Chemical Communications

Electronic Supporting Information

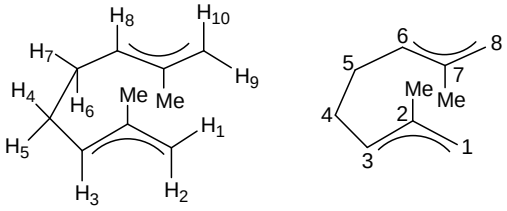
An efficient ruthenium(IV) catalyst for the selective hydration of nitriles to amides in water under mild conditions

Eder Tomás-Mendivil, Francisco J. Suárez, Josefina Díez and Victorio Cadierno*

Laboratorio de Compuestos Organometálicos y Catálisis (Unidad Asociada al CSIC), Red ORFEO-CINQA - Centro de Innovación en Química Avanzada, Departamento de Química Orgánica e Inorgánica, IUQOEM, Universidad de Oviedo, 33006 Oviedo, Spain. Fax: +34985103446; Tel: +34985103453; E-mail: vcm@uniovi.es.

EXPERIMENTAL SECTION

General methods: Synthetic procedures were performed under an atmosphere of dry nitrogen using vacuum-line and standard Schlenk techniques. Organic solvents were dried by standard methods and distilled under nitrogen before use. All reagents were obtained from commercial suppliers and used without any further purification, with the exception of complex [{RuCl(μ-Cl)(η^3:η^3-$C_{10}H_{16}$)}$_2$] (**1**),[1] the secondary phosphine oxides $R_2P(=O)H$ (R = Ph, Me)[2] and 4-cyano-1-(2,6-difluorobenzyl)-1*H*-1,2,3-triazole (**3aa**),[3] which were prepared by following the methods reported in the literature. Infrared spectra were recorded on a Perkin-Elmer 1720-XFT spectrometer. GC measurements were made on a Hewlett-Packard HP6890 equipment using a Supelco Beta-Dex^{TM} 120 column (30 m length; 250 μm diameter). NMR spectra were recorded on a Bruker DPX-300 or AV400 instruments. The chemical shift values (δ) are given in parts per million and are referred to the residual peak of the deuterated solvent employed (^{1}H and ^{13}C) or an external 85% aqueous H_3PO_4 solution (^{31}P). DEPT experiments have been carried out for all the compounds reported. Elemental analyses were provided by the Analytical Service of the Instituto de Investigaciones Químicas (IIQ-CSIC) of Seville. The numbering for protons and carbons of the 2,7-dimethylocta-2,6-diene-1,8-diyl skeleton is as follows:



Synthesis of [RuCl$_2$(η^3:η^3-$C_{10}H_{16}$){PPh$_2$(OH)}] (2a): A solution of the dimeric precursor [{RuCl(μ-Cl)(η^3:η^3-$C_{10}H_{16}$)}$_2$] (**1**)

(0.200 g, 0.32 mmol) and $Ph_2P(=O)H$ (0.131 g, 0.64 mmol) in dichloromethane (50 mL) was stirred at room temperature for 48 h. The solvent was then removed under reduced pressure. The resulting yellow solid washed with hexanes (2 x 10 mL) and diethyl ether (2 x 10 ml), and dried under vacuum. Yield: 0.269 g (82%). IR (KBr): 3035 (s), 2909 (w), 2854 (w), 1437 (s), 1380 (m), 1216 (w), 1146 (s), 1100 (m), 1027 (w), 878 (s), 742 (m), 690 (s), 609 (w), 538 (s), 489 (s) cm^{-1}. $^{31}P\{^1H\}$ NMR ($CDCl_3$) δ = 80.2 (s) ppm. ^1H NMR ($CDCl_3$) δ = 2.14 (s, 6H, Me), 2.70-2.75 (m, 2H, H_4 and H_6), 3.15 (d, 2H, $^3J_{PH}$ = 3.3 Hz, H_2 and H_{10}), 3.52-3.58 (m, 2H, H_5 and H_7), 3.85 (d, 2H, $^3J_{PH}$ = 10.4 Hz, H_1 and H_9), 5.14-5.18 (m, 2H, H_3 and H_8), 7.39-7.44 (m, 6H, Ph), 7.75 (s, 1H, OH), 8.00-8.06 (m, 2H, Ph), 8.11-8.17 (m, 2H, Ph) ppm. $^{13}C\{^1H\}$ NMR ($CDCl_3$) δ = 21.1 (s, Me), 37.2 (s, C_4 and C_5), 67.1 (s, C_1 and C_8), 107.7 (br, C_3 and C_6), 125.2 (s, C_2 and C_7), 128.0 (d, $^2J_{CP}$ = 10.5 Hz, Ph), 128.2 (d, $^2J_{CP}$ = 10.5 Hz, Ph), 129.6-129.8 (m, Ph), 139.1 (d, $^1J_{CP}$ = 54.3 Hz, Ph), 140.8 (d, $^1J_{CP}$ = 53.6 Hz, Ph) ppm. Elemental analysis calcd. (%) for $C_{22}H_{27}Cl_2OPRu$: C 51.77, H 5.33; found: C 51.59, H 5.38.

Synthesis of $[RuCl_2(\eta^3:\eta^3\text{-}C_{10}H_{16})\{PMe_2(OH)\}]$ (2b): A solution of the dimeric precursor $[\{RuCl(\mu\text{-}Cl)(\eta^3:\eta^3\text{-}C_{10}H_{16})\}_2]$ (**1**) (0.150 g, 0.24 mmol) and $Me_2P(=O)H$ (0.037 g, 0.48 mmol) in dichloromethane (50 mL) was stirred at room temperature for 48 h. The solvent was then removed under reduced pressure and the resulting oily residue dissolved in diethyl ether (30 mL) and filtered. The filtrate was evaporated to dryness and the yellow syrup formed washed with hexanes (10 mL) to afford a dark yellow solid, which was dried under vacuum. Yield: 0.156 g (84%). IR (KBr): 3188 (m), 2962 (w), 2906 (m), 2852 (w), 1457(w), 1413 (m), 1383 (m), 1283 (m), 1137 (s), 1024 (s), 948 (s), 912 (s), 849 (s), 736 (m), 699 (s), 556 (m) cm^{-1}. $^{31}P\{^1H\}$ NMR ($CDCl_3$) δ = 108.5 (s) ppm. ^1H

NMR ($CDCl_3$) δ = 1.91 (d, 3H, $^2J_{HP}$ = 9.6 Hz, PMe), 2.03 (d, 3H, $^2J_{HP}$ = 9.5 Hz, PMe), 2.24 (s, 6H, Me), 2.84-2.88 (m, 2H, H_4 and H_6), 3.06 (d, 2H, $^3J_{PH}$ = 4.8 Hz, H_2 and H_{10}), 3.42-3.47 (m, 2H, H_5 and H_7), 4.25 (d, 2H, $^3J_{PH}$ = 8.2 Hz, H_1 and H_9), 4.91-4.95 (m, 2H, H_3 and H_8) ppm; OH signal not observed. $^{13}C\{^1H\}$ NMR ($CDCl_3$) δ = 19.8 (d, $^1J_{CP}$ = 41.1 Hz, PMe), 20.6 (s, Me), 22.2 (d, $^1J_{CP}$ = 35.7 Hz, PMe), 36.5 (C_4 and C_5), 63.4 (d, $^2J_{CP}$ = 6.2 Hz, C_1 and C_8), 108.2 (d, $^2J_{CP}$ = 9.7 Hz, C_3 and C_6), 121.0 (s, C_2 and C_7) ppm. Elemental analysis calcd. (%) for $C_{12}H_{23}Cl_2OPRu$: C 37.31, H 6.00; found: C 37.17, H 6.38.

General procedure for the catalytic hydration of nitriles with complex [$RuCl_2(\eta^3:\eta^3\text{-}C_{10}H_{16})\{PMe_2(OH)\}$] (2b): Under a nitrogen atmosphere, the corresponding nitrile **3a-ac** (1 mmol), water (3 mL), and the ruthenium complex **2b** (0.0038 g, 0.01 mmol; except for **3h** and **3ab,ac** which required 0.03 and 0.05 mmol of **2b**, respectively) were introduced into a Teflon-capped sealed tube, and the reaction mixture stirred at 60 °C for the indicated time (see Table 2 and Schemes 2-3). The course of the reaction was monitored regularly taking samples of *ca.* 20 μL, which, after extraction with CH_2Cl_2 (3 mL), were analyzed by GC (in the case of **3ab,ac** the reactions were monitored by ^1H NMR spectroscopy). Once the reaction is completed, two different procedures were applied to isolate the primary amide product depending on its solubility. For amides which partially precipitate in the medium, the reaction mixture was cooled at r.t., or 0 ºC in an ice bath, until complete precipitation/crystallization of the product. The suspension was then filtered, the solid washed with water (2 x 3 mL) and hexanes (3 mL) and dried in vacuo. For those amides that do not precipitate, isolation was carried out as follows: After elimination of the water solvent under

reduced pressure, the crude reaction mixture was pulverized in diethyl ether and the resulting solid filtered, washed and dried in vacuo. In some cases additional purification by column chromatography was required (silica gel with CH_2Cl_2 as the eluent). The identity of the amide products was assessed by comparison of their NMR spectroscopic data with those reported in the literature (copies of the NMR spectra are included below).

Recycling of complex [$RuCl_2(\eta^3:\eta^3\text{-}C_{10}H_{16})\{PMe_2(OH)\}$] (2b): Under a nitrogen atmosphere, pentafluorobenzonitrile **3b** (0.193 g, 1 mmol), water (3 mL), and the ruthenium complex **2b** (0.0038 g, 0.01 mmol) were introduced into a Teflon-capped sealed tube, and the reaction mixture was stirred at 60 °C for 30 min. After this time, a sample of *ca.* 20 μL was taken and, after extraction with CH_2Cl_2 (3 mL), analyzed by GC to determine the conversion of **3b** into **4b**. The reaction mixture was then cooled at 0 ºC in an ice bath until the complete crystallization of **4b**. The suspension was filtered and pentafluorobenzonitrile **3b** (0.193 g, 1 mmol) added to the aqueous filtrate containing **2b**. The resulting mixture was introduced into a Teflon-capped sealed tube, and stirred at 60 °C for 30 min. The same procedure was repeated for 10 times.

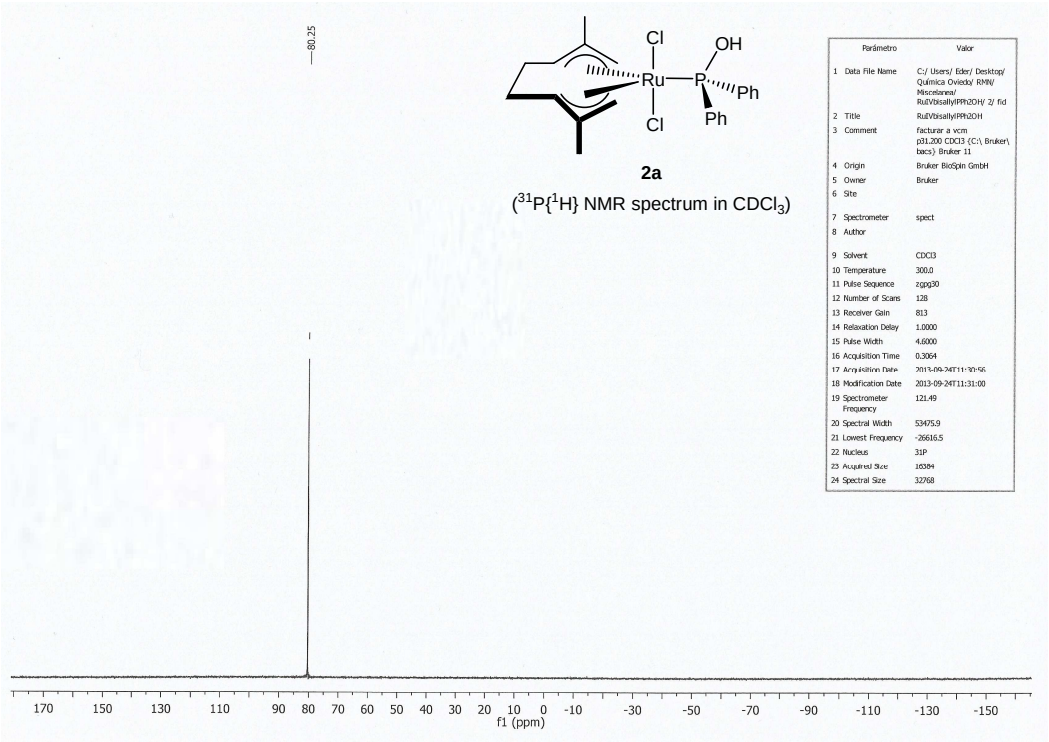
References

1 (*a*) L. Porri, M. C. Gallazzi, A. Colombo and G. Allegra, *Tetrahedron Lett.*, 1965, **47**, 4187; (*b*) A. Salzer, A. Bauer, S. Geyser and F. Podewils, *Inorg. Synth.*, 2004, **34**, 59.

2 (*a*) A. Camp, C. P. Healy, I. D. Jenkins, B. W. Skelton and A. H. White, *J. Chem. Soc., Perkin Trans. 1*, 1991, 1323; (*b*) H. R. Hays, *J. Org. Chem.*, 1968, **33**, 3690.

3 R. Portmann, *PCT Int. Appl.* WO 98/02423, 1998.

COPIES OF THE NMR SPECTRA OF COMPLEXES 2a,b AND AMIDES 4a-ac



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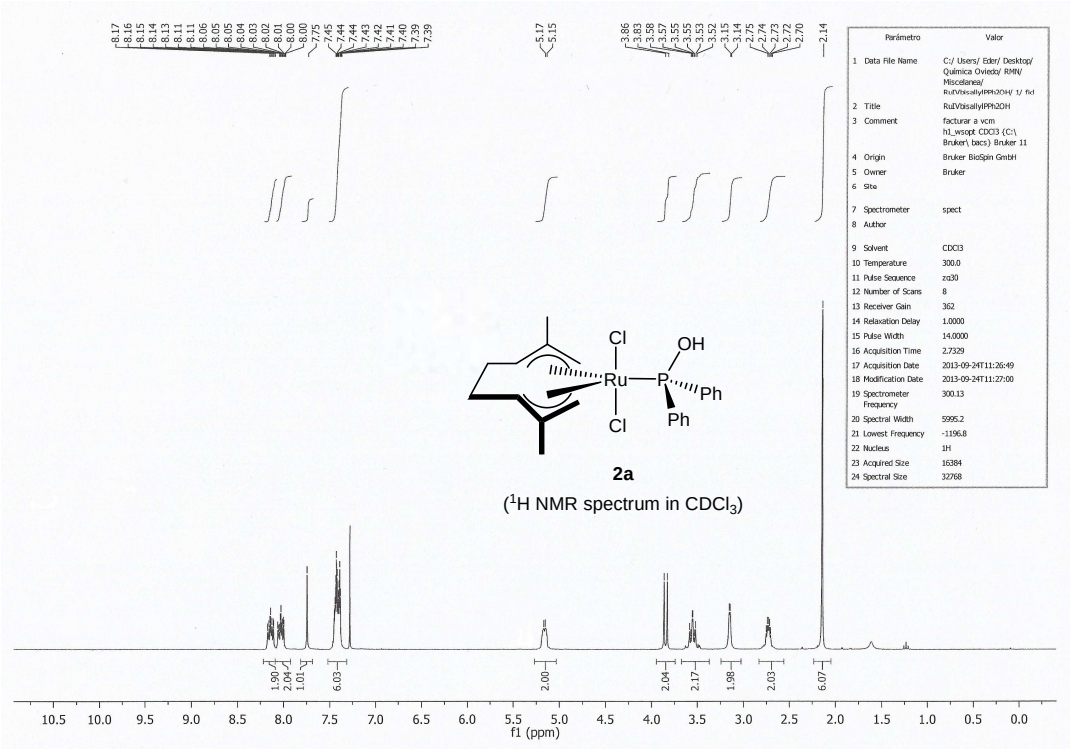
22	Nucleus	31P
23	Acquired Size	16384
24	Spectral Size	32768



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6	Site	
7	Spectrometer	spect
8	Author	
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15	Pulse Width	14.0000
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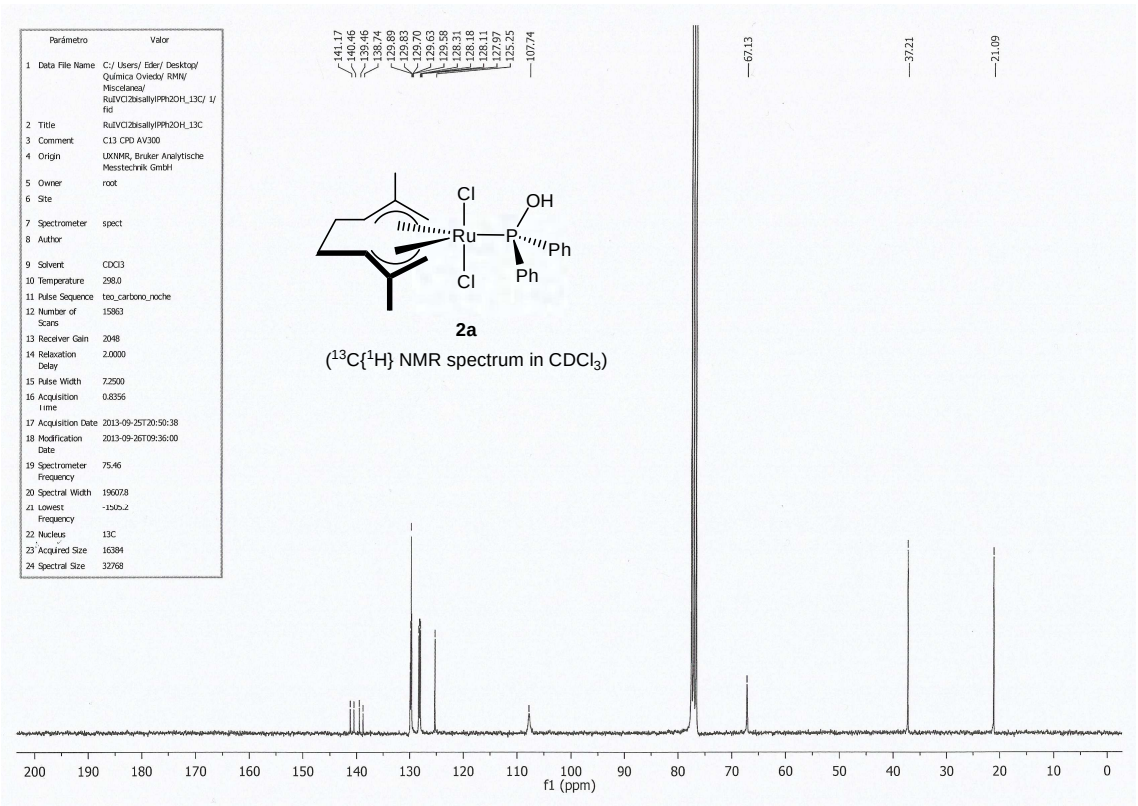
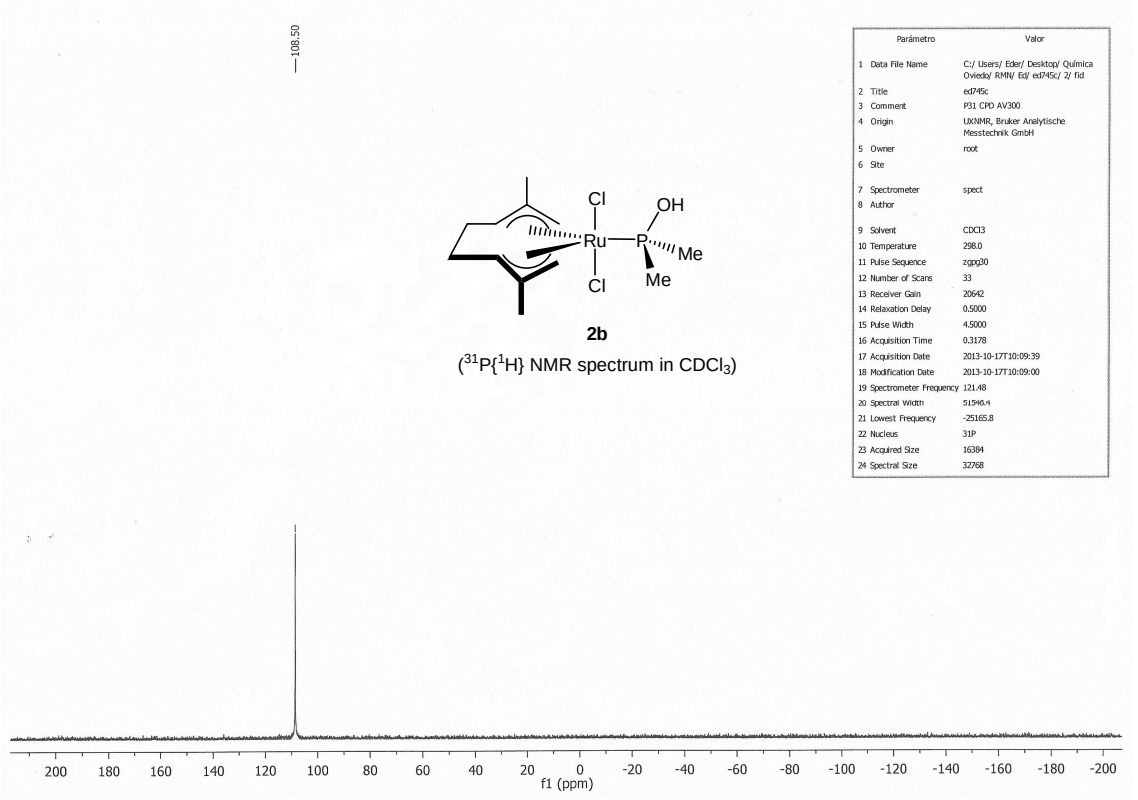


2a

($^{13}C\{^{1}H\}$ NMR spectrum in $CDCl_3$)



2b

($^{31}P\{^{1}H\}$ NMR spectrum in $CDCl_3$)

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7	Spectrometer	spect
8	Author	
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10	Temperature	298.0
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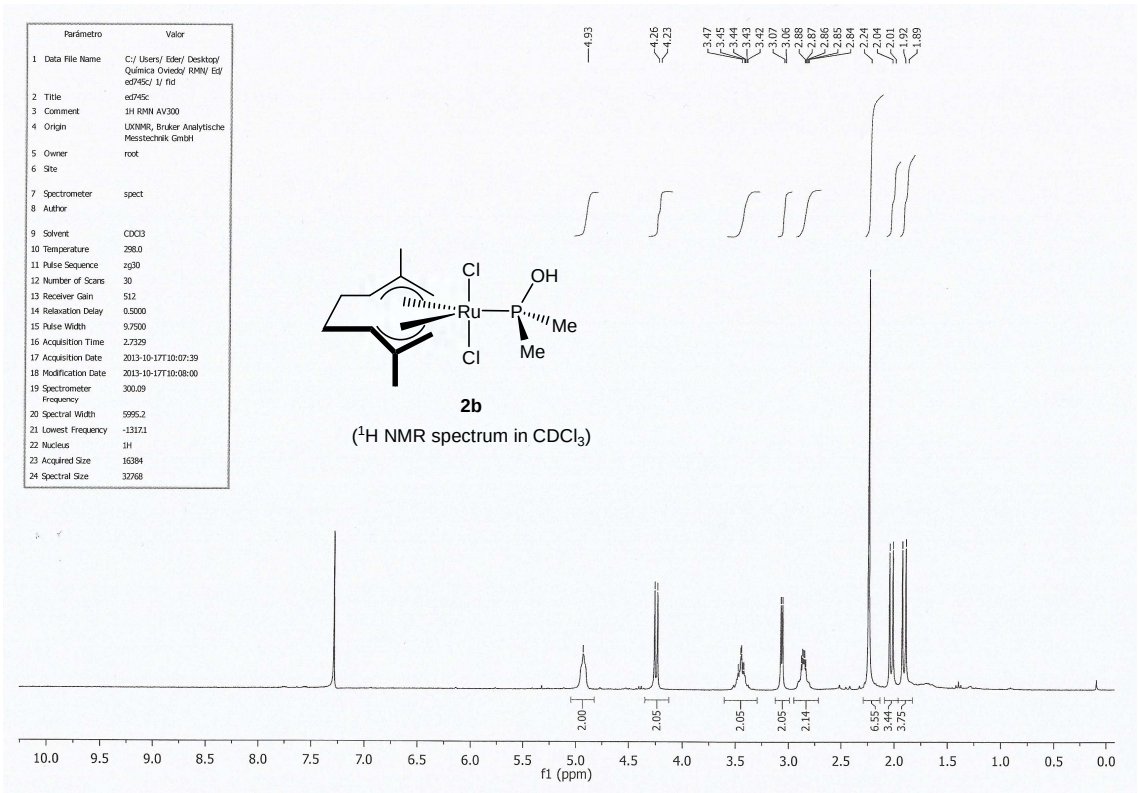
22	Nucleus	1H
23	Acquired Size	16384
24	Spectral Size	32768



2b

(^{1}H NMR spectrum in $CDCl_3$)

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5	Owner	Bruker
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7	Spectrometer	spect
8	Author	
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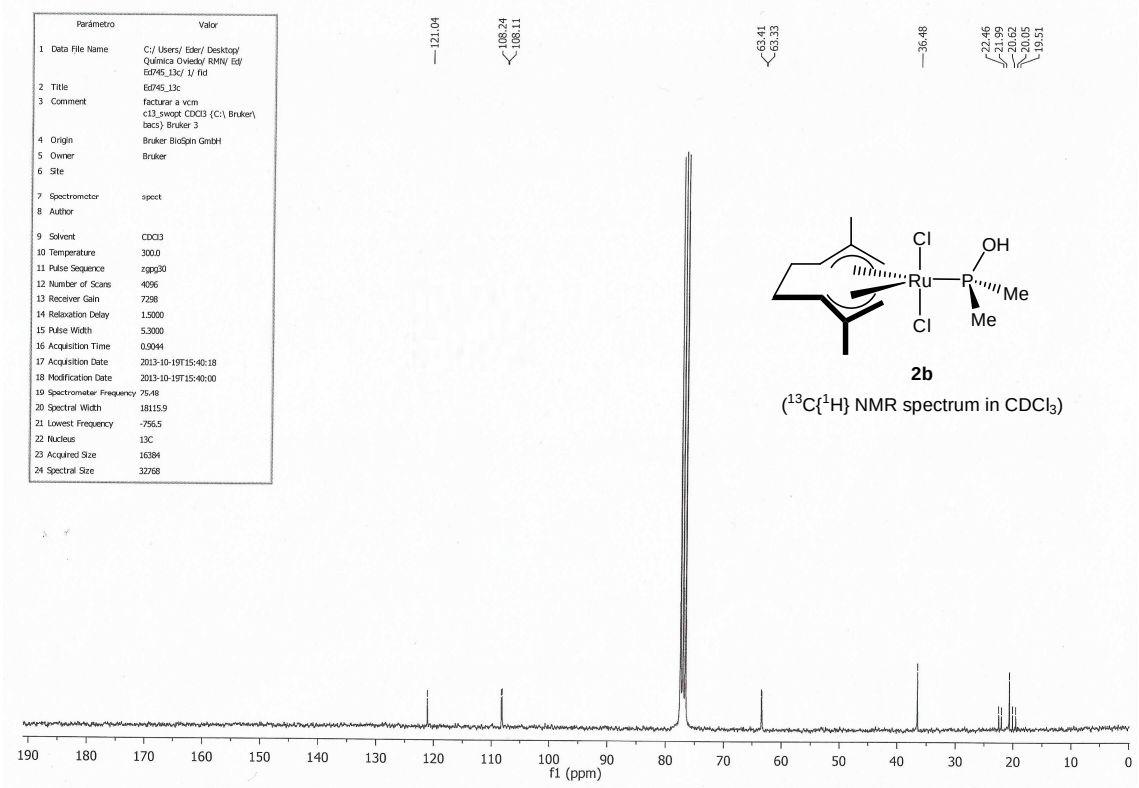
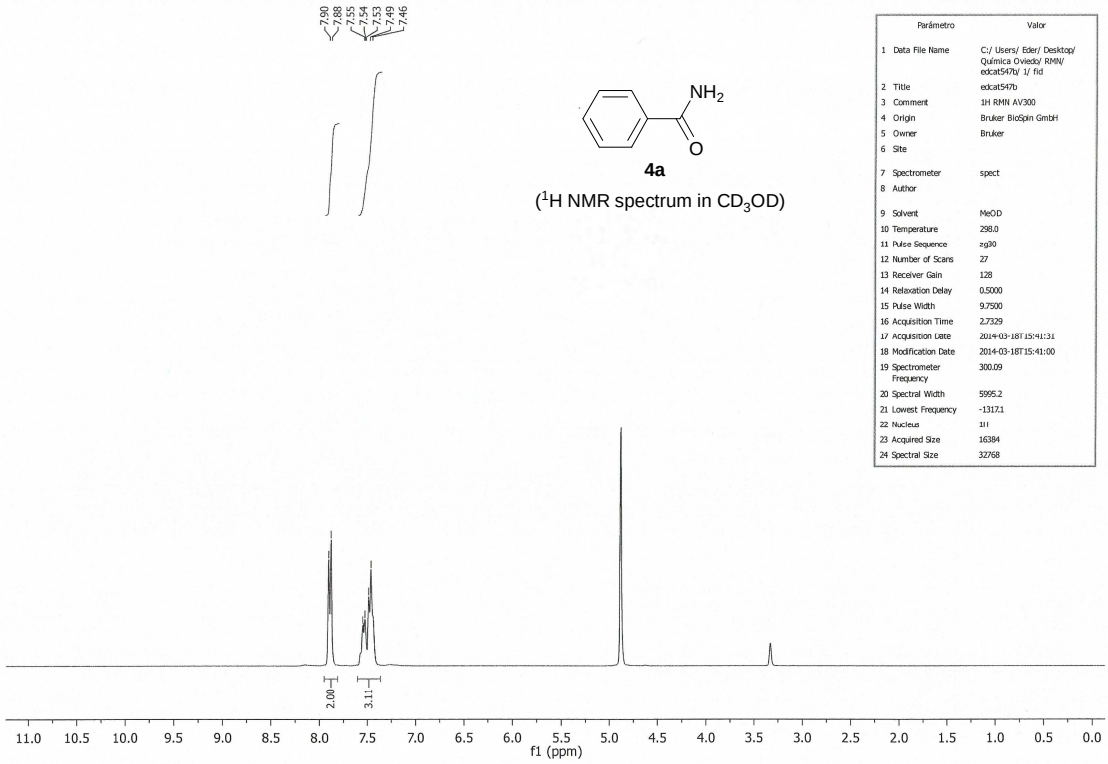


