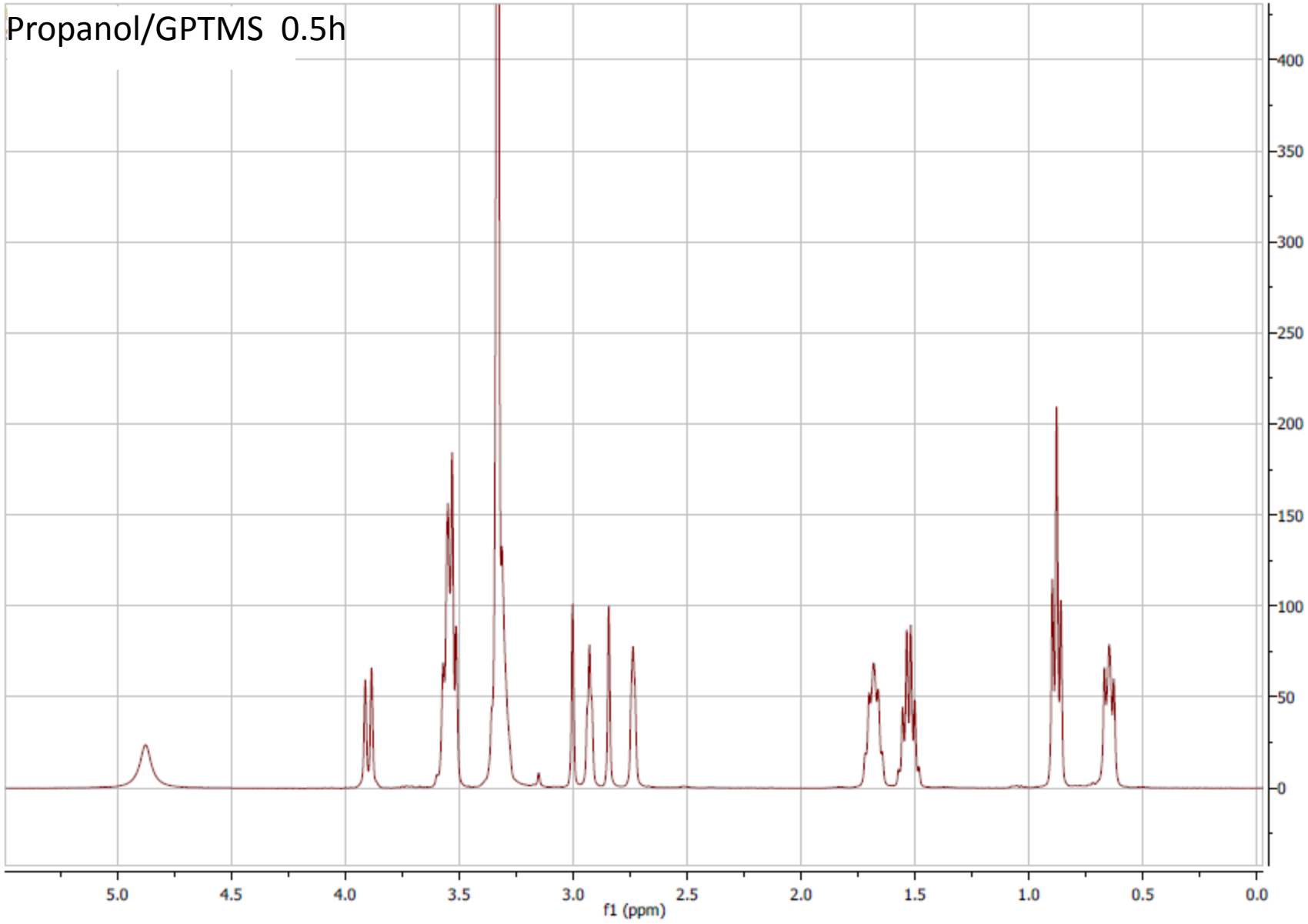
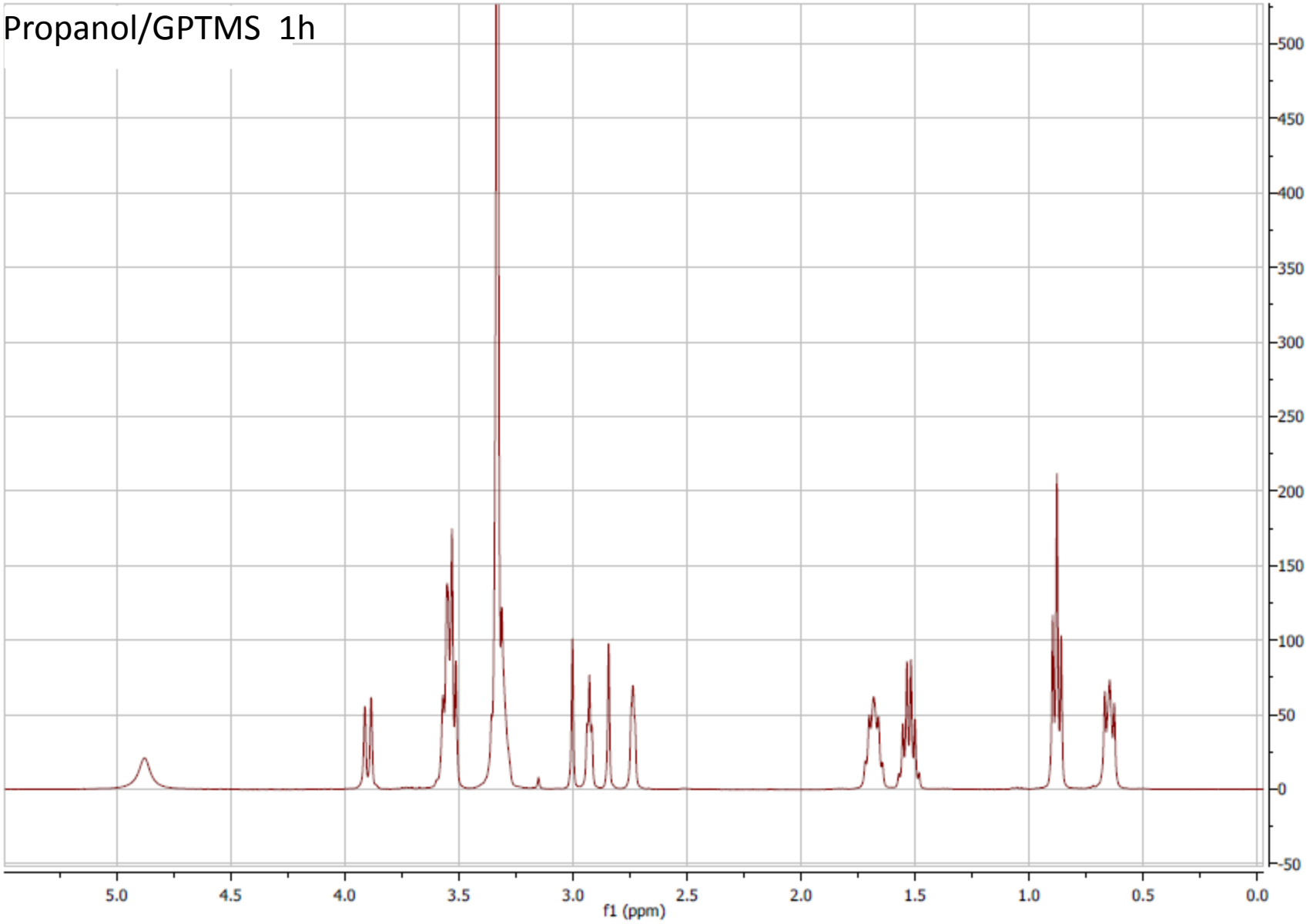
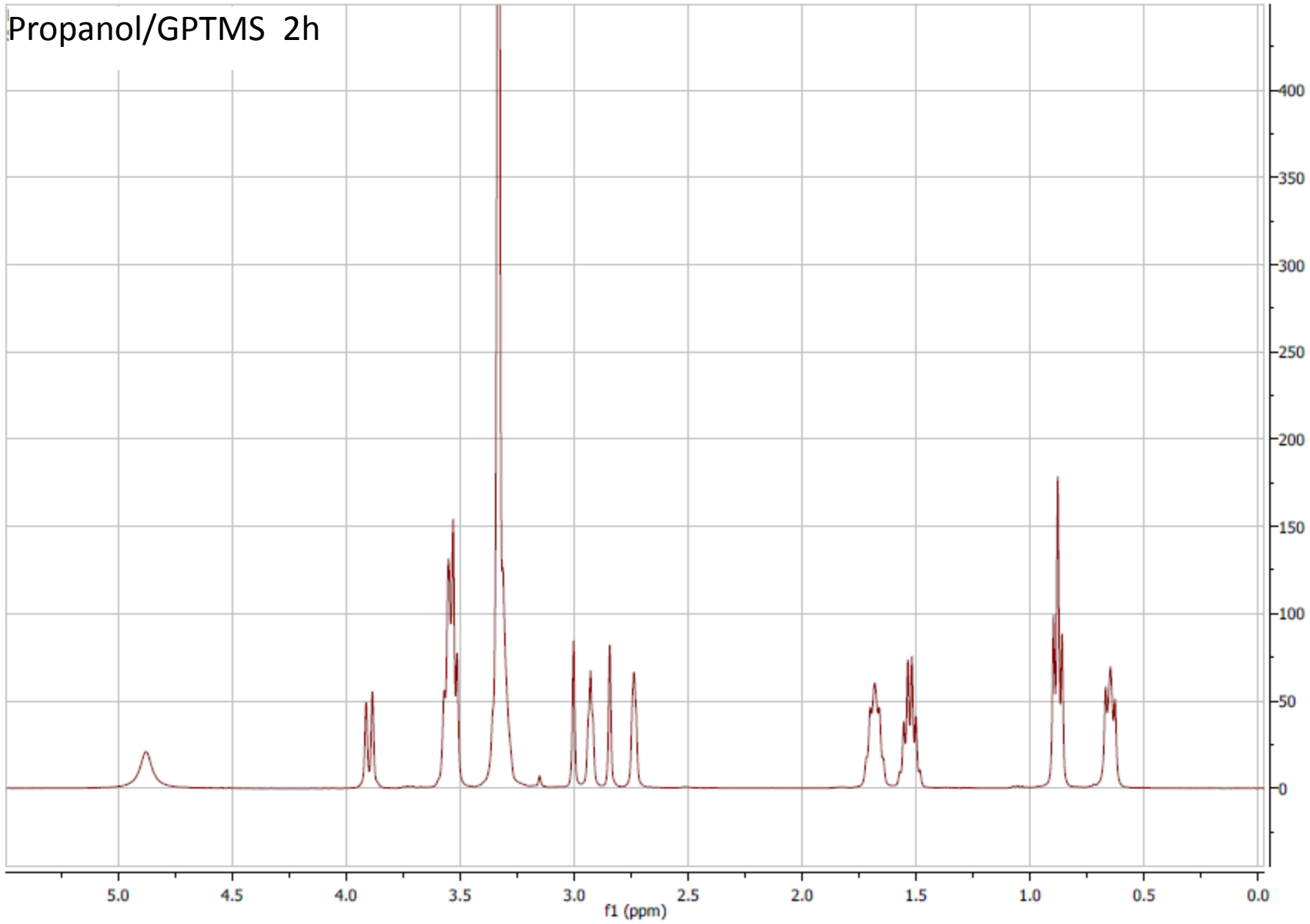
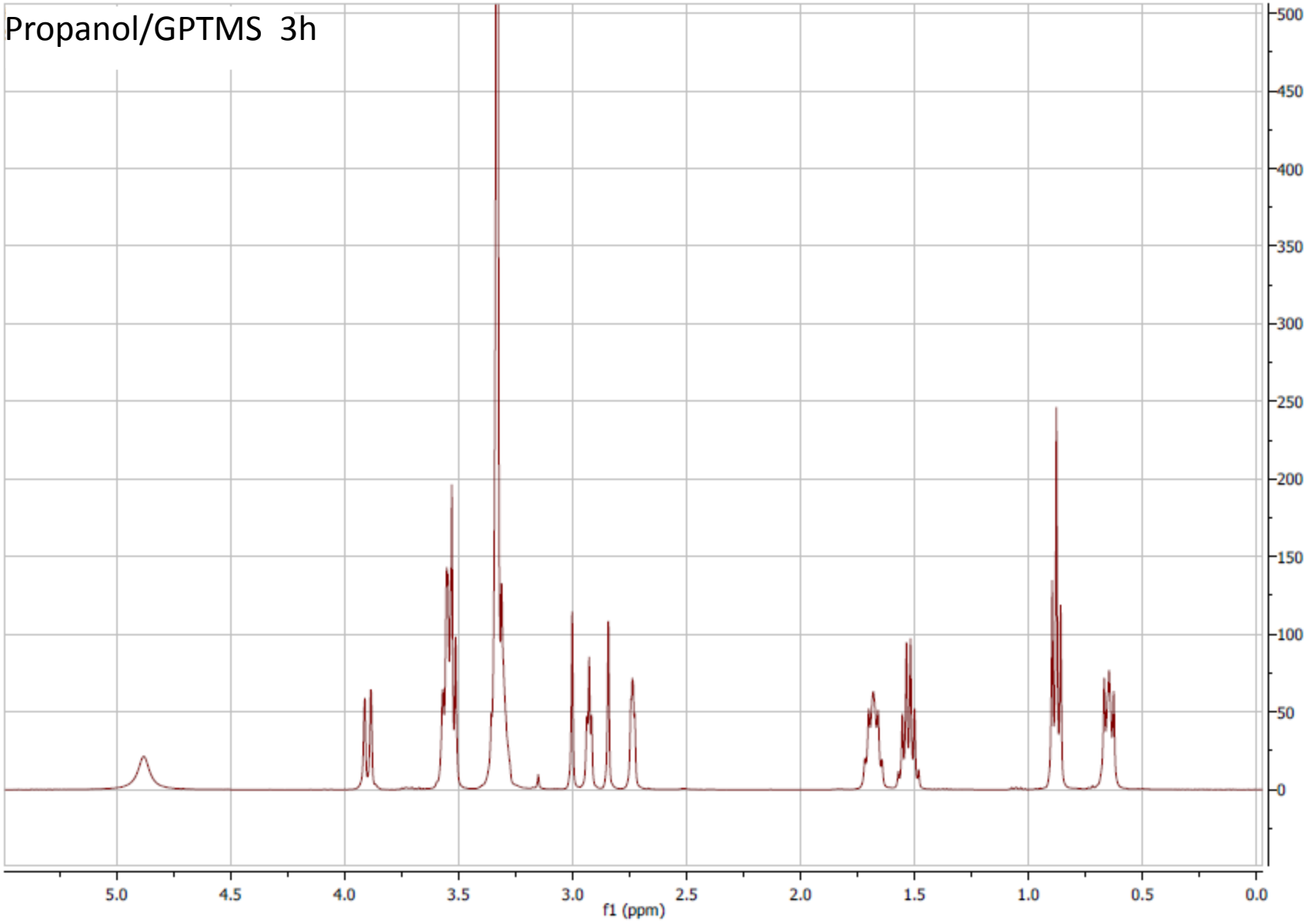
Electronic Supplementary Material (ESI) for RSC Advances

