

Supplementary Material

Reaction of trifluoromethyl 1,3-dicarbonyl compounds with formaldehyde and esters of natural α -aminoacids

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Ufa, 450 054, Russian Federation

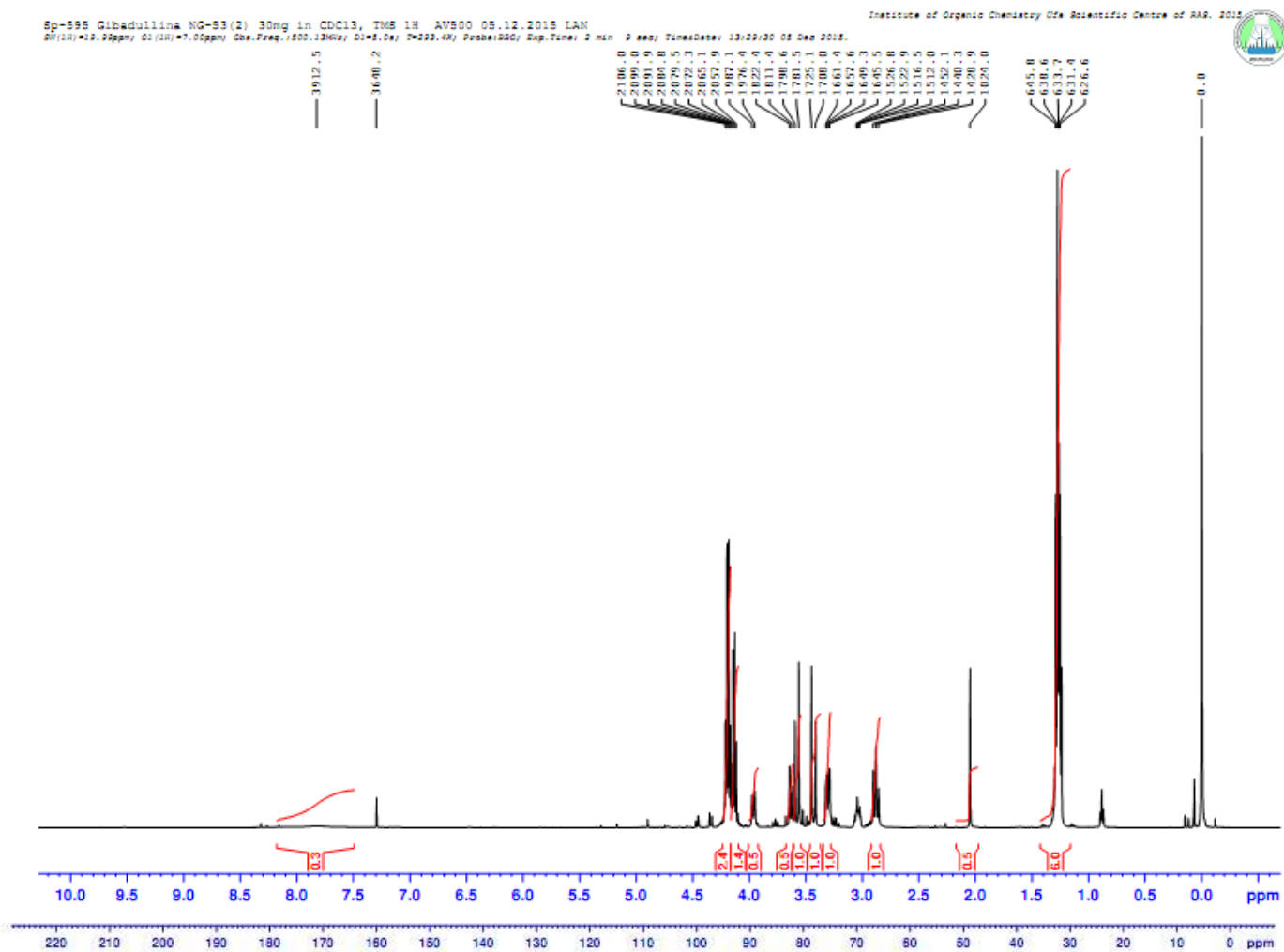
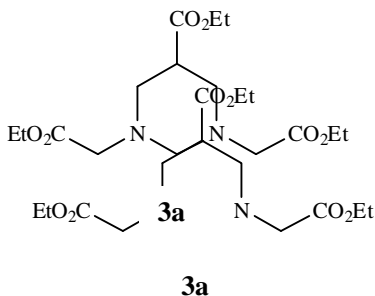
^b Ufa State Aviation Technical University, 12 K. Marx Street, Ufa, 450 008, Russian Federation

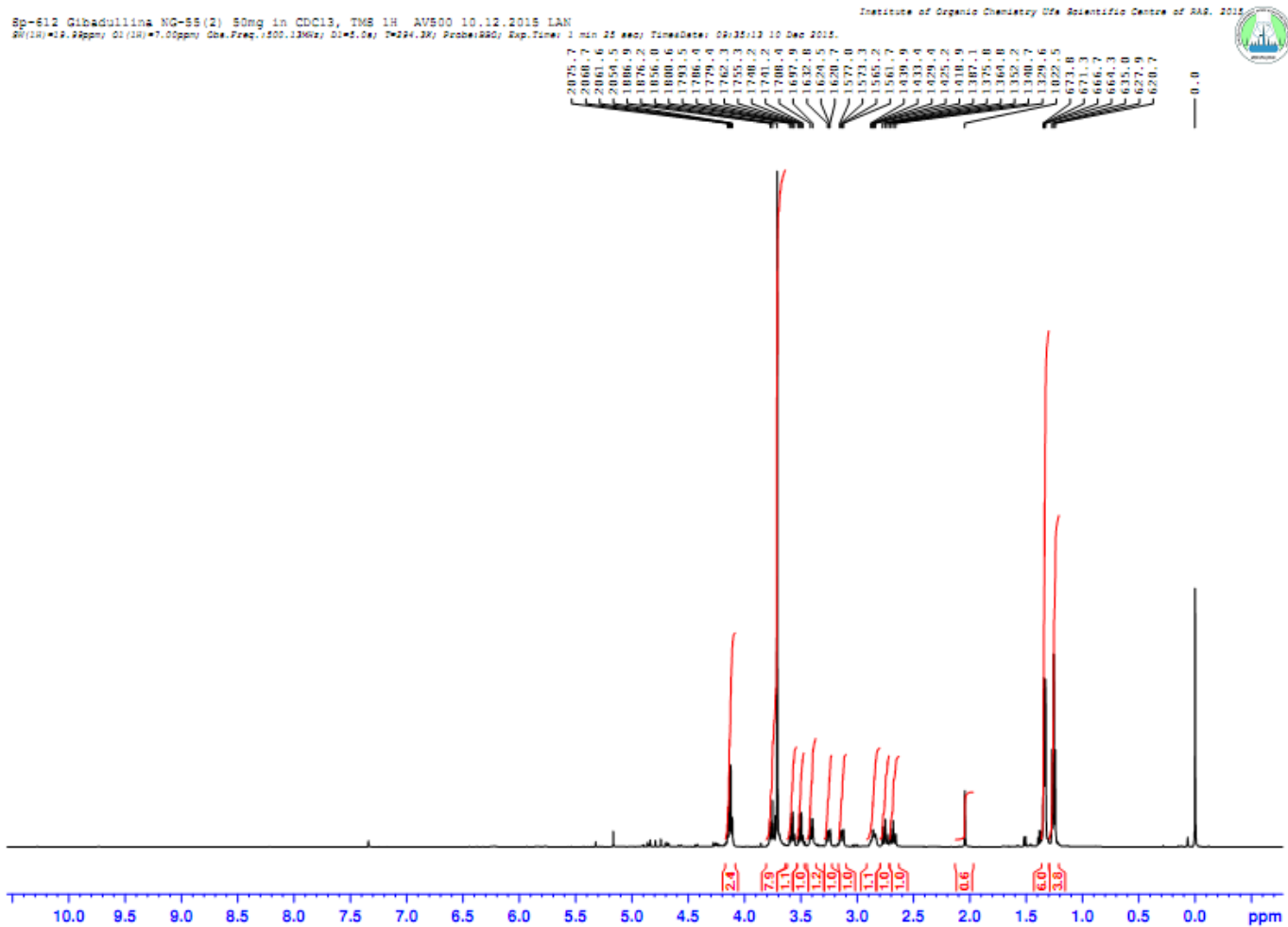
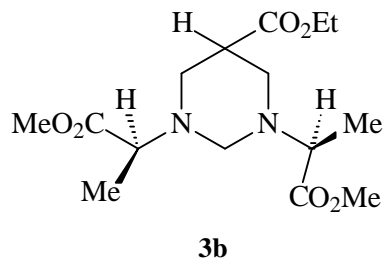
^c N. D. Zelinsky Institute of Organic Chemistry, Russian Academy of Sciences, 47 Leninsky prosp.,
119 991 Moscow, Russian Federation

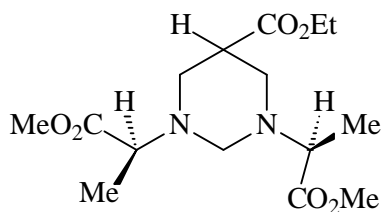
E-mail: novikovfff@bk.ru

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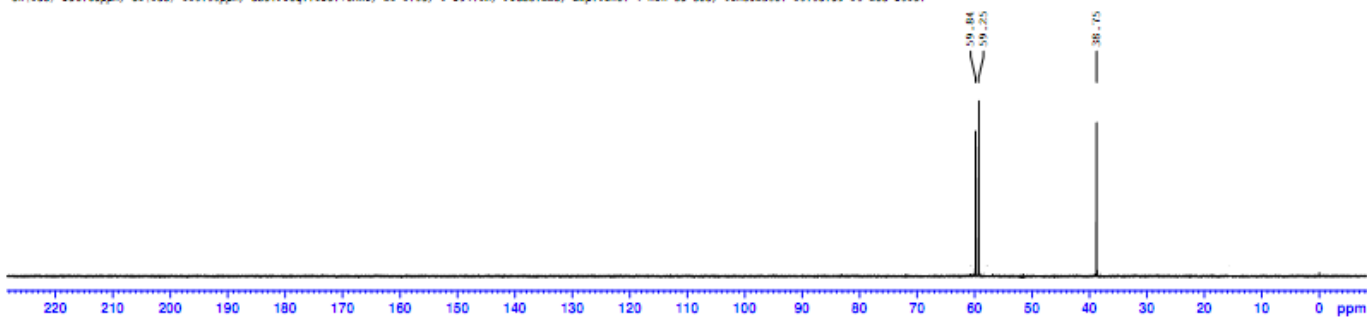




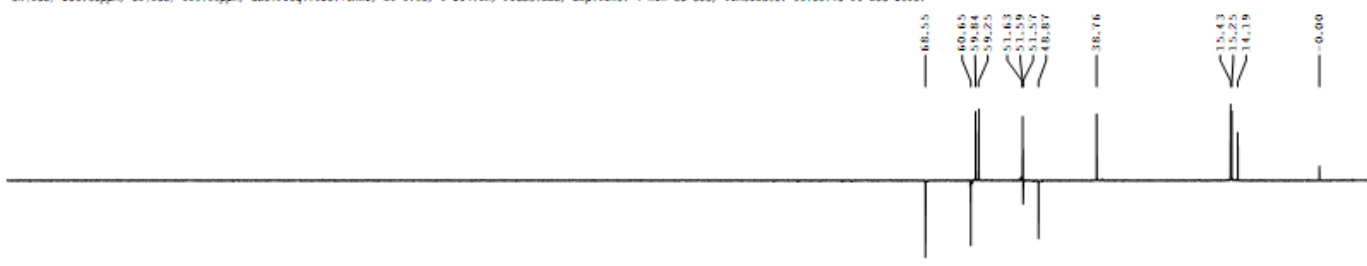


3b

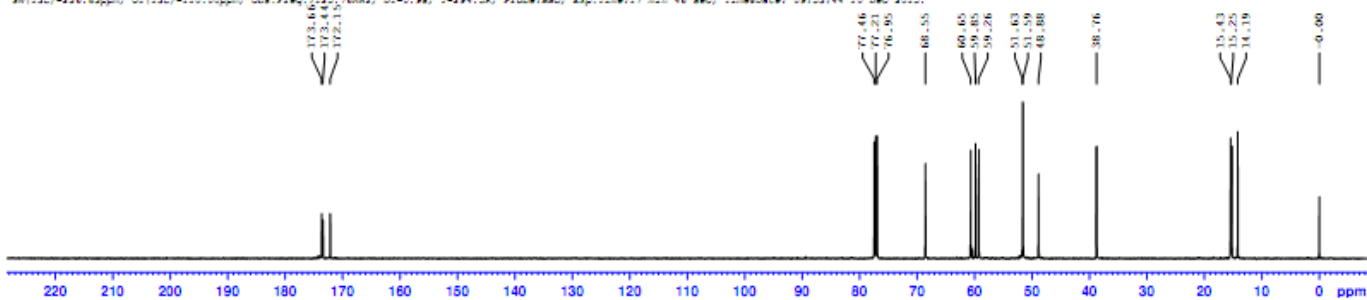
Sp-612 Gibadullina NG-55(2) 50mg in CDCl3, TMS 13C{1H} dept90 AV500 10.12.2015 LAN
 20(13C)=236.63ppm O1(13C)=110.00ppm Obs.Freq.:125.766MHz O1=1.0s T=224.1K Probe:800 Exp.Time: 4 min 53 sec TimeDate: 10/03/15 10 Dec 2015.

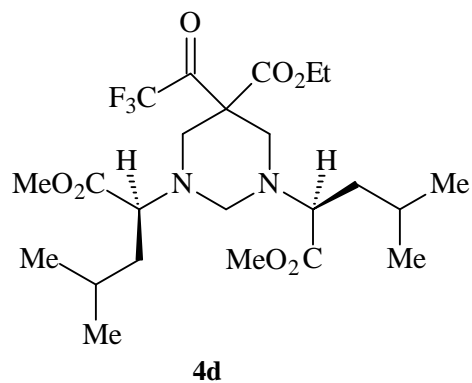


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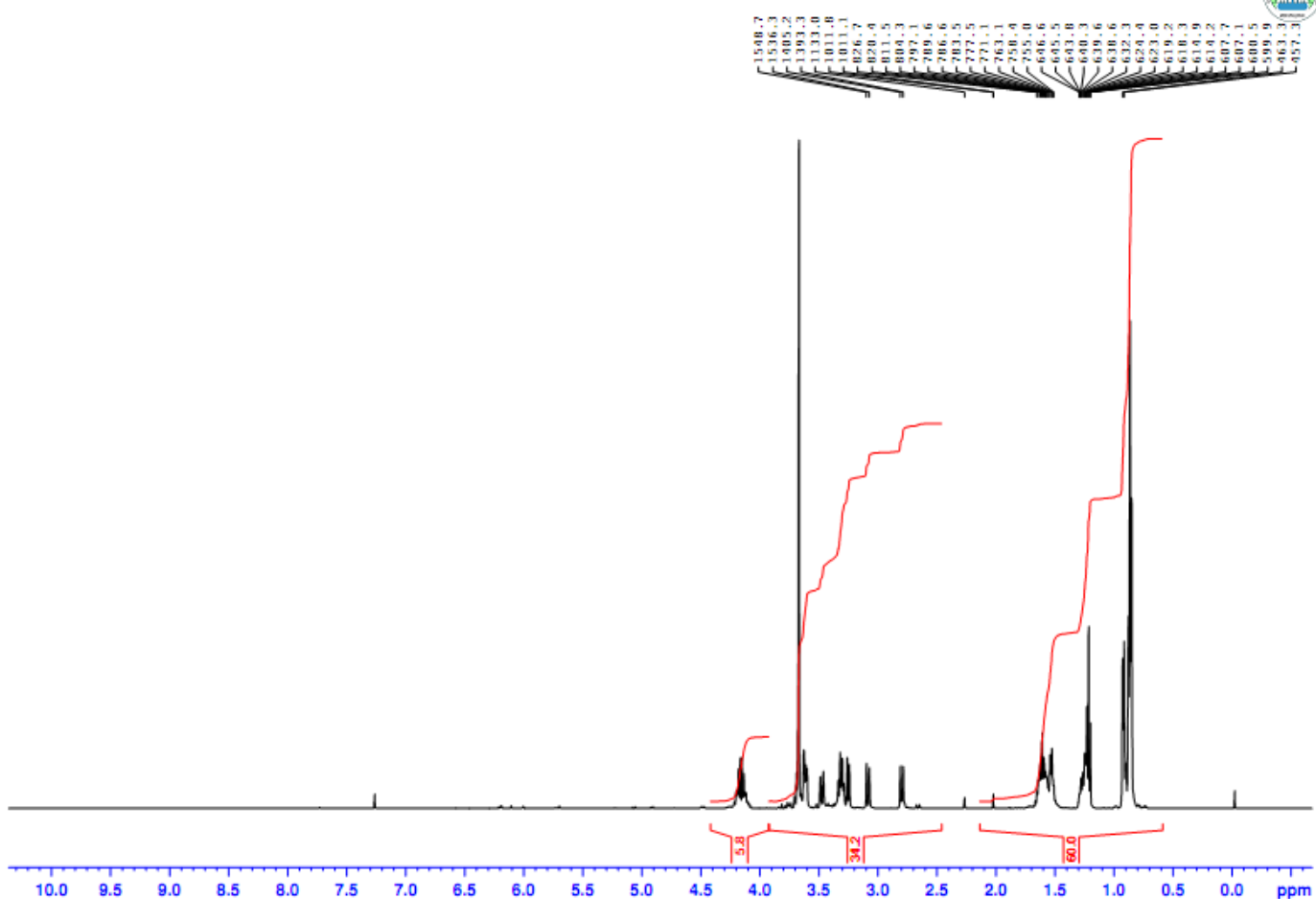
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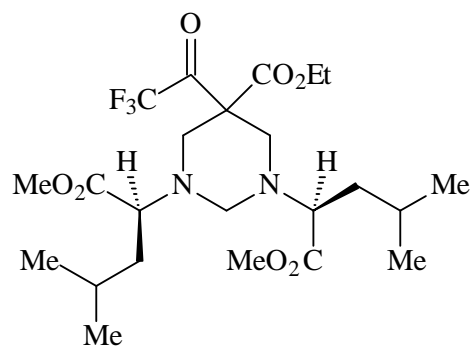




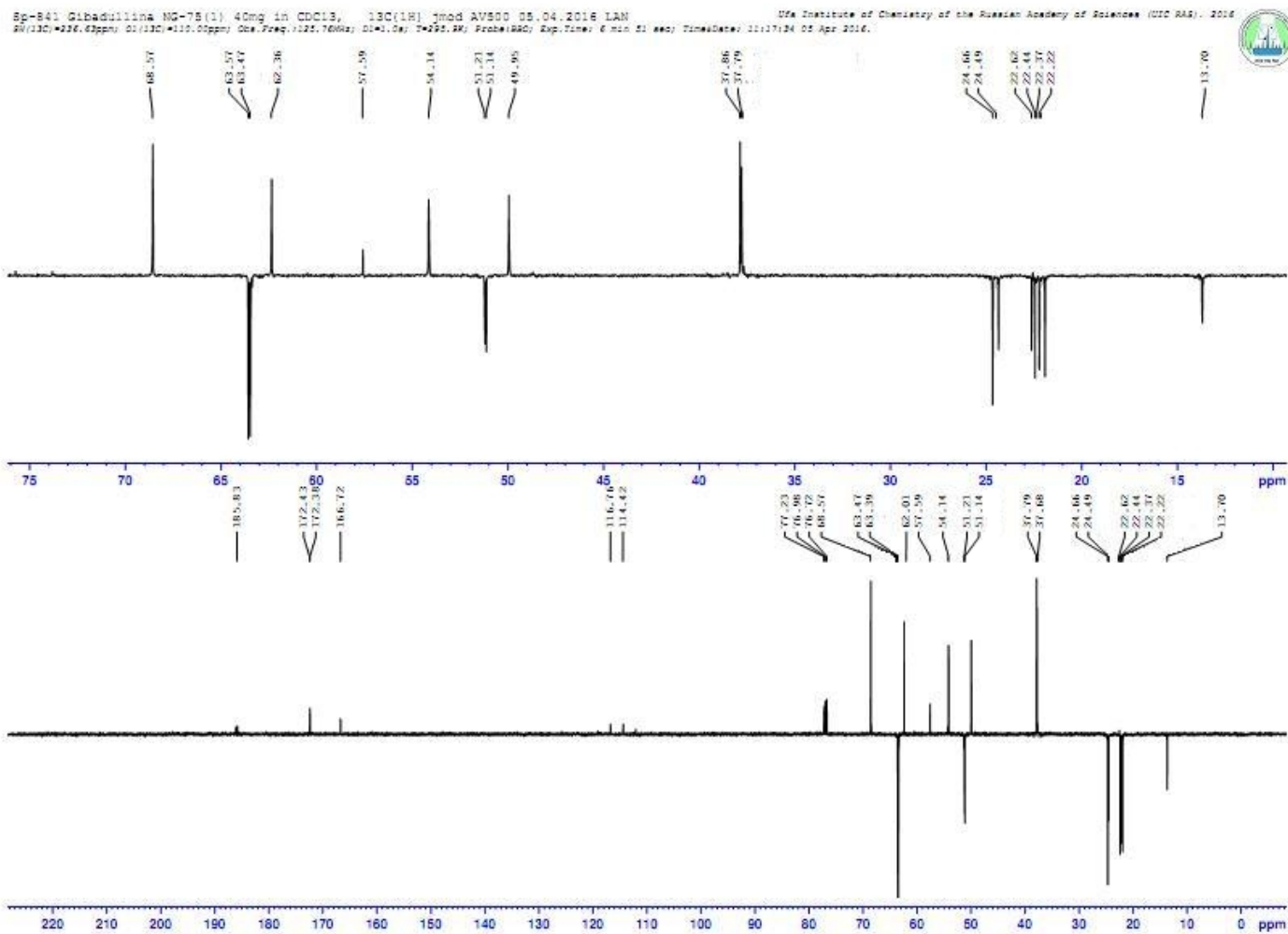
Sp-841 Gibadullina NG-75(1) 40mg in CDCl₃, 1H AV500 05.04.2016 LAN
 S/N(1H)=12.48ppm; G1(1H)=7.00ppm; Obs.Freq.:500.136KHz; G1=2.0s; T=295.1K; Probe:QNP; Exp.Time: 7 sec; TimeDate: 11/01/16 05 Apr 2016.

