Electronic Supplementary Information (ESI)

Covalently immobilized tris(triazolyl)methanol-Cu(I) complexes: Highly active and recyclable catalysts for CuAAC reactions.

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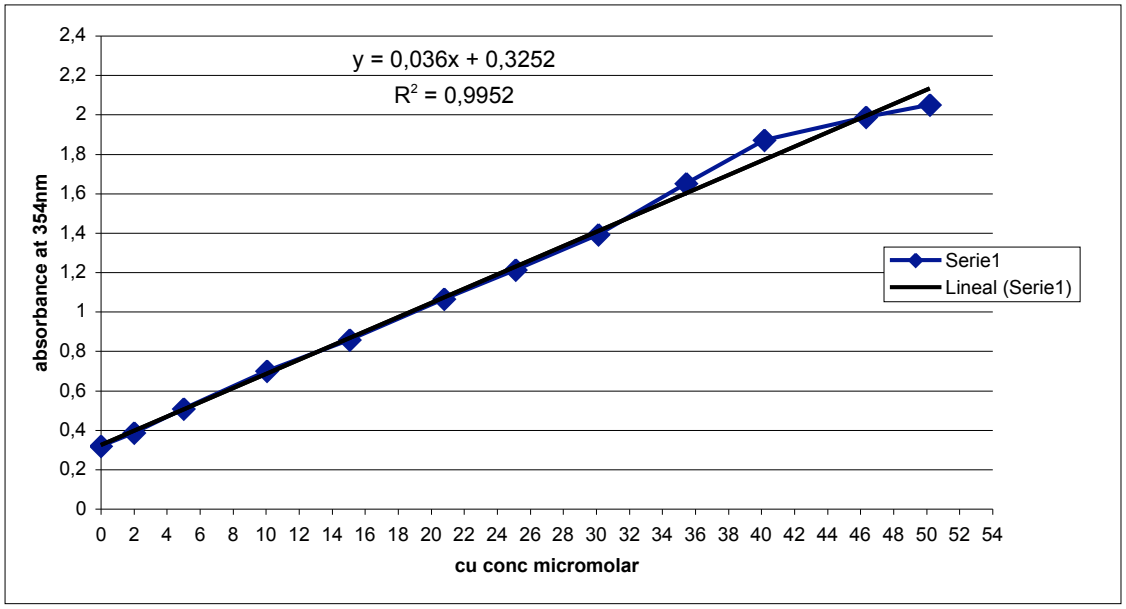
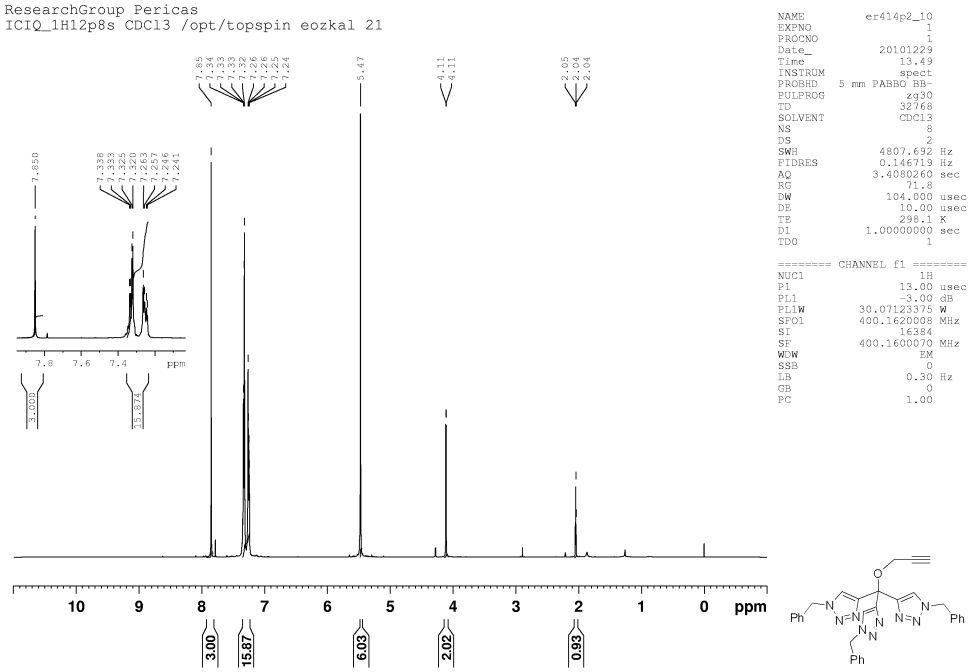
I. General Methods.

