

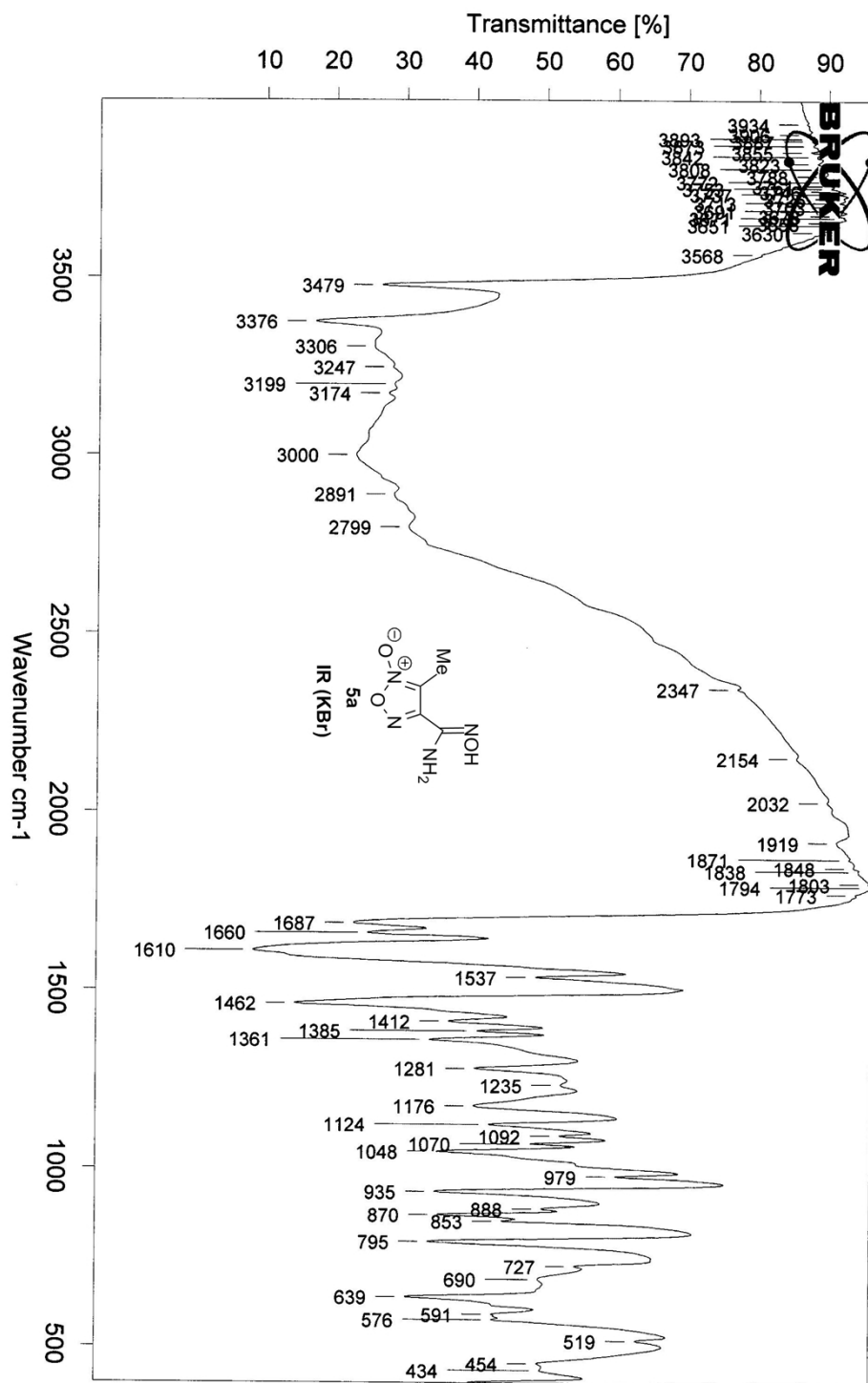
Supporting information

Efficient Assembly of Mono- and Bis(1,2,4-oxadiazol-3-yl)furoxan Scaffolds via Tandem Reactions of Furoxanylamidoximes

Leonid L. Fershtat,^a Ivan V. Ananyev,^b Nina N. Makhova^{a*}

^a N.D. Zelinsky Institute of Organic Chemistry, Leninsky prosp. 47, 119991 Moscow, Russian Federation. Fax: +7 499 135 53 28. E-mail: nnn@ioc.ac.ru

^b A.N. Nesmeyanov Institute of Organoelement Compounds, Vavilova str. 28, 119991 Moscow, Russian Federation.

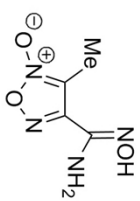


D:\EDL\LEO-638.0 ФЕРИТАТ. LEO-638, прессовка с KBr, 1/200.

03.12.2014

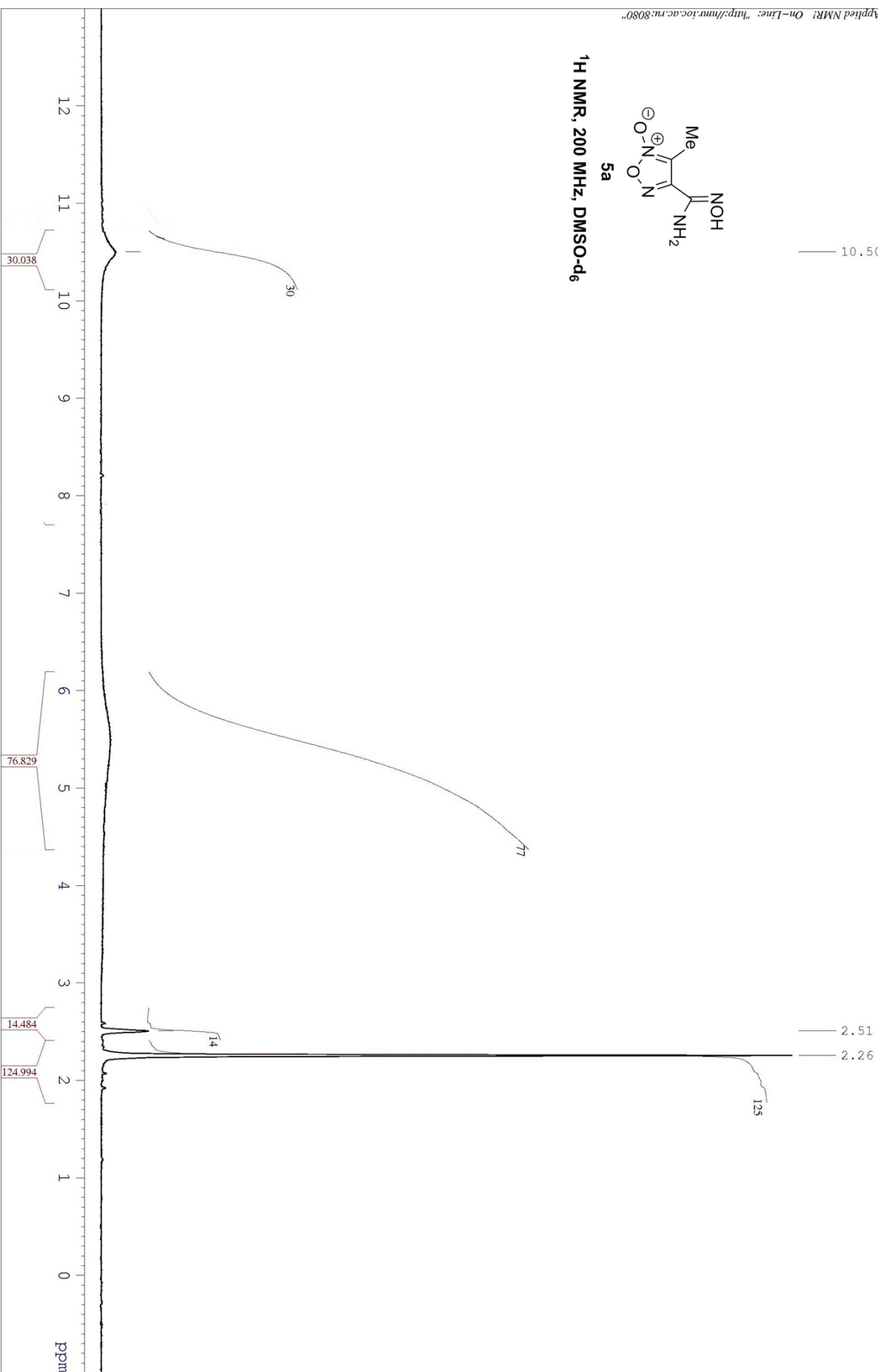
/USED 638

10.50

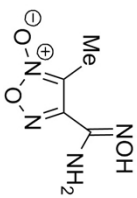


5a

¹H NMR, 200 MHz, DMSO-d₆

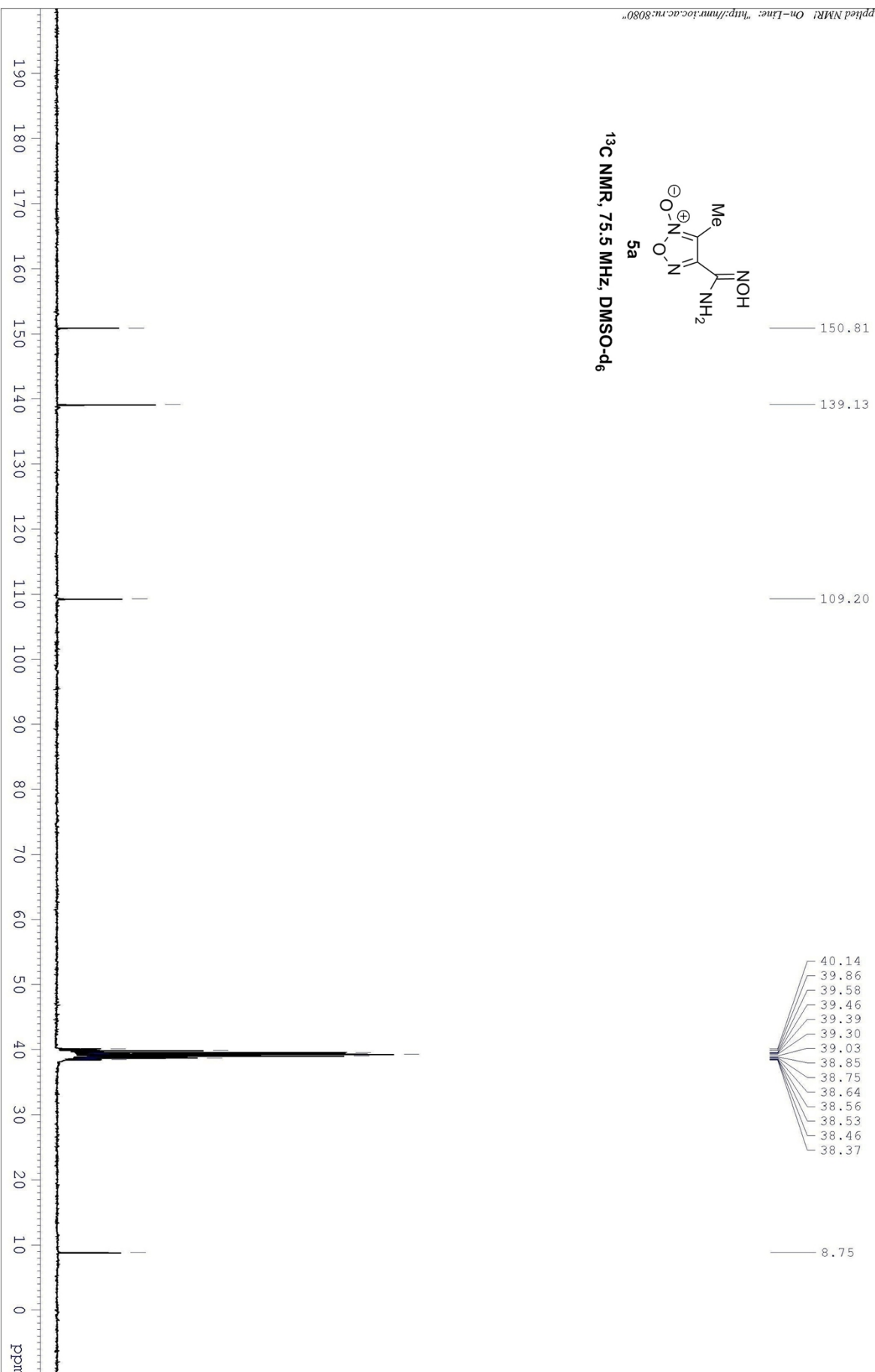


/USED 638



5a

¹³C NMR, 75.5 MHz, DMSO-d₆

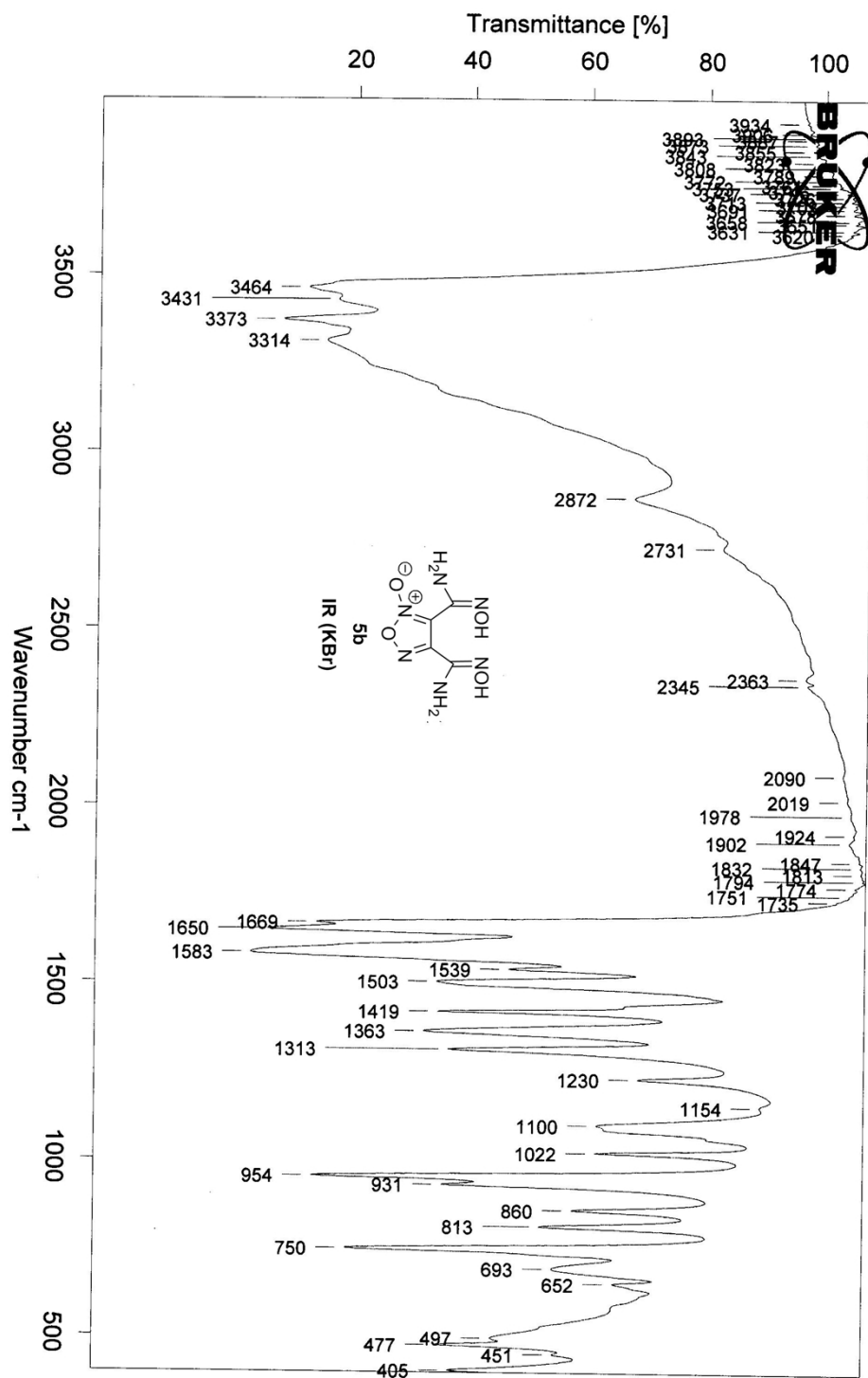


D:\EDL\LEO-660.0

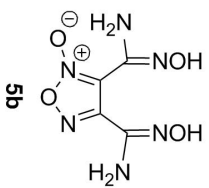
ФЕРУЛАТ. LEO-660, прессовка с KBr, 1/200.

04.12.2014

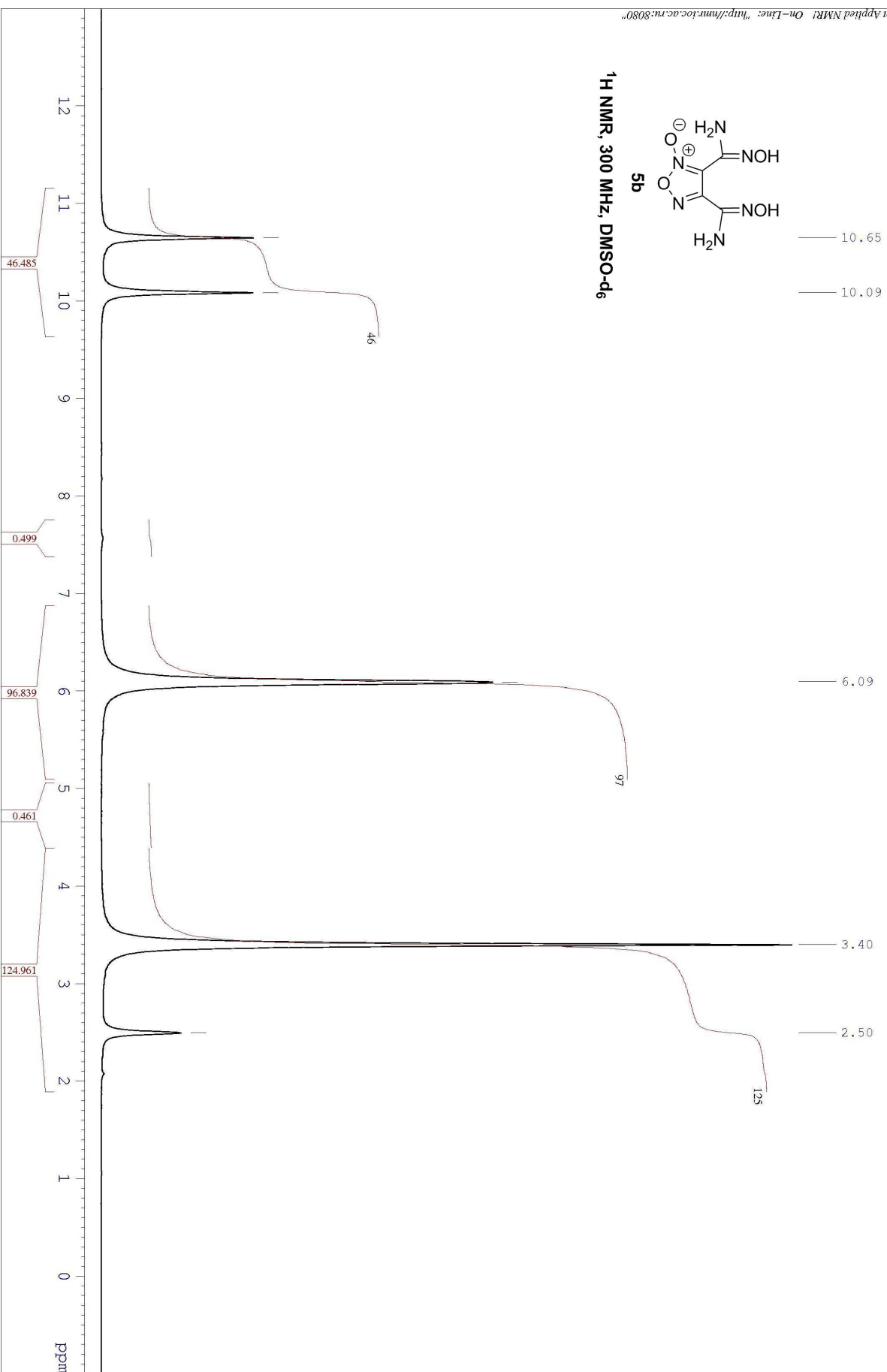
Page 1/1



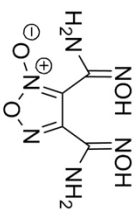
/USED 660



¹H NMR, 300 MHz, DMSO-d₆

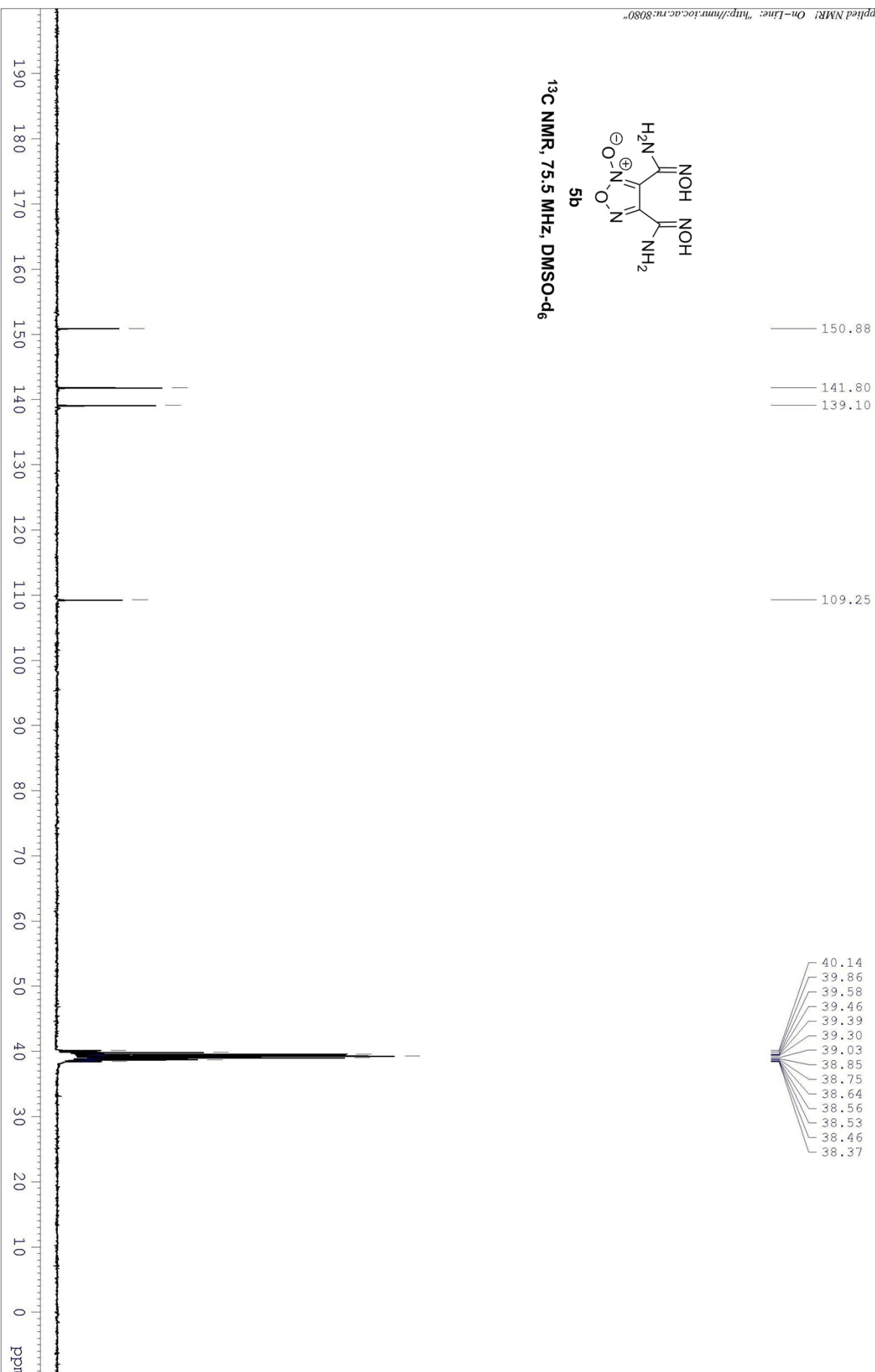


/USED 660

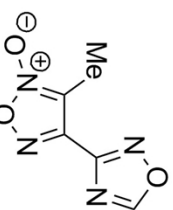


5b

¹³C NMR, 75.5 MHz, DMSO-d₆

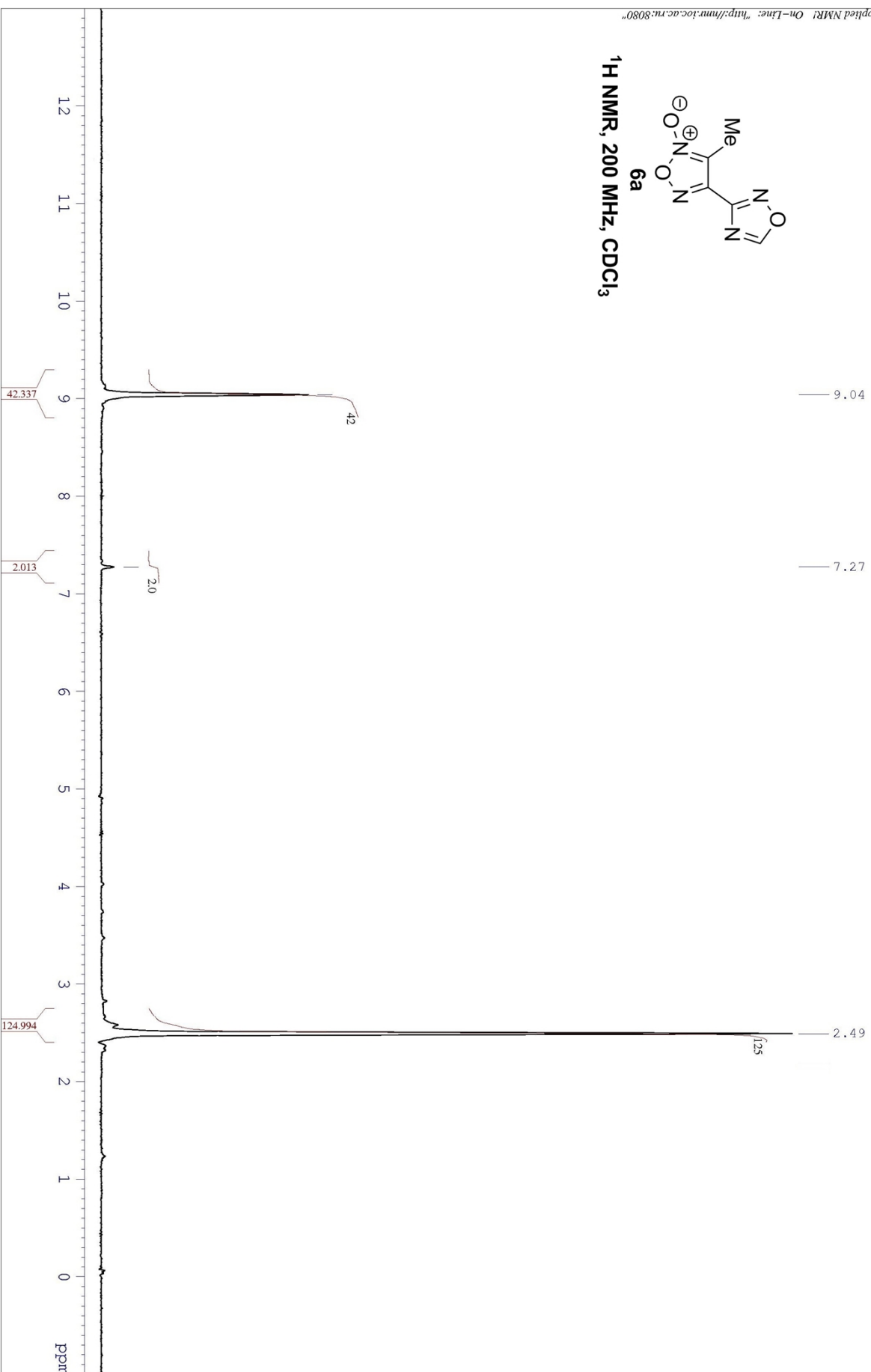


/USED 641

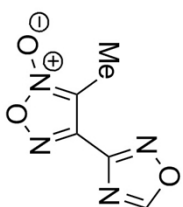


6a

¹H NMR, 200 MHz, CDCl₃

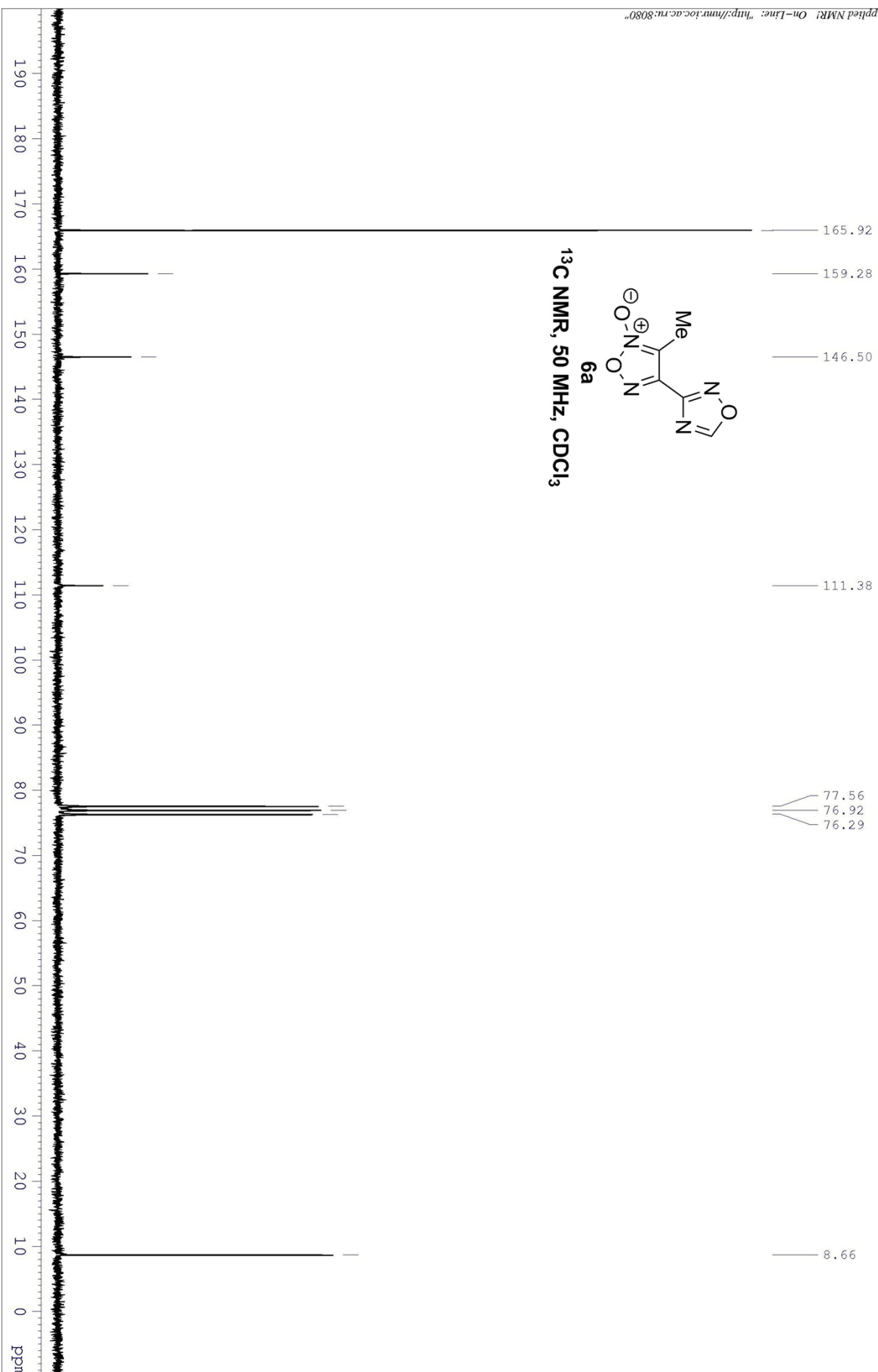


/USED 641.C13



6a

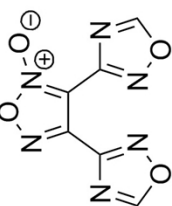
¹³C NMR, 50 MHz, CDCl₃



/USED 672

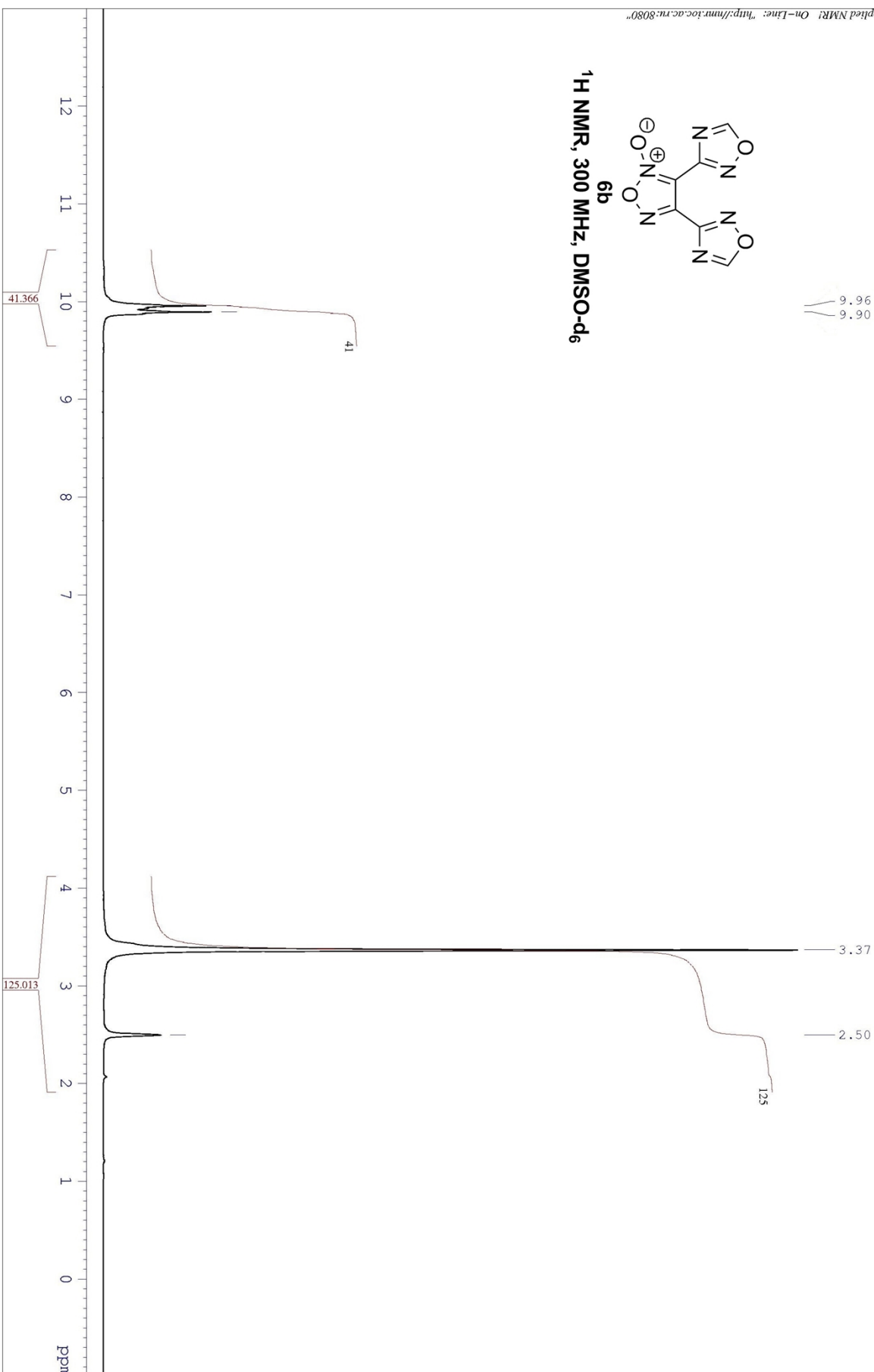
© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=300.13 MHz [1H] SF=16K SW=9009.01=2401 PW=9.0 AQ=0.901 RD=3.00 NS=1 SR=-1.12 TE=296K 30 September 2014 Opr: Struchkova M.I.; Solv: DMSO-d6;