2b

($^{13}C\{^{1}H\}$ NMR spectrum in $CDCl_3$)



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23	Acquired Size	16384
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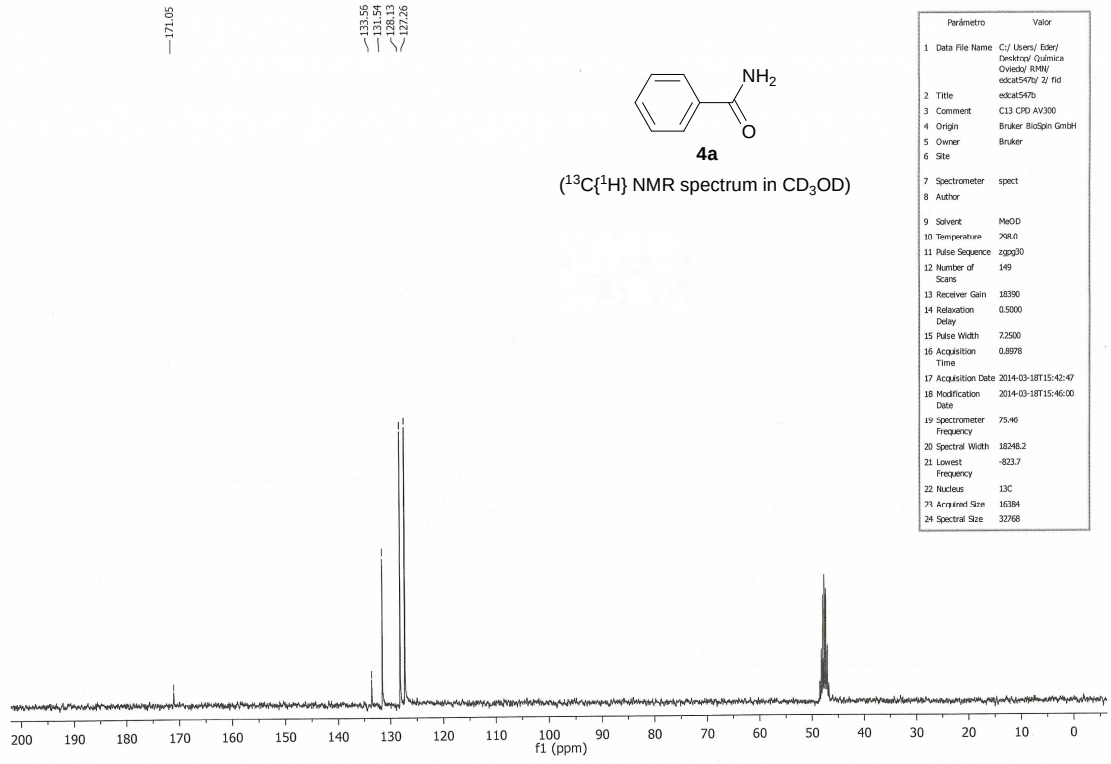
4a

(^{1}H NMR spectrum in CD_3OD)



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22	Nucleus	13C
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4a

($^{13}C\{^1H\}$ NMR spectrum in CD_3OD)

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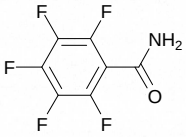
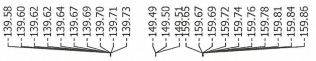
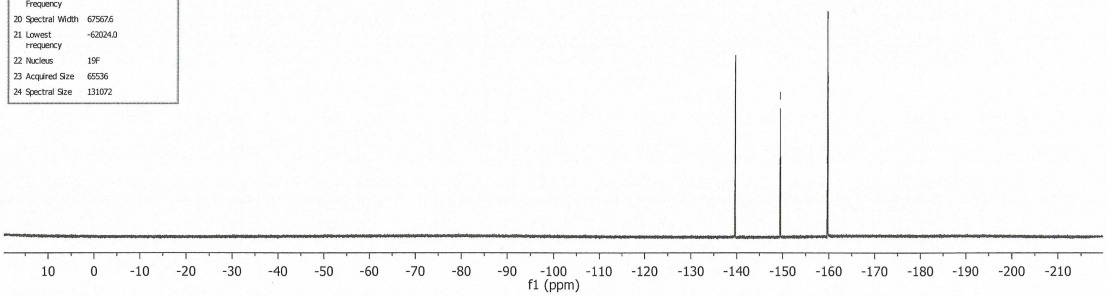
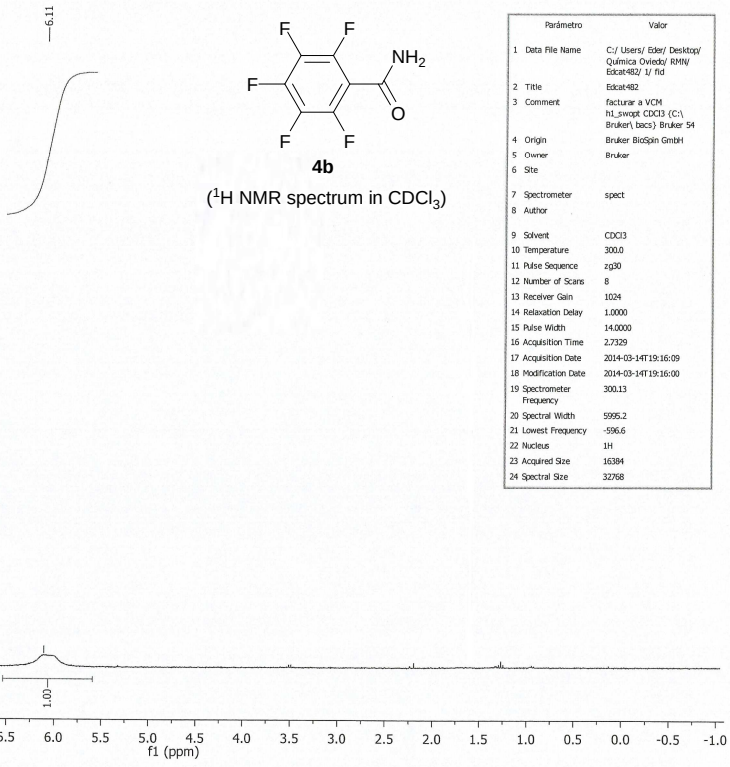


4b

($^{19}F\{^{1}H\}$ NMR spectrum in $CDCl_3$)







4b

(^{1}H NMR spectrum in $CDCl_3$)

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8	Author	
9	Solvent	CDCl3
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22	Nucleus	1H
23	Acquired Size	16384
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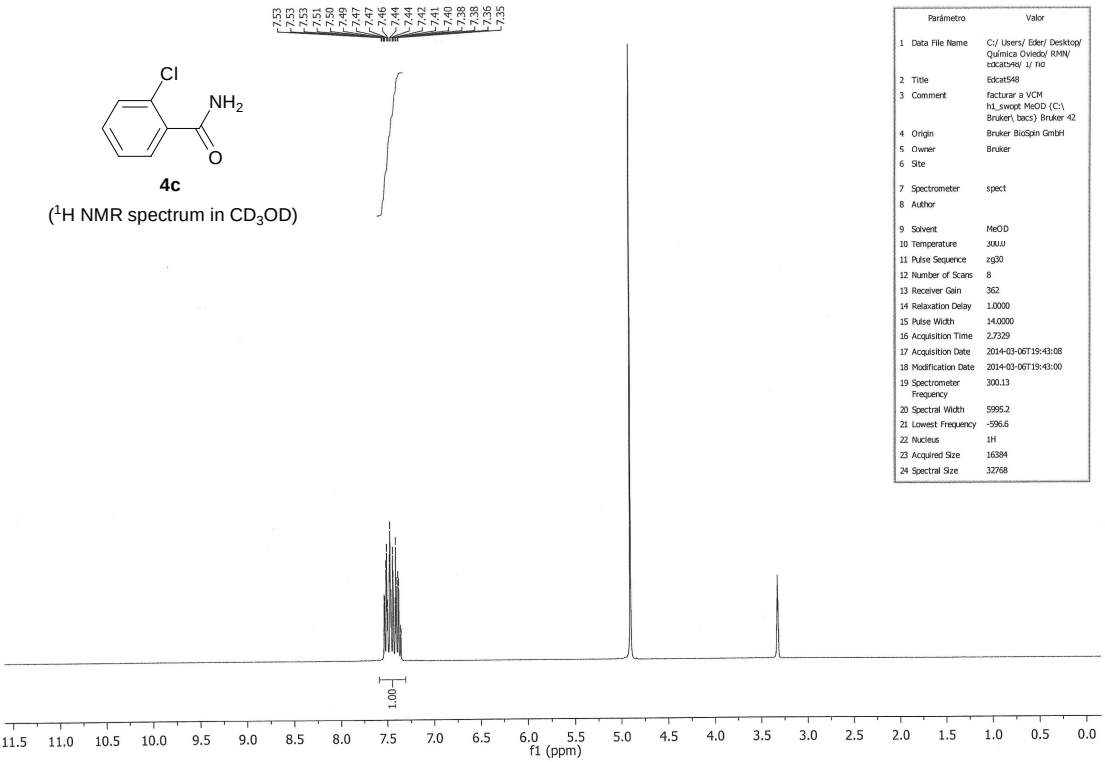
4c

(^{1}H NMR spectrum in CD_3OD)



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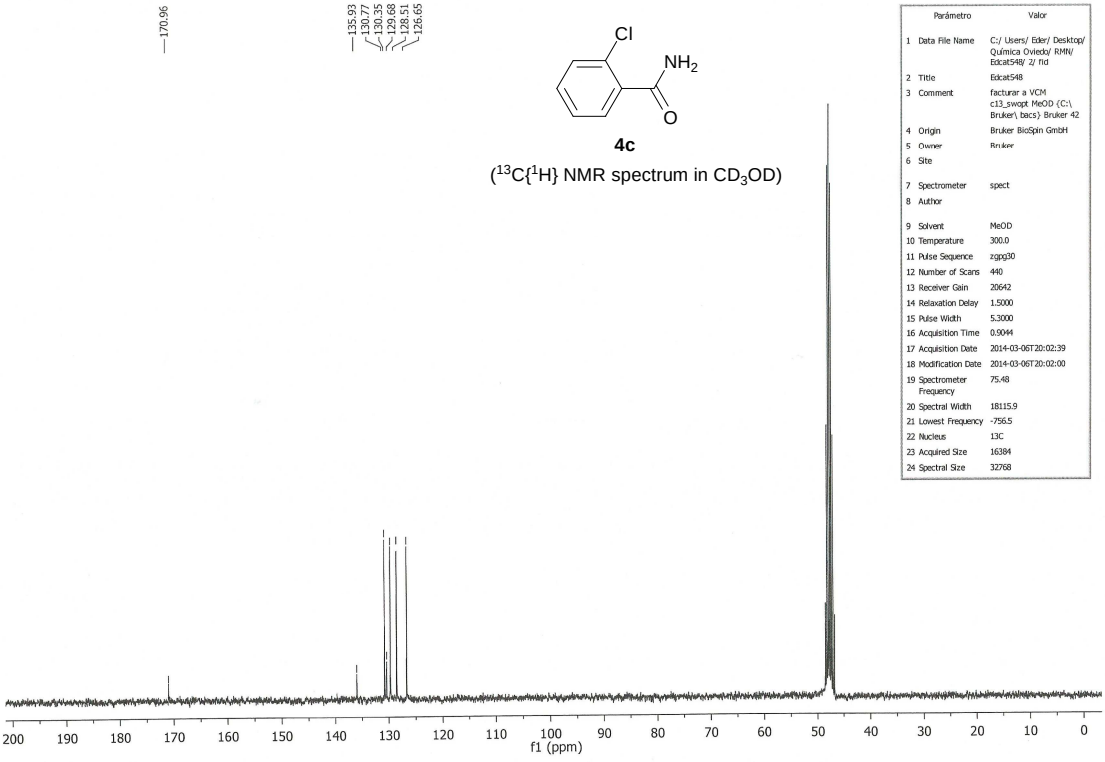
24	Spectral Size	32768

4c

($^{13}C\{^1H\}$ NMR spectrum in CD_3OD)



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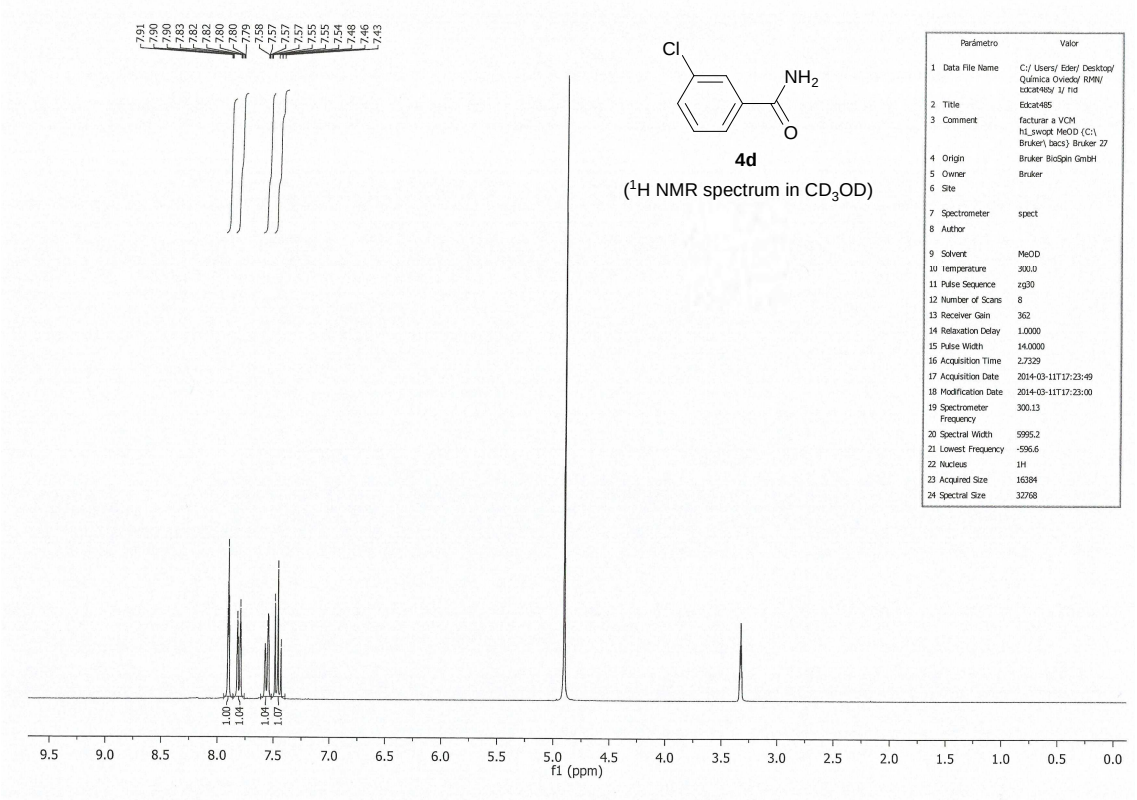
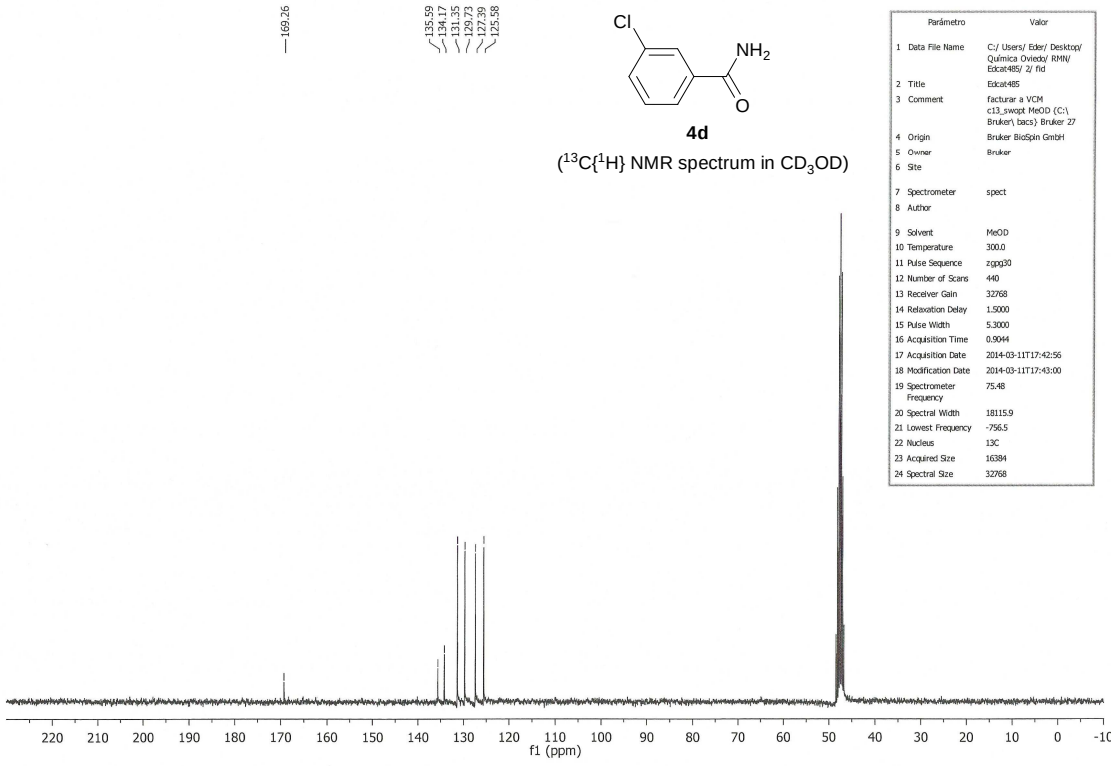
14	Relaxation Delay	1.5000
15	Pulse Width	5.3000
16	Acquisition Time	0.9044
17	Acquisition Date	2014-03-06T20:02:39
18	Modification Date	2014-03-06T20:02:00
19	Spectrometer Frequency	75.48
20	Spectral Width	18115.9
21	Lowest Frequency	-756.5
22	Nucleus	13C
23	Acquired Size	16384
24	Spectral Size	32768



4d

(^{1}H NMR spectrum in CD_3OD)

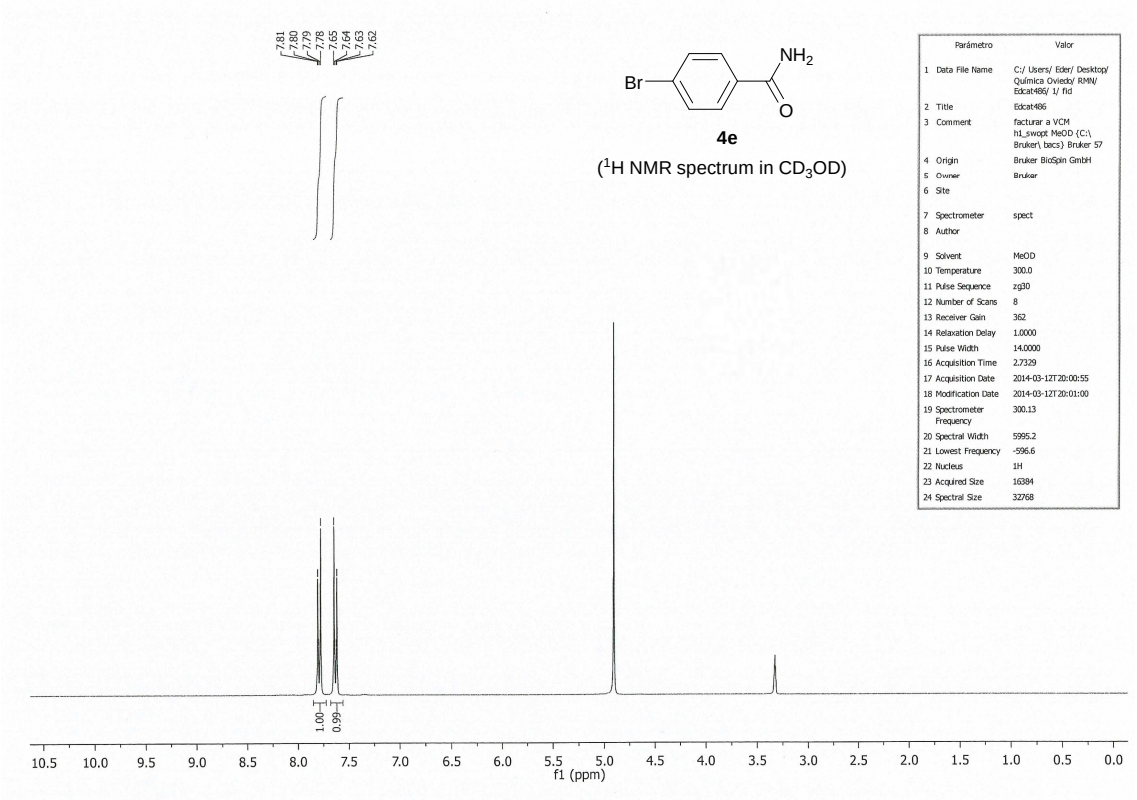
	Parámetro	Valor
1	Data File Name	C:/ Users/ Eder/ Desktop/ Química Oviedo/ RMN/ Edcat485/ 1/ fid
2	Title	Edcat485
3	Comment	facturar a VCM h1_swopt MeOD {C:\ Bruker\ bacs} Bruker 27
4	Origin	Bruker BioSpin GmbH
5	Owner	Bruker
6	Site	
7	Spectrometer	spect
8	Author	
9	Solvent	MeOD
10	Temperature	300.0
11	Pulse Sequence	zg30
12	Number of Scans	8
13	Receiver Gain	362
14	Relaxation Delay	1.0000
15	Pulse Width	14.0000
16	Acquisition Time	2.7329
17	Acquisition Date	2014-03-11T17:23:49
18	Modification Date	2014-03-11T17:23:00
19	Spectrometer Frequency	300.13
20	Spectral Width	5995.2
21	Lowest Frequency	-596.6
22	Nucleus	1H
23	Acquired Size	16384
24	Spectral Size	32768



4d

($^{13}C\{^1H\}$ NMR spectrum in CD_3OD)

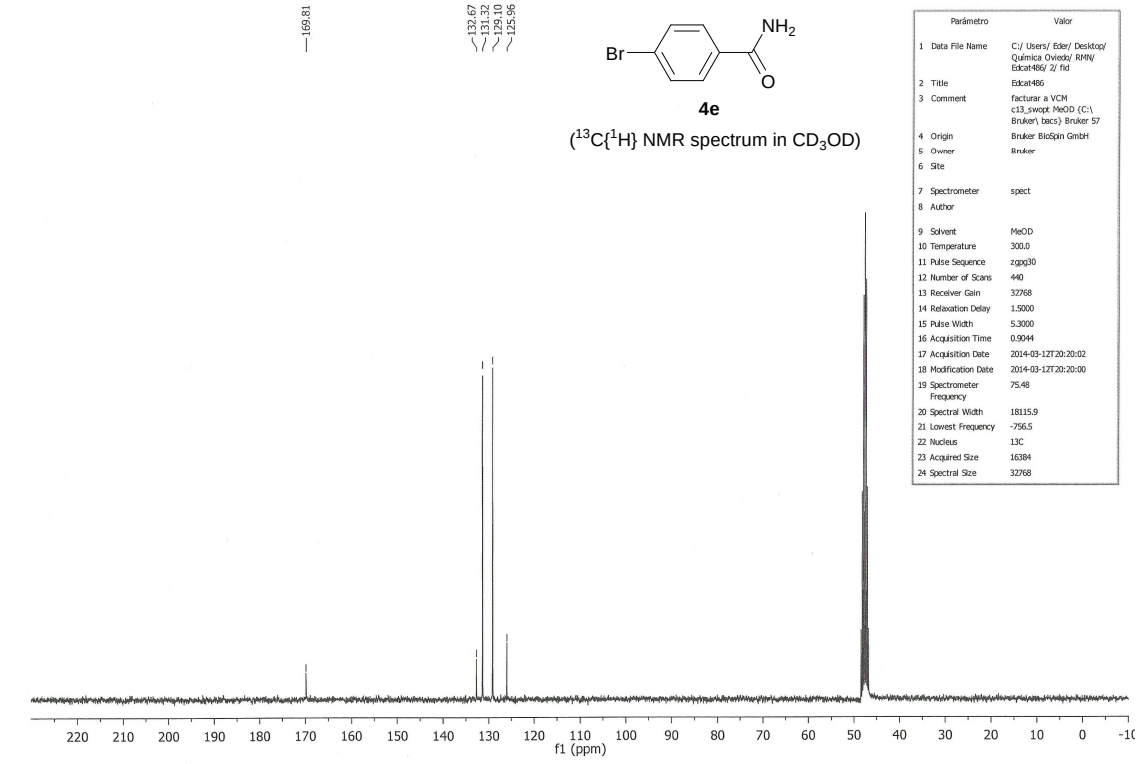
	Parámetro	Valor
1	Data File Name	C:/ Users/ Eder/ Desktop/ Química Oviedo/ RMN/ Edcat485/ 2/ fid
2	Title	Edcat485
3	Comment	facturar a VCM c13_swopt MeOD {C:\ Bruker\ bacs} Bruker 27
4	Origin	Bruker BioSpin GmbH
5	Owner	Bruker
6	Site	
7	Spectrometer	spect
8	Author	
9	Solvent	MeOD
10	Temperature	300.0
11	Pulse Sequence	zgpg30
12	Number of Scans	440
13	Receiver Gain	32768
14	Relaxation Delay	1.5000
15	Pulse Width	5.3000
16	Acquisition Time	0.9044
17	Acquisition Date	2014-03-11T17:42:56
18	Modification Date	2014-03-11T17:43:00
19	Spectrometer Frequency	75.48
20	Spectral Width	18115.9
21	Lowest Frequency	-756.5
22	Nucleus	13C
23	Acquired Size	16384
24	Spectral Size	32768



4e

(^{1}H NMR spectrum in CD_3OD)

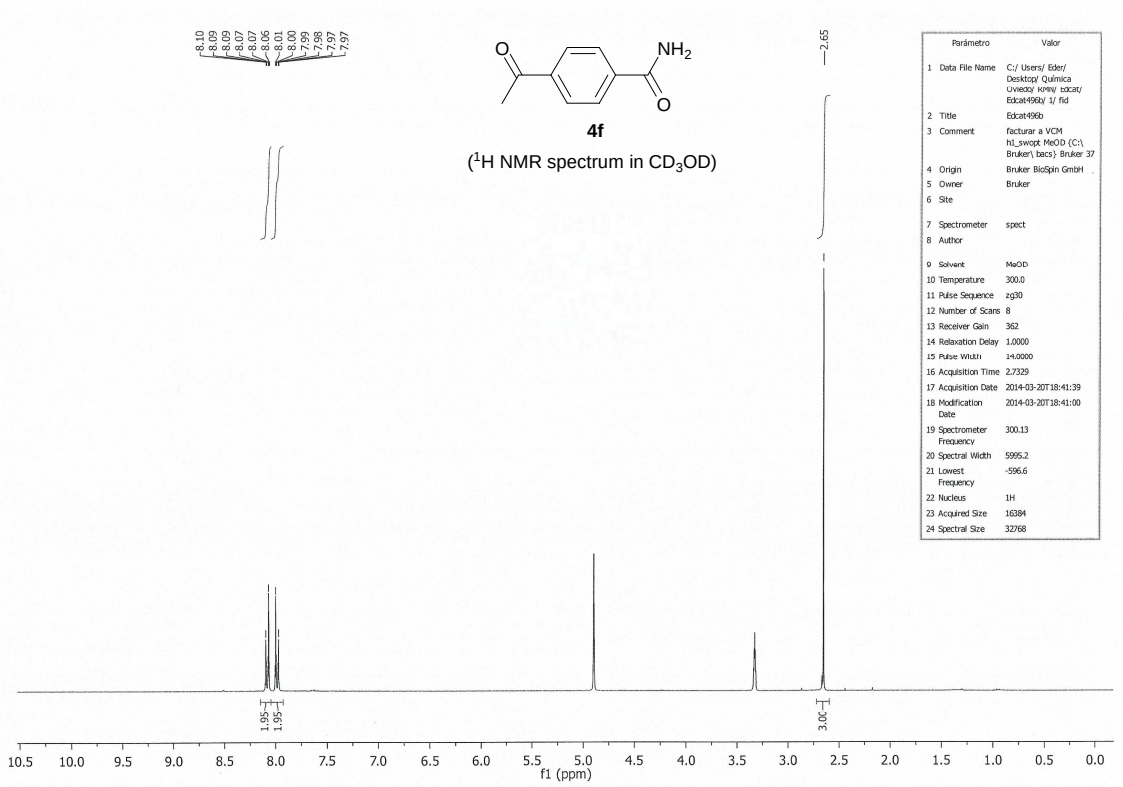
	Parámetro	Valor
1	Data File Name	C:/ Users/ Eder/ Desktop/ Química Oviedo/ RMN/ Edcat486/ 1/ fid
2	Title	Edcat486
3	Comment	facturar a VCM h1_swopt MeOD {C:\ Bruker\ bacs} Bruker 57
4	Origin	Bruker BioSpin GmbH
5	Owner	Bruker
6	Site	
7	Spectrometer	spect
8	Author	
9	Solvent	MeOD
10	Temperature	300.0
11	Pulse Sequence	zg30
12	Number of Scans	8
13	Receiver Gain	362
14	Relaxation Delay	1.0000
15	Pulse Width	14.0000
16	Acquisition Time	2.7329
17	Acquisition Date	2014-03-12T20:00:55
18	Modification Date	2014-03-12T20:01:00
19	Spectrometer Frequency	300.13
20	Spectral Width	5995.2
21	Lowest Frequency	-596.6
22	Nucleus	1H
23	Acquired Size	16384
24	Spectral Size	32768