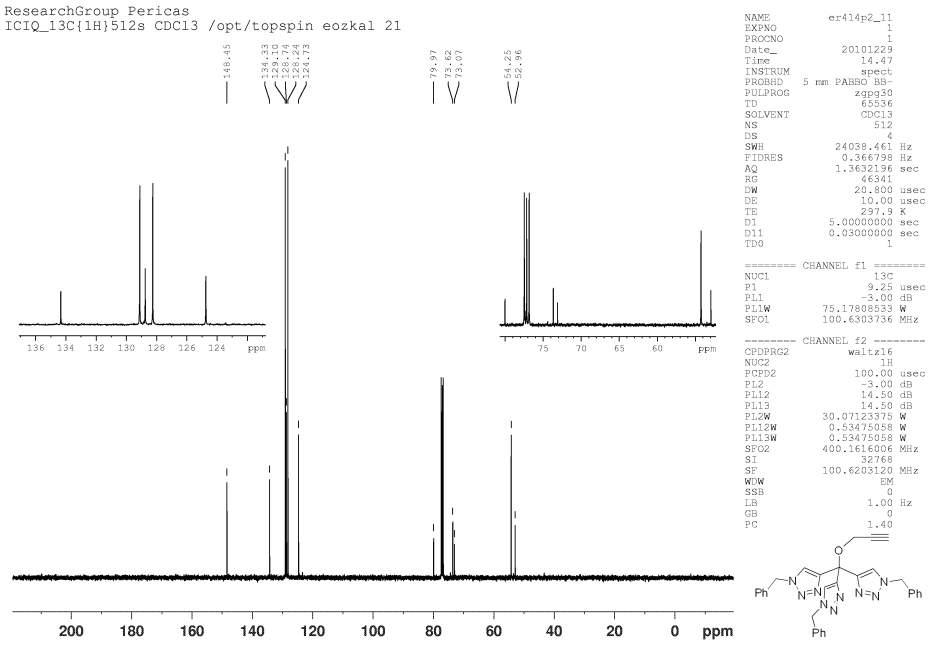
All reagents were used as purchased. All reported yields are isolated yields. CuAAC reactions were performed on vials at open air. Merrifield resin (1% DVB, f = 1.1 mmol Cl g^{-1} resin) was obtained from Novabiochem. All flash chromatography was carried out using 60 mesh silica gel and dry-packed columns. ^{1}H (400.13 MHz) and ^{13}C (100.63 MHz) Nuclear Magnetic Resonance (NMR) spectra were recorded on a Bruker Advance 400 Ultrashield spectrometer in $CDCl_3$ at room temperature (unless otherwise stated). Chemical shifts (δ) are reported with respect to tetramethylsilane as internal standard, or to the corresponding solvent residual peak, in ppm. IR spectra were recorded on a Bruker Tensor 27 FT-IR spectrometer. Elemental analyses made in C.A.I. Microanálisis Elemental, Universidad Complutense de Madrid (Spain). High Resolution Mass Spectra (HRMS) were performed by the High Resolution Mass Spectromety Service at the Institute of Chemical Research of Catalonia. UV spectra were recorded on a Shimadzu UV-1700 spectrophotometer. The optical rotation was recorded on Jasco P-1030 Polarimeter.

