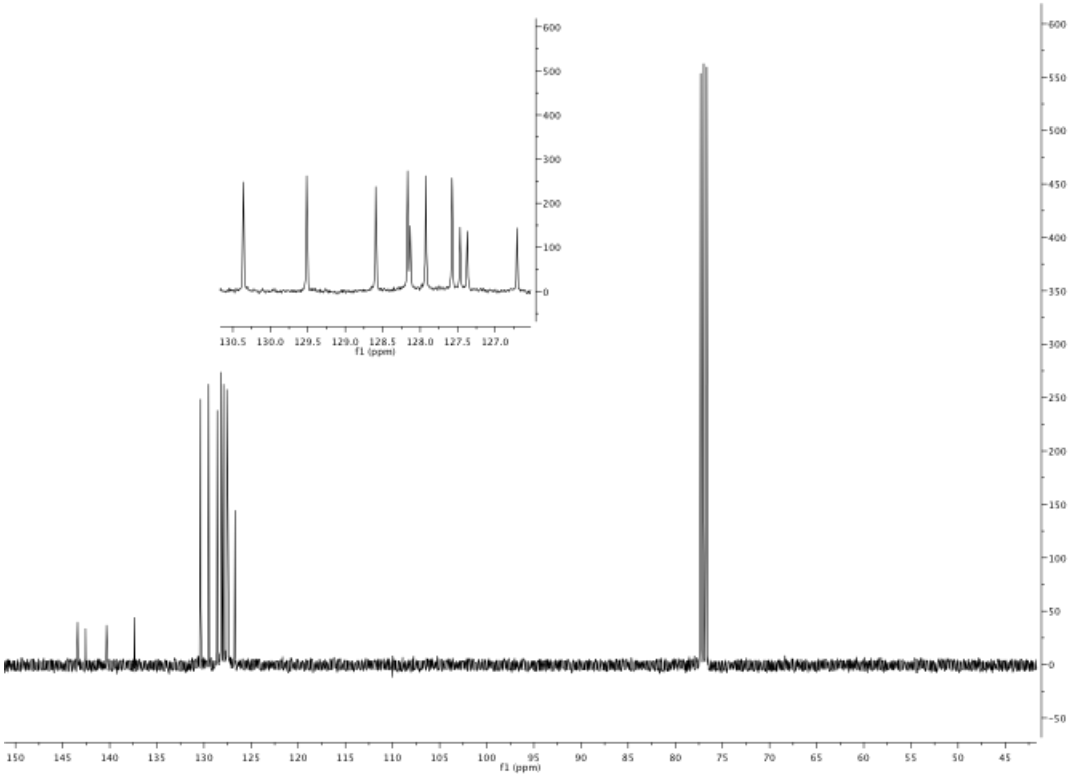
^{1}H and ^{13}C NMR (500 and 100 MHz, respectively in $CDCl_3$) of (*E*)-(1-(4-Methoxyphenyl)ethene-1,2-diyl)dibenzene, **(*E*)-*para*-3b**.



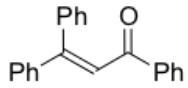


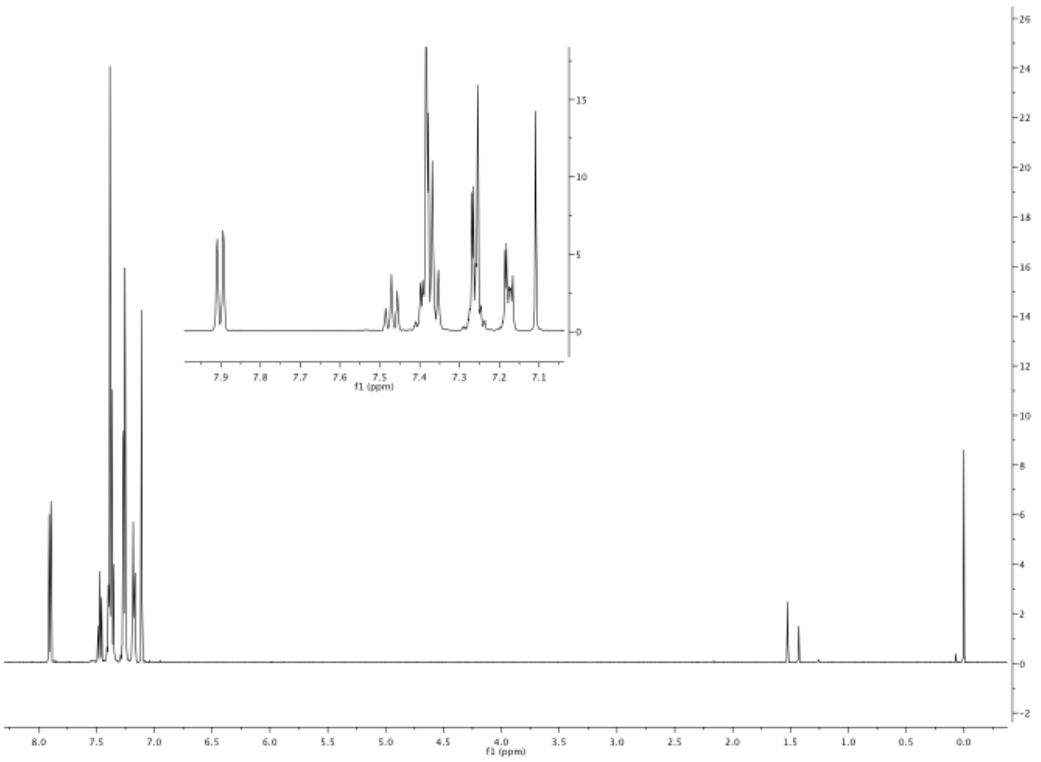
^{1}H and ^{13}C NMR (500 and 100 MHz, respectively in $CDCl_3$) of triphenylethylene, **3c**.

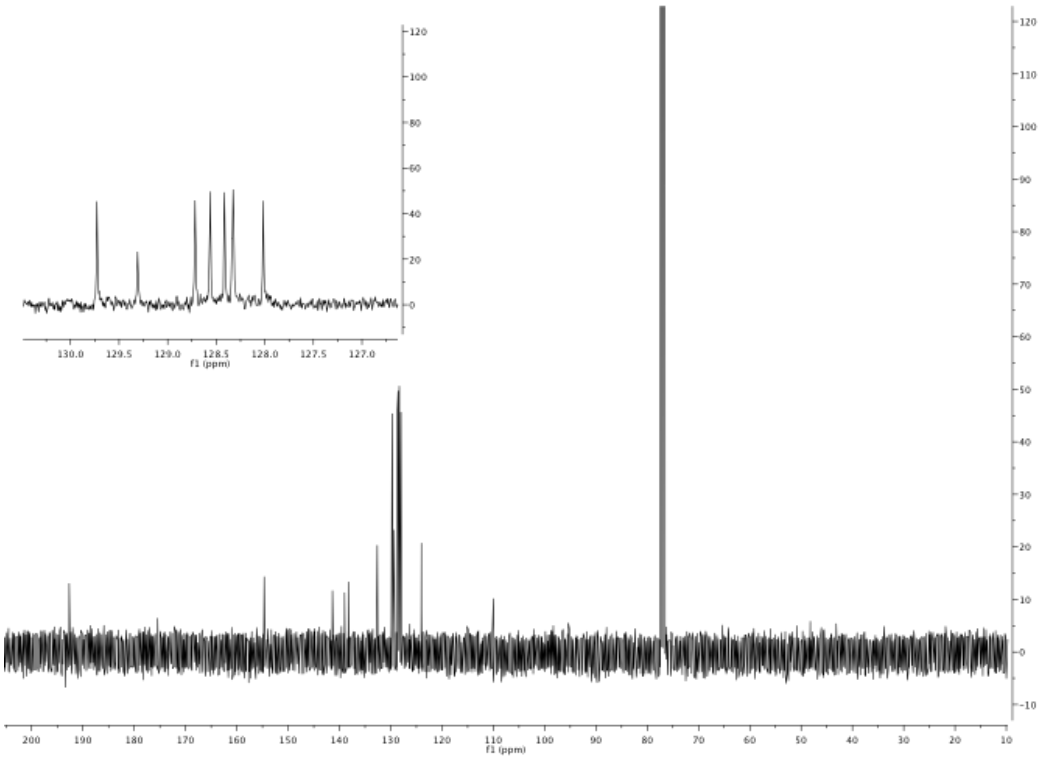




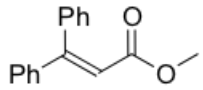
^{1}H and ^{13}C NMR (500 and 100 MHz, respectively in $CDCl_3$) of 3,3-diphenylacrylophenone, **3d**

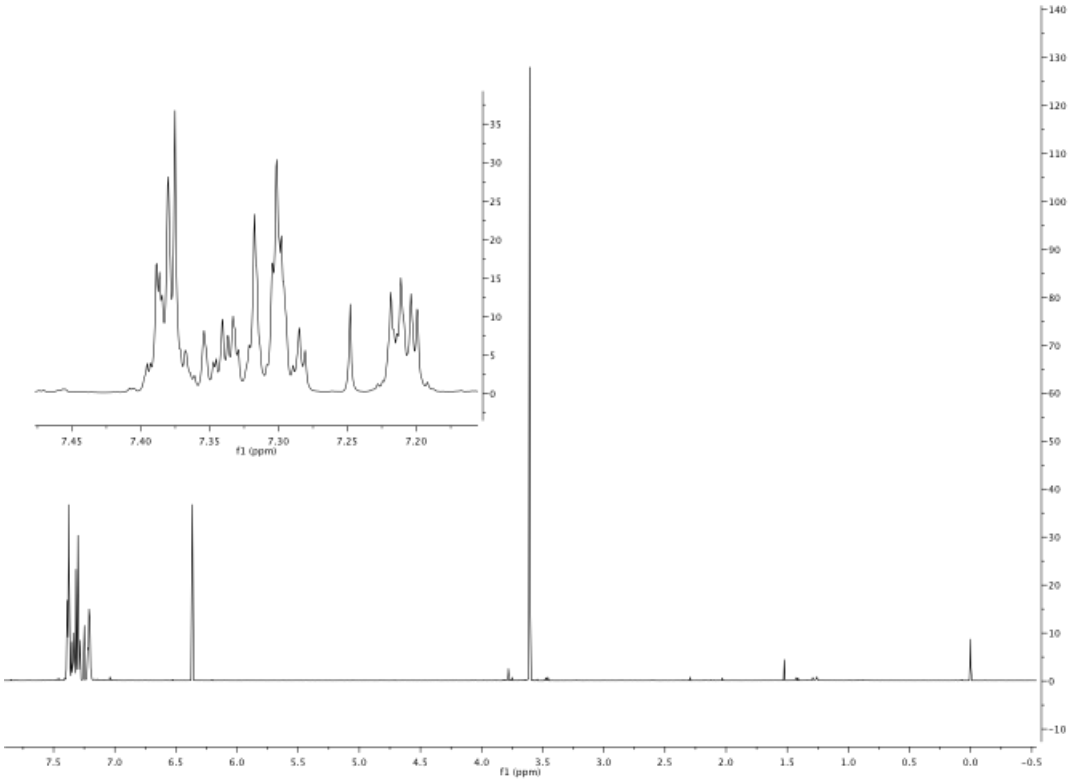


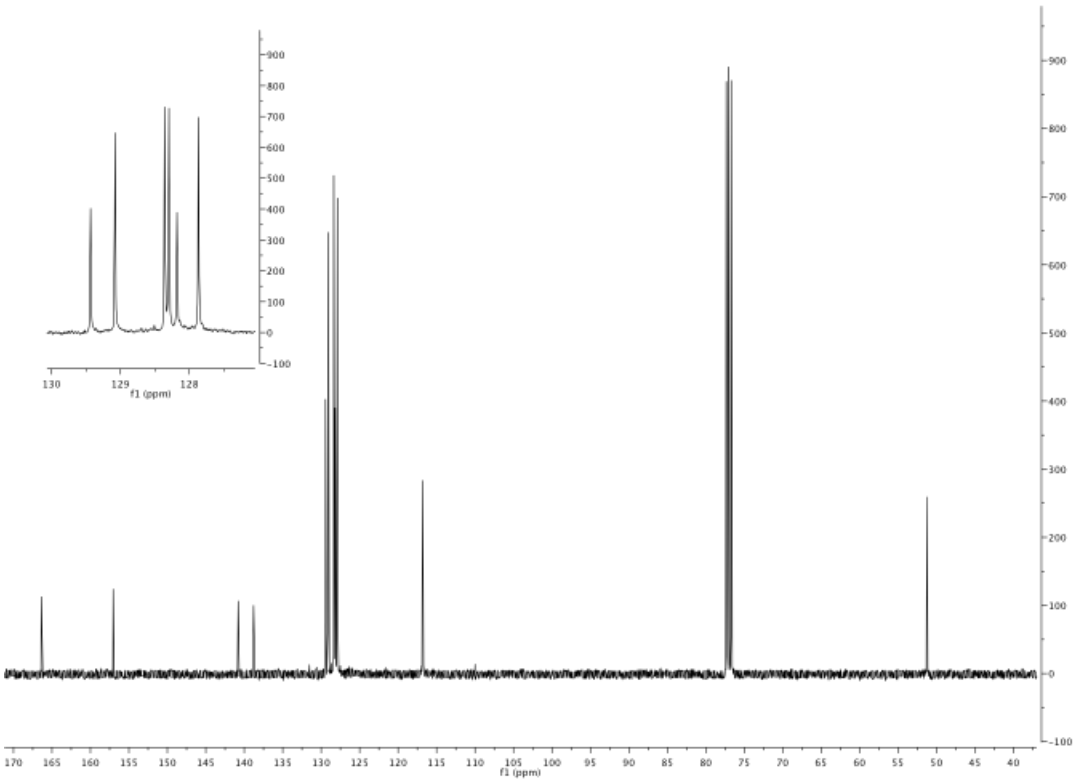




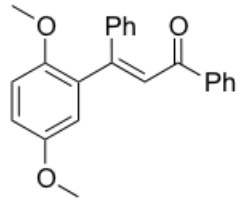
^{1}H and ^{13}C NMR (500 and 100 MHz, respectively in $CDCl_3$) of methyl 3,3-diphenyl-2-propenoate, **3e**.

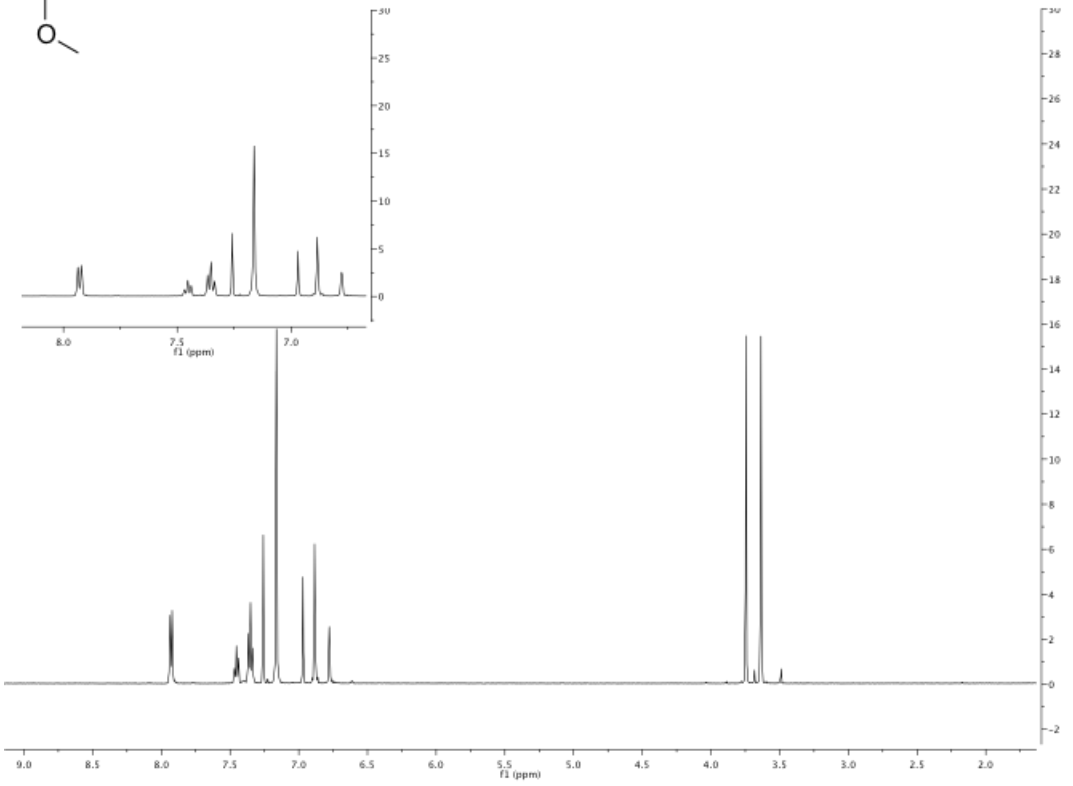


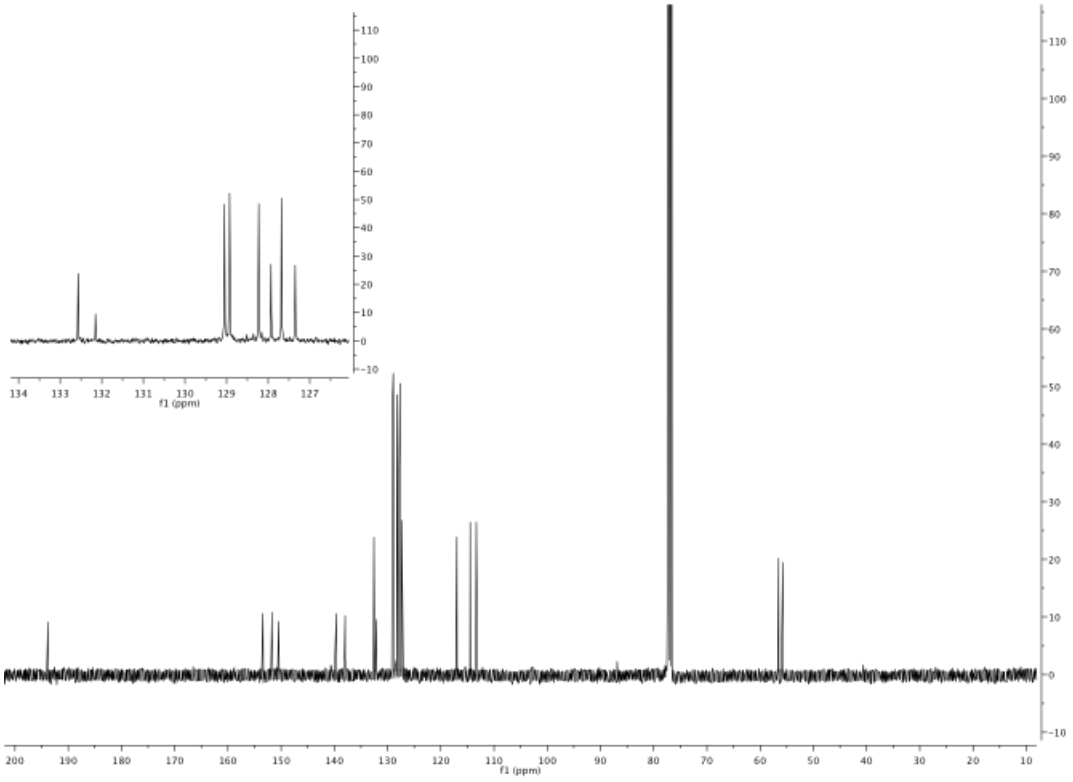




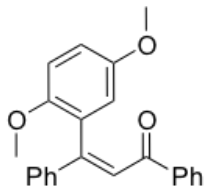
^{1}H and ^{13}C NMR (500 and 100 MHz, respectively in $CDCl_3$) of (*E*)-3-(2,5-dimethoxyphenyl)-1,3-diphenylpropenone, **(*E*)-3f**.

