Synthesis and Optical Properties of

Trioxatriangulenium Dyes with One and Two

Peripheral Amino Substituents

Thomas J. Sørensen and Bo W. Laursen*

Nano-Science Center and Department of Chemistry, University of Copenhagen, Universitetsparken 5, DK-2100 København Ø, Denmark

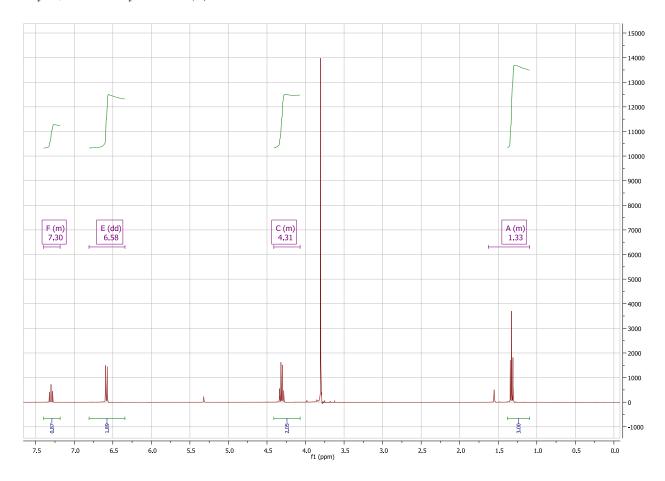
bwl@nano.ku.dk

Table of contents:

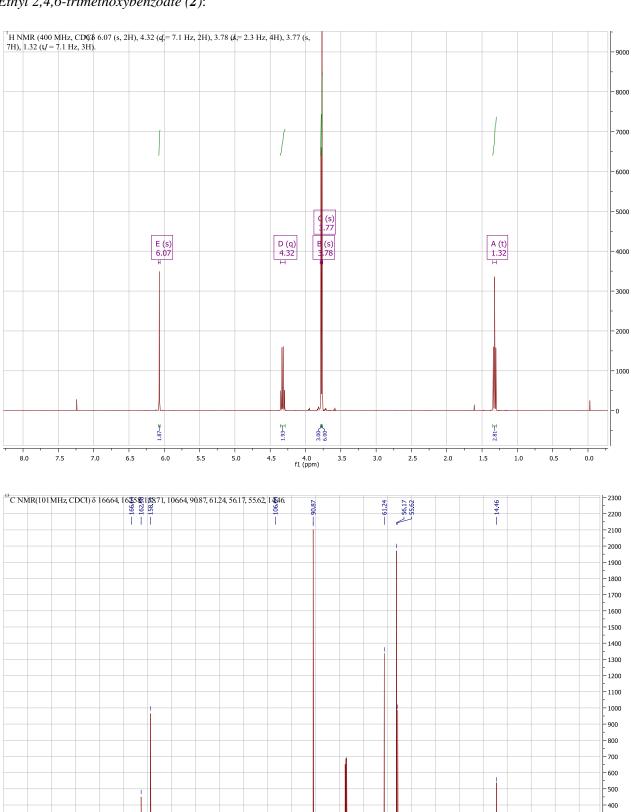
NMR spectra of the synthesized compounds	2
Compound 1, ¹ H NMR	2
Compound 2, ¹ H NMR & ¹³ C NMR	3
Compound 3 , ¹ H NMR & ¹³ C NMR	4
Compound 4, ¹ H NMR & ¹³ C NMR	5
Compound 5a , ¹ H NMR & ¹³ C NMR	6
Compound 5b , ¹ H NMR & ¹³ C NMR	7
Compound 5c , ¹ H NMR & ¹³ C NMR	8
Compound 6a , ¹ H NMR & ¹³ C NMR	9
Compound 6b , ¹ H NMR	10
Compound 6c , ¹ H NMR & ¹³ C NMR	11
Compound 7, ¹ H NMR & ¹³ C NMR	12
Compound 8a , ¹ H NMR	13
Compound 8b , ¹ H NMR & ¹³ C NMR	14
Compound 9a , ¹ H NMR & ¹³ C NMR	15
Compound 9b, none	17

NMR spectra of the synthesized compounds

Ethyl 2,6-dimethoxybenzoate (1):



Ethyl 2,4,6-trimethoxybenzoate (2):



100 90 f1 (ppm)