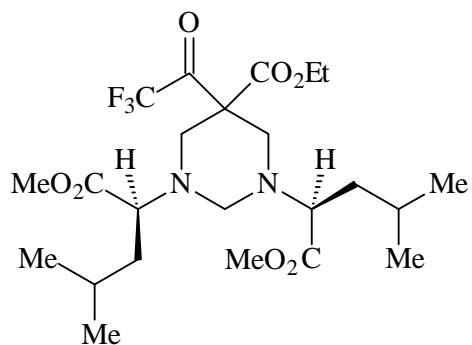
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4d



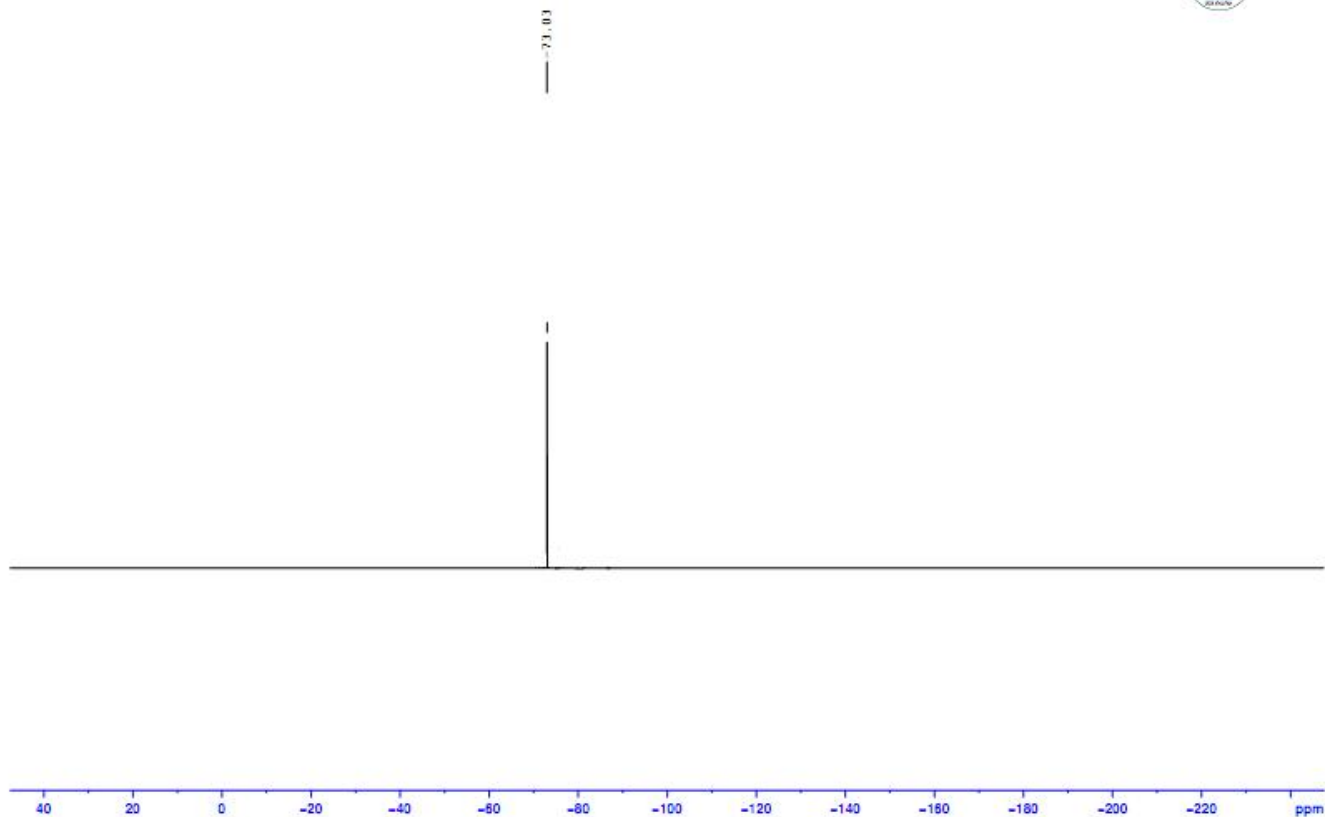


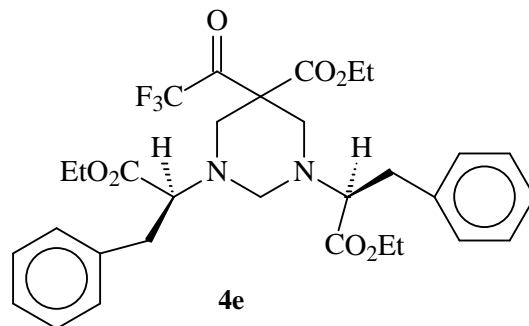
4d

Sp-1627 Qibadyllina NG-30 80mg in CDCl₃, TMS 19F(1H) Inv.gated spectrum AV500 07.08.2014 20V

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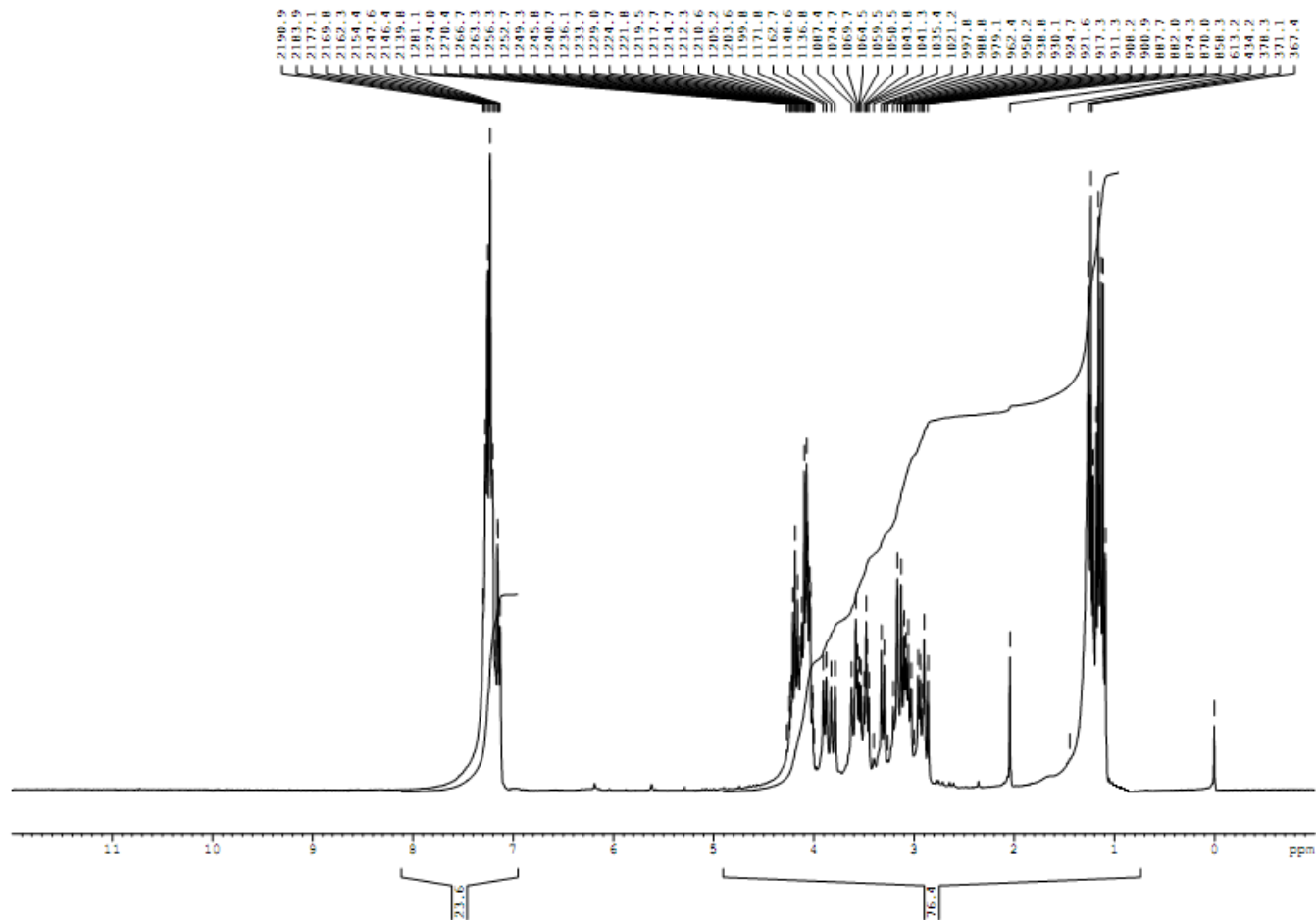
SW(19F) = 295.17ppm; Q1(19F) = -100.00ppm; Obs.Freq.: 470.59MHz; D1=1.0s; T=295.1K; Probe:BB0; Exp.Time:12 min 58 sec; TimeDate: 17:22:12 07 Aug 2014.

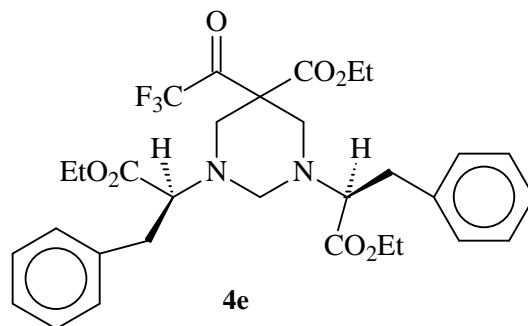




Sp-903 Gibadyllina NG-41-1 in CDCl₃ 1H AMX300 14.04.15 BIF

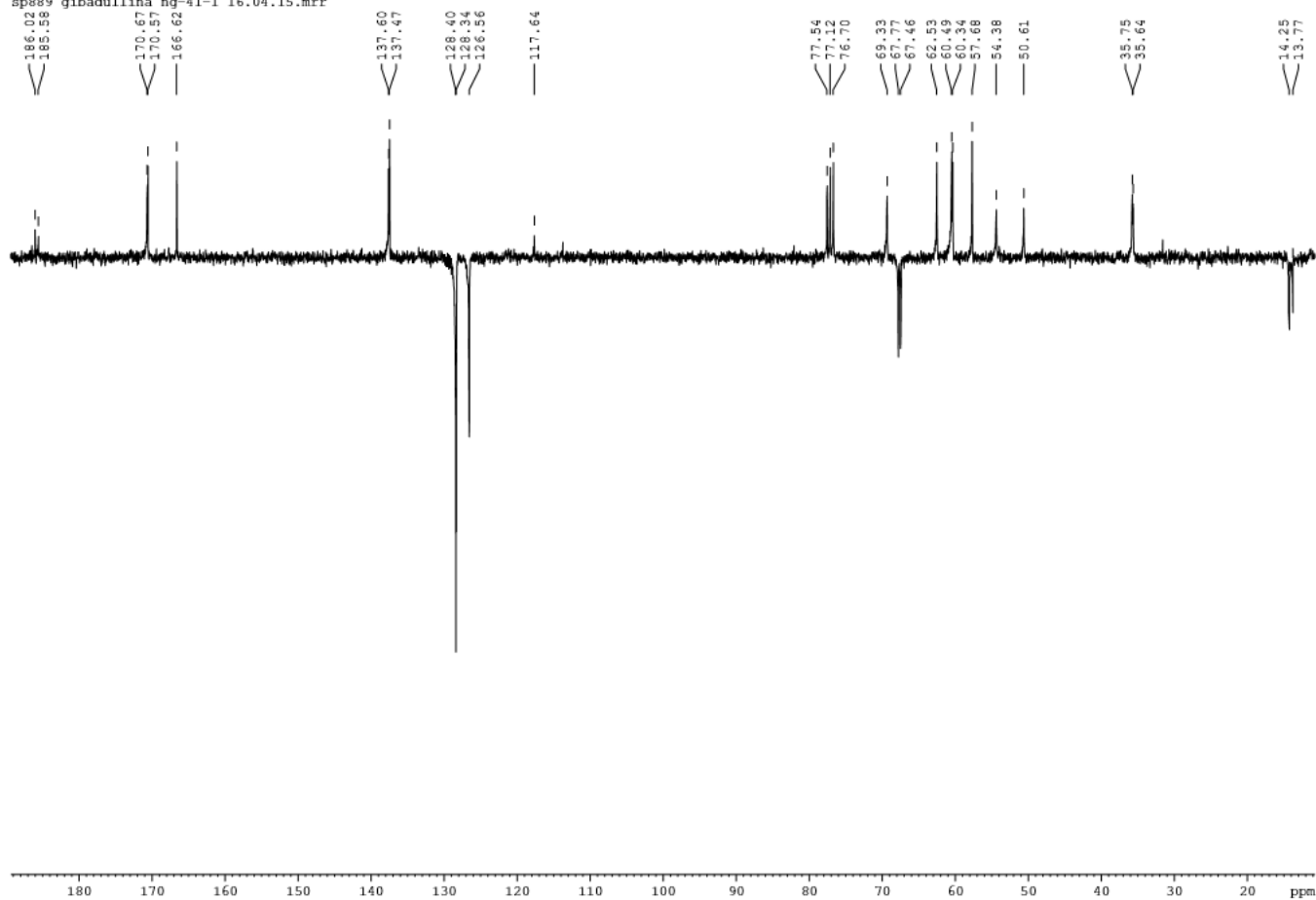
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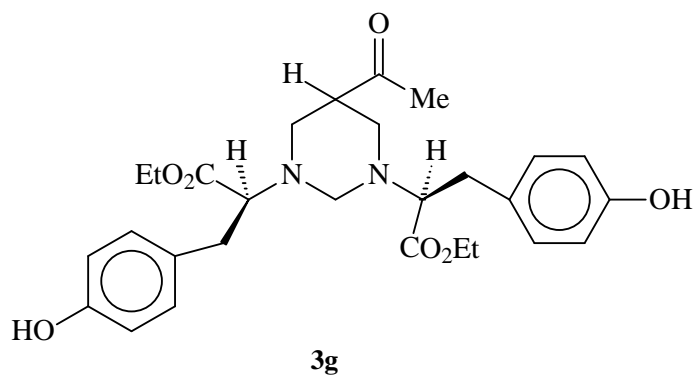




sp889 gibadullina ng-41-1 16.04.15.mrr

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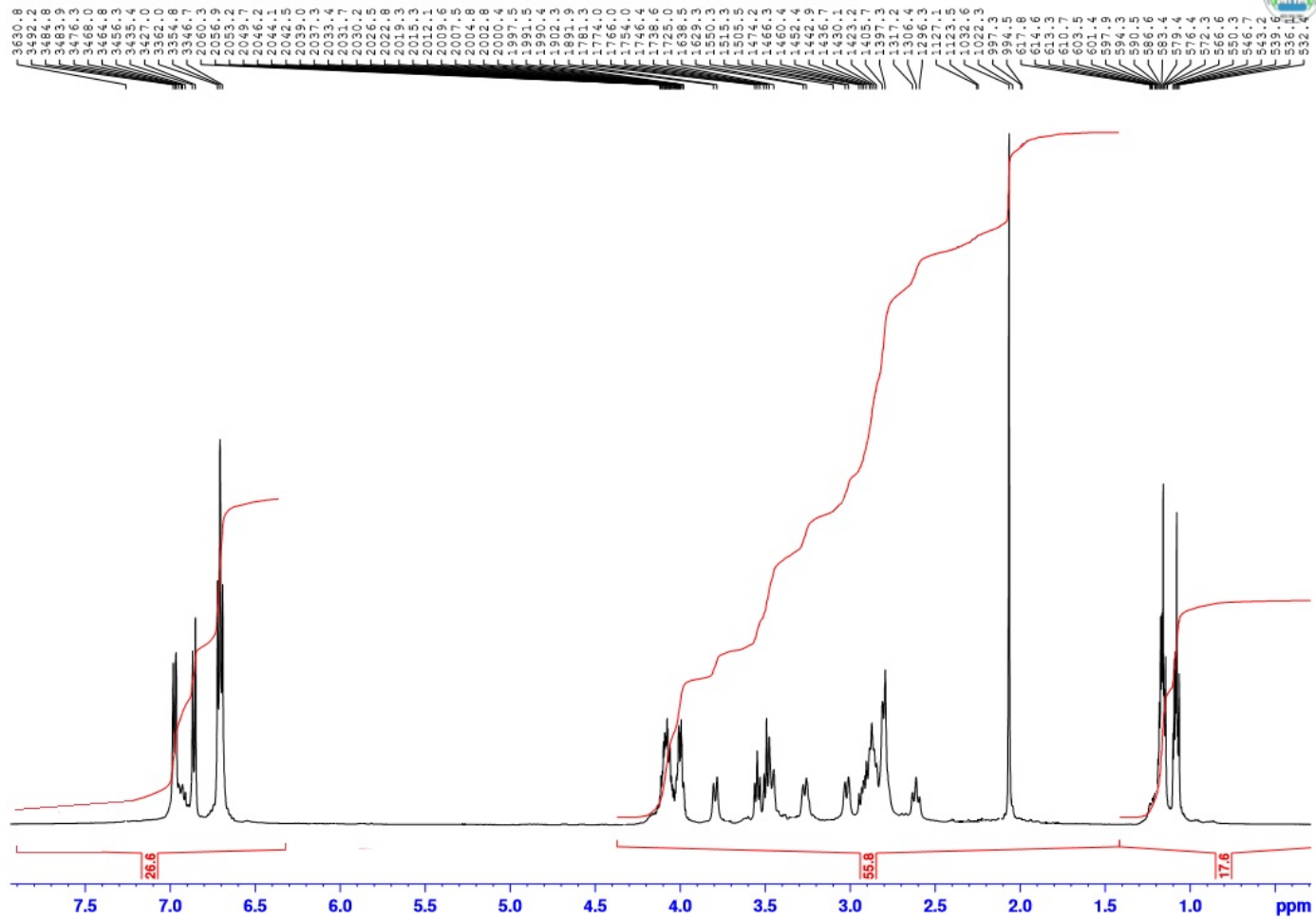