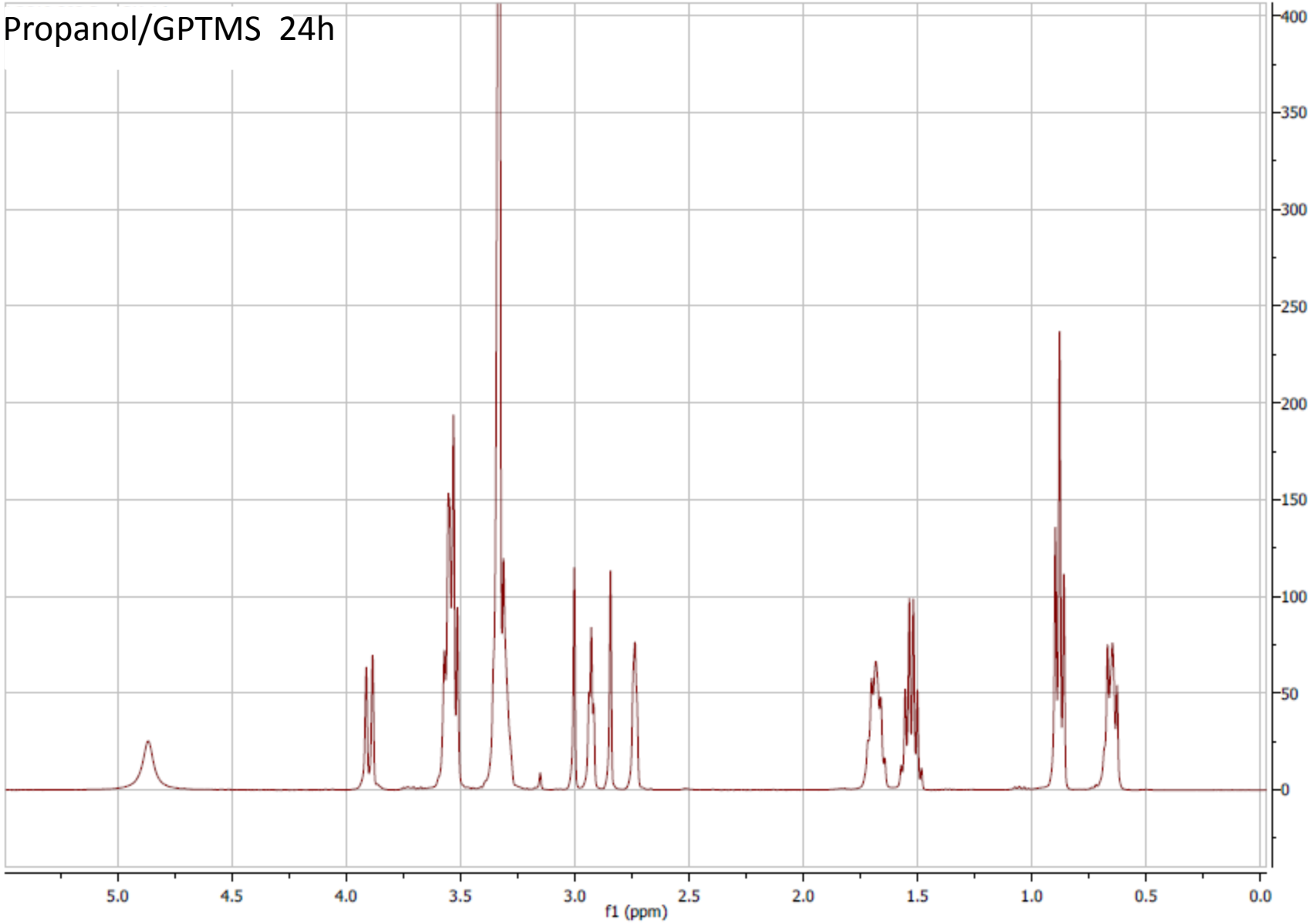
Propanol reaction NMR spectra





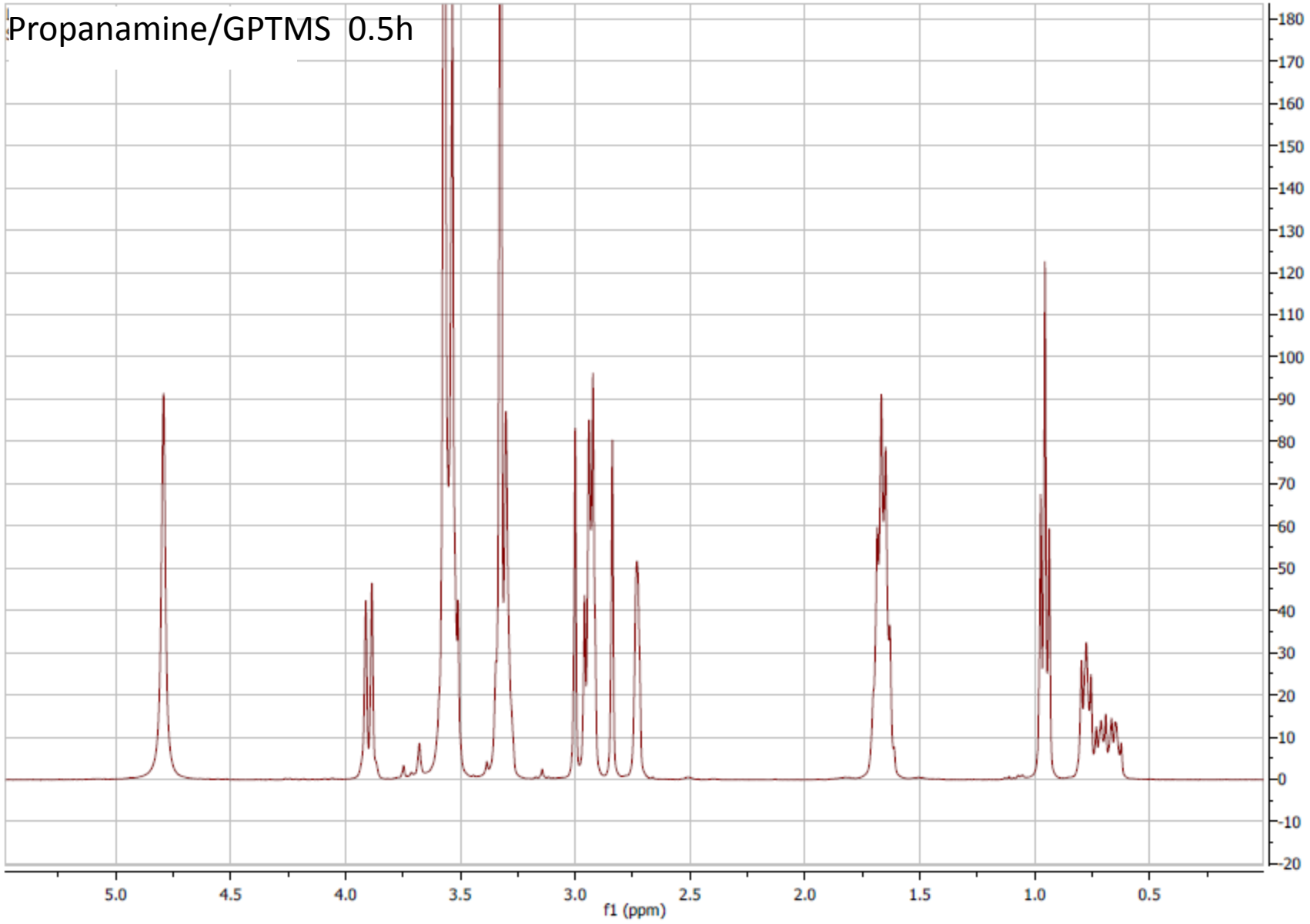


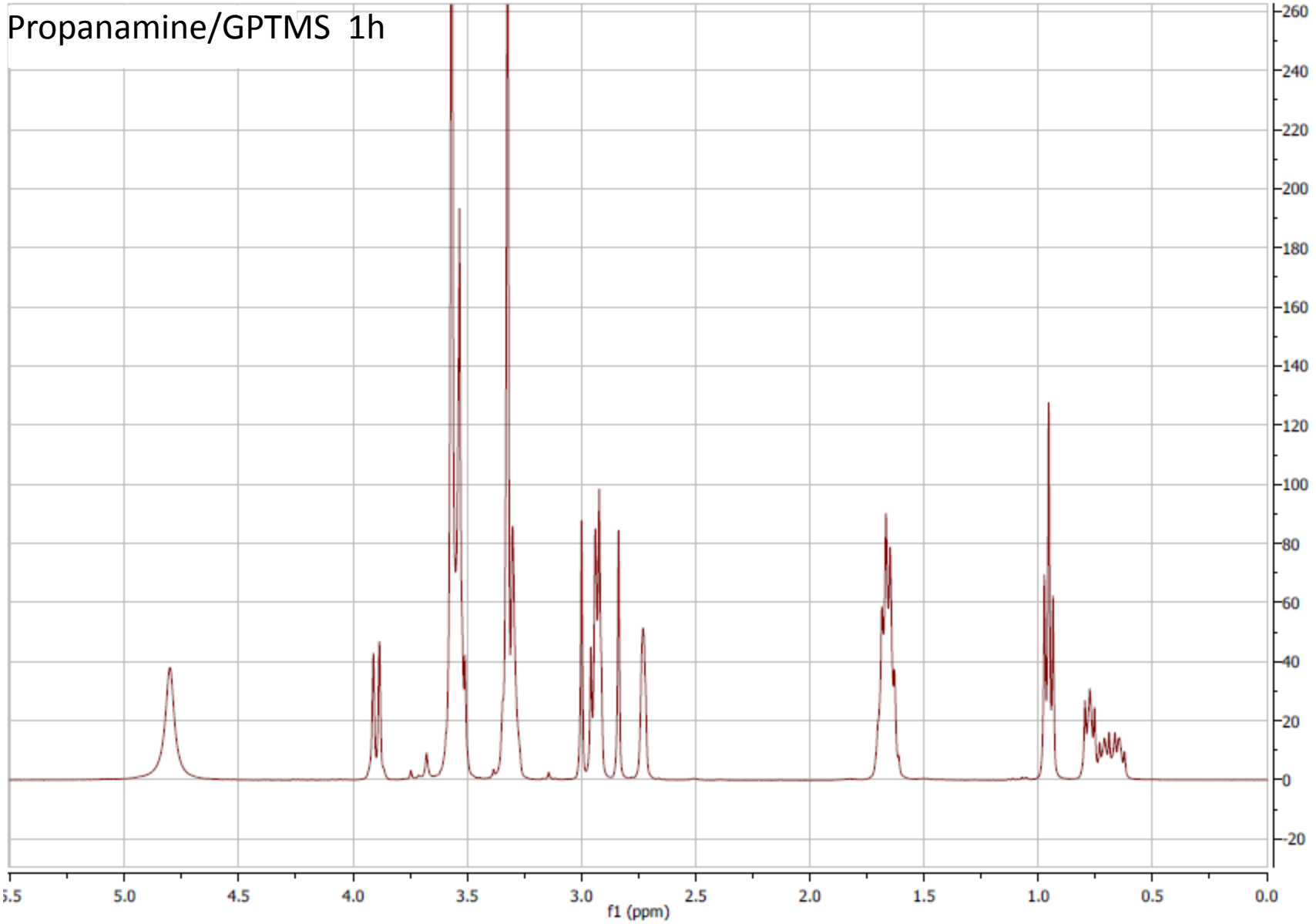


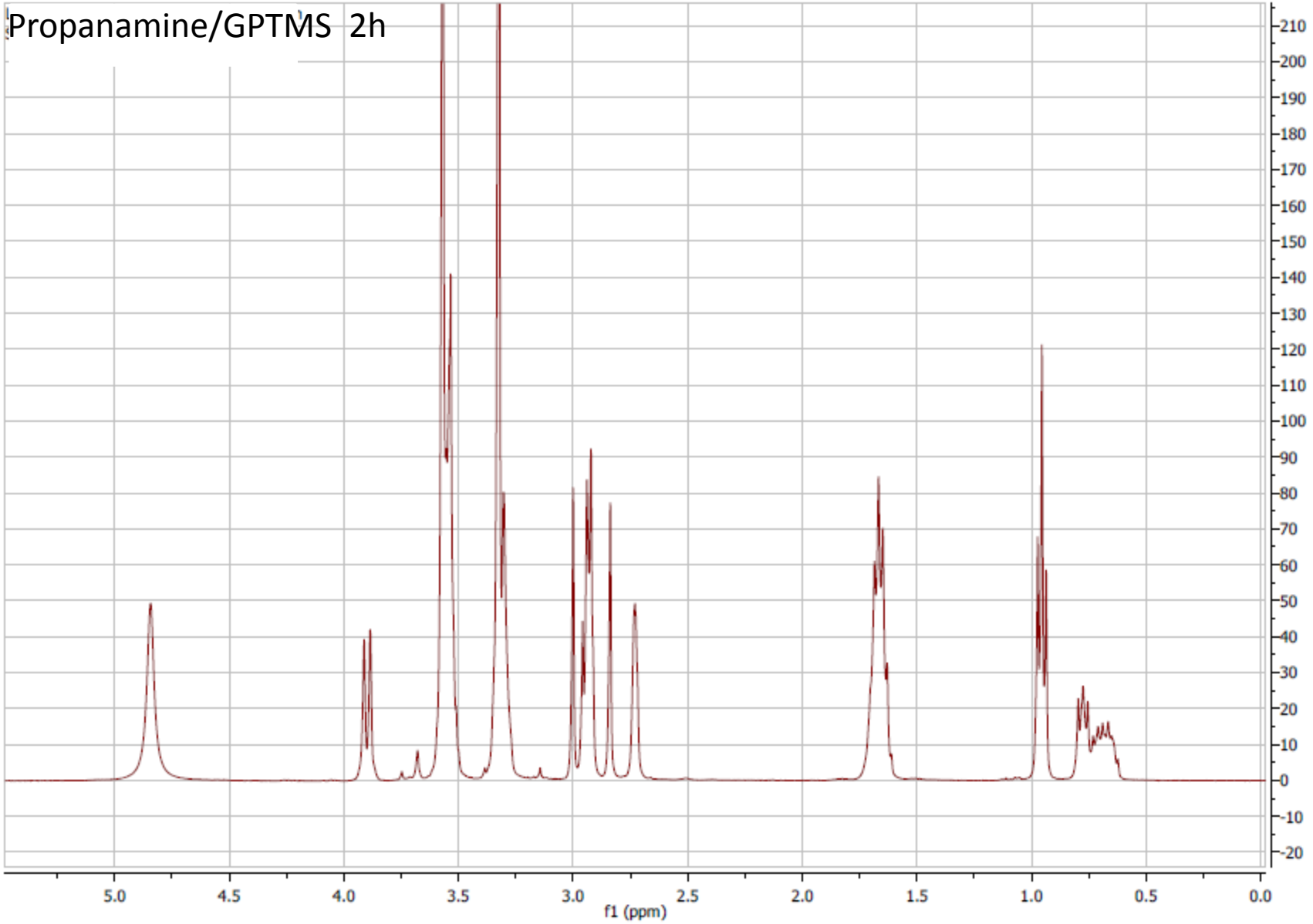


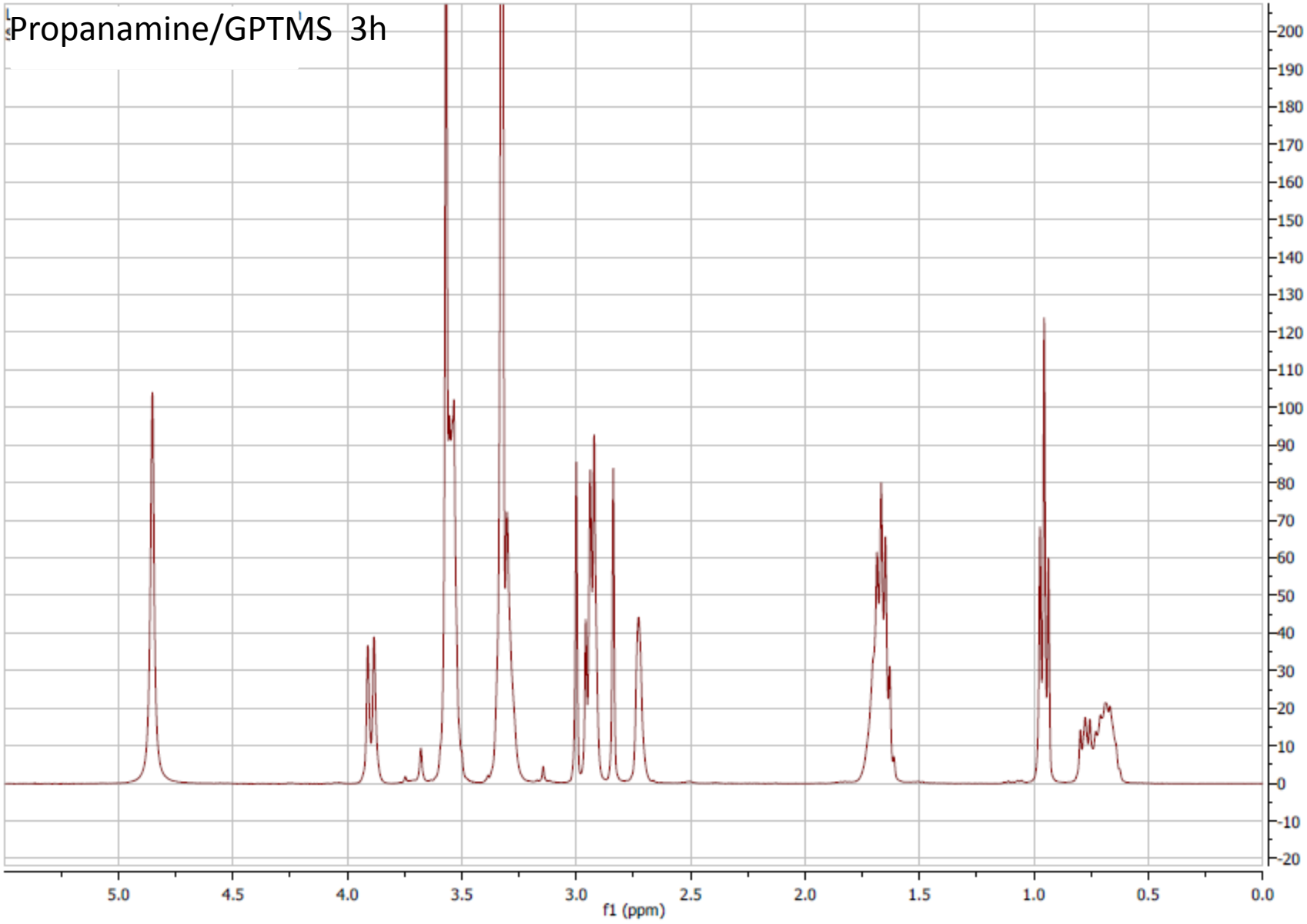
Electronic Supplementary Material (ESI) for RSC Advances

