Electronic Supplementary Material (ESI) for CrystEngComm.

Hydrogen Bond-Directed Assembly of Silsesquioxanes Cubes: Synthesis of Carboxylic Acid POSS Derivatives and the Solid State Structure of Octa[2-(p-carboxyphenyl)ethyl] Silsesquioxane

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Supporting Information

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Experimental Details:

General: Nuclear magnetic resonance spectra (NMR) were recorded on a Bruker 400 MHz in deuterated chloroform or DMSO. MS spectra were obtained on a Synapt G2-S from Waters with a quadrupole cell collision. IR spectra were done with a Spectrum 100 FT-IR spectrometer from Perkin Elmer.

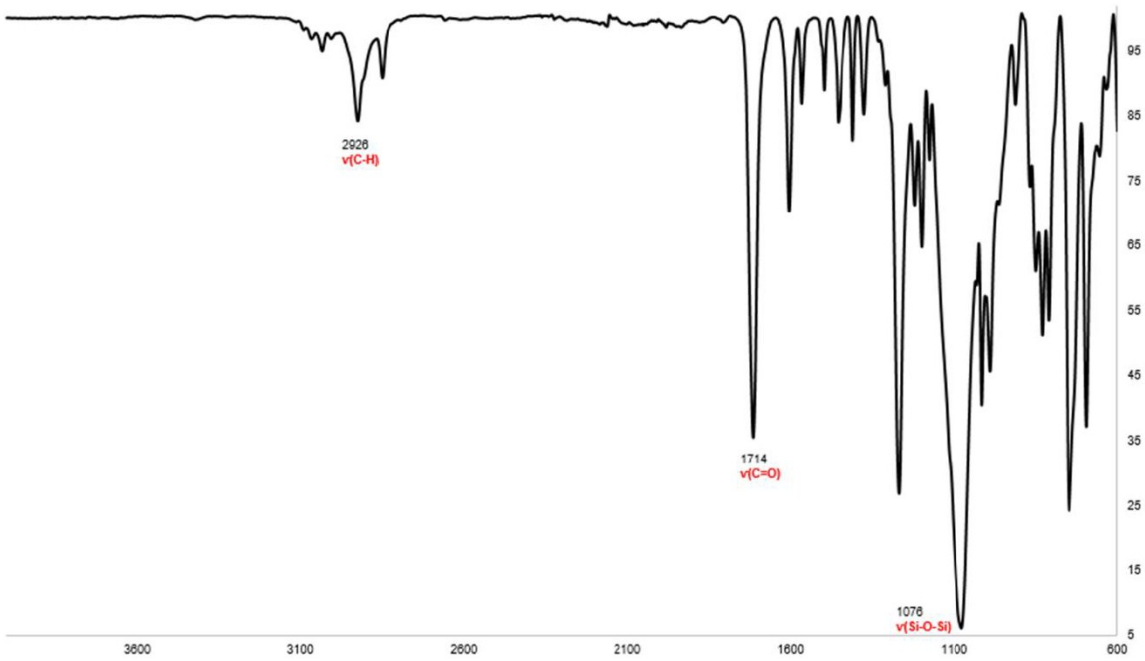


Figure S1. ATR - FTIR spectrum of **Octa[2-(p-benzyloxycarbonylphenyl)ethenyl] silsesquioxane**

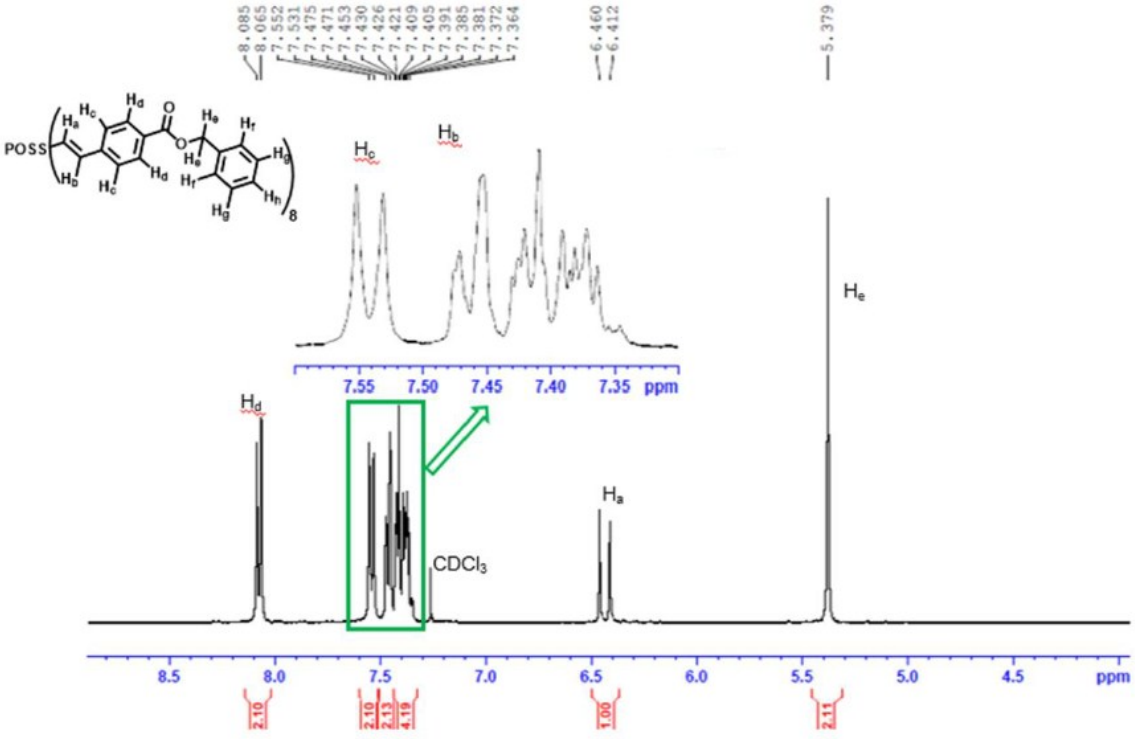


Figure S2. ^{1}H NMR spectrum of **Octa[2-(p-benzyloxycarbonylphenyl)ethenyl] silsesquioxane** in $CDCl_3$.

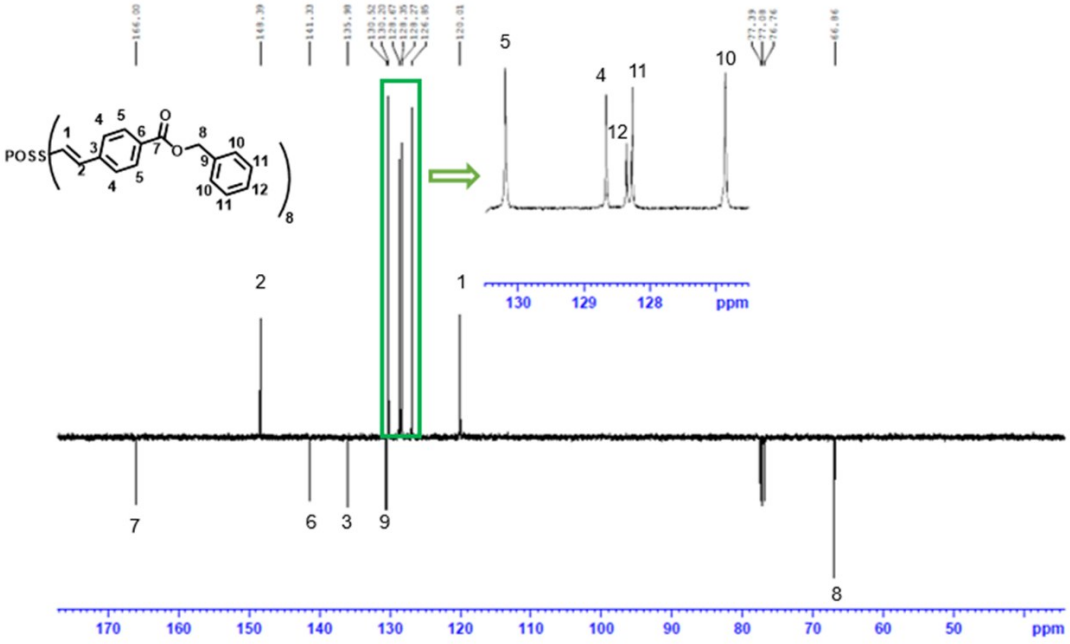


Figure S3. ^{13}C NMR spectrum of **Octa[2-(p-benzyloxycarbonylphenyl)ethenyl] silsesquioxane** in $CDCl_3$.