4e

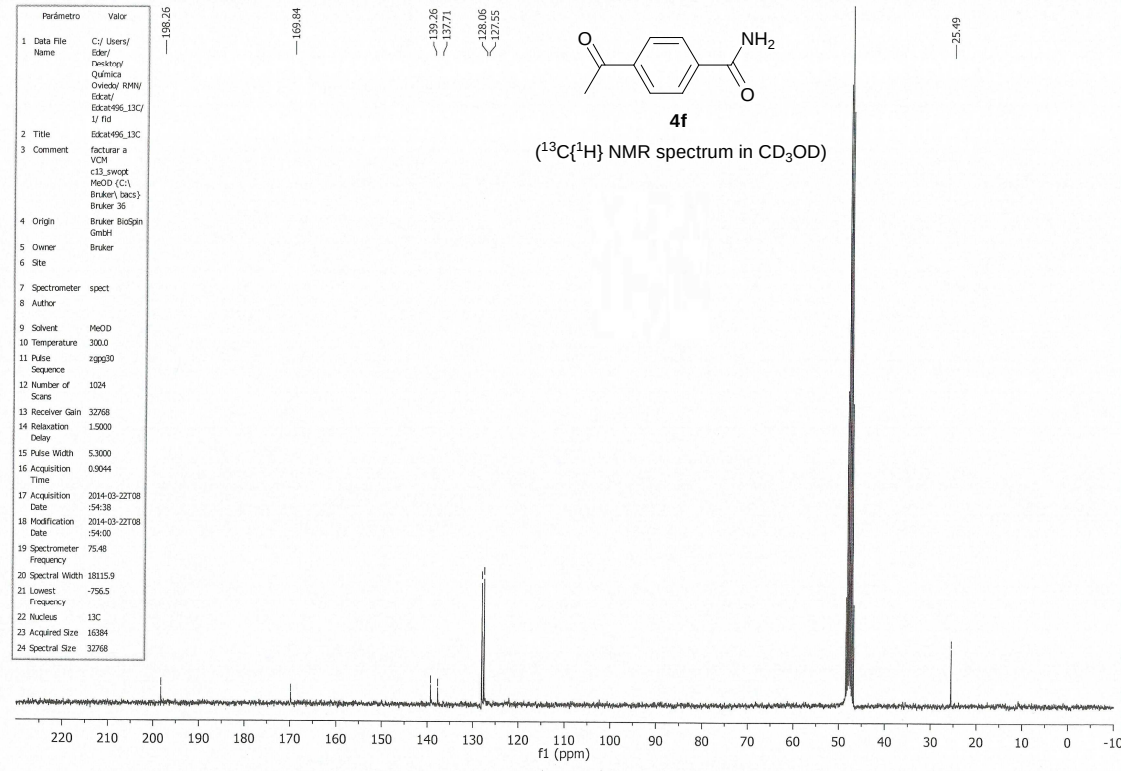
($^{13}C\{^1H\}$ NMR spectrum in CD_3OD)

	Parámetro	Valor
1	Data File Name	C:/ Users/ Eder/ Desktop/ Química Oviedo/ RMN/ Edcat486/ 2/ fid
2	Title	Edcat486
3	Comment	facturar a VCM c13_swopt MeOD {C:\ Bruker\ bacs} Bruker 57
4	Origin	Bruker BioSpin GmbH
5	Owner	Bruker
6	Site	
7	Spectrometer	spect
8	Author	
9	Solvent	MeOD
10	Temperature	300.0
11	Pulse Sequence	zgpg30
12	Number of Scans	440
13	Receiver Gain	32768
14	Relaxation Delay	1.5000
15	Pulse Width	5.3000
16	Acquisition Time	0.9044
17	Acquisition Date	2014-03-12T20:20:02
18	Modification Date	2014-03-12T20:20:00
19	Spectrometer Frequency	75.48
20	Spectral Width	18115.9
21	Lowest Frequency	-756.5
22	Nucleus	13C
23	Acquired Size	16384
24	Spectral Size	32768



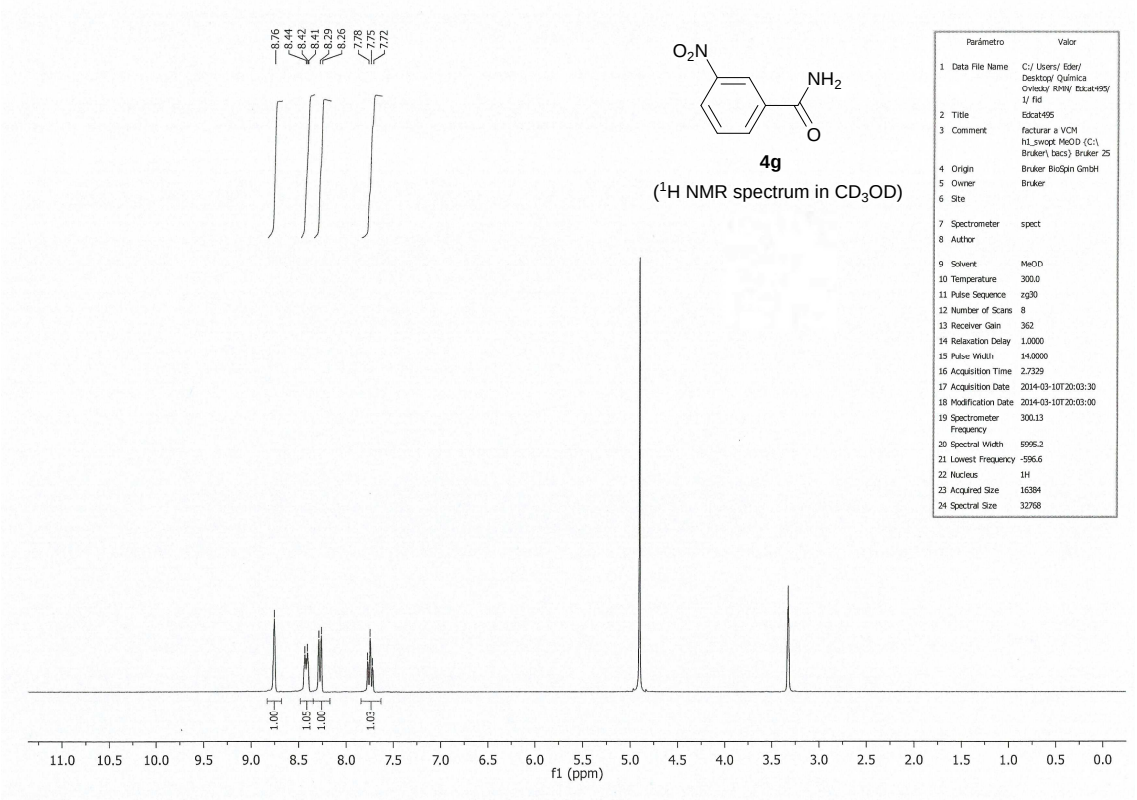
4f

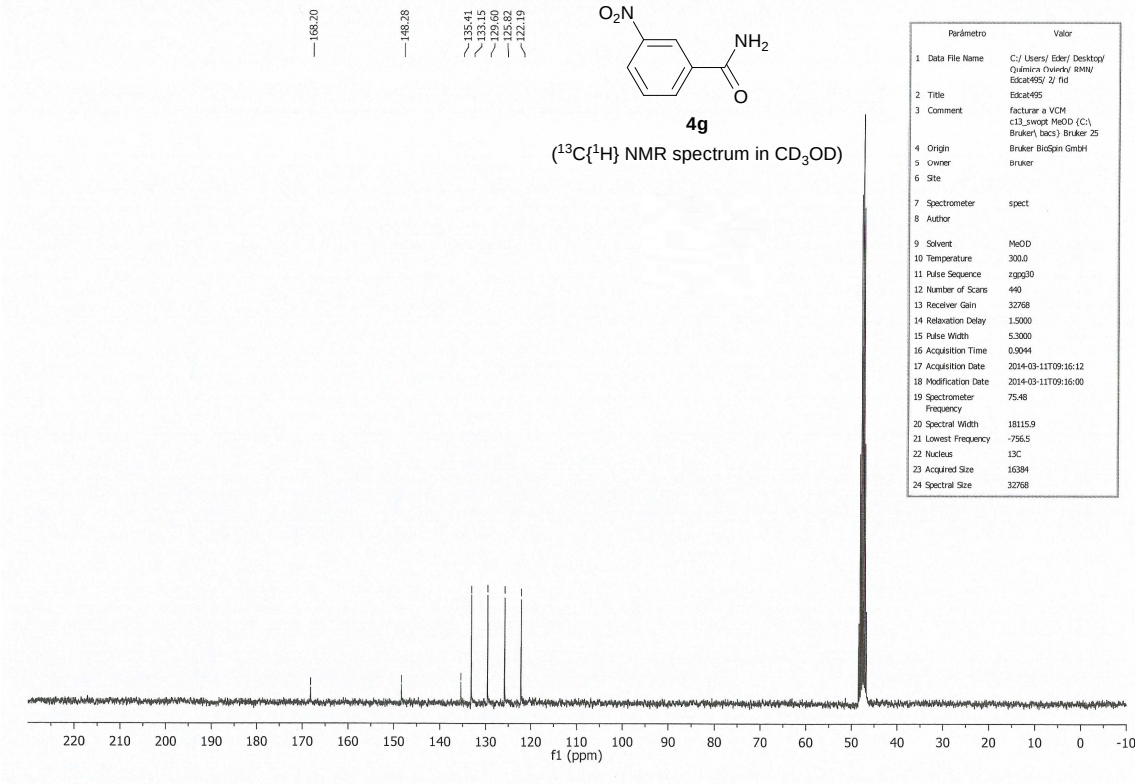
(^{1}H NMR spectrum in CD_3OD)

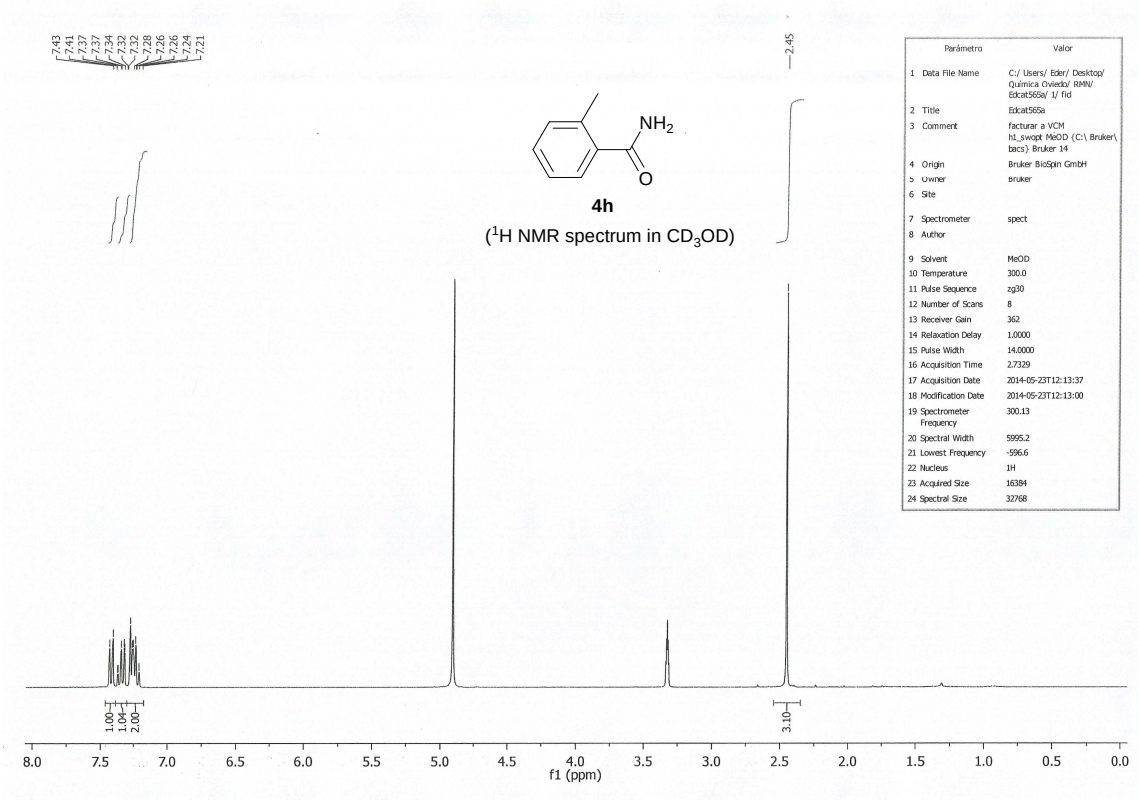


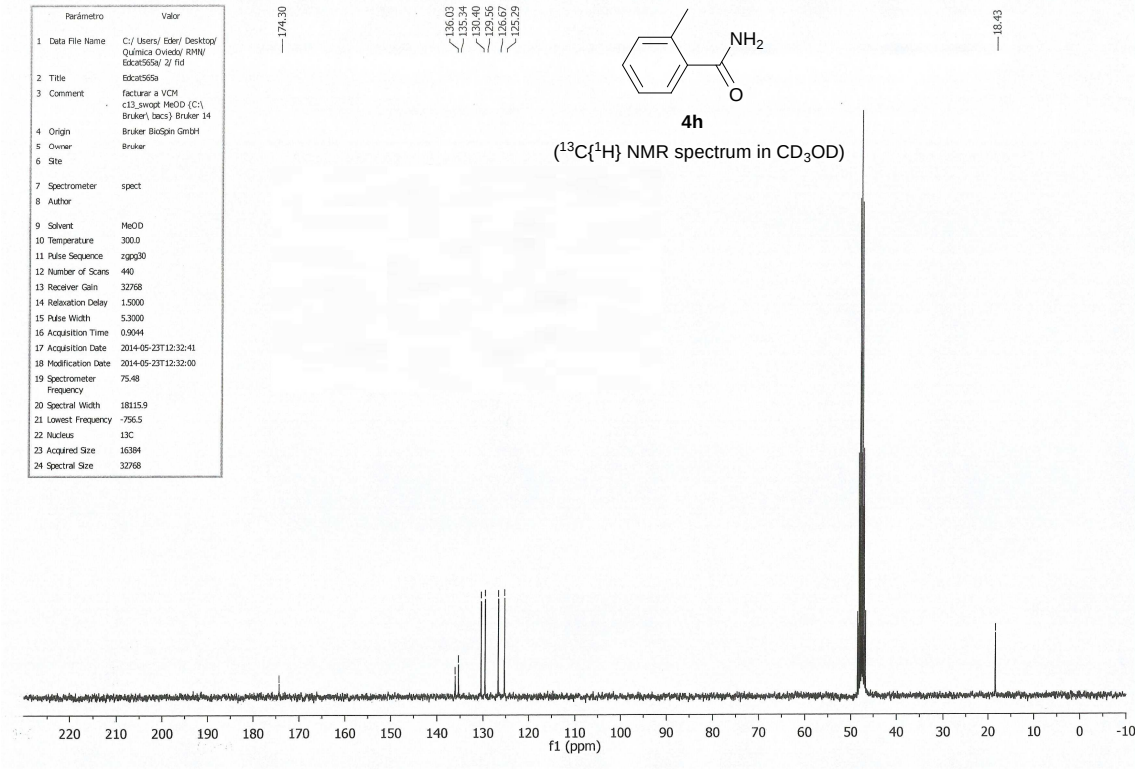
4f

($^{13}C\{^1H\}$ NMR spectrum in CD_3OD)


8.76
8.44
8.42
8.41
8.29
8.26
7.78
7.75
7.72
O2N
NH2
O
4g
(1H NMR spectrum in CD3OD)
Parámetro Valor
1 Data File Name C:/ Users/ Eder/ Desktop/ Química Oviedo/ RMN/ Edcat495/ 1/ fid
2 Title Edcat495
3 Comment facturar a VCM h1_swopt MeOD {C:\ Bruker\ bacs} Bruker 25
4 Origin Bruker BioSpin GmbH
5 Owner Bruker
6 Site
7 Spectrometer spect
8 Author
9 Solvent MeOD
10 Temperature 300.0
11 Pulse Sequence zg30
12 Number of Scans 8
13 Receiver Gain 362
14 Relaxation Delay 1.0000
15 Pulse Width 14.0000
16 Acquisition Time 2.7329
17 Acquisition Date 2014-03-10T20:03:30
18 Modification Date 2014-03-10T20:03:00
19 Spectrometer Frequency 300.13
20 Spectral Width 5995.2
21 Lowest Frequency -596.6
22 Nucleus 1H
23 Acquired Size 16384
24 Spectral Size 32768
1.00
1.05
1.00
1.03
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


168.20
148.28
135.41
133.15
129.60
125.82
122.19
O2N
NH2
O
4g
(13C{1H} NMR spectrum in CD3OD)
Parámetro Valor
1 Data File Name C:/ Users/ Eder/ Desktop/ Química Oviedo/ RMN/ Edcat495/ 2/ fid
2 Title Edcat495
3 Comment facturar a VCM c13_swopt MeOD {C:\ Bruker\ bacs} Bruker 25
4 Origin Bruker BioSpin GmbH
5 Owner Bruker
6 Site
7 Spectrometer spect
8 Author
9 Solvent MeOD
10 Temperature 300.0
11 Pulse Sequence zgpg30
12 Number of Scans 440
13 Receiver Gain 32768
14 Relaxation Delay 1.5000
15 Pulse Width 5.3000
16 Acquisition Time 0.9044
17 Acquisition Date 2014-03-11T09:16:12
18 Modification Date 2014-03-11T09:16:00
19 Spectrometer Frequency 75.48
20 Spectral Width 18115.9
21 Lowest Frequency -756.5
22 Nucleus 13C
23 Acquired Size 16384
24 Spectral Size 32768
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


7.43
7.41
7.37
7.37
7.34
7.32
7.32
7.28
7.26
7.26
7.24
7.21
2.45
NH2
O
4h
(1H NMR spectrum in CD3OD)
Parámetro Valor
1 Data File Name C:/ Users/ Eder/ Desktop/ Química Oviedo/ RMN/ Edcat565a/ 1/ fid
2 Title Edcat565a
3 Comment facturar a VCM h1_swopt MeOD {C:\ Bruker\ bacs} Bruker 14
4 Origin Bruker BioSpin GmbH
5 Owner bruker
6 Site
7 Spectrometer spect
8 Author
9 Solvent MeOD
10 Temperature 300.0
11 Pulse Sequence zg30
12 Number of Scans 8
13 Receiver Gain 362
14 Relaxation Delay 1.0000
15 Pulse Width 14.0000
16 Acquisition Time 2.7329
17 Acquisition Date 2014-05-23T12:13:37
18 Modification Date 2014-05-23T12:13:00
19 Spectrometer Frequency 300.13
20 Spectral Width 5995.2
21 Lowest Frequency -596.6
22 Nucleus 1H
23 Acquired Size 16384
24 Spectral Size 32768
1.00
1.04
2.00
3.10
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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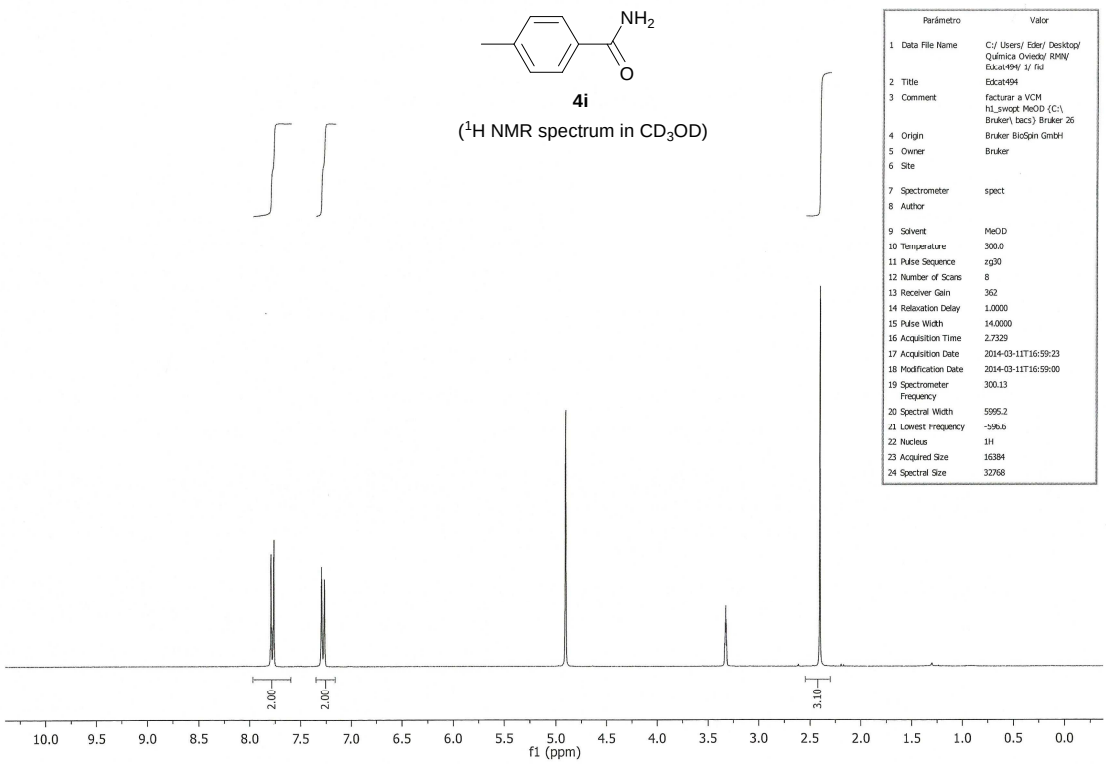
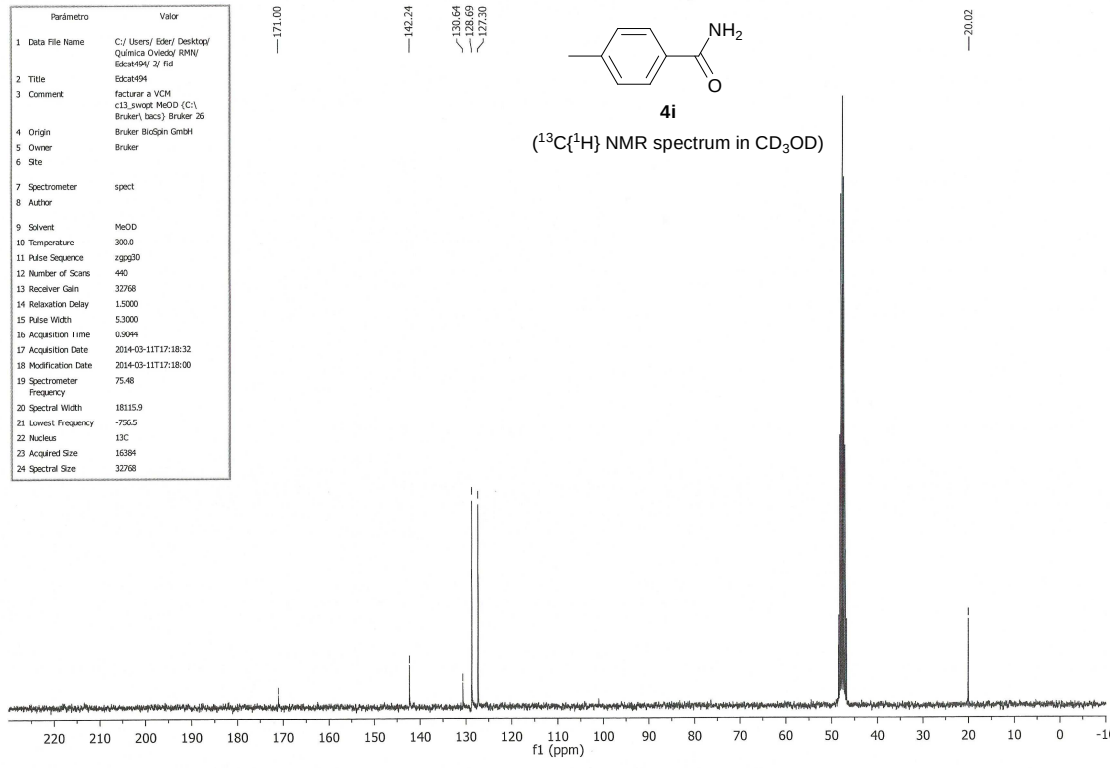
1 Data File Name C:/ Users/ Eder/ Desktop/ Química Oviedo/ RMN/ Edcat565a/ 2/ fid
2 Title Edcat565a
3 Comment facturar a VCM c13_swopt MeOD {C:\ Bruker\ bacs} Bruker 14
4 Origin Bruker BioSpin GmbH
5 Owner Bruker
6 Site
7 Spectrometer spect
8 Author
9 Solvent MeOD
10 Temperature 300.0
11 Pulse Sequence zgpg30
12 Number of Scans 440
13 Receiver Gain 32768
14 Relaxation Delay 1.5000
15 Pulse Width 5.3000
16 Acquisition Time 0.9044
17 Acquisition Date 2014-05-23T12:32:41
18 Modification Date 2014-05-23T12:32:00
19 Spectrometer Frequency 75.48
20 Spectral Width 18115.9
21 Lowest Frequency -756.5
22 Nucleus 13C
23 Acquired Size 16384
24 Spectral Size 32768
174.30
136.03
135.34
130.40
125.56
125.57
125.29
18.43
NH2
O
4h
(13C{1H} NMR spectrum in CD3OD)
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


NH2
O
4i
(1H NMR spectrum in CD3OD)
Parámetro Valor
1 Data File Name C:/ Users/ Eder/ Desktop/ Química Oviedo/ RMN/ Edcat494/ 1/ fid
2 Title Edcat494
3 Comment facturar a VCM h1_swopt MeOD {C:\ Bruker\ bacs} Bruker 26
4 Origin Bruker BioSpin GmbH
5 Owner Bruker
6 Site
7 Spectrometer spect
8 Author
9 Solvent MeOD
10 Temperature 300.0
11 Pulse Sequence zg30
12 Number of Scans 8
13 Receiver Gain 362
14 Relaxation Delay 1.0000
15 Pulse Width 14.0000
16 Acquisition Time 2.7329
17 Acquisition Date 2014-03-11T16:59:23
18 Modification Date 2014-03-11T16:59:00
19 Spectrometer Frequency 300.13
20 Spectral Width 5995.2
21 Lowest Frequency -596.6
22 Nucleus 1H
23 Acquired Size 16384
24 Spectral Size 32768
2.00
2.00
3.10
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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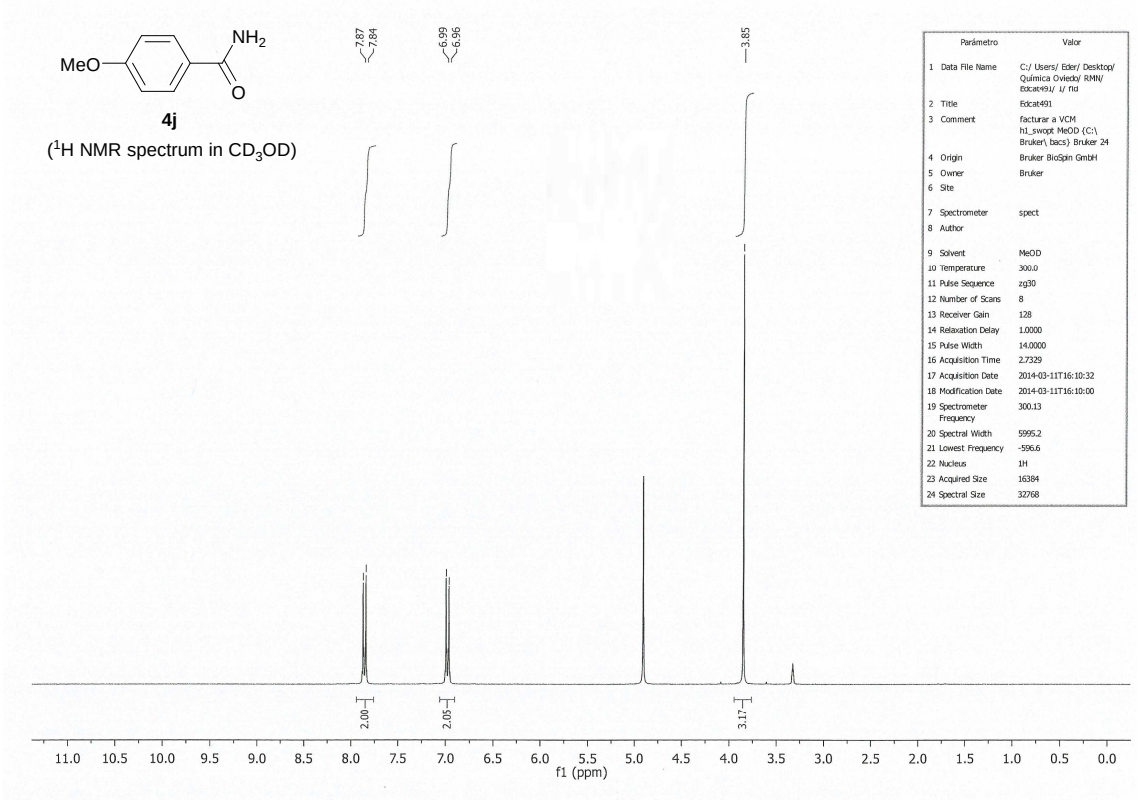
1 Data File Name C:/ Users/ Eder/ Desktop/ Química Oviedo/ RMN/ Edcat494/ 2/ fid
2 Title Edcat494
3 Comment facturar a VCM c13_swopt MeOD {C:\ Bruker\ bacs} Bruker 26
4 Origin Bruker BioSpin GmbH
5 Owner Bruker
6 Site
7 Spectrometer spect
8 Author
9 Solvent MeOD
10 Temperature 300.0
11 Pulse Sequence zgpg30
12 Number of Scans 440
13 Receiver Gain 32768
14 Relaxation Delay 1.5000
15 Pulse Width 5.3000
16 Acquisition Time 0.9044
17 Acquisition Date 2014-03-11T17:18:32
18 Modification Date 2014-03-11T17:18:00
19 Spectrometer Frequency 75.48
20 Spectral Width 18115.9
21 Lowest Frequency -756.5
22 Nucleus 13C
23 Acquired Size 16384
24 Spectral Size 32768
171.00
142.24
130.64
128.69
127.30
20.02
NH2
O
4i
(13C{1H} NMR spectrum in CD3OD)
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

