

Electronic Supplementary Information for

Reductive Activation of Arenes by Potassium Metal with Potassium Salts

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General Details

All reagents were purchased from commercial sources and used without further purification, except where stated. Anhydrous diethyl ether, tetrahydrofuran, dichloromethane and hexane were dried using a Pure-Solv 400 solvent purification system (Innovative Technology Inc., U.S.A.). Anhydrous benzene was purchased from Sigma Aldrich and dried over 3 Å molecular sieves, previously activated by microwave heating. Thin layer chromatography analyses were carried out on silica gel pre-coated aluminium foil sheets and were visualised using UV light (254 nm). Flash column chromatography was carried out using slurry packed silica gel (SiO₂), 35-75 µm particle size, 60 Å pore size, under a light positive pressure, eluting with the specified solvent system.

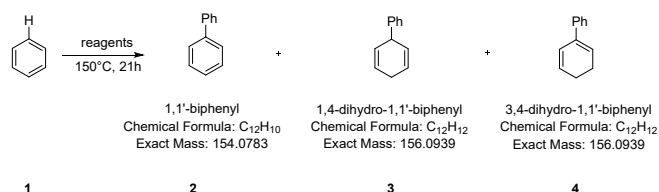
Where reactions were carried out in a glovebox, the atmosphere used was nitrogen and the glovebox was supplied by Innovative Technology Inc., USA. Throughout this paper, all reactions were worked up by addition of water (following removal of any unreacted potassium metal for separate work up in *t*-BuOH – Caution!)

¹H-NMR, ²H-NMR and ¹³C-NMR spectra were recorded on Bruker spectrometers operating at 400 or 500 MHz, 61 MHz and 101 or 126 MHz, respectively. All spectral data were acquired at 295 K. Chemical shifts (δ) are quoted in parts per million (ppm). Coupling constants (*J*) are reported in Hertz (Hz) to the nearest 0.1 Hz. The multiplicity abbreviations used are: s (singlet), d (doublet), t (triplet), q (quartet), qn (quintet), sextet (st), m (multiplet). Infrared (IR) spectra were recorded using an FTIR-ATR spectrometer. High resolution mass spectrometry was performed at the University of Swansea, in the EPSRC National Mass Spectrometry Centre. Accurate mass measurements were obtained using a LTQ Orbitrap XL using Atmospheric Pressure Chemical Ionisation (APCI) or High Resolution Nano-Electrospray (HNESP) using Electrospray Ionisation (ESI). Low resolution spectrometry was recorded by gas-phase chromatography (GCMS) using electron ionization (EI) in the University of Strathclyde. Data were recorded using an Agilent Technologies 7890A GC system coupled to a 5975C inert XL EI/CI MSD detector. Separation was performed using the DB5MS-UI column (30 m x 0.25 mm x 0.25 µm) at a temperature of 320 °C, using helium as the carrier gas.

Calculations of the yields of reactions using the internal standard 1,3,5-trimethoxybenzene (¹H-NMR internal standard) were performed as follows: 1,3,5-trimethoxybenzene (8.4 mg, 0.050 mmol, 10 mol%) was added as a solid to the reaction mixture. CDCl₃ (~1 mL) was added and the solution stirred for 5 min. A portion of the solution was taken and diluted for NMR analysis. The yields were therefore calculated using the integration of similar types of peaks: aromatic peaks of the compound were compared with the aromatic peak of the internal standard, aliphatic peaks of the compounds were compared with the aliphatic peaks of the internal standard.

Experimental details.

Reactions of Benzene:



Experiments related to Table 1. General reaction procedure: Freshly cut potassium metal (58.5 mg, 1.5 mmol), KO^tBu (168 mg, 1.5 mmol) and anhydrous benzene **1** (5 mL) were added to an oven-dried pressure tube (58.5 mg, 1.5 mmol), KO^tBu (168 mg, 1.5 mmol) and anhydrous benzene (5 mL) in a glovebox. The tube was sealed and transferred in a fumehood. The reaction was stirred at 150 °C. After 21h, the reaction was cooled to 0 °C, it was quenched carefully with isopropanol/water (20 mL), the pH was adjusted to neutral by using hydrochloric acid 5% and the organic fraction was extracted with diethyl ether (3 x 20 mL). The combined organic layers were dried over sodium sulfate, filtered and concentrated *in vacuo* to afford the crude product. The calculations of the *in situ* reaction yields *via* internal standard 1,3,5-trimethoxybenzene (¹H-NMR internal standard) were performed as a general procedure. Where specified, the crude material was purified by column chromatography. on silica gel.

Table 1

(Table 1, **Entry 1**) The results of four replicate experiments using different reagents are reported.

(a) **2** (108 mg), **3** (traces), **4** (traces) (using Legacy batch of K metal and KOtBu stored in the glovebox);
(b) **2** (115 mg), **3** (traces), **4** (traces). [using the same batches of K and KOtBu metal used in (a)];
(c) **2** (182 mg), **3** (28 mg, 0.17 mmol), **4** (6 mg, 0.04 mmol). (using a fresh batch of K metal);
(d) **2** (83 mg, 0.54 mmol), **3** (14 mg, 0.09 mmol), **4** (11 mg, 0.07 mmol) (Different batch of K metal and different batch of KOtBu).

Average of four experiments: **2** (122 mg, 0.79 mmol), **3** (12 mg, 0.07 mmol), **4** (3 mg, 0.02 mmol).

(Entry 2) Potassium metal (58.5 mg, 1.5 mmol), and anhydrous benzene (5 mL) were added to an oven-dried pressure tube. After 21 h, work up showed that no reaction had occurred.

(Entry 3) KOtBu (168 mg, 1.5 mmol) and anhydrous benzene (5 mL) were added to an oven-dried pressure tube. After 21 h, work up showed that no reaction had occurred.

(Entry 4) Potassium metal (3.9 mg, 0.1 mmol), KOtBu (168 mg, 1.5 mmol) and anhydrous benzene (2 mL) were added to an oven-dried pressure tube. Workup afforded **2** (1 mg).

(Entry 5) Potassium metal (19.5 mg, 0.5 mmol), KOtBu (11.2 mg, 0.1 mmol) and anhydrous benzene (2 mL) were added to an oven-dried pressure tube. Workup afforded **2** (30 mg, 0.19 mmol).

(Entry 6) Potassium metal (19.5 mg, 0.5 mmol), KOtBu (11.2 mg, 0.1 mmol) and anhydrous benzene (2 mL) were added to an oven-dried pressure tube. The reaction was stirred at RT for 21h and then quenched and analysed. No reaction had occurred.

Table 2

(All can be compared with Table 1, entry 1: Potassium metal (58.5 mg, 1.5 mmol), KOtBu (168 mg, 1.5 mmol) and anhydrous benzene (5 mL) were added to an oven-dried pressure tube. The results are reported. **2** (122 mg, 0.79 mmol), **3** (12 mg, 0.07 mmol), **4** (3 mg, 0.02 mmol).

(Table 2, **Entry 1**) Potassium metal (58.5 mg, 1.5 mmol), KI (249 mg, 1.5 mmol) and anhydrous benzene (5 mL) were added to an oven-dried pressure tube. Workup afforded **2** (54 mg, 0.09 mmol), **3** (traces), **4** (traces).

(Entry 2) Potassium metal (58.5 mg, 1.5 mmol), KBr (79 mg, 1.5 mmol) and anhydrous benzene (5 mL) were added to an oven-dried pressure tube. Workup afforded **2** (47 mg, 0.30 mmol), **3** (17 mg, 0.11 mmol), **4** (5 mg, 0.03 mmol).

(Entry 3) Potassium metal (58.5 mg, 1.5 mmol), KF (87 mg, 1.5 mmol) and anhydrous benzene (5 mL) were added to an oven-dried pressure tube. Workup afforded **2** (14 mg, 0.09 mmol), **3** (11 mg, 0.07 mmol), **4** (traces).

(Entry 4) Potassium metal (58.5 mg, 1.5 mmol), KBF₄ (189 mg, 1.5 mmol) and anhydrous benzene (5 mL) were added to an oven-dried pressure tube. Workup afforded **2** (96 mg, 0.62 mmol), **3** (11 mg, 0.07 mmol), **4** (5 mg, 0.03 mmol).

(Entry 5) Potassium metal (58.5 mg, 1.5 mmol), Me₄NCl (417 mg, 1.5 mmol) and anhydrous benzene (5 mL) were added to an oven-dried pressure tube. Workup showed that no reaction had occurred.

(Entry 6) Potassium metal (58.5 mg, 1.5 mmol), NaI (225 mg, 1.5 mmol) and anhydrous benzene (5 mL) were added to an oven-dried pressure tube. Workup afforded **2** (15 mg, 0.09 mmol), **3** (6 mg, 0.04 mmol).

(Entry 7) Potassium metal (58.5 mg, 1.5 mmol), NaBF₄ (165 mg, 1.5 mmol) and anhydrous benzene (5 mL) were added to an oven-dried pressure tube. Workup afforded **3** (5 mg, 0.03 mmol).

(Entry 8) Potassium metal (58.5 mg, 1.5 mmol), NaOtBu (168 mg, 1.5 mmol) and anhydrous benzene (5 mL) were added to an oven-dried pressure tube. Workup showed that no reaction had occurred.

(Entry 9) Potassium metal (58.5 mg, 1.5 mmol), LiI (201 mg, 1.5 mmol) and anhydrous benzene (5 mL) were added to an oven-dried pressure tube. Workup showed that no reaction had occurred.

(Entry 10) Sodium metal (11.5 mg, 0.5 mmol), KOtBu (56 mg, 0.5 mmol) and anhydrous benzene (2 mL) were added to an oven-dried pressure tube. Workup showed that no reaction had occurred.

(Entry 11) Potassium metal (58.5 mg, 1.5 mmol), MgI₂ (417 mg, 1.5 mmol) and anhydrous benzene (5 mL) were added to an oven-dried pressure tube. Workup showed that no reaction had occurred.

(Entry 12) Potassium metal (58.5 mg, 1.5 mmol), MgBr₂ (276 mg, 1.5 mmol) and anhydrous benzene (5 mL) were added to an oven-dried pressure tube. Workup showed that no reaction had occurred.

(Entry 13) Potassium metal (58.5 mg, 1.5 mmol), CaI₂ (441 mg, 1.5 mmol) and anhydrous benzene (5 mL) were added. Workup afforded **2** (44 mg, 0.29 mmol), **3** (traces) and **4** (traces).

(Entry 14) Potassium metal (19.5 mg, 0.5 mmol), SrI₂ (511 mg, 0.5 mmol) and anhydrous benzene (2 mL) were added to an oven-dried pressure tube. Workup afforded **2** (5 mg, 0.03 mmol).

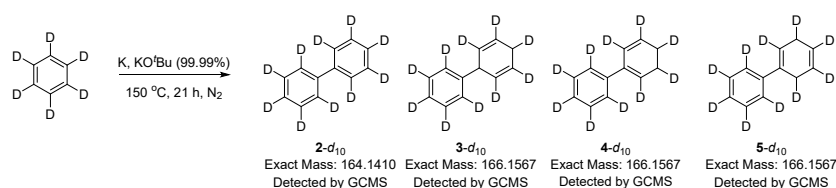
(Entry 15) Potassium metal (58.5 mg, 1.5 mmol), RbI (318 mg, 1.5 mmol) and anhydrous benzene (5 mL) were added to an oven-dried pressure tube. Workup afforded **2** (42 mg, 0.27 mmol), **3** (41 mg, 0.26 mmol).

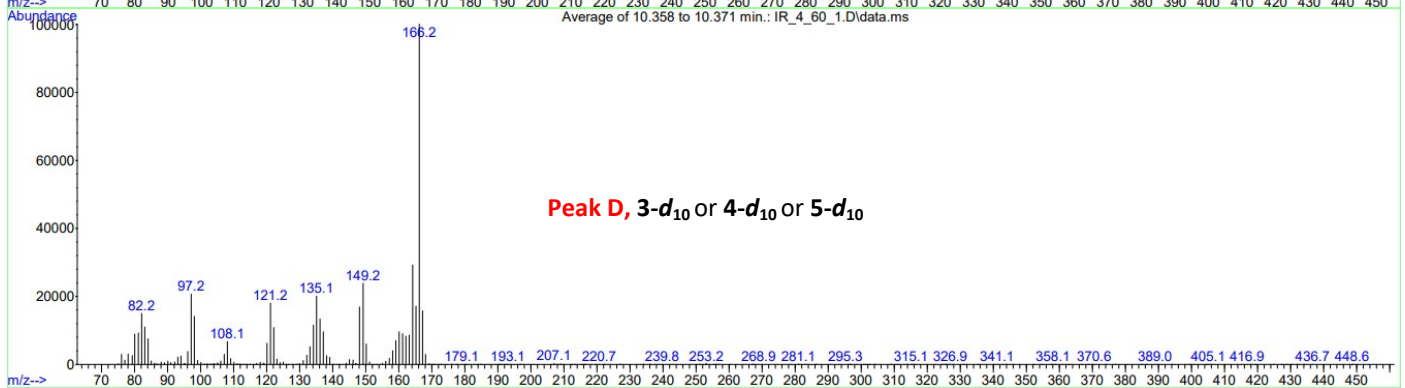
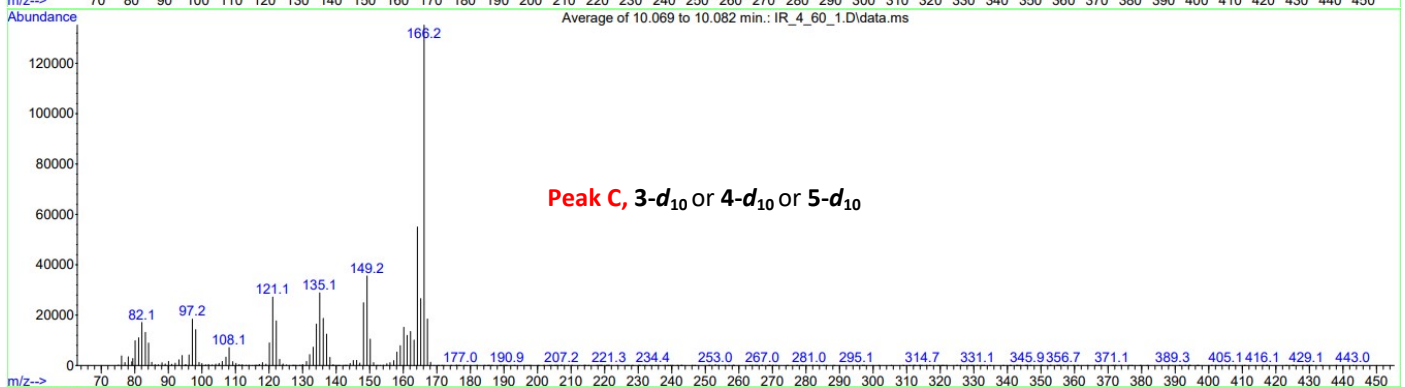
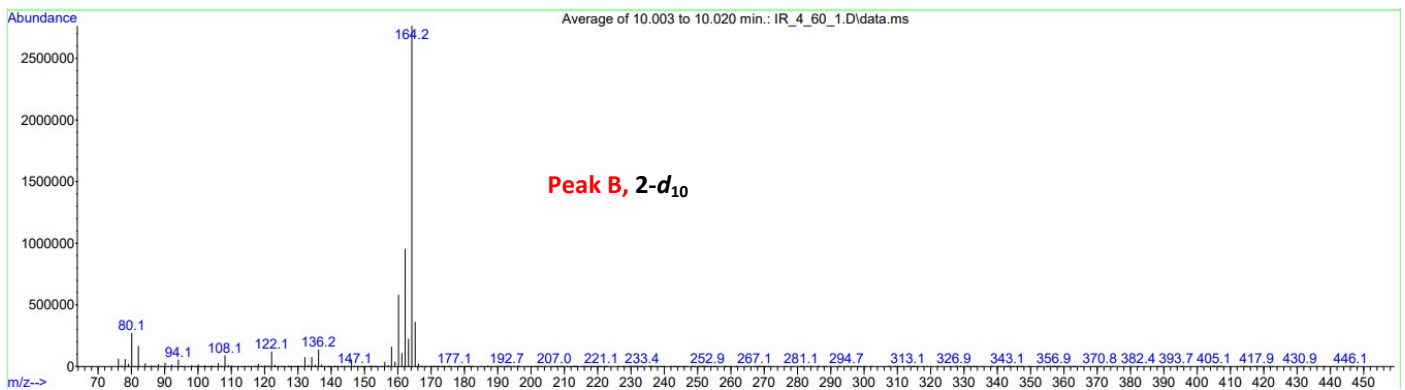
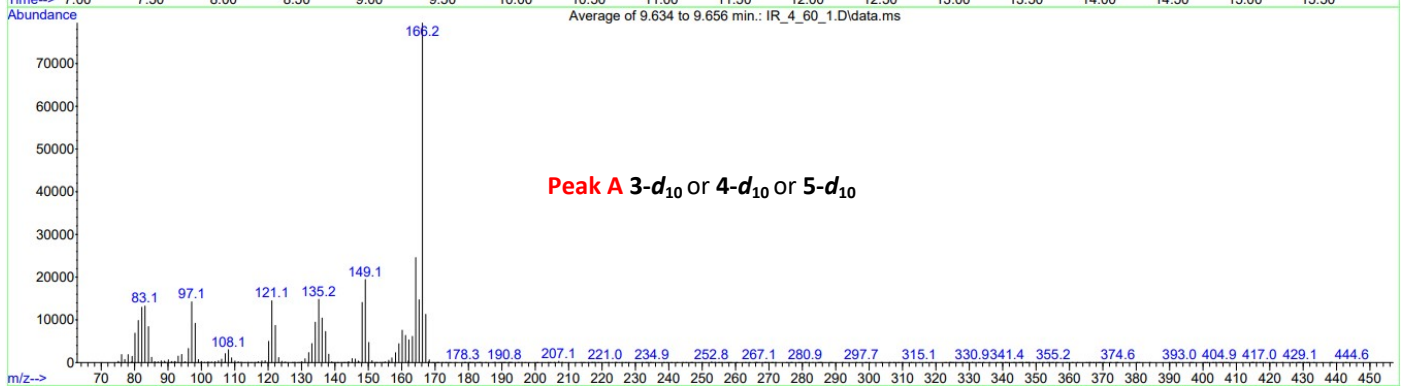
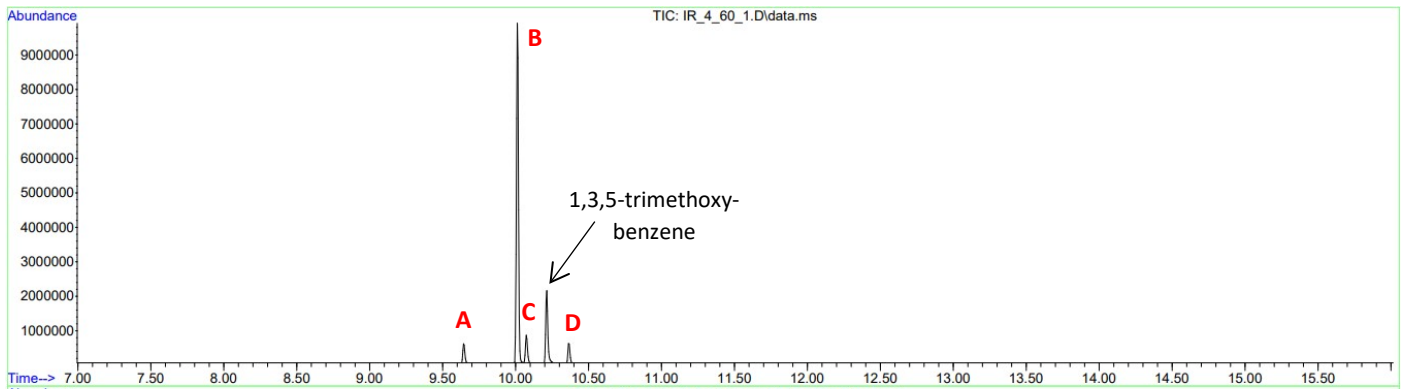
(Entry 16) Potassium metal (58.5 mg, 1.5 mmol), CsI (390 mg, 1.5 mmol) and anhydrous benzene (5 mL) were added to an oven-dried pressure tube. Workup afforded **2** (18 mg, 0.12 mmol), **3** (43 mg, 0.28 mmol), **4** (25 mg, 0.16 mmol).

