

A Total Synthesis of (+)-Negamycin through Isoxazolidine Allylation

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and Patcharaporn Sae-Lao**

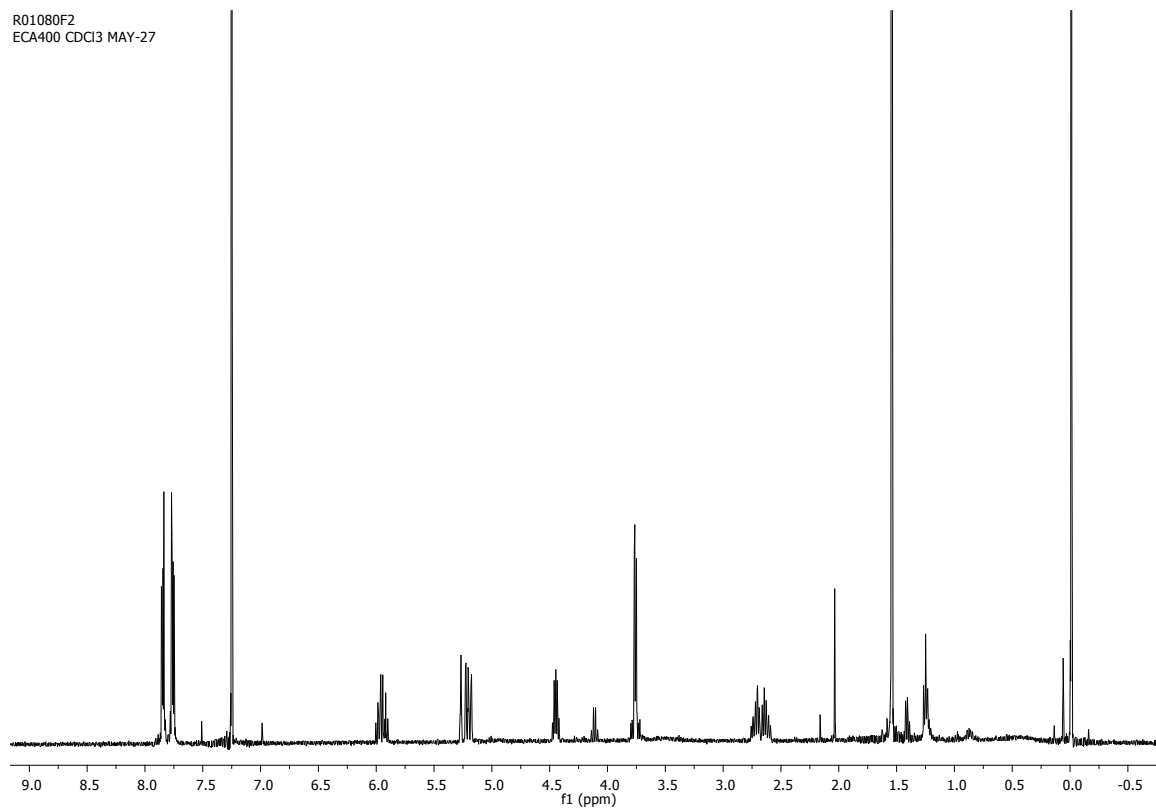
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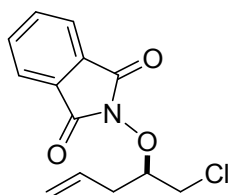
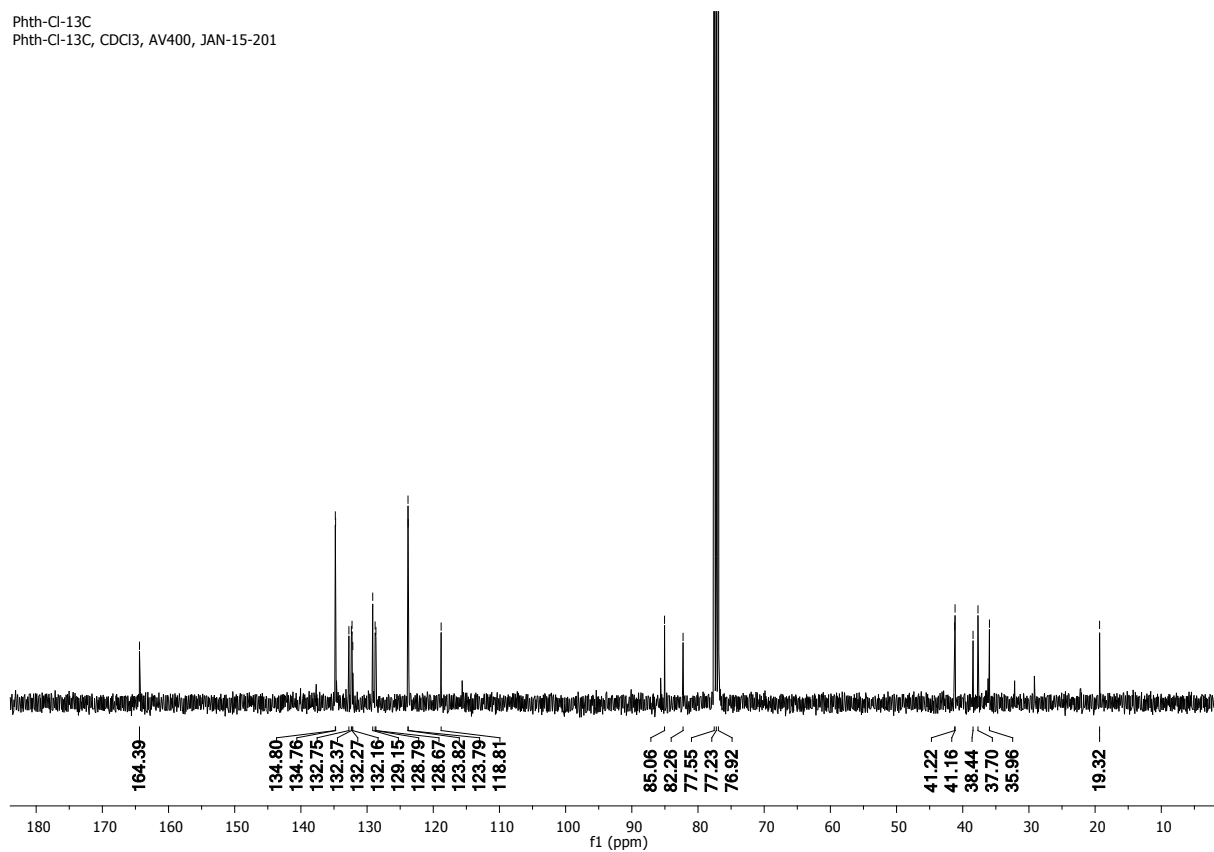
¹H and ¹³C NMR spectra. All spectra were recorded in CDCl₃ unless stated otherwise.

compound	page
phthalimide 3	2
hydroxylamine 4	3
hydroxylamine 5	4
methoxyisoxazolidine 6a	5
allyl isoxazolidine 7a	6
carboxylic acid 9	7
hydrazide 11	8
azide 12	9
negamycin 1	10
silyl ether 2b	11
hydroxylamine 3b	12
hydroxylamine 4b	13
hydroxylamine 5b	14
isoxazolidines 6b	15
allyl isoxazolidine 7b	17

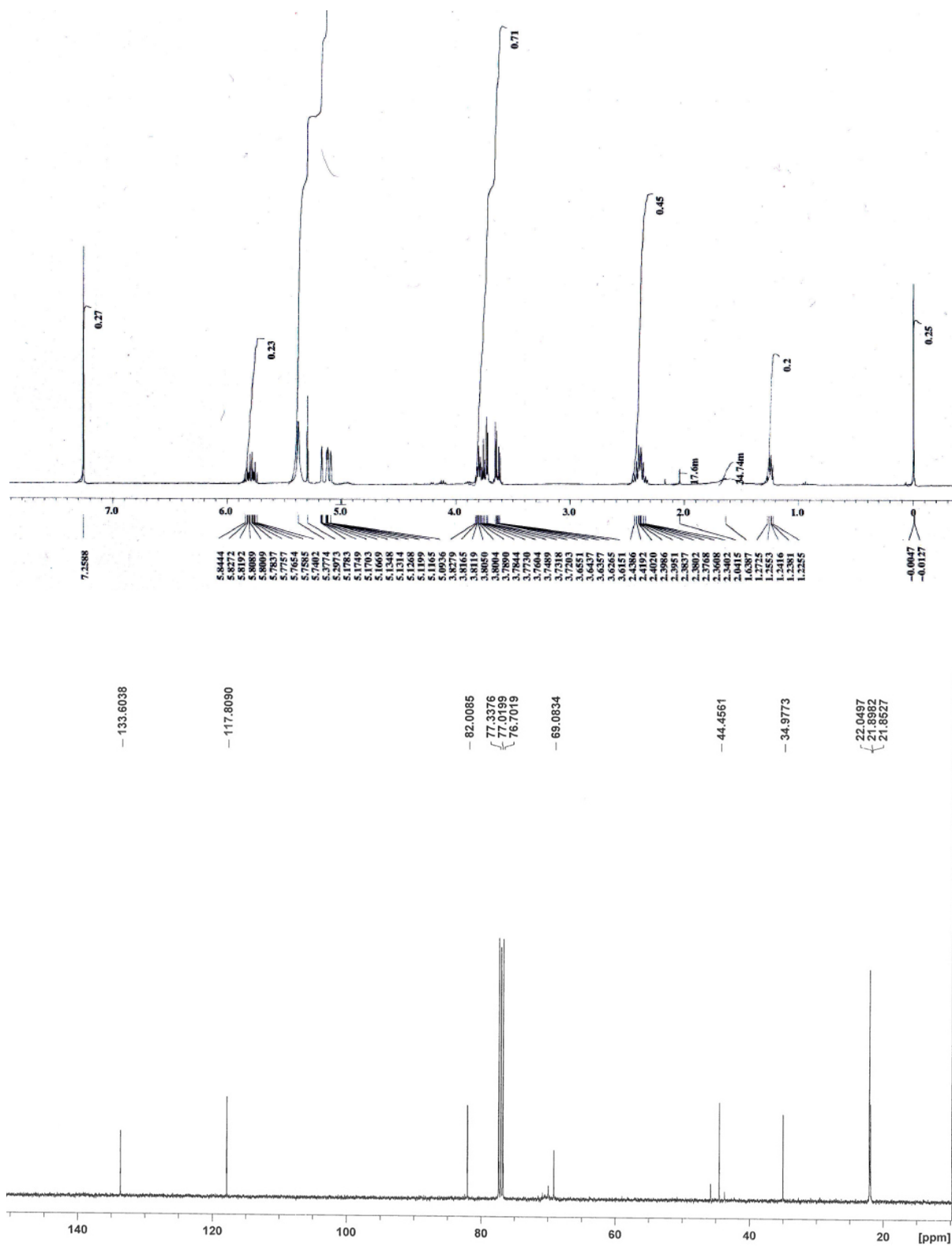
R01080F2
ECA400 CDCl3 MAY-27



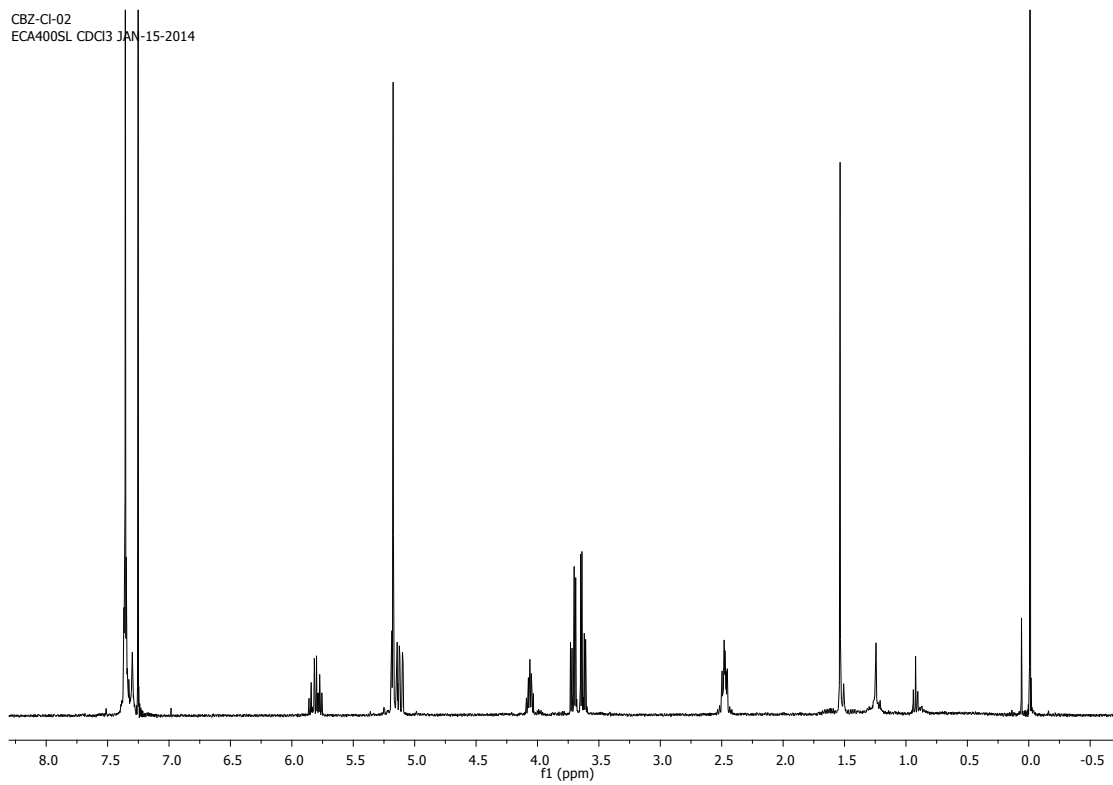
Phth-Cl-13C
Phth-Cl-13C, CDCl₃, AV400, JAN-15-201