4j

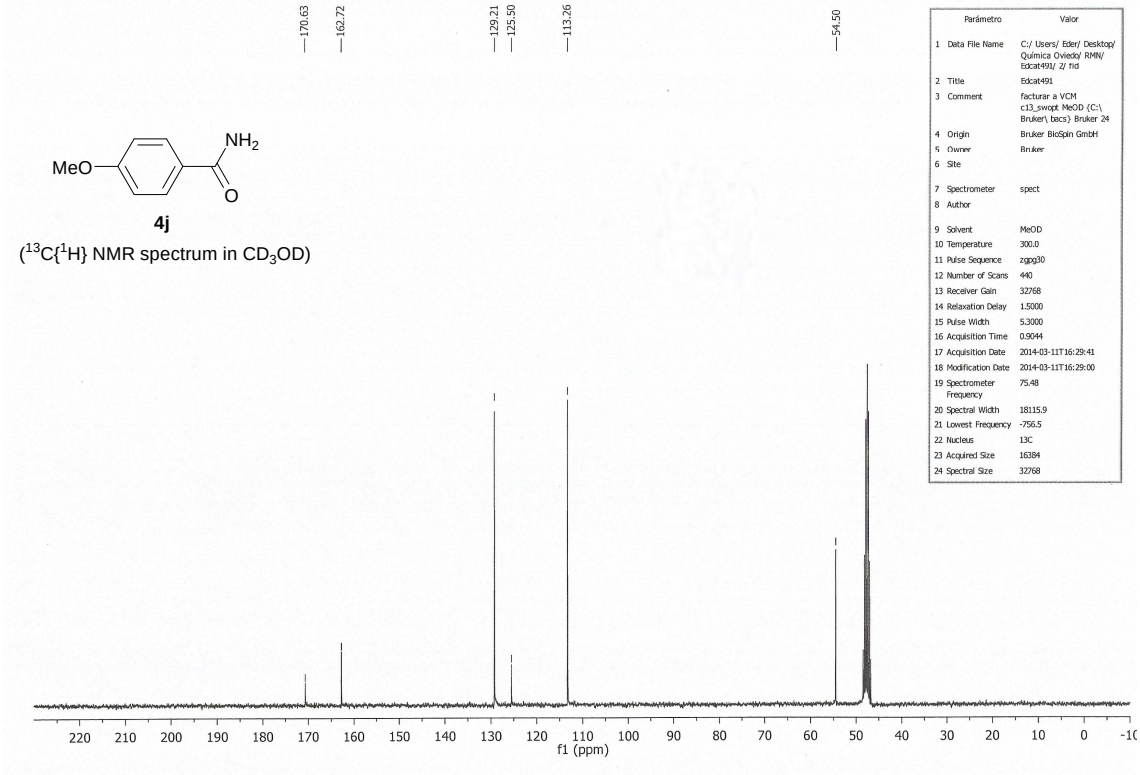
(^{1}H NMR spectrum in CD_3OD)



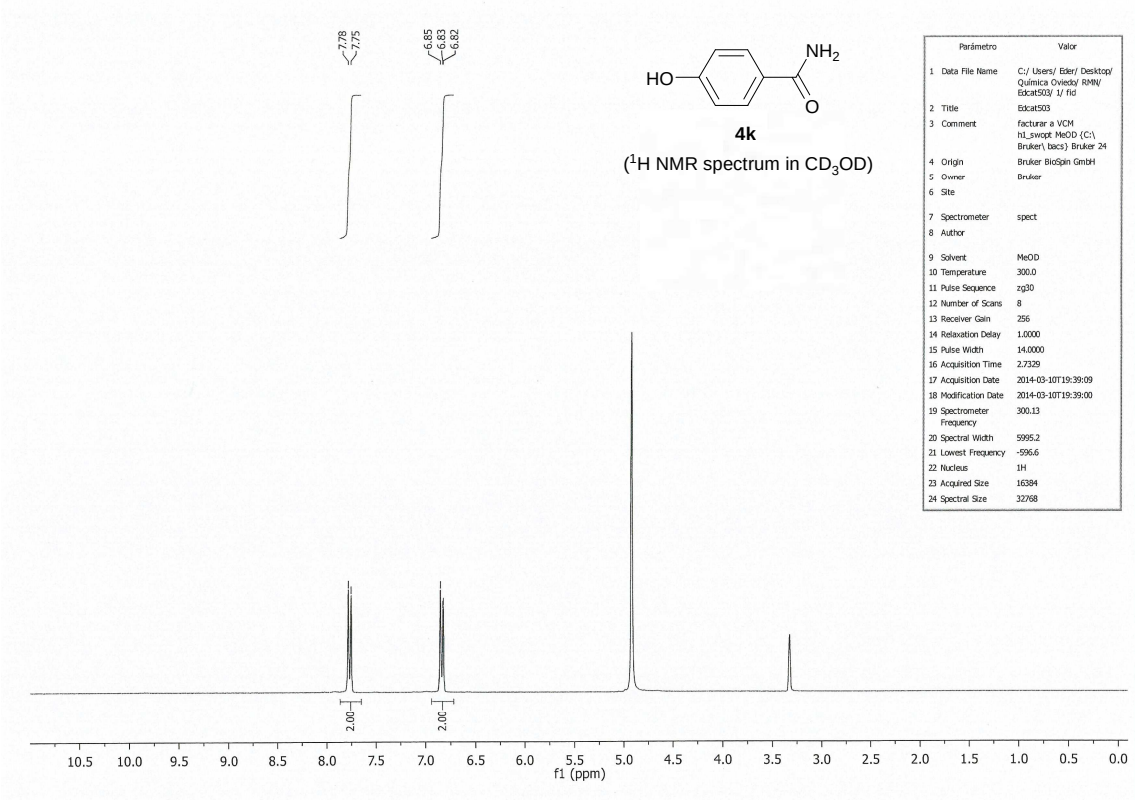
	Parámetro	Valor
1	Data File Name	C:/ Users/ Eder/ Desktop/ Química Oviedo/ RMN/ Edcat491/ 1/ fid
2	Title	Edcat491
3	Comment	facturar a VCM h1_swopt MeOD {C:\ Bruker\ bacs} Bruker 24
4	Origin	Bruker BioSpin GmbH
5	Owner	Bruker
6	Site	
7	Spectrometer	spect
8	Author	
9	Solvent	MeOD
10	Temperature	300.0
11	Pulse Sequence	zg30
12	Number of Scans	8
13	Receiver Gain	128
14	Relaxation Delay	1.0000
15	Pulse Width	14.0000
16	Acquisition Time	2.7329
17	Acquisition Date	2014-03-11T16:10:32
18	Modification Date	2014-03-11T16:10:00
19	Spectrometer Frequency	300.13
20	Spectral Width	5995.2
21	Lowest Frequency	-596.6
22	Nucleus	1H
23	Acquired Size	16384
24	Spectral Size	32768

4j

($^{13}C\{^1H\}$ NMR spectrum in CD_3OD)



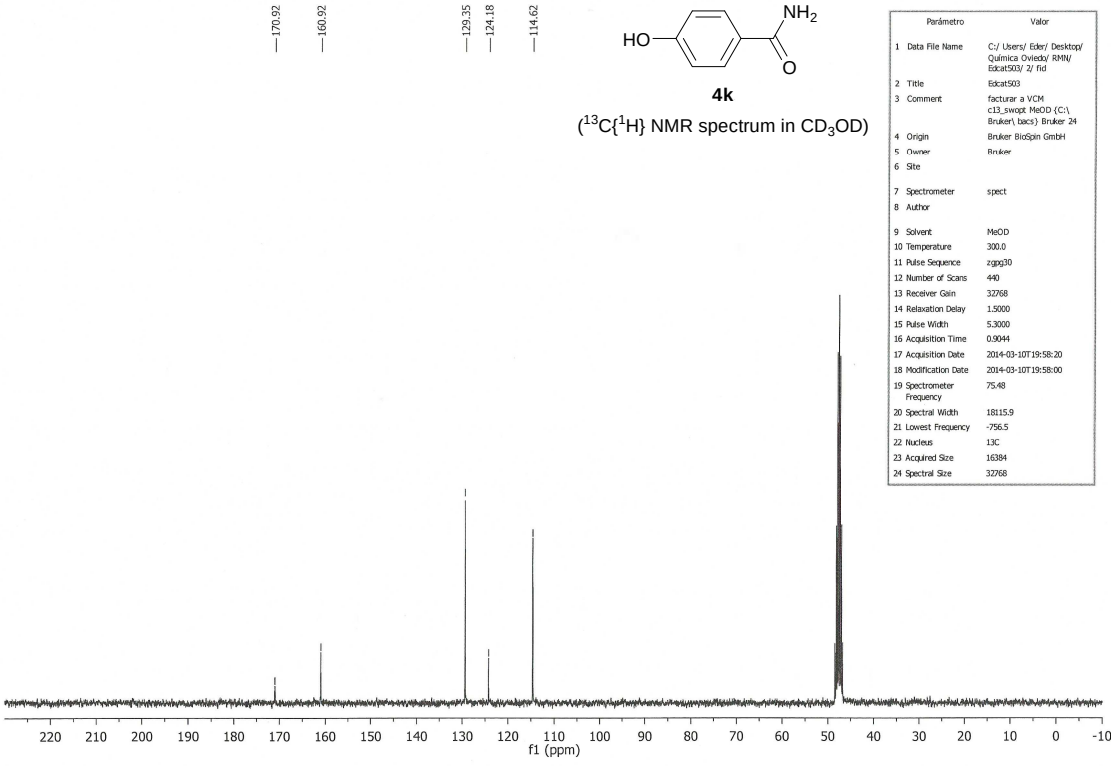
	Parámetro	Valor
1	Data File Name	C:/ Users/ Eder/ Desktop/ Química Oviedo/ RMN/ Edcat491/ 2/ fid
2	Title	Edcat491
3	Comment	facturar a VCM c13_swopt MeOD {C:\ Bruker\ bacs} Bruker 24
4	Origin	Bruker BioSpin GmbH
5	Owner	Bruker
6	Site	
7	Spectrometer	spect
8	Author	
9	Solvent	MeOD
10	Temperature	300.0
11	Pulse Sequence	zgpg30
12	Number of Scans	440
13	Receiver Gain	32768
14	Relaxation Delay	1.5000
15	Pulse Width	5.3000
16	Acquisition Time	0.9044
17	Acquisition Date	2014-03-11T16:29:41
18	Modification Date	2014-03-11T16:29:00
19	Spectrometer Frequency	75.48
20	Spectral Width	18115.9
21	Lowest Frequency	-756.5
22	Nucleus	13C
23	Acquired Size	16384
24	Spectral Size	32768



4k

(^{1}H NMR spectrum in CD_3OD)

	Parámetro	Valor
1	Data File Name	C:/ Users/ Eder/ Desktop/ Química Oviedo/ RMN/ Edcat503/ 1/ fid
2	Title	Edcat503
3	Comment	facturar a VCM h1_swopt MeOD {C:\ Bruker\ bacs} Bruker 24
4	Origin	Bruker BioSpin GmbH
5	Owner	Bruker
6	Site	
7	Spectrometer	spect
8	Author	
9	Solvent	MeOD
10	Temperature	300.0
11	Pulse Sequence	zg30
12	Number of Scans	8
13	Receiver Gain	256
14	Relaxation Delay	1.0000
15	Pulse Width	14.0000
16	Acquisition Time	2.7329
17	Acquisition Date	2014-03-10T19:39:09
18	Modification Date	2014-03-10T19:39:00
19	Spectrometer Frequency	300.13
20	Spectral Width	5995.2
21	Lowest Frequency	-596.6
22	Nucleus	1H
23	Acquired Size	16384
24	Spectral Size	32768

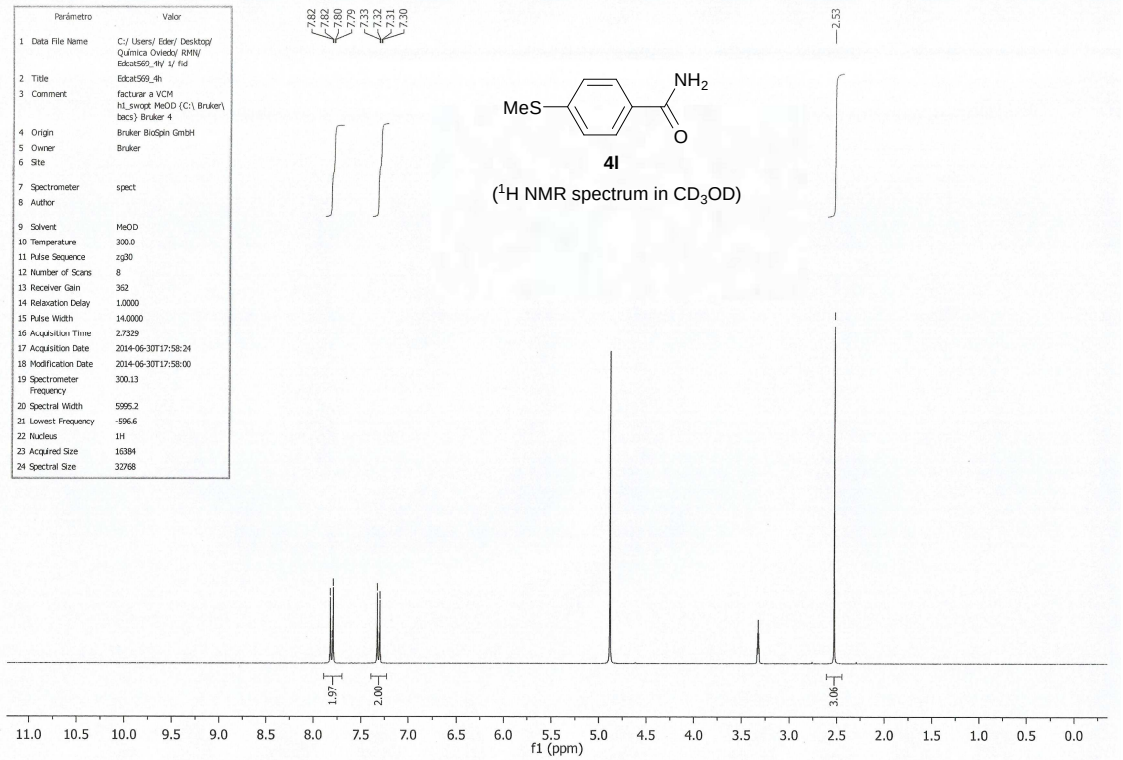


4k

($^{13}C\{^1H\}$ NMR spectrum in CD_3OD)

	Parámetro	Valor
1	Data File Name	C:/ Users/ Eder/ Desktop/ Química Oviedo/ RMN/ Edcat503/ 2/ fid
2	Title	Edcat503
3	Comment	facturar a VCM c13_swopt MeOD {C:\ Bruker\ bacs} Bruker 24
4	Origin	Bruker BioSpin GmbH
5	Owner	Bruker
6	Site	
7	Spectrometer	spect
8	Author	
9	Solvent	MeOD
10	Temperature	300.0
11	Pulse Sequence	zgpg30
12	Number of Scans	440
13	Receiver Gain	32768
14	Relaxation Delay	1.5000
15	Pulse Width	5.3000
16	Acquisition Time	0.9044
17	Acquisition Date	2014-03-10T19:58:20
18	Modification Date	2014-03-10T19:58:00
19	Spectrometer Frequency	75.48
20	Spectral Width	18115.9
21	Lowest Frequency	-756.5
22	Nucleus	13C
23	Acquired Size	16384
24	Spectral Size	32768

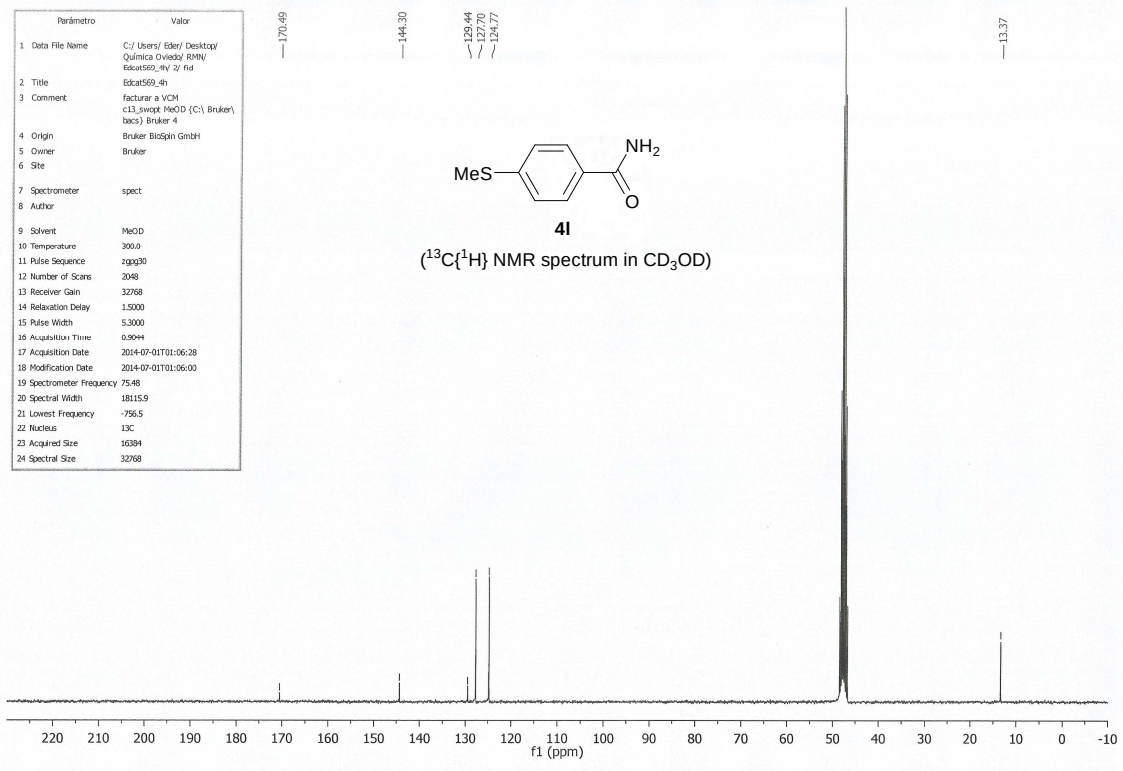
	Parámetro	Valor
1	Data File Name	C:/ Users/ Eder/ Desktop/ Química Oviedo/ RMN/ Edcat569_4h/ 1/ fid
2	Title	Edcat569_4h
3	Comment	facturar a VCM h1_swopt MeOD {C:\ Bruker\ bacs} Bruker 4
4	Origin	Bruker BioSpin GmbH
5	Owner	Bruker
6	Site	
7	Spectrometer	spect
8	Author	
9	Solvent	MeOD
10	Temperature	300.0
11	Pulse Sequence	zg30
12	Number of Scans	8
13	Receiver Gain	362
14	Relaxation Delay	1.0000
15	Pulse Width	14.0000
16	Acquisition Time	2.7329
17	Acquisition Date	2014-06-30T17:58:24
18	Modification Date	2014-06-30T17:58:00
19	Spectrometer Frequency	300.13
20	Spectral Width	5995.2
21	Lowest Frequency	-596.6
22	Nucleus	1H
23	Acquired Size	16384
24	Spectral Size	32768



4l

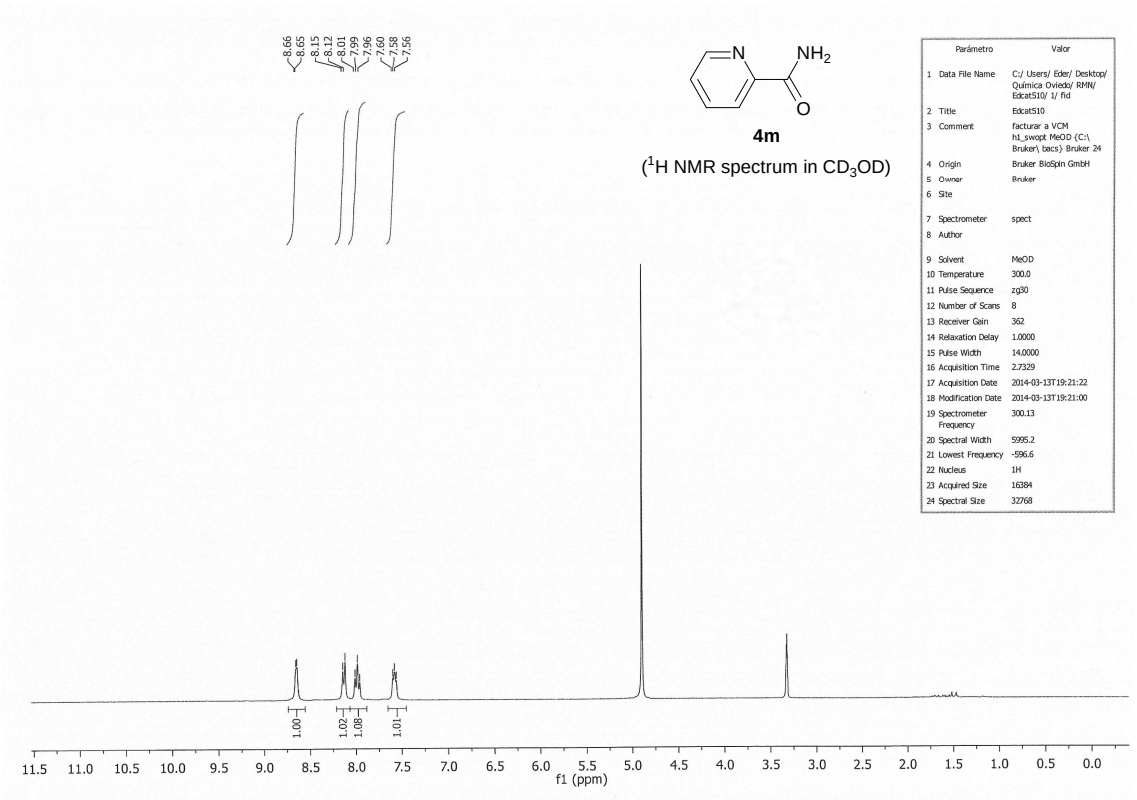
(^{1}H NMR spectrum in CD_3OD)

	Parámetro	Valor
1	Data File Name	C:/ Users/ Eder/ Desktop/ Química Oviedo/ RMN/ Edcat569_4h/ 2/ fid
2	Title	Edcat569_4h
3	Comment	facturar a VCM c13_swopt MeOD {C:\ Bruker\ bacs} Bruker 4
4	Origin	Bruker BioSpin GmbH
5	Owner	Bruker
6	Site	
7	Spectrometer	spect
8	Author	
9	Solvent	MeOD
10	Temperature	300.0
11	Pulse Sequence	zgpg30
12	Number of Scans	2048
13	Receiver Gain	32768
14	Relaxation Delay	1.5000
15	Pulse Width	5.3000
16	Acquisition Time	0.9044
17	Acquisition Date	2014-07-01T01:06:28
18	Modification Date	2014-07-01T01:06:00
19	Spectrometer Frequency	75.48
20	Spectral Width	18115.9
21	Lowest Frequency	-756.5
22	Nucleus	13C
23	Acquired Size	16384
24	Spectral Size	32768



4l

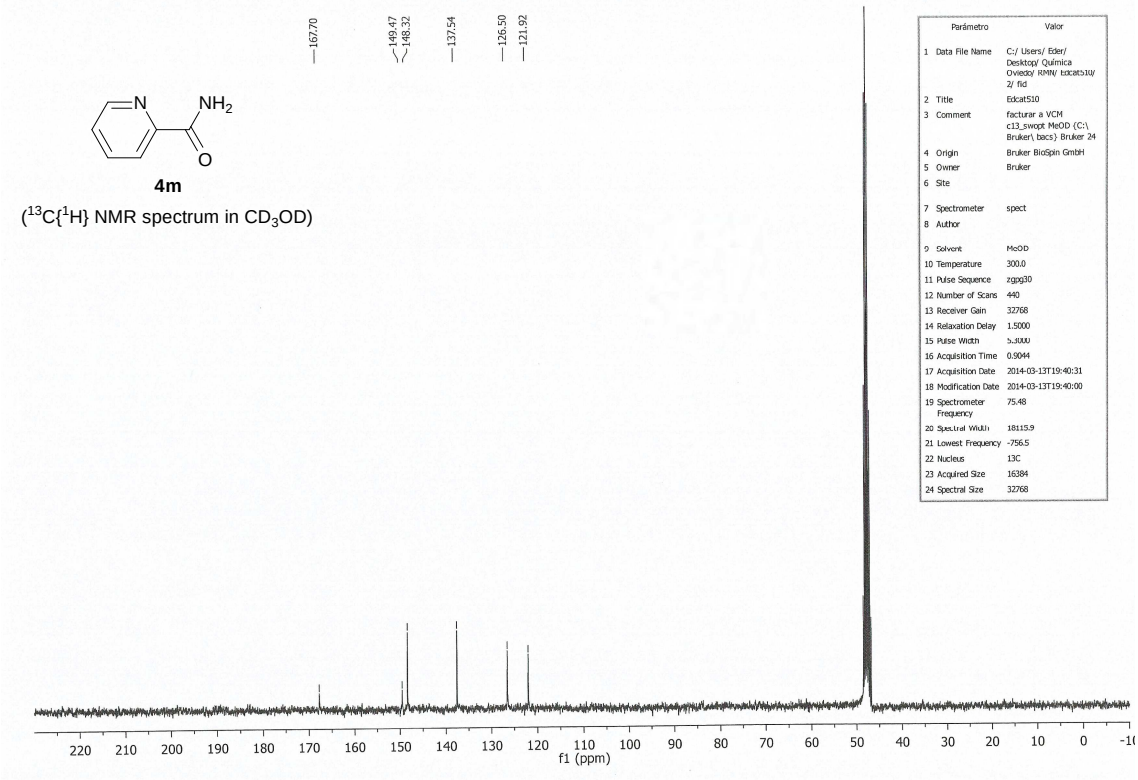
($^{13}C\{^1H\}$ NMR spectrum in CD_3OD)



4m

(^{1}H NMR spectrum in CD_3OD)

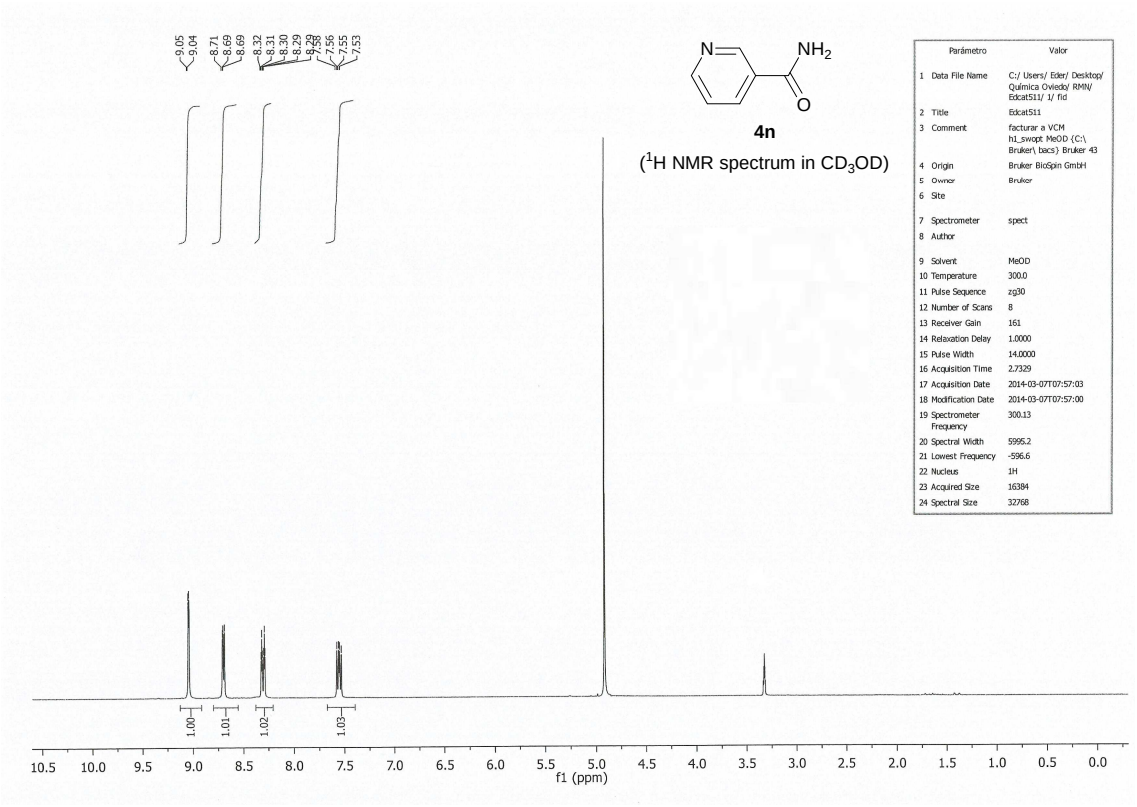
	Parámetro	Valor
1	Data File Name	C:/ Users/ Eder/ Desktop/ Química Oviedo/ RMN/ Edcat510/ 1/ fid
2	Title	Edcat510
3	Comment	facturar a VCM h1_swopt MeOD {C:\ Bruker\ bacs} Bruker 24
4	Origin	Bruker BioSpin GmbH
5	Owner	Bruker
6	Site	
7	Spectrometer	spect
8	Author	
9	Solvent	MeOD
10	Temperature	300.0
11	Pulse Sequence	zg30
12	Number of Scans	8
13	Receiver Gain	362
14	Relaxation Delay	1.0000
15	Pulse Width	14.0000
16	Acquisition Time	2.7329
17	Acquisition Date	2014-03-13T19:21:22
18	Modification Date	2014-03-13T19:21:00
19	Spectrometer Frequency	300.13
20	Spectral Width	5995.2
21	Lowest Frequency	-596.6
22	Nucleus	1H
23	Acquired Size	16384
24	Spectral Size	32768

