

Supporting Information

Synthesis and Biological Evaluation of Three New Chitosan Schiff Base

Derivatives

Nadia Q. Haj^a, Mohsin O. Mohammed,^{b*} Loqman E. Mohammood^c

^a University of Kirkuk, College of Science, Chemistry Department,009641, Kirkuk, Iraq.

^b University of Kirkuk, College of Agriculture, Basic Science Department,009641, Kirkuk, Iraq.

^c University of Kirkuk, College of Pharmacy, Chemistry Department,009641, Kirkuk, Iraq.

Table S1: shows the details for the treatment of C₁, C₂, C₃, and C₄

SAMPLE	PROCESS	TREATMENT/ DESCRIPTION
C ₁		The fish scales were washed and grinded into the powder form. The powder was treated with hot ethanol (50 - 60 °C) to kill germs and remove pungent odour. The resulting sample (designated as C ₁) was washed with ample amount of water and subjected to freeze-drying.
C ₂	Demineralization	HCl (5 %) was added slowly to the powder to remove CaCO ₃ component with volume to sample ratio 5:1. The reaction mixture was stirred for 3 hrs. After that, the powder was washed with ample amount of water until pH is neutral; 10g of samples is collected and labelled as C ₂ .
C ₃	Deproteinization	NaOH (10 %) was added to denature the powder and remove the protein at 60 °C with volume to sample ratio 5:1. The mixture was stirred for 3 hours. After that, the powder was washed with ample amount of water until pH is neutral; 10g of samples is collected and labelled as C ₃ .
C ₄	Deacetylation	NaOH (50 %) was added to deacetylate the chitin into chitosan with volume to sample ratio 5:1. The mixture was treated under reflux condition for 3 hours. After that, the powder was washed with ample amount of water until pH is neutral; 10g of samples is collected and labelled as C ₄ .

*Corresponding author. Phone: +9647701307283. E-mail addresses: althker1@uokirkuk.edu.iq, orcid: <https://orcid.org/0000-0001-5341-9727>

Table S2: illustrate the characteristic by the Acid-base titration method of standard and experimentally prepared chitosan

Sample	W	C ₁ / M	V ₁ / mL	C ₂ / M	V ₂ / mL	(-NH ₂)	DD%
C ₄	0.2909	0.1	30.00	0.1	19.6	0.0555	65.50
Chitin	0.3001	0.1	30.00	0.1	29.8	0.0011	2.09
Chitosan	0.2998	0.1	30.00	0.1	12.2	0.0949	96.10

Table S3: shows the characteristic absorption bands in the FT-IR spectra of standard and experimentally prepared chitosan

Wavenumber / cm ⁻¹				Vibration Mode
Chitin		Chitosan		
Chitin	C ₃	Chitosan	C ₄	
3441	3446	3437	3441	v(NH ₂) assoc. in primary amines v(OH) assoc. in pyranose ring
3112	3107	2874	2958	v _{as} (CH ₂) in CH ₂ OH group
2891	2891	-	2887	v(C-H) in pyranose ring
1655 (Shoulder)	1655 (Shoulder)	1602 (Shoulder)	1627 (Shoulder)	v(C=O) in NHCOCH ₃ group (Amide I band)
1416	1419	1421	1414	δ(CH ₂) in the CH ₂ OH group
1382	1382	1383	1383	δ _s (CH ₃) in NHCOCH ₃ group
1316	1316	1324	1316	δ(C-H) in pyranose ring
1264	1261	1259	1261	Complex vibrations of NHCO group (Amide III band)
1157	1157	1156	1157	v _{as} (C-O-C) (glycosidic linkage)
1074	1075	1078	1075	v _{as} (C-O-C) (glycosidic linkage)
1026	1024	1029	1024	v(C-O) in secondary OH group
953	953	-	954	v(C-O) in primary OH group
896	896	895	896	Pyranose ring skeletal vibrations
695	596	663	617	δ(NH) out of plane
598	527	578	529	δ(OH) out of a plane