II. Calibration Curve for UV-Vis Analysis of Cu Content in Triazoles 9[1]



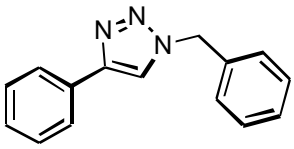
III. NMR spectra of 3-[tris(1-benzyl-1H-1,2,3-triazol-4-yl)methoxy]propyne (4).





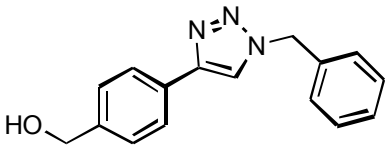
IV. ^{1}H and ^{13}C NMR Data/Spectra of 1,2,3-triazoles.

1-benzyl-4-phenyl-1H-1,2,3-triazole (9a):



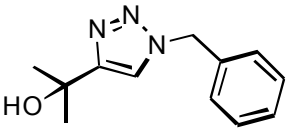
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.81 – 7.77 (m, 2H), 7.66 (s, 1H), 7.41 – 7.29 (m, 8H), 5.57 (s, 2H) ppm.
^{13}C NMR (100 MHz, $(CD_3)_2SO$): δ 147.2, 136.5, 131.2, 129.3, 129.2, 128.6, 128.3, 125.7, 122.0, 53.5 ppm.
Spectroscopic data matched the literature[2].

(4-(1-benzyl-1H-1,2,3-triazol-4-yl)phenyl)methanol (9b):



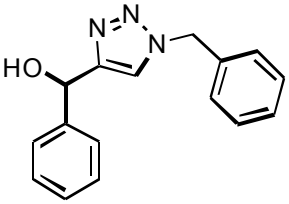
^{1}H NMR (400 MHz, $(CD_3)_2SO$): δ 8.61 (s, 1H), 7.82 (s, 2H), 7.40 – 7.37 (m, 7H), 5.64 (s, 2H), 5.24 – 5.22 (m, 1H), 4.53 (s, 2H) ppm.
^{13}C NMR (100 MHz, $(CD_3)_2SO$): δ 147.2, 142.8, 136.5, 129.6, 129.3, 128.6, 128.4, 127.4, 125.4, 121.8, 63.1, 53.5 ppm.
Spectroscopic data matched the literature[3].

2-(1-benzyl-1H-1,2,3-triazol-4-yl)propan-2-ol (9c):



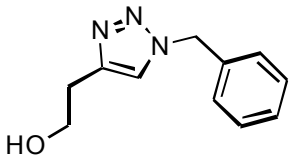
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.43 (s, 1H), 7.25 – 7.15 (m, 5H), 5.36 (s, 2H), 4.09 (br, 1H), 1.51 (s, 6H) ppm.
^{13}C NMR (100 MHz, $CDCl_3$): δ 156.3, 133.8, 129.0, 128.5, 128.0, 119.7, 69.4, 53.9, 30.5 ppm.
Spectroscopic data matched the literature[4].

(1-benzyl-1H-1,2,3-triazol-4-yl)(phenyl)methanol (9d):



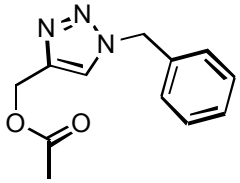
^{1}H NMR (400 MHz, $(CD_3)_2SO$): δ 7.95 (s, 1H), 7.43 – 7.25 (m, 10H), 5.99 (d, J = 4.6 Hz, 1H), 5.84 (d, J = 4.6 Hz, 1H), 5.56 (s, 2H) ppm.
^{13}C NMR (100 MHz, $(CD_3)_2SO$): δ 152.3, 144.5, 136.6, 129.2, 128.6, 128.5, 128.5, 127.5, 126.8, 122.6, 68.5, 53.2 ppm.
Spectroscopic data matched the literature[5].