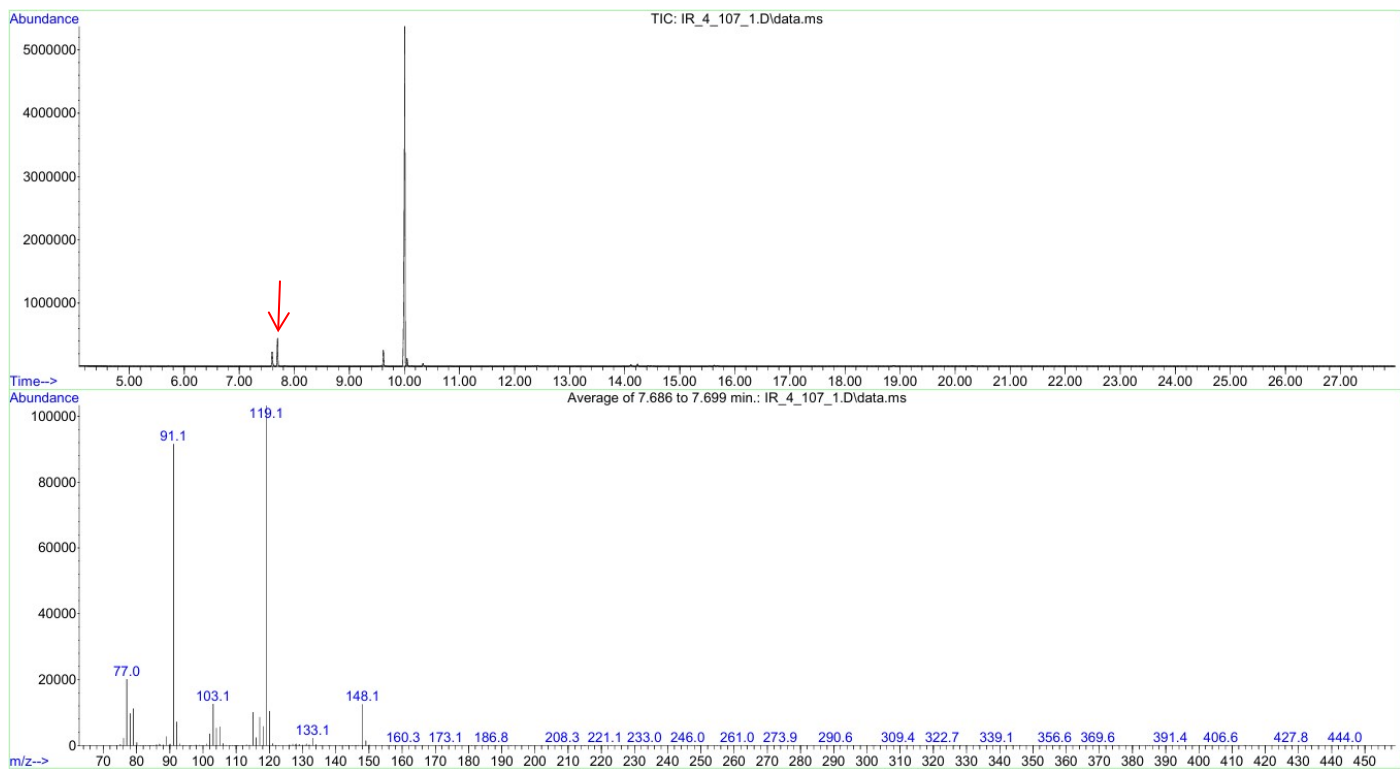
From some experiments (Table 1, entries 5 and 6) biphenyl, **2**, was isolated as sole product as a white solid. Mp: 64–67 °C (lit: 65–67 °C).¹ ν_{\max} (neat, cm⁻¹) 3061, 3034, 1477, 1429, 1344, 1182, 1169, 902, 725, 692. ¹H-NMR (400 MHz, CDCl₃) δ ppm 7.71 – 7.64 (4 H, m, ArH), 7.55 – 7.48 (4 H, m, ArH), 7.45 – 7.39 (2 H, m, ArH). ¹³C-NMR (101 MHz, CDCl₃) δ ppm 140.8, 128.3, 126.8, 126.8. GC-MS (EI) m/z 154.1. Data were consistent with the literature.²

In the other experiments, the mixtures of dihydrobiphenyls and biphenyl were inseparable, but identified from the literature: 1,4-dihydrobiphenyl, **3**,³ and 3,4-dihydrobiphenyl **4**.⁴

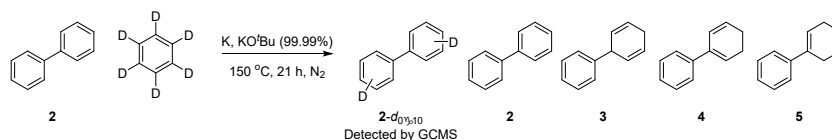
GCMS data from reaction of benzene-*d*₆ carried out according to the **General Procedure** with 1,3,5-trimethoxybenzene as calibrant.



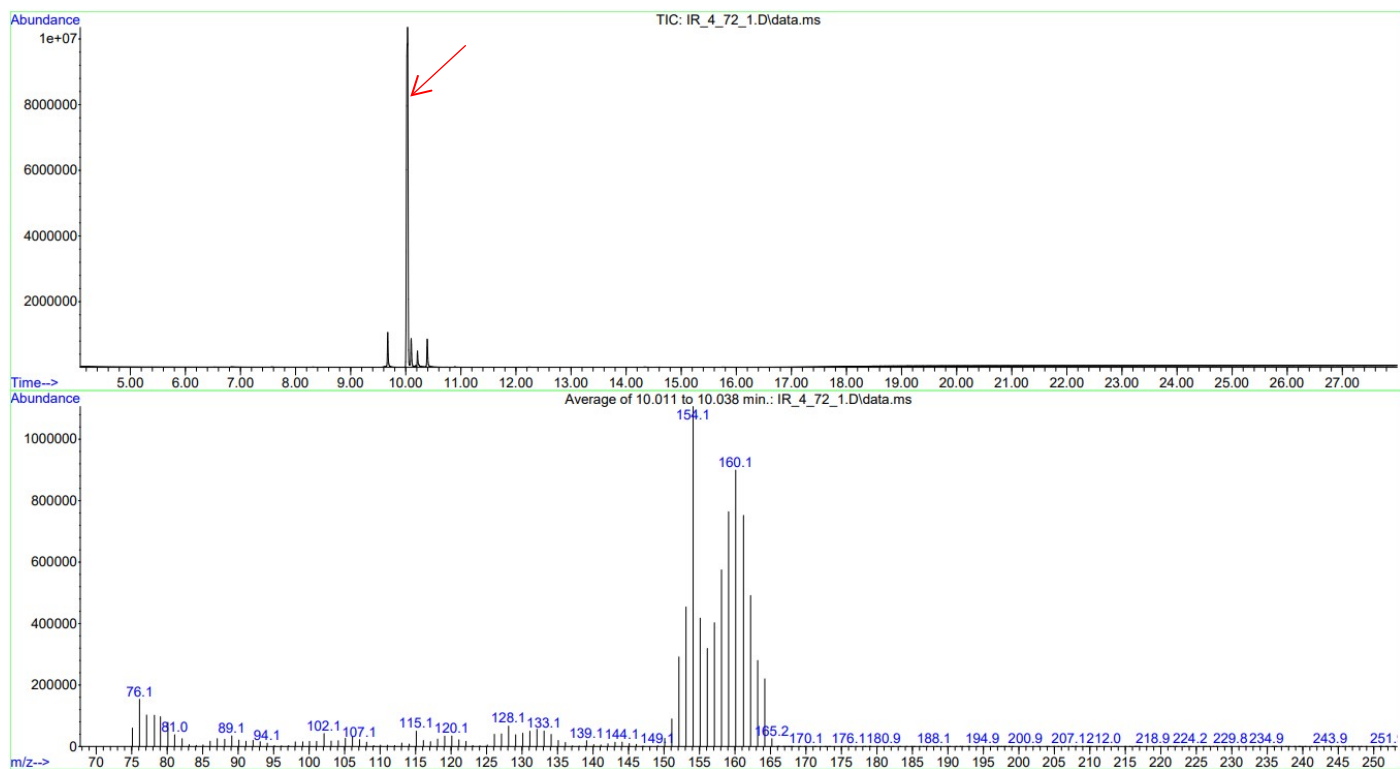




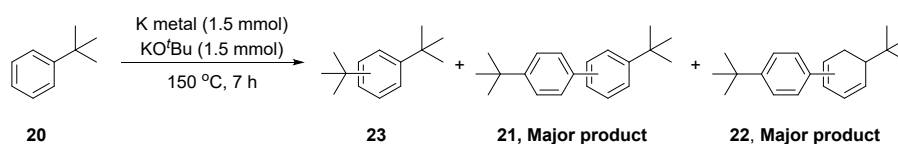
Biphenyl (**2**) as additive with benzene- d_6



This experiment was carried out according to the **General Procedure** using anhydrous benzene- d_6 (5.00 mL) with the addition of biphenyl (**2**) (231 mg, 1.50 mmol). GCMS data are shown below. NMR yields of biphenyl (**2**, 44.5 mg), 1,4-dihydro-1,1'-biphenyl (**3**, 9.2 mg),⁶ 3,4-dihydro-1,1'-biphenyl (**4**, 5.9 mg)⁴ and 2,5-dihydro-1,1'-biphenyl (**5**, 6.7 mg)⁷ (spectrum of mixture is shown on page S24) were calculated as shown (page S28) using 1,3,5-trimethoxybenzene as internal standard.

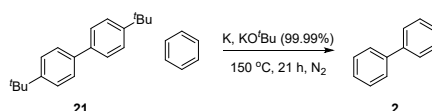


tert-butylbenzene (**20**)



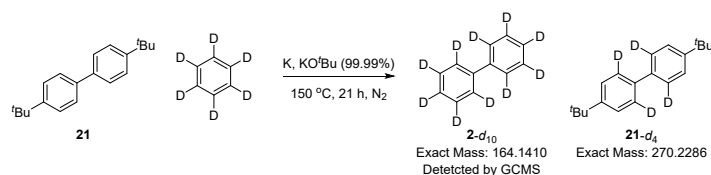
This experiment was carried out according to the **General Procedure**. The potassium was consumed – Products above were tentatively identified from GCMS. *t*Bu-C₆H₄-*t*Bu **23** (10.2 min) *m/z* 190.2; *t*Bu-C₆H₄-C₆H₆-*t*Bu **22** (14.0 and 14.4 min, isomers) *m/z* 268.3; *t*Bu-C₆H₄-C₆H₄-*t*Bu **21** (14.5, 14.9 and 15.2 min, isomers) *m/z* 266

4,4'-Di-*tert*-butyl-1,1'-biphenyl (**21**) with benzene



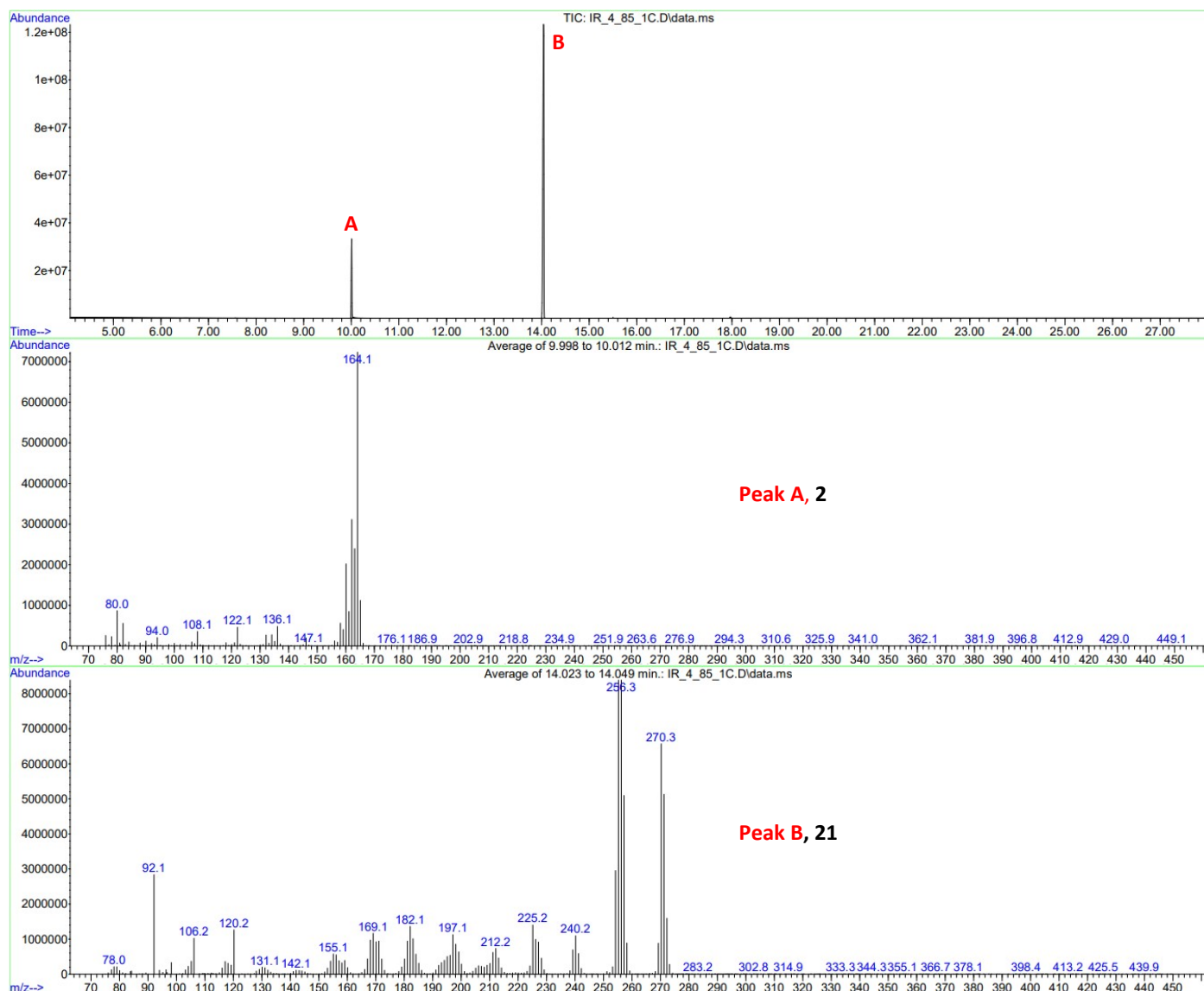
This experiment was carried out according to **General Procedure** with the addition of 4,4'-di-*tert*-butyl-1,1'-biphenyl (**21**) (400 mg, 1.50 mmol). An NMR yield of 91.5 mg of biphenyl (**2**) [as well as residual **21** (400 mg)] was calculated using 1,3,5-trimethoxybenzene as internal standard. The ¹H NMR of the crude reaction product is shown on S24.

