

Supplementary information

**Synthesis of 3,3'-Carbonyl-bis(chromones) and their Activity as
Mammalian Alkaline Phosphatase Inhibitors**

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^d *Leibniz Institut für Katalyse an der Universität Rostock e.V., Albert Einstein Str. 29a, 18059 Rostock, Germany*

peter.langer@uni-rostock.de

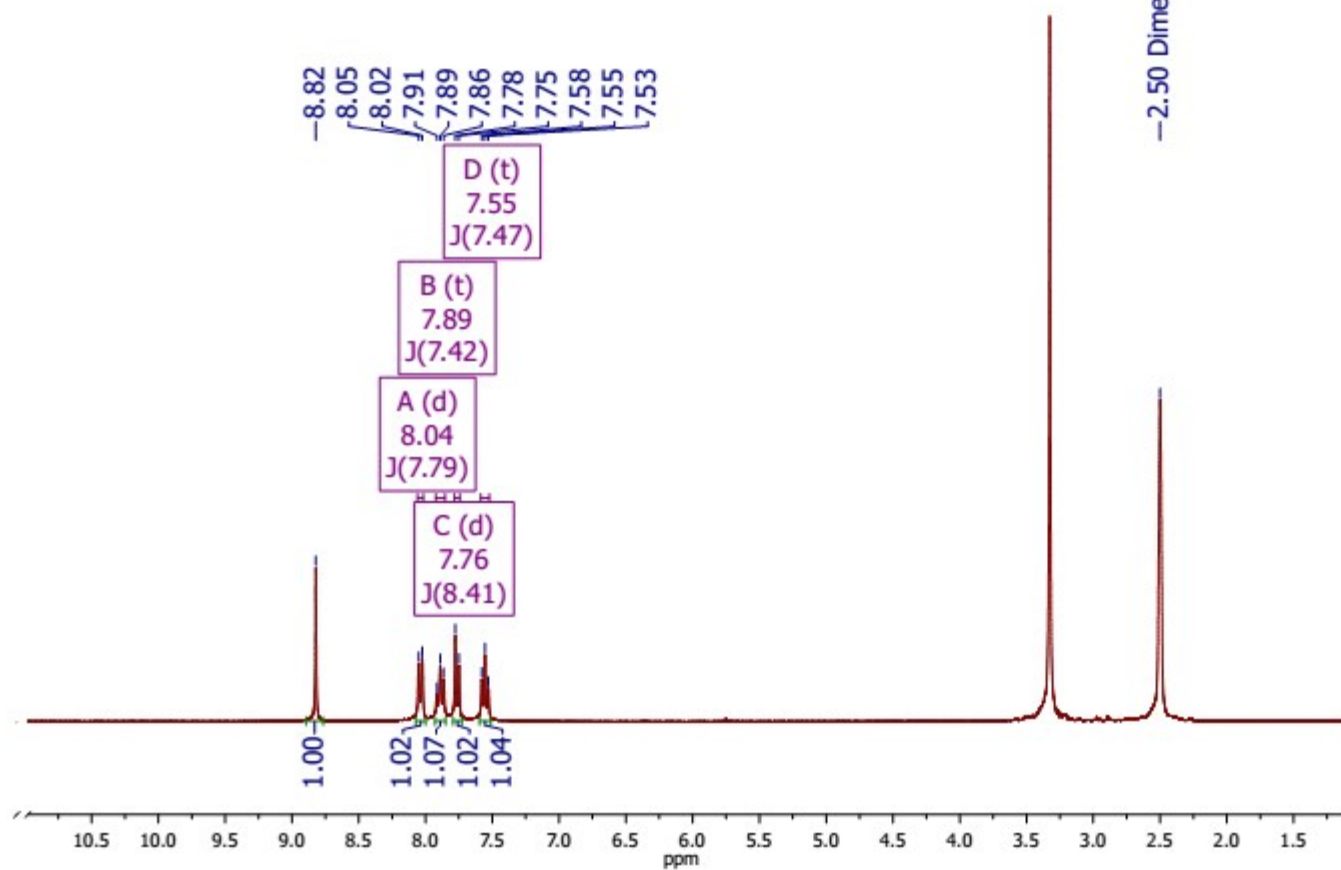
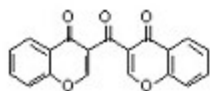
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Copies of ¹ H and ¹³ C NMR spectra.....	2
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3,3'-Carbonylbis(4*H*-chromen-4-one) 8a.

130708.u310

Miliutina MM-63.14-1 1H DMSO

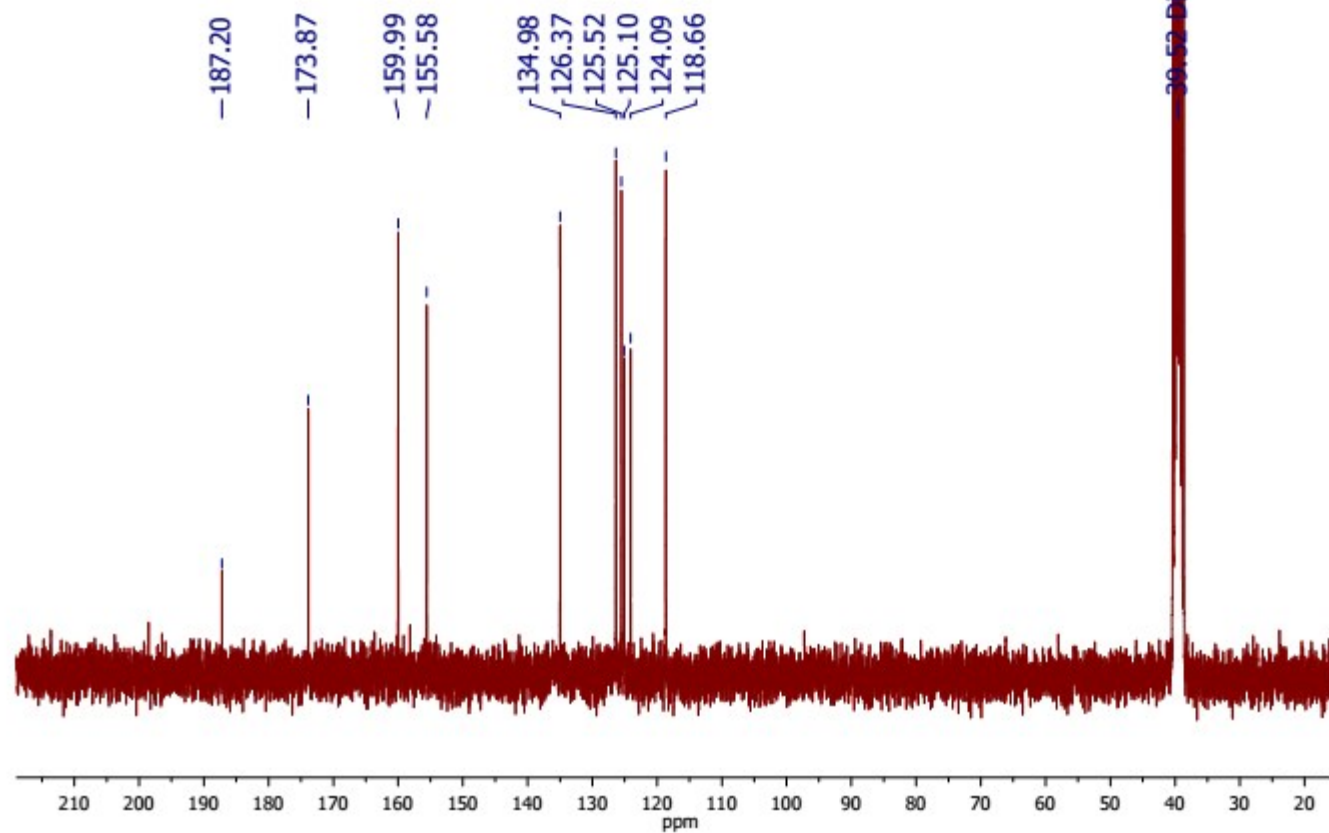
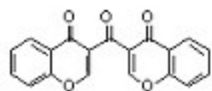


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Spectrometer	spect
Author	
Solvent	DMSO
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Pulse Width	10.0000
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Spectral Size	65536

3,3'-Carbonylbis(4*H*-chromen-4-one) 8a.

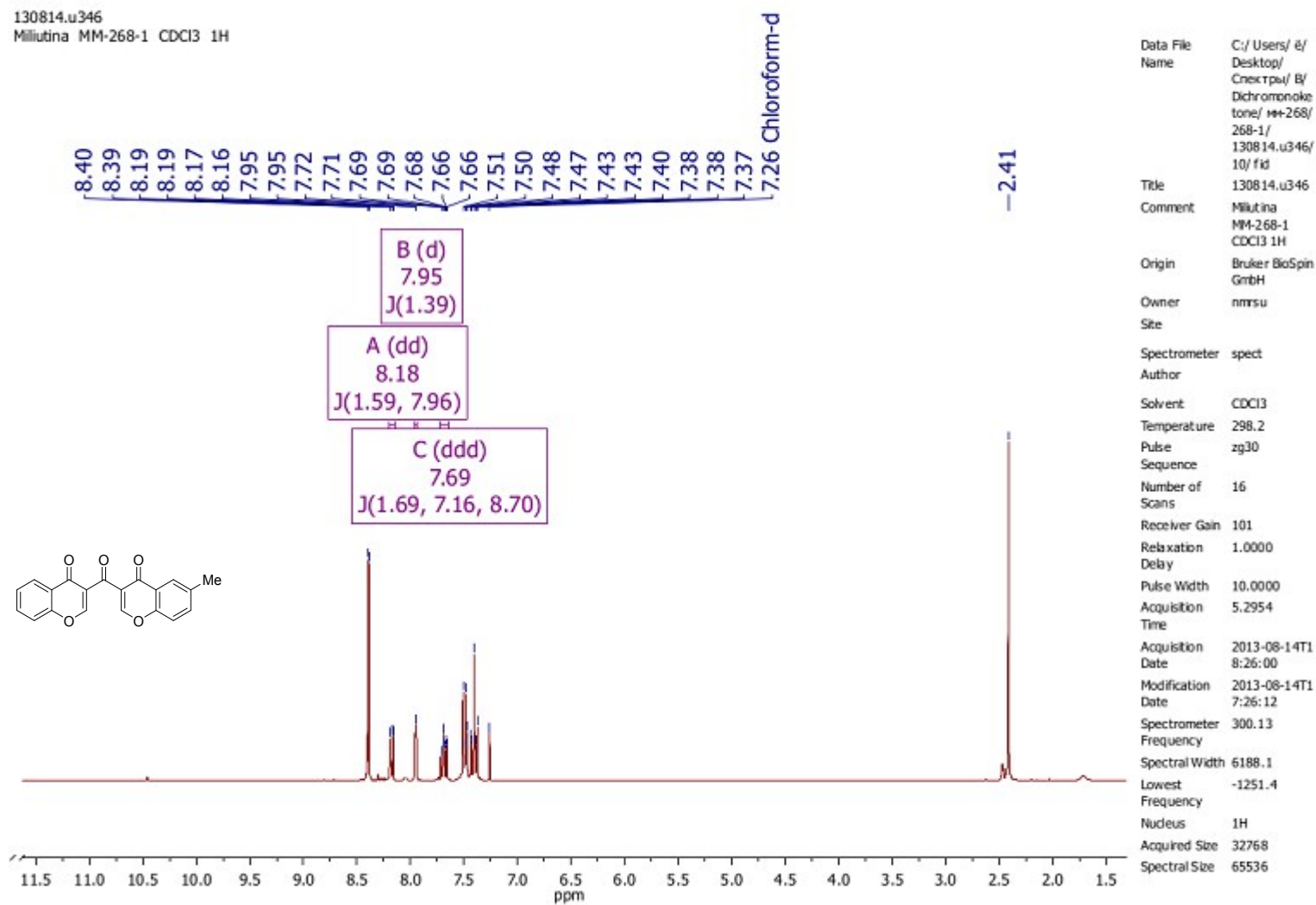
130708.u310

Miliutina MM-63.14-1 13C DMSO



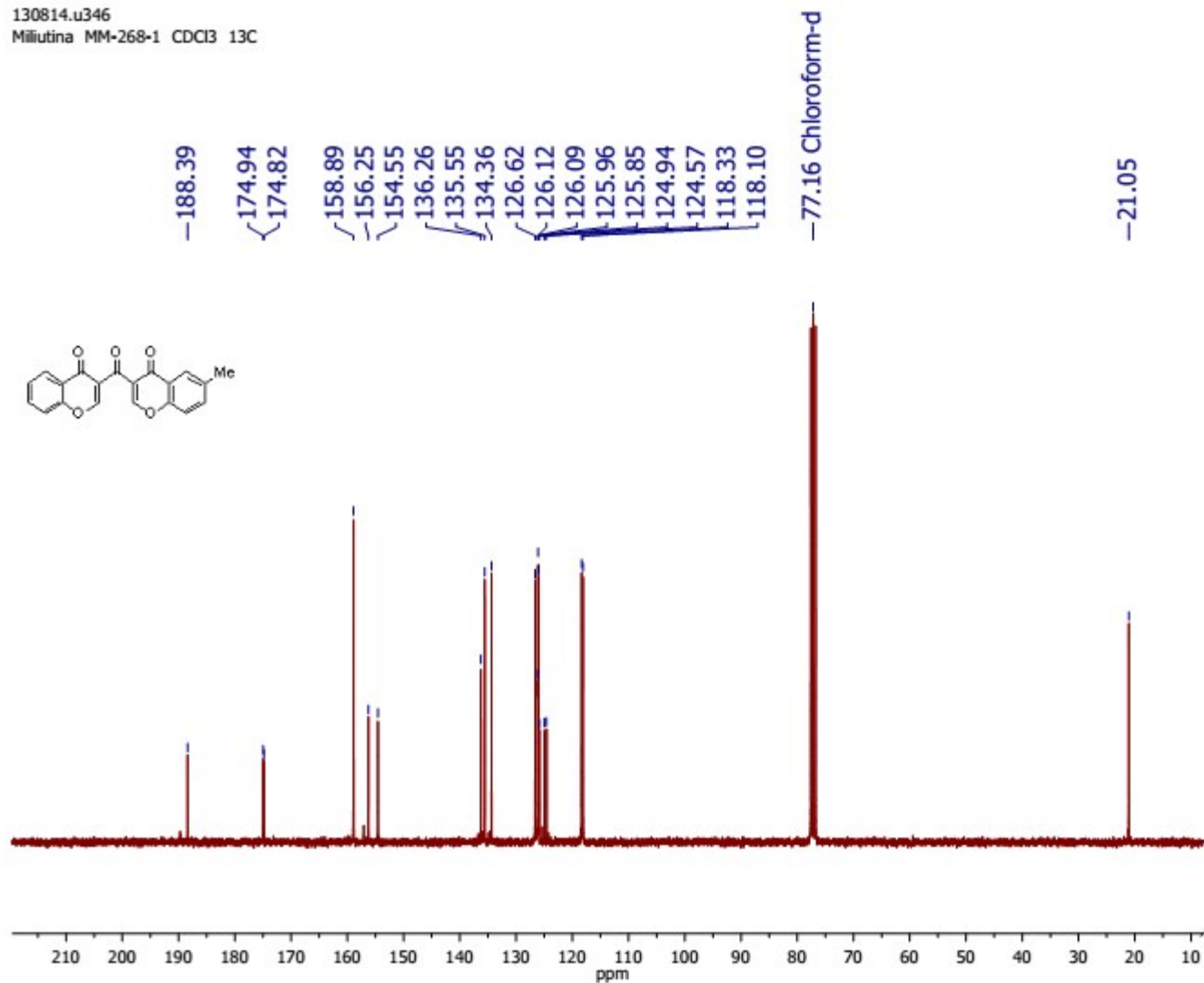
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 Author:
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 Relaxation
 Delay: 2.0000
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 Date: 2013-07-08T1
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 Lowest
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 Spectral Size: 65536

6-Methyl-3-(4-oxo-4*H*-chromene-3-carbonyl)-4*H*-chromen-4-one 8b.