2-(1-benzyl-1H-1,2,3-triazol-4-yl)ethanol (9e):



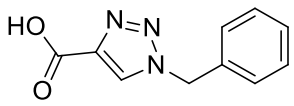
^{1}H NMR (400 MHz, $(CD_3)_2SO$): δ 7.89 (s, 1H), 7.38 – 7.29 (m, 5H), 5.54 (s, 2H), 4.67 (t, J = 5.3 Hz, 1H), 3.62 (q, J = 6.3 Hz, 2H), 2.76 (t, J = 4.6 Hz, 2H) ppm.
^{13}C NMR (100 MHz, $(CD_3)_2SO$): δ 145.2, 136.7, 129.2, 128.5, 128.4, 123.0, 60.8, 53.1, 29.6 ppm.
Spectroscopic data matched the literature[4].

(1-benzyl-1H-1,2,3-triazol-4-yl)methyl acetate (9f):



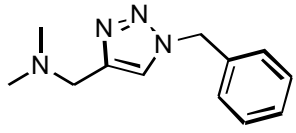
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.54 (s, 1H), 7.33 – 7.22 (m, 5H), 5.47 (s, 2H), 5.13 (s, 2H), 1.99 (s, 3H) ppm.
^{13}C NMR (100 MHz, $CDCl_3$): δ 170.7, 143.1, 134.5, 129.1, 128.8, 128.1, 123.7, 57.6, 54.1, 20.8 ppm.
Spectroscopic data matched the literature[6].

1-benzyl-1H-1,2,3-triazole-4-carboxylic acid (9g):



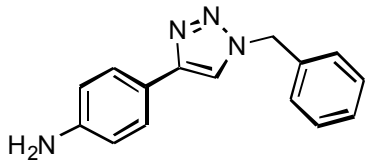
^{1}H NMR (400 MHz, $(CD_3)_2SO$): δ 8.77 (s, 1H), 7.38 – 7.34 (m, 5H), 5.64 (s, 2H) ppm.
Spectroscopic data matched the literature[7].

1-(1-benzyl-1H-1,2,3-triazol-4-yl)-N,N-dimethylmethanamine (9h):



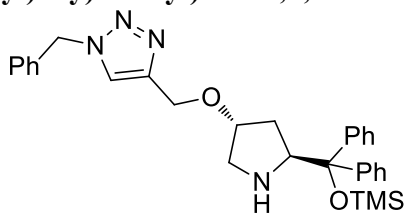
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.37 (s, 1H), 7.25-7.11 (m, 5H) 5.39 (s, 2H), 3.46 (s, 2H), 2.13 (s, 6H) ppm.
^{13}C NMR (100 MHz, $CDCl_3$): δ 145.4, 134.8, 128.9, 128.5, 127.9, 122.5, 54.2, 53.9, 45.0 ppm.
Spectroscopic data matched the literature[8].

4-(1-benzyl-1H-1,2,3-triazol-4-yl)aniline (9i):



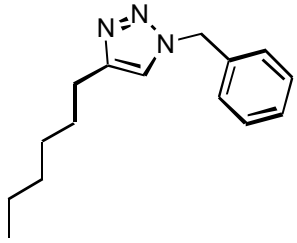
^{1}H NMR (400 MHz, $(CD_3)_2SO$): δ 8.31 (s, 1H), 7.50 – 7.48 (d, J = 8.5 Hz, 2H), 7.37 – 7.33 (m, 5H), 6.61 – 6.58 (d, J = 8.6 Hz, 2H), 5.58 (s, 2H), 5.22 (br, 2H) ppm.
^{13}C NMR (100 MHz, $(CD_3)_2SO$): δ 149.1, 136.7, 129.2, 128.5, 128.3, 126.6, 119.7, 118.8, 114.3, 53.3 ppm.
Spectroscopic data matched the literature[9].