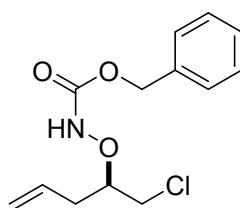
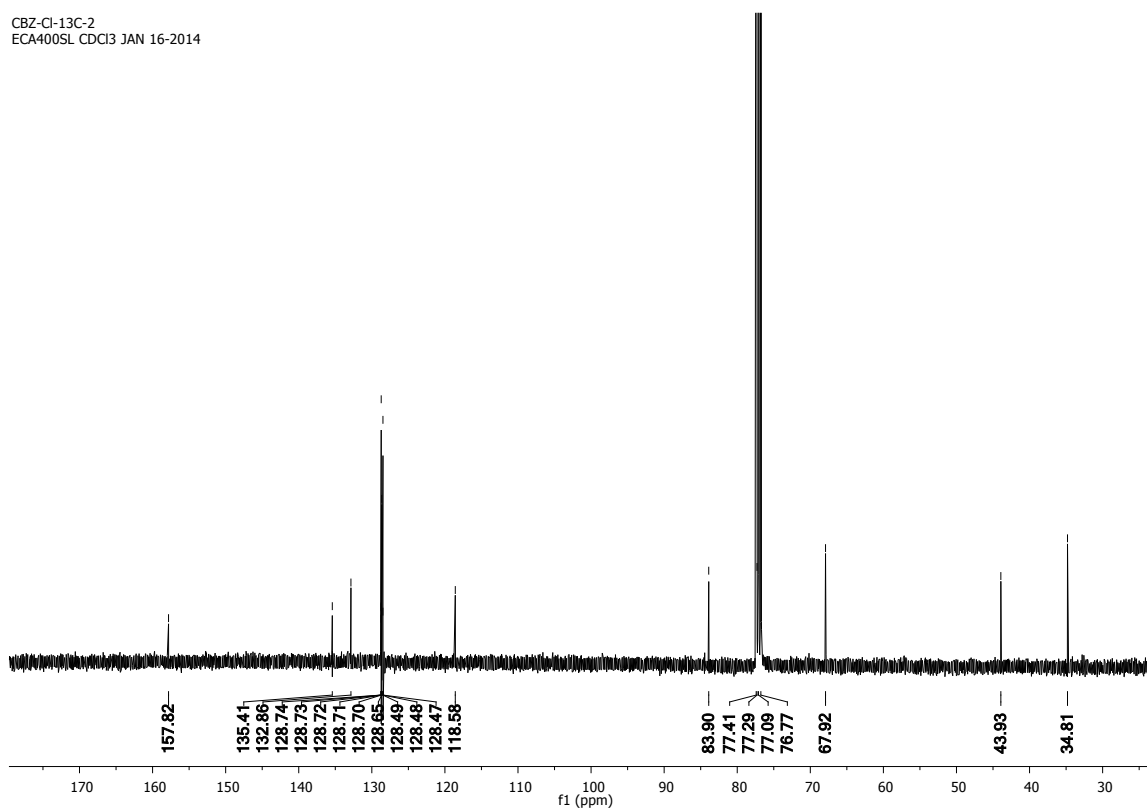
phthalimide 3



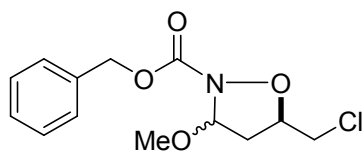
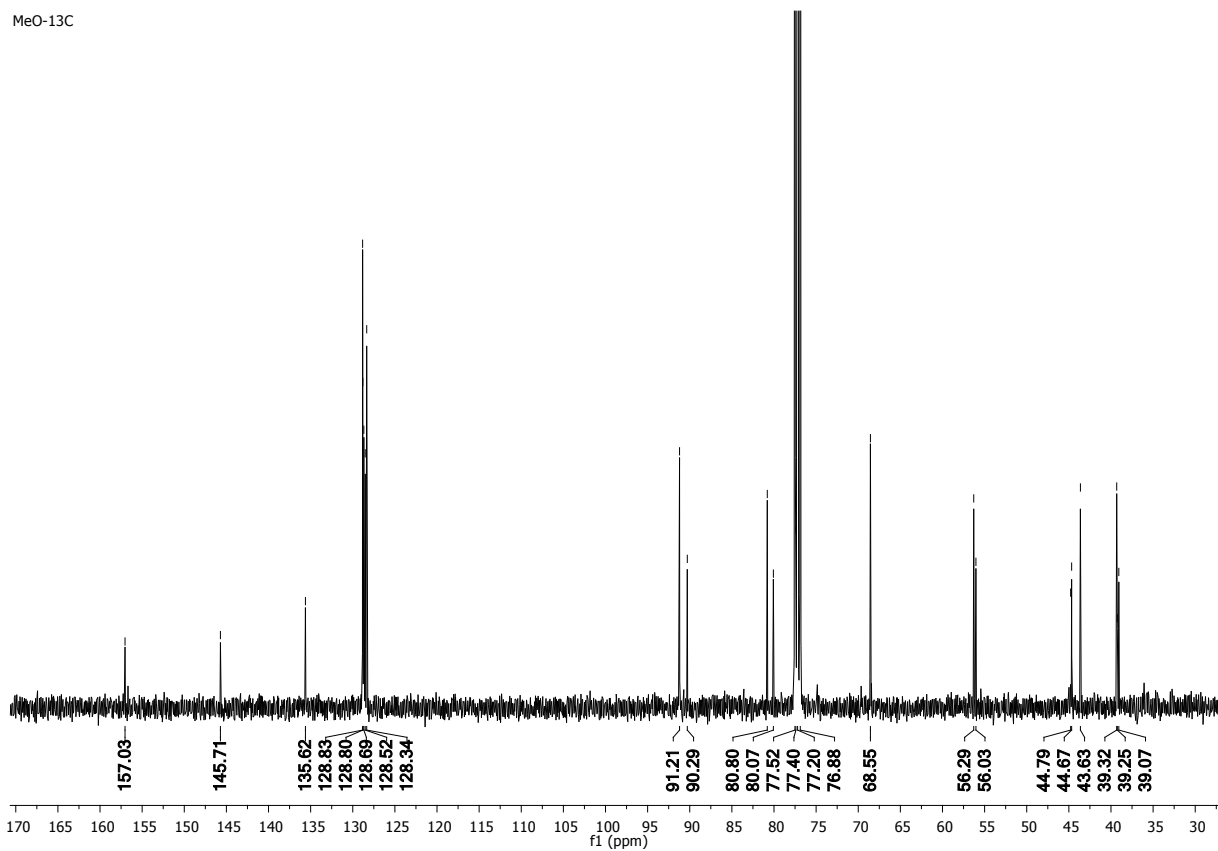
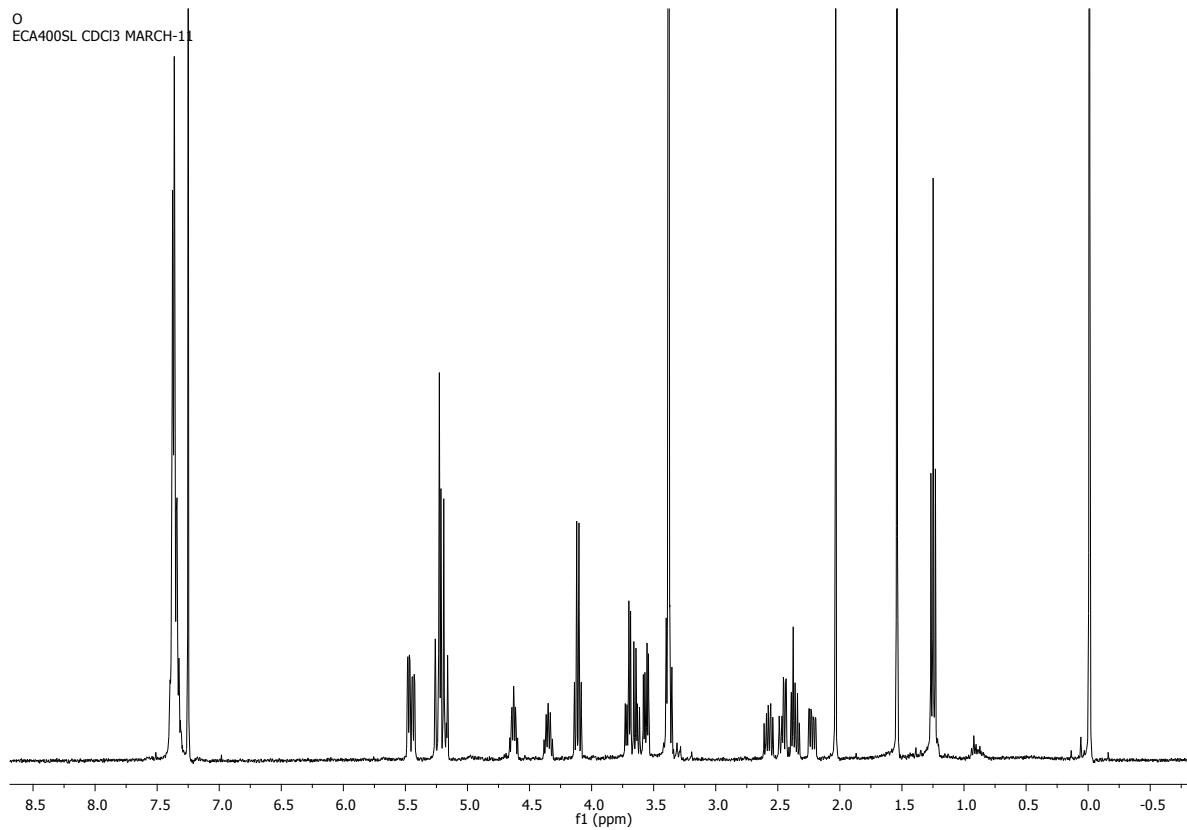
CBZ-Cl-02
ECA400SL CDCl3 JAN-15-2014