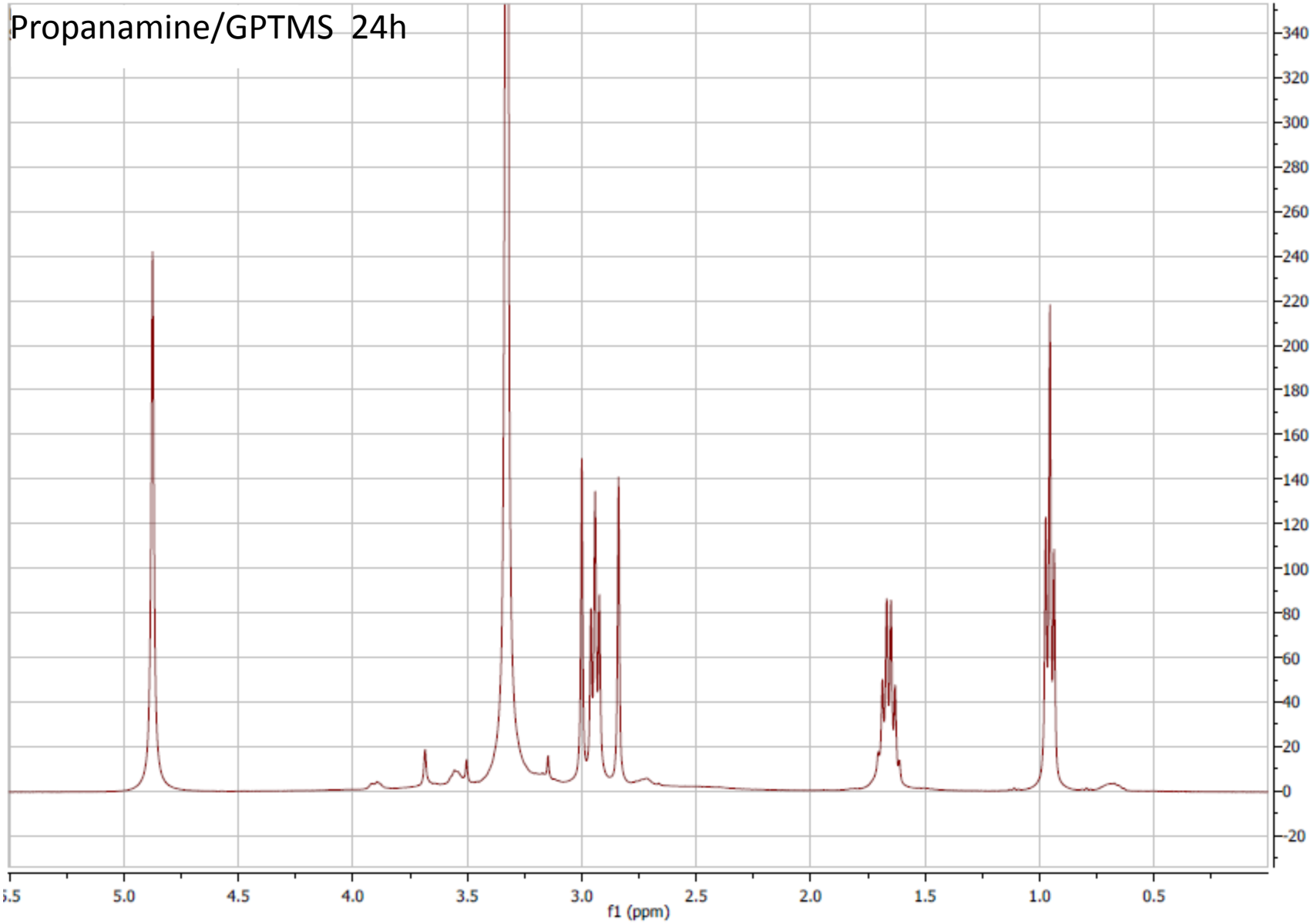
Propanamine reaction NMR spectra





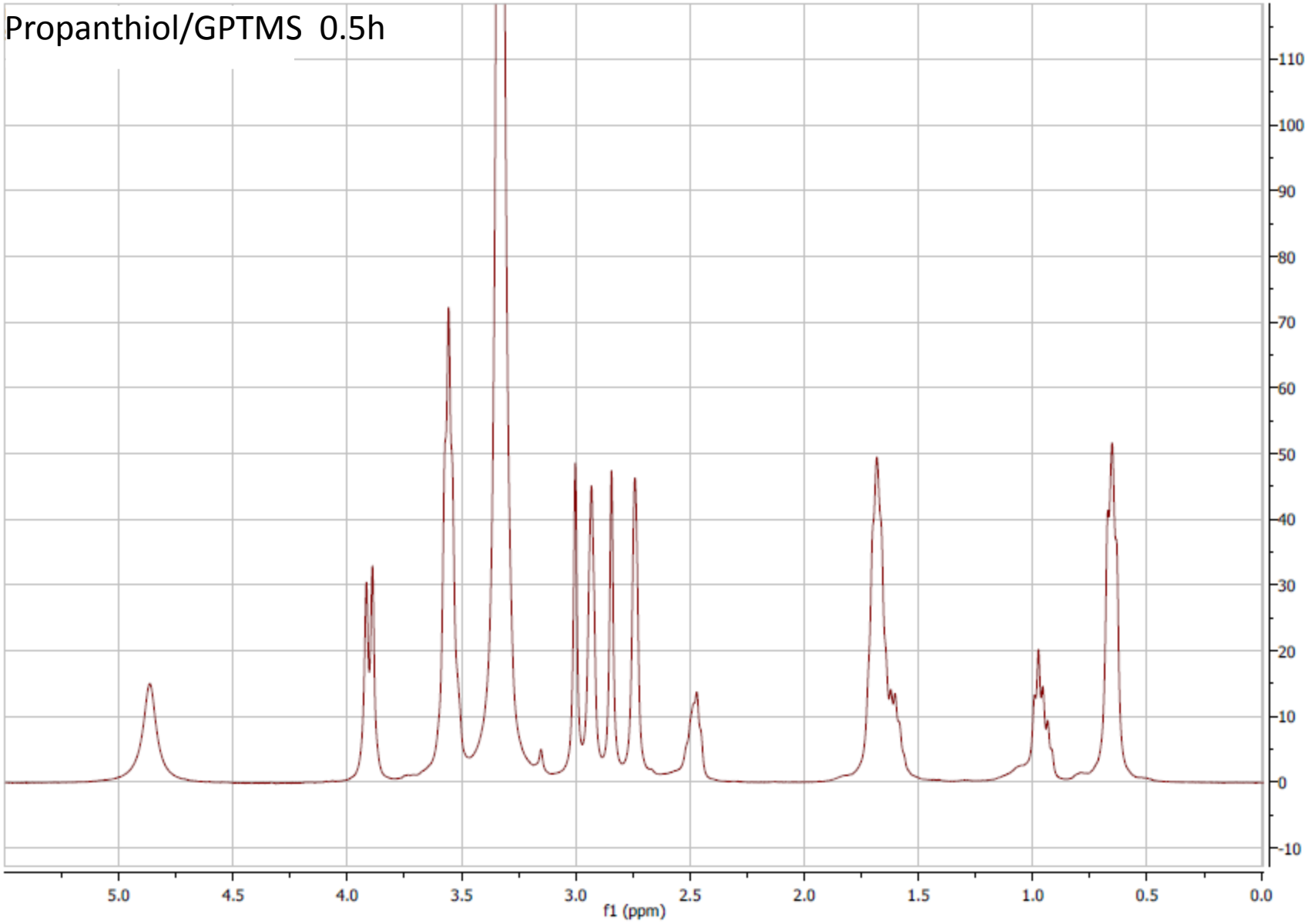


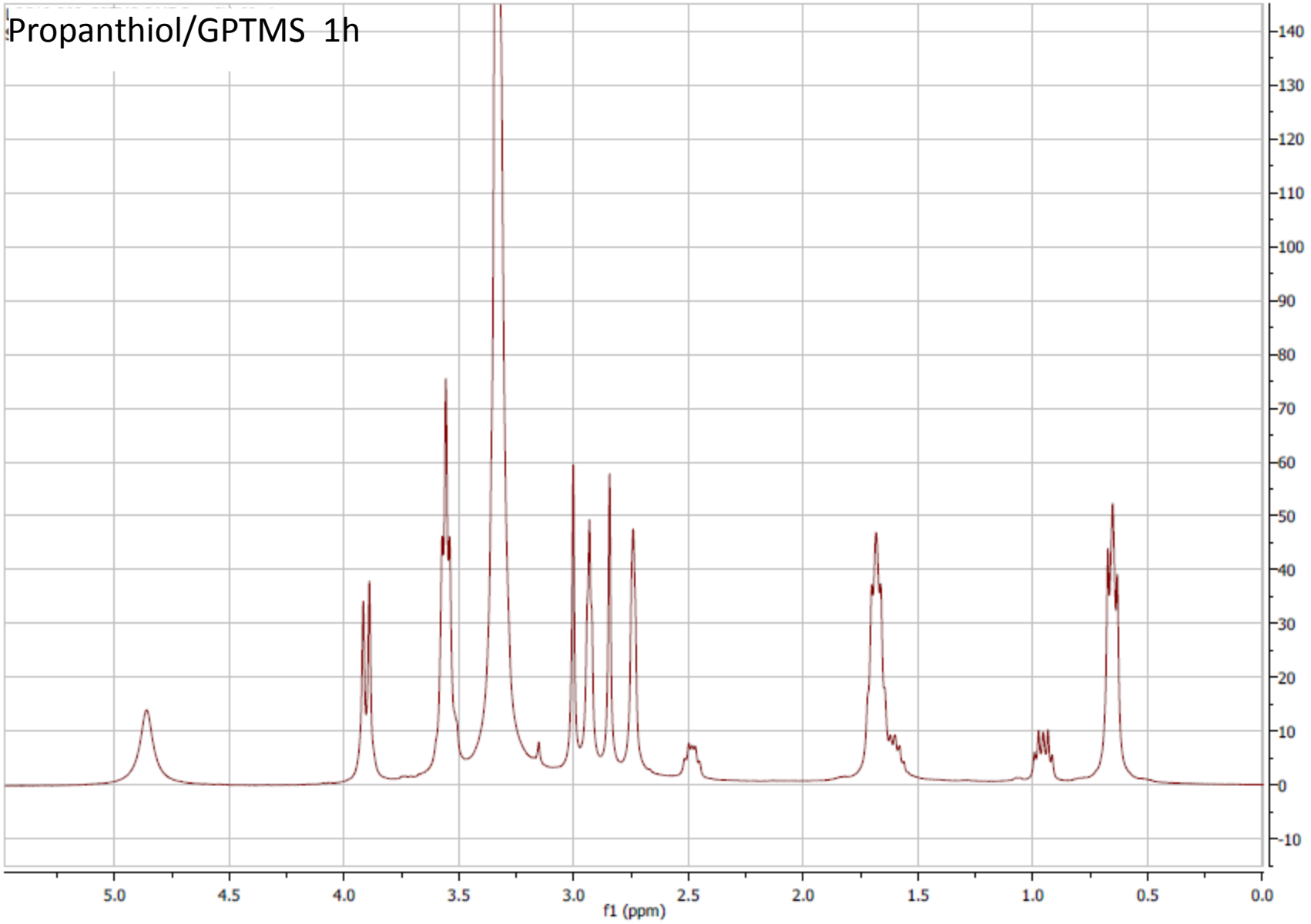


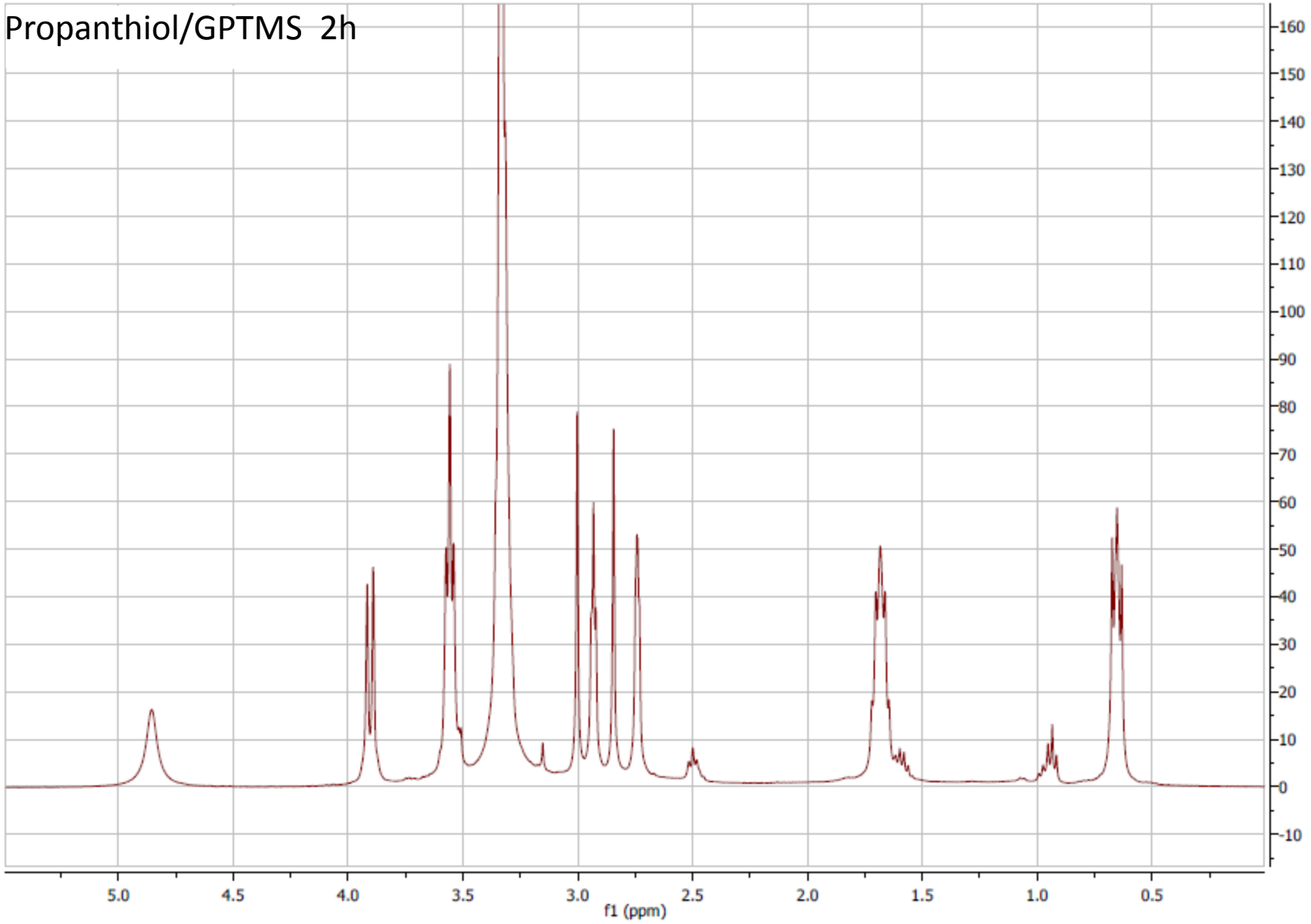


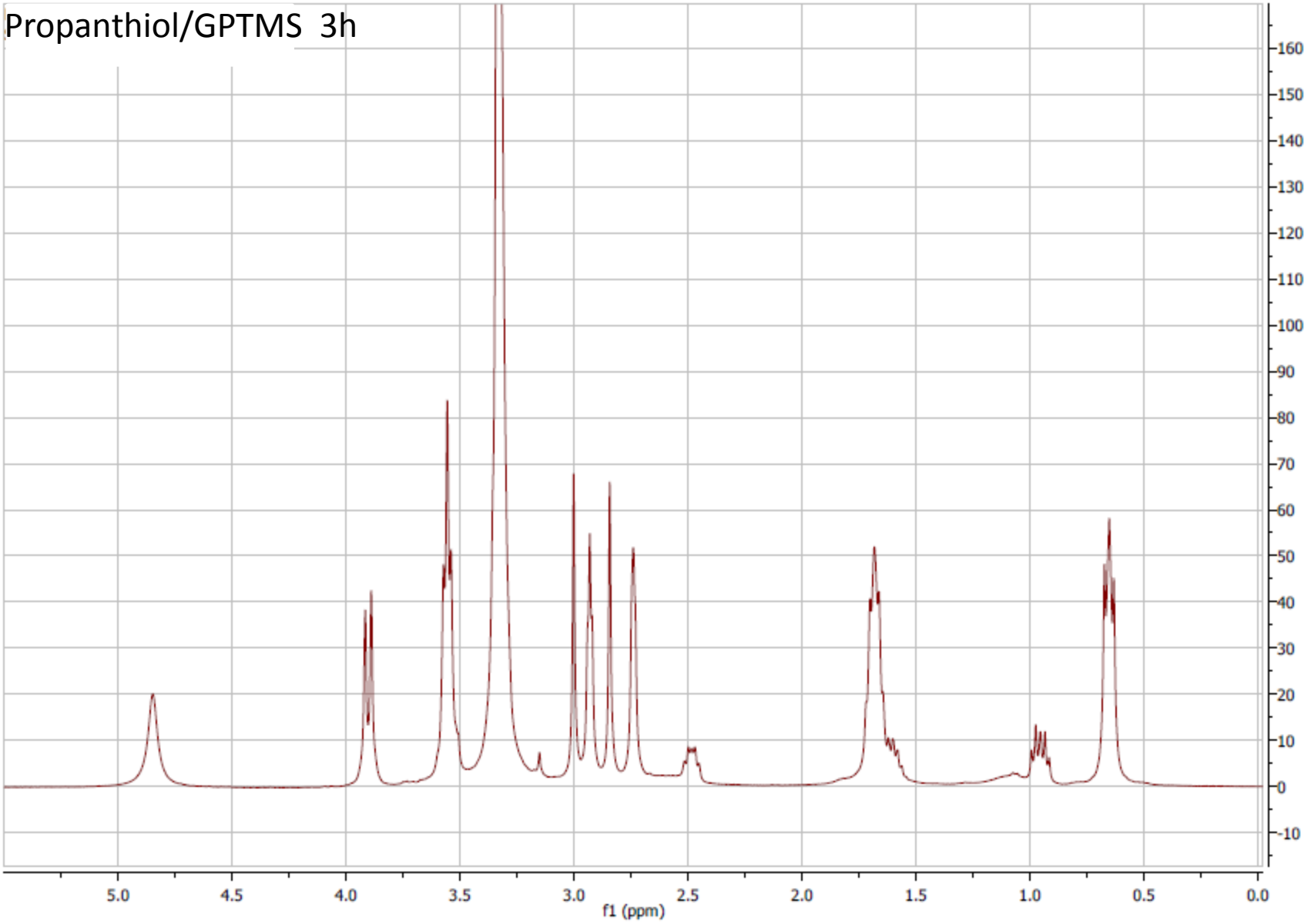
Electronic Supplementary Material (ESI) for RSC Advances

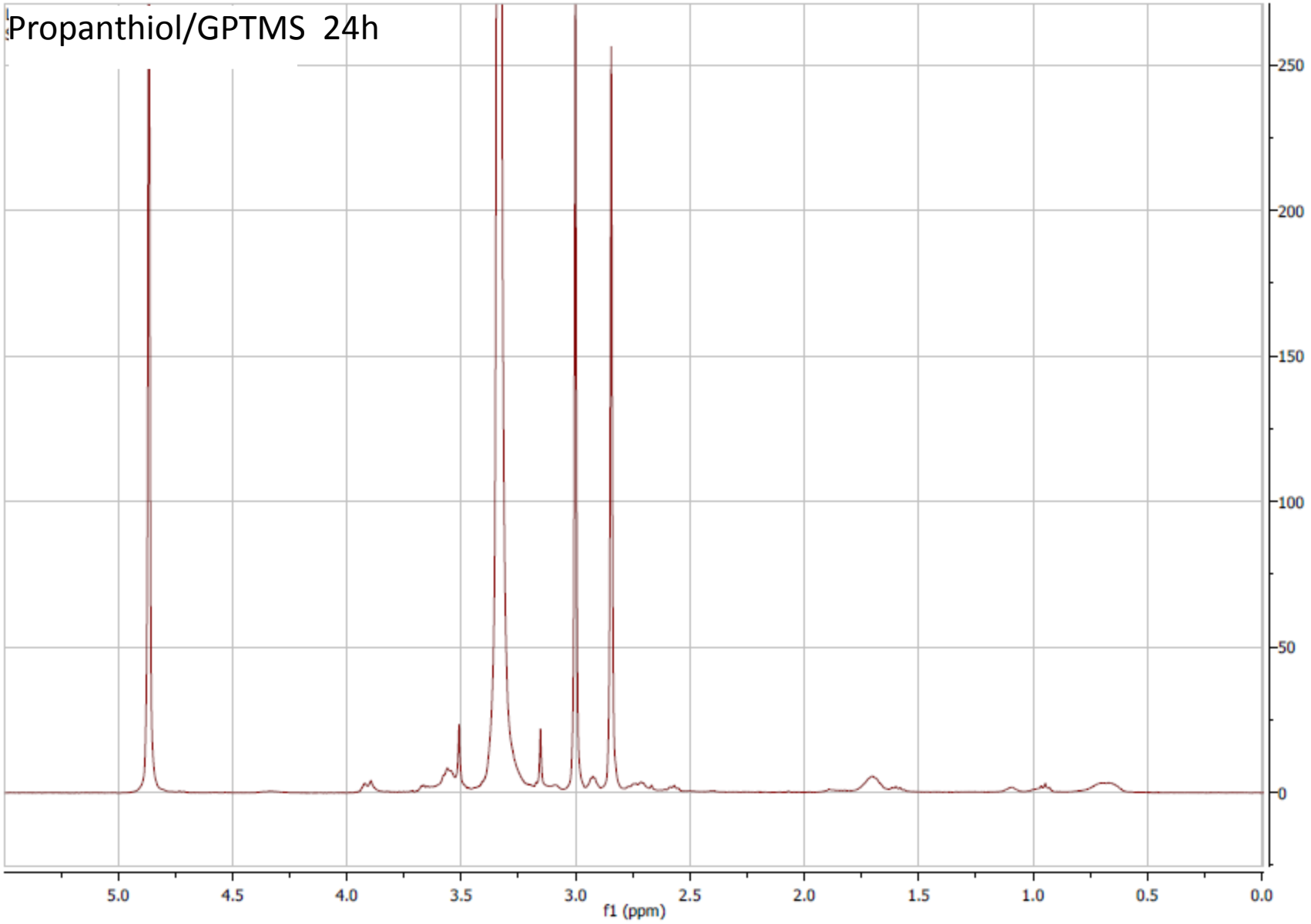
Propanthiol reaction NMR spectra





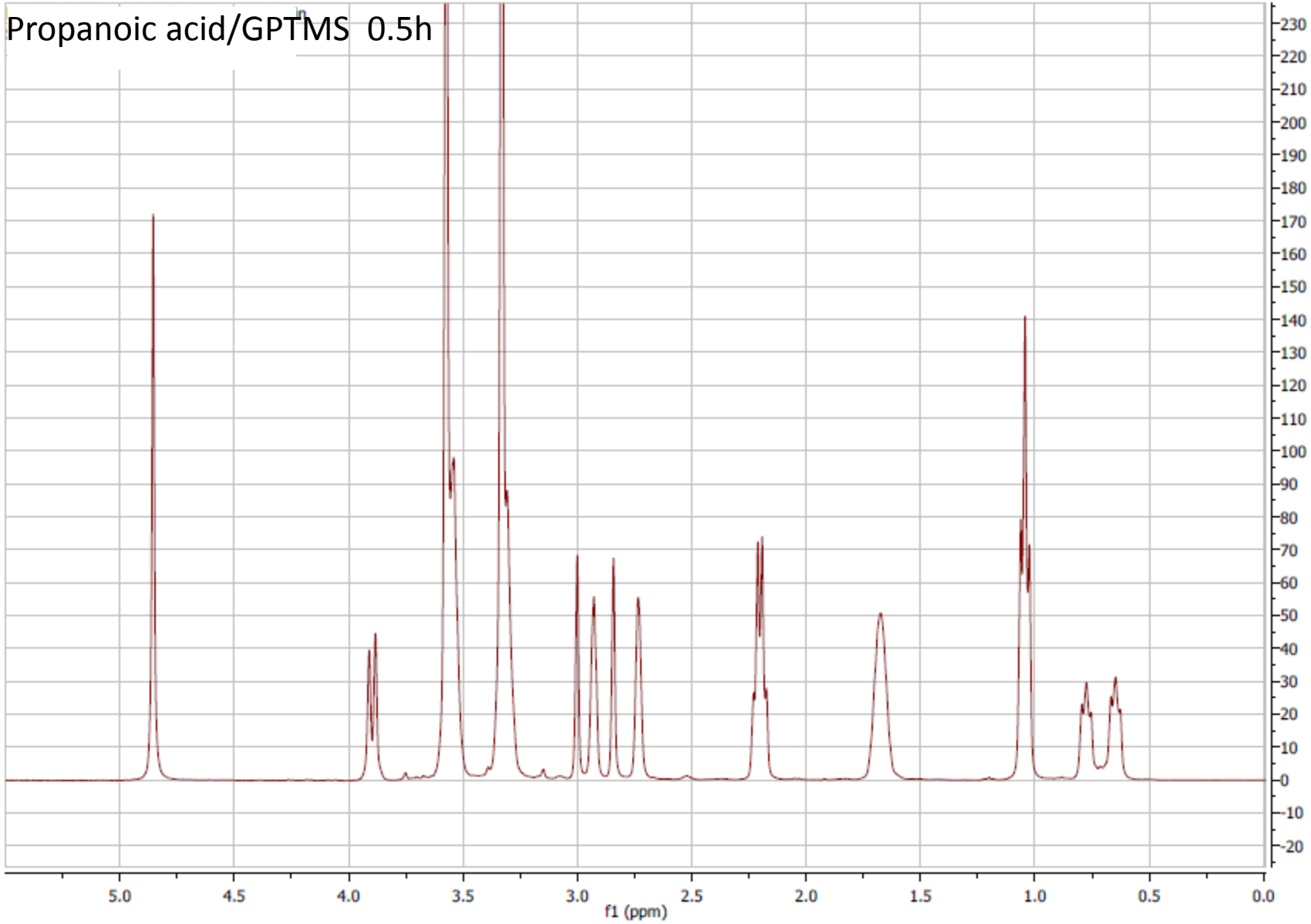


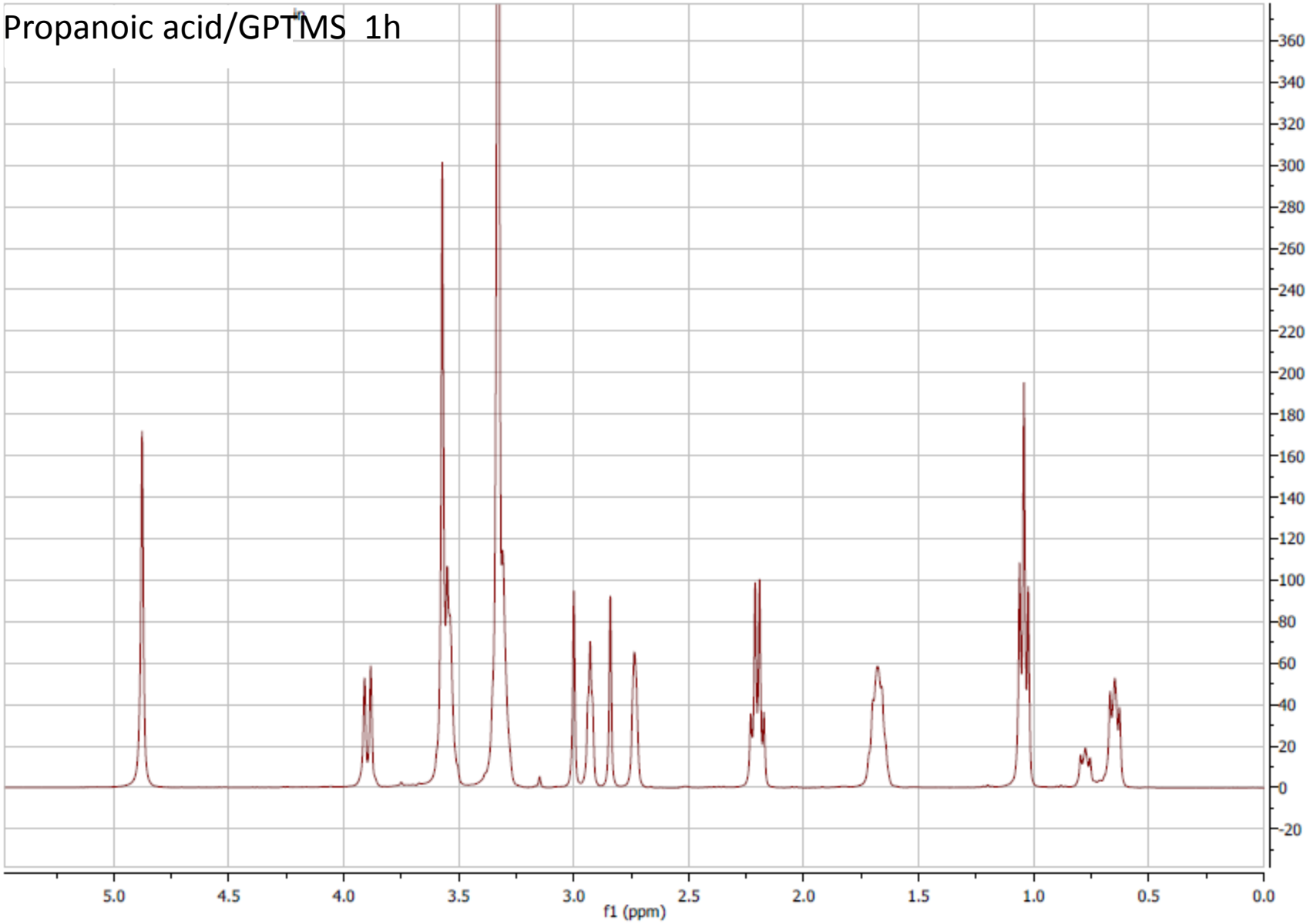


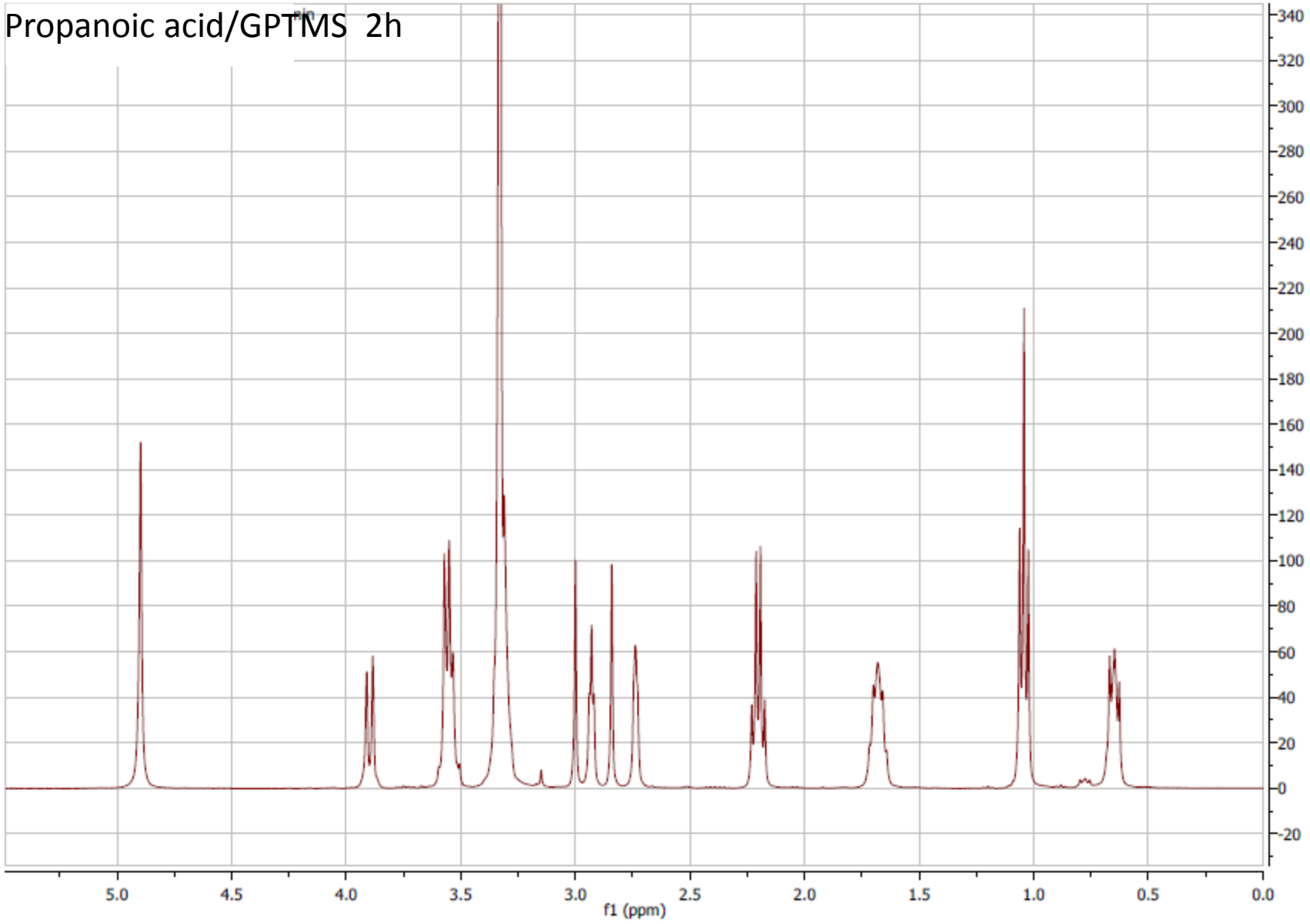


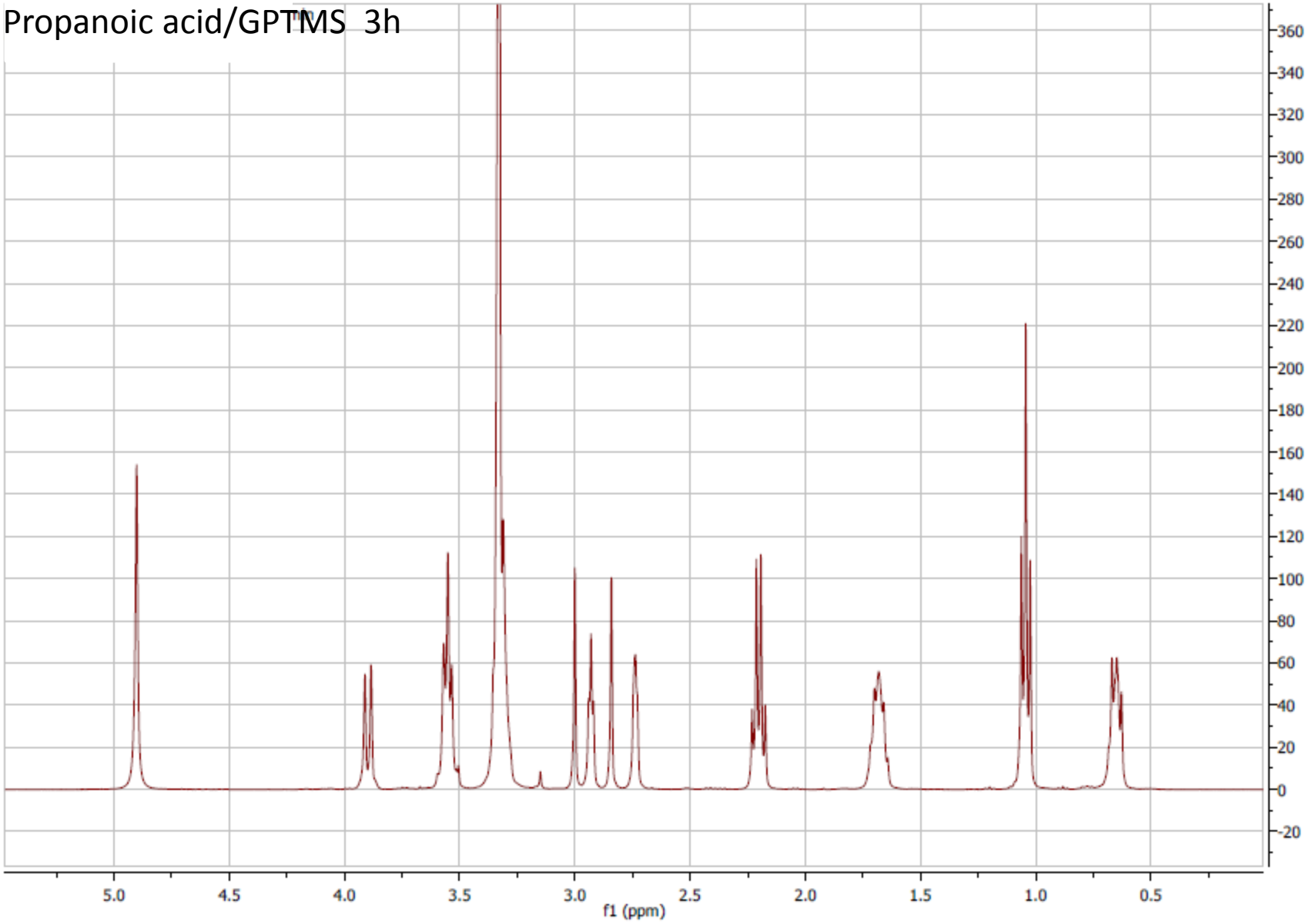
Electronic Supplementary Material (ESI) for RSC Advances