1-benzyl-4-((((3R,5S)-5-(diphenyl((trimethylsilyl)oxy)methyl)pyrrolidin-3-yl)oxy)methyl)-1H-1,2,3-triazole (9j):



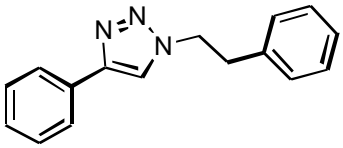
Physical form: thick orange oil.
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.57 (s, 1H), 7.48-7.46 (d, J = 7.15 Hz, 2H), 7.38-7.23 (m, 13H), 5.51-5.50 (d, J = 2.20 Hz, 2H), 4.53 (d, J = 2.15 Hz, 2H), 4.44 (t, J = 8.03 Hz, 1H), 3.90 (brs, 1H), 3.08-3.06 (d, J = 11.60 Hz, 1H), 2.85-2.82 (dd, J = 11.90, 4.70 Hz, 1H), 1.82-1.81 (dd, J = 12.15 Hz, 2H), -0.078 (s, 9H) ppm.
^{13}C NMR (100 MHz, $CDCl_3$): δ 145.6, 145.5, 134.6, 129.1, 128.7, 128.4, 128.1, 127.9, 127.8, 127.7, 127.3, 127.2, 122.7, 82.6, 78.8, 64.0, 62.5, 54.2, 52.2, 34.3, 2.16 ppm.
IR (ATR): ν = 3306.02, 3054.07, 2953.28, 1592.51, 1404.37, 1343.21, 1131.95, 917.53.
HRMS calculated for $C_{30}H_{36}N_4O_2SiNa$: 535.2505. Found: 535.2523 ($[M^+Na]^+$).
$[\alpha]_D^{26}$ = -2.65 ± 0.693 (c=0.97 in $CHCl_3$).

1-benzyl-4-hexyl-1H-1,2,3-triazole (9k):



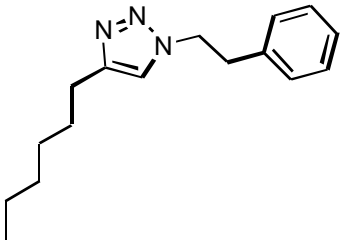
^{1}H NMR (400 MHz, $(CD_3)_2SO$): δ 7.89 (s, 1H), 7.36 – 7.27 (m, 5H), 5.54 (s, 2H), 2.61 – 2.57 (t, J = 7.5 Hz, 2H), 1.59 – 1.55 (t, J = 6.8 Hz, 2H), 1.26 (s, 6H), 0.86 – 0.83 (t, J = 6.3 Hz, 3H) ppm.
^{13}C NMR (100 MHz, $(CD_3)_2SO$): δ 147.8, 136.8, 129.2, 128.5, 128.2, 122.4, 53.1, 31.4, 29.4, 28.7, 25.5, 22.5, 14.3 ppm.
Spectroscopic data matched the literature[10].

1-phenethyl-4-phenyl-1H-1,2,3-triazole (9l):



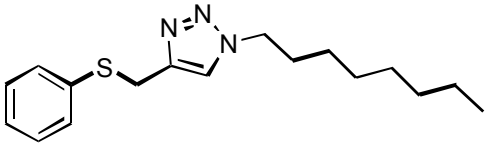
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.80 – 7.79 (d, J = 7.1 Hz, 2H), 7.51 (s, 1H), 7.43 – 7.15 (m, 8H), 4.65 – 4.62 (t, J = 7.3 Hz, 2H), 3.28 – 3.25 (t, J = 7.3 Hz, 2H) ppm.
^{13}C NMR (100 MHz, $CDCl_3$): δ 147.5, 137.1, 130.7, 128.9, 128.8, 128.7, 128.0, 127.1, 125.7, 120.0, 51.7, 36.8 ppm.
Spectroscopic data matched the literature[4].

4-hexyl-1-phenethyl-1H-1,2,3-triazole (9m):



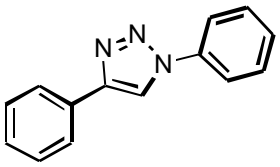
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.27 – 7.07 (m, 5H), 7.00 (s, 1H), 4.54 – 4.50 (t, J = 7.3 Hz, 2H), 3.18 – 3.15 (t, J = 7.3 Hz, 2H), 2.66 – 2.63 (t, J = 7.6 Hz, 2H), 1.63 – 1.56 (m, 2H), 1.31 – 1.26 (m, 6H), 0.89 – 0.85 (m, 3H) ppm.
^{13}C NMR (100 MHz, $CDCl_3$): δ 148.1, 137.3, 128.7, 128.6, 126.9, 120.9, 51.4, 36.8, 31.6, 29.4, 28.8, 25.6, 22.6, 14.1 ppm.
Spectroscopic data matched the literature[11].