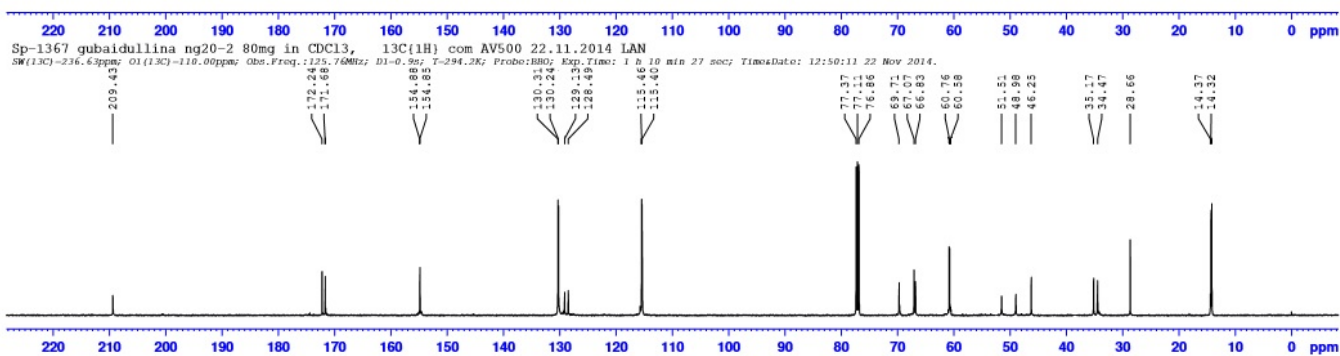
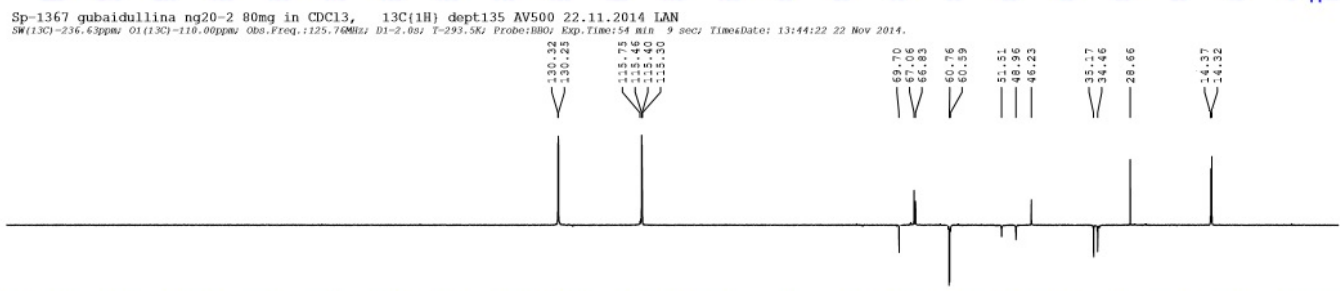
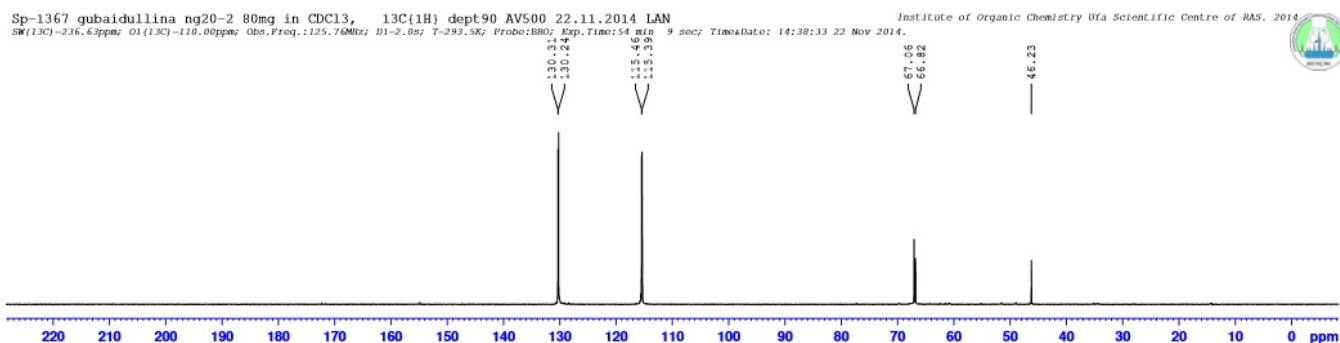
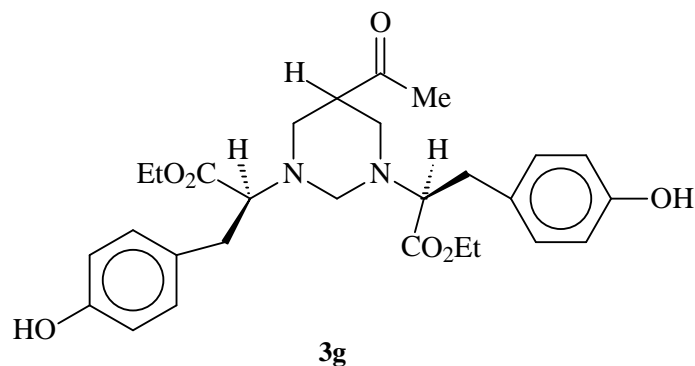


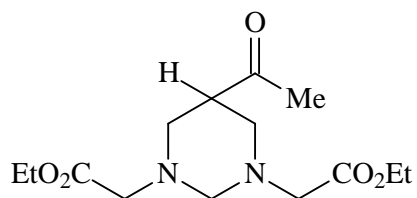
Sp-1367 gubaidullina ng20-2 80mg in CDCl₃, 1H AV500 22.11.2014 IAN

SW(1H)-19.99ppm 01(1H)-7.00ppm Obs.Freq.:500.13MHz; D1=5.0s; T=293.2K; Probe:BBO; Exp.Time: 1 min 25 sec; TimeDate: 11:39:36 22 Nov 2014.

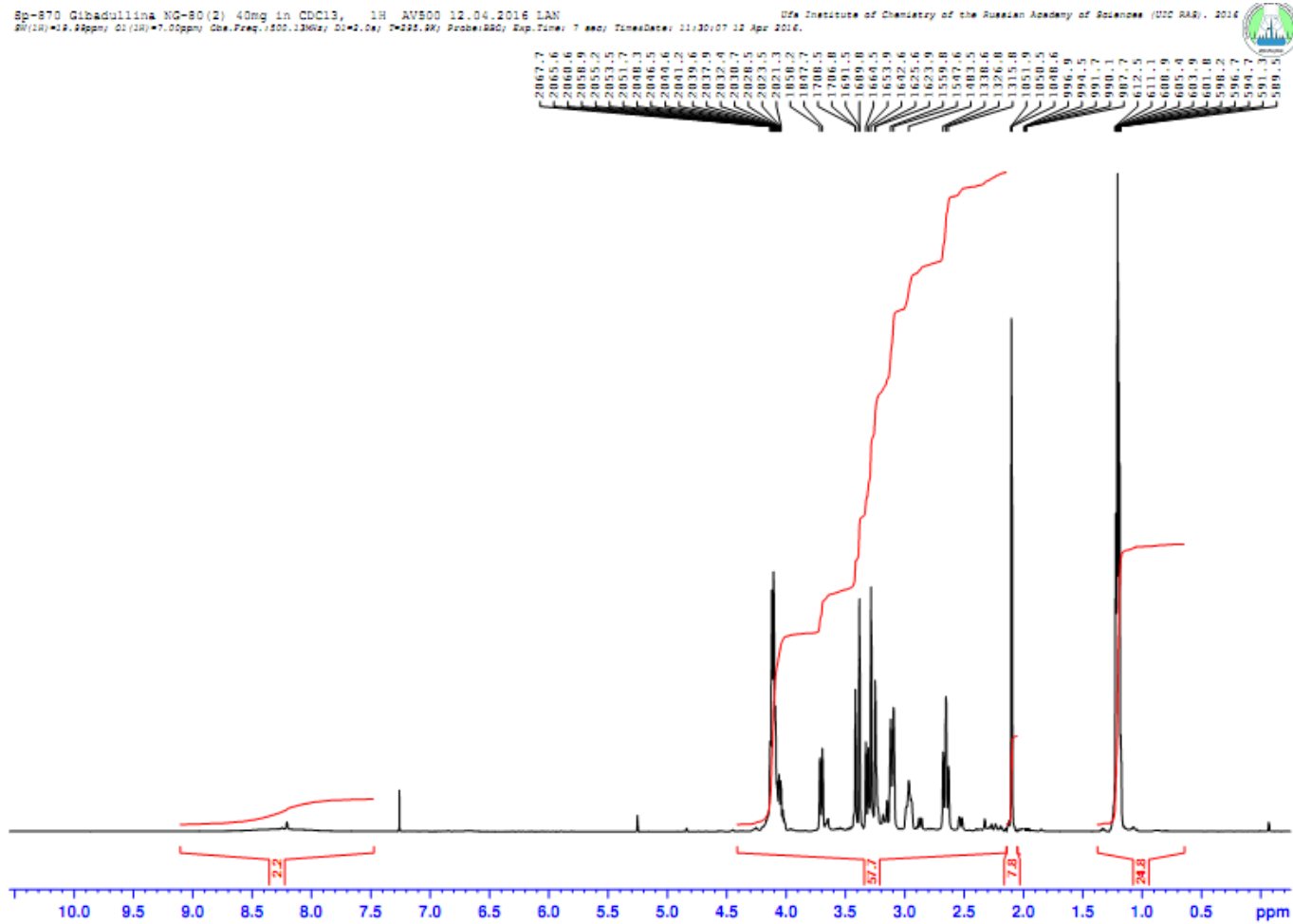
Institute of Organic Chemistry Ufa Scientific Centre of RAS, 2014

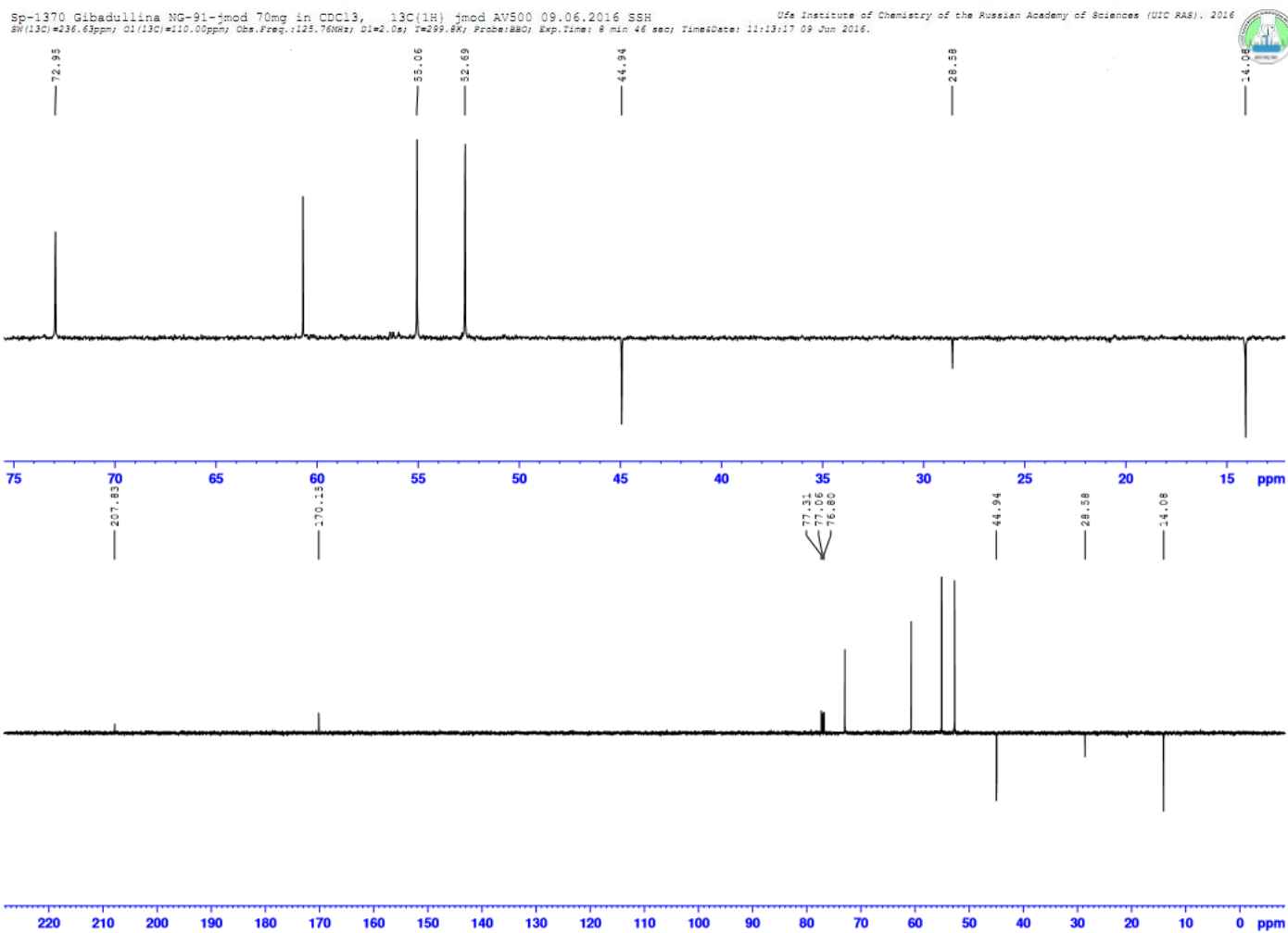
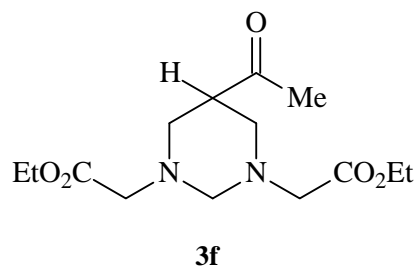


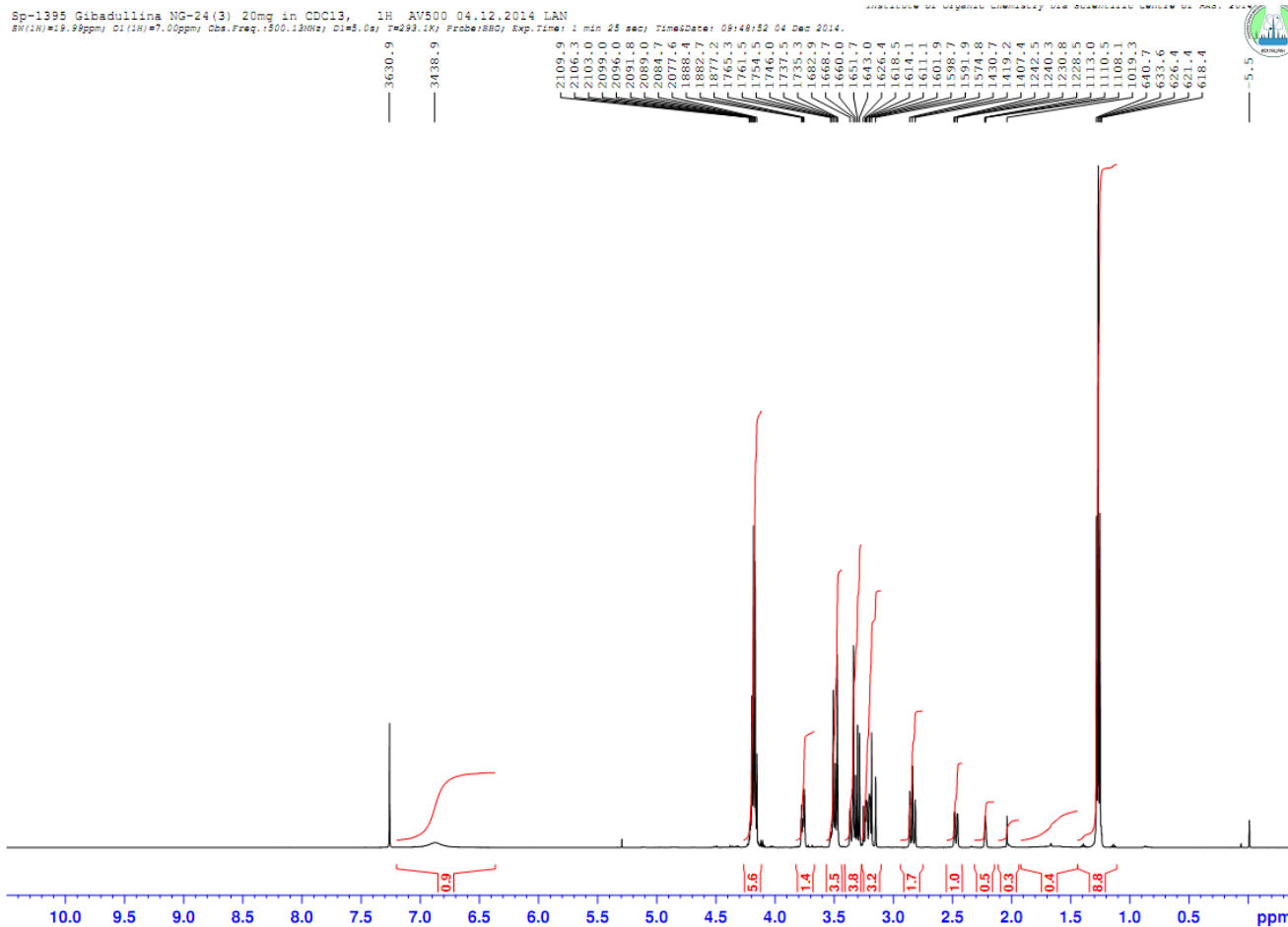
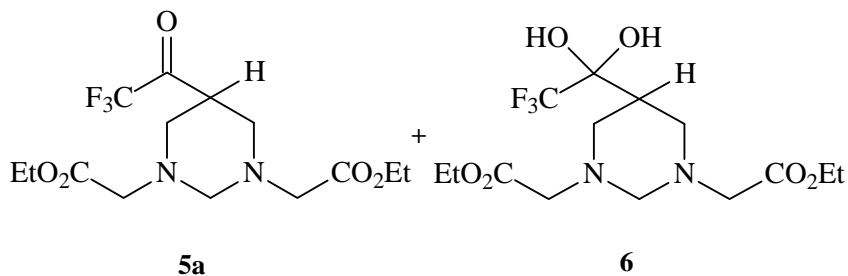