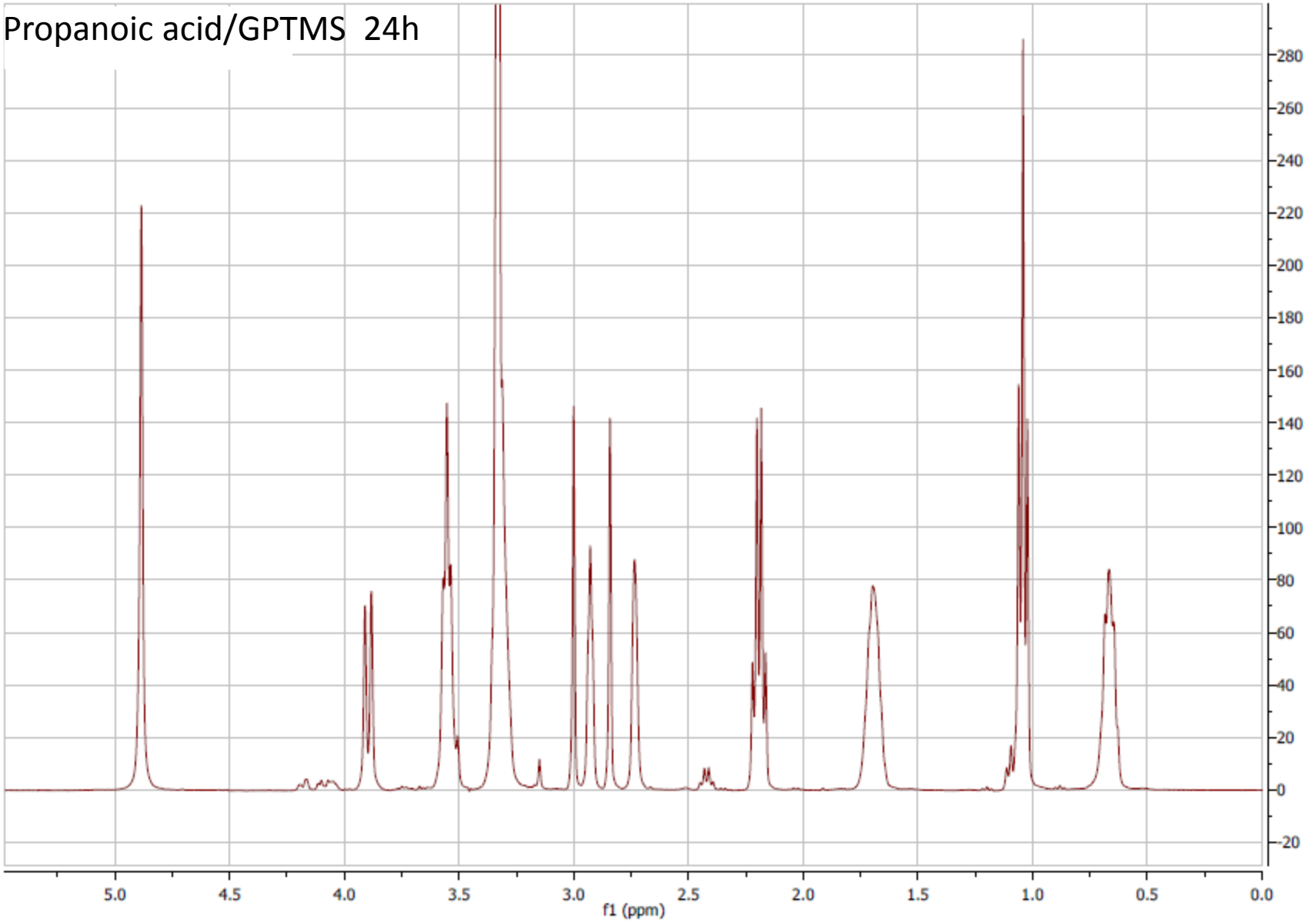
Propanoic acid reaction NMR spectra

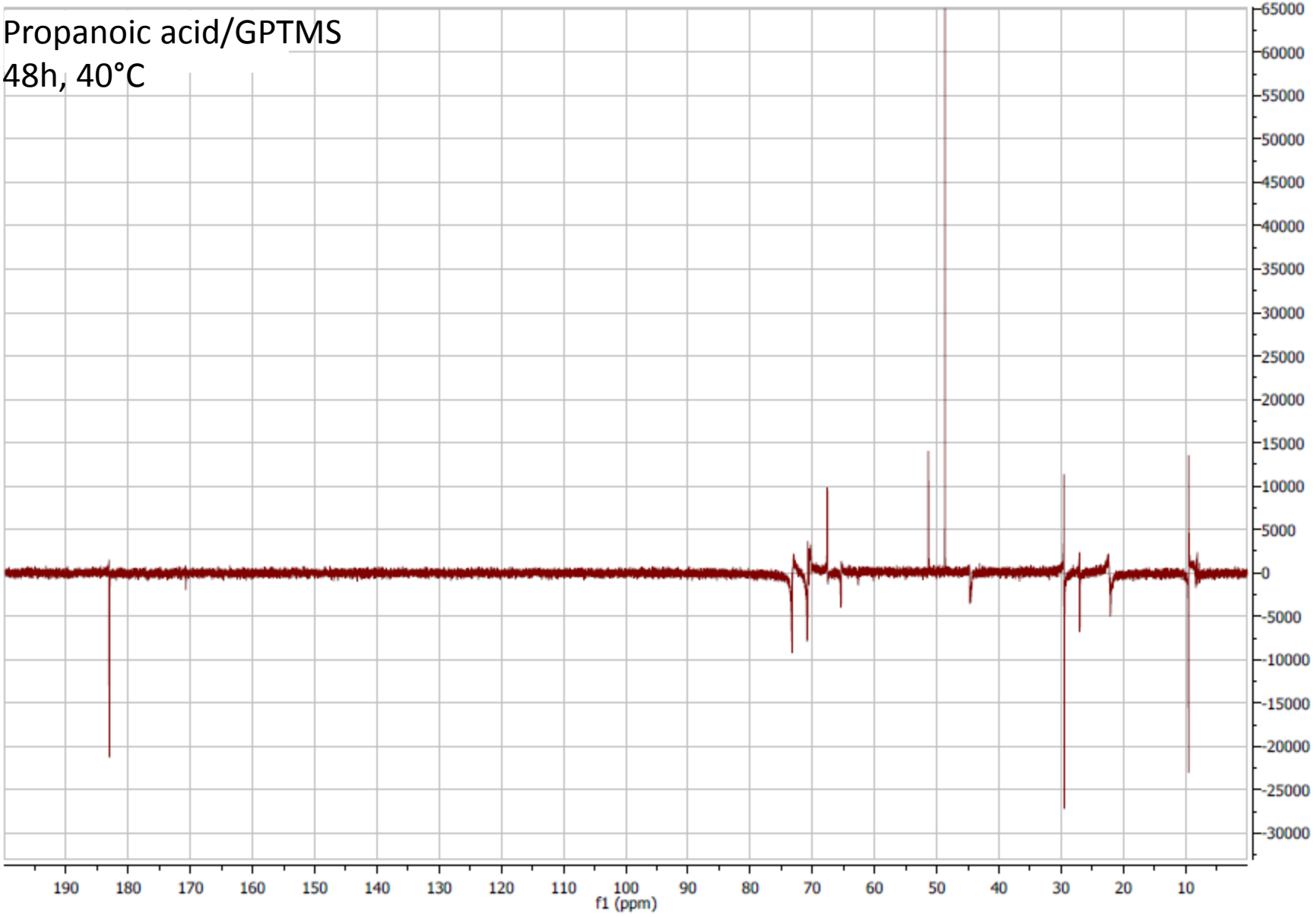




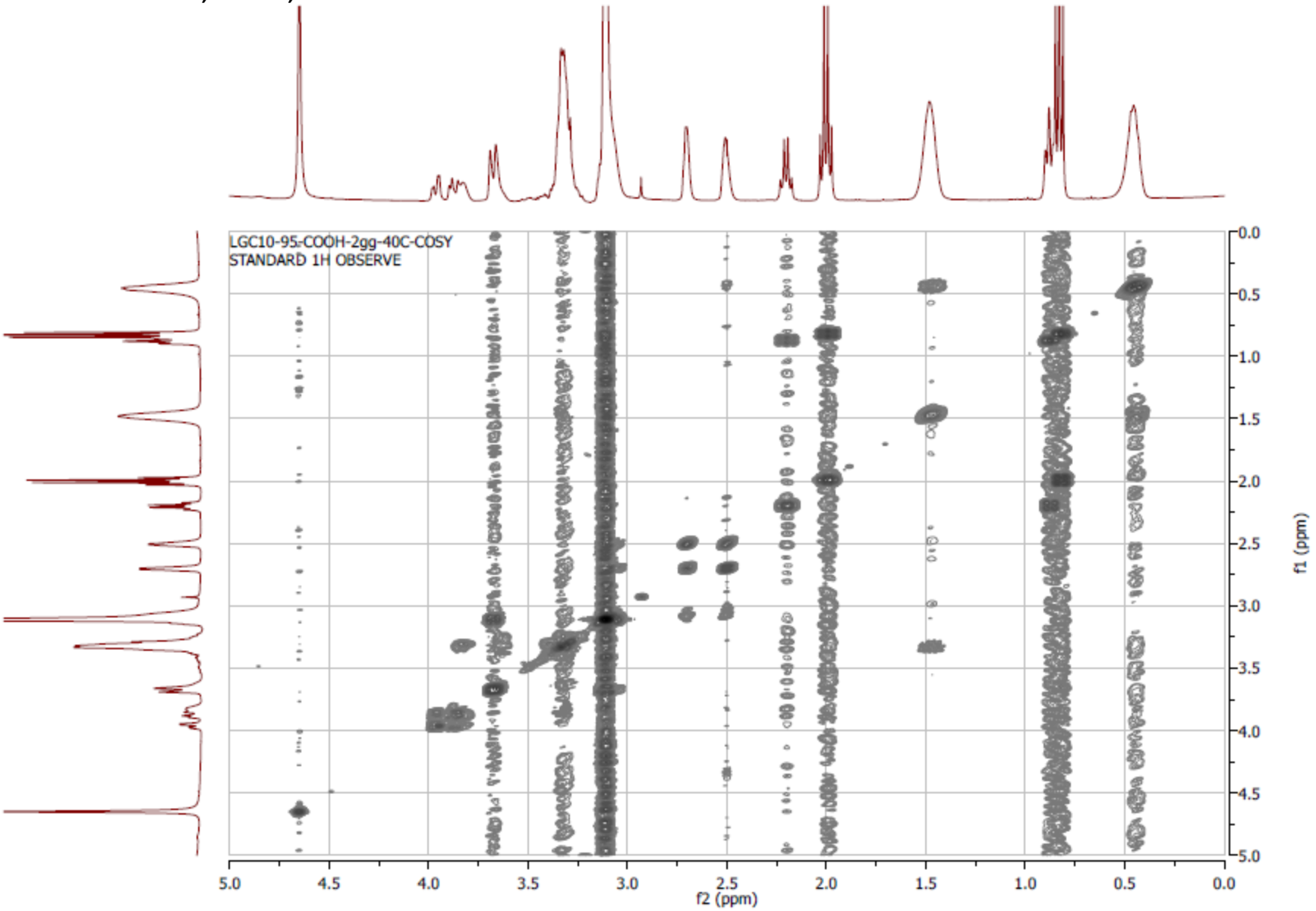




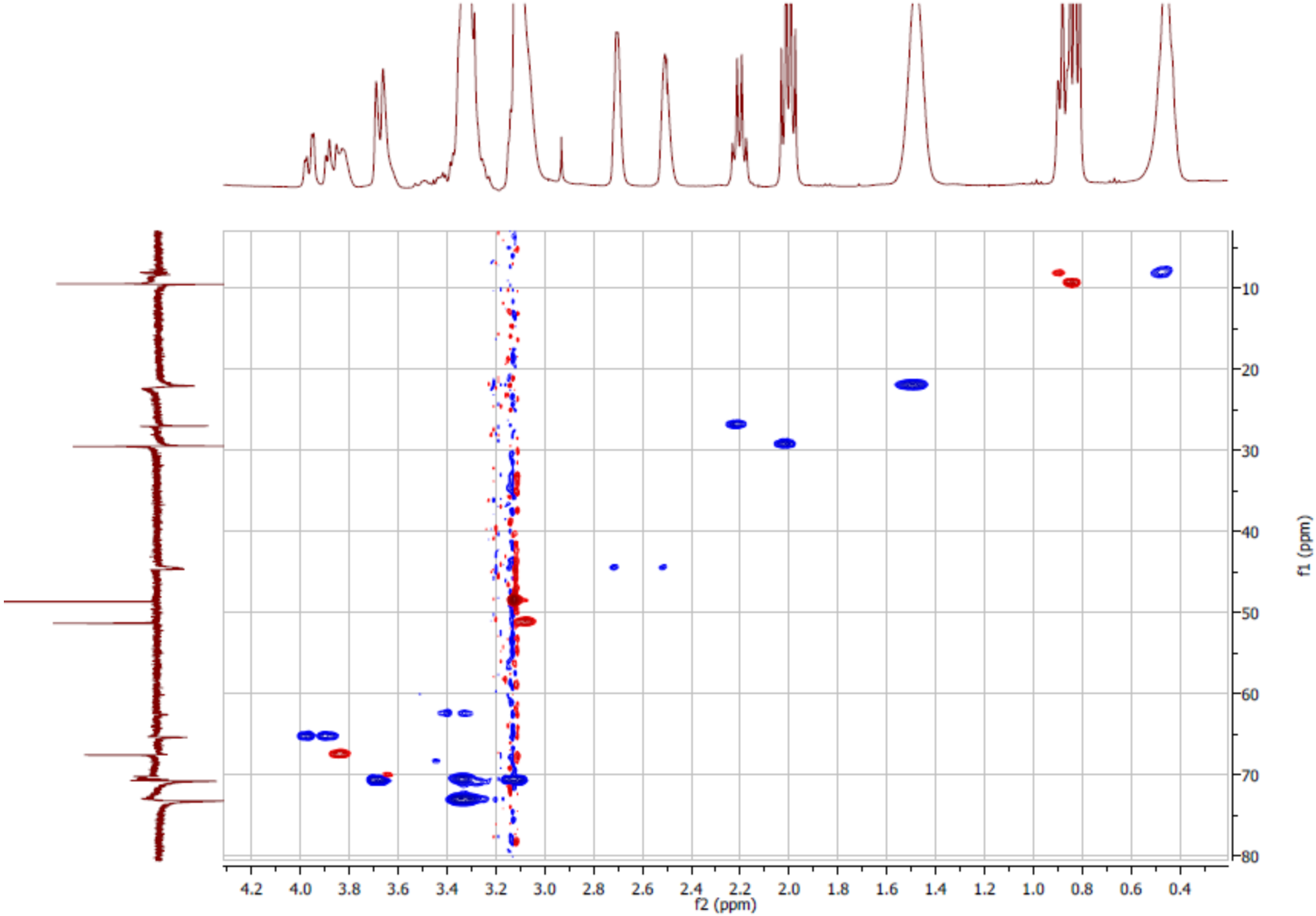




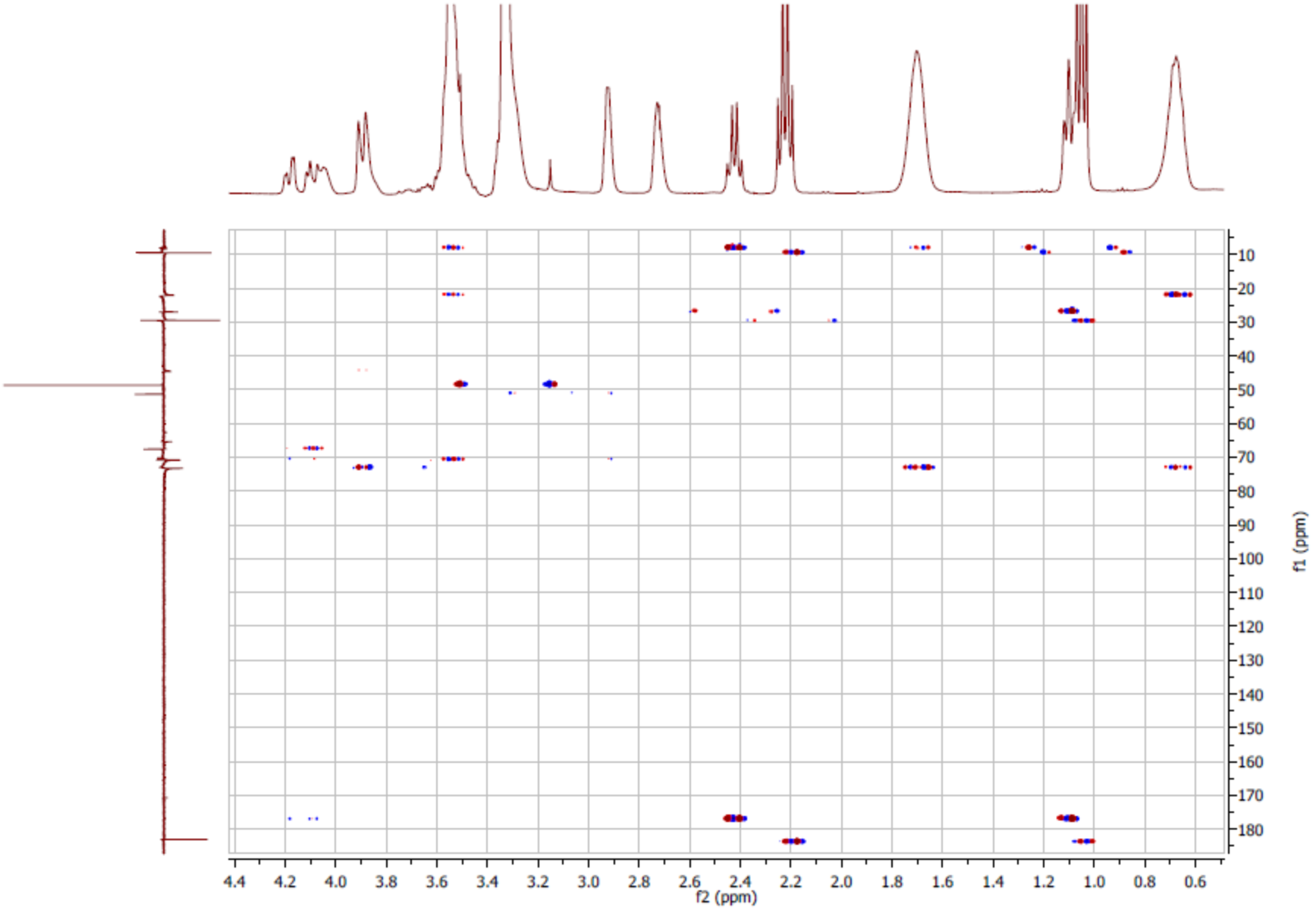
propanoic acid/GPTMS
48h, 40°C, COSY



propanoic acid/GPTMS
48h, 40°C, HSQC

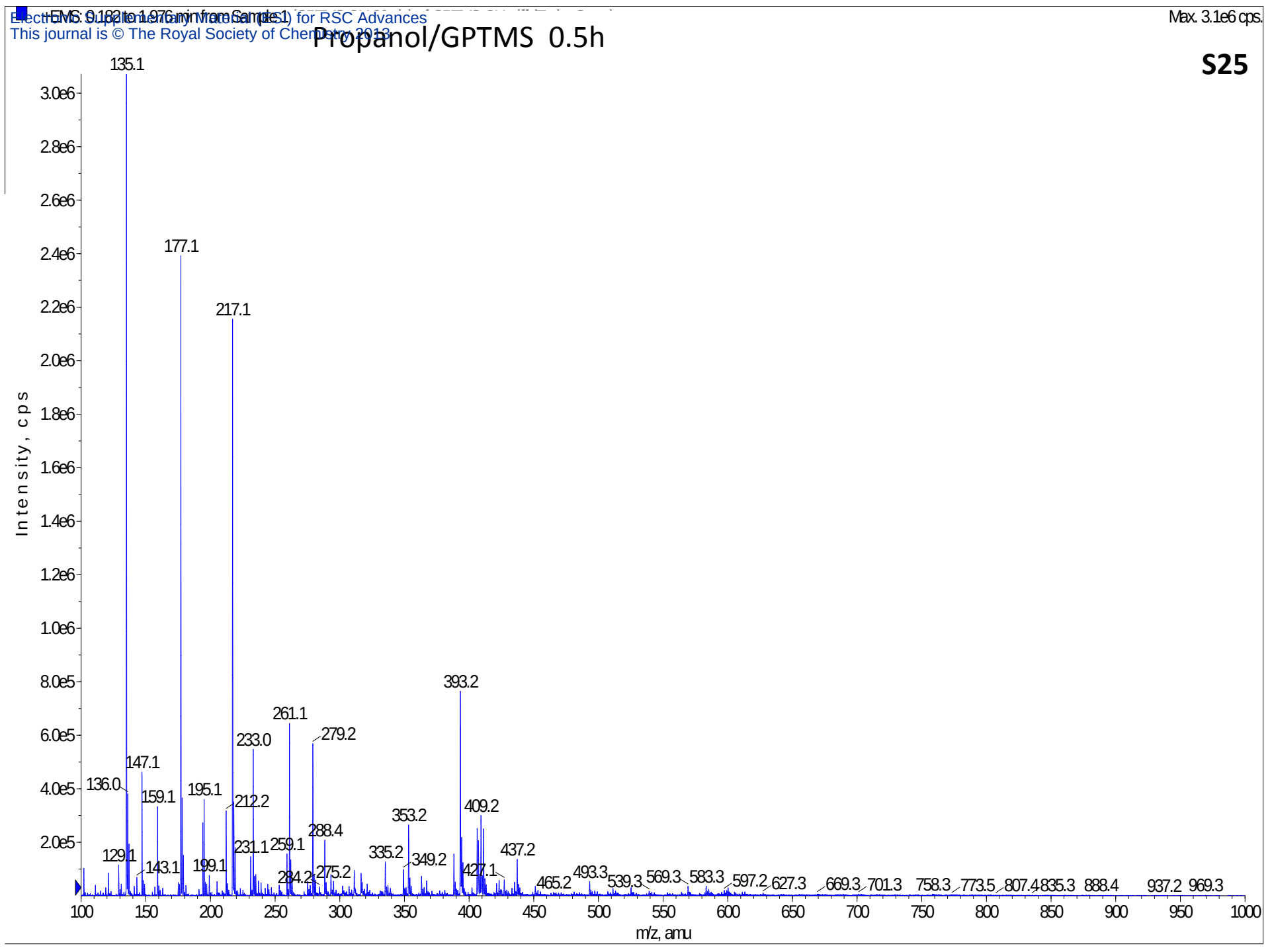


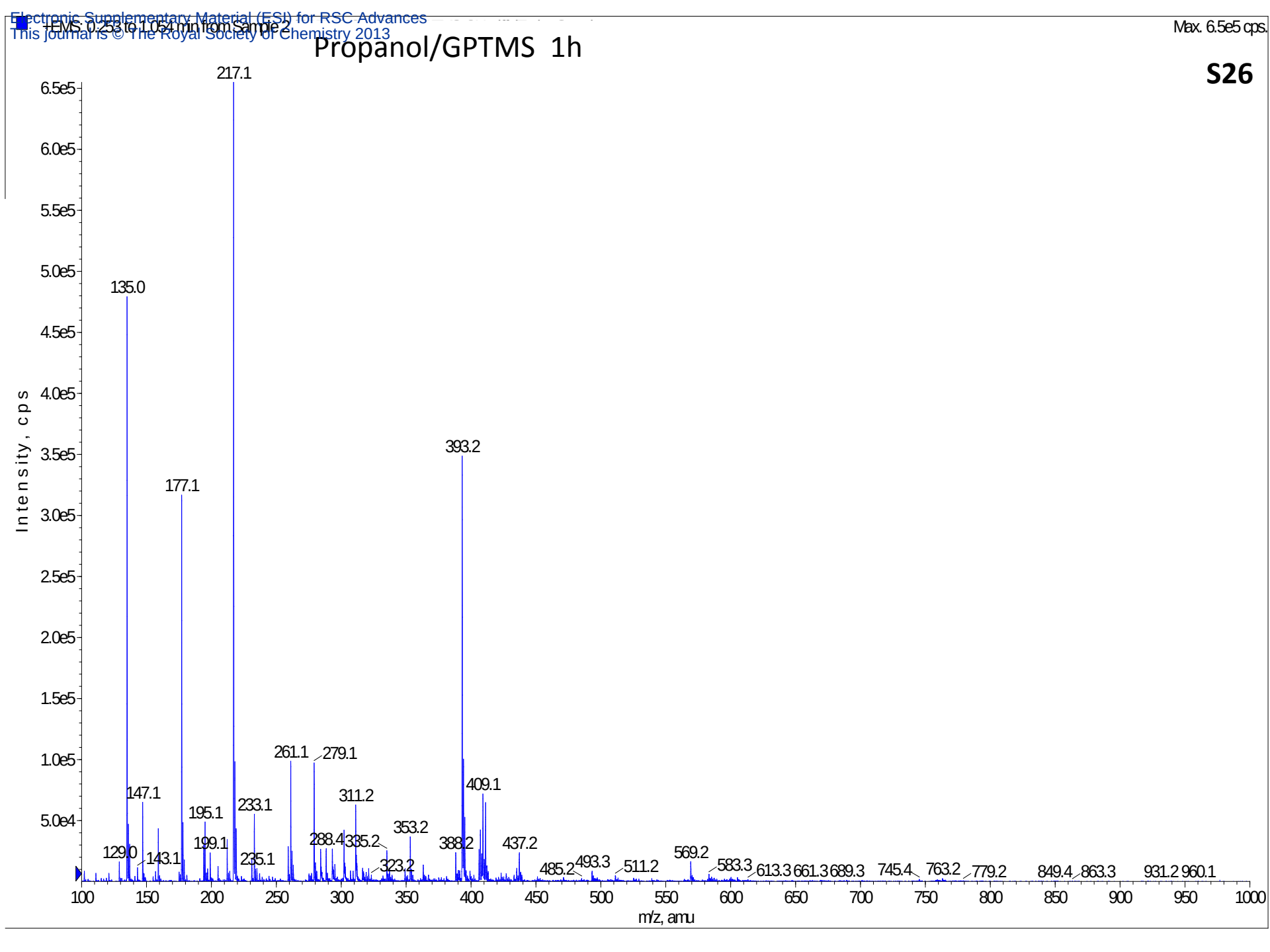
propanoic acid:GPTMS
48h, 40°C, HMBC

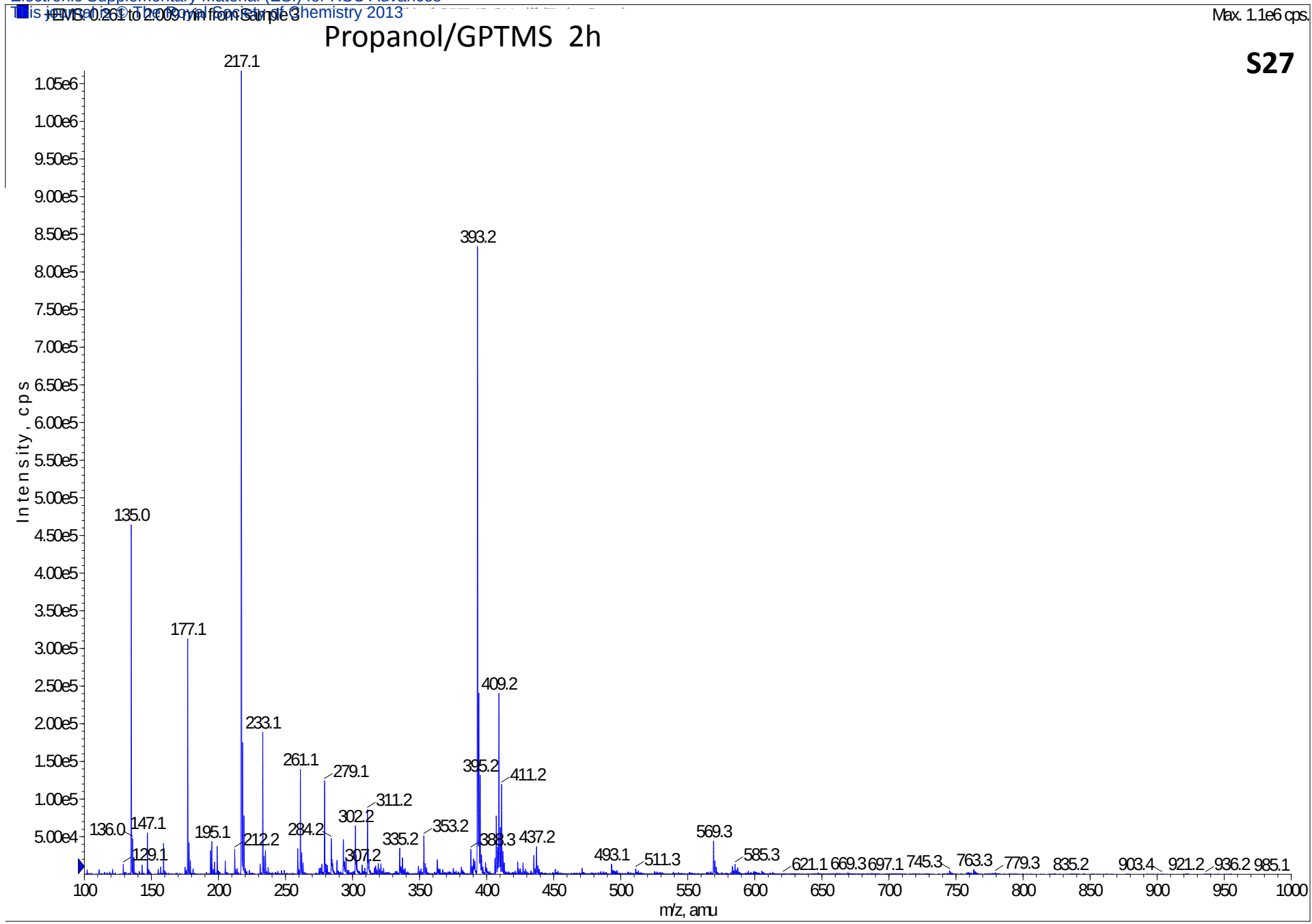


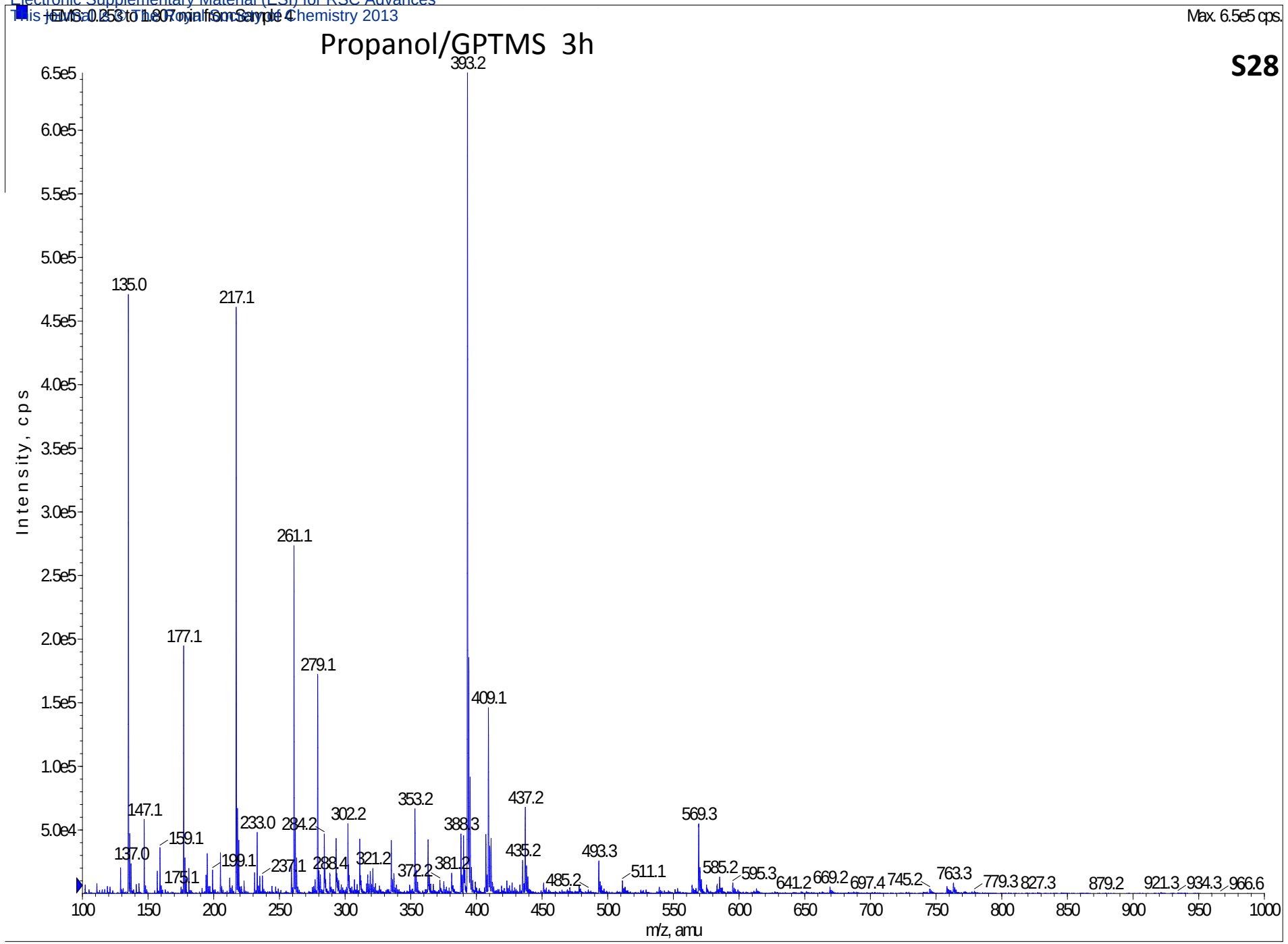
