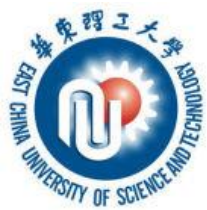


Spectroscopie de résonance magnétique nucléaire (RMN)

25-10-2023

Lecture 7



IV.8 Applications structurales et données de corrélations

Méthode générale d'analyse des spectres RMN ^{13}C

Tenir compte de l'information concernant la **multiplicité obtenue par un spectre « off-résonance » ou une DEPT.**

Utiliser les tables de corrélation pour prévoir les déplacements chimiques de tous les carbones pour chaque structure proposée

IV.7 Test « DEPT »













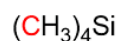
	CH_3	CH_2	CH	C
Découplage à large bande				
Un spectre DEPT-90				
Un spectre DEPT-135				

Table des déplacements chimique de ^{13}C

Approximate Values of Chemical Shifts for ^{13}C NMR



0



8 - 30



15 - 55



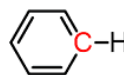
20 - 60



30 - 50



100 - 150



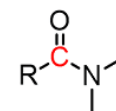
110 - 170



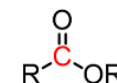
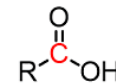
150 - 170



110 - 140



155 - 185



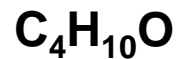
165 - 185



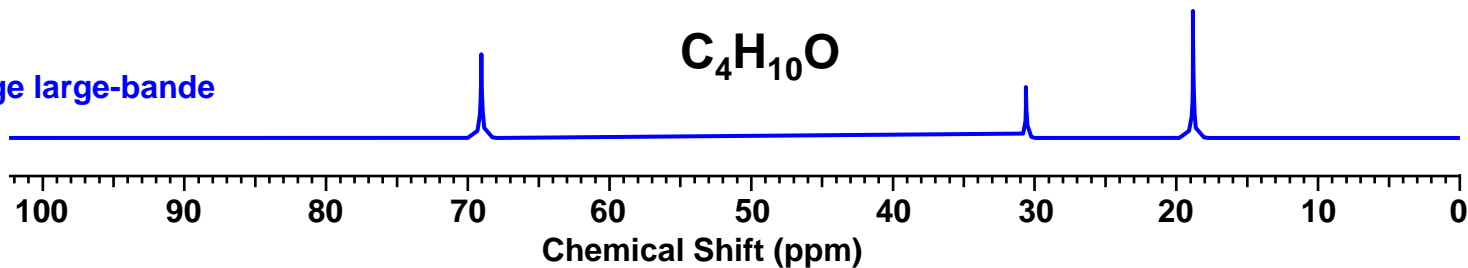
190 - 220

IV.7 Test « DEPT »

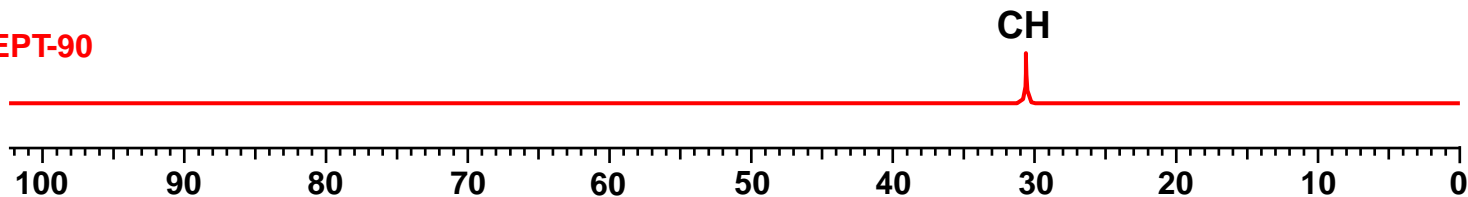
Exemple



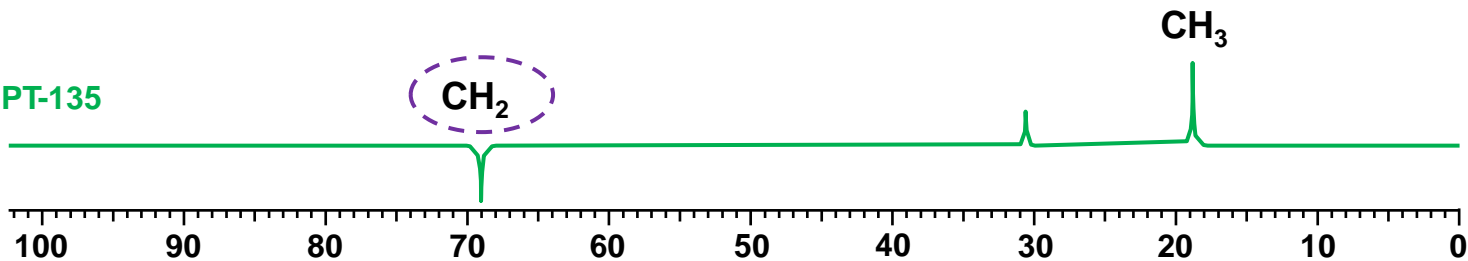
Découplage large-bande



DEPT-90

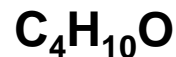


DEPT-135

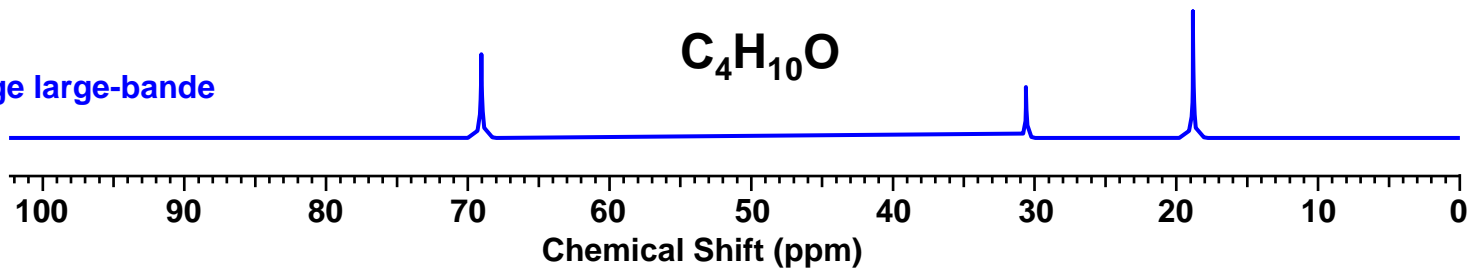


IV.7 Test « DEPT »

