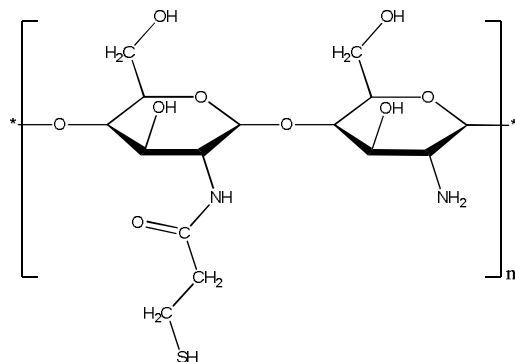


Sample Name: Thiolated chitosan
(thiolated with mercaptopropionic acid)

Sample #: P16029-TCS

Structure:

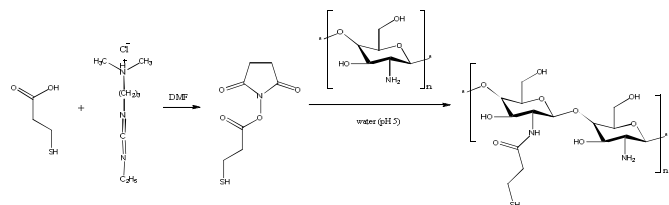


Composition:

| | |
|---|------------------------------|
| <i>Dynamic viscosity of native chitosan:</i> | <i>Degree of thiolation:</i> |
| 50–100 mPa·s, (0.5% in 0.5% Acetic Acid at 20°C) | 16 mol% |

Synthesis procedure:

The thiolated chitosan was prepared from chitosan using mercaptopropionic acid and EDAC•HCl. A scheme of reaction is shown below.



Purification:

The obtained thiolated chitosan was extensively dialyzed through cellulose membrane (MW cut-off, 5000) against water containing 5 mM HCl and 1% NaCl. The product was filtered off and lyophilized (freeze-dried).

The recommended storage temperature: around 4°C.

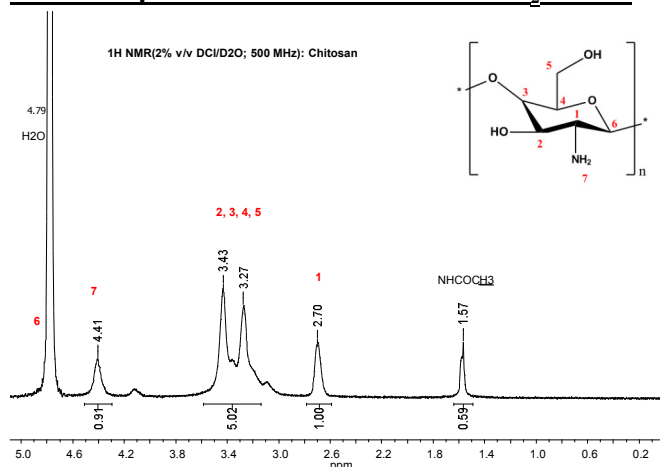
Characterization:

The product was analyzed by proton NMR and FT-IR spectroscopies. The degree of -SH functionalization was calculated from elemental analysis data.

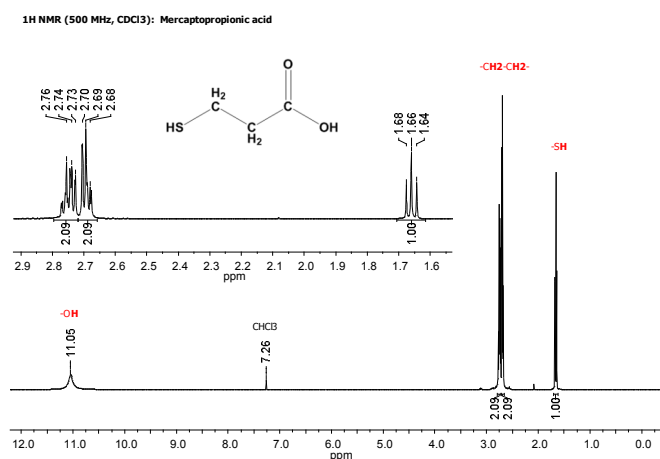
Solubility:

The polymer is soluble in water and aqueous hydrochloric acid solution, and has a limited solubility in DMSO.