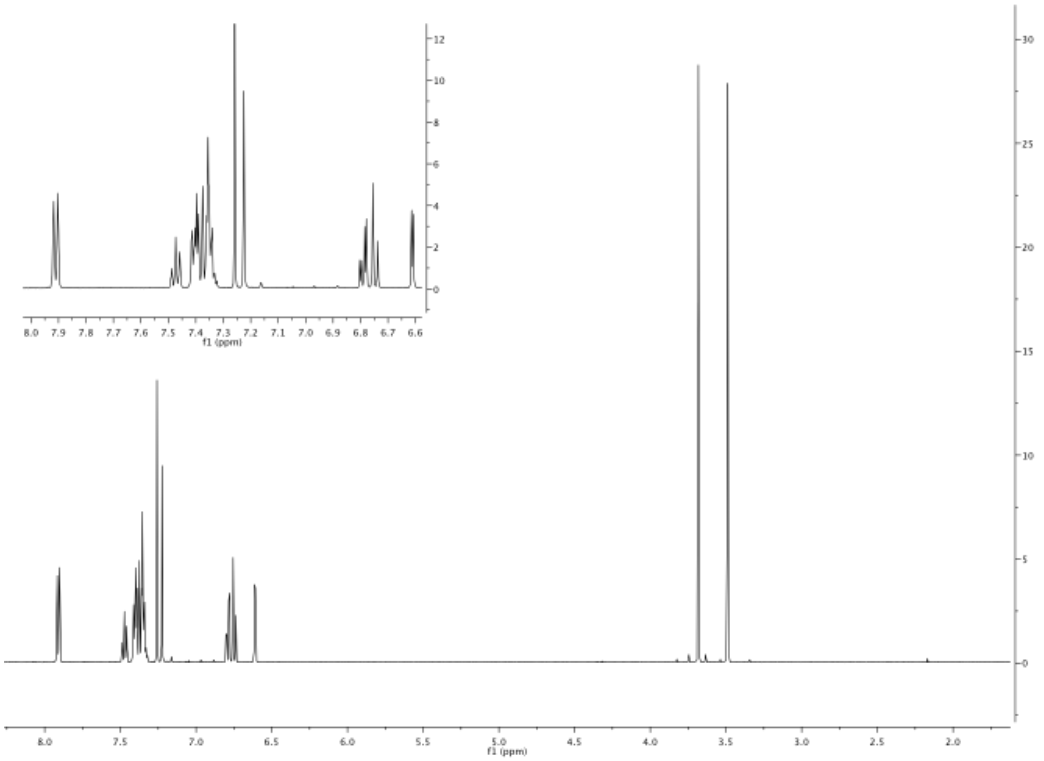


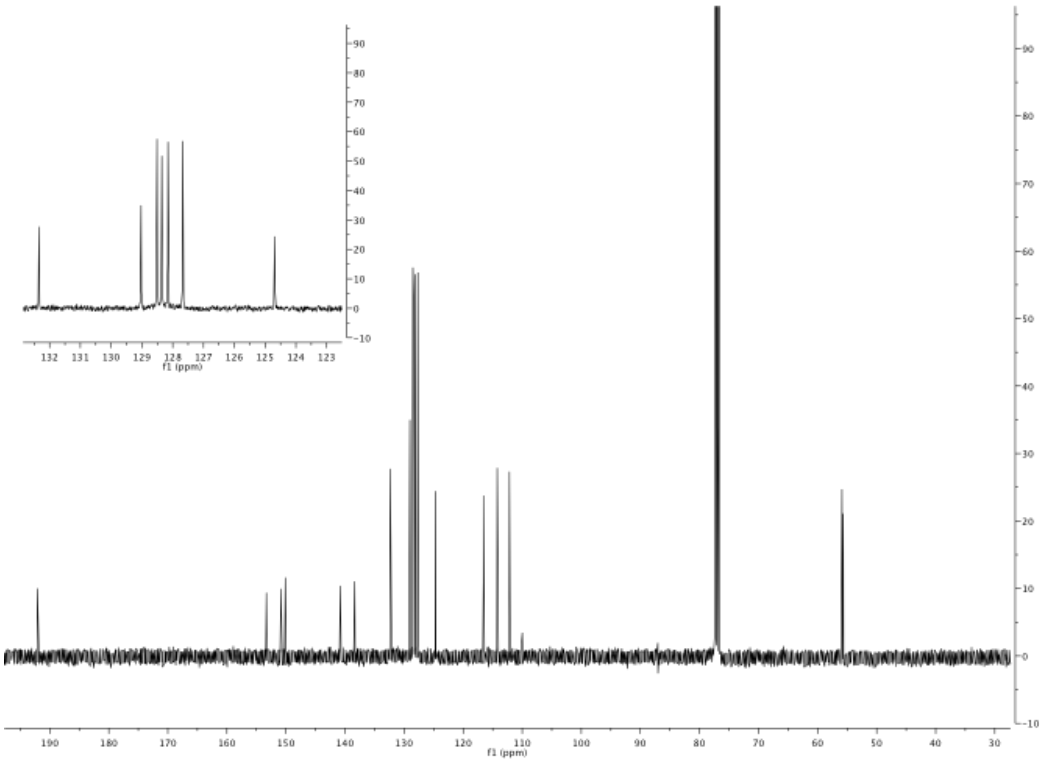




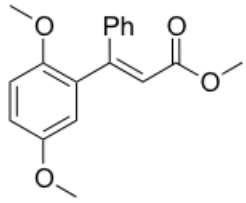
^{1}H and ^{13}C NMR (500 and 100 MHz, respectively in $CDCl_3$) of (*Z*)-3-(2,5-dimethoxyphenyl)-1,3-diphenylpropenone, **(*Z*)-3f**.

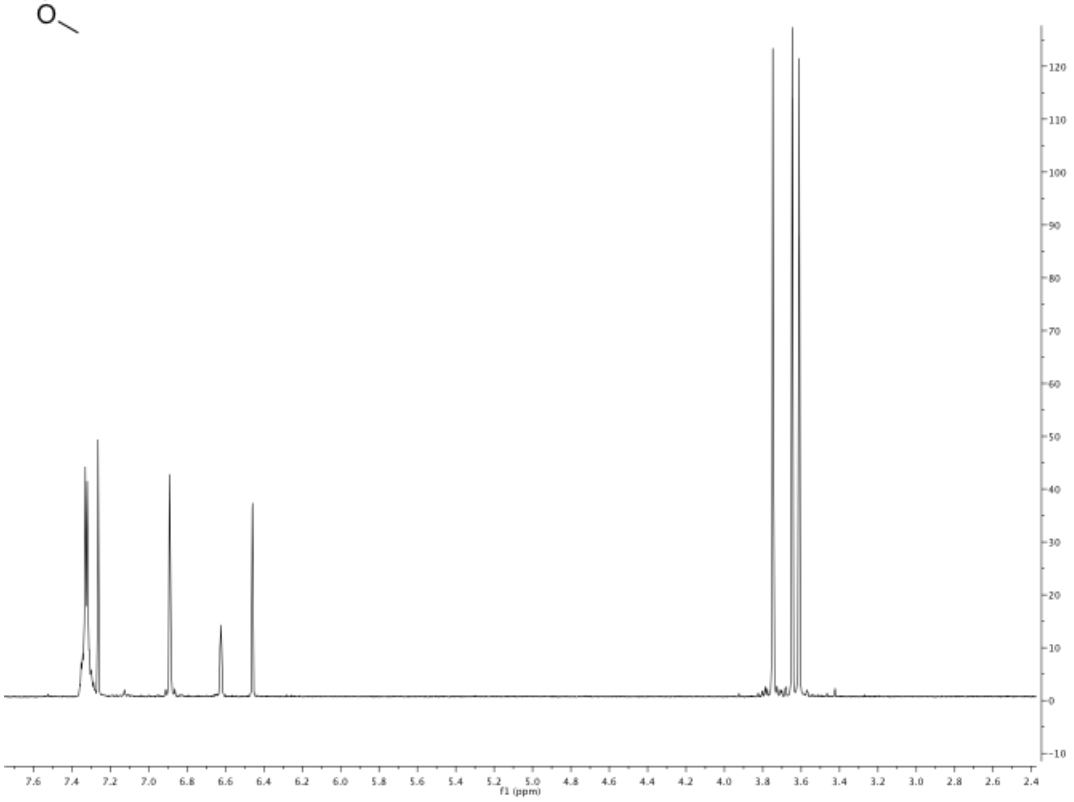


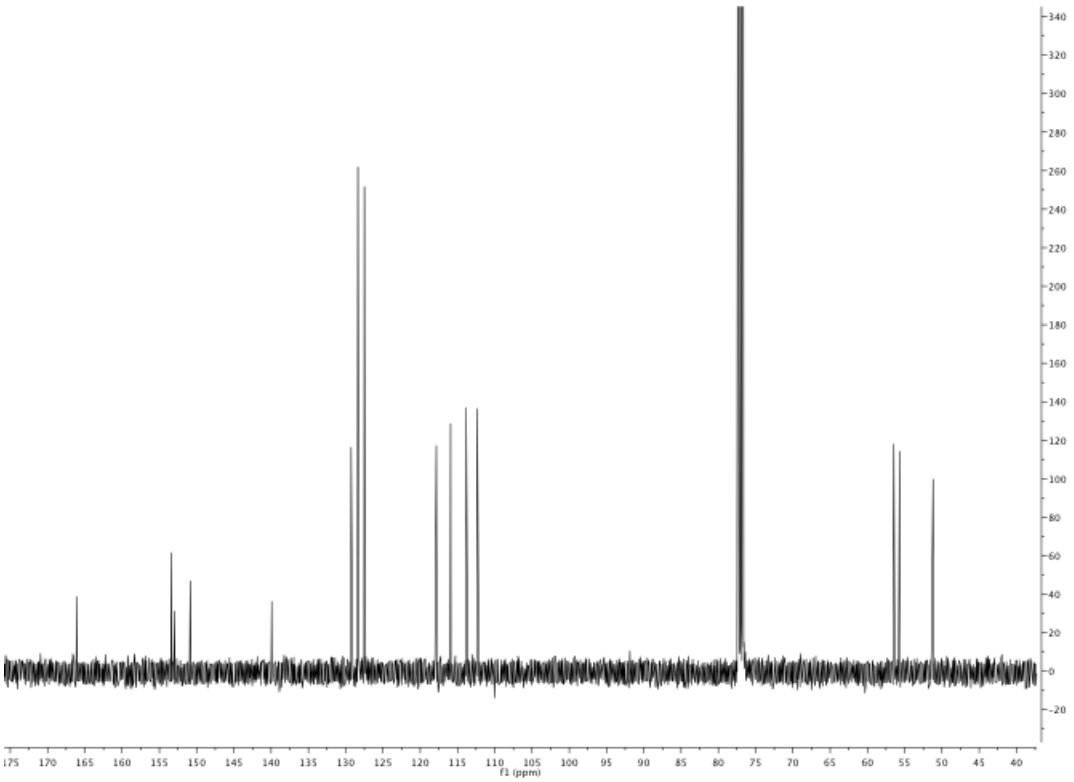




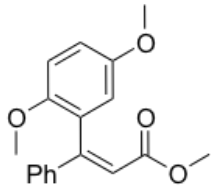
^{1}H and ^{13}C NMR (500 and 100 MHz, respectively in $CDCl_3$) of (*E*)-(2,5-dimethoxyphenyl)-3-phenylacrylic acid methyl ester, **(*E*)-3g**.

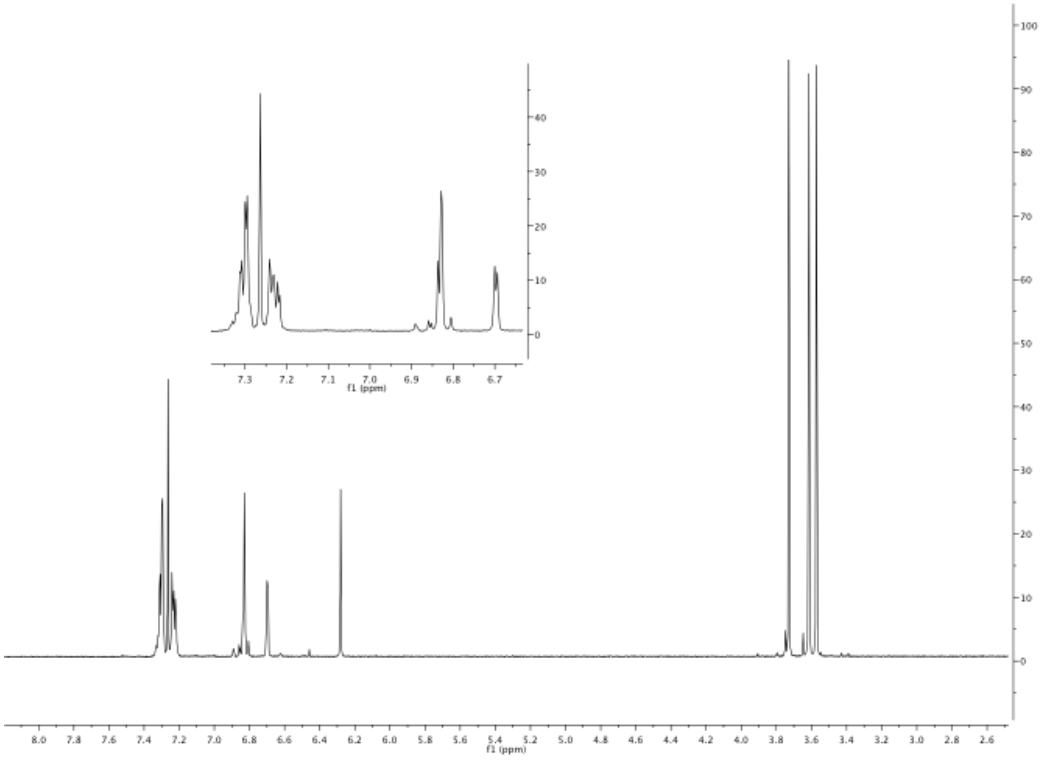


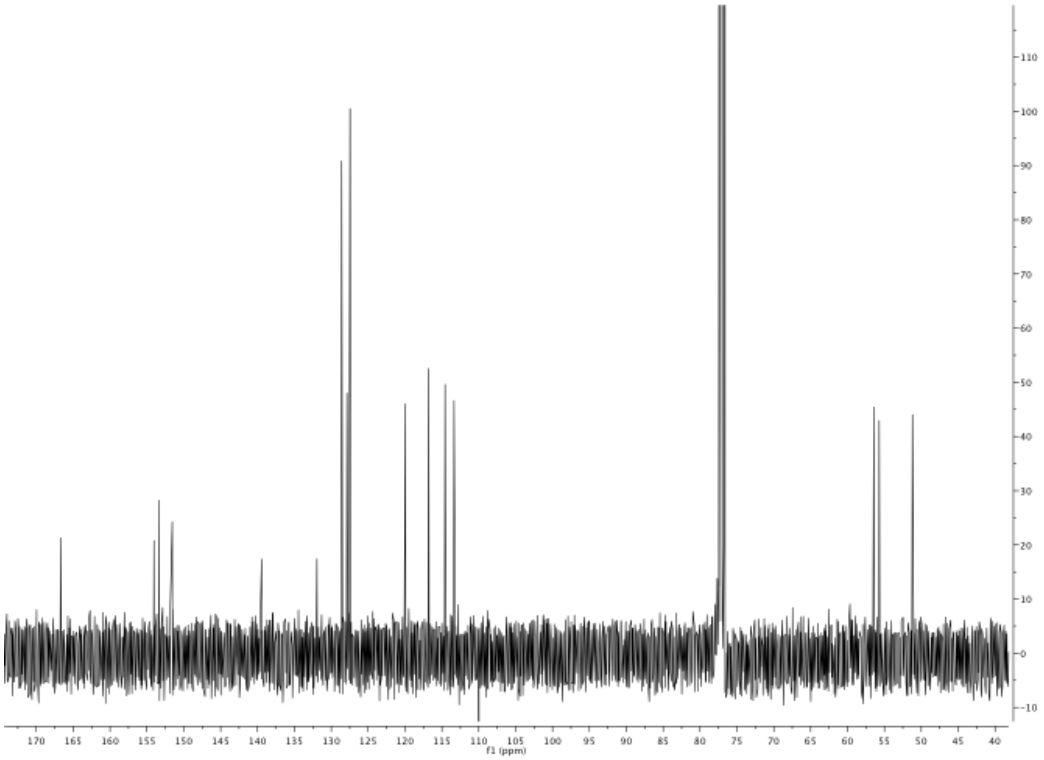




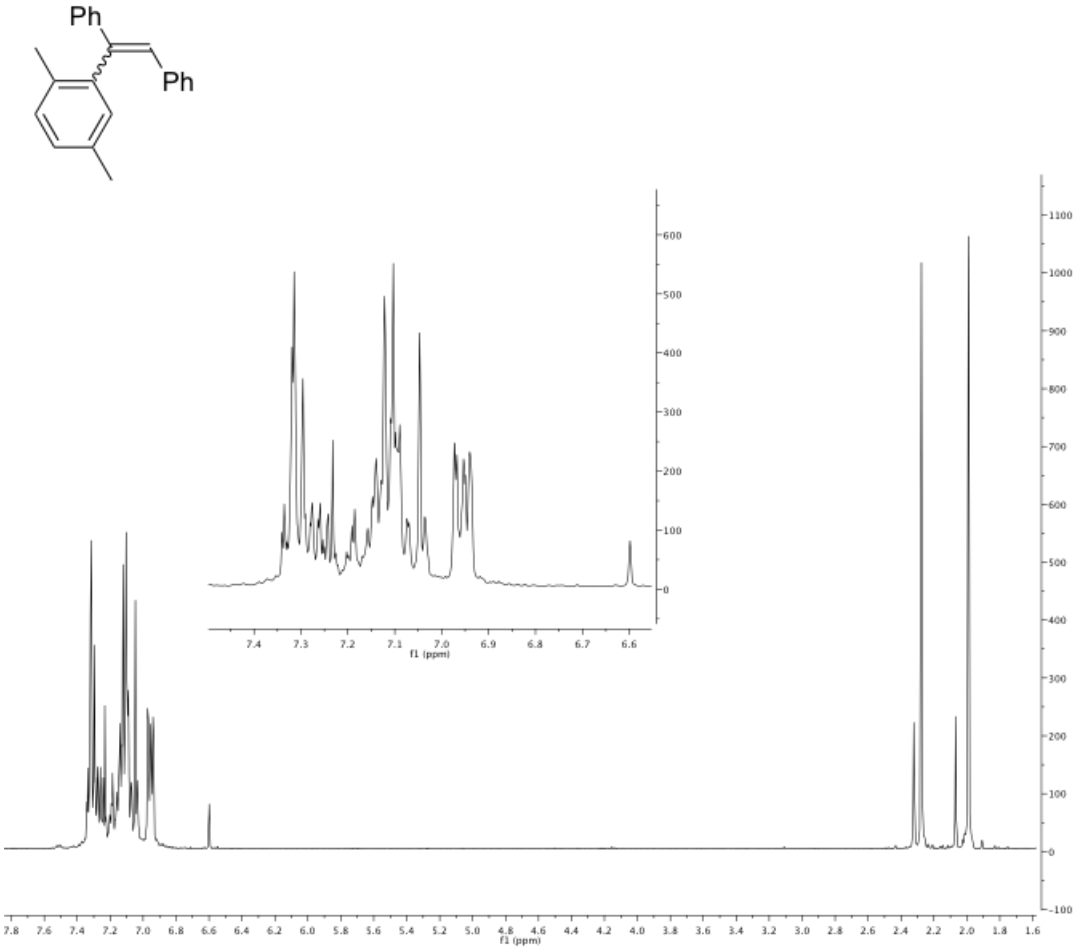
^{1}H and ^{13}C NMR (500 and 100 MHz, respectively in $CDCl_3$) of (*Z*)-(2,5-dimethoxyphenyl)-3-phenylacrylic acid methyl ester, **(Z)-3g**

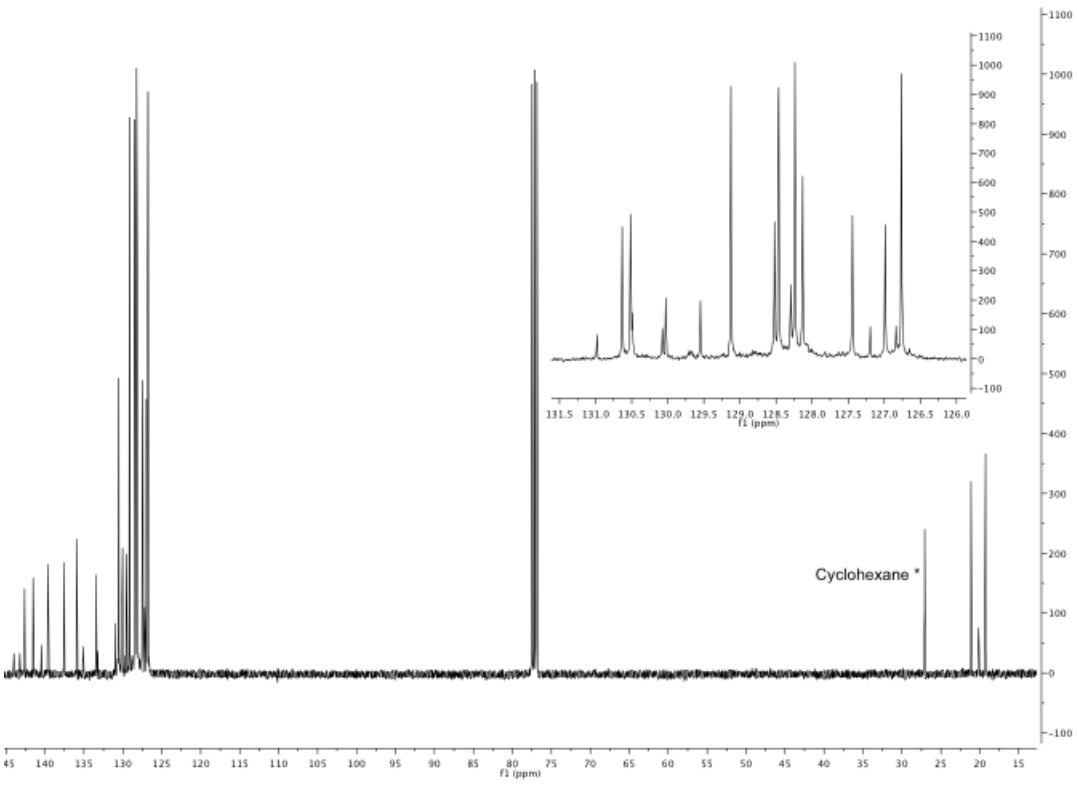




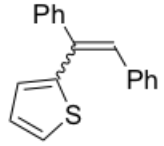


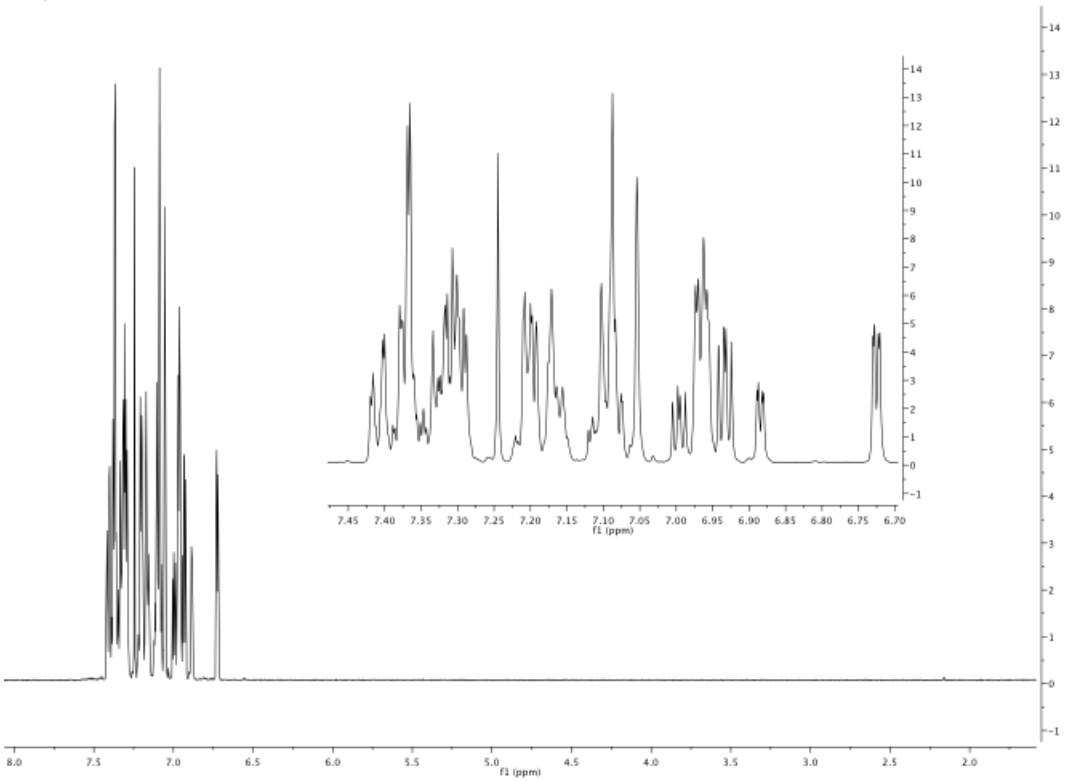
^{1}H and ^{13}C NMR (500 and 100 MHz, respectively in $CDCl_3$) of (*E*/*Z*)-1-(2,5-dimethylphenyl)-1,2-diphenylethene, **3h**.