CBZ-Cl-13C-2
ECA400SL CDCl3 JAN 16-2014

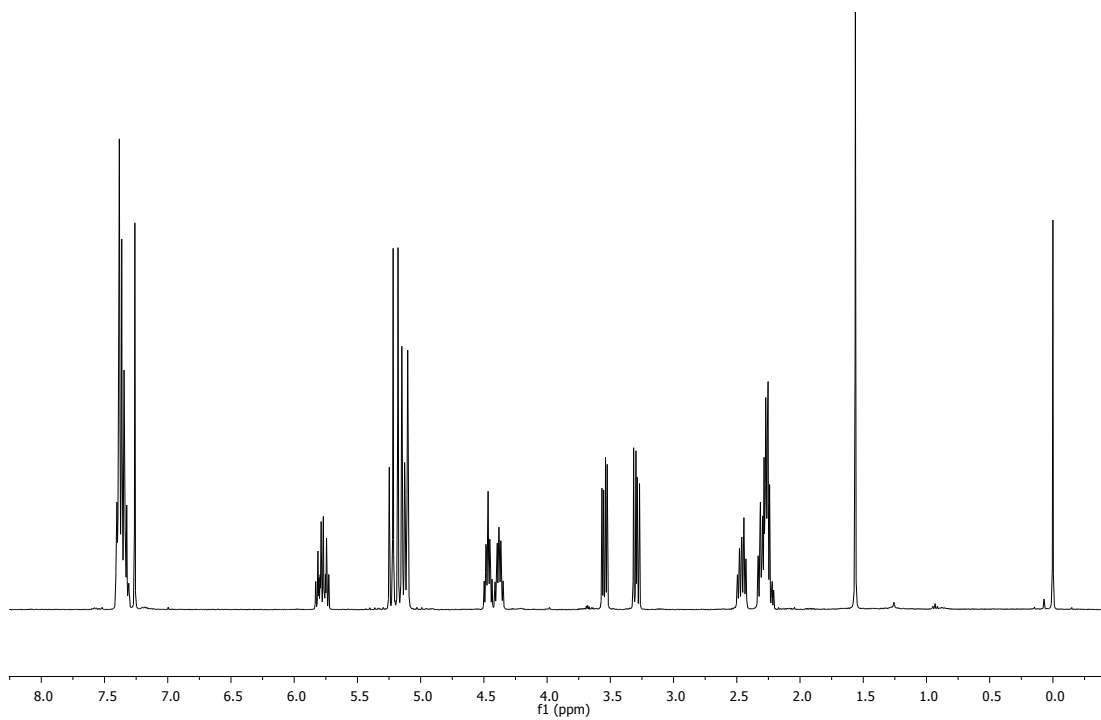


hydroxylamine **5**

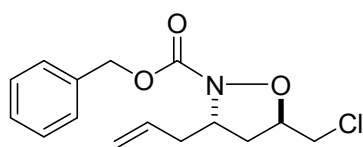
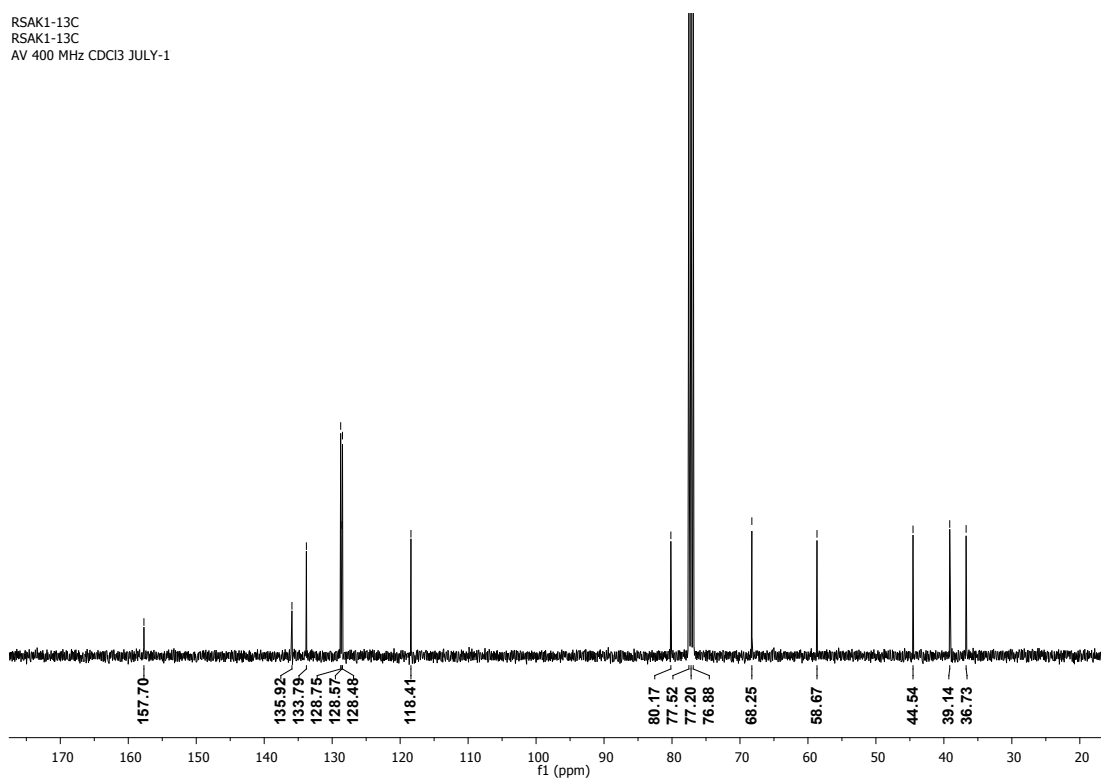


methoxyisoxazolidines **6a**

RSAK1
 RSAK1, 1H
 AV 400 MHz CDCl3 JULY-1

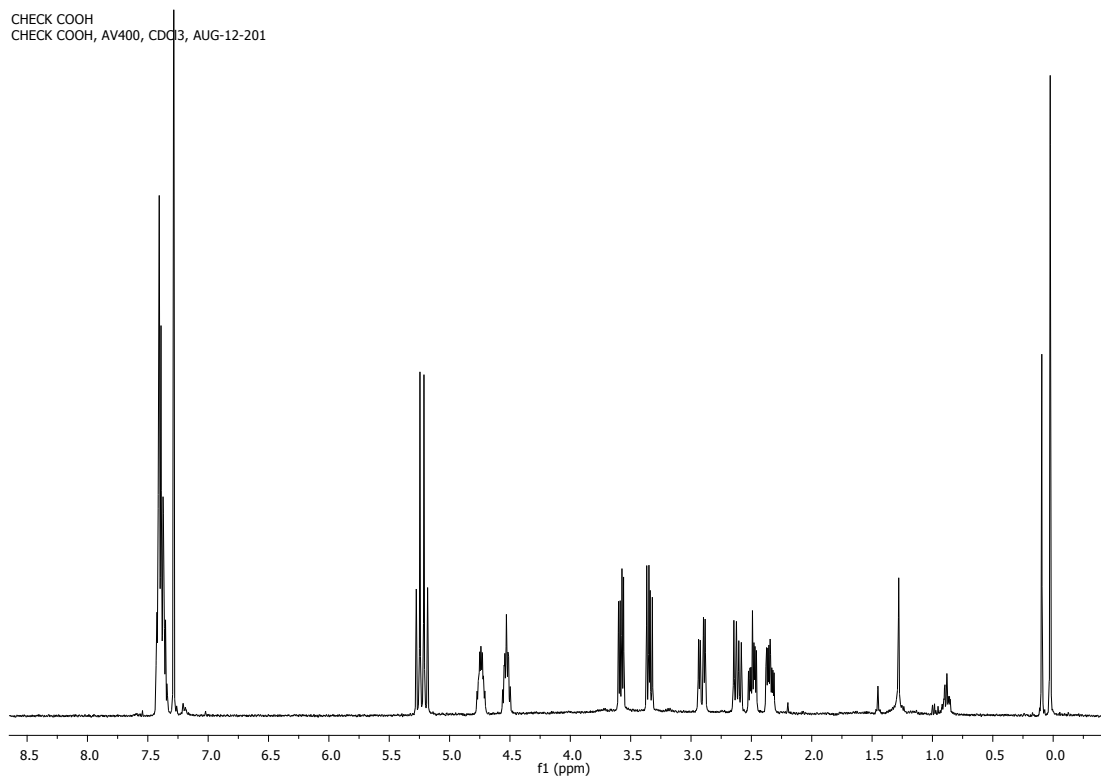


RSAK1-13C
 RSAK1-13C
 AV 400 MHz CDCl3 JULY-1

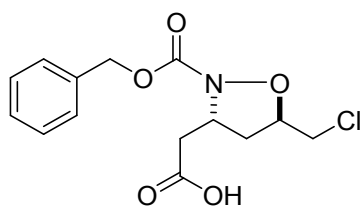
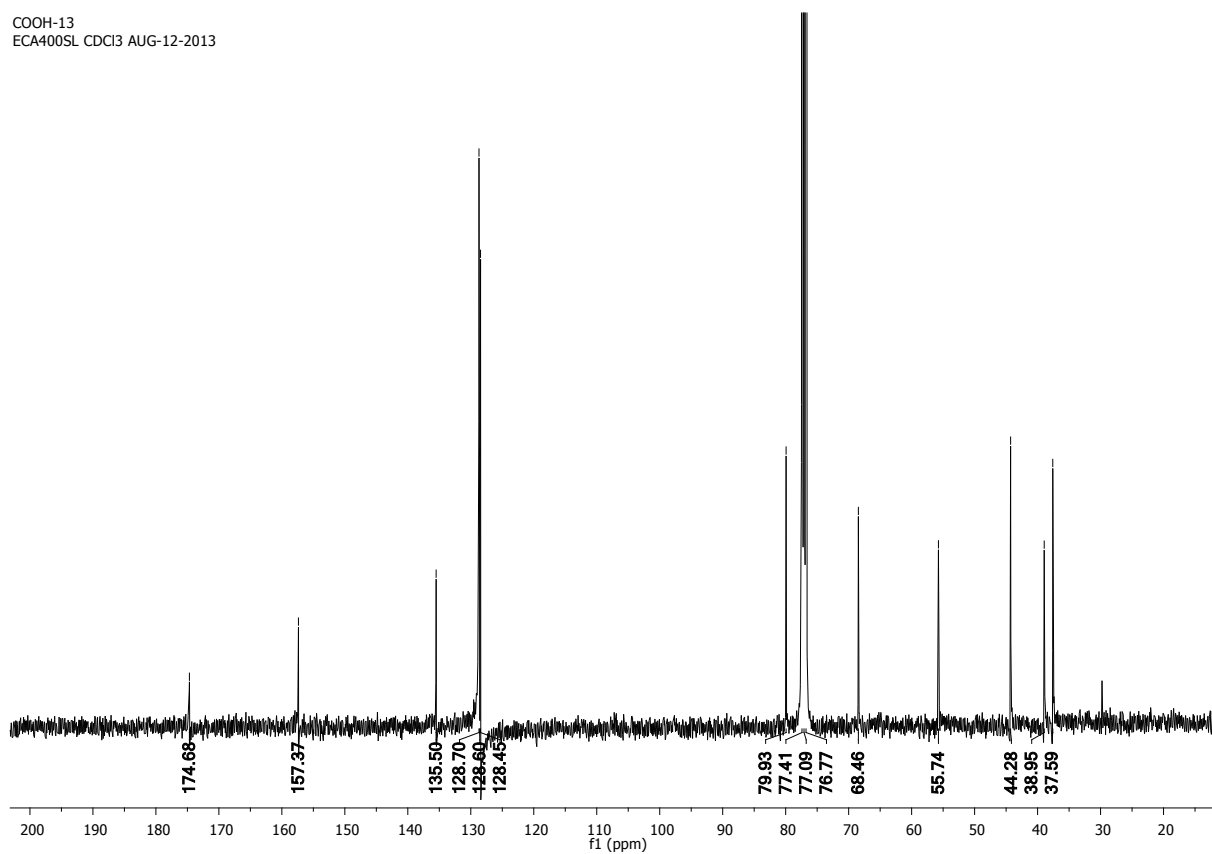