4,4'-Di-*tert*-butyl-1,1'-biphenyl (**21**) with benzene-*d*₆

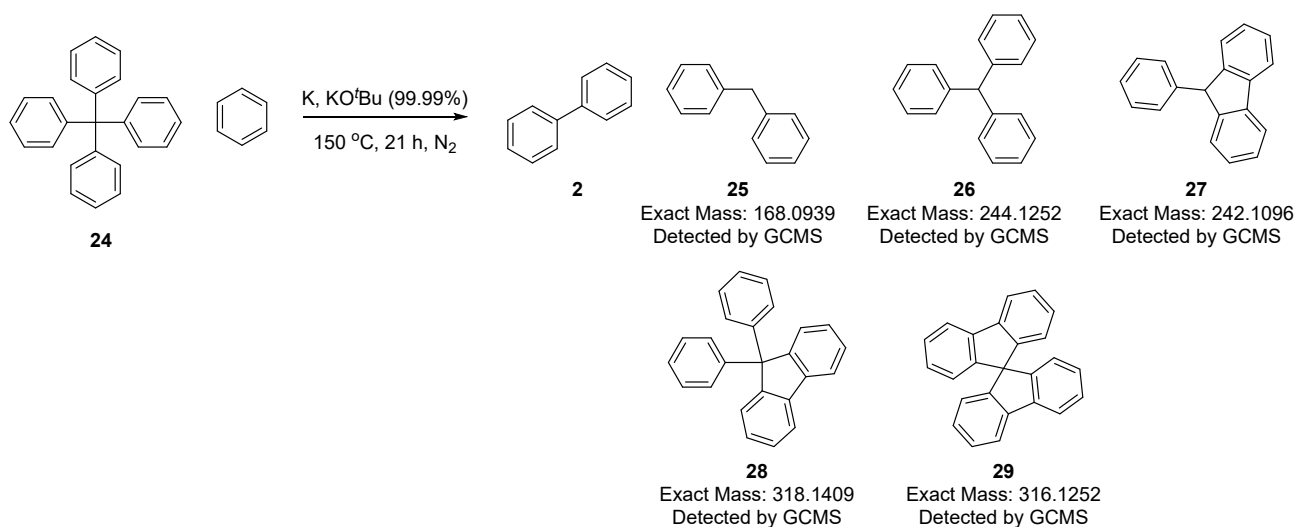


This experiment was carried out according to the **General Procedure** using benzene-*d*₆ with the addition of 4,4'-di-*tert*-butyl-1,1'-biphenyl (**21**) (400 mg, 1.50 mmol). GCMS data shown below. Clean conversion to 4,4'-di-*tert*-butyl-1,1'-biphenyl-*d*₄ + *d*₅ (**21-*d*₄** + **21-*d*₅**) was observed by ¹H NMR and the regioselectivity of the deuteration was confirmed by

nOesy NMR. The ^1H NMR spectrum of the crude reaction product is shown on S25 and the nOeSY NMR data are shown on page S27.



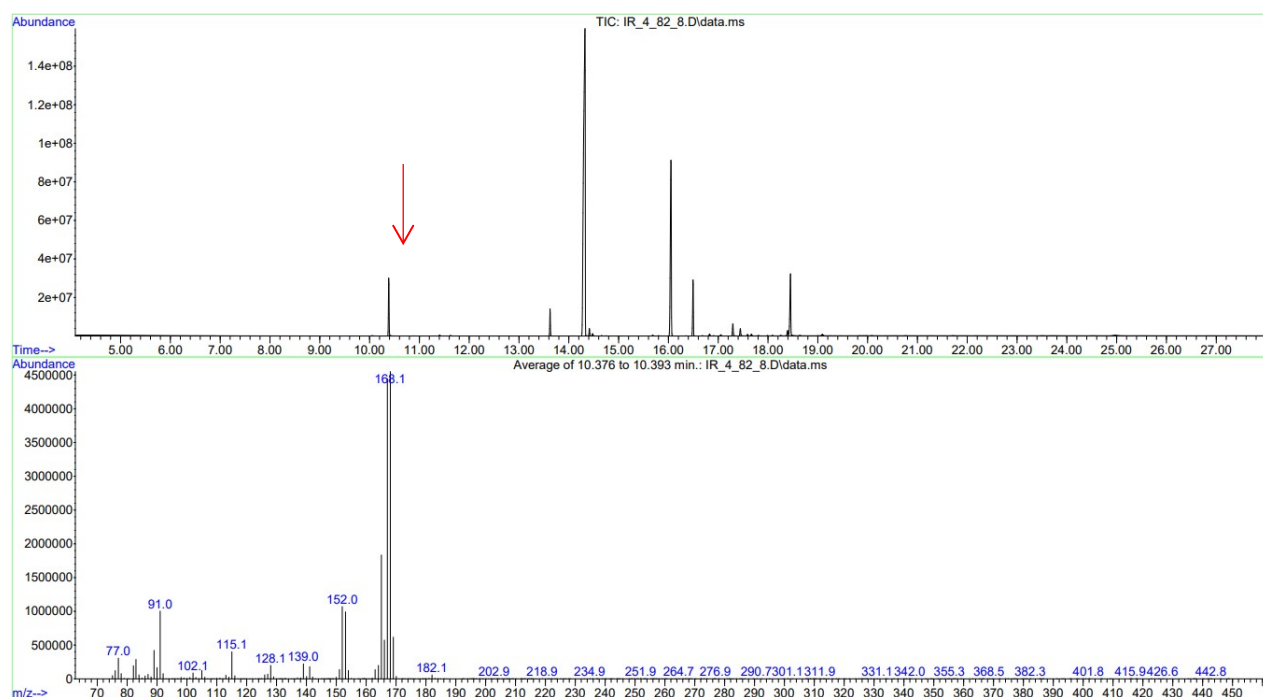
Tetraphenylmethane (24) with benzene



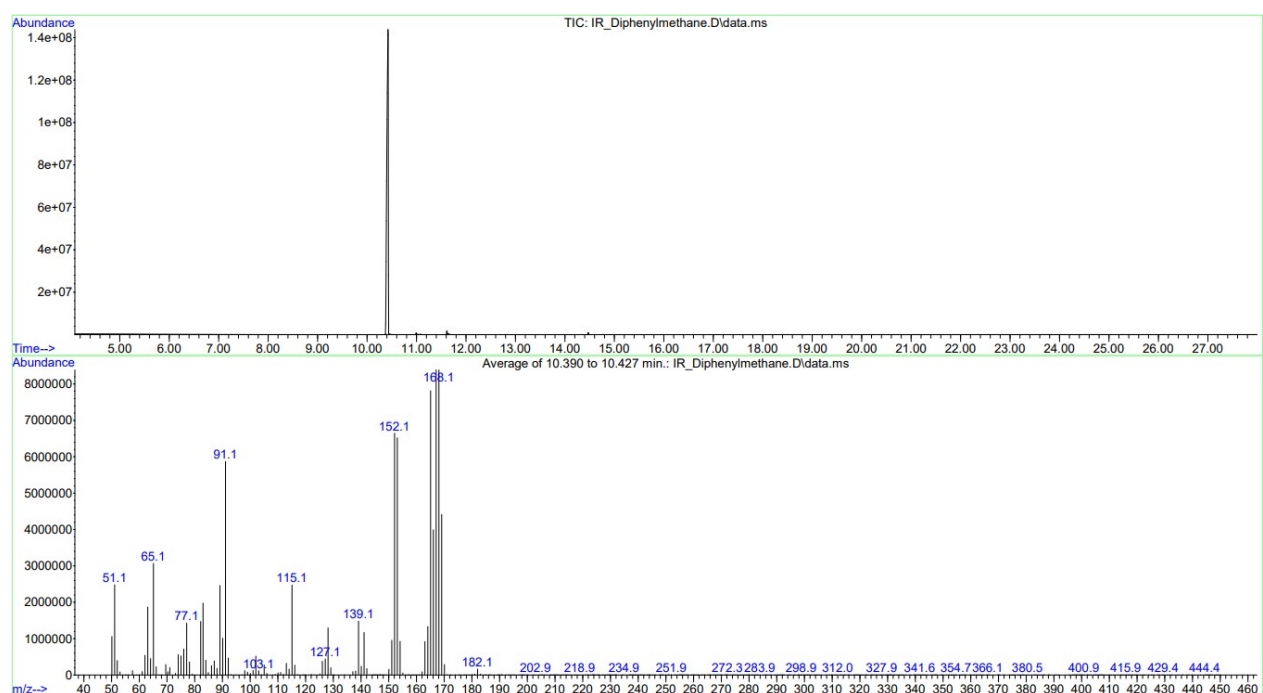
This experiment was carried out according to the **General Procedure** with the addition of tetraphenylmethane (**24**) (481 mg, 1.50 mmol). ¹⁴ Work up gave a mixture of products (126 mg) comprising biphenyl **2** as well as

24-29 (see Scheme 4 in the paper). Purification by column chromatography (0%–4% EtOAc in hexane) gave biphenyl (**2**) (11 mg, 0.071 mmol) as a white solid. M.pt.: 66 – 68 °C (lit: 68 – 70 °C)⁸; ¹H NMR (400 MHz, CDCl₃) δ 7.65 – 7.58 (m, 4H, 4 x ArH), 7.50 – 7.42 (m, 4H, 4 x ArH), 7.40 – 7.33 (m, 2H, 2 x ArH); ¹³C NMR (101 MHz, CDCl₃) δ 140.8, 128.3, 126.8, 126.7; IR ν_{max} /cm⁻¹ (neat): 3063 (w), 3037 (w), 1482 (m), 1431 (m), 731 (vs), 696 (vs); *m/z* (EI): 154.1 ([M]⁺,100), 128.1 (9), 76.1 (17). The NMR spectral data agree with the literature.⁹ Tetraphenylmethane **24** (5 mg) was also recovered in addition to a mixture of products (74 mg). Repeated purification by chromatography (0%–2% EtOAc in hexane) afforded products **25-29** as an inseparable mixture (57 mg) and characterised through GCMS analysis and comparison with a pure commercially available sample: diphenylmethane (**25**) and triphenylmethane (**26**) or literature mass spectra: 9-phenyl-9*H*-fluorene (**27**),^{10,11} 9,9-diphenyl-9*H*-fluorene (**28**)^{12,13} and 9,9'-spirobi[fluorene] (**29**).^{14,15} GCMS data are shown below.

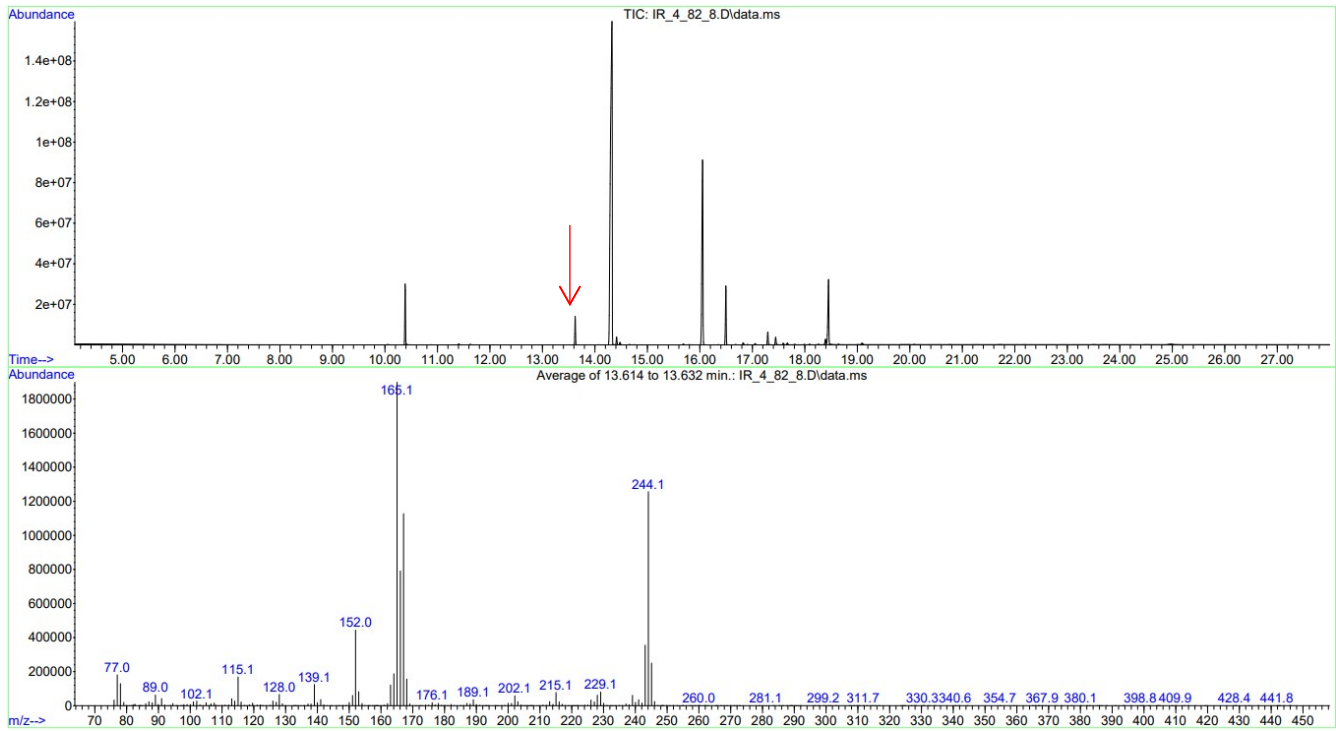
Diphenylmethane (**25**) in mixture



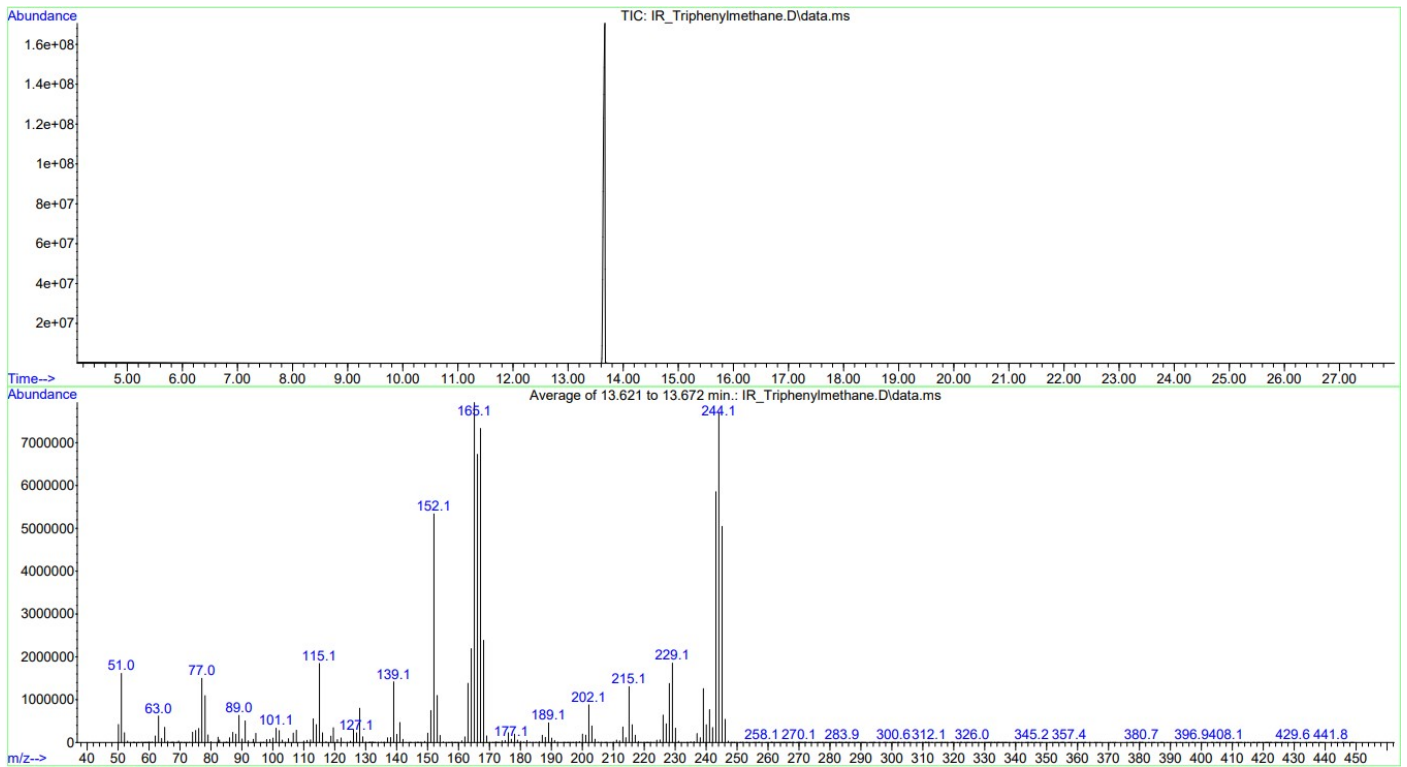
Diphenylmethane (**25**) commercially sourced