Electronic Supplementary Material (ESI) for RSC Advances

Propanol reation MS spectra








+EMS 0.253 to 1.807 min from Sample 4
Max. 6.5e5 cps.
Propanol/GPTMS 3h
S28
Intensity, cps
m/z, amu
393.2
135.0
217.1
261.1
177.1
279.1
409.1
147.1
159.1
137.0
233.0
284.2
302.2
353.2
388.3
437.2
569.3
199.1
237.1
288.4
321.2
372.2
381.2
435.2
493.3
175.1
485.2
511.1
585.2
595.3
641.2
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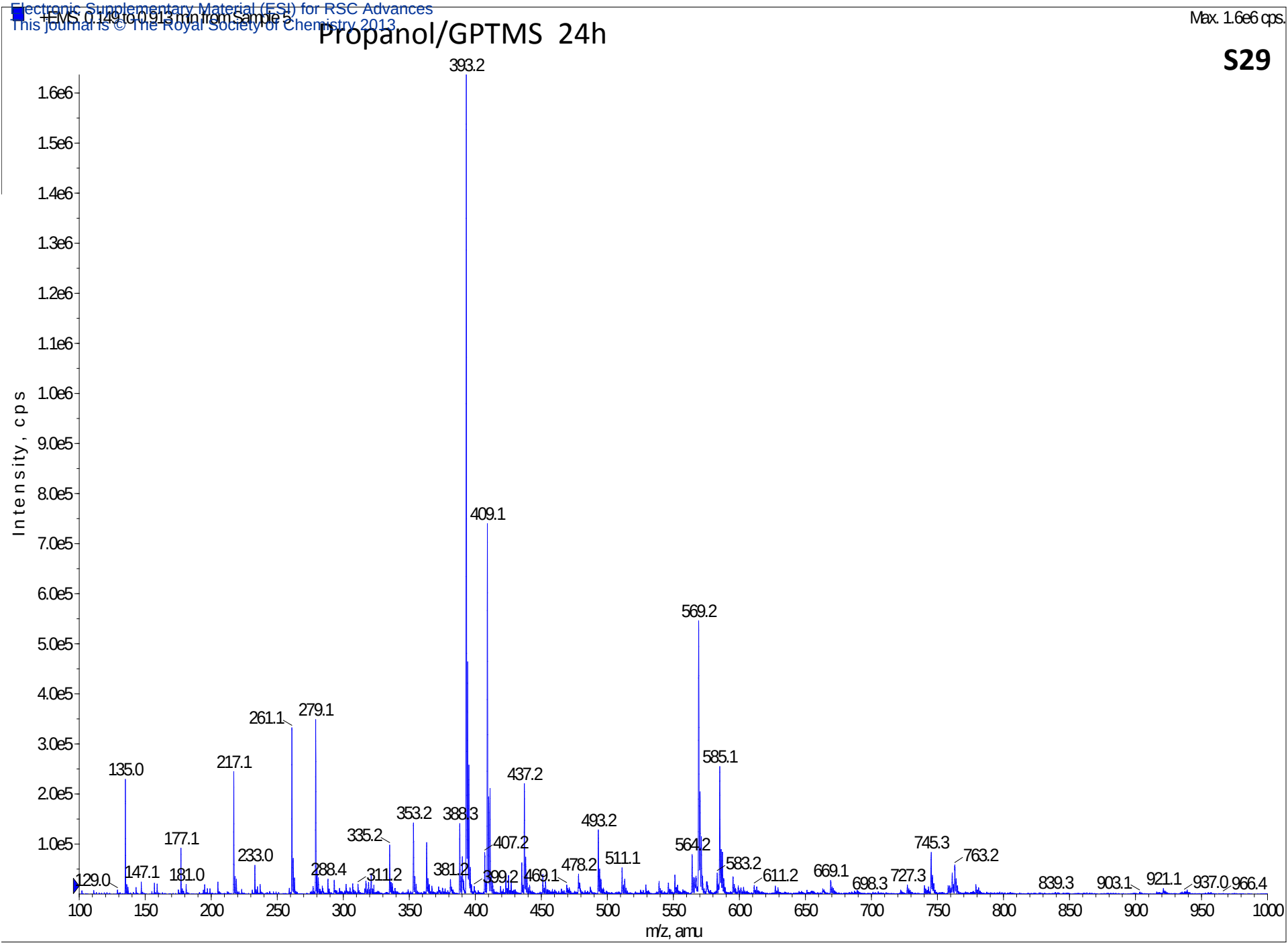
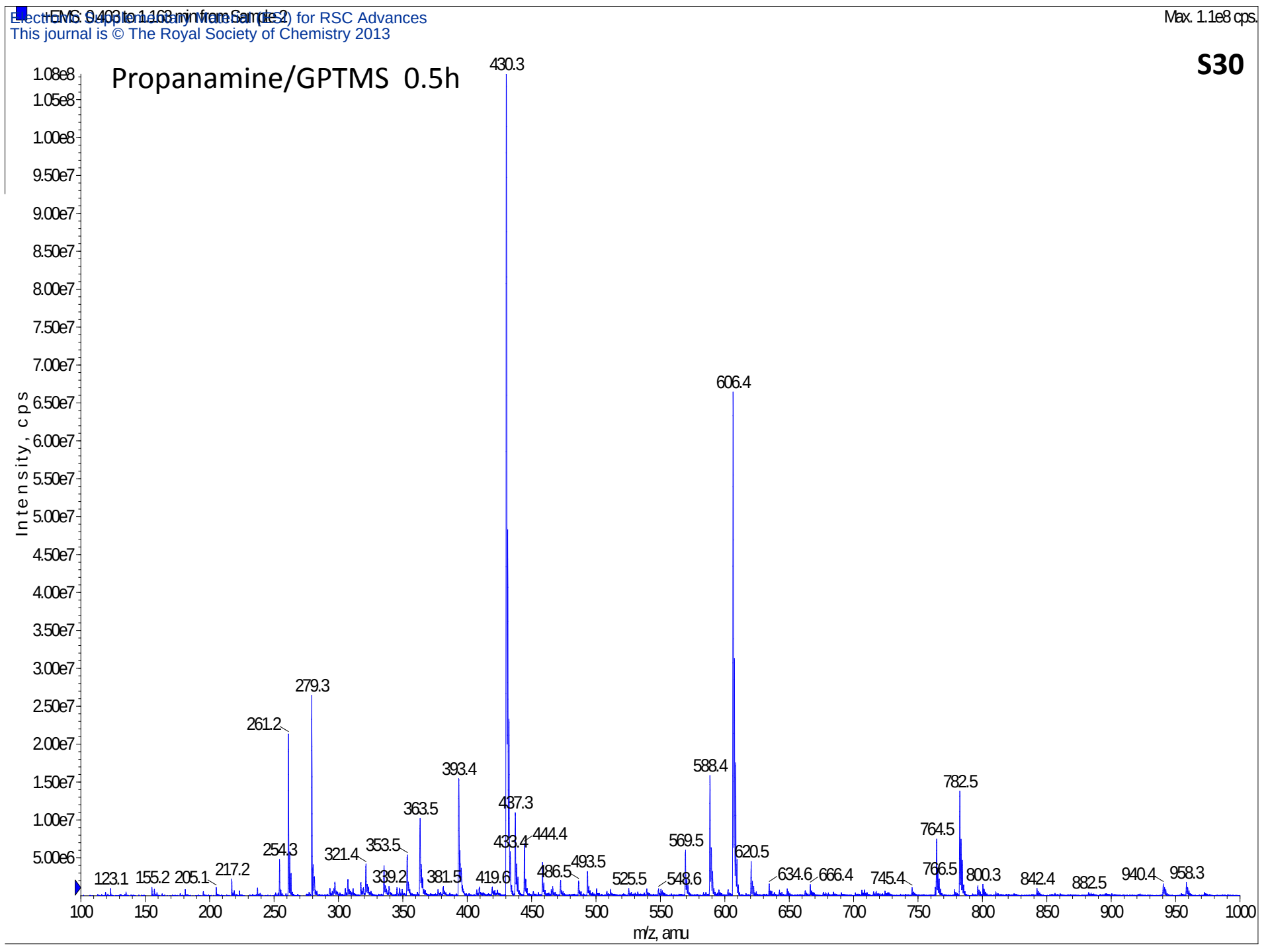
697.4
745.2
763.3
779.3
827.3
879.2
921.3
934.3
966.6