1-octyl-4-((phenylthio)methyl)-1H-1,2,3-triazole (9n)[1]:



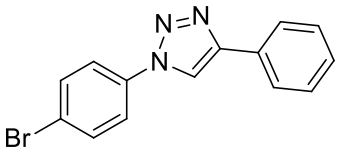
^{1}H NMR (400 MHz, $CDCl_3$): δ 7.31 – 7.14 (m, 6H), 4.24 – 4.19 (m, 4H), 1.80 – 1.77 (m, 2H), 1.22 (s, 10H), 0.87 – 0.84 (t, J = 6.9 Hz, 3H) ppm.
^{13}C NMR (100 MHz, $CDCl_3$): δ 144.7, 135.6, 129.5, 128.9, 126.4, 121.9, 50.3, 31.7, 30.2, 29.0, 28.9, 28.9, 26.4, 22.6, 14.1 ppm.
Spectroscopic data matched the literature[8].

1,4-diphenyl-1H-1,2,3-triazole (9o):



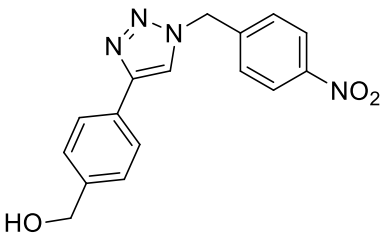
^{1}H NMR (400 MHz, $CDCl_3$): δ 8.19 (s, 1H), 7.92 – 7.26 (m, 10H) ppm.
^{13}C NMR (100 MHz, $CDCl_3$): δ 148.4, 137.1, 130.3, 129.8, 129.0, 128.8, 128.4, 125.9, 120.5, 117.6 ppm.
Spectroscopic data matched the literature[5].

1-(4-bromophenyl)-4-phenyl-1H-1,2,3-triazole (9p):