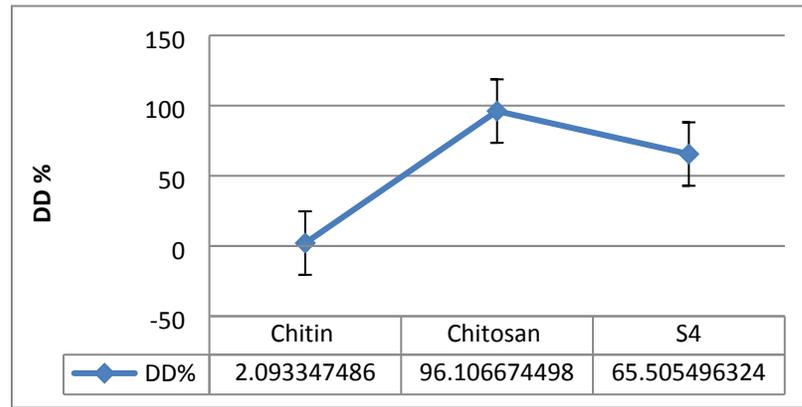


Figure S1: DD % of S₄, chitin and chitosan samples

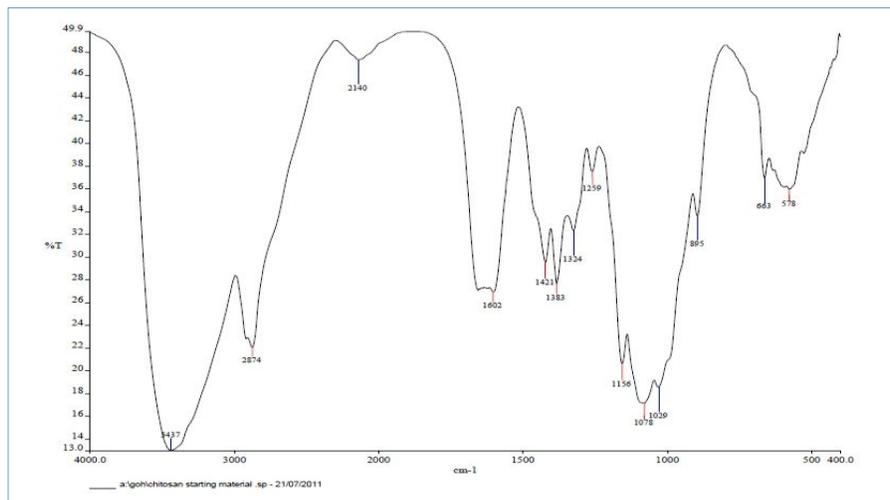


Figure S2: FT-IR spectrum of CS (Amrec Sirim; DD %=96.10 %)

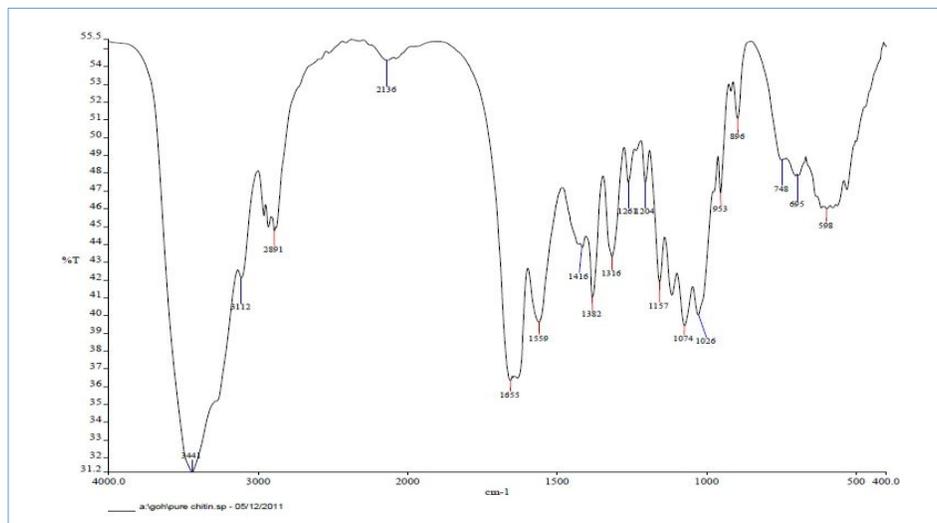


Figure S3: FT-IR spectrum of CH (Himedia; DD %=2.09%)

