

Synthesis of novel polycyano-containing organic ligands via double carbanion cleavage of 1',3'-dioxo-1',3'-dihydrospiro[cyclopropane-1,2'-indene] derivatives

Supporting information

Sergey V. Karpov,^{a*} Yakov S. Kaukov,^a Arthur A. Grigor'ev,^a Oleg E. Nasakin,^a Olga V. Kaukova^b, Viktor A. Tafeenko^c

- a** Dr. S. V. Karpov, Dr. Ya. S. Kaukov, Dr. A. A. Grigor'ev, Prof. Dr. O. E. Nasakin
Department of Chemistry and Pharmacy
I.N. Ul'yanov Chuvash state University
Moskovskii pr. 15, Cheboksary, 428015 (Russia)
E-mail: serg31.chem@mail.ru
- b** Dr. O. V. Kaukova
Chuvash State Agricultural Academy
K. Marx St., 29. Cheboksary, 428015 (Russia)
- c** Prof. Dr. V. A. Tafeenko
Department of Chemistry
Lomonosov Moscow State University
Leninskiye Gory 1-3, Moscow, 119991 (Russia)

TABLE OF CONTENTS

1',3'-Dioxo-1',3'-dihydrospiro[cyclopropane-1,2'-indene]-2,2,3,3-tetracarbonitrile (1)	2	Potassium 1,1,3,3-tetracyano-2-(2'-(isobutoxycarbonyl)benzoyl)prop-2-en-1-ide (3f)	14
Dimethyl 2,3-dicyano-1',3'-dioxo-1',3'-dihydrospiro[cyclopropane-1,2'-indene]-2,3-dicarboxylate (2)	3	Potassium (Z)-2,4-dicyano-1,5-dimethoxy-3-(2'-(methoxycarbonyl)benzoyl)-1,5-dioxopent-3-en-2-ide (4a)	16
Potassium 1,1,3,3-tetracyano-2-(2'-(methoxycarbonyl)benzoyl)prop-2-en-1-ide (3a)	4	Potassium (Z)-2,4-dicyano-1,5-dimethoxy-3-(2'-(ethoxycarbonyl)benzoyl)-1,5-dioxopent-3-en-2-ide (4b)	18
Potassium 1,1,3,3-tetracyano-2-(2'-(ethoxycarbonyl)benzoyl)prop-2-en-1-ide (3b)	6	Potassium (Z)-2,4-dicyano-1,5-dimethoxy-3-(2'-(propoxycarbonyl)benzoyl)-1,5-dioxopent-3-en-2-ide (4c)	20
Potassium 1,1,3,3-tetracyano-2-(2'-(propoxycarbonyl)benzoyl)prop-2-en-1-ide (3c)	8	Potassium (Z)-2,4-dicyano-1,5-dimethoxy-3-(2'-(isopropoxycarbonyl)benzoyl)-1,5-dioxopent-3-en-2-ide (4d)	22
Potassium 1,1,3,3-tetracyano-2-(2'-(isopropoxycarbonyl)benzoyl)prop-2-en-1-ide (3d)	10	Potassium (Z)-2,4-dicyano-1,5-dimethoxy-3-(2'-(butoxycarbonyl)benzoyl)-1,5-dioxopent-3-en-2-ide (4e)	24
Rubidium 1,1,3,3-tetracyano-2-(2'-(isopropoxycarbonyl)benzoyl)prop-2-en-1-ide (3d_Rb)	10	Potassium (Z)-2,4-dicyano-1,5-dimethoxy-3-(2'-(isobutoxycarbonyl)benzoyl)-1,5-dioxopent-3-en-2-ide (4f)	26
Potassium 1,1,3,3-tetracyano-2-(2'-(butoxycarbonyl)benzoyl)prop-2-en-1-ide (3e)	12		

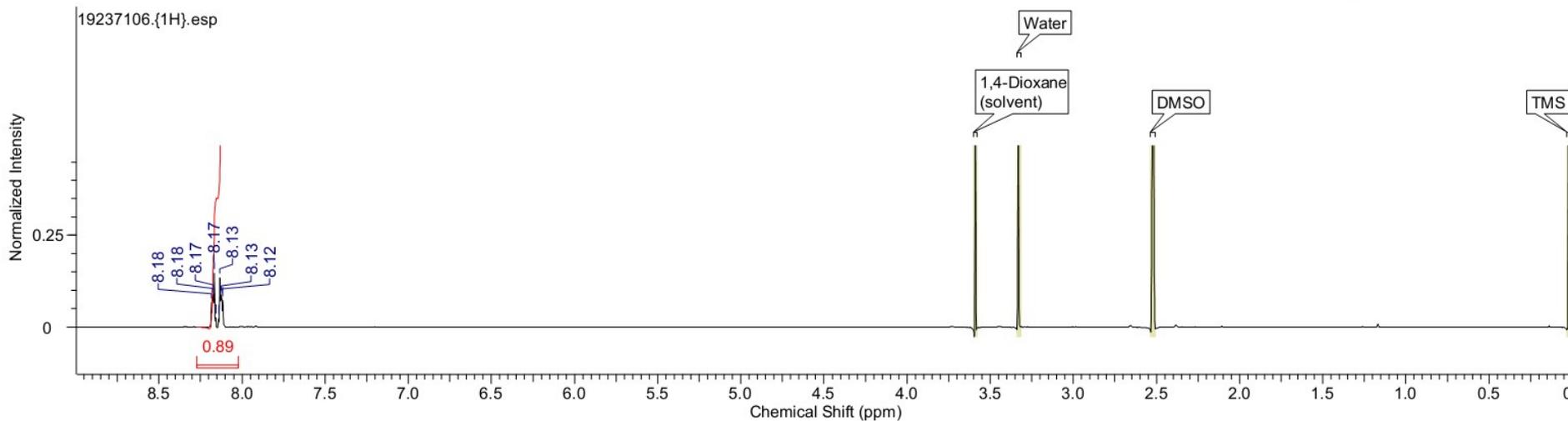
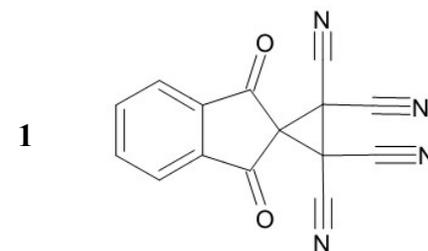
1',3'-Dioxo-1',3'-dihydrospiro[cyclopropane-1,2'-indene]-2,2,3,3-tetracarbonitrile (1)

Formula C₁₅H₄N₄O₂ FW 272.2179

Acquisition Time (sec) 1.6312 Comment NMR/19237106 Opr: Strelenko Yu.A.; Prep: L-7571; Date 16 Apr 2009 20:38:24

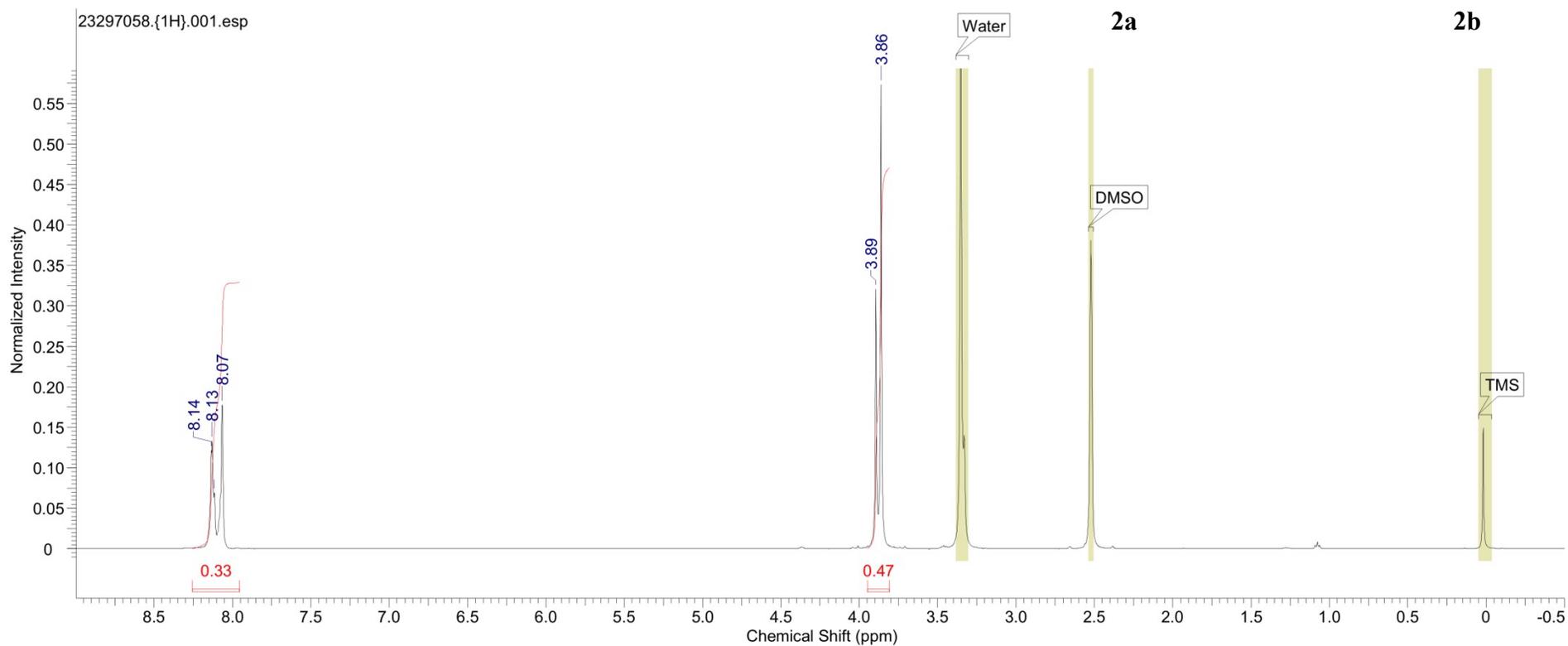
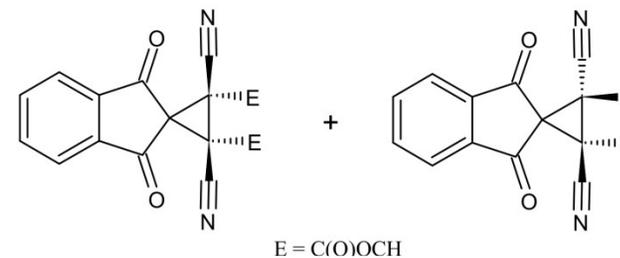
Date Stamp 16 Apr 2009 20:38:24

Nucleus	1H	Number of Transients	1	Origin	spect	Frequency (MHz)	500.13
Owner	strel	Points Count	16384	Pulse Sequence	zg	Original Points Count	16312
SW(cyclical) (Hz)	10000.00	Solvent	DMSO-d6	Spectrum Offset (Hz)	4005.8181	Receiver Gain	128.00
Sweep Width (Hz)	9999.39	Temperature (degree C)	26.900	Spectrum Type	STANDARD		



Dimethyl 2,3-dicyano-1',3'-dioxo-1',3'-dihydrospiro[cyclopropane-1,2'-indene]-2,3-dicarboxylate (2)

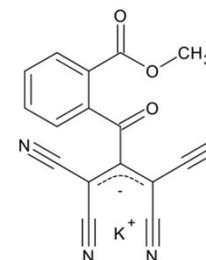
Acquisition Time (sec)	1.6312	Comment	NMR/23297058 Opr: Strelenko Yu.A.; Prep: L-8429;		Date	01 Oct 2013 17:47:44	
Date Stamp	01 Oct 2013 17:47:44						
Frequency (MHz)	500.13	Nucleus	1H	Number of Transients	1	Origin	spect
Original Points Count	16312	Owner	strel	Points Count	16384	Pulse Sequence	zg
Receiver Gain	128.00	SW(cyclical) (Hz)	10000.00	Solvent	DMSO-d6	Spectrum Offset (Hz)	4005.8181
Spectrum Type	STANDARD	Sweep Width (Hz)	9999.39	Temperature (degree C)	25.000		



