

Synthesis, structure and pyrolysis of stabilised phosphonium ylides containing saturated oxygen heterocycles

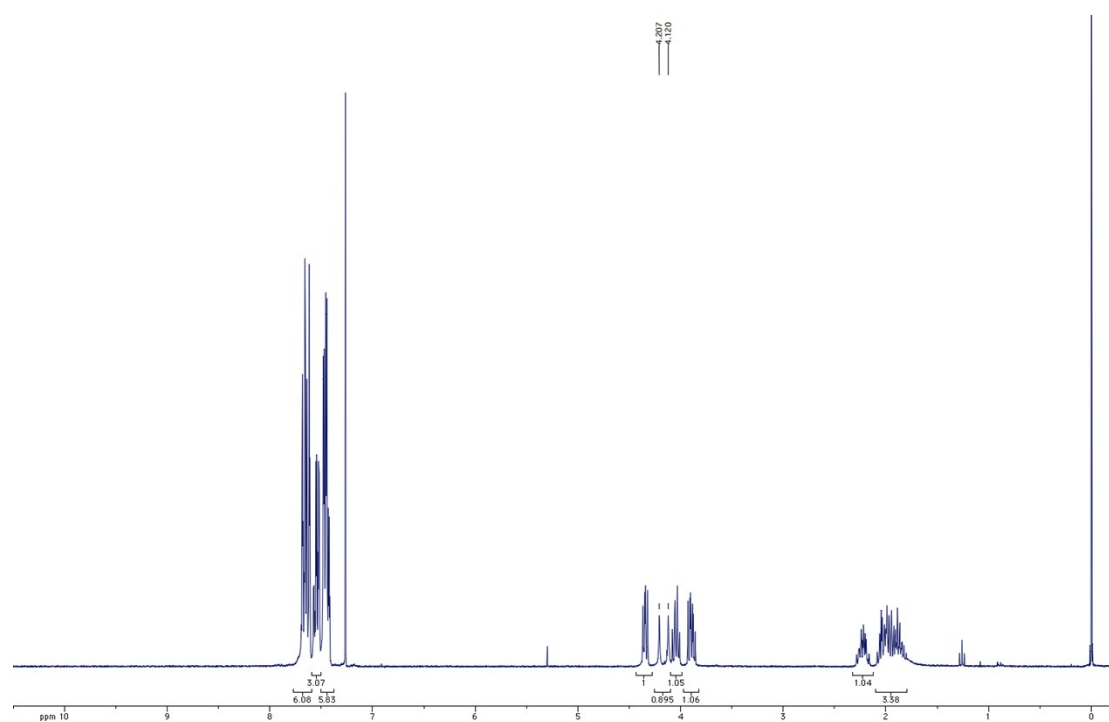
R. Alan Aitken,^{a*} Nazira Karodia,^{b,c*} Hollie B. McCarron,^a Cécile Rouxel,^a Nina Sahabo^b
and Alexandra M. Z. Slawin^a

Supplementary Information

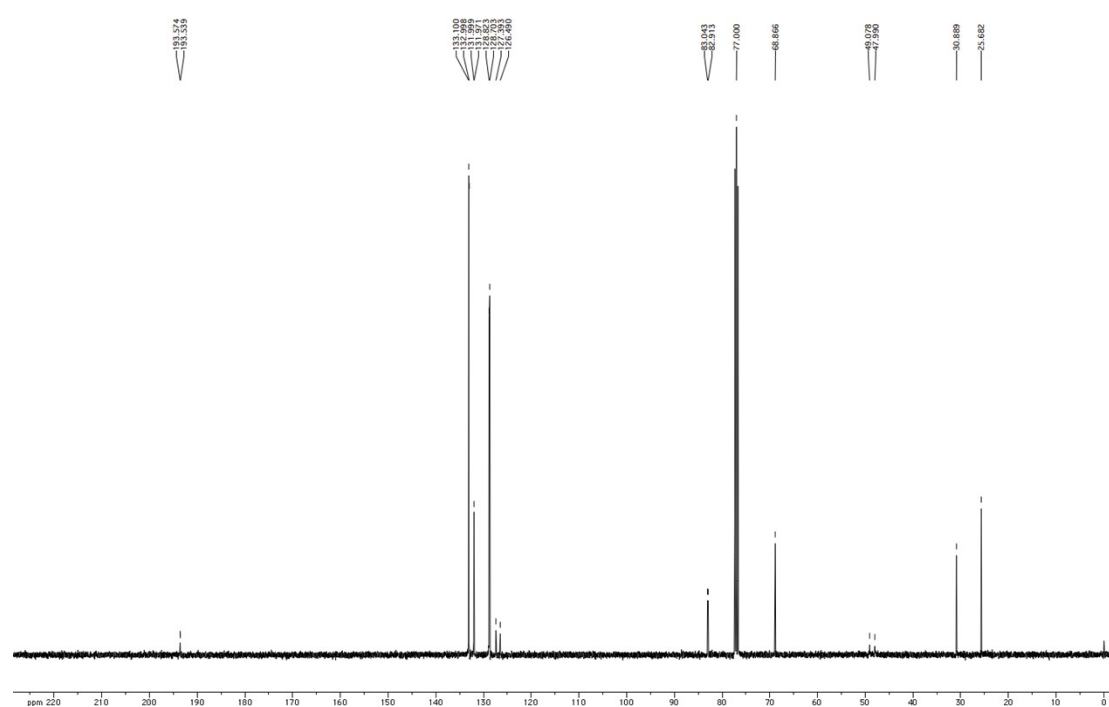
¹H and ¹³C NMR Spectra

| | Page |
|---|--------|
| Ylide 6 | 2 |
| Ylide 7 | 3 |
| Ylide 8 | 4 |
| Ylide 9 | 5 |
| Ylide 10 | 6 |
| Ylide 11 | 7 |
| Ylide 12 | 8 |
| Ylide 13 | 9 |
| Ylide 14 | 10 |
| Ylide 15 | 11 |
| Ylide 16 | 12 |
| Ylide 17 | 13 |
| Pyrolysis of 6 giving 18 | 14 |
| Pyrolysis of 12 giving 19 | 15 |
| Pyrolysis of 7 giving 22 and 24 | 16 |
| Pyrolysis of 13 giving 25 | 17 |
| Pyrolysis of 8 giving 26 | 18 |
| Pyrolysis of 14 giving 27 and 28 | 19 |
| Pyrolysis of 16 giving 29 and 30 | 20 |
| Pyrolysis of 9 giving 37 , 39 and 41 | 21, 22 |
| Pyrolysis of 10 giving 38 , 40 and 42 | 23, 24 |
| Pyrolysis of 11 giving 45 and 18 | 25 |
| Pyrolysis of 15 giving 46 | 26 |
| Pyrolysis of 17 giving 47 and 48 | 27, 28 |