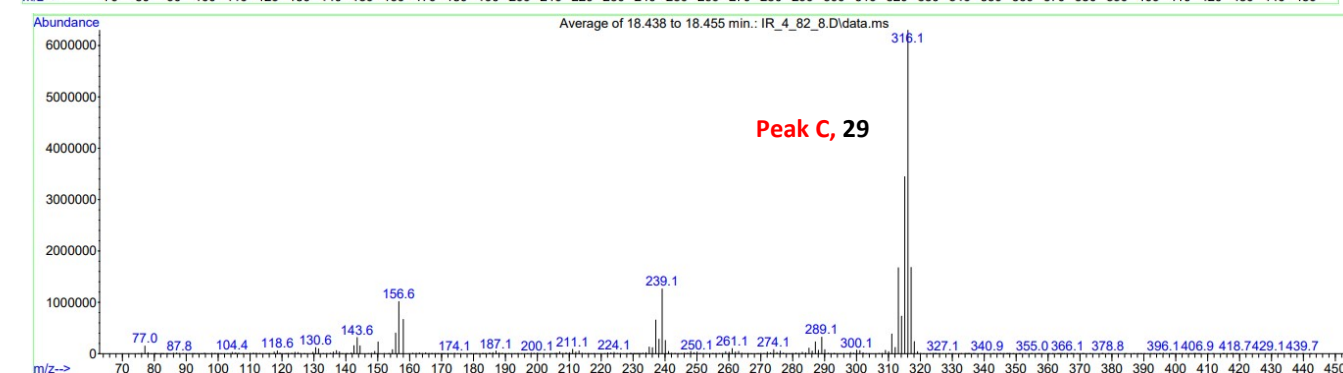
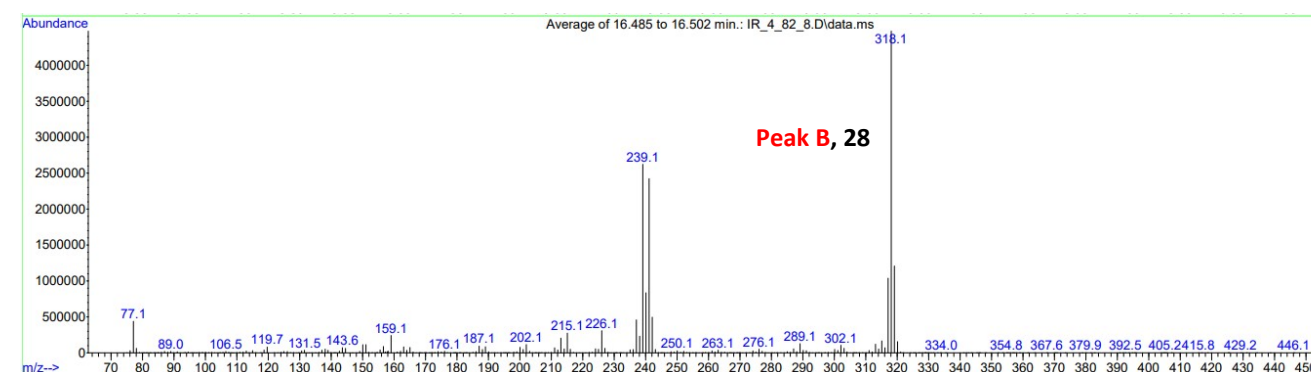
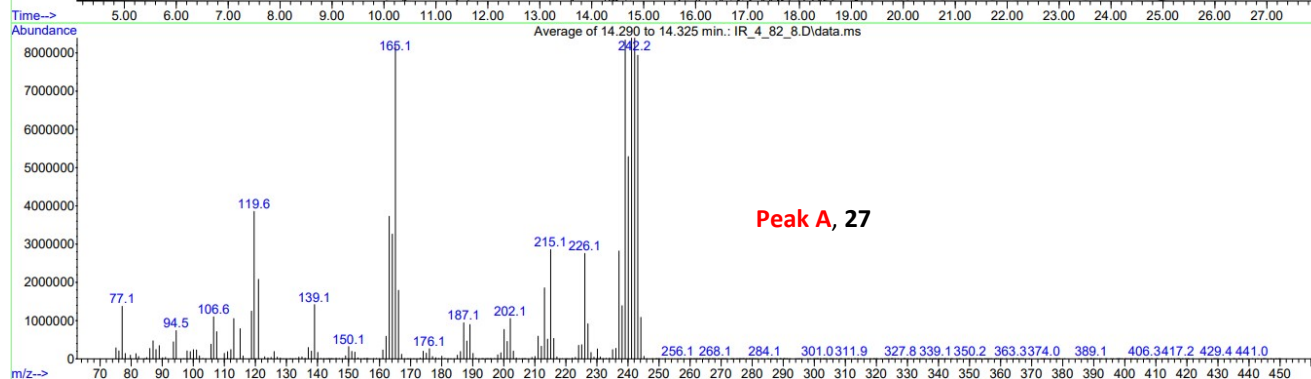
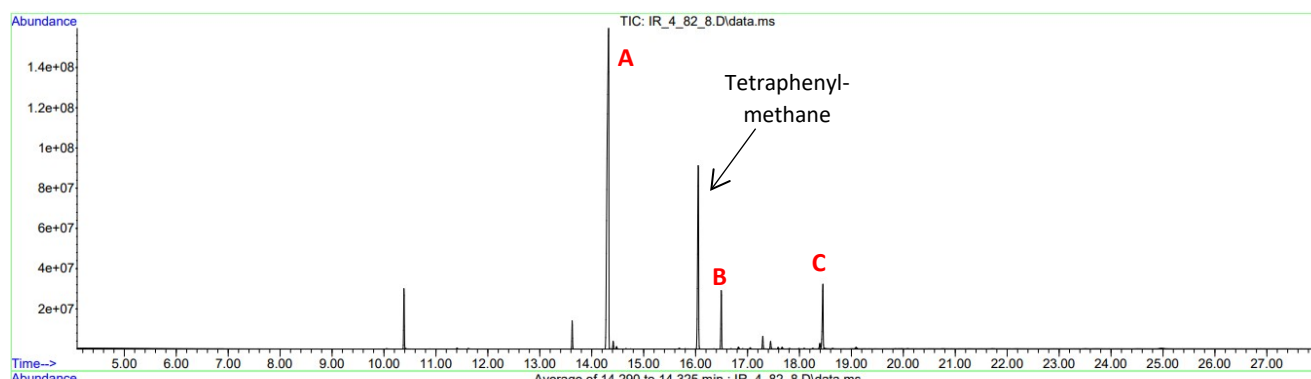
Triphenylmethane (26) in mixture



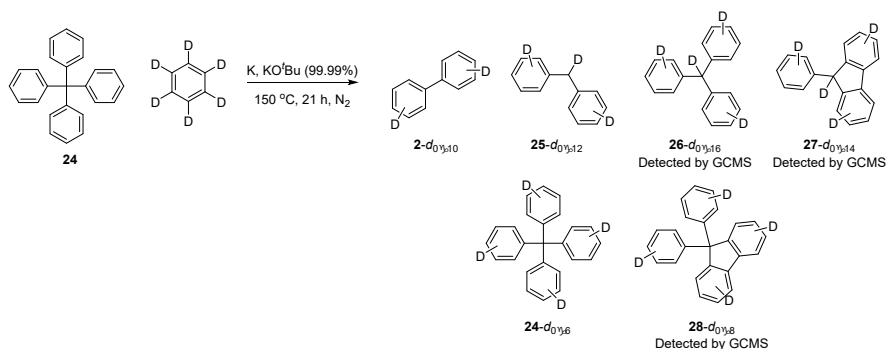
Triphenylmethane (26) commercially sourced



9-Phenyl-9H-fluorene (**27**, m/z 242), 9,9-diphenyl-9H-fluorene (**28**, m/z 318) and 9,9'-spirobi[fluorene] (**29**, m/z 316) in the mixture.



Tetraphenylmethane (**24**) with benzene- d_6 (see Scheme 6 in the paper).

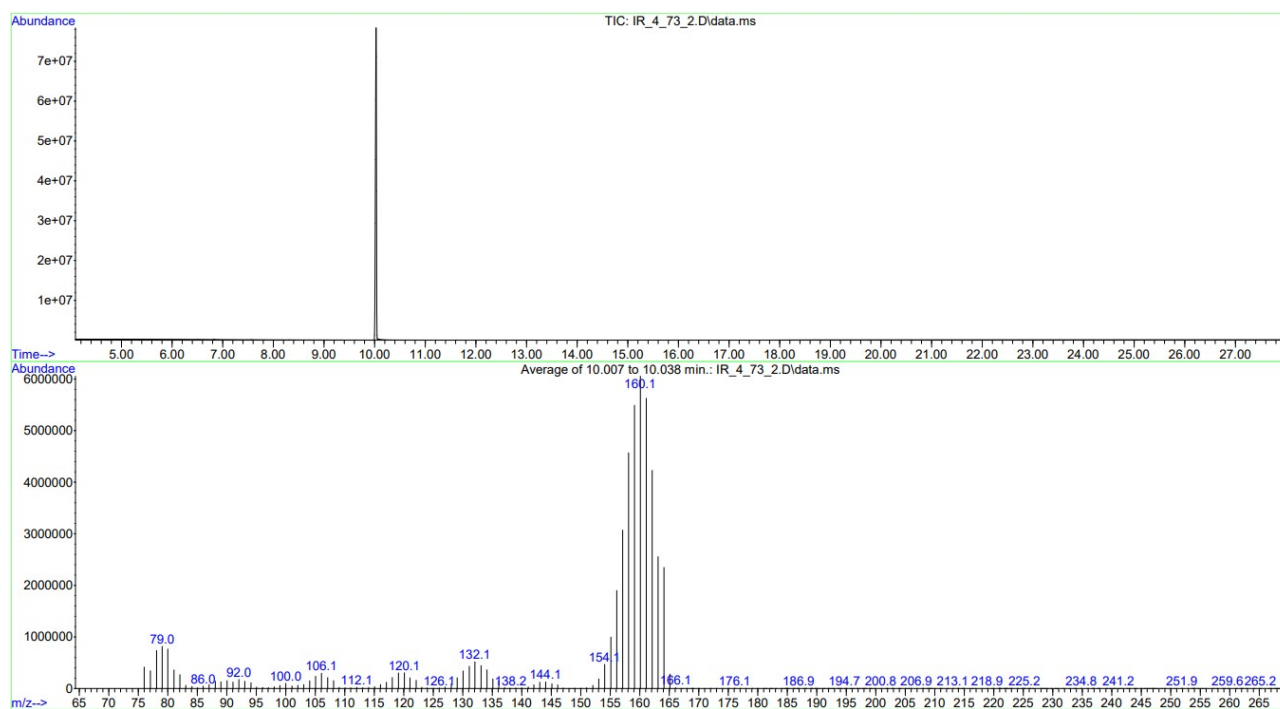


This reaction was carried out according to **General Procedure** using benzene- d_6 with the addition of tetraphenylmethane (**24**) (481 mg, 1.50 mmol). Workup of this reaction gave a mixture of products (176 mg). This was purified by column chromatography (0%–2% EtOAc in hexane) which gave biphenyl- d_{10} (**2**- d_{10}) (13 mg) as a white solid, diphenylmethane- d_{12} (**25**- d_{12}) (4 mg) as an off-white solid and tetraphenylmethane- d_6 (**24**- d_6) (2 mg) as a white solid. A ^1H NMR spectrum for each of these products is shown on pages S21–S22.

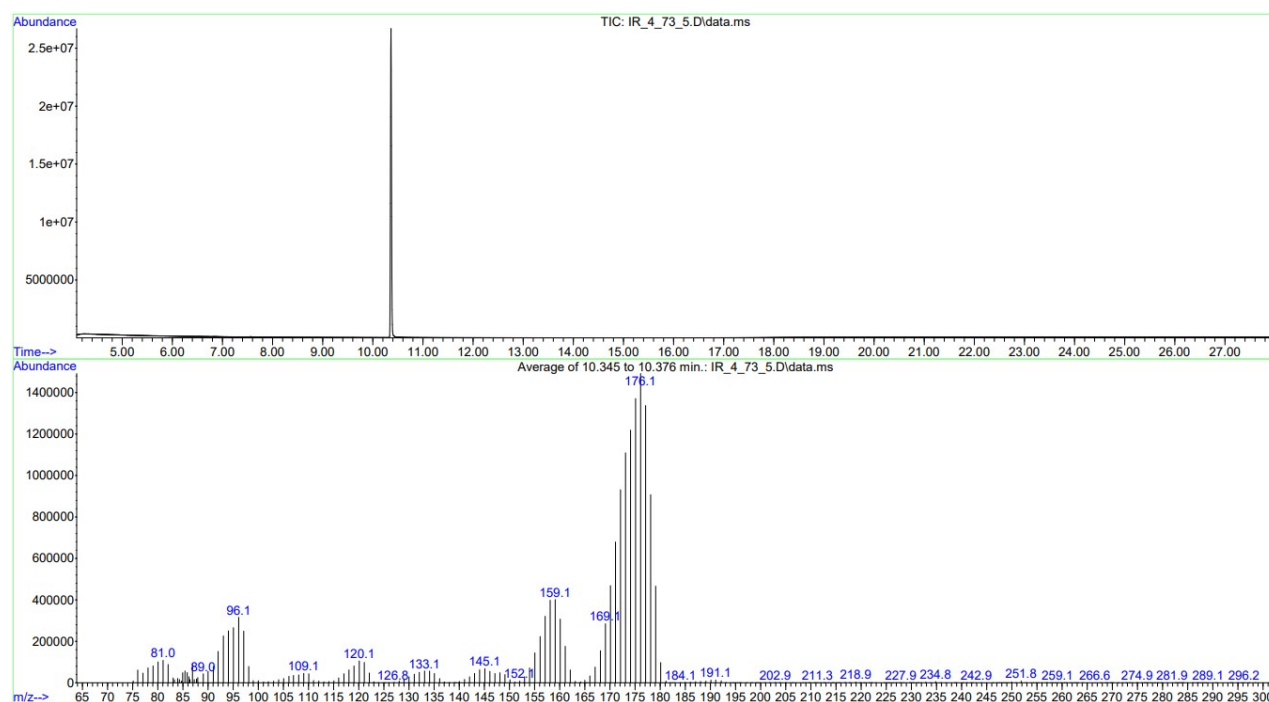
Products **26**- d_{16} , **27**- d_{14} and more **25**- d_{12} , together with traces of **26**- d_{16} , and **28**- d_8 , were recovered as an inseparable mixture (98 mg) and were characterised through GCMS analysis.

GCMS data are shown below.