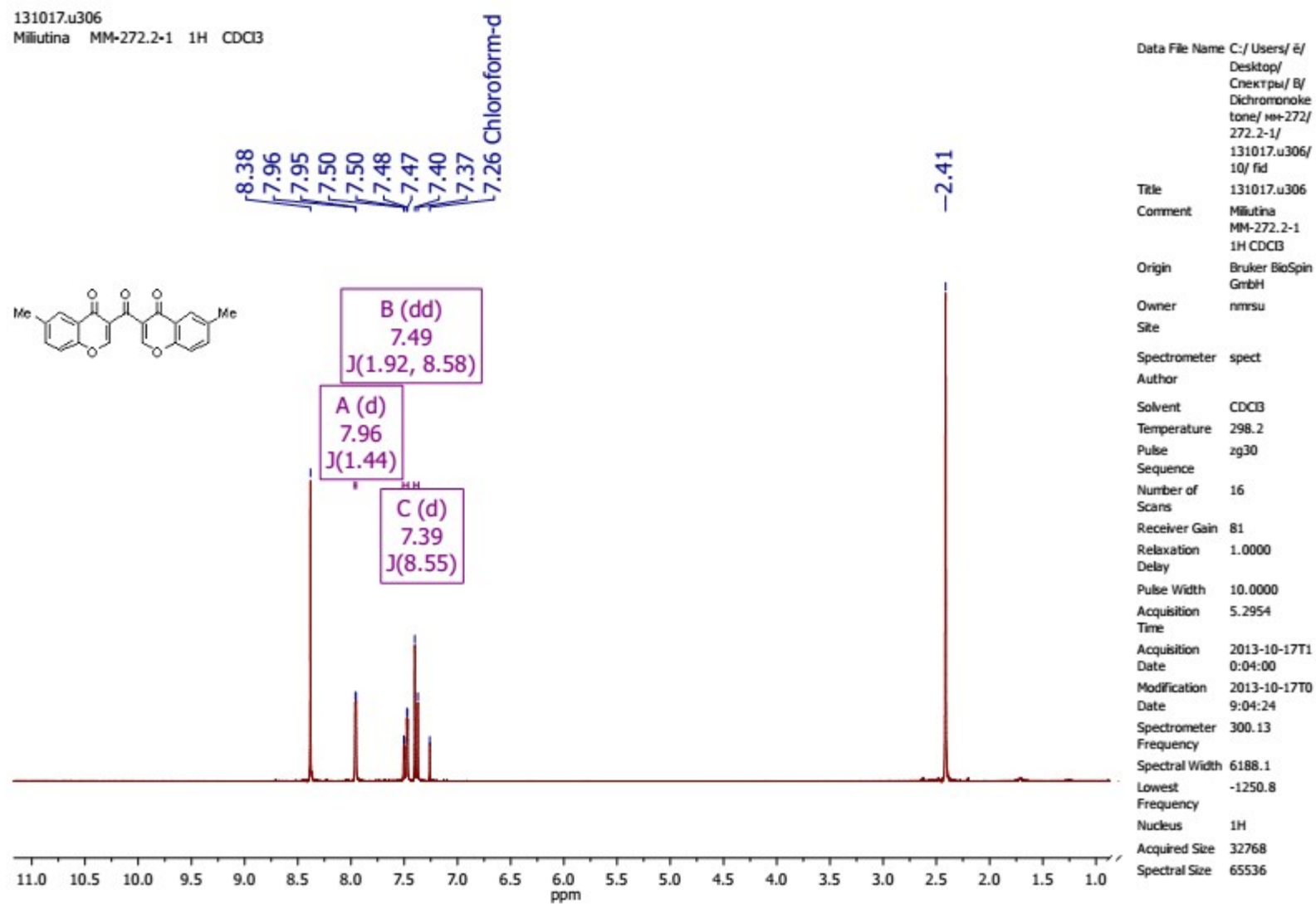
6-Methyl-3-(4-oxo-4*H*-chromene-3-carbonyl)-4*H*-chromen-4-one 8b.

130814.u346
Miliutina MM-268-1 CDCl₃ 13C

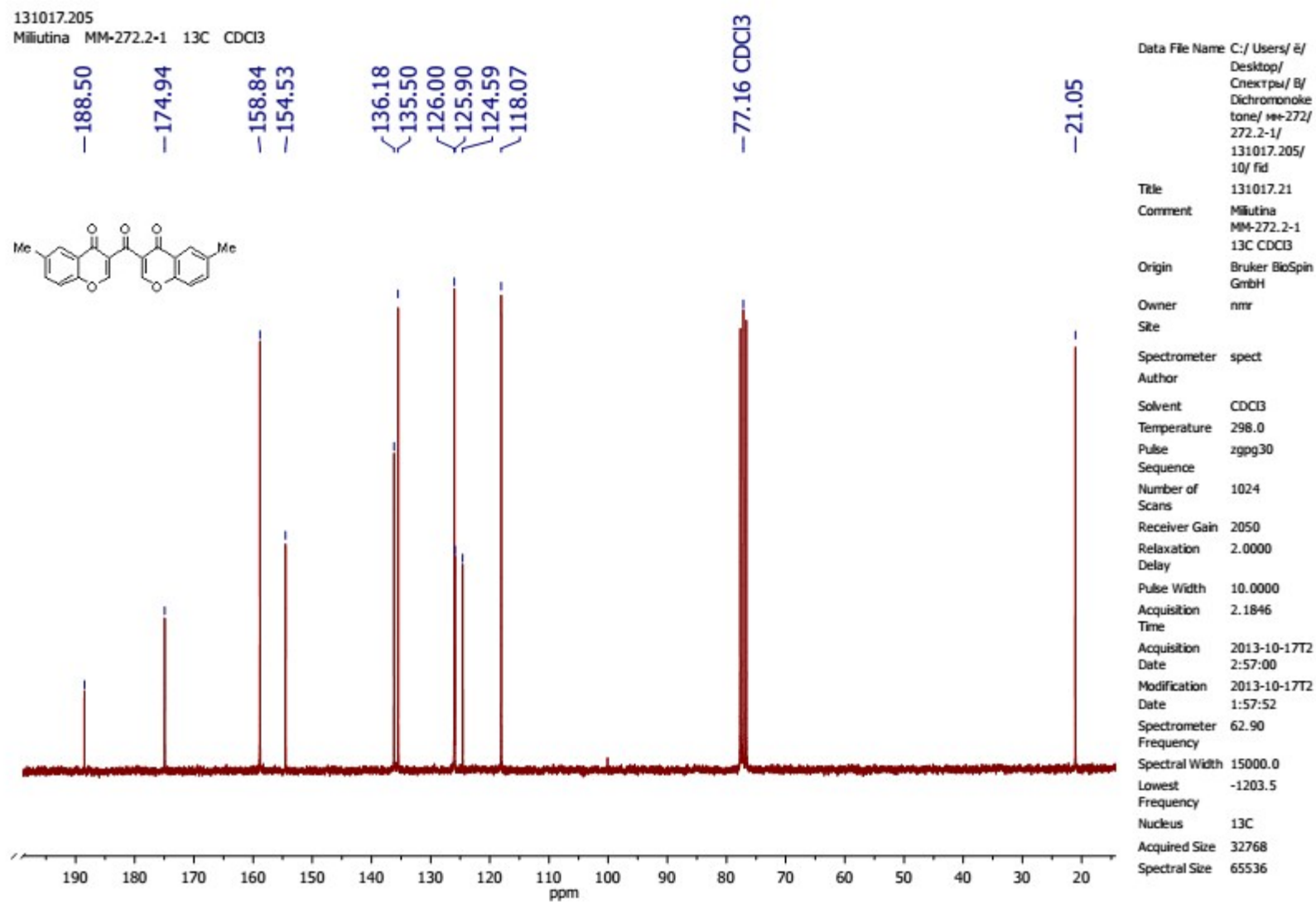


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Owner	nmrsu
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Author	
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Spectral Size	65536

3,3'-Carbonylbis(6-methyl-4*H*-chromen-4-one) 8c.

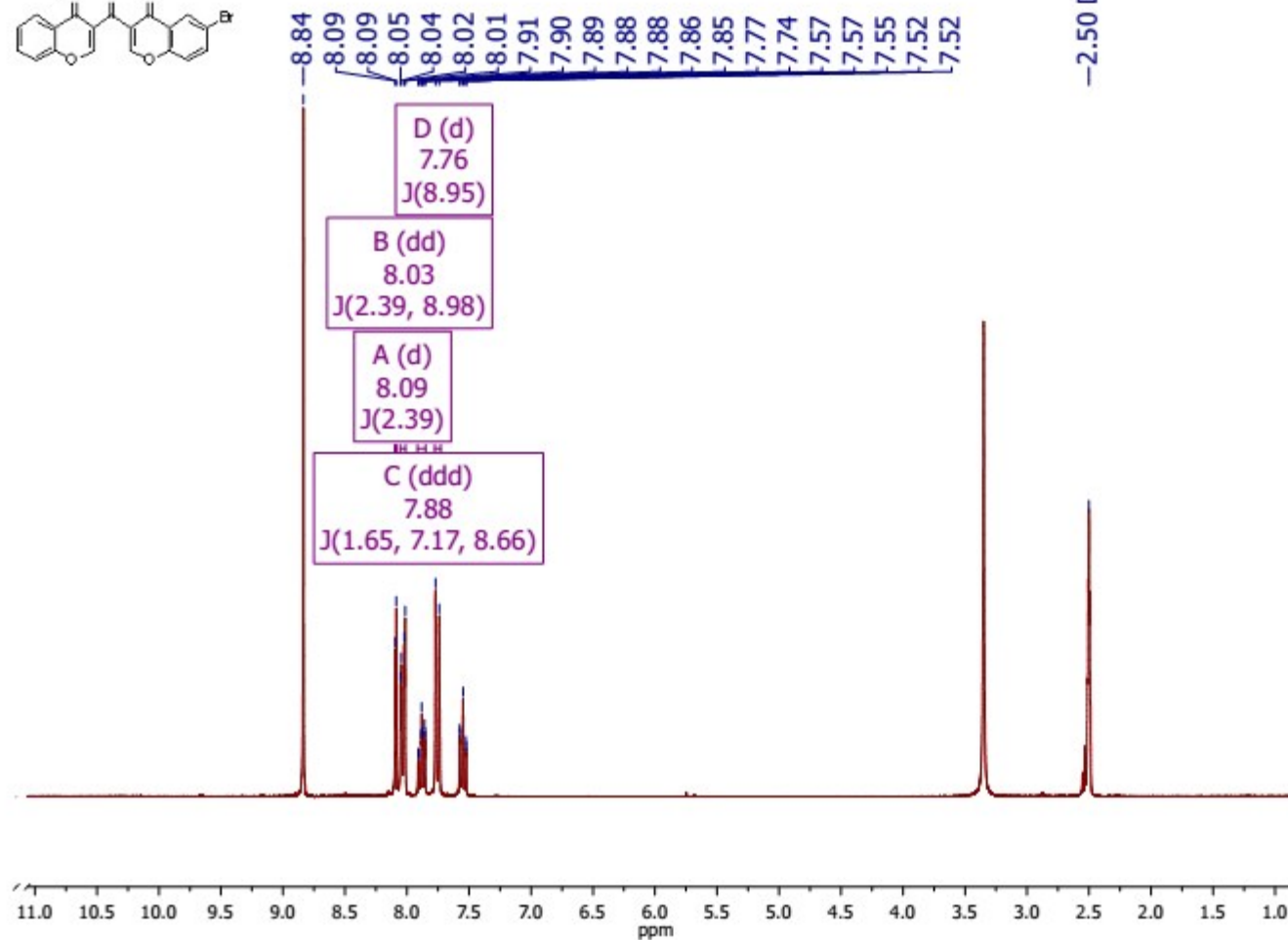
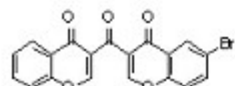


3,3'-Carbonylbis(6-methyl-4*H*-chromen-4-one) 8c.



6-Bromo-3-(4-oxo-4*H*-chromene-3-carbonyl)-4*H*-chromen-4-one 8d.