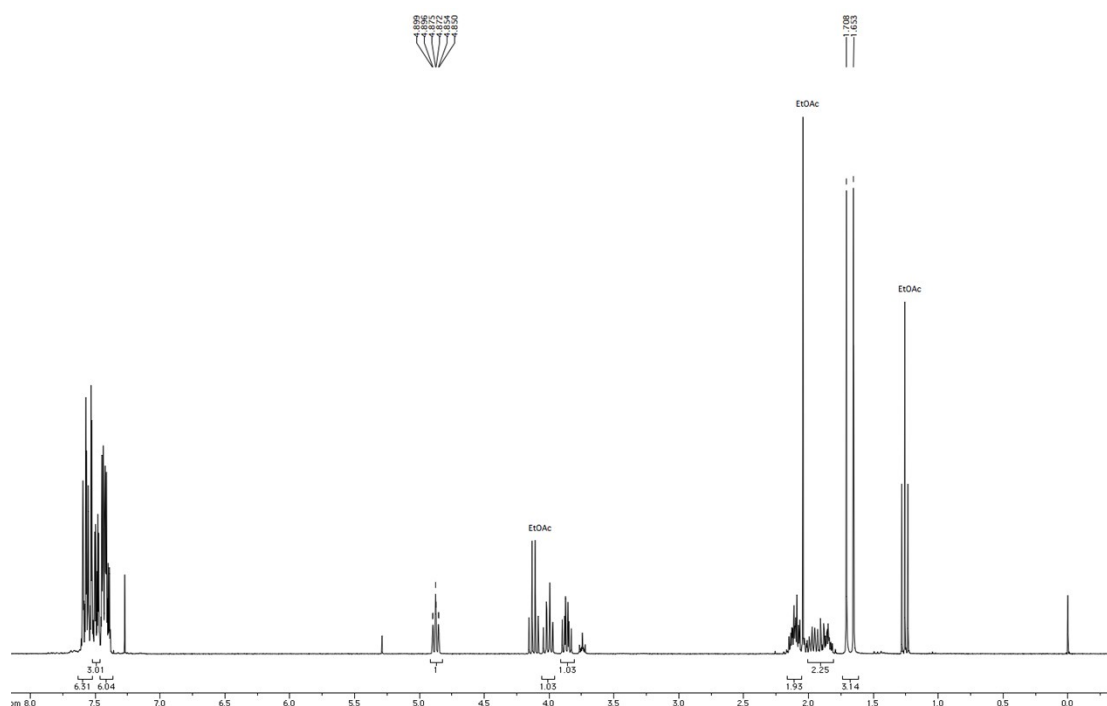
^1H NMR spectrum of ylide **6**



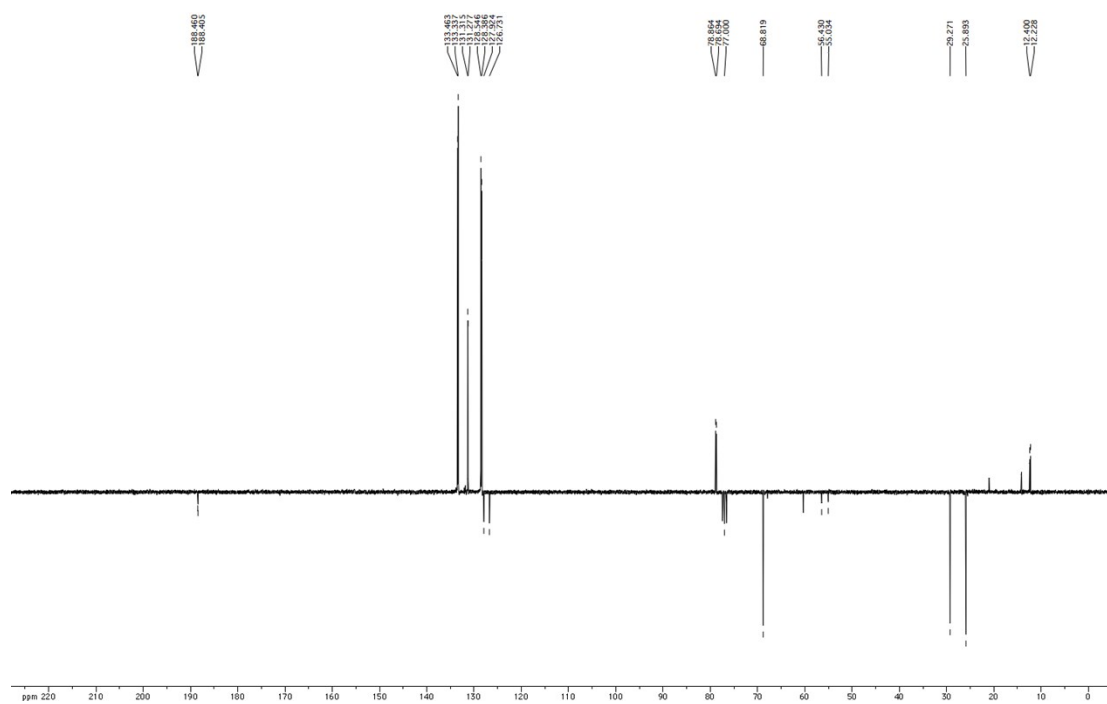
^{13}C NMR spectrum of ylide **6**



^1H NMR spectrum of ylide **7**

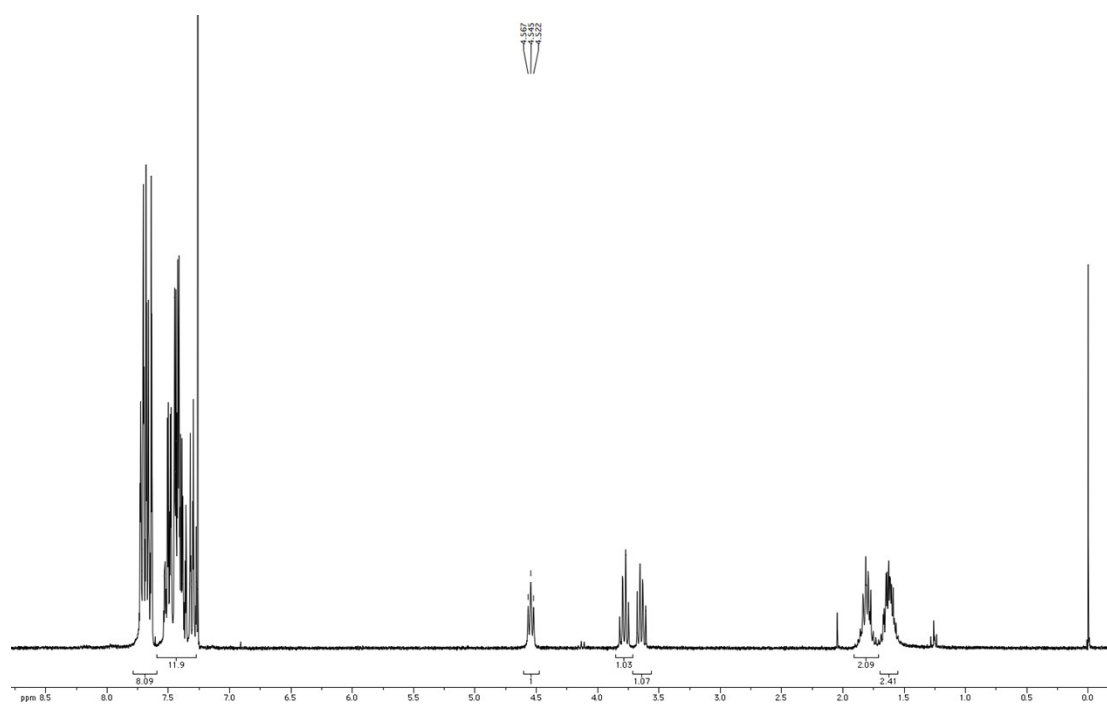


^{13}C NMR (DEPTQ) spectrum of ylide **7**

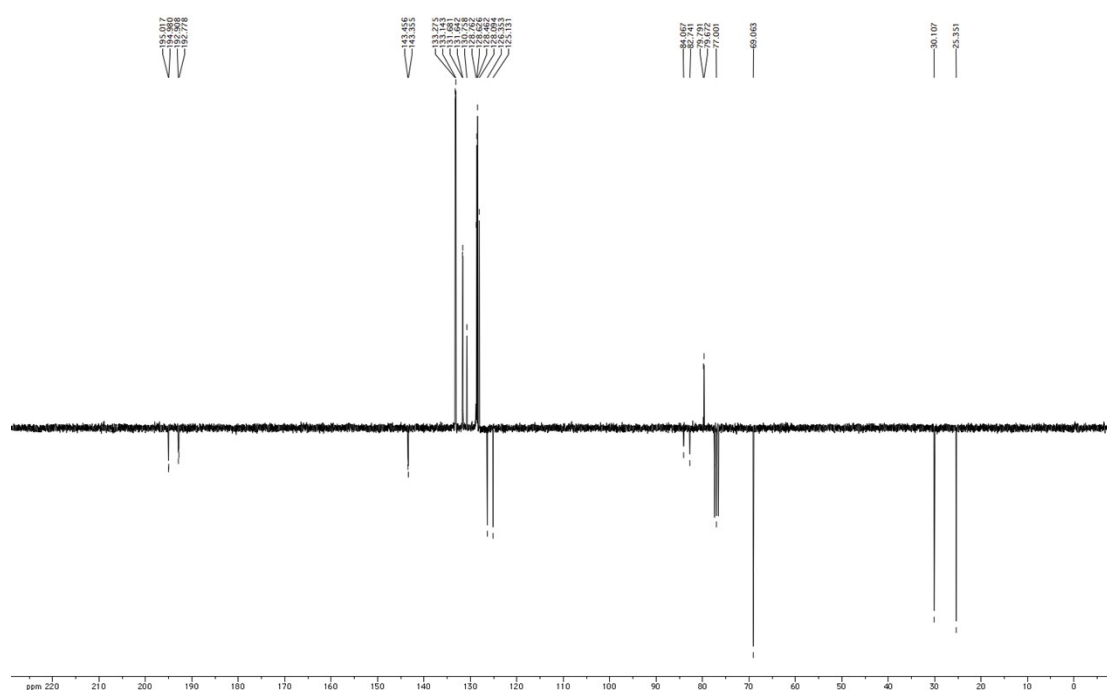


13C NMR spectrum of 2,4-dichlorobenzonitrile. The spectrum shows peaks at 195.243, 192.710, 192.610, 133.034, 128.692, 128.590, 125.737, 86.530, 80.971, 80.856, 77.001, 68.801, 30.169, 30.147, and 25.312 ppm. The x-axis ranges from 0 to 220 ppm.

^1H NMR spectrum of ylide **10**



^{13}C NMR (DEPTQ) spectrum of ylide **10**

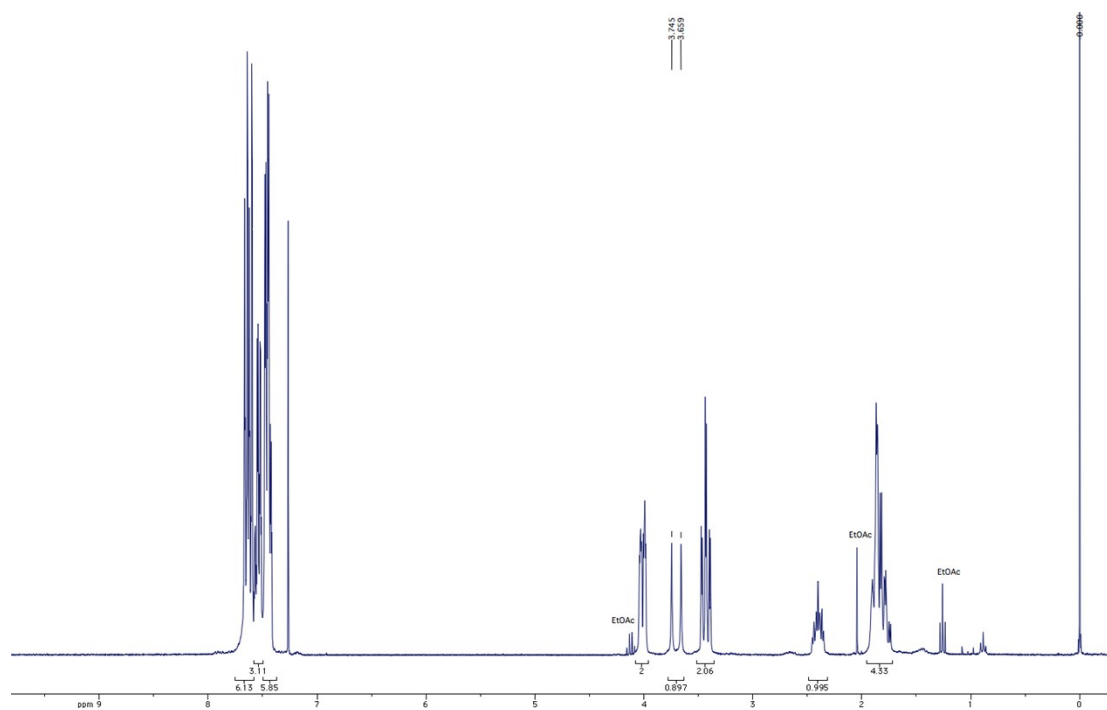


¹H NMR spectrum (CDCl₃) of compound 6j. The spectrum displays several characteristic peaks:

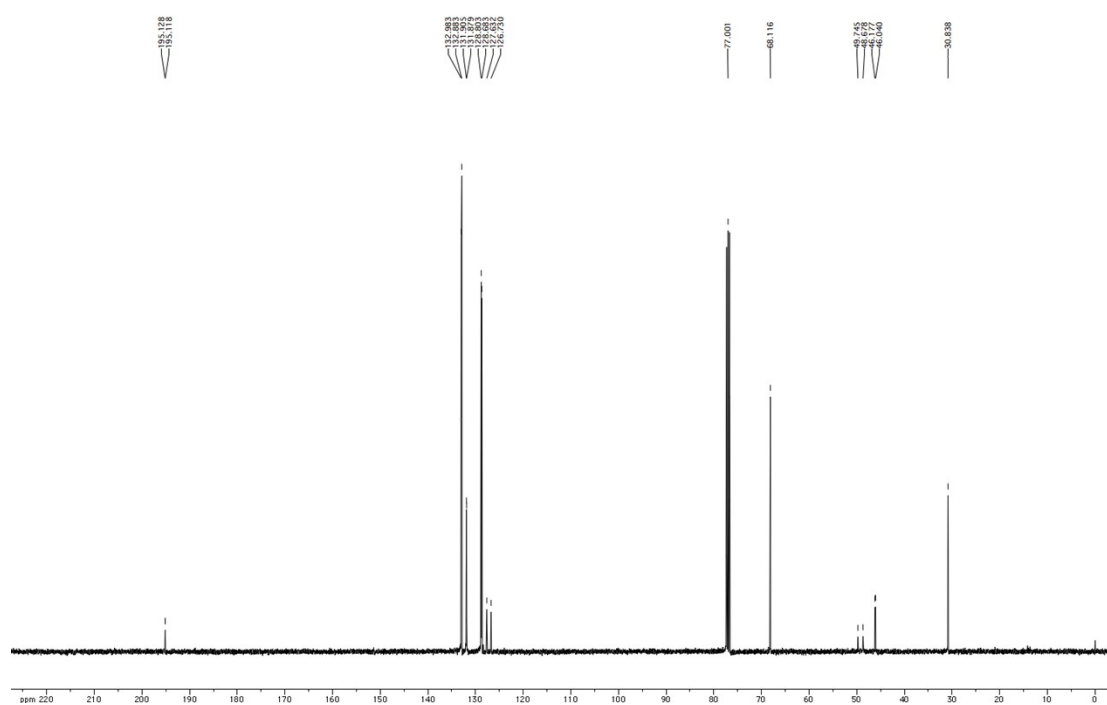
- Aromatic region (7.0–7.8 ppm): Multiple sharp peaks corresponding to the aromatic protons.
- Singlet (~5.5 ppm): A distinct peak, likely representing a specific proton environment.
- Multiplet (~3.8 ppm): Labeled "EtOAc" with an integration value of 2.03.
- Multiplet (~2.4 ppm): Labeled "EtOAc" with an integration value of 1.03.
- Complex region (1.5–2.2 ppm): Contains multiple overlapping peaks with integration values of 1.14 and 2.08.
- Small peak (~1.3 ppm): Labeled "EtOAc".
- Solvent peak (~7.2 ppm): A prominent triplet for CDCl₃.

The x-axis represents the chemical shift in ppm, ranging from 0.0 to 8.5.