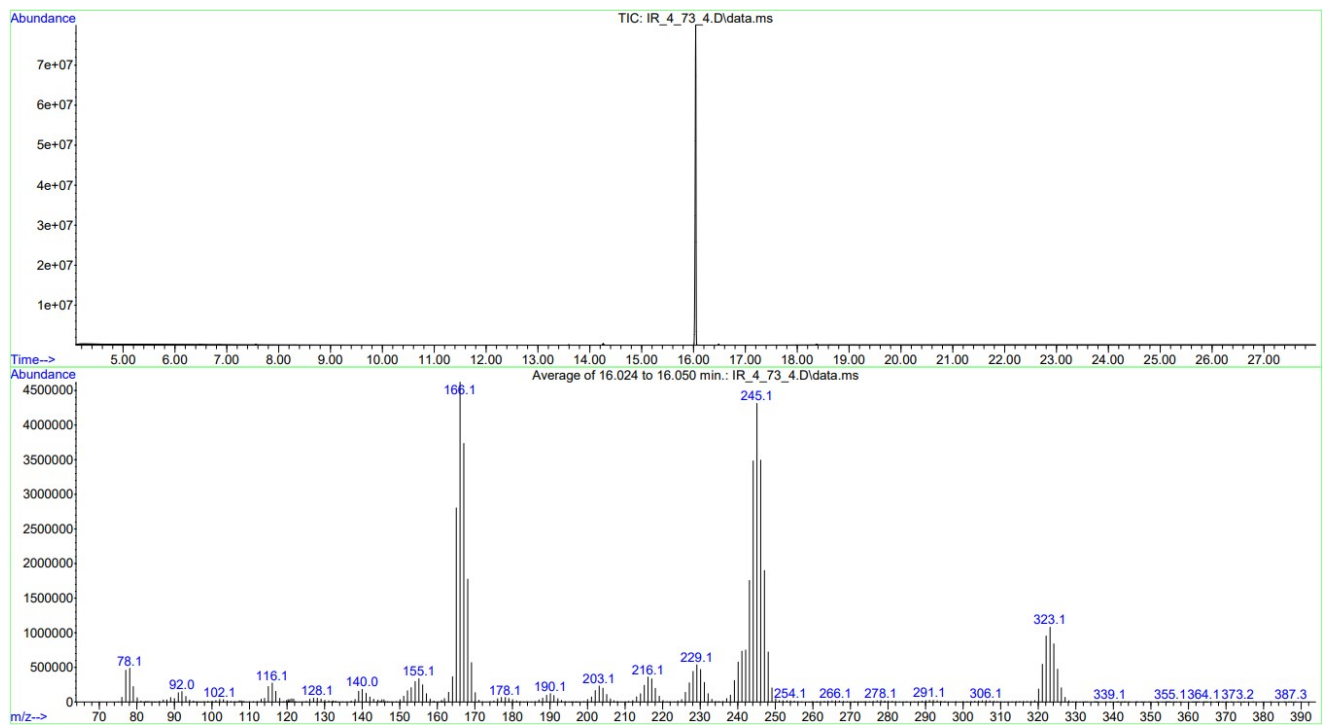
Biphenyl **2**- d_{10}



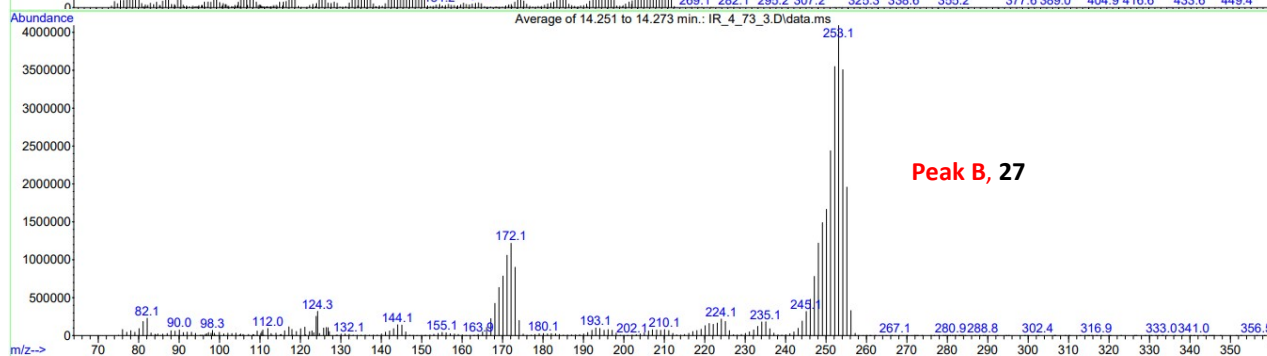
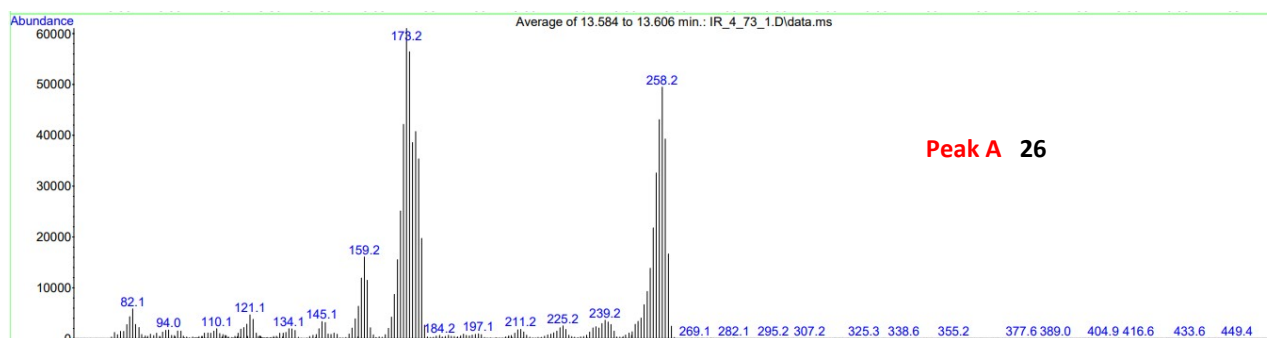
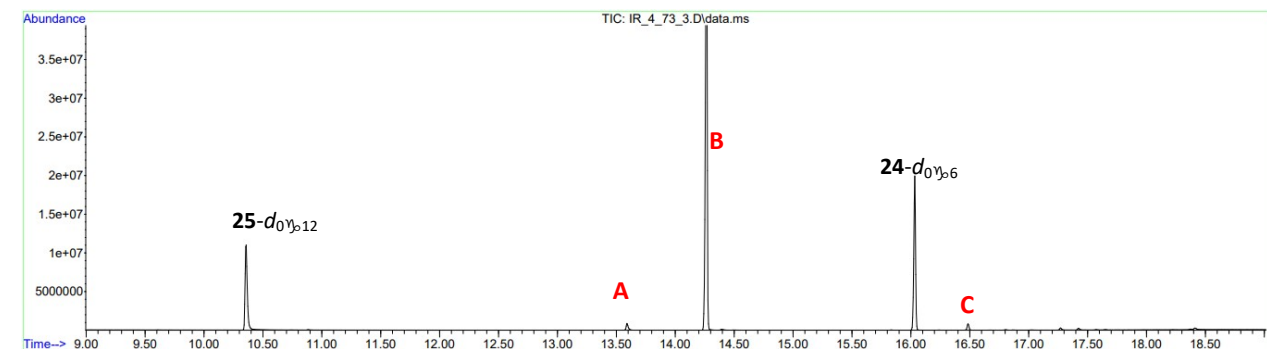
Diphenylmethane- d_{12} (**25**- d_{12})

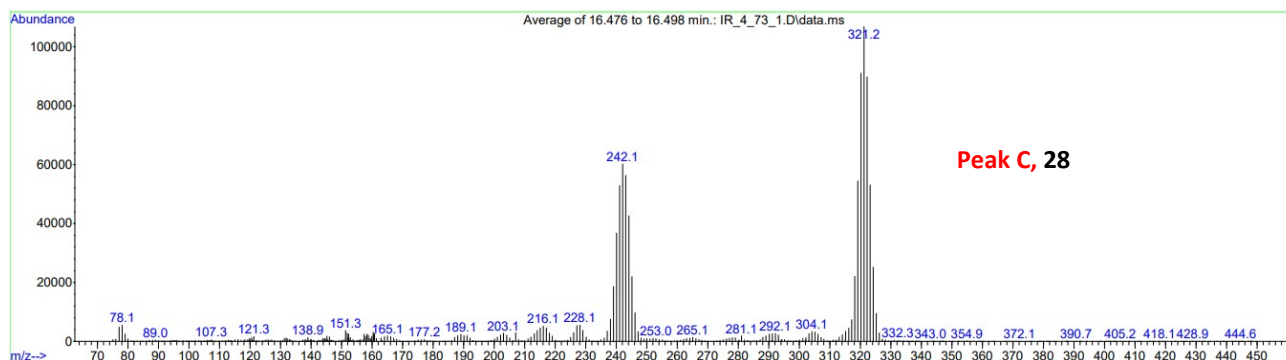


Tetraphenylmethane- $d_{0\gamma_6}$ ($24-d_{0\gamma_6}$)

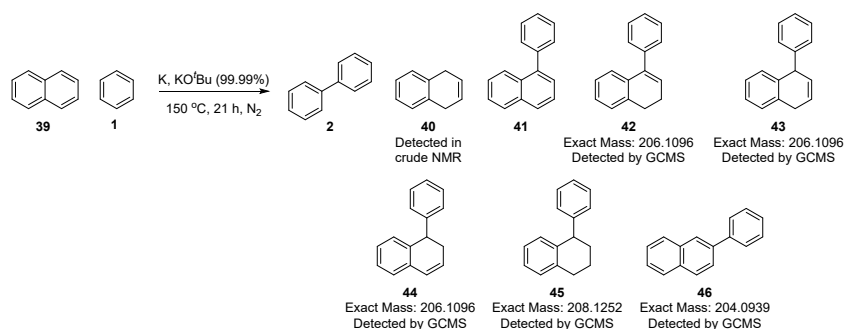


Triphenylmethane ($26-d_{0\gamma_{16}}$), 9-Phenyl-9H-fluorene ($27-d_{0\gamma_{14}}$) and 9,9-diphenyl-9H-fluorene ($28-d_{0\gamma_{18}}$) in mixture





Naphthalene (**39**) with benzene (see Scheme 7 in the paper)

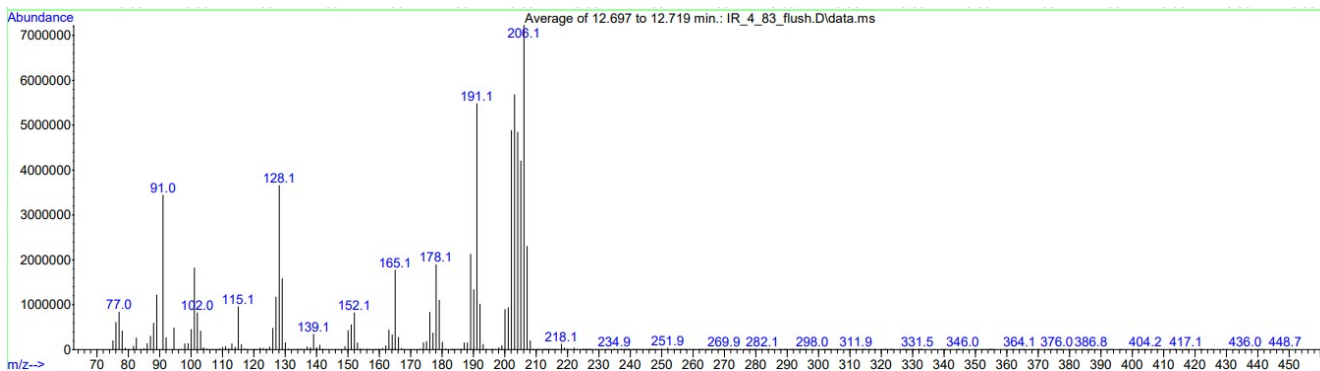
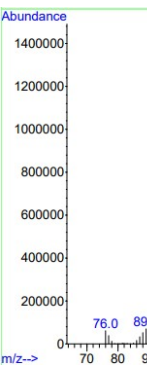
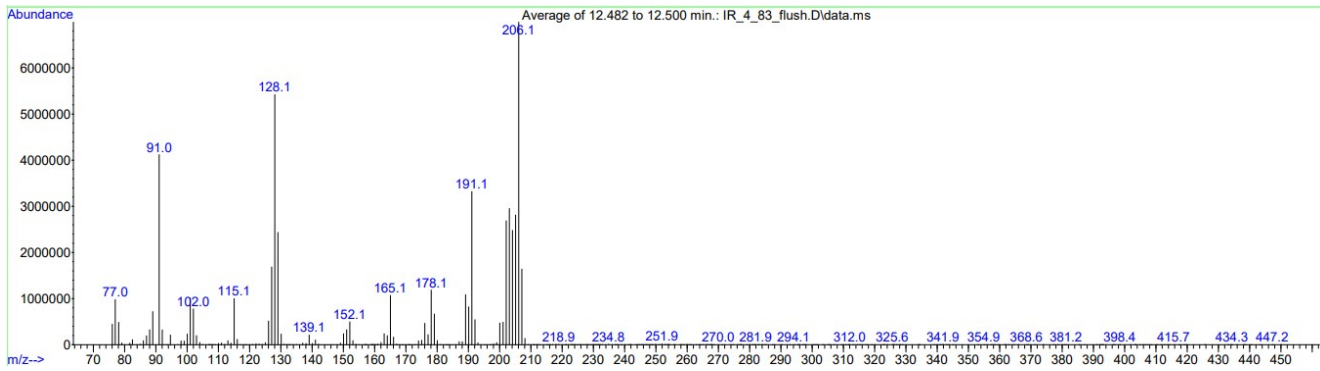
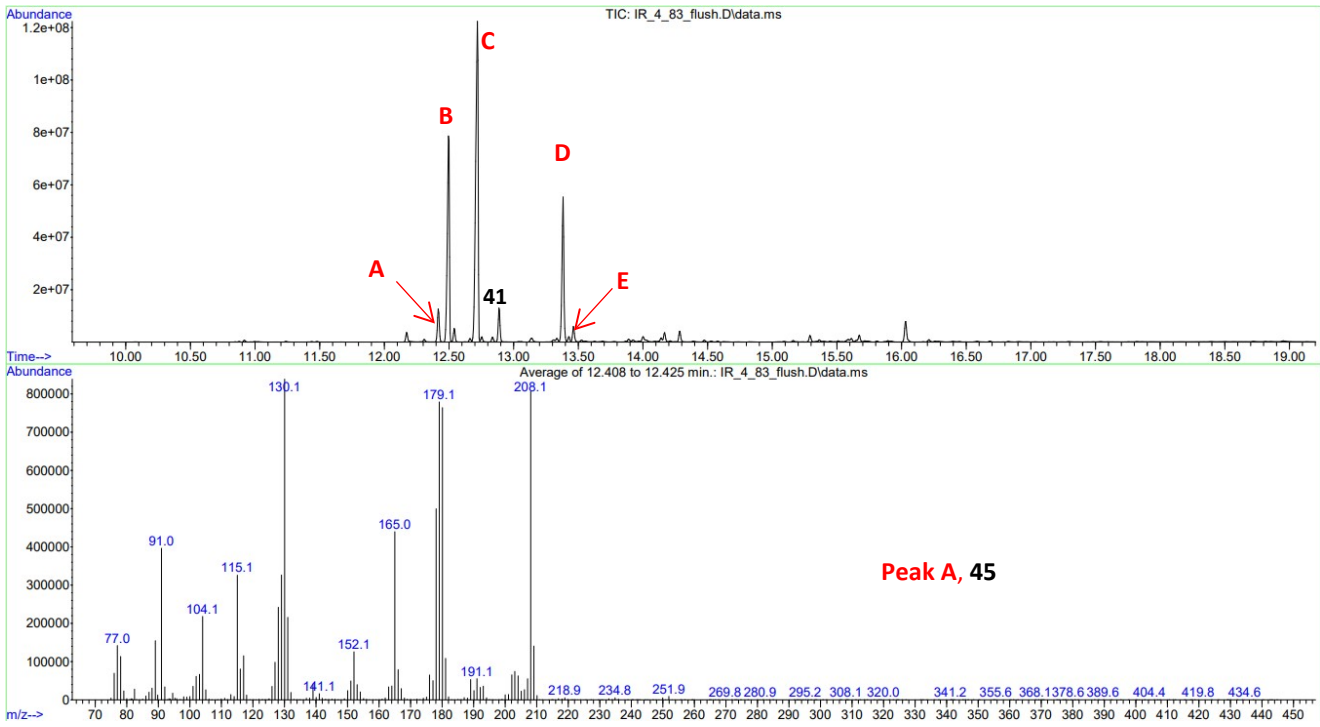


This reaction was carried out according to the **General Procedure** with the addition of naphthalene (**39**) (192 mg, 1.50 mmol) to give a mixture of products (127 mg), consisting principally of naphthalene **39**, dihydronaphthalene **40** and biphenyl **2**. This was subjected to column chromatography (hexane) which gave biphenyl (**2**) (5 mg, 0.032 mmol) as a white solid and also 1-phenylnaphthalene (**41**) (2 mg, 0.01 mmol), <1% as a yellow oil. ^1H NMR (400 MHz, CDCl_3) δ 7.95 – 7.82 (m, 3H, 3 x ArH), 7.57 – 7.39 (m, 9H, 9 x ArH); ^{13}C NMR (101 MHz, CDCl_3) δ 140.3, 139.8, 133.3, 131.1, 129.6, 127.8, 127.1, 126.7, 126.4, 125.5, 125.3, 124.9; IR $\nu_{\text{max}}/\text{cm}^{-1}$ (neat): 3060 (w), 1596 (w), 1399 (m), 780 (vs); m/z (EI): 204.1 ($[\text{M}]^+$, 96), 203.1 (100), 176.0 (9), 101.0 (23). The NMR spectral data are in agreement with literature.¹⁶

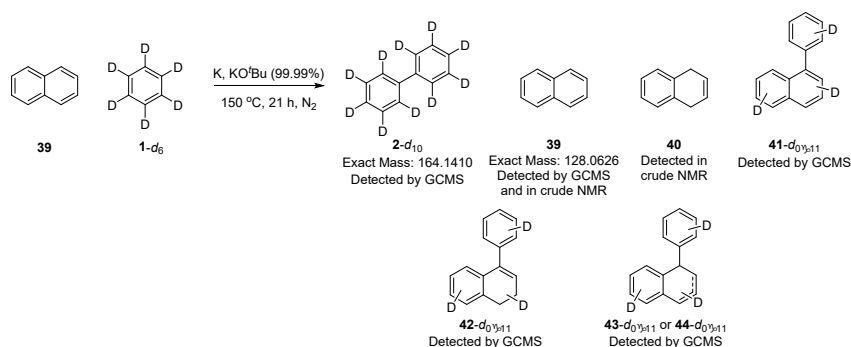
1,4-Dihydronaphthalene (**40**) was detected in the crude ^1H NMR spectrum which shows prominent characteristic peaks which are in agreement with literature.¹⁷ ^1H NMR spectrum is shown on page S25.

Products **42-46** were recovered from chromatography as an inseparable mixture (which also contained **41**) (15 mg) and were therefore tentatively characterised through GCMS analysis and comparison with literature mass spectra: 4-phenyl-1,2-dihydronaphthalene (**42**),¹⁸ 1-phenyl-1,4-dihydronaphthalene (**43**),¹⁹ 1-phenyl-1,2-dihydronaphthalene (**44**),²⁰ 1-phenyl-1,2,3,4-tetrahydronaphthalene (**45**)²¹ and 2-phenylnaphthalene (**46**).²² GCMS data shown below.

^1H NMR of analysis of the mixture shows prominent characteristic peaks which also support the formation of isomer (**42**) which are in agreement with literature.²³ The ^1H NMR spectrum is shown on page S26.