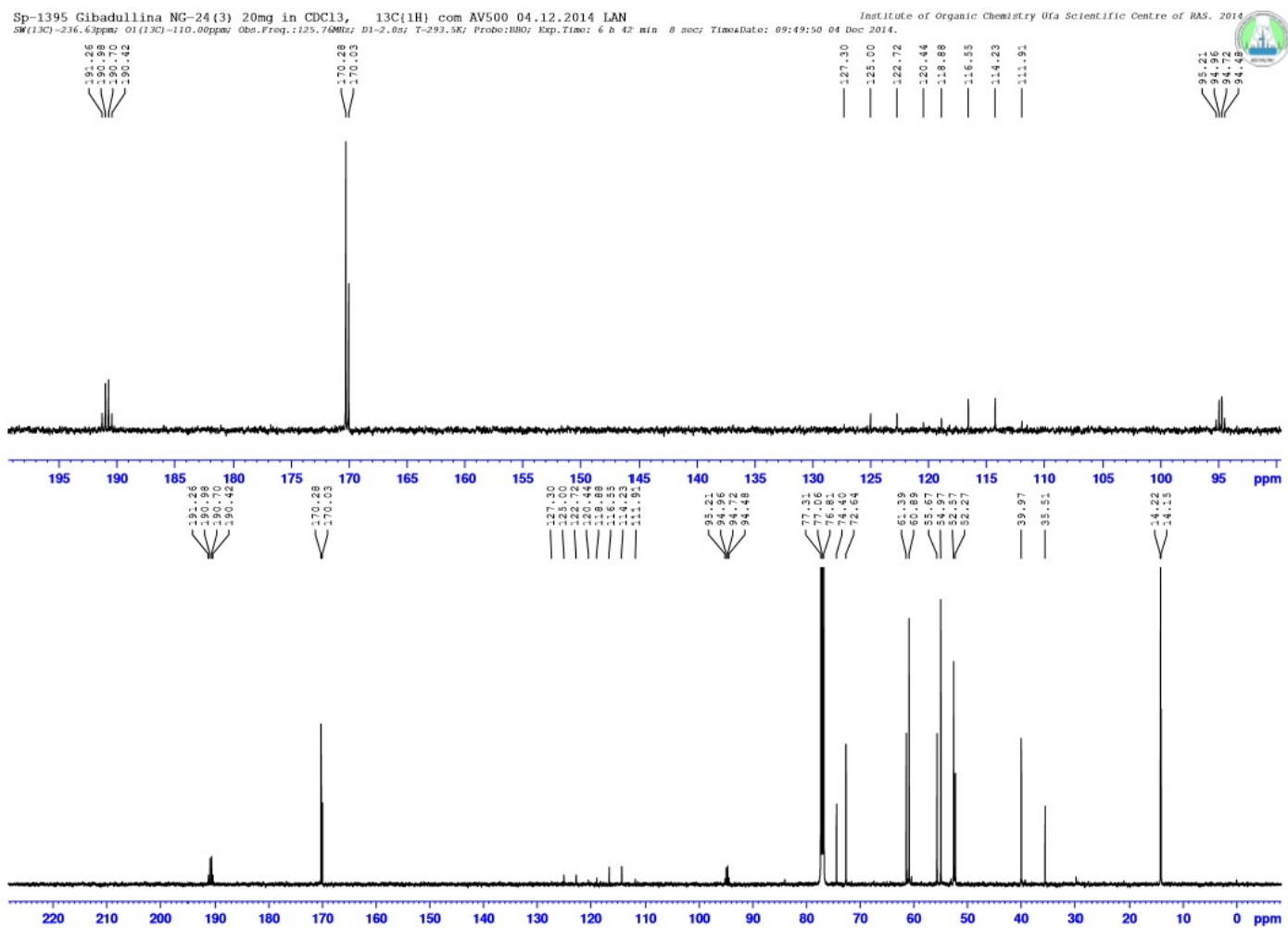
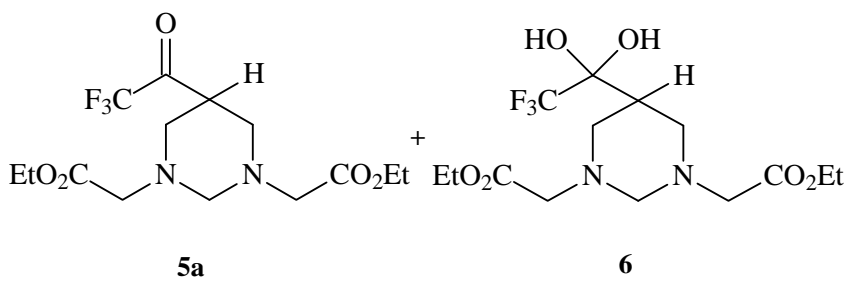


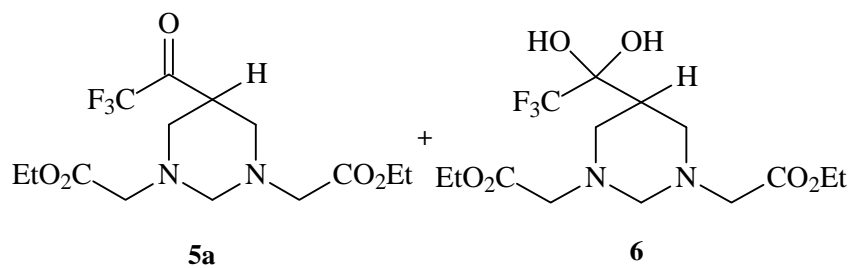
3f





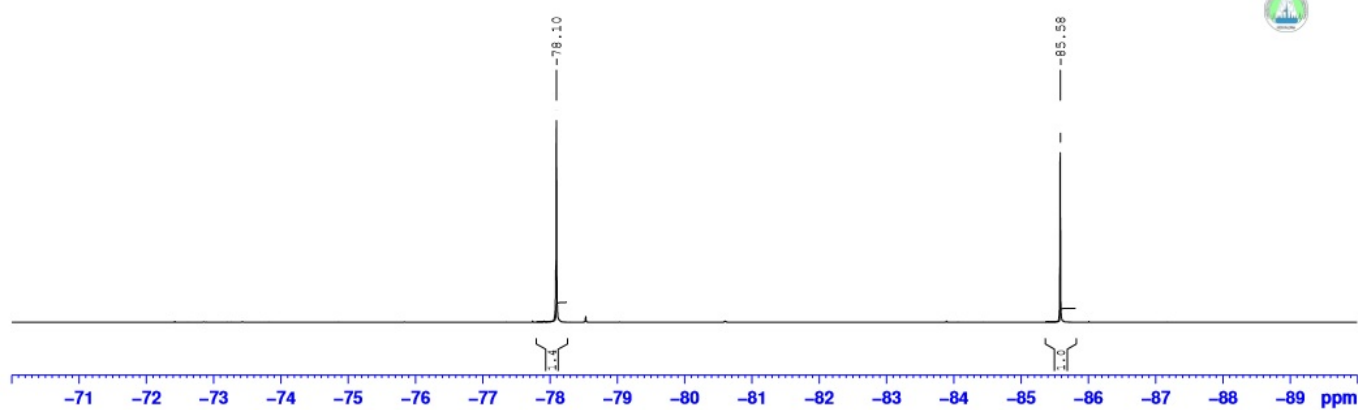




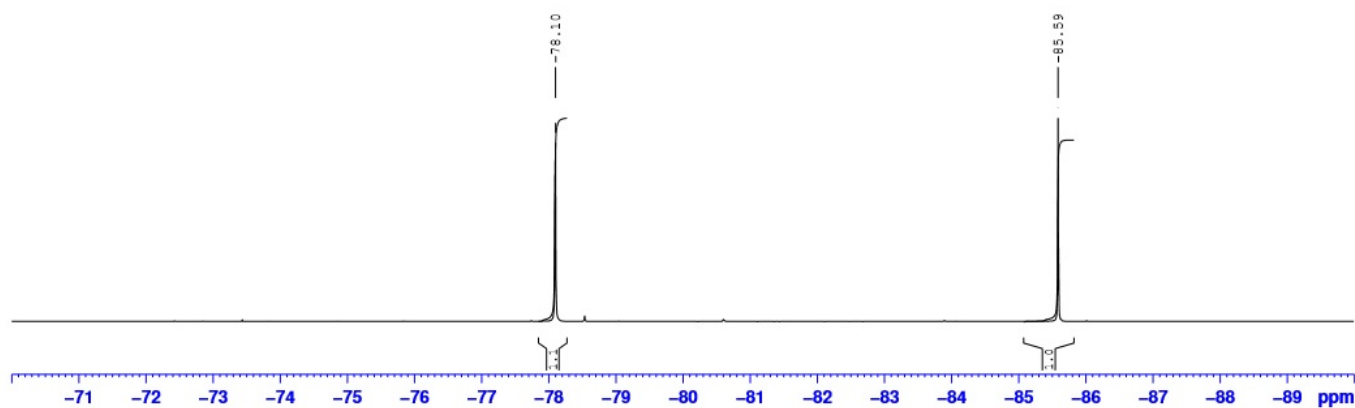


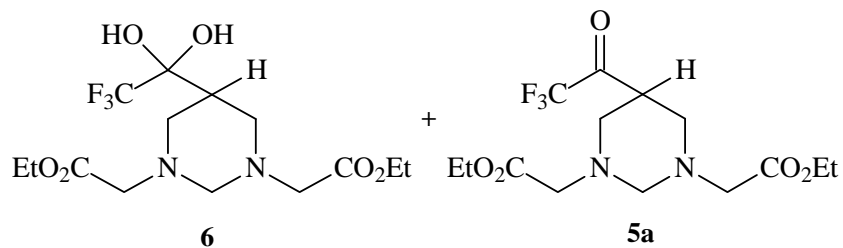
Sp-1395 Gibadullina NG-24(3) 20mg in CDCl₃, ¹⁹F spectrum AV500 04.12.2014 LAN
 SW(19F)-60.37ppm; O1(19F)-80.00ppm; Obs.Freq.:470.59MHz; D1-1.0s; T-293.3K; Probe:BBQ; Exp.Time: 3 min 49 sec; Time&Date: 09:41:54 04 Dec 2014.

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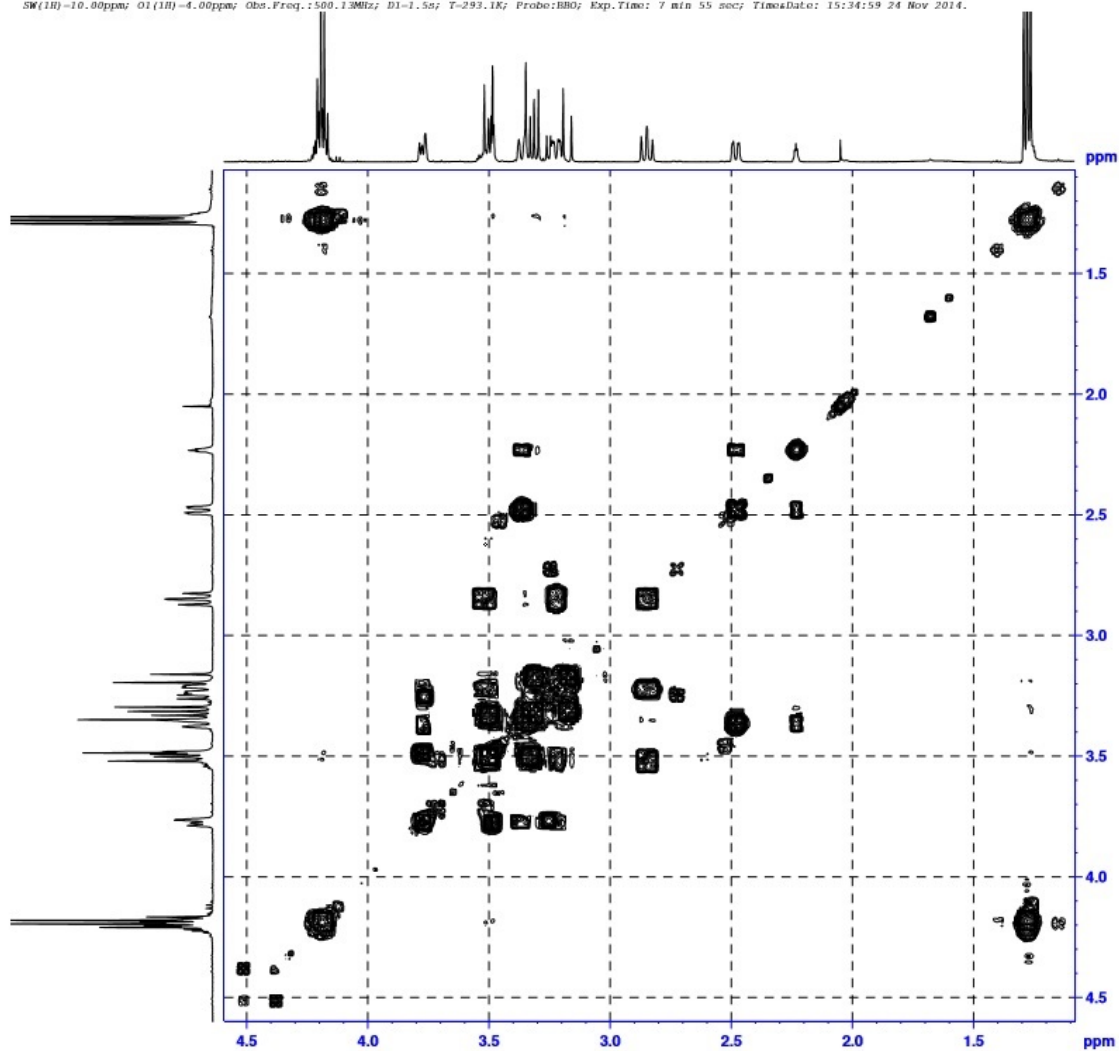
Sp-1395 Gibadullina NG-24(3) 20mg in CDCl₃, ¹⁹F{¹H} inv.gated spectrum AV500 04.12.2014 LAN
 SW(19F)-498.09ppm; O1(19F)-100.00ppm; Obs.Freq.:470.59MHz; D1-1.0s; T-293.3K; Probe:BBQ; Exp.Time: 3 min 49 sec; Time&Date: 09:34:49 04 Dec 2014.

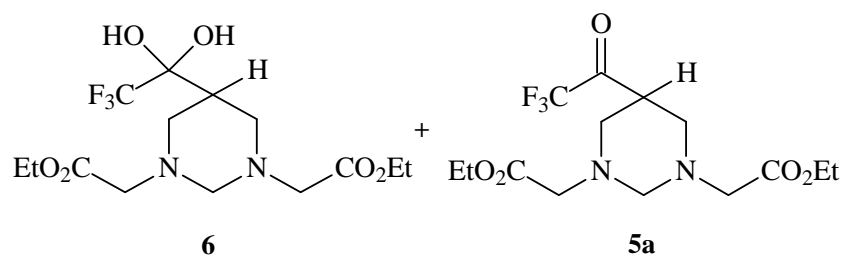




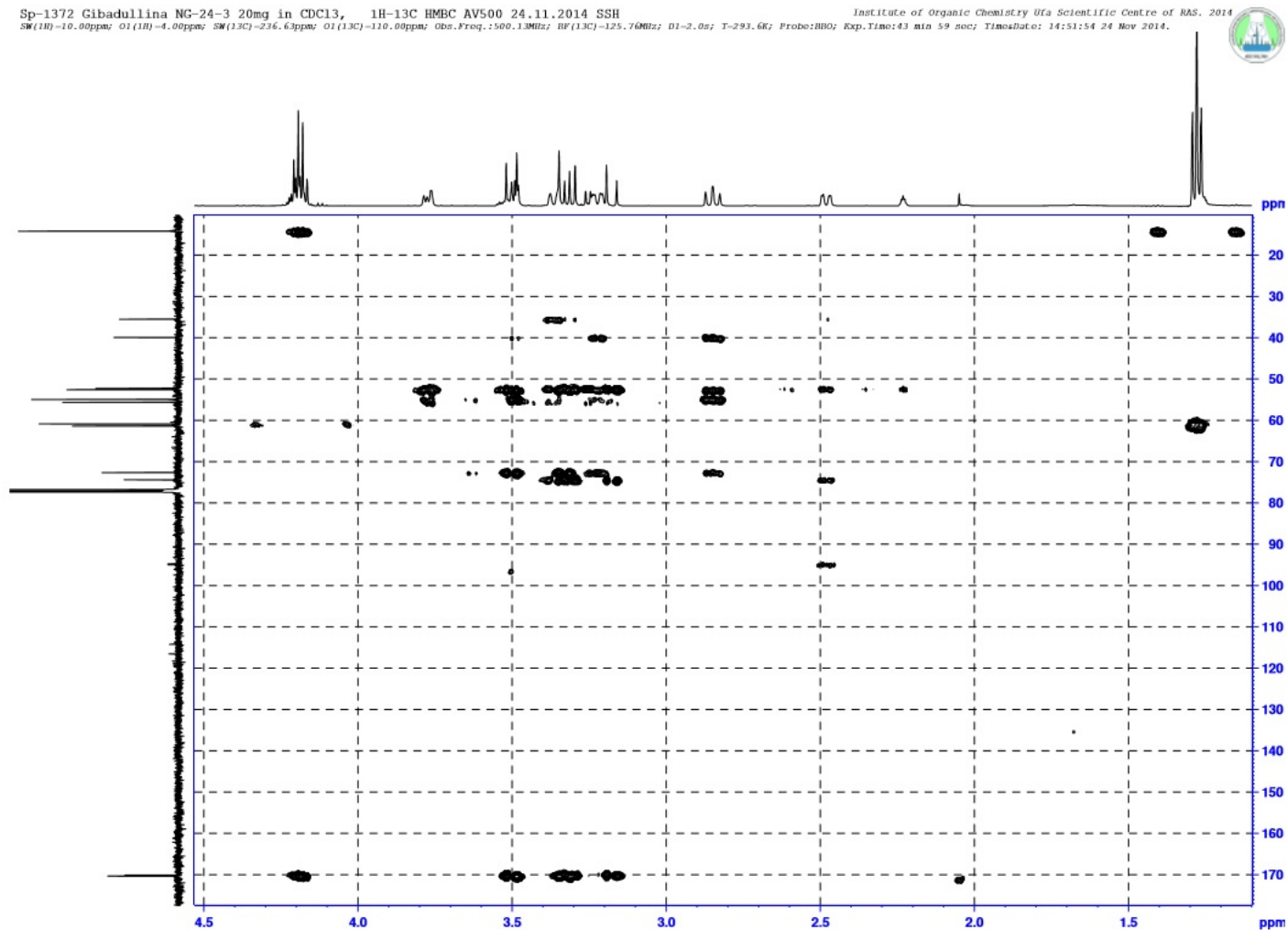
Sp-1372 Gibadullina NG-24-3 20mg in CDCl₃, 1H-1H COSY AV500 24.11.2014 SSH
SW(H1)-10.00ppm; O1(H1)-4.00ppm; Obs.Freq.:500.13MHz; D1-1.5s; T-293.1K; Probe:BBO; Exp.Time: 7 min 55 sec; TimeDate: 15:34:59 24 Nov 2014.

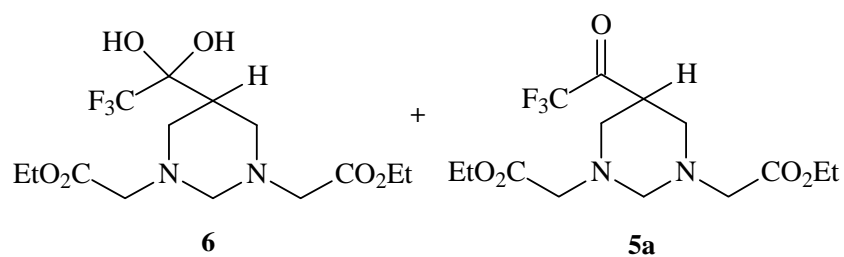
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Sp-1372 Gibadullina NG-24-3 20mg in CDCl₃, 1H-13C HMBEC AV500 24.11.2014 SSH
SW(1H)-10.00ppm; O1(1H)-4.00ppm; SW(13C)-236.63ppm; O1(13C)-110.00ppm; Obs.Freq.:500.13MHz; RF(13C)-125.76MHz; D1-3.0s; T-293.6K; Probe:BB0; Exp.Time:43 min 59 sec; TimeDate: 14:51:54 24 Nov 2014.