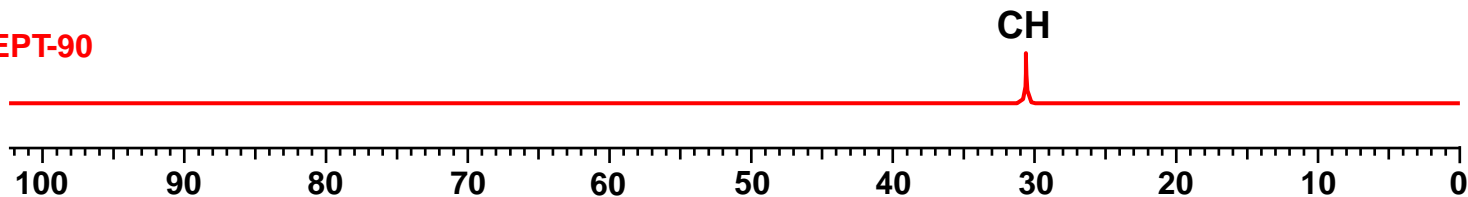
Exemple



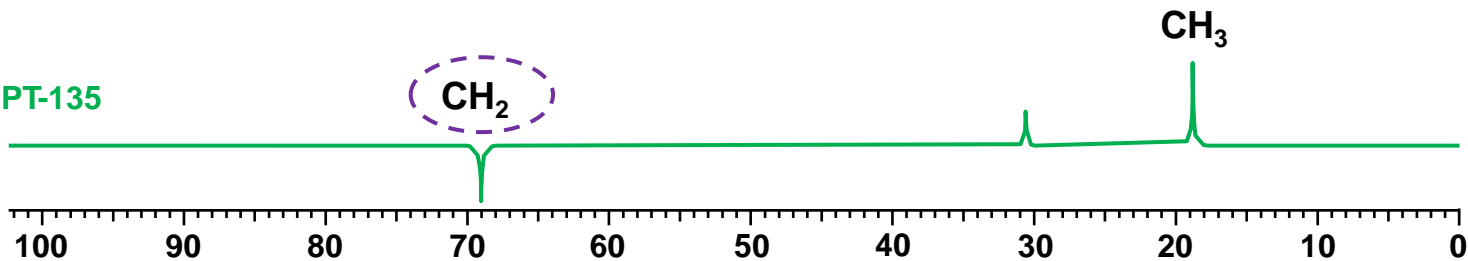
Découplage large-bande



DEPT-90



DEPT-135




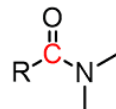
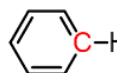
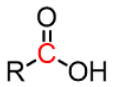
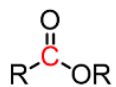
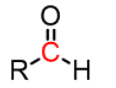
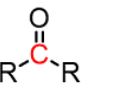
3 C et 6 H

Il manque 1 C 4 H

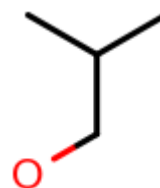
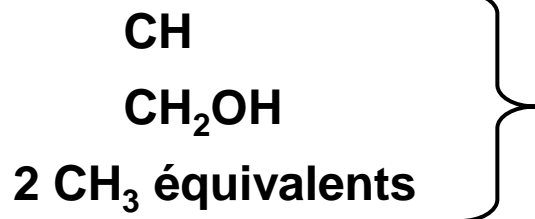
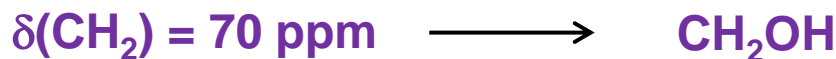
Comme il n'y a qu'un carbone supplémentaire, cela signifie qu'il y a un CH_3 additionnel plus un proton non attribué \Rightarrow ce dernier ne peut qu'être localisé sur l'oxygène : OH

IV.8 Applications structurales et données de corrélations

Approximate Values of Chemical Shifts for ^{13}C NMR

$(\text{CH}_3)_4\text{Si}$	0	* $\text{I}-\text{C}$	-20 - 40		100 - 150		155 - 185
$\text{R}-\text{CH}_3$	8 - 30	$\text{Br}-\text{C}$	25 - 65		110 - 170		165 - 185
$\text{R}-\text{CH}_2-\text{R}$	15 - 55	$\text{Cl}-\text{C}$	35 - 80	$\text{C}=\text{N}$	150 - 170		165 - 185
R_3CH	20 - 60	$\text{O}-\text{C}$	40 - 80	$\text{C}\equiv\text{N}$	110 - 140		190 - 220
R_4C	30 - 50	$\text{N}-\text{C}$	30 - 65				190 - 220