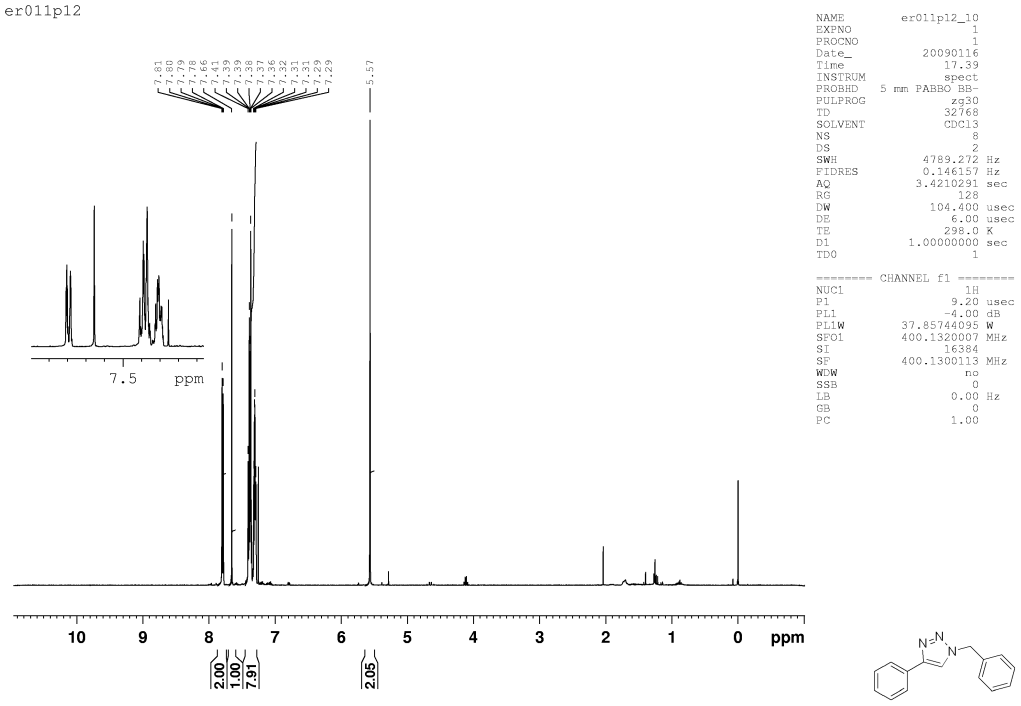


^{1}H NMR (400 MHz, $(CD_3)_2SO$): δ 9.35 (s, 1H), 7.95 – 7.40 (m, 9H) ppm.
Spectroscopic data matched the literature[7].

(4-(1-(4-nitrobenzyl)-1H-1,2,3-triazol-4-yl)phenyl)methanol (9q):



^{1}H NMR (400 MHZ, CD_3OD): δ 8.37 (s, 1H), 7.94 (d, J = 8 Hz, 2H), 7.55 (d, J = 8 Hz, 2H), 7.29 (d, J = 8 Hz, 2H), 7.13 (d, J = 8 Hz, 2H), 5.55 (s, 2H), 5.05 (t, J = 4 Hz, 1H), 4.27 (d, J = 4 Hz ,1H) ppm.
^{13}C NMR (100 MHz, $(CD_3)_2SO$): δ 147.3, 147.0, 143.4, 142.5, 129.1, 129.0, 127.1, 125.2, 124.0, 121.8, 62.8, 52.3 ppm.
Spectroscopic data matched the literature[8].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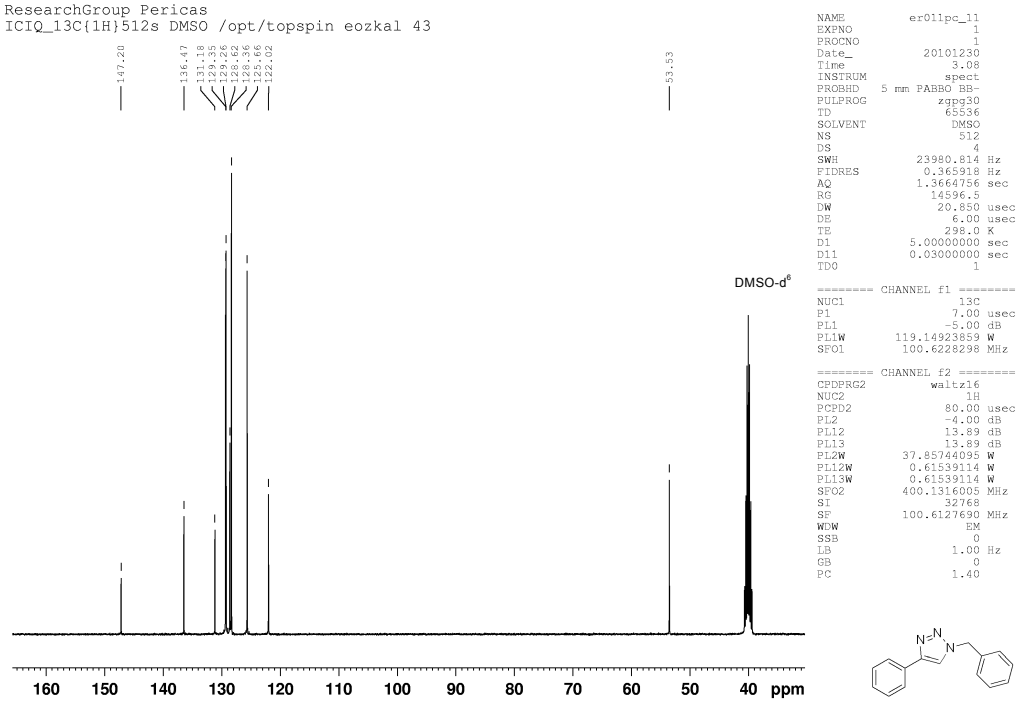
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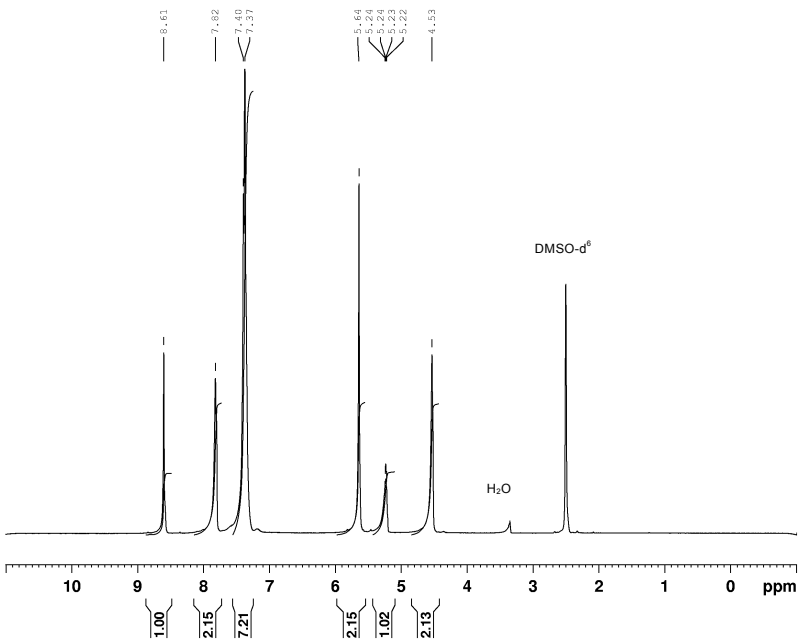