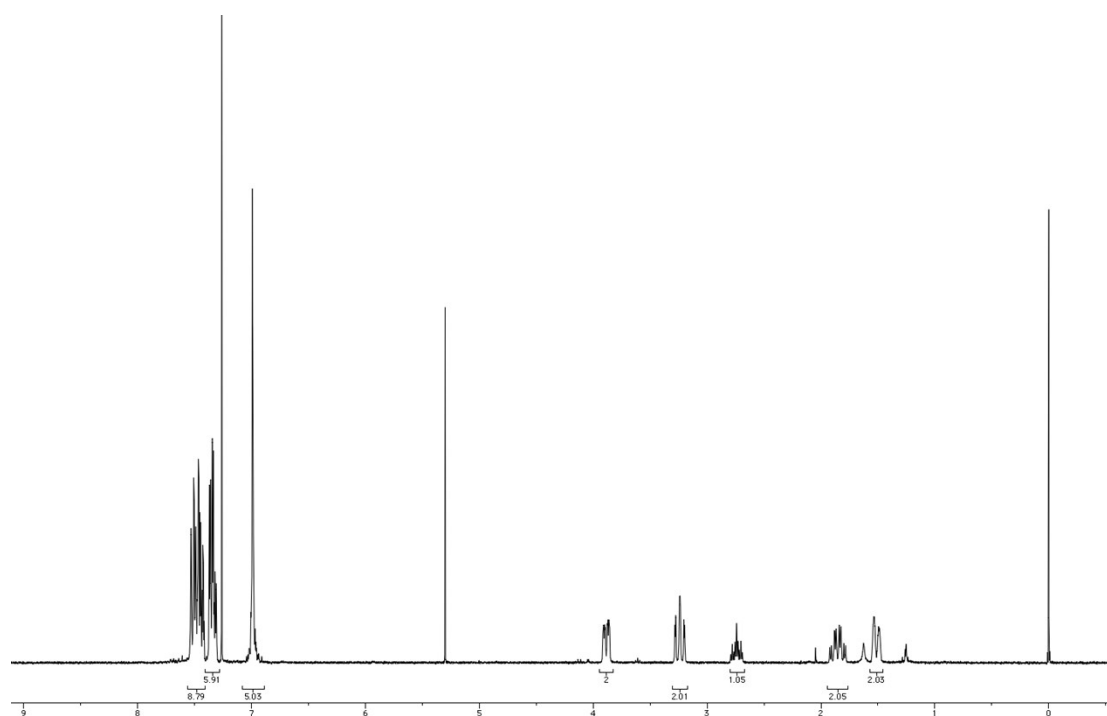
^1H NMR spectrum of ylide **12**



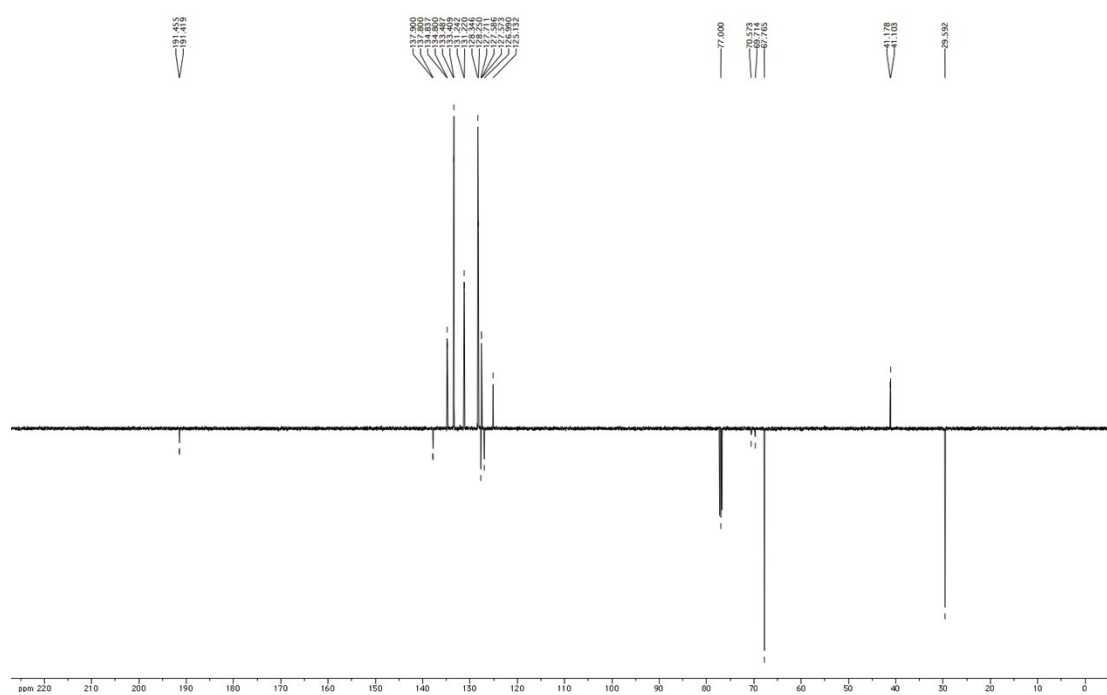
^{13}C NMR spectrum of ylide **12**



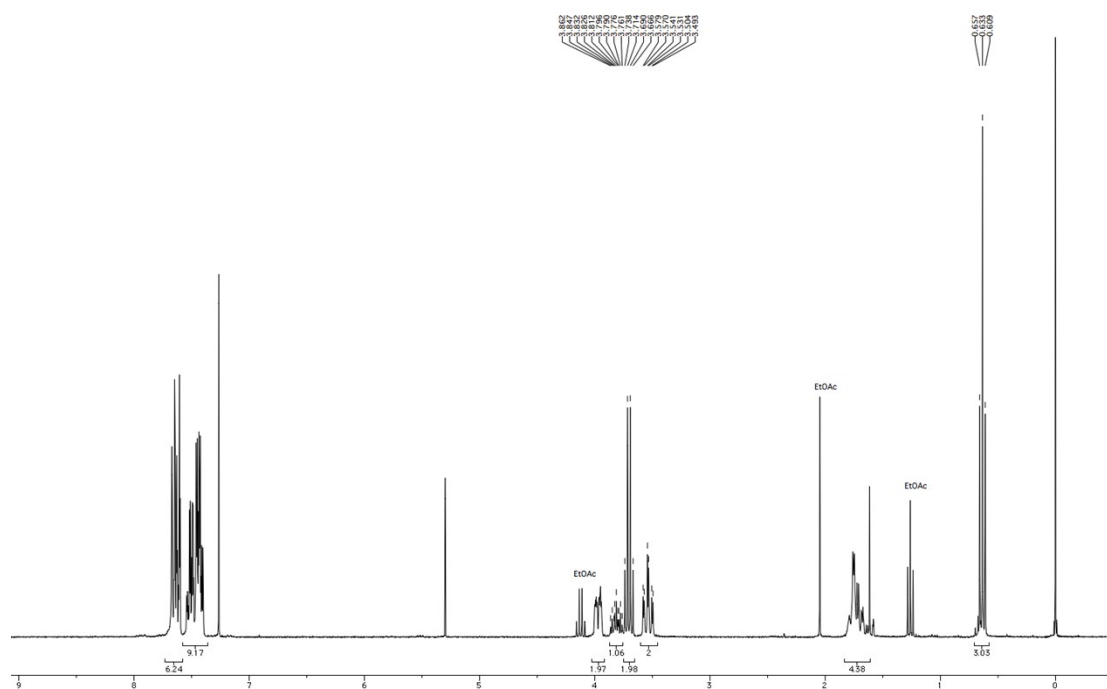
^1H NMR spectrum of ylide **14**



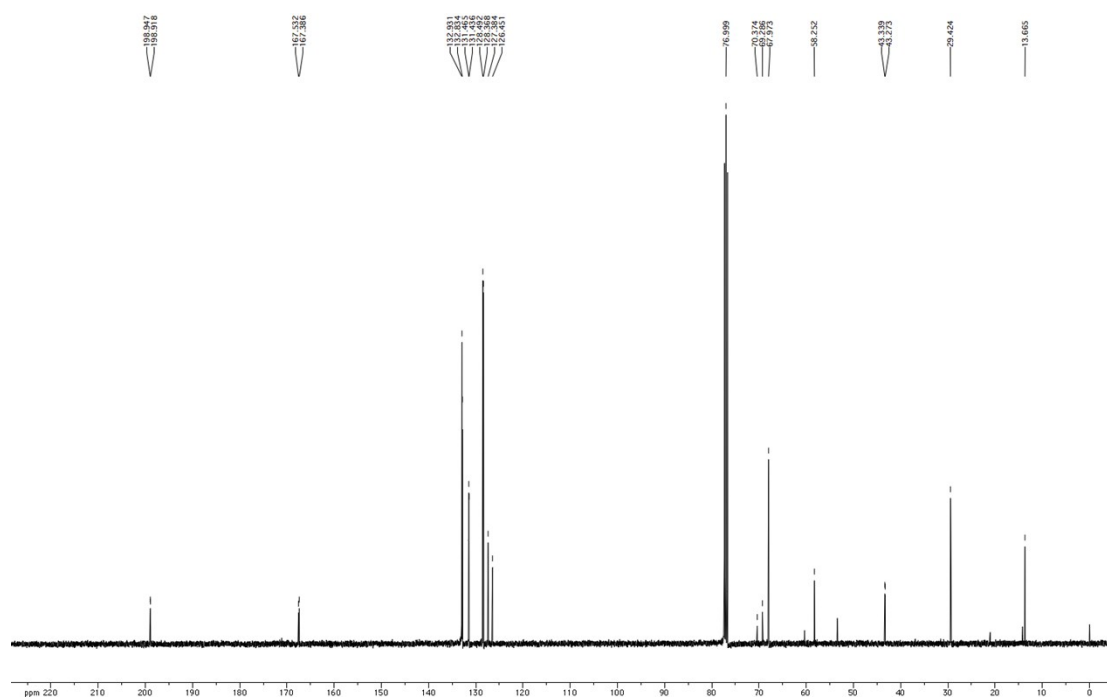
^{13}C NMR (DEPTQ) spectrum of ylide **14**



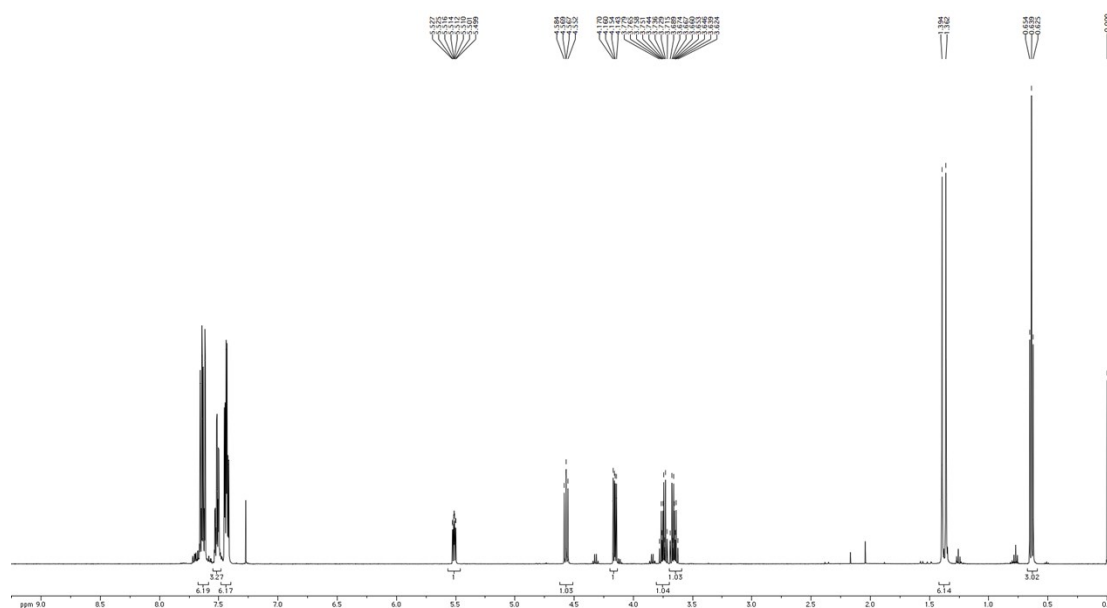
^1H NMR spectrum of ylide **15**



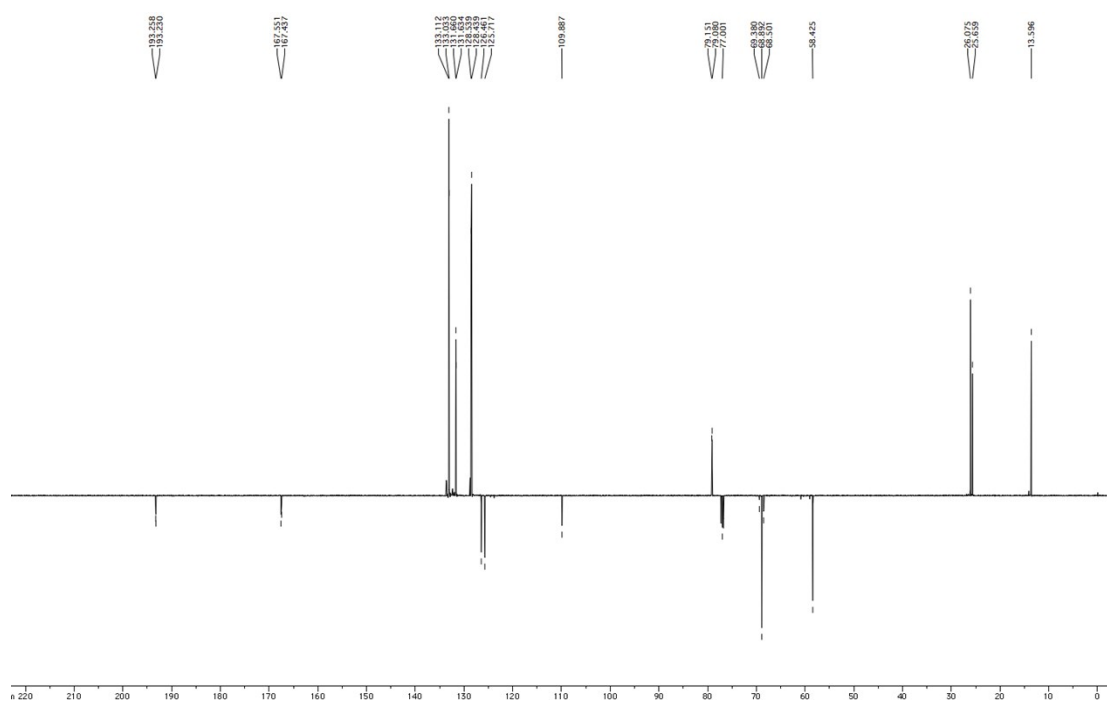
^{13}C NMR spectrum of ylide **15**



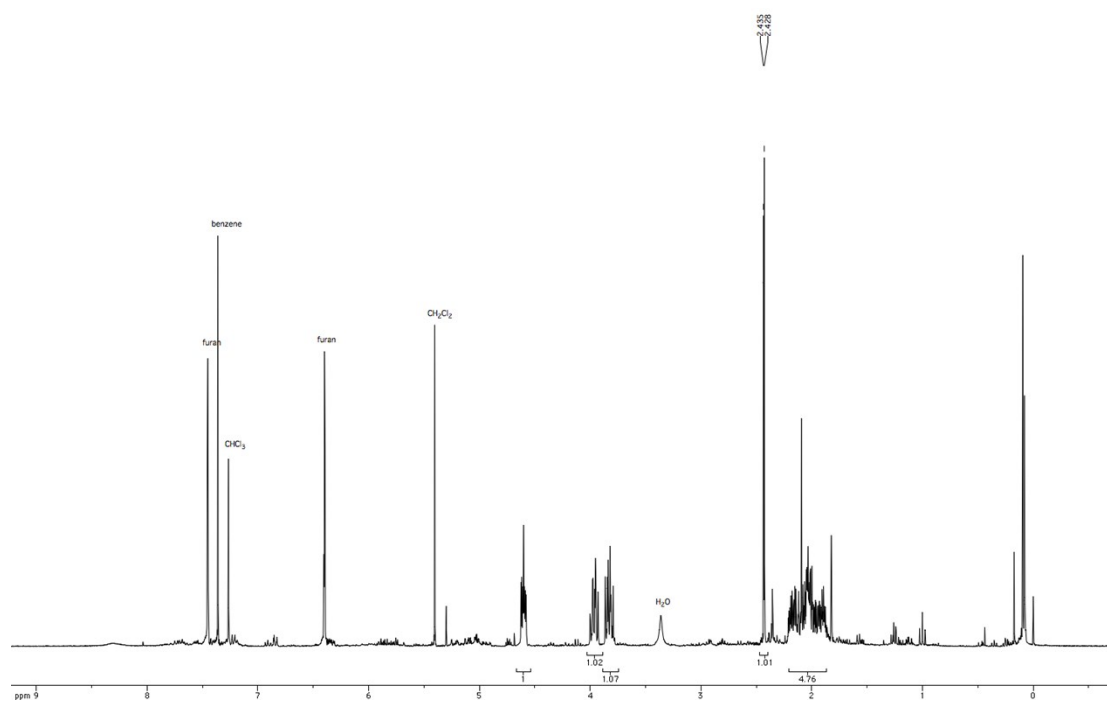
^1H NMR spectrum of ylide **17**



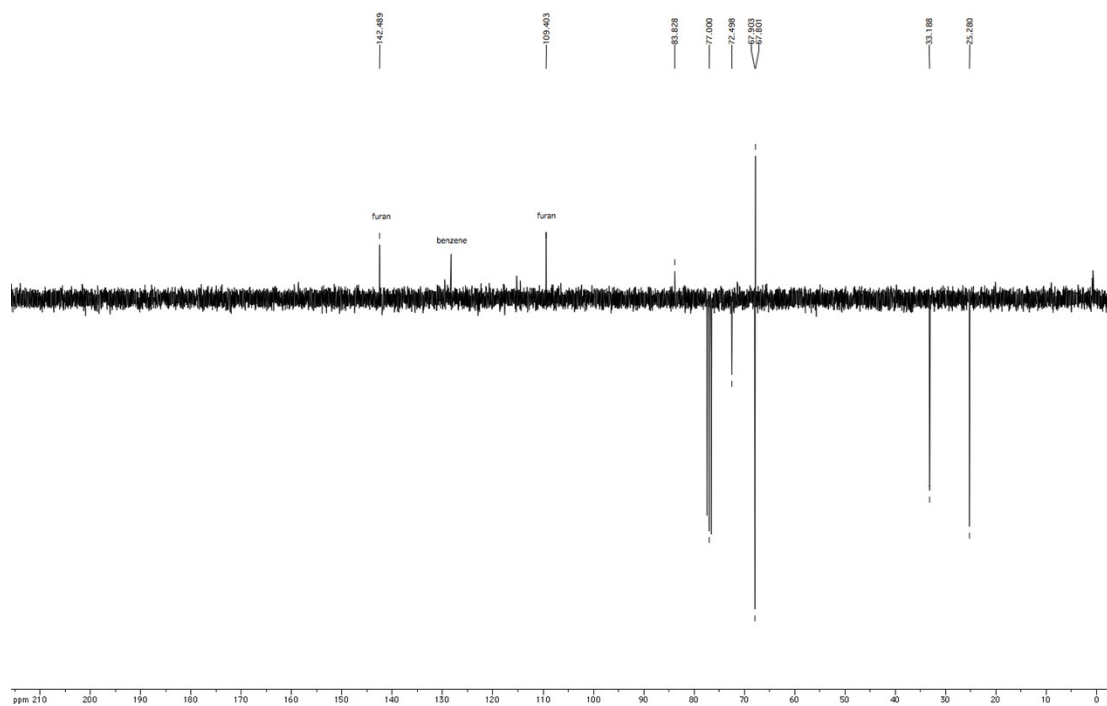
^{13}C NMR (DEPTQ) spectrum of ylide **17**