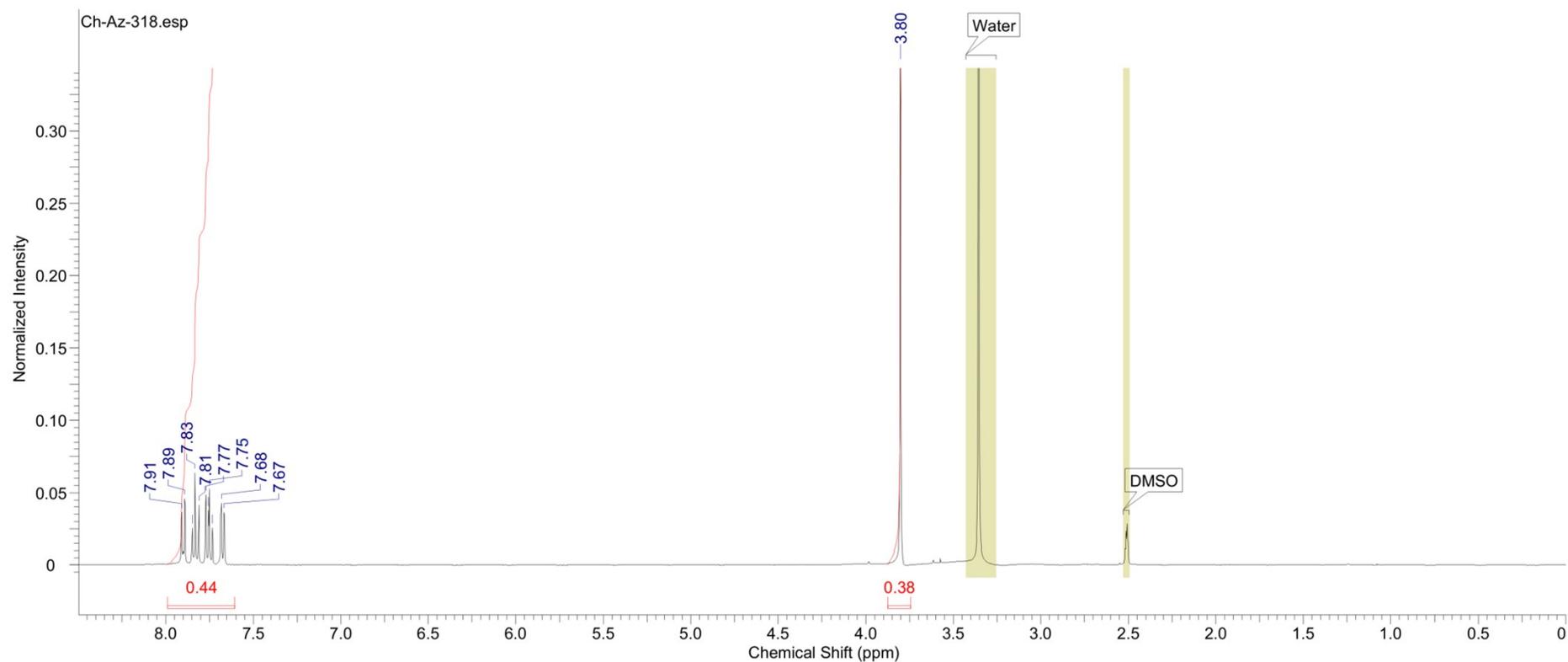
Potassium 1,1,3,3-tetracyano-2-(2'-(methoxycarbonyl)benzoyl)prop-2-en-1-ide (3a)

Formula C ₁₆ H ₇ KN ₄ O ₃	FW 342.3512 (303.2534+39.0978)
--	---------------------------------------

Acquisition Time (sec) 0.9961	Date 26 Oct 2015 13:08:00	Frequency (MHz) 400.17	Date Stamp 26 Oct 2015 13:08:00
Origin spect	Original Points Count 8192	Owner nmr	Nucleus 1H
Receiver Gain 106.79	SW(cyclical) (Hz) 8223.68	Solvent DMSO-d6	Points Count 8192
Sweep Width (Hz) 8222.68	Temperature (degree C) 22.881	Spectrum Offset (Hz) 3201.3608	Number of Transients 32
			Pulse Sequence zg
			Spectrum Type STANDARD



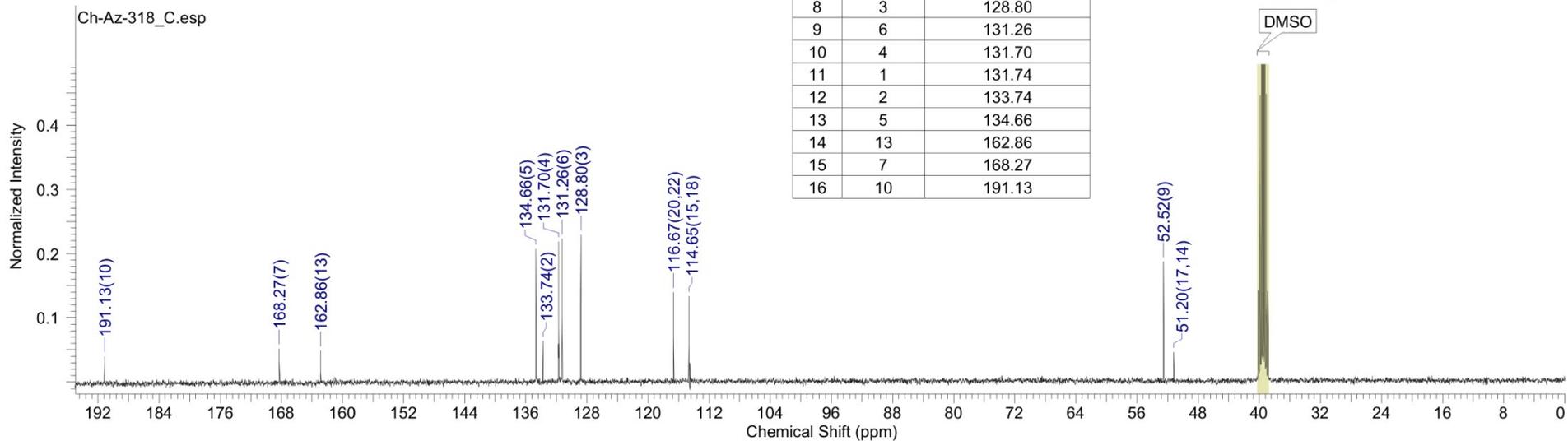
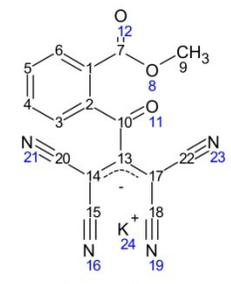
3a



Formula C₁₆H₇KN₄O₃ **FW** 342.3512 (303.2534+39.0978)

Acquisition Time (sec)	0.6816	Date	27 Oct 2015 02:49:20	Date Stamp	27 Oct 2015 02:49:20
Origin	spect	Original Points Count	16384	Frequency (MHz)	100.62
Receiver Gain	189.90	SW(cyclical) (Hz)	24038.46	Nucleus	13C
Sweep Width (Hz)	24036.99	Temperature (degree C)	23.810	Owner	nmr
				Points Count	16384
				Solvent	DMSO-d6
				Spectrum Offset (Hz)	11521.3105
				Number of Transients	2000
				Pulse Sequence	zgpgg
				Spectrum Type	STANDARD

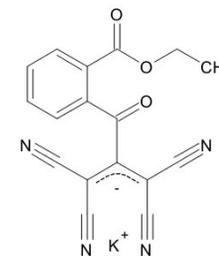
No.	Atom	Exp. Shift (ppm)
1	17	51.20
2	14	51.20
3	9	52.52
4	15	114.65
5	18	114.65
6	20	116.67
7	22	116.67
8	3	128.80
9	6	131.26
10	4	131.70
11	1	131.74
12	2	133.74
13	5	134.66
14	13	162.86
15	7	168.27
16	10	191.13



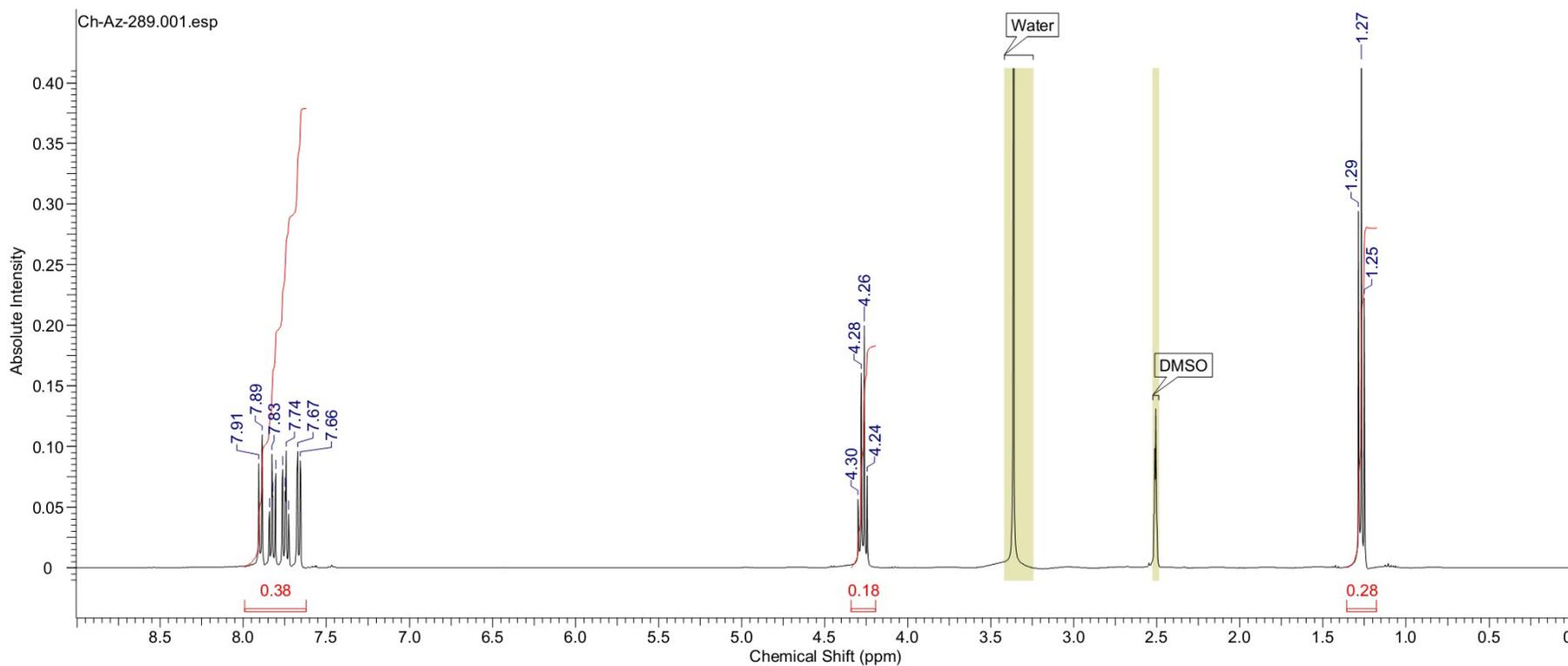
Potassium 1,1,3,3-tetracyano-2-(2'-(ethoxycarbonyl)benzoyl)prop-2-en-1-ide (3b)

Formula C ₁₇ H ₉ KN ₄ O ₃	FW 356.3778 (317.2800+39.0978)
--	---------------------------------------

Acquisition Time (sec) 0.9961	Date 18 Sep 2015 09:32:32	Date Stamp 18 Sep 2015 09:32:32
Number of Transients 16	Origin spect	Original Points Count 8192
Pulse Sequence zg	Receiver Gain 106.79	SW(cyclical) (Hz) 8223.68
Spectrum Type STANDARD	Sweep Width (Hz) 8222.68	Temperature (degree C) 19.771
	Frequency (MHz) 400.17	Nucleus 1H
	Owner nmr	Points Count 8192
	Solvent DMSO-d6	Spectrum Offset (Hz) 3201.3608

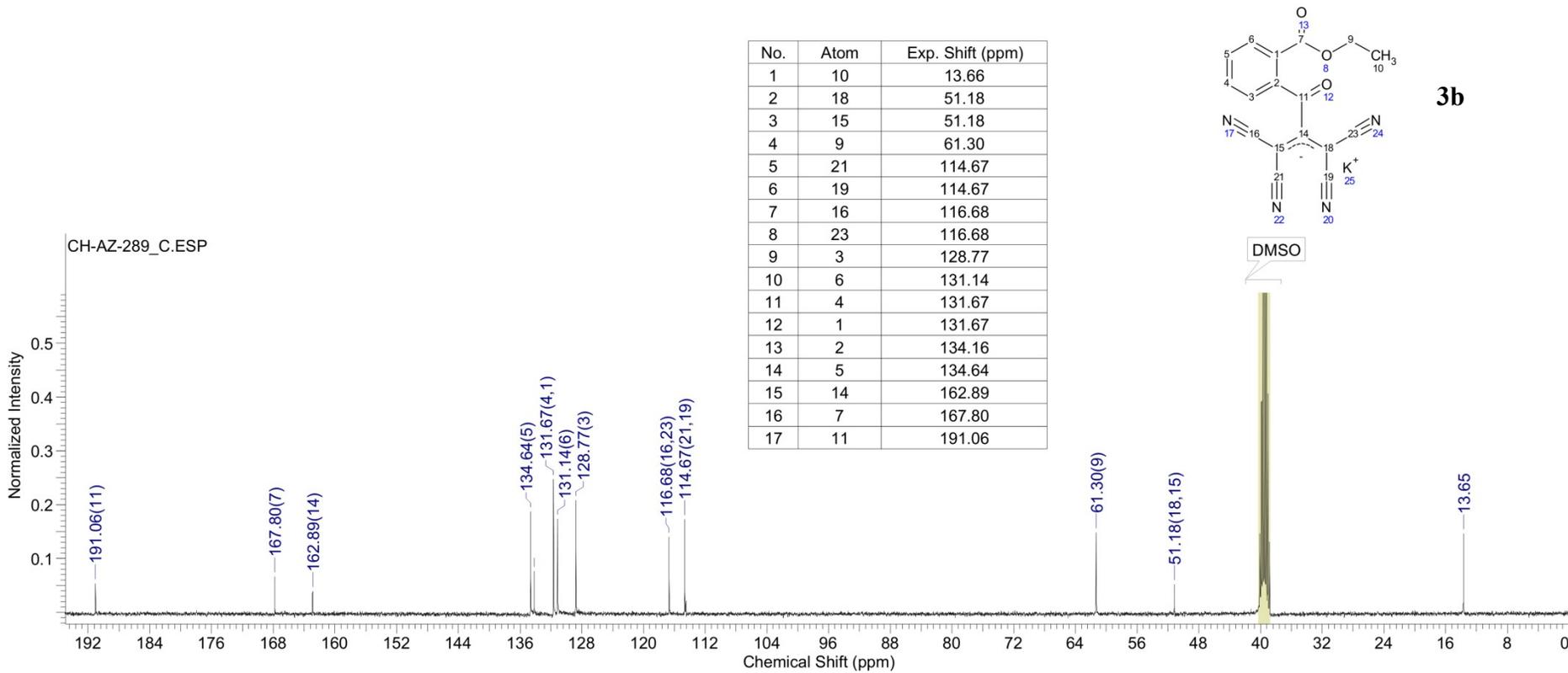


3b



Formula $C_{17}H_9KN_4O_3$ FW 356.3778 (317.2800+39.0978)