allyl isoxazolidine **7a**

CHECK COOH
CHECK COOH, AV400, CDCl₃, AUG-12-201

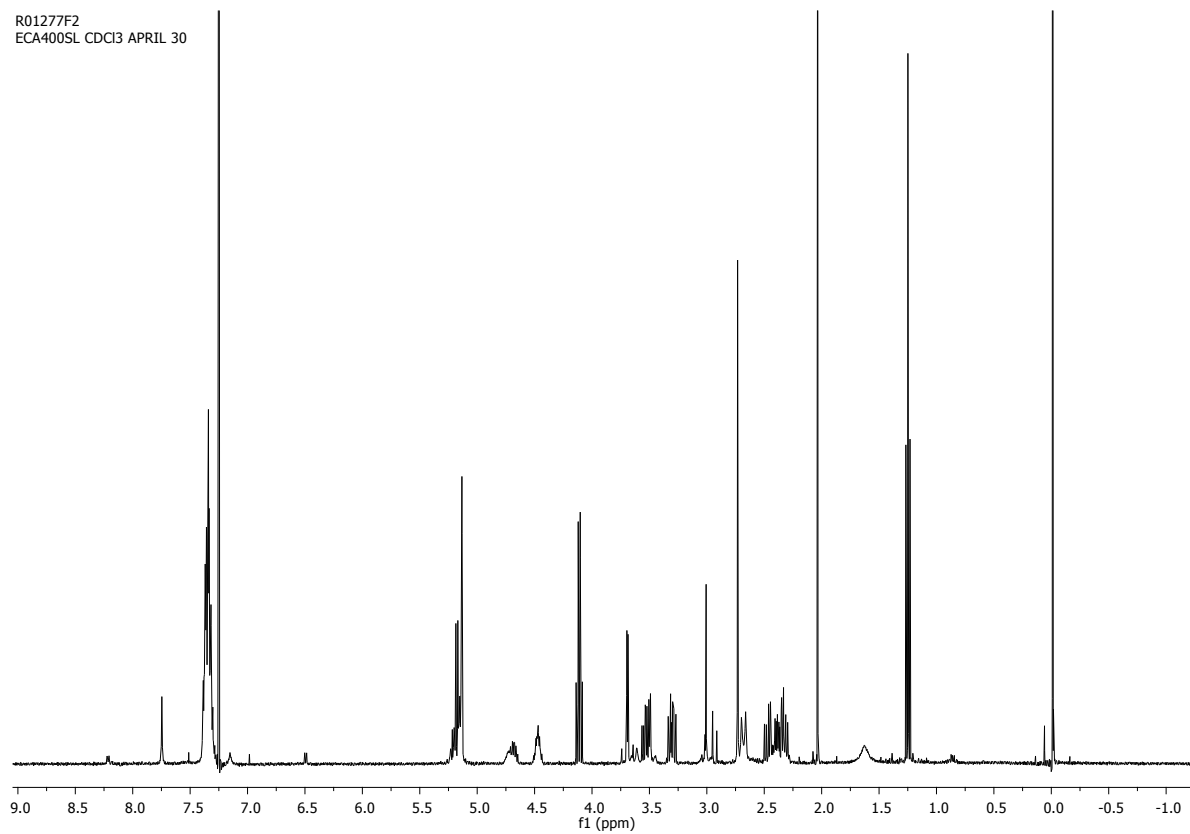


COOH-13
ECA400SL CDCl₃ AUG-12-2013

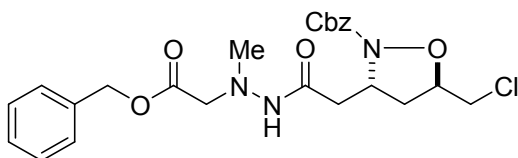
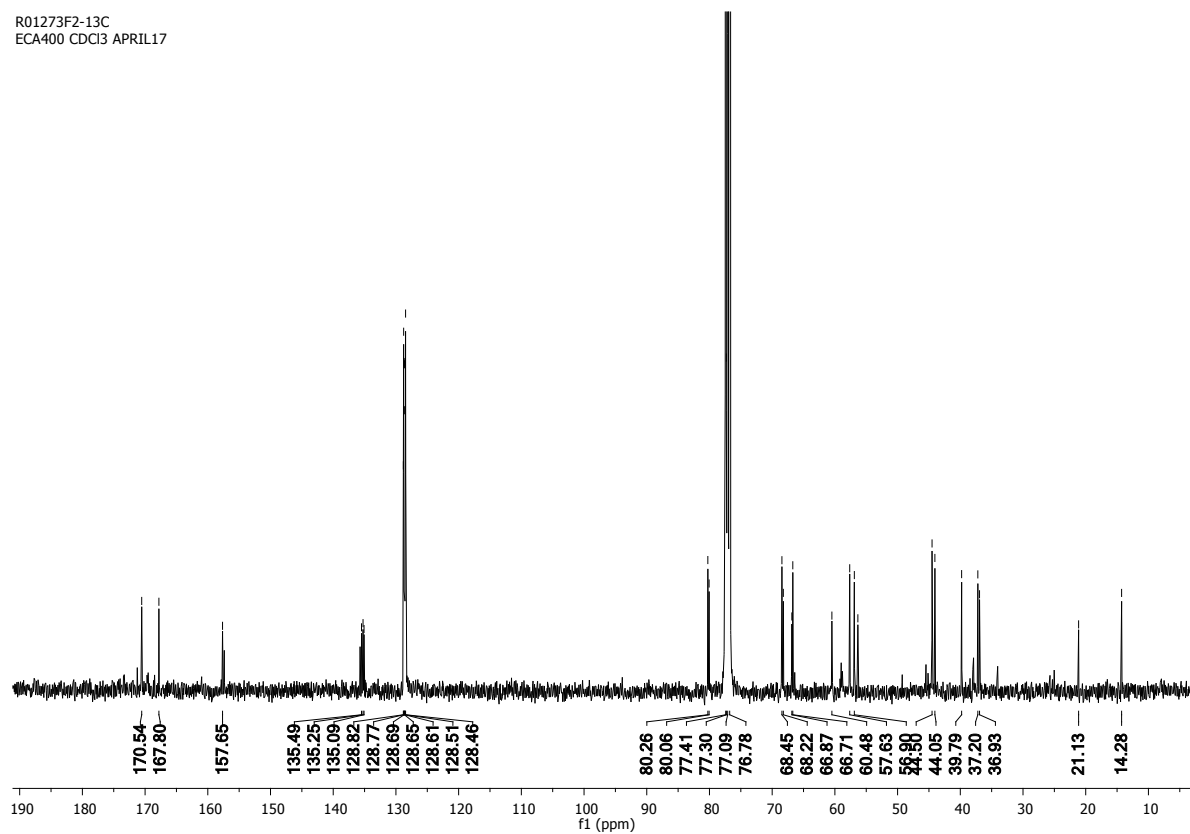


carboxylic acid **9**

R01277F2
ECA400SL CDCl3 APRIL 30

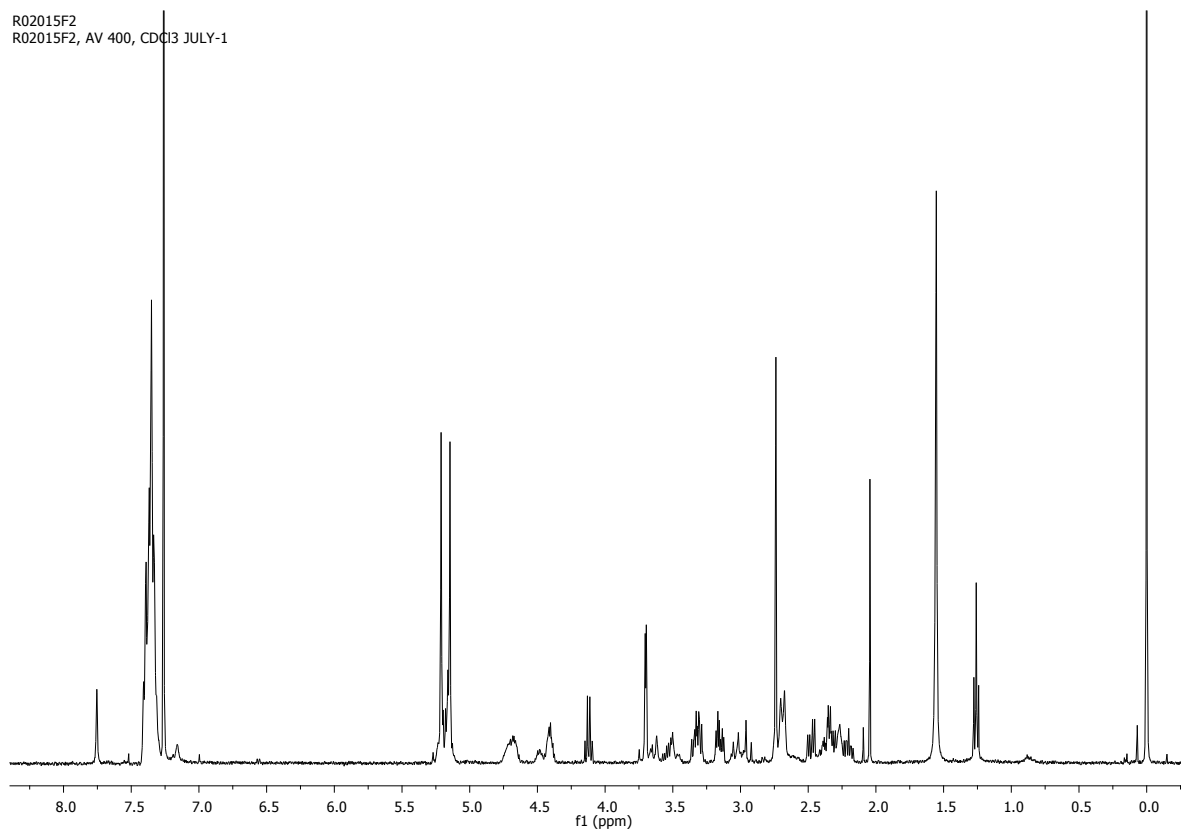


R01273F2-13C
ECA400 CDCl3 APRIL 17

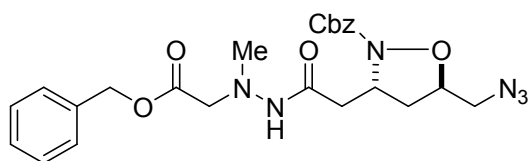
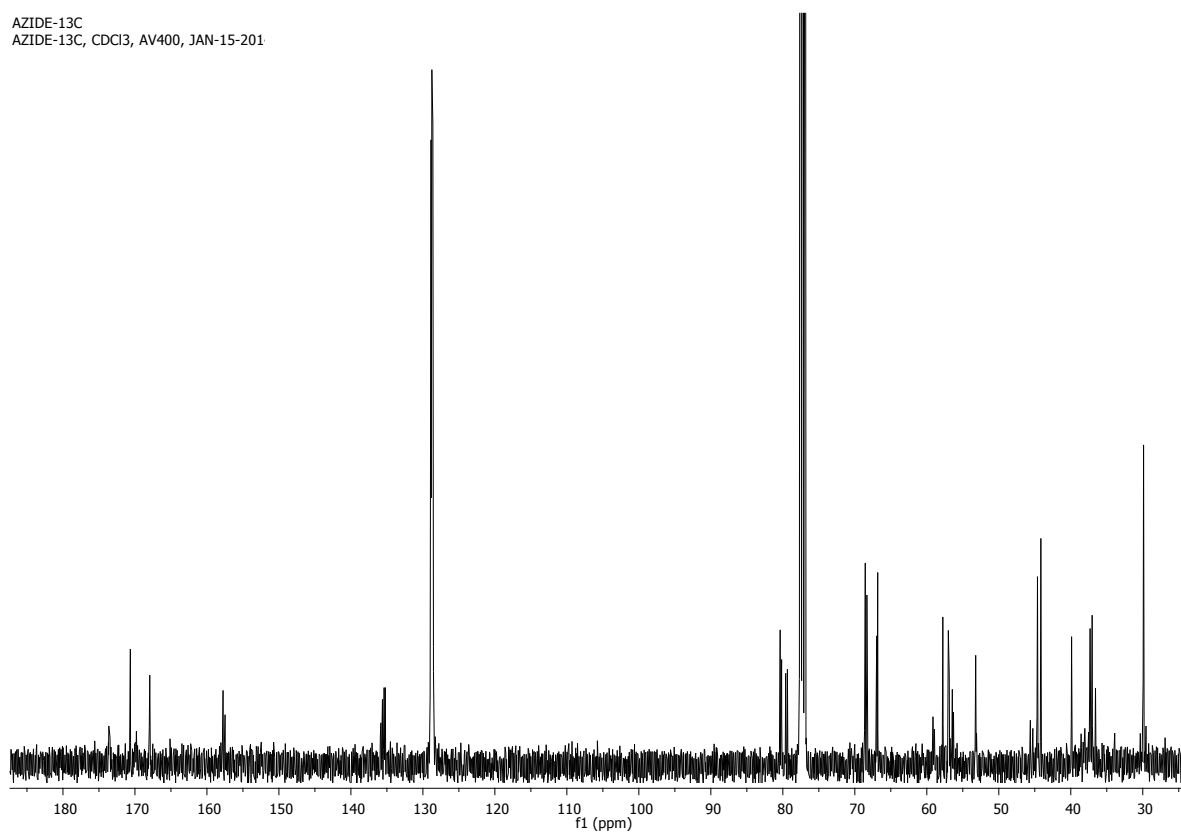