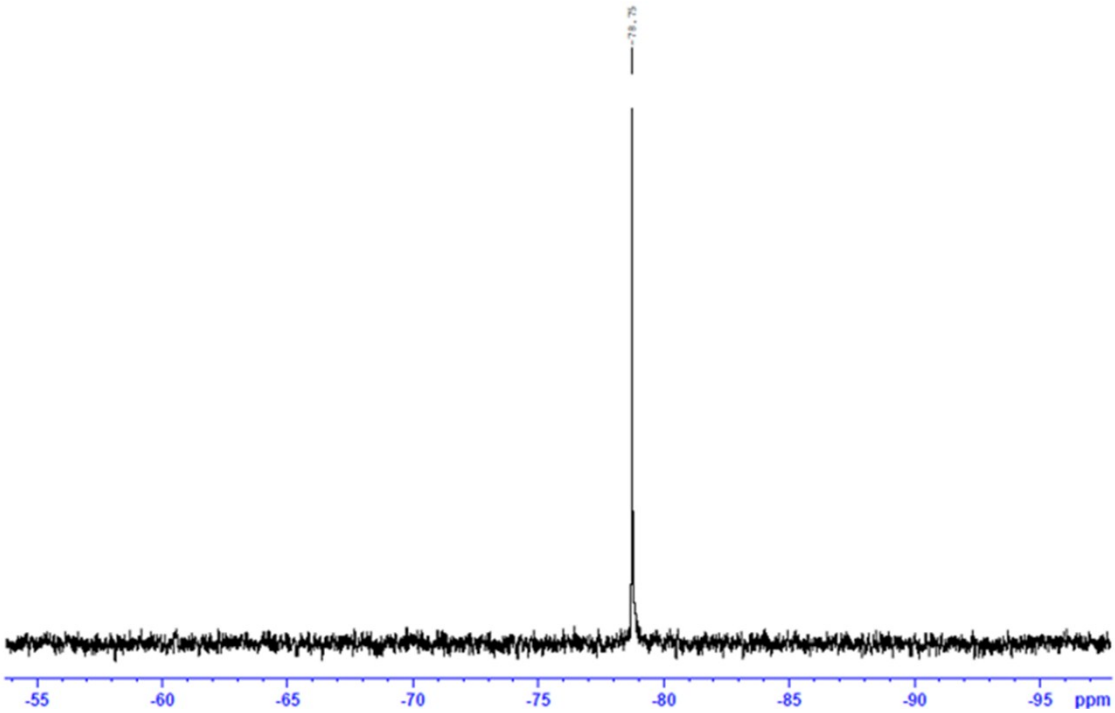


Figure S4. ^{29}Si NMR spectrum of **Octa[2-(p-benzyloxycarbonylphenyl)ethenyl] silsesquioxane** in $CDCl_3$.

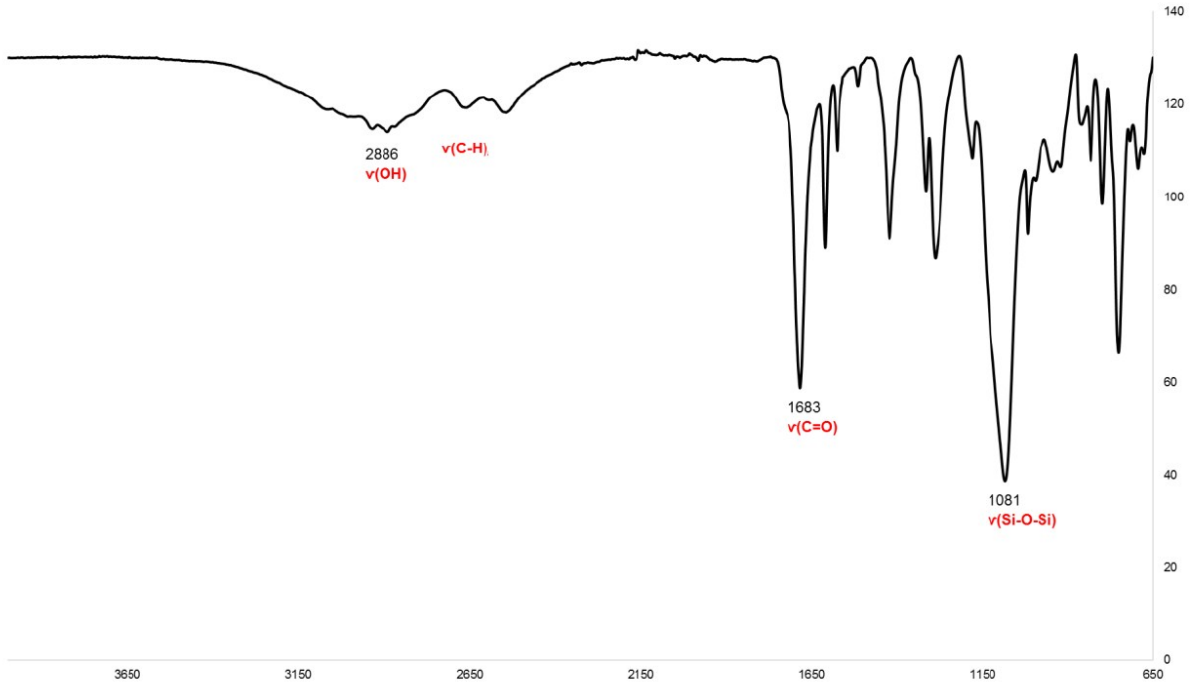


Figure S5. ATR - FTIR spectrum of **Octa[2-(p-carboxyphenyl)ethyl] silsesquioxane**

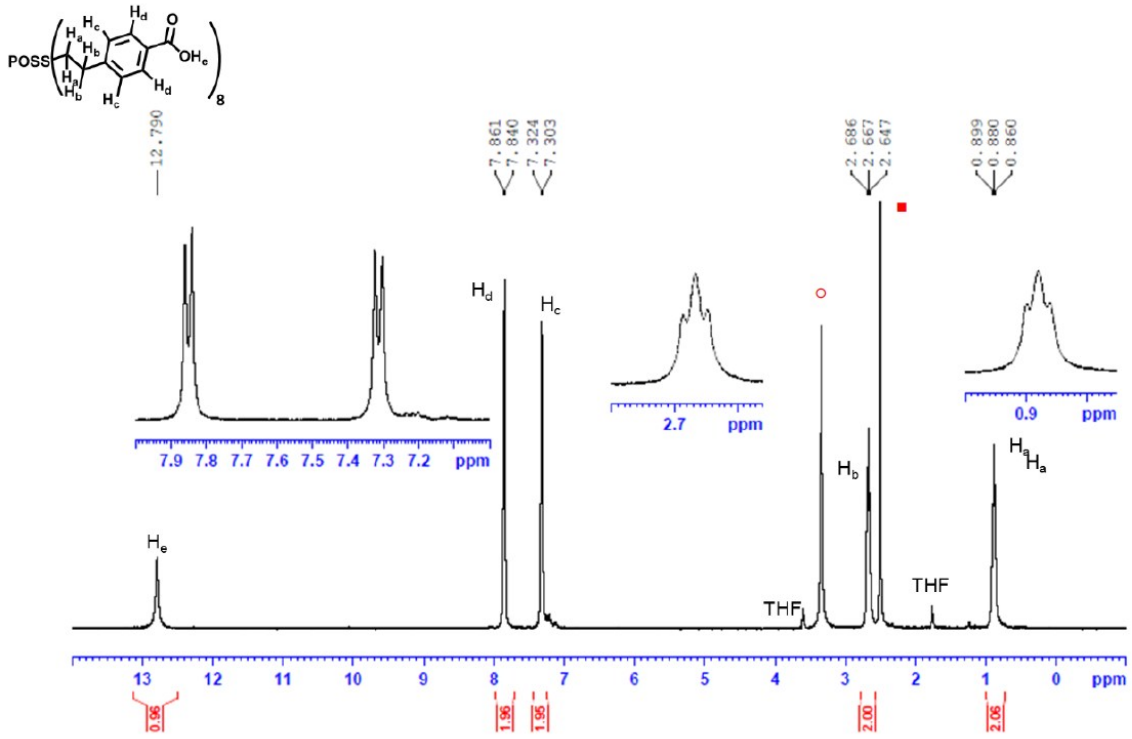


Figure S6. ^{1}H NMR spectrum of **Octa[2-(p-carboxyphenyl)ethyl] silsesquioxane** in DMSO-d6. ■DMSO, ○H_2O

