Inclusion Complexes of EMPO Derivatives with 2,6-di-O-Methyl-β-Cyclodextrin: Synthesis, NMR and EPR Investigations for Enhanced Superoxide Detection

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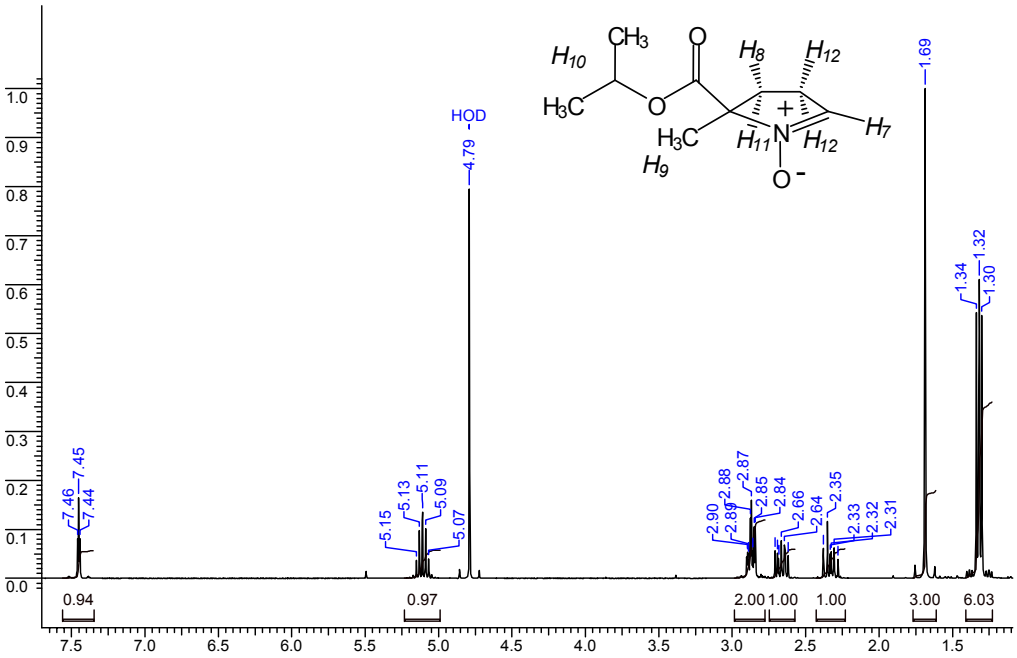
Yields in crude and isolated products for each compound issu from the four steps synthesis

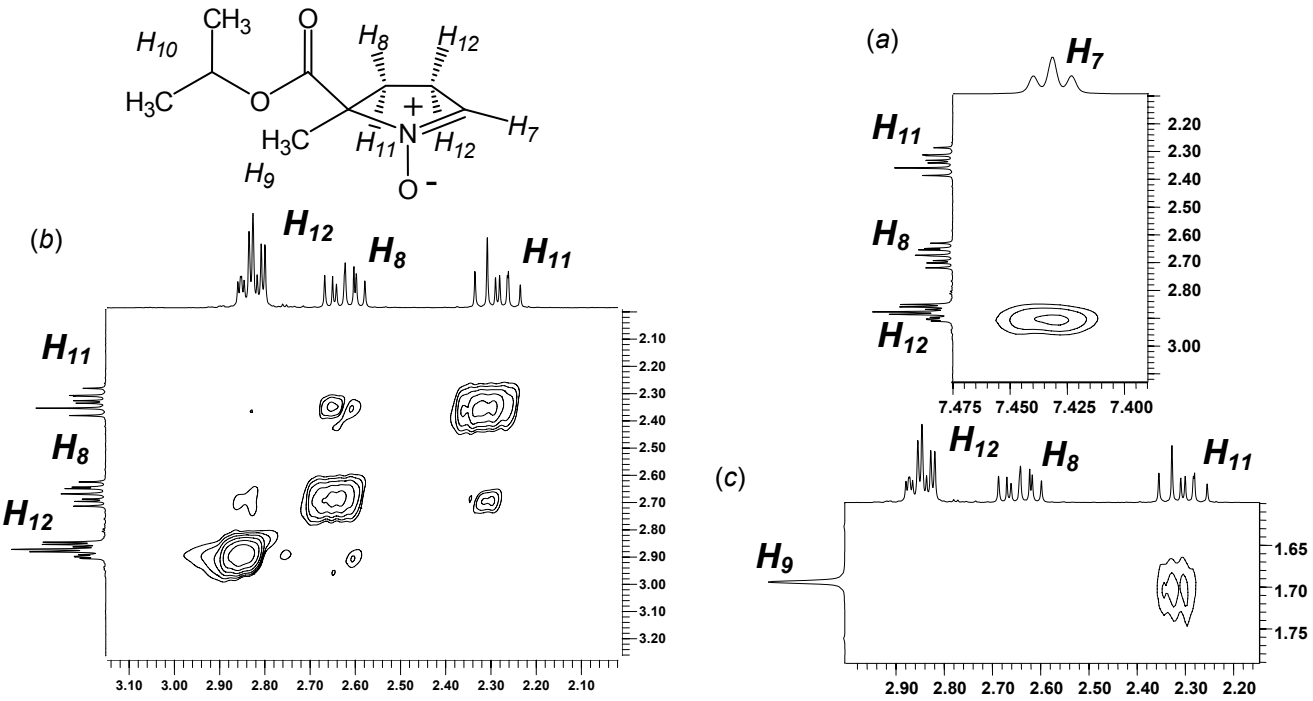
Nitrone	Yield step 1[a]	Yield step 2[b]	Yield step 3[a]	Yield step 4[b]
2d	62	84	53	20
4d	50	23	84	56
5d	78	58	88	33
6d	57	37	95	10
7d	73	43	73	14
8d	97	38	92	42
9d	66	60	97	9