hydrazide **11**

R02015F2
R02015F2, AV 400, CDCl₃ JULY-1

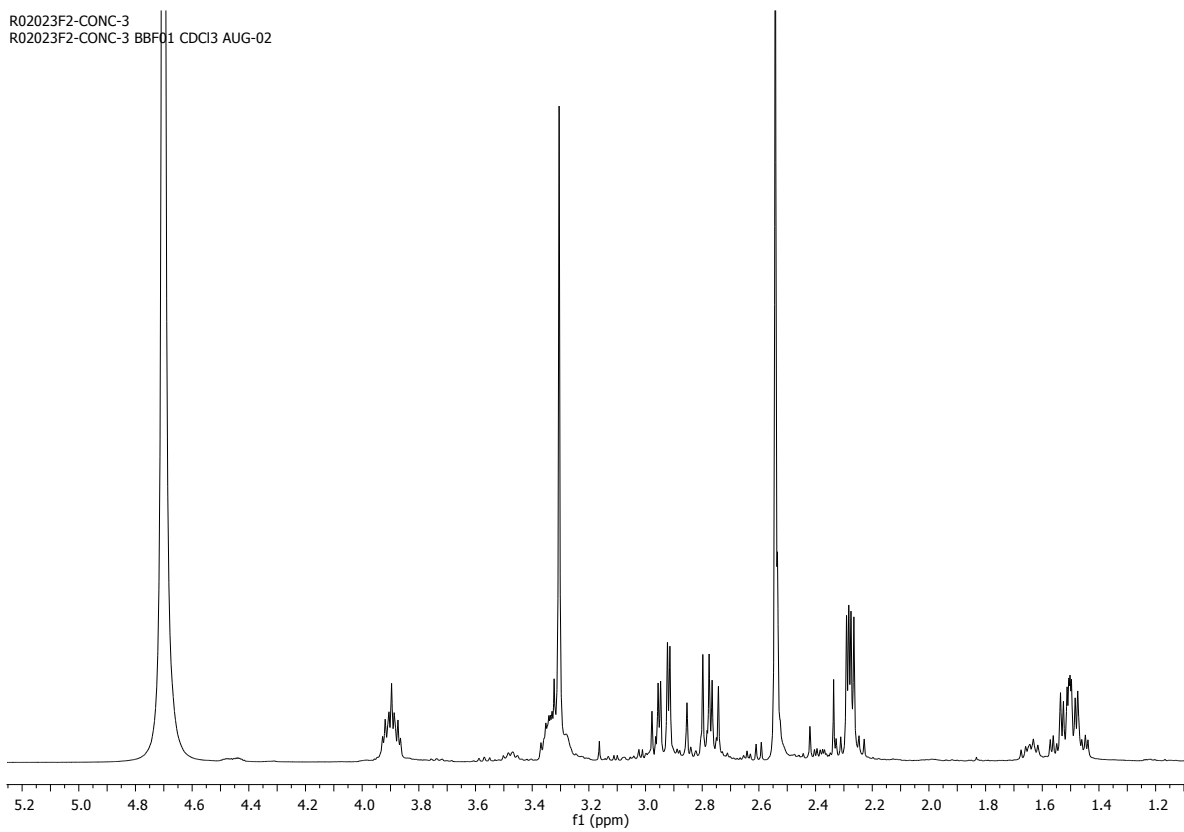


AZIDE-13C
AZIDE-13C, CDCl₃, AV400, JAN-15-201

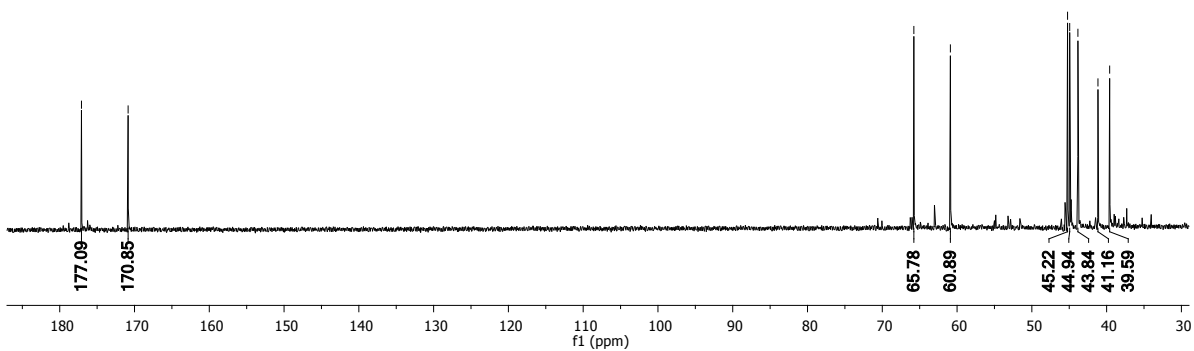


azide **12**

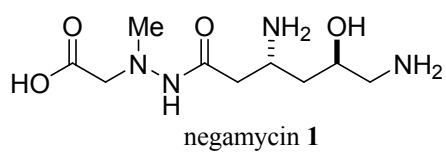
R02023F2-CONC-3
R02023F2-CONC-3 BBF01 CDCl3 AUG-02

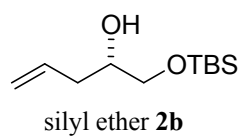
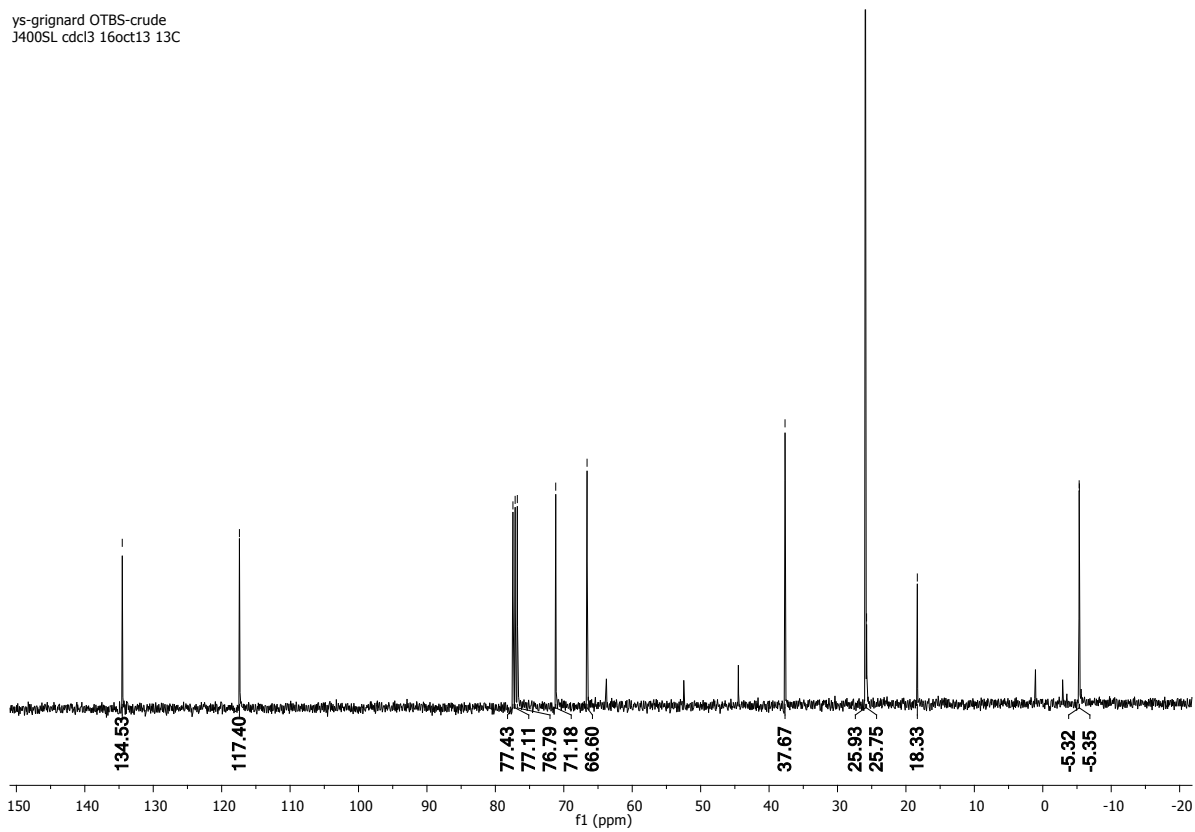
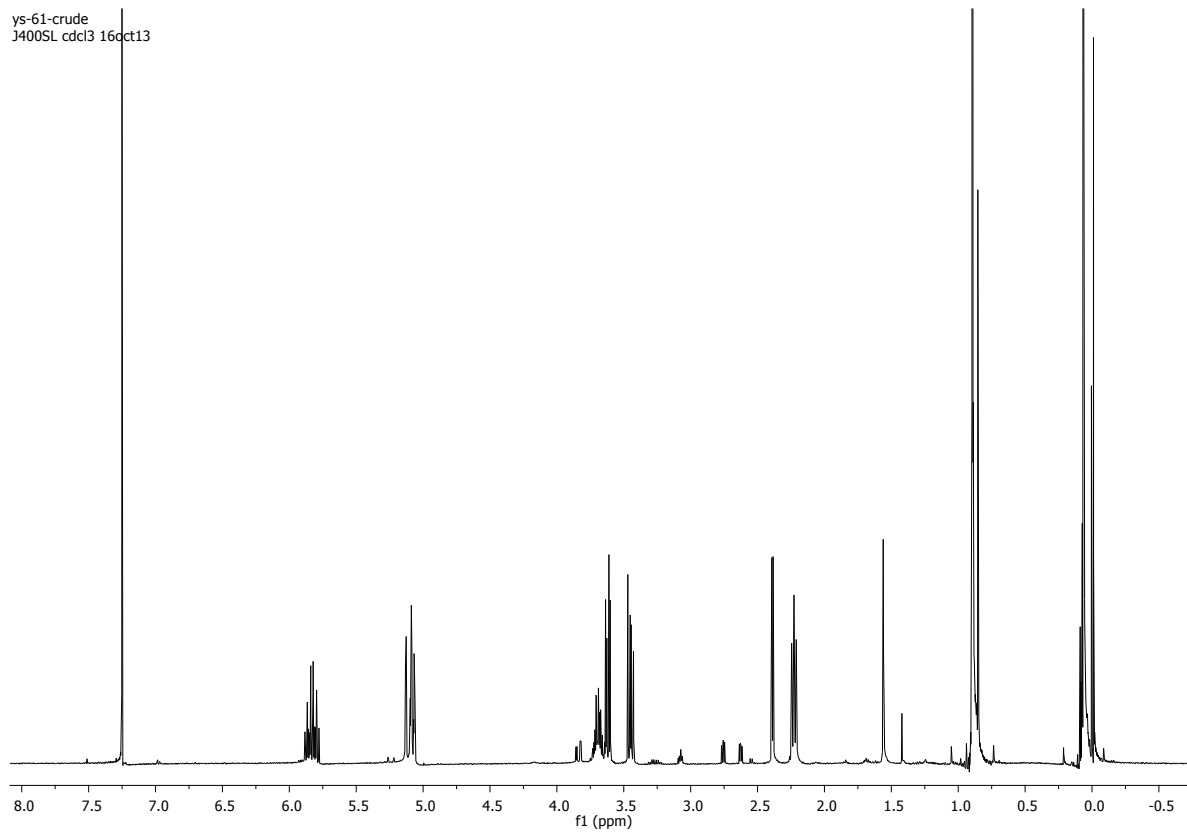