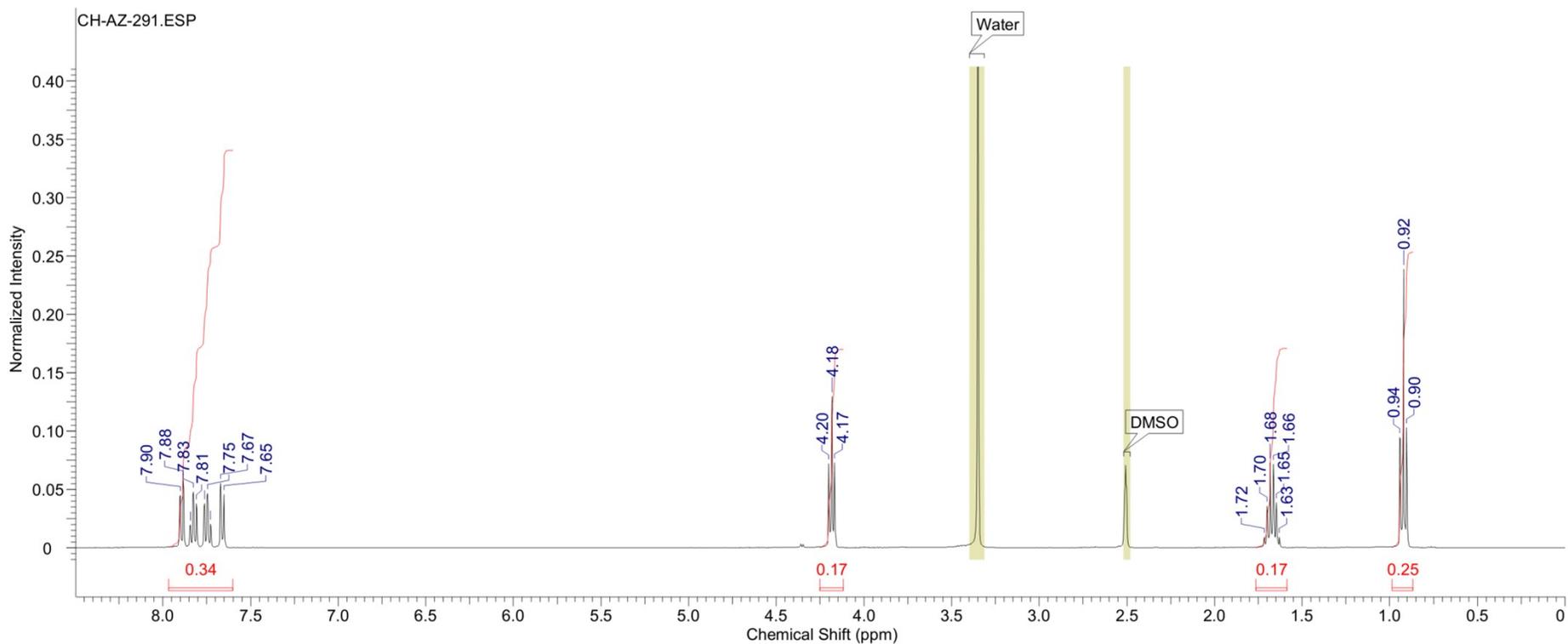
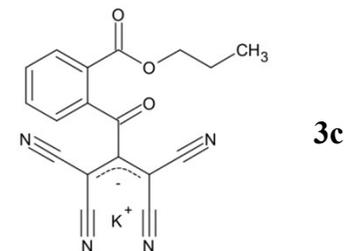
Acquisition Time (sec)	0.6816	Date	19 Sep 2015 19:23:28	Date Stamp	19 Sep 2015 19:23:28
Number of Transients	3000	Origin	spect	Frequency (MHz)	100.62
Pulse Sequence	zgpg	Receiver Gain	189.90	Nucleus	^{13}C
Spectrum Type	STANDARD	Sweep Width (Hz)	24036.99	Owner	nmr
		Original Points Count	16384	Solvent	DMSO-d6
		SW(cyclical) (Hz)	24038.46	Points Count	16384
		Temperature (degree C)	20.365	Spectrum Offset (Hz)	11521.3105



Potassium 1,1,3,3-tetracyano-2-(2'-(propoxycarbonyl)benzoyl)prop-2-en-1-ide (3c)

Formula C ₁₈ H ₁₁ KN ₄ O ₃	FW 370.4043 (331.3066+39.0978)
---	---------------------------------------

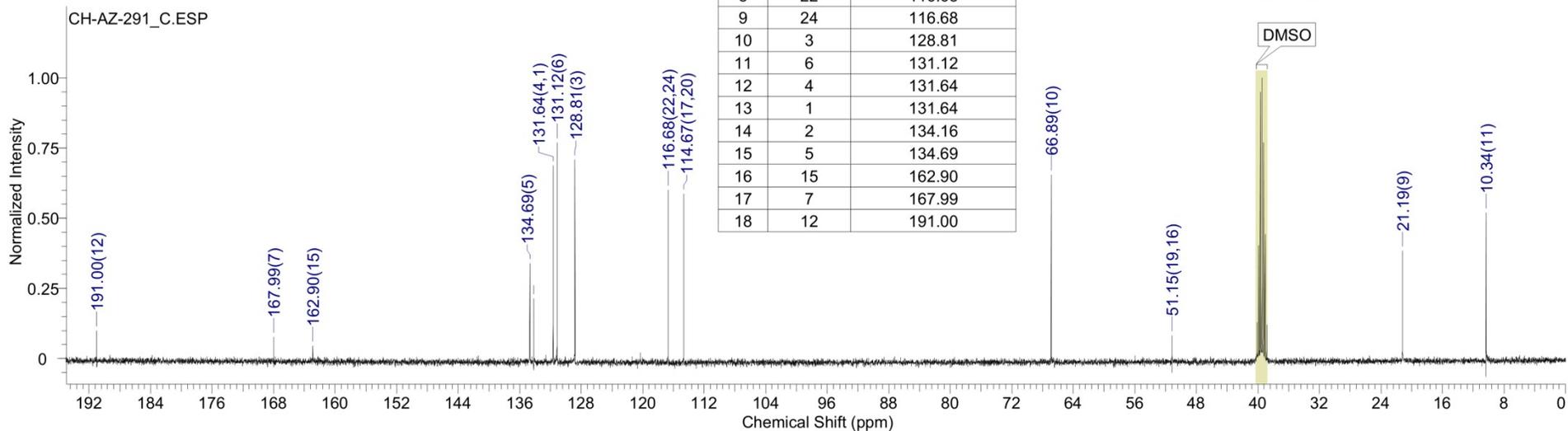
Acquisition Time (sec) 0.9961	Date 03 Oct 2015 17:30:24	Date Stamp 03 Oct 2015 17:30:24
Nucleus 1H	Number of Transients 9	Origin spect
Owner nmr	Points Count 8192	Pulse Sequence zg
SW(cyclical) (Hz) 8223.68	Solvent DMSO-d6	Spectrum Offset (Hz) 3201.3608
Sweep Width (Hz) 8222.68	Temperature (degree C) 24.760	Spectrum Type STANDARD



Formula $C_{18}H_{11}KN_4O_3$ FW 370.4043 (331.3066+39.0978)

Acquisition Time (sec)	0.6816	Date	06 Oct 2015 03:00:00	Date Stamp	06 Oct 2015 03:00:00
Nucleus	^{13}C	Number of Transients	1200	Origin	spect
Owner	nmr	Points Count	16384	Pulse Sequence	zgpg
SW(cyclical) (Hz)	24038.46	Solvent	DMSO-d6	Spectrum Offset (Hz)	11521.3105
Sweep Width (Hz)	24036.99	Temperature (degree C)	21.661	Original Points Count	16384
				Receiver Gain	189.90
				Spectrum Type	STANDARD

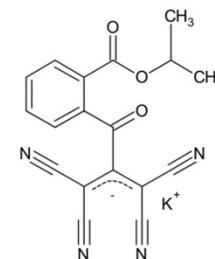
No.	Atom	Exp. Shift (ppm)
1	11	10.34
2	9	21.19
3	19	51.15
4	16	51.15
5	10	66.89
6	17	114.67
7	20	114.67
8	22	116.68
9	24	116.68
10	3	128.81
11	6	131.12
12	4	131.64
13	1	131.64
14	2	134.16
15	5	134.69
16	15	162.90
17	7	167.99
18	12	191.00



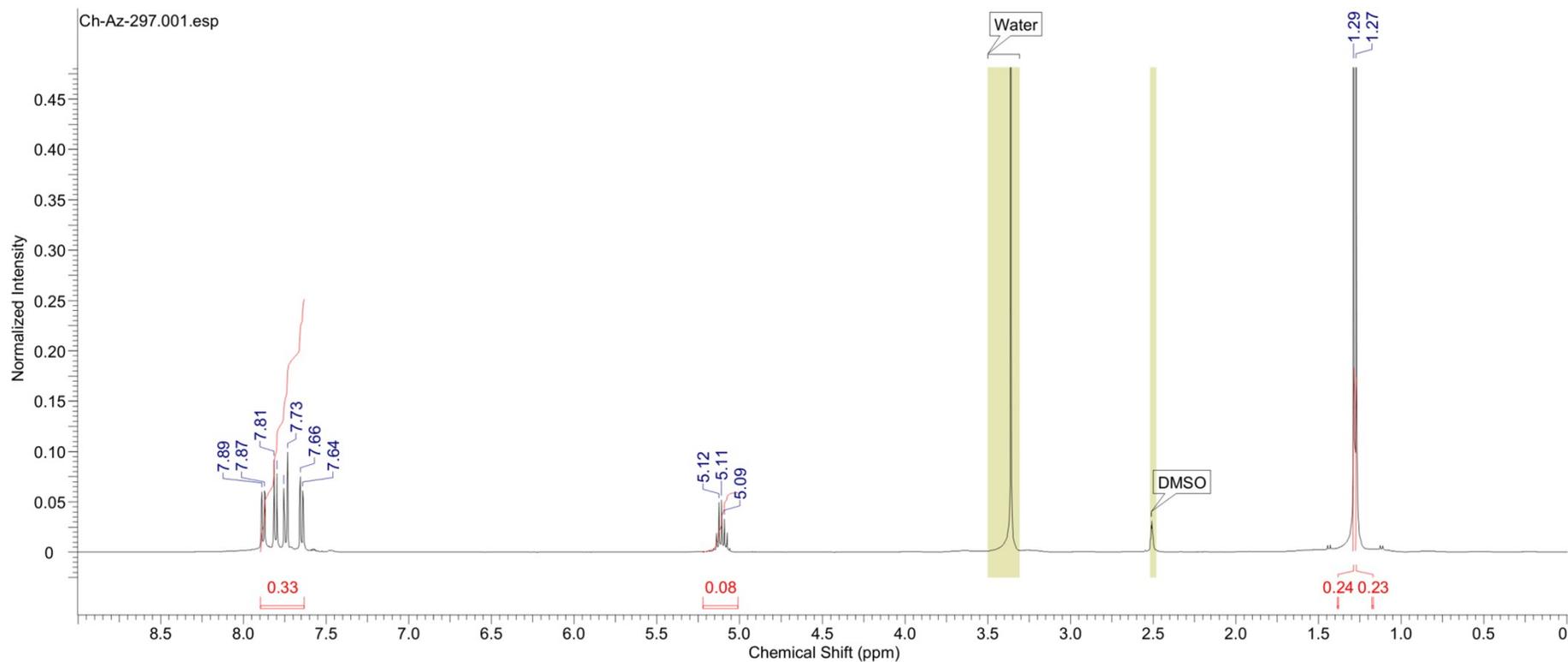
Potassium 1,1,3,3-tetracyano-2-(2'-(isopropoxycarbonyl)benzoyl)prop-2-en-1-ide (3d) and rubidium 1,1,3,3-tetracyano-2-(2'-(isopropoxycarbonyl)benzoyl)prop-2-en-1-ide (3d_Rb) ¹H NMR and ¹³C NMR data for 3d_Rb are completely analogously for 3d.