Electronic Supplementary Material (ESI) for RSC Advances

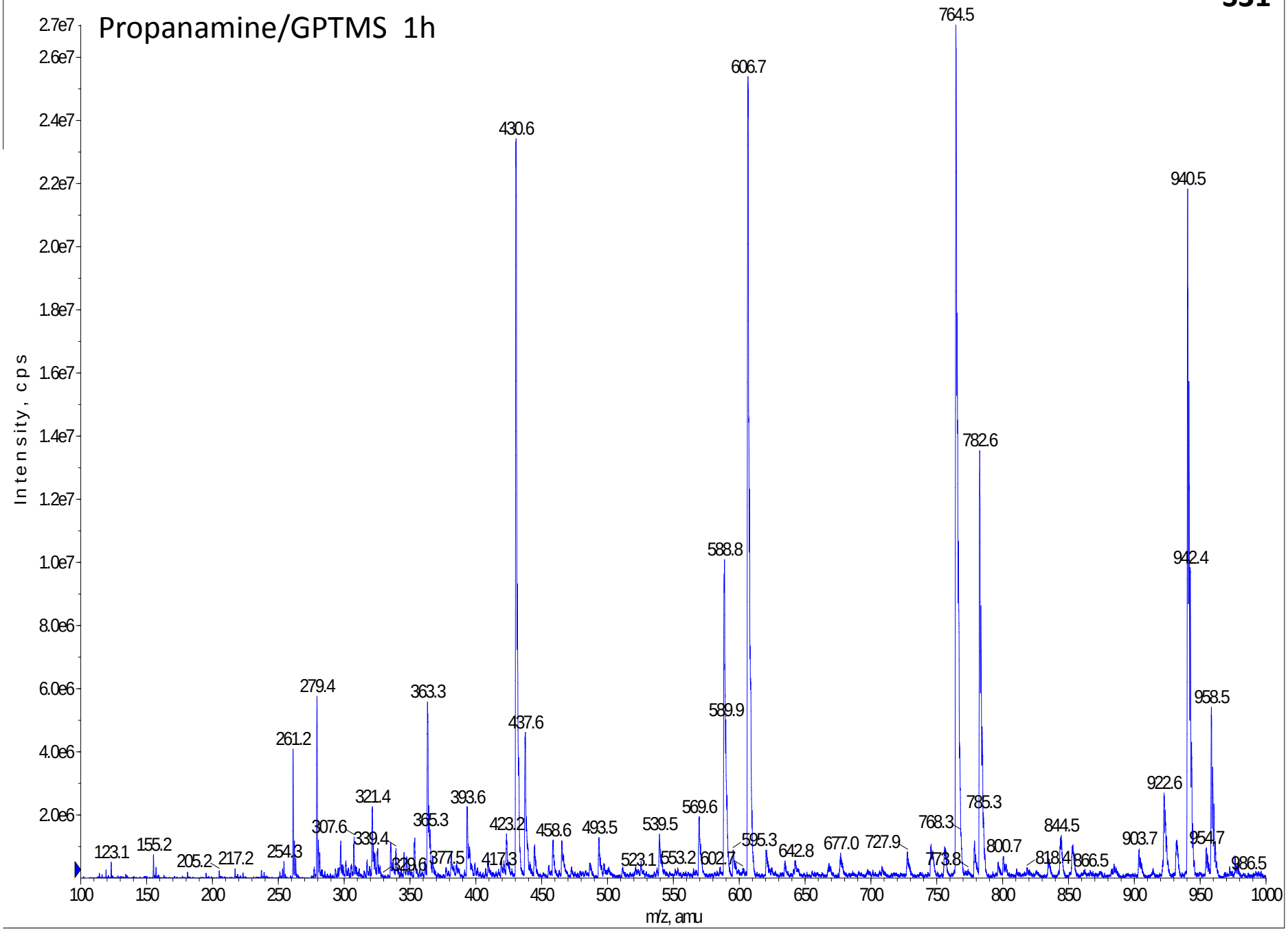
Propanamine reation MS spectra

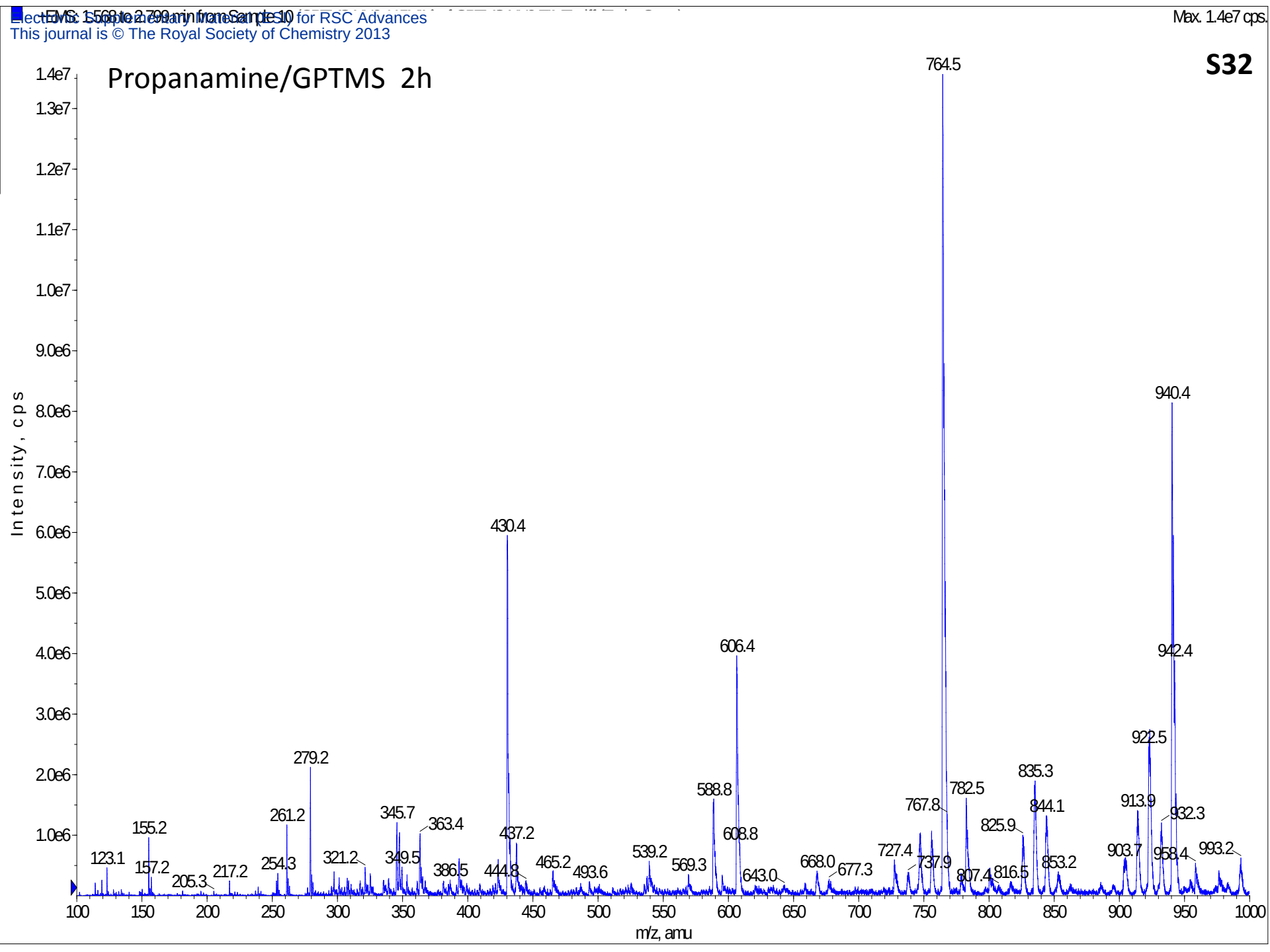


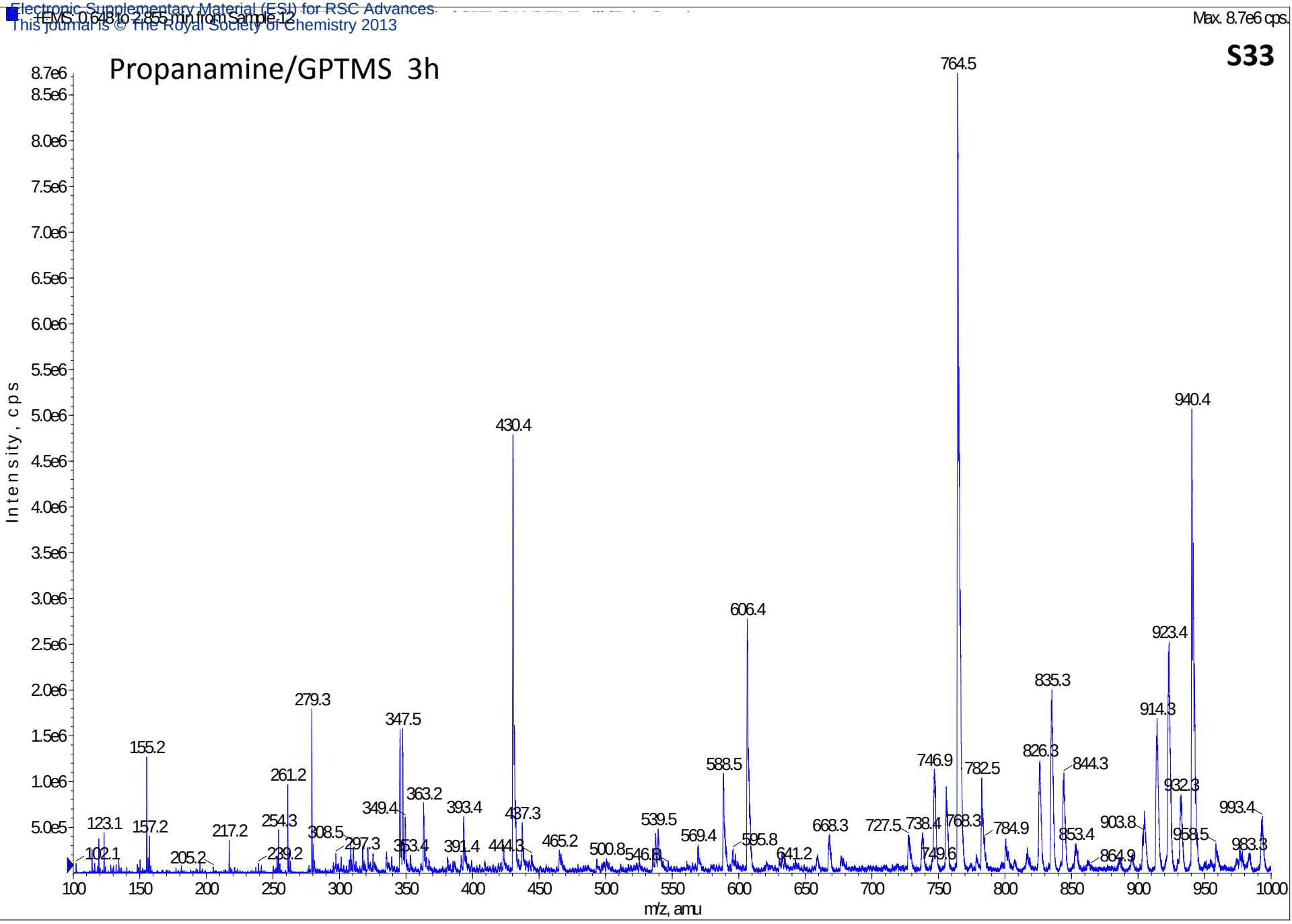
+EMS 0.504 to 1.614 min from Sample 6

Max. 2.7e7 cps.