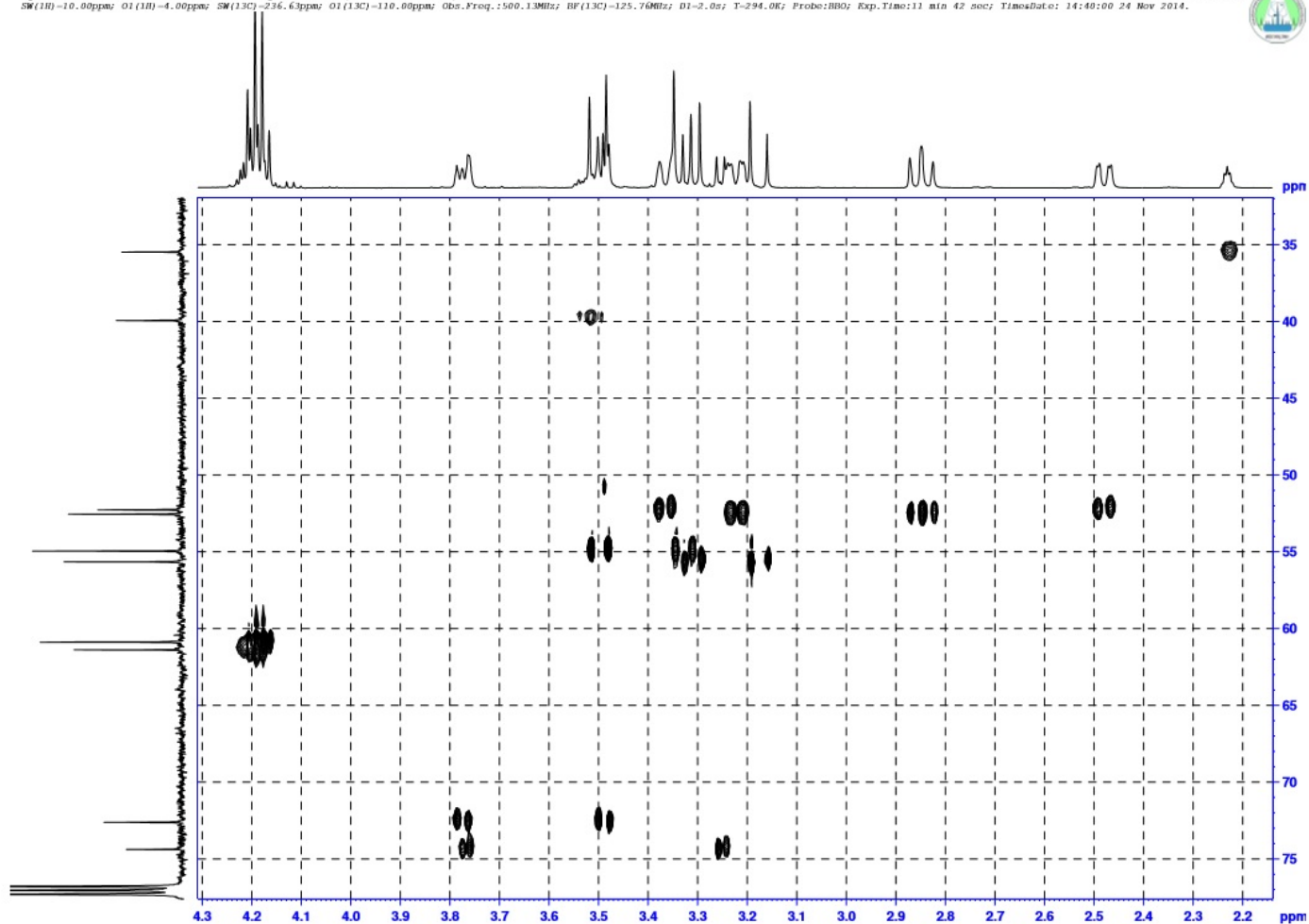


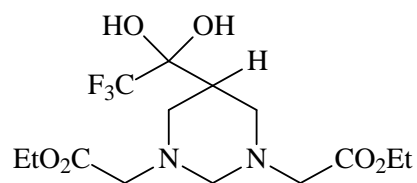


Sp-1372 Gibadullina NG-24-3 20mg in CDCl₃, 1H-13C HSQC AV500 24.11.2014 SSH

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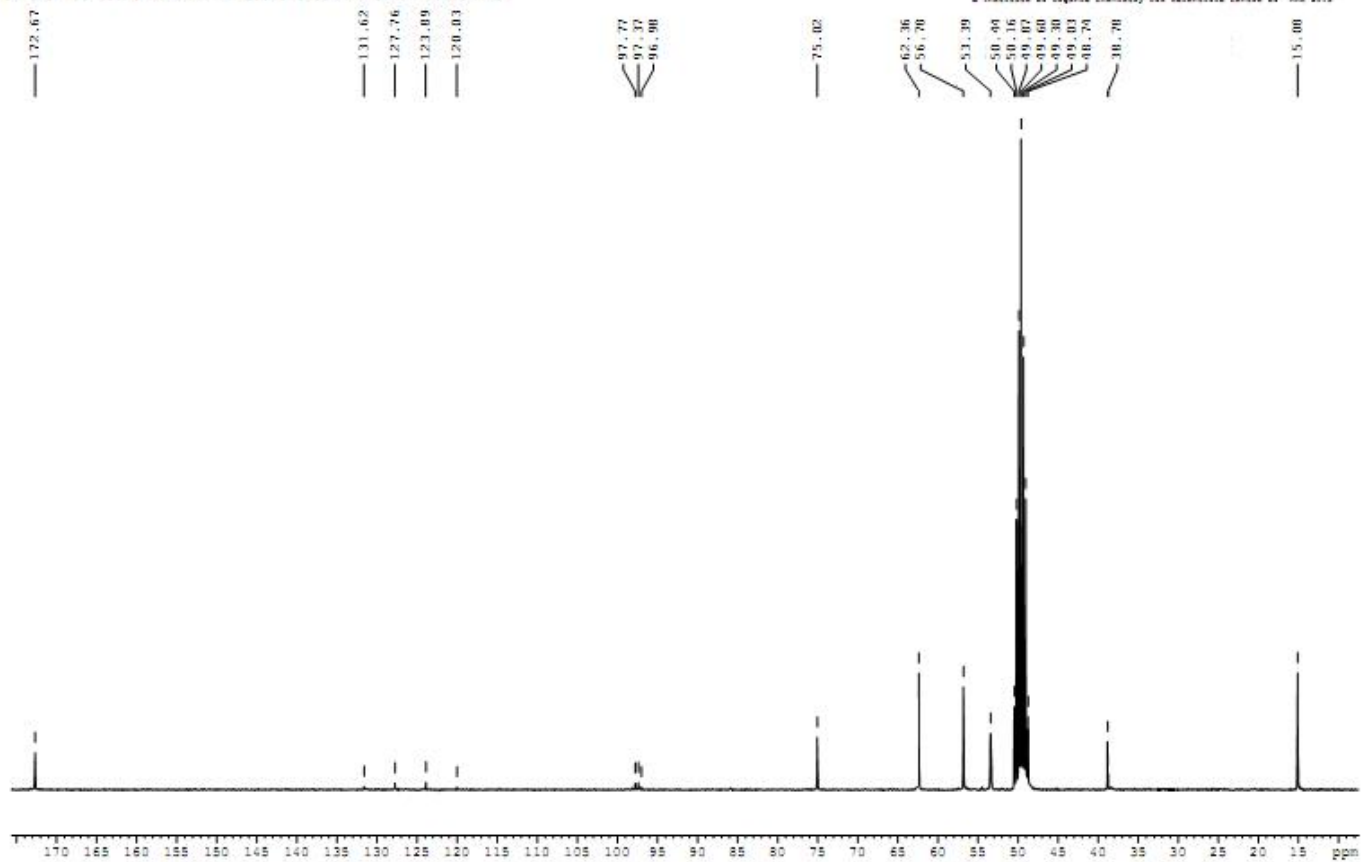
SW(1H)-10.00ppm; O1(1H)-4.00ppm; SW(13C)-236.63ppm; O1(13C)-110.00ppm; Obs.Freq.:500.13MHz; RF(13C)-125.76MHz; D1-2.0s; T-294.0K; Probe:BBO; Exp.Time:11 min 42 sec; TimesDate: 14:40:00 24 Nov 2014.

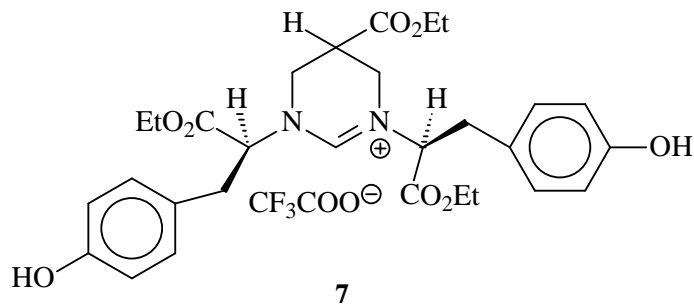


**6**

SP-549 13C COM GIBADULLINA NG33-3 CD3OD 10MG. 04-05.03 LVS.

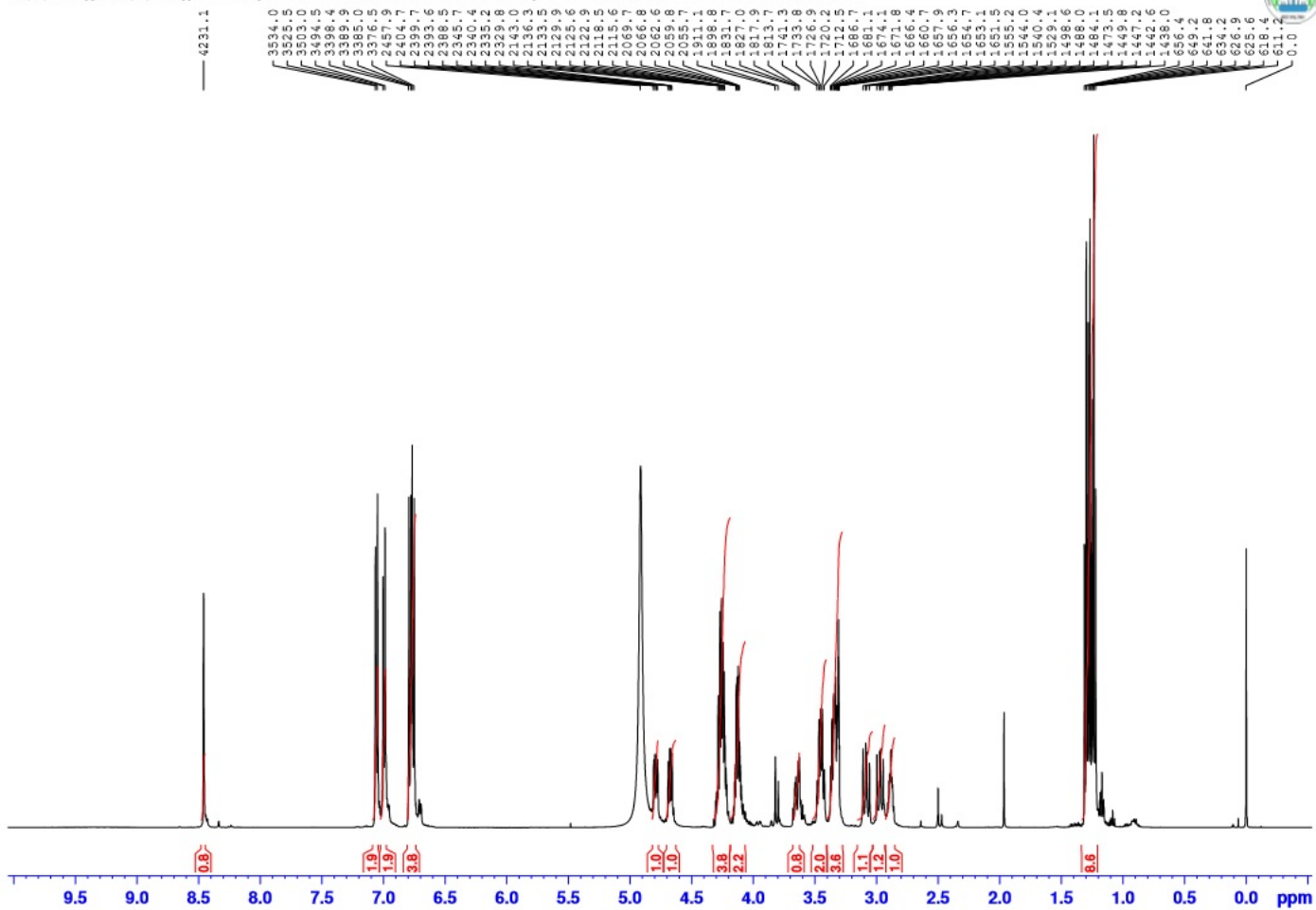
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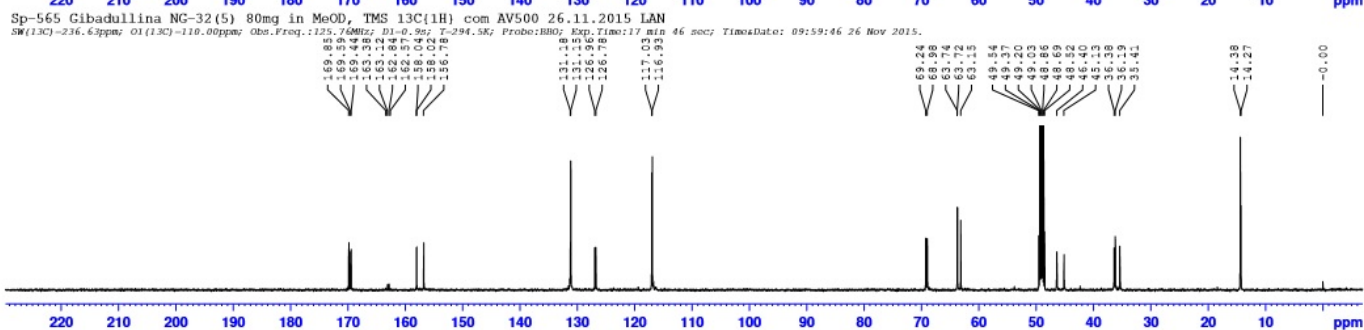
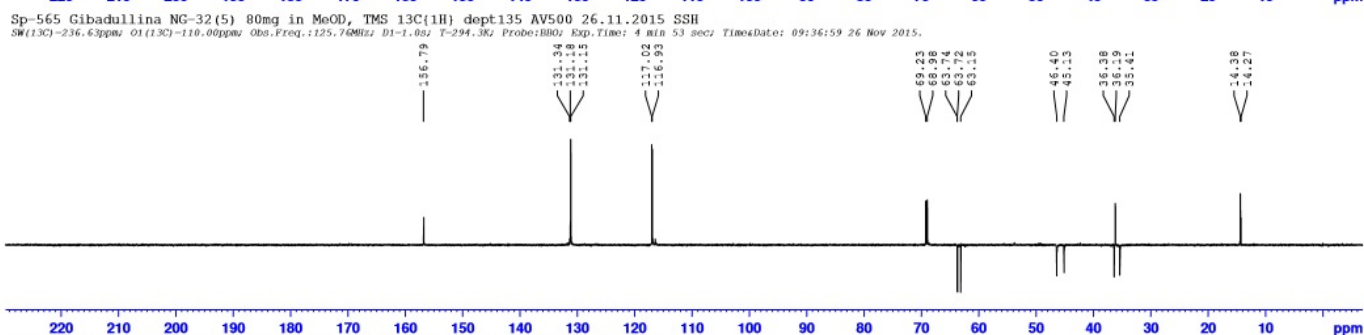
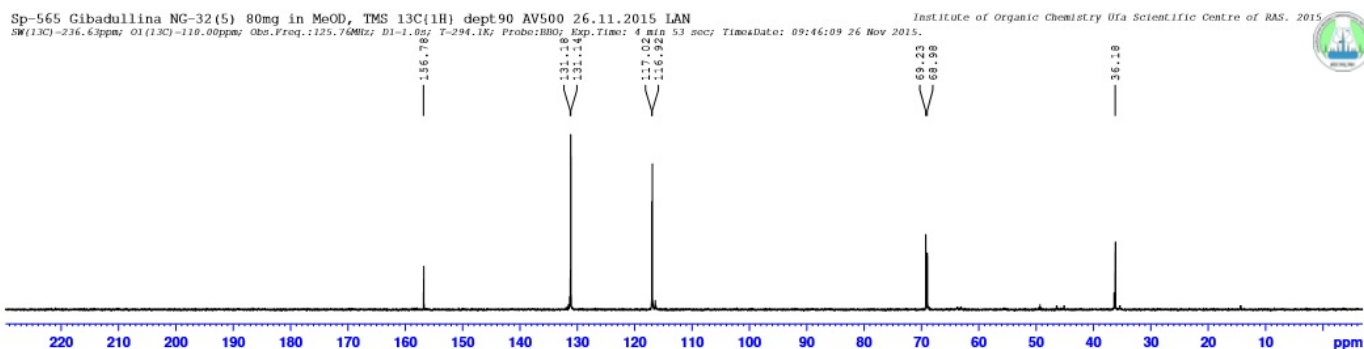
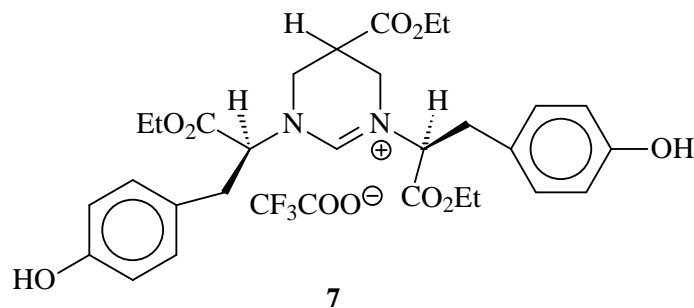