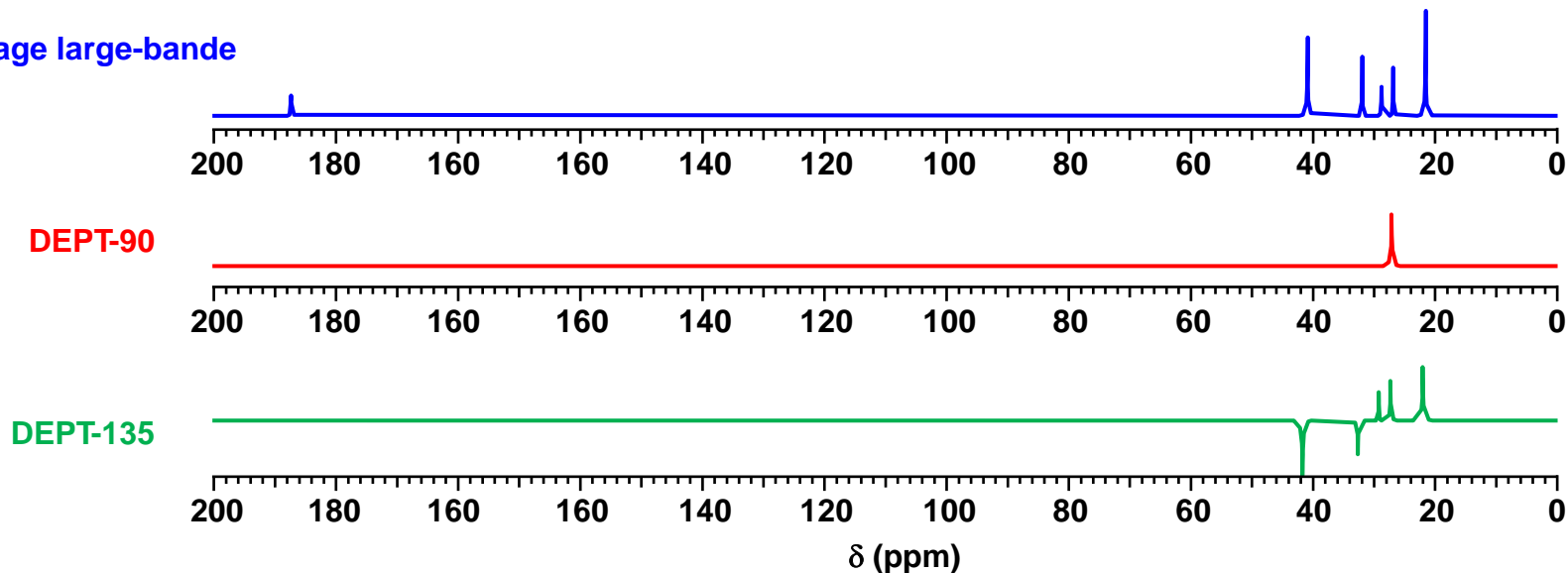
Le déplacement chimique de C-O (40 - 80 ppm) confirme la présence d'un groupe alcool **OH**



Un autre DEPT...



Découplage large-bande



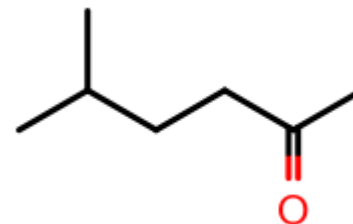
Il existe plusieurs structures qui peuvent correspondre à ces spectres.

➡ Nécessite un spectre H^1 NMR

Celui-ci donne 5 signaux dont:

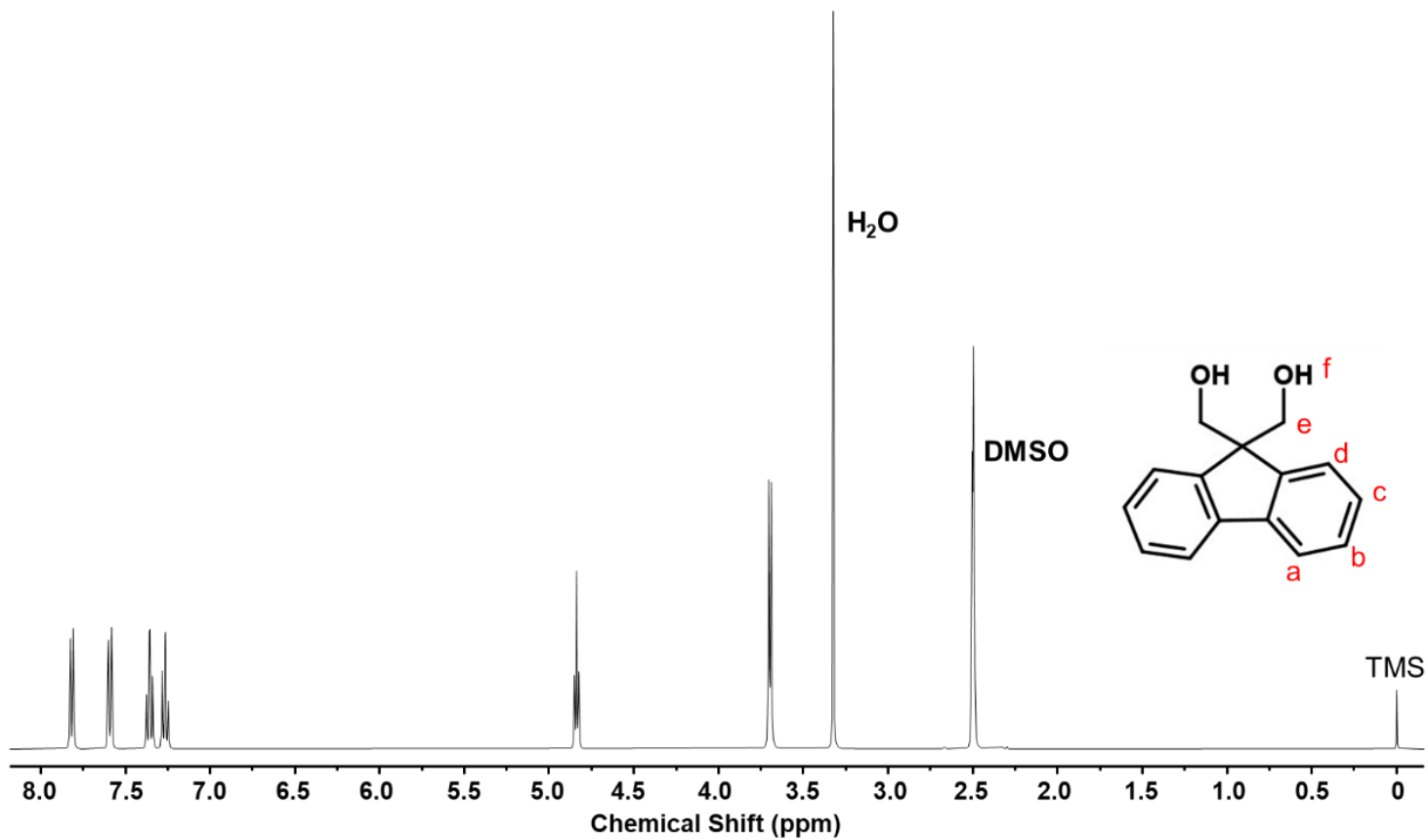
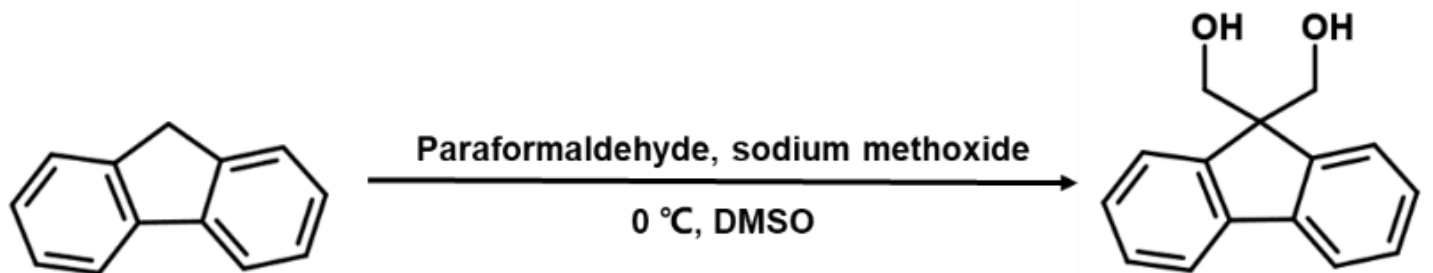
0.9 δ (6H, doublet)