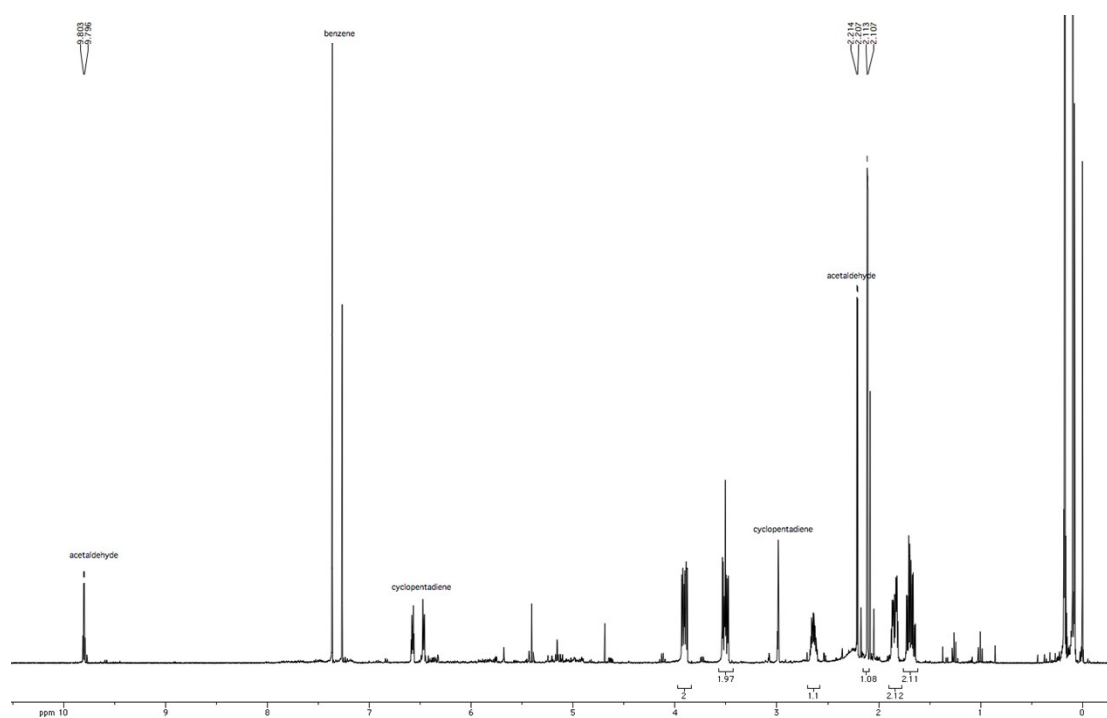
^1H NMR spectrum of FVP product from **6** containing alkyne **18**



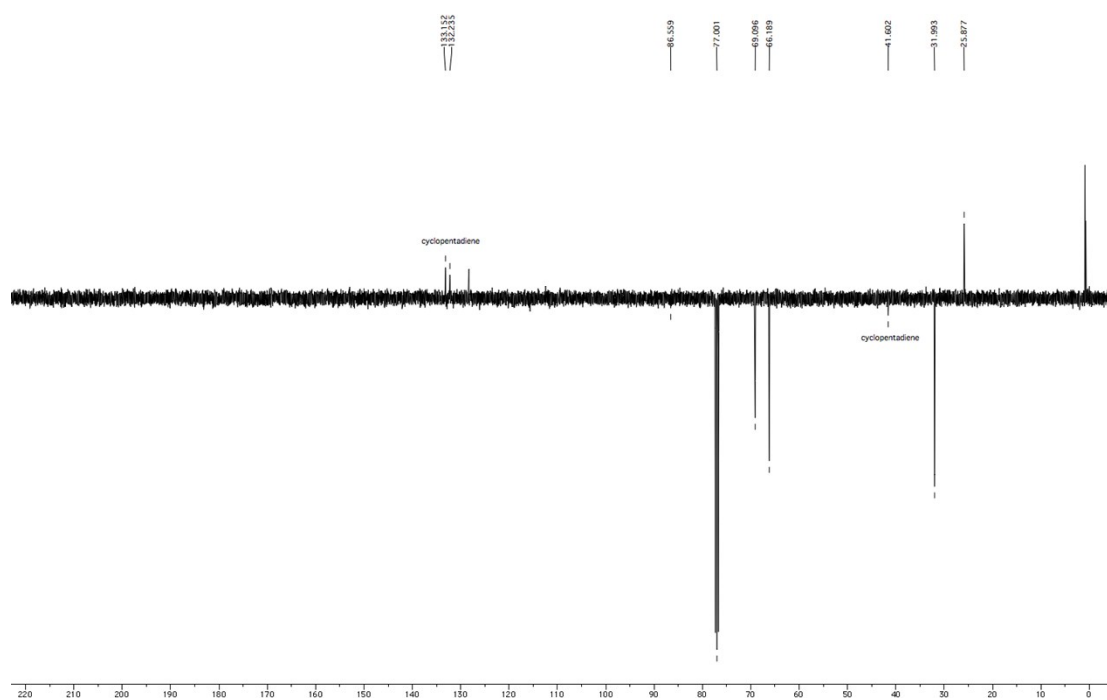
^{13}C NMR (DEPTQ) spectrum of FVP product from **6** containing alkyne **18**



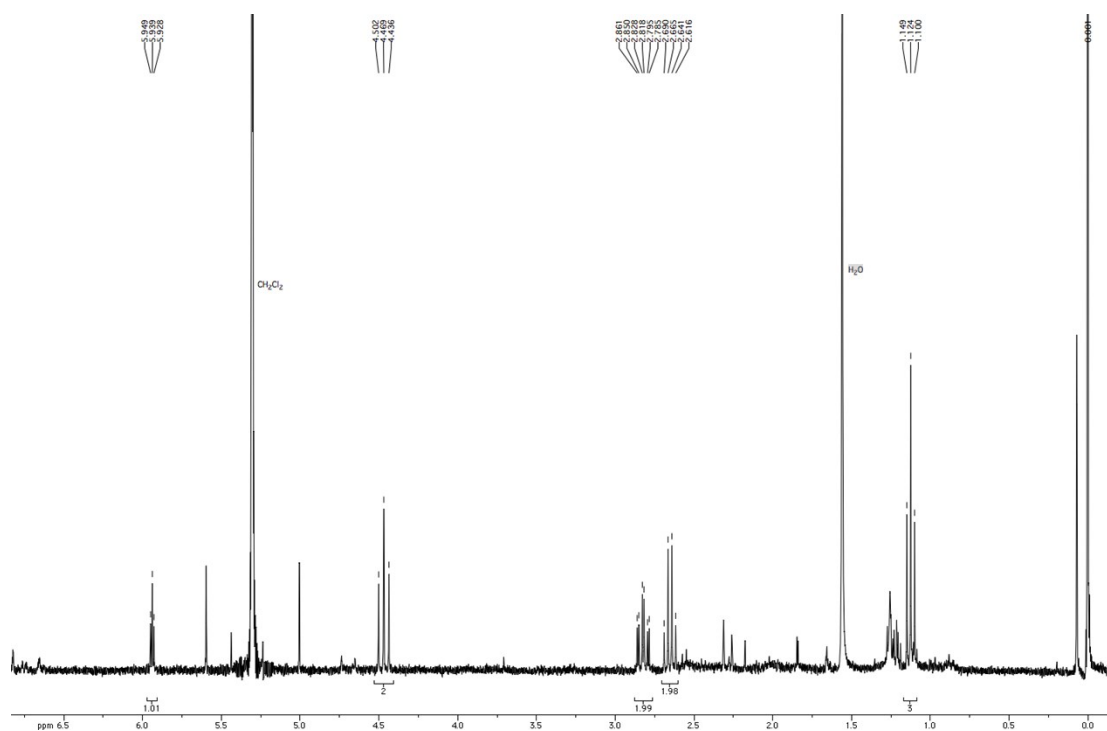
^1H NMR spectrum of FVP product from **12** containing alkyne **19**



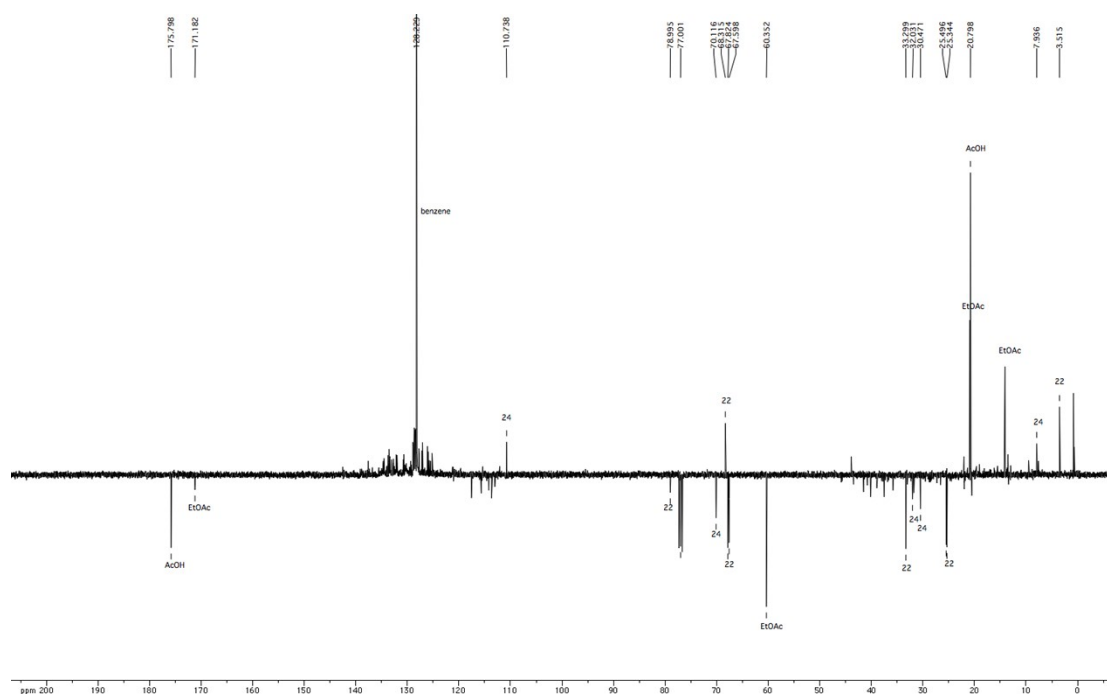
^{13}C NMR (DEPTQ) spectrum of FVP product from **12** containing alkyne **19**



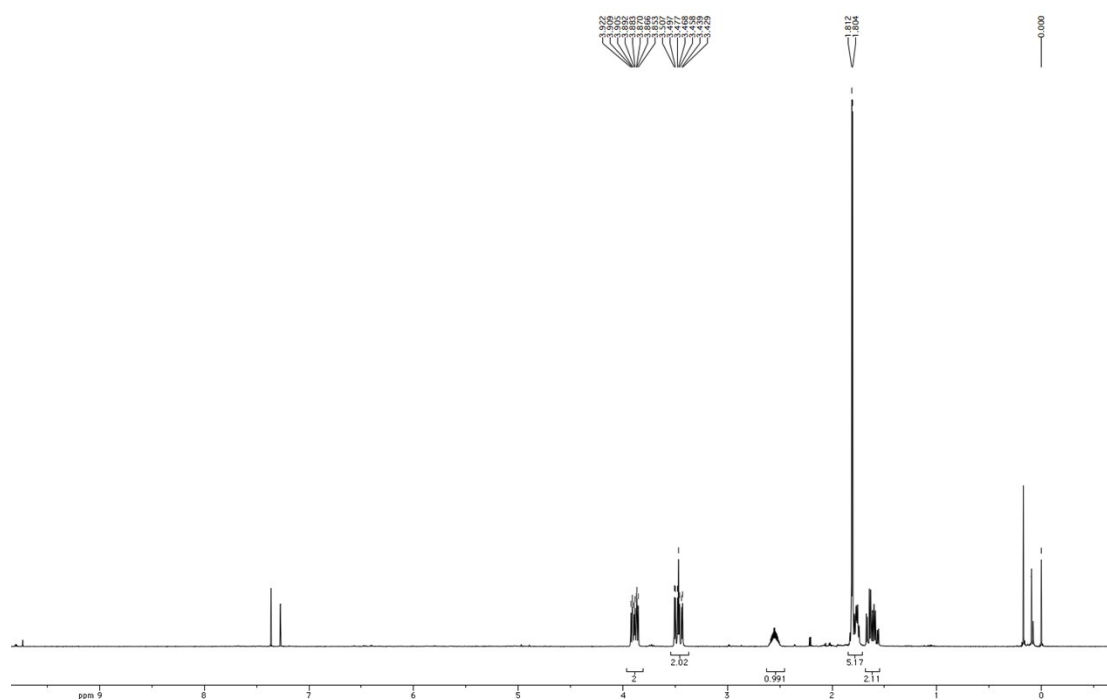
^1H NMR spectrum of FVP product from **7** containing compound **24** (after prep. TLC)