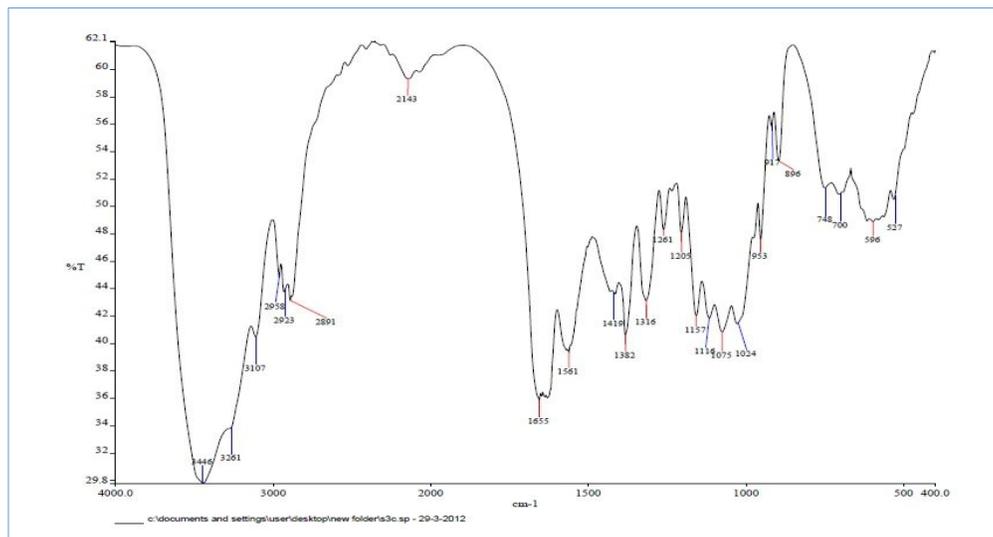


Figure S4: Shows the FT-IR spectrum of C₃

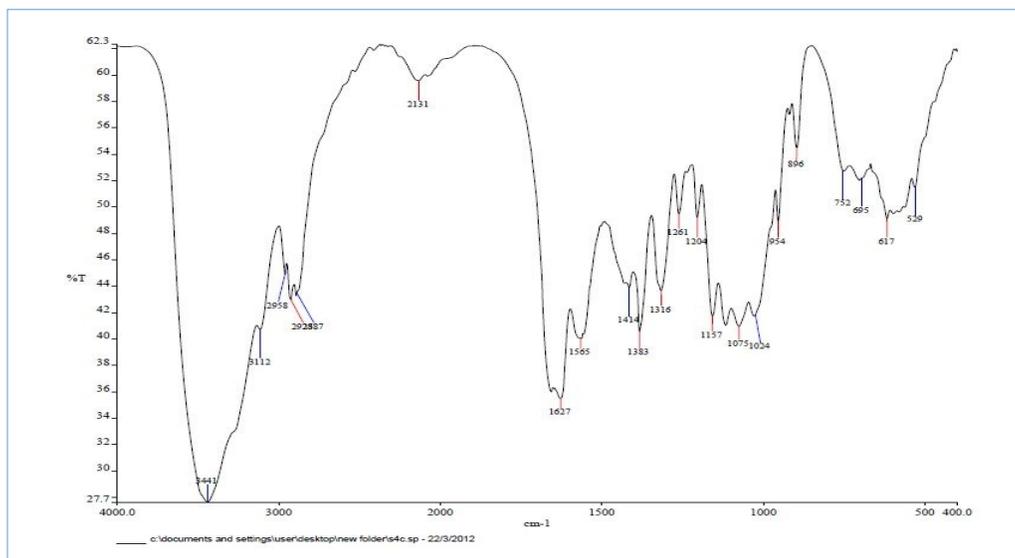


Figure S5: FT-IR spectrum of C₄ (DD %=65.50 %)

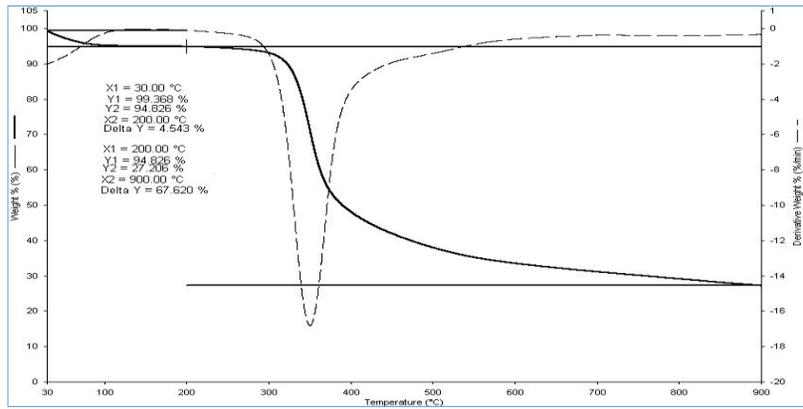


Figure S6: TG and DTG plots of CS (Amrec Sirim; DD %=96.10 %)

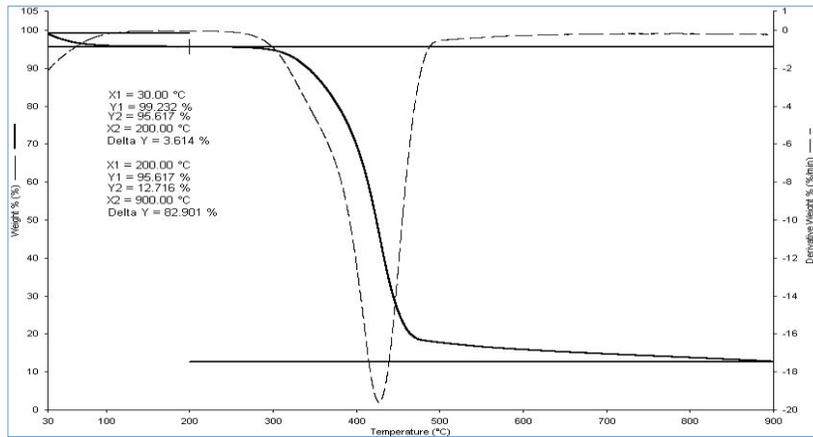


Figure S7: TG and DTG plots of CH (HiMedia; DD %=2.09%)