9.96
9.90

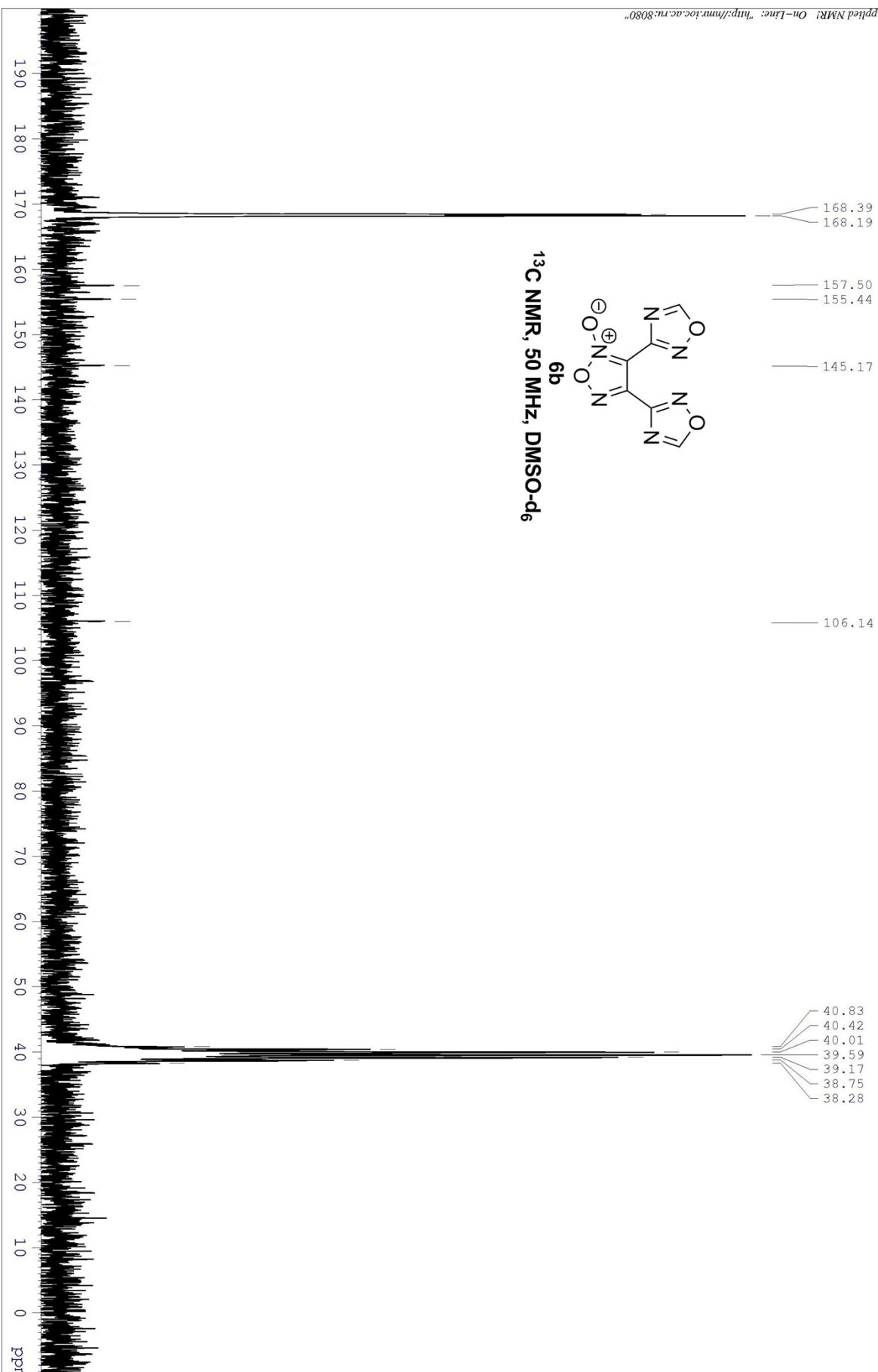


6b

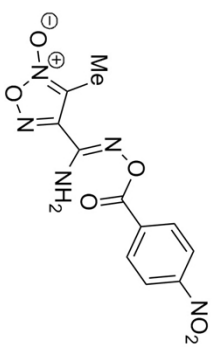
¹H NMR, 300 MHz, DMSO-d₆



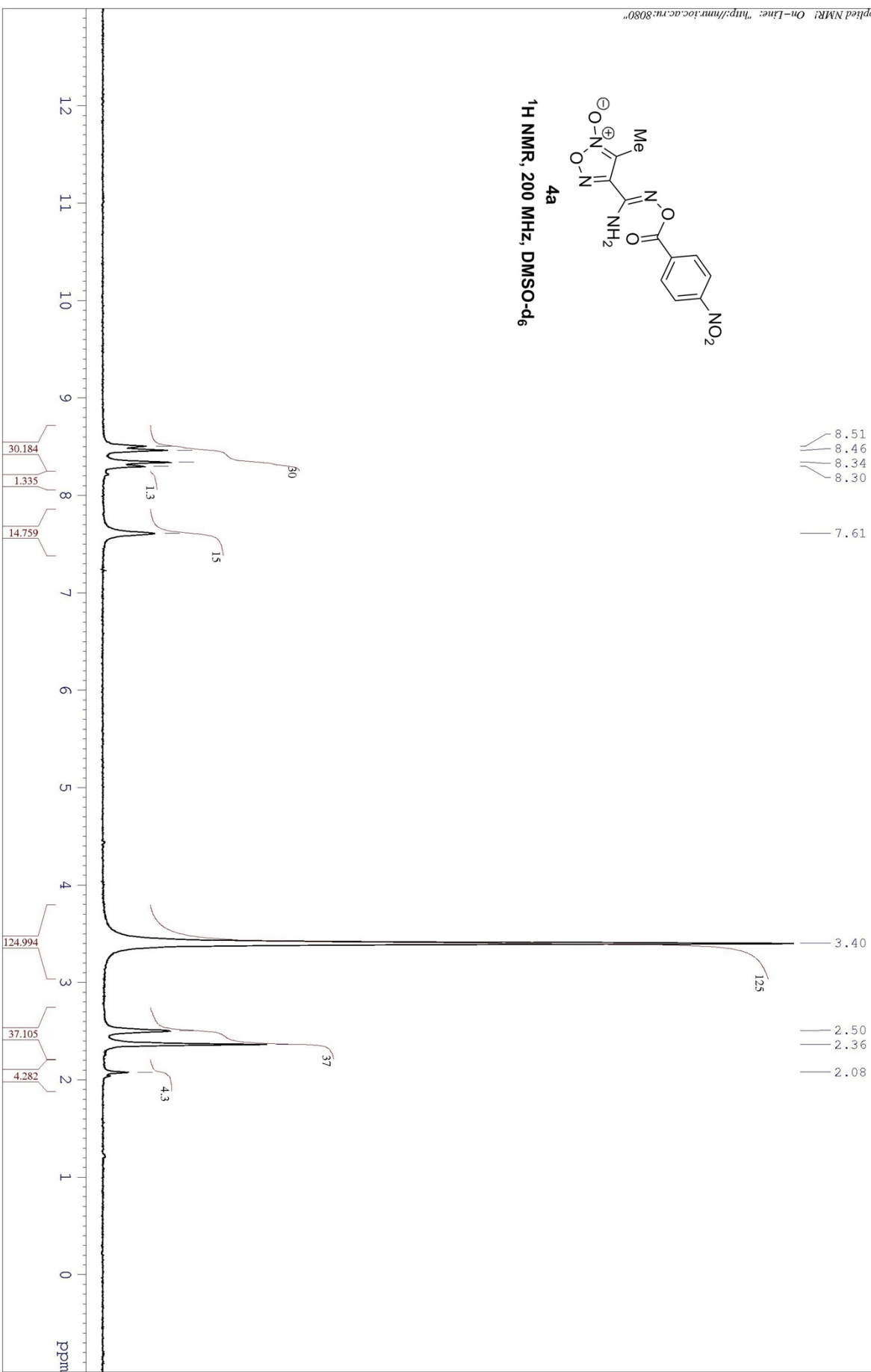
/USED 672.C13



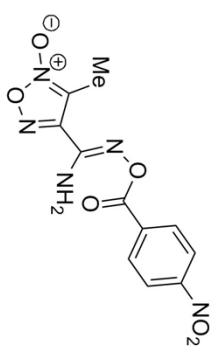
/USED 669



4a
¹H NMR, 200 MHz, DMSO-d₆

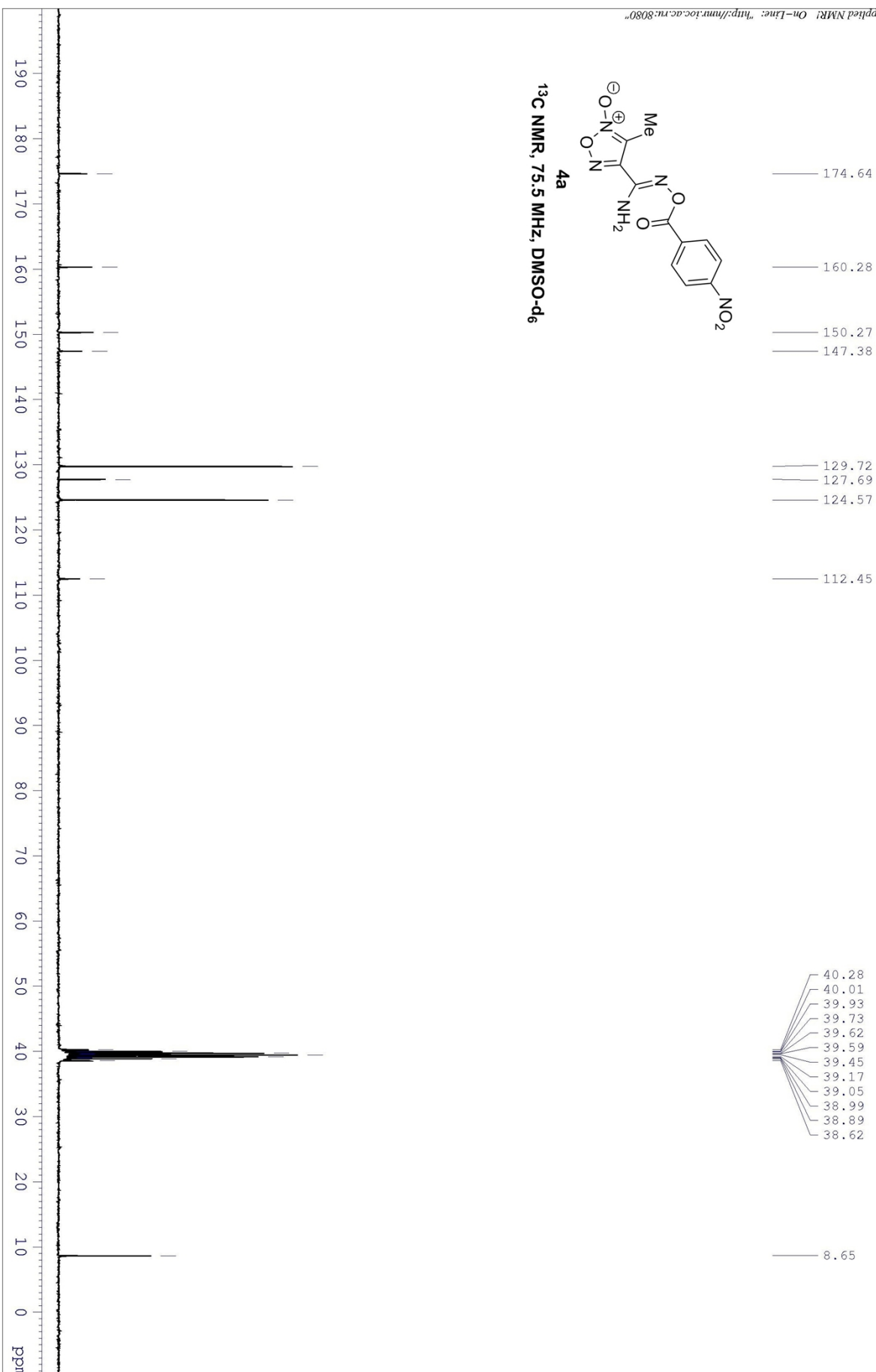


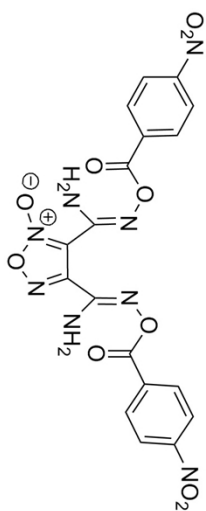
/USED 669



4a

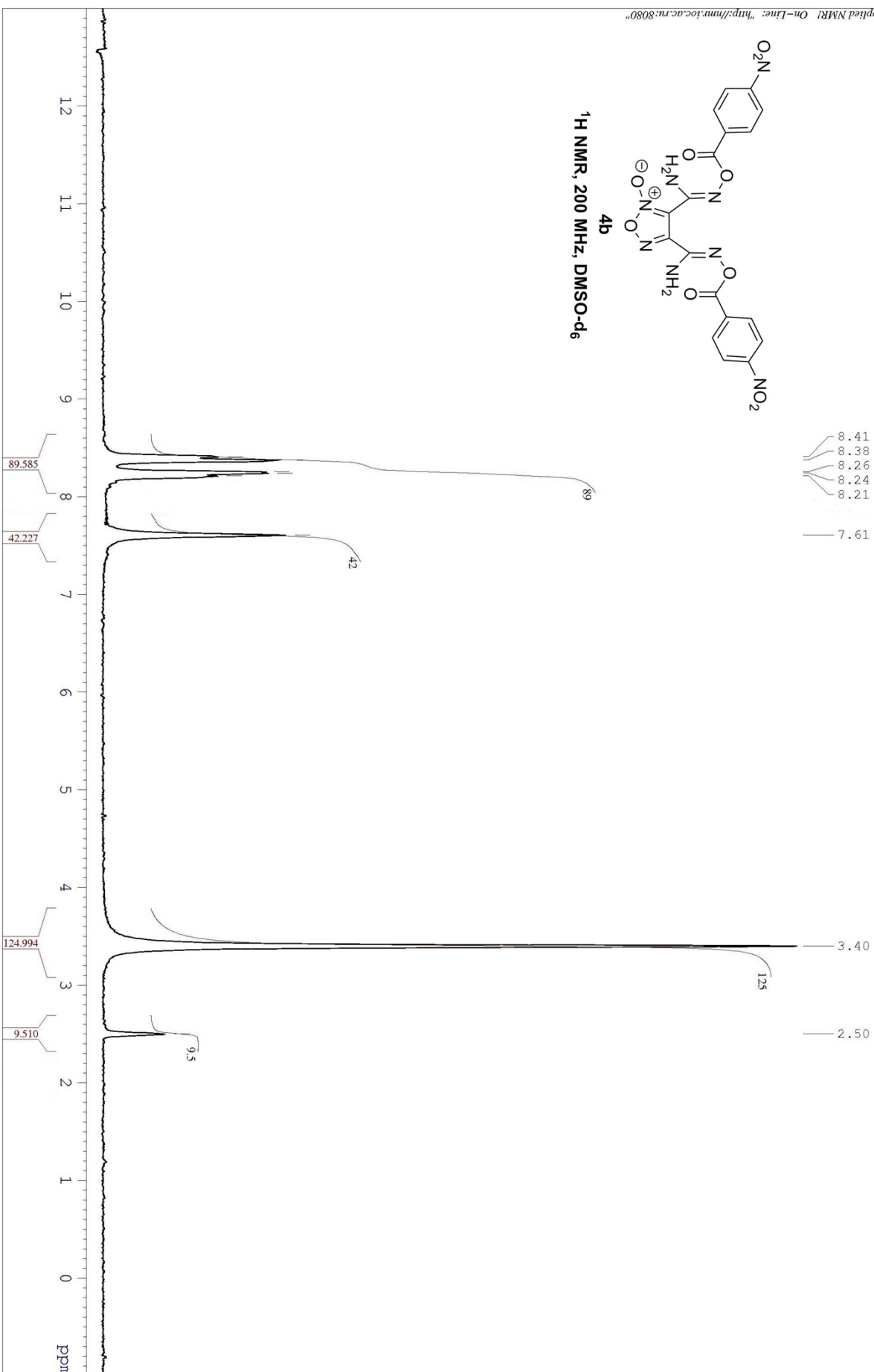
¹³C NMR, 75.5 MHz, DMSO-d₆



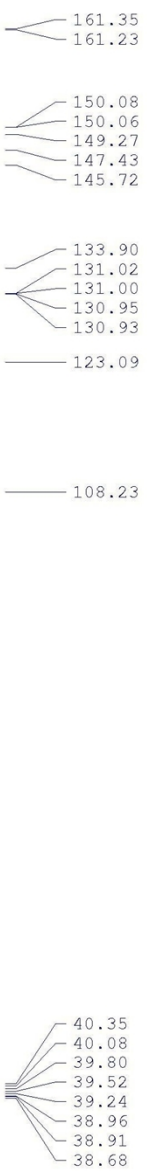


4b

¹H NMR, 200 MHz, DMSO-d₆

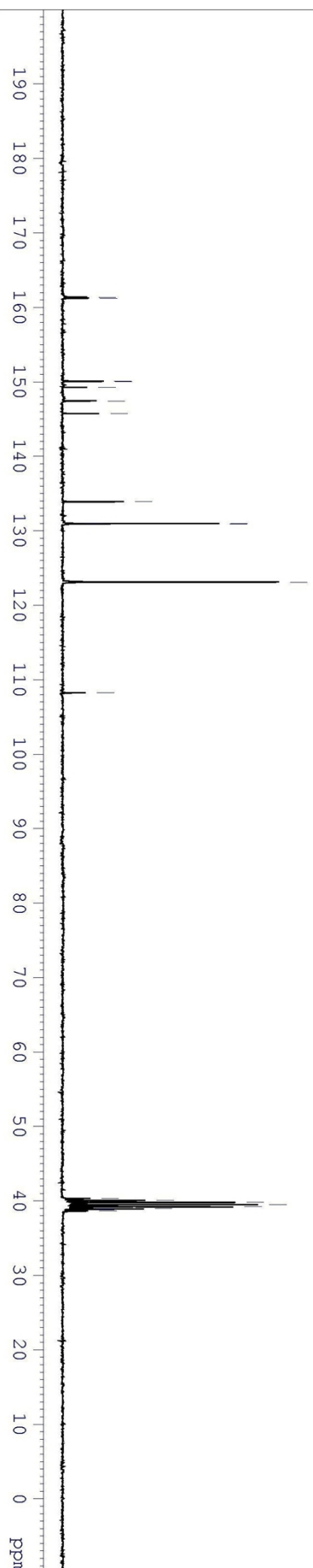


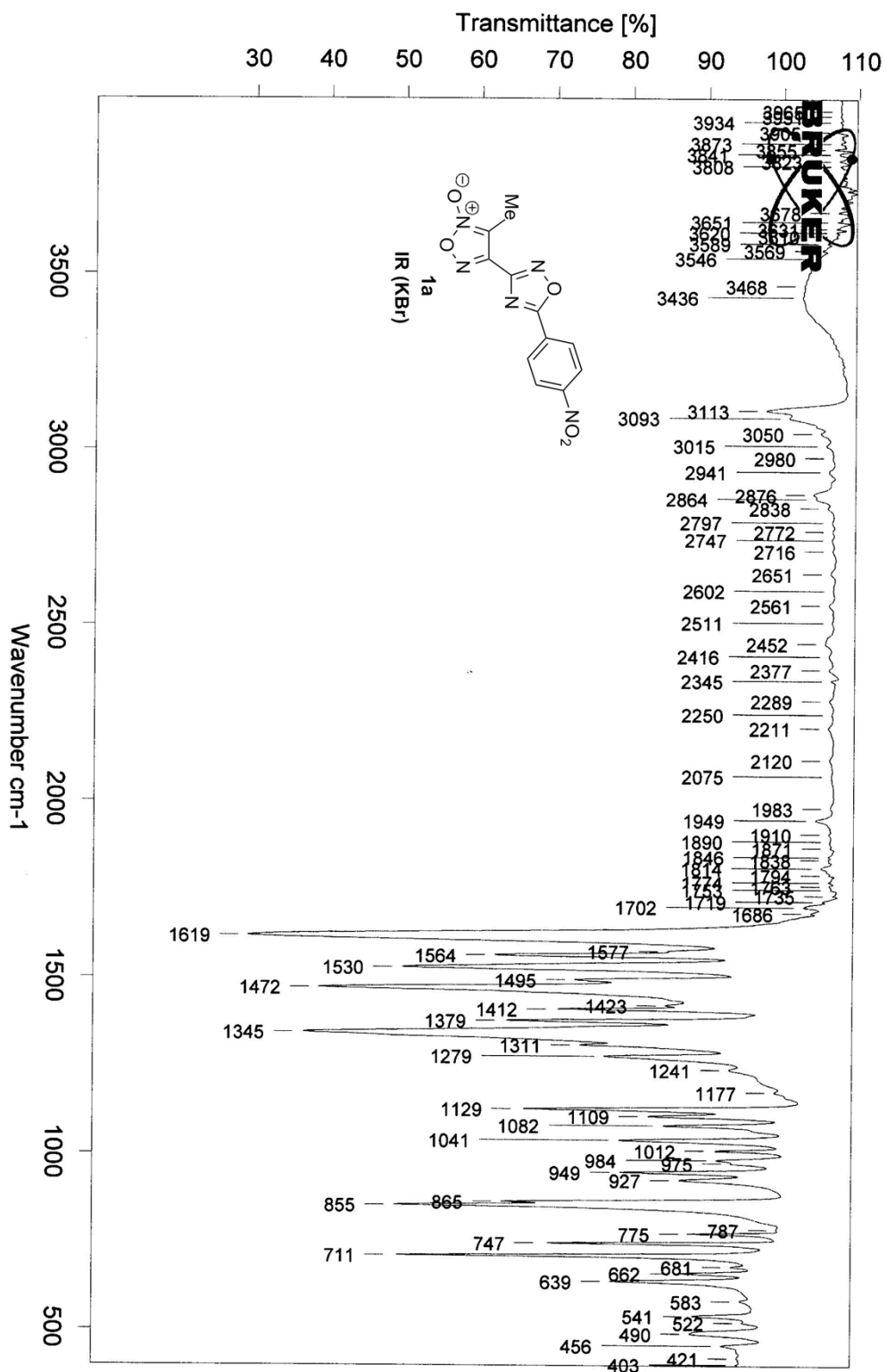
/USED 683



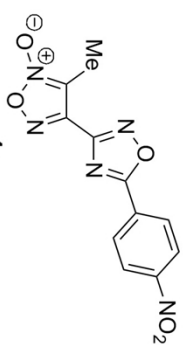
4b

¹³C NMR, 75.5 MHz, DMSO-d₆

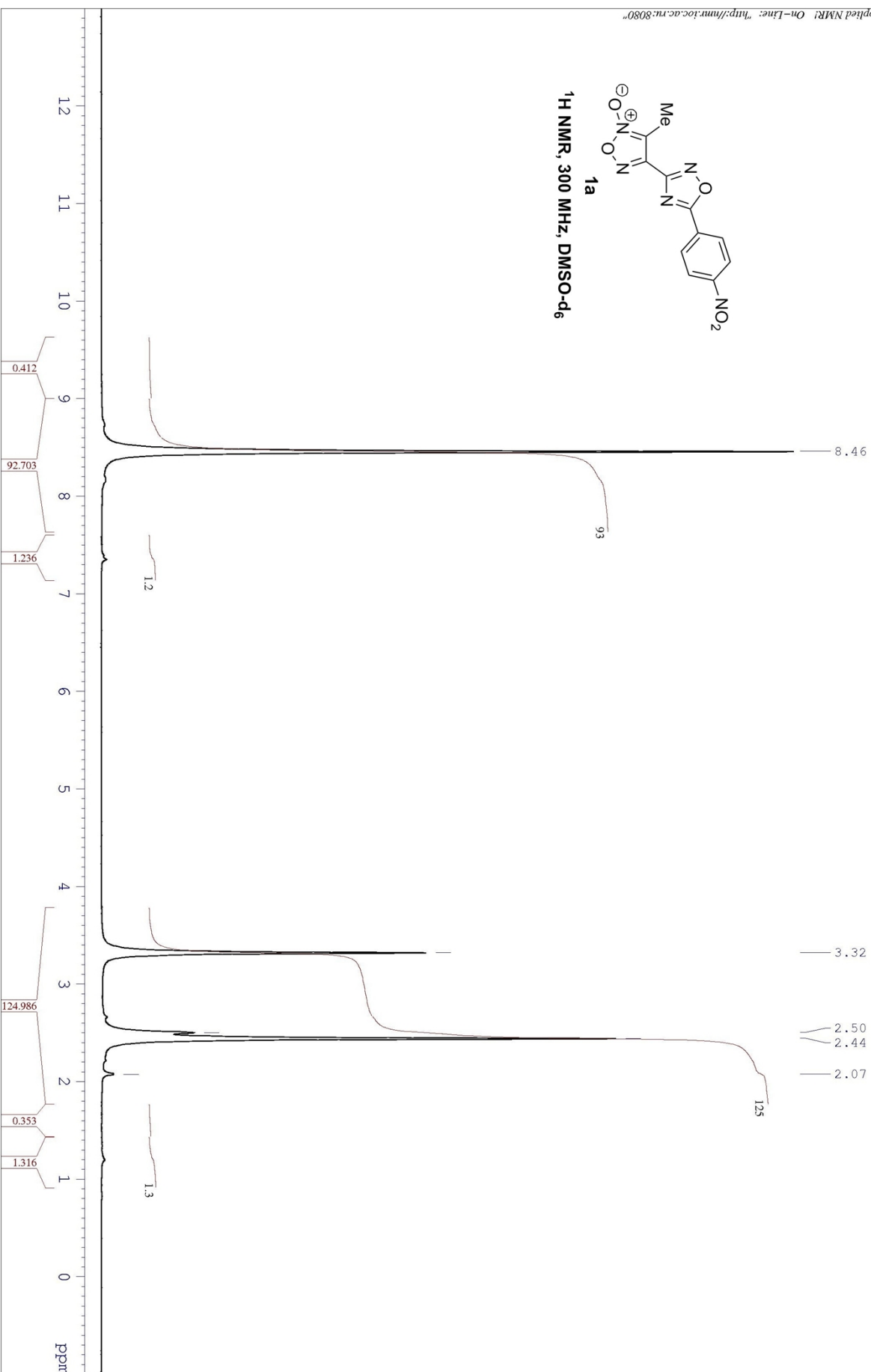




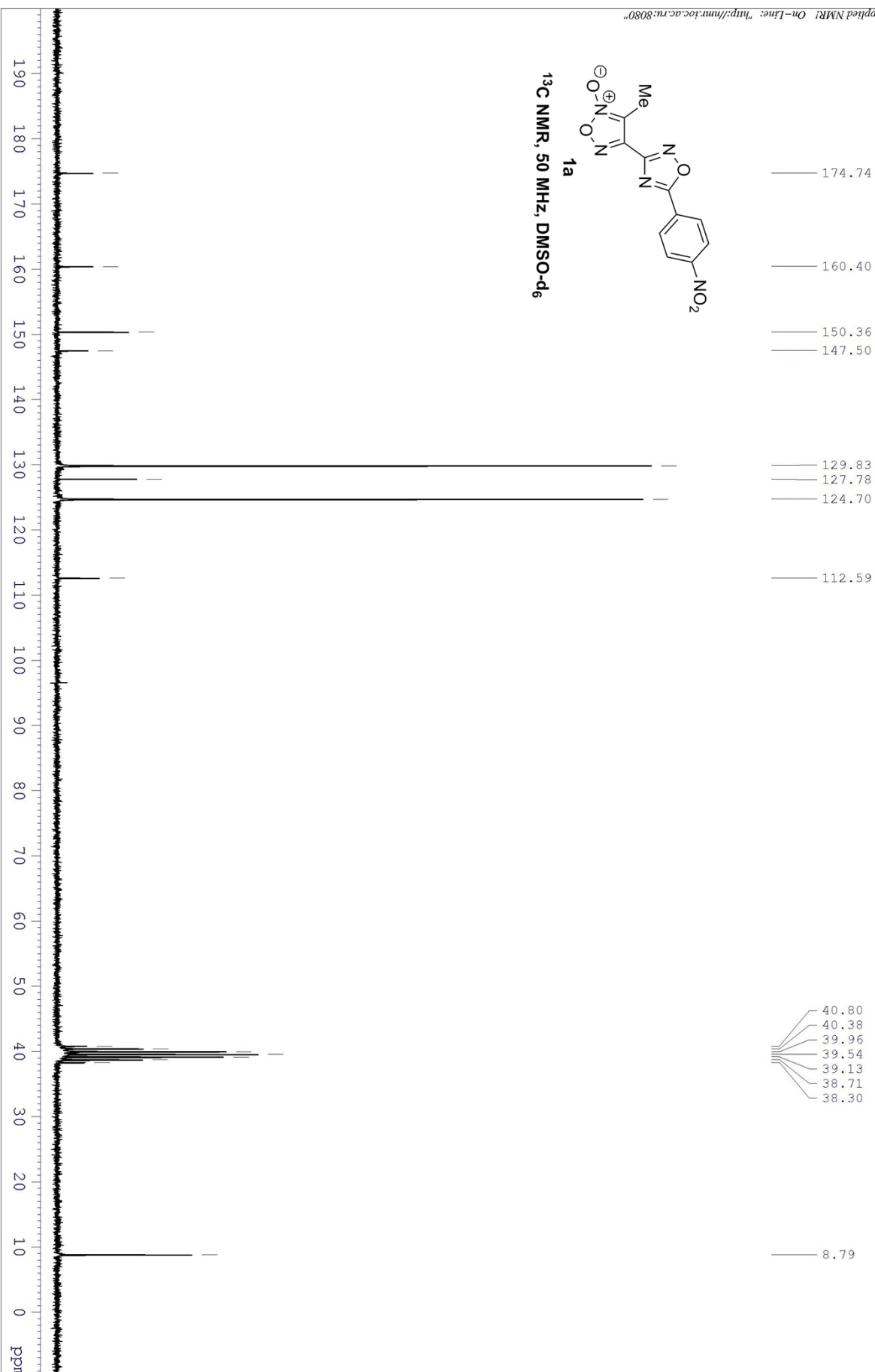
/USED 751



¹H NMR, 300 MHz, DMSO-d₆

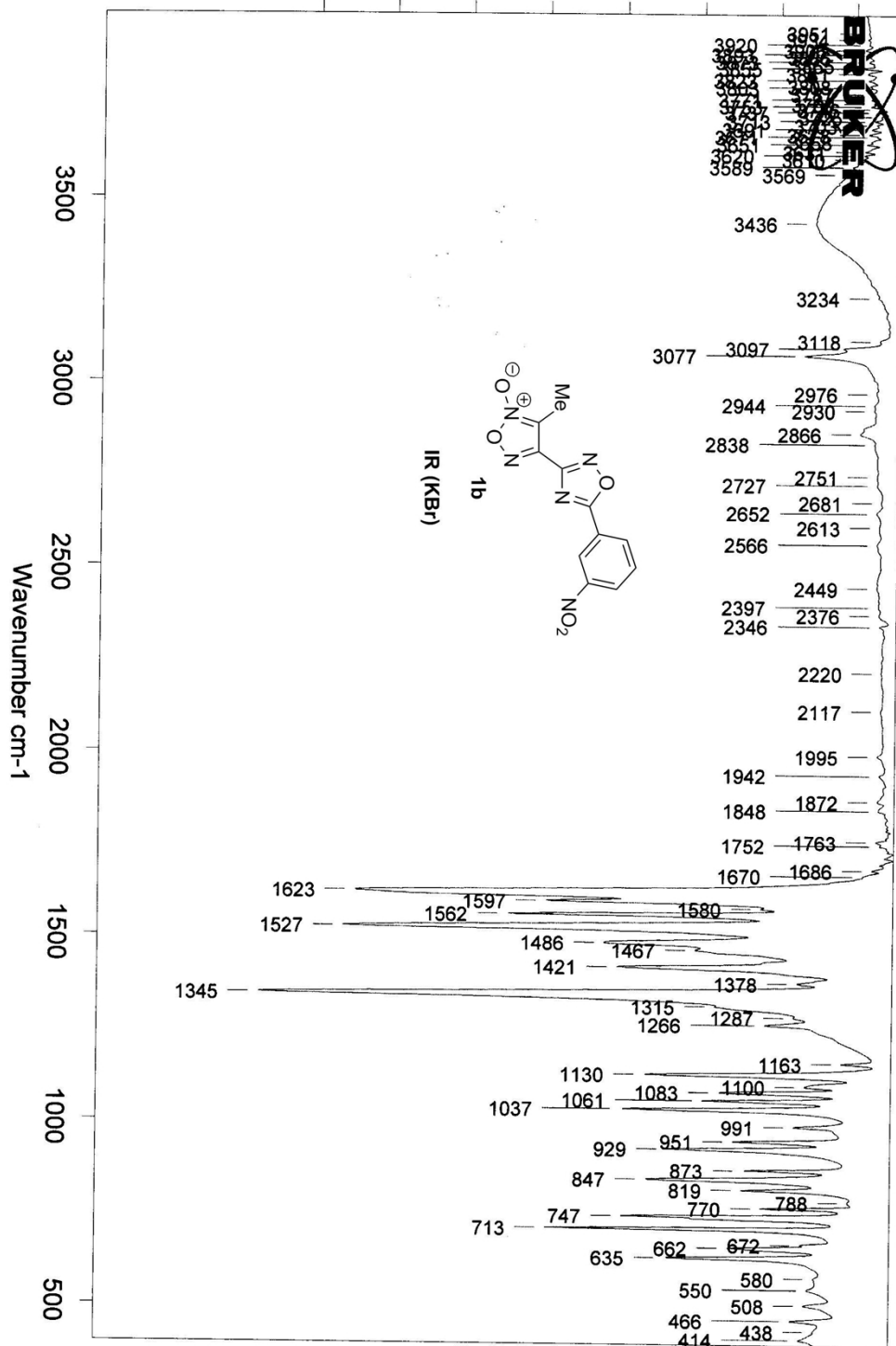


/USED 751



Transmittance [%]

40 50 60 70 80 90 100 110



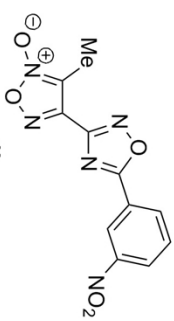
D:\EDL\LEO-726.0

ФЕПУТАТ. LEO-726, пробова с KBr, 0.5/200.