130729.u309
Miliutina precipitate MM-267 1H DMSO



-2.50 DMSO-d6

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Origin: Bruker BioSpin GmbH

Owner: nmrsu

Site:

Spectrometer: spect

Author:

Solvent: DMSO

Temperature: 298.2

Pulse Sequence: zg30

Number of Scans: 16

Receiver Gain: 114

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Pulse Width: 10.0000

Acquisition Time: 5.2954

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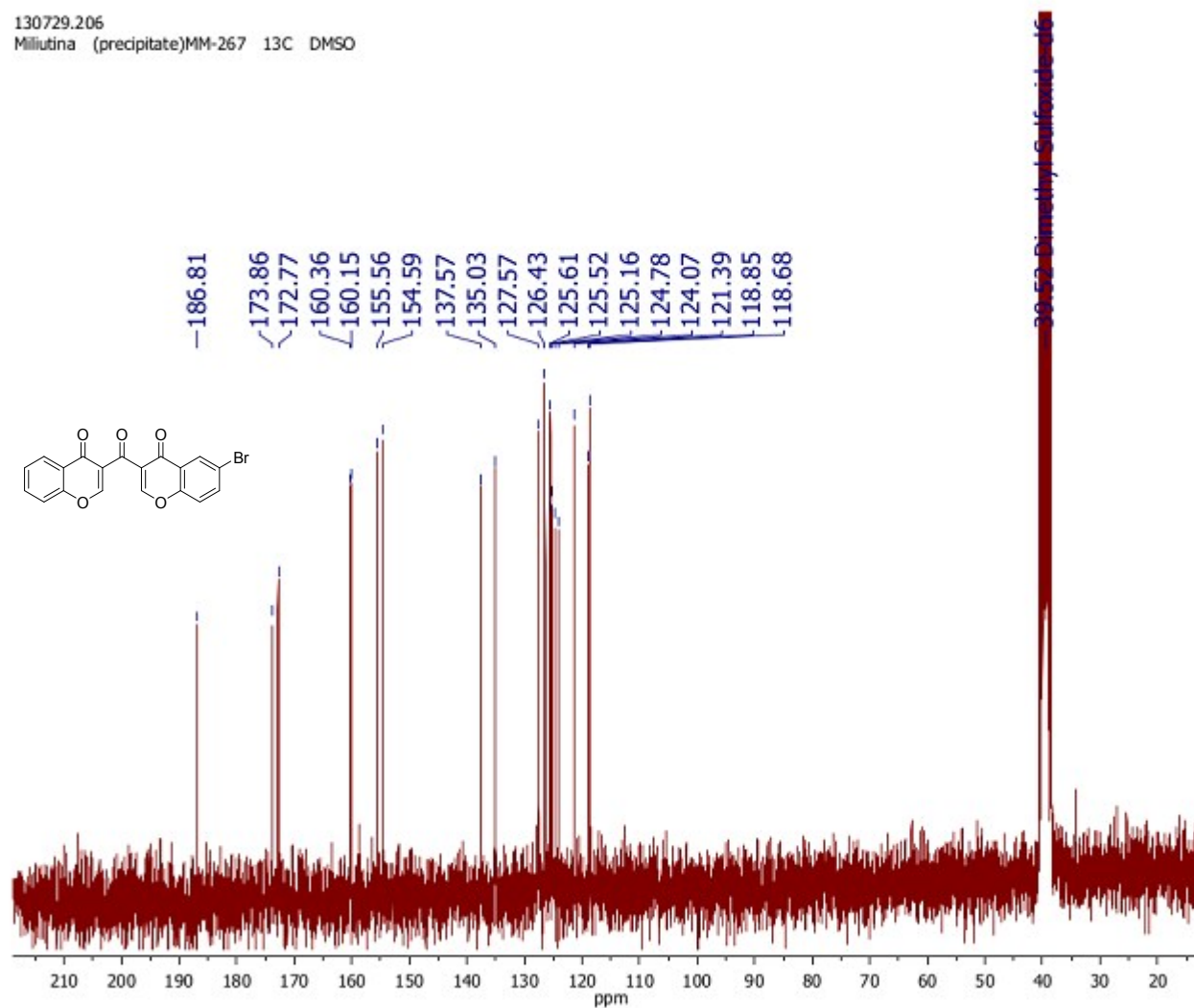
Nucleus: 1H

Acquired Size: 32768

Spectral Size: 65536

6-Bromo-3-(4-oxo-4H-chromene-3-carbonyl)-4H-chromen-4-one 8d.

130729.206
Miliutina (precipitate)MM-267 13C DMSO



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Title 130729.21

Comment Miliutina
(precipitate)MM
-267 13C DMSO

Origin Bruker BioSpin
GmbH

Owner nmr

Site

Spectrometer spect

Author

Solvent DMSO

Temperature 298.3

Pulse Sequence zgpg30

Number of
Scans 1024

Receiver Gain 2050

Relaxation
Delay 2.0000

Pulse Width 10.0000

Acquisition Time 2.1846

Acquisition Date 2013-07-29T22
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Modification 2013-07-29T21
:34:18

Spectrometer
Frequency 62.90

Spectral Width 15000.0

Lowest
Frequency -1239.9

Nucleus 13C

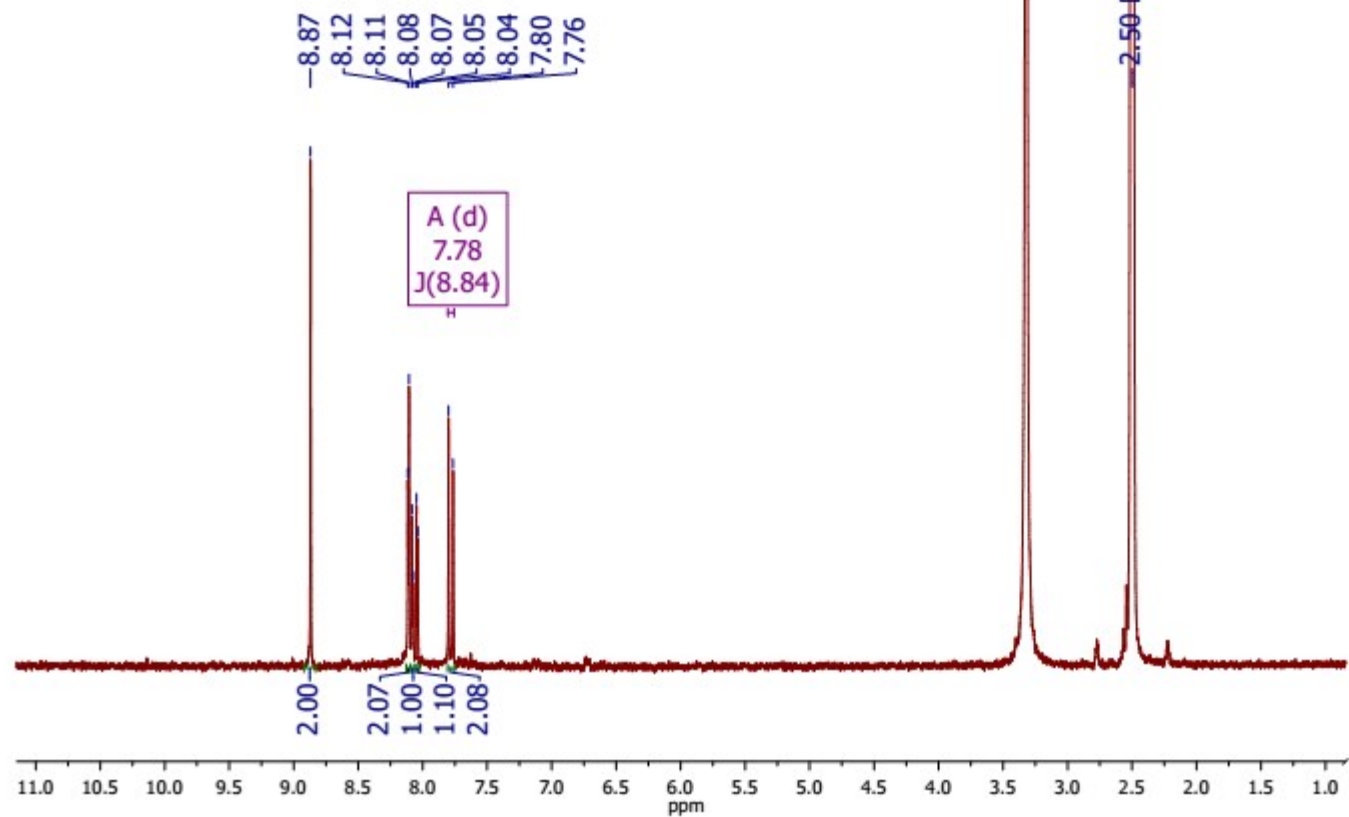
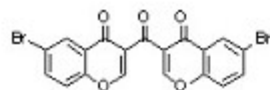
Acquired Size 32768

Spectral Size 65536

3,3'-Carbonylbis(6-bromo-4H-chromen-4-one) 8e.

130829.206

Miliutina MM-281cryst. 1H DMSO



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Comment	Miliutina MM-281cryst. 1H DMSO
Origin	Bruker BioSpin GmbH
Owner	nmr
Site	
Spectrometer	spect
Author	
Solvent	DMSO
Temperature	298.2
Pulse	zg30
Sequence	
Number of Scans	16
Receiver Gain	1820
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Nucleus	1H
Acquired Size	32768
Spectral Size	65536

3,3'-Carbonylbis(6-bromo-4H-chromen-4-one) 8e.

140423.501

M. Miliutina, MM-281, 13C in DMSO, 100 grd_C

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—172.12

—159.46

—154.19

—136.89

—127.18

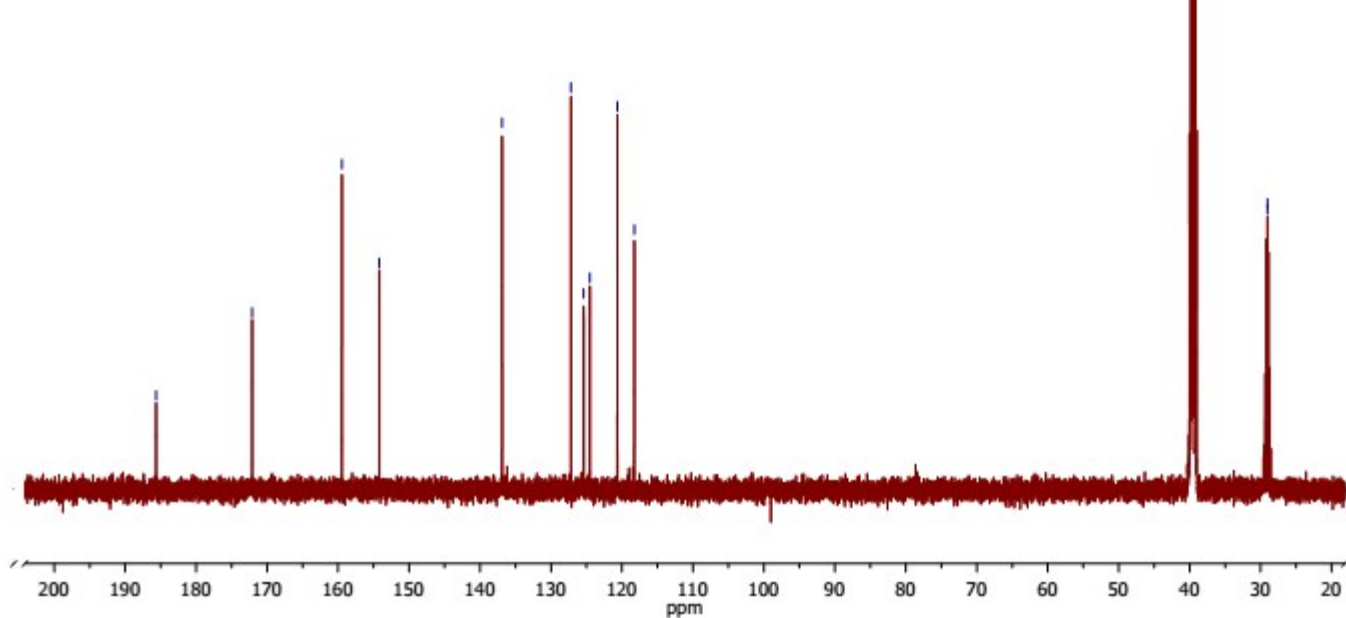
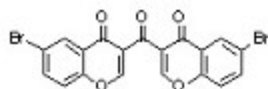
—125.39