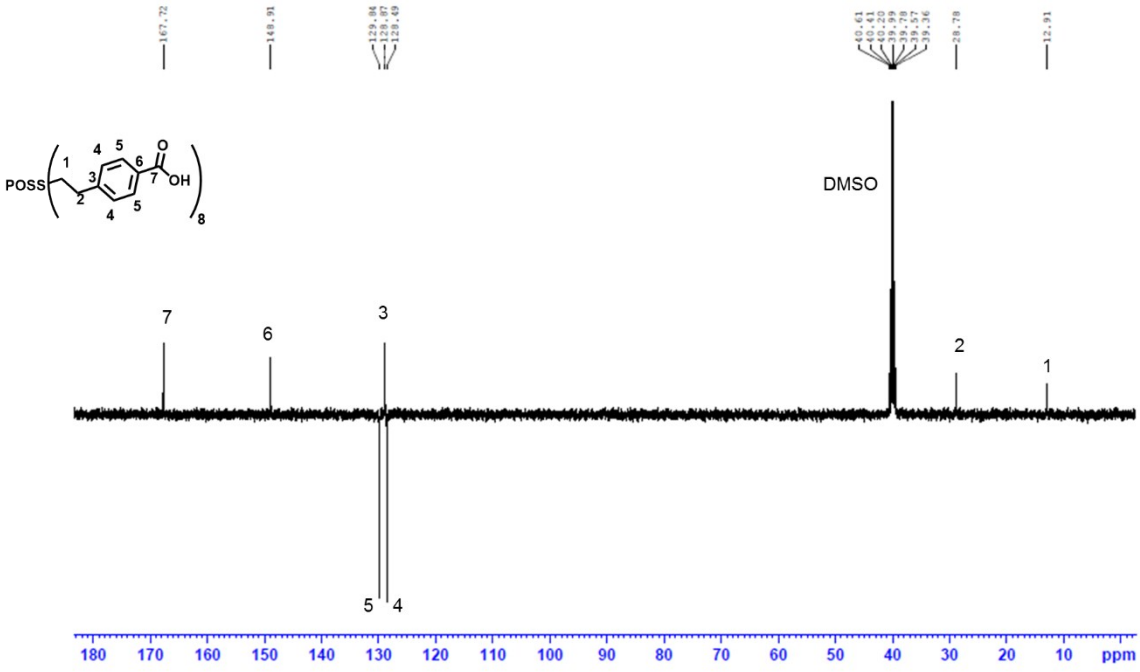


Figure S7. ^{13}C NMR spectrum of **Octa[2-(p-carboxyphenyl)ethyl] silsesquioxane** in DMSO-d6.

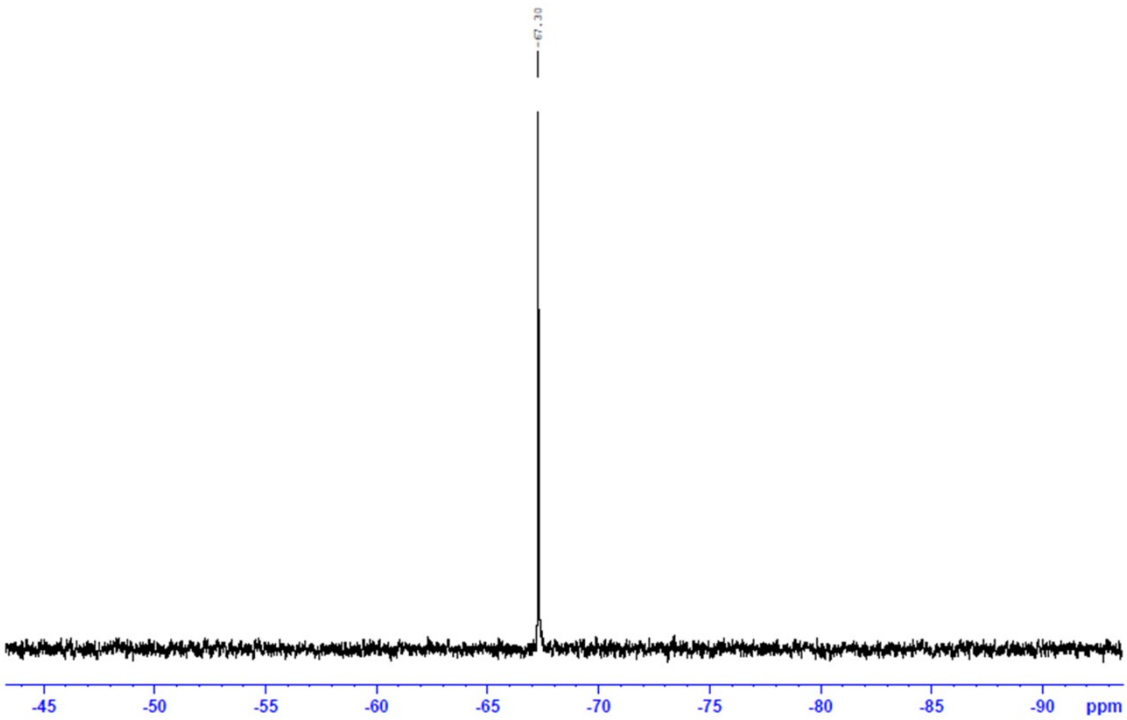


Figure S8. ^{29}Si NMR spectrum of **Octa[2-(p-carboxyphenyl)ethyl] silsesquioxane** in DMSO-d6.