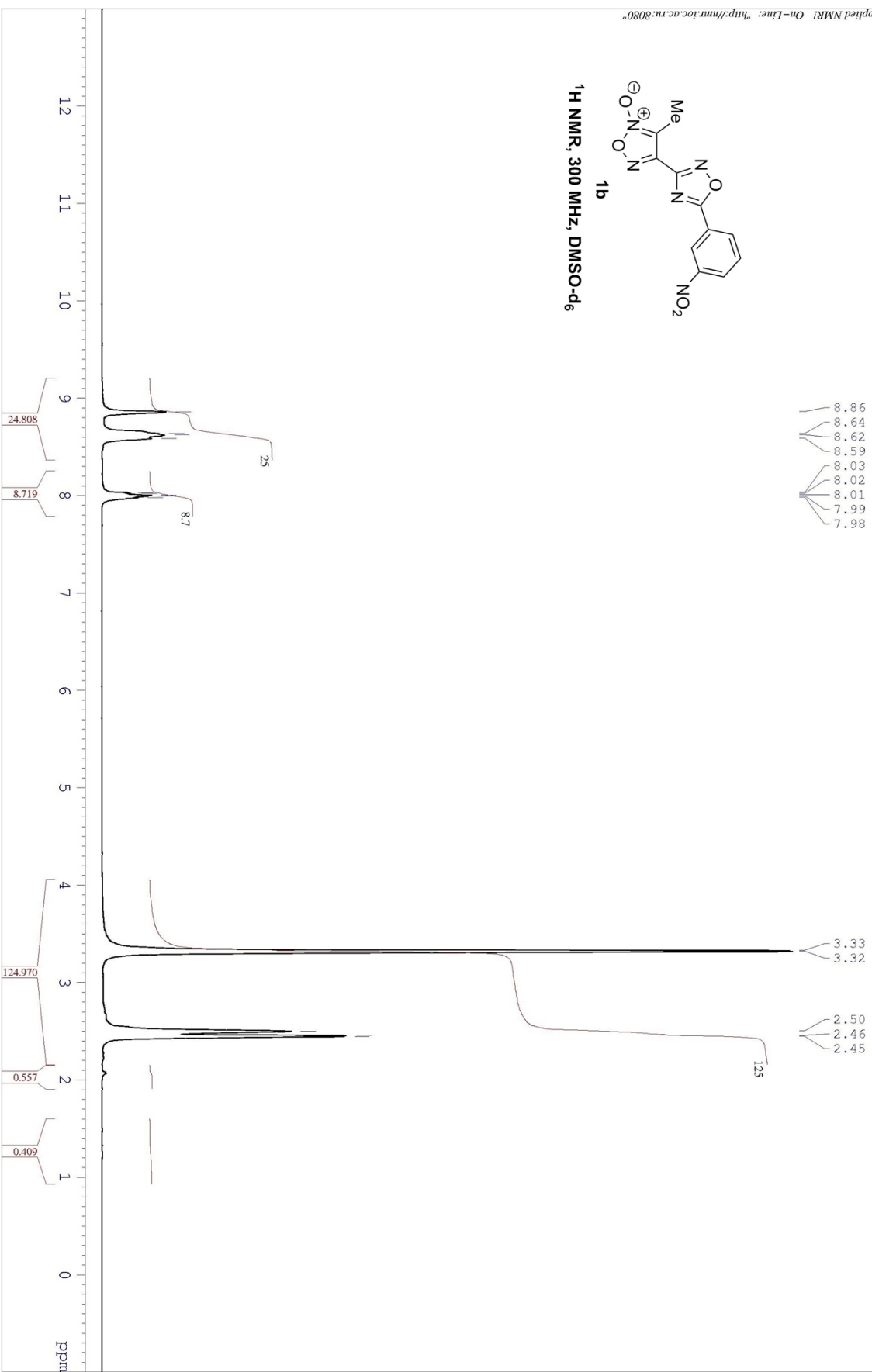
04.12.2014

/USED 726

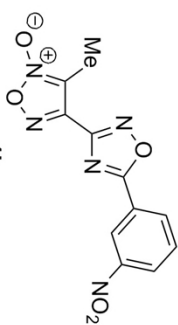
© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=300.13 MHz (1H) SI=16K SW=9009.01=2401 PW=9.0 AQ=0.901 RD=3.00 NS=1 SR=-4.12 TF=300K 21 November 2014 Opn: Stuchkova M.I.; Solv: DMSO-d6;



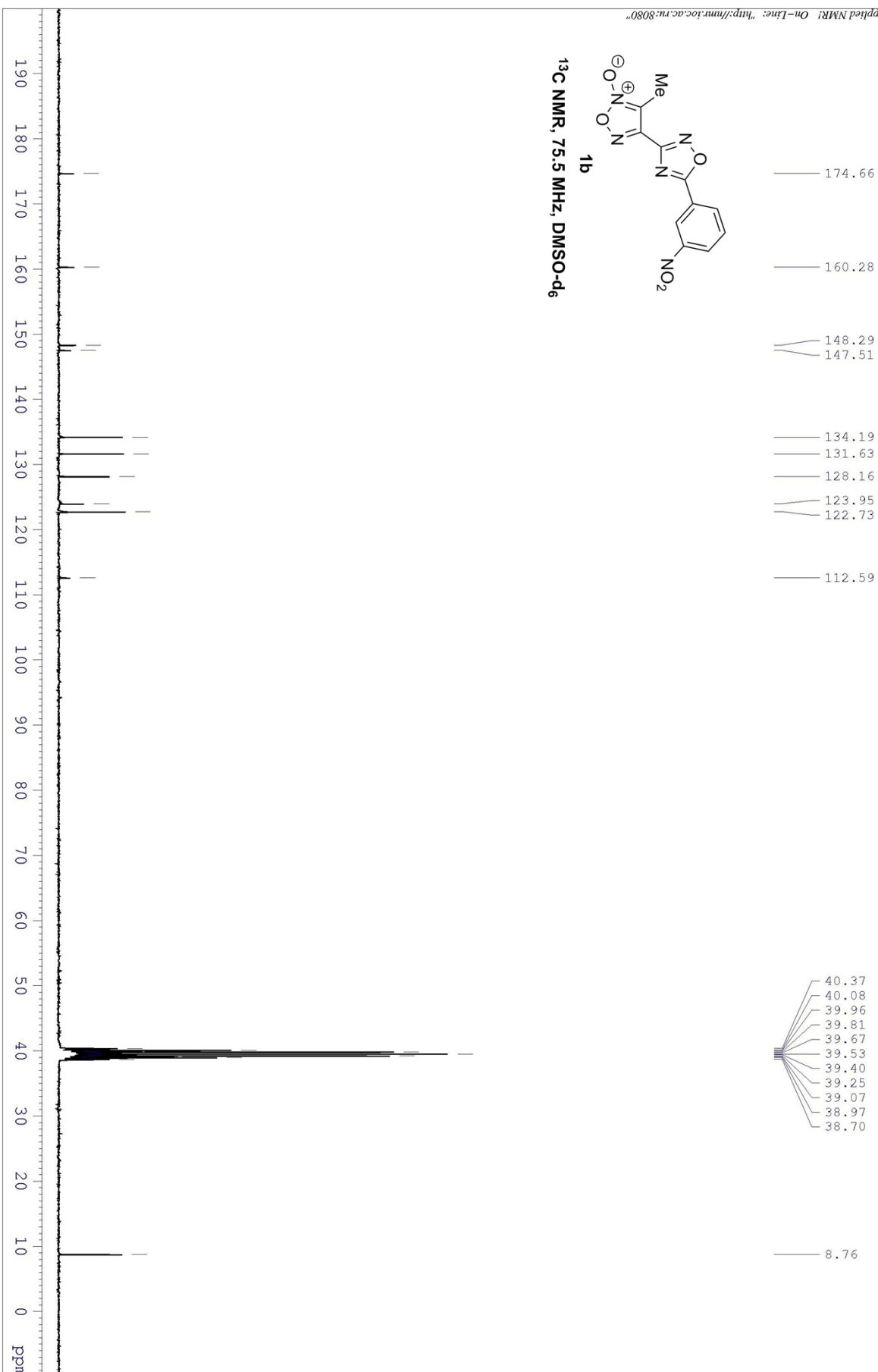
¹H NMR, 300 MHz, DMSO-d₆



/USED 726



¹³C NMR, 75.5 MHz, DMSO-d₆



Display Report

Analysis Info

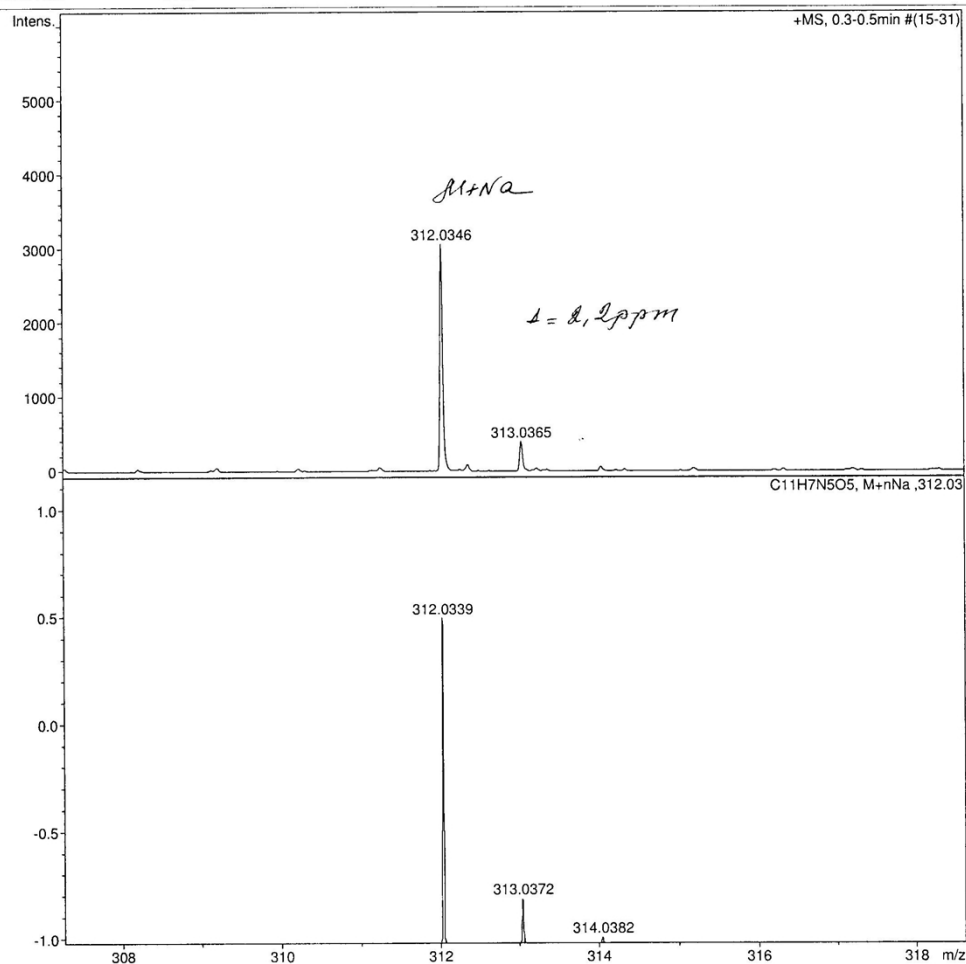
Analysis Name D:\Data\Kolotyrkina\2014\Fershtat\1210003.d
Method tune_low.m
Sample Name /USED 726
Comment C11H7N5O5 mw 289 calibrant added

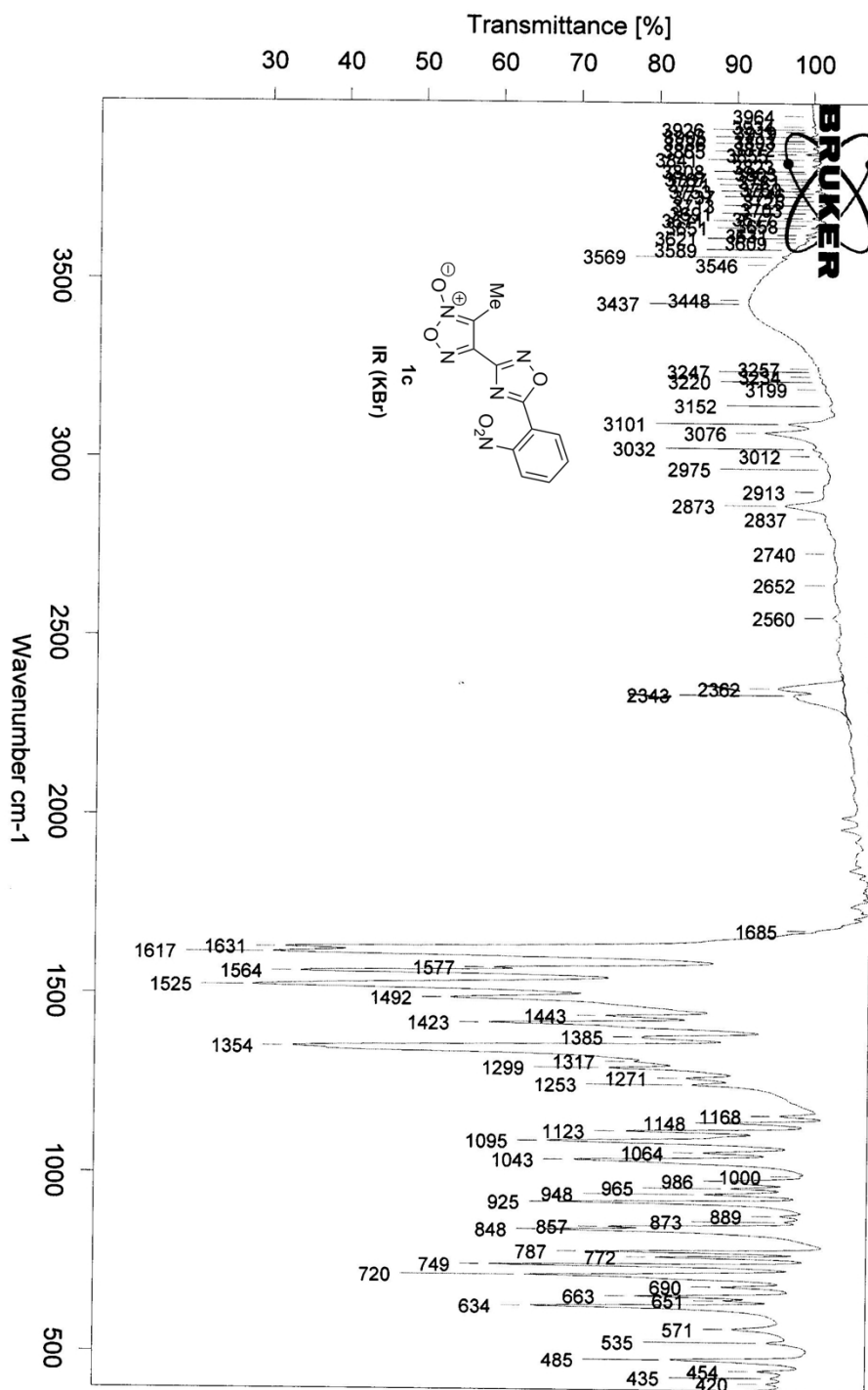
Acquisition Date 10.12.2014 14:55:31

Operator BDAL@DE
Instrument / Ser# micrOTOF 10248

Acquisition Parameter

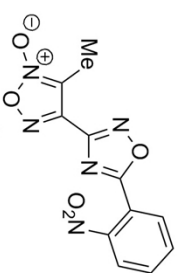
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



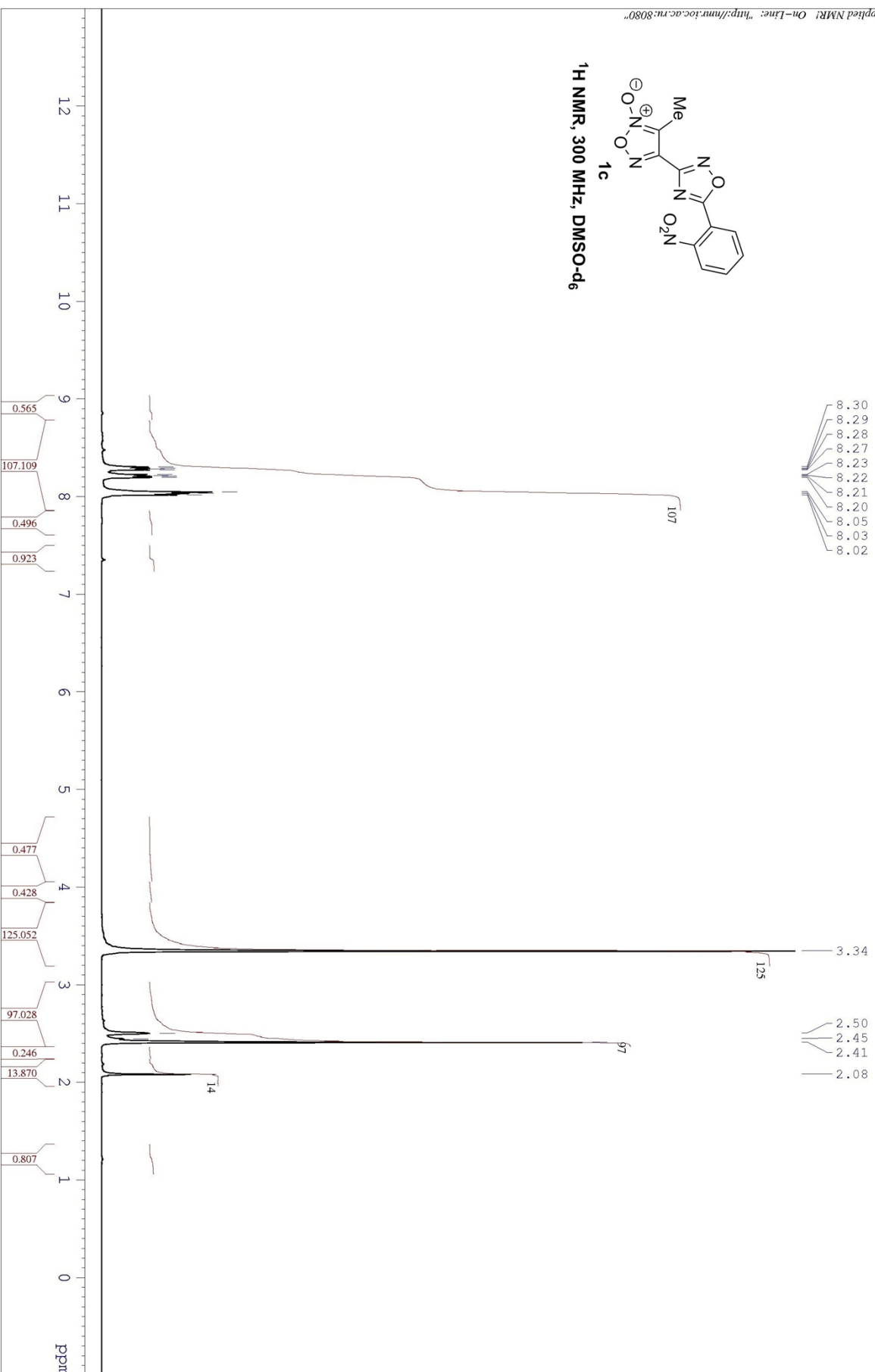


/USED 753

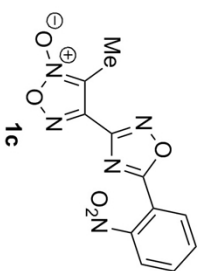
© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=300.13 MHz (1H) SI=16K SW=6010.01=2401 PW=9.0 AQ=1.351 RD=3.00 NS=1 SR=-1.17 TE=301K 16 December 2014 Op: Struchkova M.I.; Solv: DMSO-d6;
The Best Applied NMR! On-Line: "http://nmr.toc.ac.ru:8080"



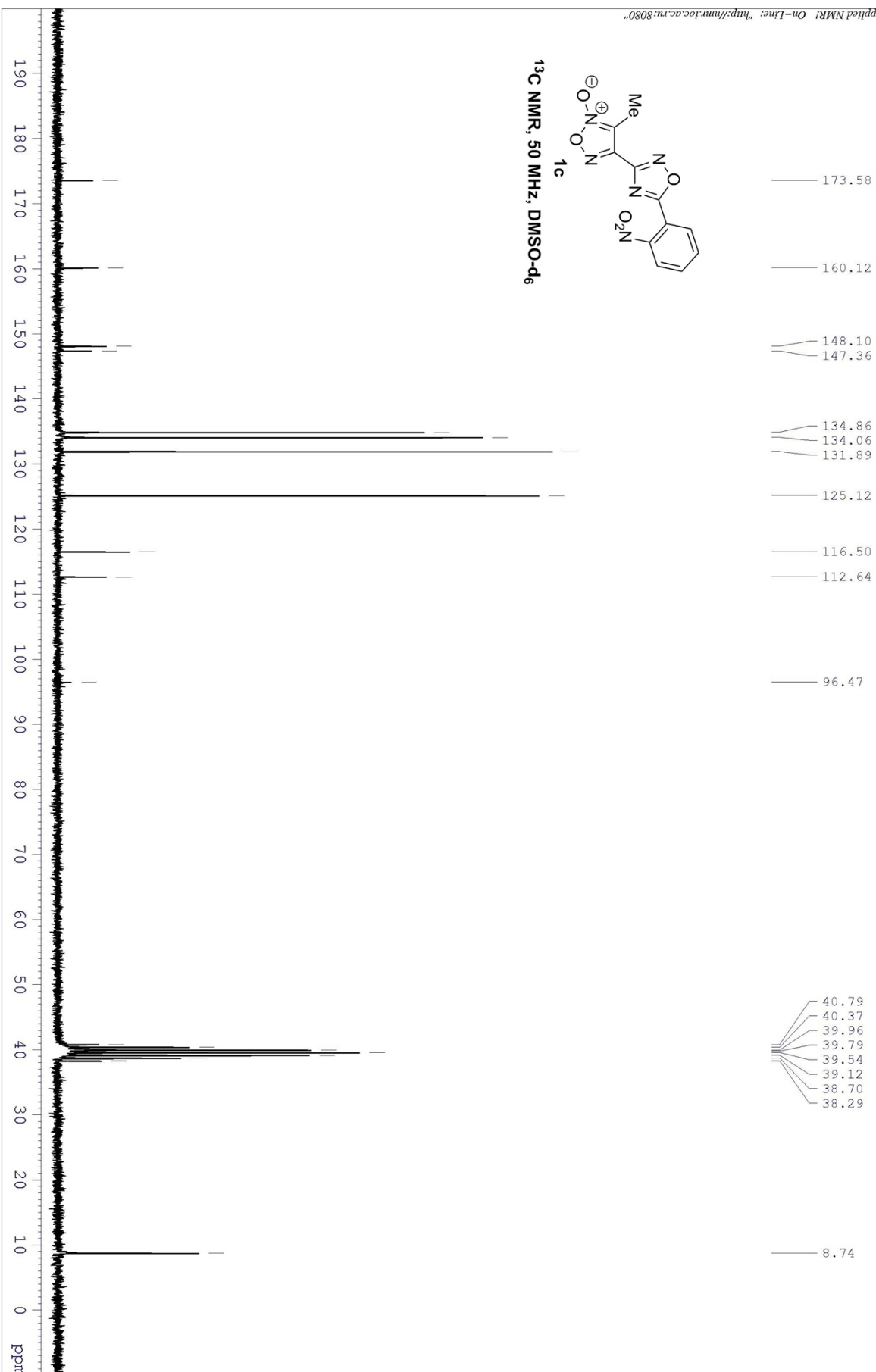
¹H NMR, 300 MHz, DMSO-d₆

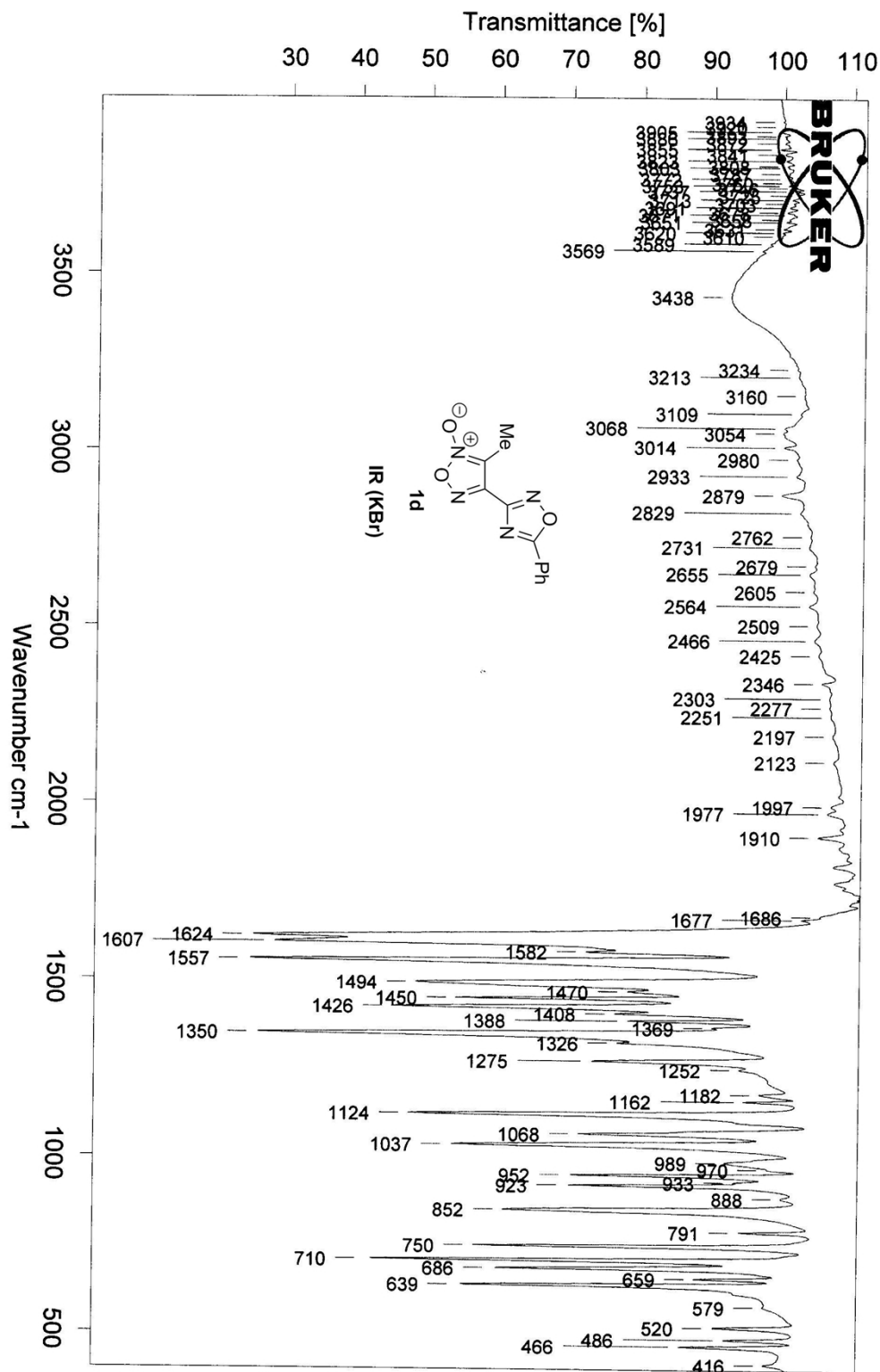


/USED 753



¹³C NMR, 50 MHz, DMSO-d₆

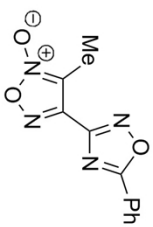




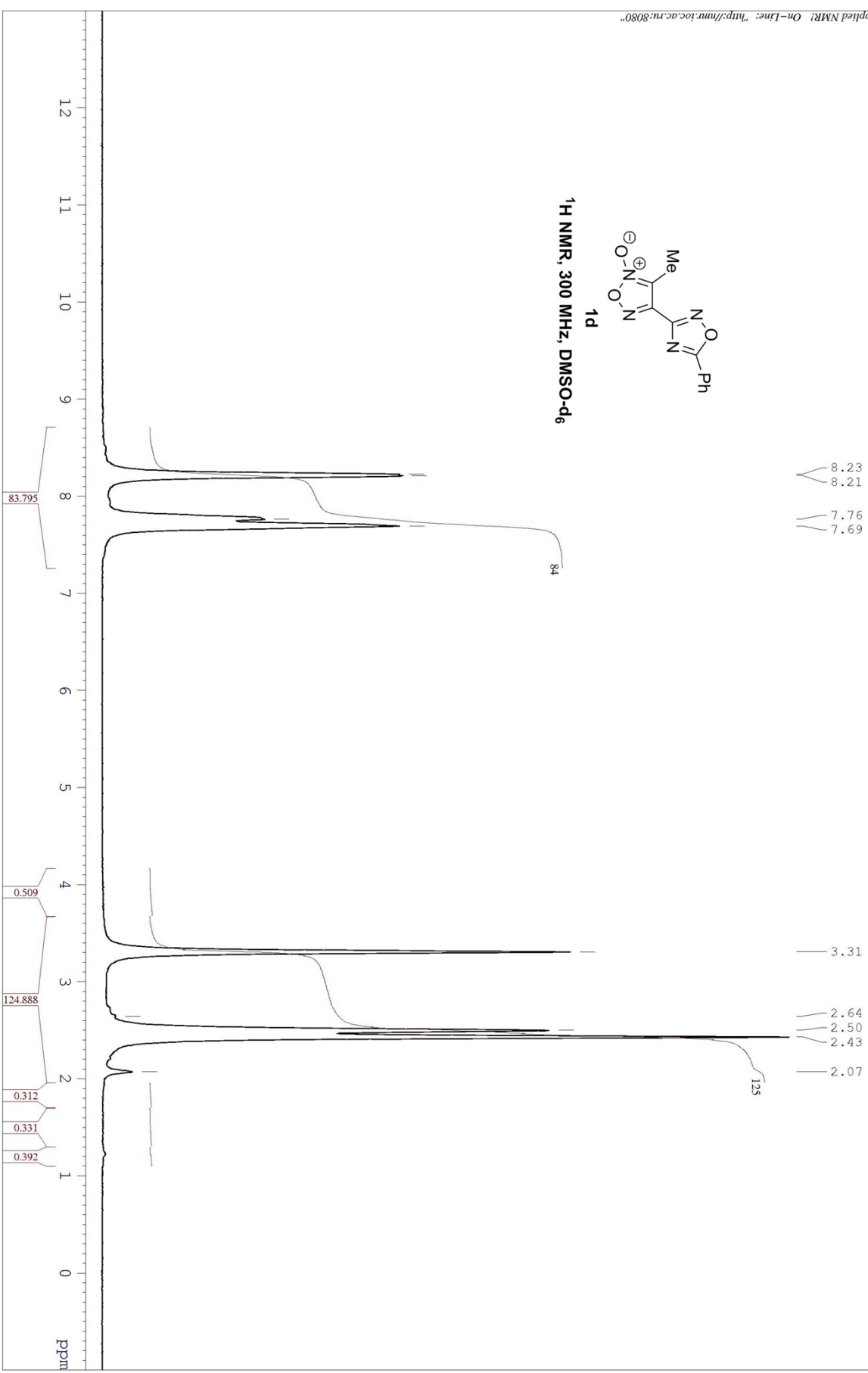
D:\EDL\LEO-705.0

ФЕРИТАТ. LEO-705, пресовка с KBr. 1/200.