Formula	C ₁₈ H ₁₁ KN O ₃	FW	370.4043 (331.3066+39.0978)
---------	---	----	-----------------------------

Acquisition Time (sec)	0.9961	Date	27 Sep 2015 05:48:32	Date Stamp	27 Sep 2015 05:48:32
Number of Transients	32	Origin	spect	Original Points Count	8192
Pulse Sequence	zg	Receiver Gain	88.88	SW(cyclical) (Hz)	8223.68
Spectrum Type	STANDARD	Sweep Width (Hz)	8222.68	Temperature (degree C)	21.415
				Frequency (MHz)	400.17
				Nucleus	1H
				Owner	nmr
				Points Count	8192
				Solvent	DMSO-d6
				Spectrum Offset (Hz)	3201.3608



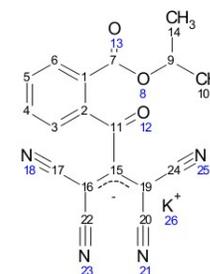
3d



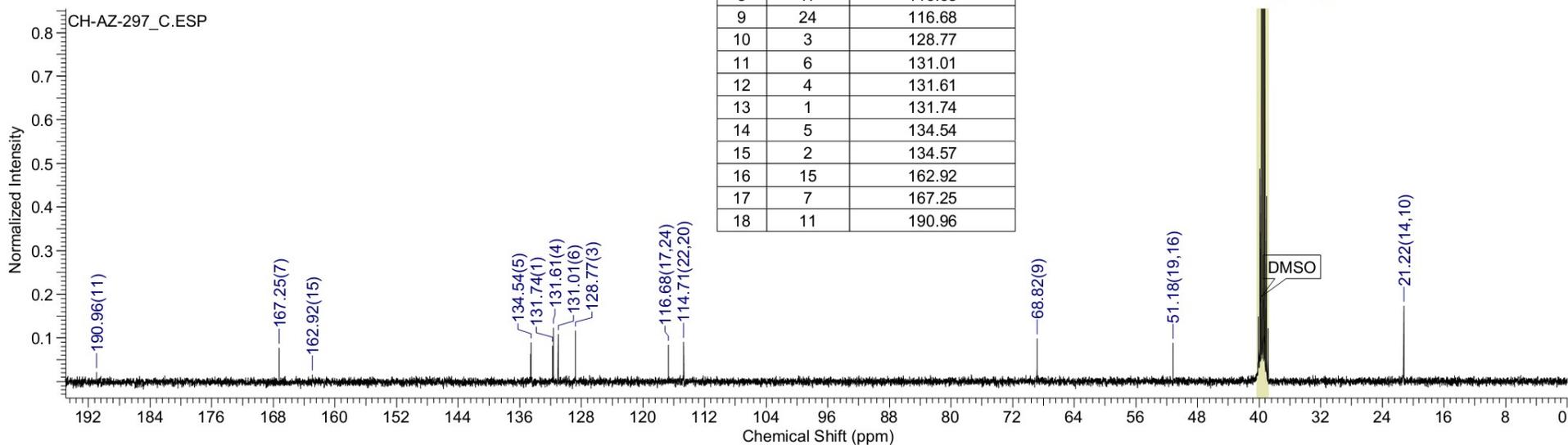
Formula C₁₈H₁₁KN₄O₃ **FW** 370.4043 (331.3066+39.0978)

Acquisition Time (sec) 0.6816	Date 27 Sep 2015 05:50:40		Date Stamp 27 Sep 2015 05:50:40
Number of Transients 2000	Origin spect	Original Points Count 16384	Frequency (MHz) 100.62 Nucleus 13C
Pulse Sequence zgpg	Receiver Gain 189.90	SW(cyclical) (Hz) 24038.46	Owner nmr Points Count 16384
Spectrum Type STANDARD	Sweep Width (Hz) 24036.99	Temperature (degree C) 21.507	Solvent DMSO-d6 Spectrum Offset (Hz) 11521.3105

No.	Atom	Exp. Shift (ppm)
1	14	21.22
2	10	21.22
3	19	51.18
4	16	51.18
5	9	68.82
6	22	114.71
7	20	114.71
8	17	116.68
9	24	116.68
10	3	128.77
11	6	131.01
12	4	131.61
13	1	131.74
14	5	134.54
15	2	134.57
16	15	162.92
17	7	167.25
18	11	190.96



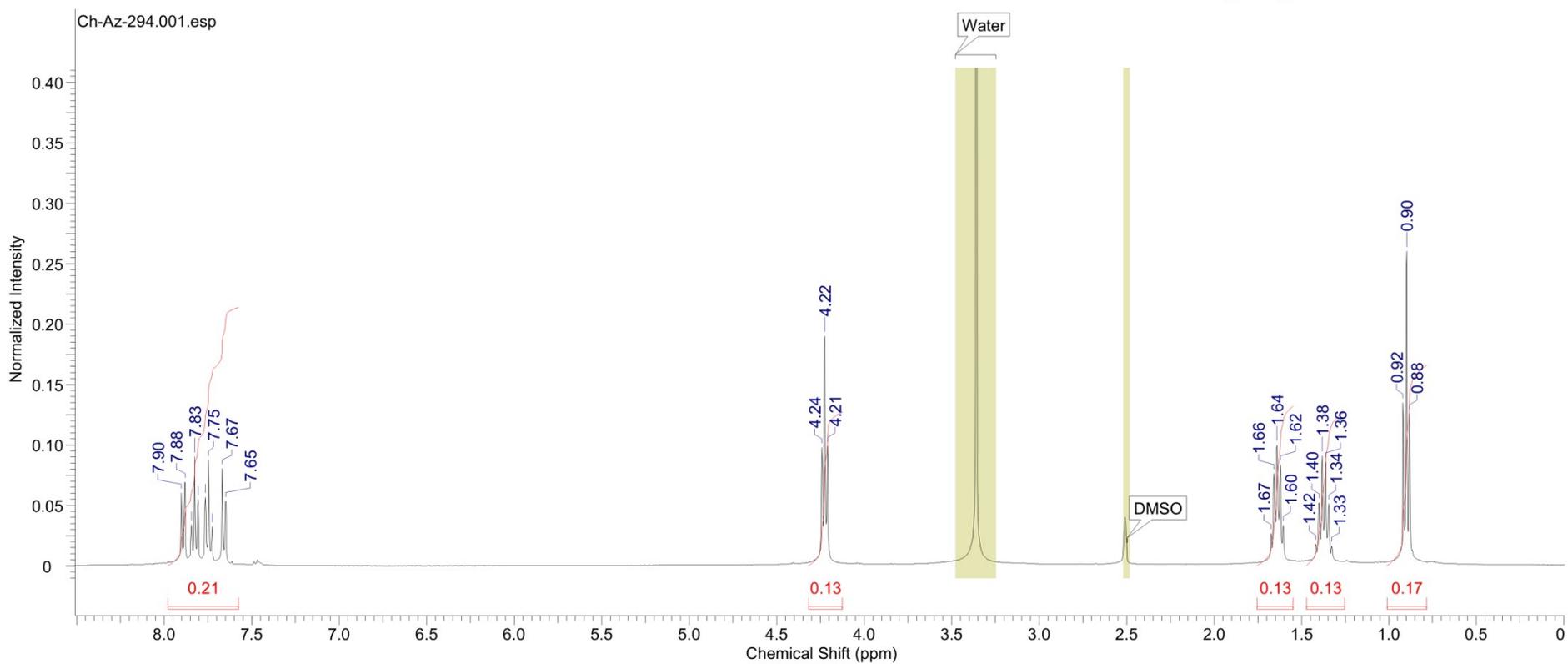
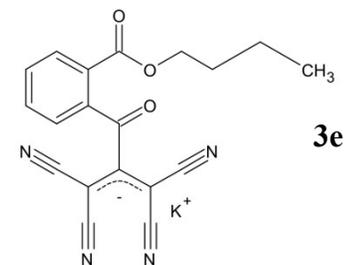
3d



Potassium 1,1,3,3-tetracyano-2-(2'-(butoxycarbonyl)benzoyl)prop-2-en-1-ide (3e)

Formula C ₁₉ H ₁₃ KN ₄ O ₃	FW 384.4309 (345.3332+39.0978)
---	---------------------------------------

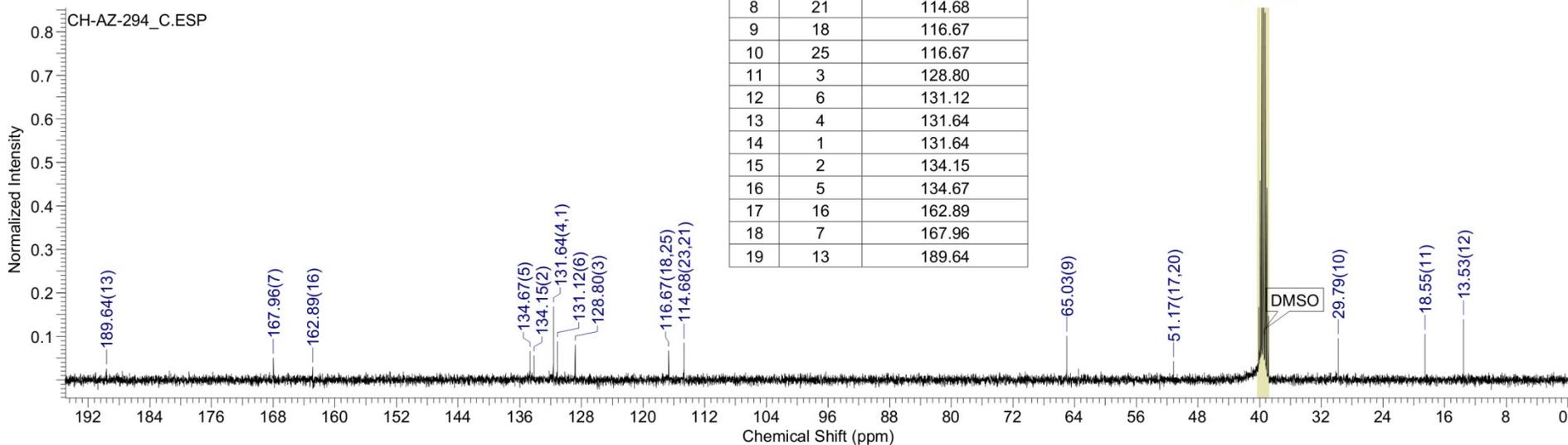
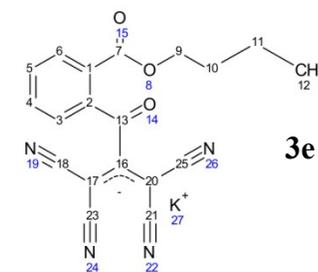
Acquisition Time (sec) 0.9961	Date 27 Sep 2015 03:17:04	Date Stamp 27 Sep 2015 03:17:04
Number of Transients 32	Origin spect	Original Points Count 8192
Pulse Sequence zg	Receiver Gain 106.79	SW(cyclical) (Hz) 8223.68
Spectrum Type STANDARD	Sweep Width (Hz) 8222.68	Temperature (degree C) 22.227
	Frequency (MHz) 400.17	Nucleus 1H
	Owner nmr	Points Count 8192
	Solvent DMSO-d6	Spectrum Offset (Hz) 3201.3608



Formula C₁₉H₁₃KN₄O₃ FW 384.4309 (345.3332+39.0978)

Acquisition Time (sec)	0.6816	Date	27 Sep 2015 03:21:20	Date Stamp	27 Sep 2015 03:21:20
Number of Transients	2000	Origin	spect	Original Points Count	16384
Pulse Sequence	zgpgg	Receiver Gain	189.90	SW(cyclical) (Hz)	24038.46
Spectrum Type	STANDARD	Sweep Width (Hz)	24036.99	Temperature (degree C)	22.342
				Frequency (MHz)	100.62
				Nucleus	13C
				Owner	nmr
				Points Count	16384
				Solvent	DMSO-d6
				Spectrum Offset (Hz)	11521.3105

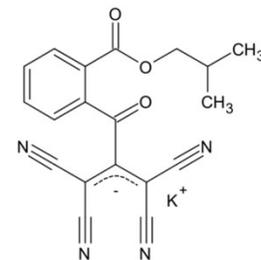
No.	Atom	Exp. Shift (ppm)
1	12	13.53
2	11	18.55
3	10	29.79
4	17	51.17
5	20	51.17
6	9	65.03
7	23	114.68
8	21	114.68
9	18	116.67
10	25	116.67
11	3	128.80
12	6	131.12
13	4	131.64
14	1	131.64
15	2	134.15
16	5	134.67
17	16	162.89
18	7	167.96
19	13	189.64