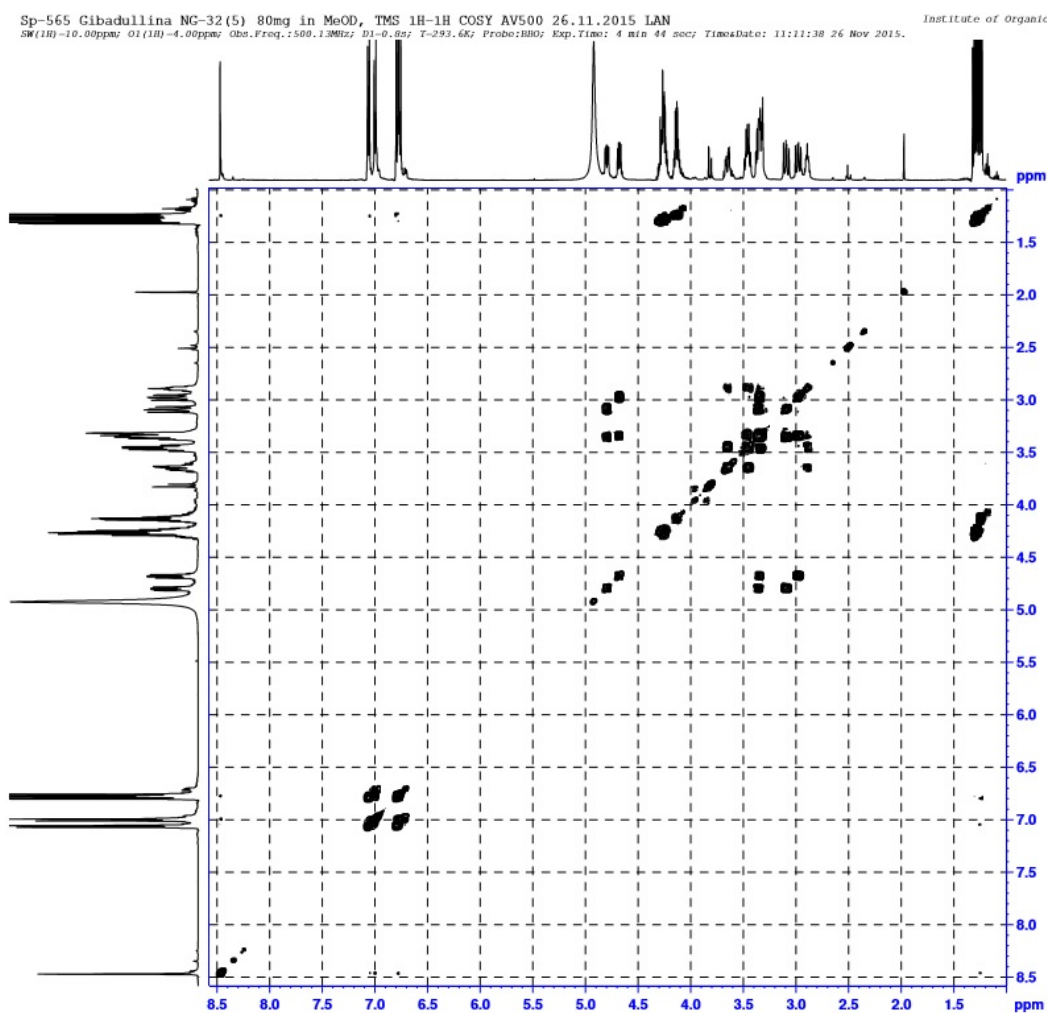
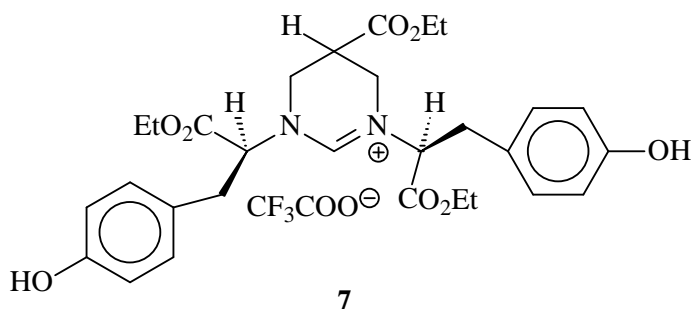


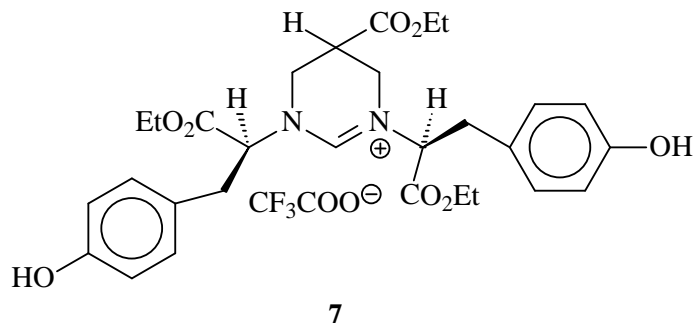
Sp-565 Cibadullina NG-32(5) 80mg in MeOD, TMS 1H AV500 26.11.2015 SSH
 SW(1H)-19.99ppm 01(1H)-7.00ppm Obs.Freq.:500.13MHz D1-5.0s T-293.6K Probe:BB04 Exp.Time: 1 min 25 sec Time4Date: 09:31:44 26 Nov 2015.

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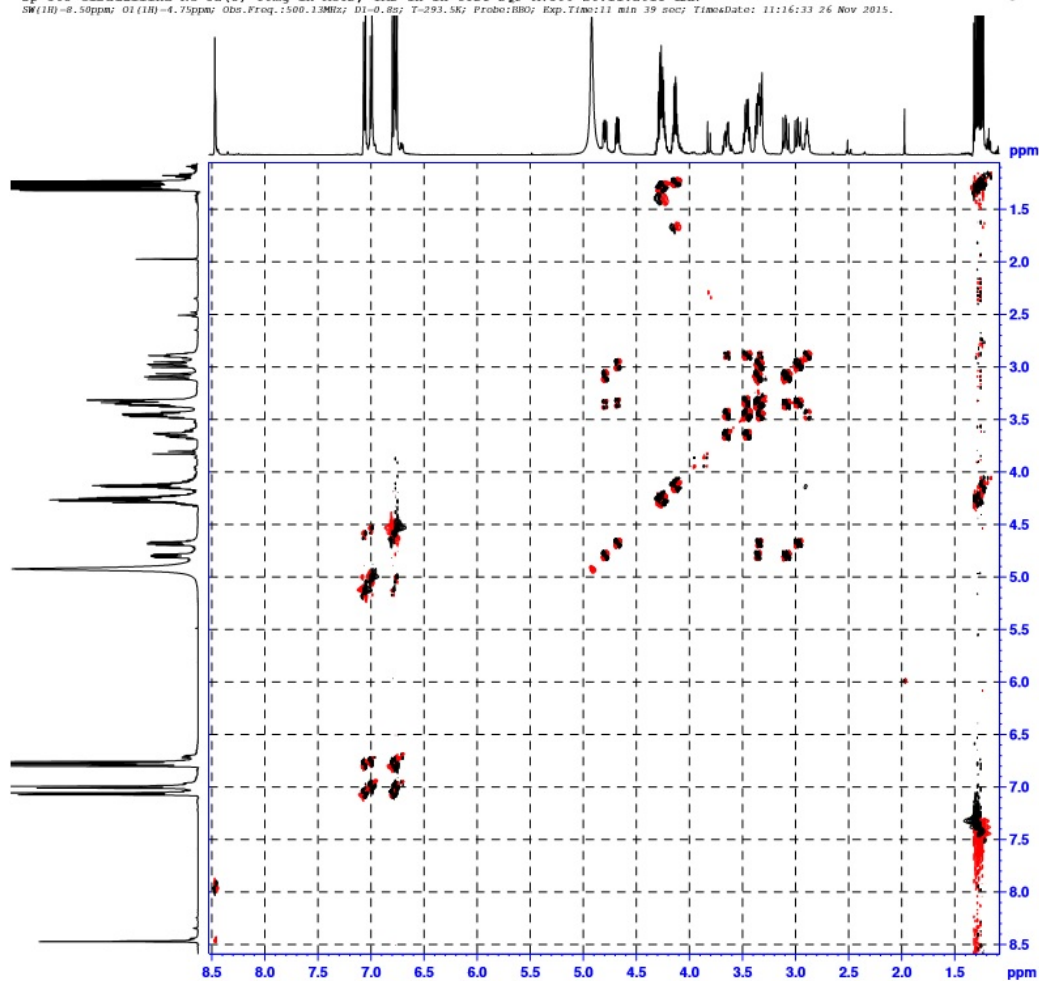


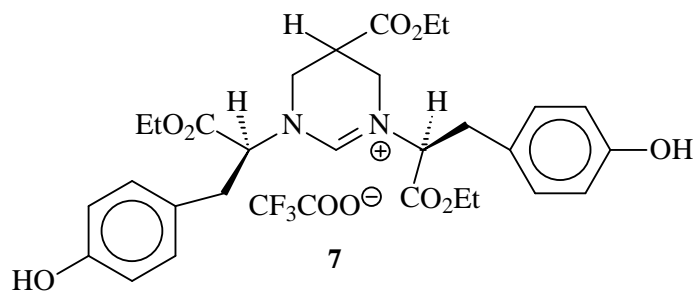




Sp-565 Gibadullina NG-32(5) 80mg in MeOD, TMS 1H-1H COSY-DQF AV500 26.11.2015 LAN
SW(1H) = 8.50ppm; O1(1H) = 4.75ppm; Obs. Freq.: 500.13MHz; D1 = 0.8s; T = 293.5K; Probe: BBO; Exp. Time: 11 min 39 sec; Time & Date: 11:16:33 26 Nov 2015.

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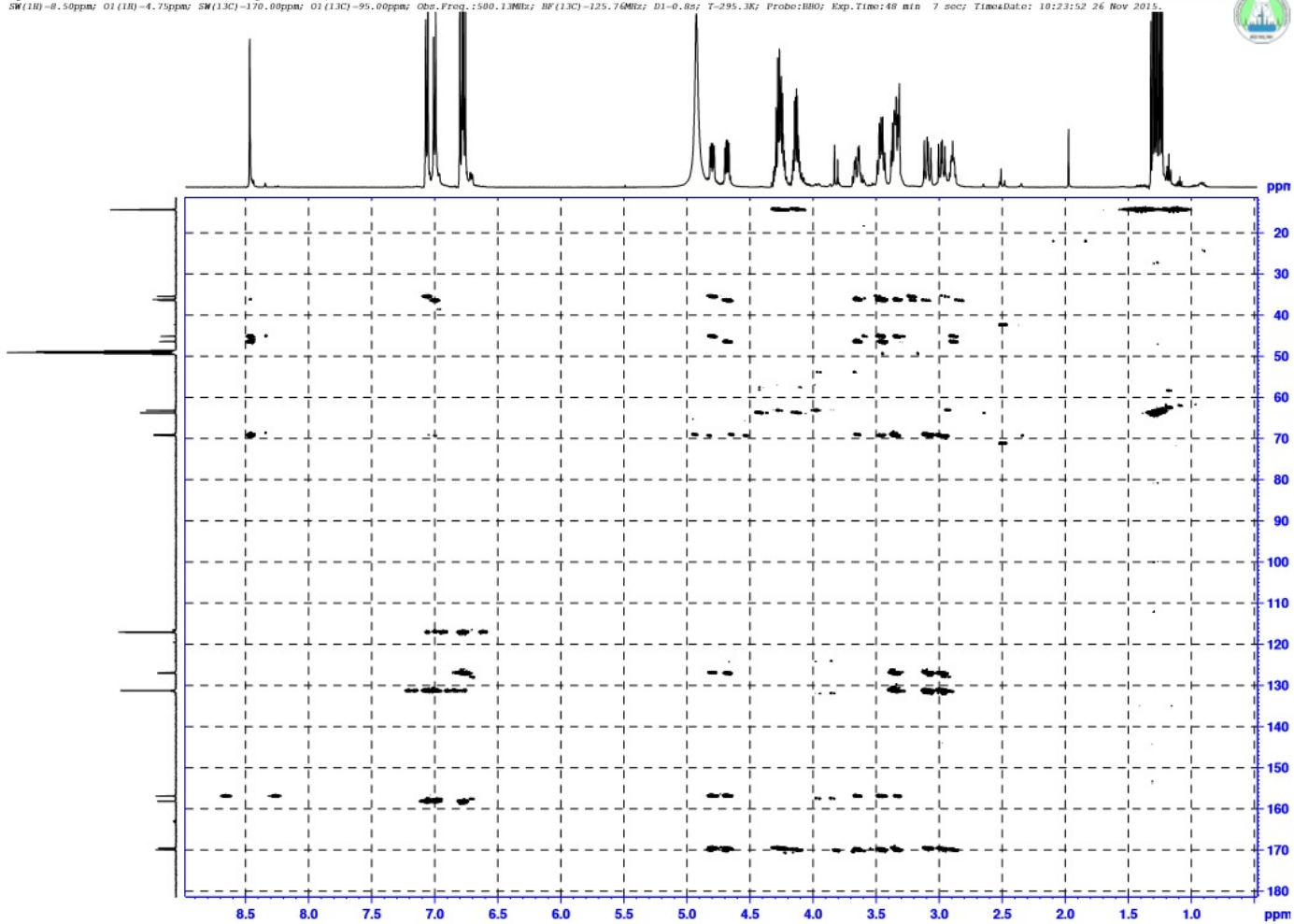


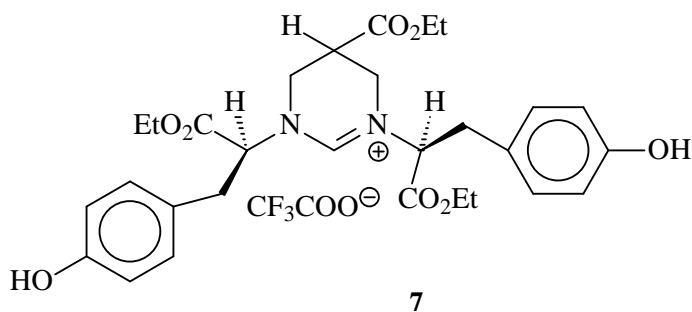


Sp-565 Gibadullina NG-32(5) 80mg in MeOD, TMS 1H-13C HMBC AV500 26.11.2015 LAN

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SW(1H)-8.50ppm; O1(1H)-4.75ppm; SW(13C)-170.00ppm; O1(13C)-95.00ppm; Obs.Prog.:500.13MHz; HF(13C)-125.76MHz; D1=0.8s; T=295.3K; Probo:HBO; Exp.Time:48 min 7 sec; TimeoDate: 10:23:52 26 Nov 2015.

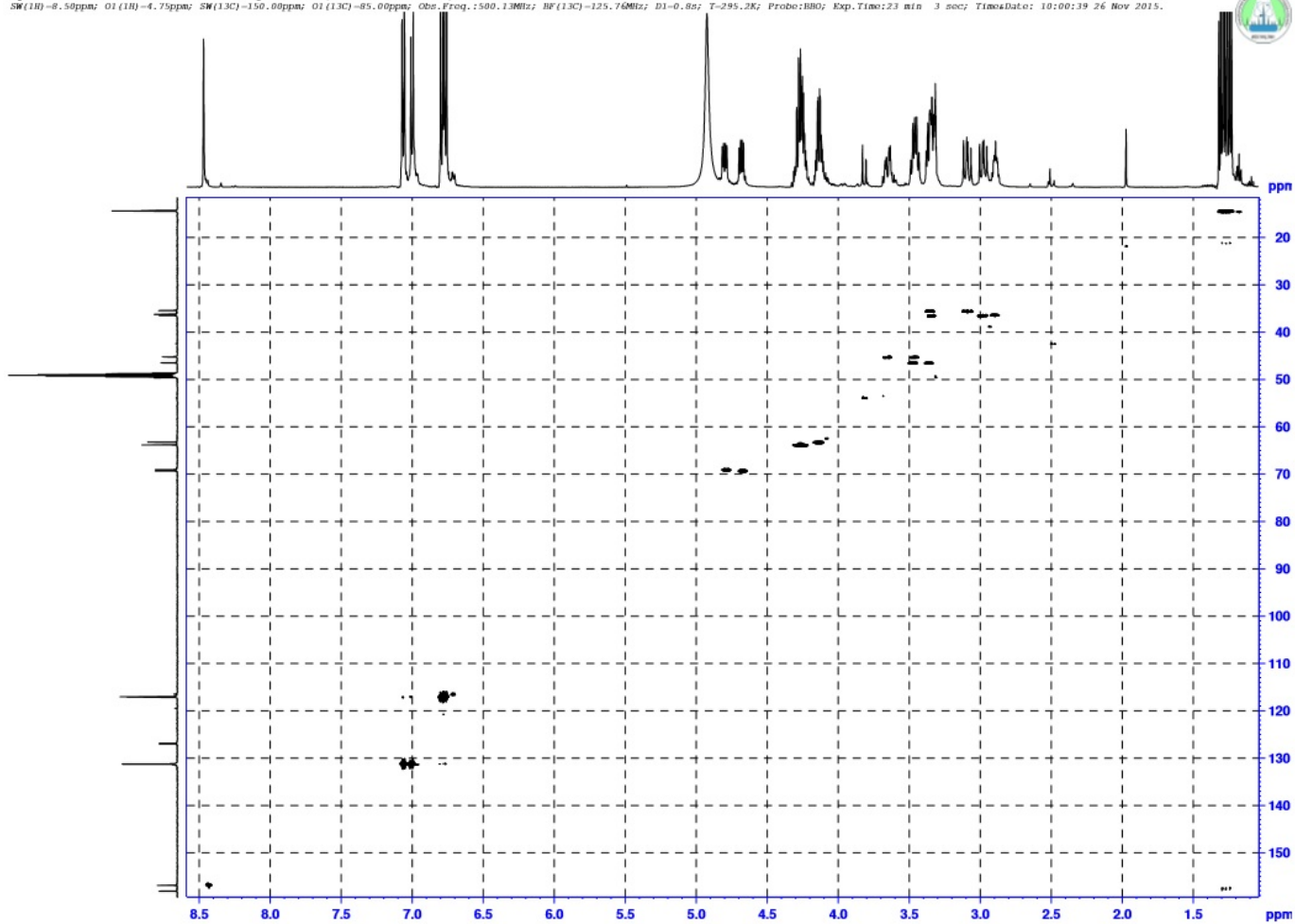


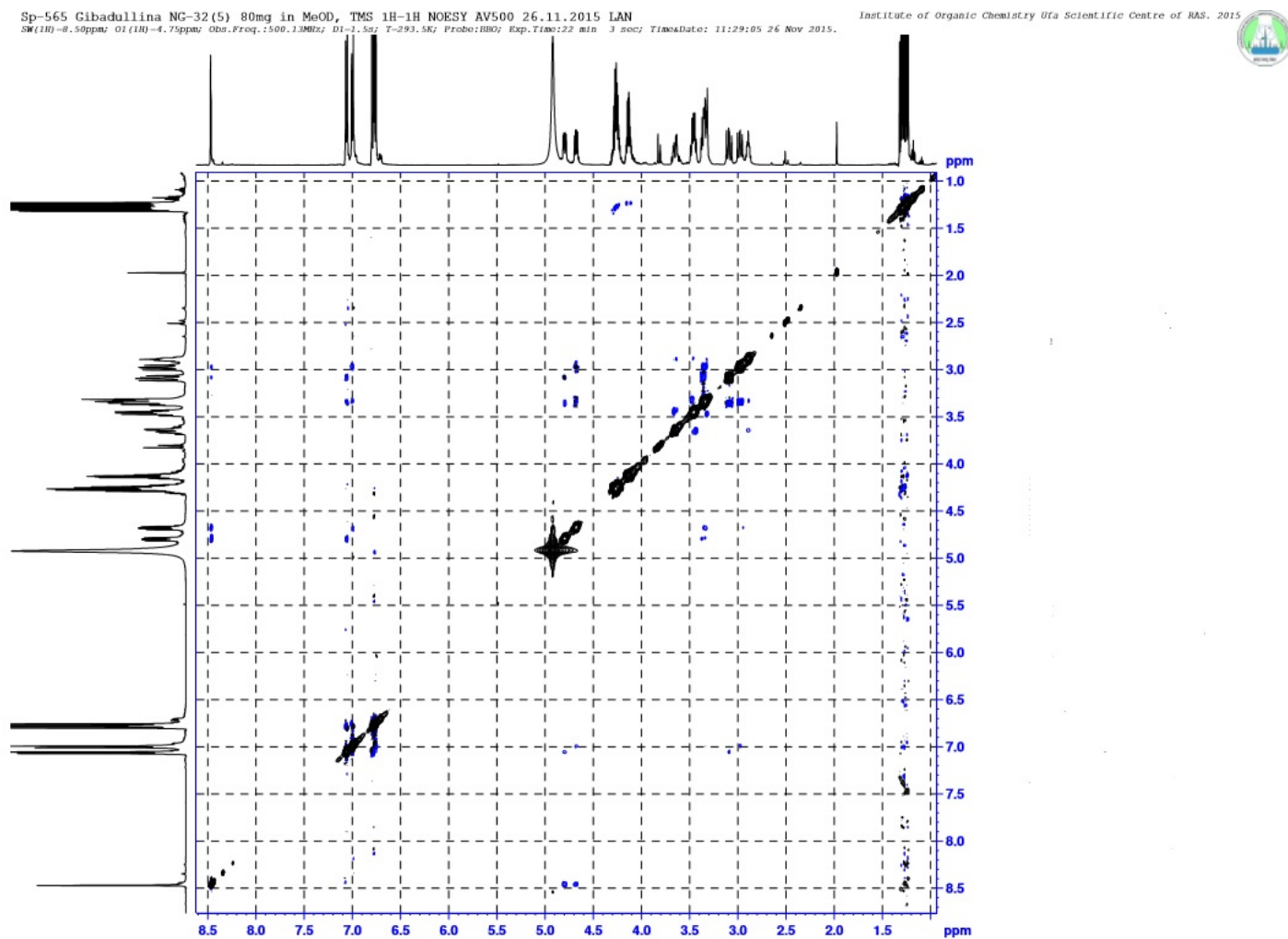
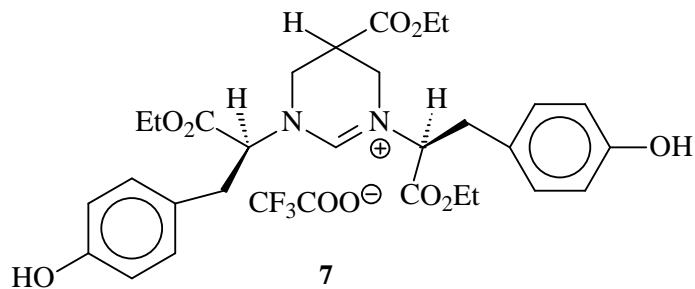