[a] : crude yield.
[b] : purified yield.

Example of a ^{1}H NMR spectrum of a nitrone compound in D_2O at 300 MHz (*i*PrMPO 4d).

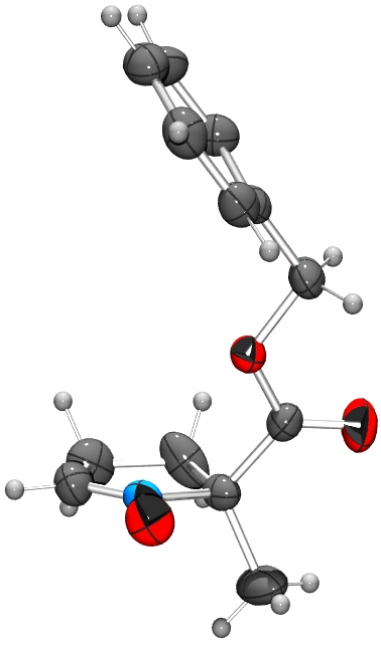


Parts of 2D-NOESY spectrum of *i*PrMPO 4d in D_2O for the attribution of pyrrolidine cycle protons.

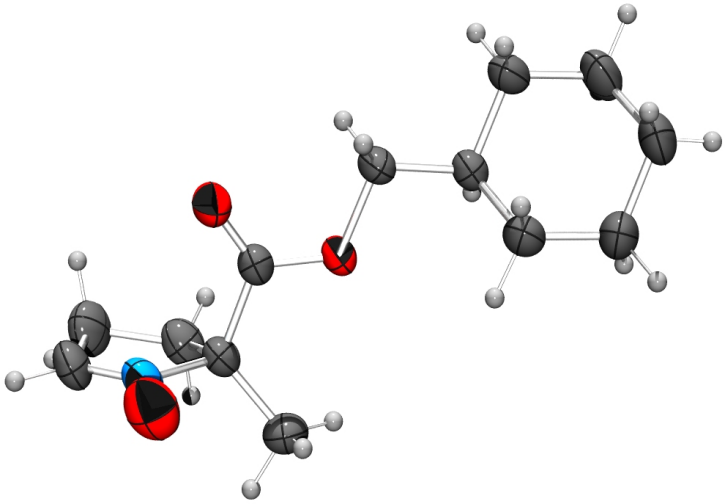


The nitronyl proton H7 (*a*) resonating nearly at 7 ppm helped in determining the signals of its nearest neighbours H12 (integration = 2). Indeed, H7 exhibited a single correlation with the third multiplet at 2.85 ppm. On the other hand, the observation of a unique correlation between the methyl group H9 and the first multiplet allowed to discriminate between the second (2.62 ppm) and the first (2.31) multiplet as H11 (*c*). Then, the observation of a last group of correlations in the region 2-3 ppm allowed the non ambiguous characterization of the H8 proton NMR signal (*b*).

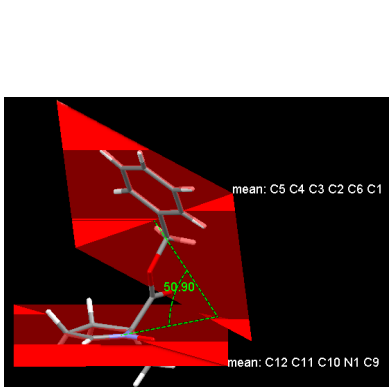
Crystal structure determination of BnMPO 6d

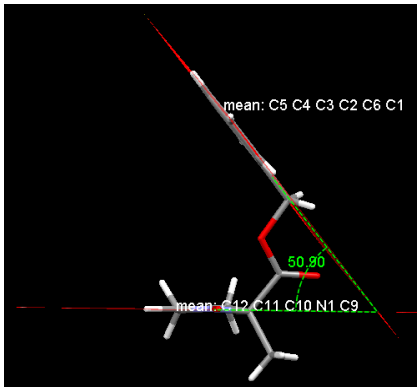


Crystal structure determination of CMMPO 9d

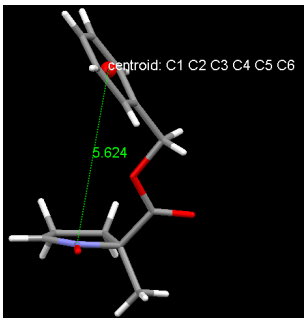


Angle between the two 6-membered and 5-membered rings of nitrone BnMPO 6d in the solid state.