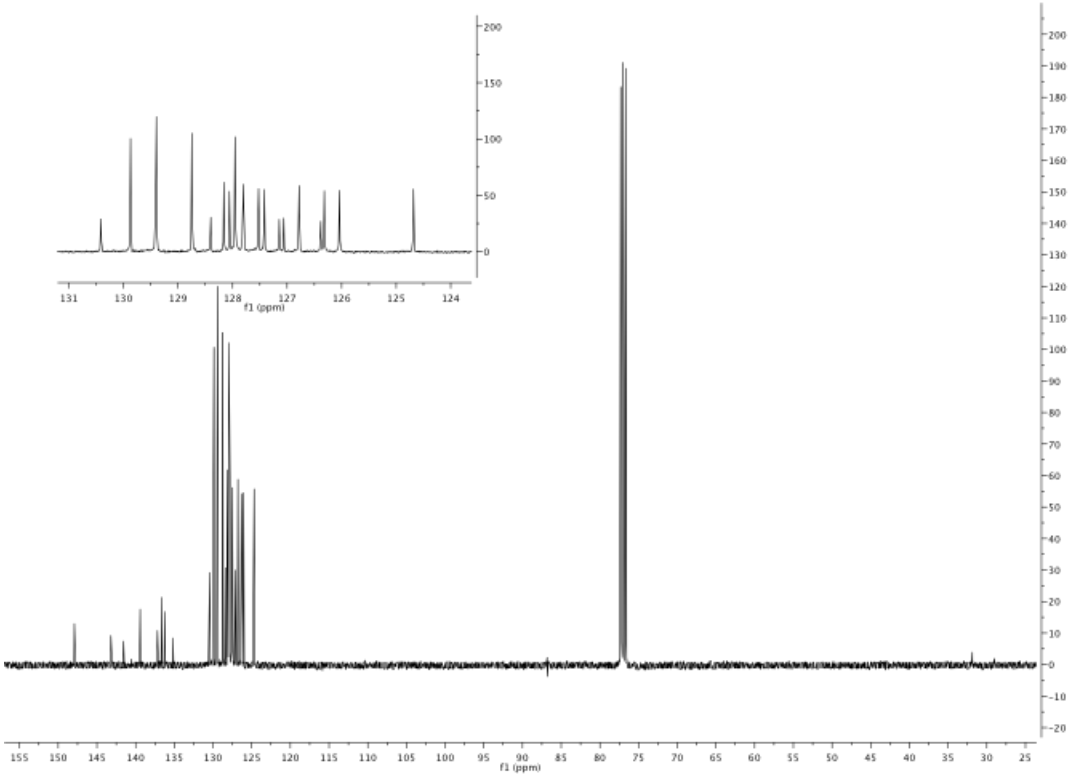




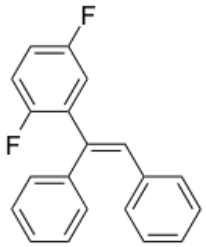
^{1}H and ^{13}C NMR (500 and 100 MHz, respectively in $CDCl_3$) of *(E/Z)*-2-(1,2-diphenylethenyl)thiophene, **3i**.

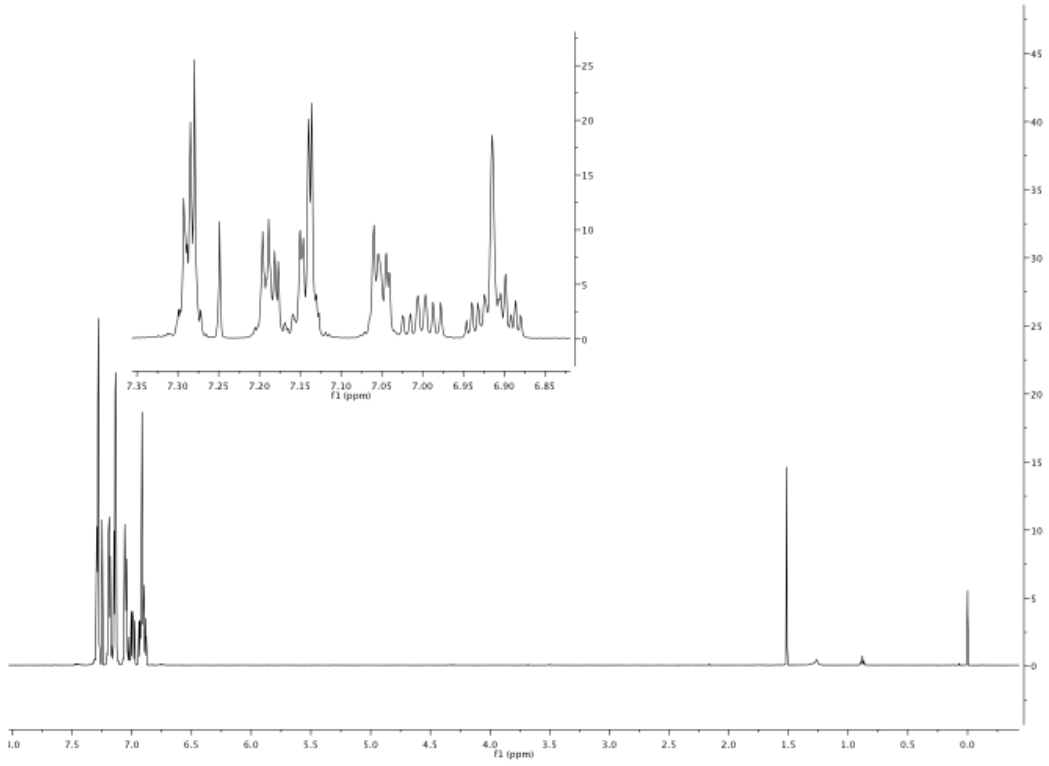


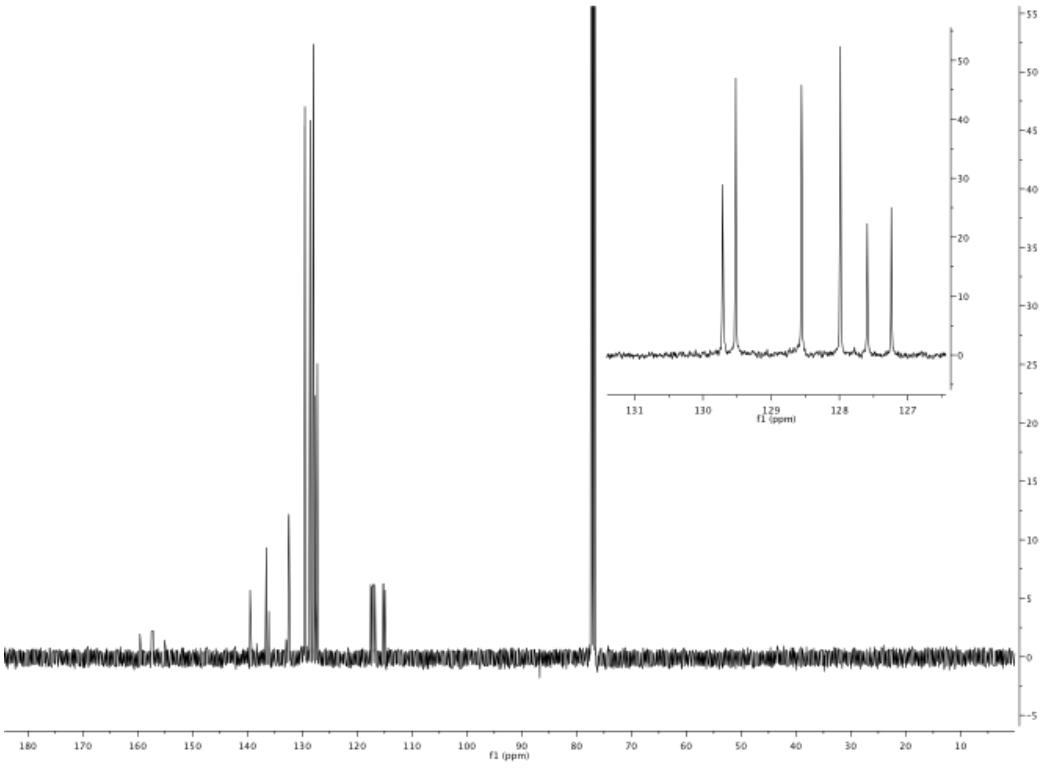


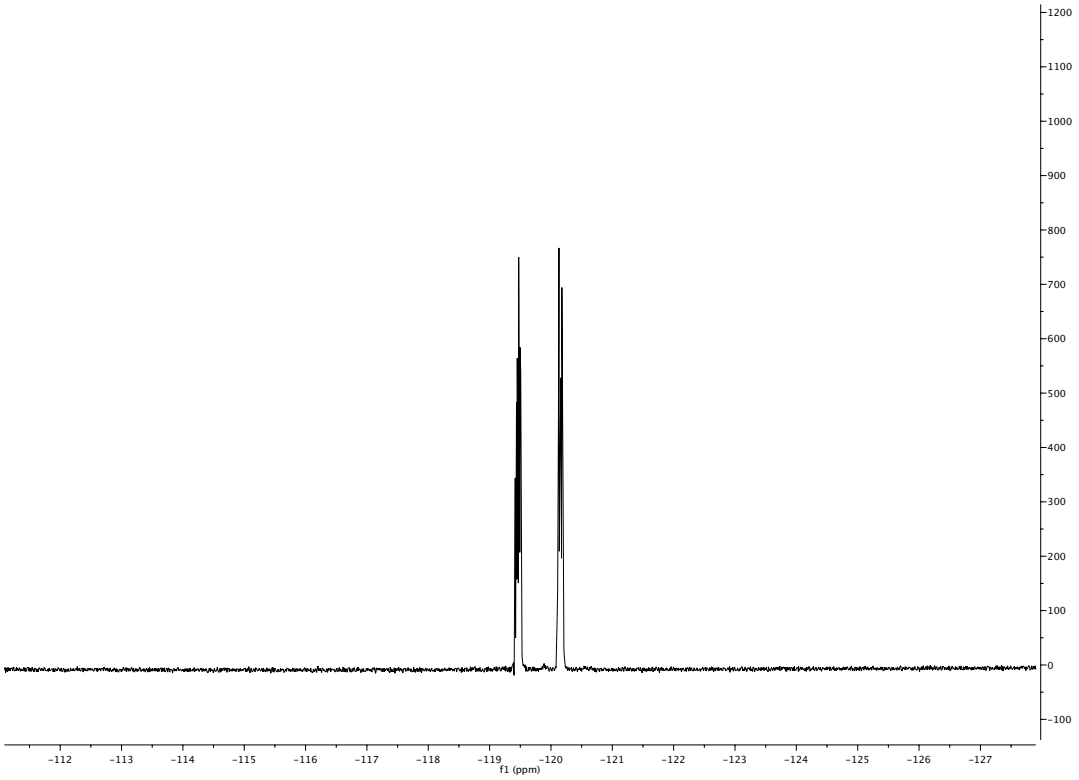


^{1}H, ^{13}C and ^{19}F NMR (500, 100 and 376 MHz, respectively in $CDCl_3$) of (*E*)-(1-(2,5-difluorophenyl)ethene-1,2-diyl)dibenzene, **(*E*)-3j**.

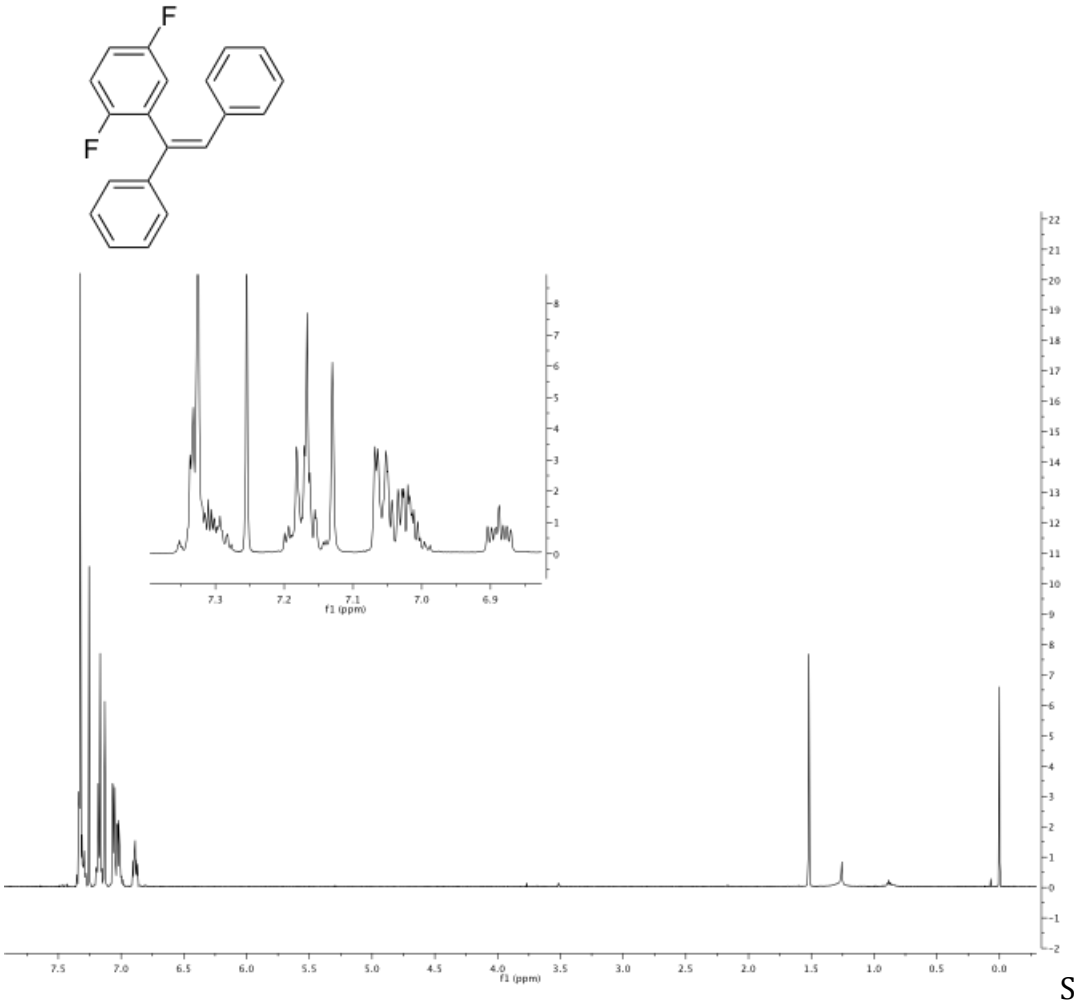


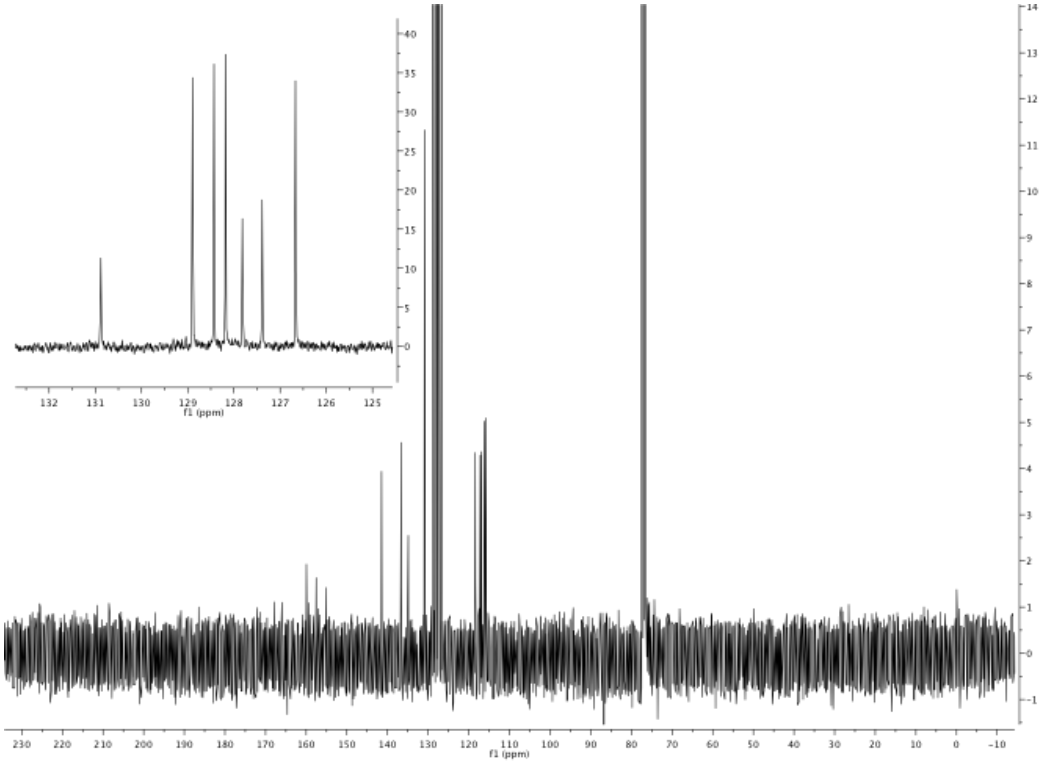


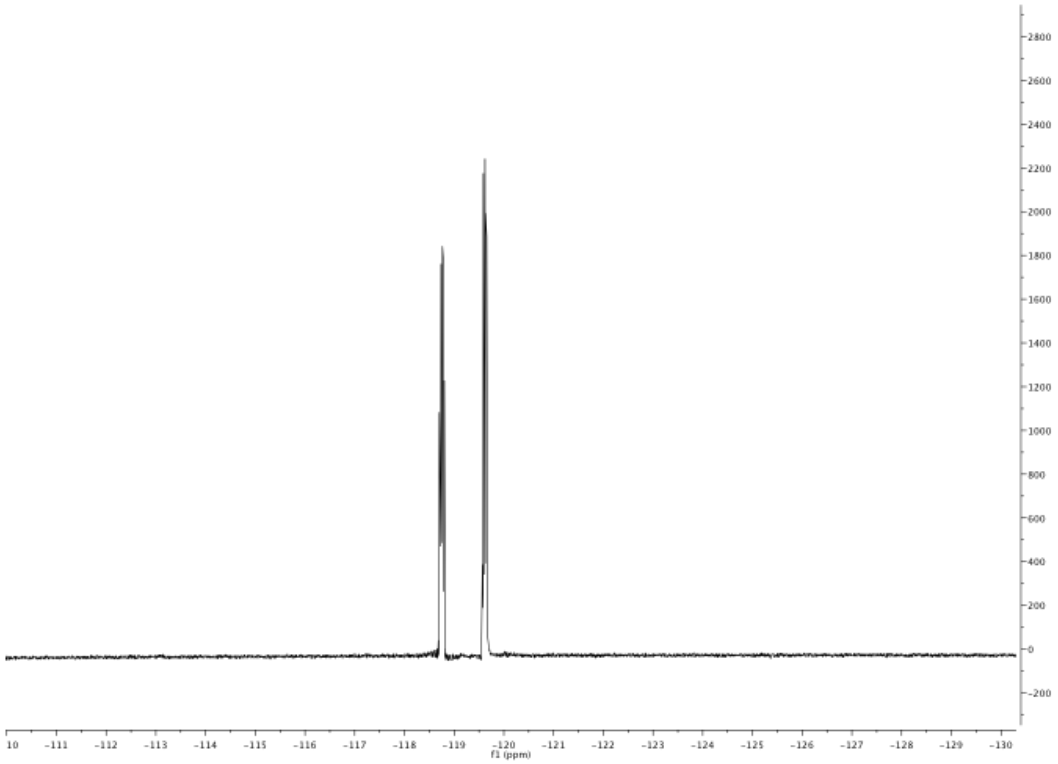




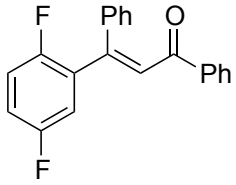
^{1}H, ^{13}C and ^{19}F NMR (500, 100 and 376 MHz, respectively in $CDCl_3$) of (*Z*)-(1-(2,5-difluorophenyl)ethene-1,2-diyl)dibenzene, **(*Z*)-3j**.