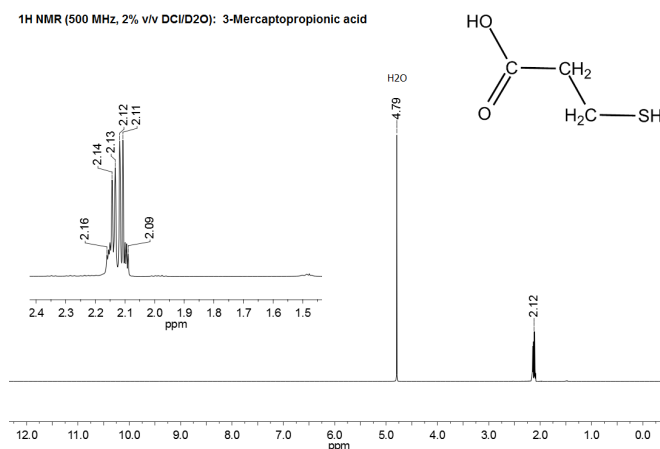
¹H NMR spectrum of native chitosan in D₂O/DCI:



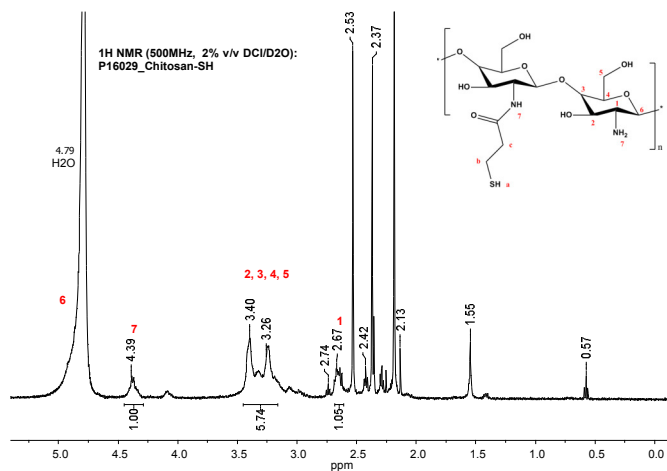
¹H NMR of 3-mercaptopropionic acid in CDCl₃:



¹H NMR of 3-mercaptopropionic acid in D₂O/DCI:



¹H NMR spectrum of P16029 in D₂O/DCI:

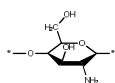


Elemental analysis of native chitosan :

Identification de l'échantillon: CHITOSAN
 Formule moléculaire: C₆ H₁₁ N O₄
 Méthode utilisée: 160531E

| Sample Name | % Nitrogen | % Carbon | % Hydrogen |
|-------------|------------|----------|------------|
| POL1-1 | 7,41 | 41,14 | 6,88 |
| POL1-2 | 7,31 | 41,12 | 6,88 |

| | % Nitrogen | % Carbon | % Hydrogen |
|----------|------------|----------|------------|
| Moyenne | 7,36 | 41,13 | 6,88 |
| Théorie: | 8,70 | 44,70 | 6,90 |



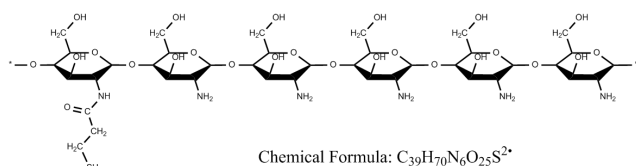
Chemical Formula: C₆H₁₁NO₄²⁺
 Elemental Analysis: C, 44,72; H, 6,88; N, 8,69; O, 39,71

Elemental analysis of P16029 product:

Identification de l'échantillon: P16029
 Formule moléculaire: (C₉ H₁₅ N O₅ S)_n
 Méthode utilisée: 160611E

| Sample Name | % Nitrogen | % Carbon | % Hydrogen | % Sulphur |
|-------------|------------|----------|------------|-----------|
| POL6-1 | 5.43 | 31.78 | 5.01 | 2.26 |
| POL6-2 | 6.63 | 36.57 | 6.00 | 3.54 |

| | % Nitrogen | % Carbon | % Hydrogen | % Sulphur |
|----------|-------------|---------------|-------------|-------------|
| Moyenne | 5.43 - 6.63 | 31.78 - 36.57 | 5.01 - 6.00 | 2.26 - 3.54 |
| Théorie: | 5.62 | 43.36 | 6.07 | 12.86 |



Chemical Formula: C₃₉H₇₀N₆O₂₅S²⁺
 Elemental Analysis: C, 44,40; H, 6,69; N, 7,97; O, 37,91; S, 3,04