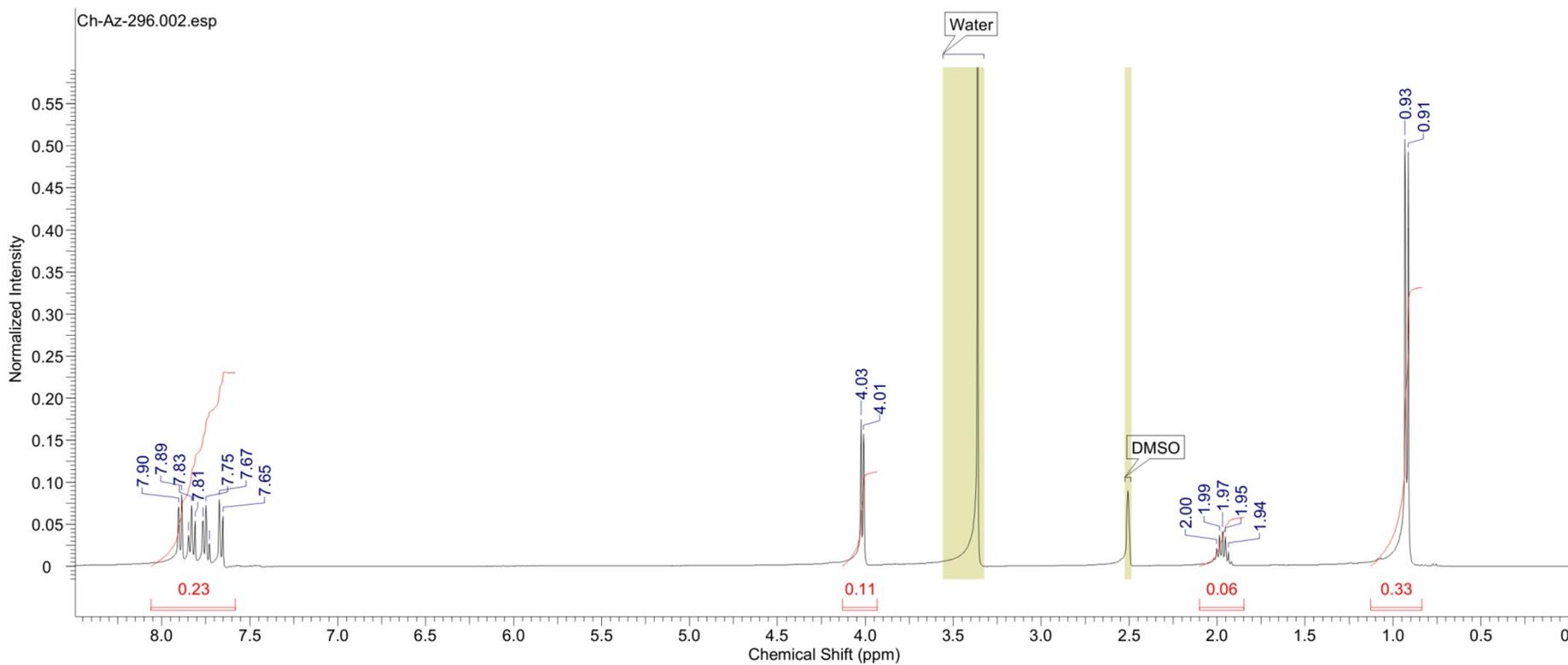
Potassium 1,1,3,3-tetracyano-2-(2'-(isobutoxycarbonyl)benzoyl)prop-2-en-1-ide (3f)

Formula C₁₉H₁₃KN₄O₃ FW 384.4309 (345.3332+39.0978)

Acquisition Time (sec)	0.9961	Date	26 Sep 2015 14:05:36	Date Stamp	26 Sep 2015 14:05:36
Number of Transients	16	Origin	spect	Frequency (MHz)	400.17
Pulse Sequence	zg	Receiver Gain	74.28	SW(cyclical) (Hz)	8223.68
Spectrum Type	STANDARD	Sweep Width (Hz)	8222.68	Temperature (degree C)	20.961
				Nucleus	1H
				Owner	nmr
				Points Count	8192
				Solvent	DMSO-d6
				Spectrum Offset (Hz)	3201.3608



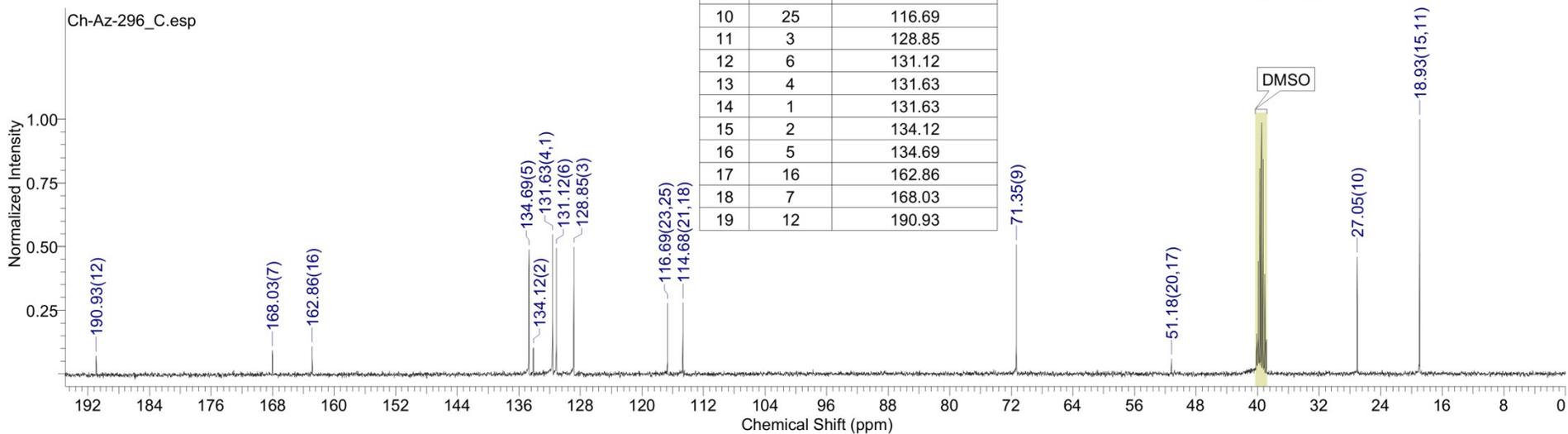
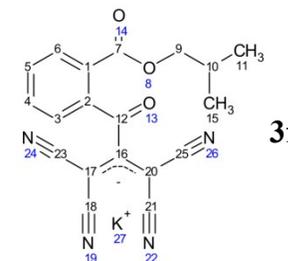
3f



Formula C₁₉H₁₃KN₄O₃ **FW** 384.4309 (345.3332+39.0978)

Acquisition Time (sec) 0.6816	Date 29 Sep 2015 20:44:32	Date Stamp 29 Sep 2015 20:44:32
Nucleus 13C	Number of Transients 1200	Origin spect
Owner nmr	Points Count 16384	Pulse Sequence zgpg
SW(cyclical) (Hz) 24038.46	Solvent DMSO-d6	Spectrum Offset (Hz) 11521.3105
Sweep Width (Hz) 24036.99	Temperature (degree C) 23.970	Spectrum Type STANDARD

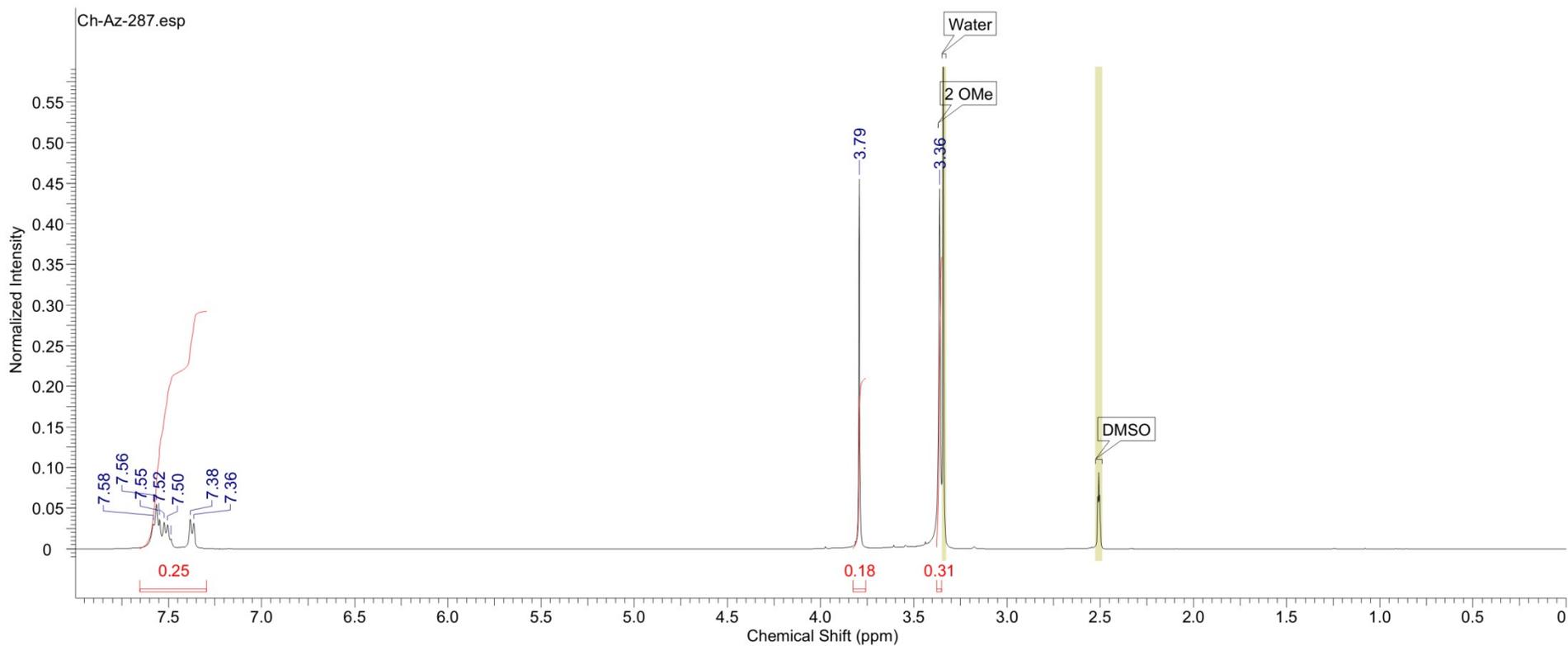
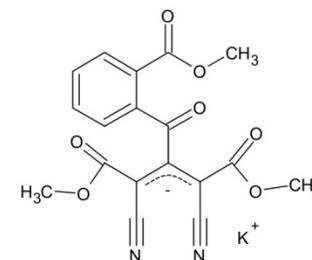
No.	Atom	Exp. Shift (ppm)
1	15	18.93
2	11	18.93
3	10	27.05
4	20	51.18
5	17	51.18
6	9	71.35
7	21	114.68
8	18	114.68
9	23	116.69
10	25	116.69
11	3	128.85
12	6	131.12
13	4	131.63
14	1	131.63
15	2	134.12
16	5	134.69
17	16	162.86
18	7	168.03
19	12	190.93



Potassium (Z)-2,4-dicyano-1,5-dimethoxy-3-(2'-(methoxycarbonyl)benzoyl)-1,5-dioxpent-3-en-2-ide (4a)

Formula $C_{18}H_{13}KN_2O_7$ FW 408.4044 (369.3067+39.0978)

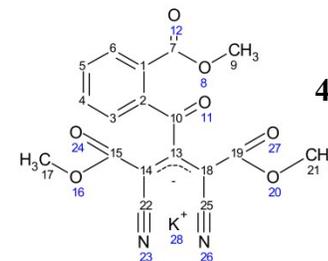
Acquisition Time (sec)	0.9961	Date	03 Oct 2015 17:19:44	Date Stamp	03 Oct 2015 17:19:44
Nucleus	1H	Number of Transients	9	Origin	spect
Owner	nmr	Points Count	8192	Pulse Sequence	zg
SW(cyclical) (Hz)	8223.68	Solvent	DMSO-d6	Spectrum Offset (Hz)	3201.3608
Sweep Width (Hz)	8222.68	Temperature (degree C)	25.774	Receiver Gain	106.79
				Spectrum Type	STANDARD



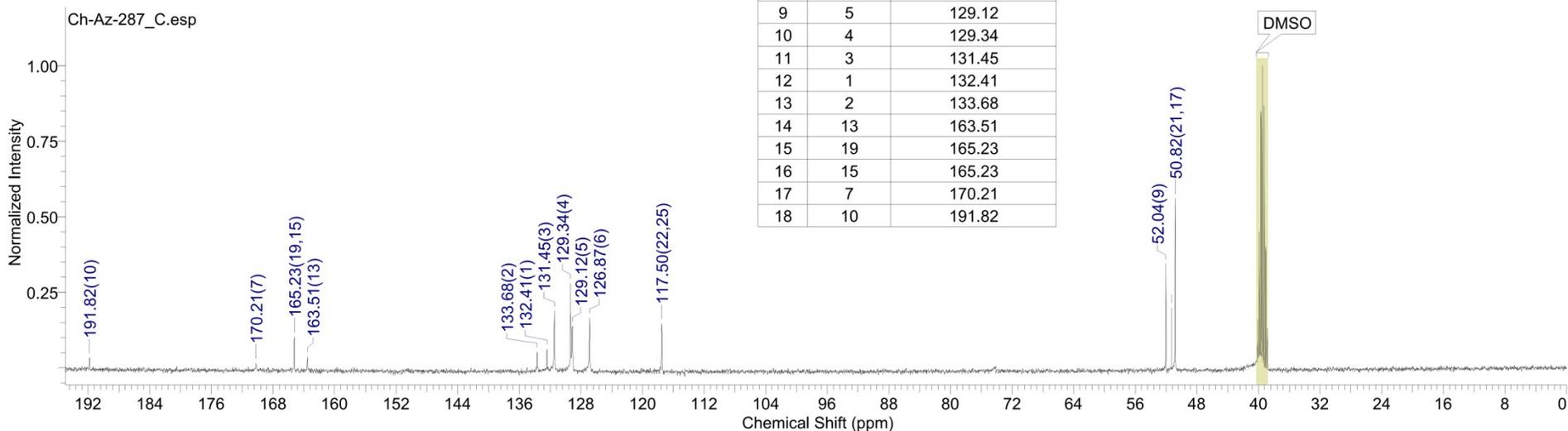
Formula C₁₈H₁₃KN₂O₇ FW 408.4044 (369.3067+39.0978)