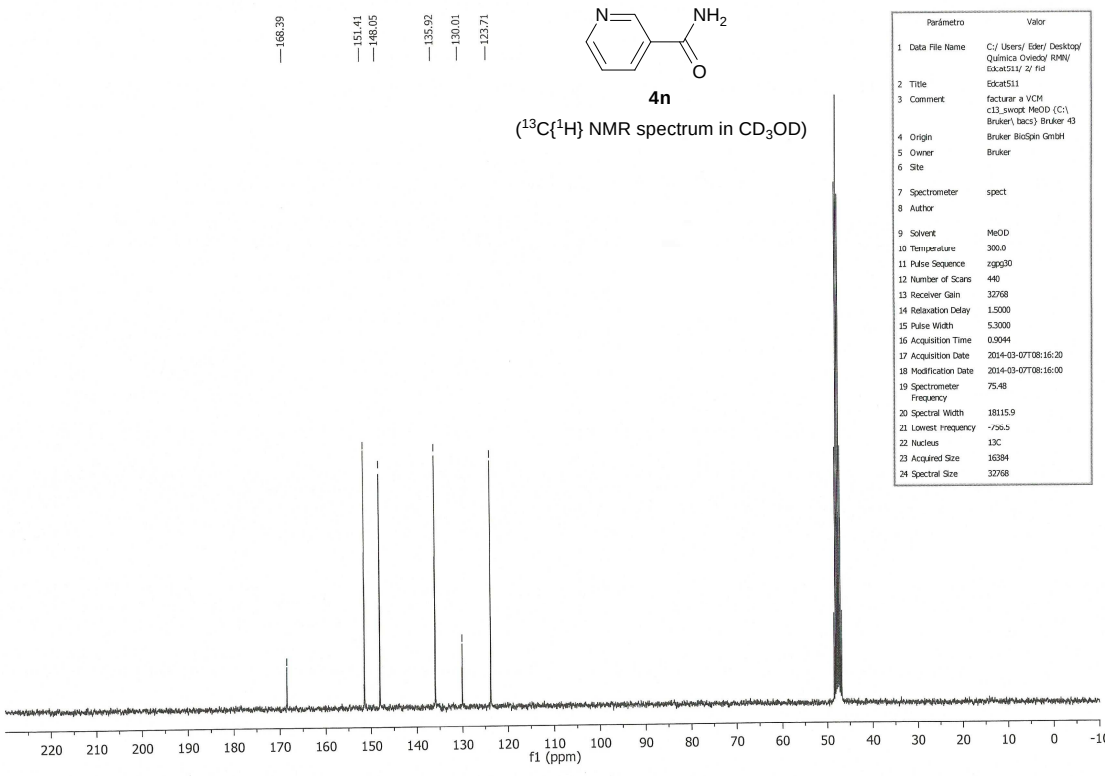


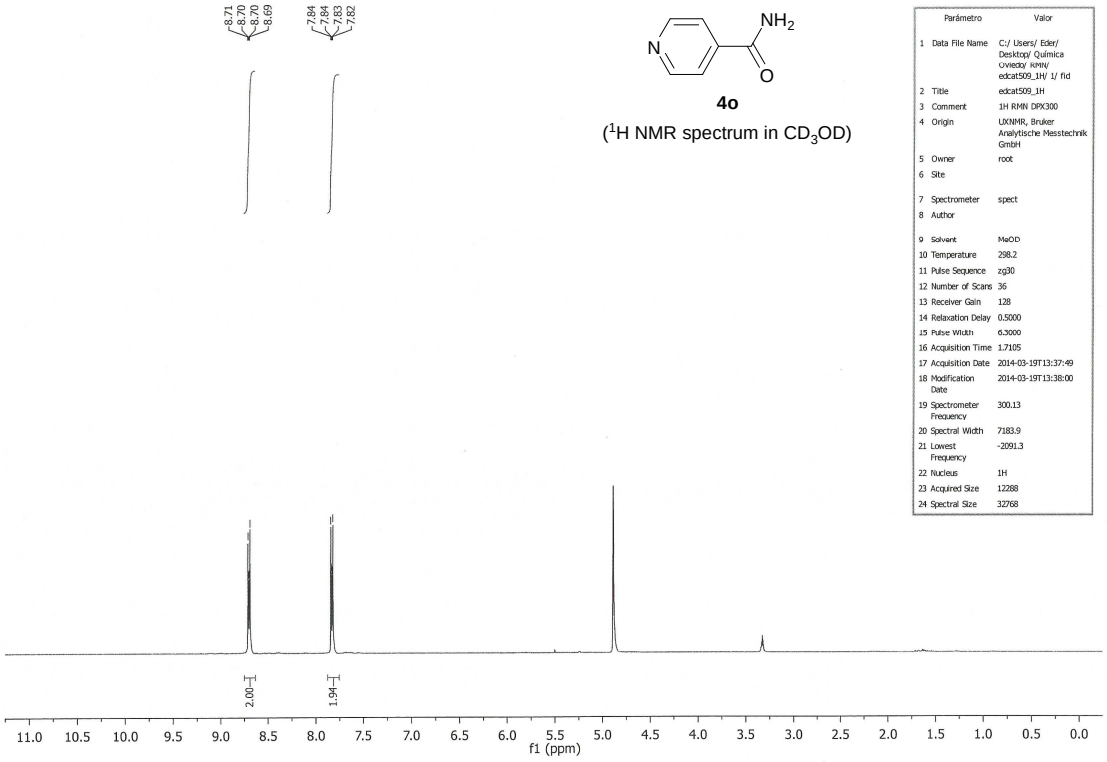
4m

($^{13}C\{^1H\}$ NMR spectrum in CD_3OD)

	Parámetro	Valor
1	Data File Name	C:/ Users/ Eder/ Desktop/ Química Oviedo/ RMN/ Edcat510/ 2/ fid
2	Title	Edcat510
3	Comment	facturar a VCM c13_swopt MeOD {C:\ Bruker\ bacs} Bruker 24
4	Origin	Bruker BioSpin GmbH
5	Owner	Bruker
6	Site	
7	Spectrometer	spect
8	Author	
9	Solvent	MeOD
10	Temperature	300.0
11	Pulse Sequence	zgpg30
12	Number of Scans	440
13	Receiver Gain	32768
14	Relaxation Delay	1.5000
15	Pulse Width	5.3000
16	Acquisition Time	0.9044
17	Acquisition Date	2014-03-13T19:40:31
18	Modification Date	2014-03-13T19:40:00
19	Spectrometer Frequency	75.48
20	Spectral Width	18115.9
21	Lowest Frequency	-756.5
22	Nucleus	13C
23	Acquired Size	16384
24	Spectral Size	32768


9.05
9.04
8.71
8.69
8.69
8.32
8.31
8.30
8.29
7.56
7.55
7.53
N
NH2
O
4n
(1H NMR spectrum in CD3OD)
Parámetro Valor
1 Data File Name C:/ Users/ Eder/ Desktop/ Química Oviedo/ RMN/ Edcat511/ 1/ fid
2 Title Edcat511
3 Comment facturar a VCM h1_swopt MeOD {C:\ Bruker\ bacs} Bruker 43
4 Origin Bruker BioSpin GmbH
5 Owner Bruker
6 Site
7 Spectrometer spect
8 Author
9 Solvent MeOD
10 Temperature 300.0
11 Pulse Sequence zg30
12 Number of Scans 8
13 Receiver Gain 161
14 Relaxation Delay 1.0000
15 Pulse Width 14.0000
16 Acquisition Time 2.7329
17 Acquisition Date 2014-03-07T07:57:03
18 Modification Date 2014-03-07T07:57:00
19 Spectrometer Frequency 300.13
20 Spectral Width 5995.2
21 Lowest Frequency -596.6
22 Nucleus 1H
23 Acquired Size 16384
24 Spectral Size 32768
1.00
1.01
1.02
1.03
f1 (ppm)

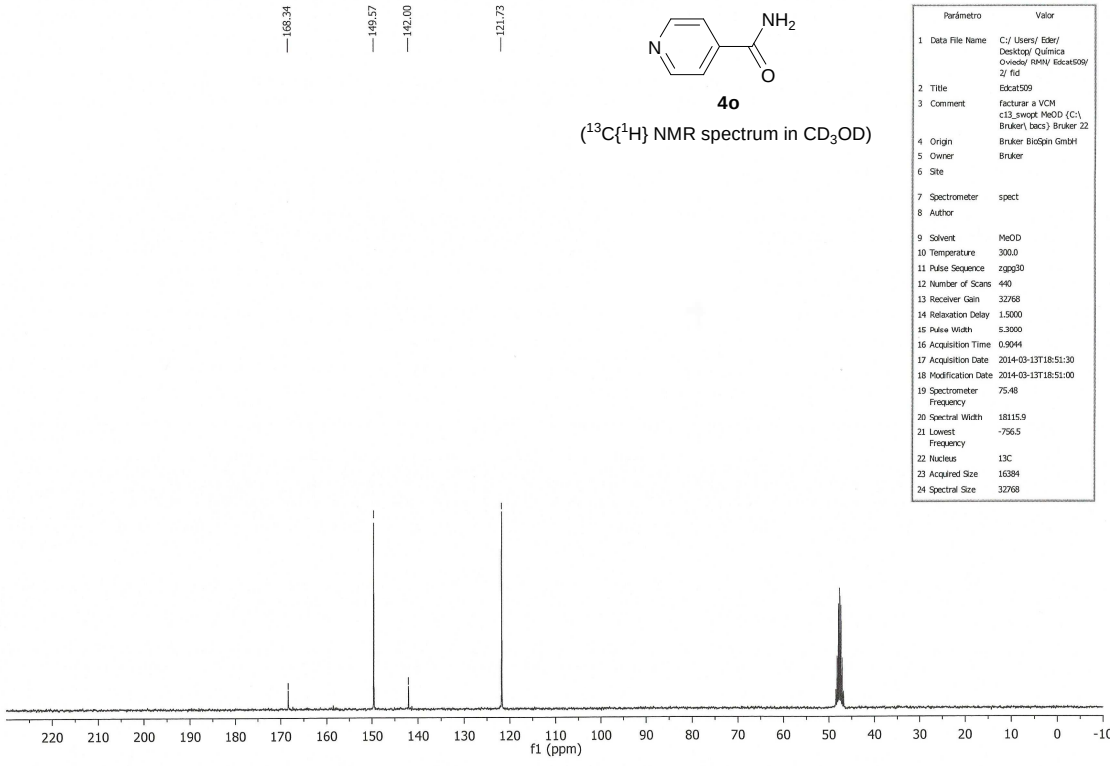

168.39
151.41
148.05
135.92
130.01
123.71
N
NH2
O
4n
(13C{1H} NMR spectrum in CD3OD)
Parámetro Valor
1 Data File Name C:/ Users/ Eder/ Desktop/ Química Oviedo/ RMN/ Edcat511/ 2/ fid
2 Title Edcat511
3 Comment facturar a VCM c13_swopt MeOD {C:\ Bruker\ bacs} Bruker 43
4 Origin Bruker BioSpin GmbH
5 Owner Bruker
6 Site
7 Spectrometer spect
8 Author
9 Solvent MeOD
10 Temperature 300.0
11 Pulse Sequence zgpg30
12 Number of Scans 440
13 Receiver Gain 32768
14 Relaxation Delay 1.5000
15 Pulse Width 5.3000
16 Acquisition Time 0.9044
17 Acquisition Date 2014-03-07T08:16:20
18 Modification Date 2014-03-07T08:16:00
19 Spectrometer Frequency 75.48
20 Spectral Width 18115.9
21 Lowest Frequency -756.5
22 Nucleus 13C
23 Acquired Size 16384
24 Spectral Size 32768
f1 (ppm)



4o

(^{1}H NMR spectrum in CD_3OD)

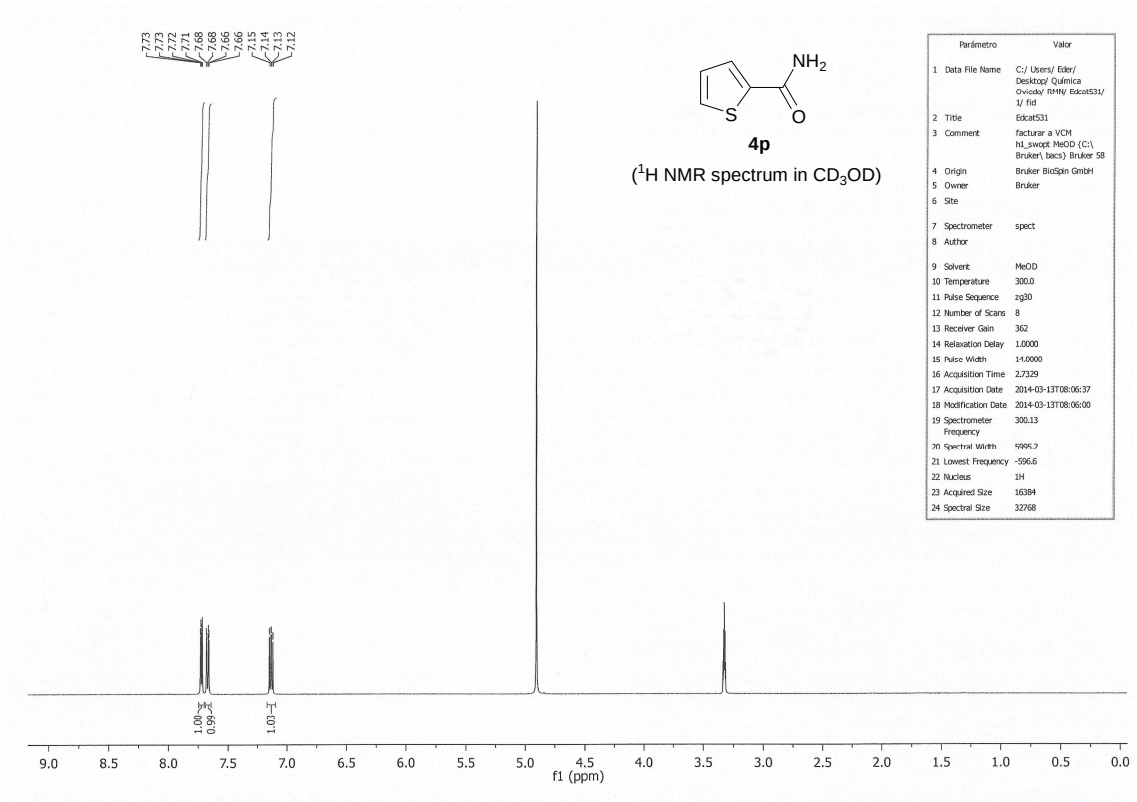
	Parámetro	Valor
1	Data File Name	C:/ Users/ Eder/ Desktop/ Química Oviedo/ RMN/ edcat509_1H/ 1/ fid
2	Title	edcat509_1H
3	Comment	1H RMN DPX300
4	Origin	UXNMR, Bruker Analytische Messtechnik GmbH
5	Owner	root
6	Site	
7	Spectrometer	spect
8	Author	
9	Solvent	MeOD
10	Temperature	298.2
11	Pulse Sequence	zg30
12	Number of Scans	36
13	Receiver Gain	128
14	Relaxation Delay	0.5000
15	Pulse Width	6.3000
16	Acquisition Time	1.7105
17	Acquisition Date	2014-03-19T13:37:49
18	Modification Date	2014-03-19T13:38:00
19	Spectrometer Frequency	300.13
20	Spectral Width	7183.9
21	Lowest Frequency	-2091.3
22	Nucleus	1H
23	Acquired Size	12288
24	Spectral Size	32768



4o

($^{13}C\{^1H\}$ NMR spectrum in CD_3OD)

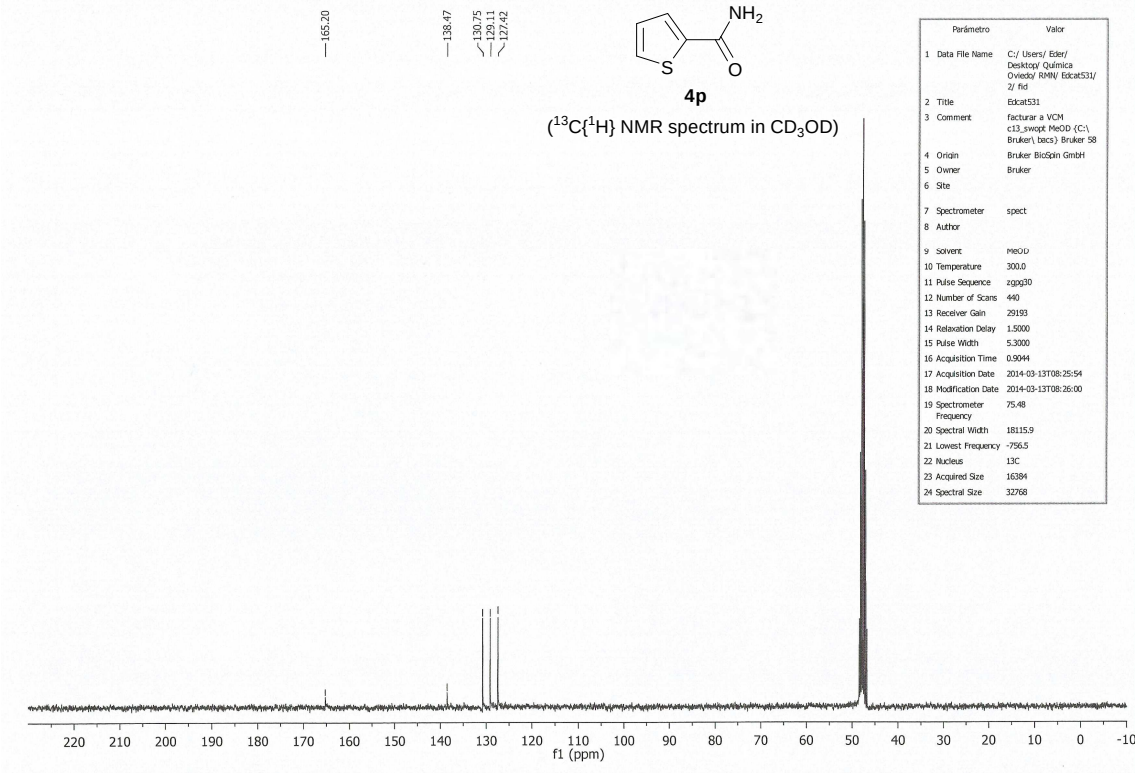
	Parámetro	Valor
1	Data File Name	C:/ Users/ Eder/ Desktop/ Química Oviedo/ RMN/ Edcat509/ 2/ fid
2	Title	Edcat509
3	Comment	facturar a VCM c13_swopt MeOD (C:\ Bruker\ bacs) Bruker 22
4	Origin	Bruker BioSpin GmbH
5	Owner	Bruker
6	Site	
7	Spectrometer	spect
8	Author	
9	Solvent	MeOD
10	Temperature	300.0
11	Pulse Sequence	zgpg30
12	Number of Scans	440
13	Receiver Gain	32768
14	Relaxation Delay	1.5000
15	Pulse Width	5.3000
16	Acquisition Time	0.9044
17	Acquisition Date	2014-03-13T18:51:30
18	Modification Date	2014-03-13T18:51:00
19	Spectrometer Frequency	75.48
20	Spectral Width	18115.9
21	Lowest Frequency	-756.5
22	Nucleus	13C
23	Acquired Size	16384
24	Spectral Size	32768



4p

(^{1}H NMR spectrum in CD_3OD)

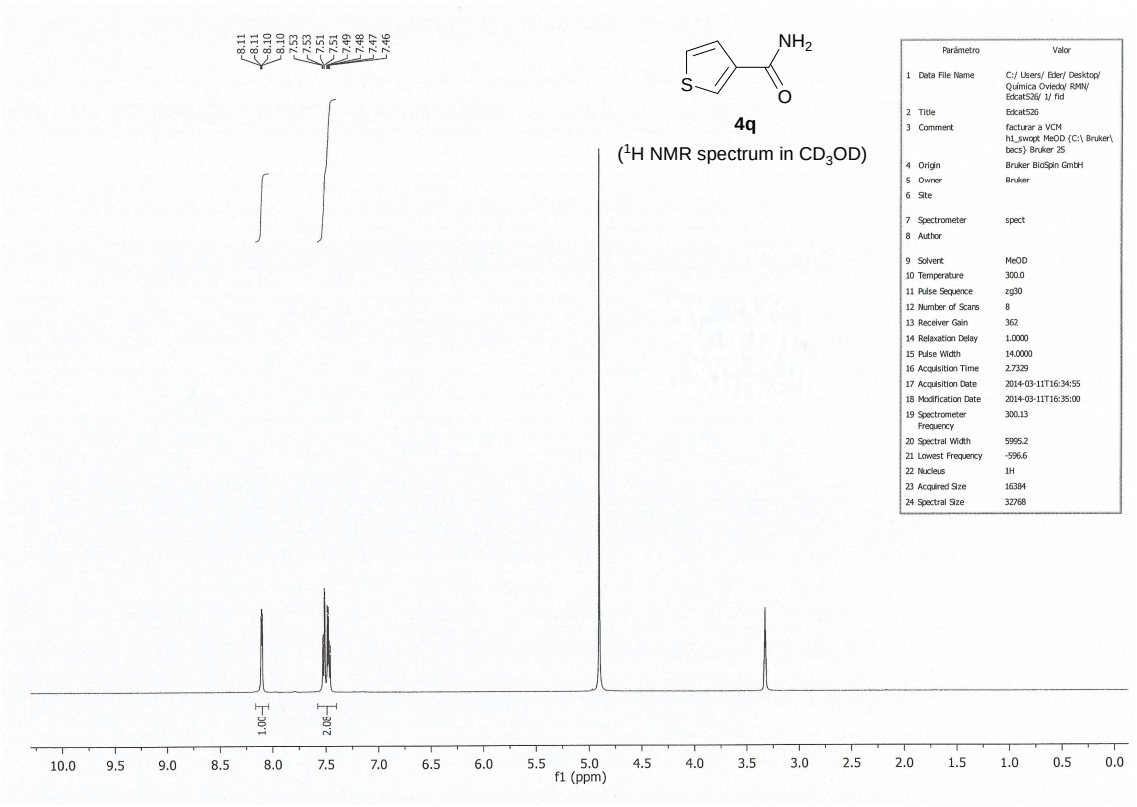
	Parámetro	Valor
1	Data File Name	C:/ Users/ Eder/ Desktop/ Química Oviedo/ RMN/ Edcat531/ 1/ fid
2	Title	Edcat531
3	Comment	facturar a VCM h1_swopt MeOD {C:\ Bruker\ bacs} Bruker 58
4	Origin	Bruker BioSpin GmbH
5	Owner	Bruker
6	Site	
7	Spectrometer	spect
8	Author	
9	Solvent	MeOD
10	Temperature	300.0
11	Pulse Sequence	zg30
12	Number of Scans	8
13	Receiver Gain	362
14	Relaxation Delay	1.0000
15	Pulse Width	14.0000
16	Acquisition Time	2.7329
17	Acquisition Date	2014-03-13T08:06:37
18	Modification Date	2014-03-13T08:06:00
19	Spectrometer Frequency	300.13
20	Spectral Width	5995.2
21	Lowest Frequency	-596.6
22	Nucleus	1H
23	Acquired Size	16384
24	Spectral Size	32768



4p

($^{13}C\{^1H\}$ NMR spectrum in CD_3OD)

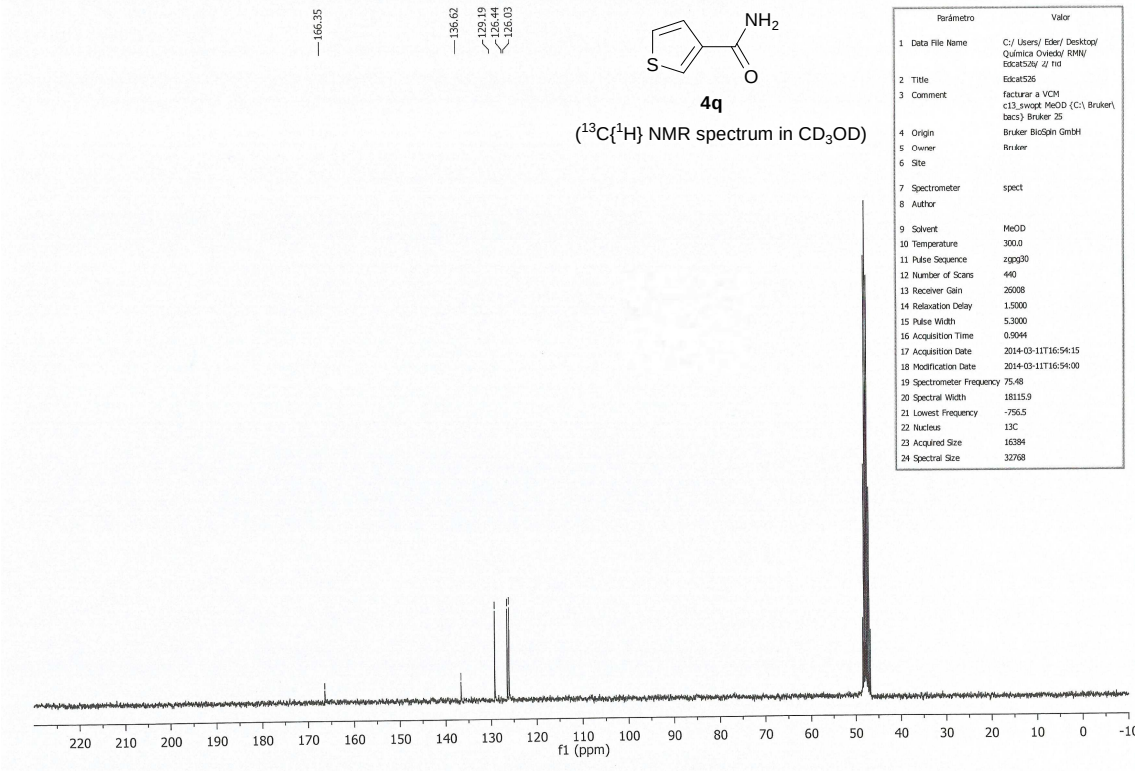
	Parámetro	Valor
1	Data File Name	C:/ Users/ Eder/ Desktop/ Química Oviedo/ RMN/ Edcat531/ 2/ fid
2	Title	Edcat531
3	Comment	facturar a VCM c13_swopt MeOD {C:\ Bruker\ bacs} Bruker 58
4	Origin	Bruker BioSpin GmbH
5	Owner	Bruker
6	Site	
7	Spectrometer	spect
8	Author	
9	Solvent	MeOD
10	Temperature	300.0
11	Pulse Sequence	zgpg30
12	Number of Scans	440
13	Receiver Gain	29193
14	Relaxation Delay	1.5000
15	Pulse Width	5.3000
16	Acquisition Time	0.9044
17	Acquisition Date	2014-03-13T08:25:54
18	Modification Date	2014-03-13T08:26:00
19	Spectrometer Frequency	75.48
20	Spectral Width	18115.9
21	Lowest Frequency	-756.5
22	Nucleus	13C
23	Acquired Size	16384
24	Spectral Size	32768



4q

(^{1}H NMR spectrum in CD_3OD)

	Parámetro	Valor
1	Data File Name	C:/ Users/ Eder/ Desktop/ Química Oviedo/ RMN/ Edcat526/ 1/ fid
2	Title	Edcat526
3	Comment	facturar a VCM h1_swopt MeOD {C:\ Bruker\ bacs} Bruker 25
4	Origin	Bruker BioSpin GmbH
5	Owner	Bruker
6	Site	
7	Spectrometer	spect
8	Author	
9	Solvent	MeOD
10	Temperature	300.0
11	Pulse Sequence	zg30
12	Number of Scans	8
13	Receiver Gain	362
14	Relaxation Delay	1.0000
15	Pulse Width	14.0000
16	Acquisition Time	2.7329
17	Acquisition Date	2014-03-11T16:34:55
18	Modification Date	2014-03-11T16:35:00
19	Spectrometer Frequency	300.13
20	Spectral Width	5995.2
21	Lowest Frequency	-596.6
22	Nucleus	1H
23	Acquired Size	16384
24	Spectral Size	32768

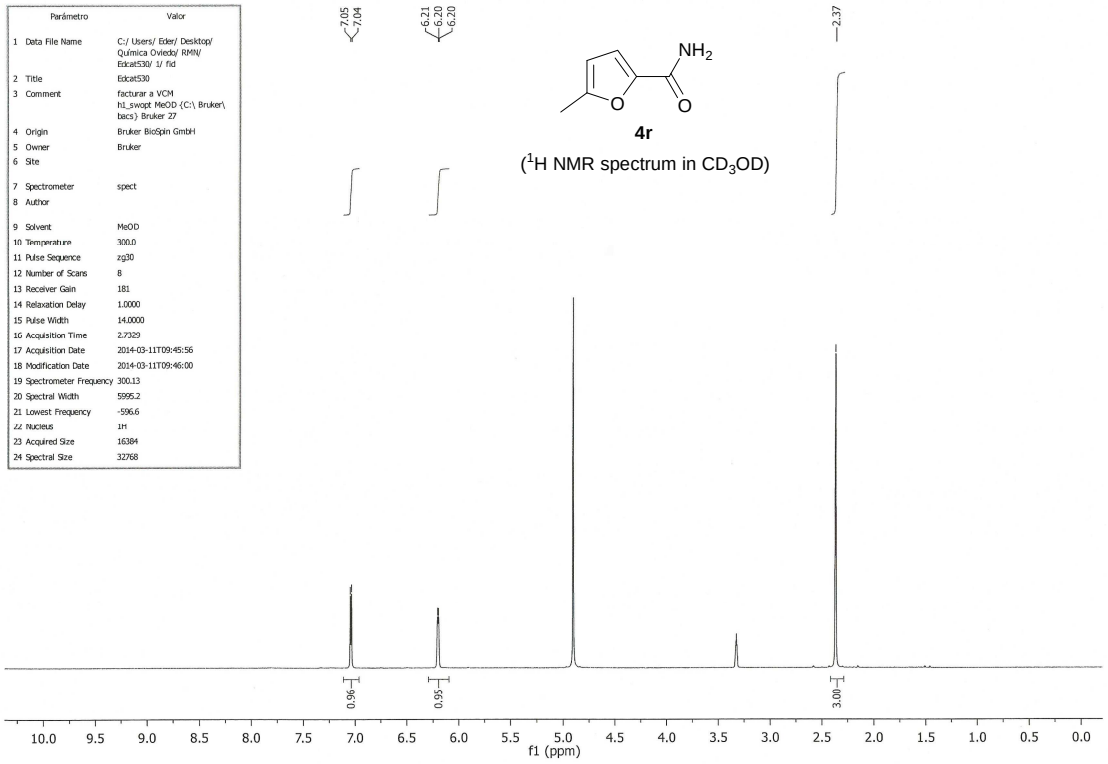


4q

($^{13}C\{^1H\}$ NMR spectrum in CD_3OD)