Propanamine/GPTMS 1h

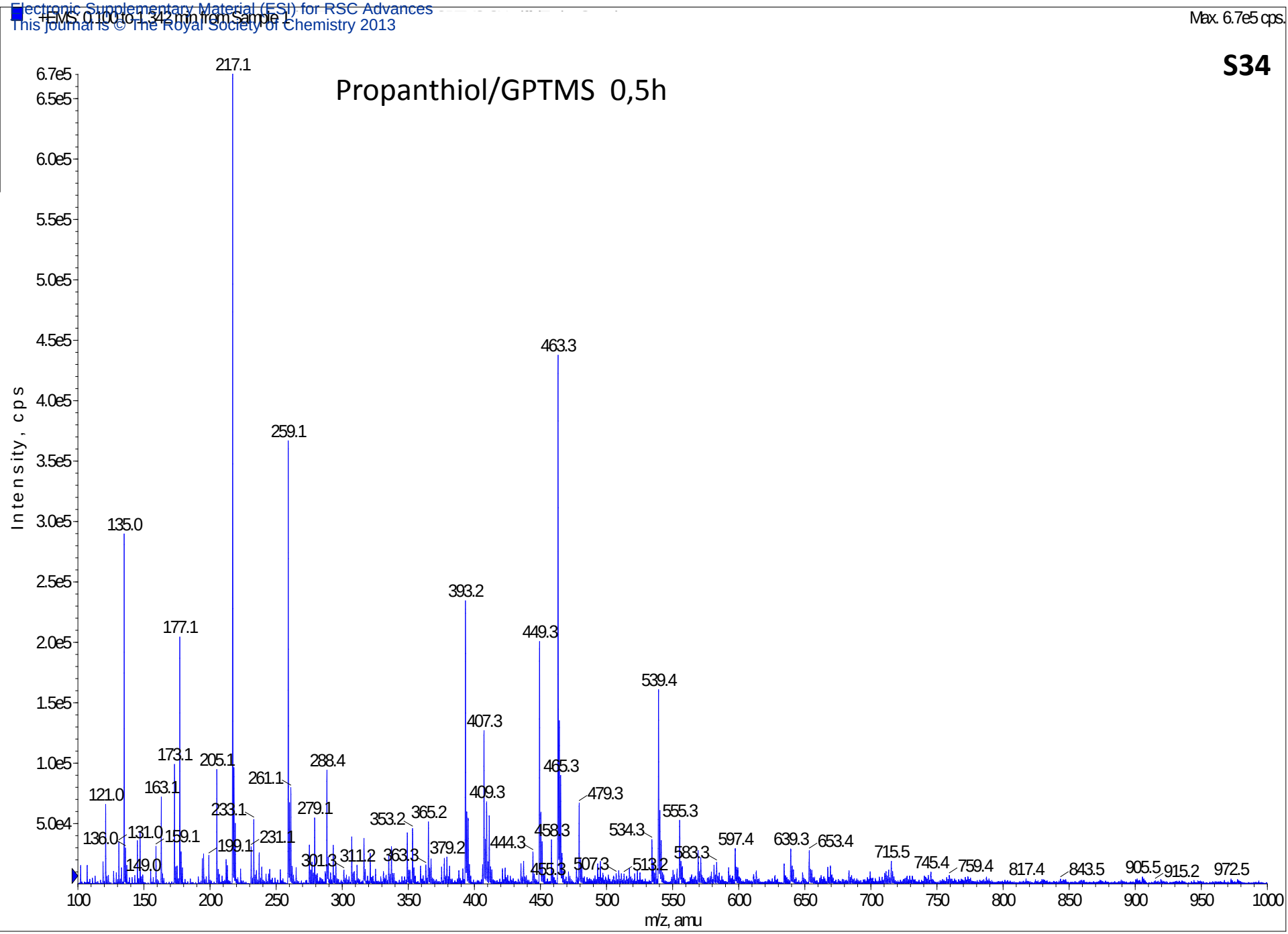






Electronic Supplementary Material (ESI) for RSC Advances

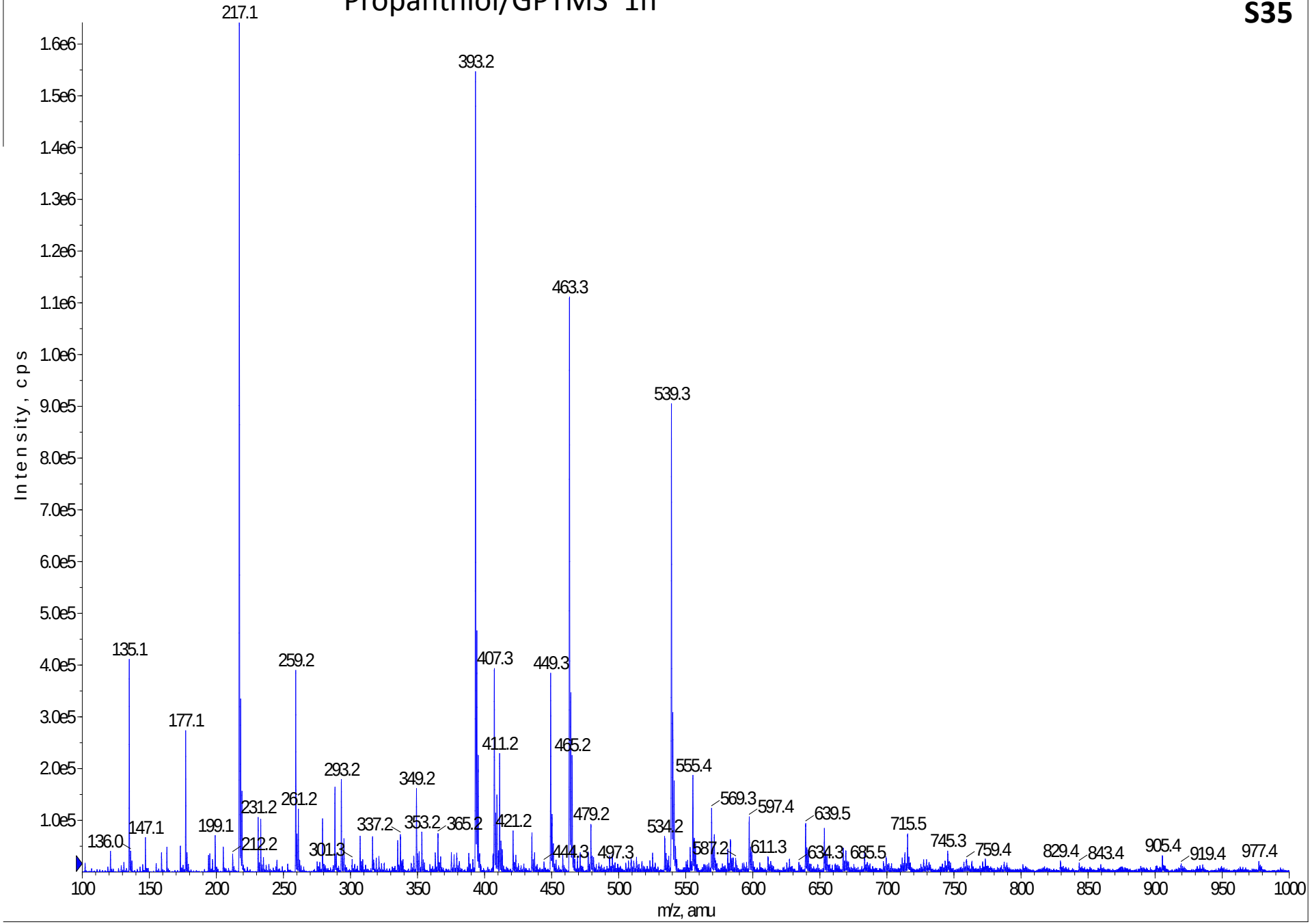
Propanthiol reation MS spectra

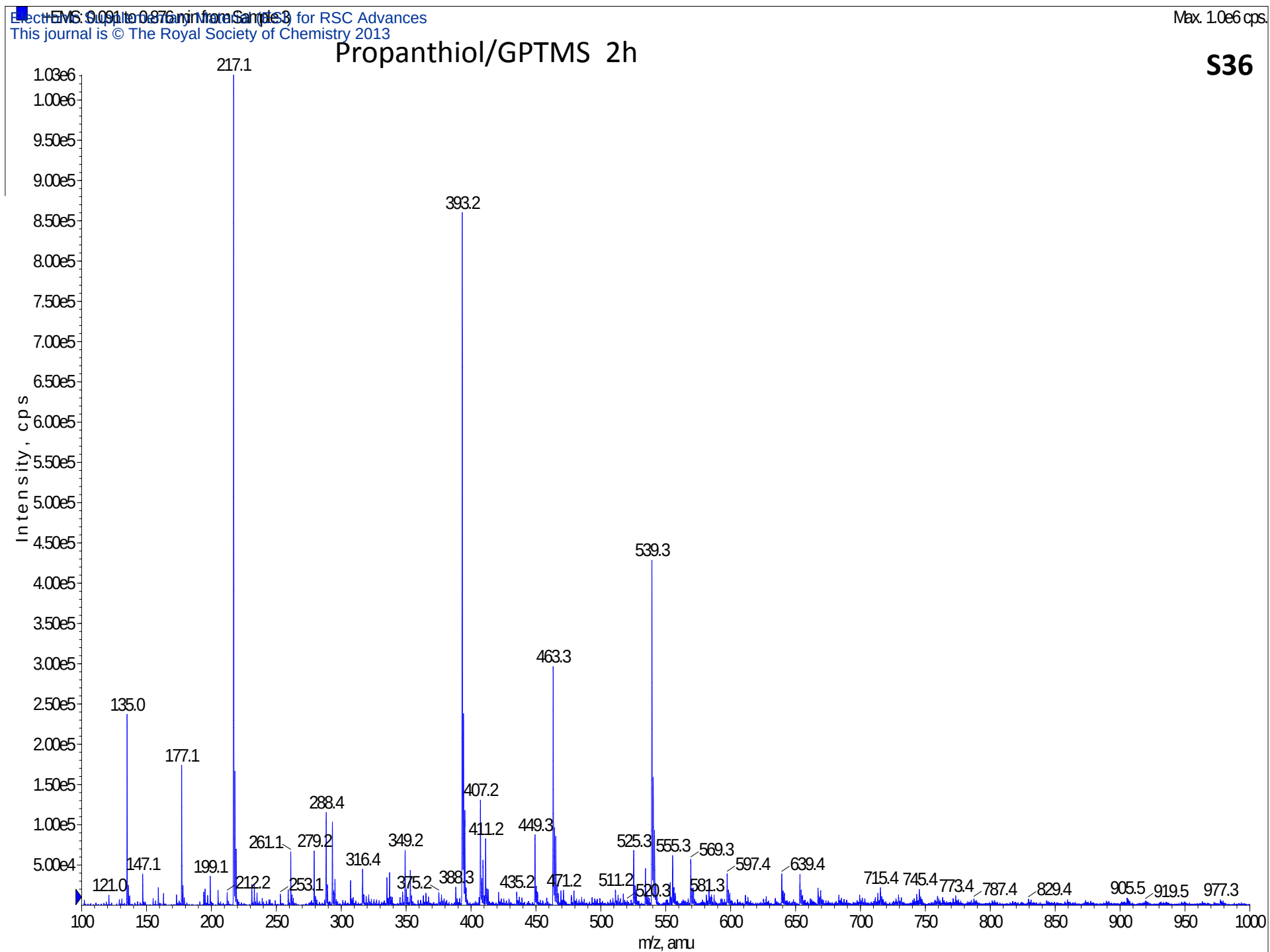


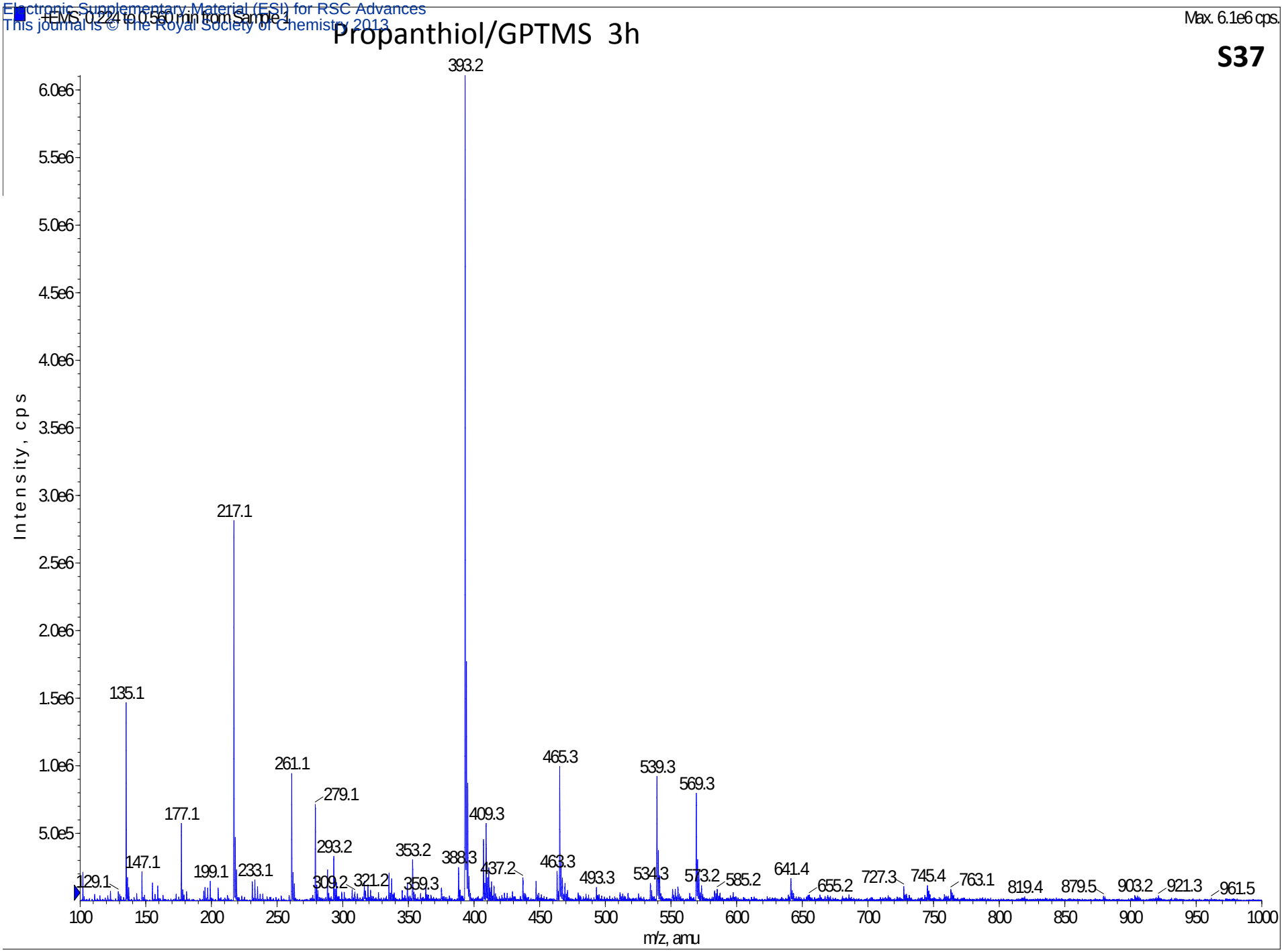
+EMS 0.437 to 1.263 min from Sample 2

Max. 1.6e6 cps.

Propanthiol/GPTMS 1h

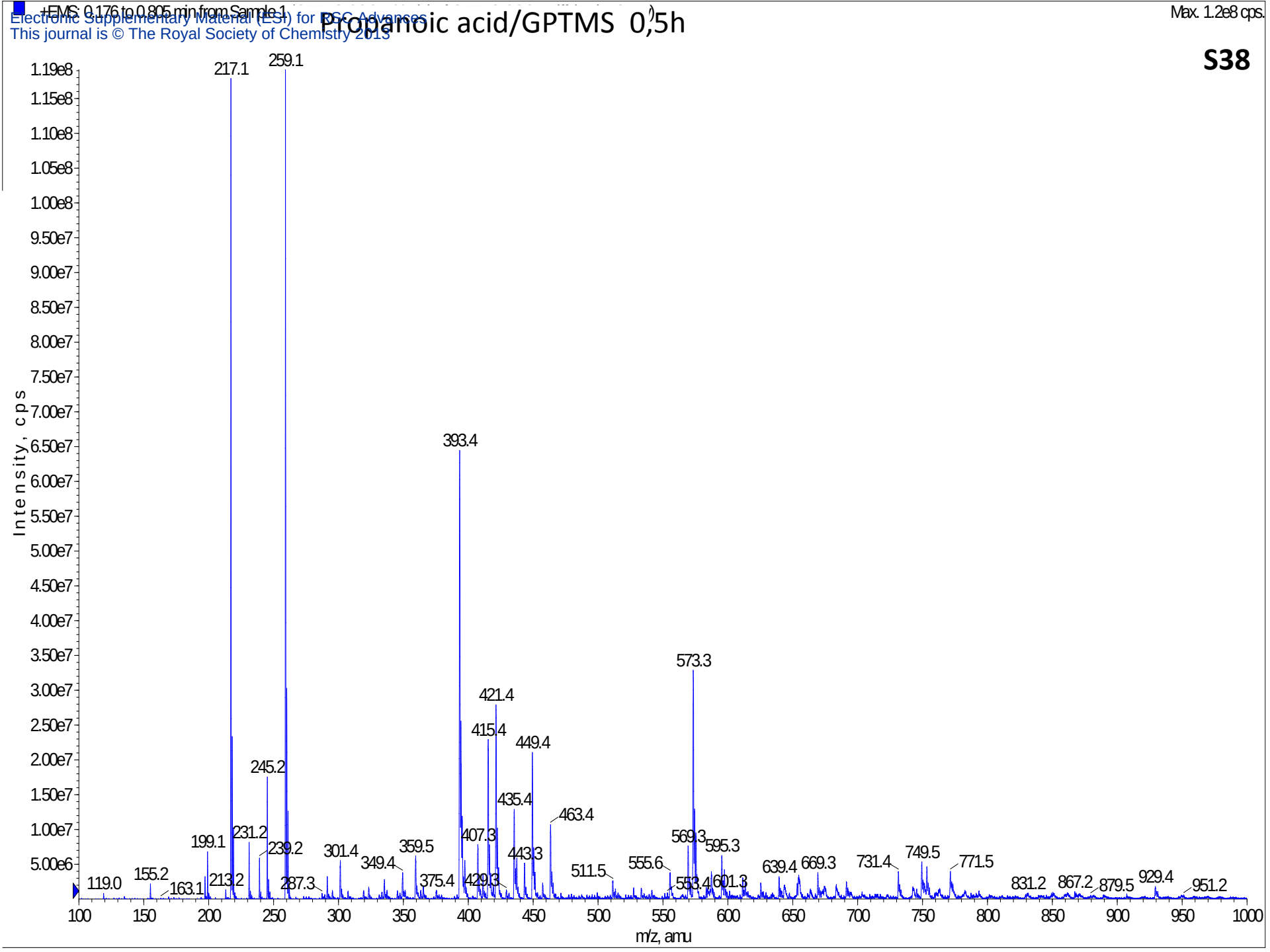


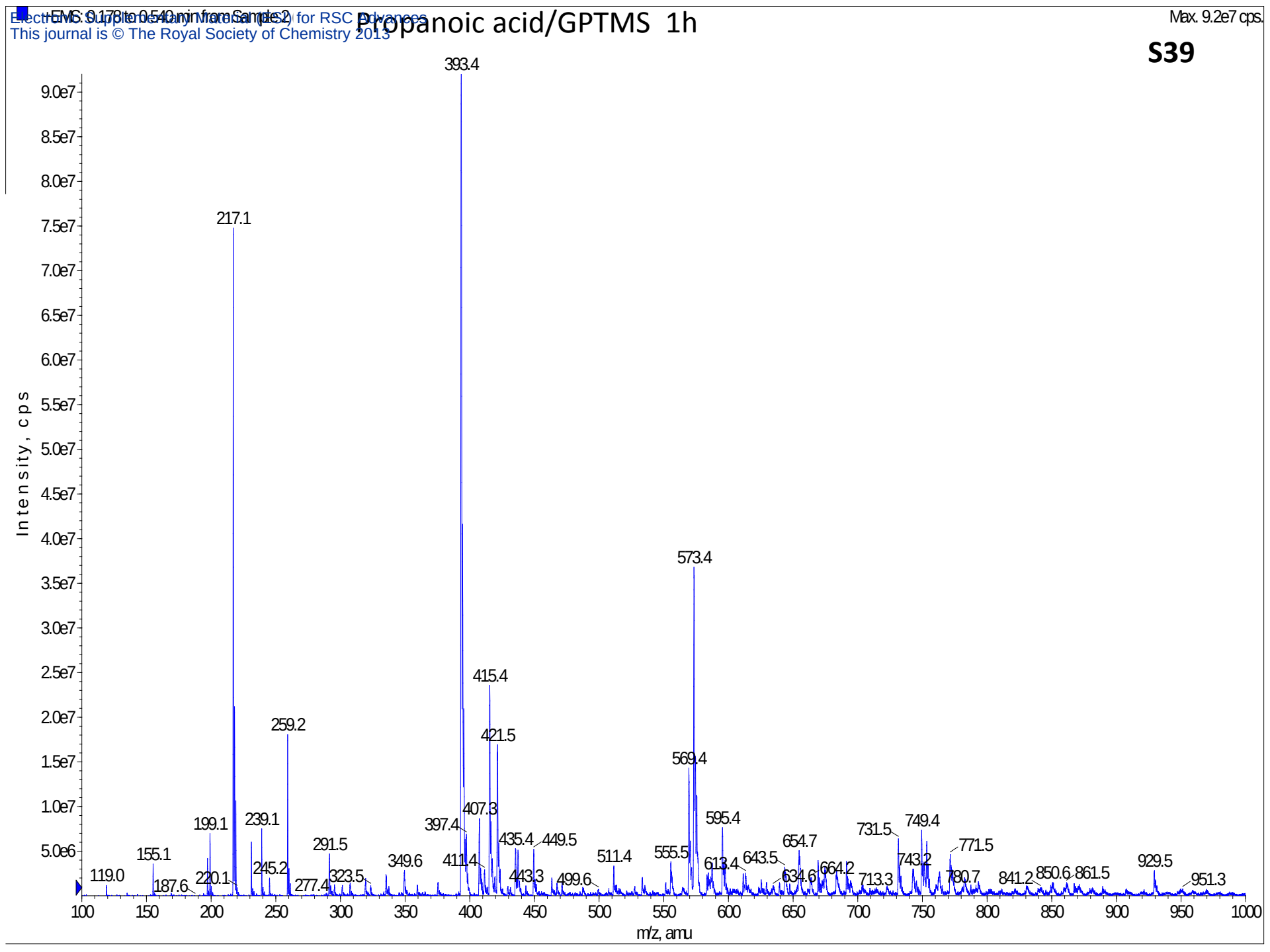


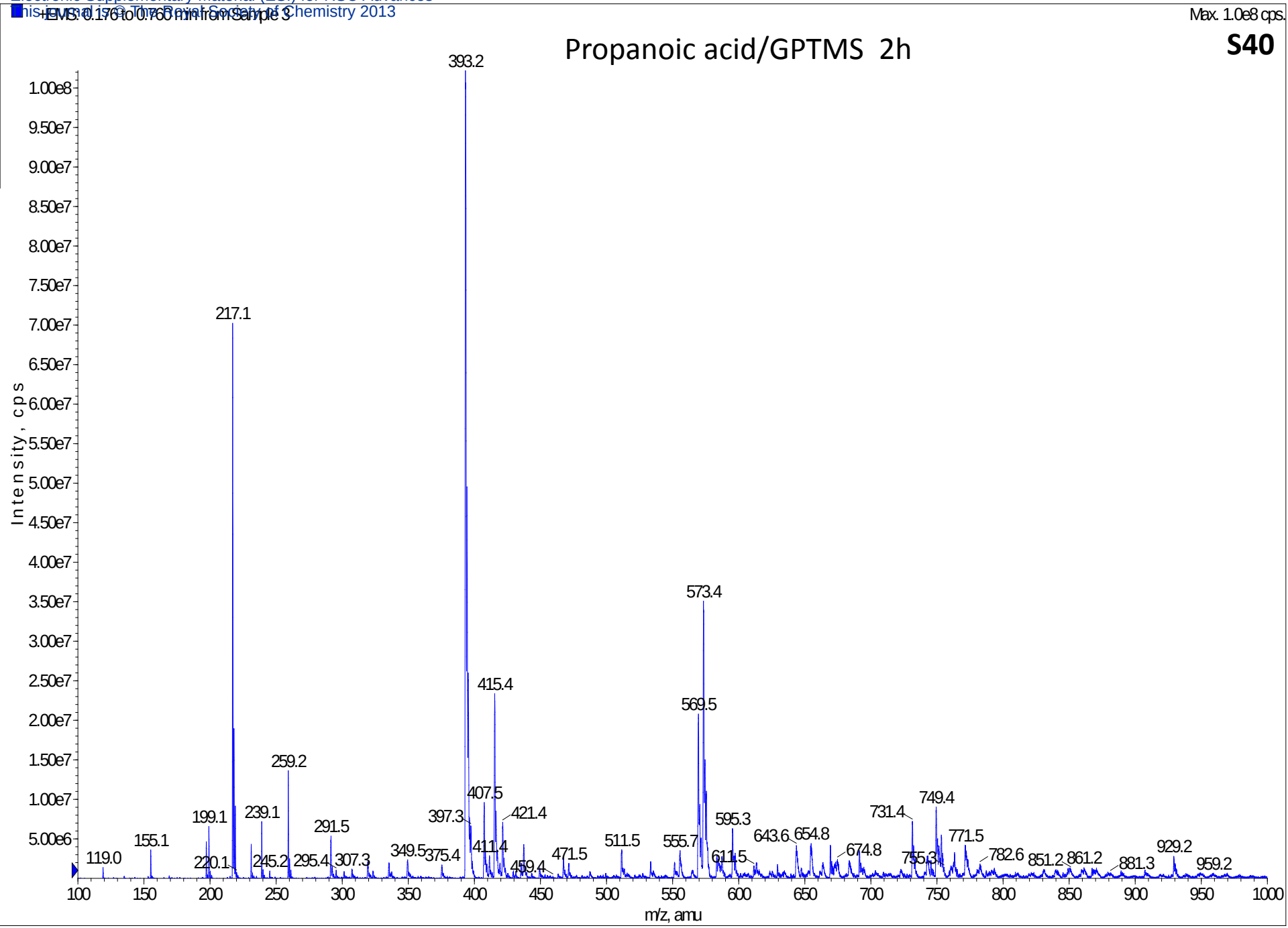


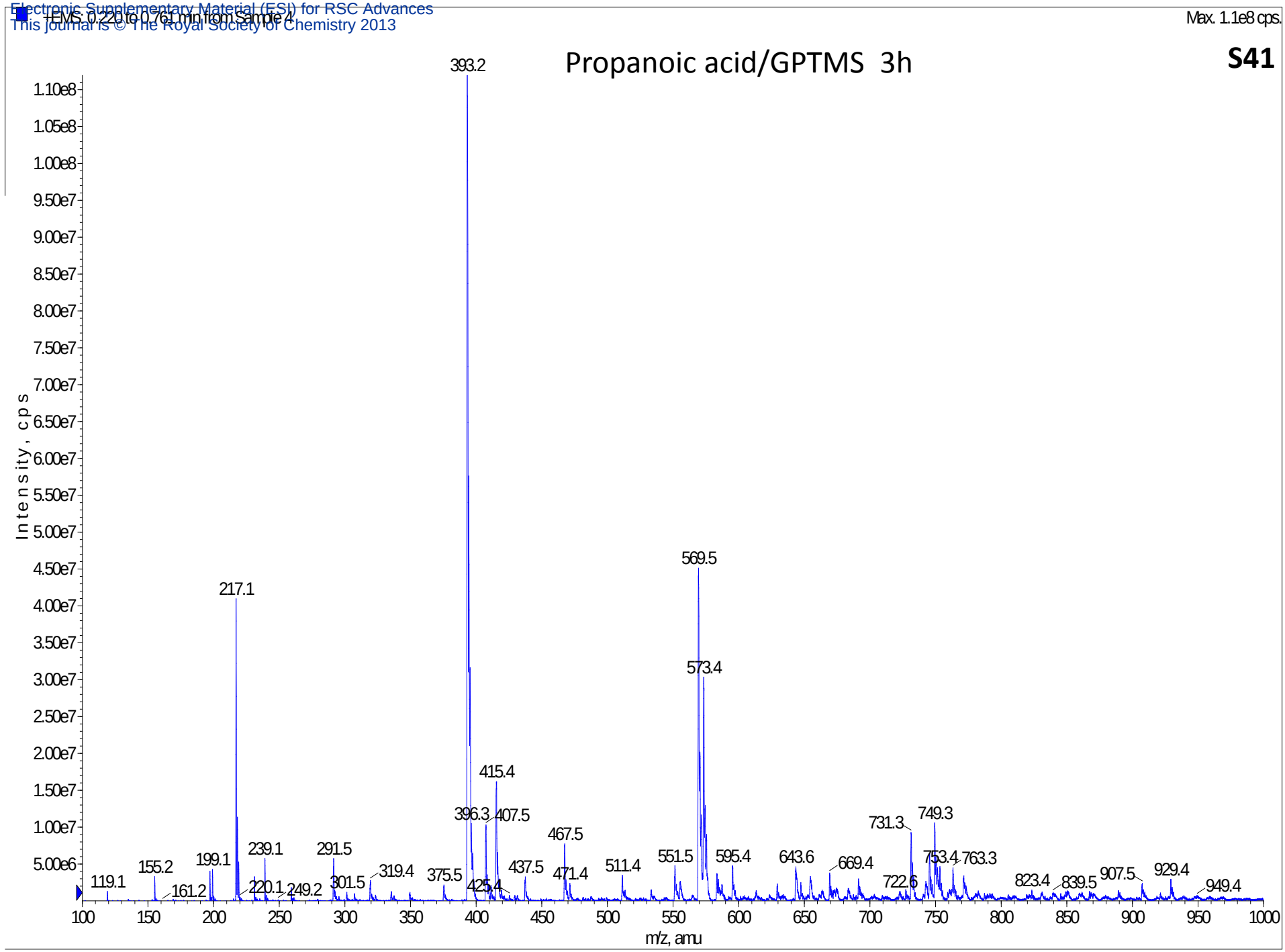
Electronic Supplementary Material (ESI) for RSC Advances