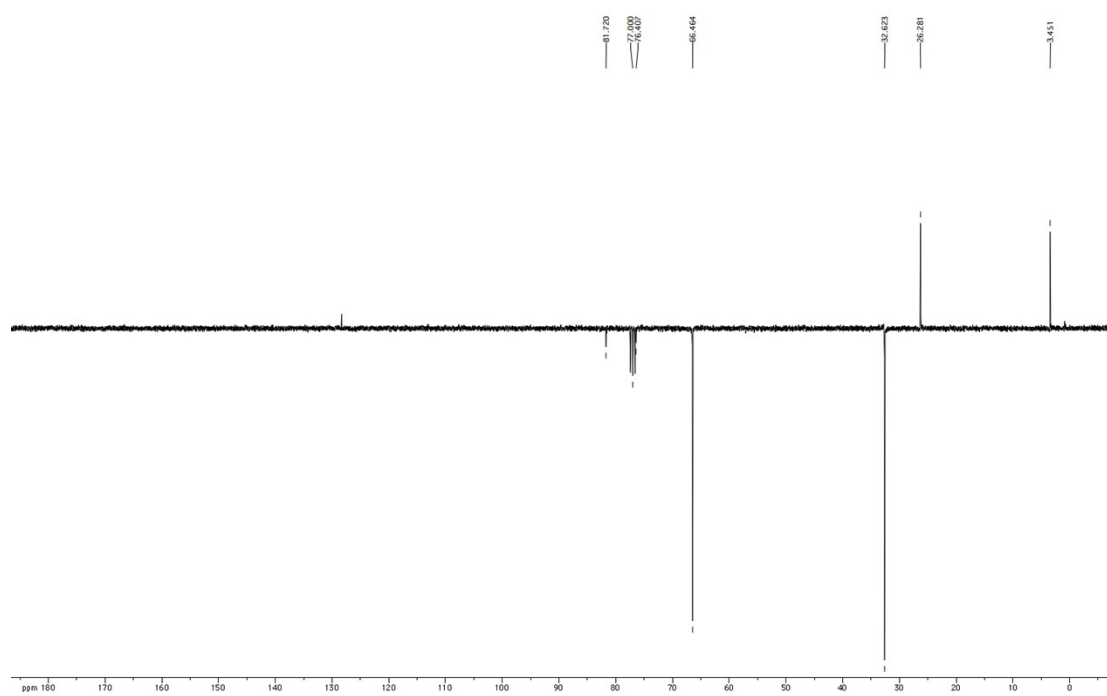
^{13}C NMR (DEPTQ) spectrum of FVP product from **7** containing compounds **22** and **24**



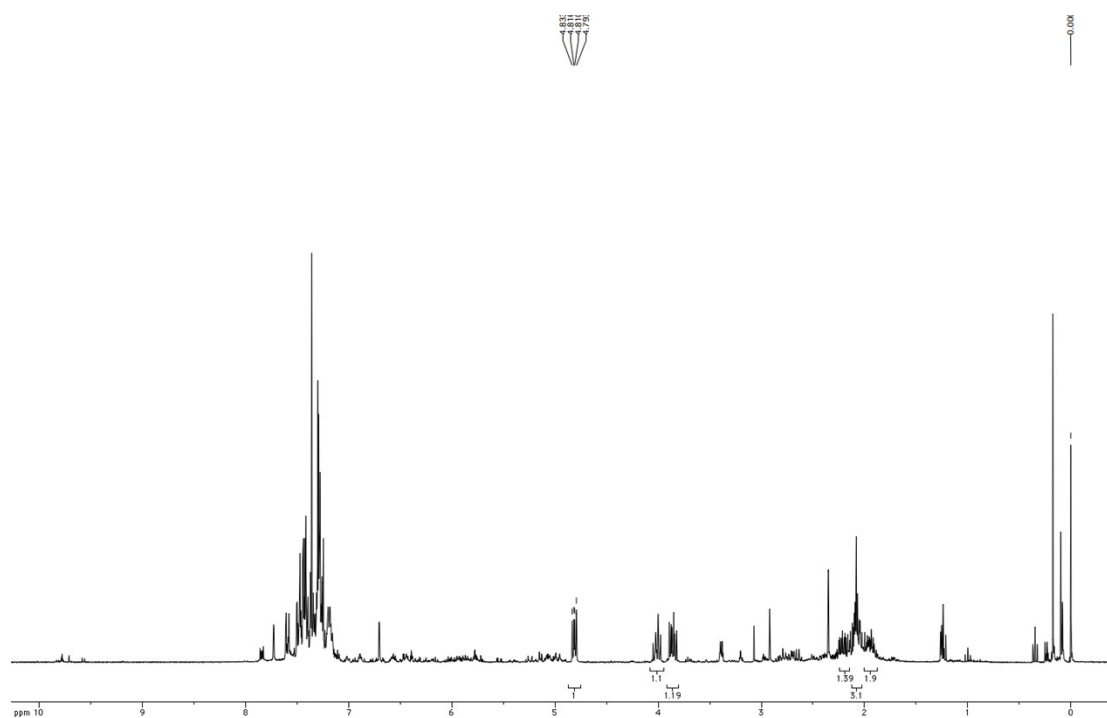
^1H NMR spectrum of FVP product from **13** containing alkyne **25**



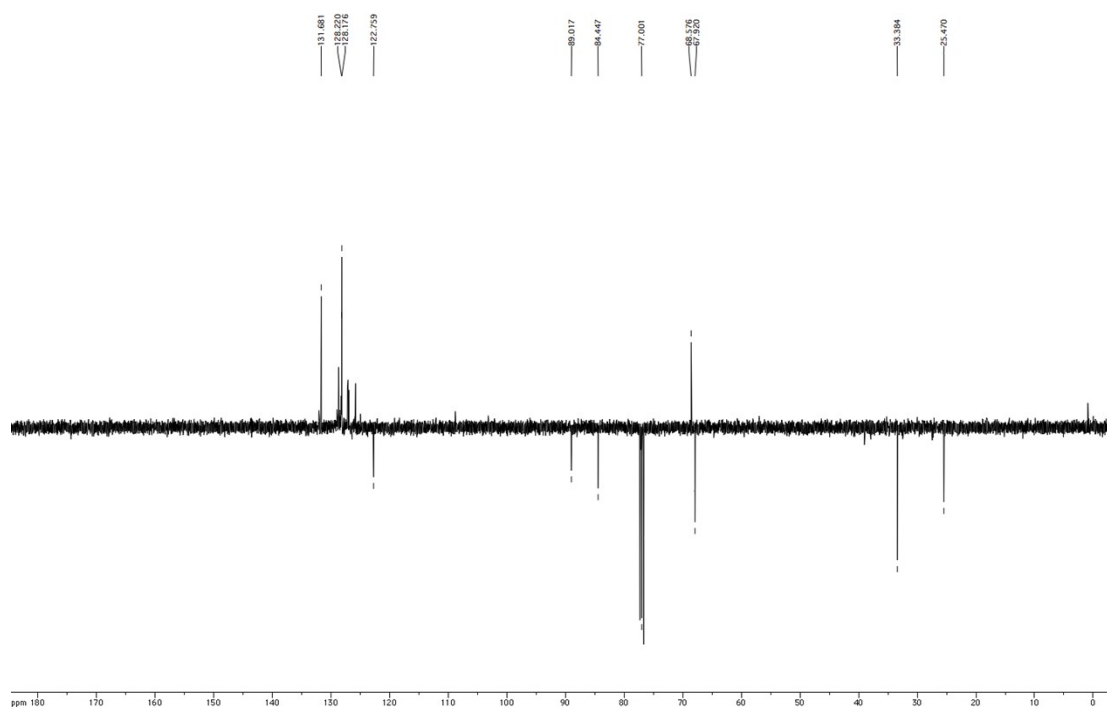
^{13}C NMR (DEPTQ) spectrum of FVP product from **13** containing alkyne **25**



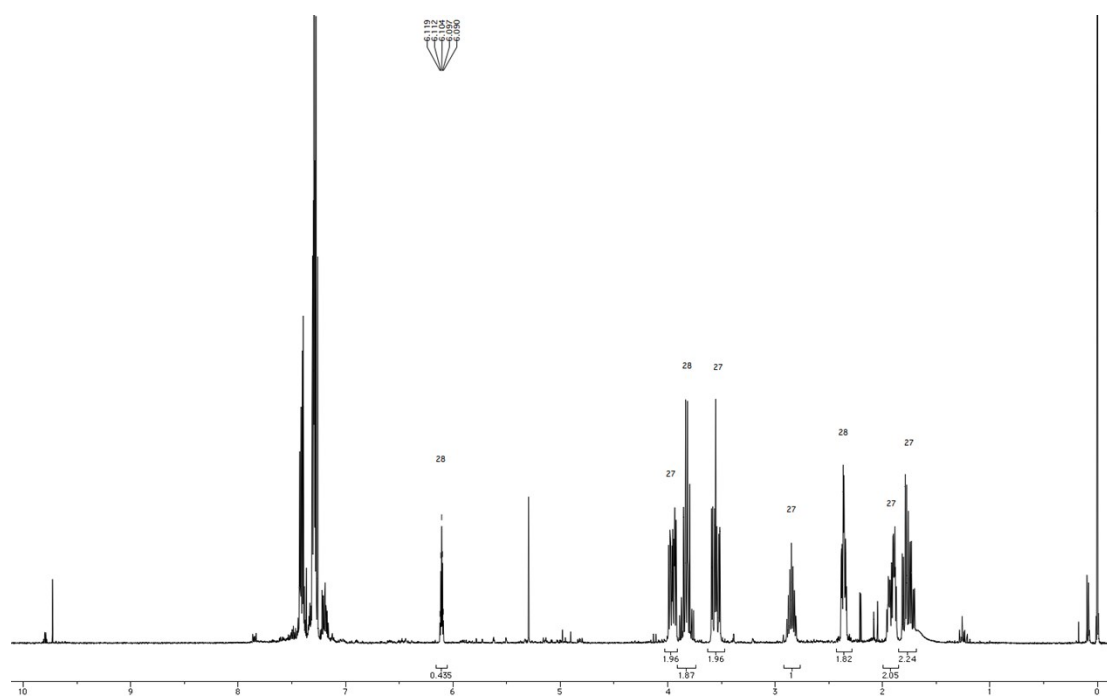
^1H NMR spectrum of FVP product from **8** containing alkyne **26**



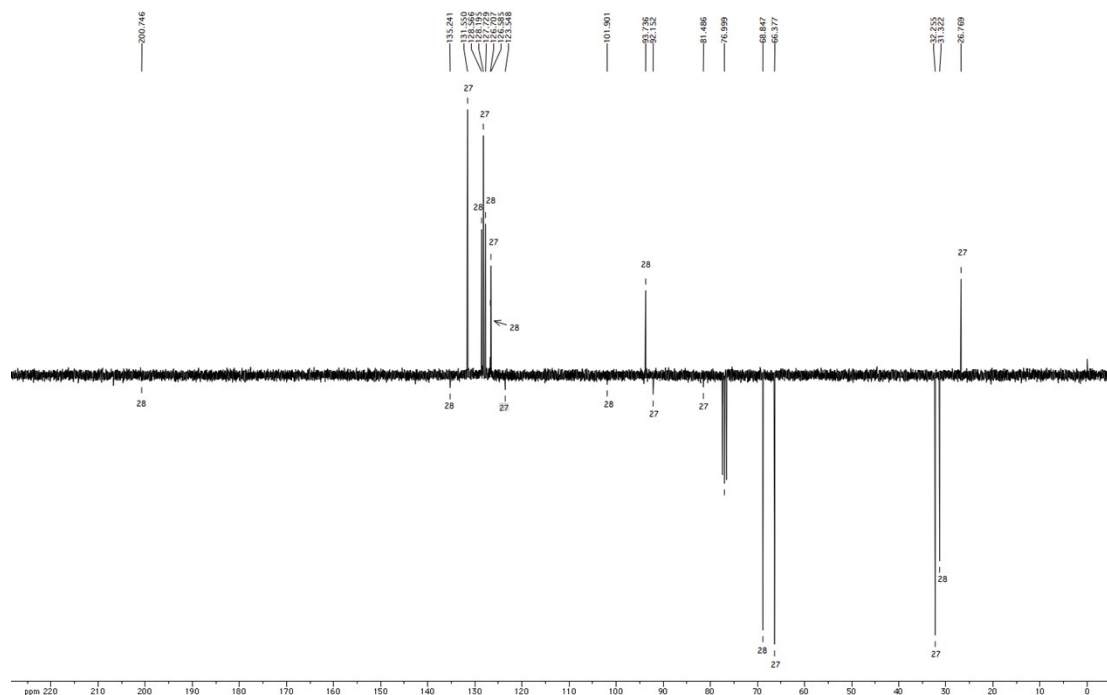
^{13}C NMR (DEPTQ) spectrum of FVP product from **8** containing alkyne **26**