—124.53

—120.65

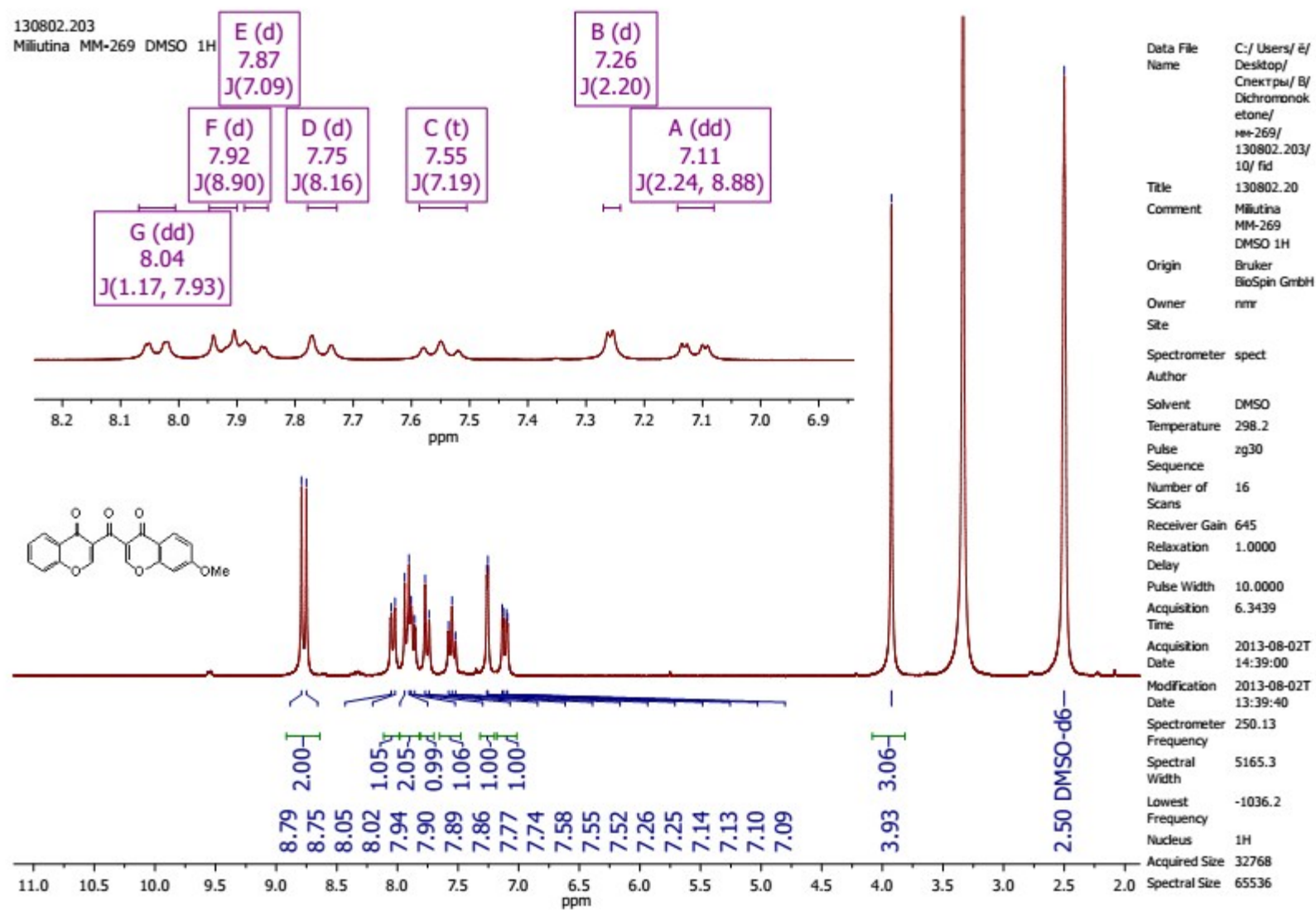
—118.26

—29.04 (CH₃)₂CO
—29.04



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Owner Site	Administrator
Spectrometer Author	spect
Solvent	DMSO
Temperature	373.0
Pulse Sequence	zgpg30
Number of Scans	1024
Receiver Gain	20642
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Acquired Size	32768
Spectral Size	65536

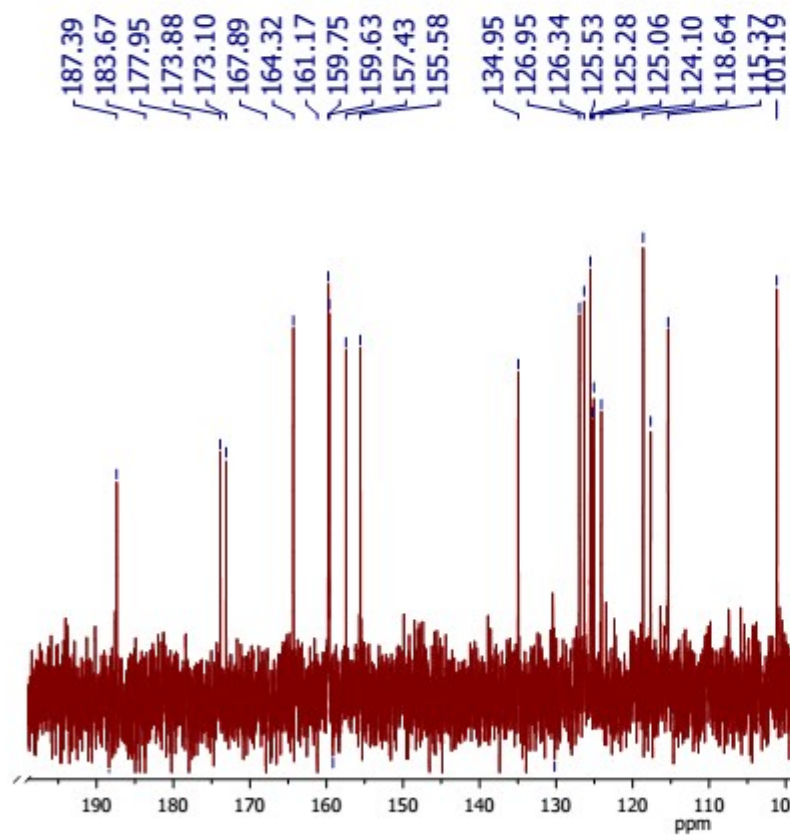
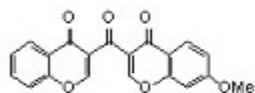
7-Methoxy-3-(4-oxo-4H-chromene-3-carbonyl)-4H-chromen-4-one 8f.



7-Methoxy-3-(4-oxo-4H-chromene-3-carbonyl)-4H-chromen-4-one 8f.

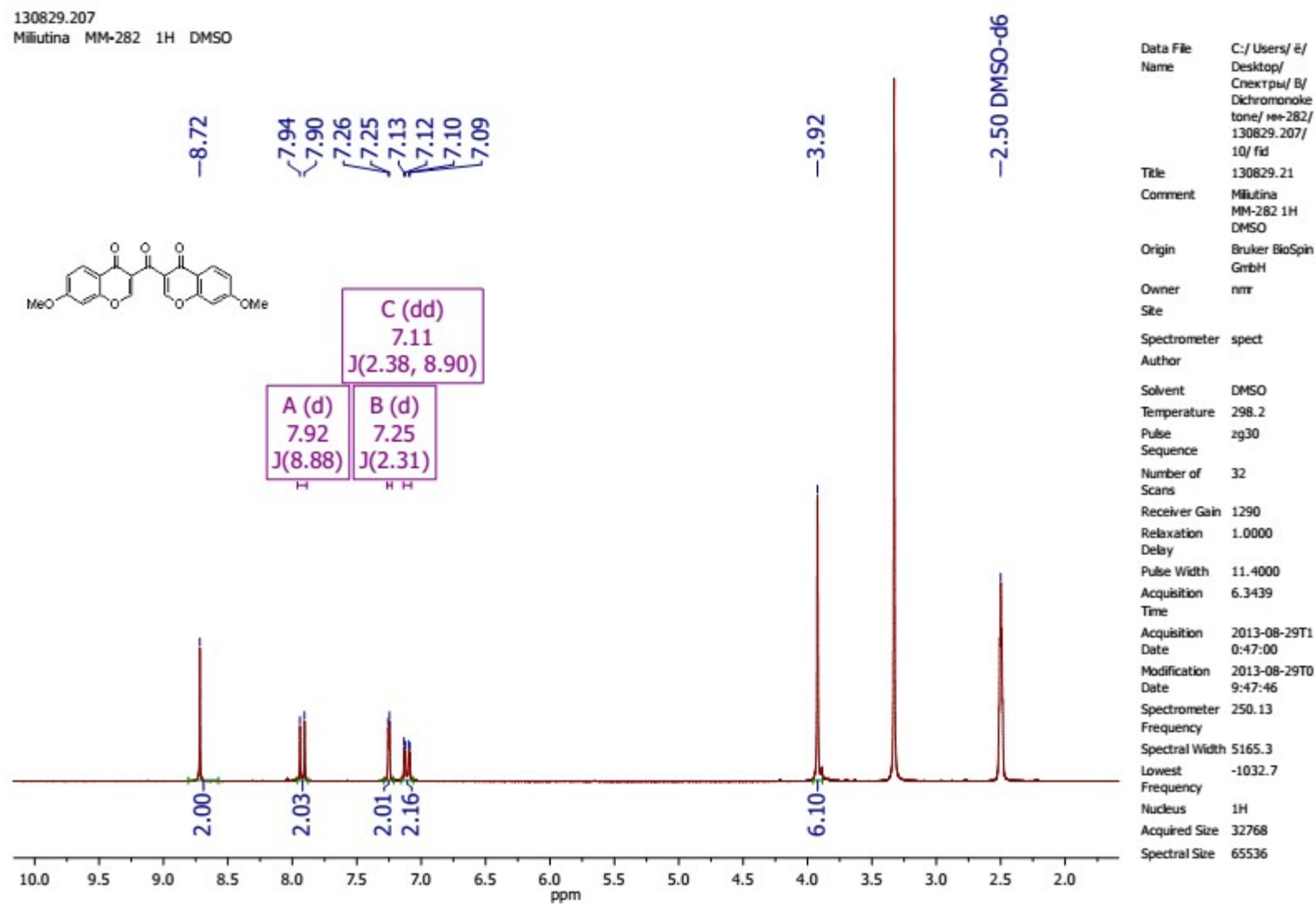
130802.203

Milutina MM-269 DMSO 13C



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Comment	Milutina MM-269 DMSO 13C
Origin	Bruker BioSpin GmbH
Owner	nmr
Site	
Spectrometer	spect
Author	
Solvent	DMSO
Temperature	298.8
Pulse	zgpg30
Sequence	
Number of Scans	1024
Receiver Gain	2050
Relaxation Delay	2.0000
Pulse Width	10.0000
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Modification Date	2013-08-02T 14:53:42
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Lowest Frequency	-1239.9
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

3,3'-carbonylbis(7-methoxy-4H-chromen-4-one) 8g.



3,3'-carbonylbis(7-methoxy-4H-chromen-4-one) 8g.

130829.207
Miliutina MM-282 13C DMSO

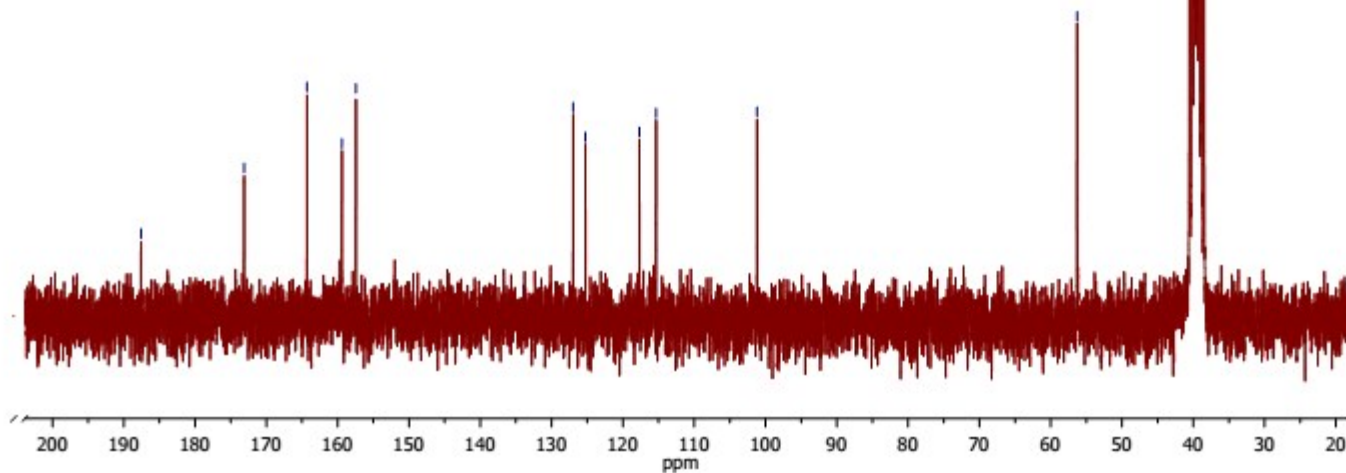
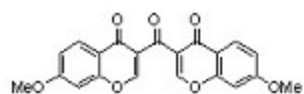
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101.18

56.30

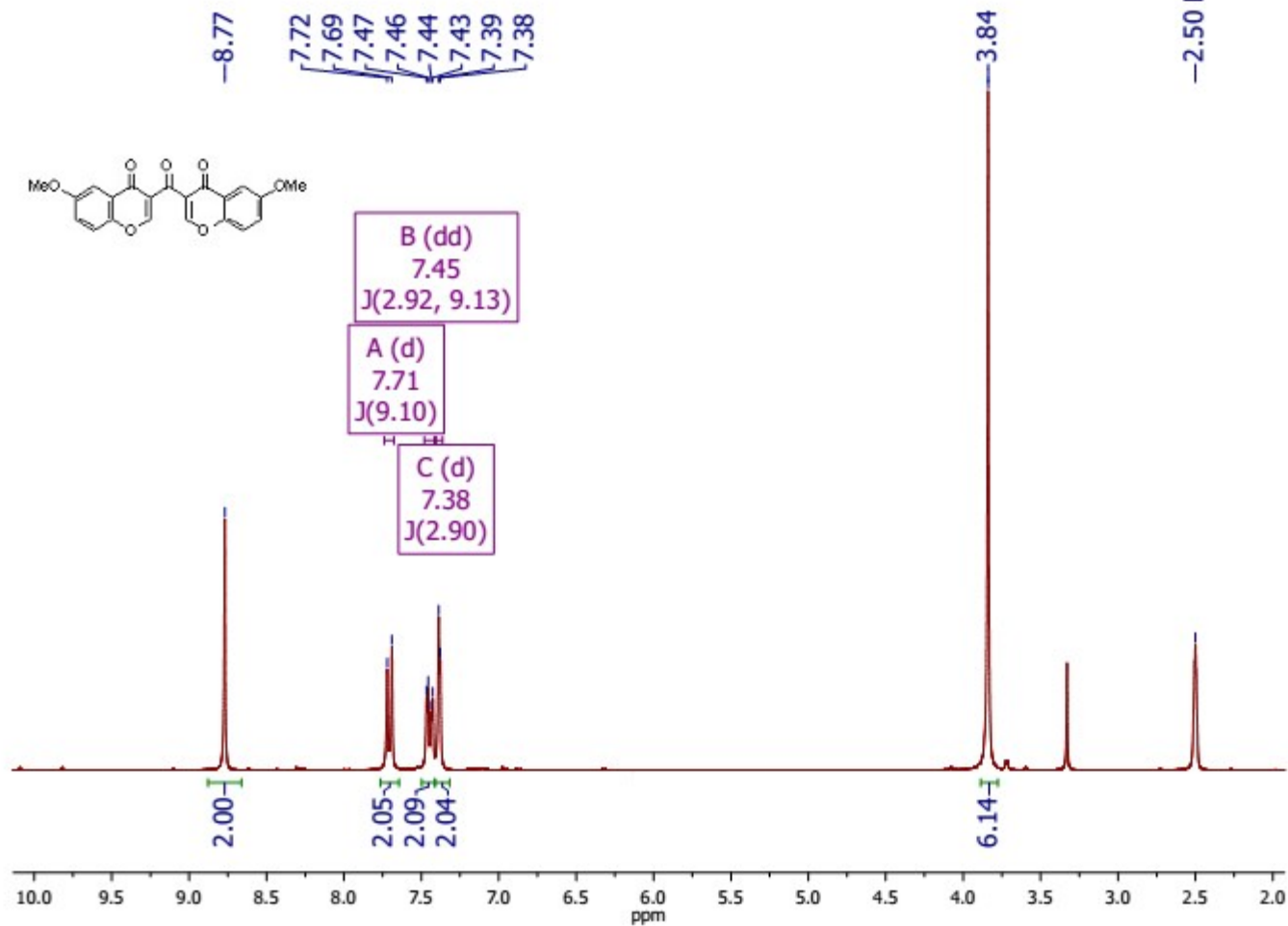
39.52 DMSO-d6



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Comment Miliutina MM-282 13C DMSO
Origin Bruker BioSpin GmbH
Owner nmr
Site
Spectrometer spect
Author
Solvent DMSO
Temperature 298.2
Pulse zgpg30
Sequence
Number of Scans 1024
Receiver Gain 2050
Relaxation Delay 2.0000
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Modification Date 2013-08-29T2 1:06:50
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Nucleus 13C
Acquired Size 32768
Spectral Size 65536

3,3'-carbonylbis(6-methoxy-4H-chromen-4-one) 8h.