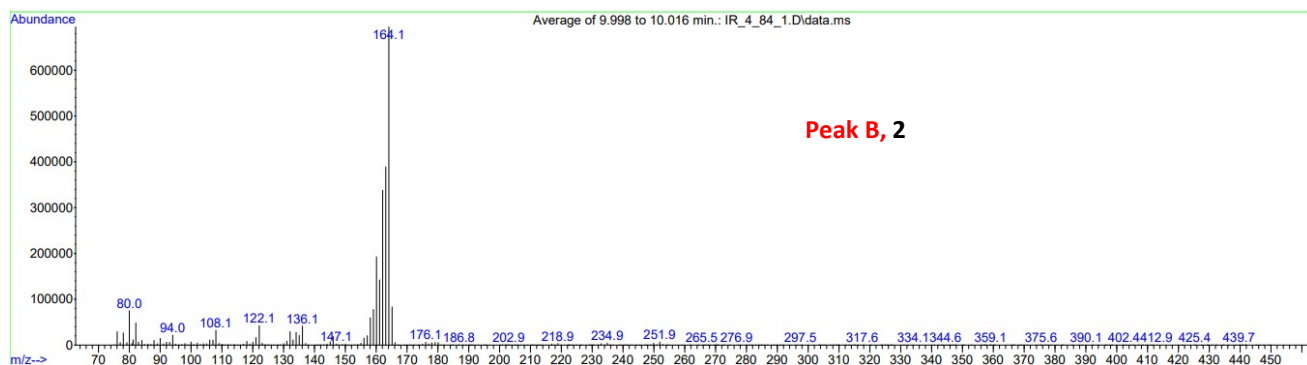
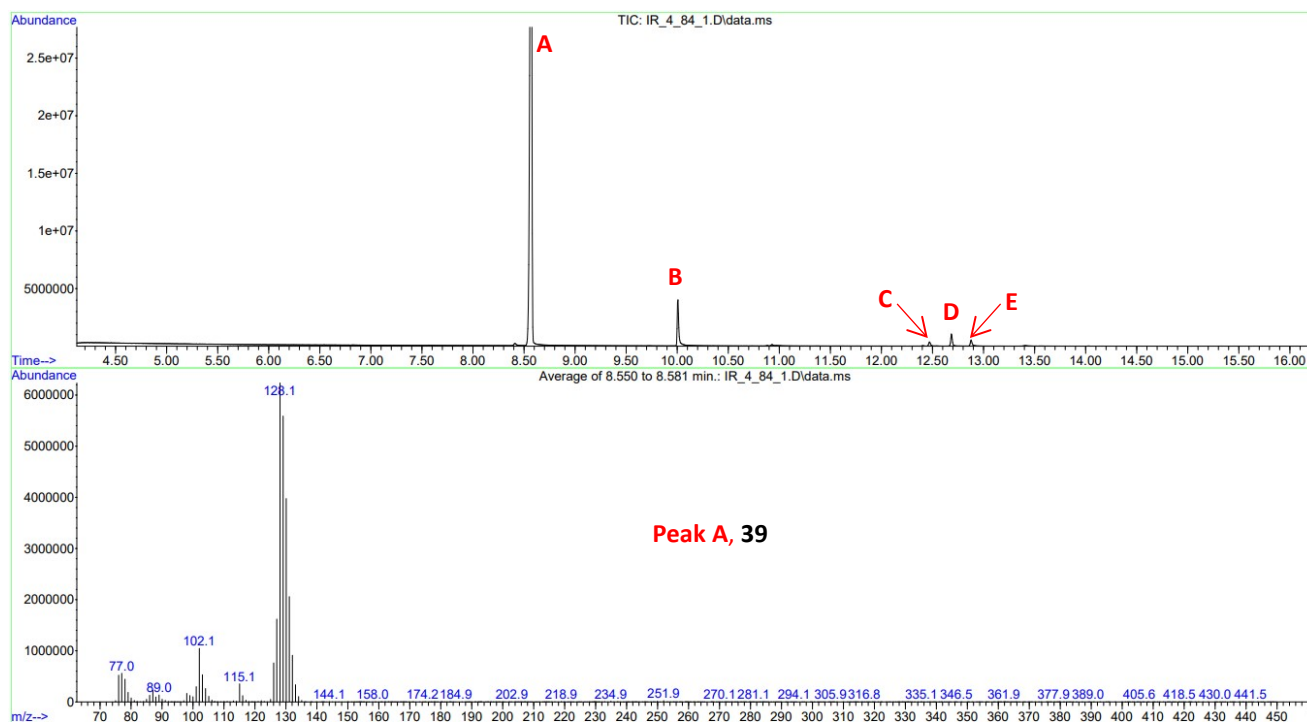


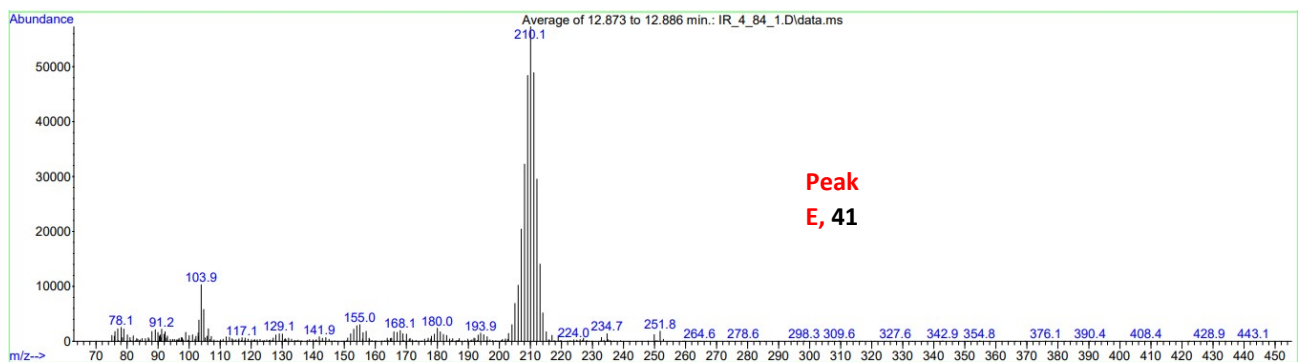
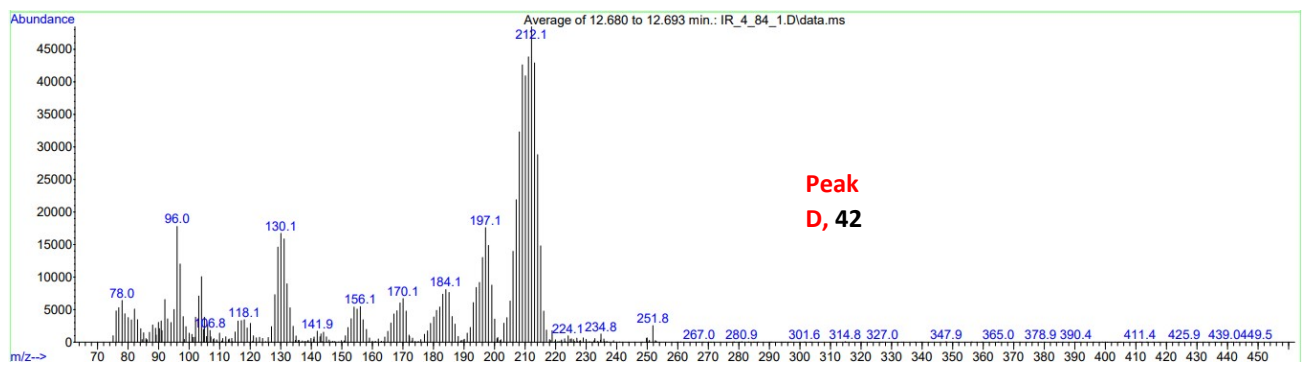
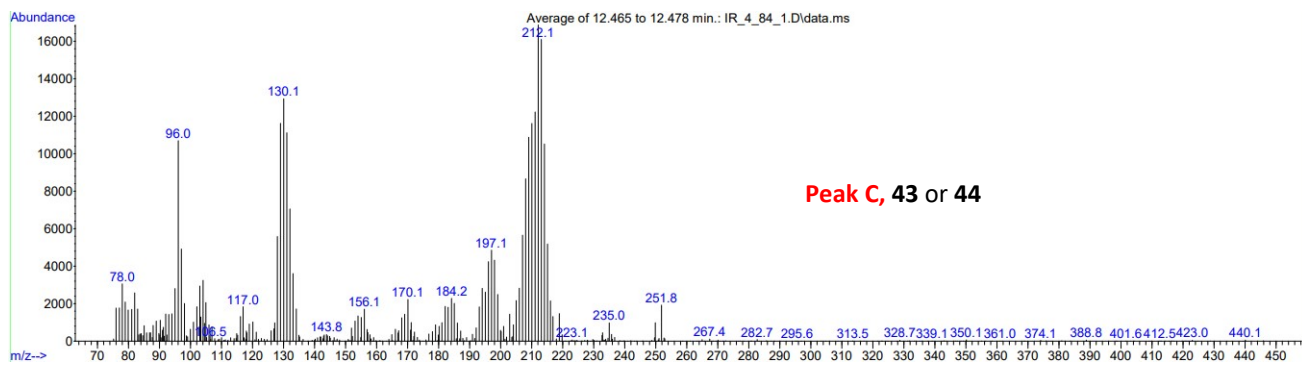
Naphthalene (**39**) with benzene- d_6 (see Scheme 8 in the paper).



This experiment was carried out according to the **General Procedure** using benzene- d_6 with the addition of naphthalene (**39**) (192 mg, 1.50 mmol). Workup and evaporation gave a mixture of products (102 mg). Biphenyl- d_{10} (**2- d_{10}**), 1-phenylnaphthalene- $d_{0y_{b11}}$ (**41- $d_{0y_{b11}}$**), 4-phenyl-1,2-dihydronaphthalene- $d_{0y_{b11}}$ (**42- $d_{0y_{b11}}$**) and 1-phenyl-1,4-dihydronaphthalene- $d_{0y_{b11}}$ (**43- $d_{0y_{b11}}$**) or 1-phenyl-1,2-dihydronaphthalene- $d_{0y_{b11}}$ (**44- $d_{0y_{b11}}$**) were all detected by GCMS analysis. GCMS data are shown below.

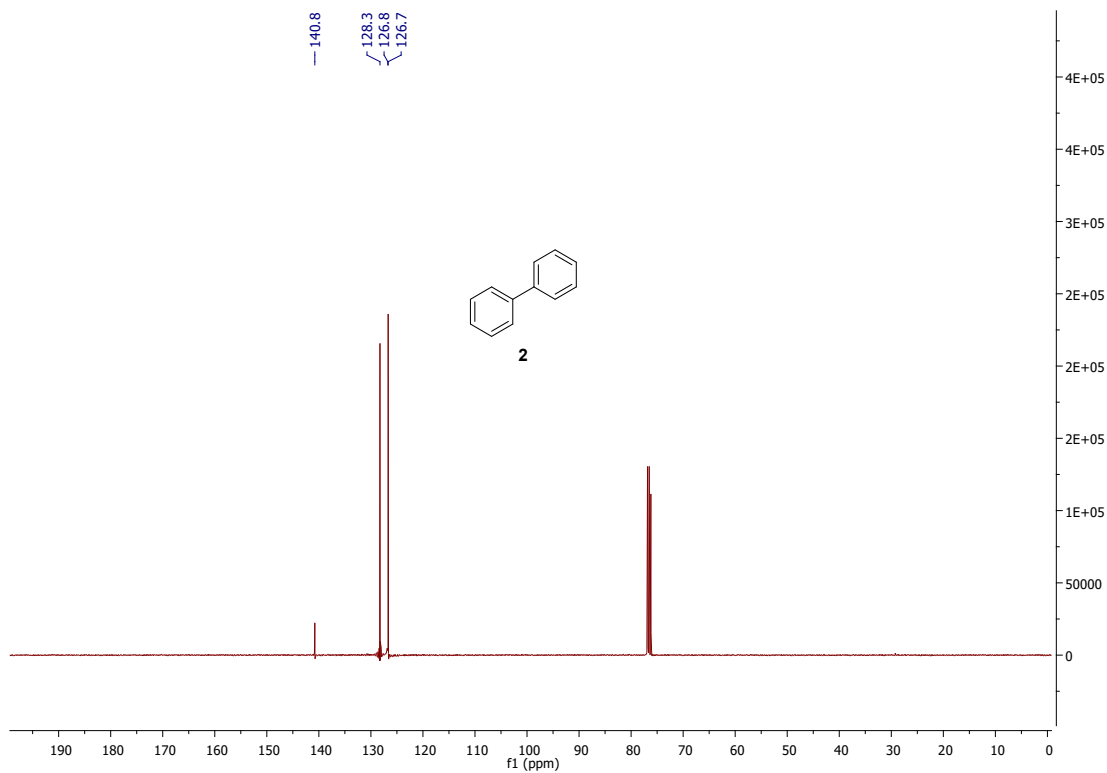
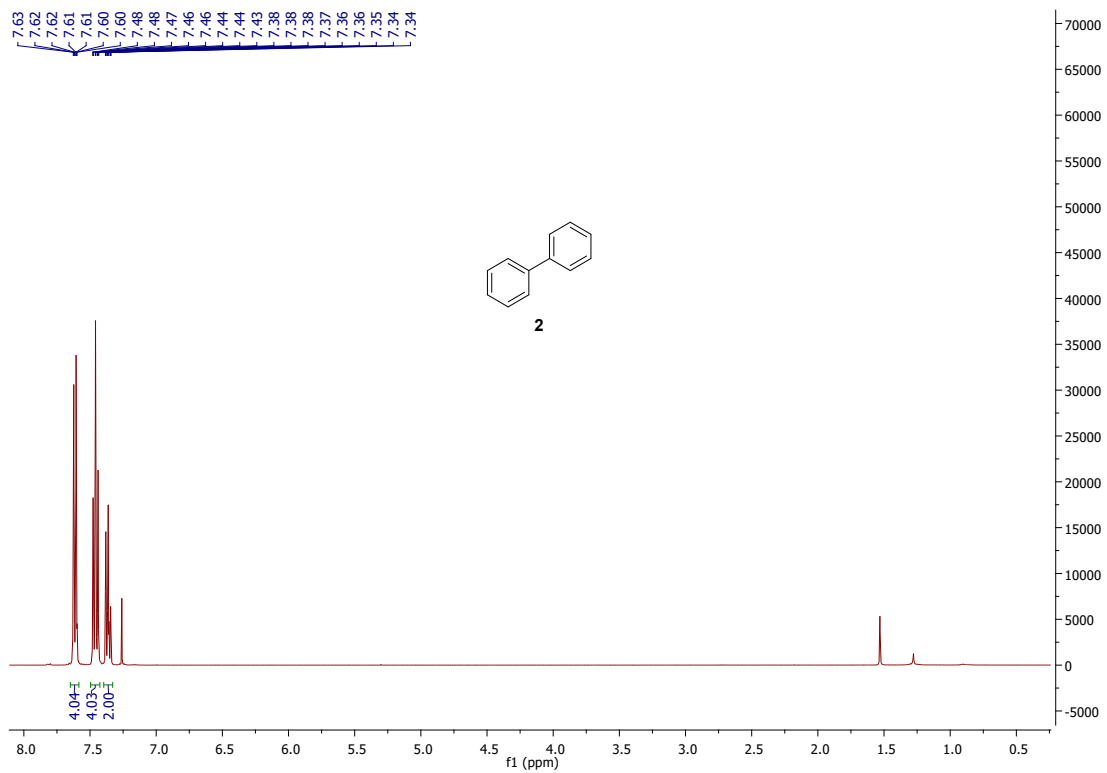
Undeuterated naphthalene (**39**) was observed in the crude ^1H NMR and by GCMS. 1,4-Dihydronaphthalene (**40**) was also detected in the crude ^1H NMR spectrum which shows prominent characteristic peaks which are in agreement with literature.¹³ ^1H NMR spectrum is shown on page S26.