Acquisition Time (sec)	0.6816	Date	06 Oct 2015 00:54:08	Date Stamp	06 Oct 2015 00:54:08
Nucleus	13C	Number of Transients	2000	Origin	spect
Owner	nmr	Points Count	16384	Pulse Sequence	zgpgg
SW(cyclical) (Hz)	24038.46	Solvent	DMSO-d6	Spectrum Offset (Hz)	11521.3105
Sweep Width (Hz)	24036.99	Temperature (degree C)	21.609	Spectrum Type	STANDARD

No.	Atom	Exp. Shift (ppm)
1	21	50.82
2	17	50.82
3	18	51.30
4	14	51.30
5	9	52.04
6	22	117.50
7	25	117.50
8	6	126.87
9	5	129.12
10	4	129.34
11	3	131.45
12	1	132.41
13	2	133.68
14	13	163.51
15	19	165.23
16	15	165.23
17	7	170.21
18	10	191.82



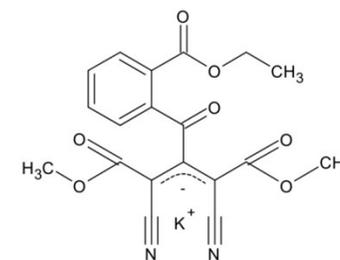
4a



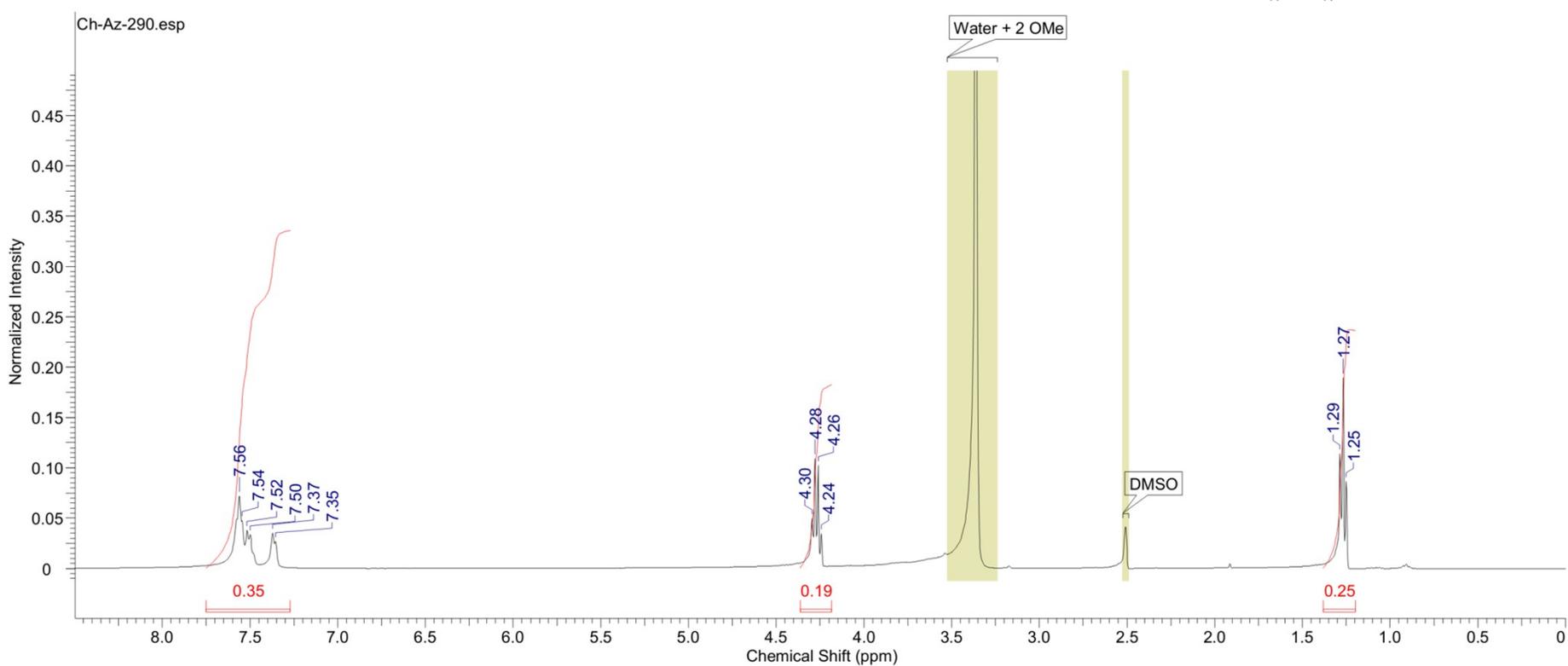
Potassium (Z)-2,4-dicyano-1,5-dimethoxy-3-(2'-(ethoxycarbonyl)benzoyl)-1,5-dioxopent-3-en-2-ide (4b)

Formula	C ₁₉ H ₁₅ KN ₂ O ₇	FW	422.4310 (383.3332+39.0978)
---------	--	----	-----------------------------

Acquisition Time (sec)	0.9961	Date	27 Sep 2015 07:28:48	Date Stamp	27 Sep 2015 07:28:48
Number of Transients	32	Origin	spect	Original Points Count	8192
Pulse Sequence	zg	Receiver Gain	106.79	SW(cyclical) (Hz)	8223.68
Spectrum Type	STANDARD	Sweep Width (Hz)	8222.68	Temperature (degree C)	21.127
				Frequency (MHz)	400.17
				Owner	nmr
				Nucleus	¹ H
				Points Count	8192
				Solvent	DMSO-d6
				Spectrum Offset (Hz)	3201.3608



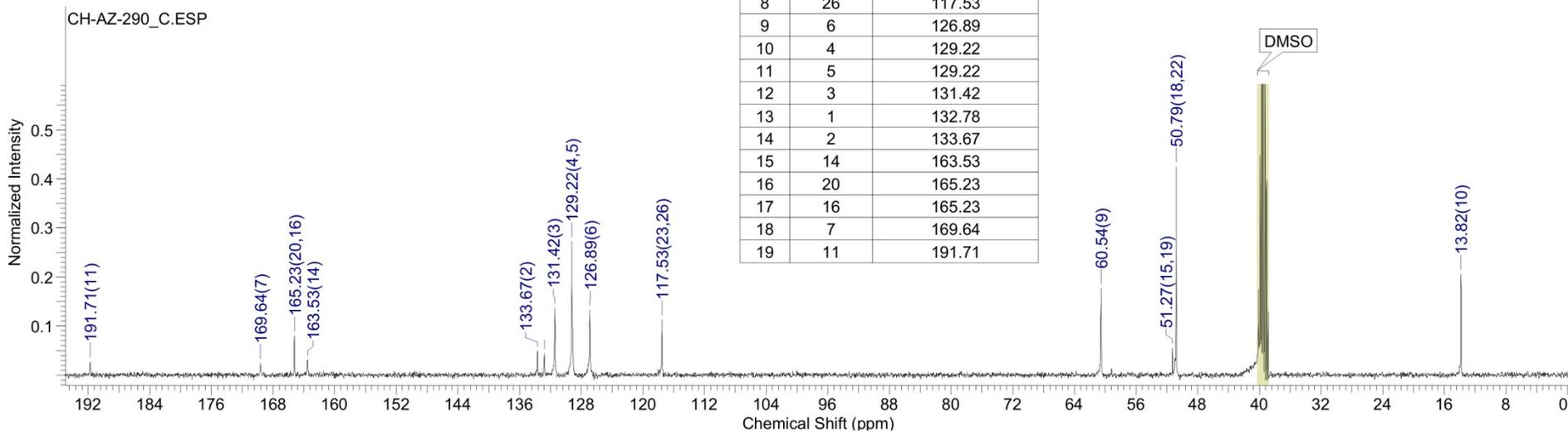
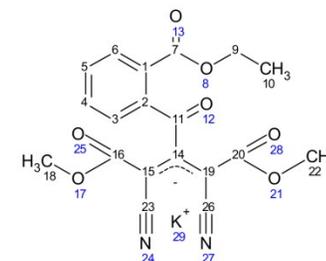
4b



Formula C₁₉H₁₅KN₂O₇ **FW** 422.4310 (383.3332+39.0978)

Acquisition Time (sec) 0.6816	Date 10 Oct 2015 00:45:36	Date Stamp 10 Oct 2015 00:45:36
Nucleus 13C	Number of Transients 4000	Origin spect
Owner nmr	Points Count 16384	Pulse Sequence zgpg
SW(cyclical) (Hz) 24038.46	Solvent DMSO-d6	Spectrum Offset (Hz) 11521.3105
Sweep Width (Hz) 24036.99	Temperature (degree C) 22.745	Frequency (MHz) 100.62
		Original Points Count 16384
		Receiver Gain 189.90
		Spectrum Type STANDARD

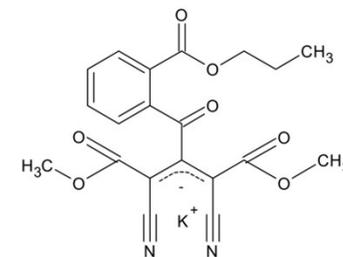
No.	Atom	Exp. Shift (ppm)
1	10	13.82
2	18	50.79
3	22	50.79
4	15	51.27
5	19	51.27
6	9	60.54
7	23	117.53
8	26	117.53
9	6	126.89
10	4	129.22
11	5	129.22
12	3	131.42
13	1	132.78
14	2	133.67
15	14	163.53
16	20	165.23
17	16	165.23
18	7	169.64
19	11	191.71



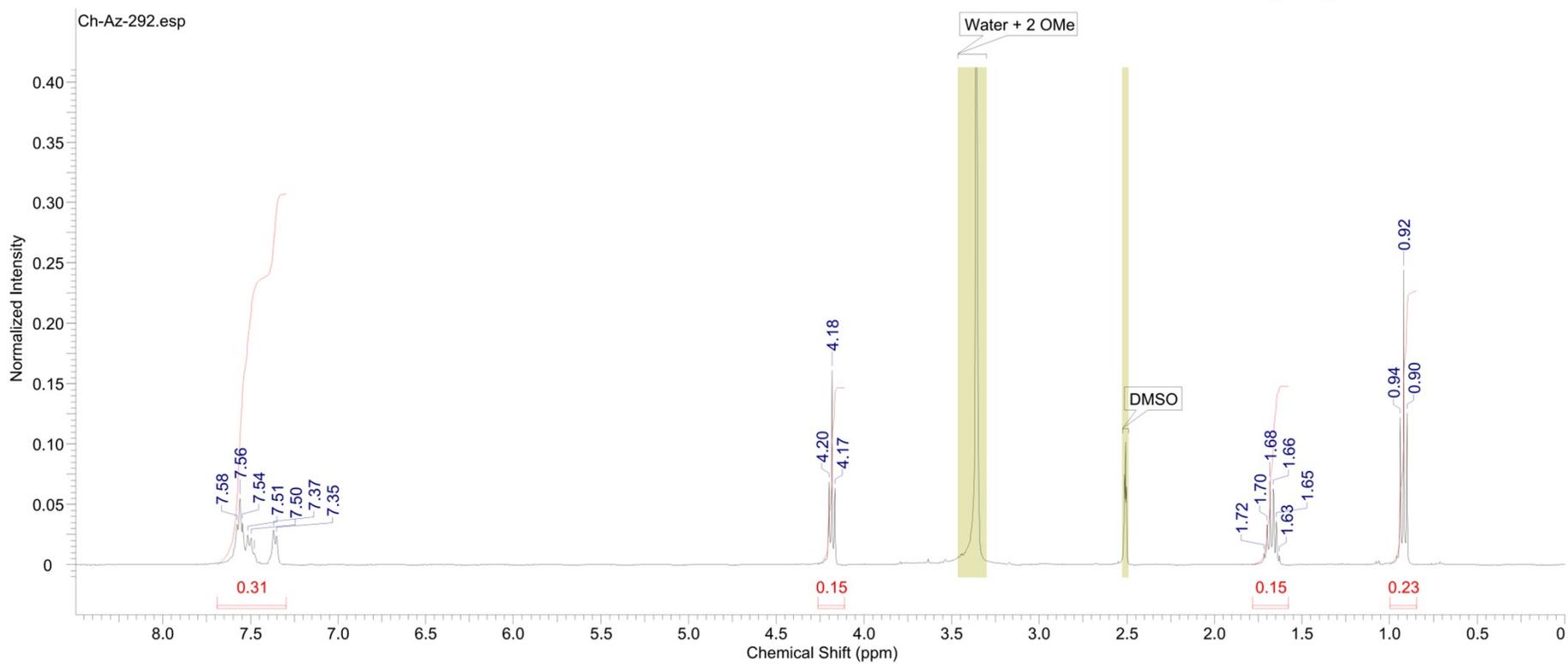
Potassium (Z)-2,4-dicyano-1,5-dimethoxy-3-(2'-(propoxycarbonyl)benzoyl)-1,5-dioxopent-3-en-2-ide (4c)