140310.u312
Miliutina MM-293-1 1H DMSO



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Title 140310.u312

Comment Miliutina MM-293-1 1H DMSO

Origin Bruker BioSpin GmbH

Owner nmrsu

Site

Spectrometer spect

Author

Solvent DMSO

Temperature 298.2

Pulse zg30

Sequence

Number of Scans 32

Receiver Gain 101

Relaxation 1.0000

Delay

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Acquisition Date 2014-03-10T1 3:49:00

Modification Date 2014-03-10T1 3:49:04

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Spectral Width 6188.1

Lowest -1253.2

Frequency

Nucleus 1H

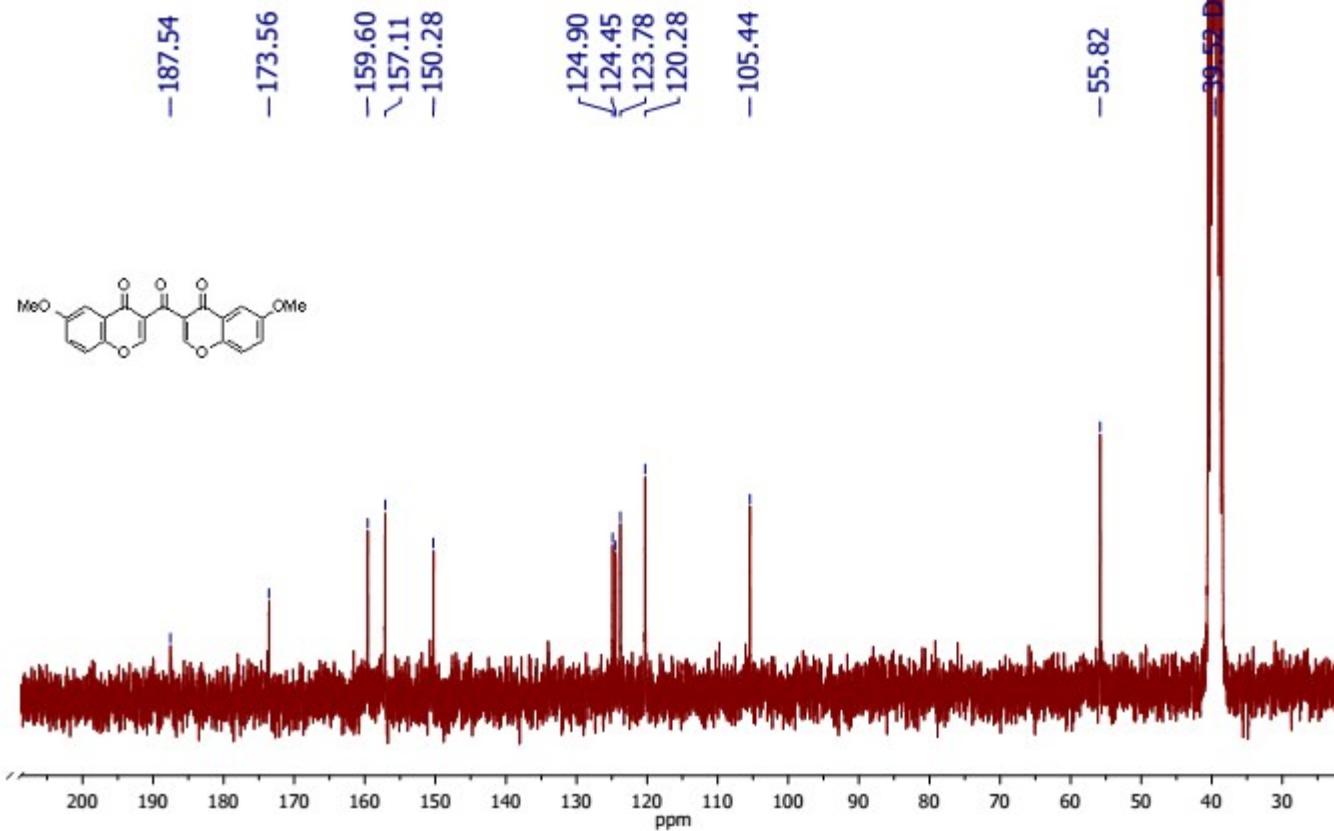
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Spectral Size 65536

3,3'-carbonylbis(6-methoxy-4H-chromen-4-one) 8h.

140310.202

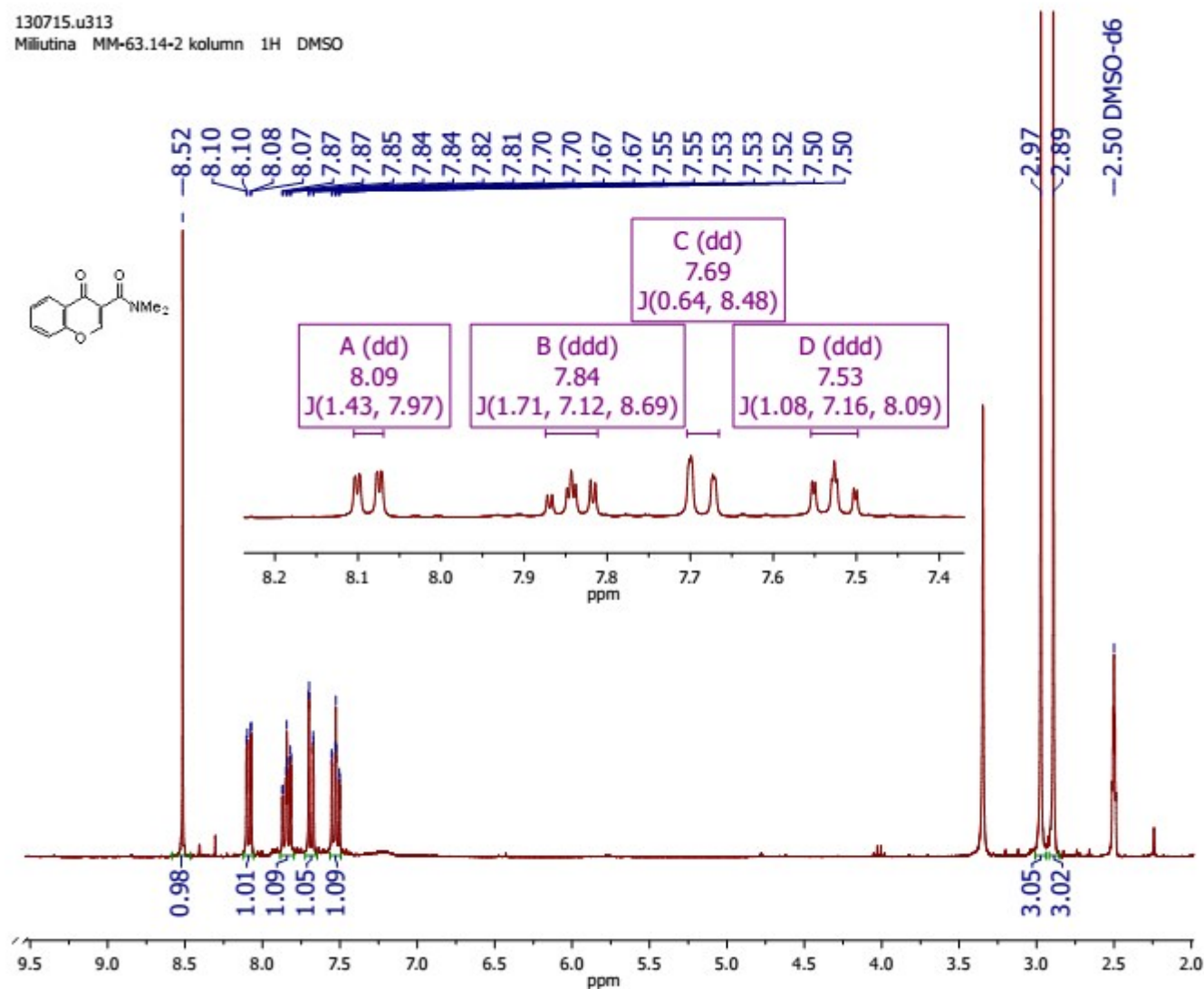
Miliutina MM-293-1 13C DMSO



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Owner	nmr
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Spectrometer	spect
Author	
Solvent	DMSO
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Lowest Frequency	-1239.9
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

***N,N*-Dimethyl-4-oxo-4*H*-chromene-3-carboxamide 12a**

130715.u313
Miliutina MM-63.14-2 kolumn 1H DMSO

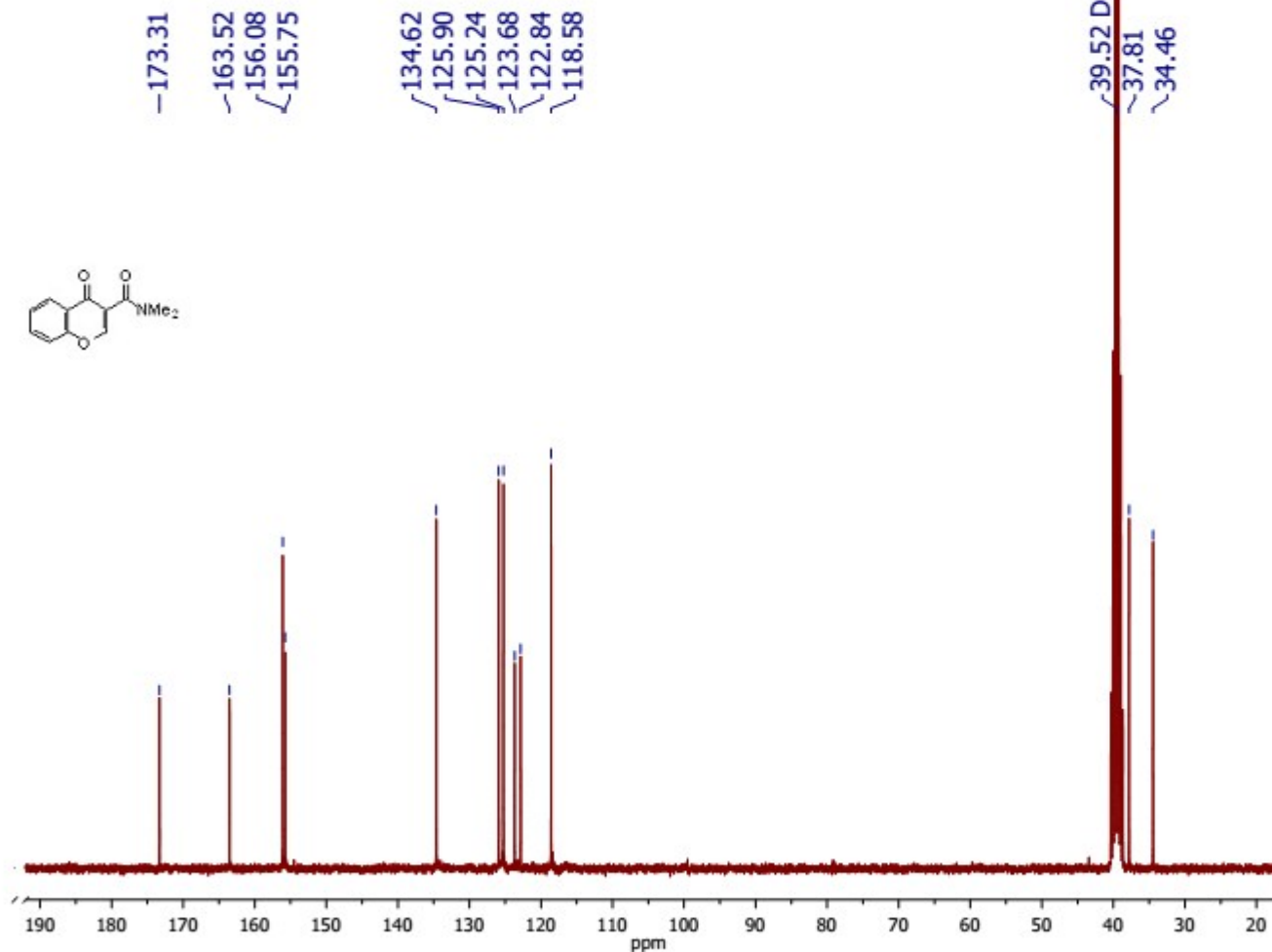
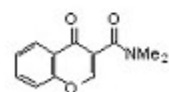