1.9 δ (3H, singlet)

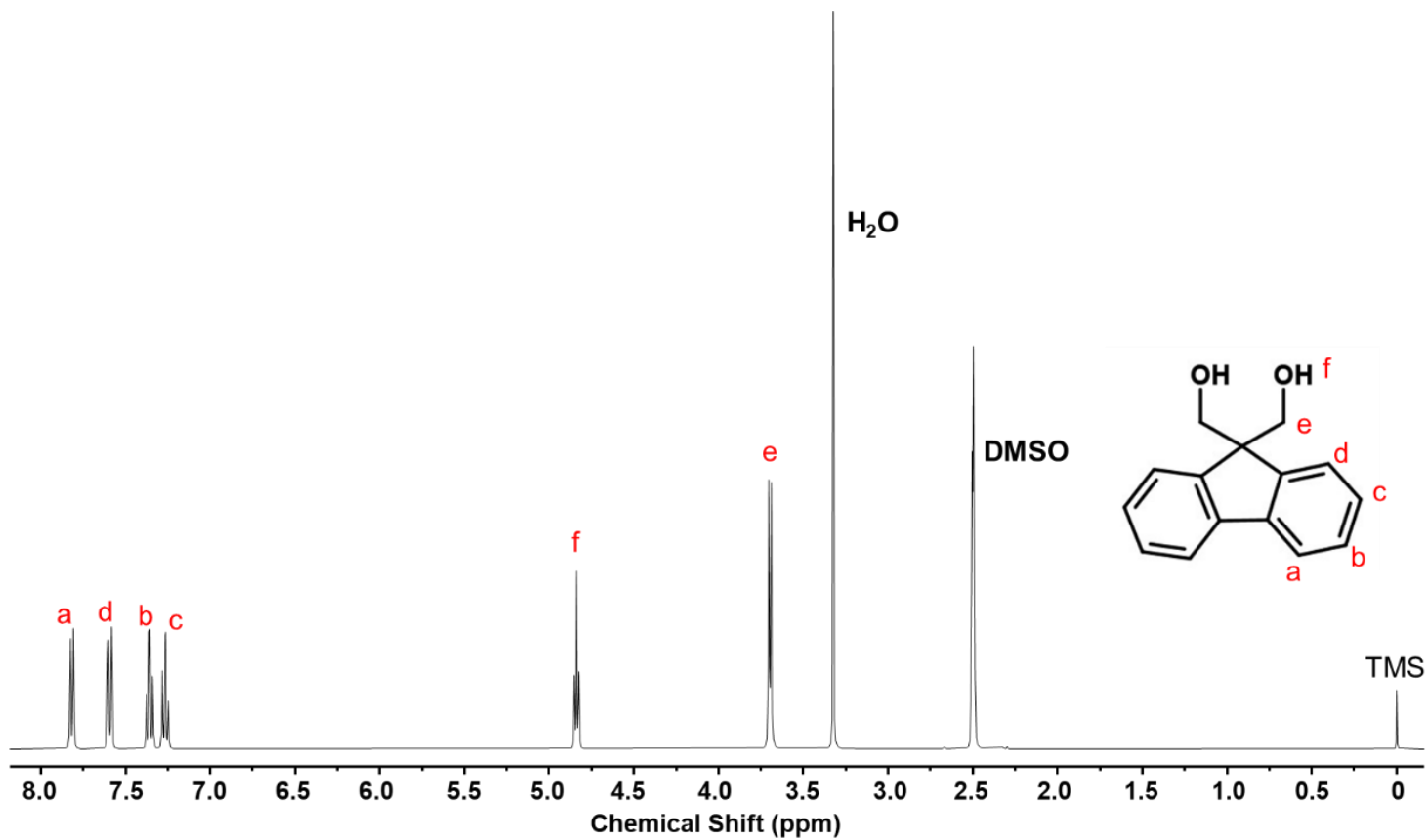
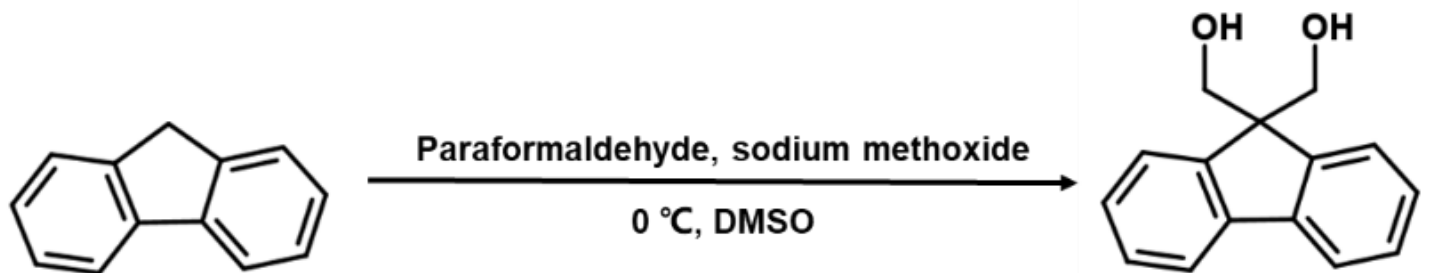