R02023F2-CONC-13C
R02023F2-CONC-13C BBF01 CDCl3 AUG-02

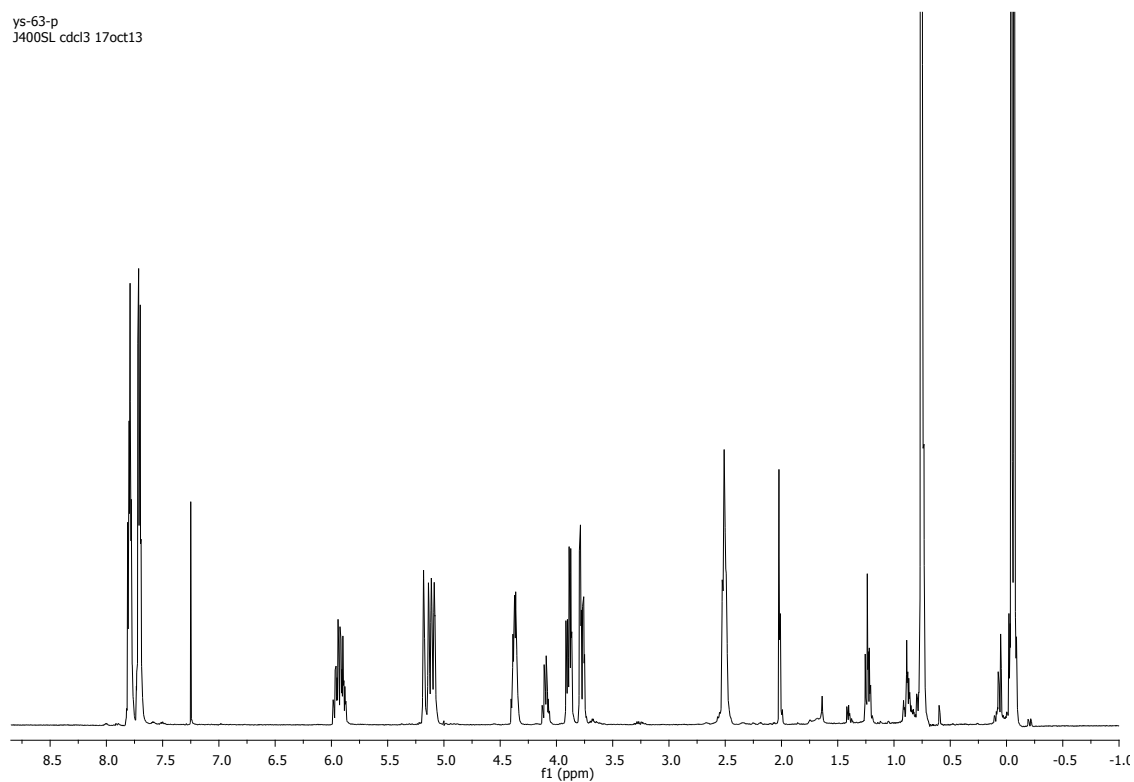


spectra recorded in D₂O

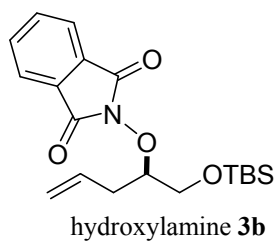
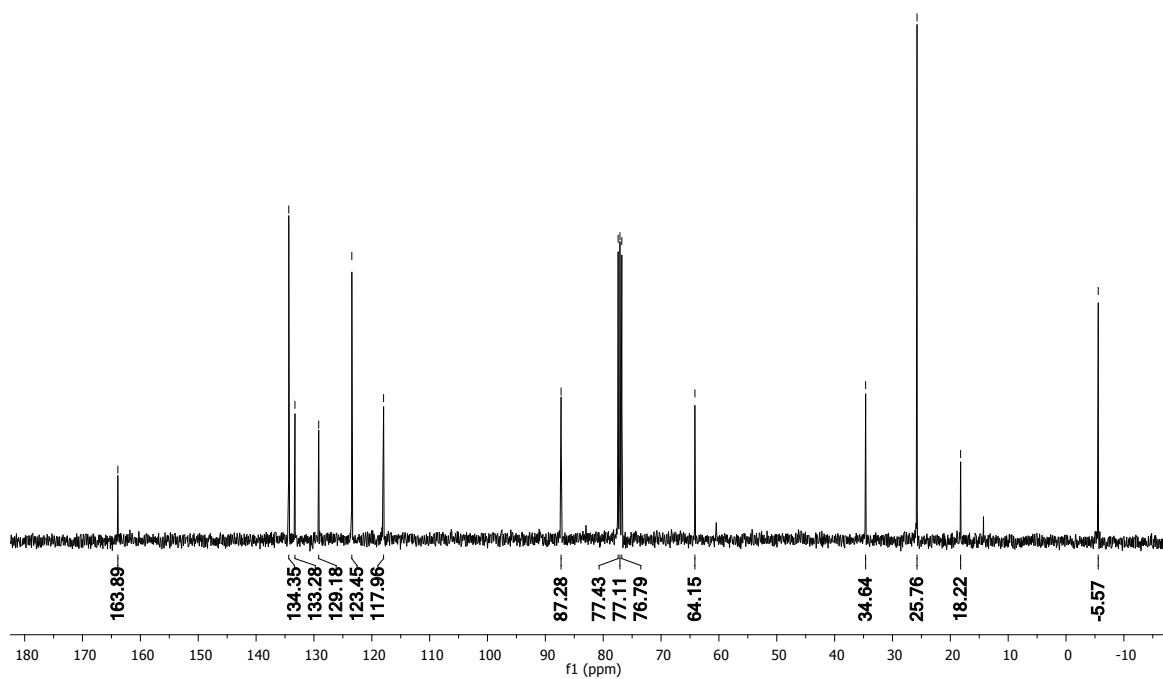




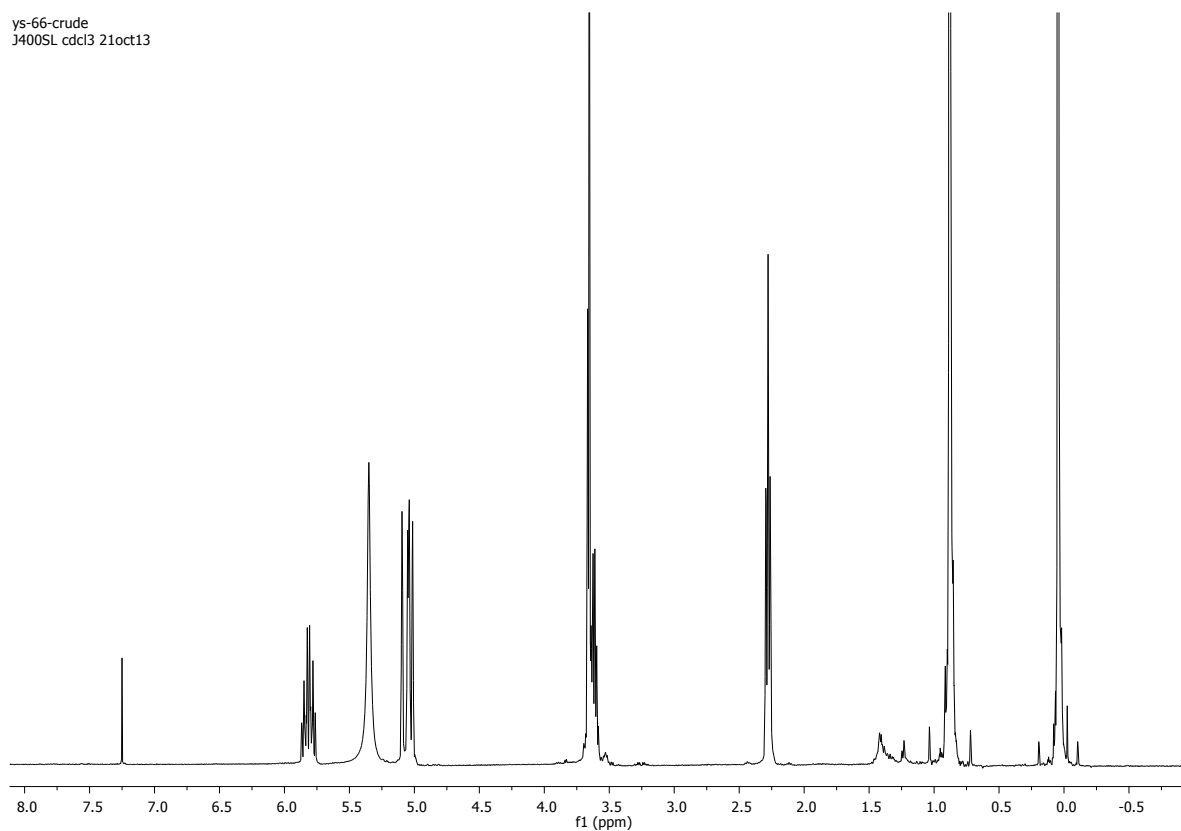
ys-63-p
J400SL cdd3 17oct13



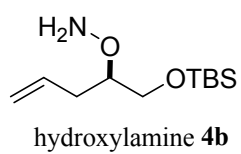
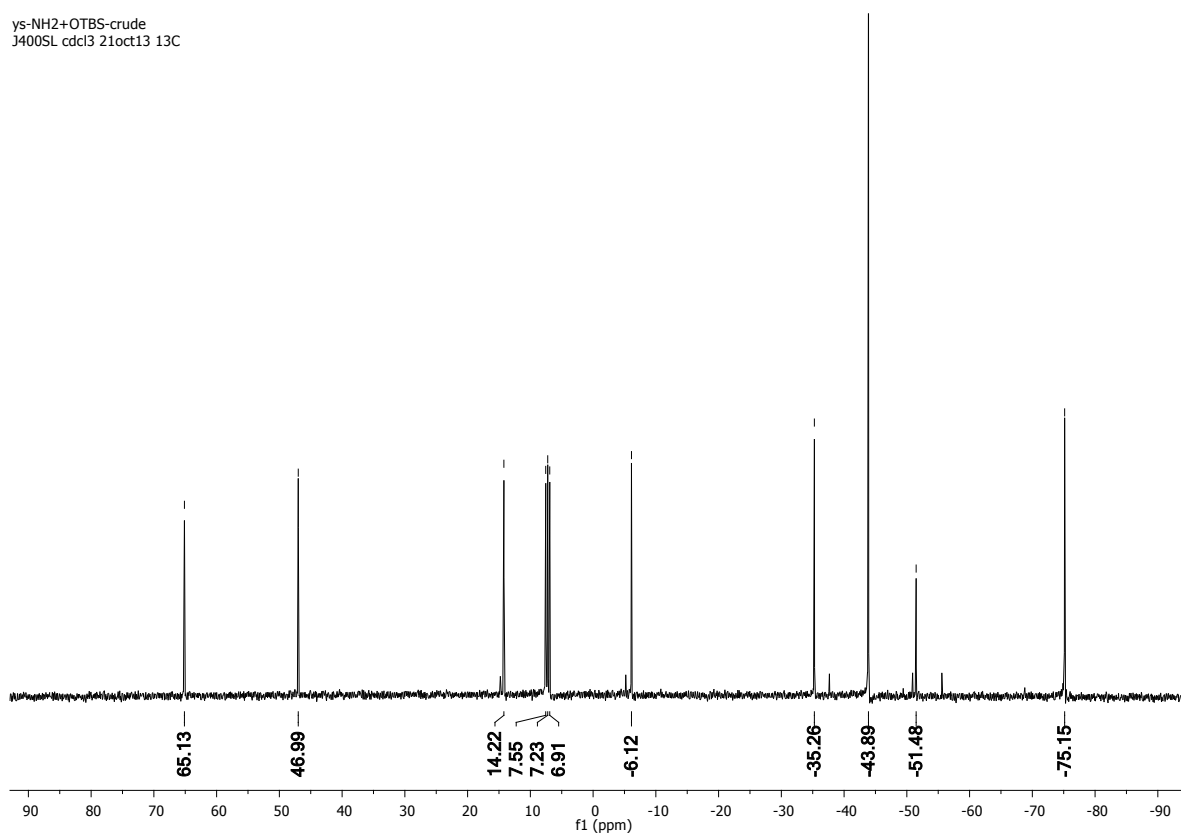
ys-mitsunobu OTBS-p
J400SL cdd3 17oct13 13C