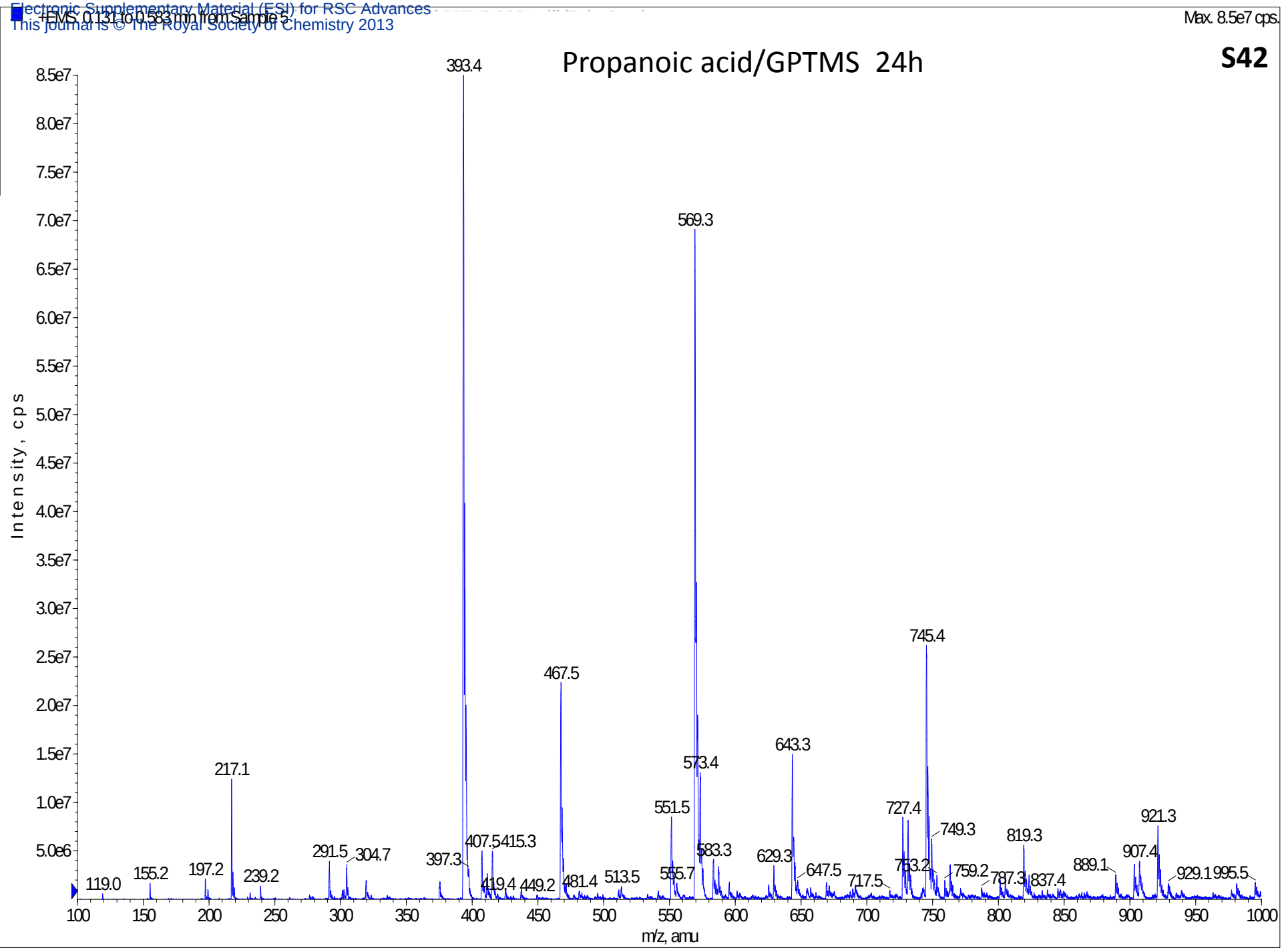
Propanoic acid reation MS spectra





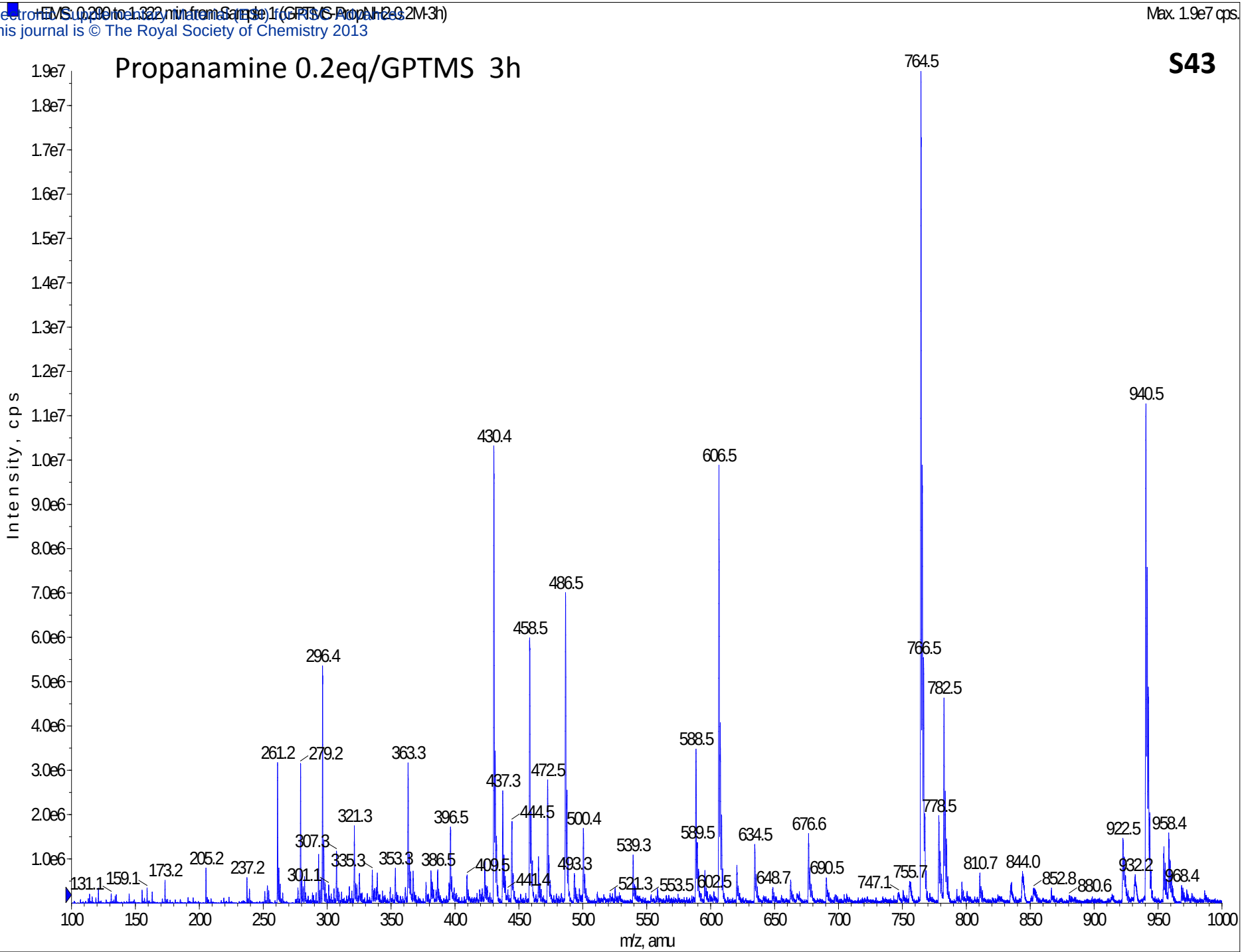


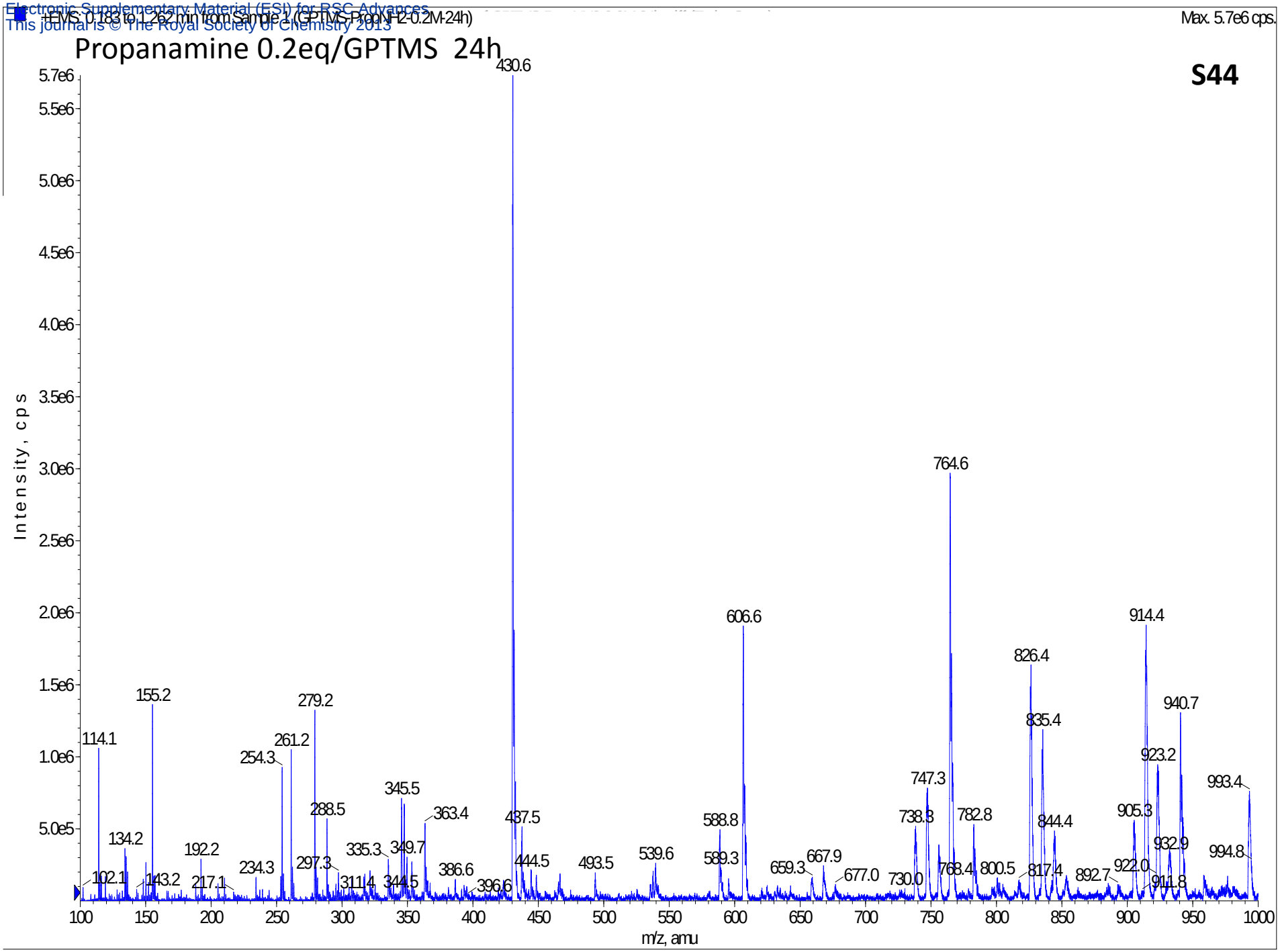




Electronic Supplementary Material (ESI) for RSC Advances

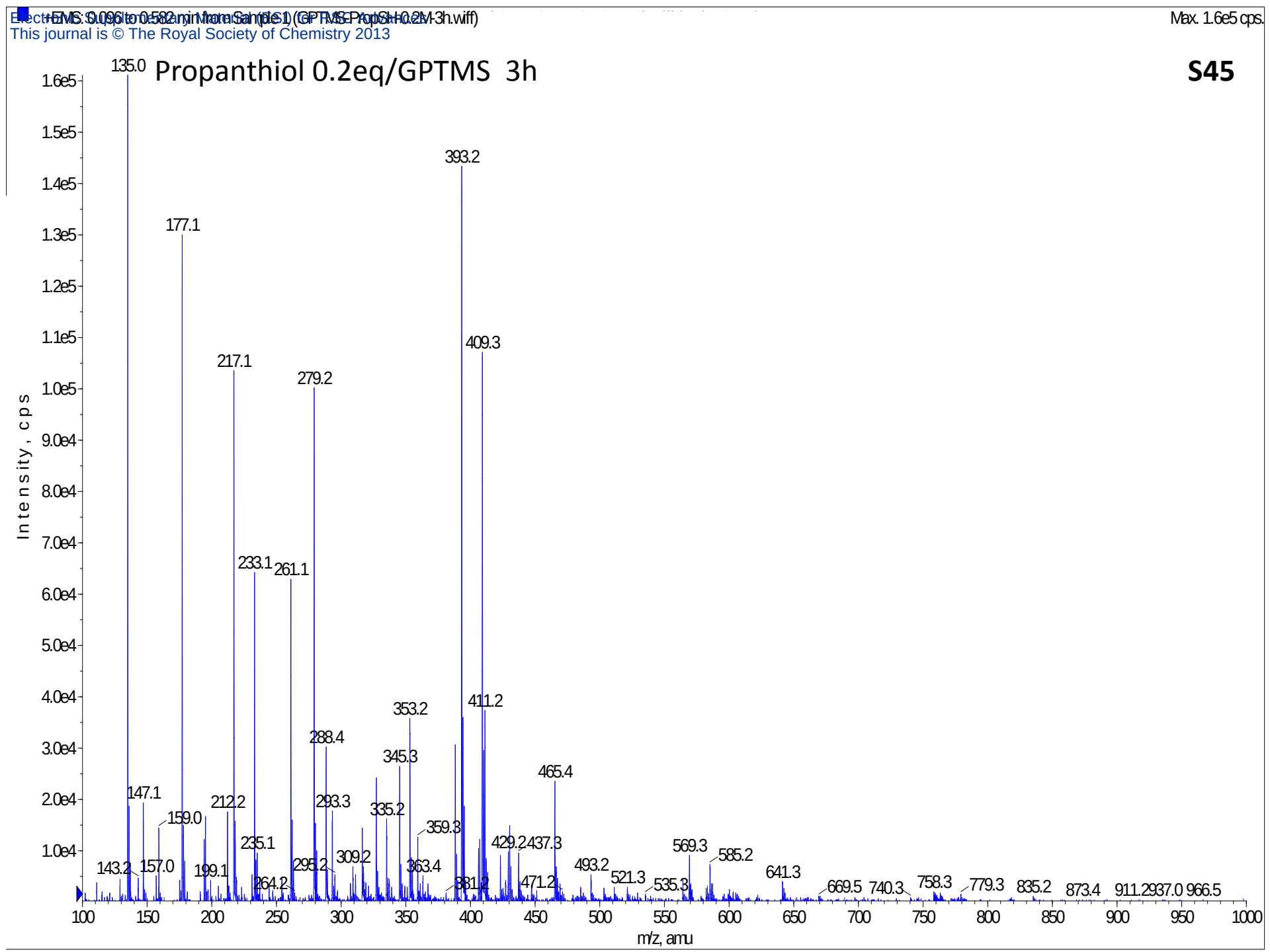
Propanamine 0.2 eq reation MS spectra

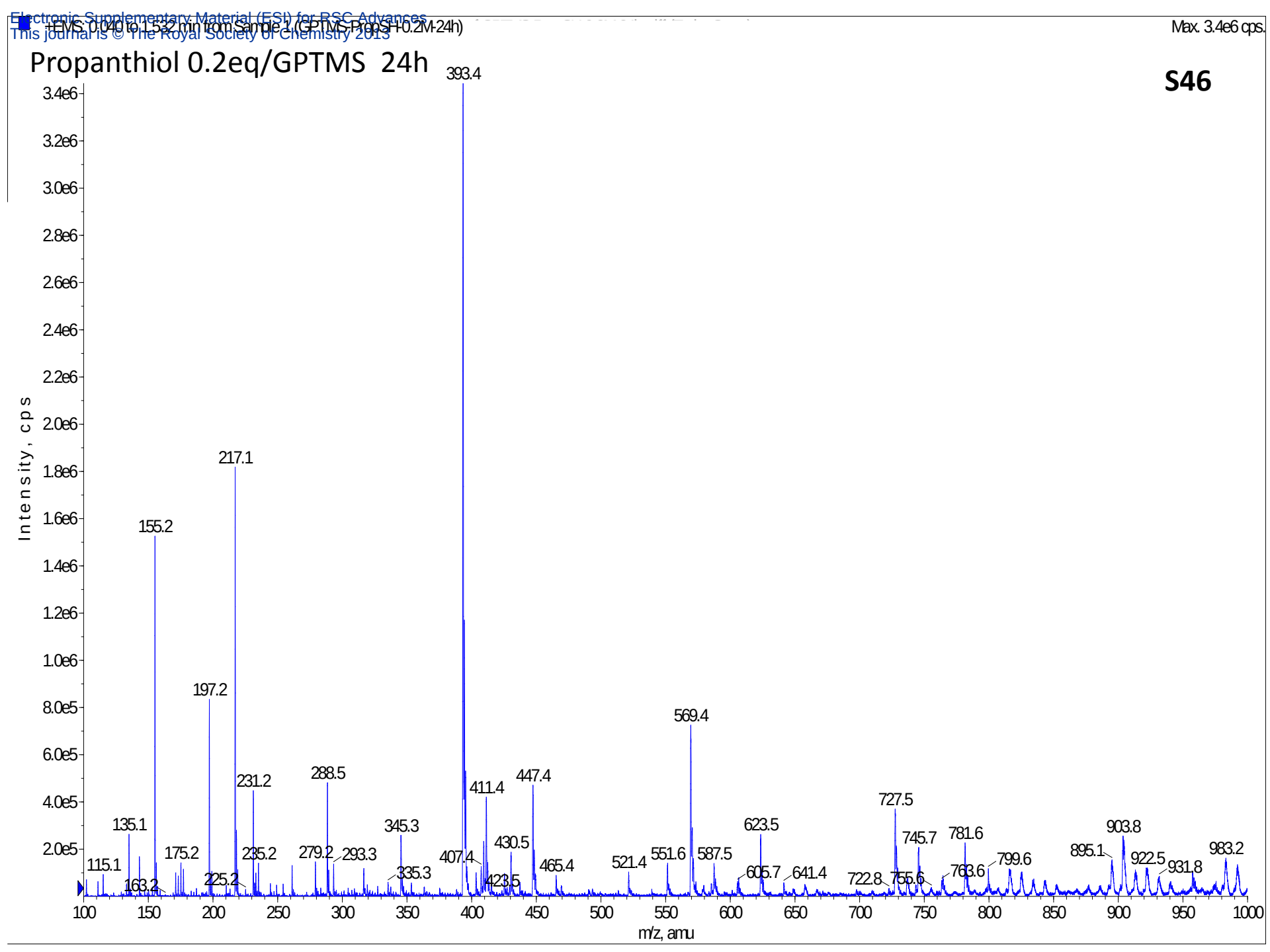




Electronic Supplementary Material (ESI) for RSC Advances

Propanthiol 0.2 eq reation MS spectra



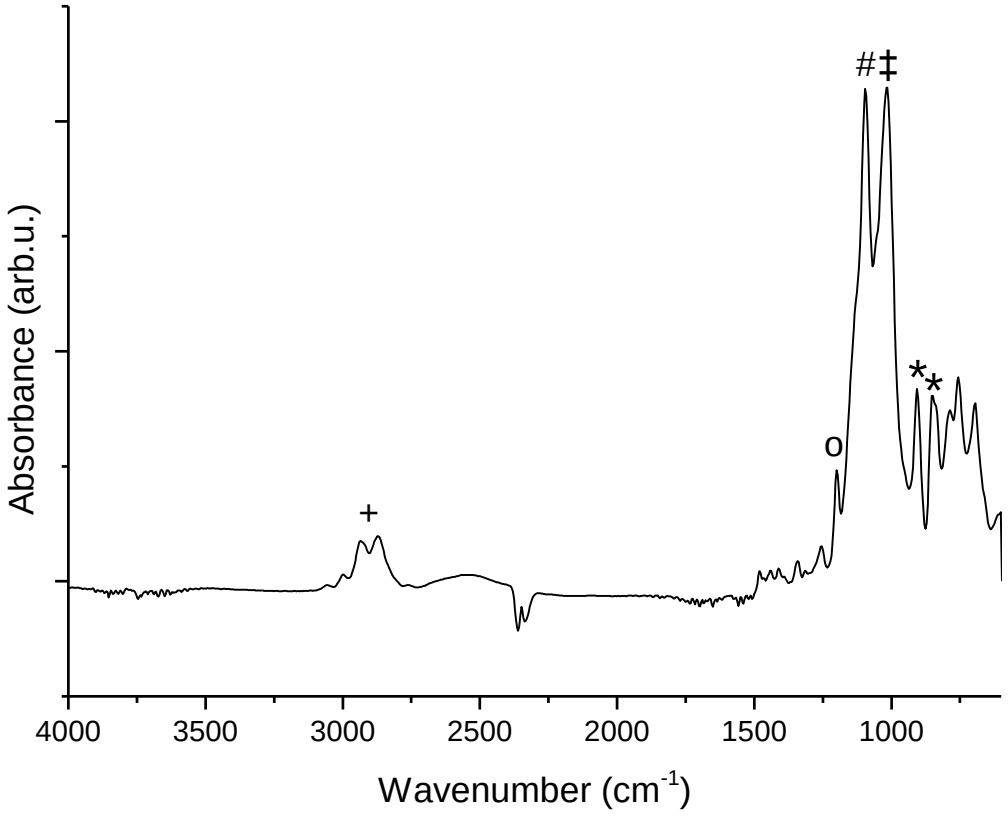


Electronic Supplementary Material (ESI) for RSC Advances

FT-IR spectra of precipitated solids

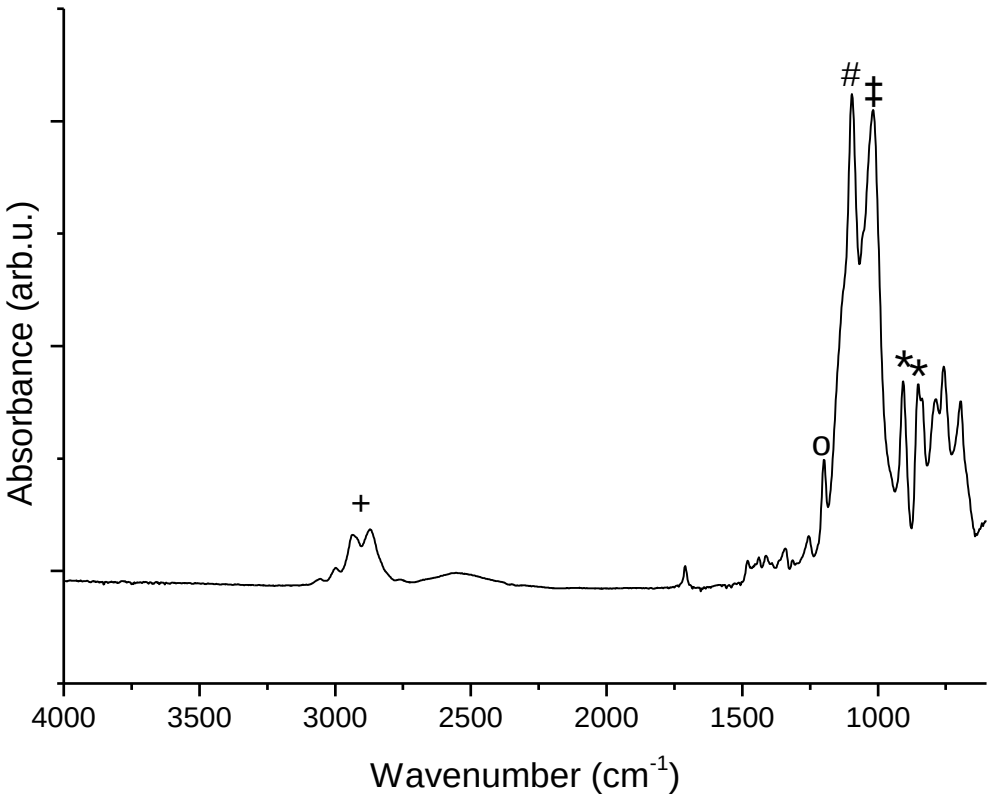
Experimental

Precipitated solids were analyzed by FT-IR using a Thermo Scientific Nicolet iN10 instrument operating in ATR mode and operating with a liquid nitrogen cooled MCT detector. Samples were ground to a fine powder and placed on a gold substrate. Spectra were recorded between wavenumbers of 4000-600 cm^{-1} with a resolution of 4 cm^{-1}.



FT-IR spectra of precipitated solids resulting from GPTMS/propylamine reaction after 24h. +: C-H alkanes, o: -Si-CH_2- #: Si-O-Si ‡: Si-OH, *: epoxide ring.

FT-IR analysis of the gel-like precipitated solids confirmed the presence of GPTMS species. The double band due to the Si-O-Si symmetric stretch of the silicate network (1094 cm^{-1}) and a surprisingly high level of non-bridging oxygens, Si-OH (1016 cm^{-1}) which shows that the silicate network is only partially condensed. Unopened epoxide ring species were observed at 904 cm^{-1} and 846 cm^{-1} providing further evidence that the eopixde ring remains unreacted within this timescale. There was no obversable bands corresponding to amine groups (typically expected around 3400-3500 cm^{-1} for N-H stretch, 1550-1650 cm^{-1} for N-H bending of primary amines) confirming that the propylamine primarily remains in solution as observed by ^1H NMR and does not react with the epoxide of GPTMS.



FT-IR spectra of precipitated solids resulting from GPTMS/propanthiol reaction after 24h. +: C-H alkanes, o: -Si-CH_2- #: Si-O-Si ‡: Si-OH, *: epoxide ring.

The FT-IR spectra of the gel-like precipitate obtained by reaction between GPTMS and propanthiol (Figure 8) showed characteristic bands from a partially condensed silicate network. The Si-O-Si symmetric stretch appeared at 1098 cm^{-1} with the non-bridging Si-OH band appearing at 1020 cm^{-1}. The unopened epoxide ring was observed at 847 and 909 cm^{-1}, indicating that no reaction had taken place between the thiol and the epoxide ring. Further confirmation of this was that no band attributed to the thiol group could be observed in the spectra.