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Comment Miliutina MM-63.14-2 kolumn 1H DMSO
Origin Bruker BioSpin GmbH
Owner nmrsu
Site
Spectrometer spect
Author
Solvent DMSO
Temperature 298.2
Pulse Sequence zg30
Number of Scans 16
Receiver Gain 51
Relaxation Delay 1.0000
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Acquisition Time 5.2954
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Nucleus 1H
Acquired Size 32768
Spectral Size 65536

***N,N*-Dimethyl-4-oxo-4*H*-chromene-3-carboxamide 12a**

130715.u313

Miliutina MM-63.14-2 kolumn 13C DMSO

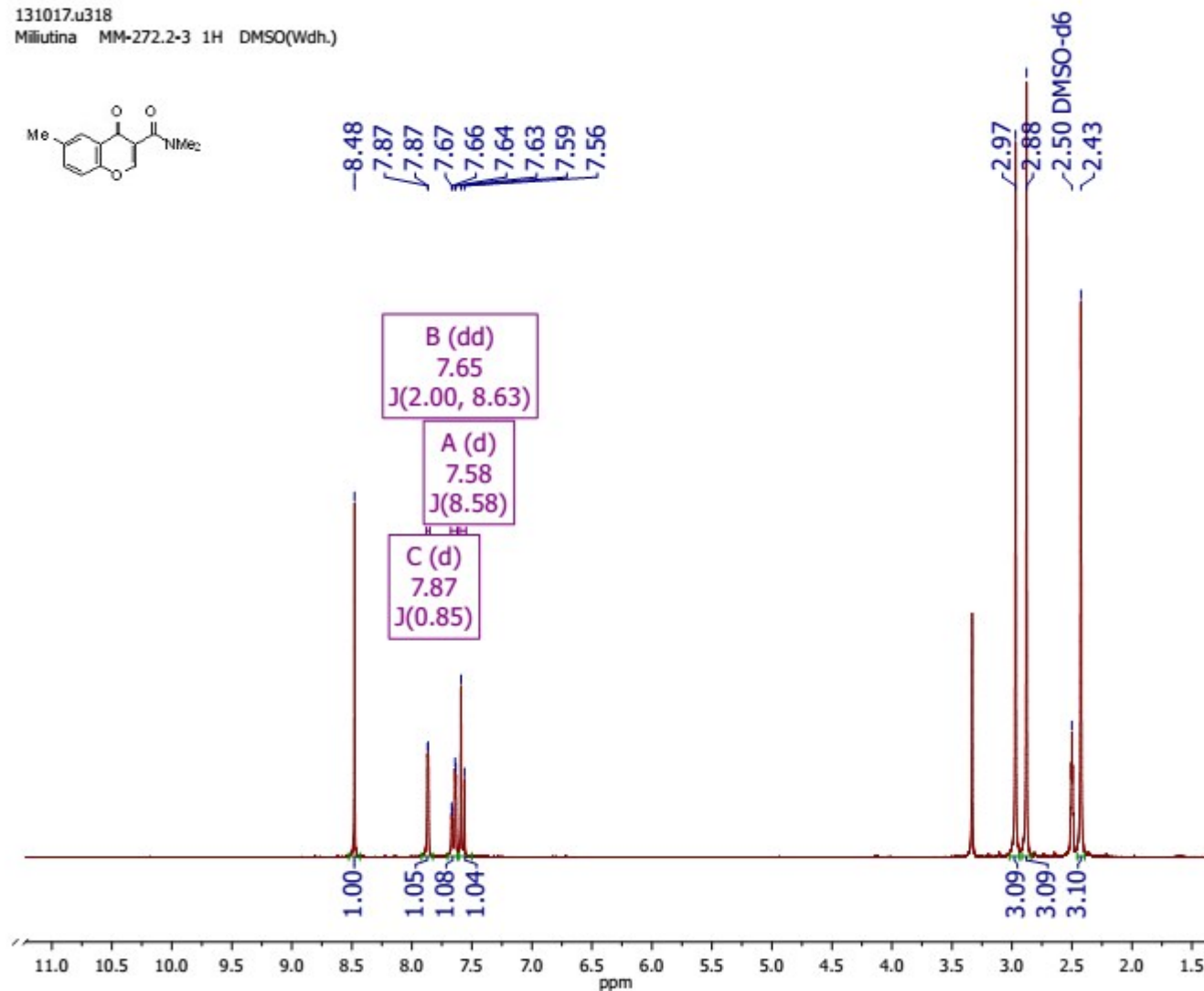
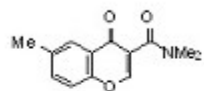


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Origin	Bruker BioSpin GmbH
Owner Site	nmsu
Spectrometer	spect
Author	
Solvent	DMSO
Temperature	298.2
Pulse Sequence	zgpg30
Number of Scans	1024
Receiver Gain	2050
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.8176
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Modification Date	2013-07-16T 02:08:18
Spectrometer Frequency	75.47
Spectral Width	18028.8
Lowest Frequency	-1505.6
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

***N,N*,6-Trimethyl-4-oxo-4*H*-chromene-3-carboxamide 12b**

131017.u318

Miliutina MM-272.2-3 1H DMSO(Wdh.)



Data File Name C:/Users/ē/Desktop/Cнектры/ B/ Dichromonoket one/ mm-272/ 272.2-3/ 131017.u318/ 10/ fid

Title 131017.u318

Comment Miliutina MM-272.2-3 1H DMSO(Wdh.)

Origin Bruker BioSpin GmbH

Owner nmrsu

Site

Spectrometer spect

Author

Solvent DMSO

Temperature 298.2

Pulse zg30

Sequence

Number of Scans 16

Receiver Gain 51

Relaxation Delay 1.0000

Pulse Width 10.0000

Acquisition Time 5.2954

Acquisition Date 2013-10-17T1 1:51:00

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Nucleus 1H

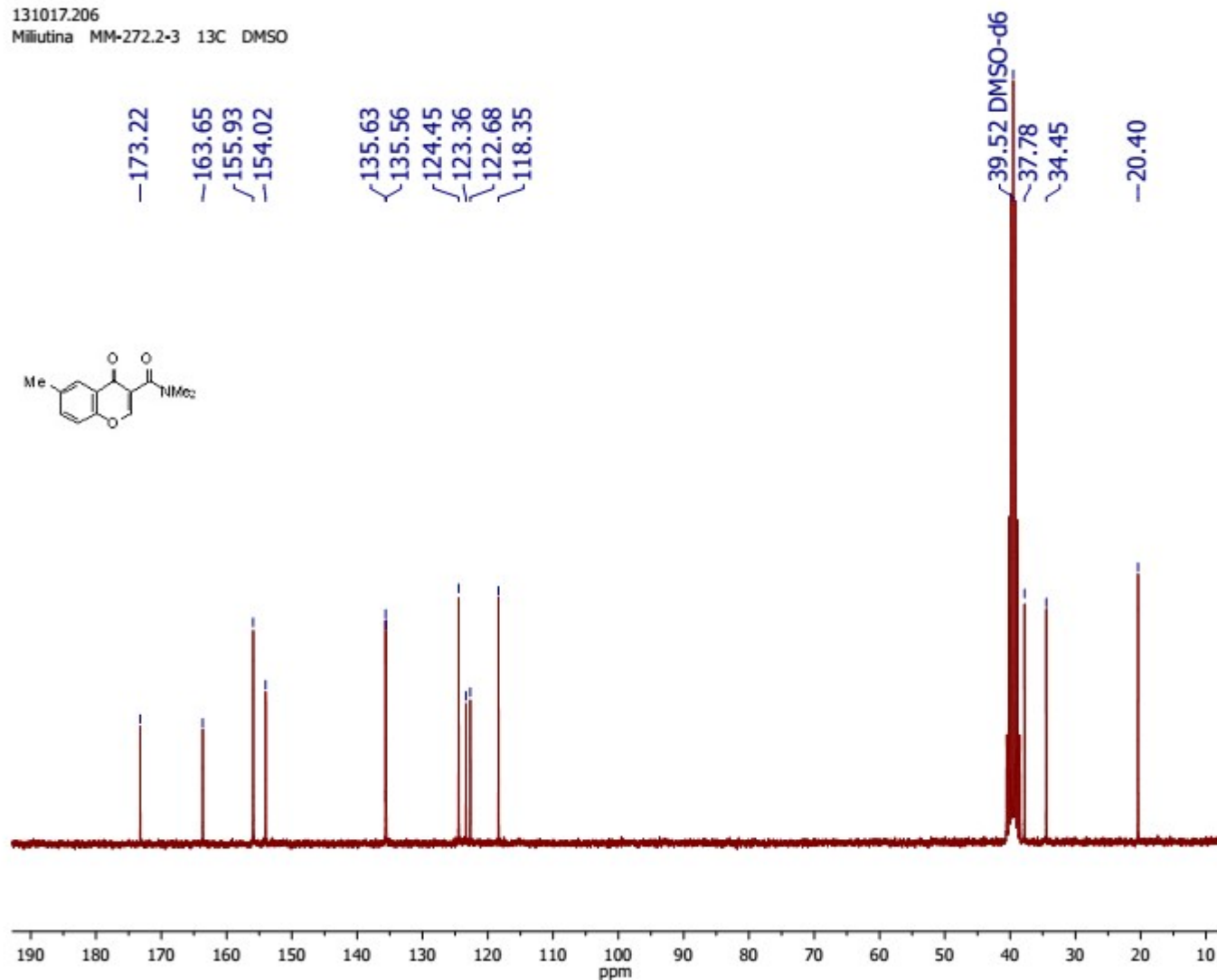
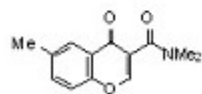
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Spectral Size 65536

***N,N*,6-Trimethyl-4-oxo-4*H*-chromene-3-carboxamide 12b**

131017.206
Miliutina MM-272.2-3 13C DMSO

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163.65
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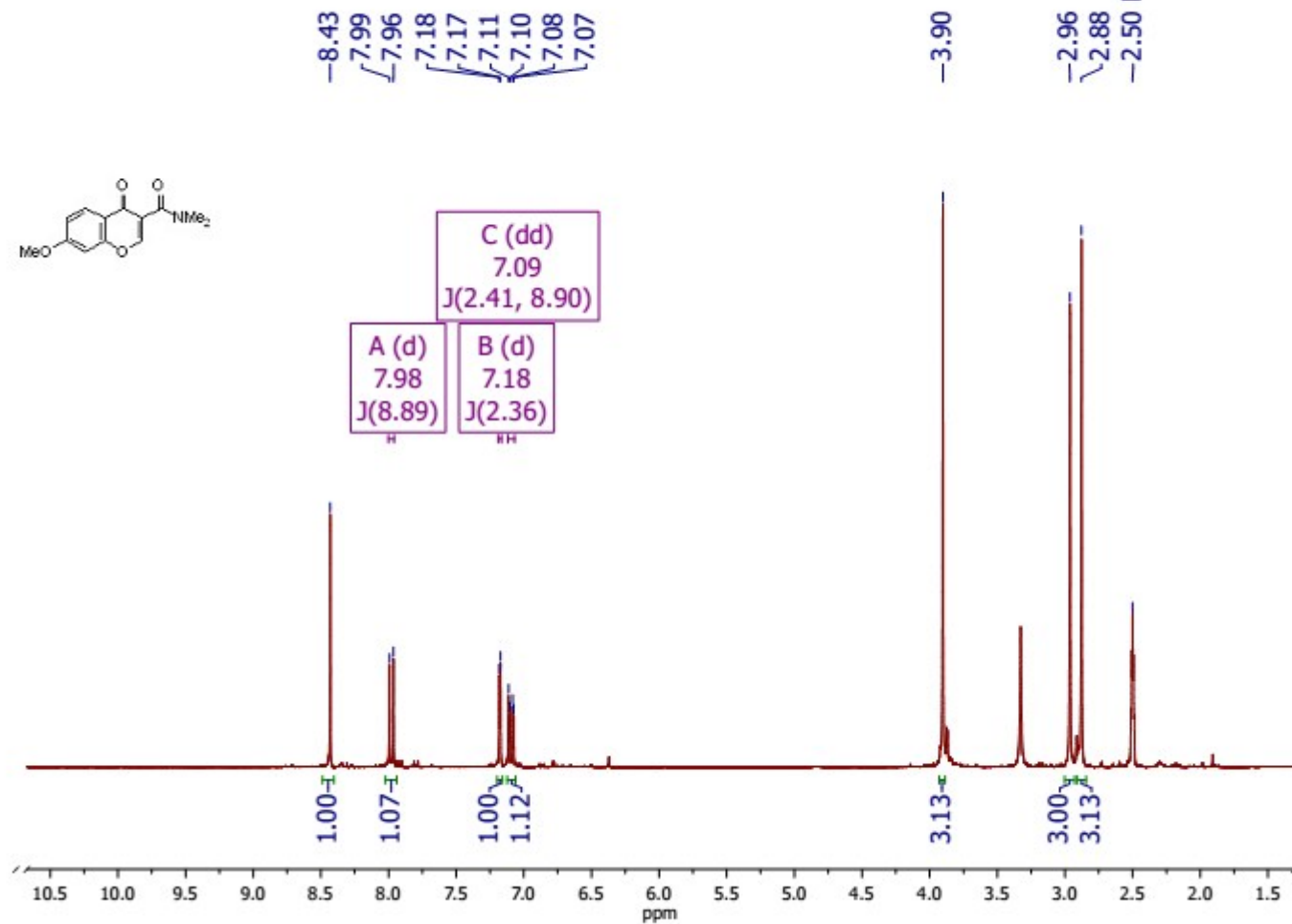
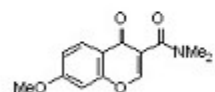


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Owner	nmr
Site	
Spectrometer	spect
Author	
Solvent	DMSO
Temperature	298.0
Pulse	zgpg30
Sequence	
Number of Scans	1024
Receiver Gain	2050
Relaxation Delay	2.0000
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Acquisition Time	2.1846
Acquisition Date	2013-10-18T00:37:00
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Spectral Width	15000.0
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Nucleus	13C
Acquired Size	32768
Spectral Size	65536