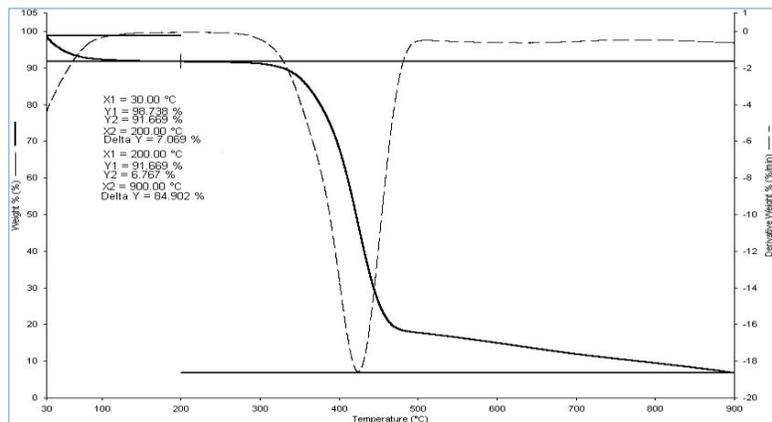


Figure S8: TG and DTG plots of C₃

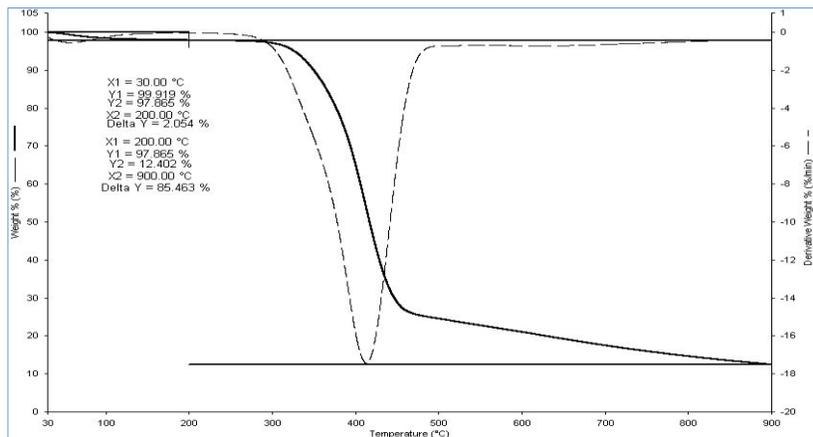


Figure S9: TG and DTG plots of C₄ (DD %=65.50 %)

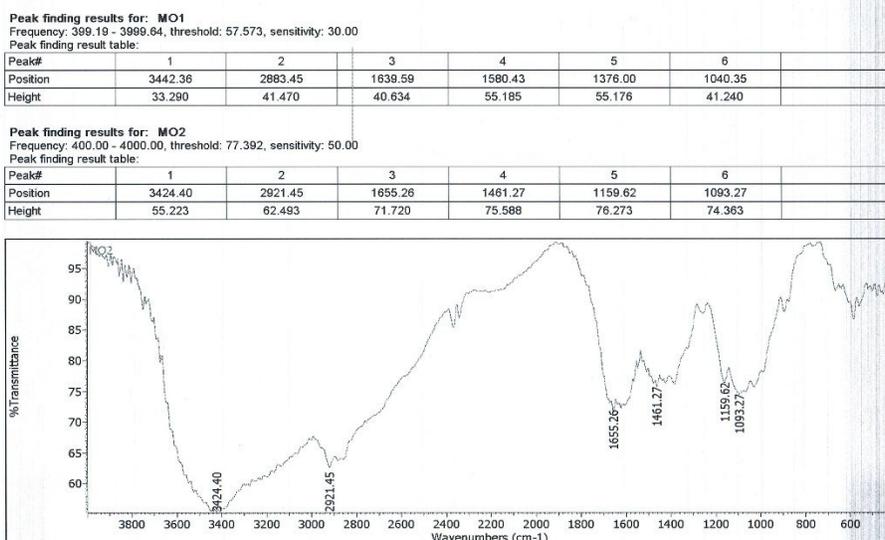


Figure S10: FT-IR spectrum of CS-P1

Peak finding results for: MO1
 Frequency: 399.19 - 3999.64, threshold: 57.573, sensitivity: 30.00
 Peak finding result table:

Peak#	1	2	3	4	5	6
Position	3442.36	2883.45	1639.59	1580.43	1376.00	1040.35
Height	33.290	41.470	40.634	55.185	55.176	41.240

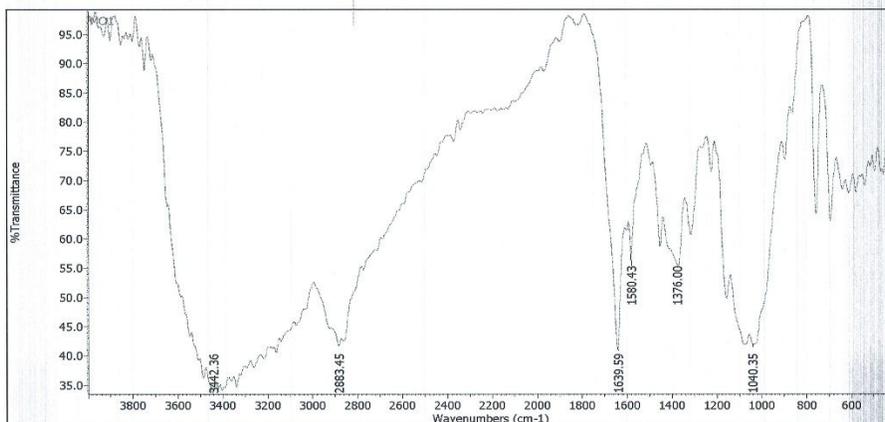


Figure S11: FT-IR spectrum of CS-P2