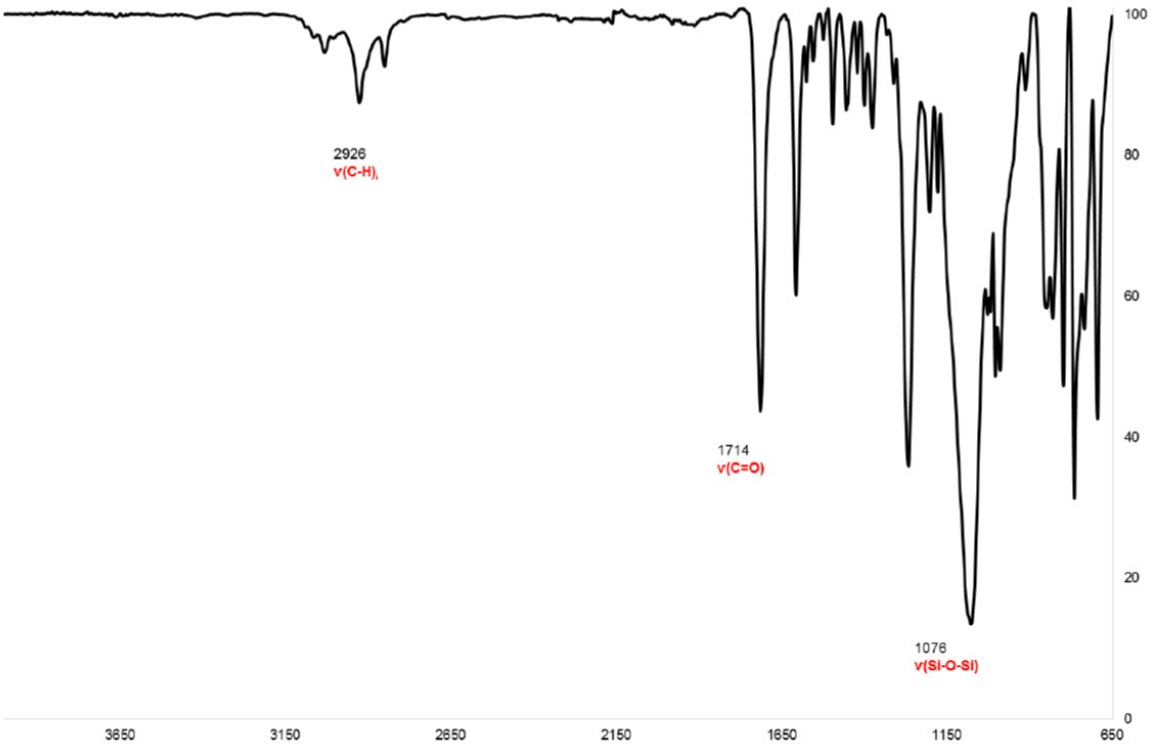


Figure S9. ATR - FTIR spectrum of **Octa[2-(4-benzyloxycarbonyl-1,1'-biphenyl) ethenyl] silsesquioxane**

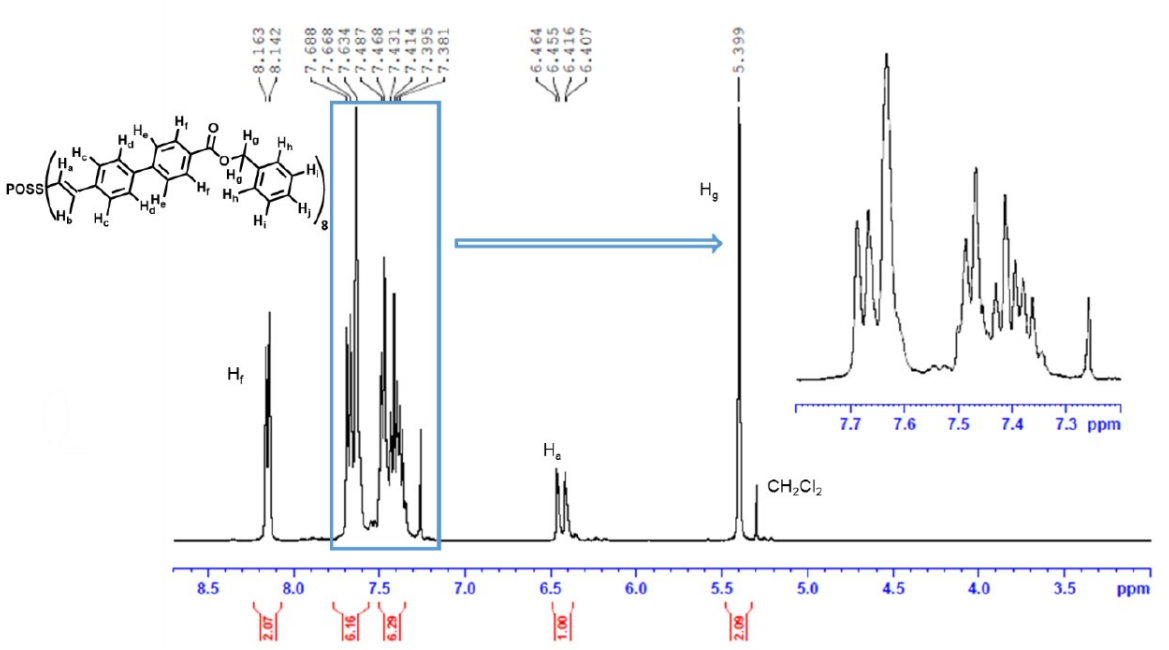


Figure S10. ^{1}H NMR spectrum of **Octa[2-(4-benzyloxycarbonyl-1,1'-biphenyl) ethenyl] silsesquioxane** in $CDCl_3$.

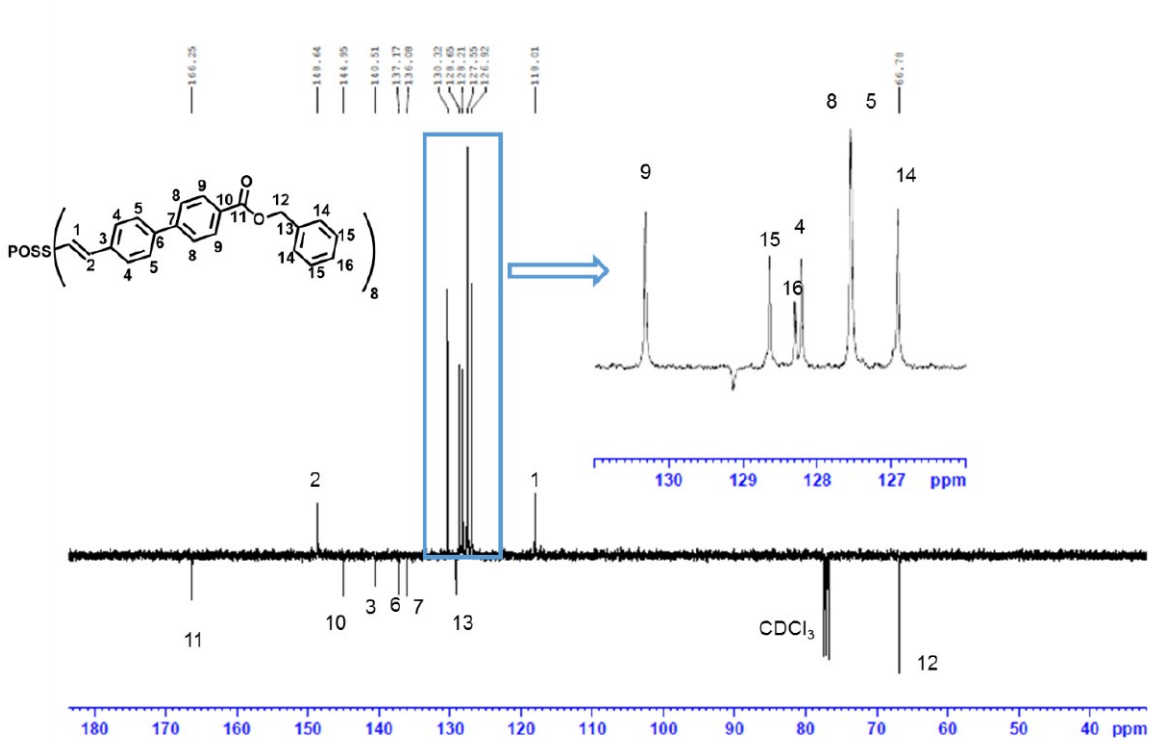


Figure S11. ^{13}C NMR spectrum of **Octa[2-(4-benzyloxycarbonyl-1,1'-biphenyl) ethenyl] silsesquioxane** in $CDCl_3$.