7-Methoxy-*N,N*-dimethyl-4-oxo-4*H*-chromene-3-carboxamide 12c

130830.u304

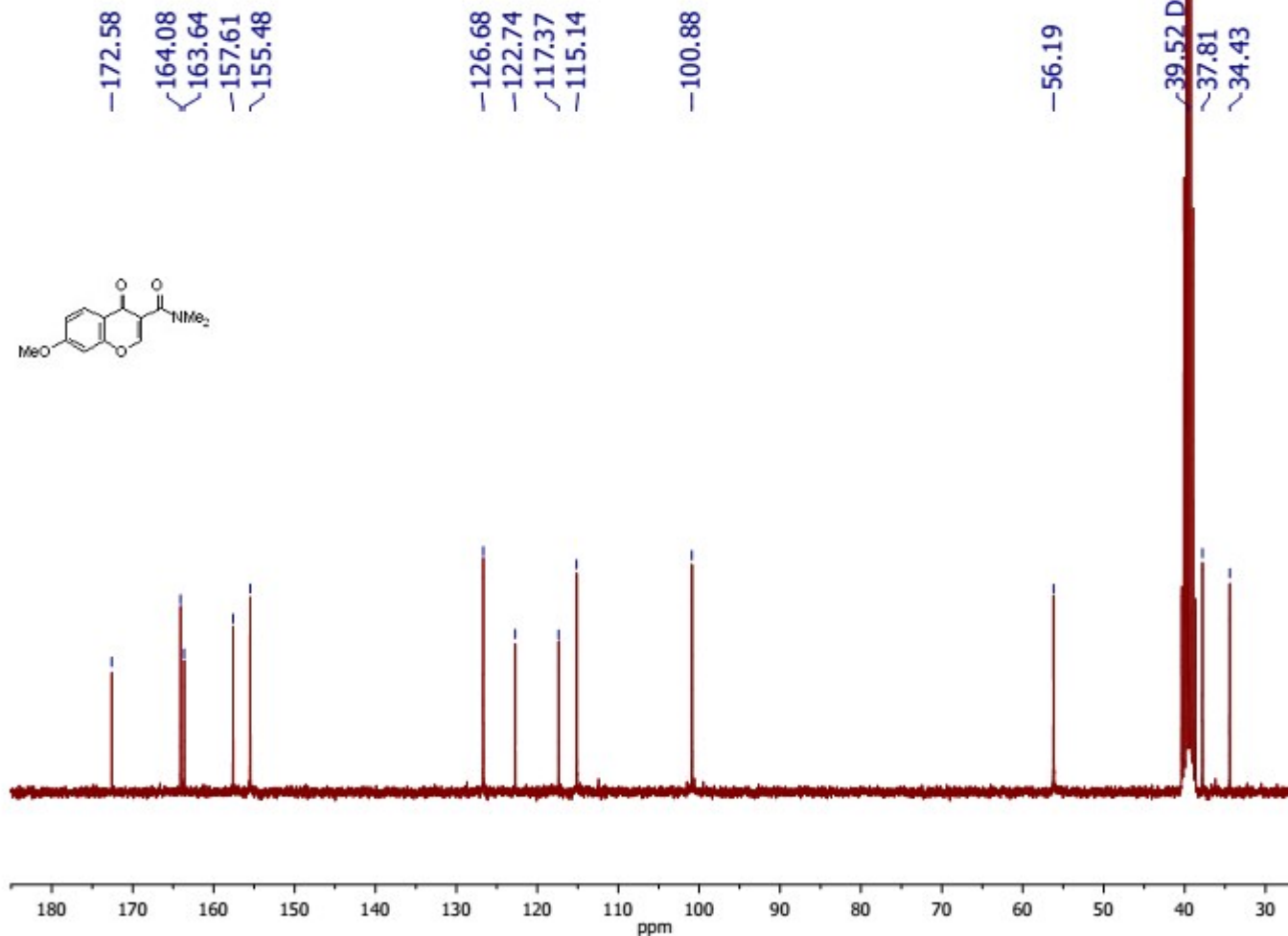
Miliutina MM-282-2 1H DMSO



Data File Name	C:/Users/é/Desktop/Спектры/ B/ Dichromonok etone/ me-282/ 282-2/ 130830.u304 / 10/ fid
Title	130830.u304
Comment	Miliutina MM-282-2 1H DMSO
Origin	Bruker BioSpin GmbH
Owner	nmrsu
Site	
Spectrometer	spect
Author	
Solvent	DMSO
Temperature	298.2
Pulse Sequence	zg30
Number of Scans	16
Receiver Gain	90
Relaxation Delay	1.0000
Pulse Width	10.0000
Acquisition Time	5.2954
Acquisition Date	2013-08-30T 10:30:00
Modification Date	2013-08-30T 09:30:22
Spectrometer Frequency	300.13
Spectral Width	6188.1
Lowest Frequency	-1255.3
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

7-Methoxy-*N,N*-dimethyl-4-oxo-4*H*-chromene-3-carboxamide 12c

130830.u304
Miliutina MM-282-2 13C DMSO

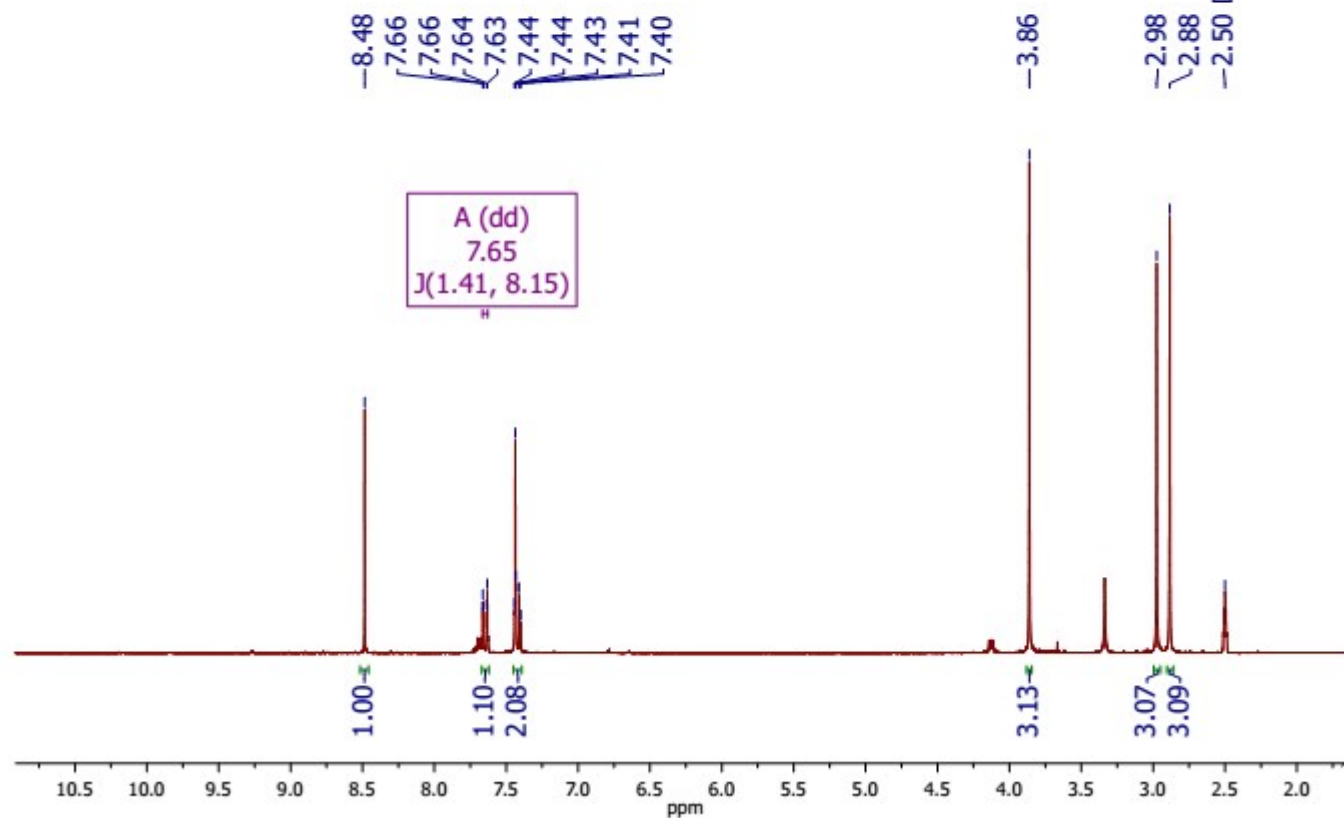
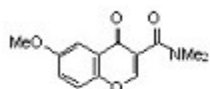


Data File Name	C:/Users/ē/Desktop/Cnextpa/B/Dichromonoketone/MM-282/282-2/130830.u304/11/fid
Title	130830.u304
Comment	Miliutina MM-282-2 13C DMSO
Origin	Bruker BioSpin GmbH
Owner	nmrsu
Site	
Spectrometer	spect
Author	
Solvent	DMSO
Temperature	298.2
Pulse Sequence	zgpg30
Number of Scans	1024
Receiver Gain	2050
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.8176
Acquisition Date	2013-08-31T 21:51:00
Modification Date	2013-08-31T 20:51:58
Spectrometer Frequency	75.47
Spectral Width	18028.8
Lowest Frequency	-1506.6
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

6-Methoxy-*N,N*-dimethyl-4-oxo-4*H*-chromene-3-carboxamide 12d

131122.u308

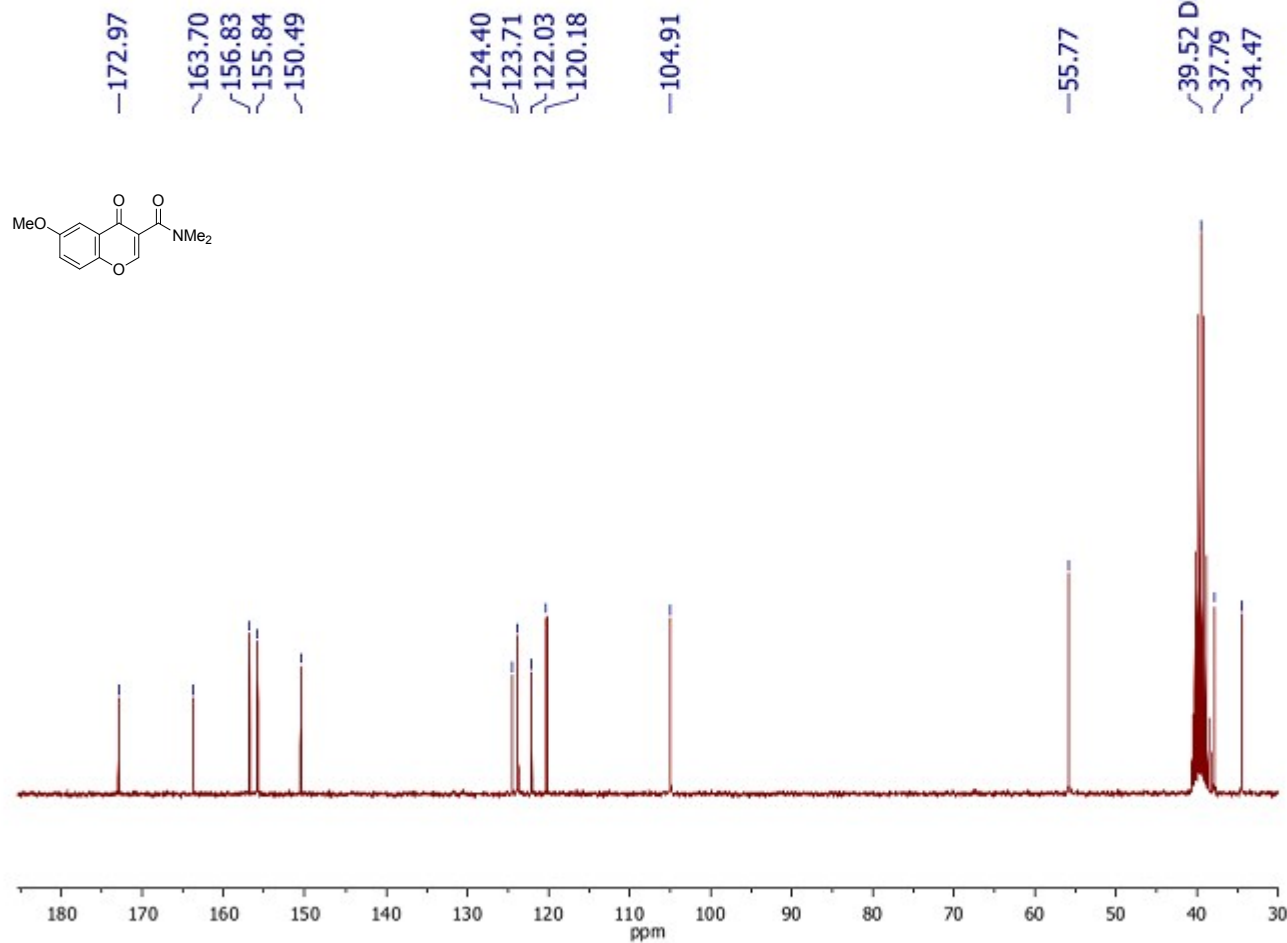
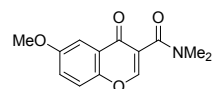
Milutina MM-293-3 1H DMSO



Data File Name	C:/Users/E/Desktop/Спектры/ B/ Dichromonoketone/ MM-293/ 293-3/ 131122.u308/ 10/ fid
Title	131122.u308
Comment	Milutina MM-293-3 1H DMSO
Origin	Bruker BioSpin GmbH
Owner	nmrsu
Site	
Spectrometer	spect
Author	
Solvent	DMSO
Temperature	298.2
Pulse	zg30
Sequence	
Number of Scans	16
Receiver Gain	51
Relaxation Delay	1.0000
Pulse Width	10.0000
Acquisition Time	5.2954
Acquisition Date	2013-11-22T08:38:00
Modification Date	2013-11-22T08:38:34
Spectrometer Frequency	300.13
Spectral Width	6188.1
Lowest Frequency	-1252.3
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