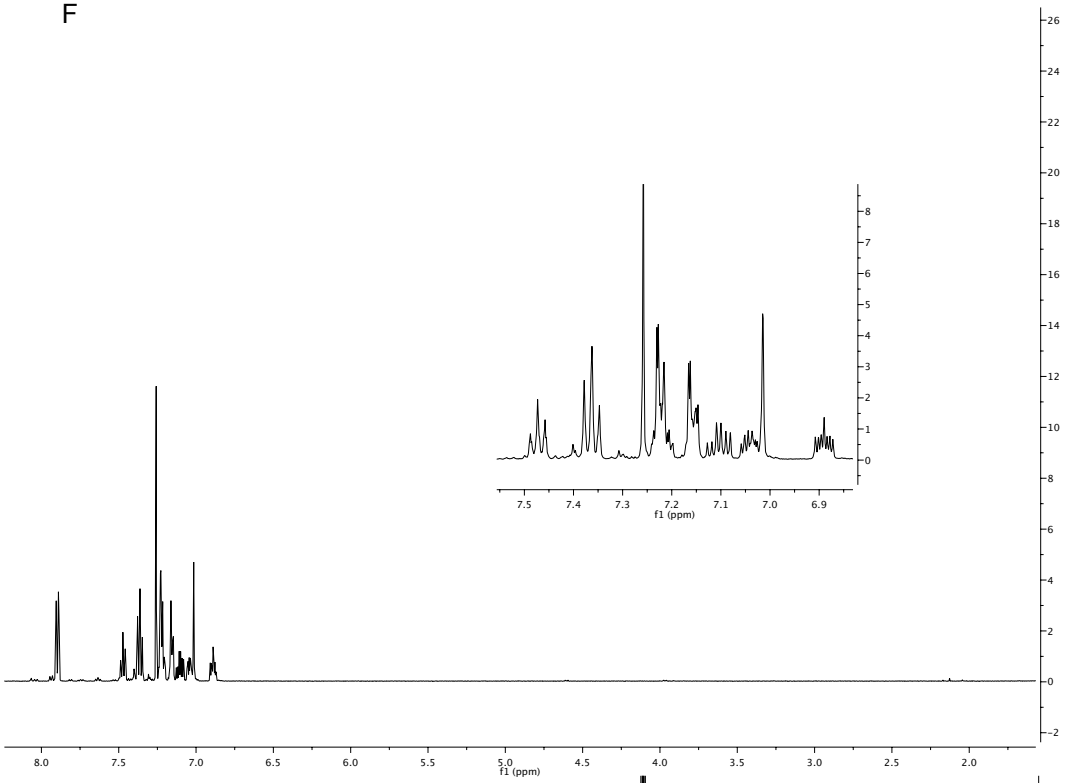


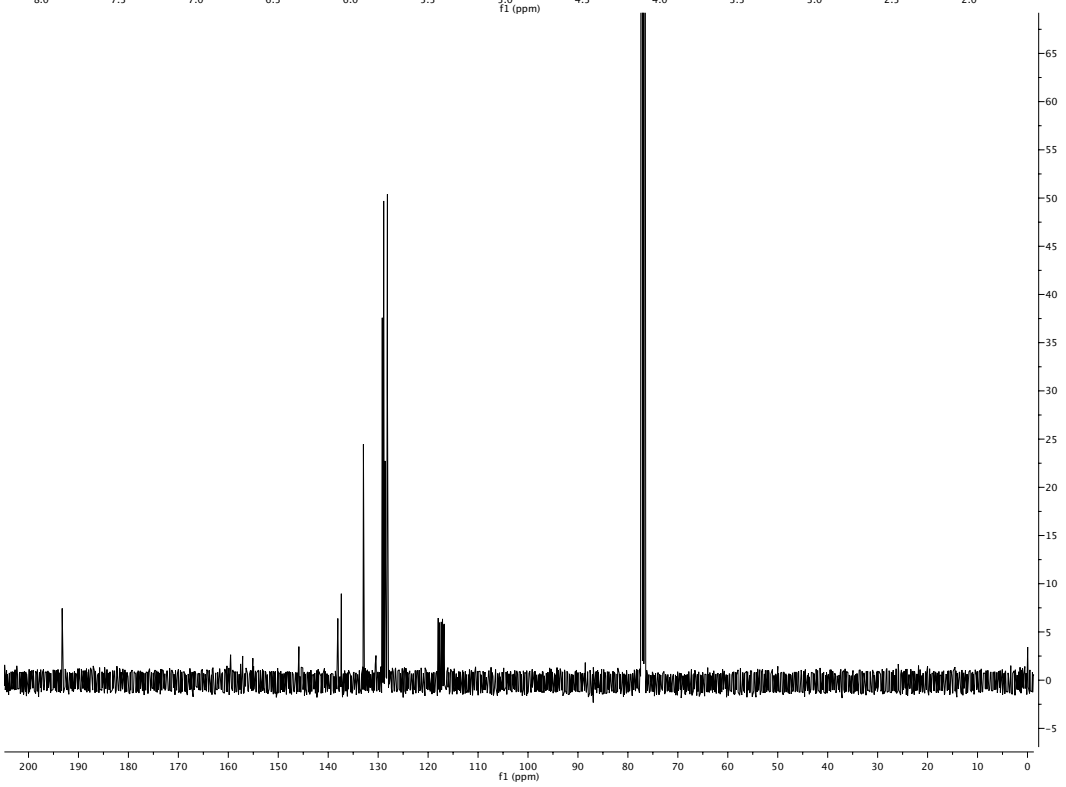

f1 (ppm)
f1 (ppm)

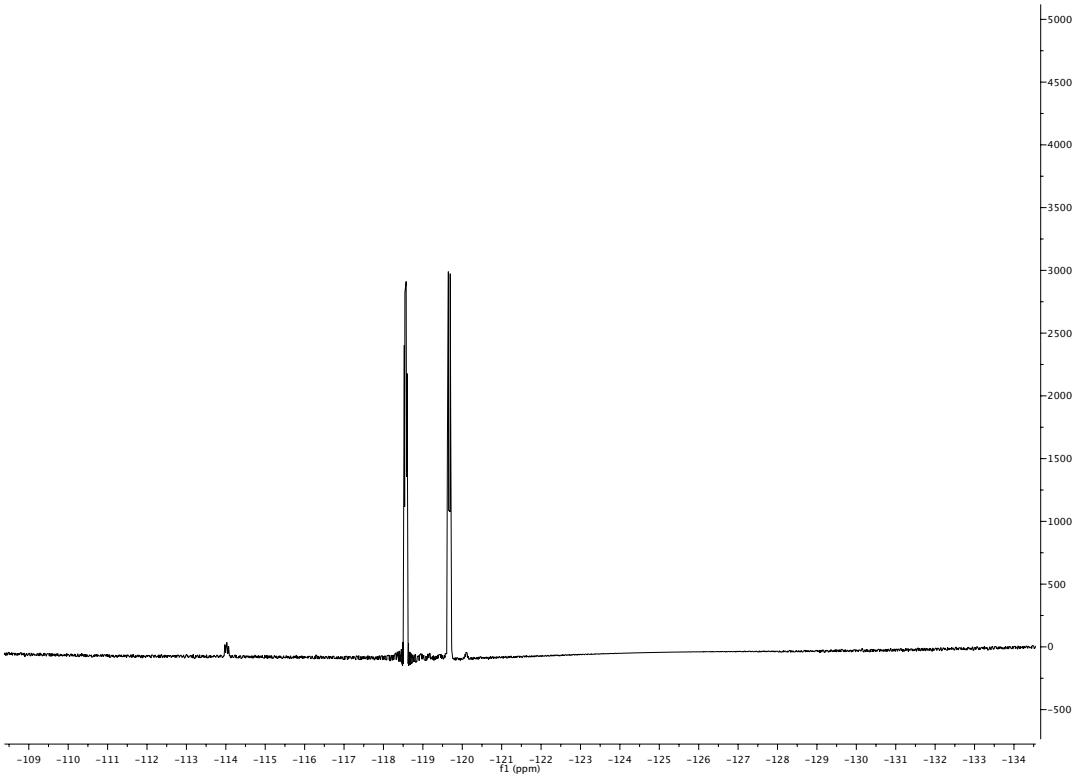

f1 (ppm)

^{1}H, ^{13}C and ^{19}F NMR (500, 100 and 376 MHz, respectively in $CDCl_3$) of (*E*)-3-(2,5-difluorophenyl)-1,3-diphenylpropenone, **(*E*)-3k**.

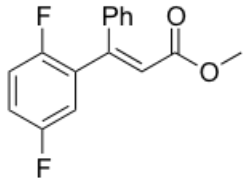


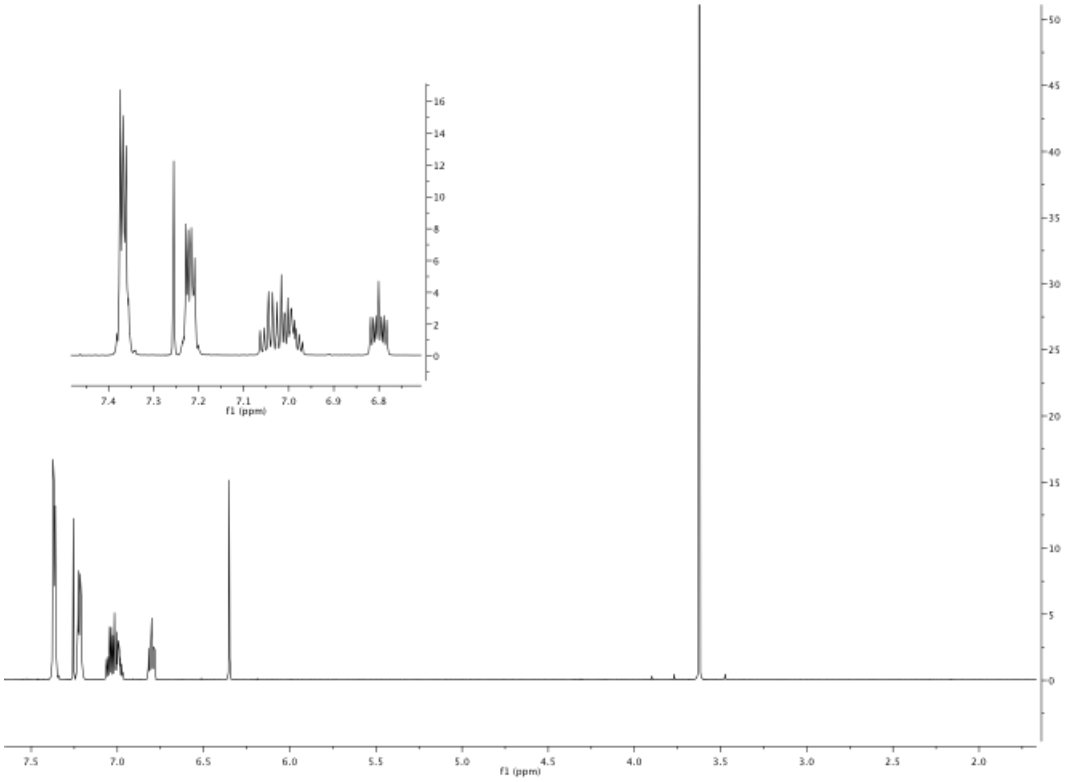


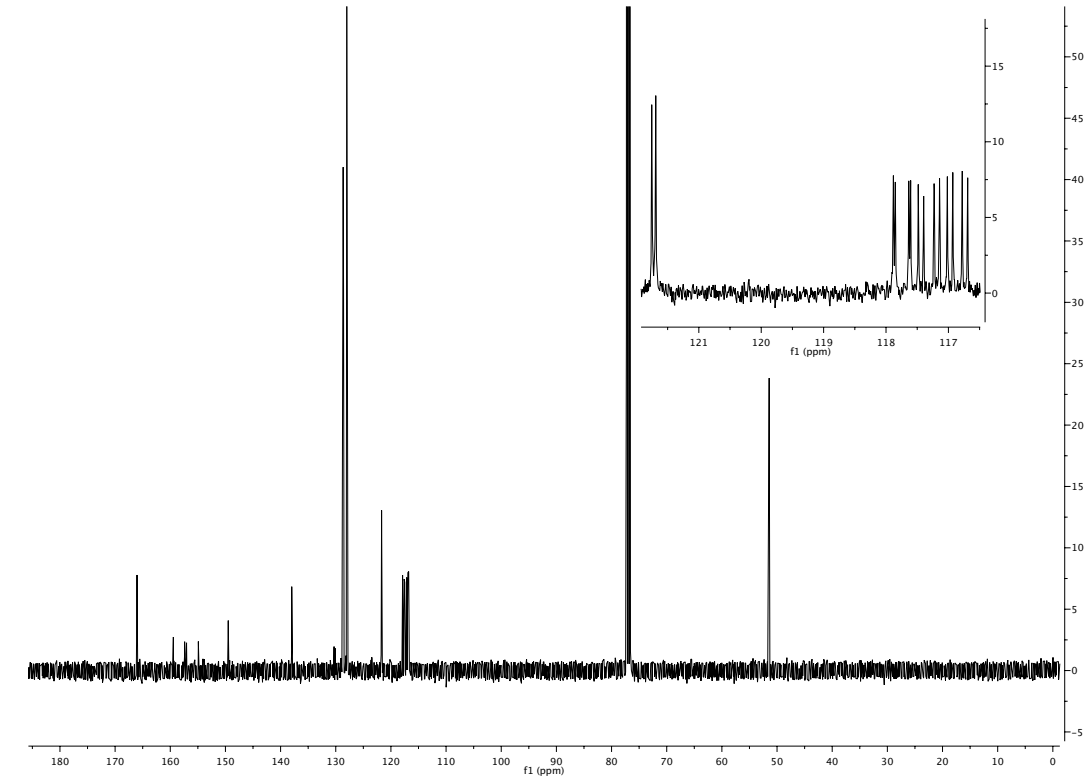


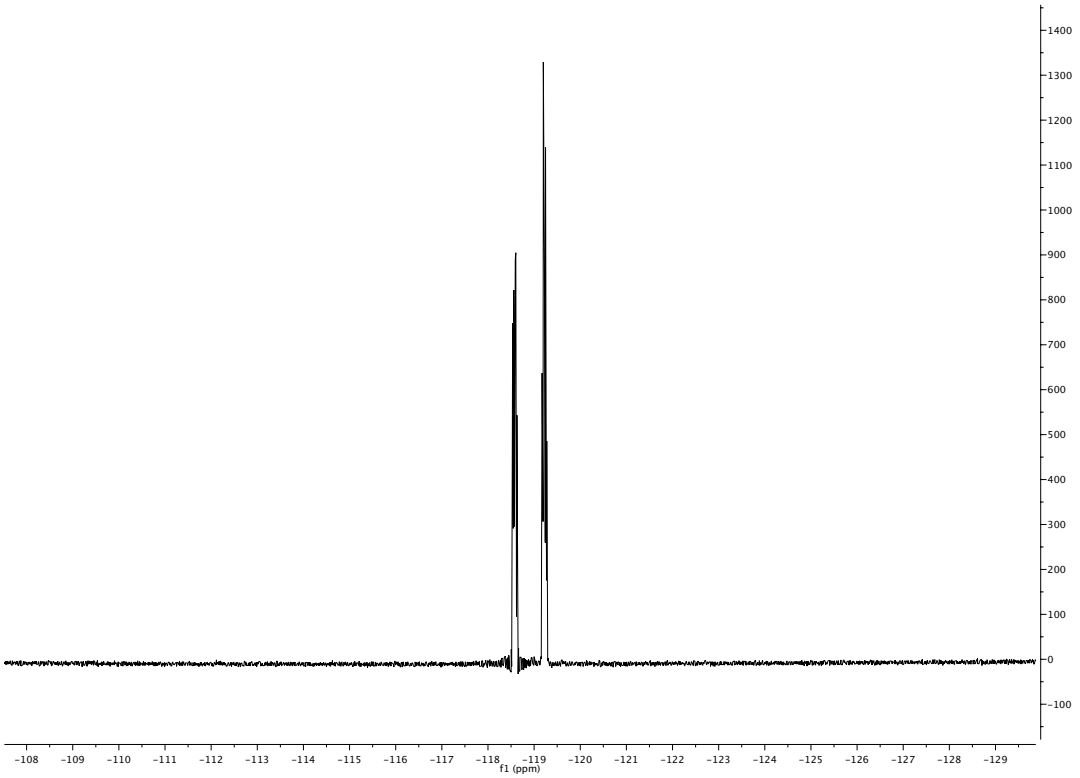


^{1}H, ^{13}C and ^{19}F NMR (500, 100 and 376 MHz, respectively in $CDCl_3$) of (*E*)-(2,5-difluorophenyl)-3-phenylacrylic acid methyl ester, **(*E*)-3l**.