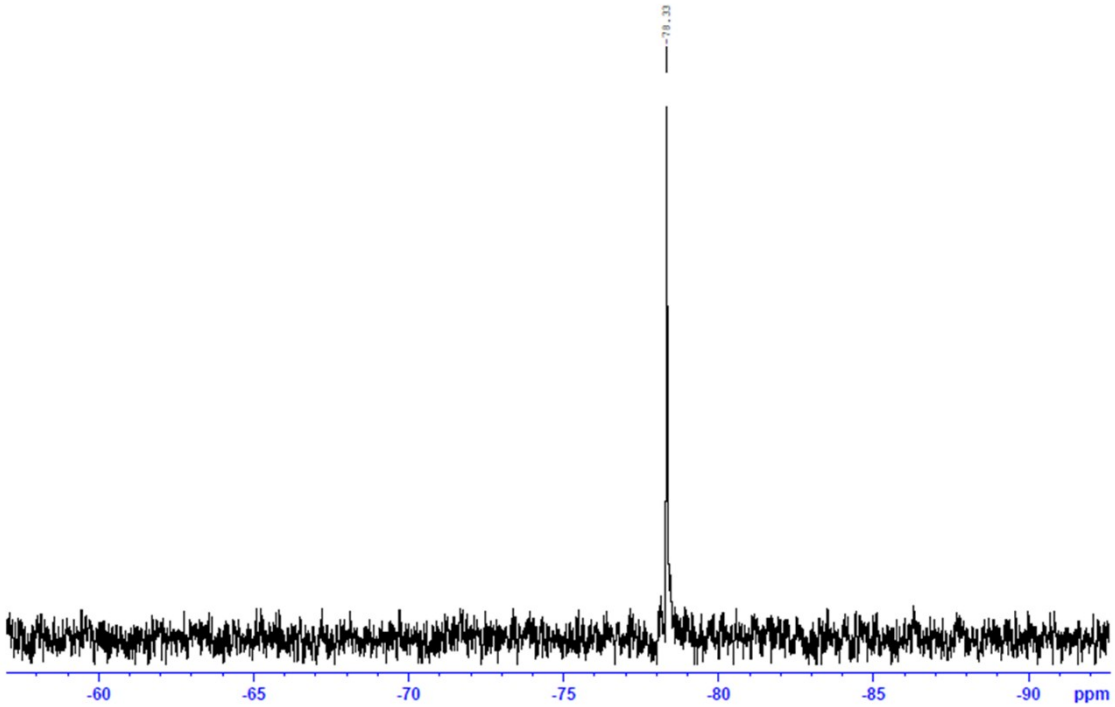


Figure S12. ^{29}Si NMR spectrum of **Octa[2-(4-benzyloxycarbonyl-1,1'-biphenyl) ethenyl] silsesquioxane** in $CDCl_3$.

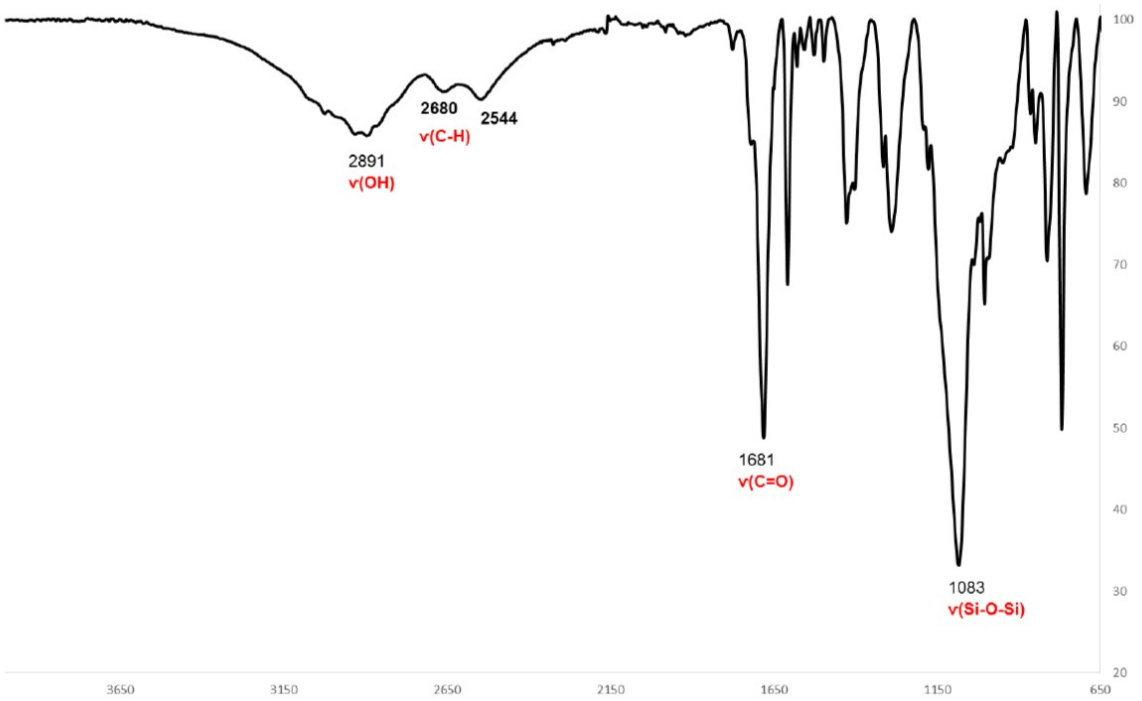


Figure S13. ATR - FTIR spectrum of **Octa[2-(4-carboxy-1,1'-biphenyl)ethyl] silsesquioxane.**

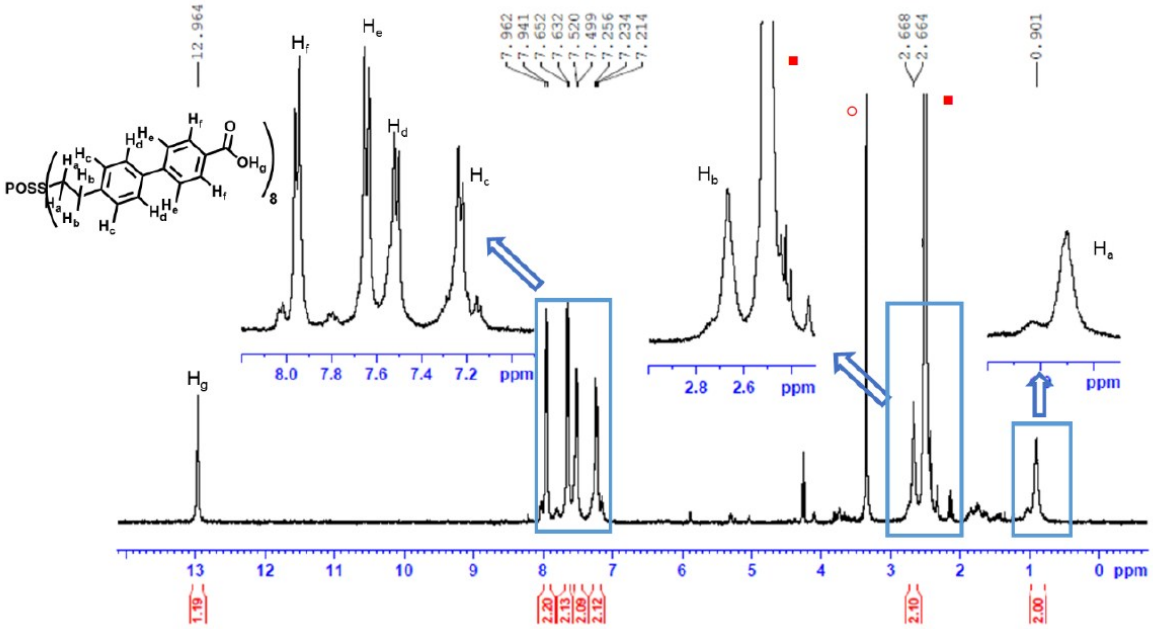


Figure S14. ^{1}H NMR spectrum of **Octa[2-(4-carboxy-1,1'-biphenyl)ethyl] silsesquioxane in** DMSO-d6. ■DMSO, oH_2O