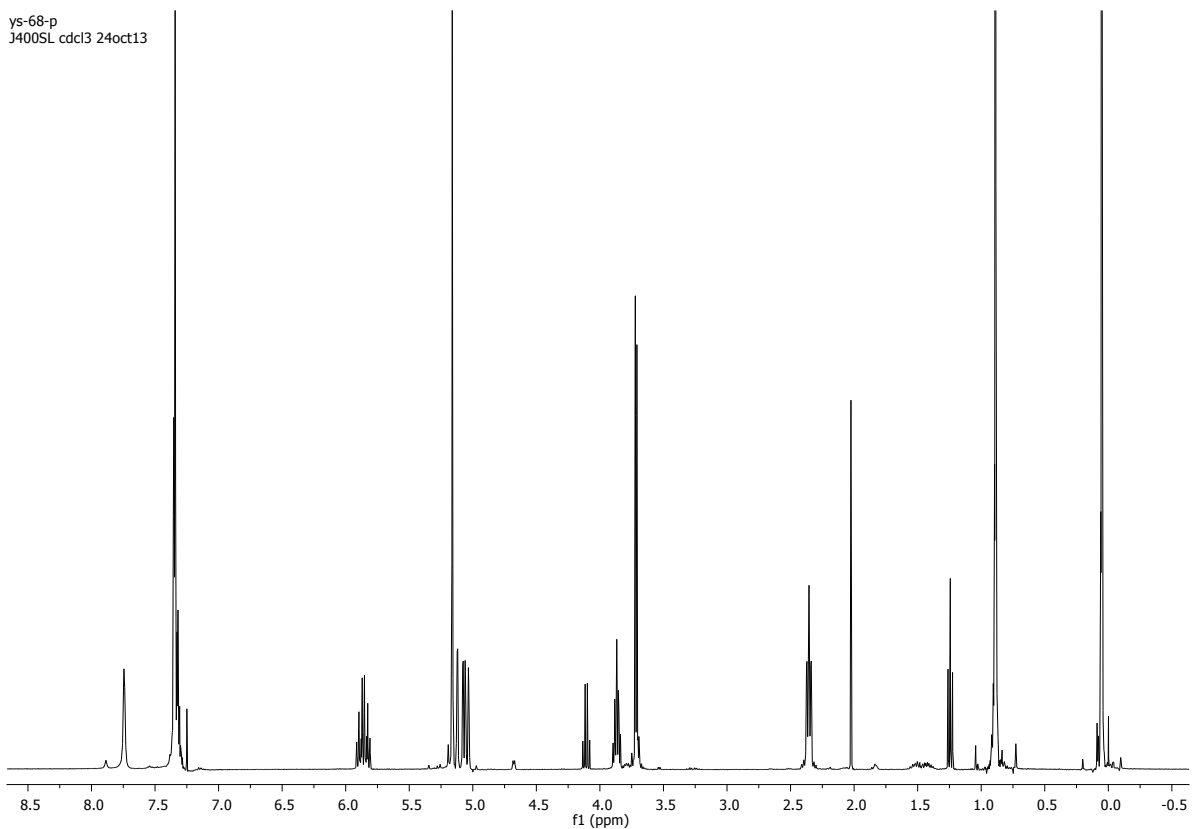
ys-66-crude
J400SL cdcl3 21oct13



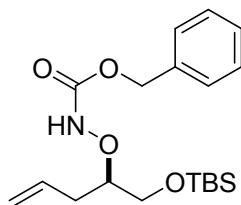
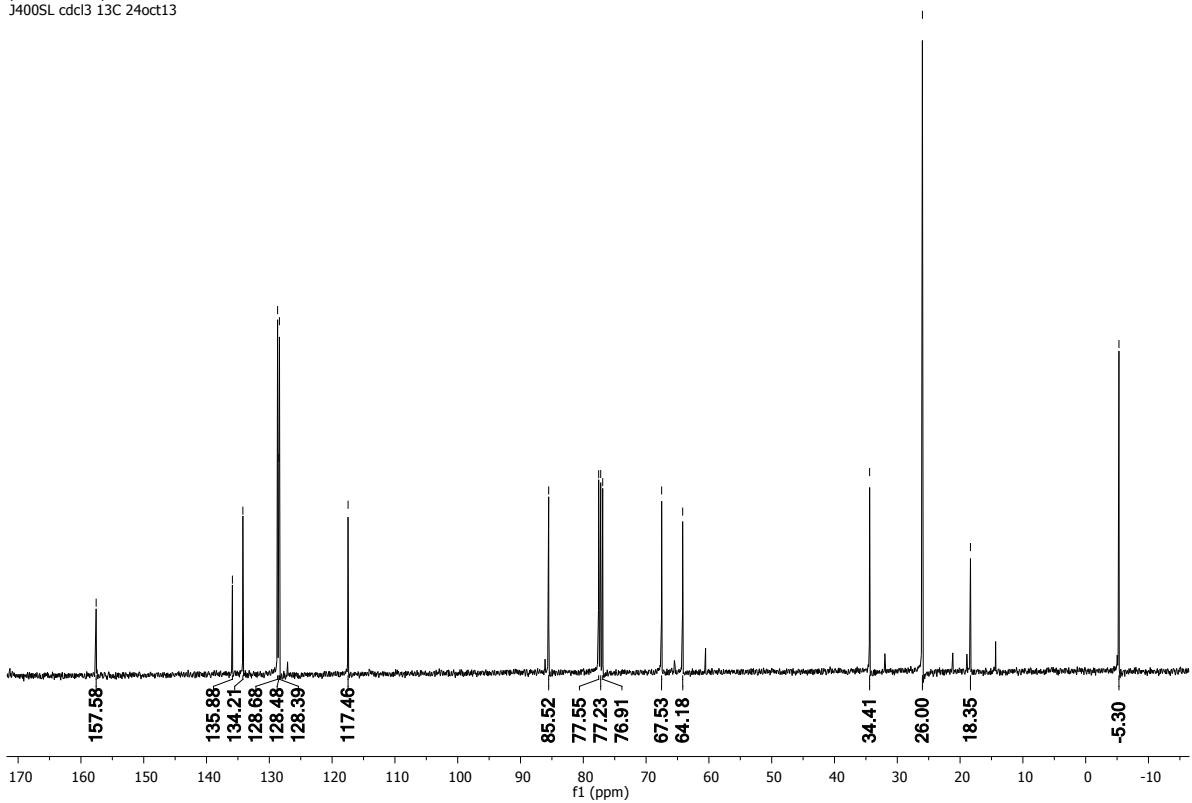
ys-NH2+OTBS-crude
J400SL cdcl3 21oct13 13C



ys-68-p
J400SL cdcl3 24oct13

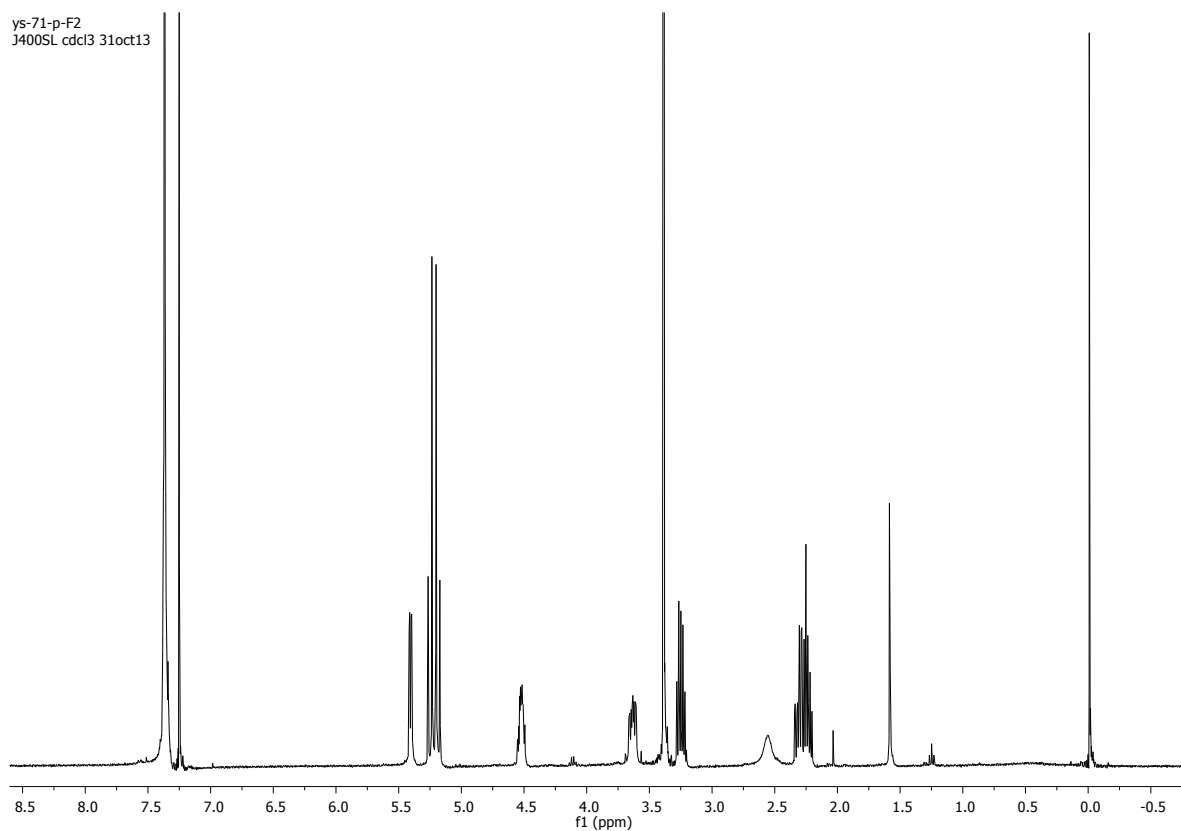


ys-cbz+OTBS-p
J400SL cdcl3 13C 24oct13

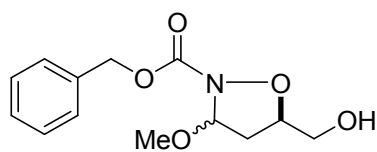
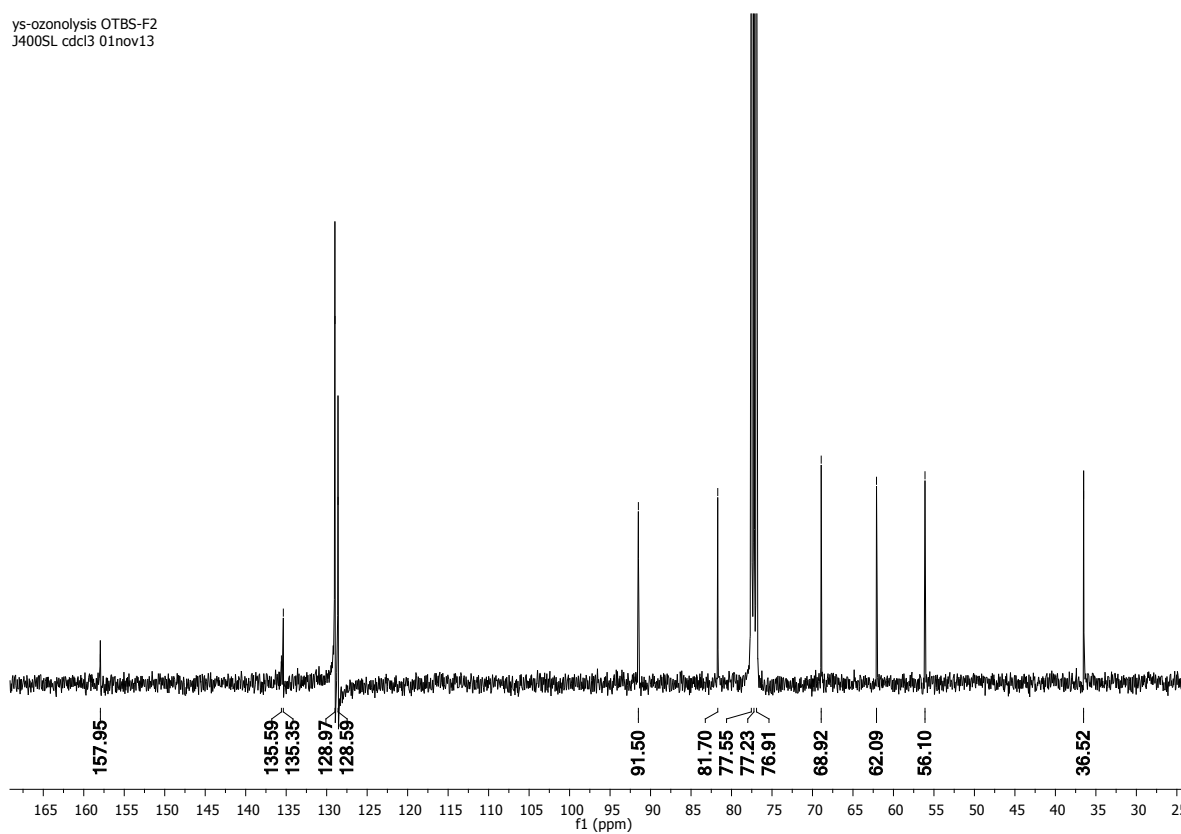