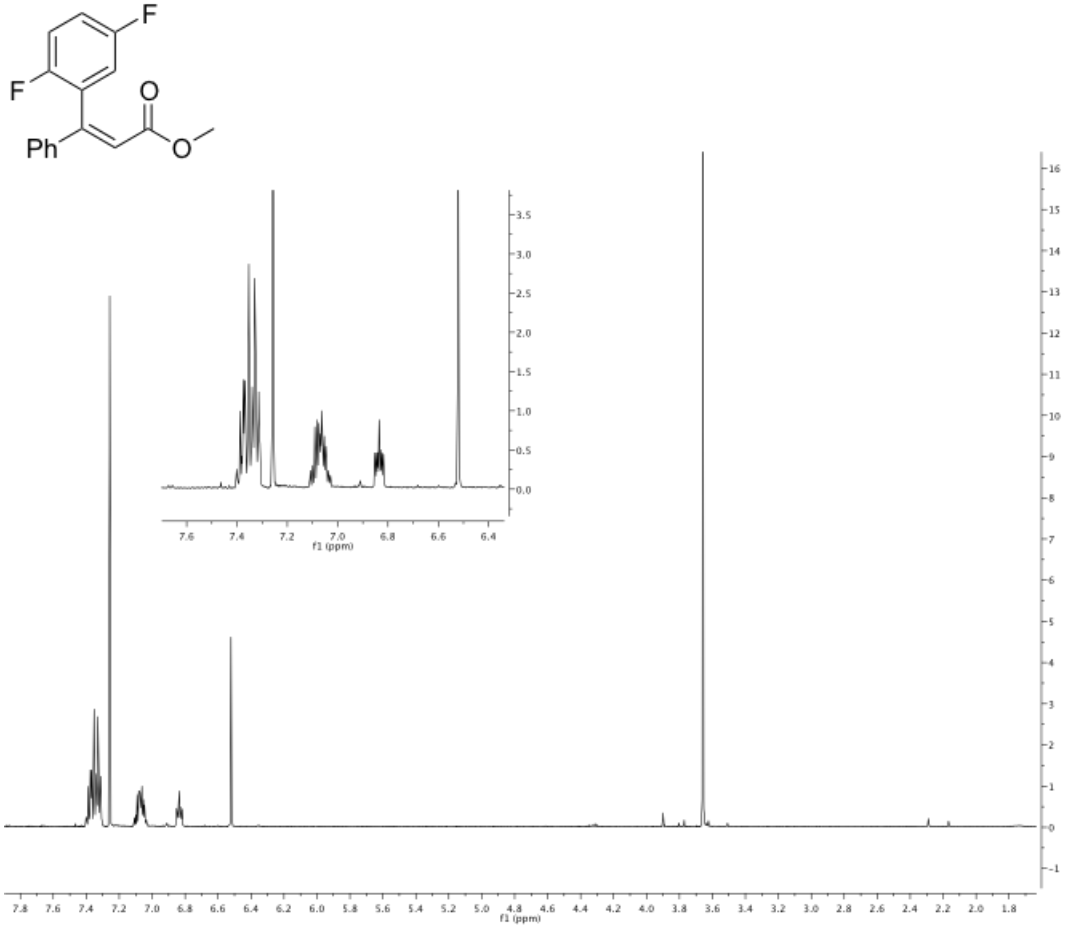


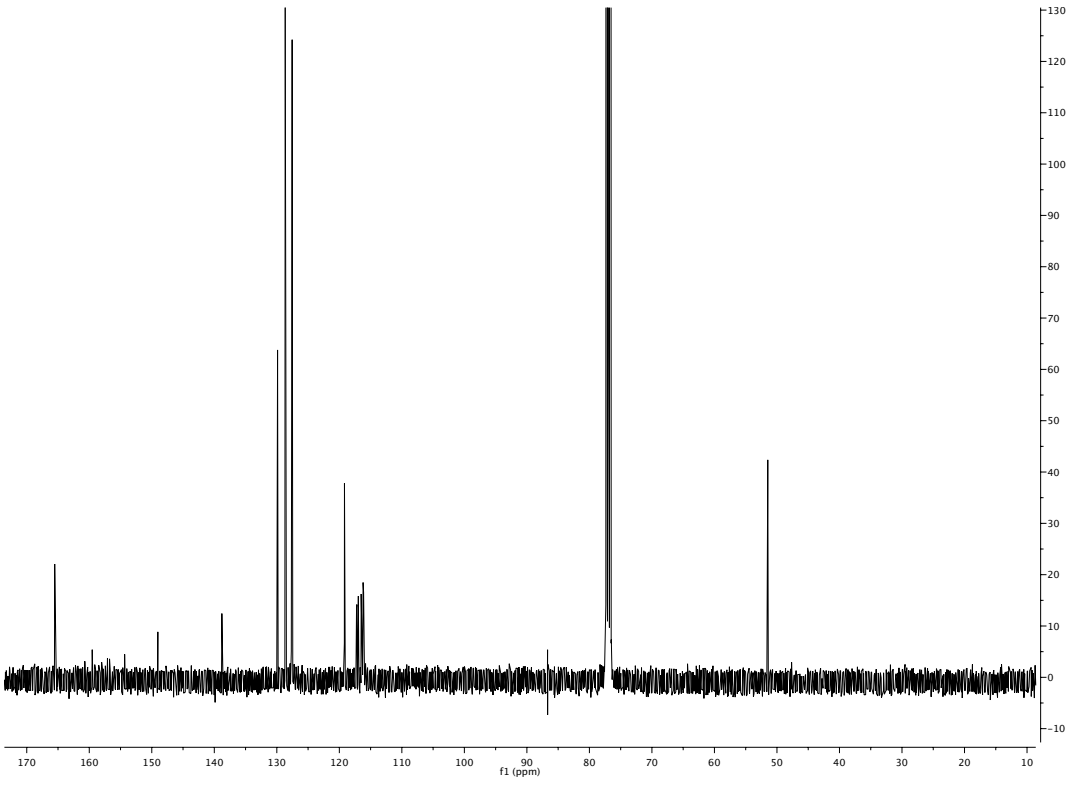


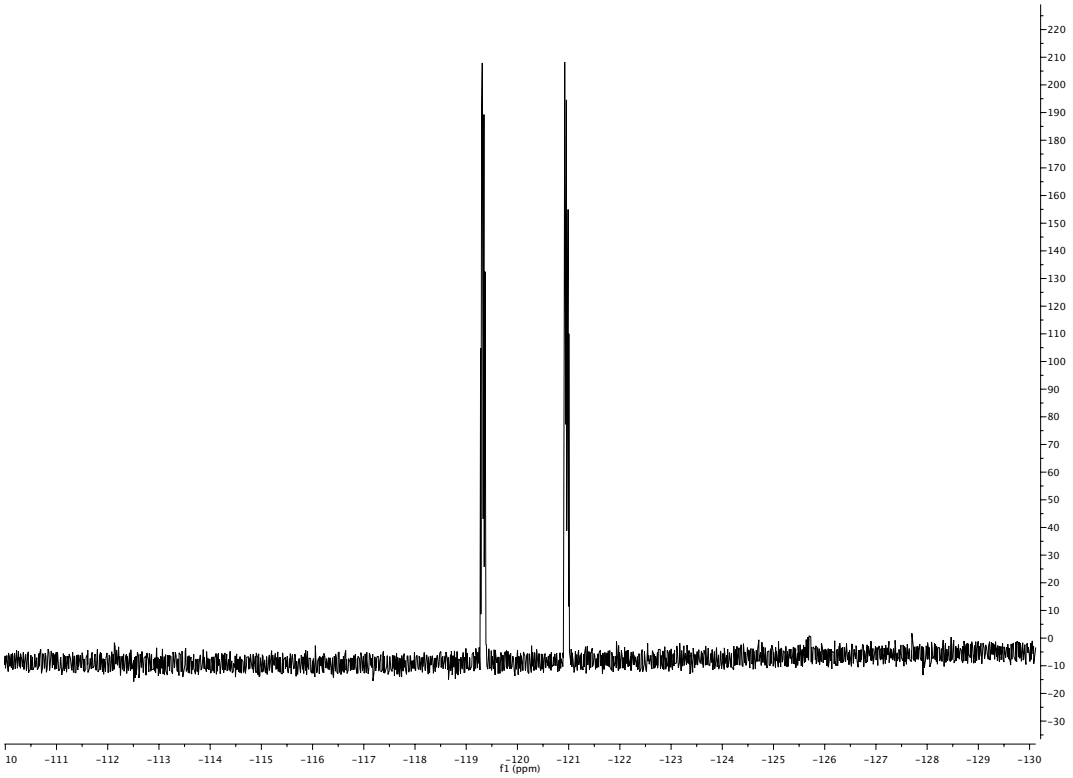

f1 (ppm)
f1 (ppm)


f1 (ppm)

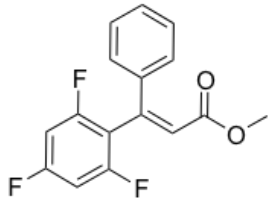
^{1}H, ^{13}C and ^{19}F NMR (500, 100 and 376 MHz, respectively in $CDCl_3$) of (*Z*)-(2,5-difluorophenyl)-3-phenylacrylic acid methyl ester, **(*Z*)-3l**.

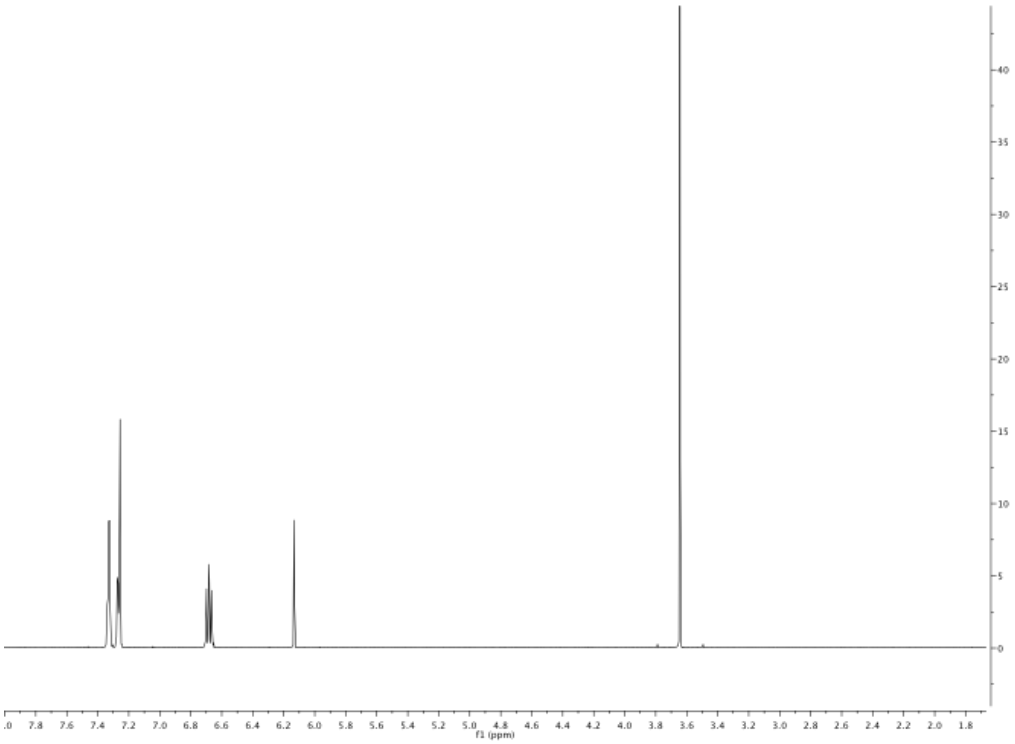


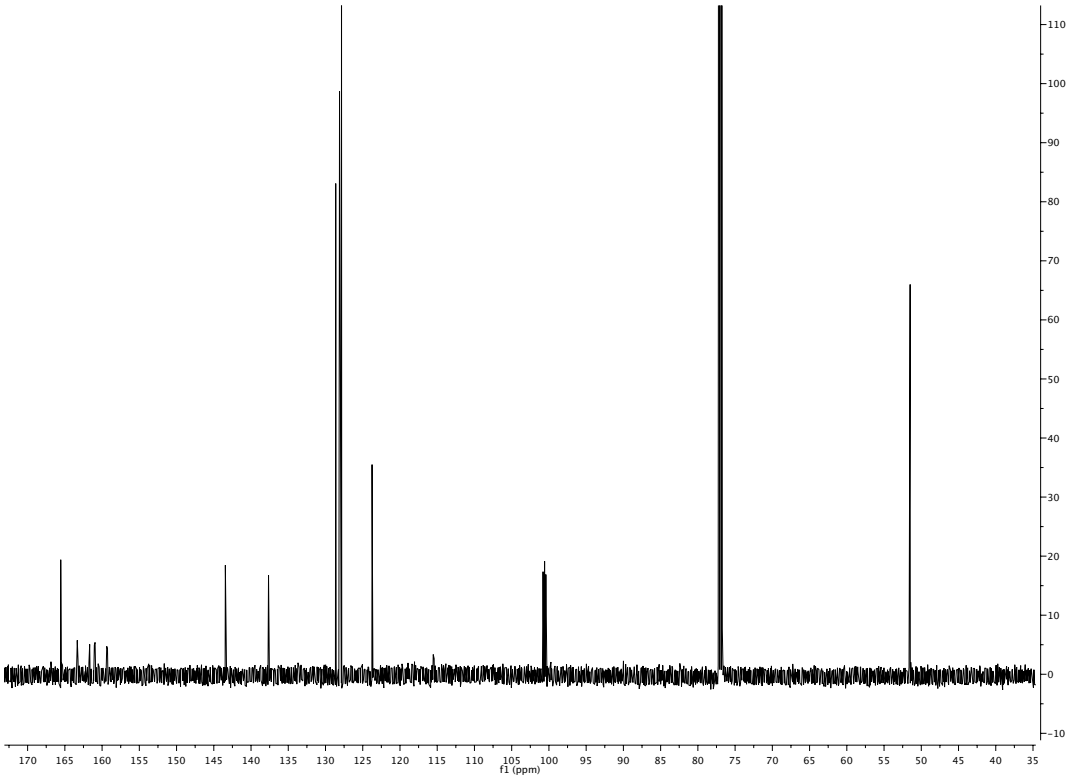


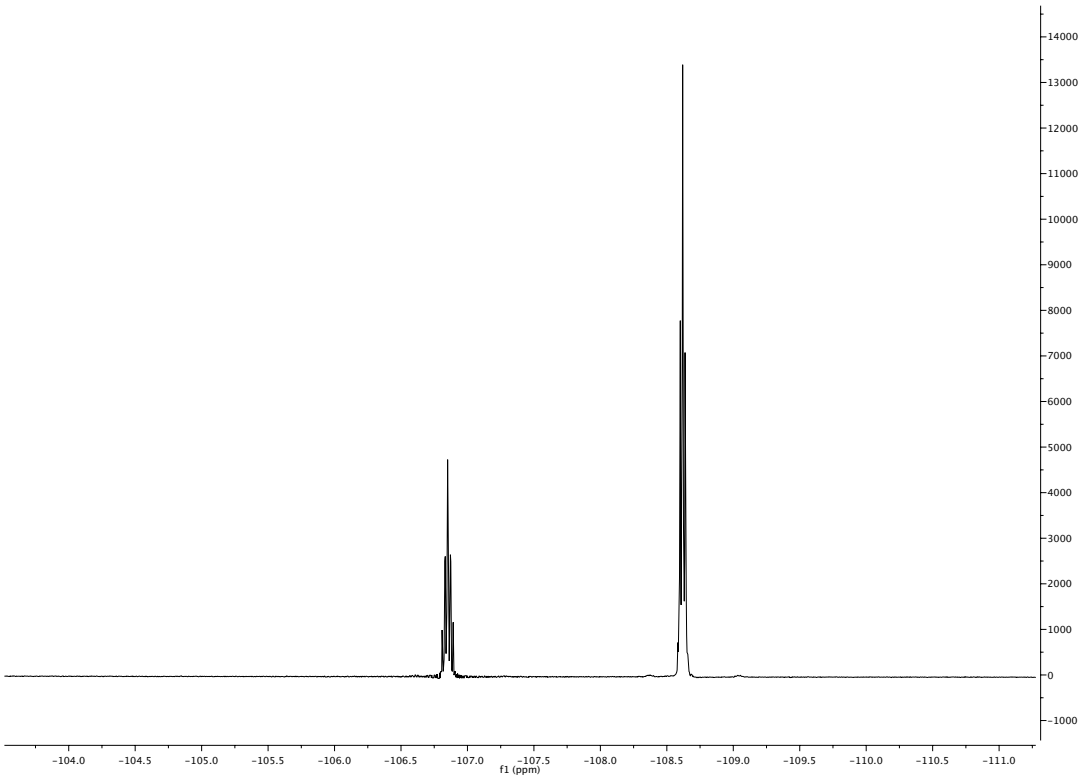


^{1}H, ^{13}C and ^{19}F NMR (500, 100 and 376 MHz, respectively in $CDCl_3$) of (*E*)-(2,4,6-trifluorophenyl)-3-phenylacrylic acid methyl ester, **(*E*)-3m**.

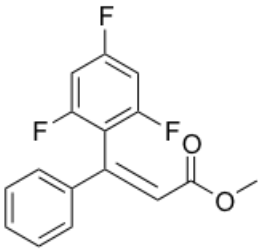


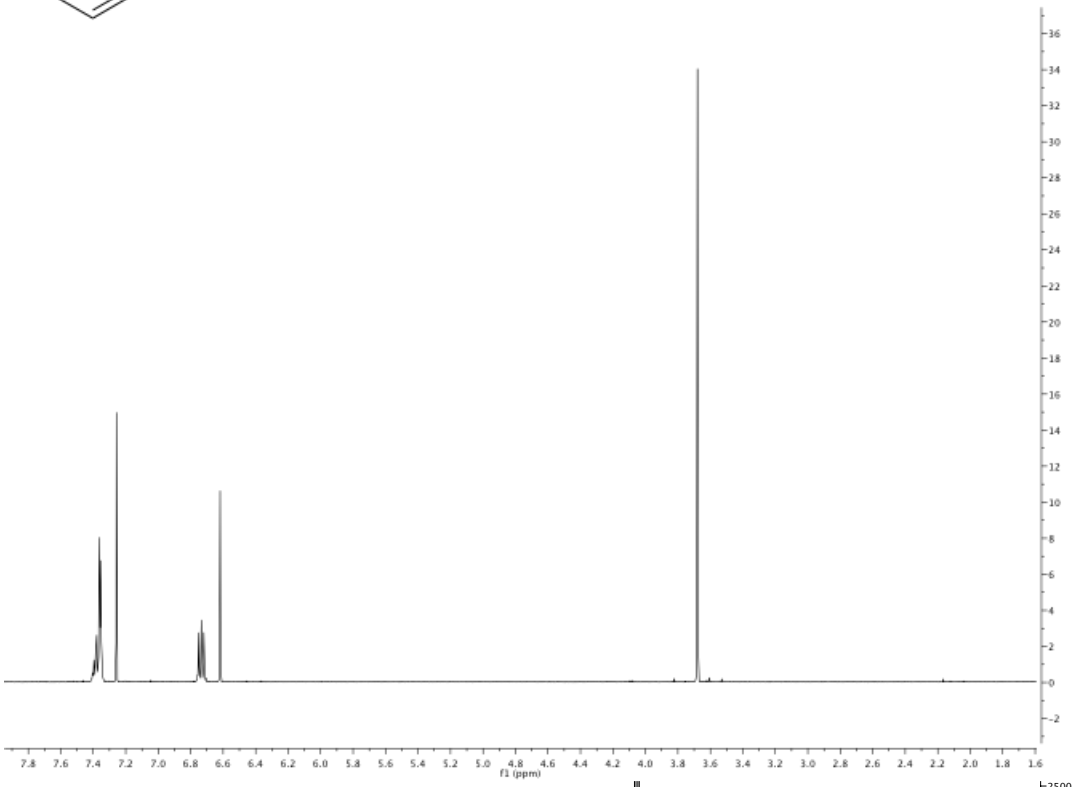


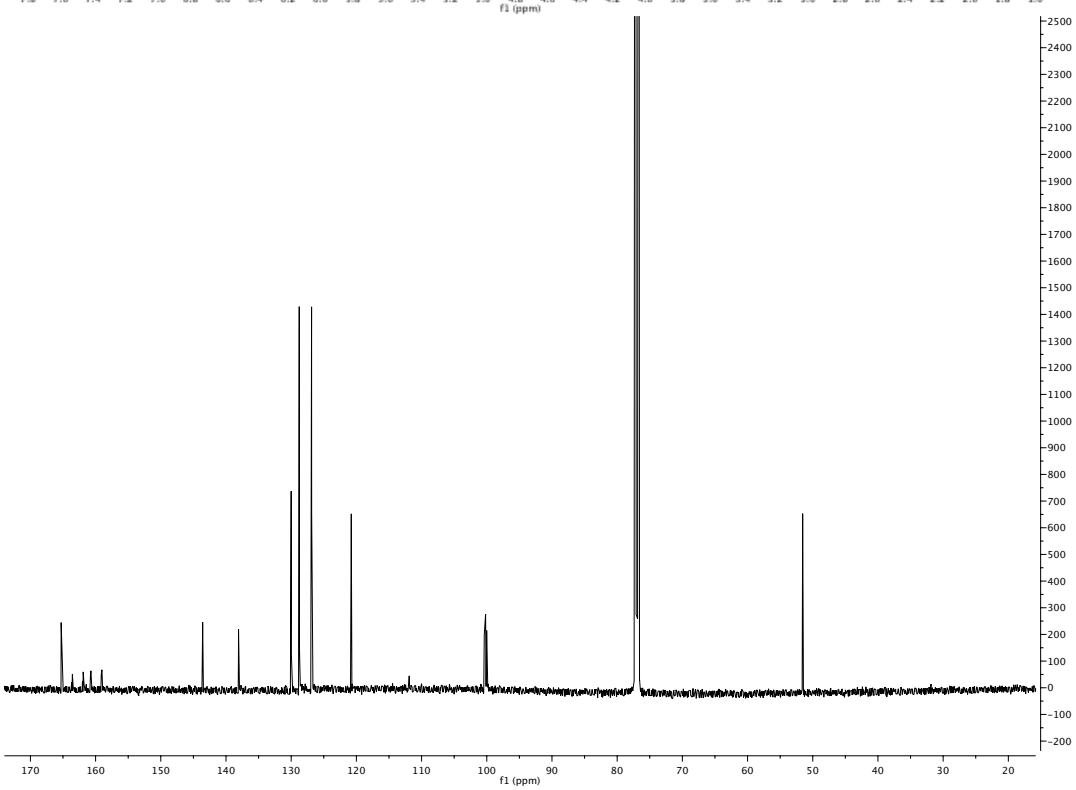

f1 (ppm)

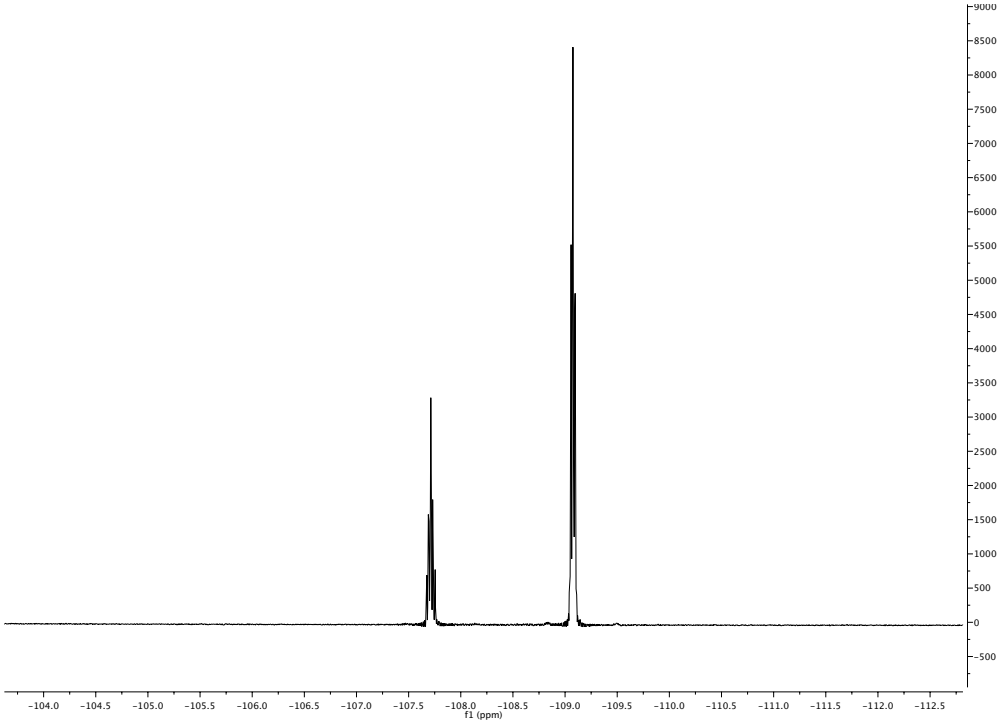

f1 (ppm)

^{1}H, ^{13}C and ^{19}F NMR (500, 100 and 376 MHz, respectively in $CDCl_3$) of (*Z*)-(2,4,6-trifluorophenyl)-3-phenylacrylic acid methyl ester, **(*Z*)-3m**.