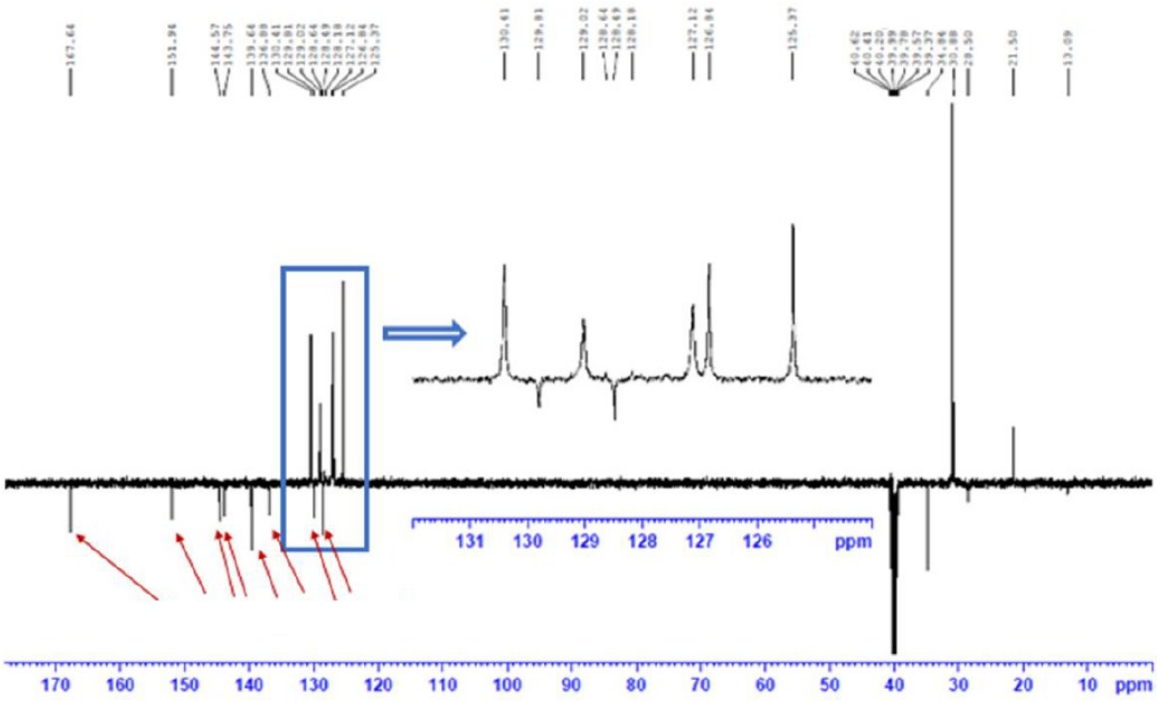


Figure S15. ^{13}C NMR spectrum of **Octa[2-(4-carboxy-1,1'-biphenyl)ethyl] silsesquioxane** in DMSO-d6.

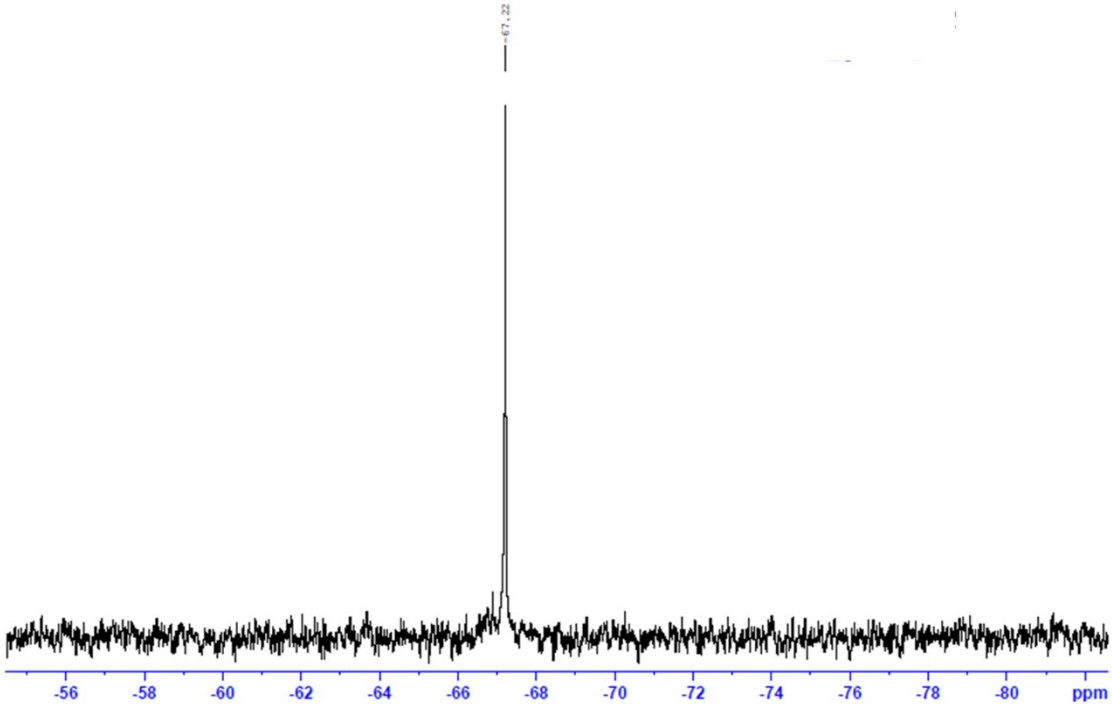


Figure S16. ^{29}Si NMR spectrum of **Octa[2-(4-carboxy-1,1'-biphenyl)ethyl] silsesquioxane** in DMSO-d6.

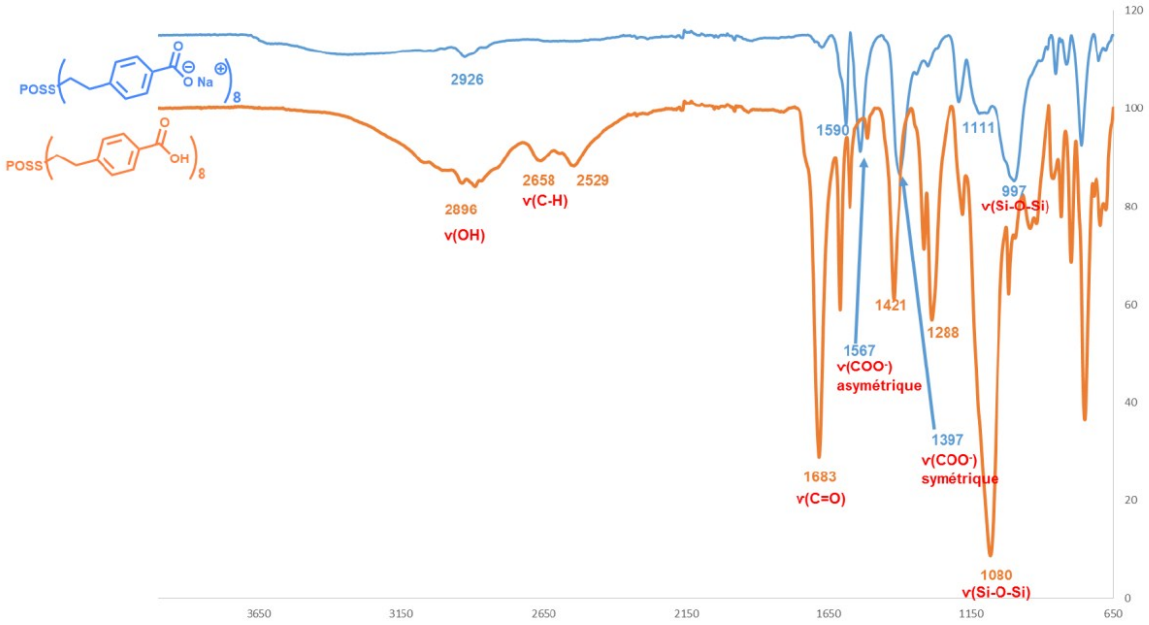


Figure S17. Comparison between ATR - FTIR spectra of **Octa[2-(4-carboxy-1,1'-biphenyl)ethyl] silsesquioxane** and the corresponding sodium salt.