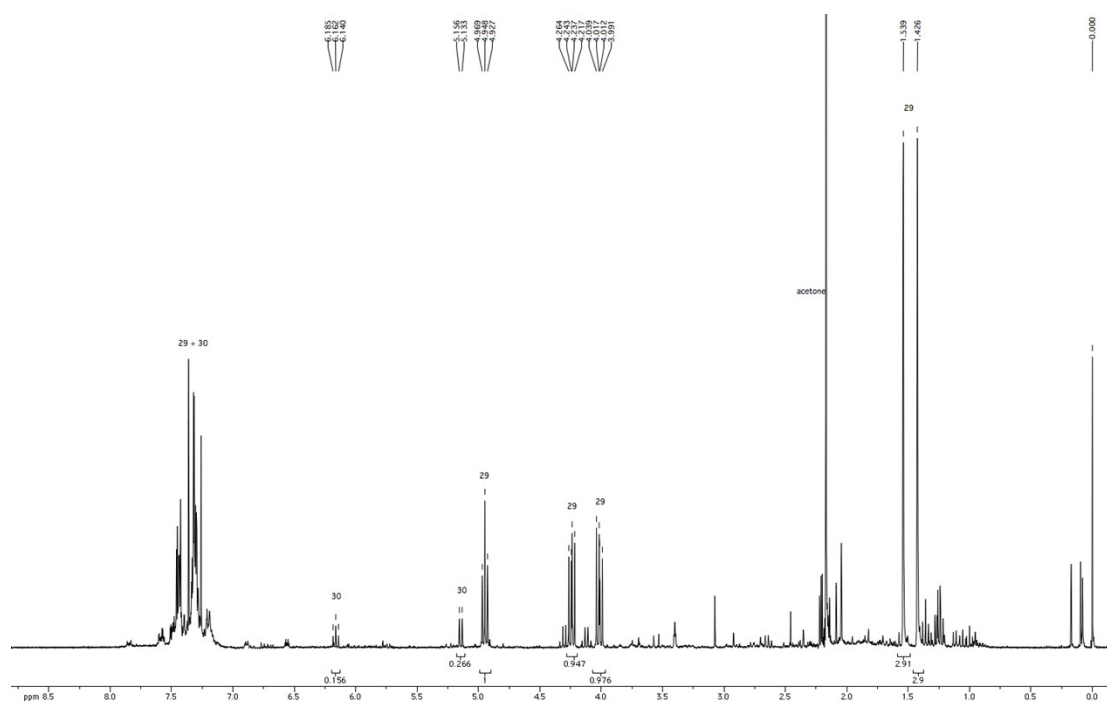
^1H NMR spectrum of FVP product from **14** containing alkyne **27** and allene **28**



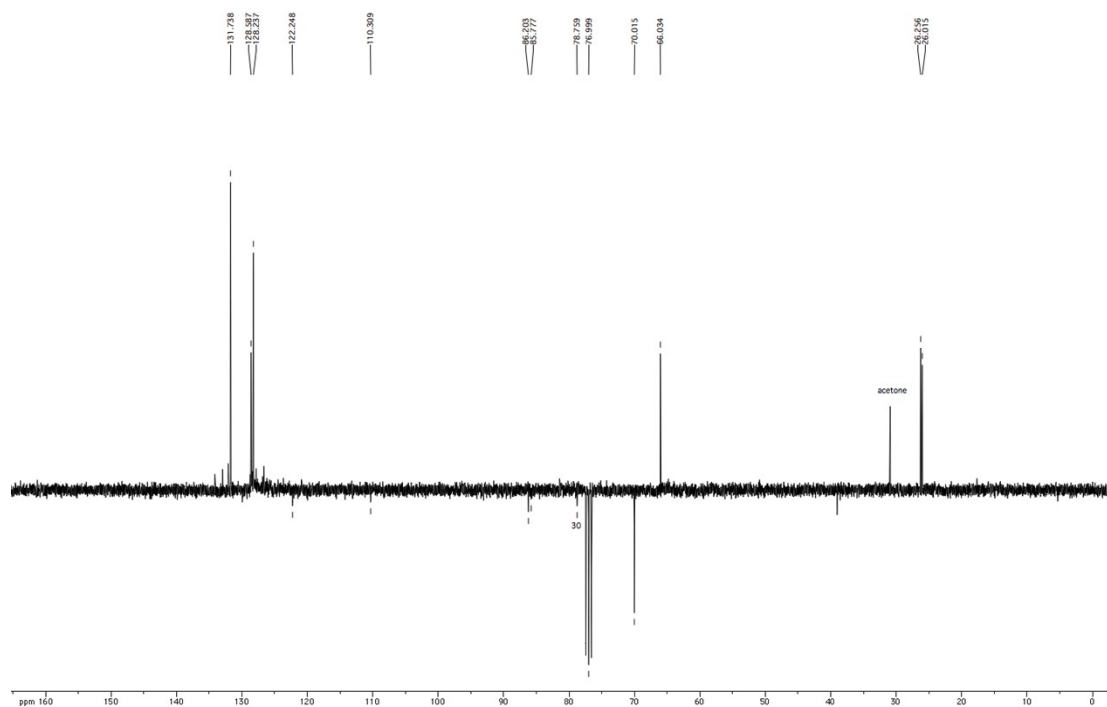
^{13}C NMR (DEPTQ) spectrum of FVP product from **14** containing alkyne **27** and allene **28**



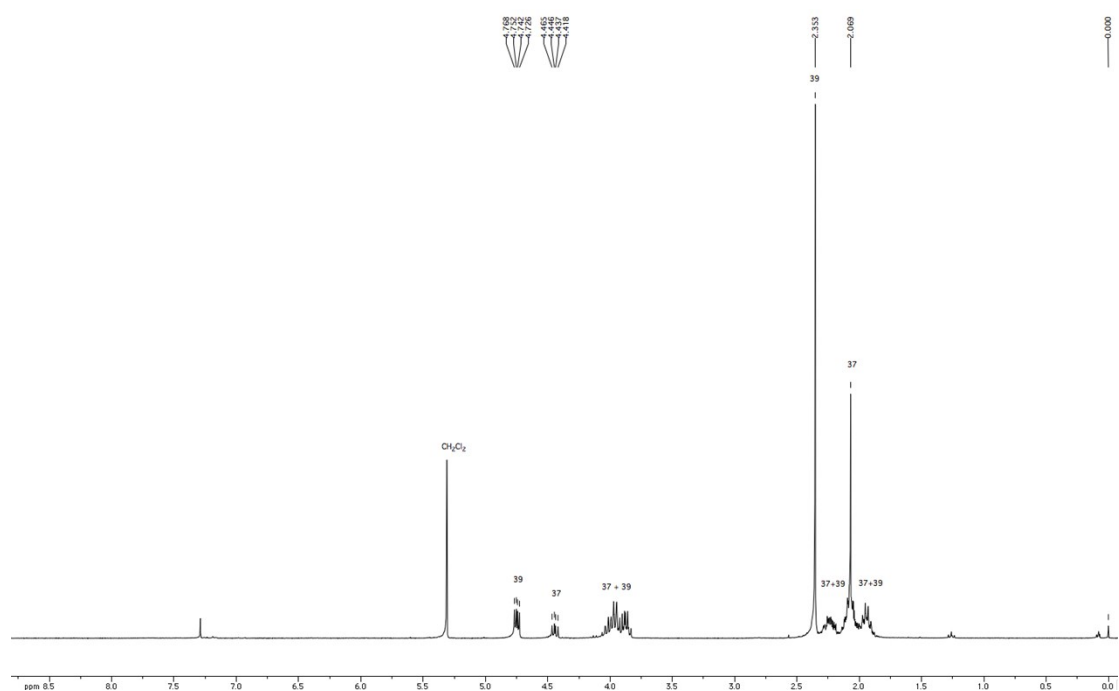
^1H NMR spectrum of FVP product from **16** containing alkyne **29** and oxete **30**



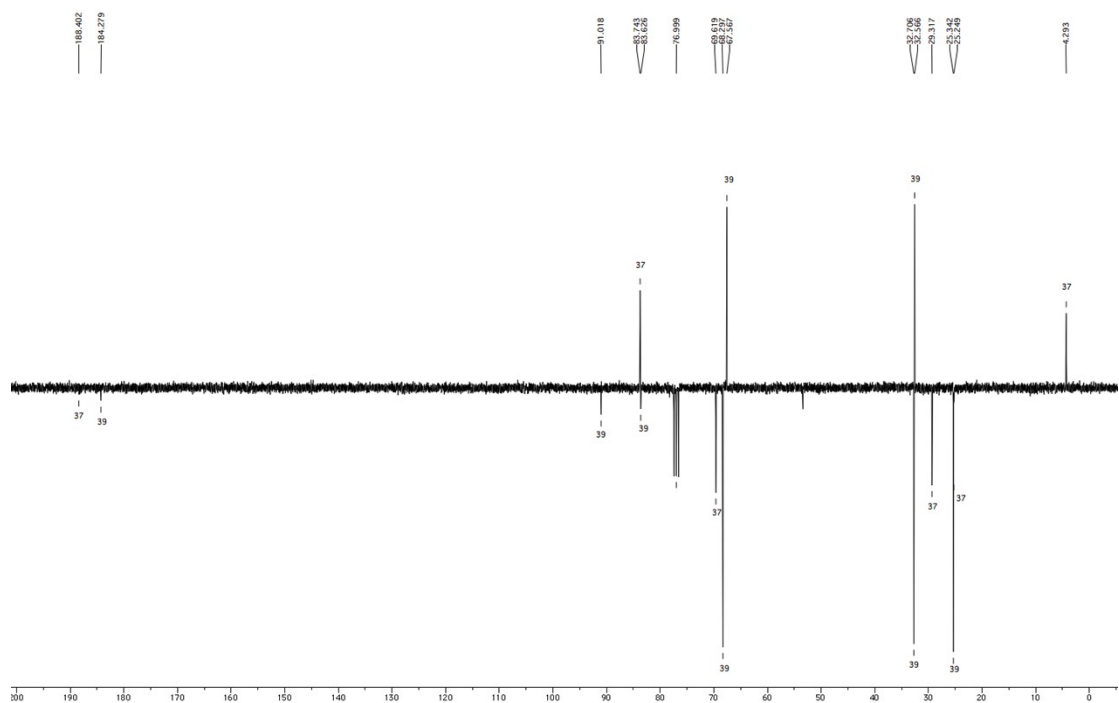
^{13}C NMR (DEPTQ) spectrum of FVP product from **16** containing alkyne **29** and oxete **30**



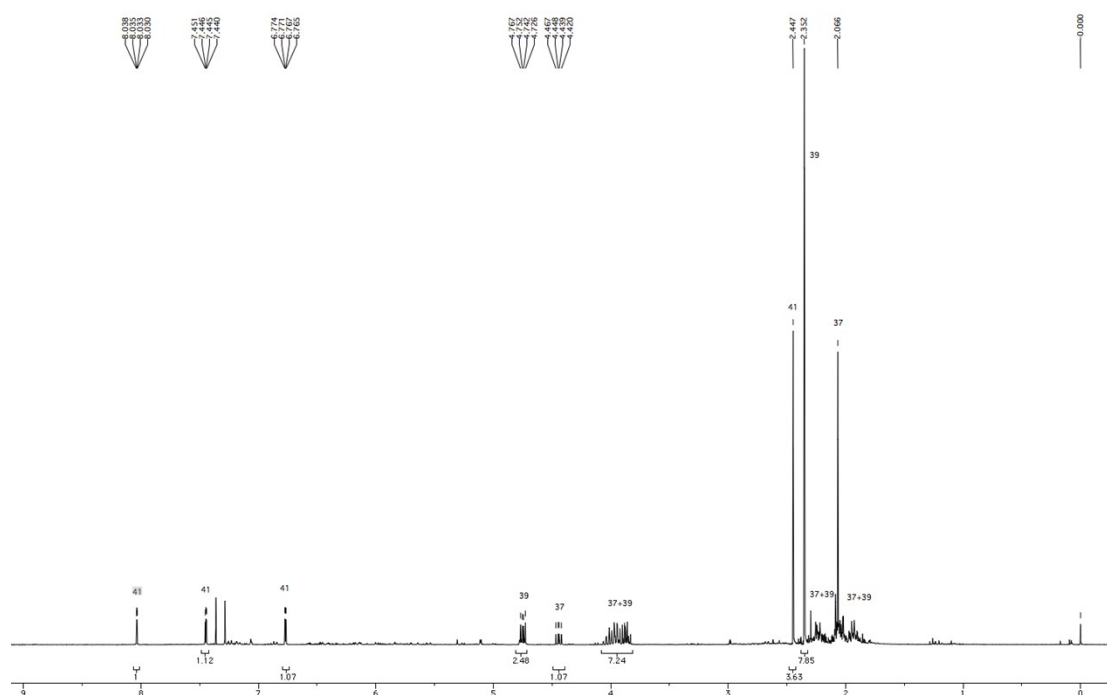
^1H NMR spectrum of 500 °C FVP product from **9** containing alkynes **37** and **39**