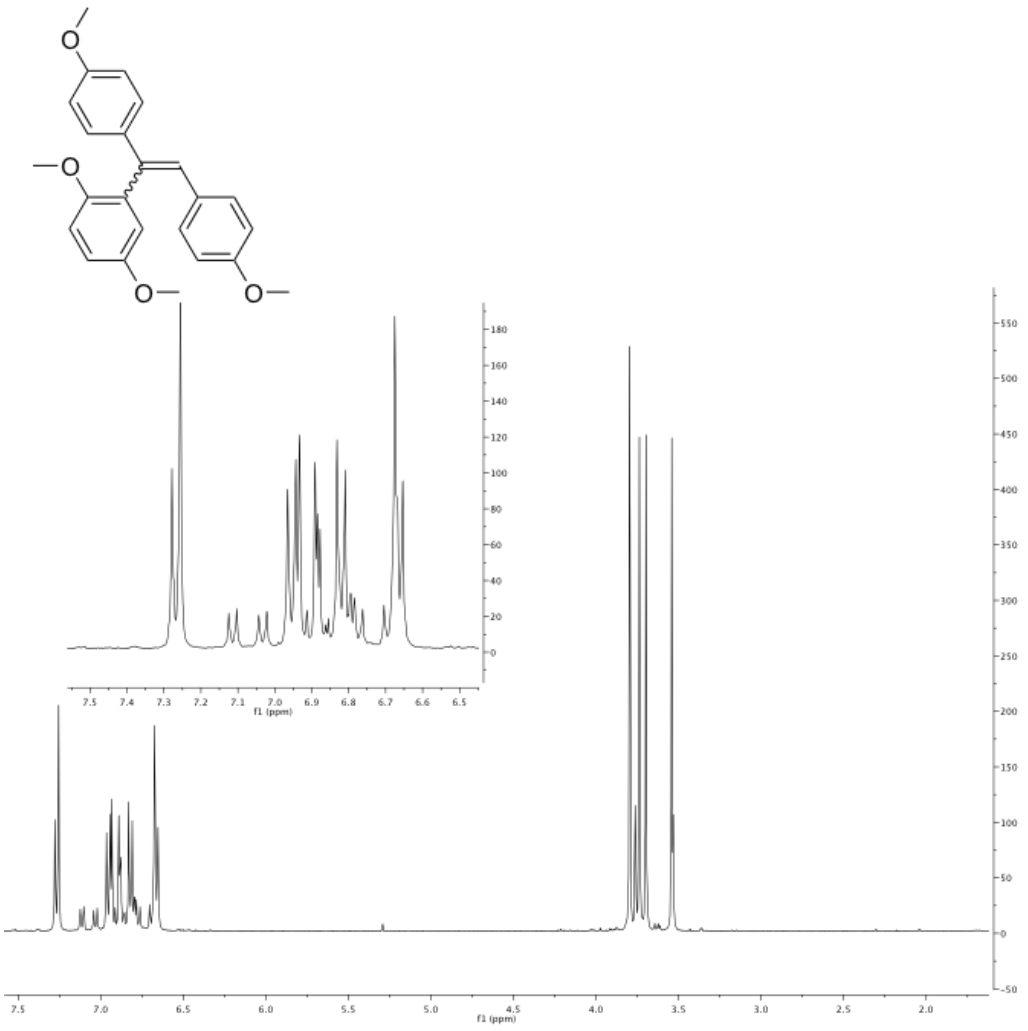


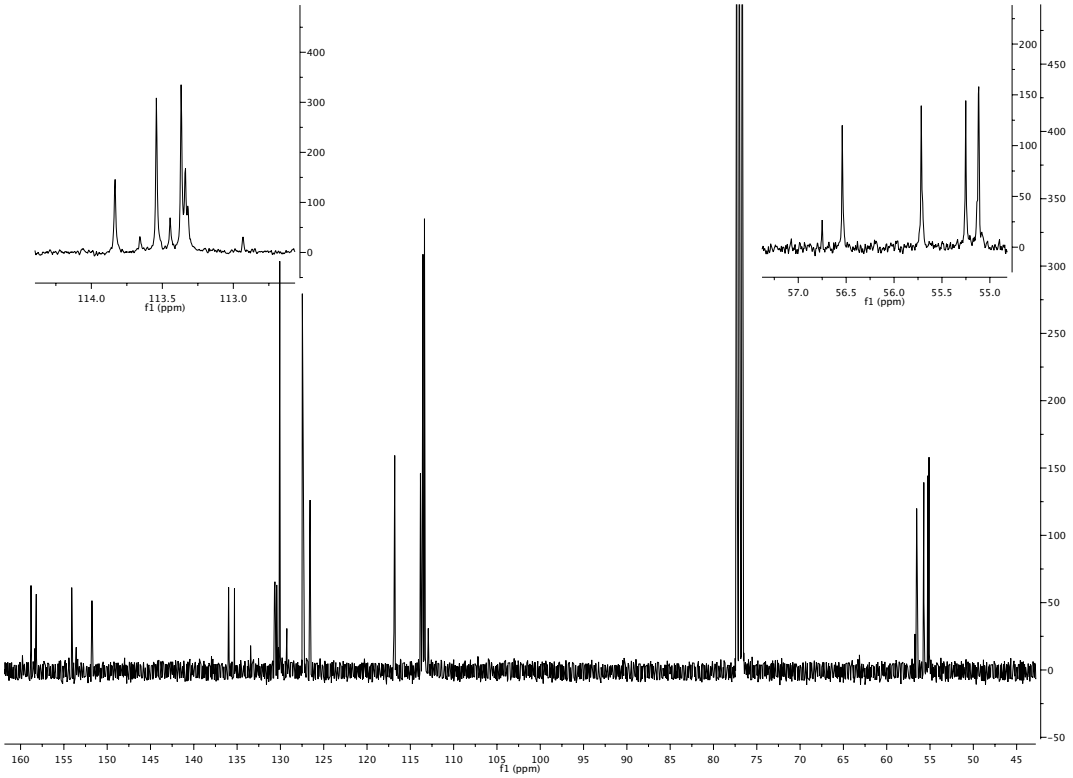




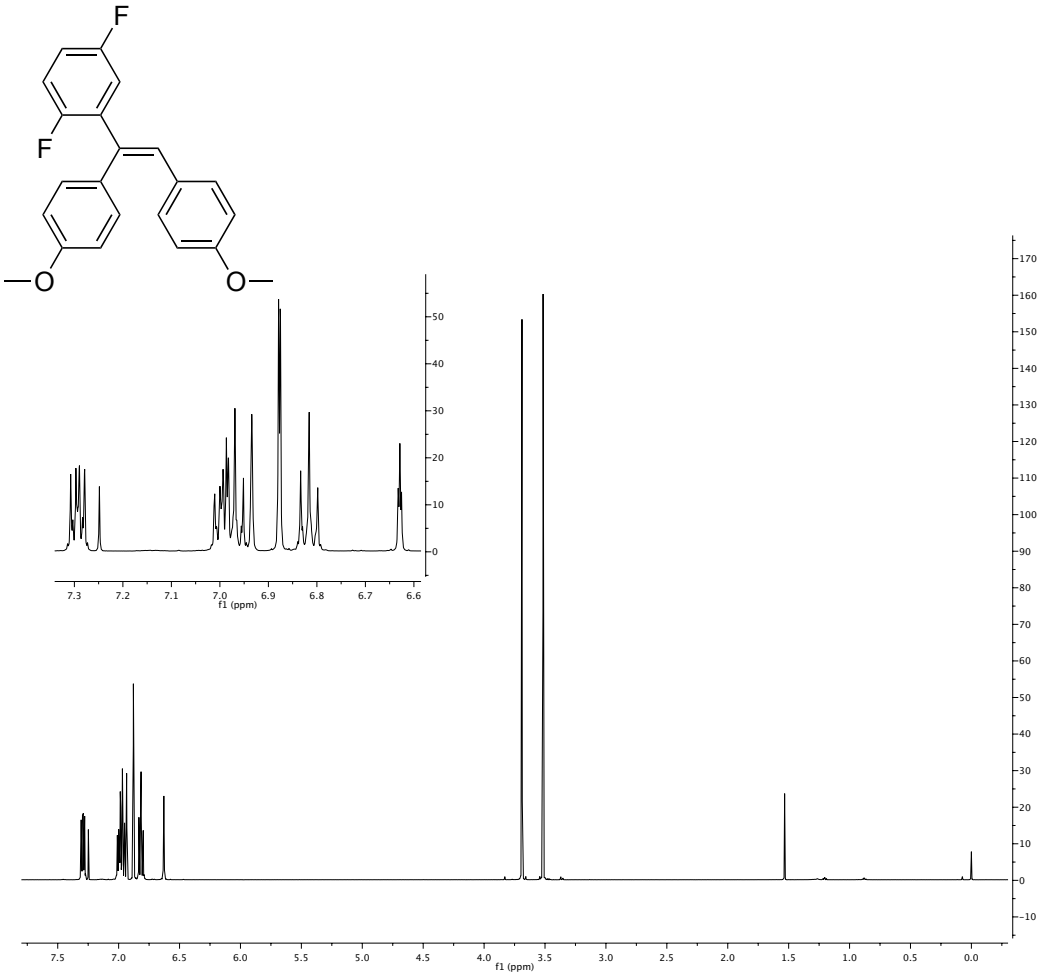


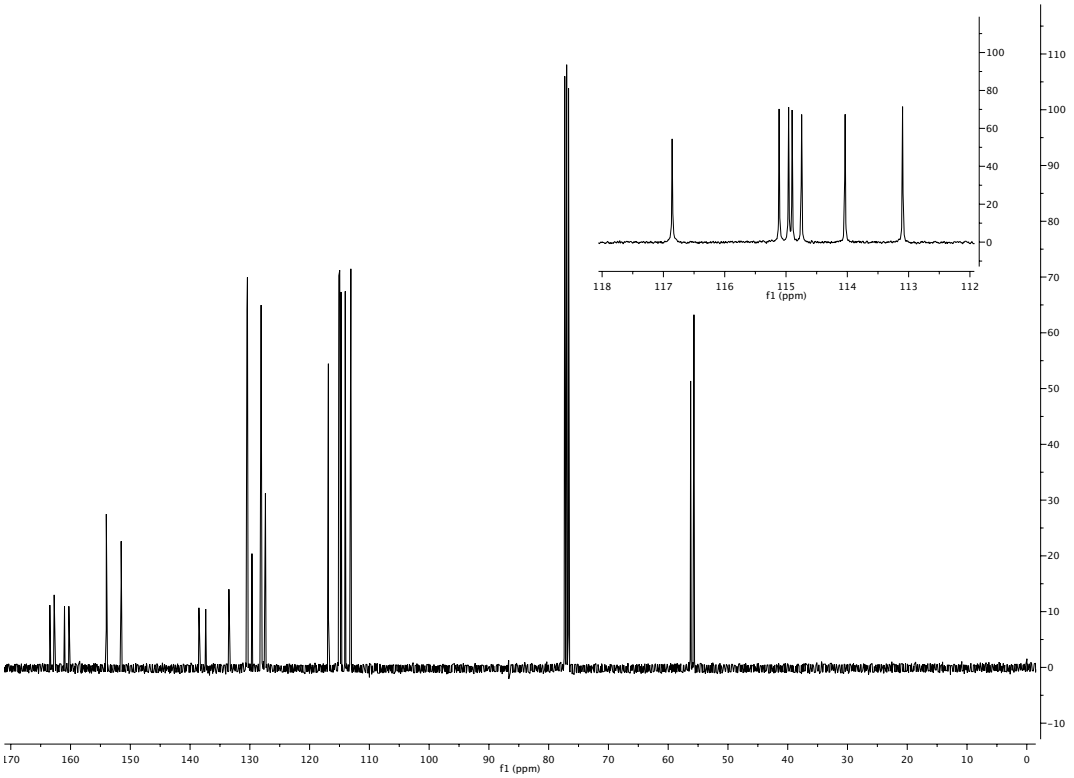
^{1}H and ^{13}C NMR (500, 100 MHz, respectively in $CDCl_3$) of (*E*/*Z*)-4,4'-(1-(2,5-dimethoxyphenyl)ethene-1,2-diyl)bis(methoxybenzene), **3n**.

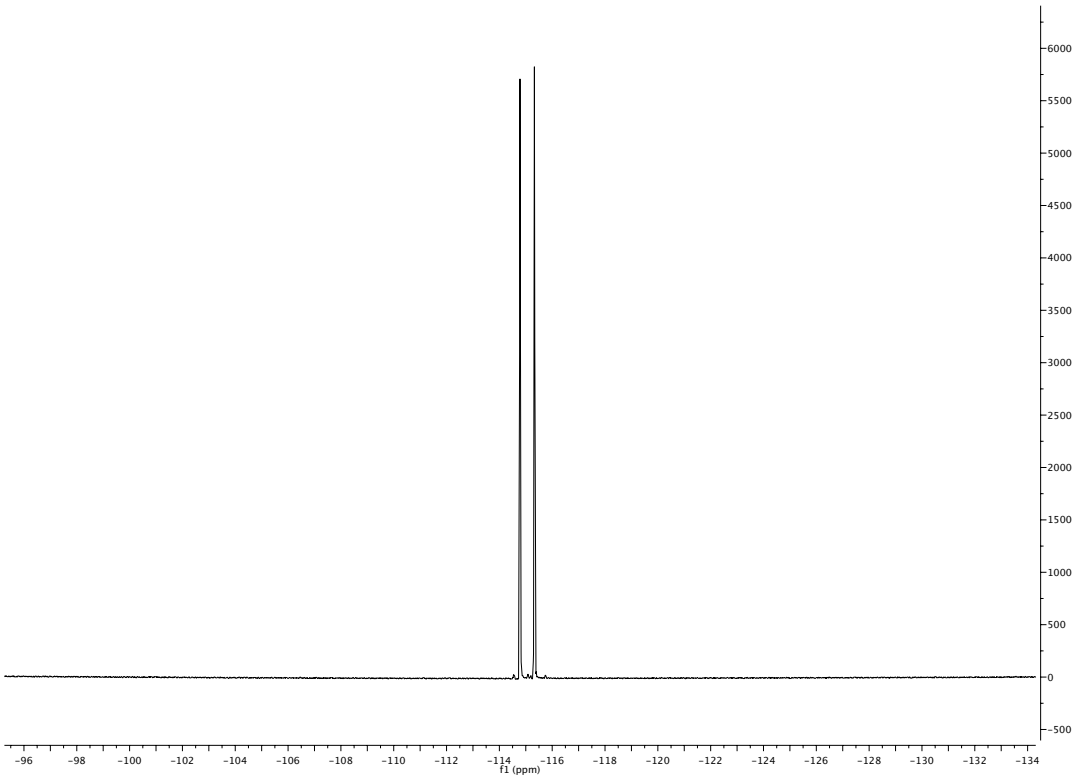




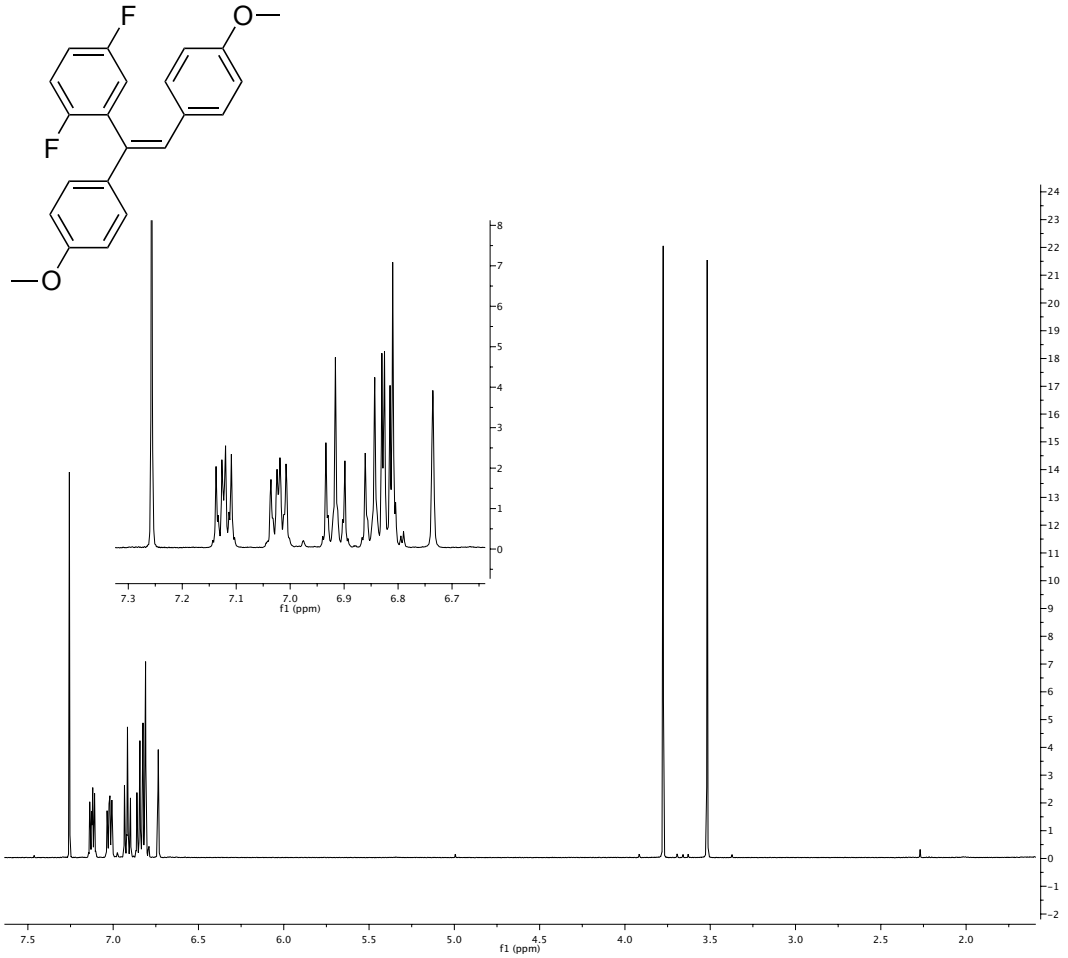
^{1}H, ^{13}C and ^{19}F NMR (500, 100 and 376 MHz, respectively in $CDCl_3$) of (*E*)-4,4'-(1-(2,5-Dimethoxyphenyl)ethene-1,2-diyl)bis(fluorobenzene), **(*E*)-3o**.