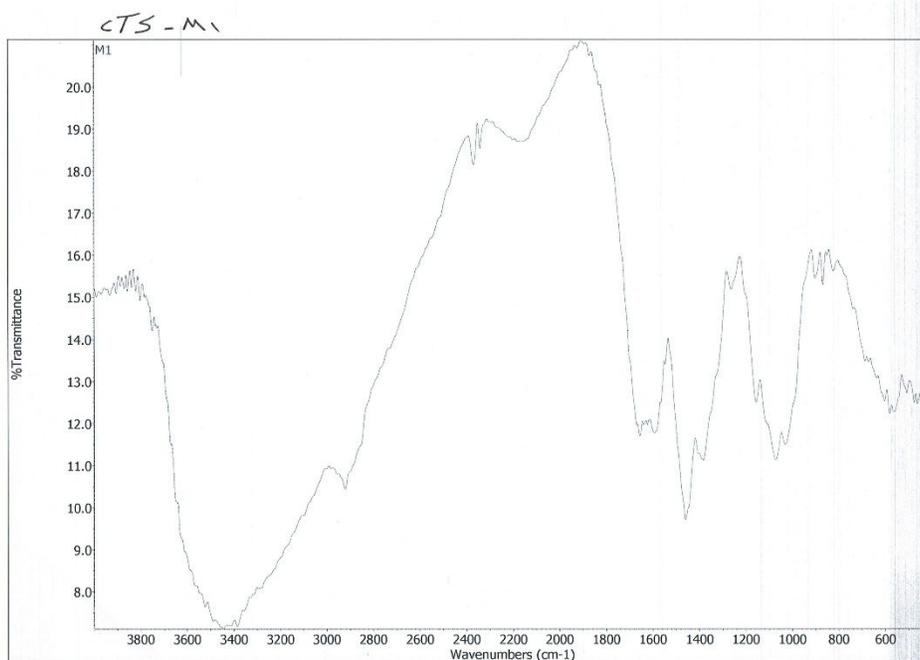


Figure S12: FT-IR spectrum of CS-P3

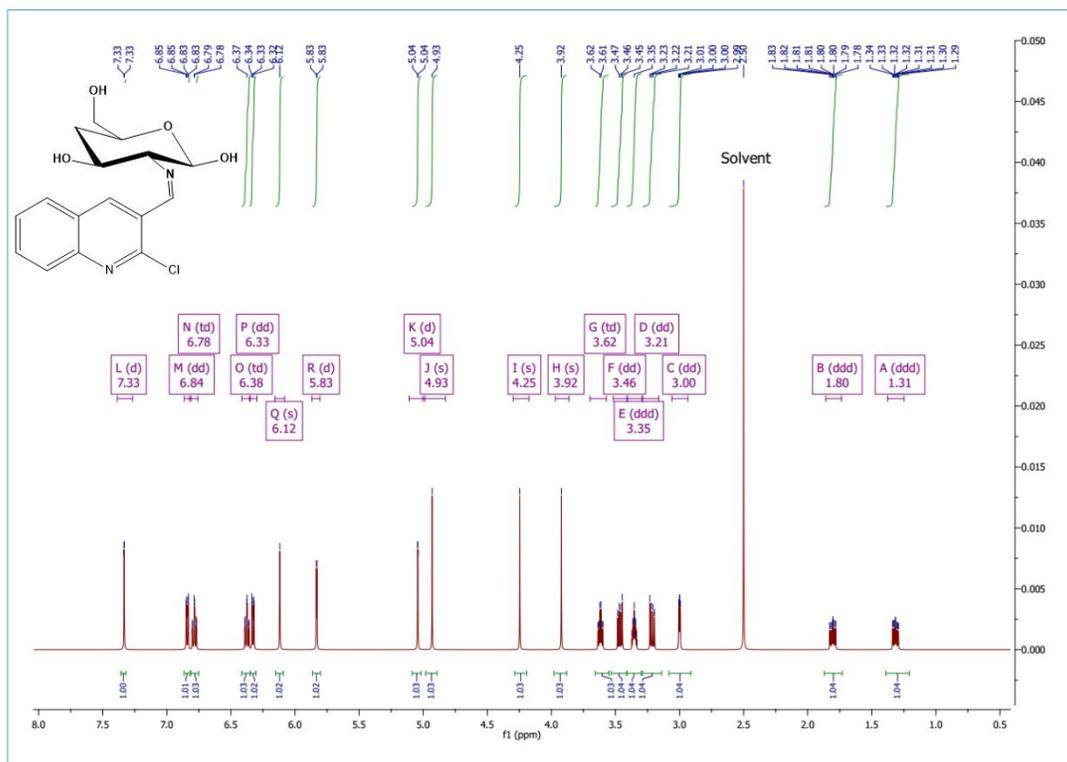


Figure S13: ^1H NMR form compound CS-P1

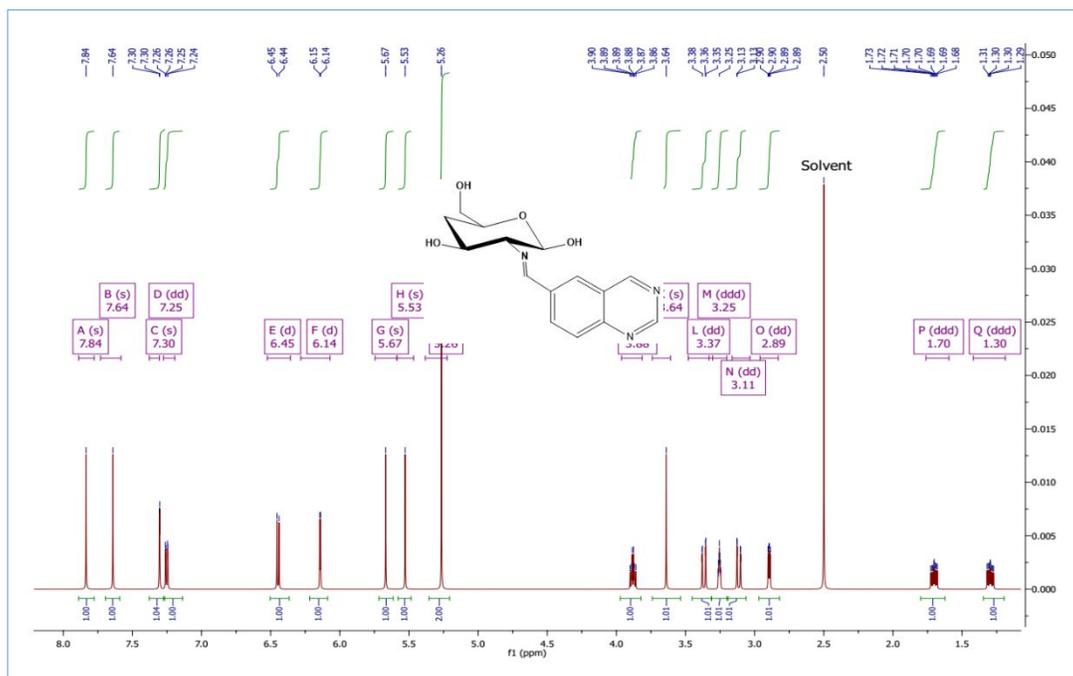