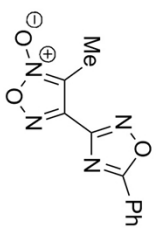
03.12.2014



1d
¹H NMR, 300 MHz, DMSO-d₆

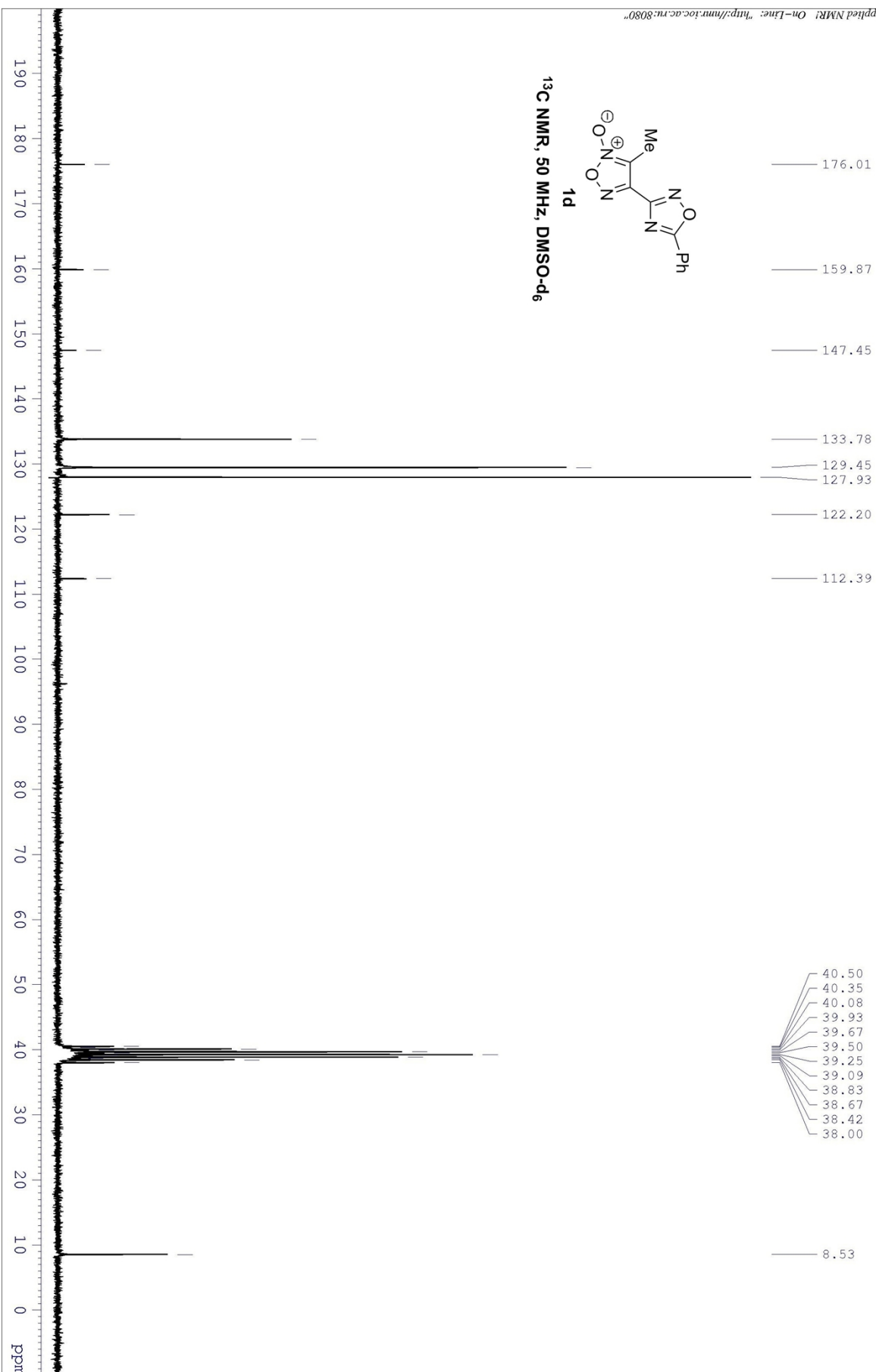


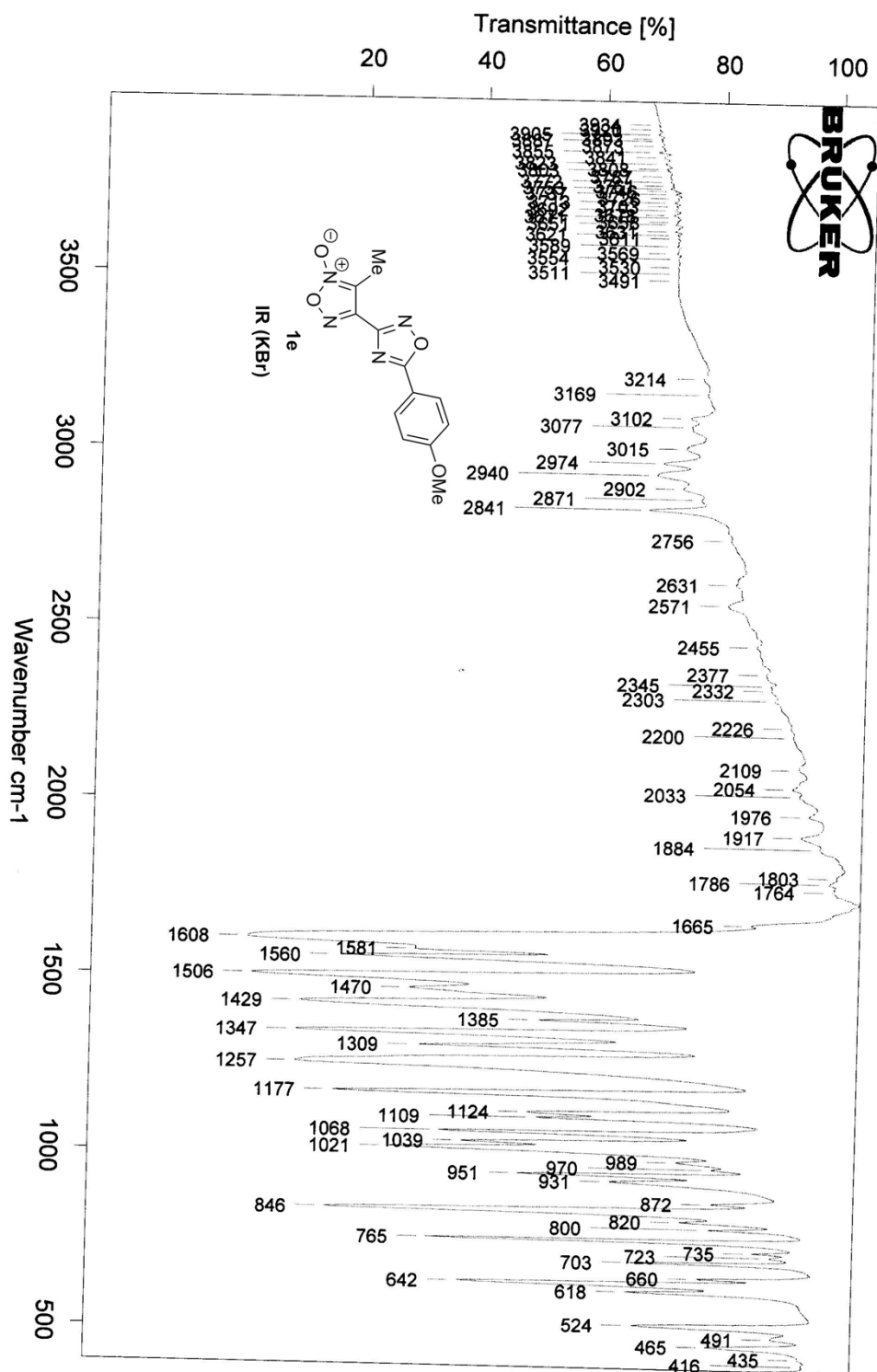
/USED 705.C13



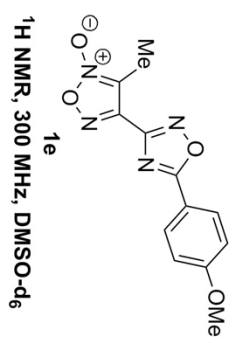
1d

¹³C NMR, 50 MHz, DMSO-d₆

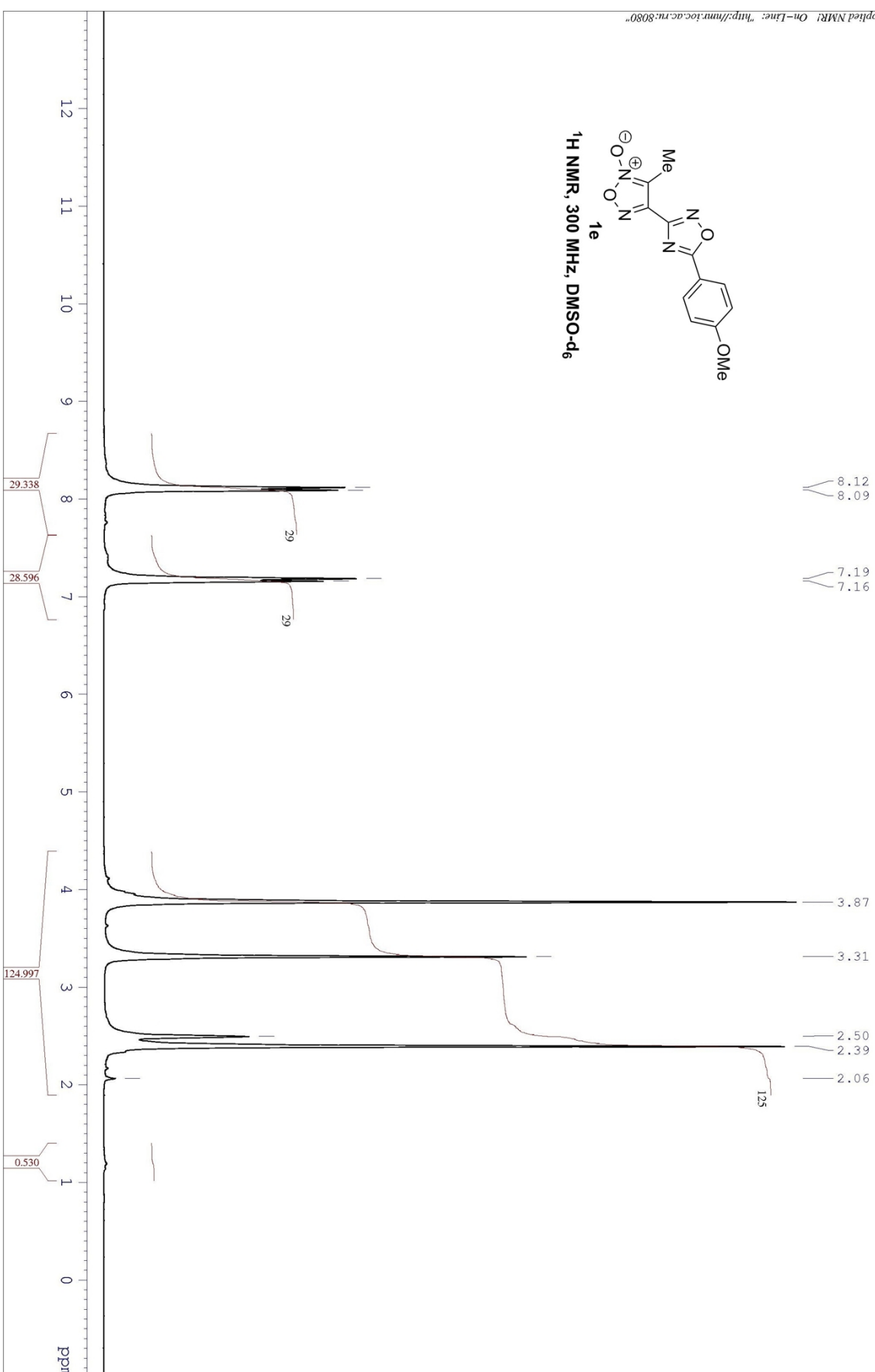




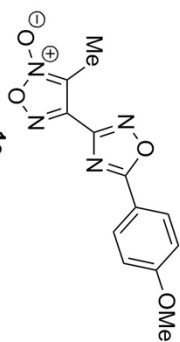
/USED 760.2



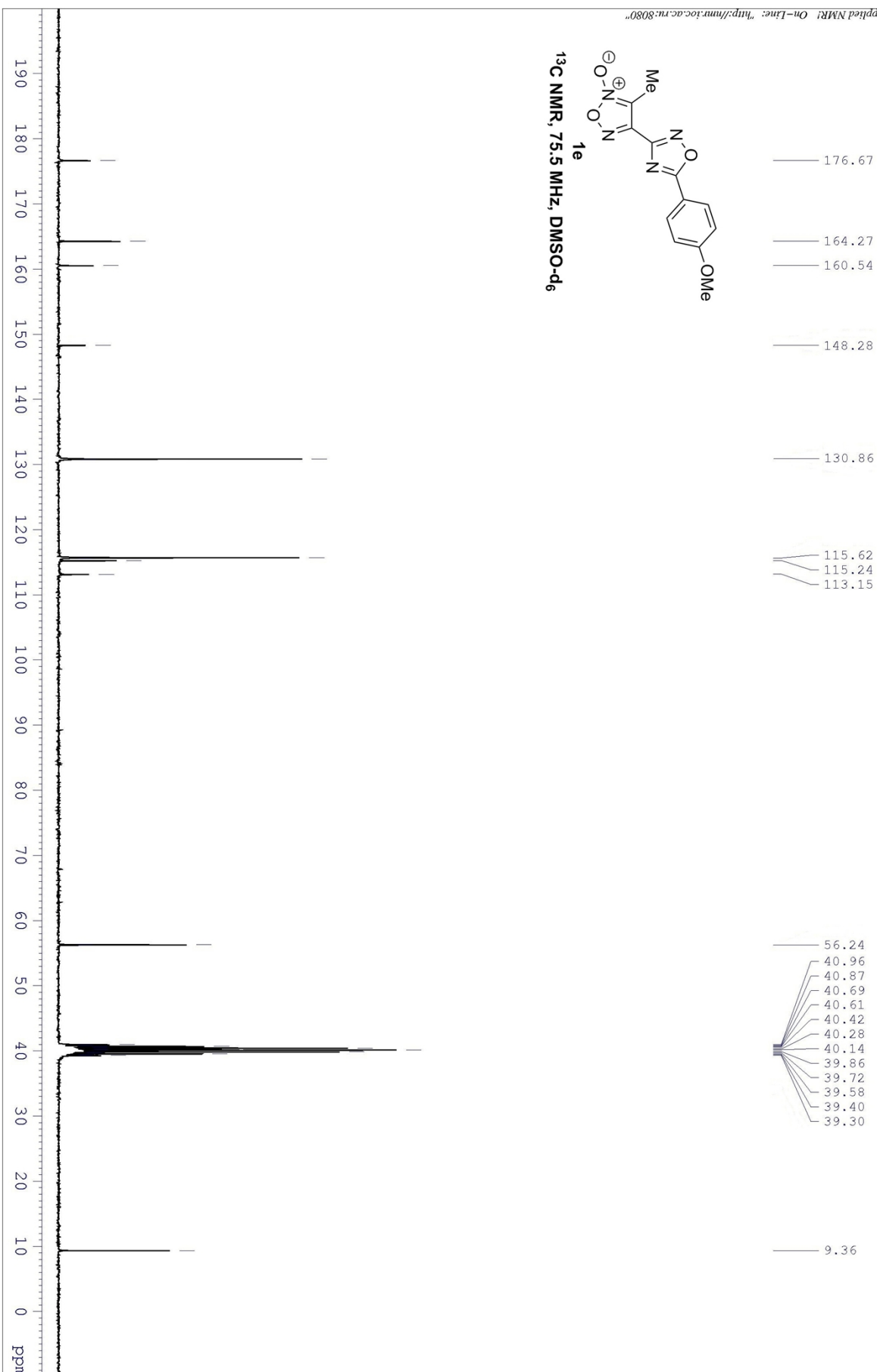
¹H NMR, 300 MHz, DMSO-d₆



/USED 760.2



13C NMR, 75.5 MHz, DMSO-d₆

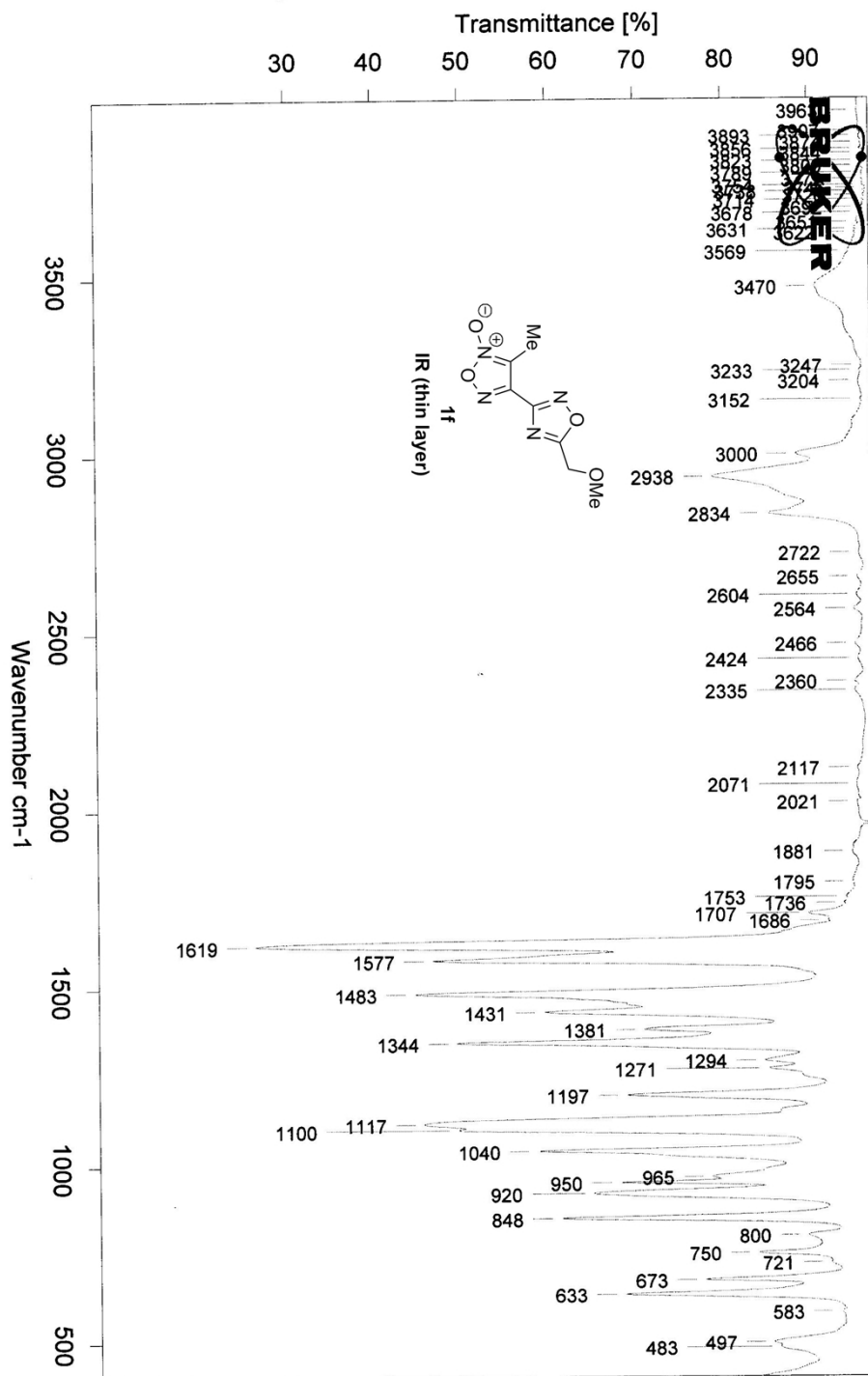


D:\EDU\LEO-744.0

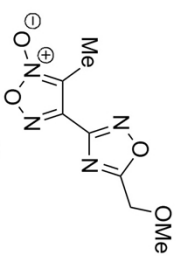
ФЕРУЛАТ. ЛЕО-744, тонкий слой, пл. КВ.

03.03.2015

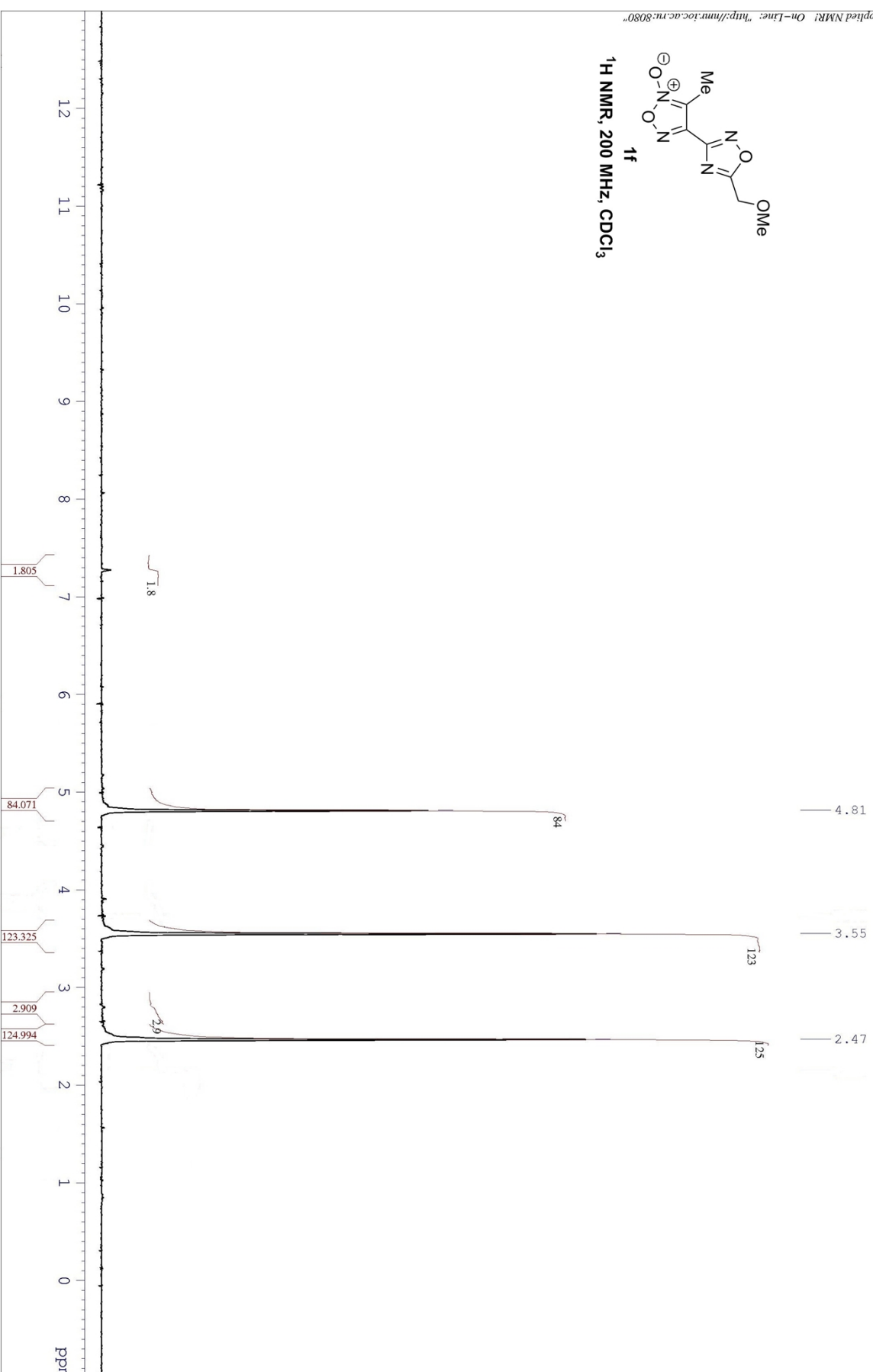
Page 1/1



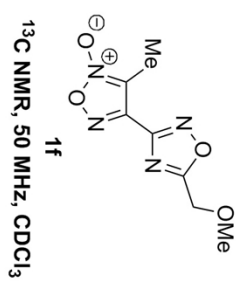
/USED 744



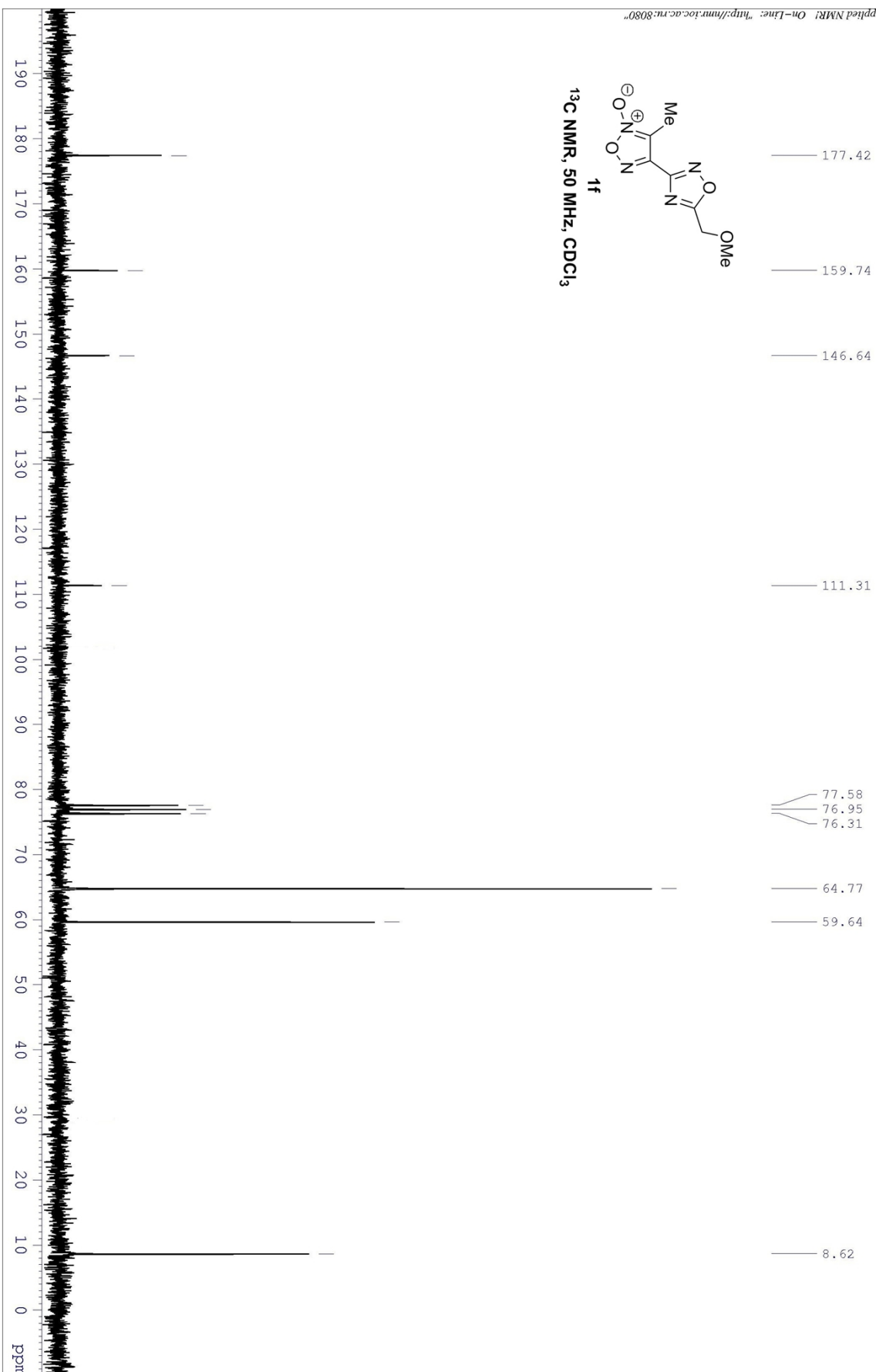
¹H NMR, 200 MHz, CDCl₃

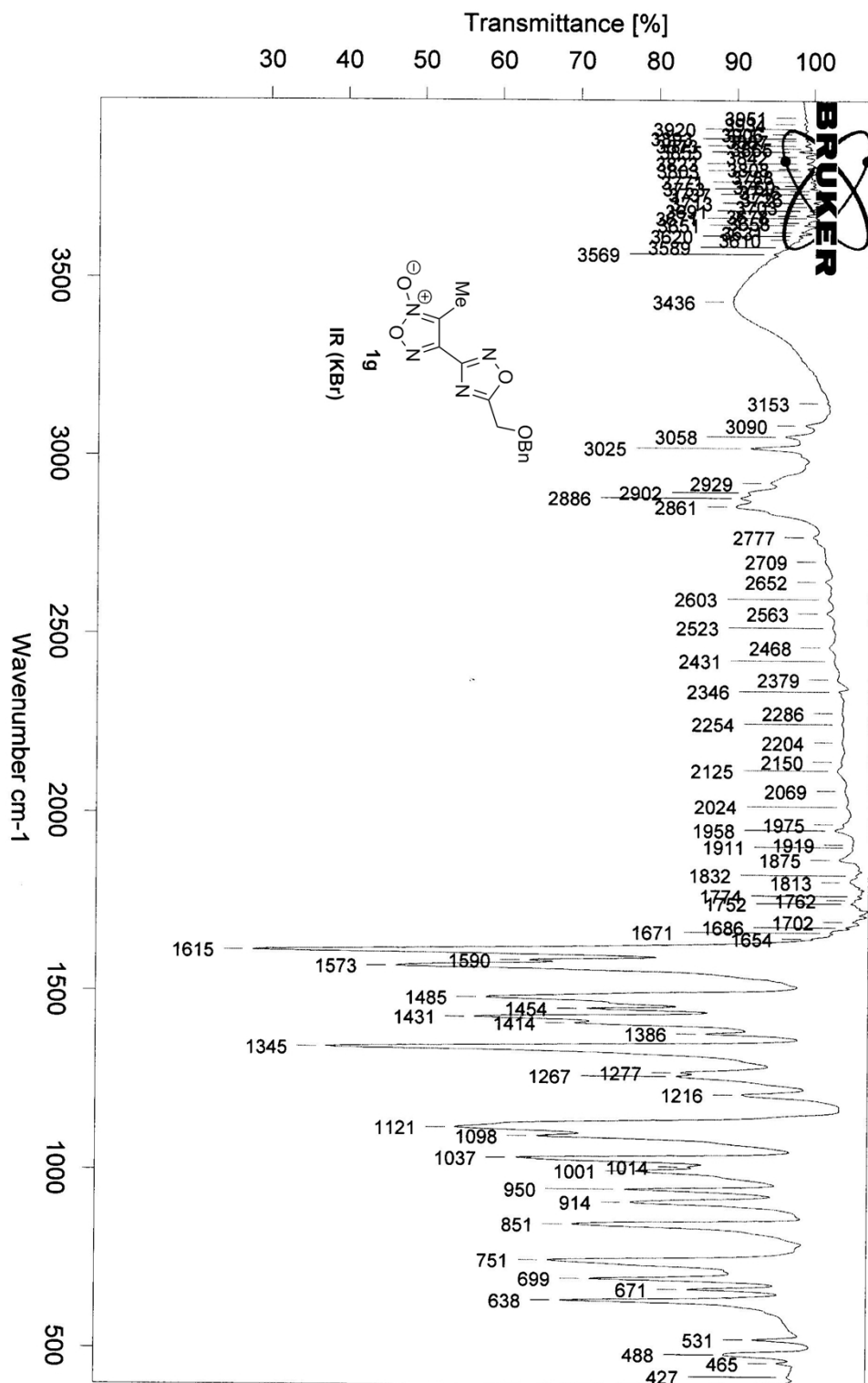


/USED 744.C13



¹³C NMR, 50 MHz, CDCl₃





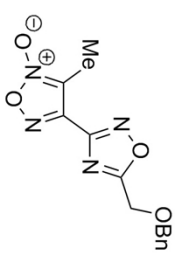
D:\ED\LEO-736.0

ФЕРИТАТ. LEO-736 , прессовка с KBr, 1/200.

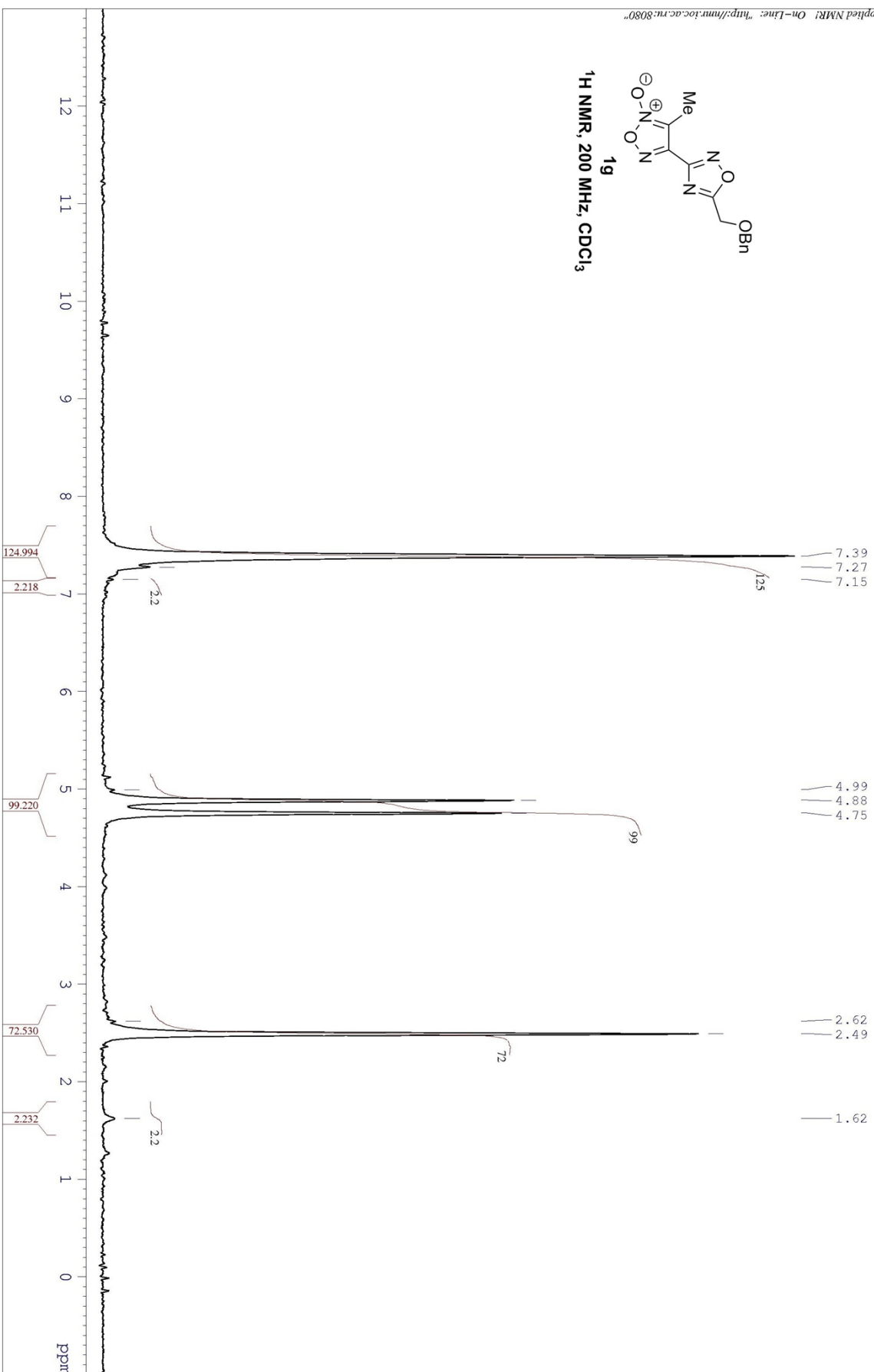
10.12.2014

/USED 736

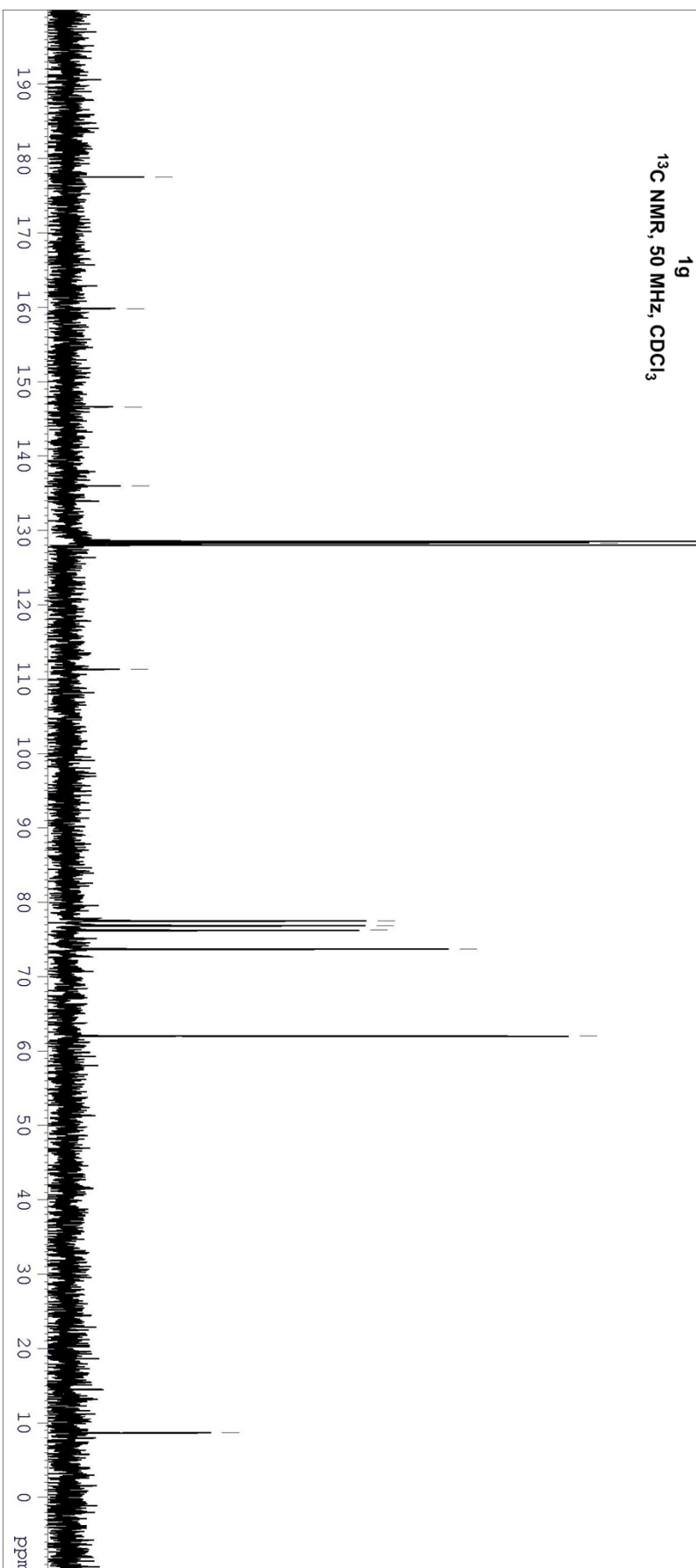
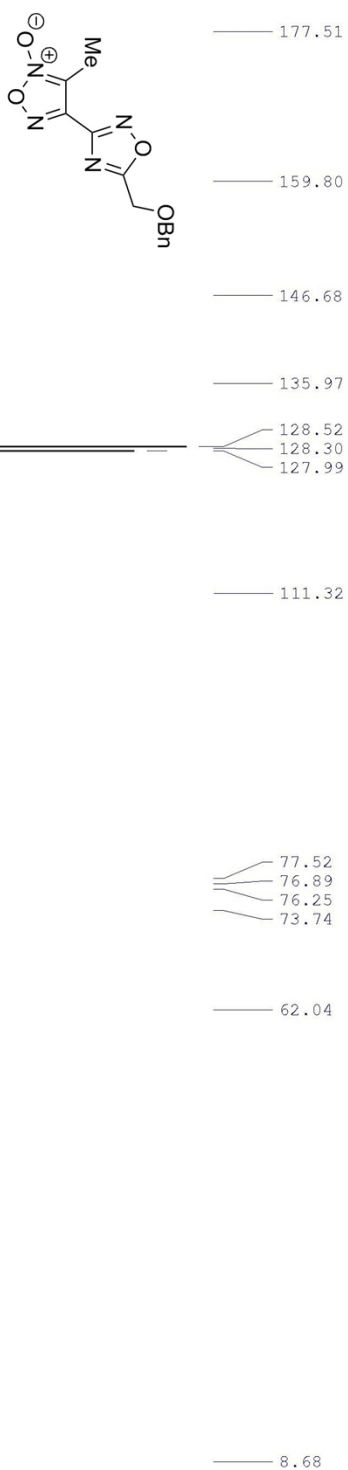
© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AC200 SF=200.13 MHz (1H) SI=16K SW=4000 OI=3939 PW=6.0 AQ=2.048 RD=3.00 NS=32 SR=2338.00 TE=297K 26 November 2014 Op: Struchkova M.I.; Solv: CDCl3;



1g
¹H NMR, 200 MHz, CDCl₃



/USED 736.C13



Display Report

Analysis Info

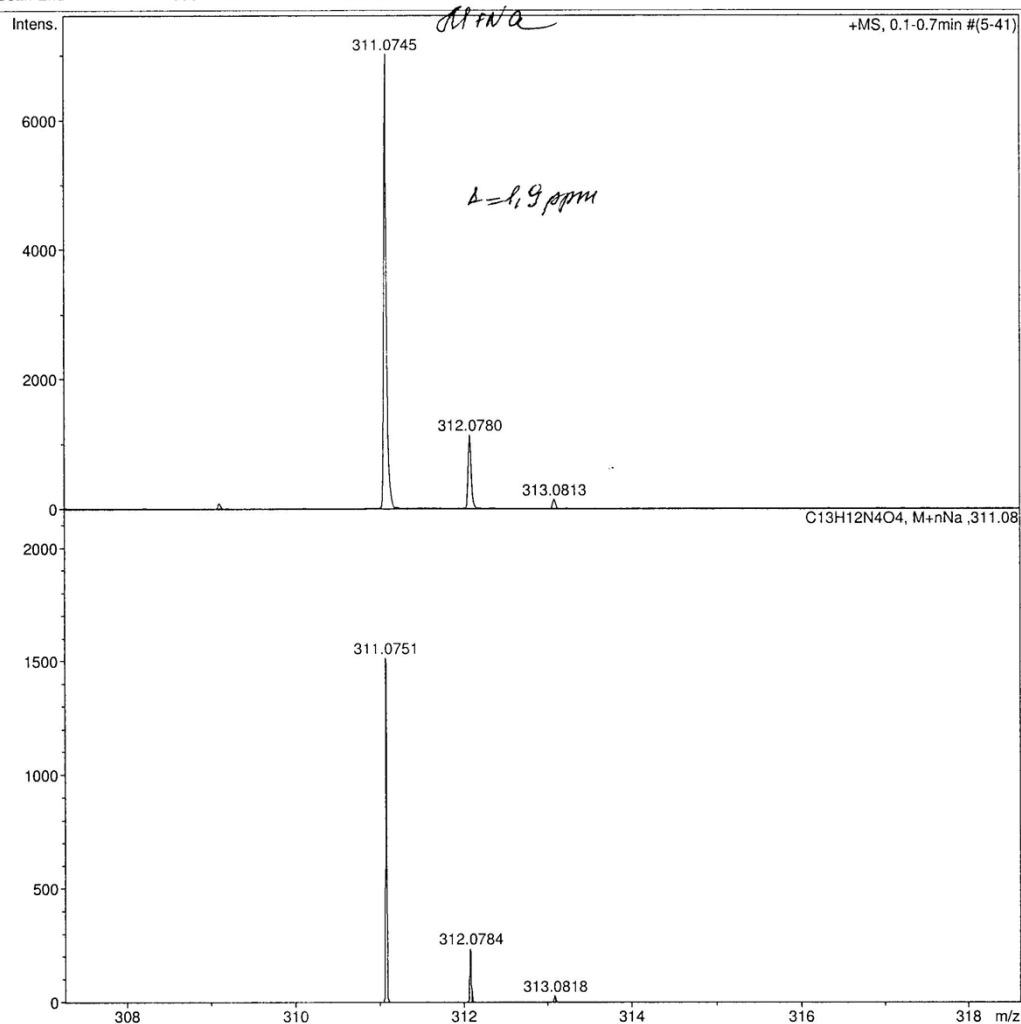
Analysis Name D:\Data\Kolotyrkina\2014\Fershtat\1210004.d
Method tune_low.m
Sample Name /USED 736
Comment C13H12N4O4 mw 288 calibrant added

Acquisition Date 10.12.2014 15:03:02

Operator BDAL@DE
Instrument / Ser# micrOTOF 10248

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste

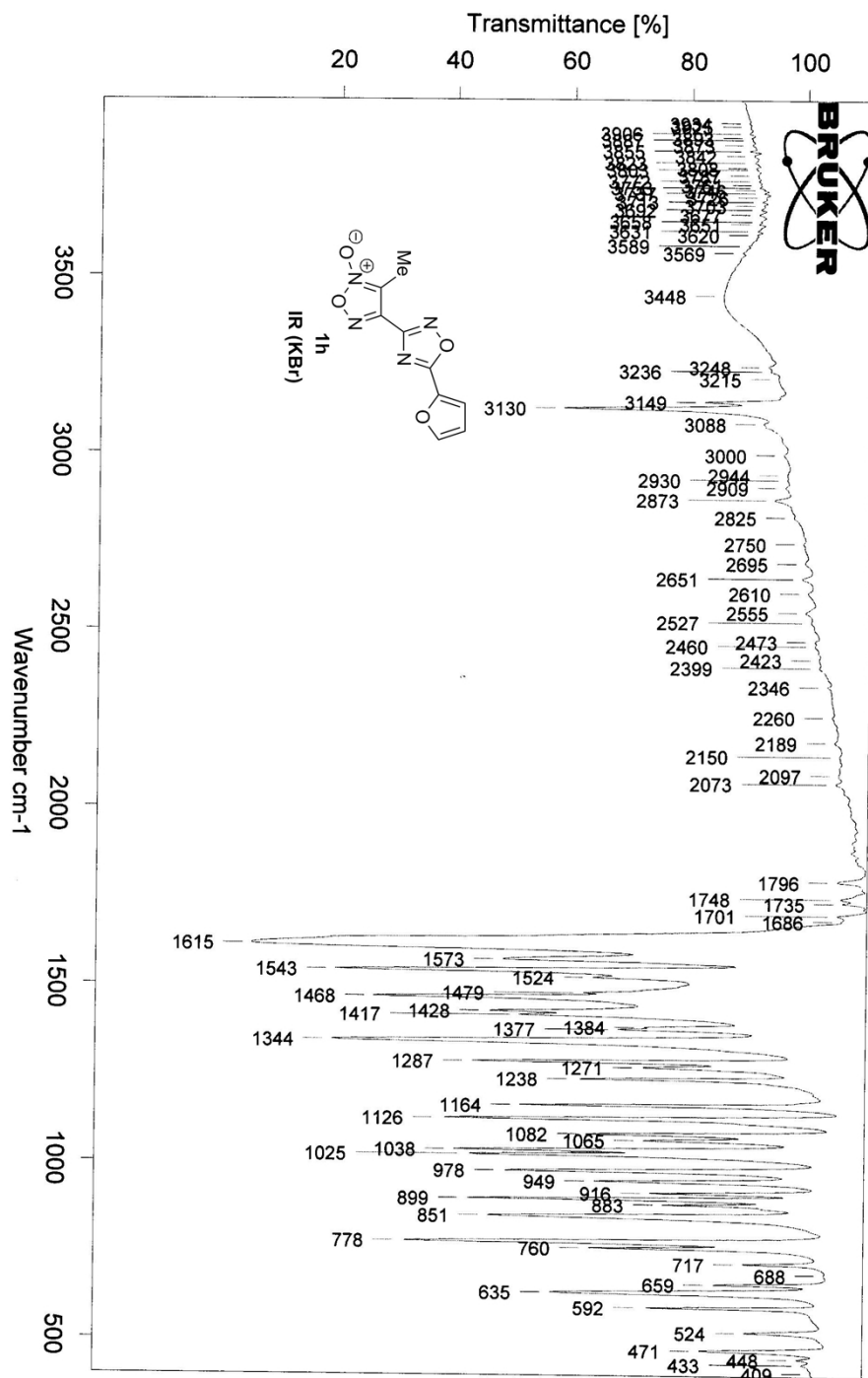


D:\EDL\LEO-747.0

ФЕРИТАТ. LEO-747, прояснения с KBr, 1/200.