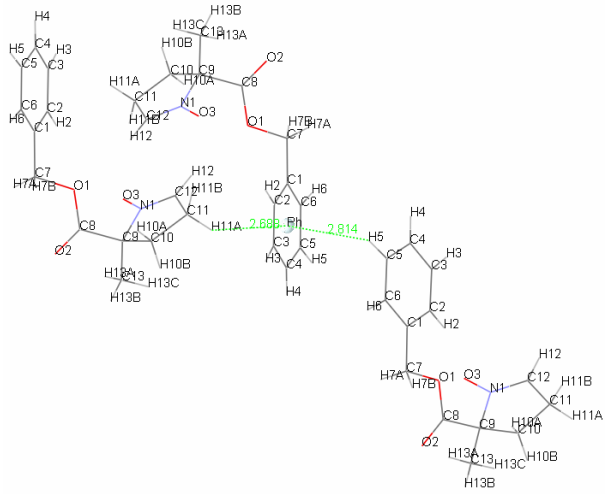




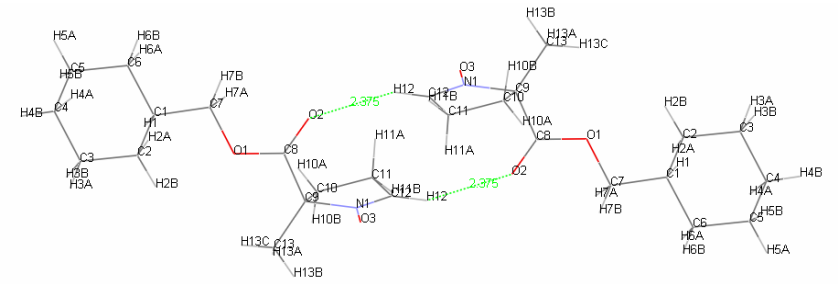
Distance between the center of the phenyl ring and the positively charged nitrogen atom of nitrone BnMPO 6d in the solid state.



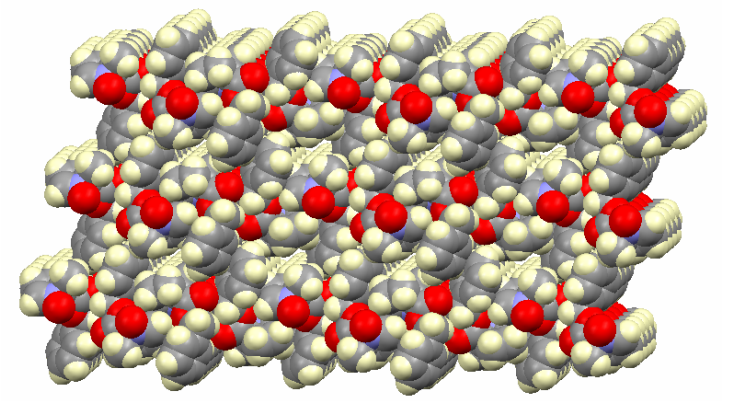
Structural features and stabilizing interactions of nitrones BnMPO 6d in the solid state.



Structural features with opposite disposition of cyclohexyl groups of nitrones CMMPO 9d in the solid state.

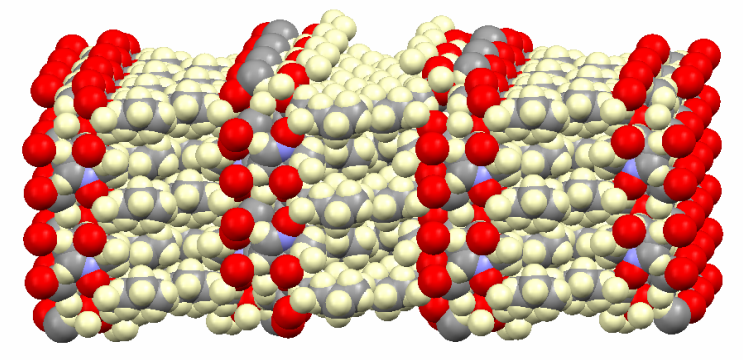


Periodical organization of the yin-yang motif formed by two BnMPO 6d nitrones in the solid state.
(see text)



Hydrophobic and hydrophilic sheets formed by the periodical organization of nitrone CMMPO 9d in the solid state.

(see text)



Thickness of both hydrophobic and hydrophilic sheets of the nitrone CMMPO 9d in the solid state.