140

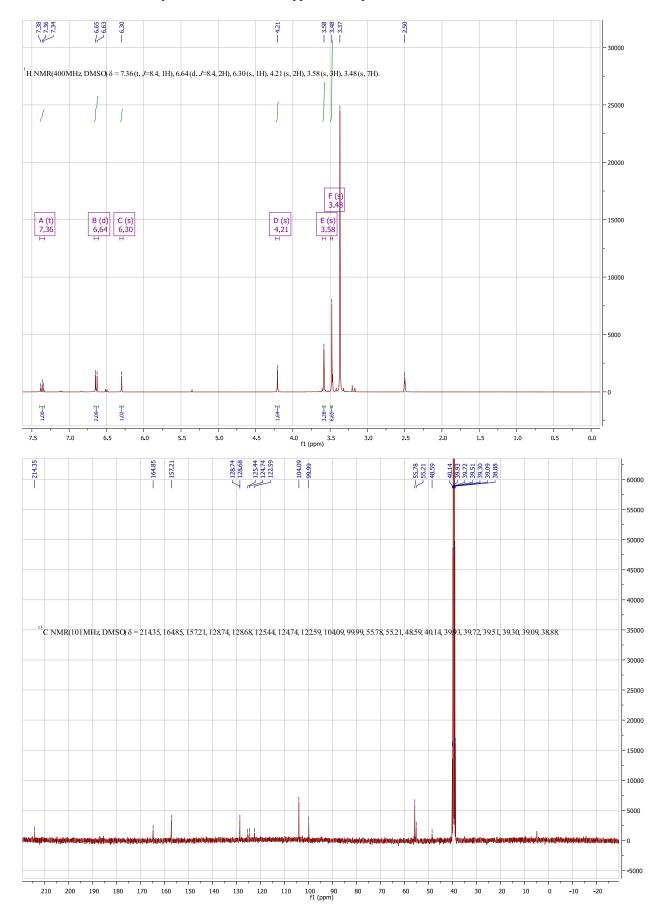
130

170 160 150

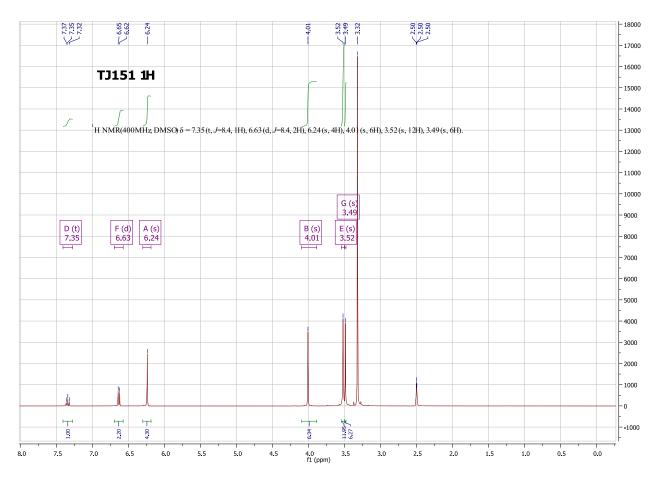
60

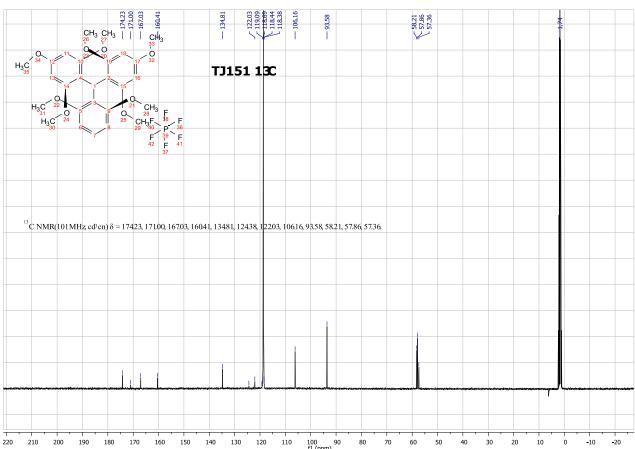
- 300 - 200 - 100 - 0 - -100

Bis(2,6-dimethoxyphenyl)-(2,4,6-trimethoxyphenyl)-methylium hexafluorophosphate (3): OBS. The compound is unstable in solution. The extra peaks in the aromatic region and the grass around 3.5 ppm is from the helicene formed by this reaction and will appear in all spectra recorded after ~5 min. after in solution.