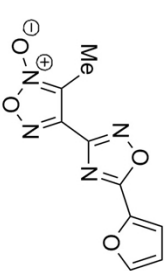
10.12.2014

Page 1/1

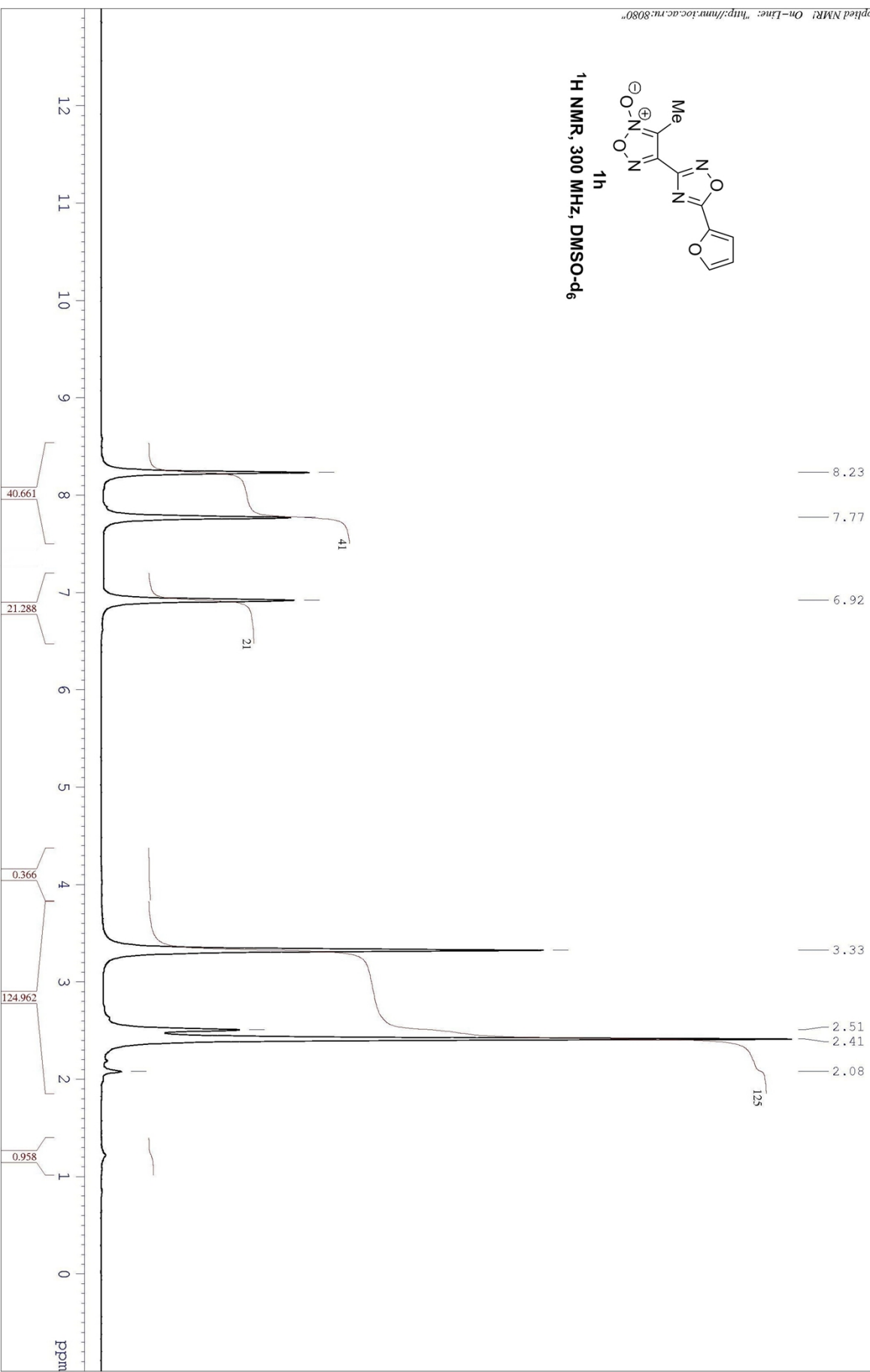


/USED 747.2

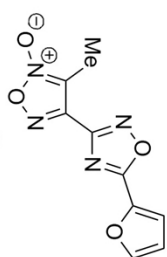
© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AM300 SF=300.13 MHz 1H SI=16K SW=6010 O1=2401 PW=9.0 AQ=1.351 RD=3.00 NS=1 SR=-4.17 TF=300K 9 December 2014 Opr: Struchkova M.I.; Solv: DMSO-d6;
 The Best Applied NMR! On-Line: "http://nmr.ioc.ac.msc:8080"



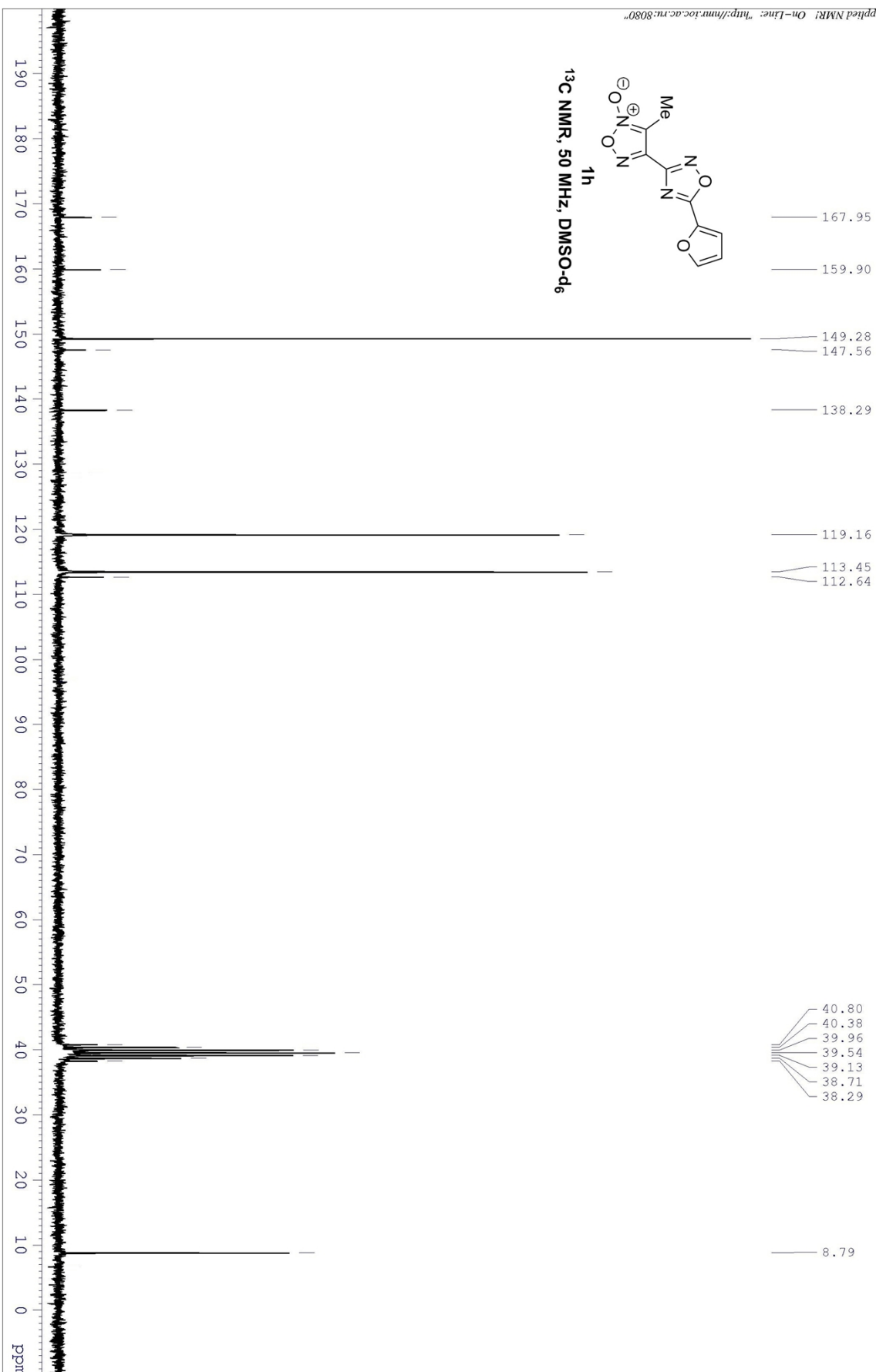
¹H NMR, 300 MHz, DMSO-d₆



/USED 747.2C13

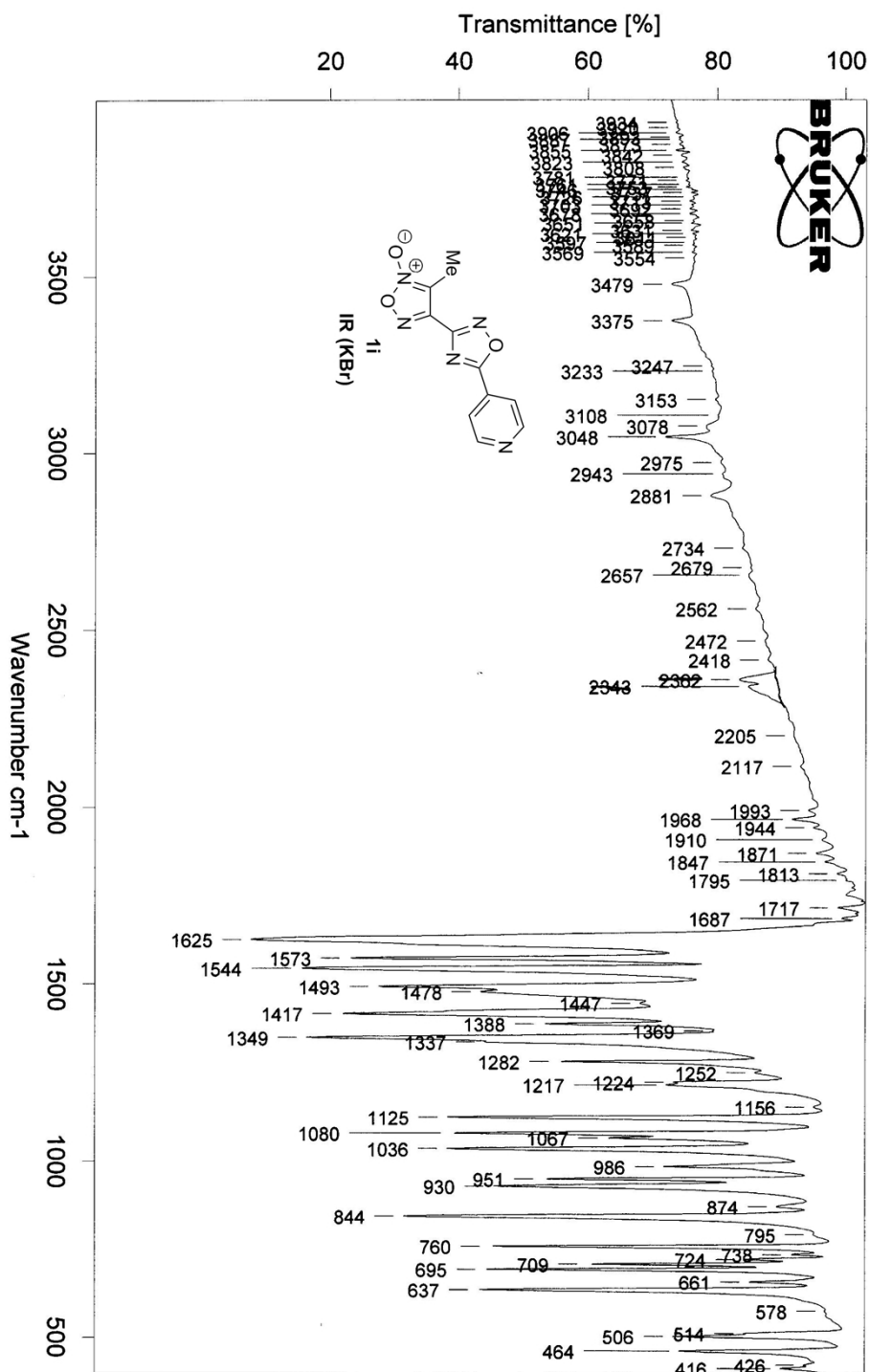


¹³C NMR, 50 MHz, DMSO-d₆

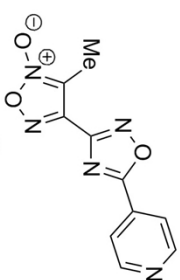


D:\EDL\756.0 ФЕРУЛАТ. 756 , препарат с KBr, 1/200.

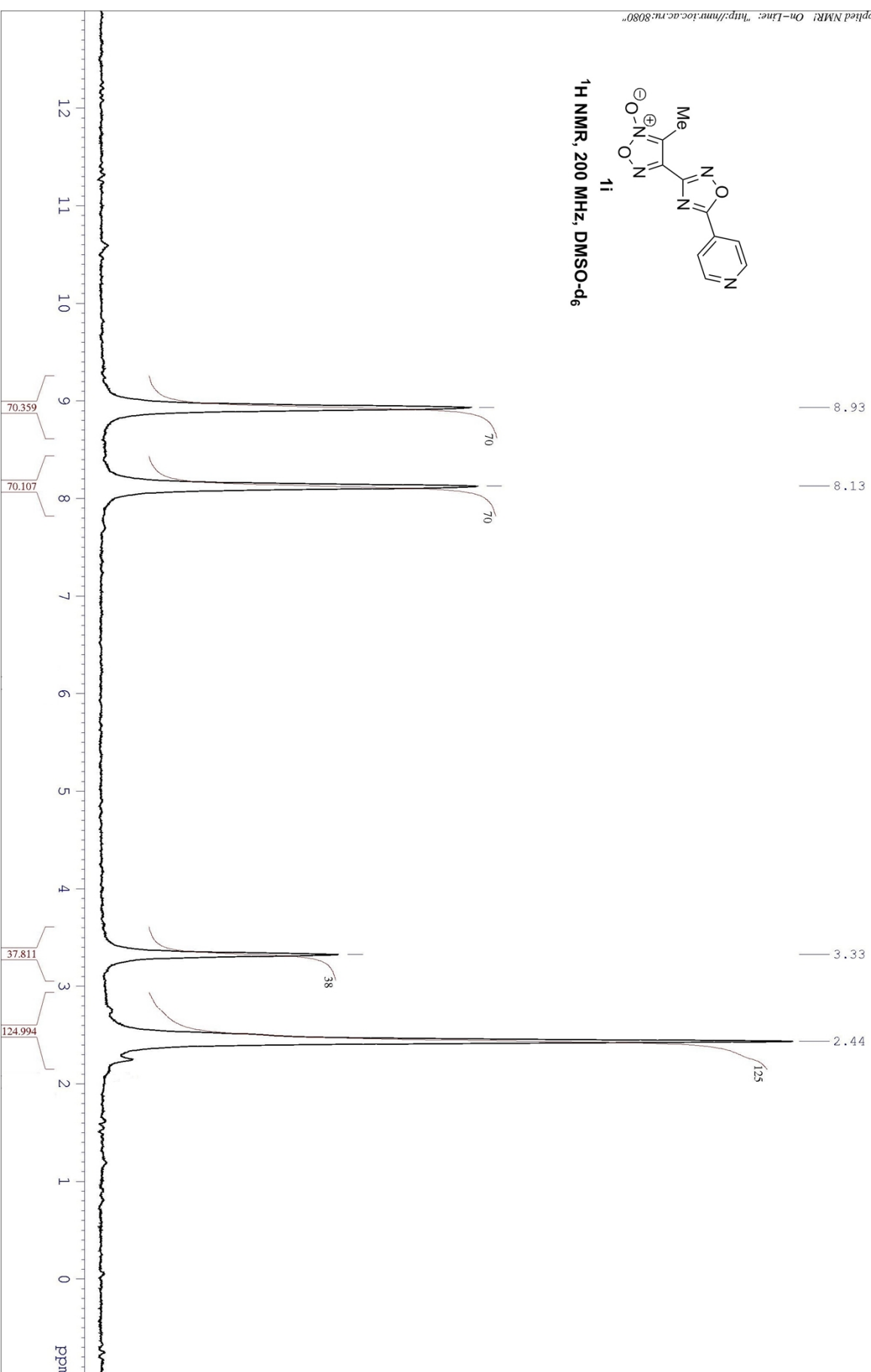
24.02.2015



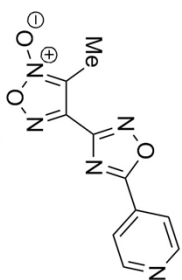
/USED 756



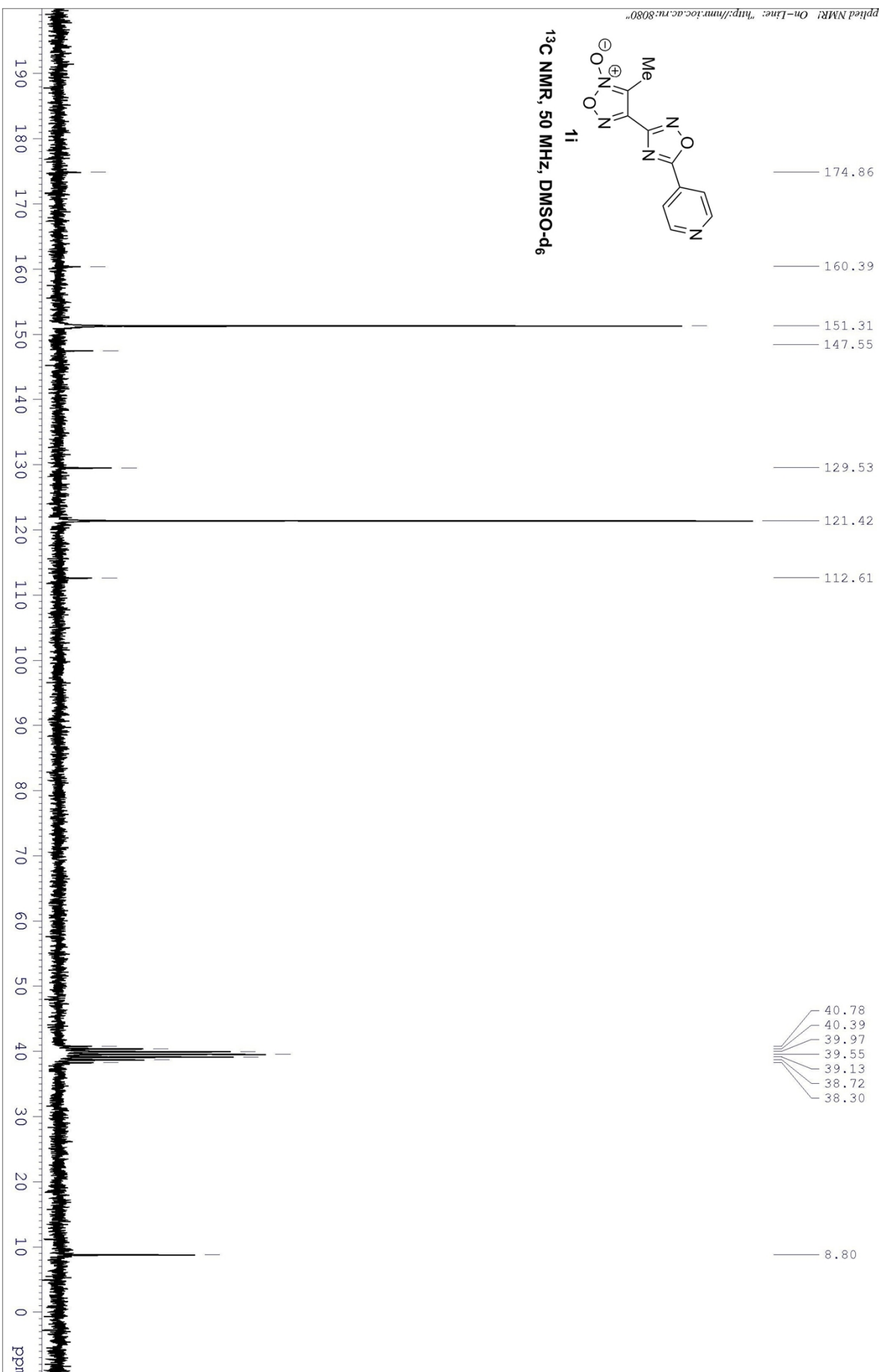
¹H NMR, 200 MHz, DMSO-d₆

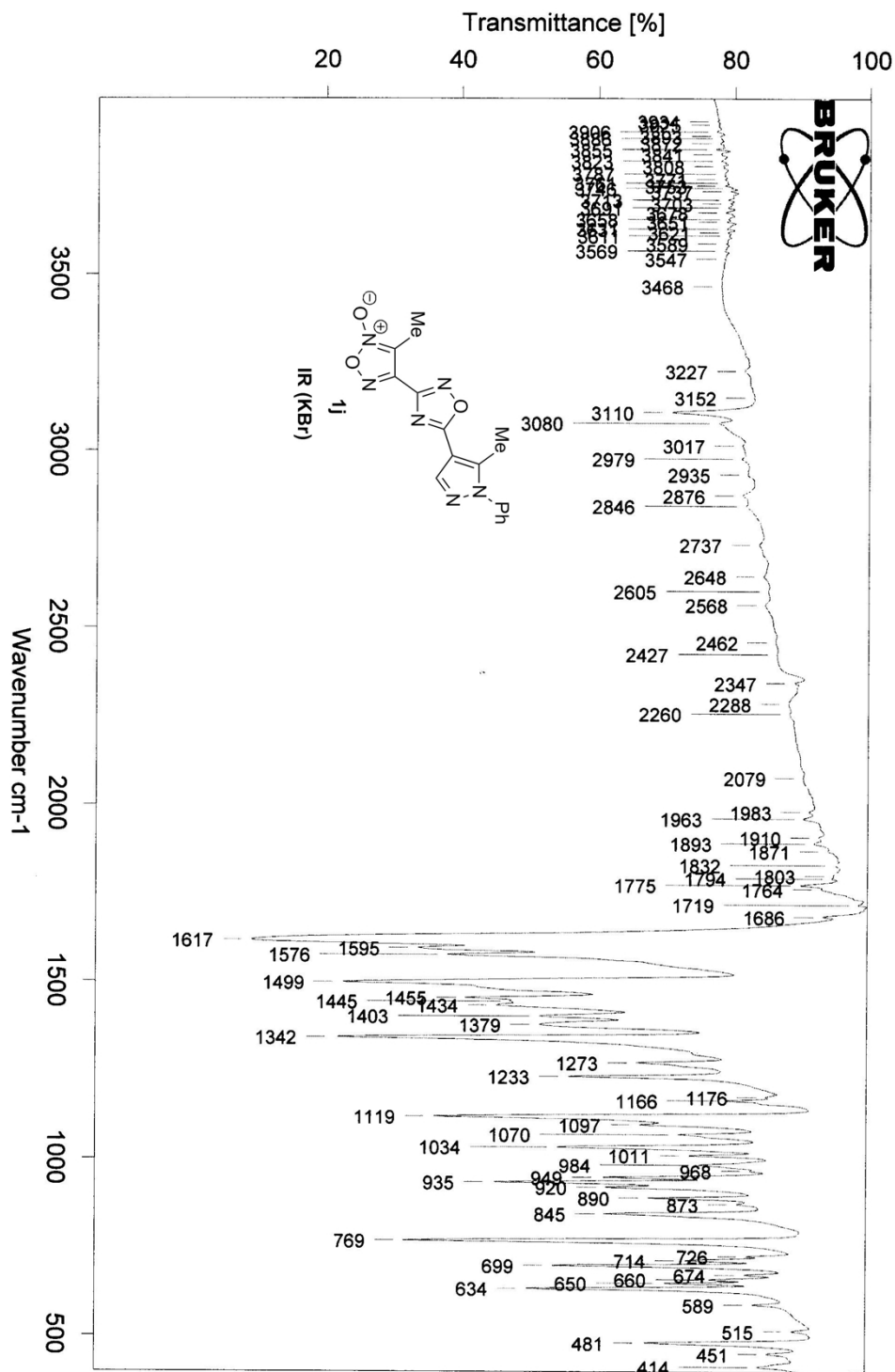


/USED 756.C13

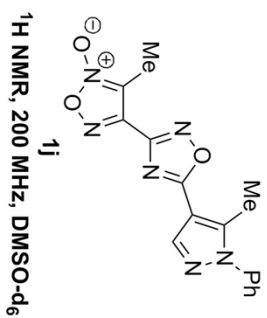


¹³C NMR, 50 MHz, DMSO-d₆

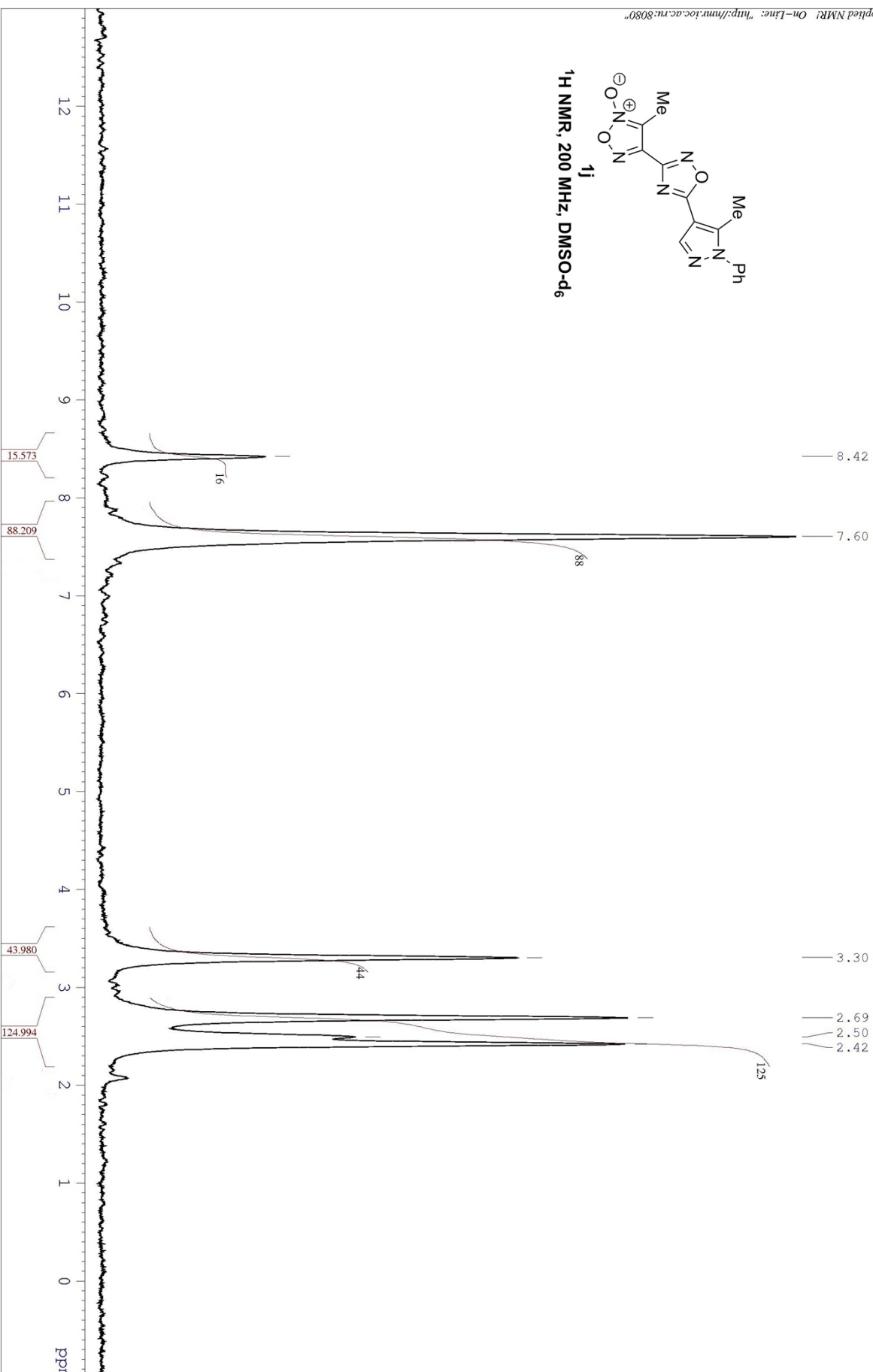




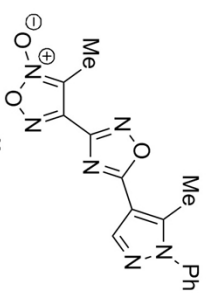
/USED 785



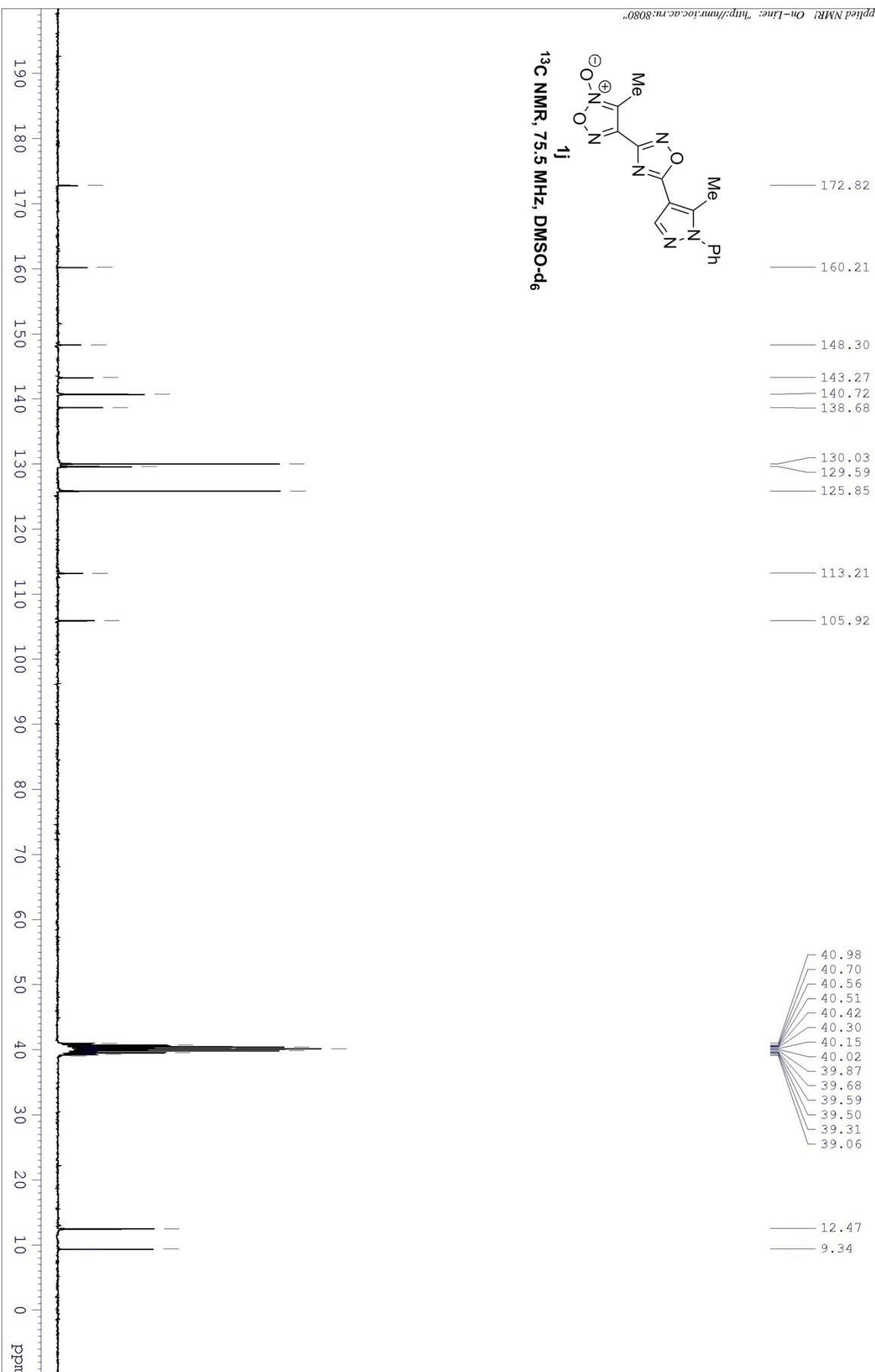
¹H NMR, 200 MHz, DMSO-d₆



/USED 785



¹³C NMR, 75.5 MHz, DMSO-d₆



Display Report

Analysis Info

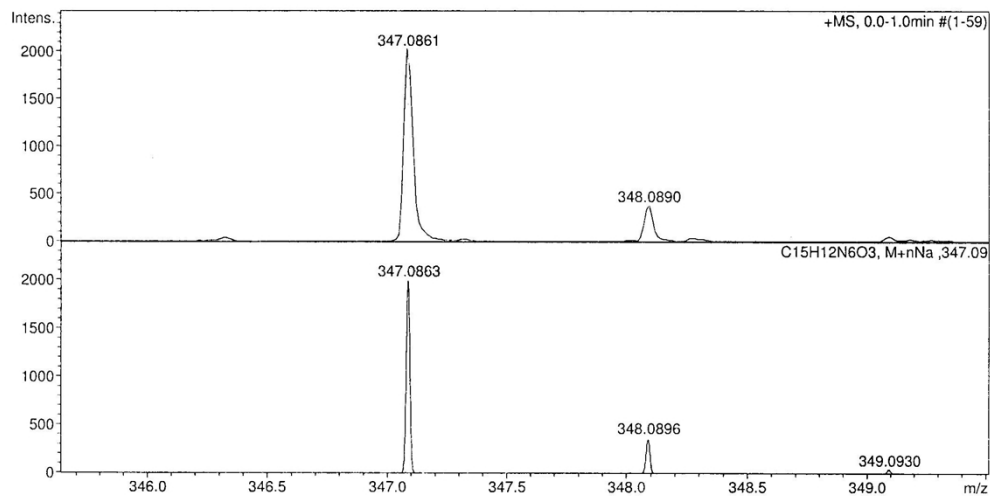
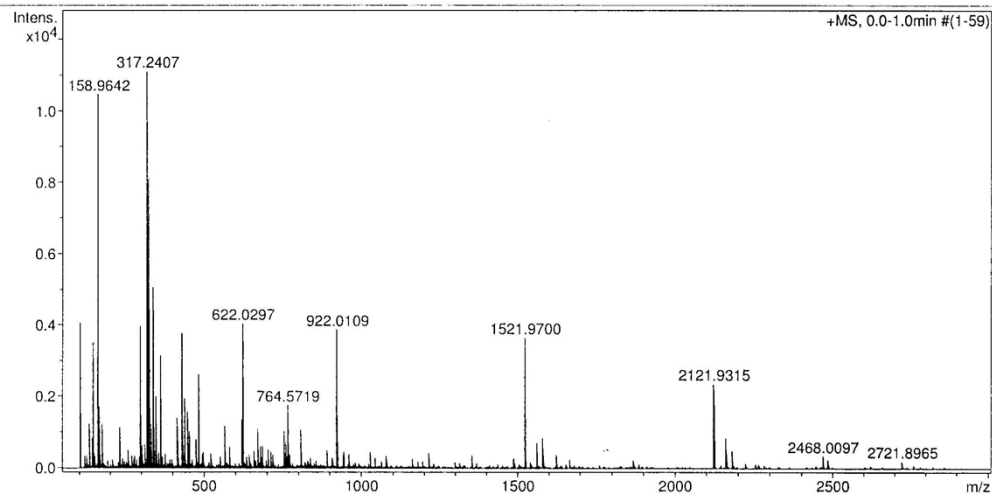
Analysis Name D:\Data\Chizhov\Makhova\Fershtat\785_&clblow.d
Method tune_low.m
Sample Name /USED 785
Comment CH3CN 100 %, dil. 200, calibrant added