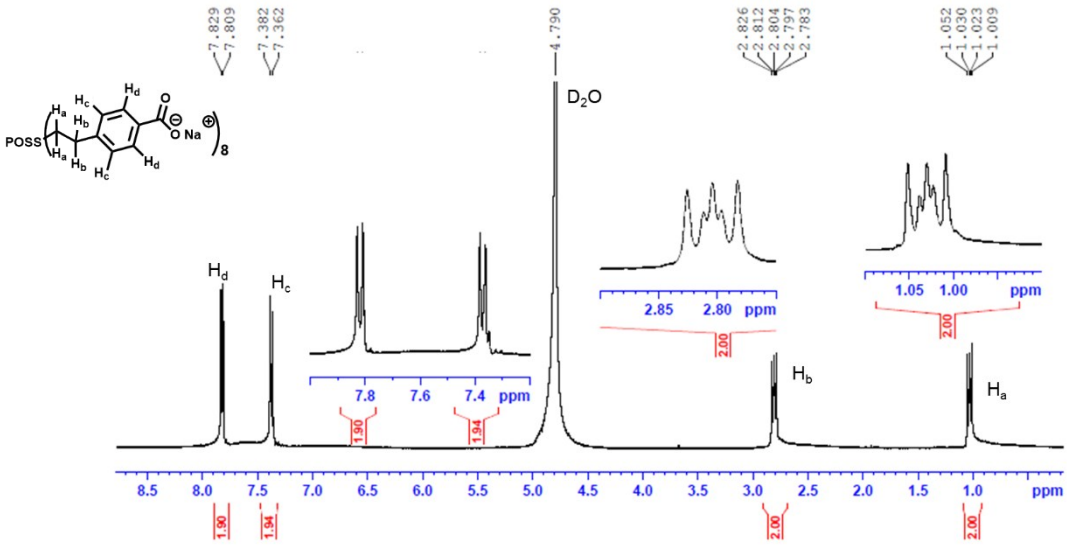
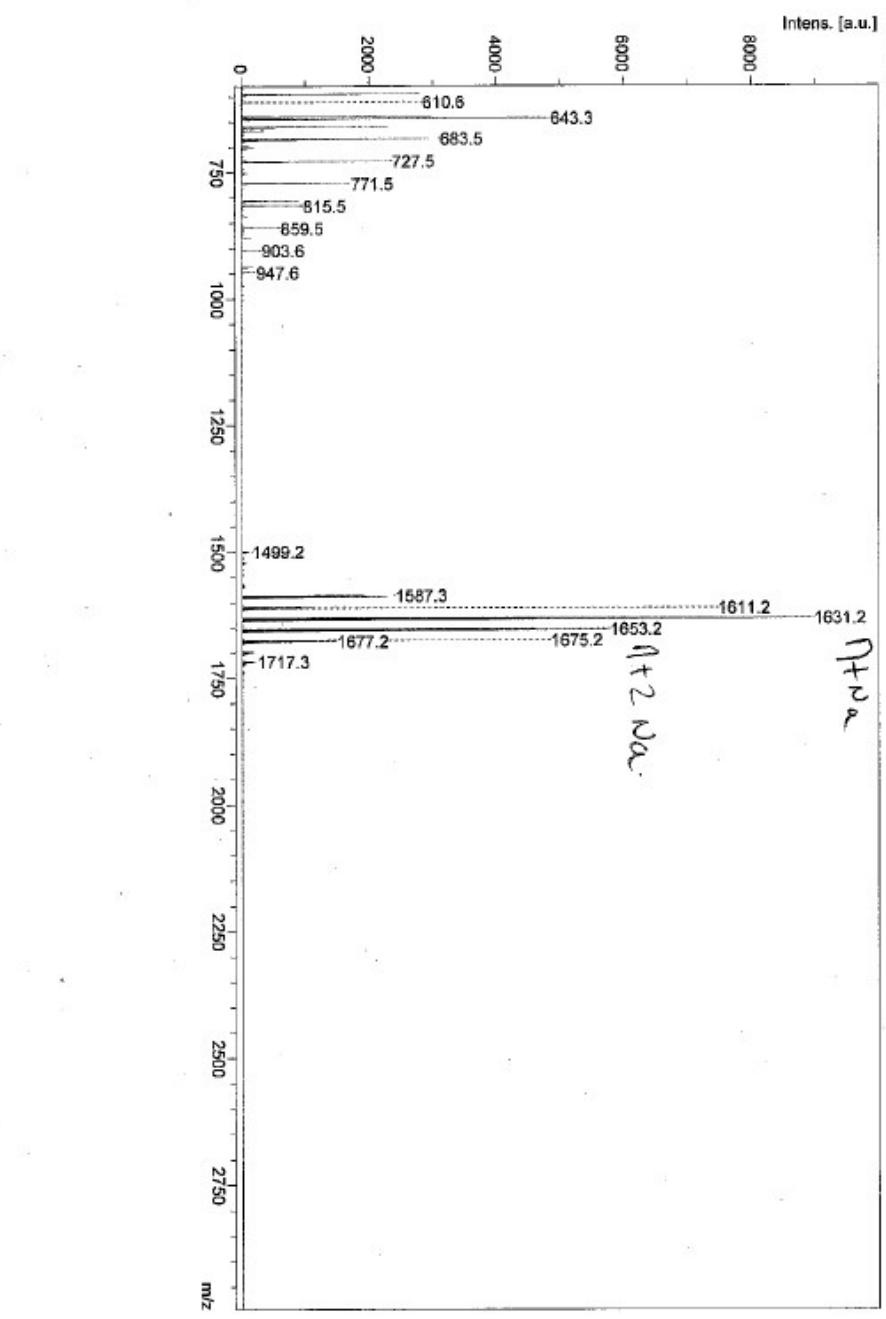


Figure S18. ^{1}H NMR of spectrum of **sodium octa[2-(p-carboxylatephenyl)ethyl] silsesquioxane** in D_2O.

Figure S19. MALDY-TOF mass spectroscopy spectrum in positive ionisation of **Octa[2-(p-benzyloxycarbonylphenyl)ethenyl] silsesquioxane.**



m/z	S/N	Res.	Intens.	Area
595.420	321.0	8205	2769.88	322
610.617	11.9	10505	102.06	10
638.643	54.8	9598	472.45	51
639.434	347.8	8356	2982.01	370
643.326	517.0	7379	4434.39	616
659.317	239.5	7799	2067.62	279
661.335	36.9	9919	318.36	34
666.670	39.7	10607	340.92	35
683.453	346.7	10068	3069.13	354
685.461	64.6	9642	560.53	67
687.365	13.2	11695	115.01	11
697.478	14.6	10507	124.80	14
699.310	19.4	10776	165.54	18
703.466	8.1	9485	70.03	9
727.473	270.4	10688	2304.69	272
751.460	10.3	10562	87.89	11
771.497	192.7	11972	1627.30	187
806.519	101.2	12058	851.52	101
815.519	106.9	12761	897.11	103
838.851	8.0	15655	67.57	6
859.542	65.0	14195	553.09	63
879.473	15.8	12322	128.37	17
903.569	31.1	15915	247.92	27
937.479	21.6	13317	173.70	23
947.590	19.1	18160	148.71	15
1499.173	19.8	25162	128.28	21
1522.160	9.5	21602	57.31	11
1567.238	6.9	20589	40.87	9
1587.250	388.2	17355	2350.51	608
1589.255	186.3	20530	1103.93	245
1609.236	195.7	20751	1154.07	263
1611.240	66.5	15969	393.06	114
1631.243	1370.9	12757	8265.56	2999
1633.260	616.5	13840	3671.57	1225
1653.232	848.4	13321	5156.40	1835
1655.249	344.8	14010	2037.54	686
1675.222	223.6	15230	1314.78	419
1677.239	71.3	16840	421.23	121
1697.213	27.9	15933	164.67	54
1699.226	11.9	13658	70.95	27
1717.284	22.7	19686	136.65	36
1739.269	6.9	21926	44.12	11

Figure S20. MALDY-TOF mass spectroscopy spectrum in positive ionisation of **Octa[2-(p-carboxyphenyl)ethyl] silsesquioxane.**

Figure S21. MALDY-TOF mass spectroscopy spectrum in negative ionisation of **Octa[2-(p-carboxyphenyl)ethyl] silsesquioxane.**