Formula $C_{20}H_{17}KN_2O_7$ FW 436.4576 (397.3598+39.0978)

Acquisition Time (sec)	0.9961	Date	03 Oct 2015 15:01:04	Date Stamp	03 Oct 2015 15:01:04
Number of Transients	9	Origin	spect	Frequency (MHz)	400.17
Pulse Sequence	zg	Receiver Gain	74.28	SW(cyclical) (Hz)	8223.68
Spectrum Type	STANDARD	Sweep Width (Hz)	8222.68	Temperature (degree C)	20.632
				Nucleus	1H
				Owner	nmr
				Solvent	DMSO-d6
				Points Count	8192
				Spectrum Offset (Hz)	3201.3608



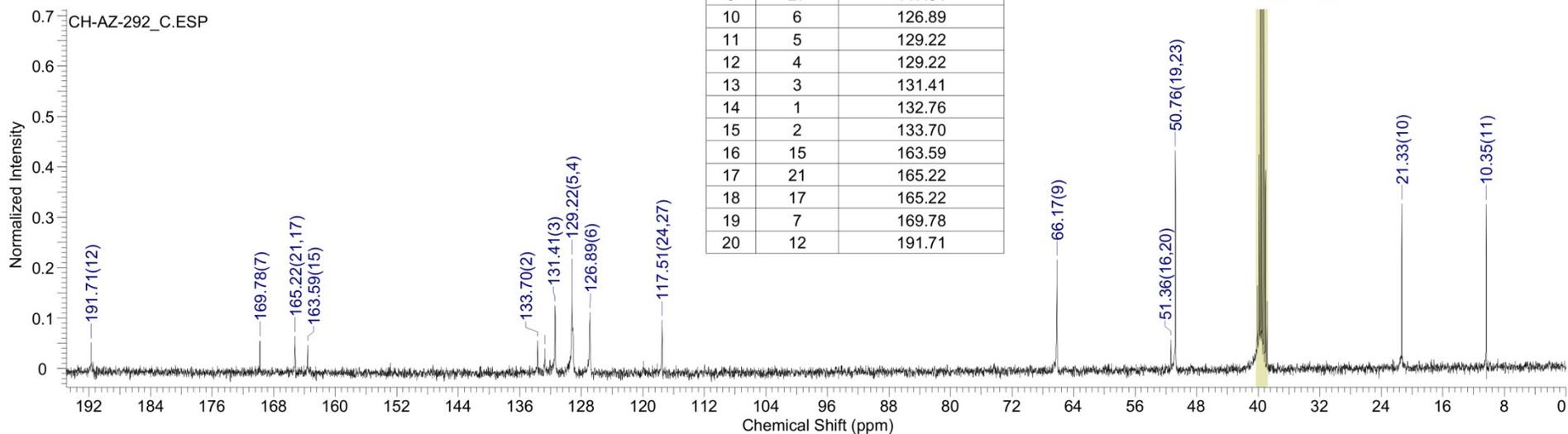
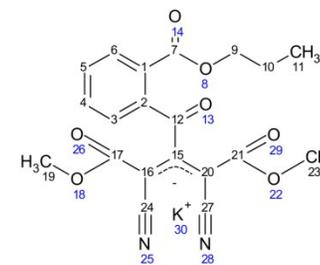
4c



Formula C₂₀H₁₇KN₂O₇ **FW** 436.4576 (397.3598+39.0978)

Acquisition Time (sec) 0.6816	Date 06 Oct 2015 00:05:04	Date Stamp 06 Oct 2015 00:05:04
Nucleus 13C	Number of Transients 2000	Origin spect
Owner nmr	Points Count 16384	Pulse Sequence zgpgg
SW(cyclical) (Hz) 24038.46	Solvent DMSO-d6	Spectrum Offset (Hz) 11521.3105
Sweep Width (Hz) 24036.99	Temperature (degree C) 21.396	Spectrum Type STANDARD

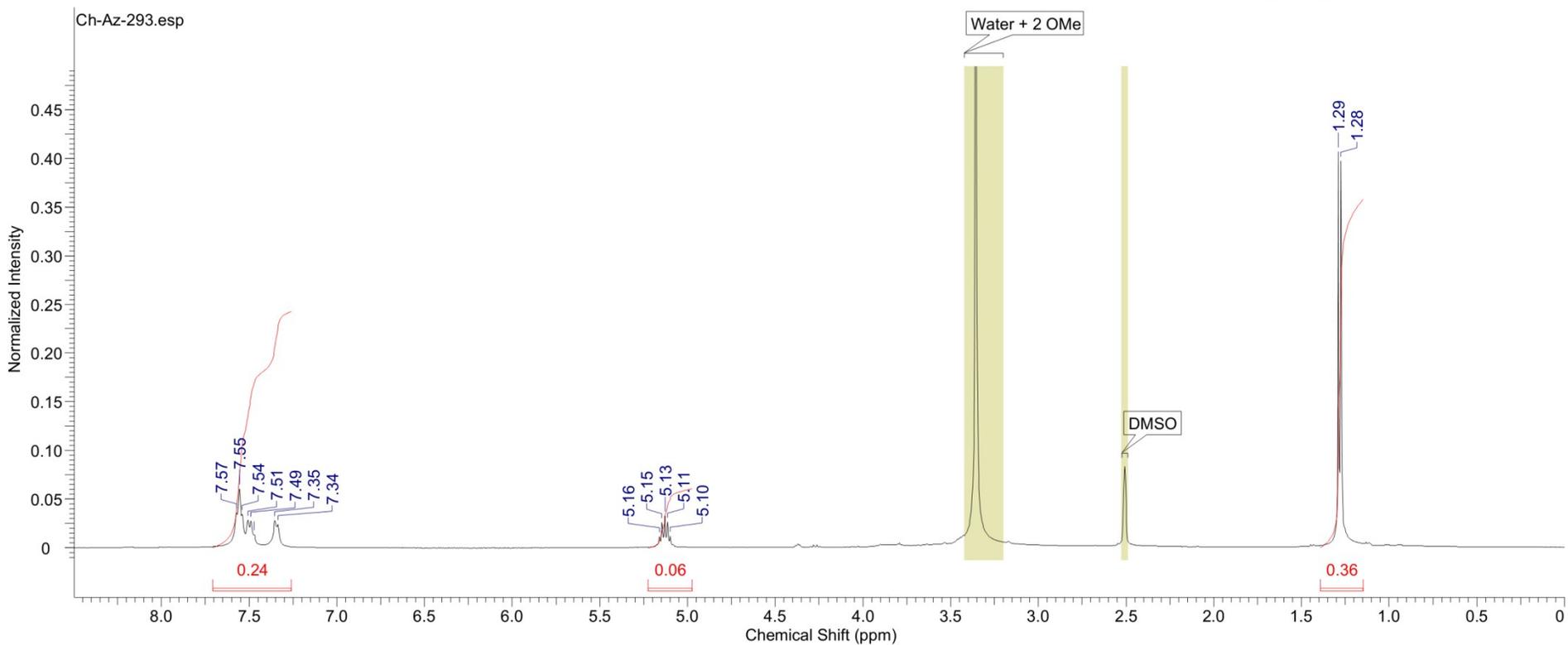
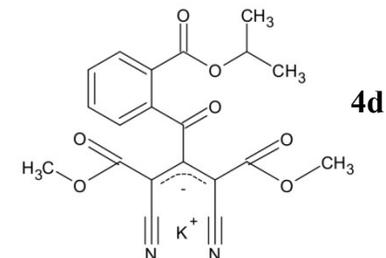
No.	Atom	Exp. Shift (ppm)
1	11	10.35
2	10	21.33
3	19	50.76
4	23	50.76
5	16	51.36
6	20	51.36
7	9	66.17
8	24	117.51
9	27	117.51
10	6	126.89
11	5	129.22
12	4	129.22
13	3	131.41
14	1	132.76
15	2	133.70
16	15	163.59
17	21	165.22
18	17	165.22
19	7	169.78
20	12	191.71



Potassium (Z)-2,4-dicyano-1,5-dimethoxy-3-(2'-(isopropoxycarbonyl)benzoyl)-1,5-dioxopent-3-en-2-ide (4d)

Formula C ₂₀ H ₁₇ KN ₂ O ₇	FW 436.4576 (397.3598+39.0978)
---	---------------------------------------

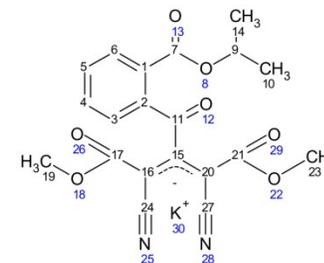
Acquisition Time (sec) 0.9961	Date 27 Sep 2015 02:28:00	Date Stamp 27 Sep 2015 02:28:00
Nucleus 1H	Number of Transients 16	Origin spect
Owner nmr	Points Count 8192	Pulse Sequence zg
SW(cyclical) (Hz) 8223.68	Solvent DMSO-d6	Spectrum Offset (Hz) 3201.3608
Sweep Width (Hz) 8222.68	Temperature (degree C) 21.288	Spectrum Type STANDARD



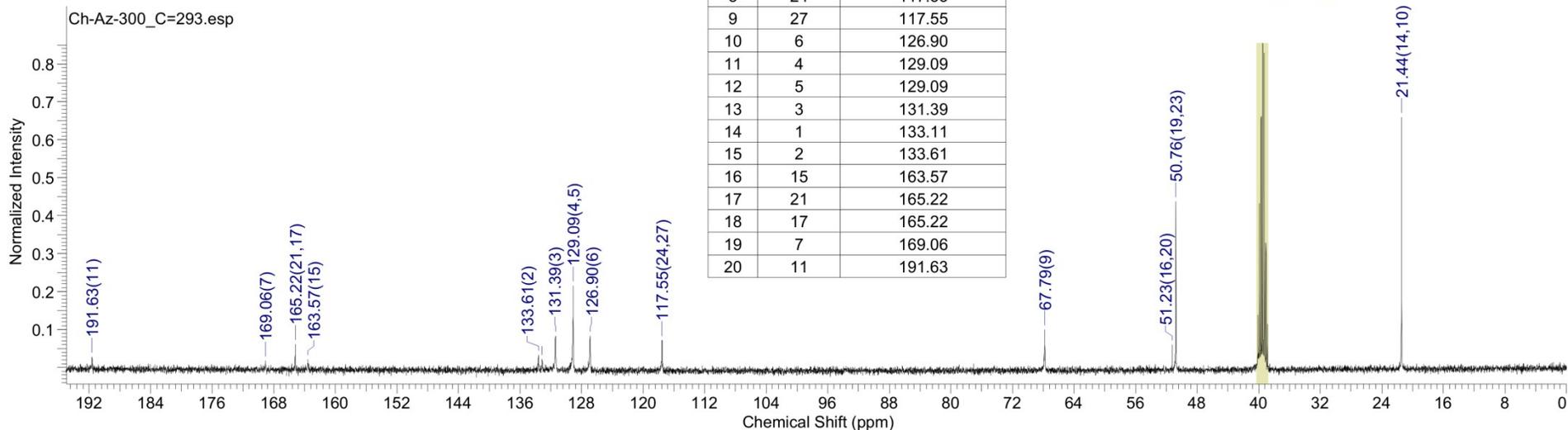
Formula C₂₀H₁₇KN₂O₇ **FW** 436.4576 (397.3598+39.0978)

Acquisition Time (sec) 0.6816	Date 06 Oct 2015 01:41:04	Date Stamp 06 Oct 2015 01:41:04
Nucleus 13C	Number of Transients 2000	Origin spect
Owner nmr	Points Count 16384	Frequency (MHz) 100.62
SW(cyclical) (Hz) 24038.46	Solvent DMSO-d6	Original Points Count 16384
Sweep Width (Hz) 24036.99	Temperature (degree C) 21.803	Receiver Gain 189.90
		Spectrum Offset (Hz) 11521.3105
		Spectrum Type STANDARD

No.	Atom	Exp. Shift (ppm)
1	14	21.44
2	10	21.44
3	19	50.76
4	23	50.76
5	16	51.23
6	20	51.23
7	9	67.79
8	24	117.55
9	27	117.55
10	6	126.90
11	4	129.09
12	5	129.09
13	3	131.39
14	1	133.11
15	2	133.61
16	15	163.57
17	21	165.22
18	17	165.22
19	7	169.06
20	11	191.63