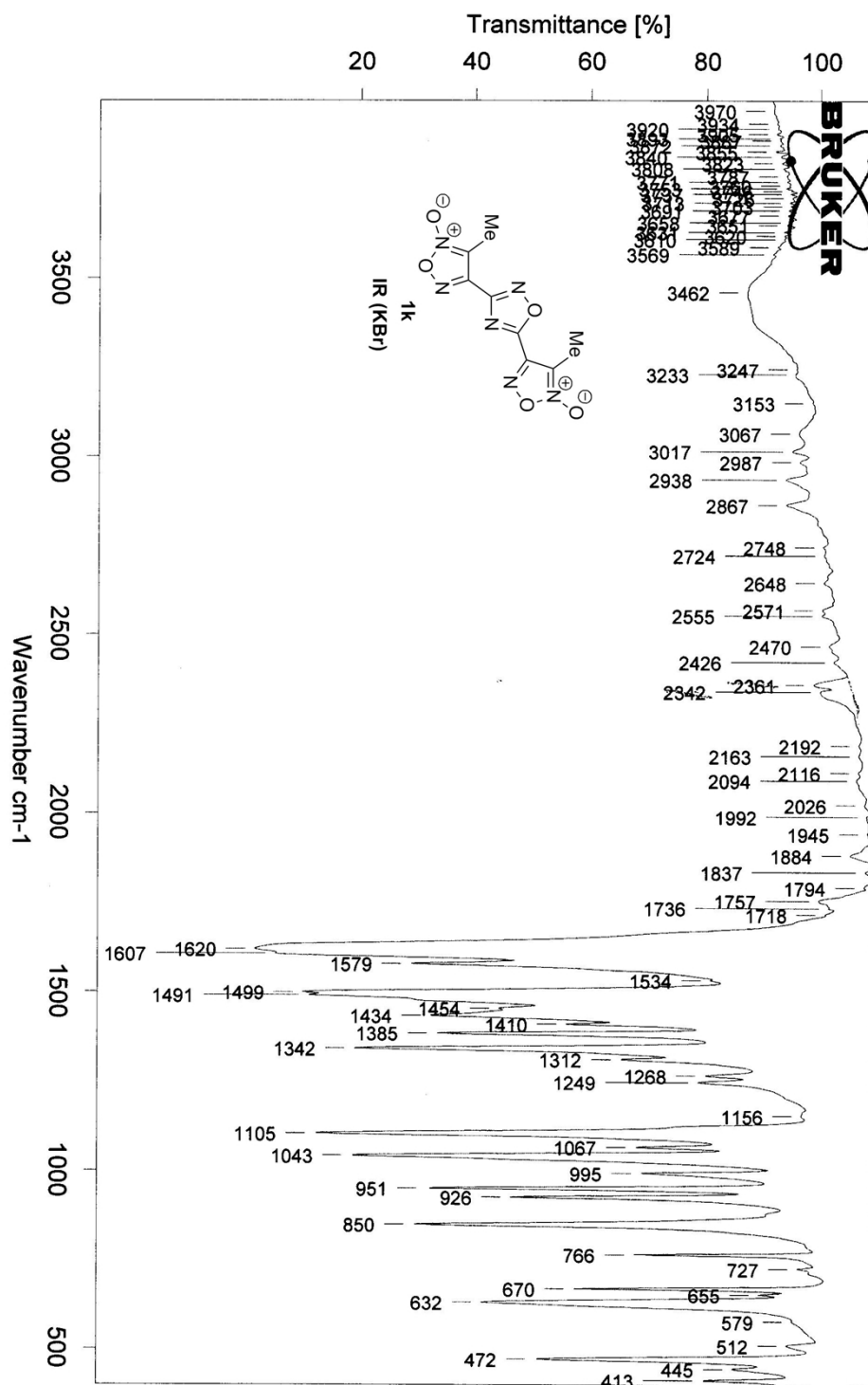
Acquisition Date 02.04.2015 14:57:07

Operator BDAL@DE
Instrument / Ser# micrOTOF 10248

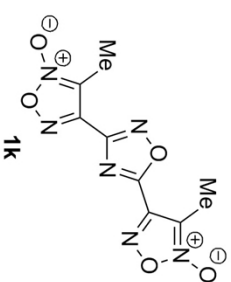
Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste

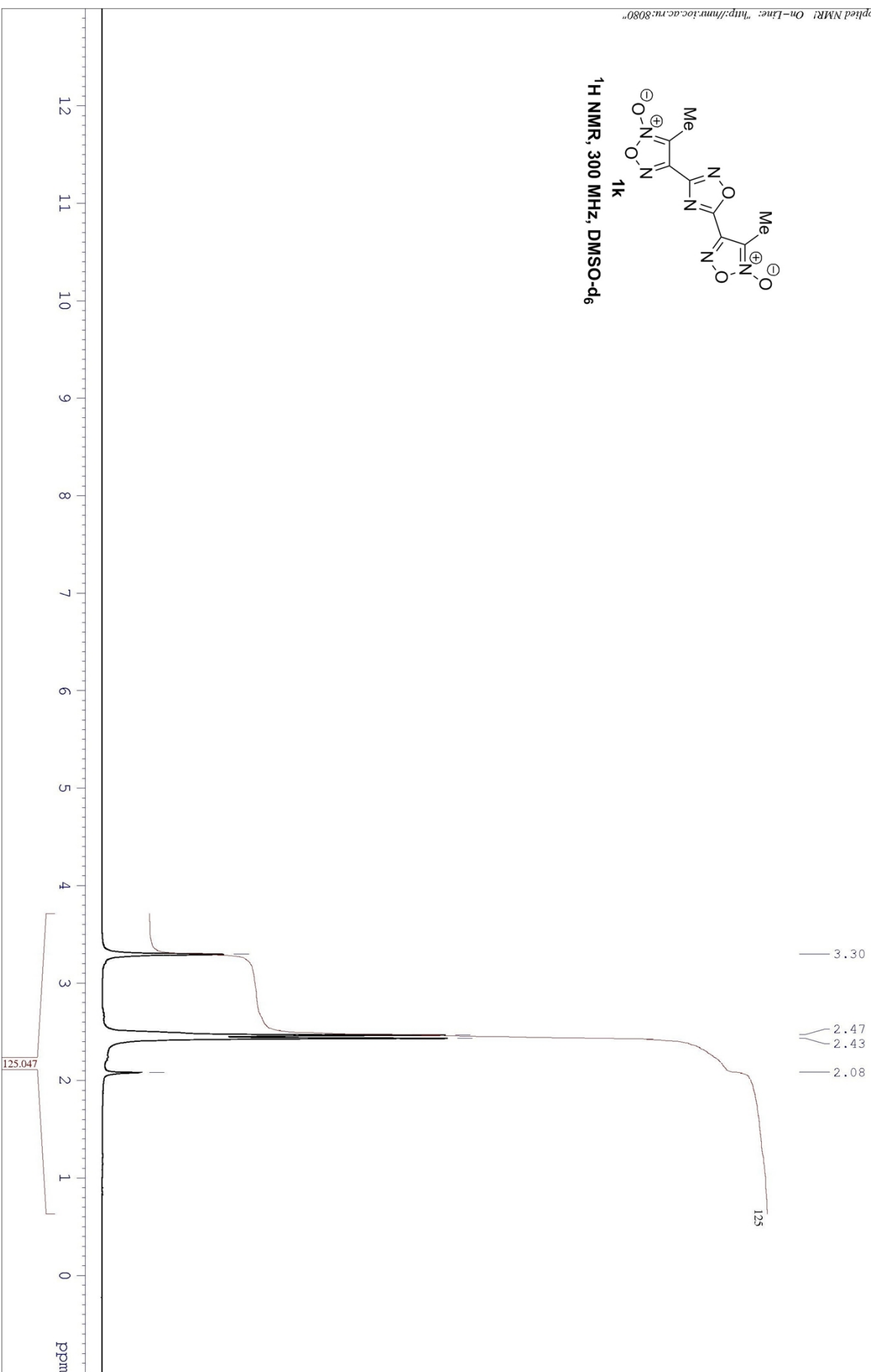




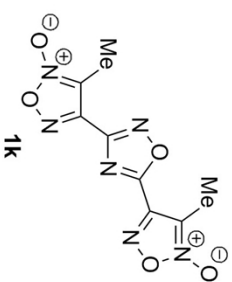
/USED 777



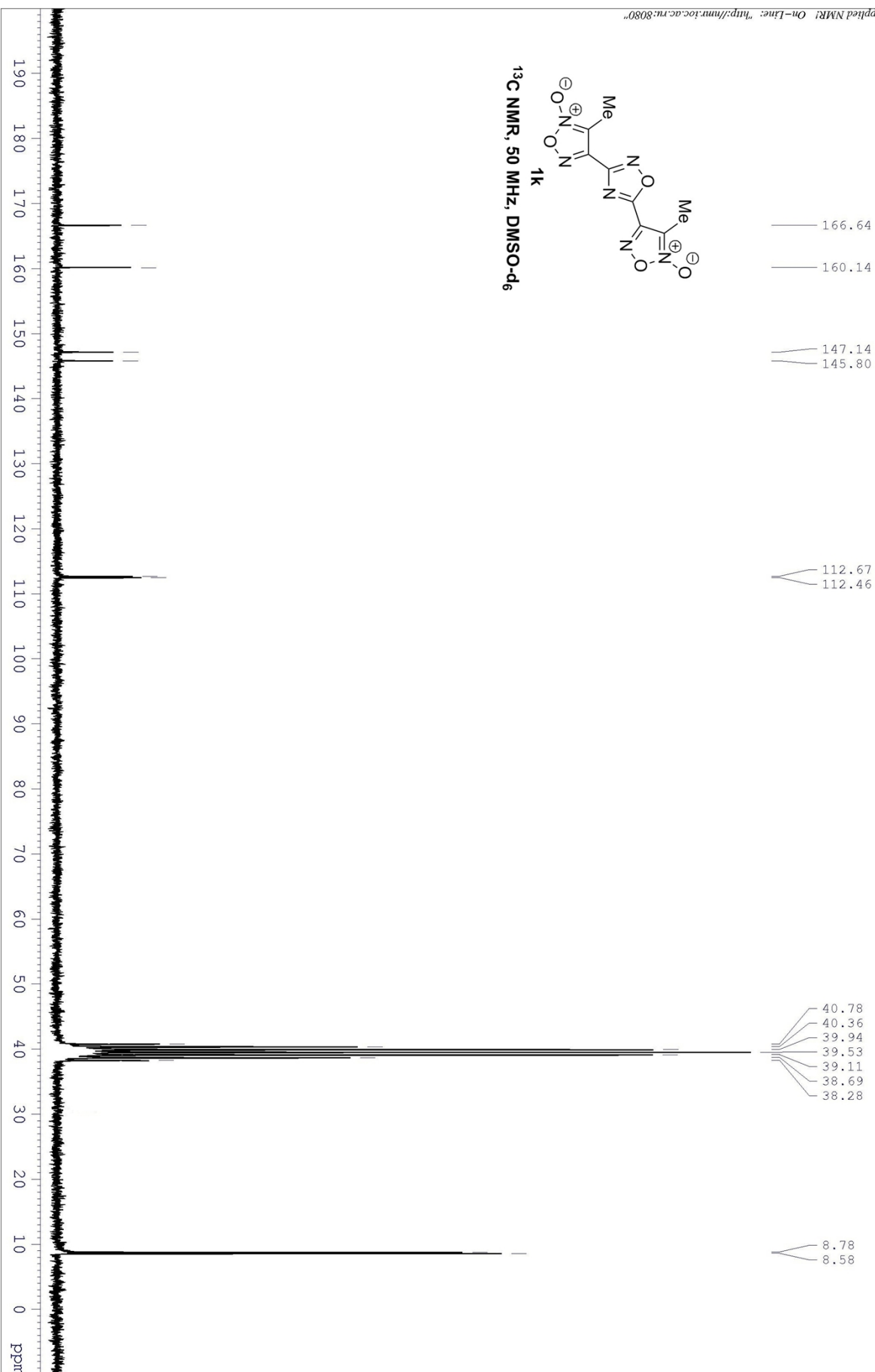
¹H NMR, 300 MHz, DMSO-d₆



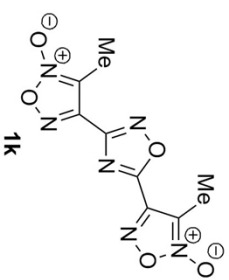
/USED 777.C13



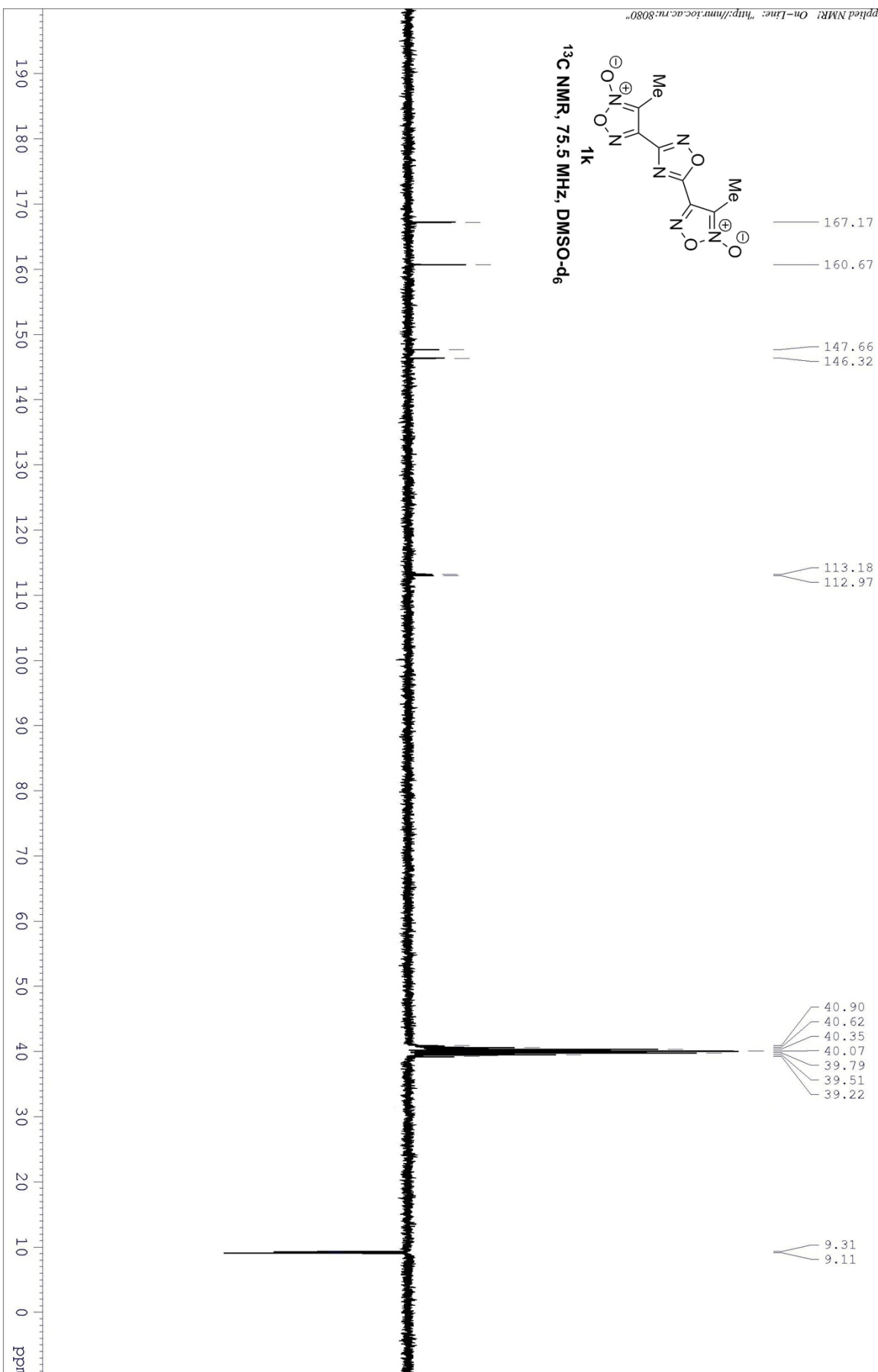
¹³C NMR, 50 MHz, DMSO-d₆



USED 777



^{13}C NMR, 75.5 MHz, DMSO- d_6



Display Report

Analysis Info

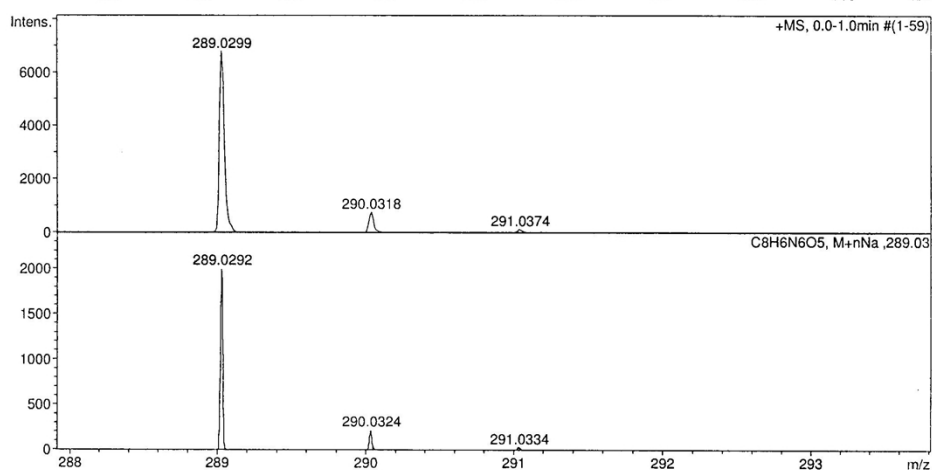
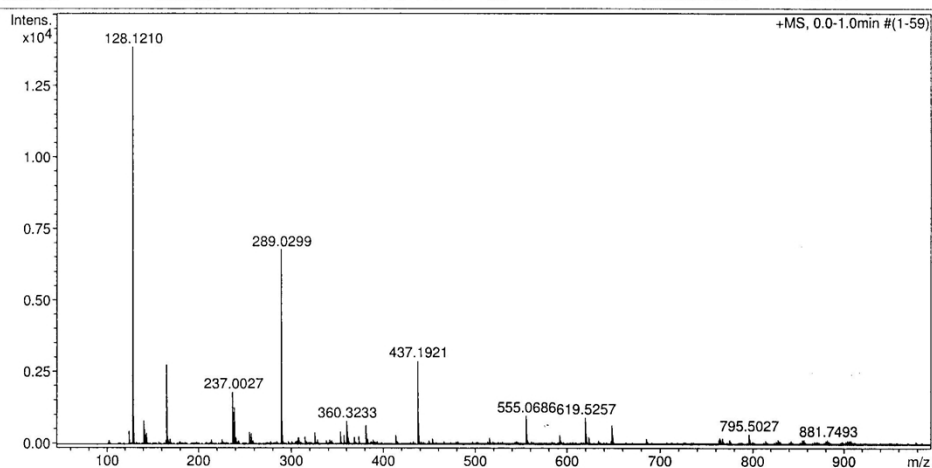
Analysis Name D:\Data\Chizhov\Makhova\Fershtat\777_low.d
Method tune_low.m
Sample Name /USED 777
Comment CH3CN 100 %, dil. 20, no calibrant added

Acquisition Date 27.02.2015 12:54:06

Operator BDAL@DE
Instrument / Ser# micrOTOF 10248

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste

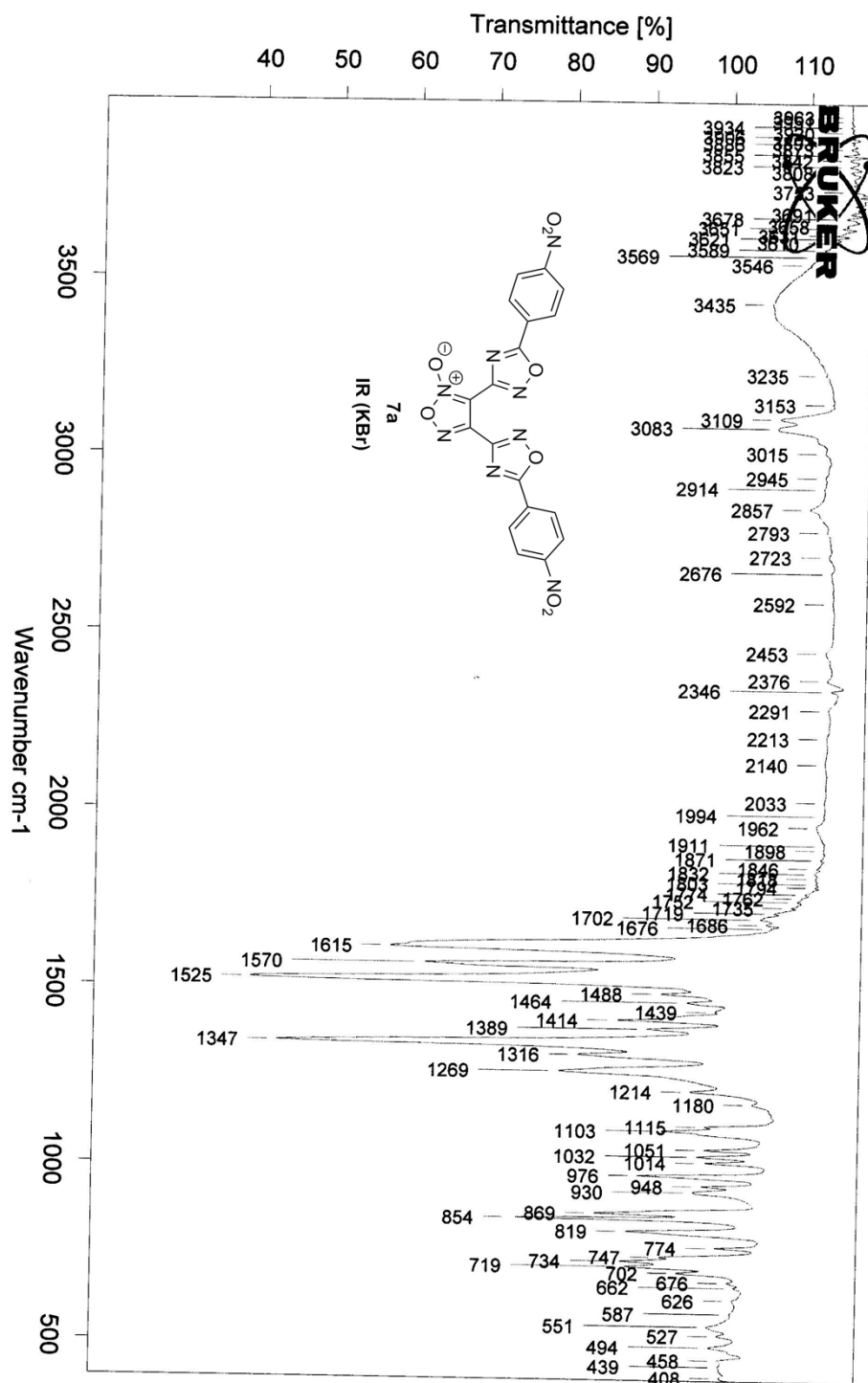


D:\EDL\LEO-752.0

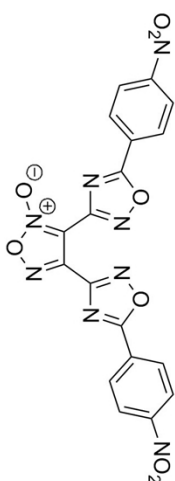
ФЕРУЛАТ, LEO-752, препарат с KBr 1/200.

17.02.2015

Page 1/1

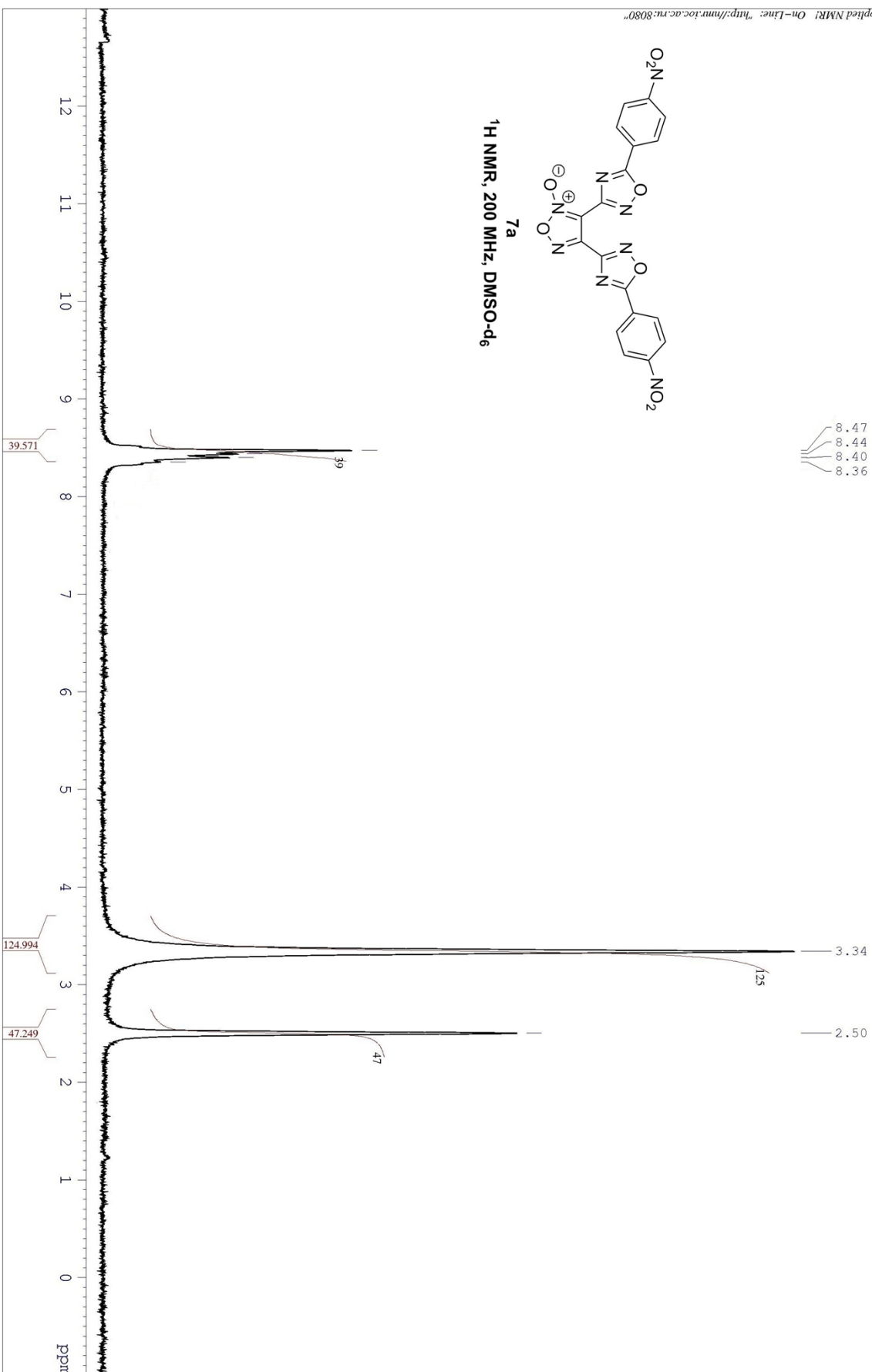


/USED 695



7a

¹H NMR, 200 MHz, DMSO-d₆



Display Report

Analysis Info

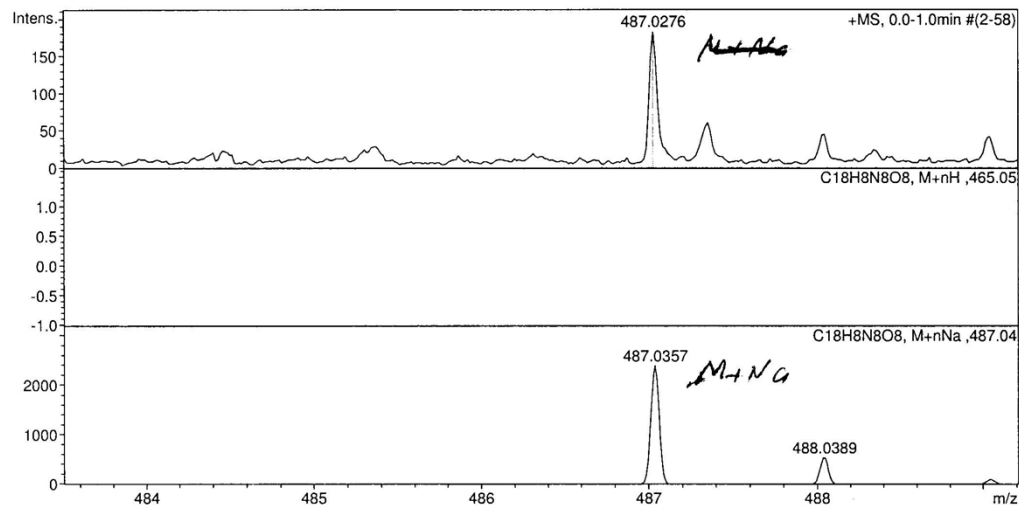
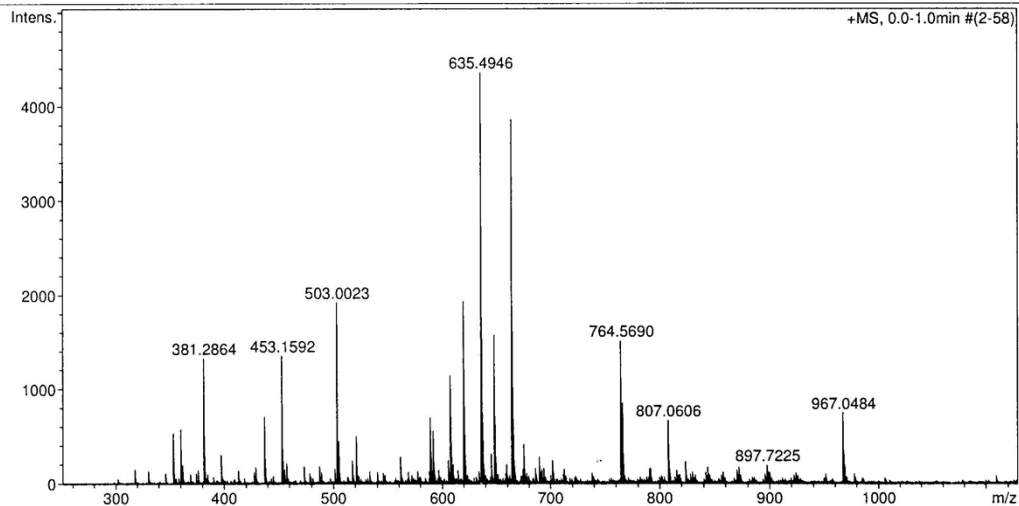
Analysis Name D:\Data\Kadentsev\2014\Fershtat\21-10-14-004.d
Method tune_wide.m
Sample Name /USED 695
Comment MeCN 100 %, Mw464 not calibrant added

Acquisition Date 21.10.2014 14:50:38

Operator BDAL@DE
Instrument / Ser# micrOTOF 10248

Acquisition Parameter

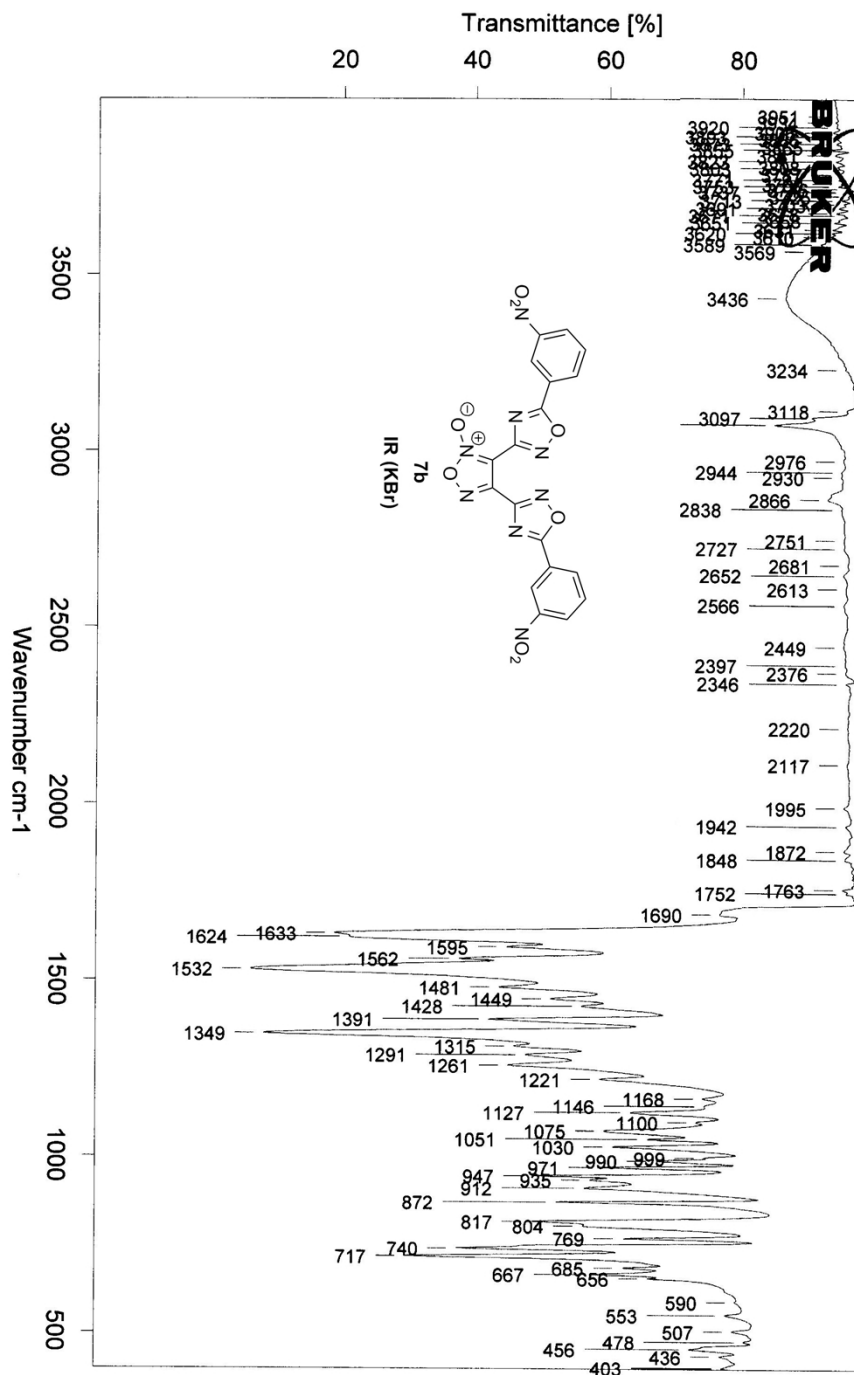
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



D:\EDL\735.0 ФЕРИТАТ. 735 , прессовка с KBr. 1/200.