Figure S14: ^1H NMR form compound CS-P2

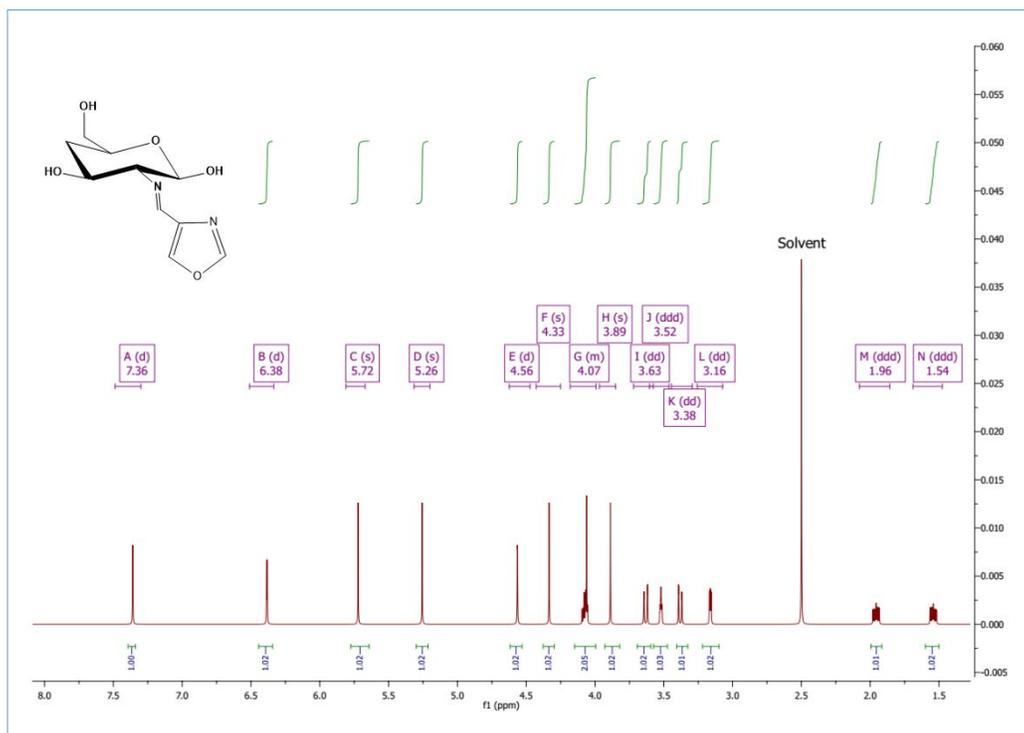


Figure S15: ¹H NMR form compound CS-P3

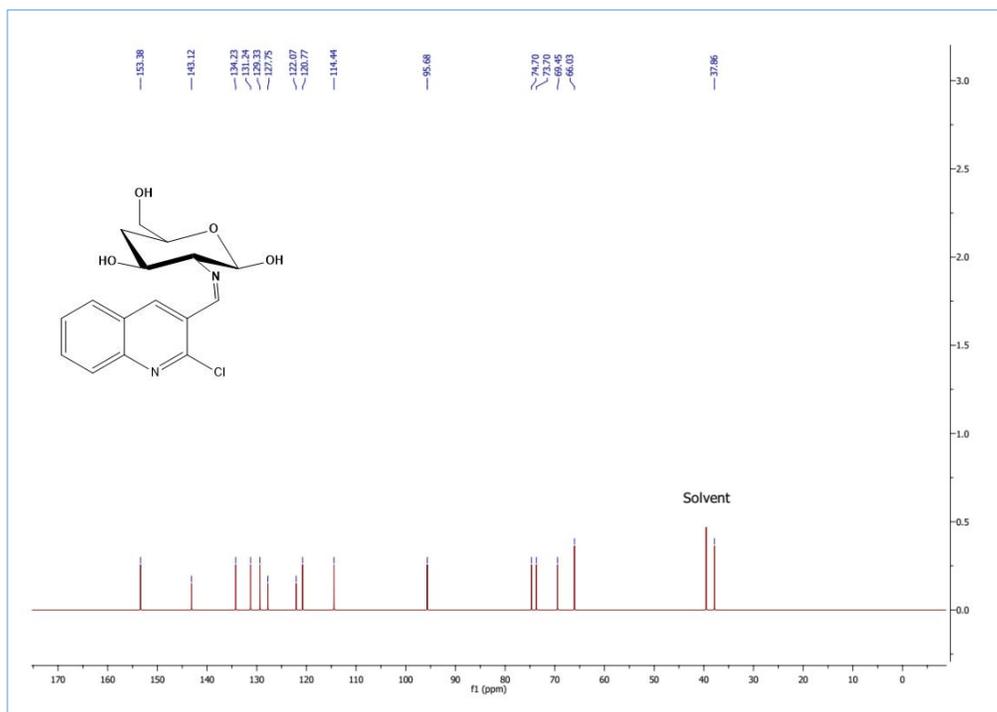