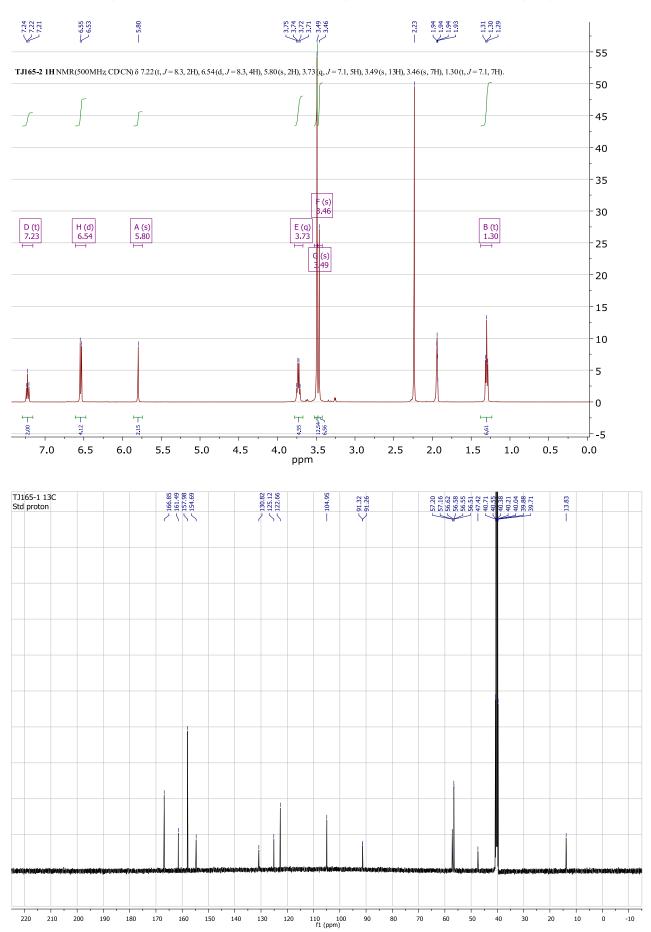


(2,6-dimethoxyphenyl)-bis(2,4,6-trimethoxyphenyl)-methylium hexafluorophosphate (4):