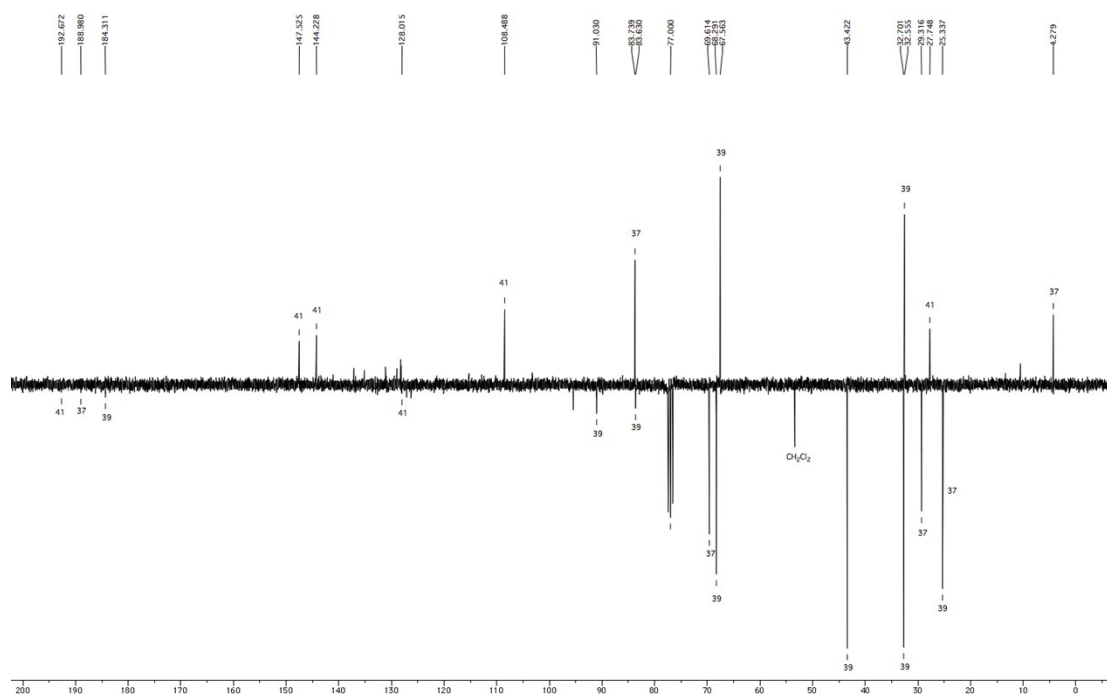
^{13}C NMR (DEPTQ) spectrum of 500 °C FVP product from **9** containing alkynes **37** and **39**



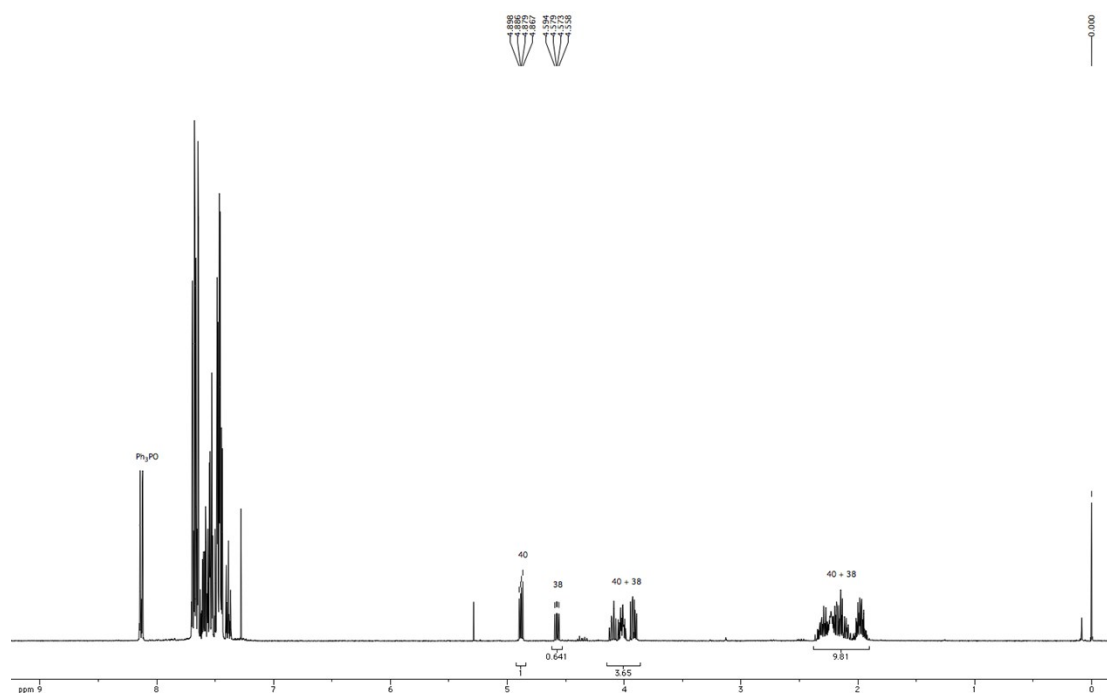
^1H NMR spectrum of 700 °C FVP product from **9** containing **37**, **39** and **41**



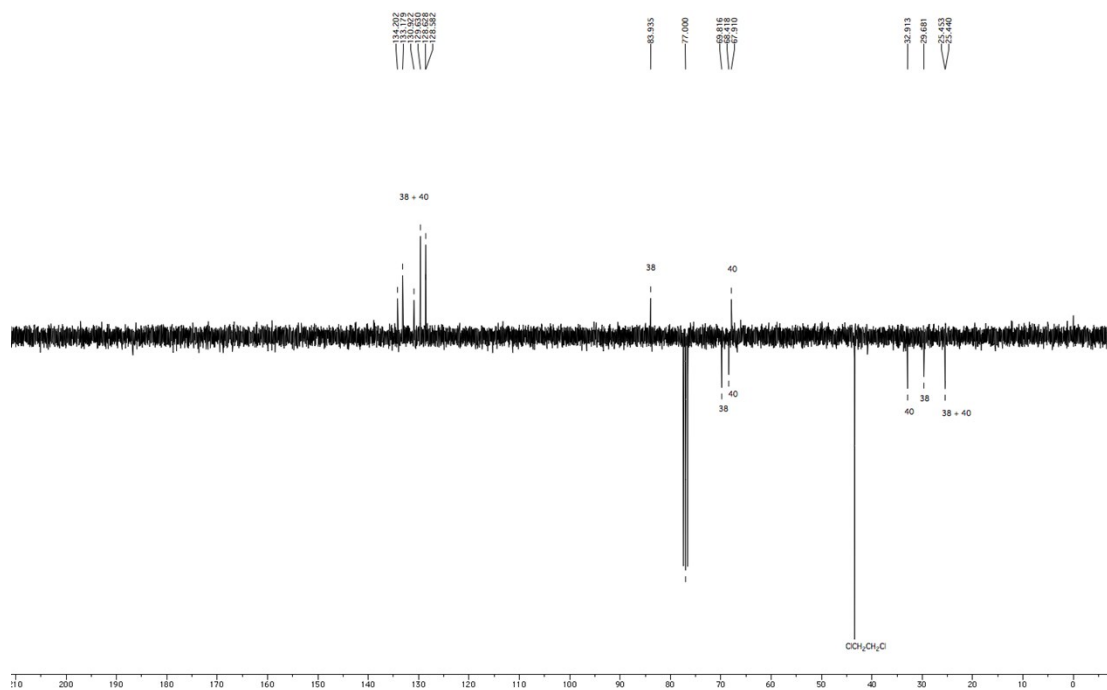
^{13}C NMR (DEPTQ) spectrum of 700 °C FVP product from **9** containing **37**, **39** and **41**



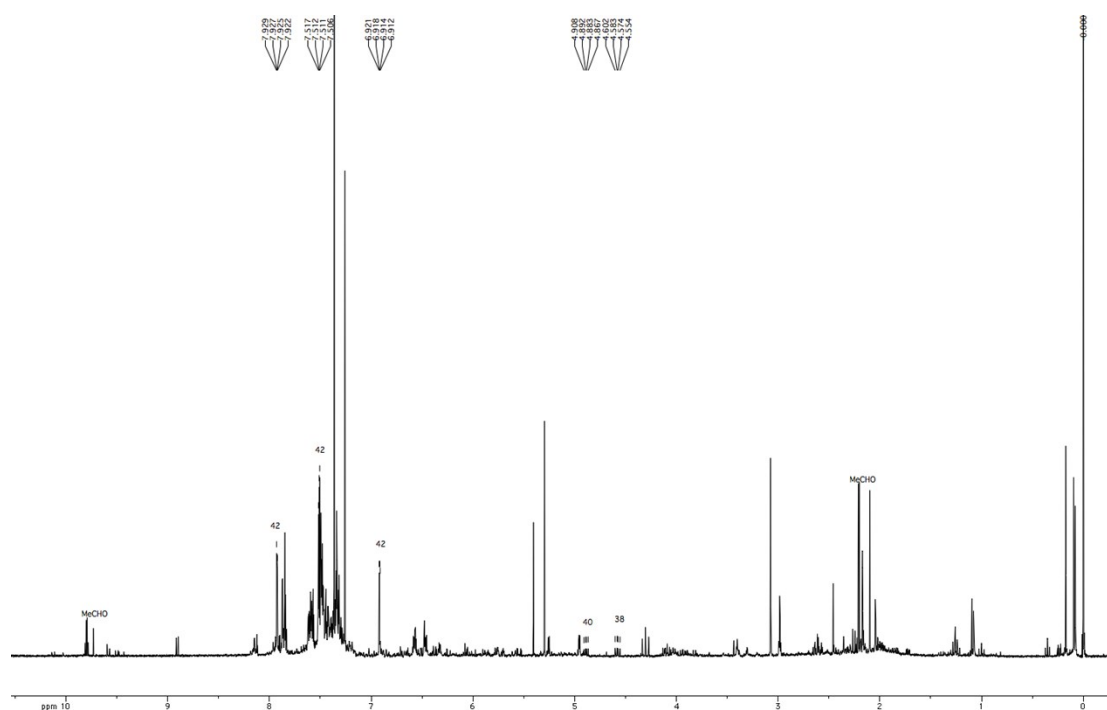
^1H NMR spectrum of 500 °C FVP product from **10** containing alkynes **38** and **40**



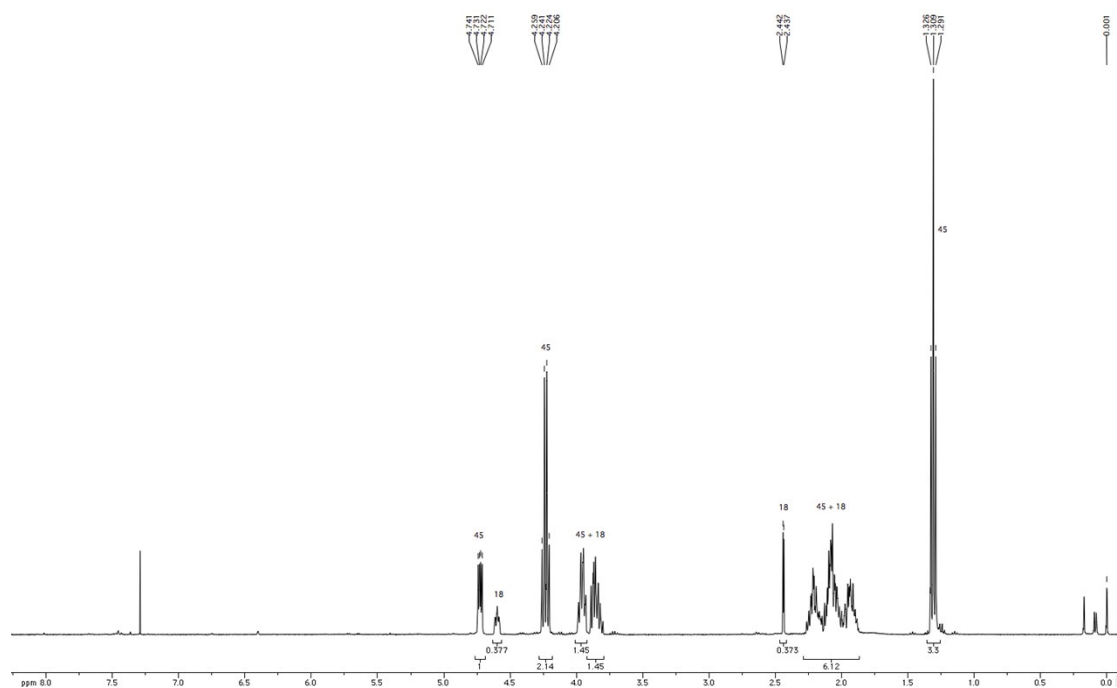
^{13}C NMR (DEPTQ) spectrum of 500 °C FVP product from **10** containing alkynes **38** and **40**



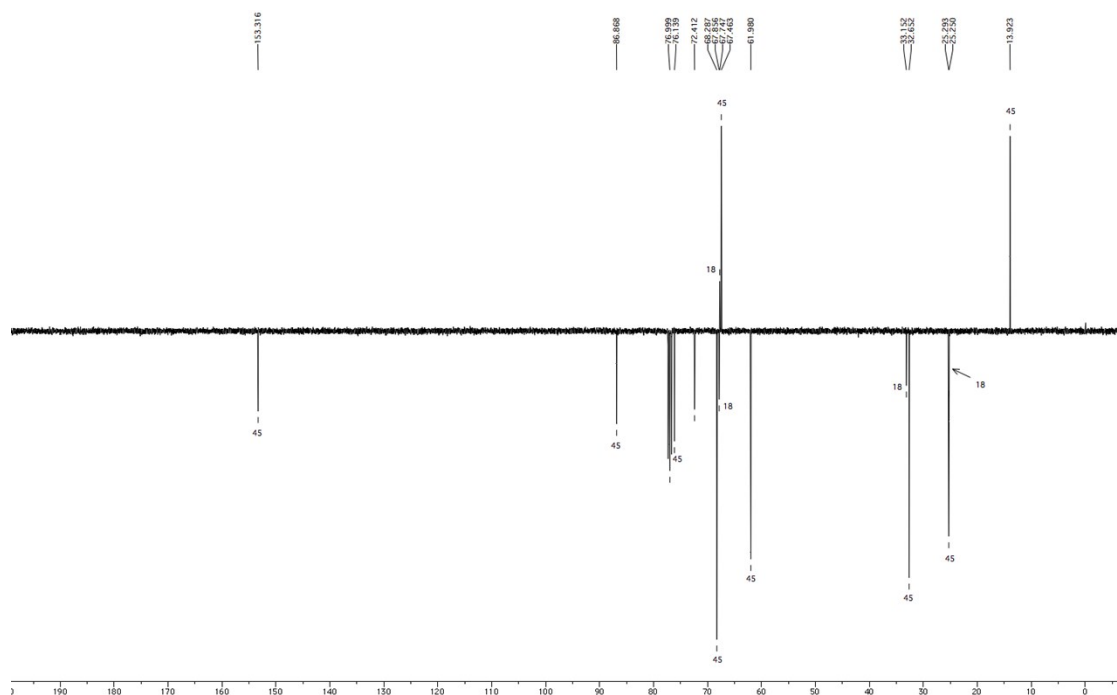
^1H NMR spectrum of 700 °C FVP product from **10** containing **38**, **40** and **42**