5 methyl-2-hexanone

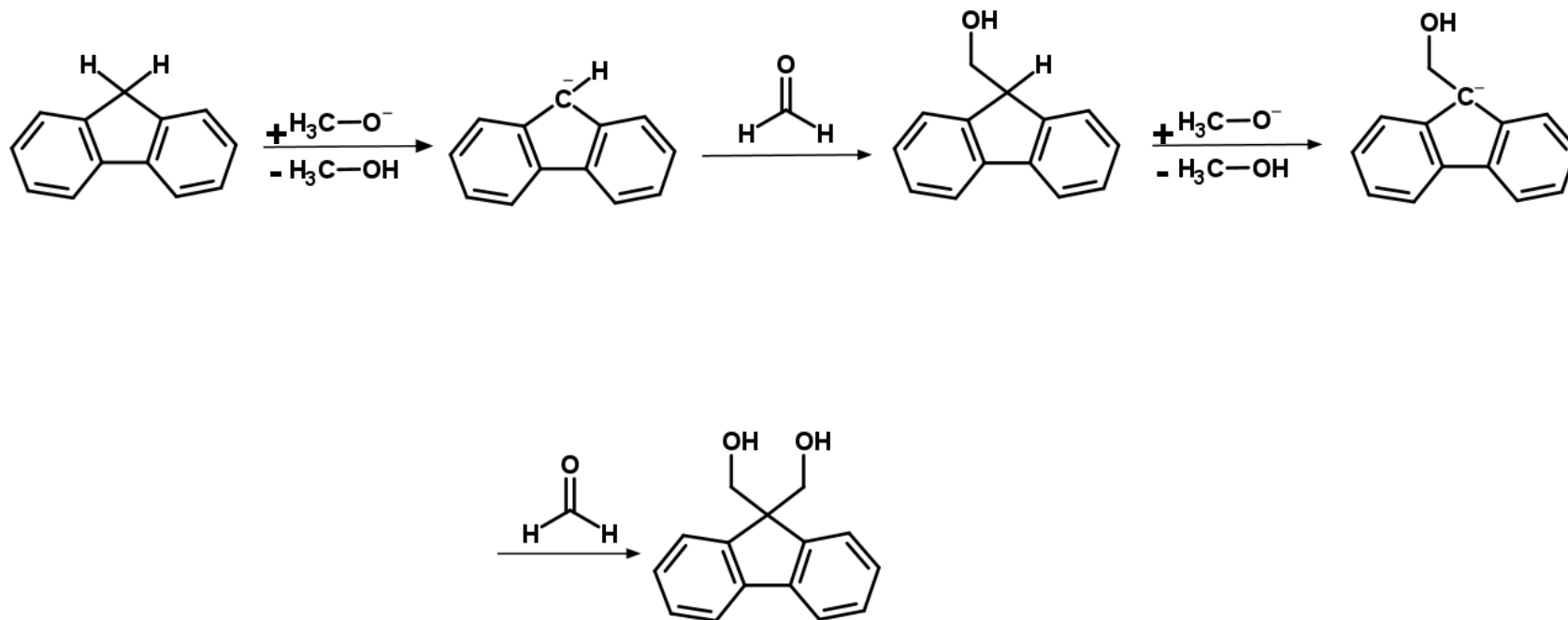
Histoire Vraie...



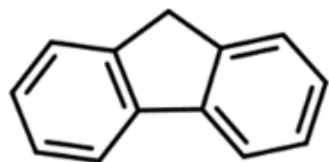
Histoire Vraie...



Mécanisme de la réaction

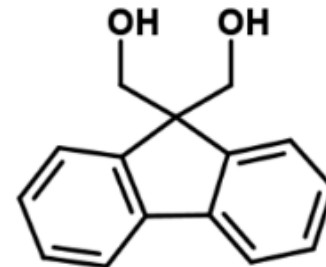


Histoire Vraie...