Figure S16: ¹³C NMR form compound CS-P1

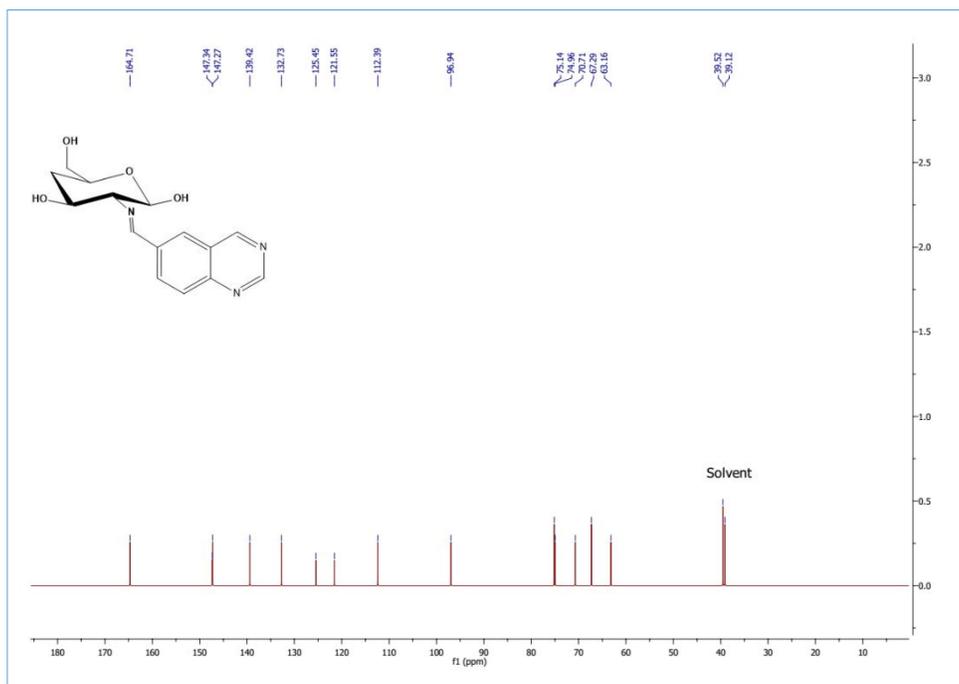


Figure S17: ^{13}C NMR form compound CS-P2

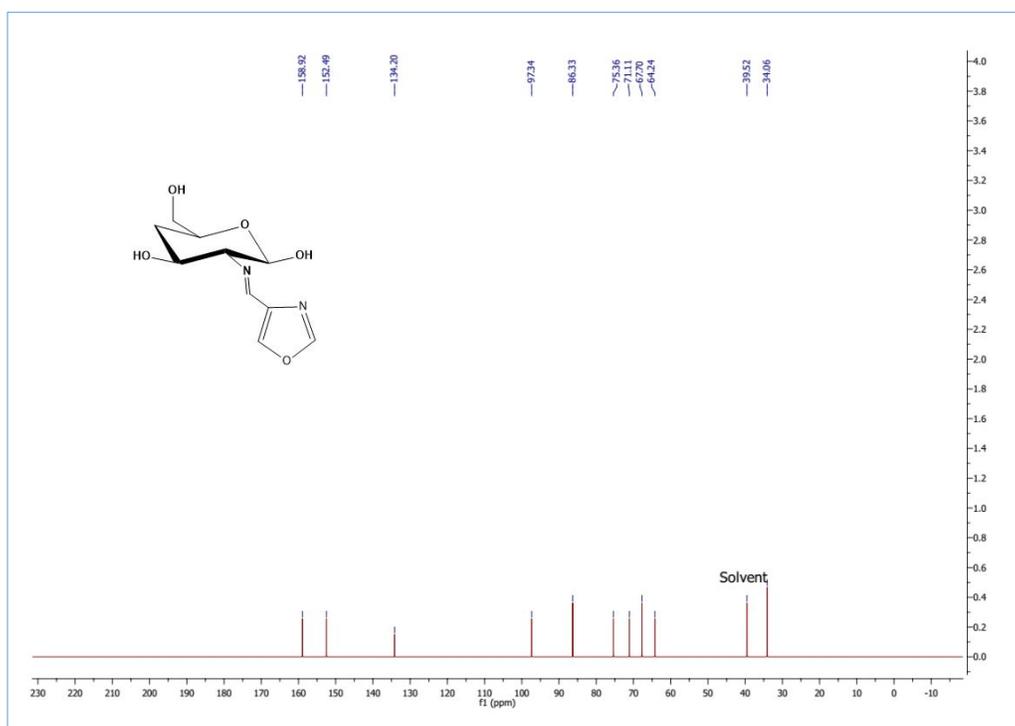


Figure S18: ^{13}C NMR form compound CS-P3