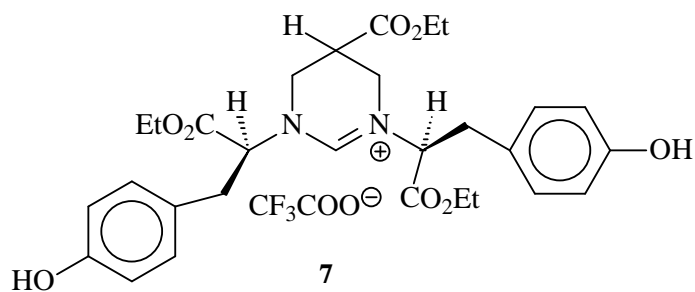
Sp-565 Gibadullina NG-32(5) 80mg in MeOD, TMS 1H-13C HSQC AV500 26.11.2015 LAN

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SW(1H)-8.50ppm; O1(1H)-4.75ppm; SW(13C)-150.00ppm; O1(13C)-85.00ppm; Obs. Freq.:500.13MHz; HF(13C)-125.74MHz; D1-0.8s; T-295.2K; Probe:BBO; Exp.Time:23 min 3 sec; TimeDate: 10:00:39 26 Nov 2015.



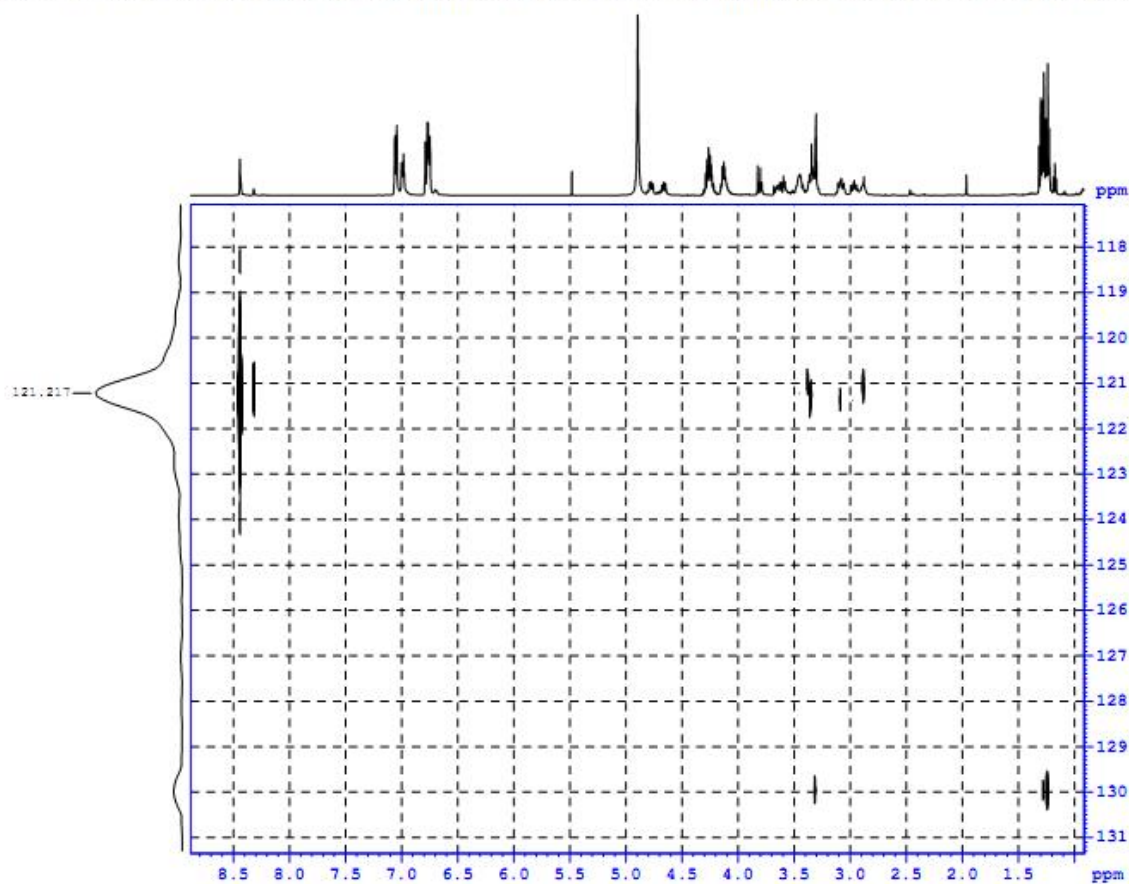


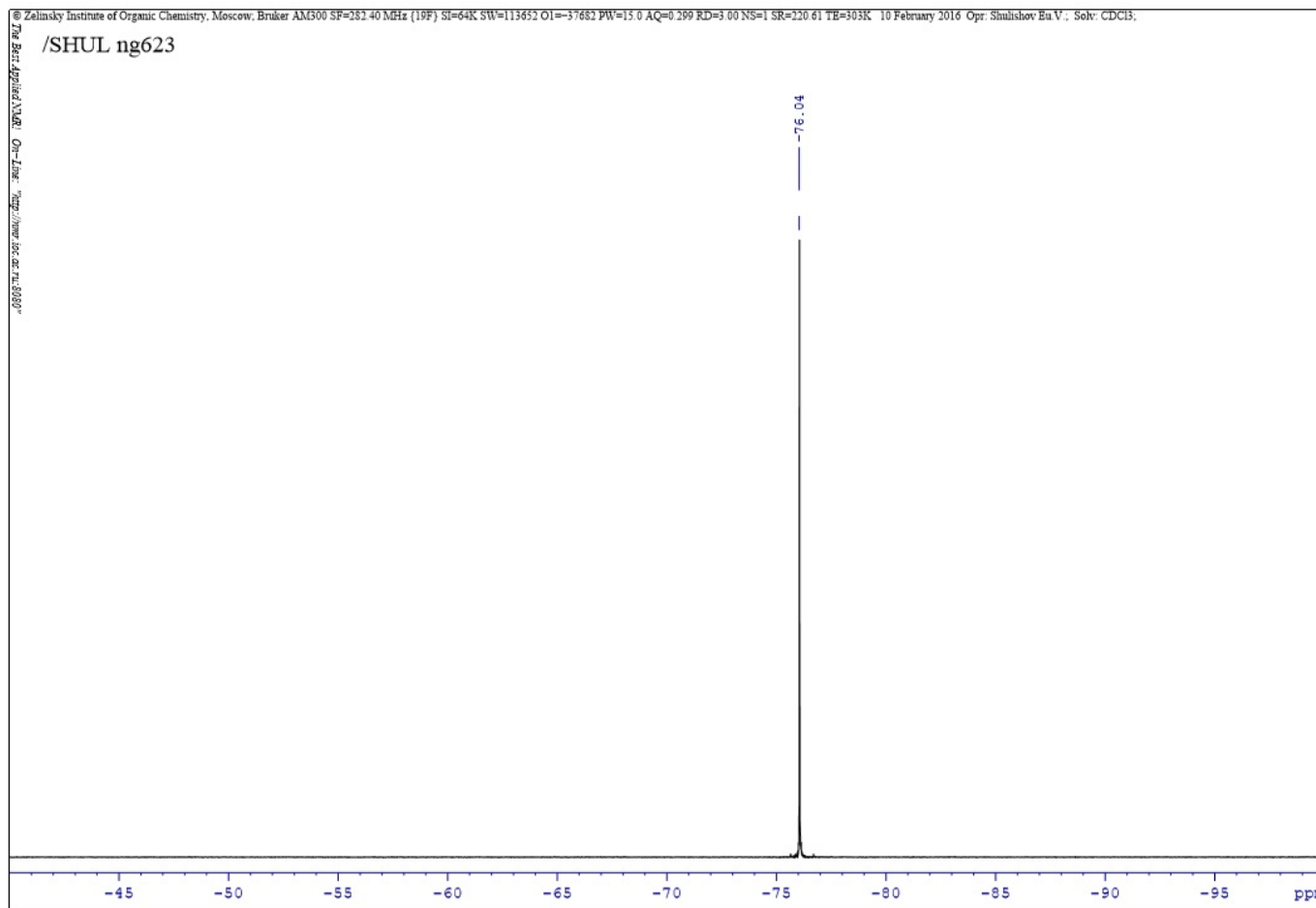
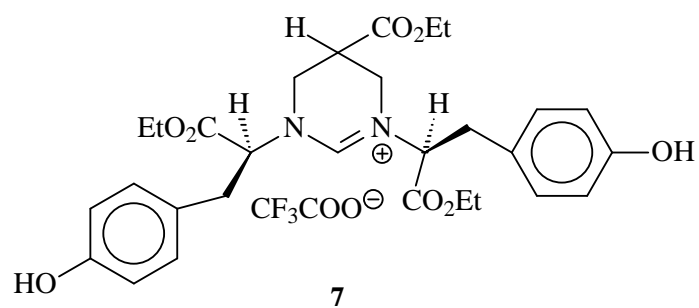


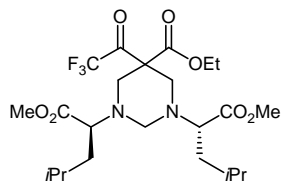
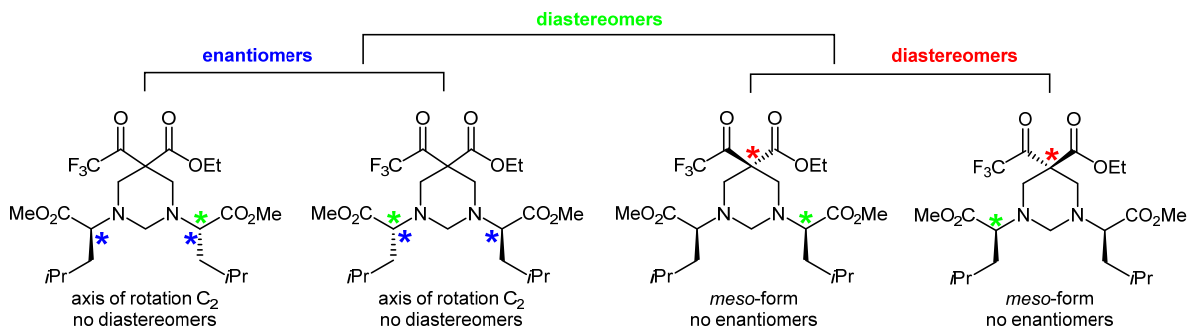
Sp-782 Gibadullina NG-32 (5)-third 40mg in MeOD, TMS 1H-15N HMBC AV500 04.02.2016 LAN

Ufa Institute of Chemistry of the Russian Academy of Sciences (IUC RAS), 2016

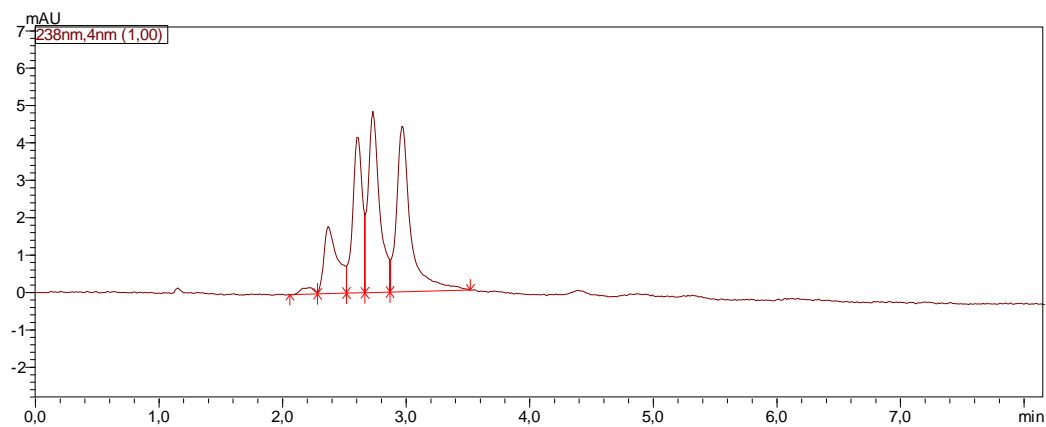
SW(1H)=9.00ppm; Q1(1H)=5.00ppm; SW(15N)=40.00ppm; Q1(15N)=120.00ppm; Obs.Freq.=500.126MHz; SP(15N)=50.68MHz; Q1=0.8s; T=294.0K; Probe:5MM; Exp.Time/24 min 55 sec; Time/Date: 15:16:11 04 Feb 2016.





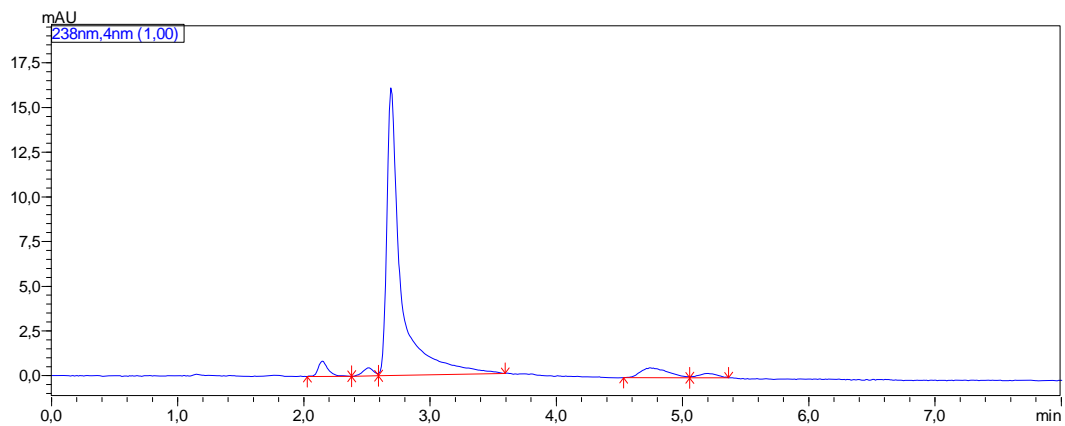
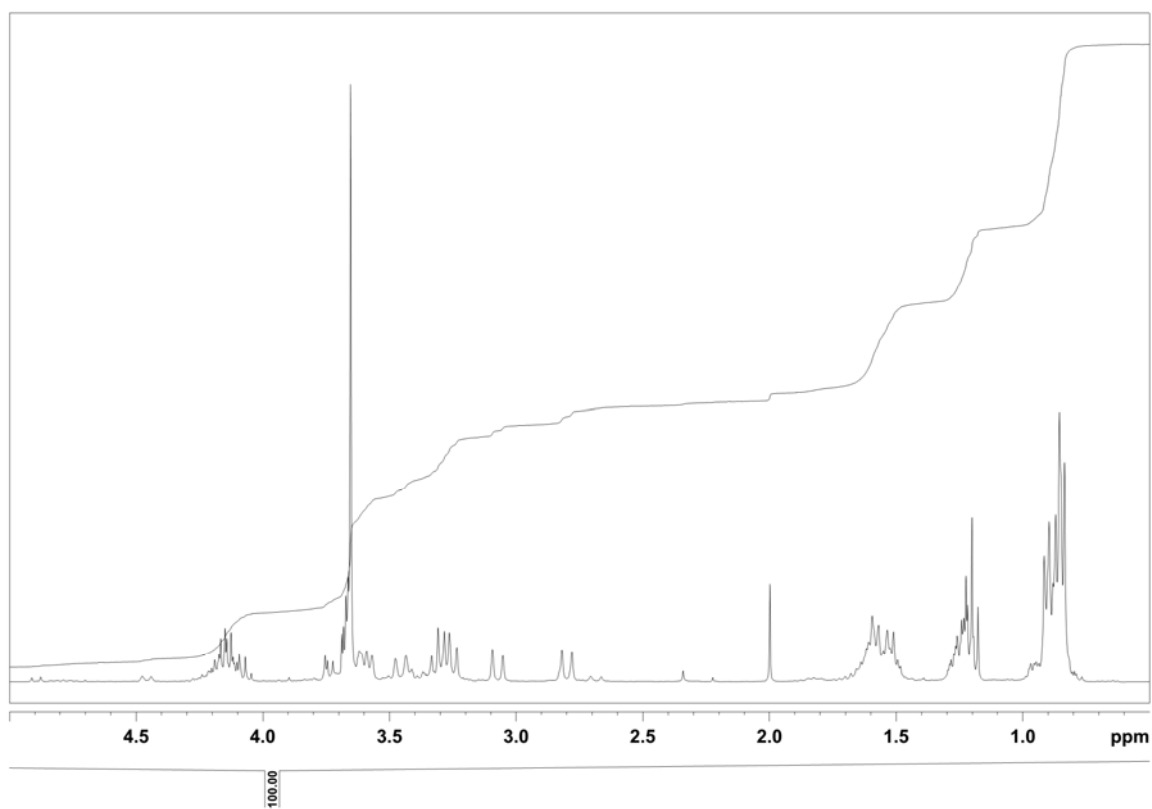
S-4d — 1 pure enantiomer**rac-4d** — 4 stereo-isomers**Chiral-HPLC** (99.5:0.5 Hex-iPrOH, 2 mL/min)**rac-4d**