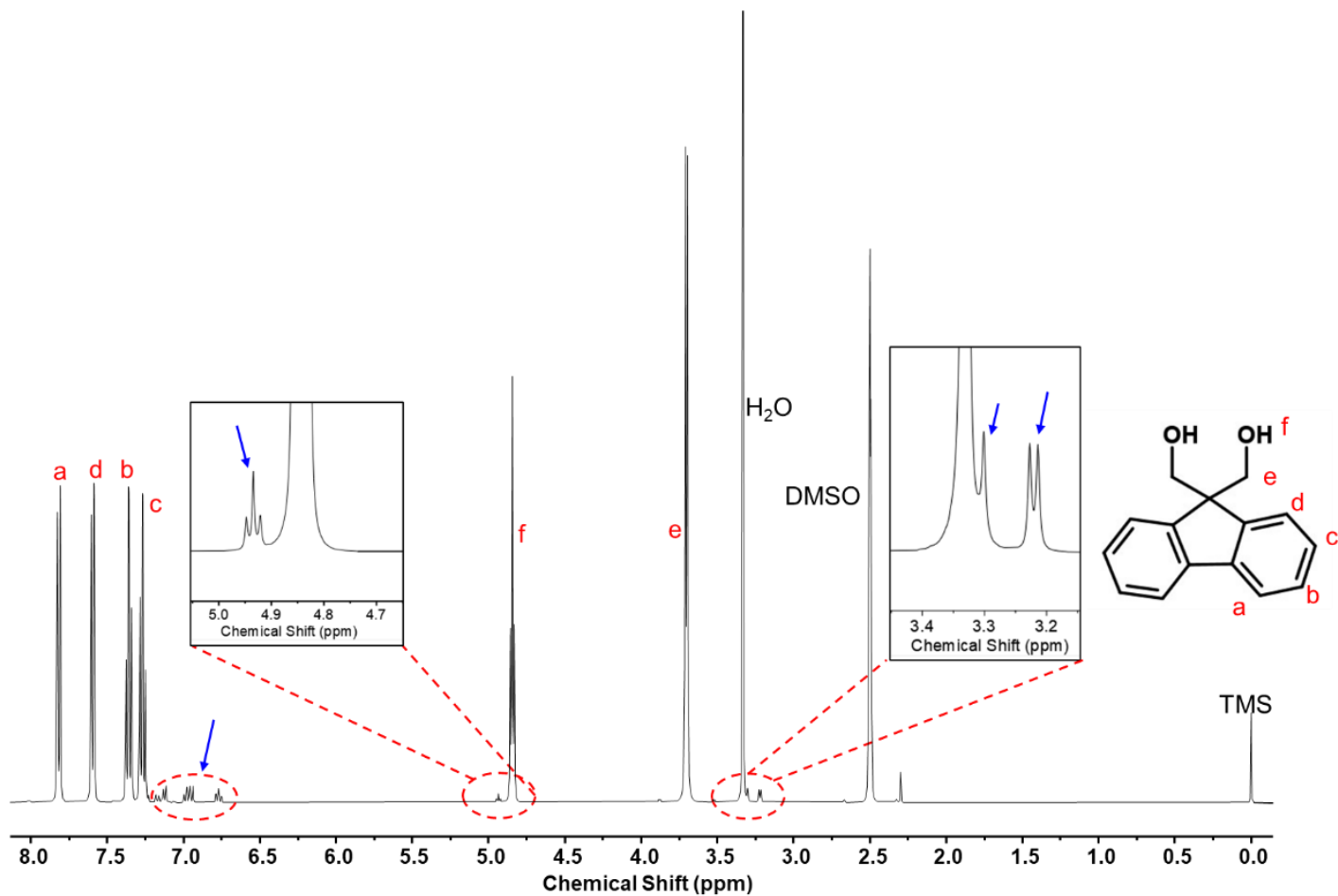


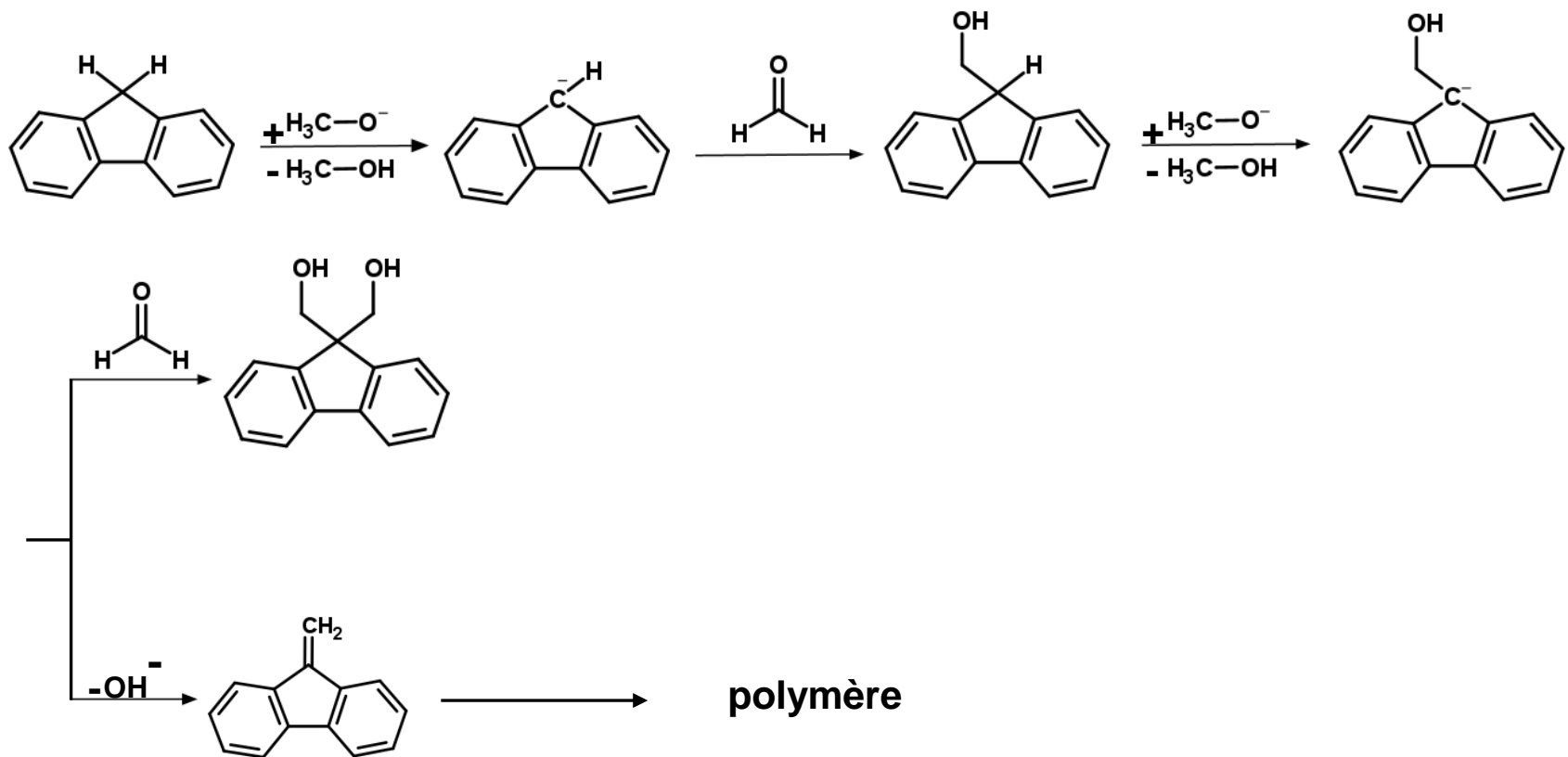
Paraformaldehyde, sodium methoxide

0 °C, DMSO

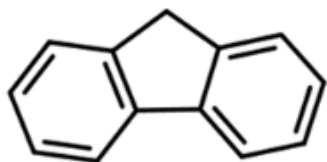


+ impureté



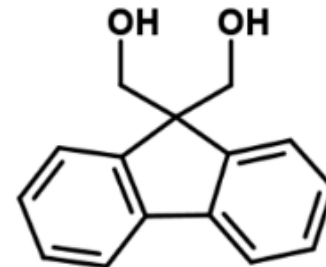


Histoire Vraie...