	Parámetro	Valor
1	Data File Name	C:/ Users/ Eder/ Desktop/ Química Oviedo/ RMN/ Edcat526/ 2/ fid
2	Title	Edcat526
3	Comment	facturar a VCM c13_swopt MeOD {C:\ Bruker\ bacs} Bruker 25
4	Origin	Bruker BioSpin GmbH
5	Owner	Bruker
6	Site	
7	Spectrometer	spect
8	Author	
9	Solvent	MeOD
10	Temperature	300.0
11	Pulse Sequence	zgpg30
12	Number of Scans	440
13	Receiver Gain	26008
14	Relaxation Delay	1.5000
15	Pulse Width	5.3000
16	Acquisition Time	0.9044
17	Acquisition Date	2014-03-11T16:54:15
18	Modification Date	2014-03-11T16:54:00
19	Spectrometer Frequency	75.48
20	Spectral Width	18115.9
21	Lowest Frequency	-756.5
22	Nucleus	13C
23	Acquired Size	16384
24	Spectral Size	32768

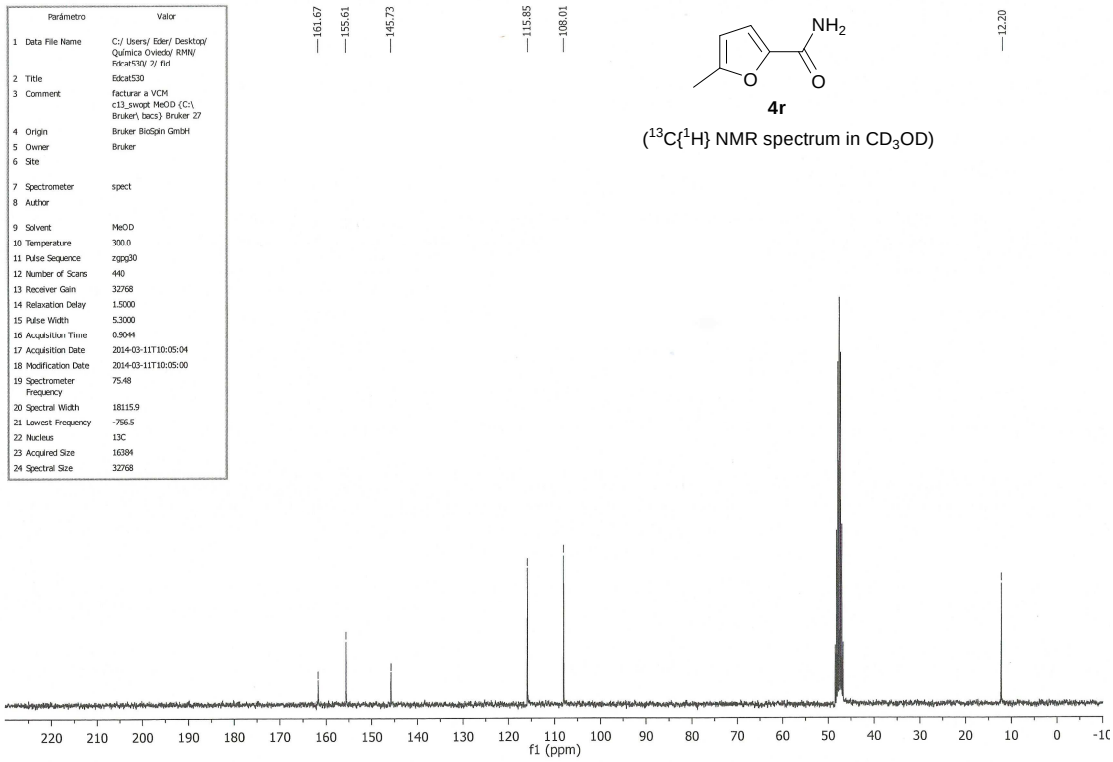
Parámetro	Valor
1 Data File Name	C:/ Users/ Eder/ Desktop/ Química Oviedo/ RMN/ Edcat530/ 1/ fid
2 Title	Edcat530
3 Comment	facturar a VCM h1_swopt MeOD {C:\ Bruker\ bacs} Bruker 27
4 Origin	Bruker BioSpin GmbH
5 Owner	Bruker
6 Site	
7 Spectrometer	spect
8 Author	
9 Solvent	MeOD
10 Temperature	300.0
11 Pulse Sequence	zg30
12 Number of Scans	8
13 Receiver Gain	181
14 Relaxation Delay	1.0000
15 Pulse Width	14.0000
16 Acquisition Time	2.7329
17 Acquisition Date	2014-03-11T09:45:56
18 Modification Date	2014-03-11T09:46:00
19 Spectrometer Frequency	300.13
20 Spectral Width	5995.2
21 Lowest Frequency	-596.6
22 Nucleus	1H
23 Acquired Size	16384
24 Spectral Size	32768



4r

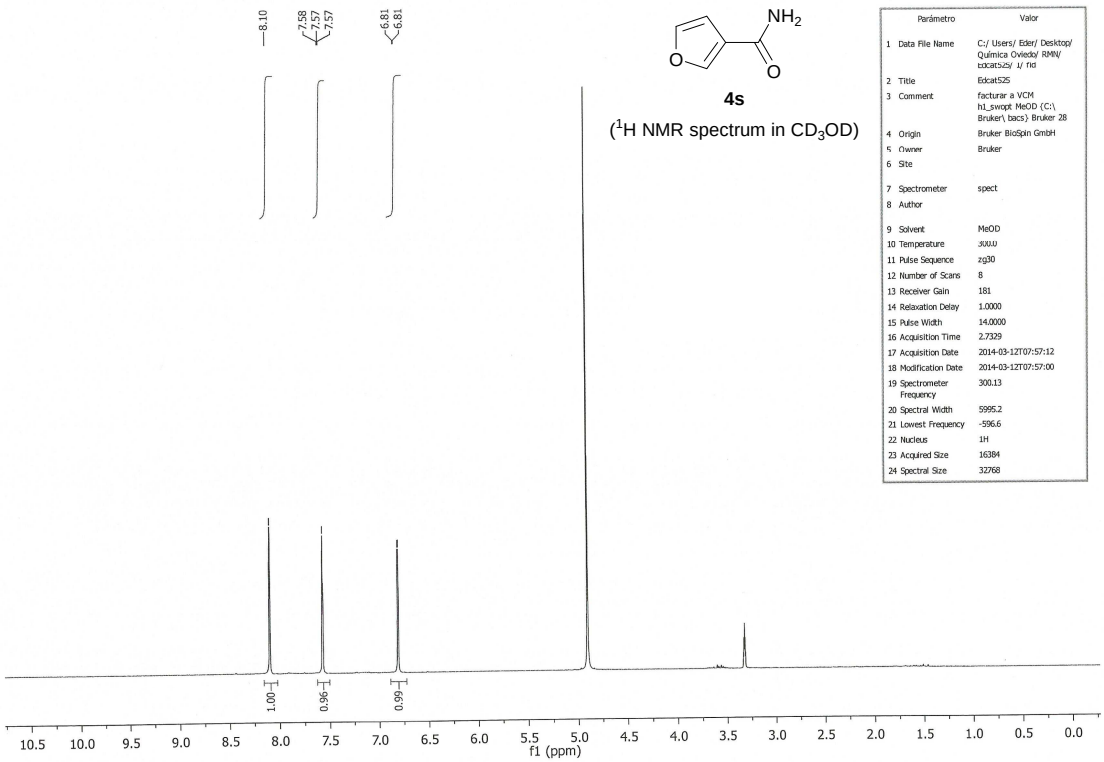
(^{1}H NMR spectrum in CD_3OD)

Parámetro	Valor
1 Data File Name	C:/ Users/ Eder/ Desktop/ Química Oviedo/ RMN/ Edcat530/ 2/ fid
2 Title	Edcat530
3 Comment	facturar a VCM c13_swopt MeOD {C:\ Bruker\ bacs} Bruker 27
4 Origin	Bruker BioSpin GmbH
5 Owner	Bruker
6 Site	
7 Spectrometer	spect
8 Author	
9 Solvent	MeOD
10 Temperature	300.0
11 Pulse Sequence	zgpg30
12 Number of Scans	440
13 Receiver Gain	32768
14 Relaxation Delay	1.5000
15 Pulse Width	5.3000
16 Acquisition Time	0.9044
17 Acquisition Date	2014-03-11T10:05:04
18 Modification Date	2014-03-11T10:05:00
19 Spectrometer Frequency	75.48
20 Spectral Width	18115.9
21 Lowest Frequency	-756.5
22 Nucleus	13C
23 Acquired Size	16384
24 Spectral Size	32768



4r

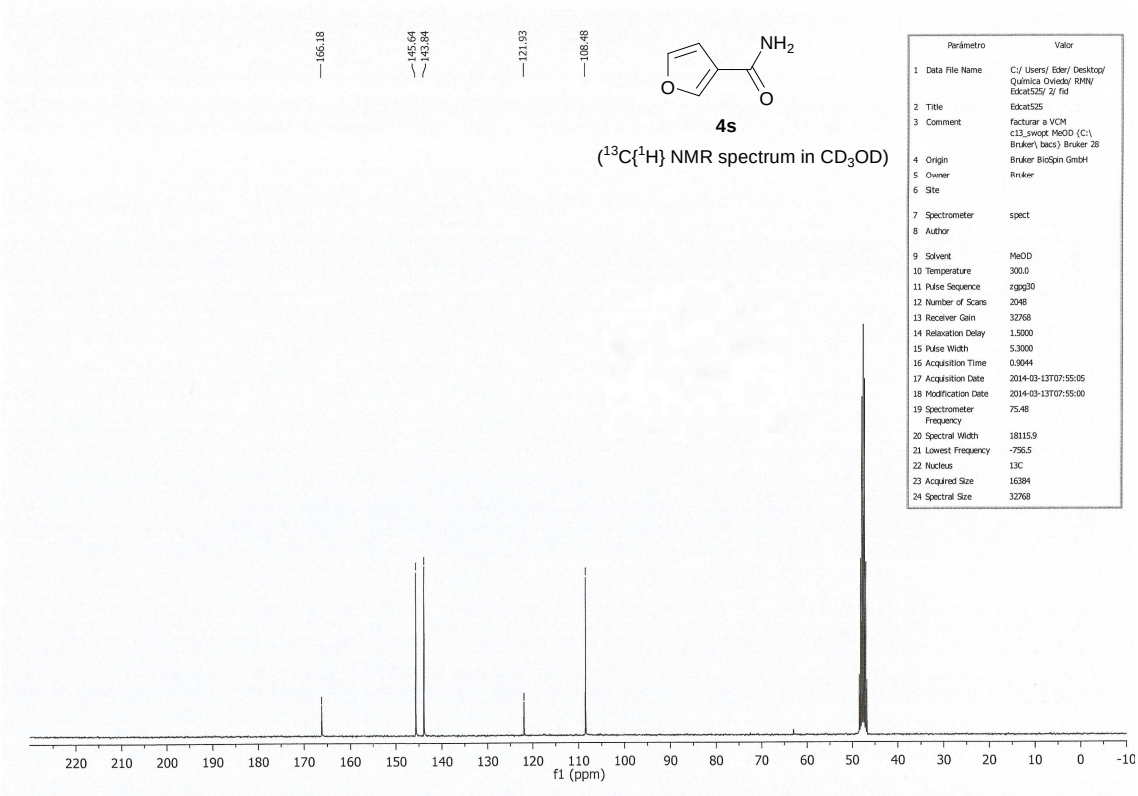
($^{13}C\{^1H\}$ NMR spectrum in CD_3OD)



4s

(^{1}H NMR spectrum in CD_3OD)

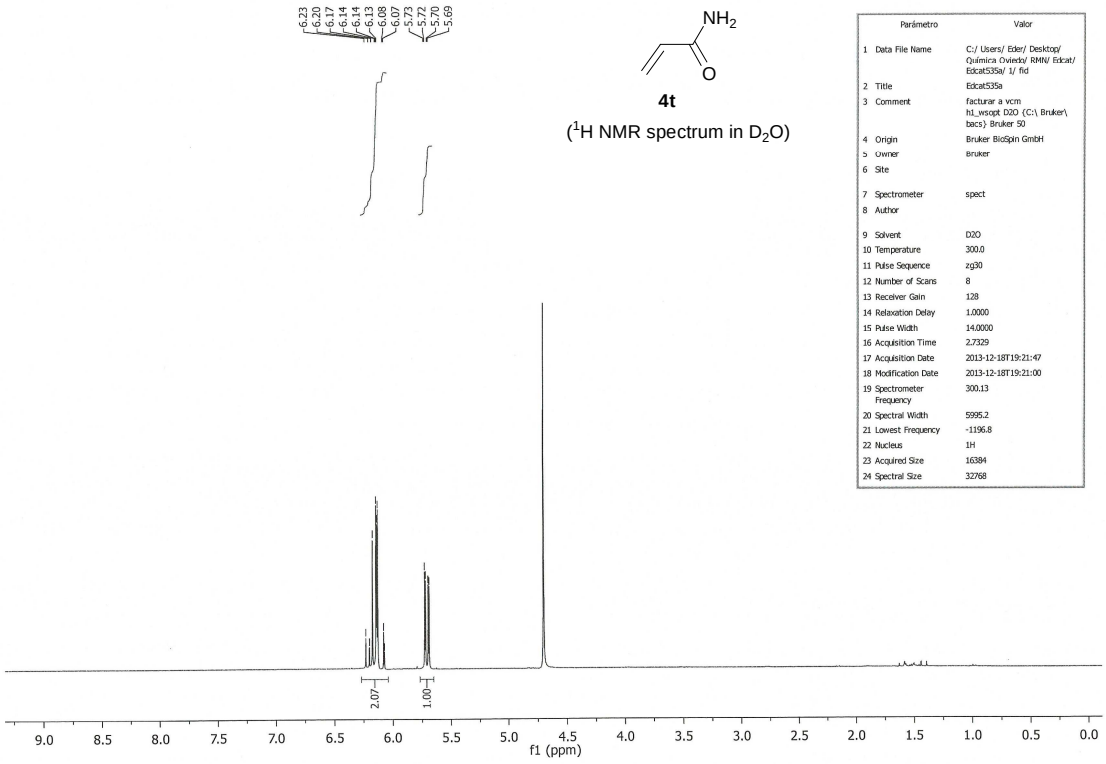
	Parámetro	Valor
1	Data File Name	C:/ Users/ Eder/ Desktop/ Química Oviedo/ RMN/ Edcat525/ 1/ fid
2	Title	Edcat525
3	Comment	facturar a VCM h1_swopt MeOD {C:\ Bruker\ bacs} Bruker 28
4	Origin	Bruker BioSpin GmbH
5	Owner	Bruker
6	Site	
7	Spectrometer	spect
8	Author	
9	Solvent	MeOD
10	Temperature	300.0
11	Pulse Sequence	zg30
12	Number of Scans	8
13	Receiver Gain	181
14	Relaxation Delay	1.0000
15	Pulse Width	14.0000
16	Acquisition Time	2.7329
17	Acquisition Date	2014-03-12T07:57:12
18	Modification Date	2014-03-12T07:57:00
19	Spectrometer Frequency	300.13
20	Spectral Width	5995.2
21	Lowest Frequency	-596.6
22	Nucleus	1H
23	Acquired Size	16384
24	Spectral Size	32768



4s

($^{13}C\{^1H\}$ NMR spectrum in CD_3OD)

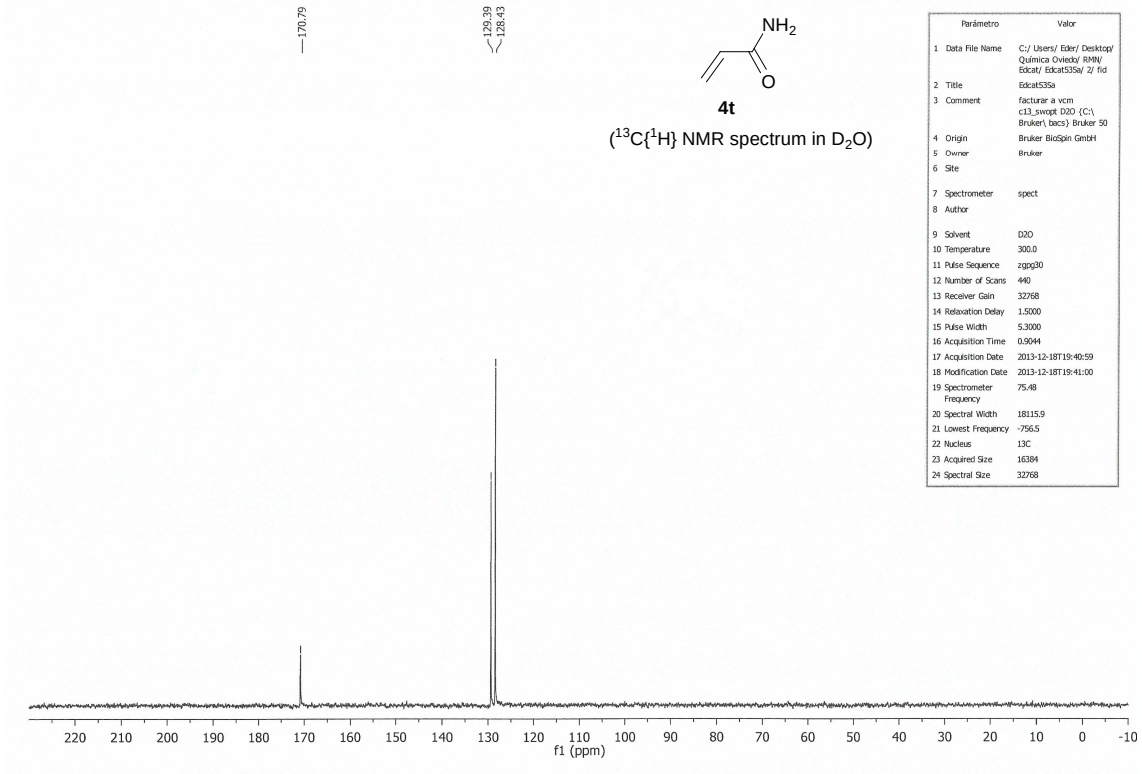
	Parámetro	Valor
1	Data File Name	C:/ Users/ Eder/ Desktop/ Química Oviedo/ RMN/ Edcat525/ 2/ fid
2	Title	Edcat525
3	Comment	facturar a VCM c13_swopt MeOD {C:\ Bruker\ bacs} Bruker 28
4	Origin	Bruker BioSpin GmbH
5	Owner	Bruker
6	Site	
7	Spectrometer	spect
8	Author	
9	Solvent	MeOD
10	Temperature	300.0
11	Pulse Sequence	zgpg30
12	Number of Scans	2048
13	Receiver Gain	32768
14	Relaxation Delay	1.5000
15	Pulse Width	5.3000
16	Acquisition Time	0.9044
17	Acquisition Date	2014-03-13T07:55:05
18	Modification Date	2014-03-13T07:55:00
19	Spectrometer Frequency	75.48
20	Spectral Width	18115.9
21	Lowest Frequency	-756.5
22	Nucleus	13C
23	Acquired Size	16384
24	Spectral Size	32768



4t

(^{1}H NMR spectrum in D_2O)

	Parámetro	Valor
1	Data File Name	C:/ Users/ Eder/ Desktop/ Química Oviedo/ RMN/ Edcat/ Edcat535a/ 1/ fid
2	Title	Edcat535a
3	Comment	facturar a vcm h1_wsopt D2O {C:\ Bruker\ bacs} Bruker 50
4	Origin	Bruker BioSpin GmbH
5	Owner	Bruker
6	Site	
7	Spectrometer	spect
8	Author	
9	Solvent	D2O
10	Temperature	300.0
11	Pulse Sequence	zg30
12	Number of Scans	8
13	Receiver Gain	128
14	Relaxation Delay	1.0000
15	Pulse Width	14.0000
16	Acquisition Time	2.7329
17	Acquisition Date	2013-12-18T19:21:47
18	Modification Date	2013-12-18T19:21:00
19	Spectrometer Frequency	300.13
20	Spectral Width	5995.2
21	Lowest Frequency	-1196.8
22	Nucleus	1H
23	Acquired Size	16384
24	Spectral Size	32768



4t

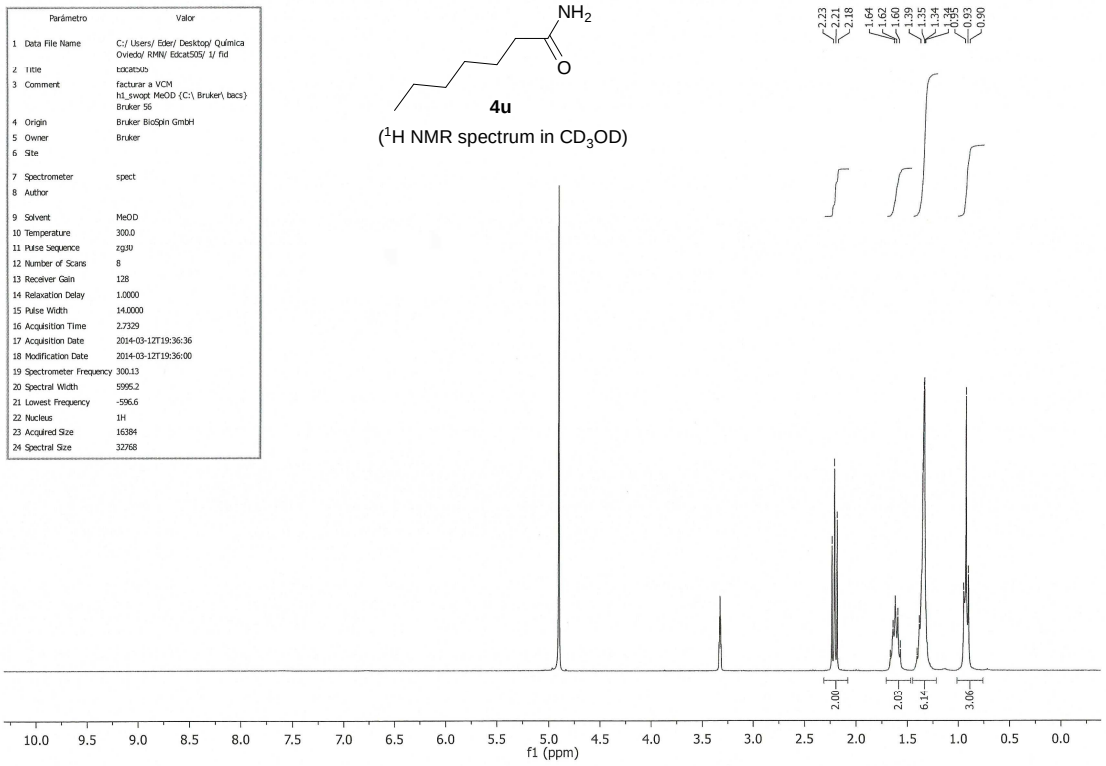
($^{13}C\{^1H\}$ NMR spectrum in D_2O)

	Parámetro	Valor
1	Data File Name	C:/ Users/ Eder/ Desktop/ Química Oviedo/ RMN/ Edcat/ Edcat535a/ 2/ fid
2	Title	Edcat535a
3	Comment	facturar a vcm c13_swopt D2O {C:\ Bruker\ bacs} Bruker 50
4	Origin	Bruker BioSpin GmbH
5	Owner	Bruker
6	Site	
7	Spectrometer	spect
8	Author	
9	Solvent	D2O
10	Temperature	300.0
11	Pulse Sequence	zgpg30
12	Number of Scans	440
13	Receiver Gain	32768
14	Relaxation Delay	1.5000
15	Pulse Width	5.3000
16	Acquisition Time	0.9044
17	Acquisition Date	2013-12-18T19:40:59
18	Modification Date	2013-12-18T19:41:00
19	Spectrometer Frequency	75.48
20	Spectral Width	18115.9
21	Lowest Frequency	-756.5
22	Nucleus	13C
23	Acquired Size	16384
24	Spectral Size	32768

	Parámetro	Valor
1	Data File Name	C:/ Users/ Eder/ Desktop/ Química Oviedo/ RMN/ Edcat505/ 1/ fid
2	Title	edcat5Ub
3	Comment	facturar a VCM h1_swopt MeOD {C:\ Bruker\ bacs} Bruker 56
4	Origin	Bruker BioSpin GmbH
5	Owner	Bruker
6	Site	
7	Spectrometer	spect
8	Author	
9	Solvent	MeOD
10	Temperature	300.0
11	Pulse Sequence	zg30
12	Number of Scans	8
13	Receiver Gain	128
14	Relaxation Delay	1.0000
15	Pulse Width	14.0000
16	Acquisition Time	2.7329
17	Acquisition Date	2014-03-12T19:36:36
18	Modification Date	2014-03-12T19:36:00
19	Spectrometer Frequency	300.13
20	Spectral Width	5995.2
21	Lowest Frequency	-596.6
22	Nucleus	1H
23	Acquired Size	16384
24	Spectral Size	32768

4u

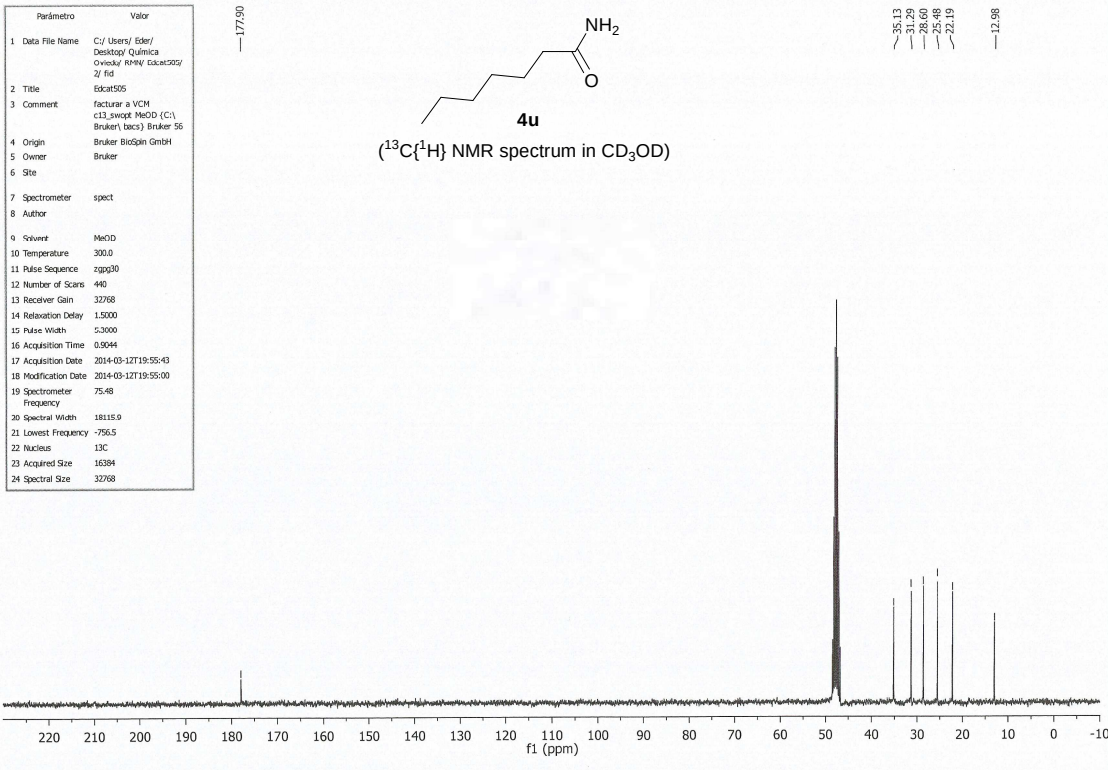
(^{1}H NMR spectrum in CD_3OD)



	Parámetro	Valor
1	Data File Name	C:/ Users/ Eder/ Desktop/ Química Oviedo/ RMN/ Edcat505/ 2/ fid
2	Title	Edcat505
3	Comment	facturar a VCM c13_swopt MeOD {C:\ Bruker\ bacs} Bruker 56
4	Origin	Bruker BioSpin GmbH
5	Owner	Bruker
6	Site	
7	Spectrometer	spect
8	Author	
9	Solvent	MeOD
10	Temperature	300.0
11	Pulse Sequence	zgpg30
12	Number of Scans	440
13	Receiver Gain	32768
14	Relaxation Delay	1.5000
15	Pulse Width	5.3000
16	Acquisition Time	0.9044
17	Acquisition Date	2014-03-12T19:55:43
18	Modification Date	2014-03-12T19:55:00
19	Spectrometer Frequency	75.48
20	Spectral Width	18115.9
21	Lowest Frequency	-756.5
22	Nucleus	13C
23	Acquired Size	16384
24	Spectral Size	32768