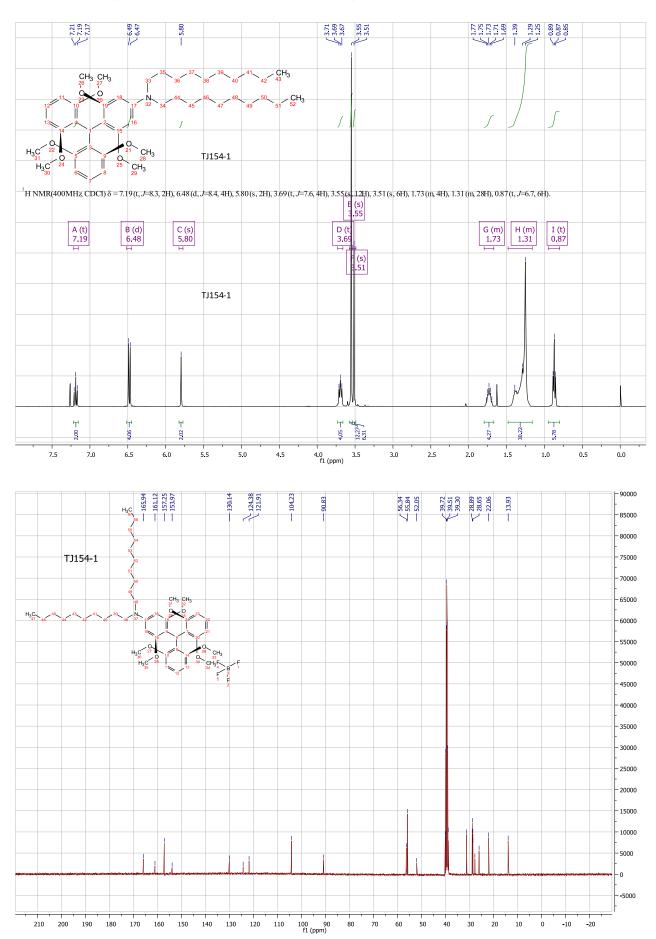




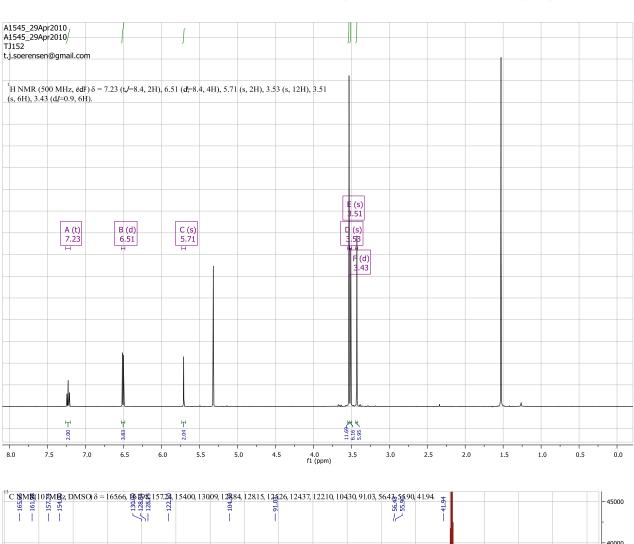
 $Bis(2,6-dimethoxyphenyl)-(4-diethylamino-2,6-trimethoxyphenyl)-methylium\ hexafluorophosphate\ ({\bf 5a}):$

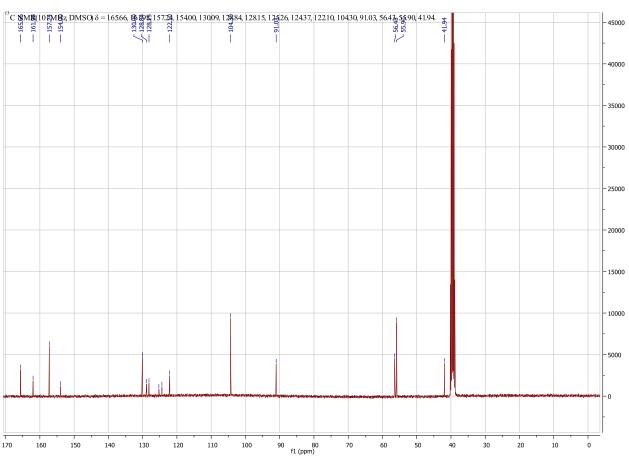


 $Bis(2,6-dimethoxyphenyl)-(4-didecylamino-2,6-trimethoxyphenyl)-methylium\ tetrafluoroborate\ ({\it 5b}):$

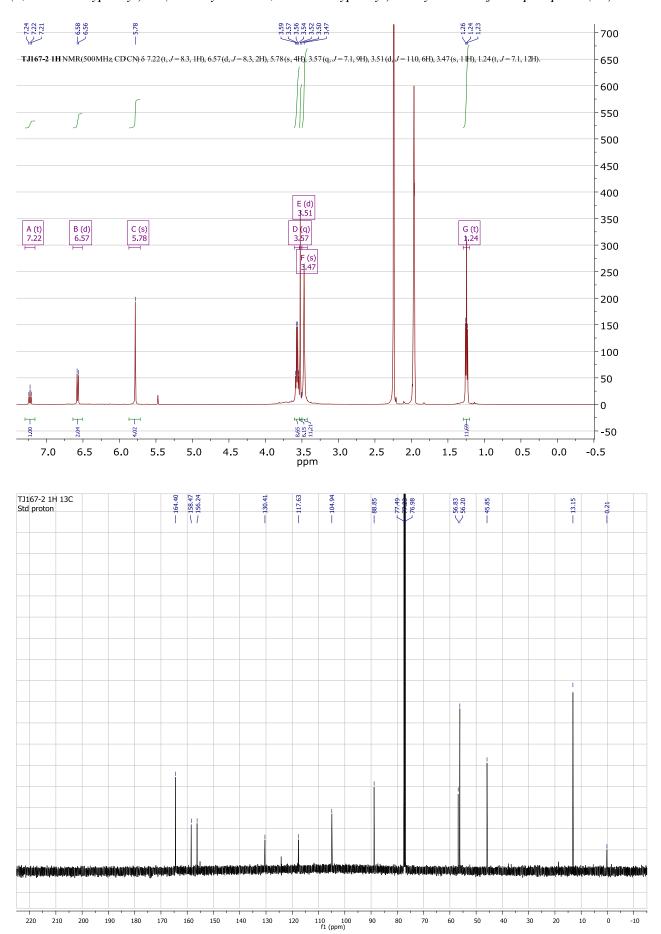


 $Bis(2,6-dimethoxyphenyl)-(4-dimethylamino-2,6-dimethoxyphenyl)-methylium\ hexafluorophosphate\ ({\it 5c}$):