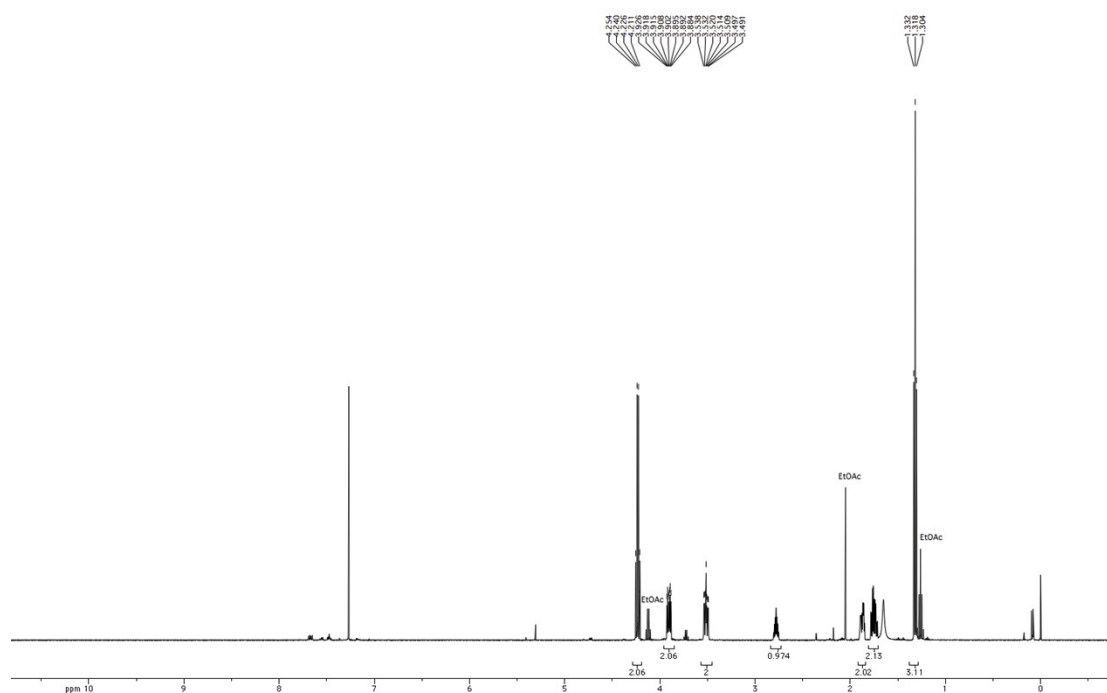
^1H NMR spectrum of FVP product from **11** containing alkynes **45** and **18**



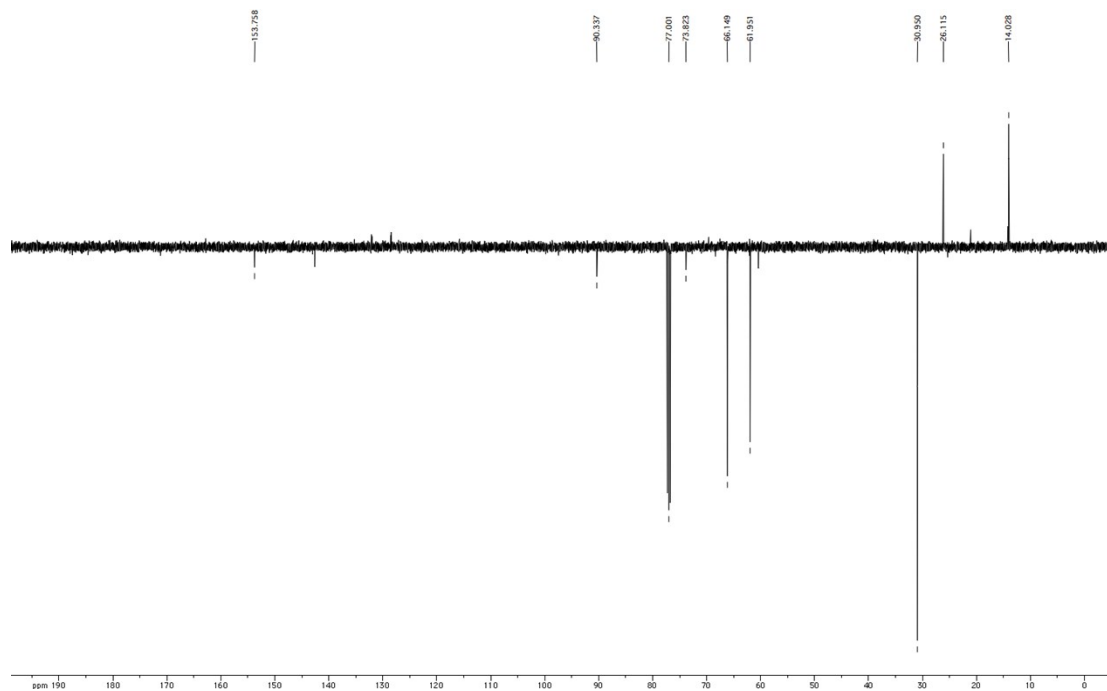
^{13}C NMR (DEPTQ) spectrum of FVP product from **11** containing alkynes **45** and **18**



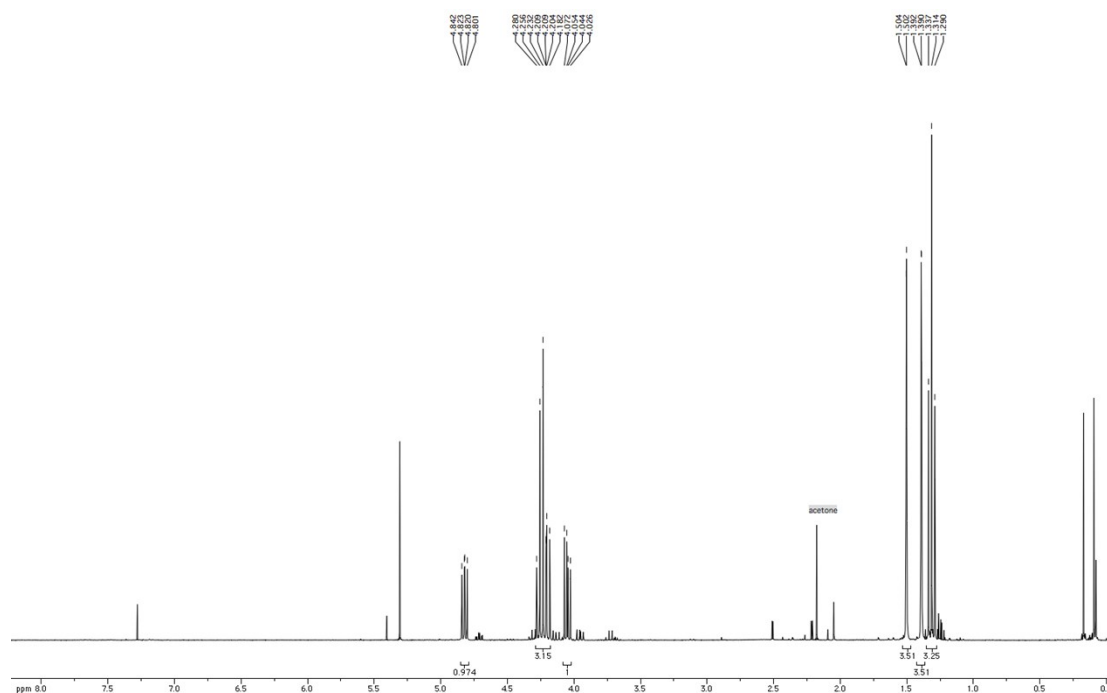
^1H NMR spectrum of FVP product from **15** containing alkyne **46**



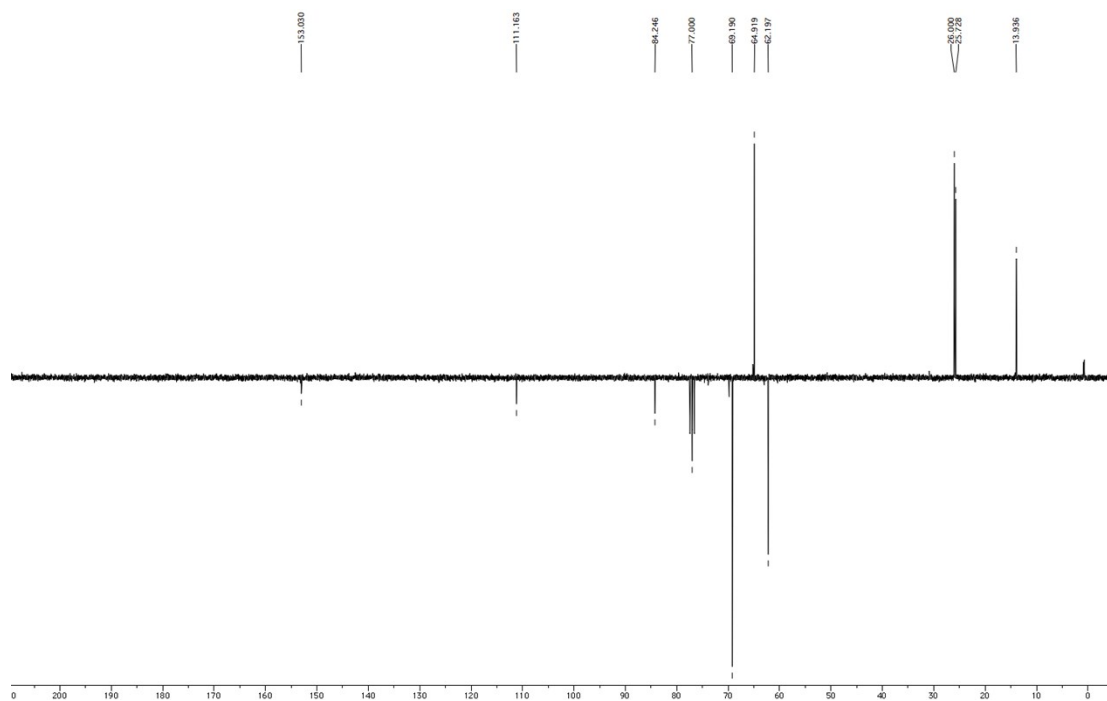
^{13}C NMR (DEPTQ) spectrum of FVP product from **15** containing alkyne **46**



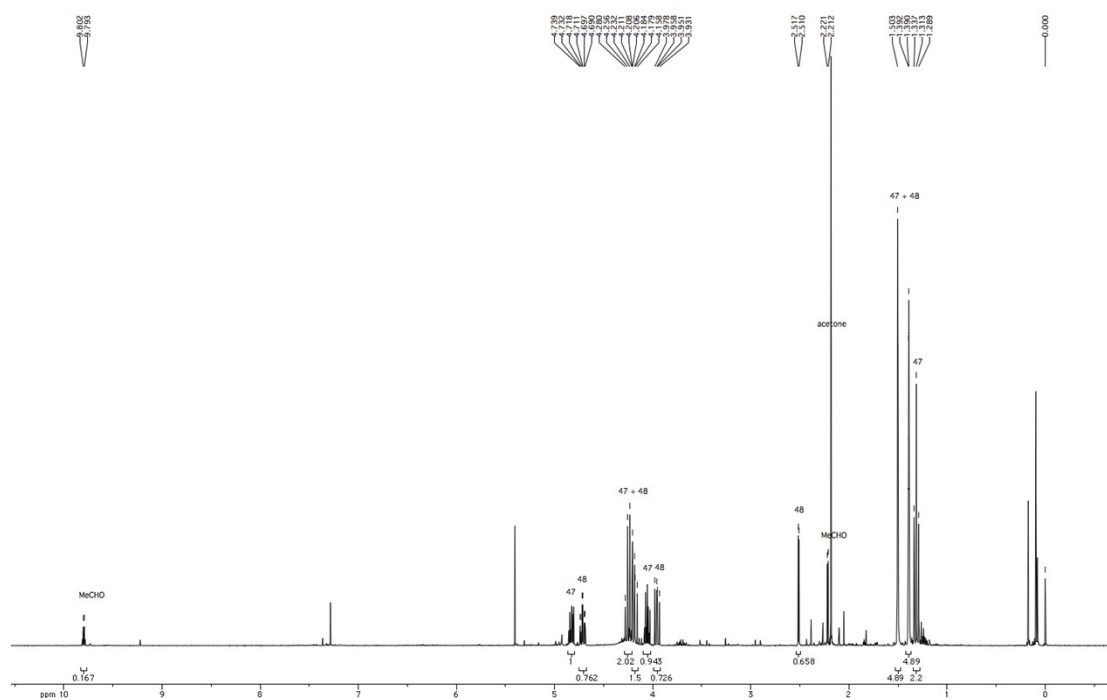
^1H NMR spectrum of 600 °C FVP product from **17** containing mainly alkyne **47**



^{13}C NMR (DEPTQ) spectrum of 600 °C FVP product from **17** containing mainly alkyne **47**



^1H NMR spectrum of 650 °C FVP product from **17** containing alkynes **47** and **48**



^{13}C NMR (DEPTQ) spectrum of 650 °C FVP product from **17** containing alkynes **47** and **48**