4u

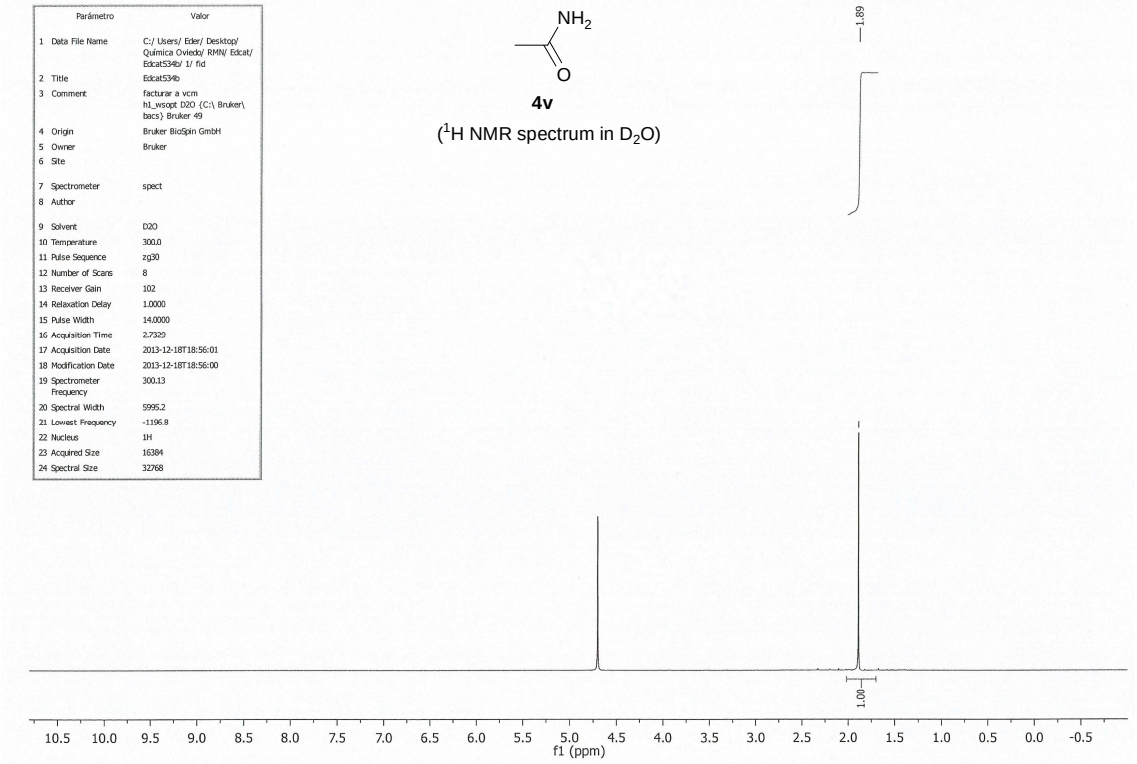
($^{13}C\{^1H\}$ NMR spectrum in CD_3OD)



	Parámetro	Valor
1	Data File Name	C:/ Users/ Eder/ Desktop/ Química Oviedo/ RMN/ Edcat/ Edcat534b/ 1/ fid
2	Title	Edcat534b
3	Comment	facturar a vcm h1_wsopt D2O {C:\ Bruker\ bacs} Bruker 49
4	Origin	Bruker BioSpin GmbH
5	Owner	Bruker
6	Site	
7	Spectrometer	spect
8	Author	
9	Solvent	D2O
10	Temperature	300.0
11	Pulse Sequence	zg30
12	Number of Scans	8
13	Receiver Gain	102
14	Relaxation Delay	1.0000
15	Pulse Width	14.0000
16	Acquisition Time	2.7329
17	Acquisition Date	2013-12-18T18:56:01
18	Modification Date	2013-12-18T18:56:00
19	Spectrometer Frequency	300.13
20	Spectral Width	5995.2
21	Lowest Frequency	-1196.8
22	Nucleus	1H
23	Acquired Size	16384
24	Spectral Size	32768

4v

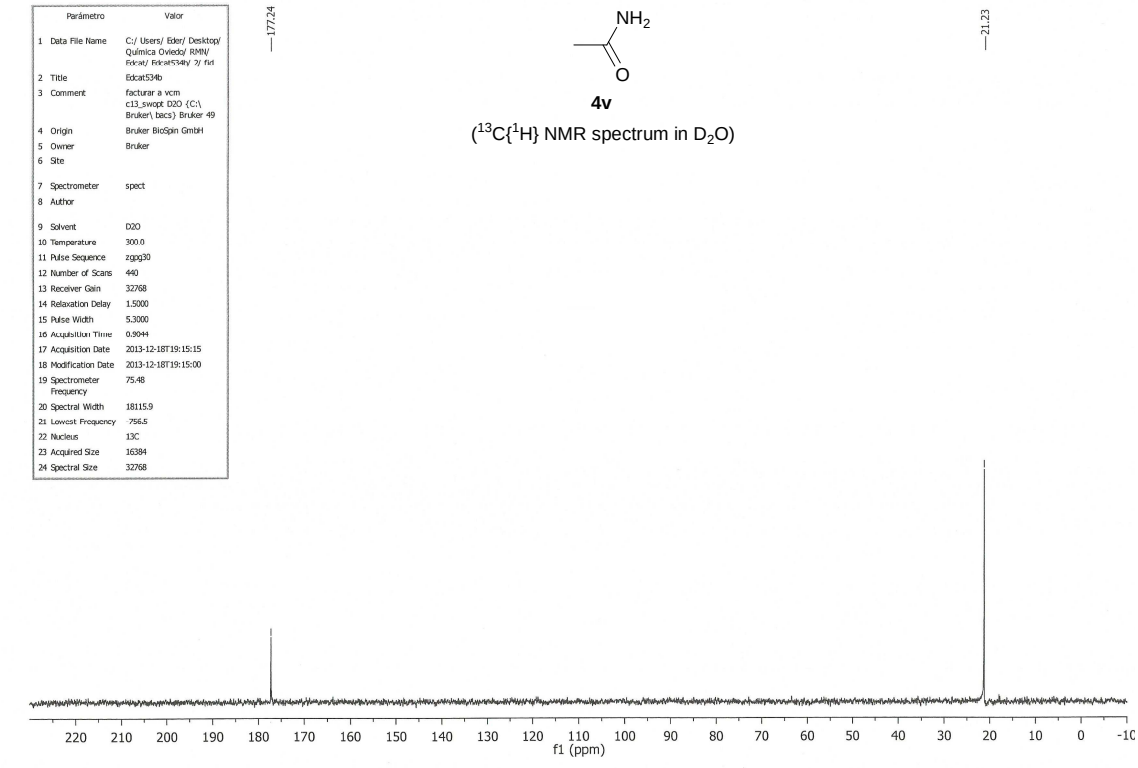
(^{1}H NMR spectrum in D_2O)

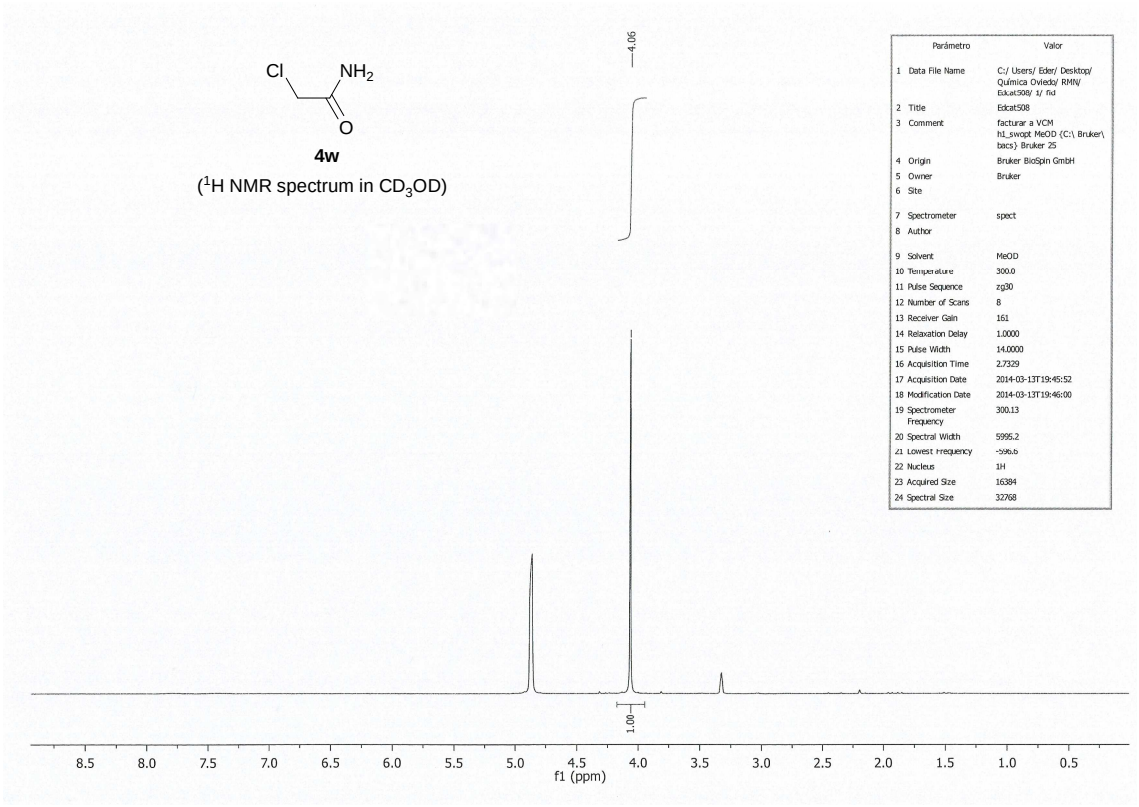


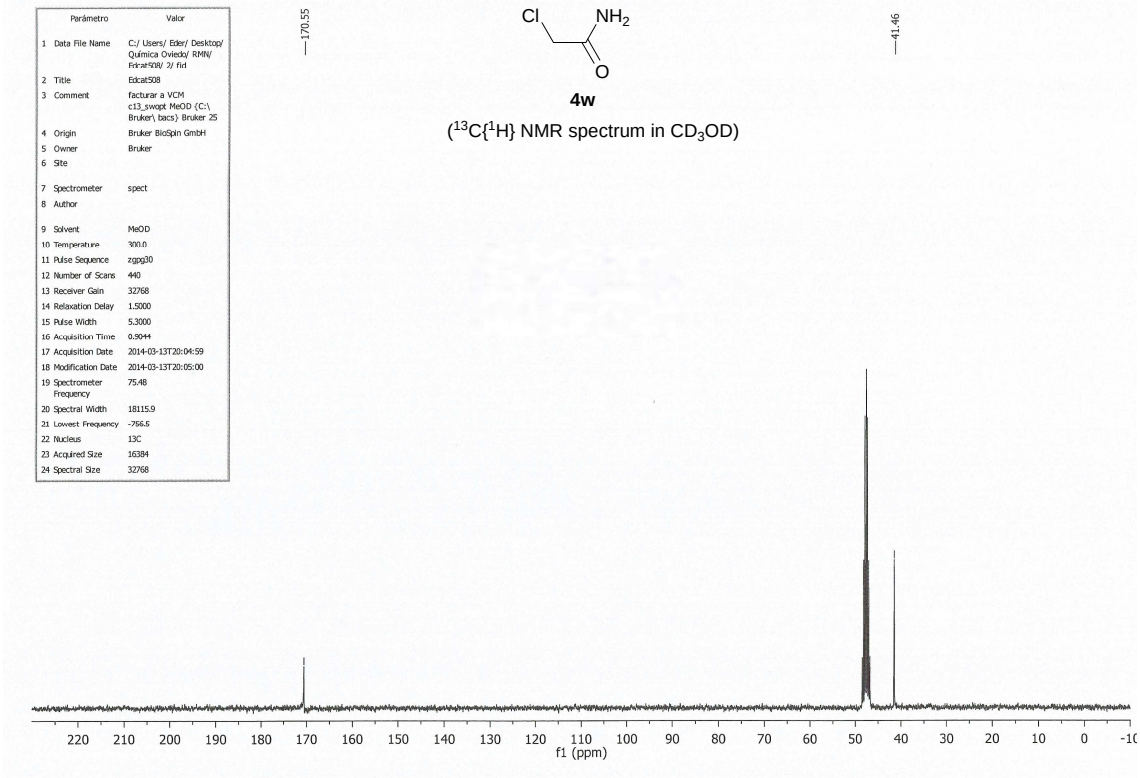
	Parámetro	Valor
1	Data File Name	C:/ Users/ Eder/ Desktop/ Química Oviedo/ RMN/ Edcat/ Edcat534b/ 3/ fid
2	Title	Edcat534b
3	Comment	facturar a vcm c13_swopt D2O {C:\ Bruker\ bacs} Bruker 49
4	Origin	Bruker BioSpin GmbH
5	Owner	Bruker
6	Site	
7	Spectrometer	spect
8	Author	
9	Solvent	D2O
10	Temperature	300.0
11	Pulse Sequence	zgpg30
12	Number of Scans	440
13	Receiver Gain	32768
14	Relaxation Delay	1.5000
15	Pulse Width	5.3000
16	Acquisition Time	0.9044
17	Acquisition Date	2013-12-18T19:15:15
18	Modification Date	2013-12-18T19:15:00
19	Spectrometer Frequency	75.48
20	Spectral Width	18115.9
21	Lowest Frequency	-756.5
22	Nucleus	13C
23	Acquired Size	16384
24	Spectral Size	32768

4v

($^{13}C\{^{1}H\}$ NMR spectrum in D_2O)

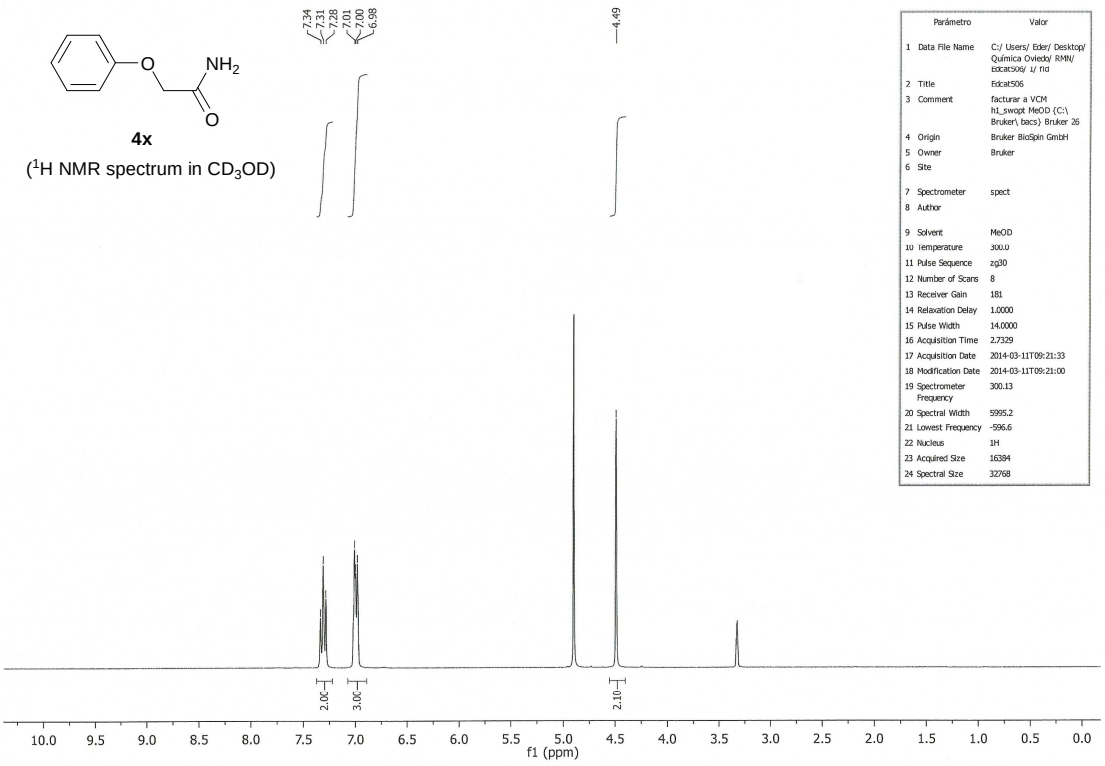



Cl
NH2
O
4w
(1H NMR spectrum in CD3OD)
4.06
1.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)
Parámetro Valor
1 Data File Name C:/ Users/ Eder/ Desktop/ Química Oviedo/ RMN/ Edcat508/ 1/ fid
2 Title Edcat508
3 Comment facturar a VCM h1_swopt MeOD {C:\ Bruker\ bacs} Bruker 25
4 Origin Bruker BioSpin GmbH
5 Owner Bruker
6 Site
7 Spectrometer spect
8 Author
9 Solvent MeOD
10 Temperature 300.0
11 Pulse Sequence zg30
12 Number of Scans 8
13 Receiver Gain 161
14 Relaxation Delay 1.0000
15 Pulse Width 14.0000
16 Acquisition Time 2.7329
17 Acquisition Date 2014-03-13T19:45:52
18 Modification Date 2014-03-13T19:46:00
19 Spectrometer Frequency 300.13
20 Spectral Width 5995.2
21 Lowest Frequency -596.6
22 Nucleus 1H
23 Acquired Size 16384
24 Spectral Size 32768


Parámetro Valor
1 Data File Name C:/ Users/ Eder/ Desktop/ Química Oviedo/ RMN/ Edcat508/ 2/ fid
2 Title Edcat508
3 Comment facturar a VCM c13_swopt MeOD {C:\ Bruker\ bacs} Bruker 25
4 Origin Bruker BioSpin GmbH
5 Owner Bruker
6 Site
7 Spectrometer spect
8 Author
9 Solvent MeOD
10 Temperature 300.0
11 Pulse Sequence zgpg30
12 Number of Scans 440
13 Receiver Gain 32768
14 Relaxation Delay 1.5000
15 Pulse Width 5.3000
16 Acquisition Time 0.9044
17 Acquisition Date 2014-03-13T20:04:59
18 Modification Date 2014-03-13T20:05:00
19 Spectrometer Frequency 75.48
20 Spectral Width 18115.9
21 Lowest Frequency -756.5
22 Nucleus 13C
23 Acquired Size 16384
24 Spectral Size 32768
170.55
41.46
Cl
NH2
O
4w
(13C{1H} NMR spectrum in CD3OD)
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

4x

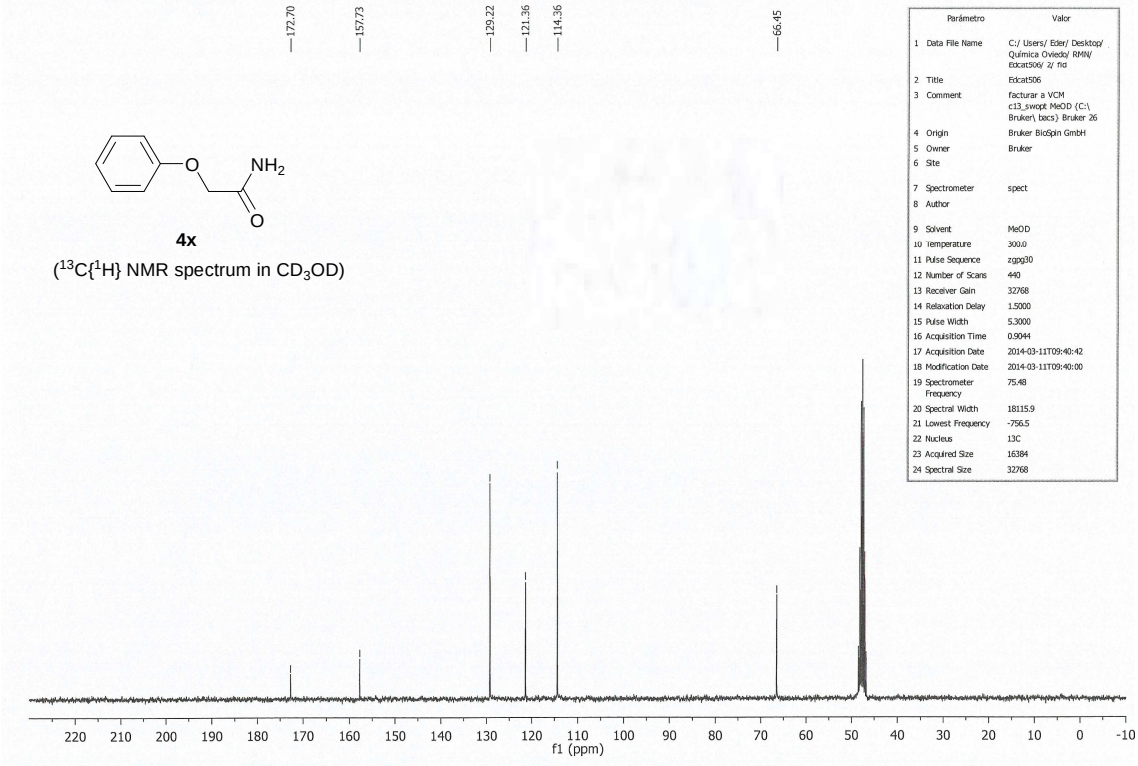
(^{1}H NMR spectrum in CD_3OD)



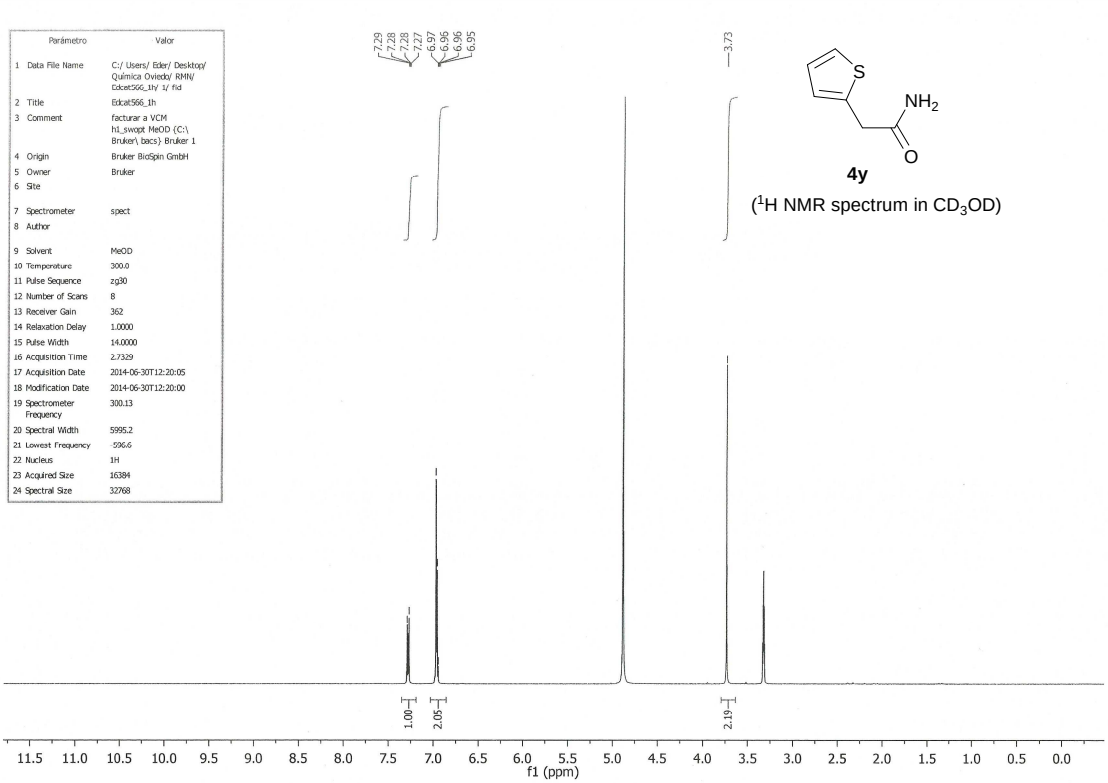
	Parámetro	Valor
1	Data File Name	C:/ Users/ Eder/ Desktop/ Química Oviedo/ RMN/ Edcat506/ 1/ fid
2	Title	Edcat506
3	Comment	facturar a VCM h1_swopt MeOD {C:\ Bruker\ bacs} Bruker 26
4	Origin	Bruker BioSpin GmbH
5	Owner	Bruker
6	Site	
7	Spectrometer	spect
8	Author	
9	Solvent	MeOD
10	Temperature	300.0
11	Pulse Sequence	zg30
12	Number of Scans	8
13	Receiver Gain	181
14	Relaxation Delay	1.0000
15	Pulse Width	14.0000
16	Acquisition Time	2.7329
17	Acquisition Date	2014-03-11T09:21:33
18	Modification Date	2014-03-11T09:21:00
19	Spectrometer Frequency	300.13
20	Spectral Width	5995.2
21	Lowest Frequency	-596.6
22	Nucleus	1H
23	Acquired Size	16384
24	Spectral Size	32768

4x

($^{13}C\{^1H\}$ NMR spectrum in CD_3OD)

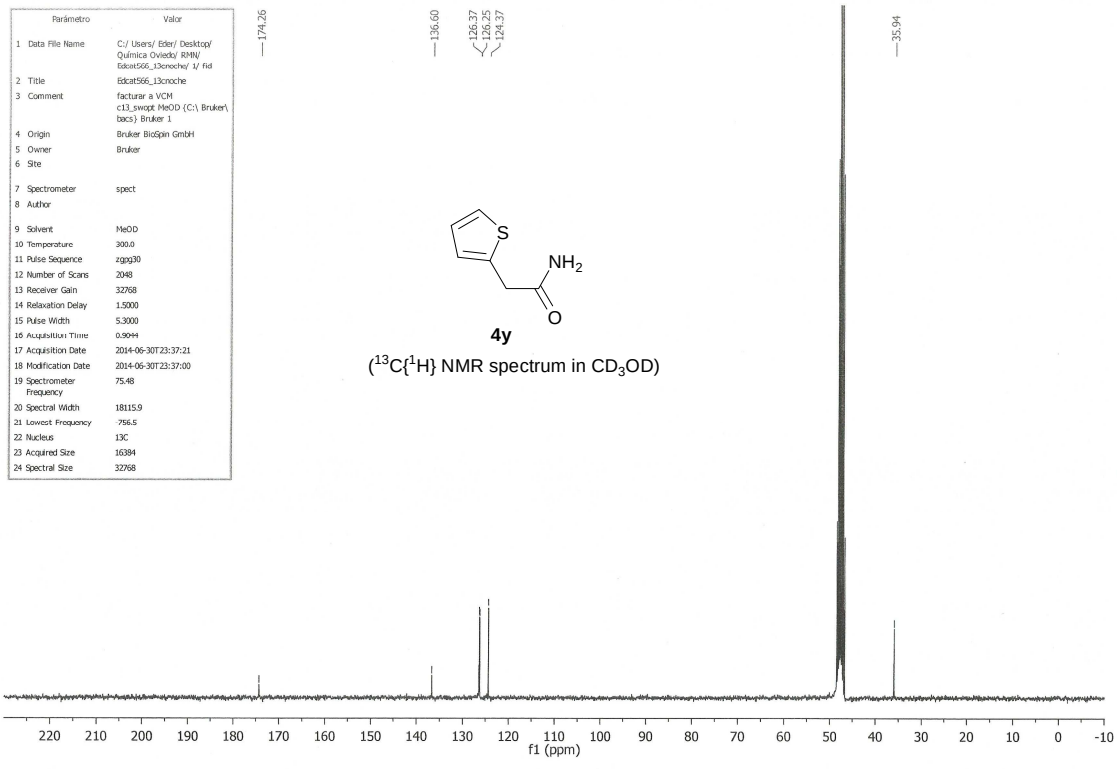


	Parámetro	Valor
1	Data File Name	C:/ Users/ Eder/ Desktop/ Química Oviedo/ RMN/ Edcat506/ 2/ fid
2	Title	Edcat506
3	Comment	facturar a VCM c13_swopt MeOD {C:\ Bruker\ bacs} Bruker 26
4	Origin	Bruker BioSpin GmbH
5	Owner	Bruker
6	Site	
7	Spectrometer	spect
8	Author	
9	Solvent	MeOD
10	Temperature	300.0
11	Pulse Sequence	zgpg30
12	Number of Scans	440
13	Receiver Gain	32768
14	Relaxation Delay	1.5000
15	Pulse Width	5.3000
16	Acquisition Time	0.9044
17	Acquisition Date	2014-03-11T09:40:42
18	Modification Date	2014-03-11T09:40:00
19	Spectrometer Frequency	75.48
20	Spectral Width	18115.9
21	Lowest Frequency	-756.5
22	Nucleus	13C
23	Acquired Size	16384
24	Spectral Size	32768



4y

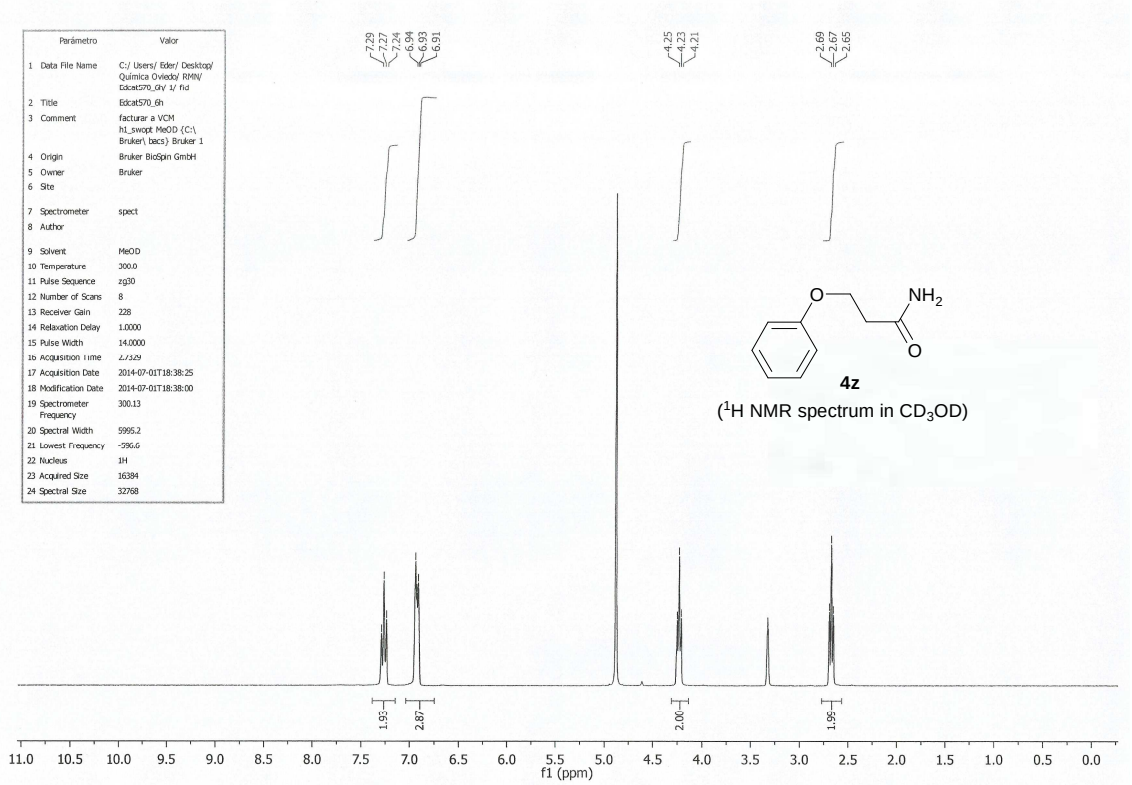
(^{1}H NMR spectrum in CD_3OD)