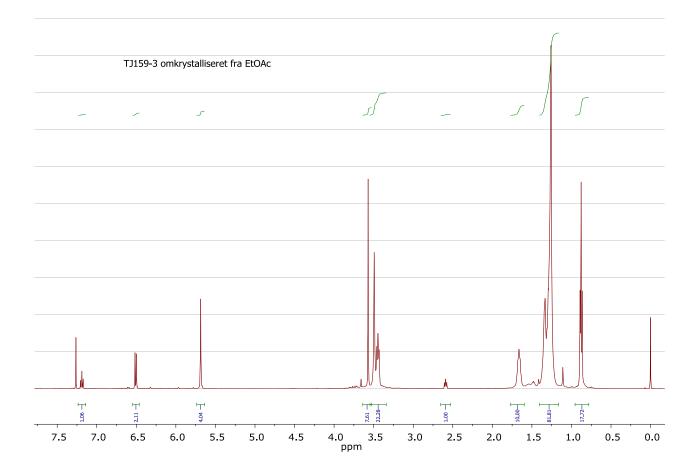




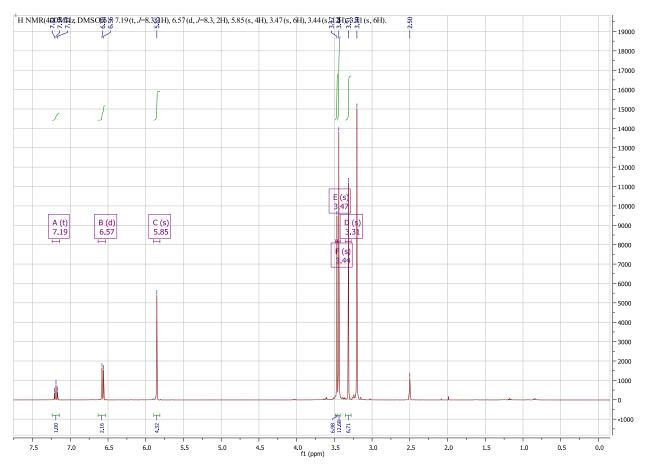
(2,6-dimethoxyphenyl)-bis(4-diethylamino-2,6-dimethoxyphenyl)-methylium hexafluorophosphate $(\mathbf{6a})$:

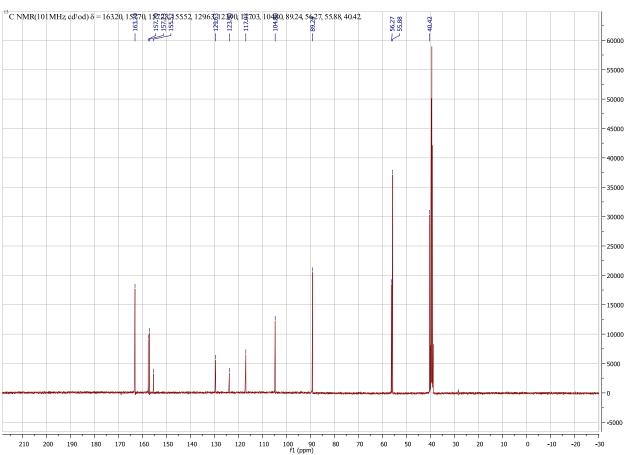


 $(2,6-dimethoxyphenyl)-bis (4-didecylamino-2,6-dimethoxyphenyl)-methylium\ hexafluorophosphate\ (\textbf{6b}):$ Co-crystallizes with didecylamine.