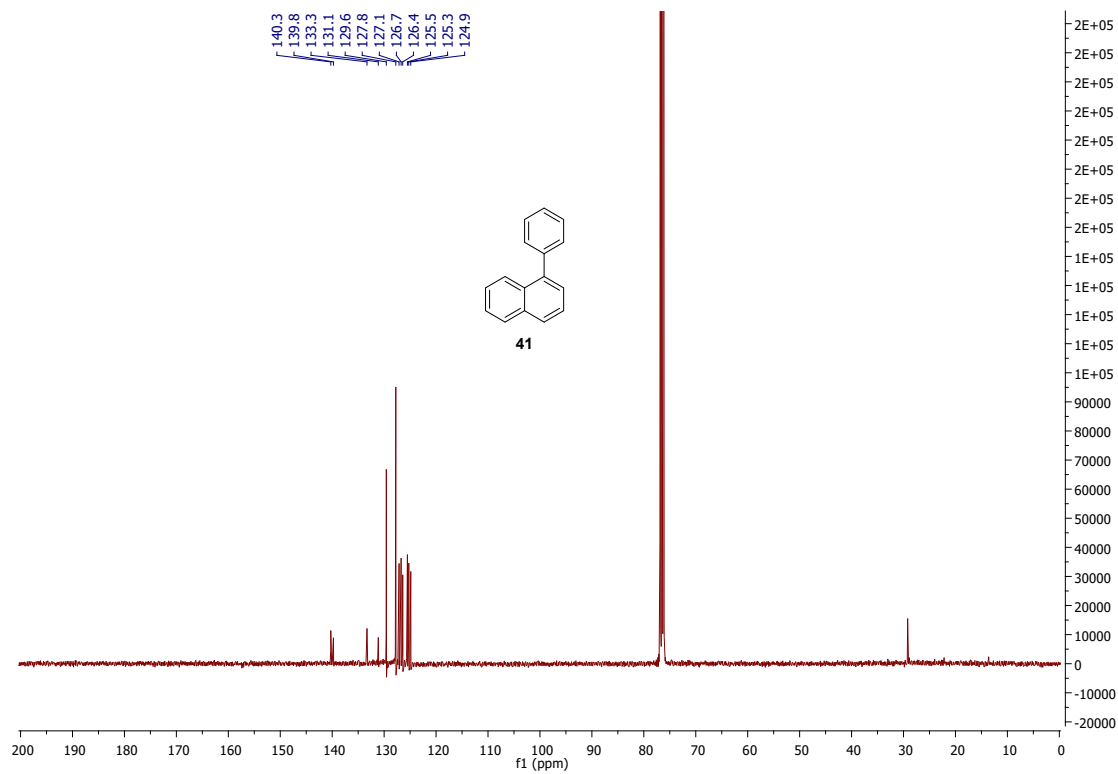
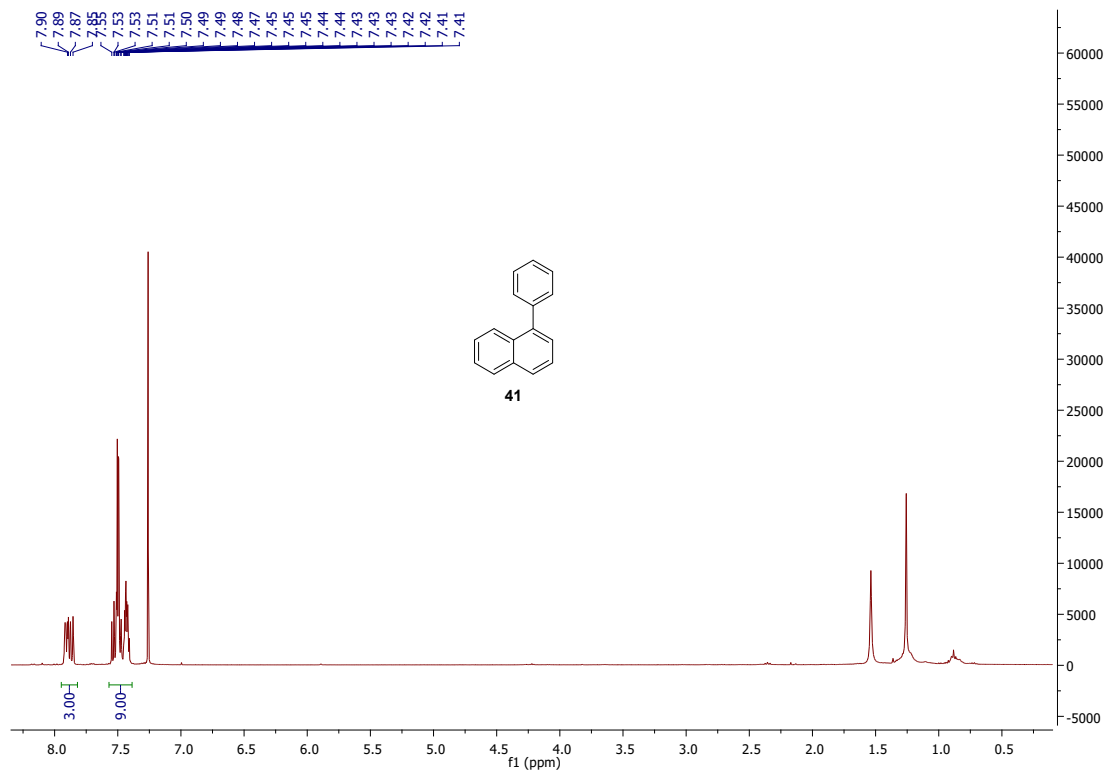


^1H and ^{13}C NMR Spectra

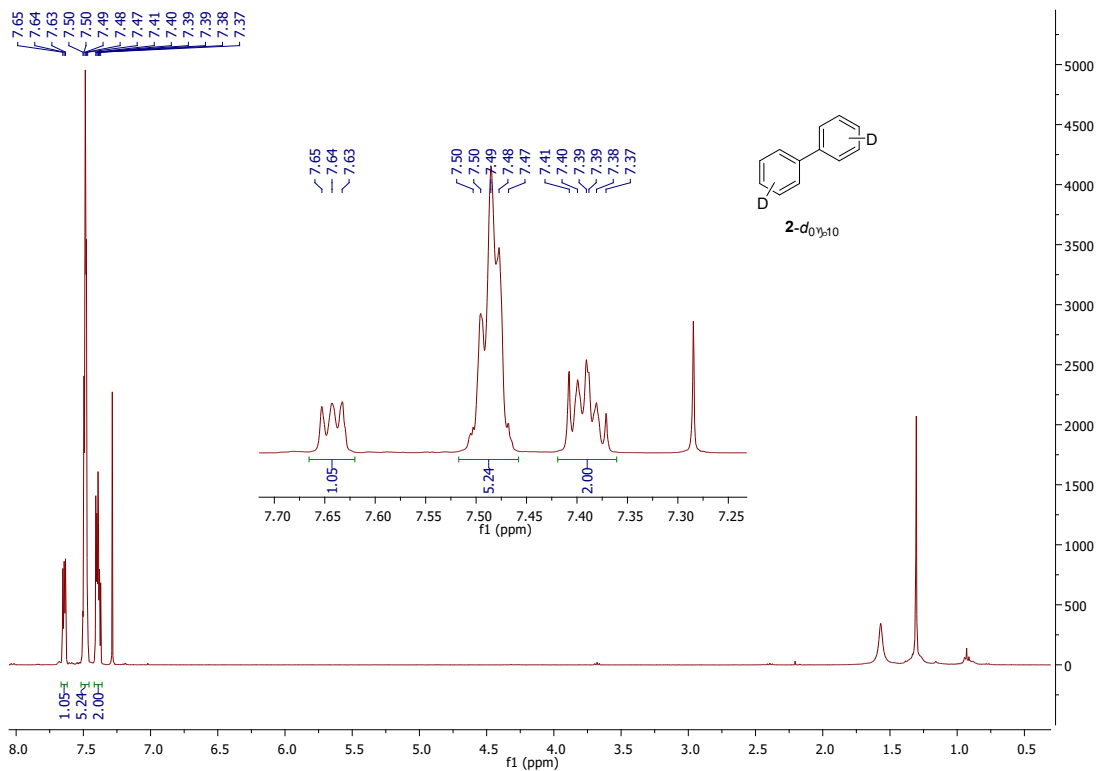
Biphenyl (**2**)



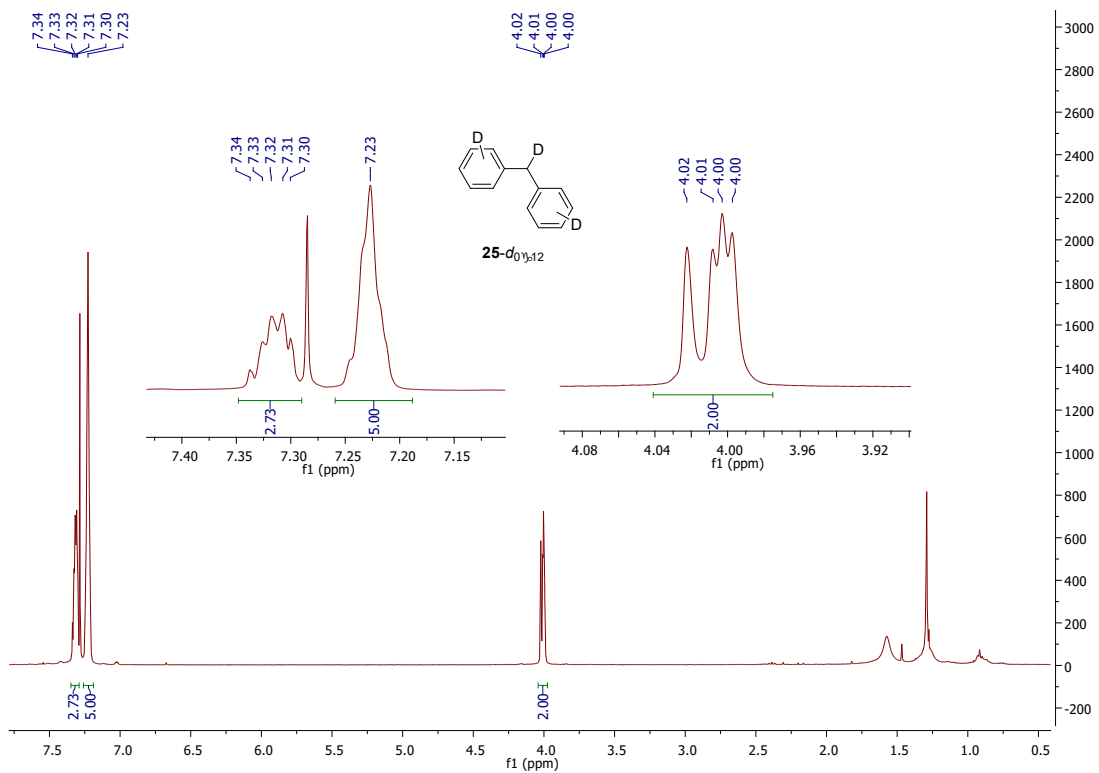
1-Phenylnaphthalene (35)



Biphenyl- $d_{0\gamma b10}$ ($2-d_{0\gamma b10}$)

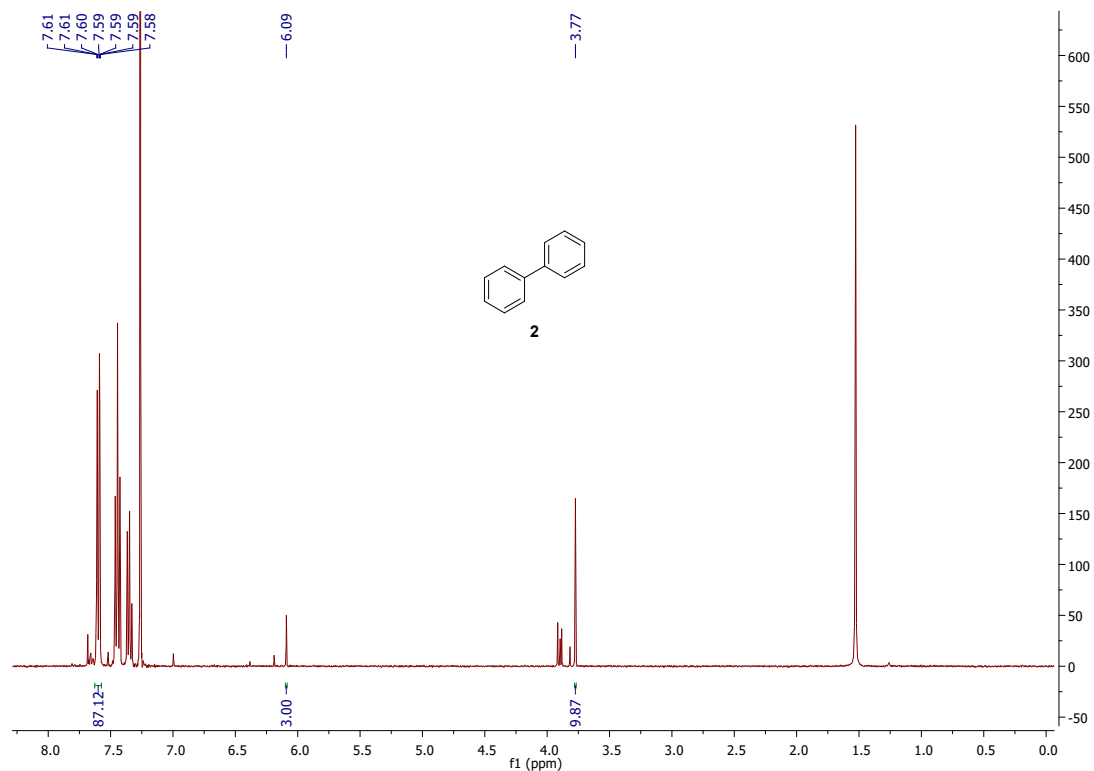


Diphenylmethane-d₀y₁₂ (25-d₀y₁₂)

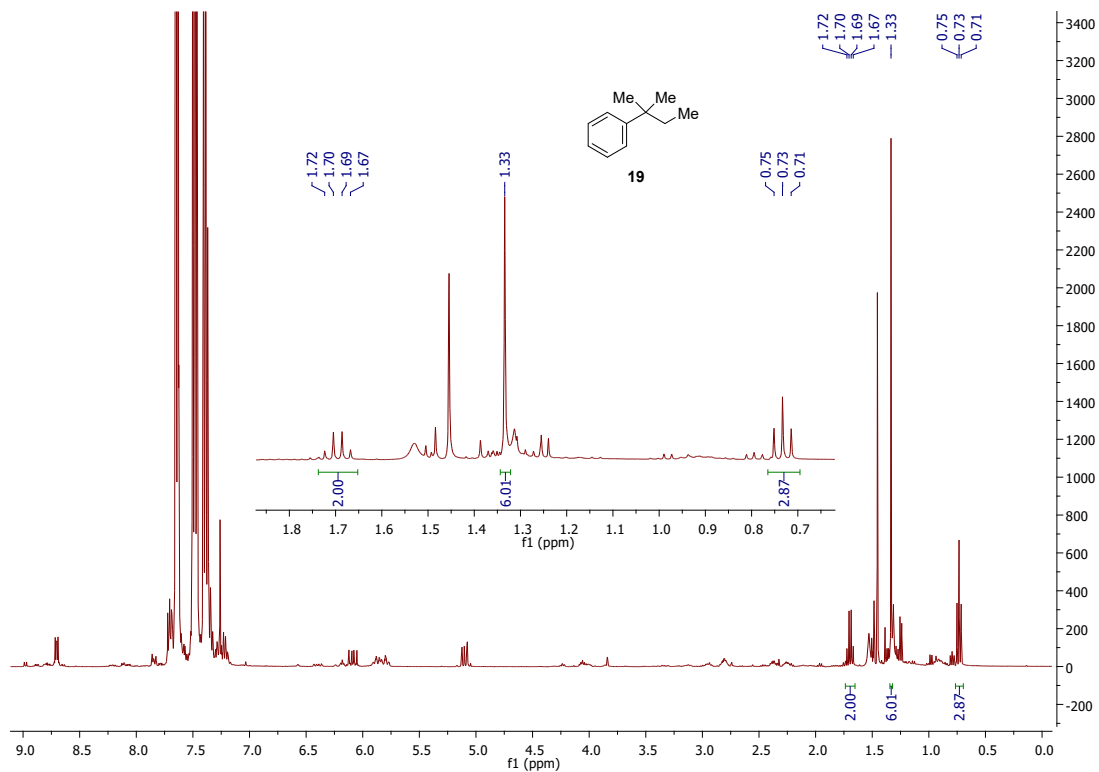


¹H Spectra of Crude Reaction products

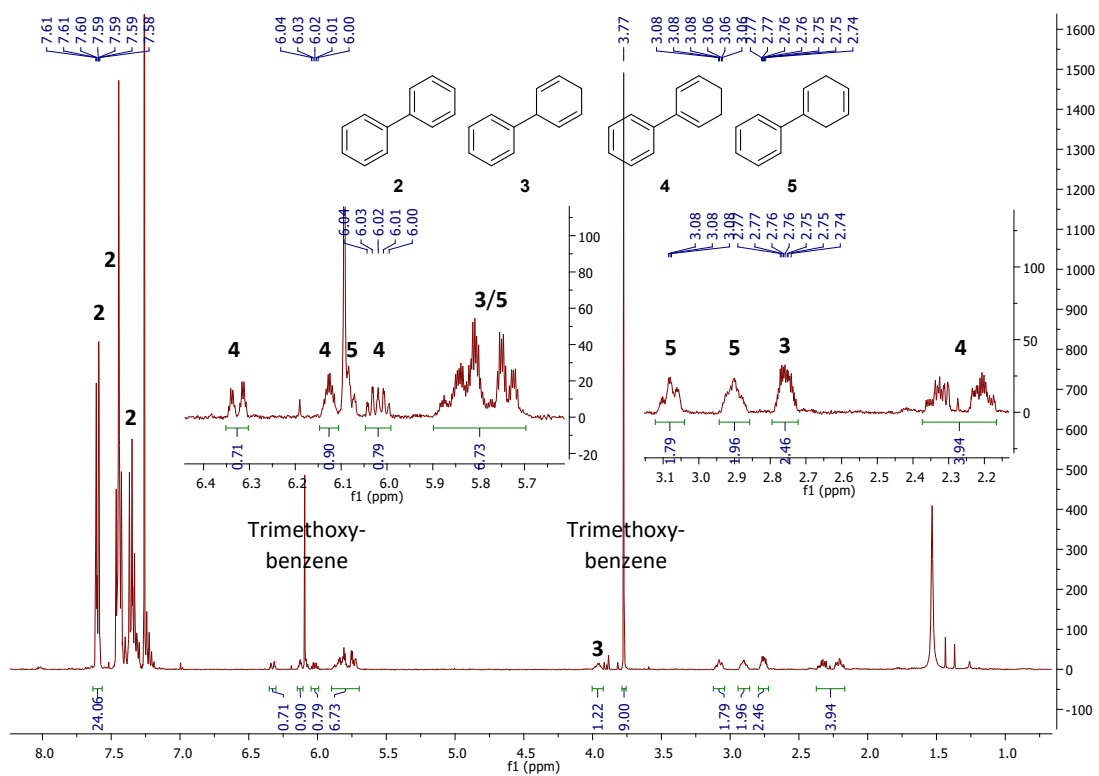
Biphenyl (**2**)



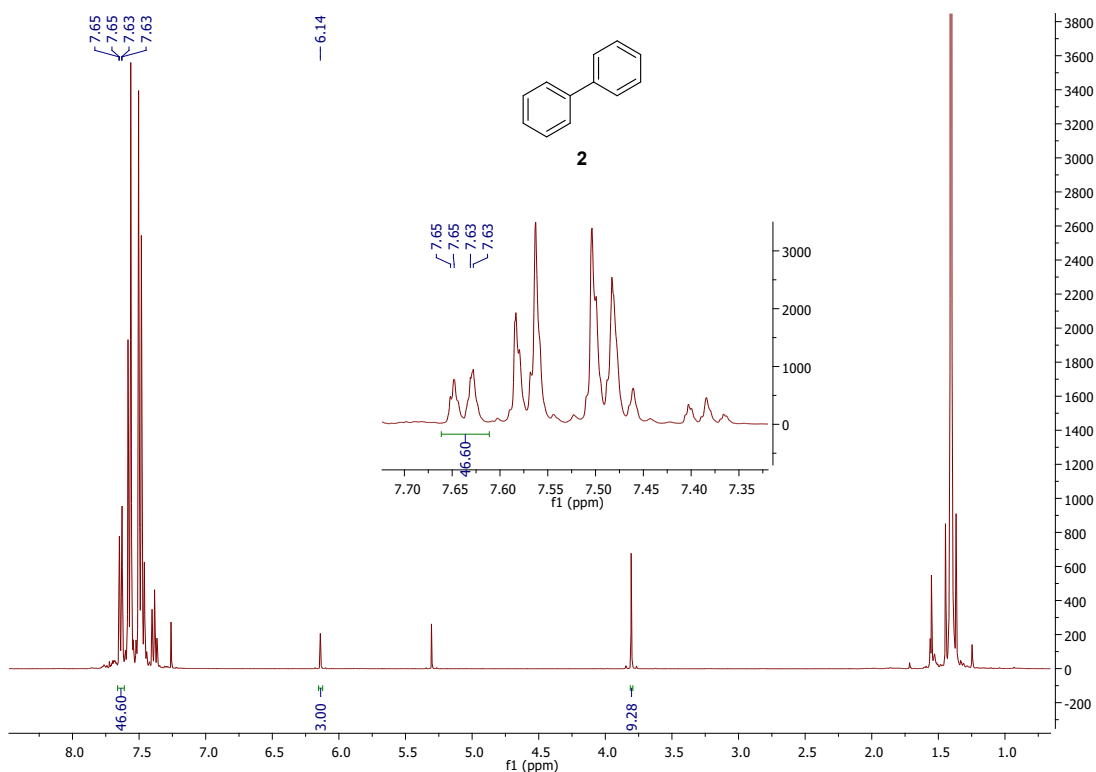
tert-Pentylbenzene (**19**) in crude reaction mixture.