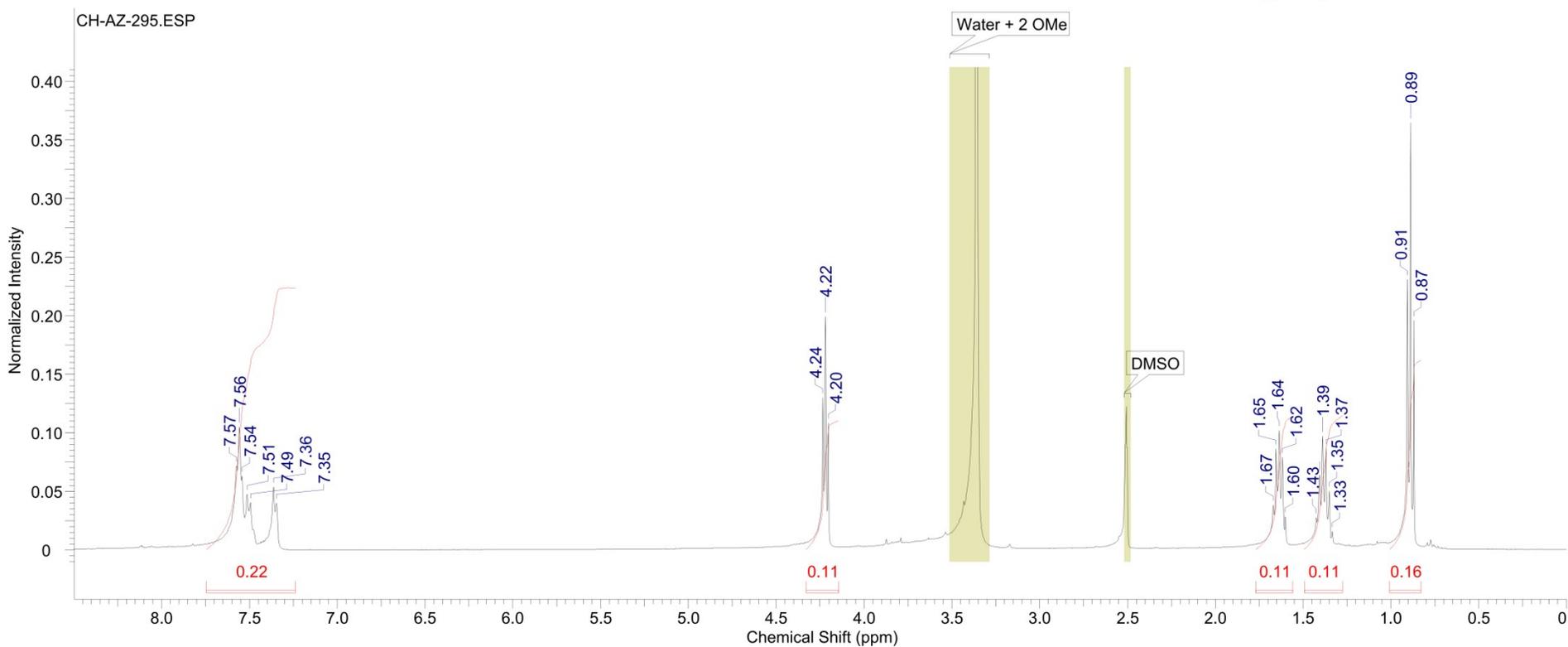
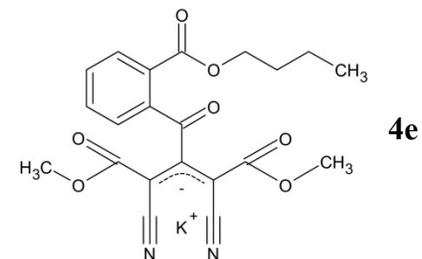
4d



Potassium (Z)-2,4-dicyano-1,5-dimethoxy-3-(2'-(butoxycarbonyl)benzoyl)-1,5-dioxopent-3-en-2-ide (4e)

Formula $C_{21}H_{19}KN_2O_7$ FW 450.4842 (411.3864+39.0978)

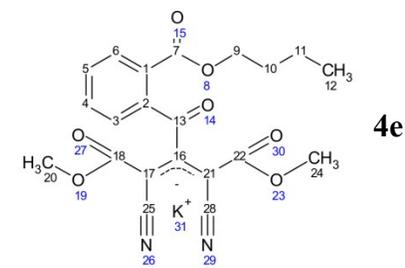
Acquisition Time (sec)	0.9961	Date	18 Sep 2015 09:24:00	Date Stamp	18 Sep 2015 09:24:00
Nucleus	1H	Number of Transients	16	Origin	spect
Owner	nmr	Points Count	8192	Pulse Sequence	zg
SW(cyclical) (Hz)	8223.68	Solvent	DMSO-d6	Spectrum Offset (Hz)	3201.3608
Sweep Width (Hz)	8222.68	Temperature (degree C)	19.747	Spectrum Type	STANDARD
				Frequency (MHz)	400.17
				Original Points Count	8192
				Receiver Gain	60.56



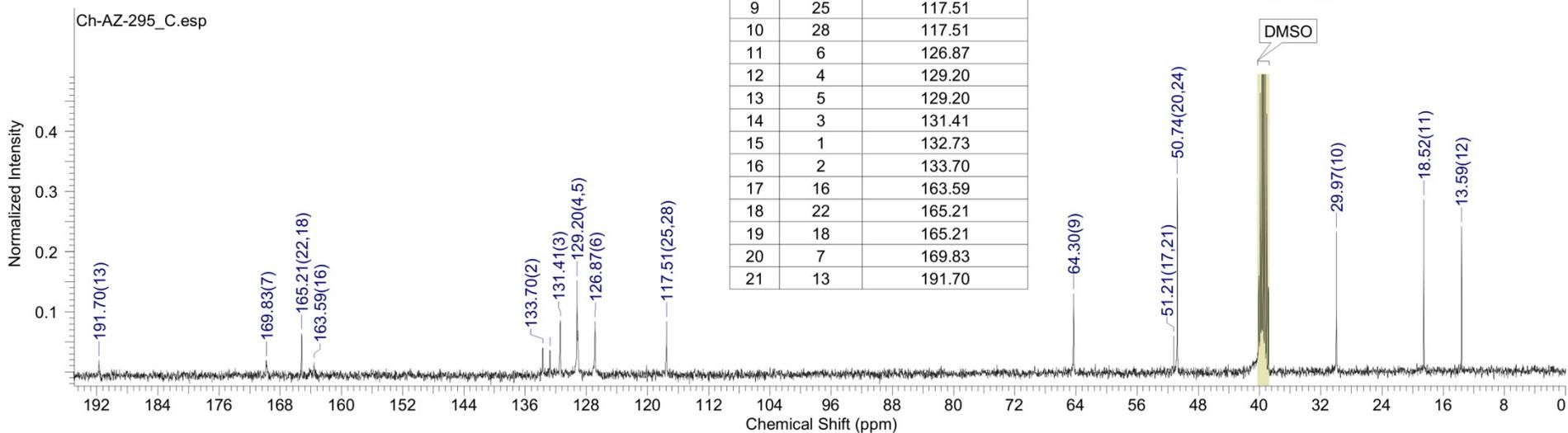
Formula C ₂₁ H ₁₉ KN ₂ O ₇	FW 450.4842 (411.3864+39.0978)
---	---------------------------------------

Acquisition Time (sec) 0.6816	Date 18 Sep 2015 16:13:36	Date Stamp 18 Sep 2015 16:13:36
Nucleus 13C	Number of Transients 1200	Origin spect
Owner nmr	Points Count 16384	Pulse Sequence zgpgg
SW(cyclical) (Hz) 24038.46	Solvent DMSO-d6	Spectrum Offset (Hz) 11521.3105
Sweep Width (Hz) 24036.99	Temperature (degree C) 20.942	Spectrum Type STANDARD

No.	Atom	Exp. Shift (ppm)
1	12	13.59
2	11	18.52
3	10	29.97
4	20	50.74
5	24	50.74
6	17	51.21
7	21	51.21
8	9	64.30
9	25	117.51
10	28	117.51
11	6	126.87
12	4	129.20
13	5	129.20
14	3	131.41
15	1	132.73
16	2	133.70
17	16	163.59
18	22	165.21
19	18	165.21
20	7	169.83
21	13	191.70



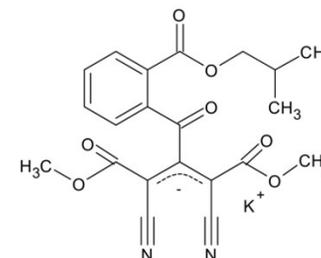
4e



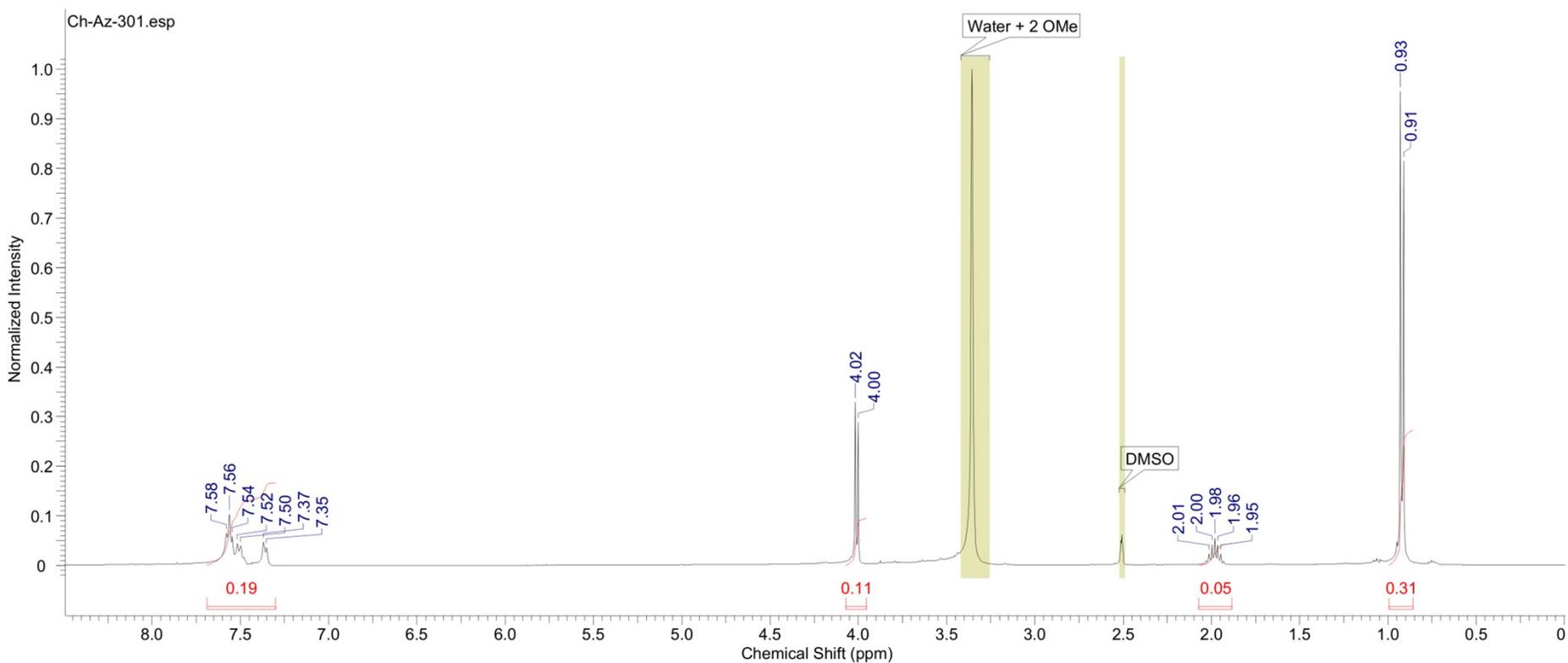
Potassium (Z)-2,4-dicyano-1,5-dimethoxy-3-(2'-(isobutoxycarbonyl)benzoyl)-1,5-dioxopent-3-en-2-ide (4f)

Formula C ₂₁ H ₁₉ KN ₂ O ₇	FW 450.4842 (411.3864+39.0978)
---	---------------------------------------

Acquisition Time (sec) 0.9961	Date 27 Sep 2015 04:57:20	Date Stamp 27 Sep 2015 04:57:20
Number of Transients 32	Origin spect	Original Points Count 8192
Pulse Sequence zg	Receiver Gain 74.28	SW(cyclical) (Hz) 8223.68
Spectrum Type STANDARD	Sweep Width (Hz) 8222.68	Temperature (degree C) 21.394
	Frequency (MHz) 400.17	Nucleus 1H
	Owner nmr	Points Count 8192
	Solvent DMSO-d6	Spectrum Offset (Hz) 3201.3608



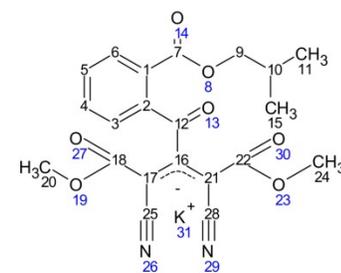
4f



Formula C ₂₁ H ₁₉ KN ₂ O ₇	FW 450.4842 (411.3864+39.0978)
---	---------------------------------------

Acquisition Time (sec) 0.6816	Date 27 Sep 2015 05:01:36	Date Stamp 27 Sep 2015 05:01:36
Number of Transients 2000	Origin spect	Original Points Count 16384
Pulse Sequence zgpg	Receiver Gain 189.90	SW(cyclical) (Hz) 24038.46
Spectrum Type STANDARD	Sweep Width (Hz) 24036.99	Temperature (degree C) 21.516
	Frequency (MHz) 100.62	Nucleus 13C
	Owner nmr	Points Count 16384
	Solvent DMSO-d6	Spectrum Offset (Hz) 11521.3105

No.	Atom	Exp. Shift (ppm)
1	15	18.99
2	11	18.99
3	10	27.11
4	20	50.74
5	24	50.74
6	21	51.34
7	17	51.34
8	9	70.27
9	25	117.60
10	28	117.60
11	6	126.92
12	5	129.22
13	4	129.22
14	3	131.39
15	1	132.73
16	2	133.73
17	16	163.62
18	22	165.21
19	18	165.21
20	7	168.88
21	12	191.67



4f