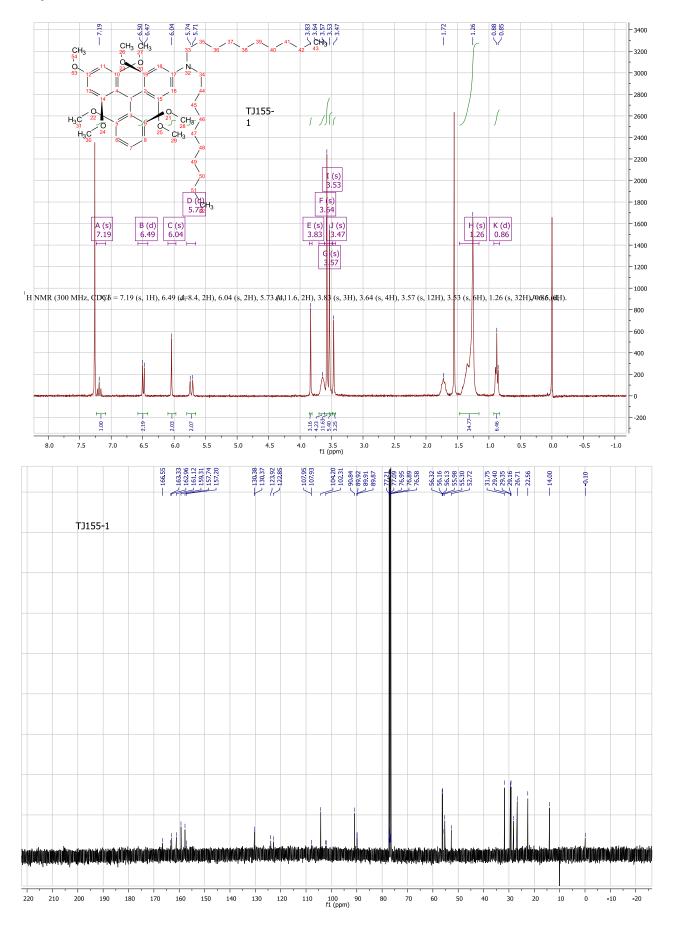


(2,6-dimethoxyphenyl)-bis(4-dimethylamino-2,6-dimethoxyphenyl)-methylium hexafluorophosphate $(\boldsymbol{6c})$:



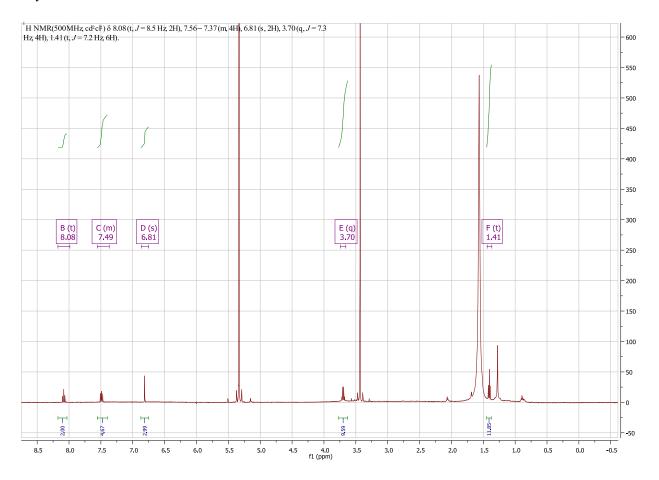


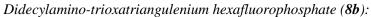
(2,6-dimethoxyphenyl)-(2,4,6-trimethoxyphenyl)- (4-didecylamino-2,6-trimethoxyphenyl)-methylium tetrafluoroborate (7):