6-Methoxy-*N,N*-dimethyl-4-oxo-4*H*-chromene-3-carboxamide 12d

131122.216
Miliutina MM-293-3 13C DMSO

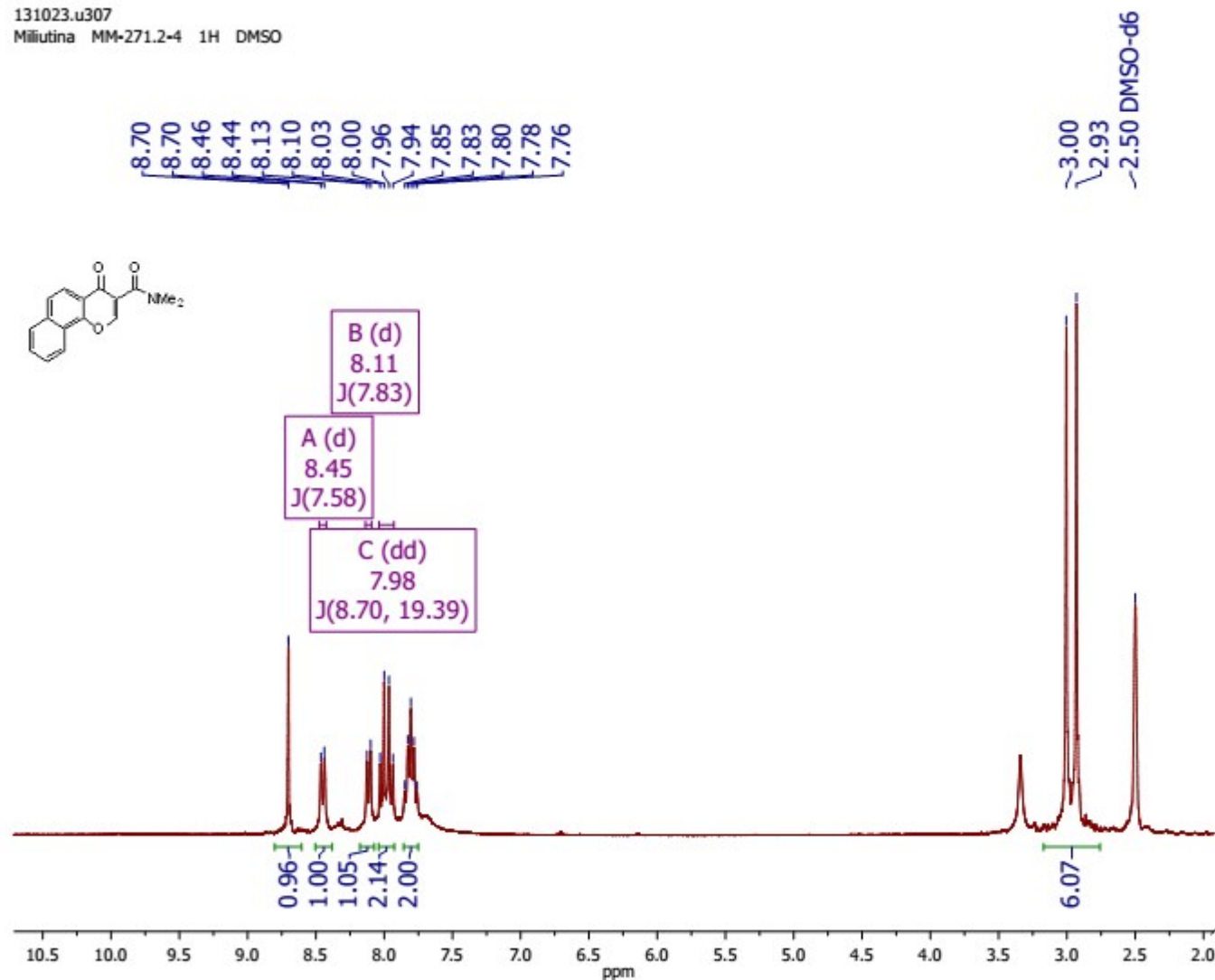


Data File Name: C:/Users/ë/Desktop/Cнекрпы/ B/ Dichromonoket one/ see-293/ 293-3/ 131122.216/ 10/ ftd
Title: 131122.22
Comment: Miliutina MM-293-3 13C DMSO
Origin: Bruker BioSpin GmbH
Owner: nmr
Site:
Spectrometer: spect
Author:
Solvent: DMSO
Temperature: 298.0
Pulse: zgpg30
Sequence:
Number of Scans: 1024
Receiver Gain: 2050
Relaxation Delay: 2.0000
Pulse Width: 10.0000
Acquisition Time: 2.1846
Acquisition Date: 2013-11-23T1 1:56:00
Modification Date: 2013-11-23T1 1:57:06
Spectrometer Frequency: 62.90
Spectral Width: 15000.0
Lowest Frequency: -1239.4
Nucleus: 13C
Acquired Size: 32768
Spectral Size: 65536

N,N-Dimethyl-4-oxo-4*H*-benzo[*h*]chromene-3-carboxamide 12e

131023.u307

Miliutina MM-271.2-4 1H DMSO

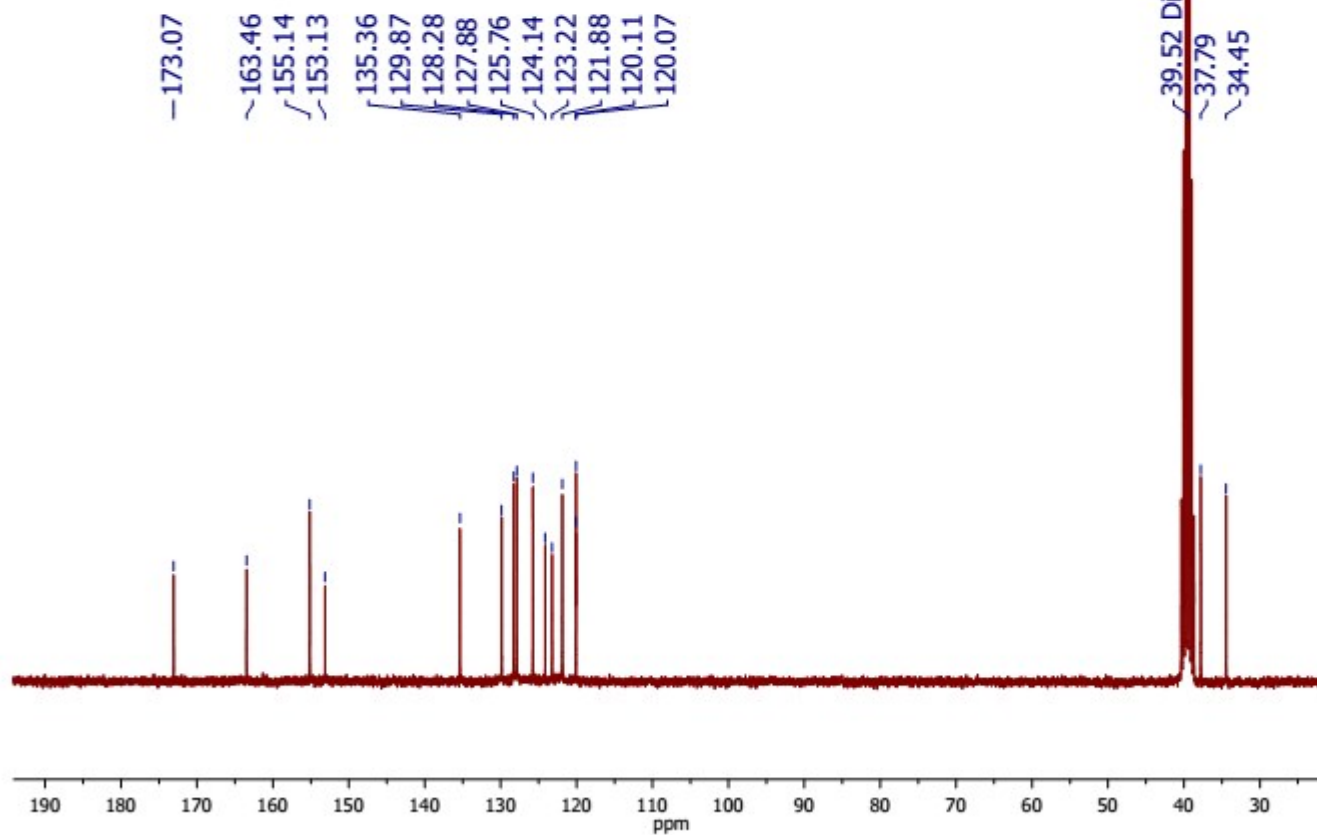
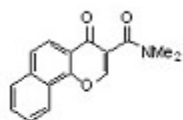


Data File Name	C:/Users/ij/Desktop/Спектры/ B/ Dichromonoke tone/ mm-271/ 271.2-4/ 131023.u307/ 10/ fid
Title	131023.u307
Comment	Miliutina MM-271.2-4 1H DMSO
Origin	Bruker BioSpin GmbH
Owner	nmrsu
Site	
Spectrometer	spect
Author	
Solvent	DMSO
Temperature	298.2
Pulse Sequence	zg30
Number of Scans	16
Receiver Gain	90
Relaxation Delay	1.0000
Pulse Width	10.0000
Acquisition Time	5.2954
Acquisition Date	2013-10-23T09:53:00
Modification Date	2013-10-23T08:53:46
Spectrometer Frequency	300.13
Spectral Width	6188.1
Lowest Frequency	-1316.3
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

***N,N*-Dimethyl-4-oxo-4*H*-benzo[*h*]chromene-3-carboxamide 12e**

131023.u307

Milutina MM-271.2-4 13C DMSO

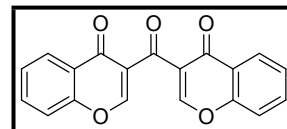


Data File Name	C:/Users/e/Desktop/Cnecrps/B/Dichromonoketone/mm-271/271.2-4/131023.u307/11/fid
Title	131023.u307
Comment	Milutina MM-271.2-4 13C DMSO
Origin	Bruker BioSpin GmbH
Owner Site	nmrsu
Spectrometer	spect
Author	
Solvent	DMSO
Temperature	298.2
Pulse	zgpg30
Sequence	
Number of Scans	1024
Receiver Gain	2050
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.8176
Acquisition Date	2013-10-23T 22:36:00
Modification Date	2013-10-23T 21:36:36
Spectrometer Frequency	75.47
Spectral Width	18028.8
Lowest Frequency	-1506.6
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

X-Ray structures of compounds 8a and 8h

X-Ray structure of compound 8a

Identification code	is_mm62	
Empirical formula	C ₁₉ H ₁₀ O ₅	
Formula weight	318.27	
Temperature	173(2) K	
Wavelength	0.71073 Å	
Crystal system	monoclinic	
Space group (H.-M.)	<i>P</i> 2 ₁ / <i>c</i>	
Space group (Hall)	- <i>P</i> 2 ₁ yc	
Unit cell dimensions	<i>a</i> = 16.6528(7) Å	α = 90.00°
	<i>b</i> = 6.6881(3) Å	β = 113.809(2)°
	<i>c</i> = 13.5348(6) Å	γ = 90.00°
Volume	1379.16(10) Å ³	
Z	4	
Calculated density	1.533 mg/m ³	
Absorption coefficient	0.112 mm ⁻¹	
F(000)	656	
Crystal size	0.61 x 0.19 x 0.10 mm ³	
θ range for data collection	2.67 to 29.99°	
Index ranges	-22 ≤ <i>h</i> ≤ 23, -9 ≤ <i>k</i> ≤ 9, -19 ≤ <i>l</i> ≤ 19	
Reflections collected	15596	
Independent reflections	4026 [<i>R</i> (int) = 0.0218]	
Completeness to θ = 28.00°	100.0%	
Absorption correction	Multi-scan	
Max. and min. transmission	0.9889 and 0.9346	
Refinement method	Full-matrix least-squares on <i>F</i> ²	
Data / restraints / parameters	3478 / 0 / 217	
Goodness-of-fit on <i>F</i> ²	1.021	
Final <i>R</i> indices [<i>I</i> > 2σ(<i>I</i>)]	<i>R</i> 1 = 0.0410, <i>wR</i> 2 = 0.1032	
<i>R</i> indices (all data)	<i>R</i> 1 = 0.0484, <i>wR</i> 2 = 0.1076	
Largest diff. peak and hole	0.357 and -0.207 e.Å ⁻³	



X-Ray structure of compound 8h

Identification code	ah_mm293-1
Empirical formula	C ₂₁ H ₁₄ O ₇
Formula weight	378.32
Temperature	173(2) K
Wavelength	0.71073 Å
Crystal system	triclinic
Space group (H.-M.)	$P\bar{1}$
Space group (Hall)	-P 1
Unit cell dimensions	$a = 6.9459(3) \text{ Å}$ $b = 8.6534(4) \text{ Å}$ $c = 14.8426(7) \text{ Å}$
	$\alpha = 74.281(2)^\circ$ $\beta = 81.211(2)^\circ$ $\gamma = 68.824(2)^\circ$
Volume	799.27(6) Å ³
Z	2
Calculated density	1.572 mg/m ³
Absorption coefficient	0.120 mm ⁻¹
F(000)	392
Crystal size	0.270 x 0.160 x 0.070 mm ³
θ range for data collection	2.654 to 28.997°
Index ranges	$-9 \leq h \leq 9, -11 \leq k \leq 11, -20 \leq l \leq 20$
Reflections collected	20387
Independent reflections	4243 [R(int) = 0.0268]
Completeness to $\theta = 28.00^\circ$	99.8%
Absorption correction	Multi-scan
Max. and min. transmission	0.7460 and 0.7029
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	3472 / 0 / 255
Goodness-of-fit on F ²	1.062
Final R indices [$I > 2\sigma(I)$]	R1 = 0.0387, wR2 = 0.0984
R indices (all data)	R1 = 0.0491, wR2 = 0.1057
Largest diff. peak and hole	0.395 and -0.241 e.Å ⁻³