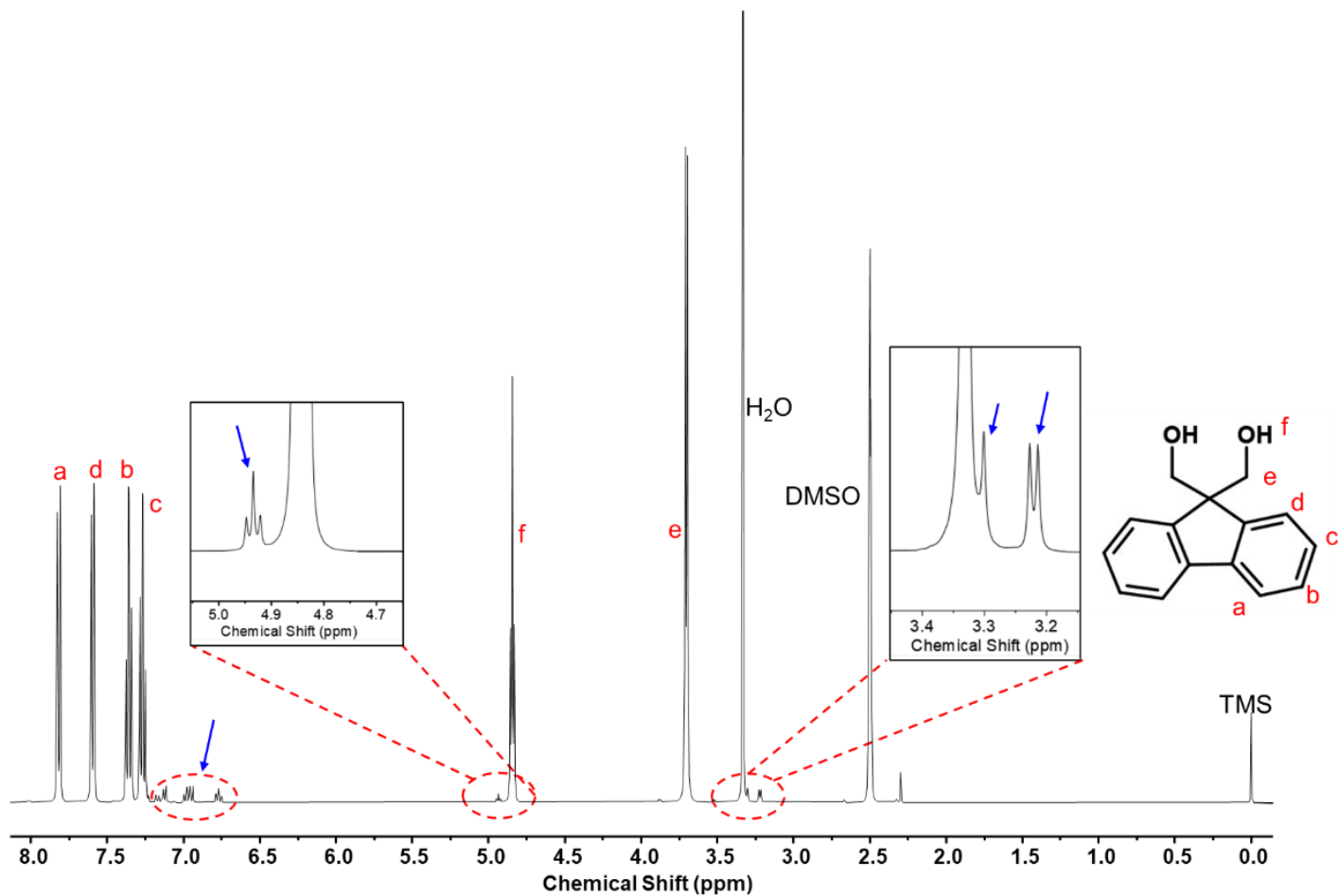


Paraformaldehyde, sodium methoxide

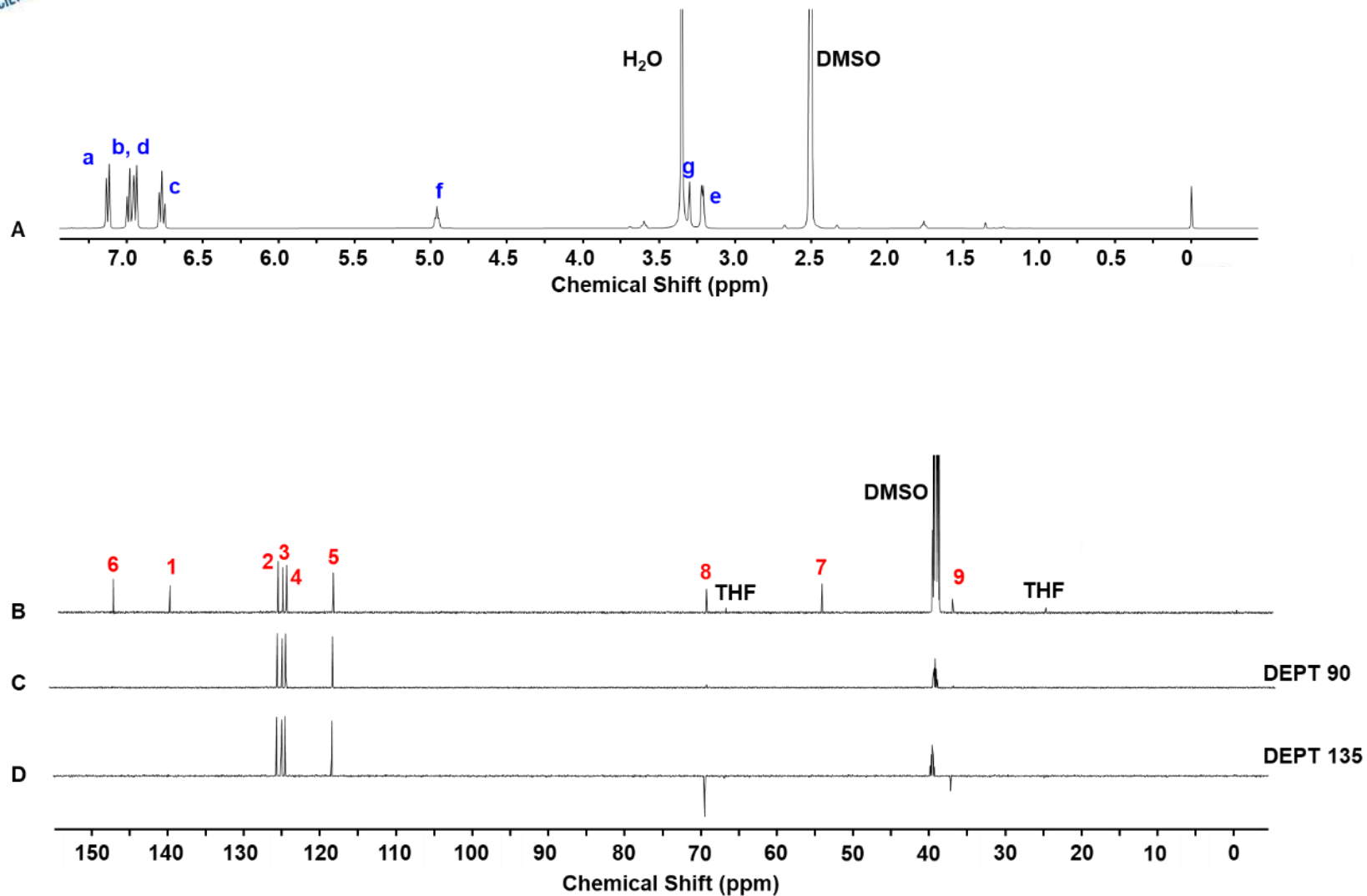
0 °C, DMSO



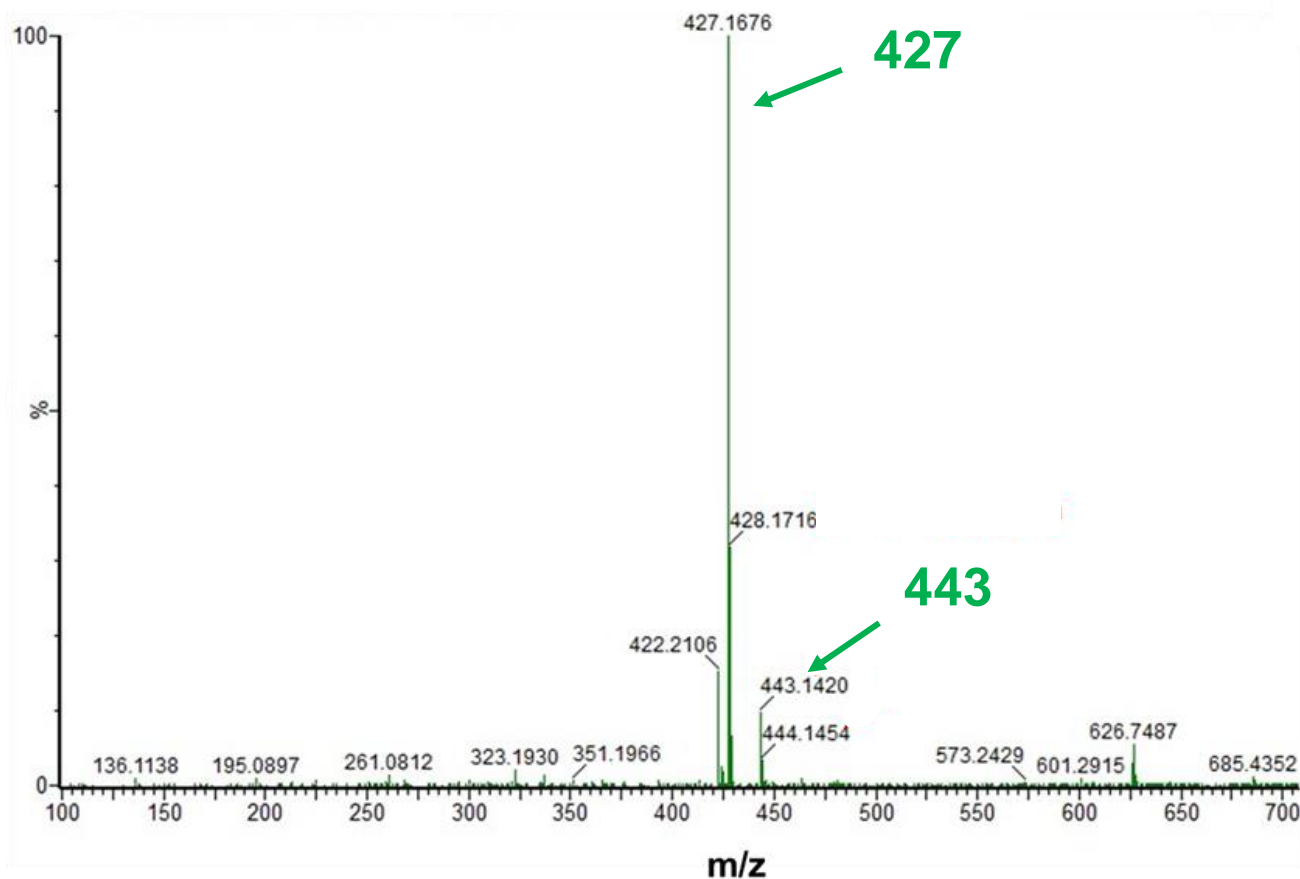
+ impureté



Spectres ^1H RMN et DEPT de l'impureté



Spectre de masse de l'impureté



L'échantillon est incorporé dans une matrice qui contient des ions sodium (**23 g/mol**) et potassium (**39 g/mol**)

La masse molaire de l'impureté est donc de $427 - 23 = 443 - 39 = 404$ g/mol