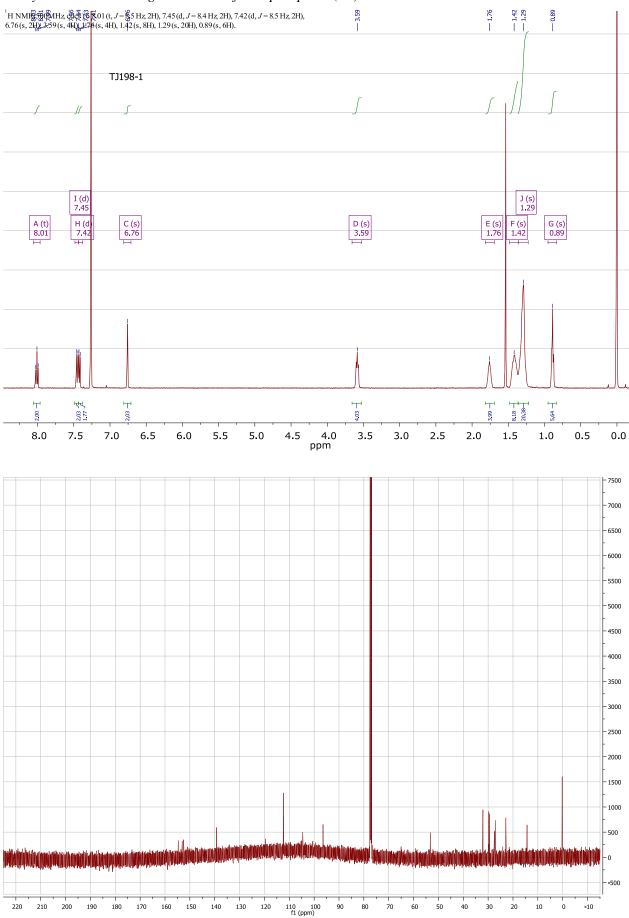


Diethylamino-trioxatriangulenium hexafluorophosphate (8a):

Only traces isolated!

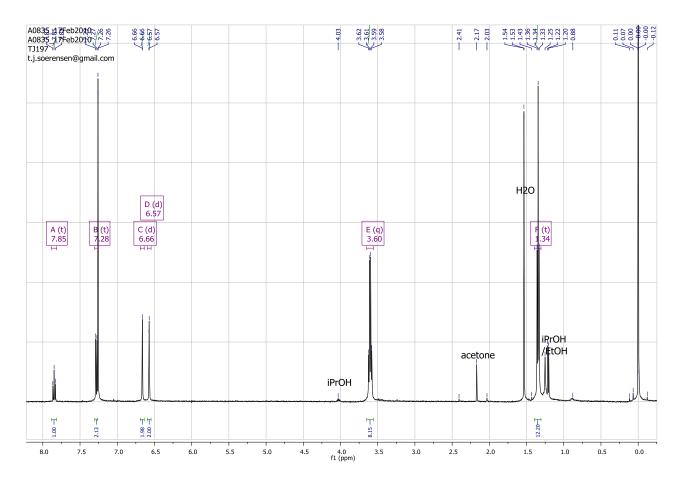




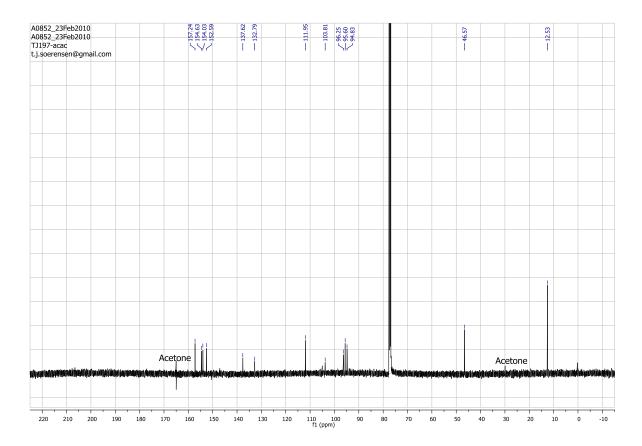


${\it Bis} (diethylamino) \hbox{-} triox atriangulenium\ hexafluorophosphate\ (\textbf{9a}) \hbox{:}$

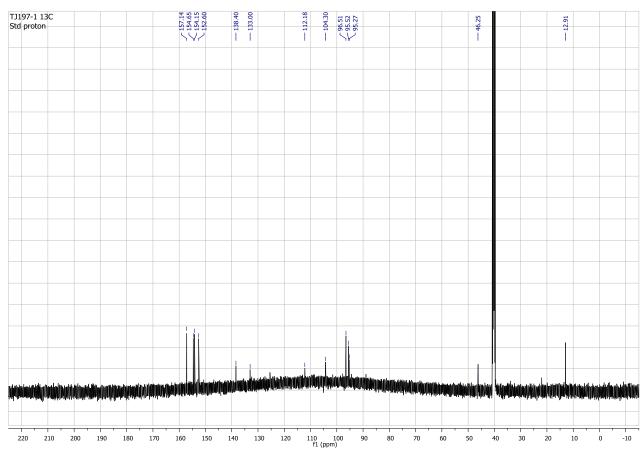
H NMR



C NMR with Cr(acac)₃



C NMR



 $Bis (didecy lamino) \text{-} triox atriangulenium\ hexafluorophosphate\ (\textbf{9b}):$

Only traces isolated!