4y

($^{13}C\{^1H\}$ NMR spectrum in CD_3OD)

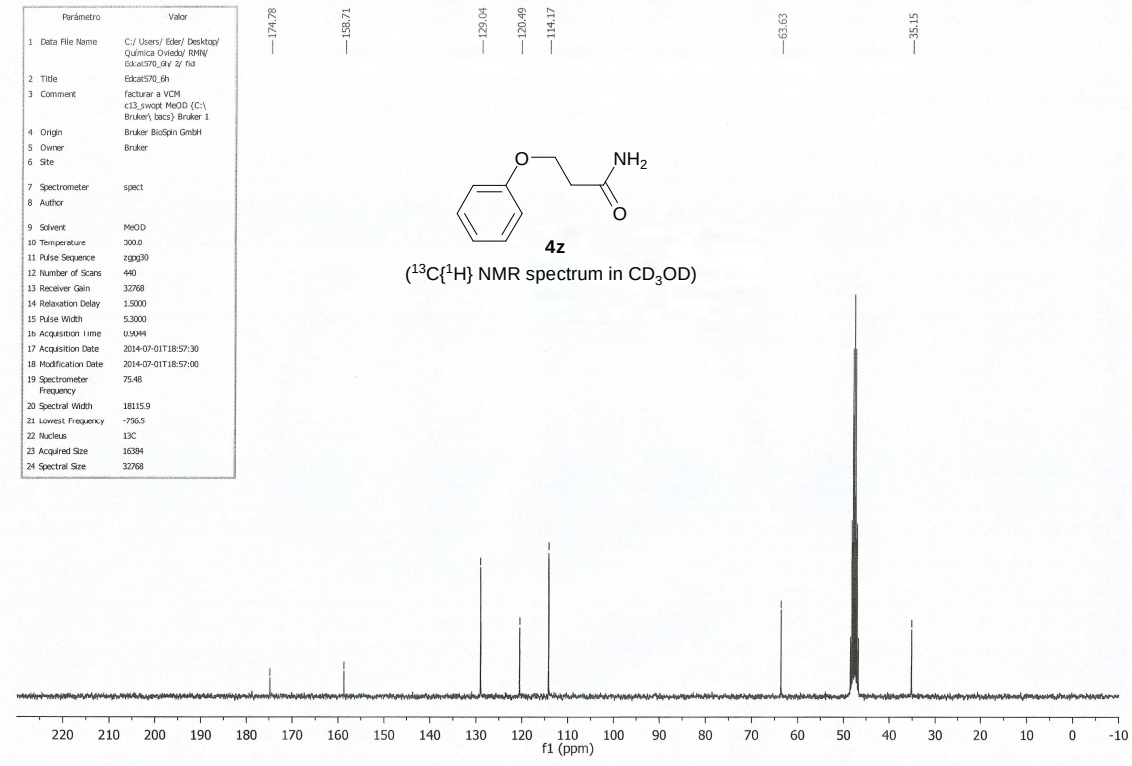
Parámetro	Valor
1 Data File Name	C:/ Users/ Eder/ Desktop/ Química Oviedo/ RMN/ Edcat570_6h/ 1/ fid
2 Title	Edcat570_6h
3 Comment	facturar a VCM h1_swopt MeOD {C:\ Bruker\ bacs} Bruker 1
4 Origin	Bruker BioSpin GmbH
5 Owner	Bruker
6 Site	
7 Spectrometer	spect
8 Author	
9 Solvent	MeOD
10 Temperature	300.0
11 Pulse Sequence	zg30
12 Number of Scans	8
13 Receiver Gain	228
14 Relaxation Delay	1.0000
15 Pulse Width	14.0000
16 Acquisition Time	2.7329
17 Acquisition Date	2014-07-01T18:38:25
18 Modification Date	2014-07-01T18:38:00
19 Spectrometer Frequency	300.13
20 Spectral Width	5995.2
21 Lowest Frequency	-596.6
22 Nucleus	1H
23 Acquired Size	16384
24 Spectral Size	32768



4z

(^{1}H NMR spectrum in CD_3OD)

Parámetro	Valor
1 Data File Name	C:/ Users/ Eder/ Desktop/ Química Oviedo/ RMN/ Edcat570_6h/ 2/ fid
2 Title	Edcat570_6h
3 Comment	facturar a VCM c13_swopt MeOD {C:\ Bruker\ bacs} Bruker 1
4 Origin	Bruker BioSpin GmbH
5 Owner	Bruker
6 Site	
7 Spectrometer	spect
8 Author	
9 Solvent	MeOD
10 Temperature	300.0
11 Pulse Sequence	zgpg30
12 Number of Scans	440
13 Receiver Gain	32768
14 Relaxation Delay	1.5000
15 Pulse Width	5.3000
16 Acquisition Time	0.9044
17 Acquisition Date	2014-07-01T18:57:30
18 Modification Date	2014-07-01T18:57:00
19 Spectrometer Frequency	75.48
20 Spectral Width	18115.9
21 Lowest Frequency	-756.5
22 Nucleus	13C
23 Acquired Size	16384
24 Spectral Size	32768

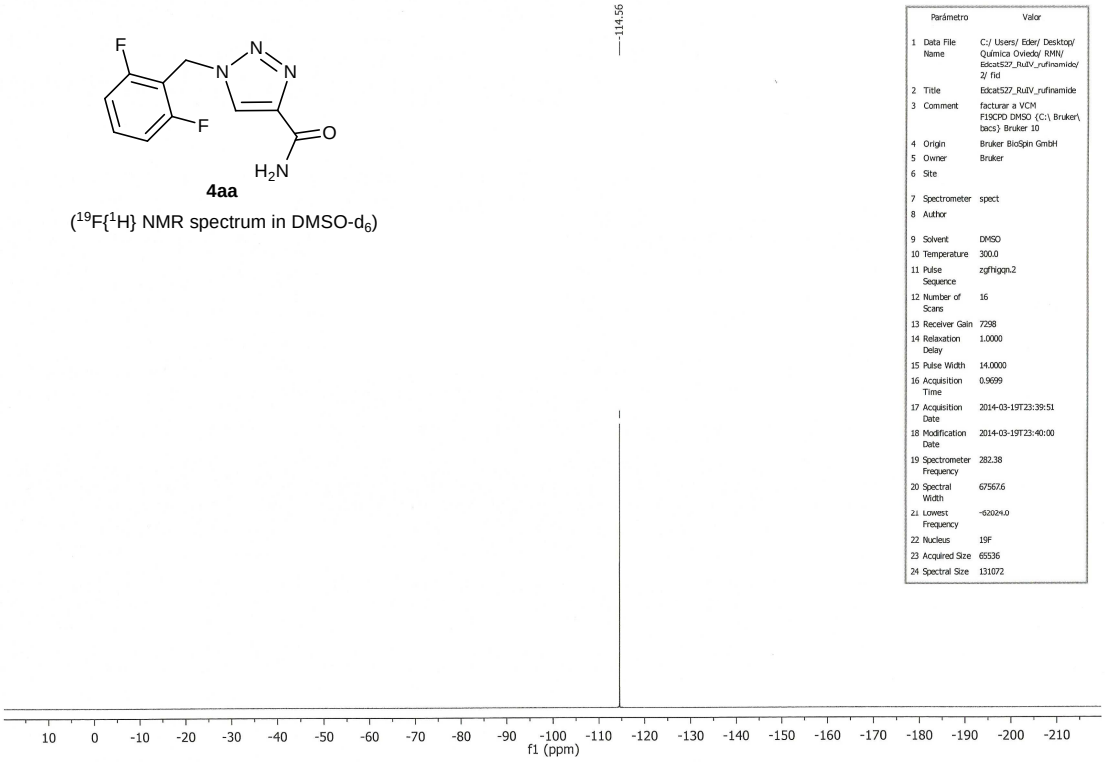


4z

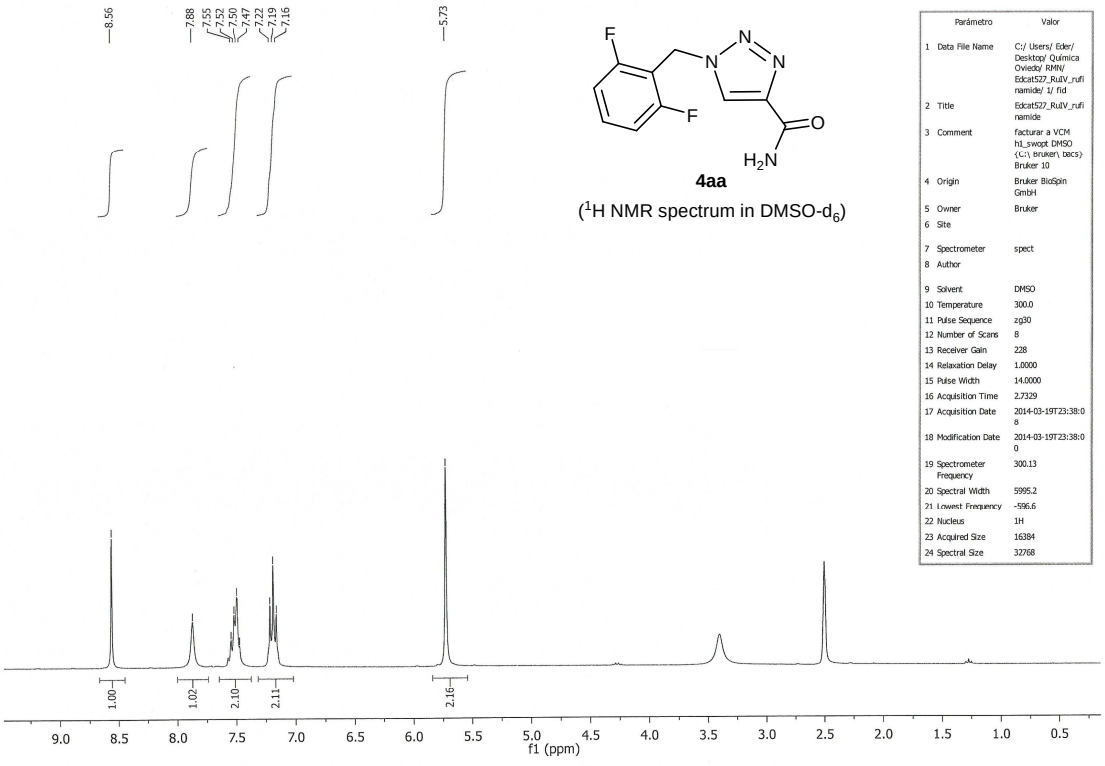
($^{13}C\{^1H\}$ NMR spectrum in CD_3OD)

4aa

($^{19}F\{^1H\}$ NMR spectrum in DMSO-d_6)



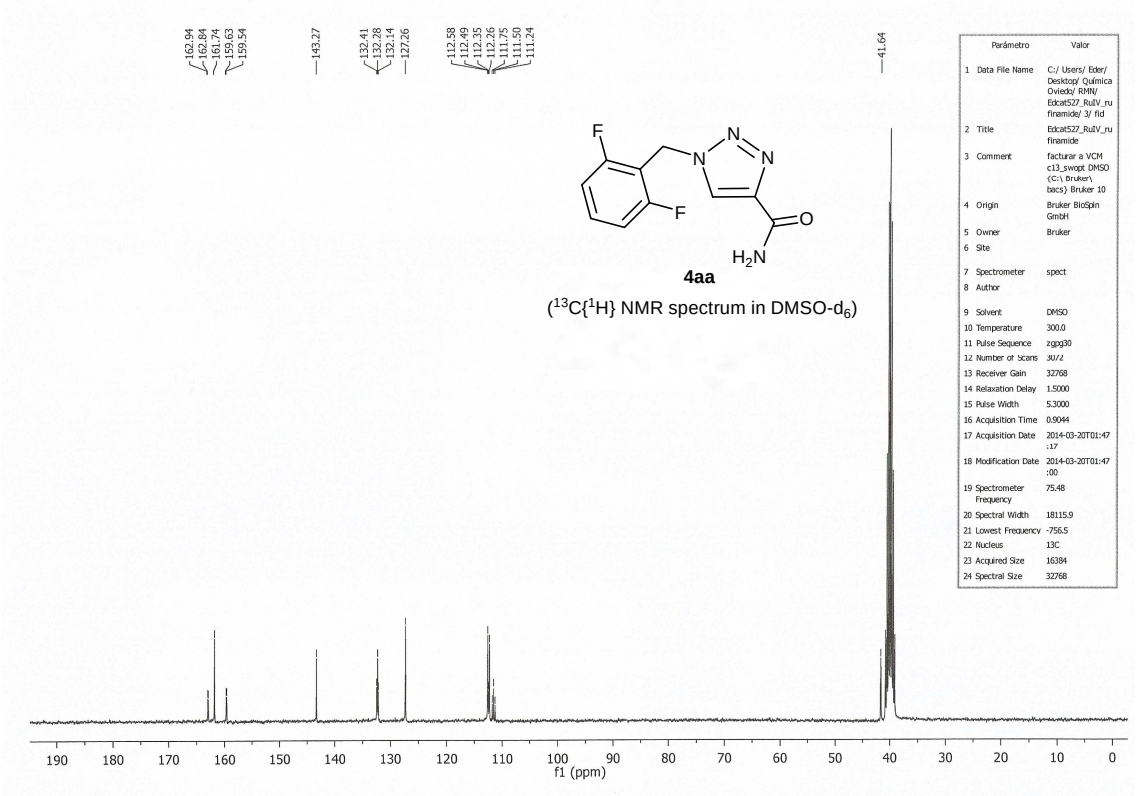
	Parámetro	Valor
1	Data File Name	C:/ Users/ Eder/ Desktop/ Química Oviedo/ RMN/ Edcat527_RuIV_rufinamide/ 2/ fid
2	Title	Edcat527_RuIV_rufinamide
3	Comment	facturar a VCM F19CPD DMSO {C:\ Bruker\ bacs} Bruker 10
4	Origin	Bruker BioSpin GmbH
5	Owner	Bruker
6	Site	
7	Spectrometer	spect
8	Author	
9	Solvent	DMSO
10	Temperature	300.0
11	Pulse Sequence	zgfhigqn.2
12	Number of Scans	16
13	Receiver Gain	7298
14	Relaxation Delay	1.0000
15	Pulse Width	14.0000
16	Acquisition Time	0.9699
17	Acquisition Date	2014-03-19T23:39:51
18	Modification Date	2014-03-19T23:40:00
19	Spectrometer Frequency	282.38
20	Spectral Width	67567.6
21	Lowest Frequency	-6202.4.0
22	Nucleus	19F
23	Acquired Size	65536
24	Spectral Size	131072



4aa

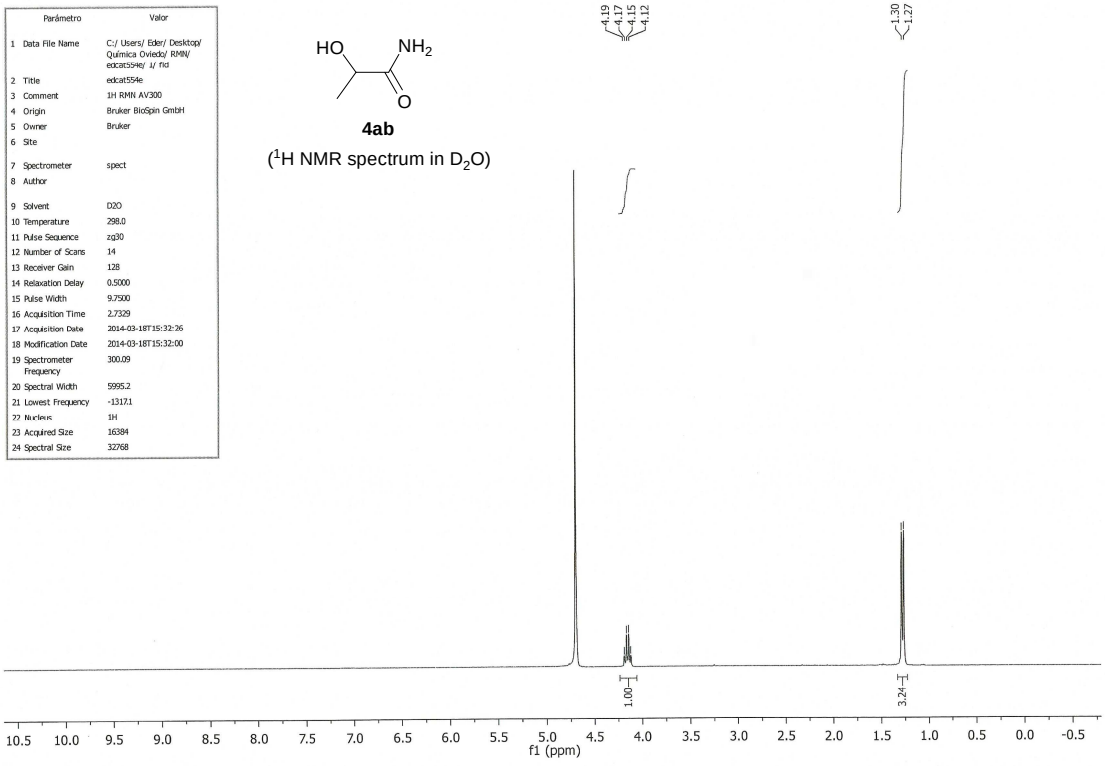
(^1H NMR spectrum in DMSO-d_6)

	Parámetro	Valor
1	Data File Name	C:/ Users/ Eder/ Desktop/ Química Oviedo/ RMN/ Edcat527_RuIV_rufinamide/ 1/ fid
2	Title	Edcat527_RuIV_rufinamide
3	Comment	facturar a VCM h1_swopt DMSO {C:\ bruker\ bacs} Bruker 10
4	Origin	Bruker BioSpin GmbH
5	Owner	Bruker
6	Site	
7	Spectrometer	spect
8	Author	
9	Solvent	DMSO
10	Temperature	300.0
11	Pulse Sequence	zg30
12	Number of Scans	8
13	Receiver Gain	228
14	Relaxation Delay	1.0000
15	Pulse Width	14.0000
16	Acquisition Time	2.7329
17	Acquisition Date	2014-03-19T23:38:08
18	Modification Date	2014-03-19T23:38:00
19	Spectrometer Frequency	300.13
20	Spectral Width	5995.2
21	Lowest Frequency	-596.6
22	Nucleus	1H
23	Acquired Size	16384
24	Spectral Size	32768



4aa

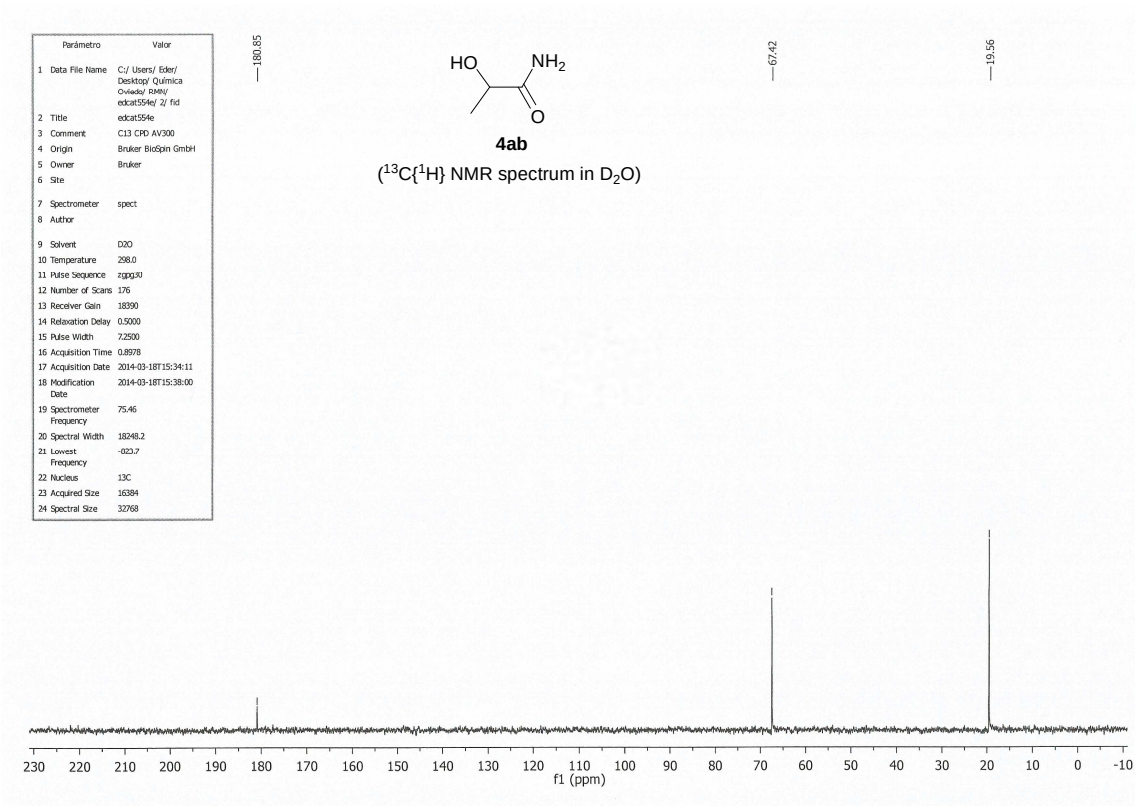
($^{13}C\{^{1}H\}$ NMR spectrum in DMSO-d_6)



4ab

(^{1}H NMR spectrum in D_2O)

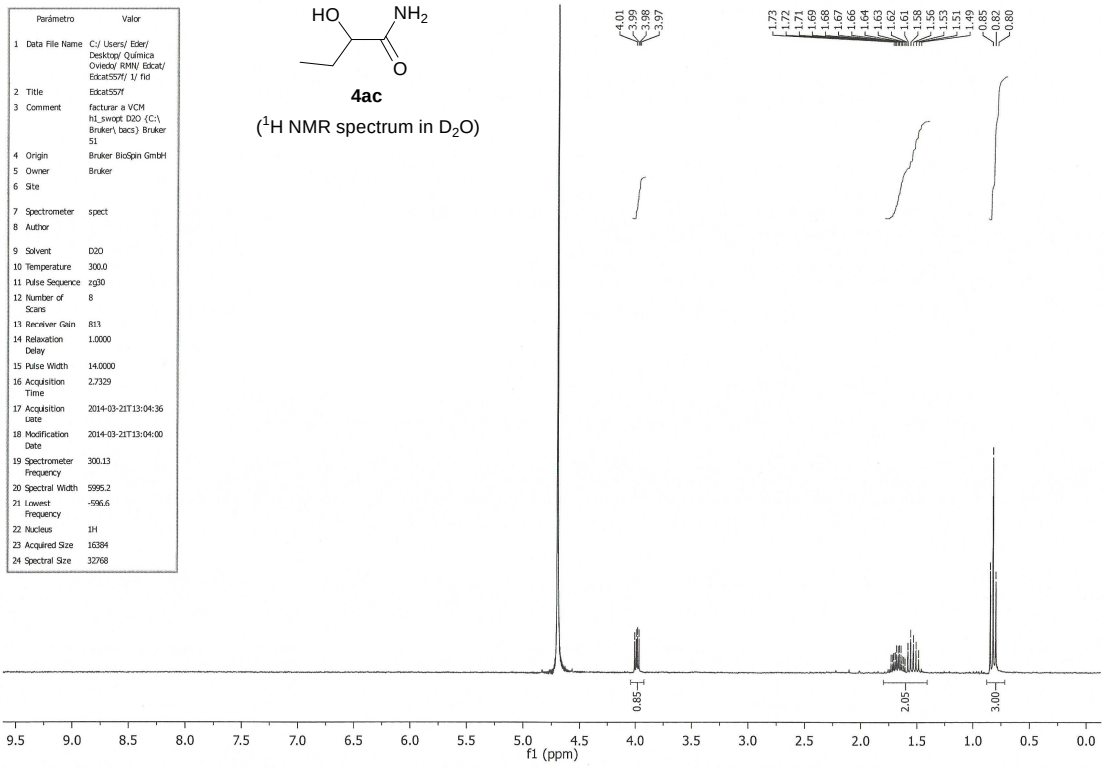
	Parámetro	Valor
1	Data File Name	C:/ Users/ Eder/ Desktop/ Química Oviedo/ RMN/ edcat554e/ 2/ fid
2	Title	edcat554e
3	Comment	C13 CPD AV300
4	Origin	Bruker BioSpin GmbH
5	Owner	Bruker
6	Site	
7	Spectrometer	spect
8	Author	
9	Solvent	D2O
10	Temperature	298.0
11	Pulse Sequence	zgpg30
12	Number of Scans	176
13	Receiver Gain	18390
14	Relaxation Delay	0.5000
15	Pulse Width	7.2500
16	Acquisition Time	0.8978
17	Acquisition Date	2014-03-18T15:34:11
18	Modification Date	2014-03-18T15:38:00
19	Spectrometer Frequency	75.46
20	Spectral Width	18248.2
21	Lowest Frequency	-023.7
22	Nucleus	13C
23	Acquired Size	16384
24	Spectral Size	32768



4ab

($^{13}C\{^{1}H\}$ NMR spectrum in D_2O)

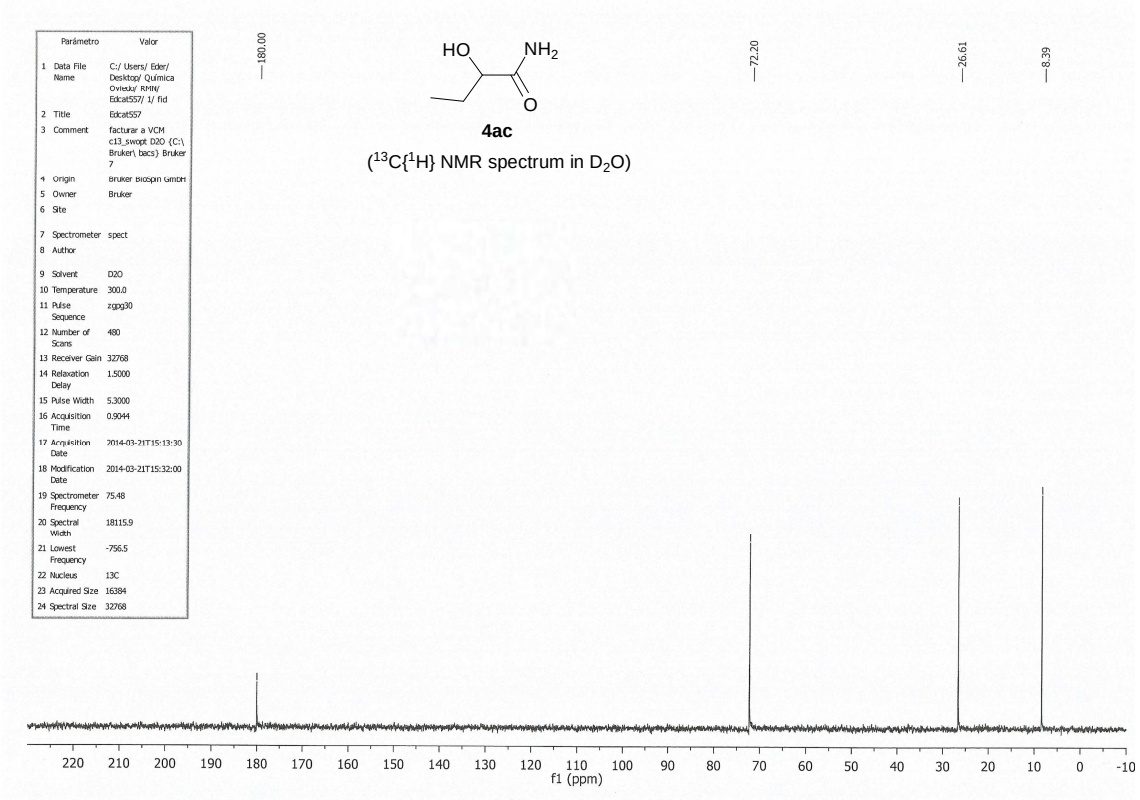
	Parámetro	Valor
1	Data File Name	C:/ Users/ Eder/ Desktop/ Química Oviedo/ RMN/ Edcat/ Edcat557f/ 1/ fid
2	Title	Edcat557f
3	Comment	facturar a VCM h1_swopt D2O {C:\ Bruker\ bacs} Bruker 51
4	Origin	Bruker BioSpin GmbH
5	Owner	Bruker
6	Site	
7	Spectrometer	spect
8	Author	
9	Solvent	D2O
10	Temperature	300.0
11	Pulse Sequence	zg30
12	Number of Scans	8
13	Receiver Gain	813
14	Relaxation Delay	1.0000
15	Pulse Width	14.0000
16	Acquisition Time	2.7329
17	Acquisition Date	2014-03-21T13:04:36
18	Modification Date	2014-03-21T13:04:00
19	Spectrometer Frequency	300.13
20	Spectral Width	5995.2
21	Lowest Frequency	-596.6
22	Nucleus	1H
23	Acquired Size	16384
24	Spectral Size	32768



4ac

(^{1}H NMR spectrum in D_2O)

	Parâmetro	Valor
1	Data File Name	C:/ Users/ Eder/ Desktop/ Química Oviedo/ RMN/ Edcat557/ 1/ fid
2	Title	Edcat557
3	Comment	facturar a VCM c13_swopt D2O {C:\ Bruker\ bacs} Bruker 7
4	Origin	Bruker Biospin GmbH
5	Owner	Bruker
6	Site	
7	Spectrometer	spect
8	Author	
9	Solvent	D2O
10	Temperature	300.0
11	Pulse Sequence	zgpg30
12	Number of Scans	480
13	Receiver Gain	32768
14	Relaxation Delay	1.5000
15	Pulse Width	5.3000
16	Acquisition Time	0.9044
17	Acquisition Date	2014-03-21T15:13:30
18	Modification Date	2014-03-21T15:32:00
19	Spectrometer Frequency	75.48
20	Spectral Width	18115.9
21	Lowest Frequency	-756.5
22	Nucleus	13C
23	Acquired Size	16384
24	Spectral Size	32768



4ac

($^{13}C\{^{1}H\}$ NMR spectrum in D_2O)