hydroxylamine **5b**

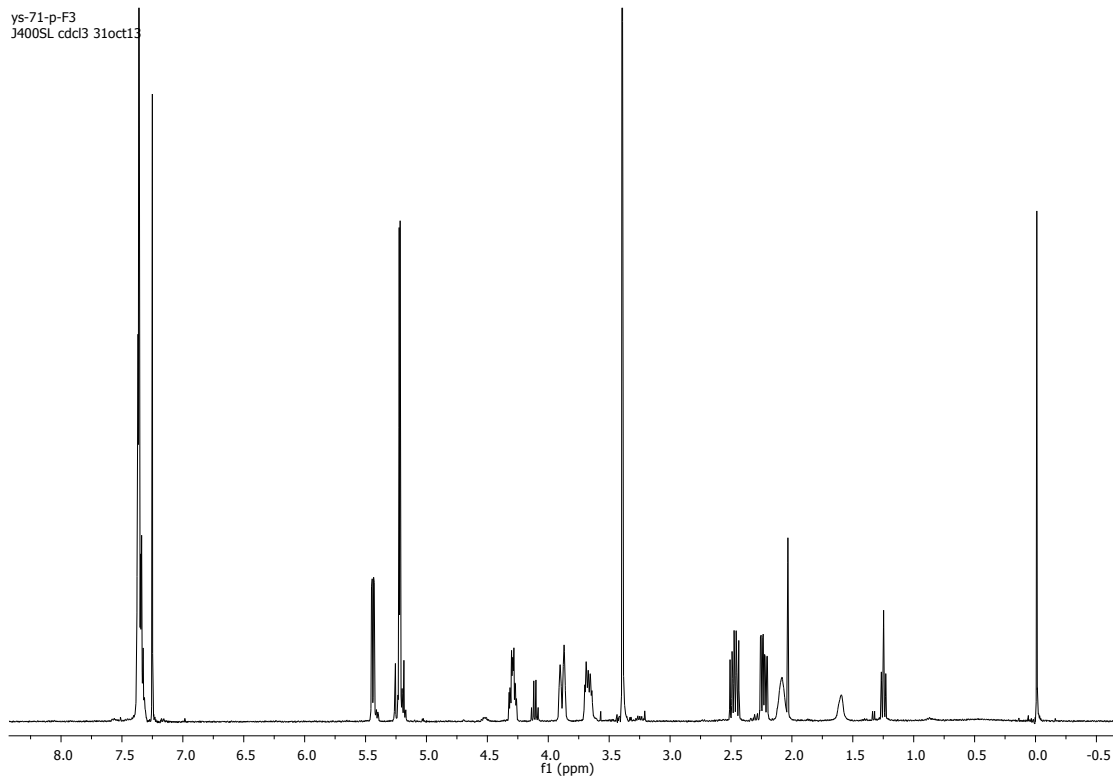
ys-71-p-F2
J400SL cdcl3 31oct13



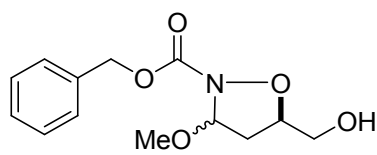
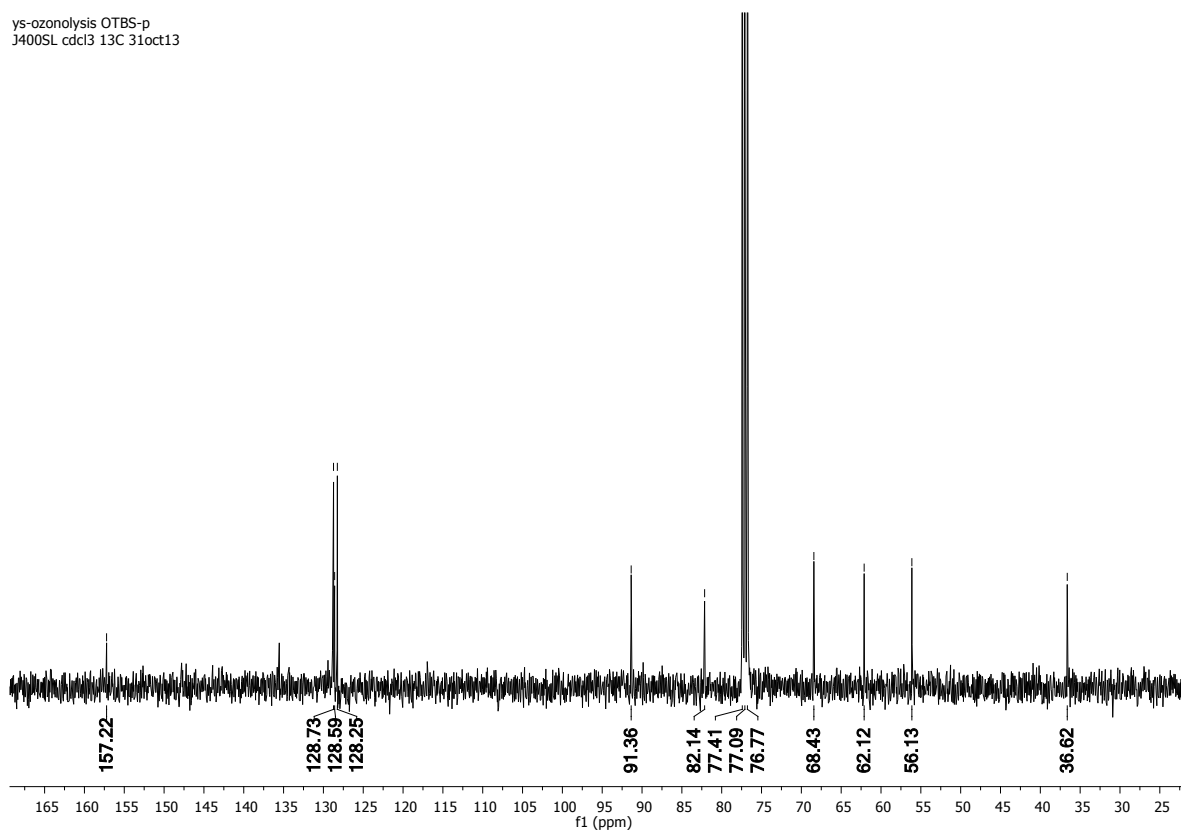
ys-ozonolysis OTBS-F2
J400SL cdcl3 01nov13



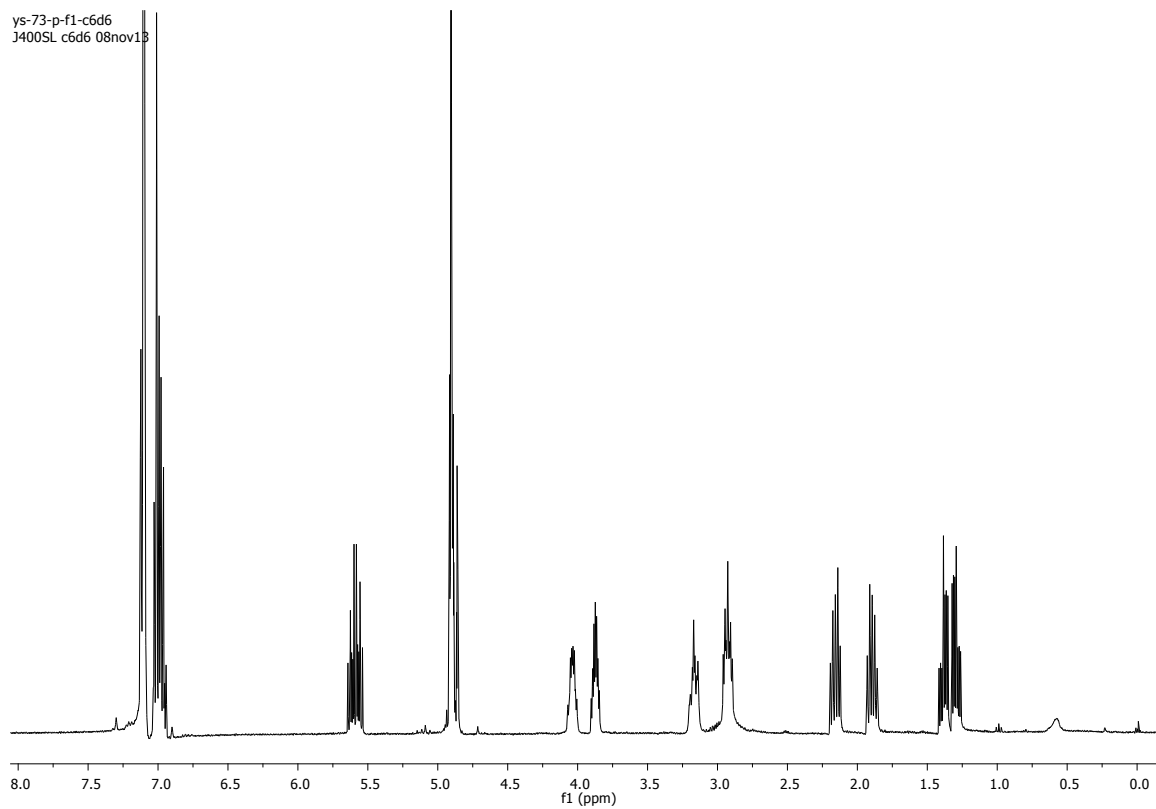
isoxazolidines **6b**



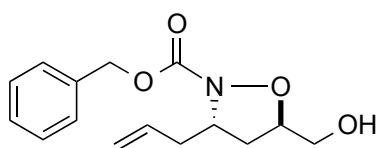
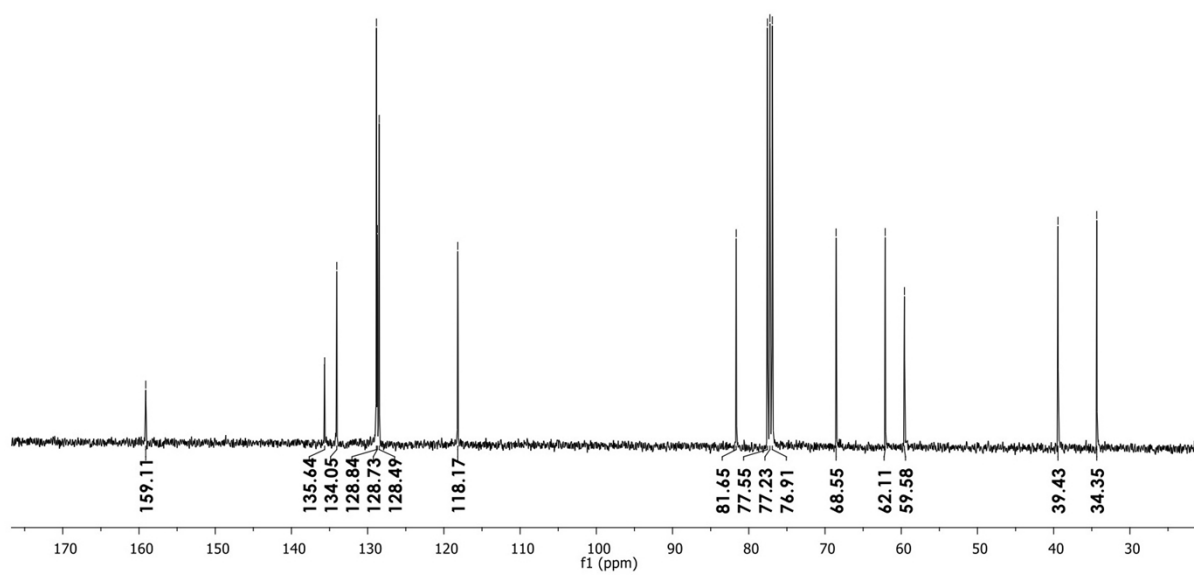
ys-ozonolysis OTBS-p
J400SL cdcl3 13C 31oct13



isoxazolidines **6b**



The ^1H spectrum was recorded in C_6D_6 to give better resolution of the signals



allyl isoxazolidine **7b**