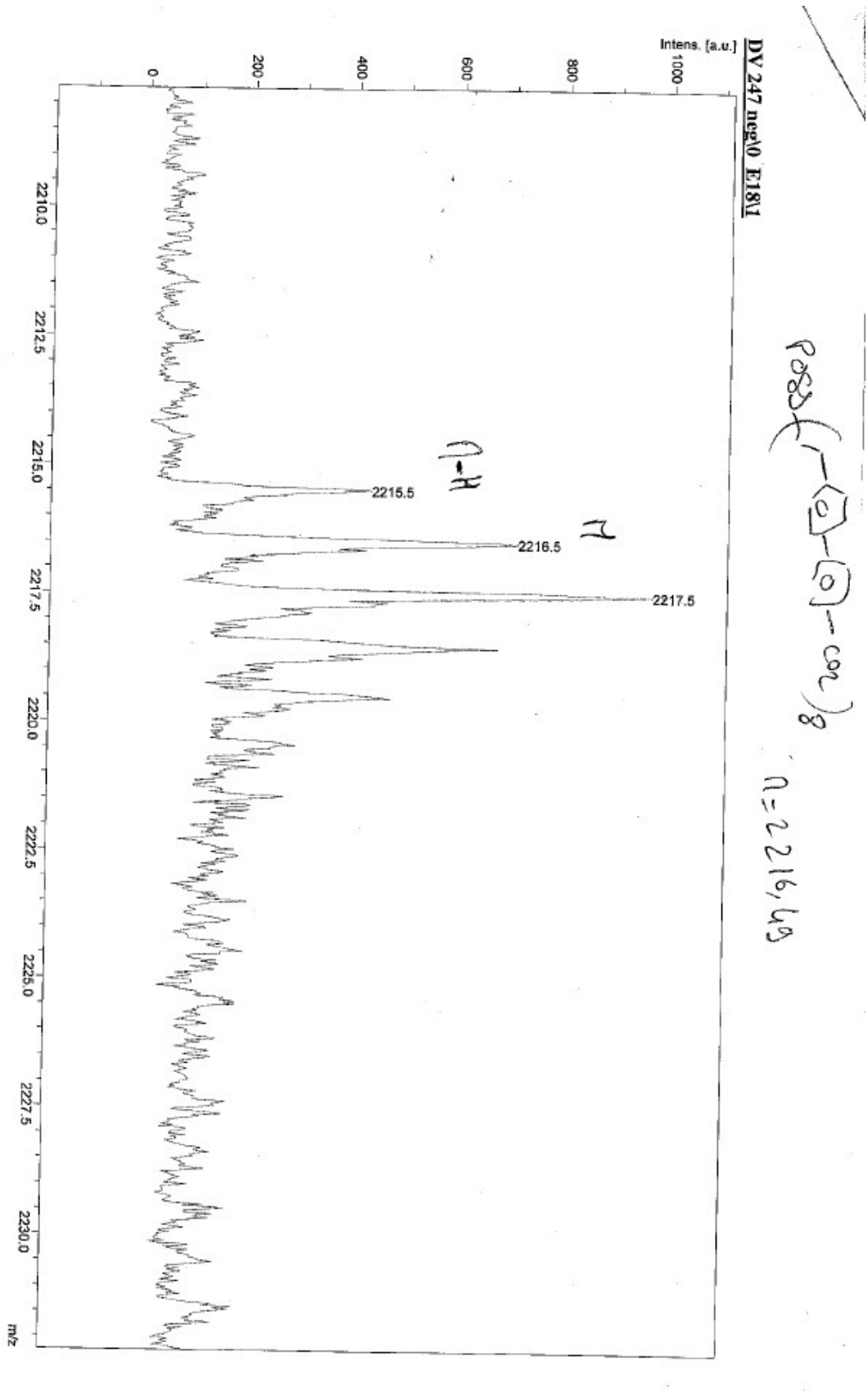


Figure S22. MALDY-TOF mass spectroscopy spectum in negative ionisation of **Octa[2-(4-carboxy-1,1'-biphenyl)ethyl] silsesquioxane.**

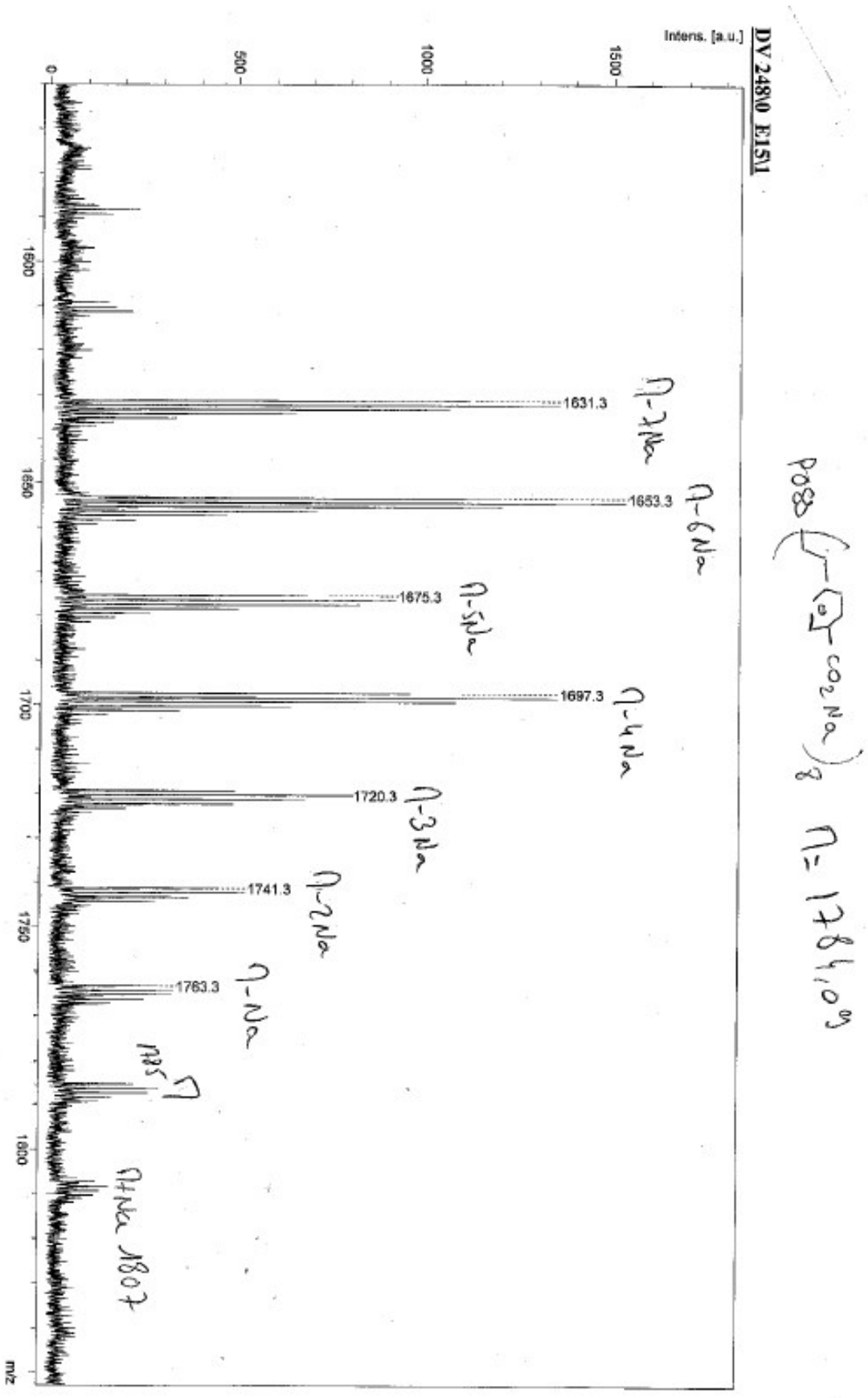


Figure S23. MALDY-TOF mass spectroscopy spectrum of **sodium octa[2-(p-carboxylatephenyl)ethyl] silsesquioxane.**