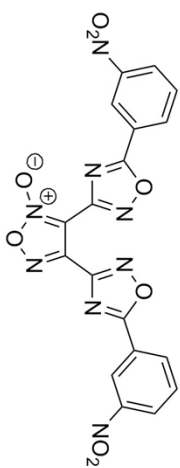
24.02.2015

Page 1/1

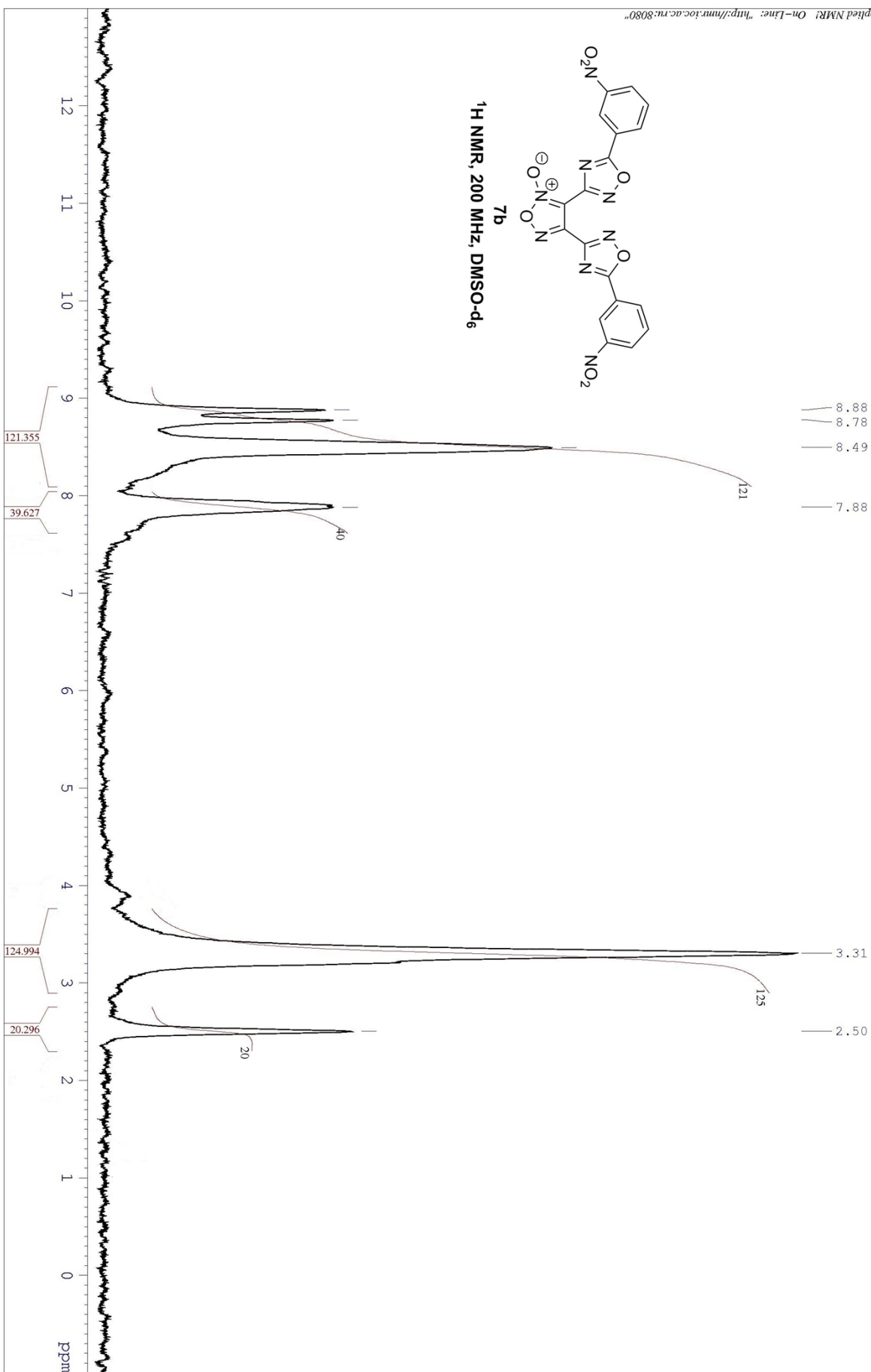


/USED 735

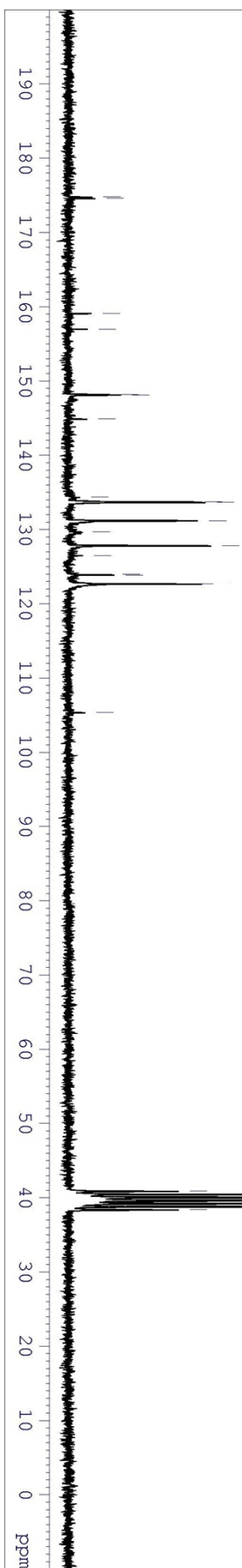
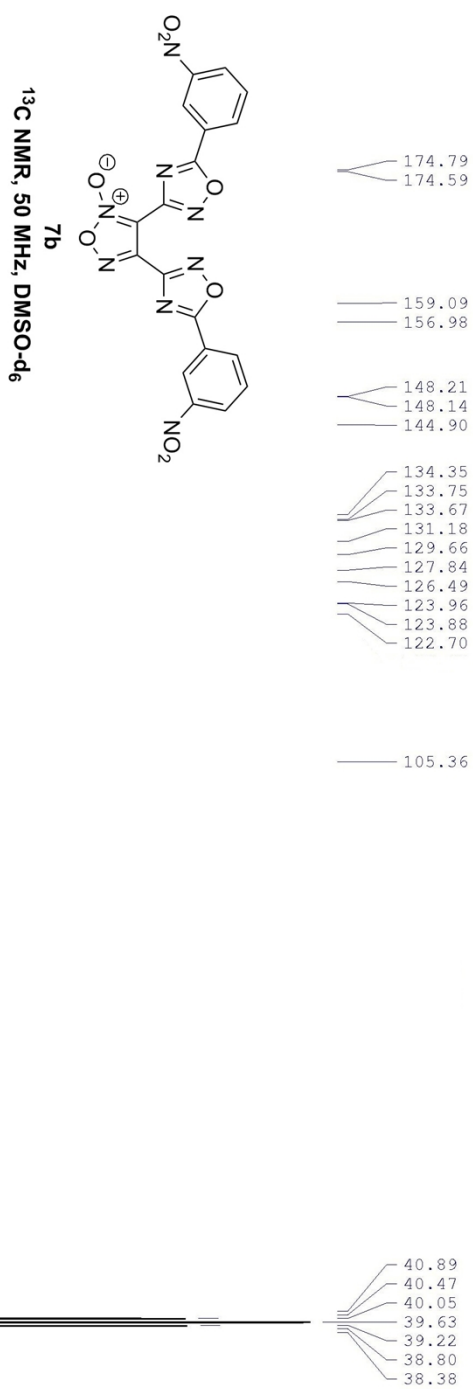
© Zelinsky Institute of Organic Chemistry, Moscow; Broker AC200 SF=200.13 MHz [1H] SI=16K SW=4000 OI=4888 PW=6.0 AQ=2.048 RD=3.00 NS=12 SR=3287.07 TF=297K 25 November 2014 Opr: Smutkova M.I.; Solv: DMSO-d6;



¹H NMR, 200 MHz, DMSO-d₆



/USED 735.C13



Display Report

Analysis Info

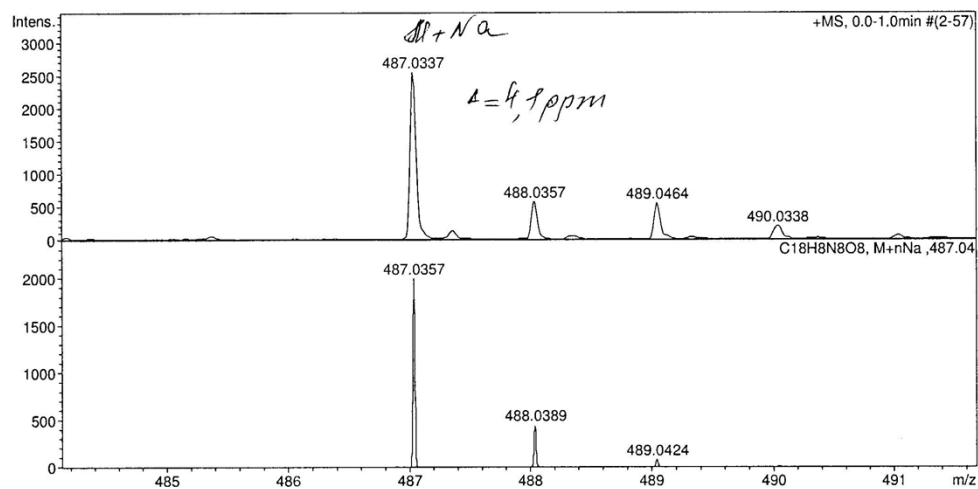
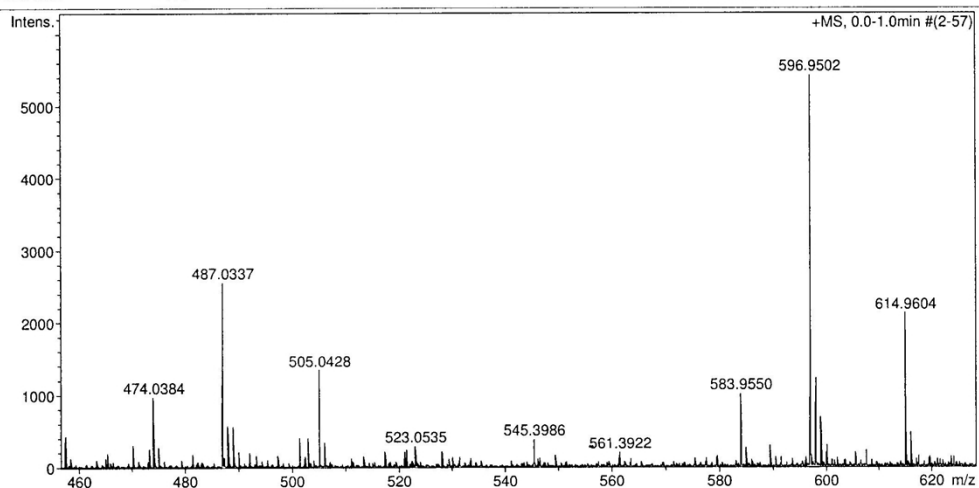
Analysis Name D:\Data\Kolotyrkina\2014\Fershtat\1211004.d
Method tune_wide.m
Sample Name /USED 735
Comment C18H8N8O8 mw 464 calibrant added

Acquisition Date 11.12.2014 12:07:02

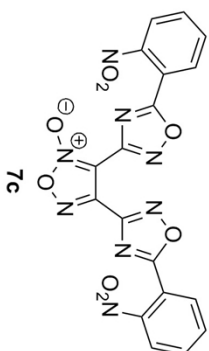
Operator BDAL@DE
Instrument / Ser# micrOTOF 10248

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



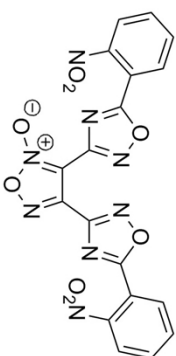
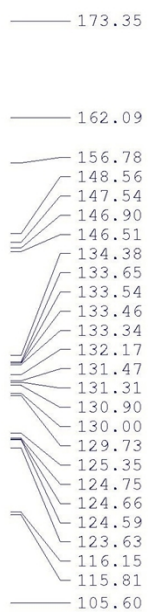
/USED 757



7c
¹H NMR, 300 MHz, DMSO-d₆

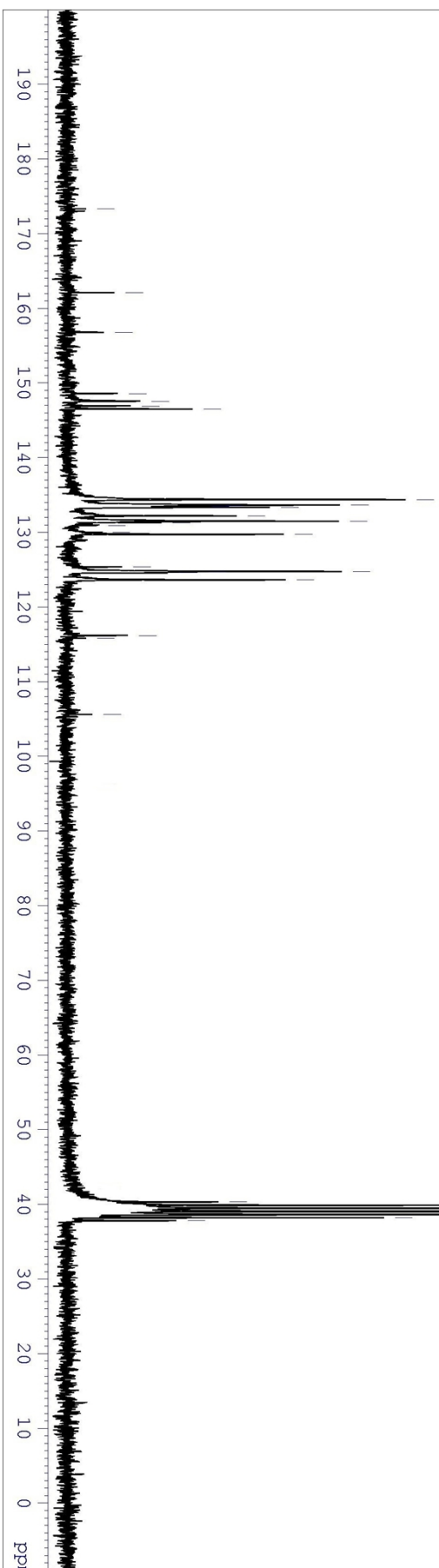
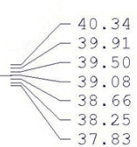


/USED 757.C13



7c

¹³C NMR, 50 MHz, DMSO-d₆

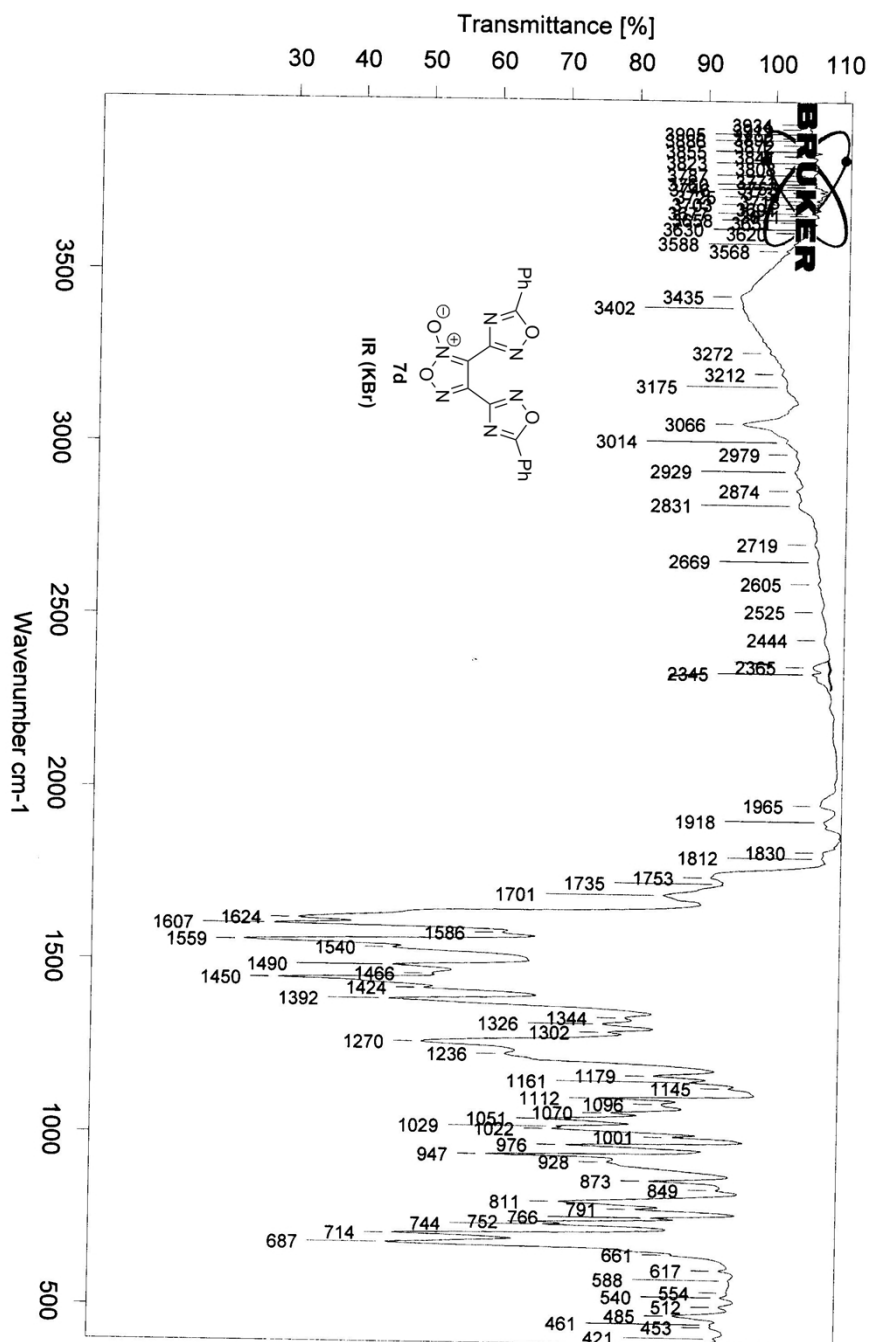


D:\EDULEO-742.0

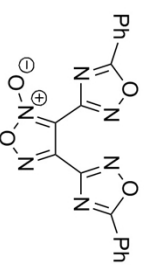
ФЕПУТАТ. LEO-742, , прекобра с KBr, 1/200.

27.02.2015

Page 1/1

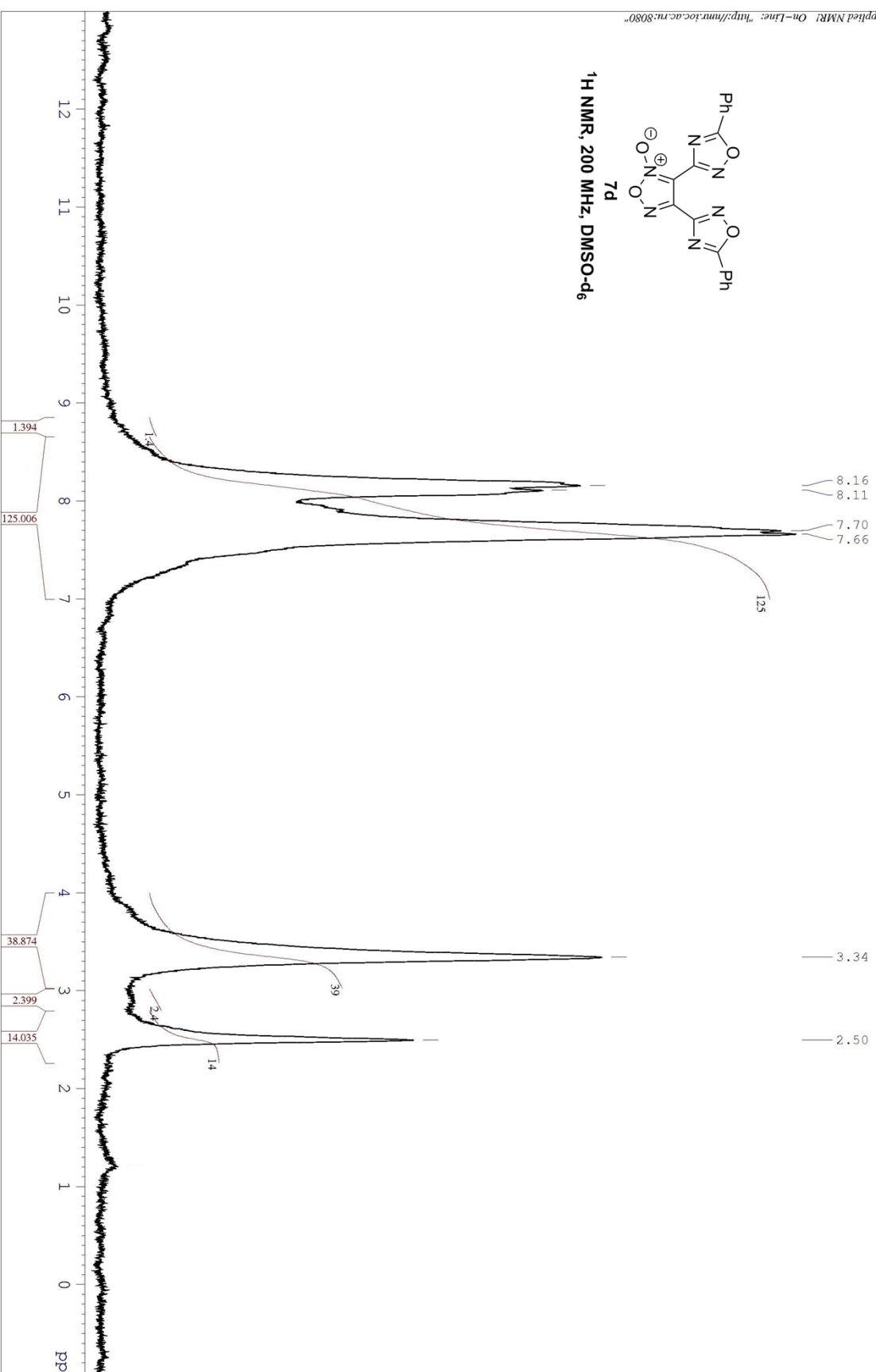


/USED 742



7d

¹H NMR, 200 MHz, DMSO-d₆



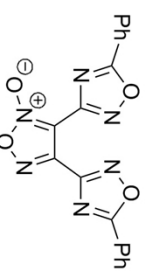
/USED 742

179.11
176.53

158.99
156.93

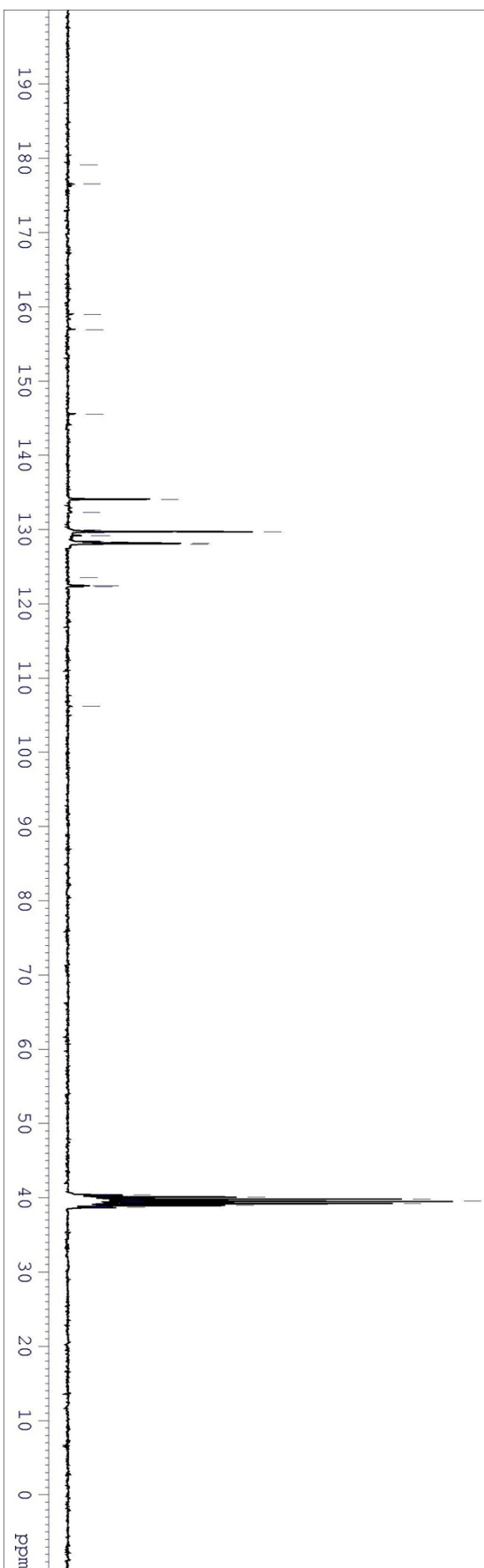
145.55
134.06
132.32
129.95
129.81
129.70
129.55
129.19
129.15
128.41
128.32
128.15
128.03
123.51
122.45
122.42
122.31

40.45
40.39
40.29
40.11
39.83
39.73
39.55
39.45
39.28
39.00
38.86
38.81
38.72



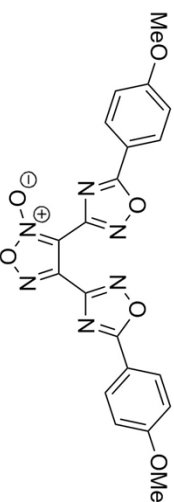
7d

¹³C NMR, 75.5 MHz, DMSO-d₆

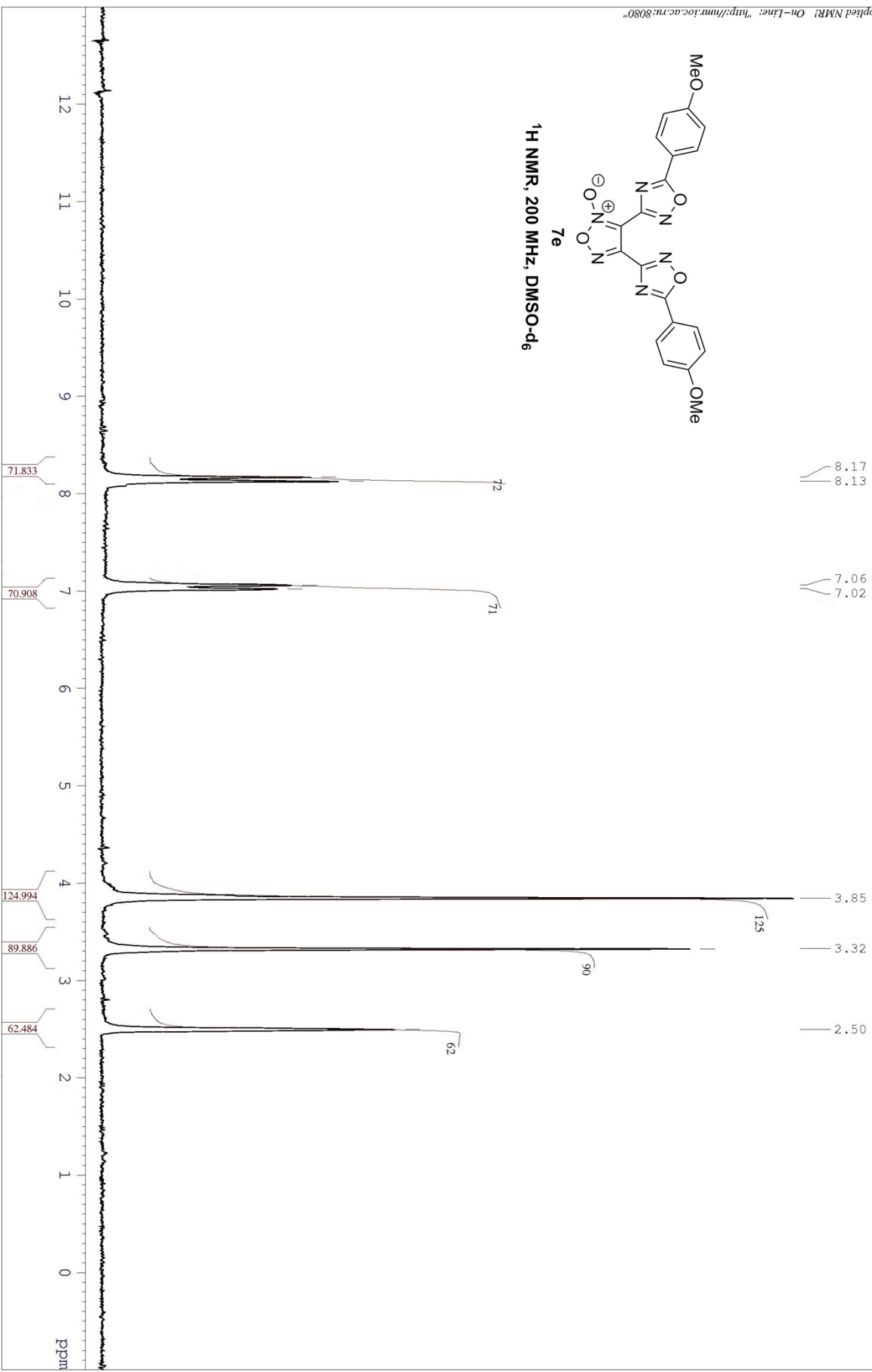


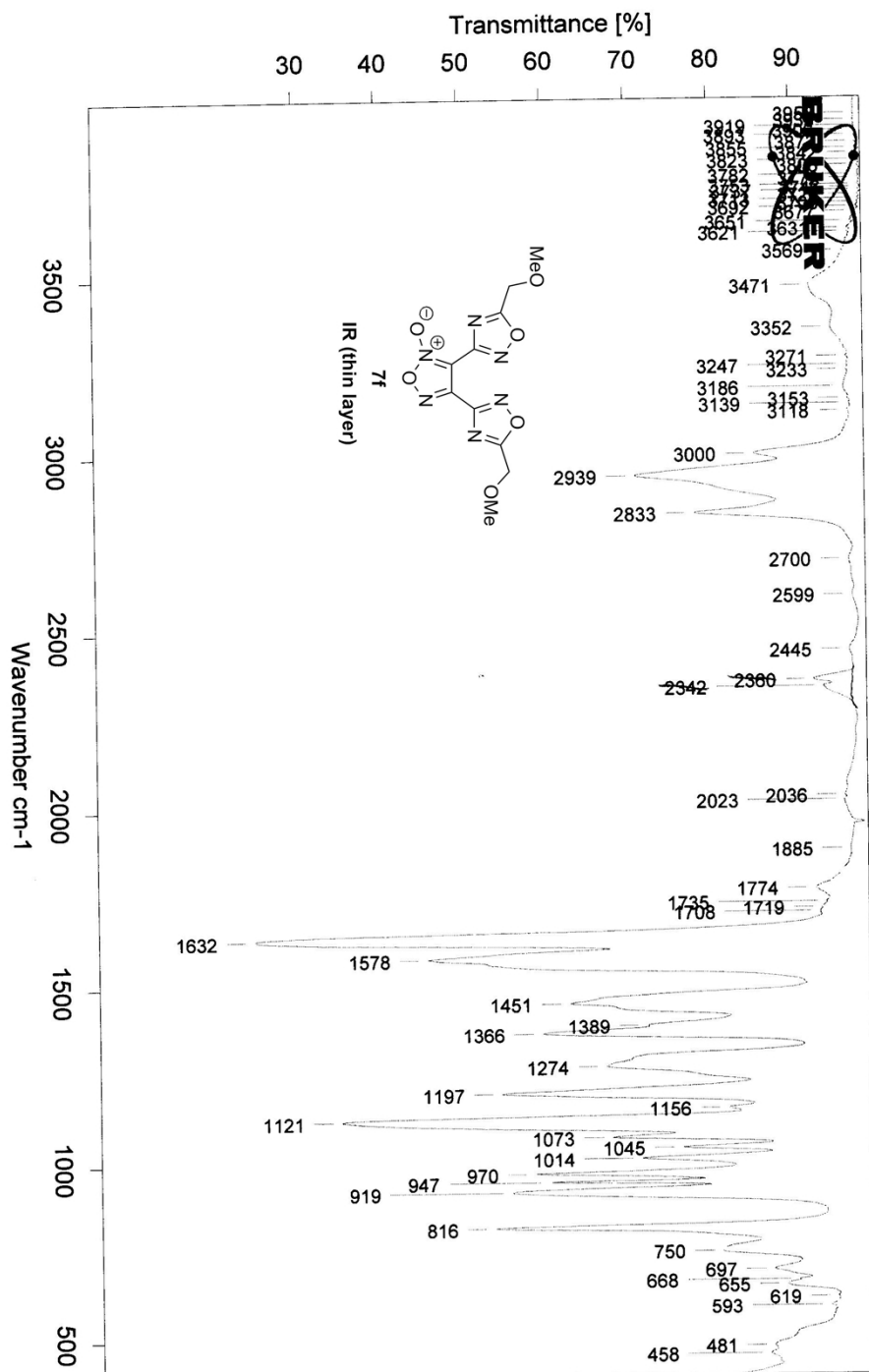
/USED 789

© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AC200 SF=200.13 MHz [1H] SI=16K SW=4000 OI=4888 PW=6.0 AQ=2.048 RD=3.00 NS=12 SR=3289.08 TF=297K 11 February 2015 Op: Struchkova M.I.; Solv: DMSO-d₆.



¹H NMR, 200 MHz, DMSO-d₆





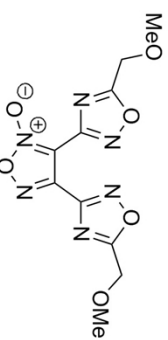
D:\EDL\LEO-746.0

ФЕРУЛАТ. LEO-746, тонкий слой, пл. KBr.