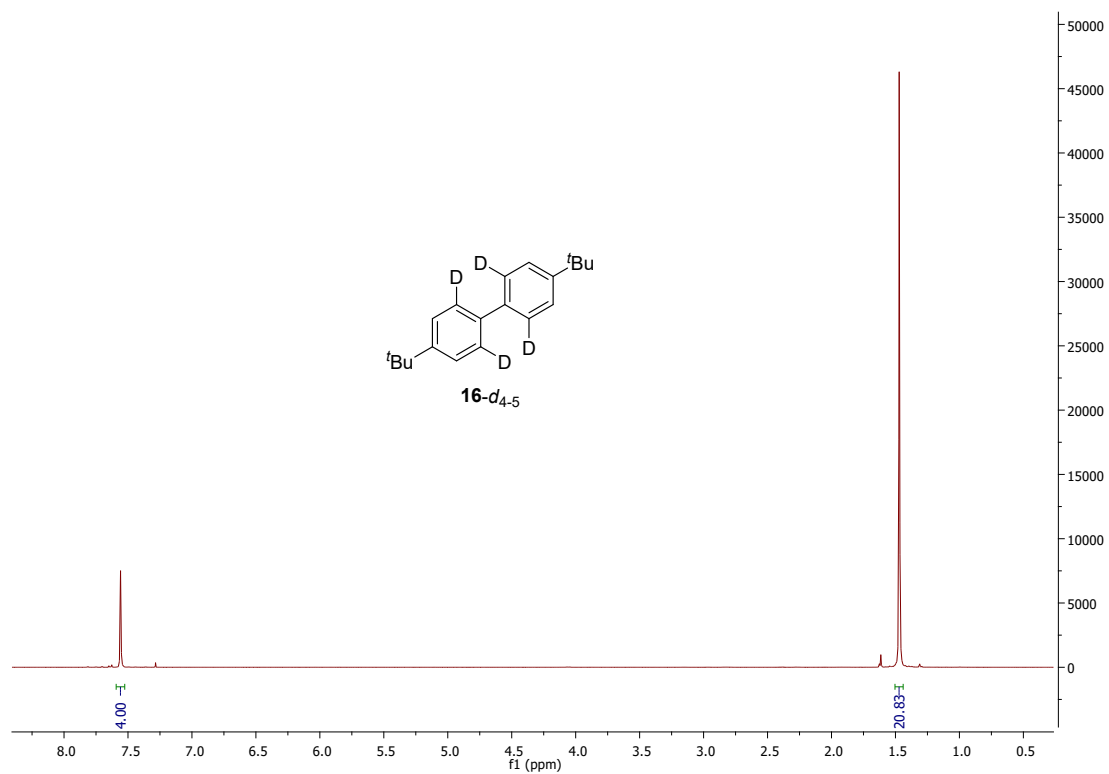
Biphenyl (**2**), 1,4-dihydro-1,1'-biphenyl (**3**), 3,4-dihydro-1,1'-biphenyl (**4**) and 2,5-dihydro-1,1'-biphenyl (**5**)



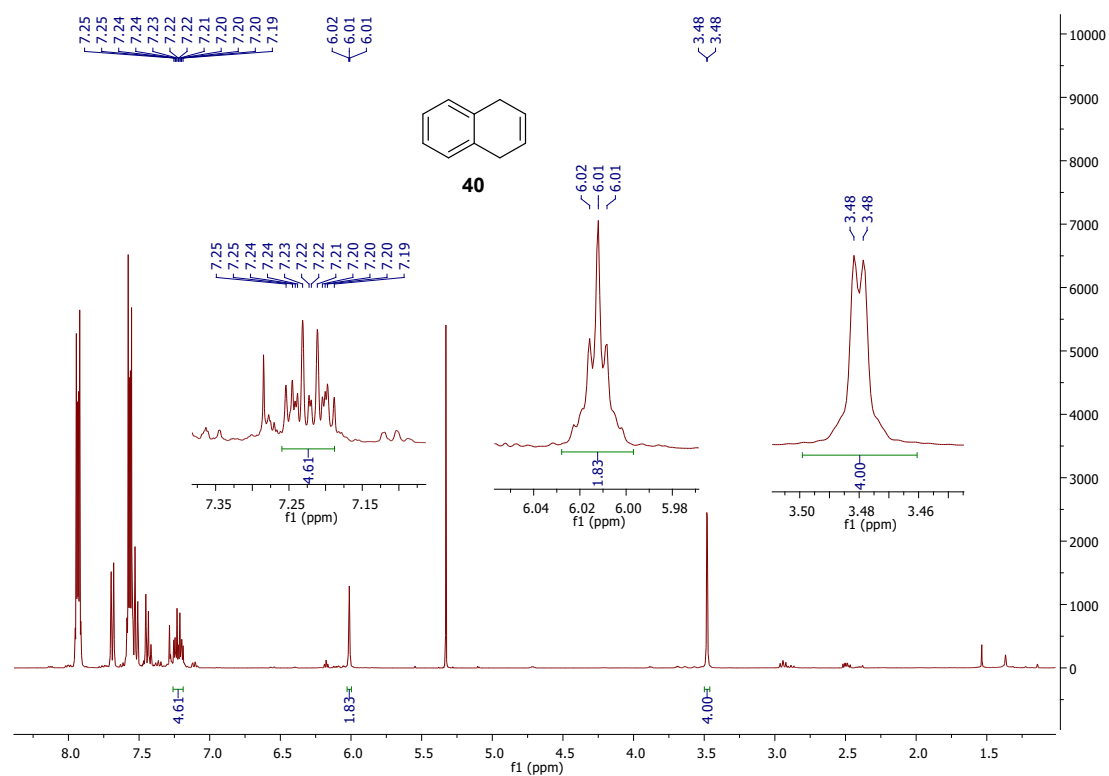
Biphenyl (**2**) – reaction with 4,4'-di-*tert*-butyl-1,1'-biphenyl (**21**)



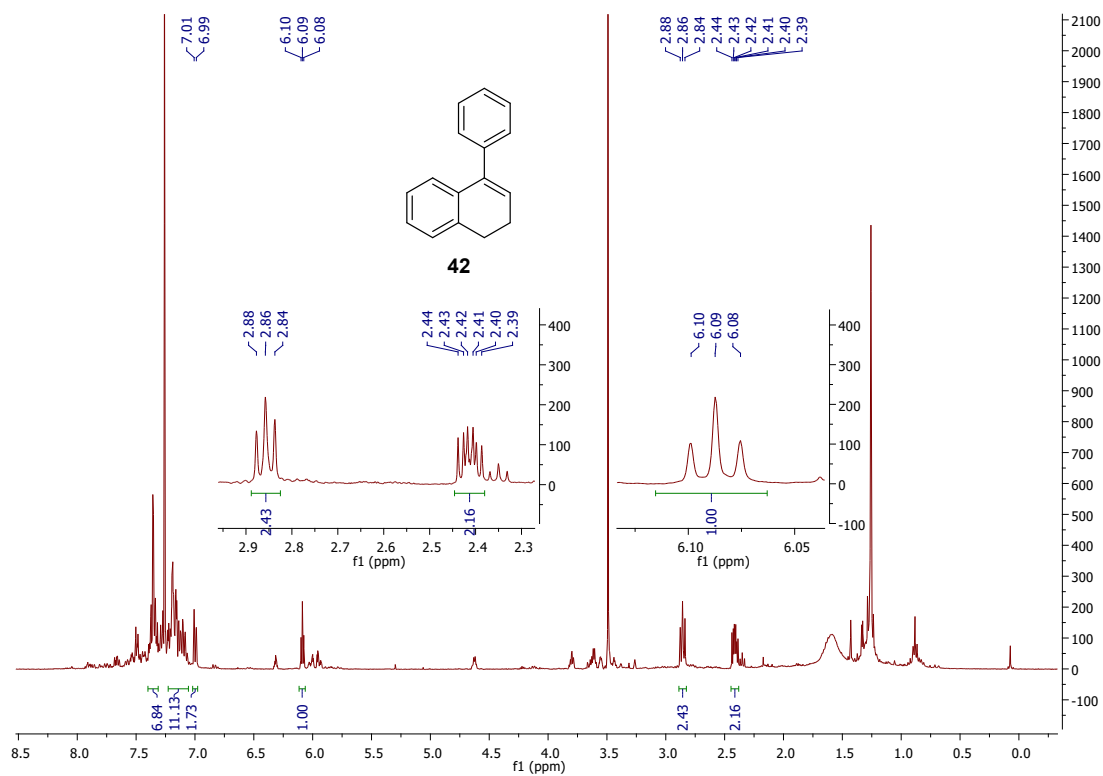
4,4'-Di-*tert*-butyl-1,1'-biphenyl-*d*₄ (**21-d₄** + *d*₅)



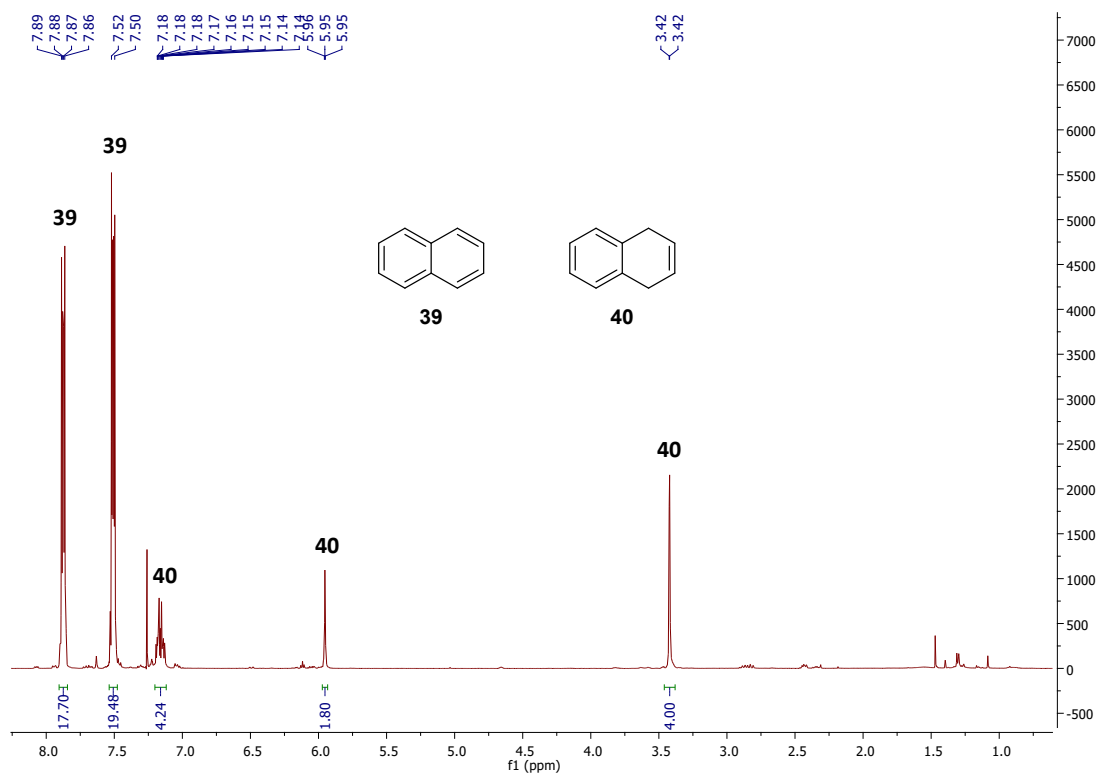
1,4-Dihydronaphthalene (**40**) – reaction with benzene



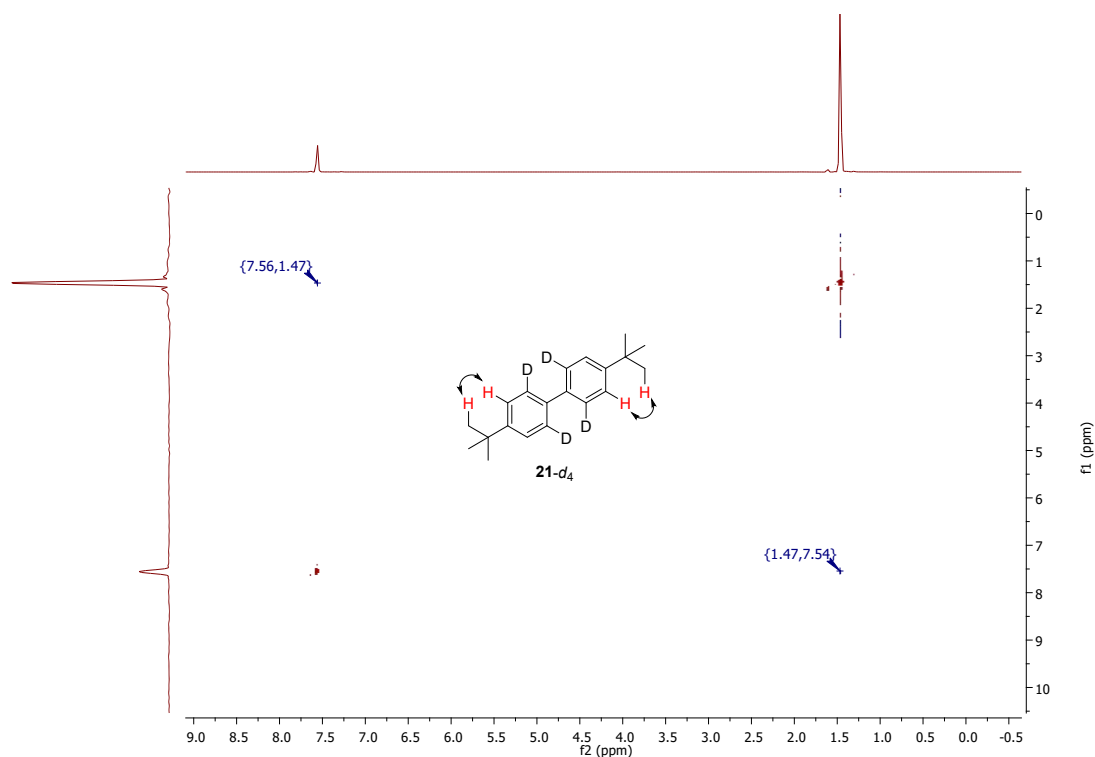
4-Phenyl-1,2-dihydronaphthalene (**42**)



Naphthalene (**39**) and 1,4-Dihydronaphthalene (**40**) – reaction with benzene-*d*₆



nOesy NMR of 4,4'-Di-*tert*-butyl-1,1'-biphenyl-*d*₄ + *d*₅ (**21-d**₄ + *d*₅)



Example Calculation of NMR Yields: (This example calculation relates to the amount of biphenyl **2** in the reaction using biphenyl in C₆D₆). The spectrum is shown at bottom of page S23

$$n_{IS} = \frac{m}{MW}$$

$$n_{IS} = \frac{7.1}{168.2}$$

$$n_{IS} = 0.042 \text{ mmol}$$

$$\frac{n_p}{n_{IS}} = \frac{(\text{integral of product peak} \div \text{no. of protons})}{(\text{integral of IS peak} \div \text{no. of protons})}$$

$$\frac{n_p}{n_{IS}} = \frac{(24.06 \div 4)}{(9.00 \div 9)}$$

$$\frac{n_p}{n_{IS}} = 6.015$$

$$\therefore n_p = 6.015 \times n_{IS}$$

$$n_p = 6.015 \times 0.042$$

$$n_p = 0.289 \text{ mmol}$$

$$\text{NMR Yield} = n_p \times MW$$

$$\text{NMR Yield} = 0.289 \times 154.1$$

$$\text{NMR Yield} = 44.5 \text{ mg}$$

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