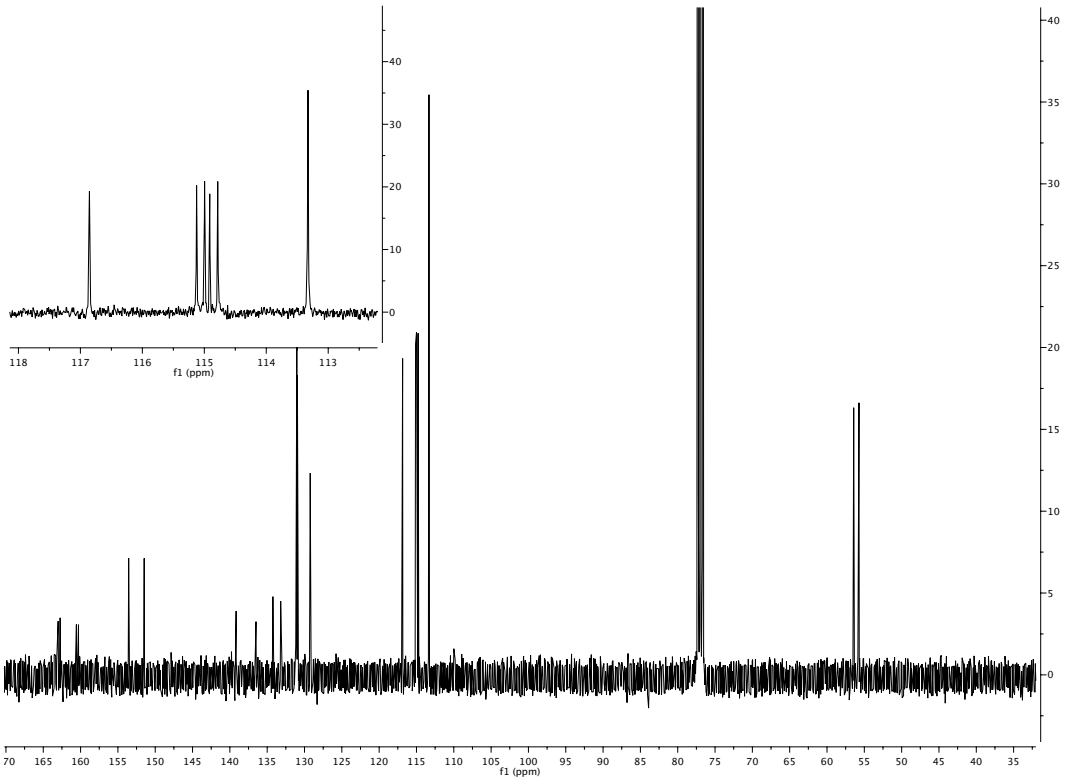


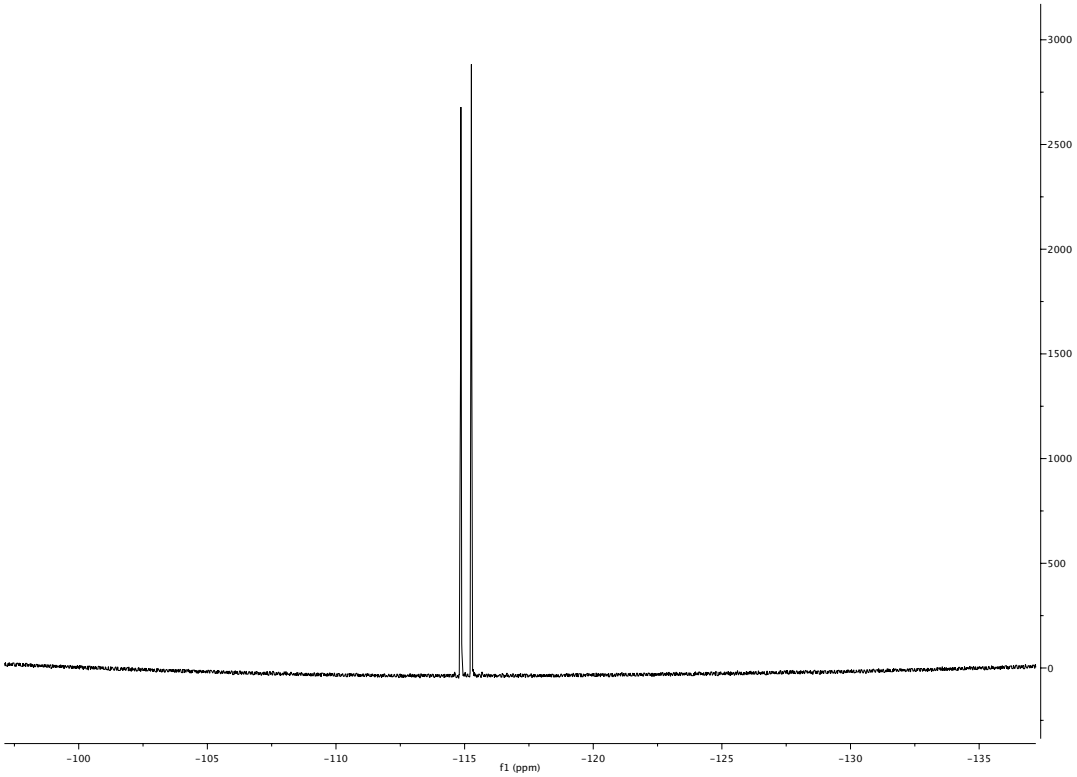

f1 (ppm)


f1 (ppm)

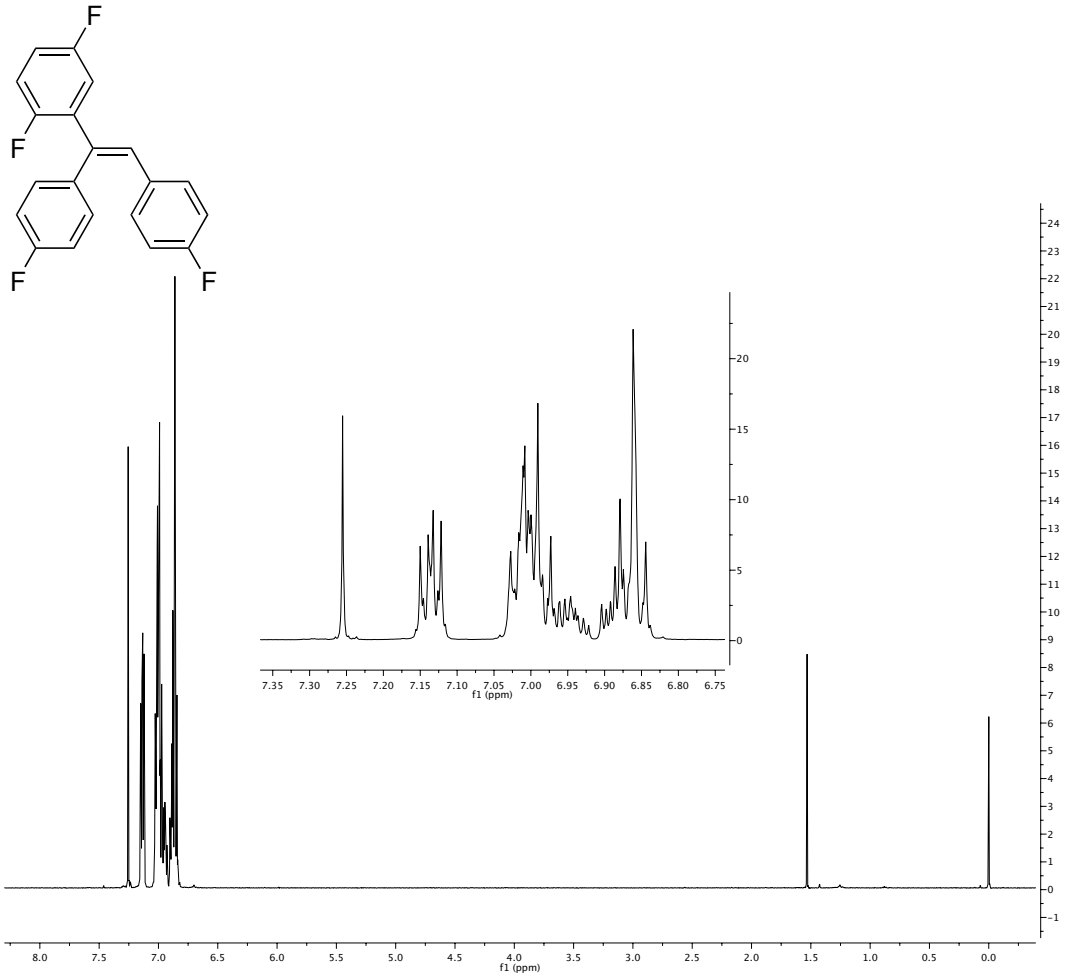
^{1}H, ^{13}C and ^{19}F NMR (500, 100 and 376 MHz, respectively in $CDCl_3$) of (*Z*)-4,4'-(1-(2,5-Dimethoxyphenyl)ethene-1,2-diyl)bis(fluorobenzene), **(Z)-3o**.

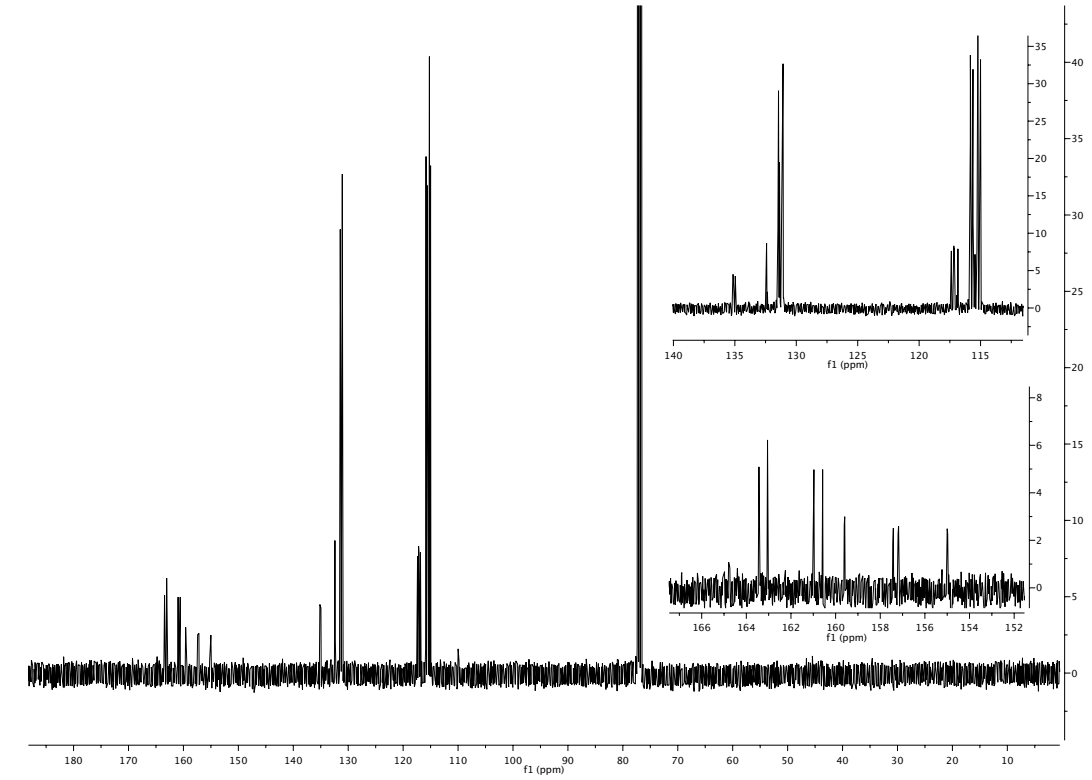


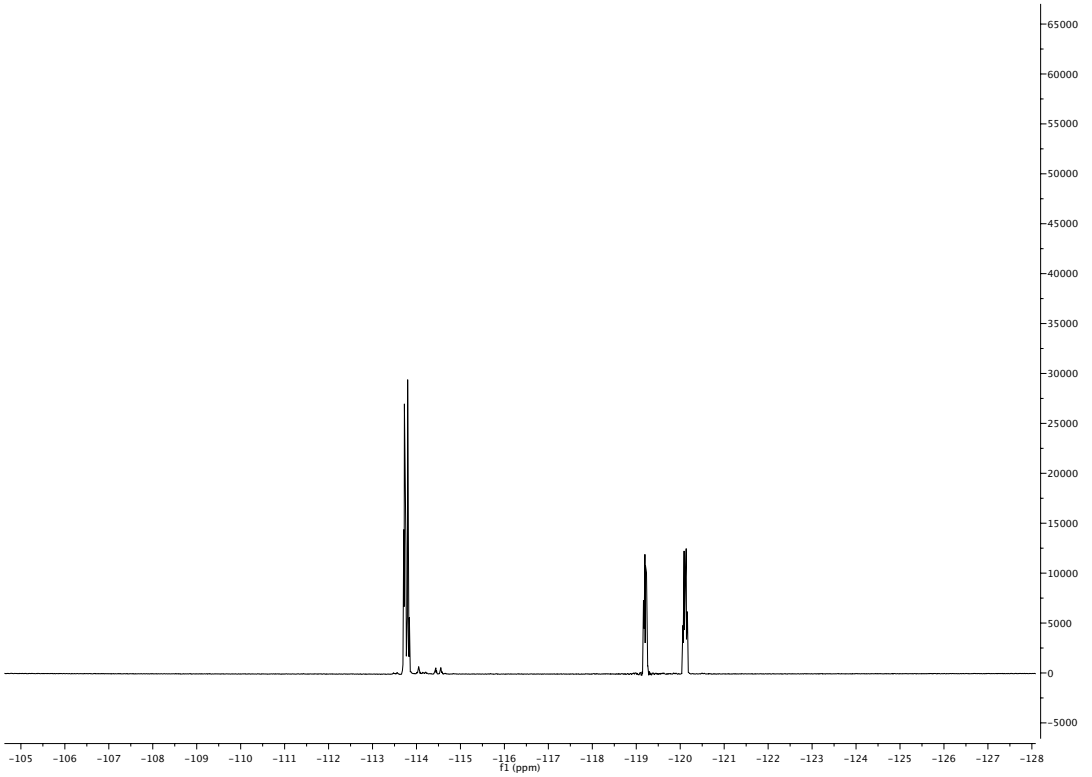




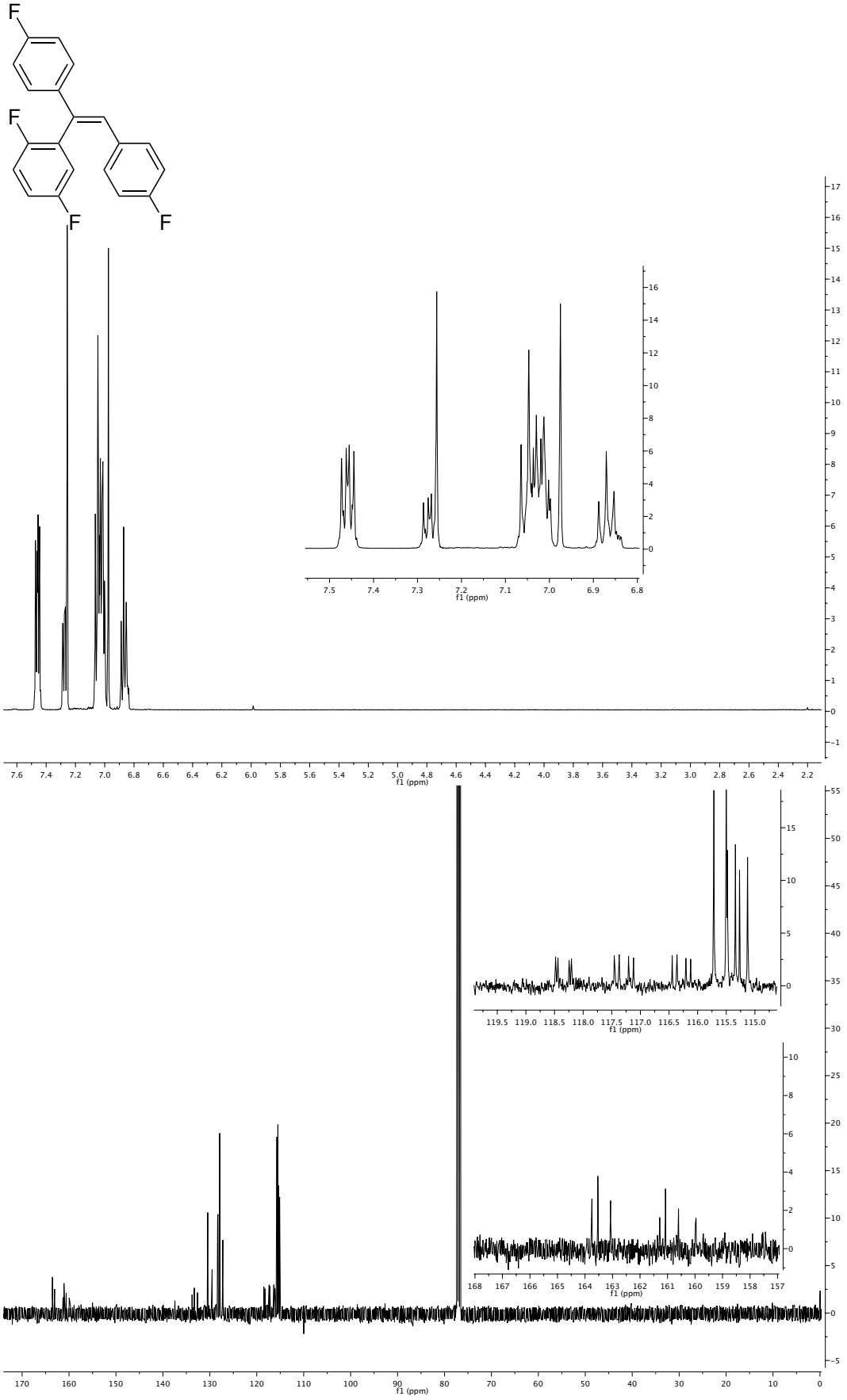
^{1}H, ^{13}C and ^{19}F NMR (500, 100 and 376 MHz, respectively in $CDCl_3$) of (*E*)-4,4'-(1-(2,5-difluorophenyl)ethene-1,2-diyl)bis(fluorobenzene), **(*E*)-3p**.

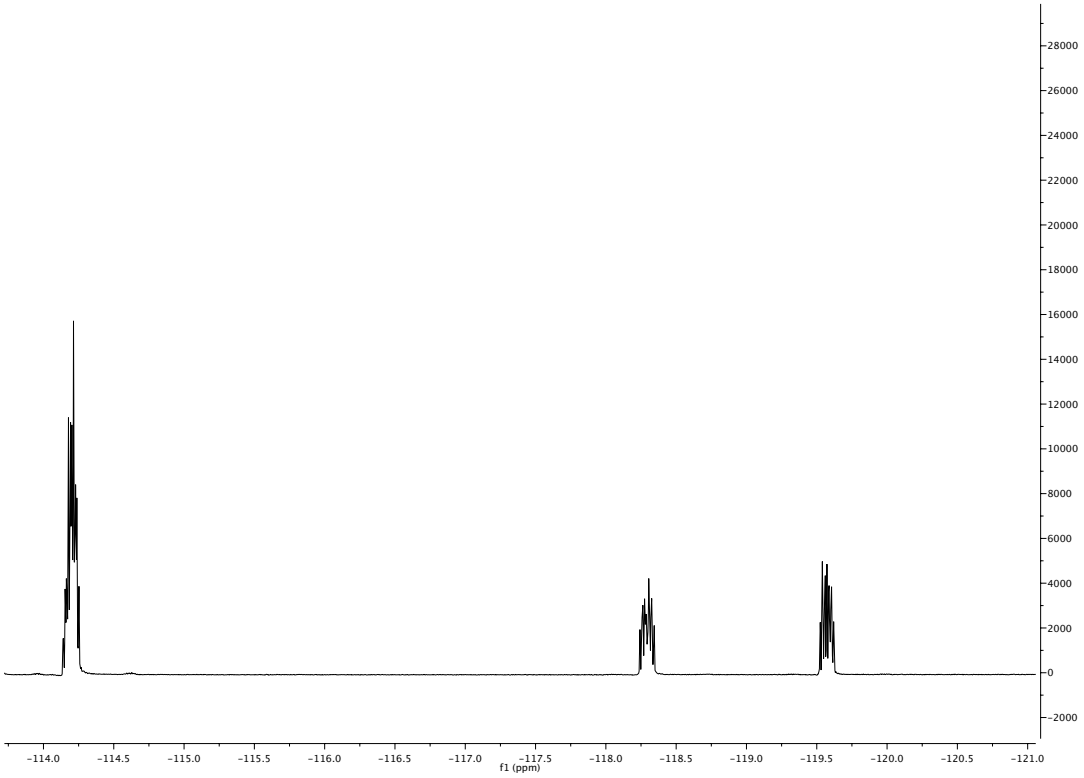



f1 (ppm)
f1 (ppm)
f1 (ppm)

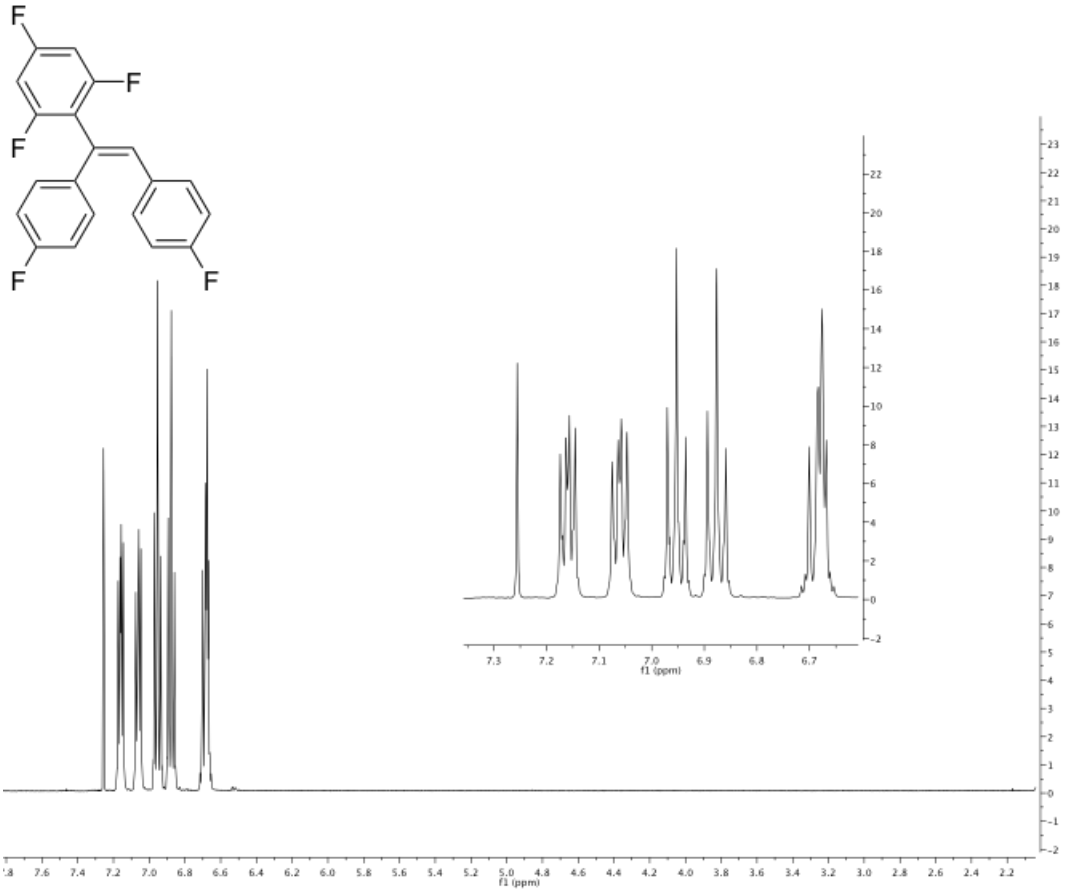

f1 (ppm)

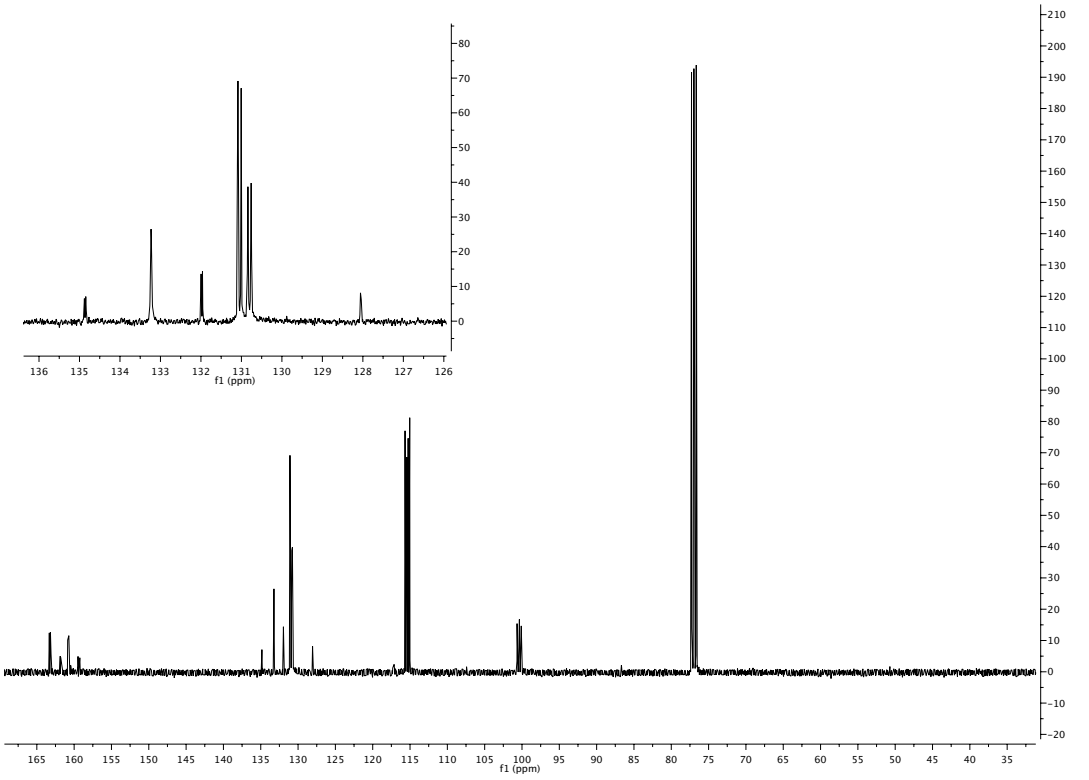
^{1}H, ^{13}C and ^{19}F NMR (500, 100 and 376 MHz, respectively in $CDCl_3$) of (*Z*)-4,4'-(1-(2,5-difluorophenyl)ethene-1,2-diyl)bis(fluorobenzene), **(*Z*)-3p**.