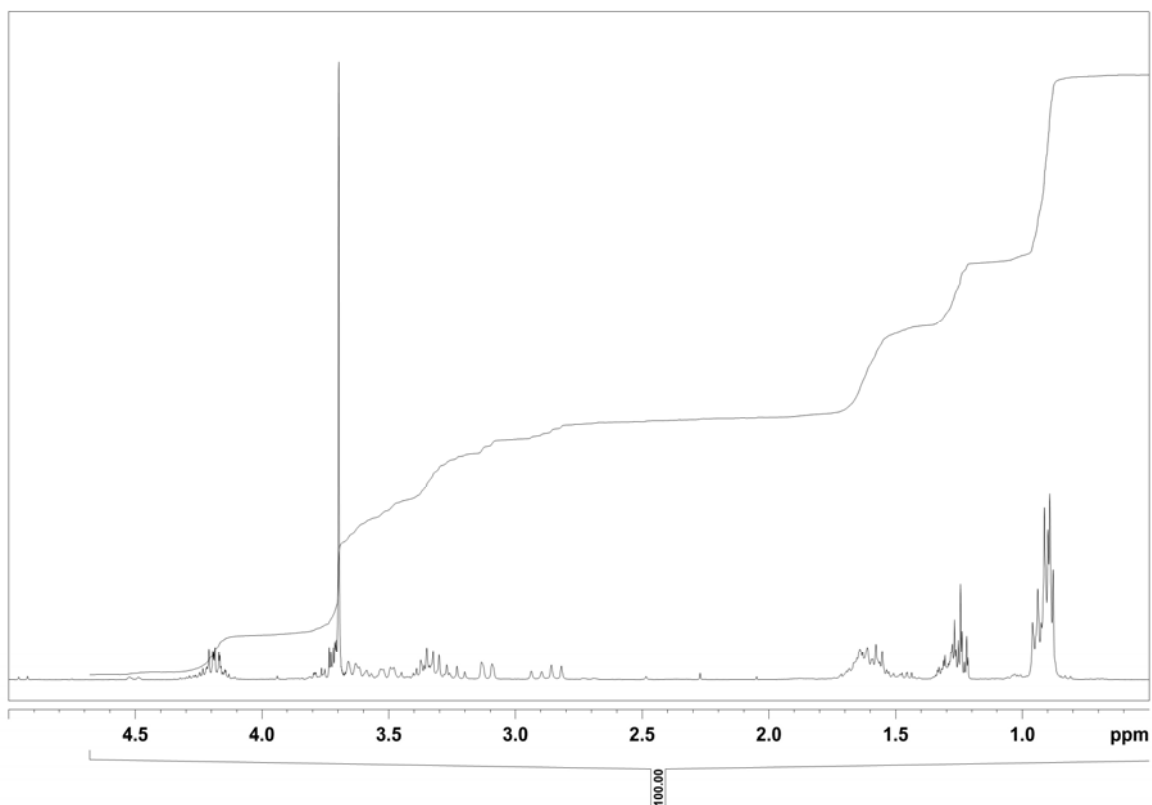
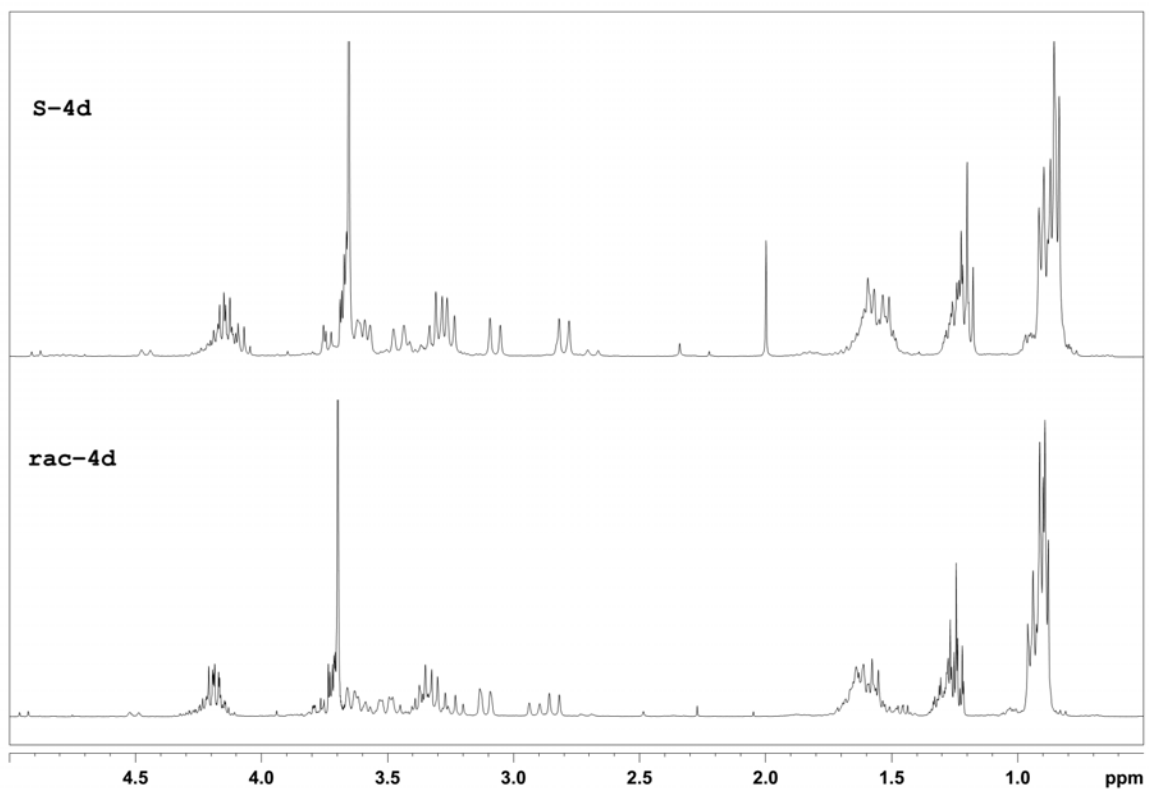
Datafile Name: NCr134_6.lcd
 Sample Name: NCr134
 Sample ID: NCr134



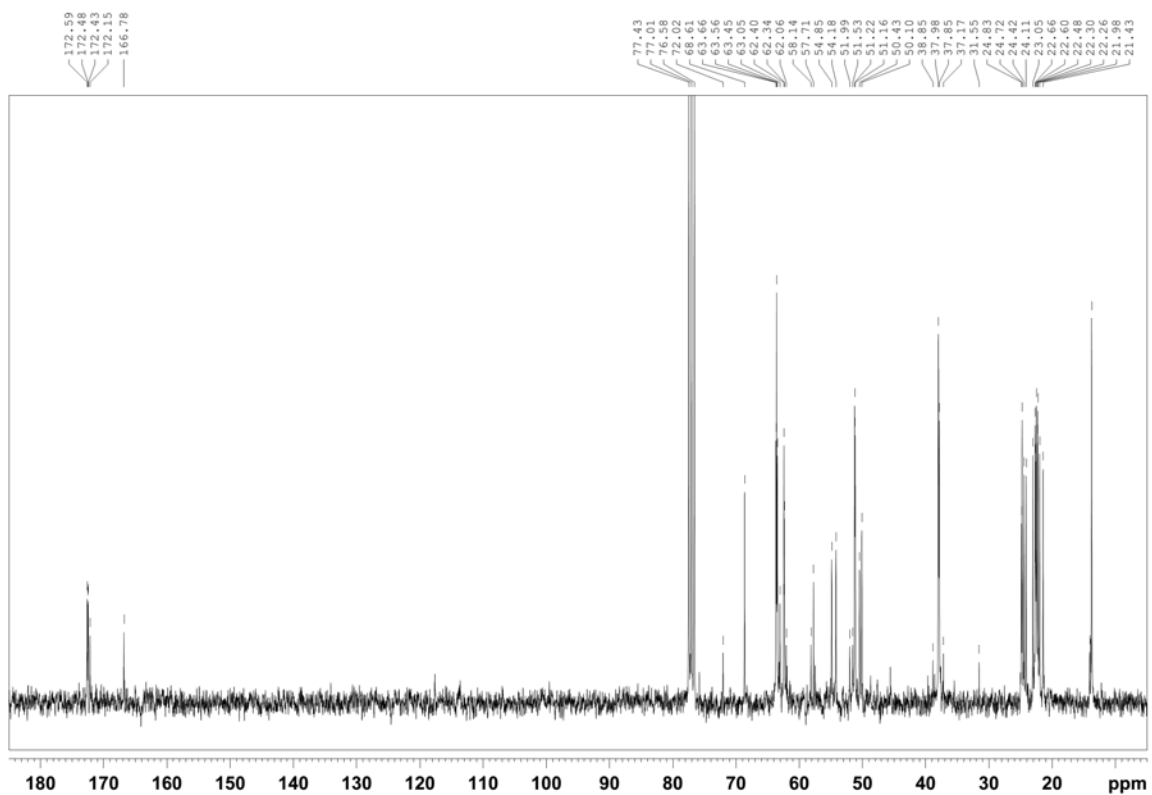
S-4d

Datafile Name: NCr135_3.lcd
Sample Name: NCr135
Sample ID: NCr135

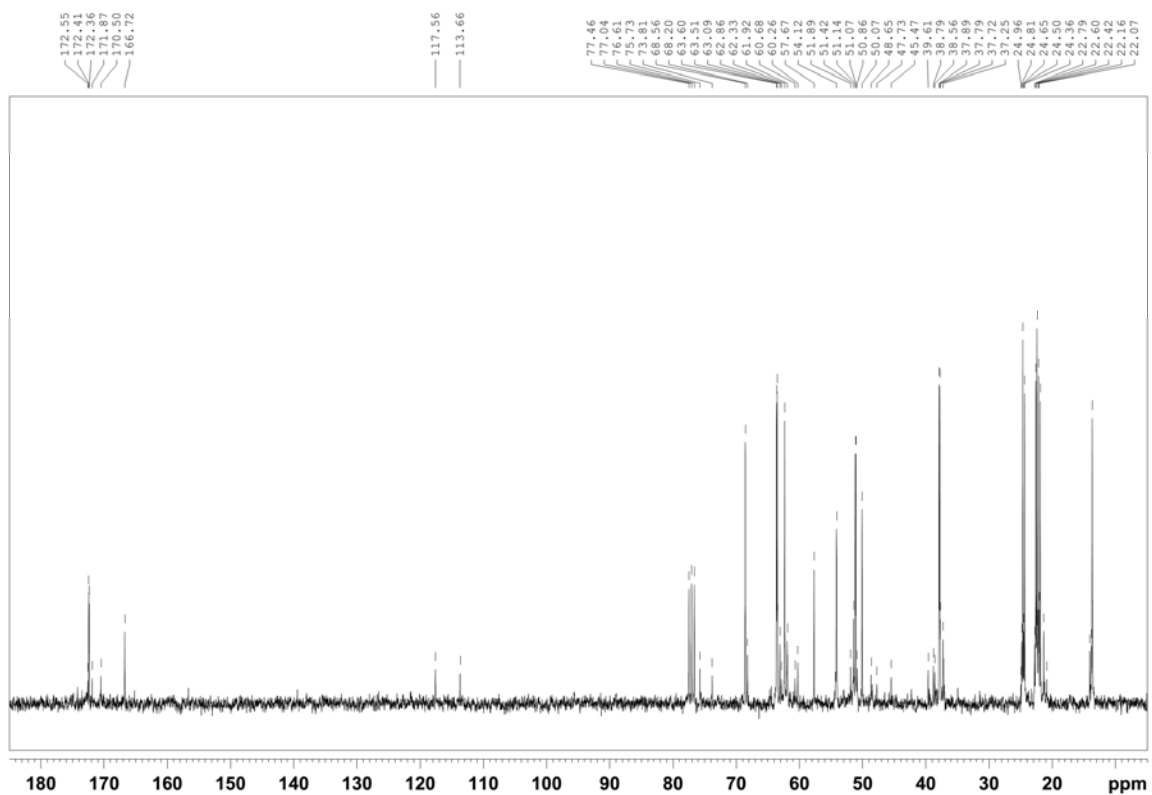
 ^1H NMR; S-4d

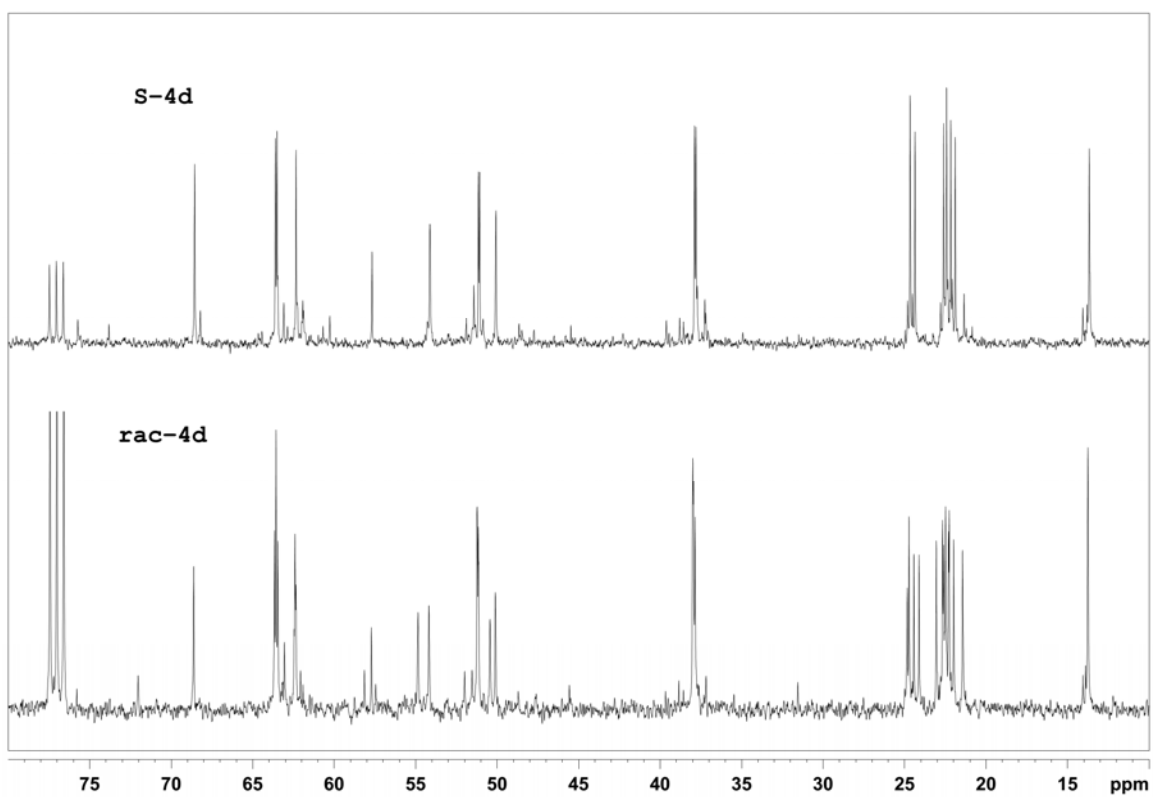
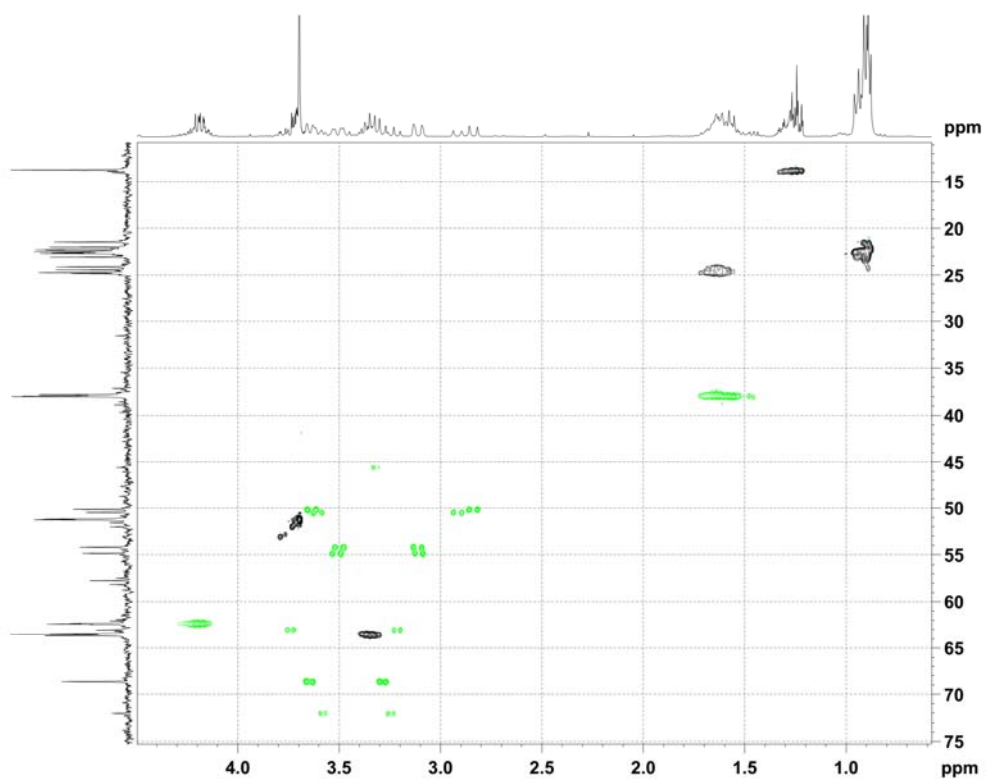
^1H NMR; **rac-4d** ^1H NMR; comparison of **S-4d** and **rac-4d**

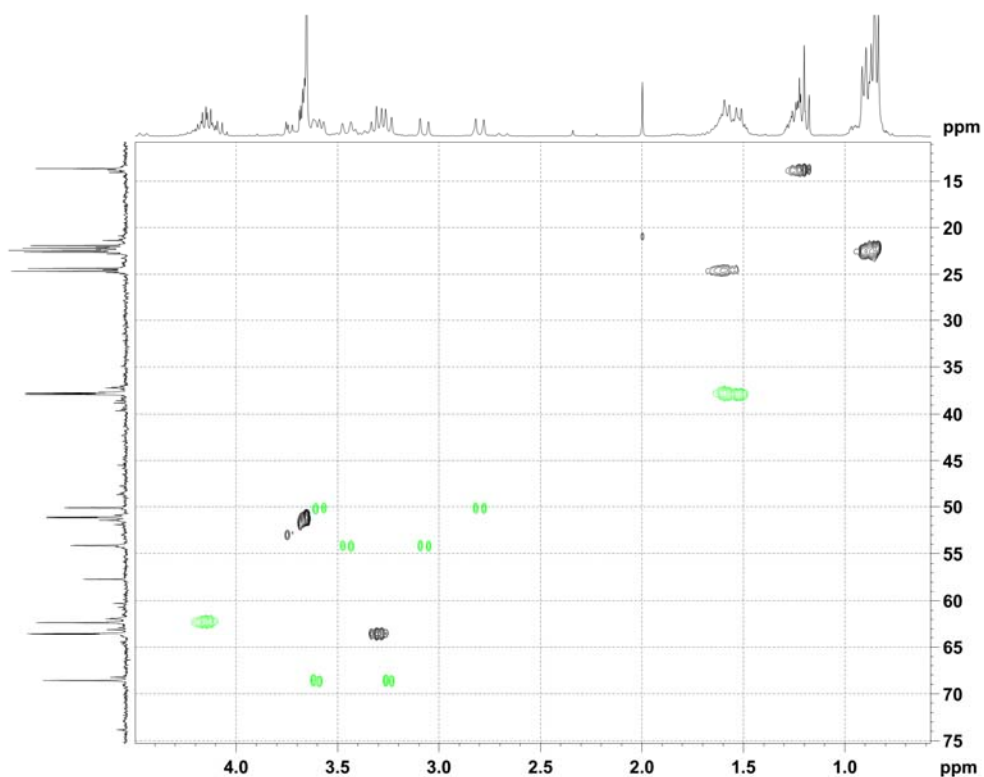
$^{13}\text{C}\{^1\text{H}\}$ NMR; rac-4d



$^{13}\text{C}\{^1\text{H}\}$ NMR; S-4d



$^{13}\text{C}\{^1\text{H}\}$ NMR; comparison of **S-4d** and **rac-4d**2D ^1H , ^{13}C edited-HSQC NMR; **rac-4d**

2D ^1H , ^{13}C edited-HSQC NMR; **S-4d**2D ^1H , ^{13}C edited-HSQC NMR; comparison of **S-4d** and **rac-4d**