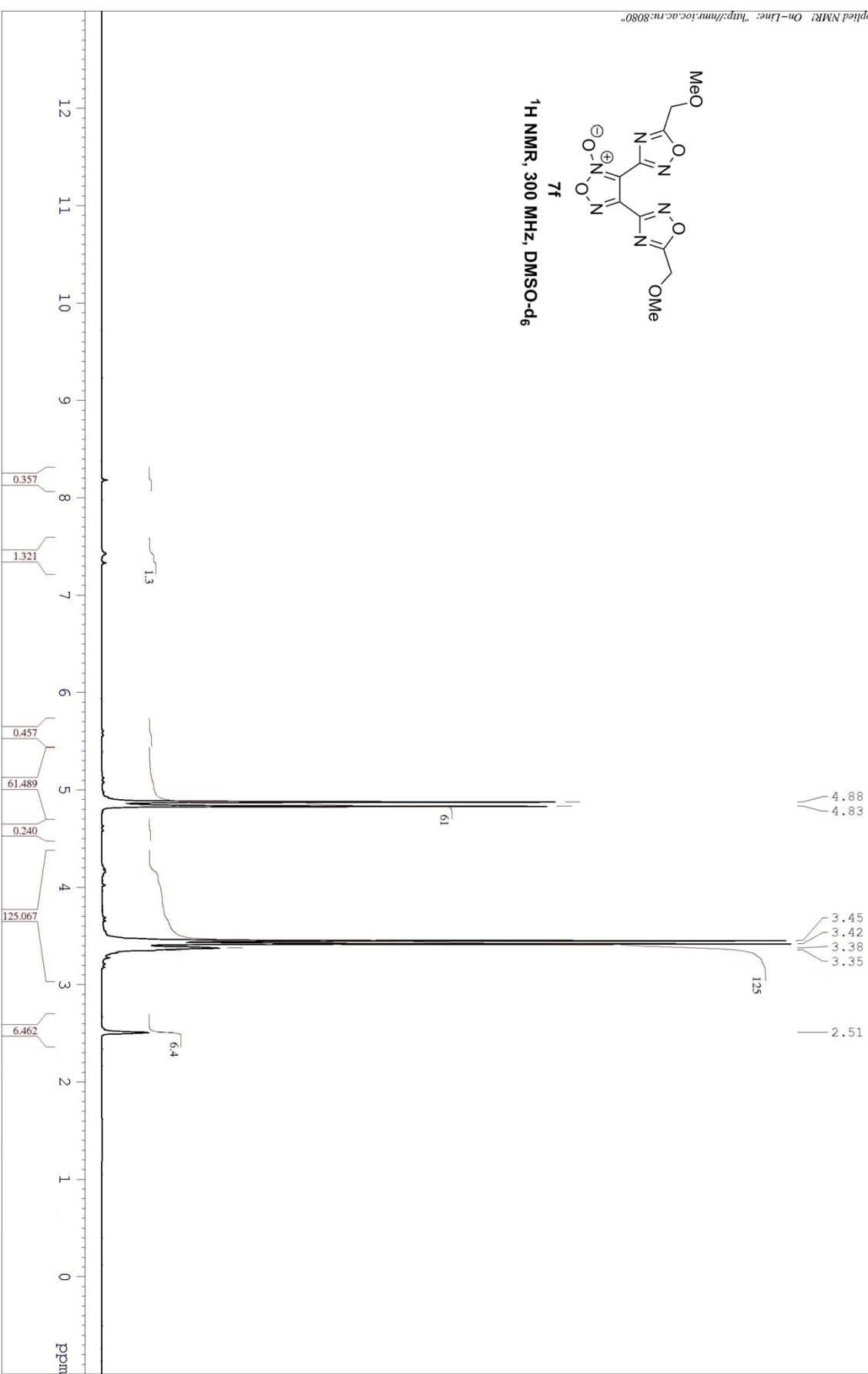
03.03.2015

/USED 746

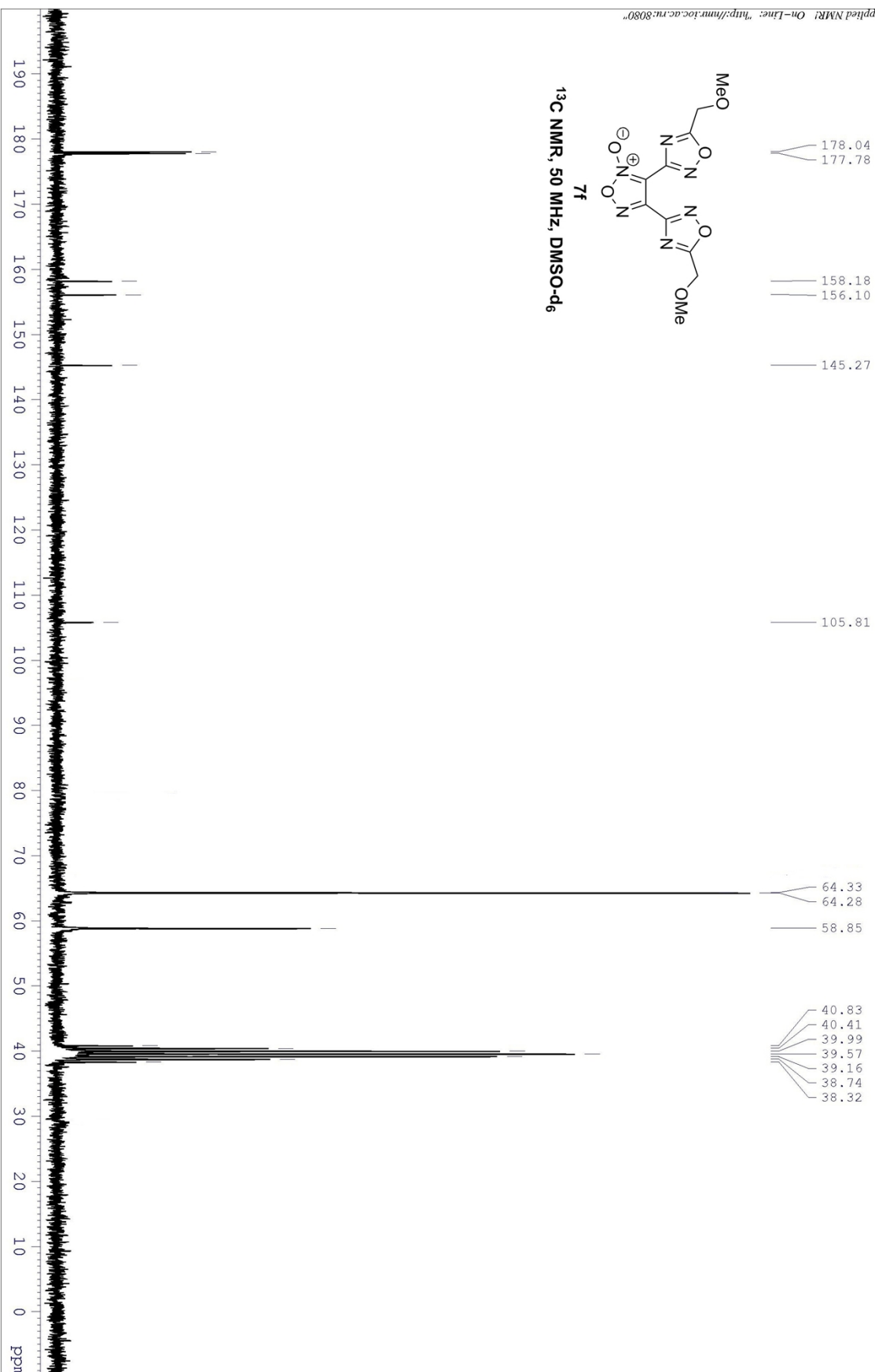
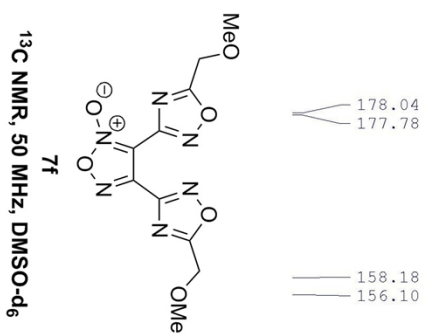
© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AMX300 SF=300.13 MHz [1H] SF=16K SW=6010 O1=2401 PW=9.0 AQ=1.351 RD=3.00 NS=1 SR=-4.17 TE=300K 11 December 2014 Opr: Stuehlova M.L.; Solv: DMSO-d6;
 The Best Applied NMR! On-Line: "http://nmr.ioc.ac.ru:8080"



7f
¹H NMR, 300 MHz, DMSO-d₆



/USED 746.C13



Display Report

Analysis Info

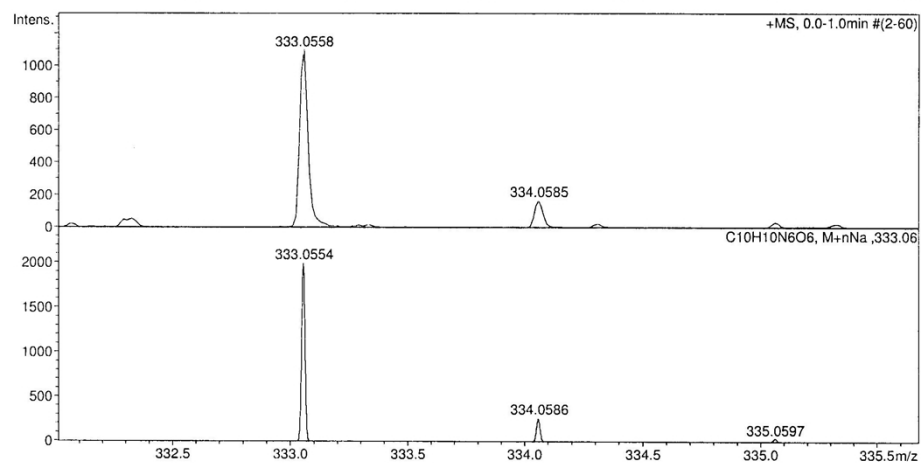
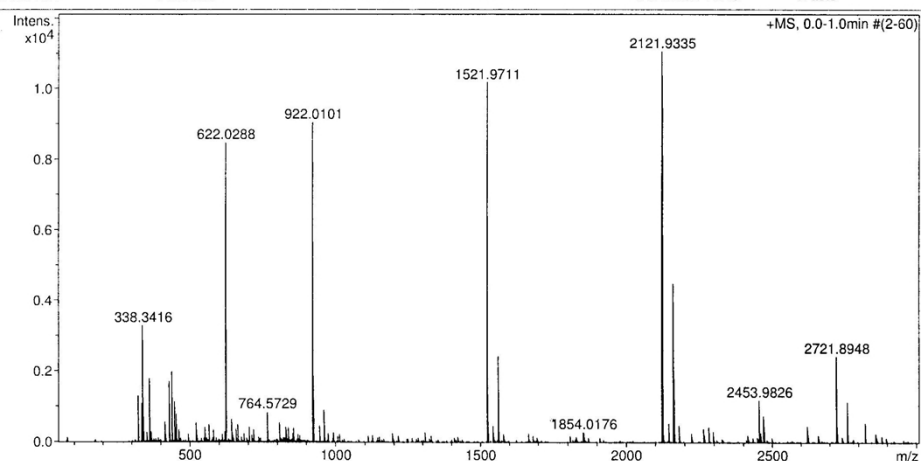
Analysis Name D:\Data\Chizhov\Makhova\Fershtat\746_&clb.d
Method tune_wide.m
Sample Name /USED 746
Comment CH3CN 100 %, dil. 200, calibrant added

Acquisition Date 02.04.2015 14:52:02

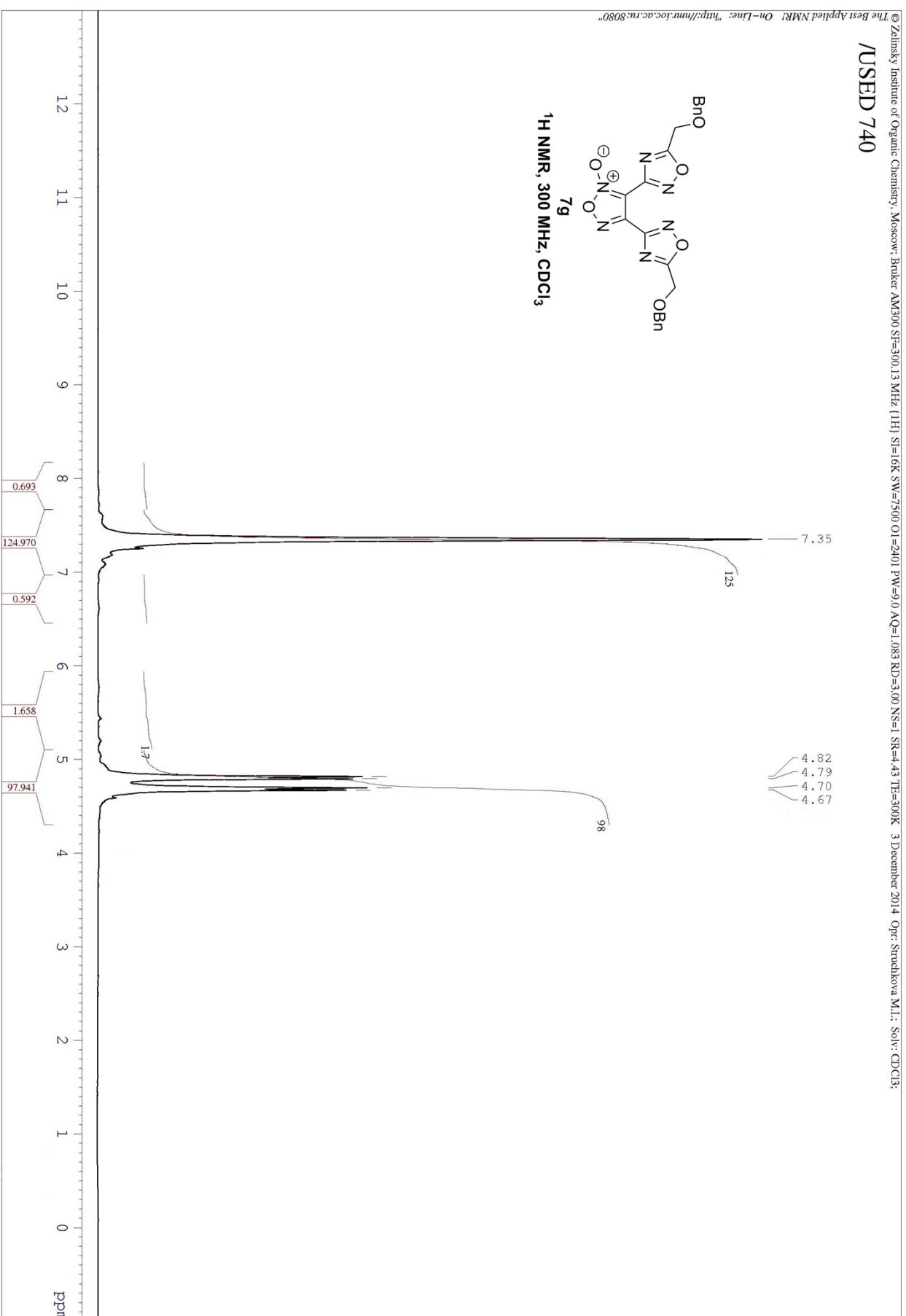
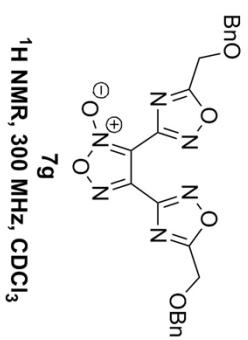
Operator BDAL@DE
Instrument / Ser# micrOTOF 10248

Acquisition Parameter

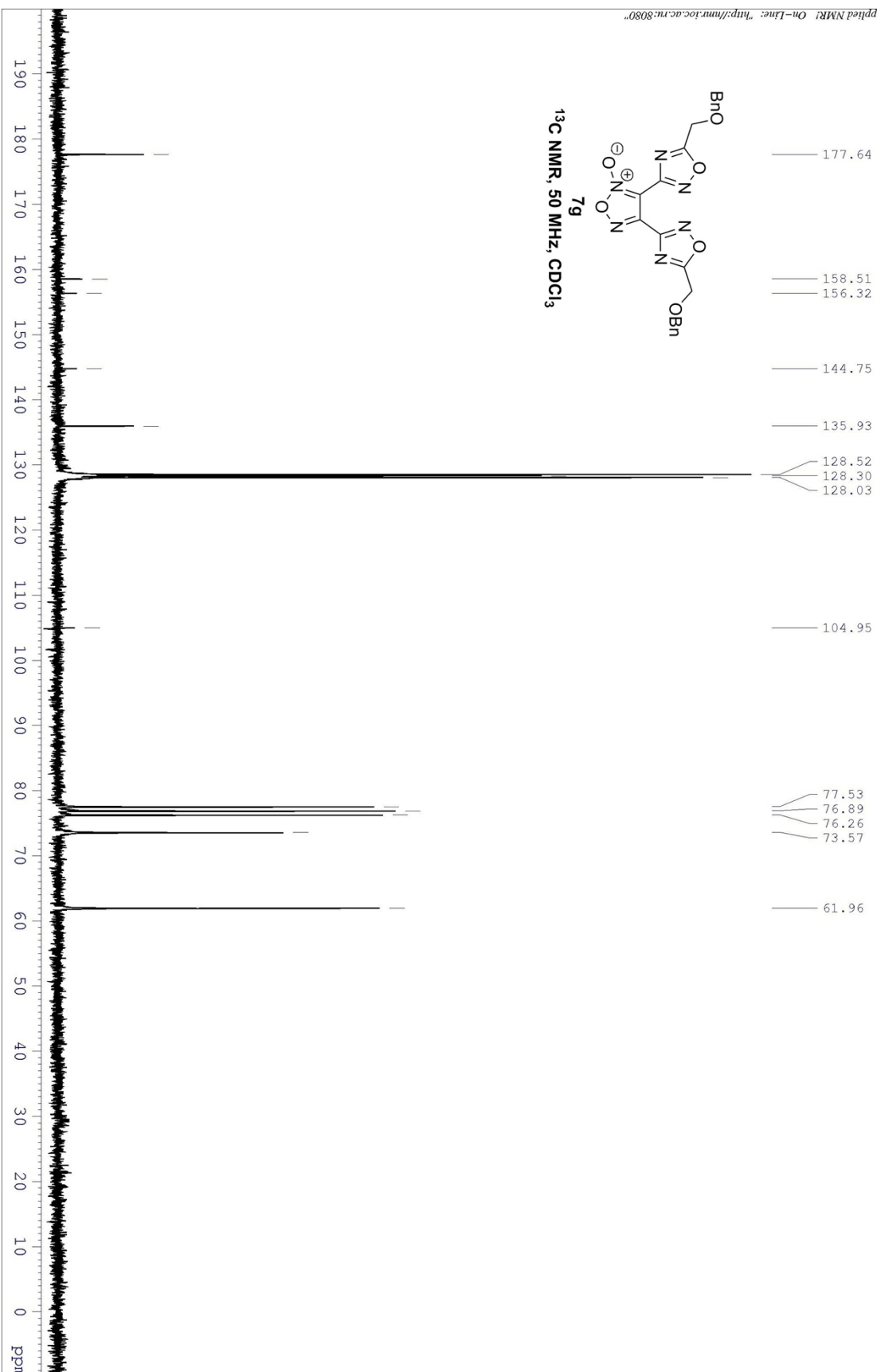
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



/USED 740



/USED 740.C13



Display Report

Analysis Info

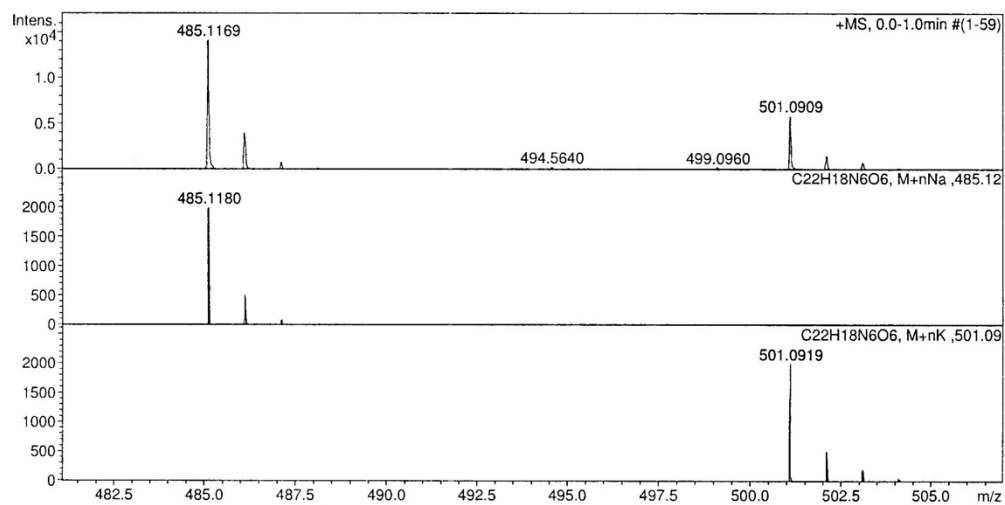
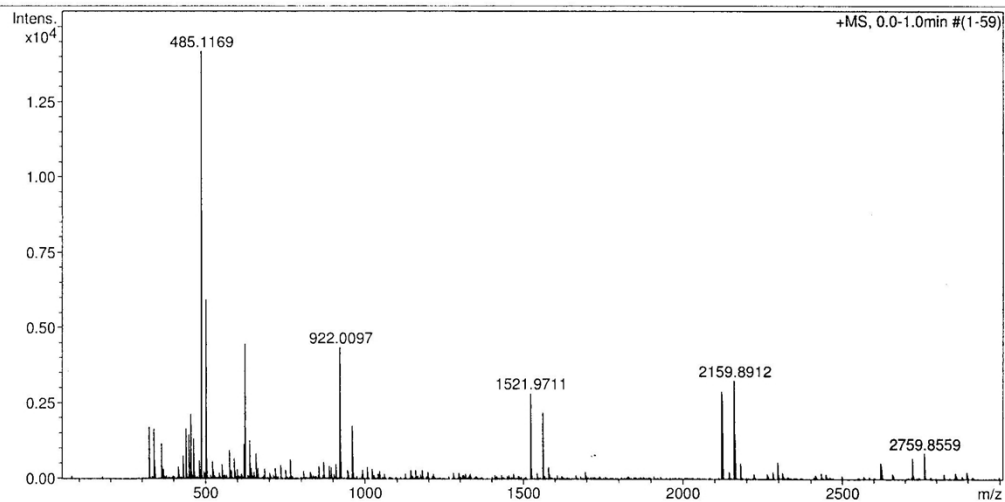
Analysis Name D:\Data\Chizhov\Makhova\Fershtat\740_&clb.d
Method tune_wide.m
Sample Name /USED 740
Comment CH3CN 100 %, dil. 200, calibrant added

Acquisition Date 02.04.2015 14:46:31

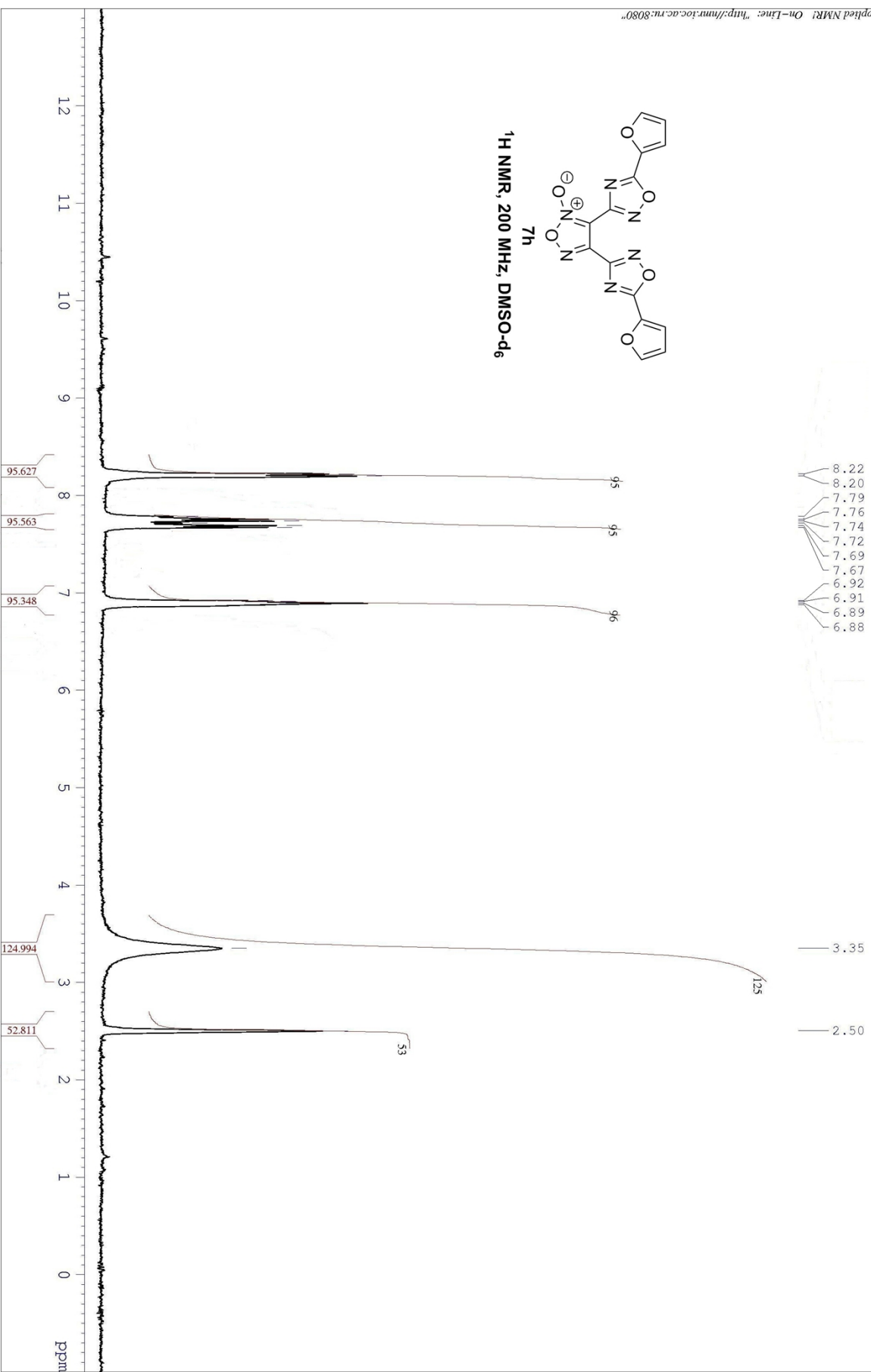
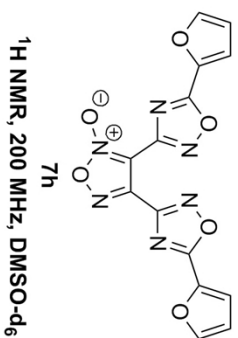
Operator BDAL@DE
Instrument / Ser# micrOTOF 10248

Acquisition Parameter

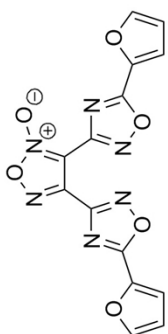
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Not active			Set Dry Heater	180 °C
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste



/USED 754

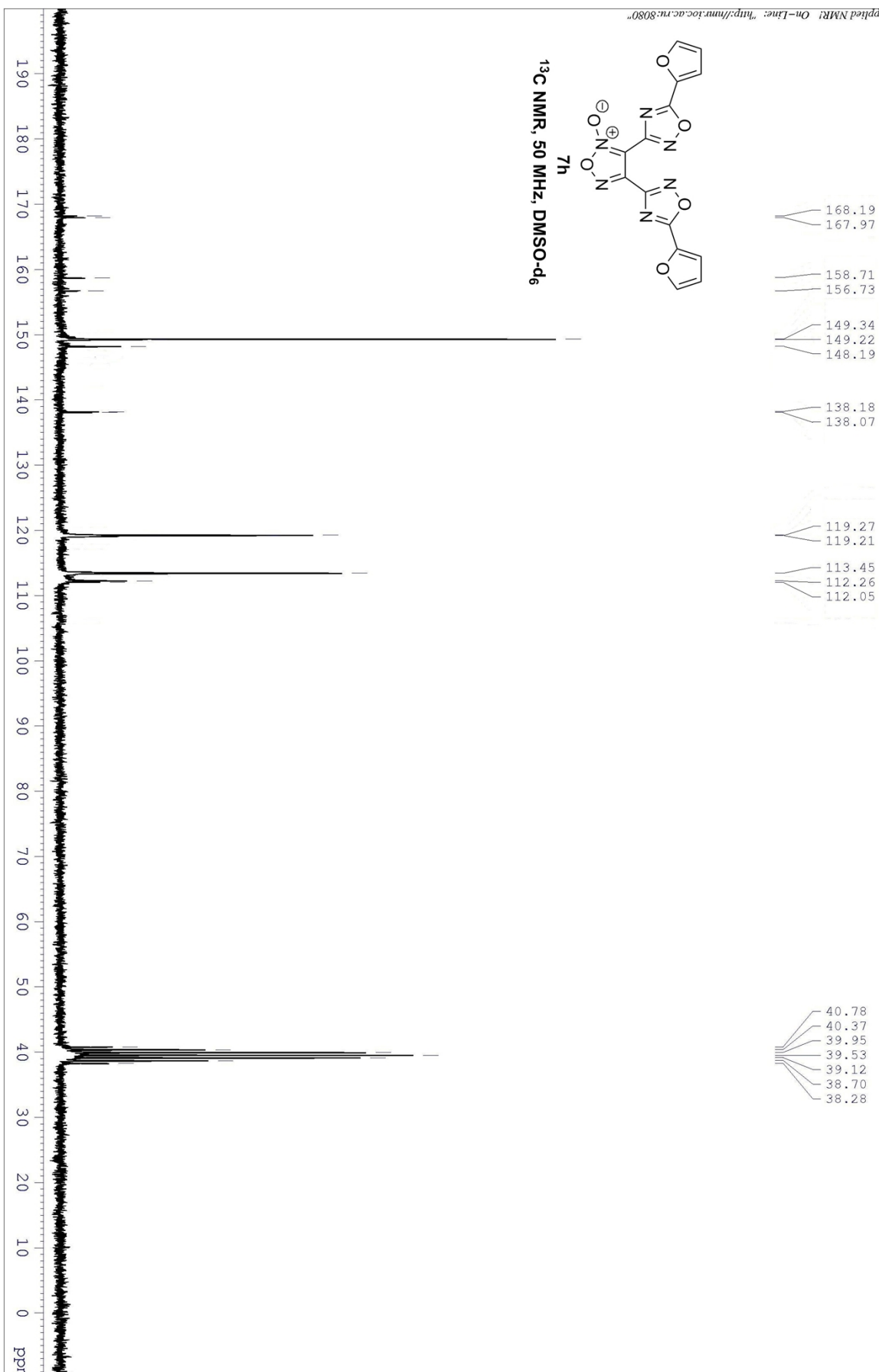


/USED 754.C13

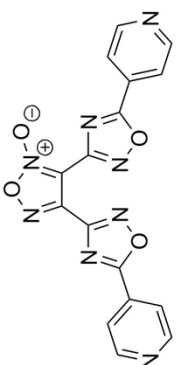


7h

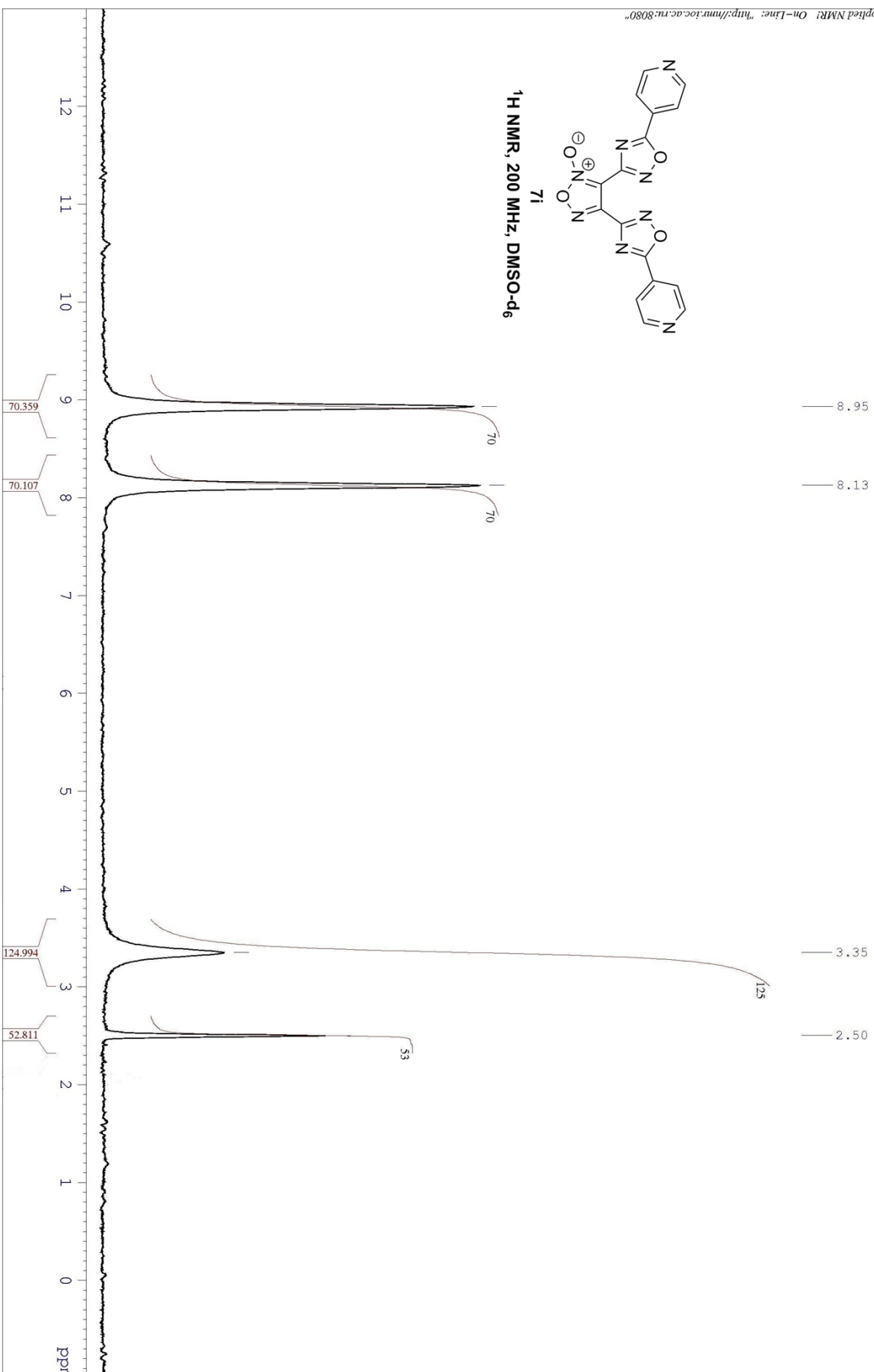
¹³C NMR, 50 MHz, DMSO-d₆



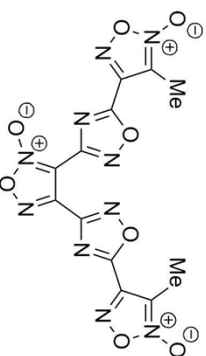
/USED 755



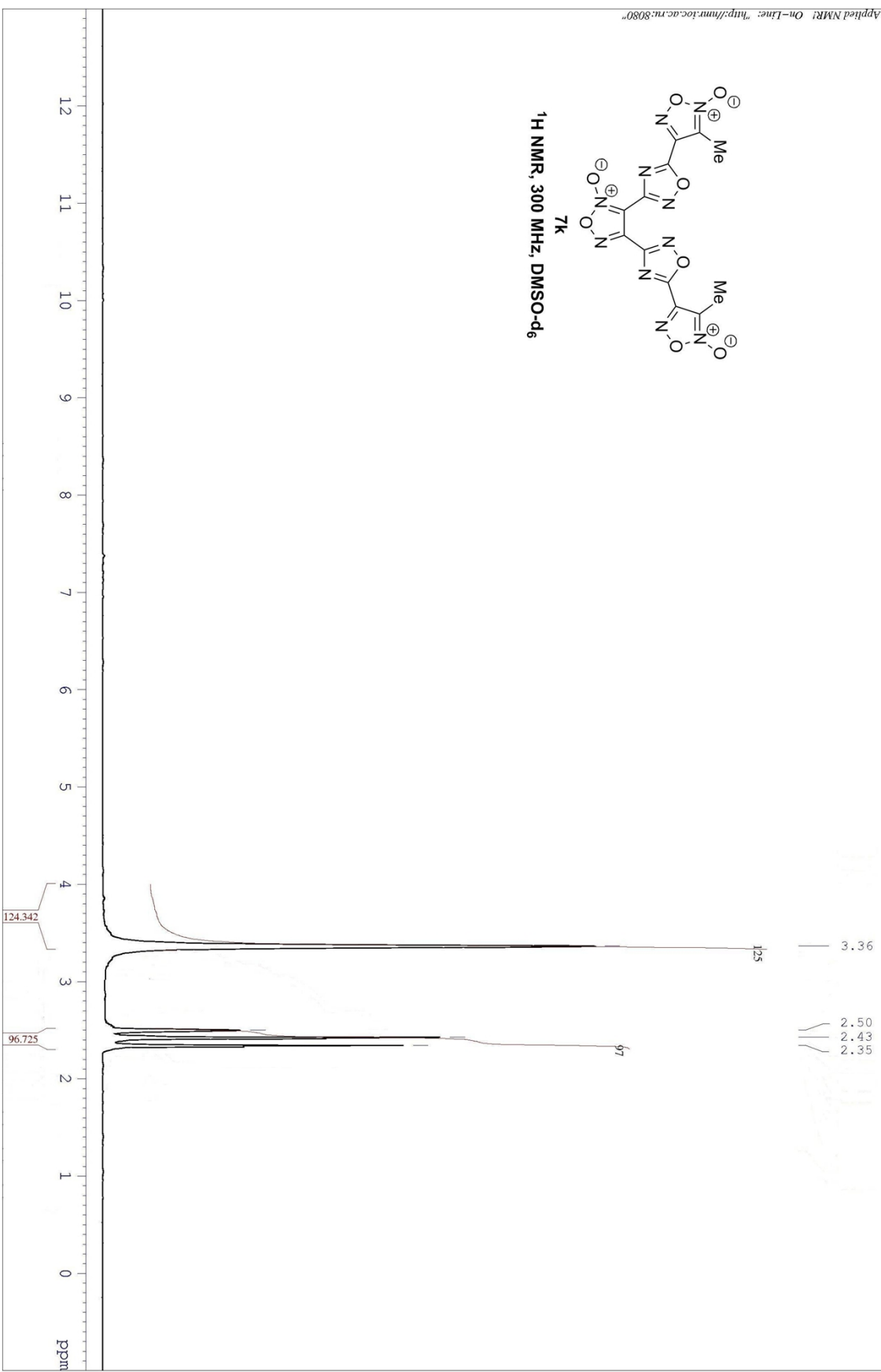
¹H NMR, 200 MHz, DMSO-d₆



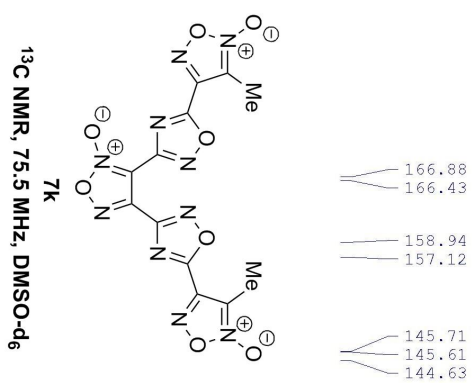
/USED 795



7k
¹H NMR, 300 MHz, DMSO-d₆



/USED 795.c13



166.88
166.43

158.94
157.12

145.71
145.61
144.63

112.40
112.37
109.25

40.30
40.02
39.74
39.61
39.47
39.19
39.05
38.91
38.78
38.63
38.17

8.48
8.32