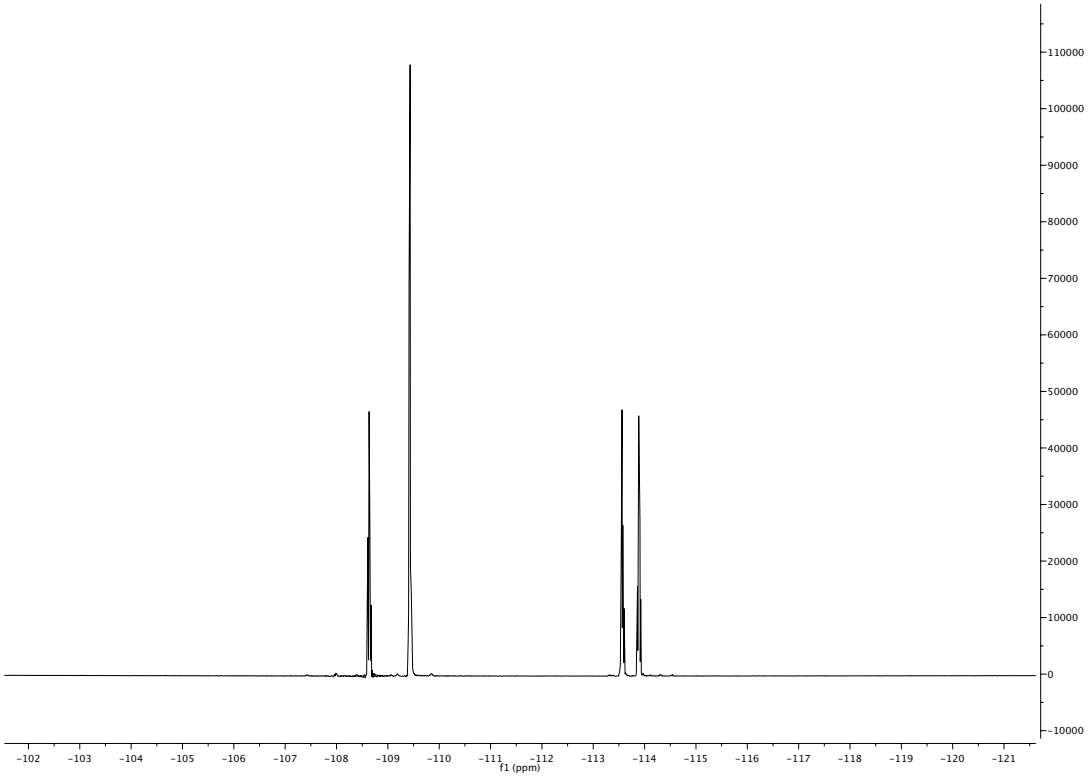




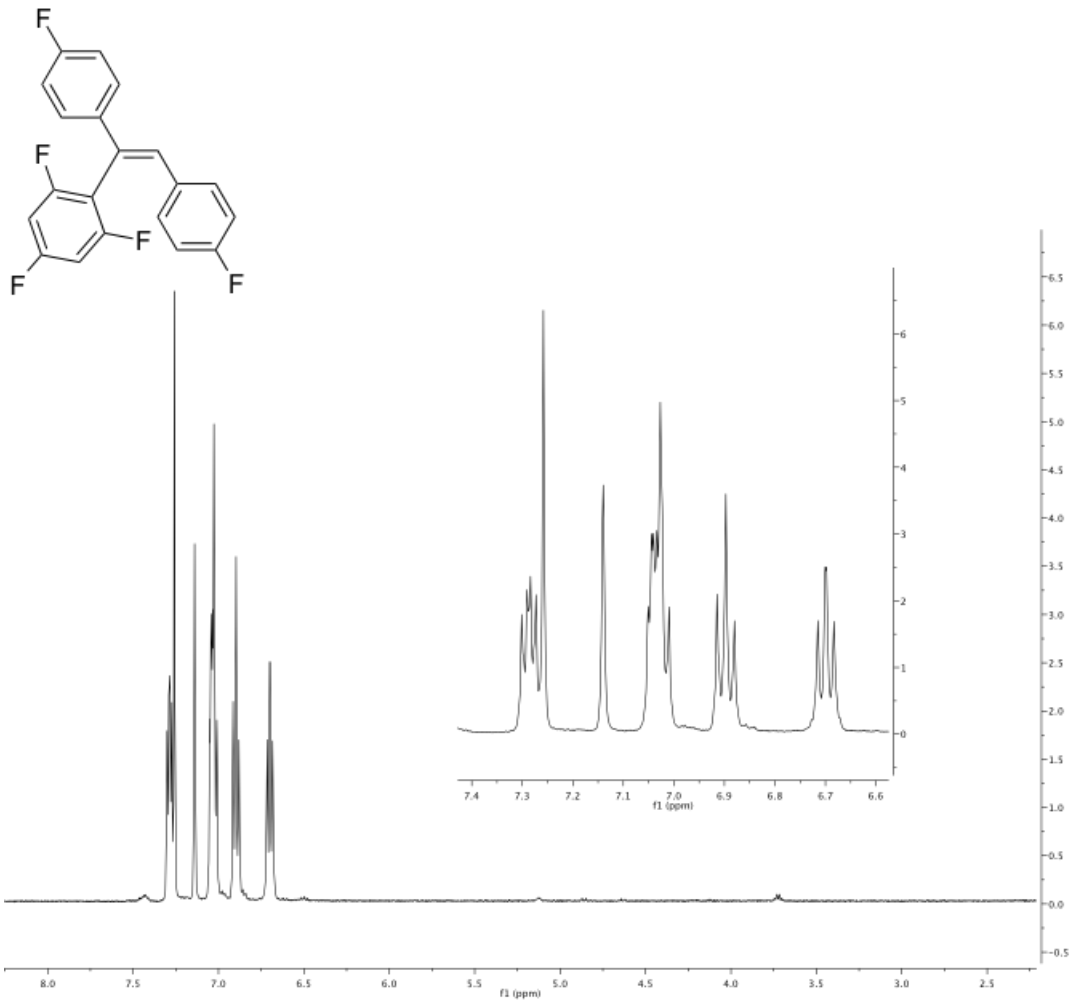
^{1}H, ^{13}C and ^{19}F NMR (500, 100 and 376 MHz, respectively in $CDCl_3$) of (*E*)-4,4'-(1-(2,4,6-trifluorophenyl)ethene-1,2-diyl)bis(fluorobenzene), **(*E*)-3q**.

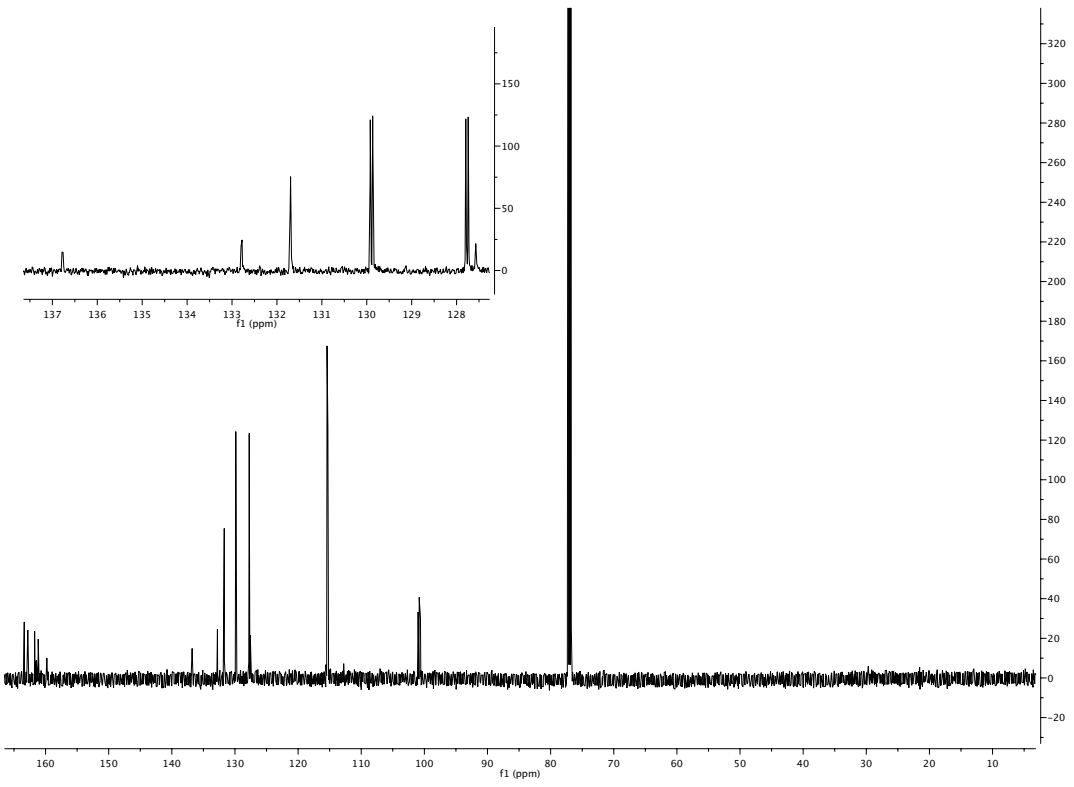


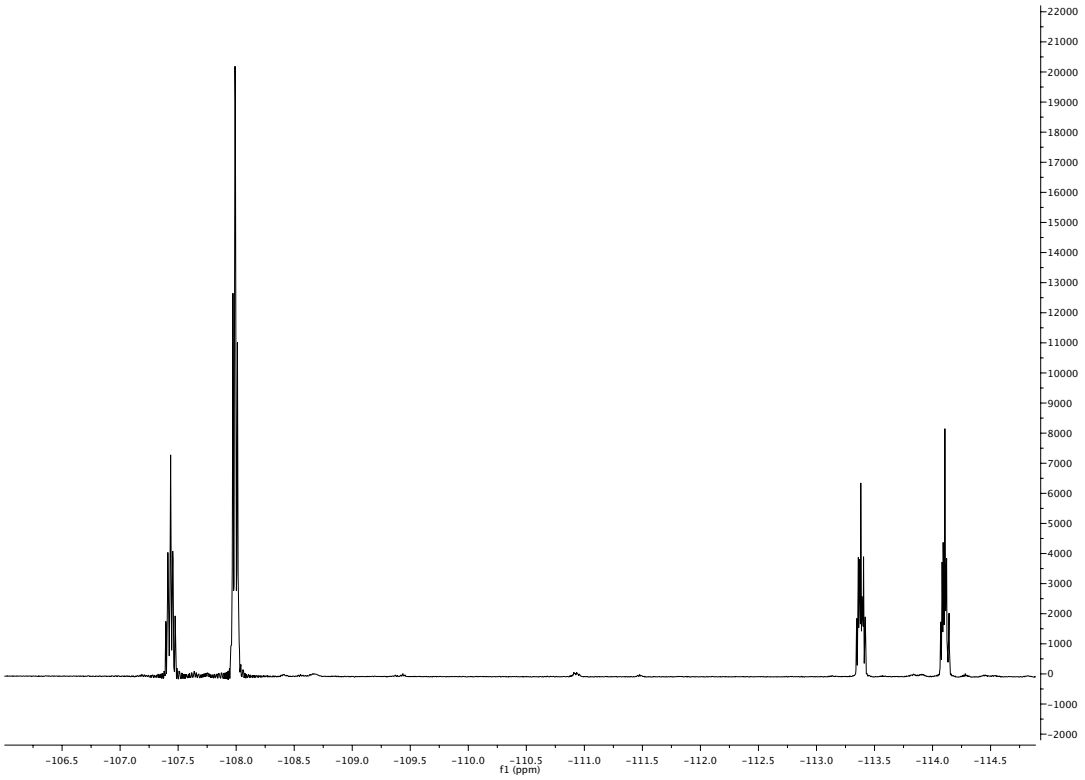

f1 (ppm)


f1 (ppm)

^{1}H, ^{13}C and ^{19}F NMR (500, 100 and 376 MHz, respectively in $CDCl_3$) of (*Z*)-4,4'-(1-(2,4,6-trifluorophenyl)ethene-1,2-diyl)bis(fluorobenzene), **(*Z*)-3q**.



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−107.0
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−108.5
−109.0
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−110.0
−110.5
−111.0
−111.5
−112.0
−112.5
−113.0
−113.5
−114.0
−114.5
f1 (ppm)

Structural determinations and crystallographic data

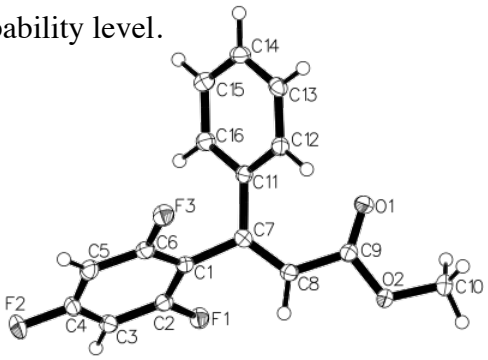
Cif files of the obtained structures can be obtained free of charge by request from the Cambridge crystallographic data centre; cif deposition numbers are 939488 and 939489 for (*E*)-3m and (*Z*)-3m respectively.

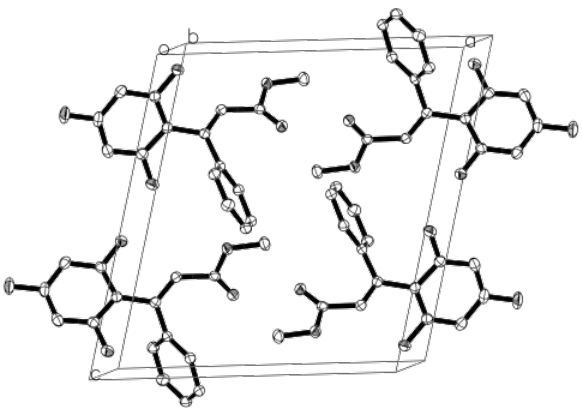
Compound **(*E*)-3m**

Data were collected at 100(2) K for crystals of **(*E*)-3m** at (λ = 1.5418 Å) by Dr. Helge Müller-Bunz using CrysAlisPro[1] (Agilent Technologies) software. The structure was solved by direct methods with SHELXS-97, refined using full-matix least squares routines against F^2 with SHELXL-97[2]. All non-hydrogen atoms were refined anisotropically, and hydrogen atoms were placed in calculated positions and refined using a riding model with fixed C-H distances of 0.95 Å (sp^2 C-H) and 0.98 Å (CH_3), and $U_{iso}(H) = 1.2U_{eq}(C)$ (sp^2) and $1.5U_{eq}(C)$ (sp^3).

Crystal data: $C_{16}H_{11}F_3O_2$, M=292.25, monoclinic, space group P-21/c, a=11.80816(9), b=9.10167(6), c=12.7085(9) Å, α=90.00, β=103.658(7), γ= 90.00^O, V= 1327.211(16) Å^3, Z= 4, D_c= 1.463 g cm^{-3}, specimen: colourless plates, 0.095 x 0.225 x 0.259 mm, 21147 measured reflections, R_{int}= 0.0210, R= 0.0310 for 2650 observed data ((I) >2σ(I)), wR= 0.0789, and GOOF= 1.053 for all data (2762).

ORTEP representations of the structure of $C_{16}H_{11}F_3O_2$ **(*E*)-3l**. Displacement ellipsoids are drawn at the 50 % probability level.



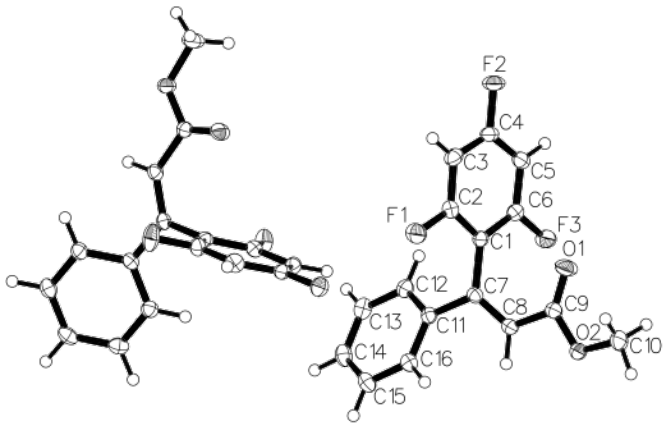


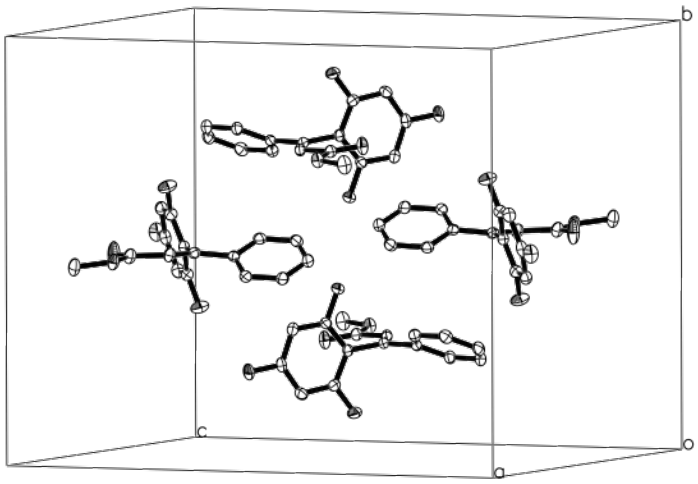
Compound **(*Z*)-3m**

Data were collected at 100(2) K for crystals of **(*Z*)-3m** at (λ = 1.5418 Å) by Dr. Helge Müller-Bunz using CrysAlisPro[1] (Agilent Technologies) software. The structure was solved by direct methods with SHELXS-97, refined using full-matix least squares routines against F^2 with SHELXL-97[2]. All non-hydrogen atoms were refined anisotropically, and hydrogen atoms were placed in calculated positions and refined using a riding model with fixed C-H distances of 0.95 Å (sp^2 C-H) and 0.98 Å (CH_3), and $U_{iso}(H) = 1.2U_{eq}(C)$ (sp^2) and $1.5U_{eq}(C)$ (sp^3).

Crystal data: $C_{16}H_{11}F_3O_2$, M=292.25, orthorhombic, space group Pbca, a=17.1451(5), b=15.8233(6), c=19.5388(7) Å, α=90.00, β=90.00, γ= 90.00°, V= 5300.71(3) Å^3, Z= 16, D_c= 1.465 g cm^{-3}, specimen: colourless blocks, 0.172 x 0.266 x 0.438 mm, 105300 measured reflections, R_{int}= 0.0338, R= 0.0317 for 5276 observed data ((I) >2σ(I)), wR= 0.0817, and GOOF= 1.025 for all data (5567).

ORTEP representations of the structure of $C_{16}H_{11}F_3O_2$ **(*Z*)-3m**. Displacement ellipsoids are drawn at the 50 % probability level.





References:

1. Agilent Technologies, release 27-06-2012 CrysAlis171.NET.
2. G. M. Sheldrick, SHELX97, Programs for Crystal Structure Analysis; Universität, Göttingen: Germany, 2008.