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TE              298.2 K
D1            1.00000000 sec
TD0                    1

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PL1              -3.00 dB
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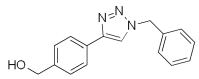
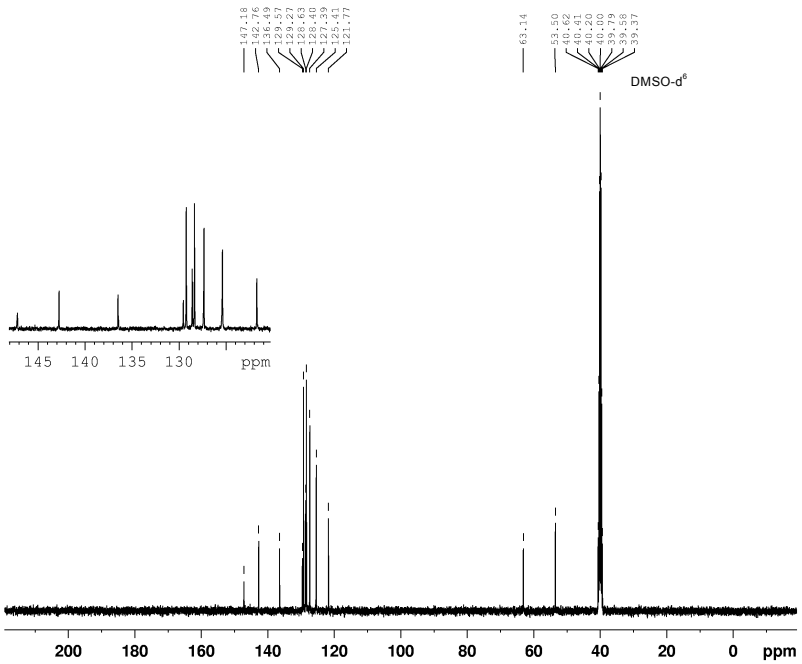
SFO1       400.1620008 MHz
SI                 16384
SF         400.1600000 MHz
WDW                   EM
SSB                    0
LB              0.30 Hz
GB                     0
PC                  1.00
```



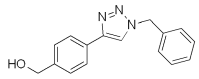
ResearchGroup Pericas
ICIQ_13C{1H}512s DMSO /opt/topspin eozkal 16

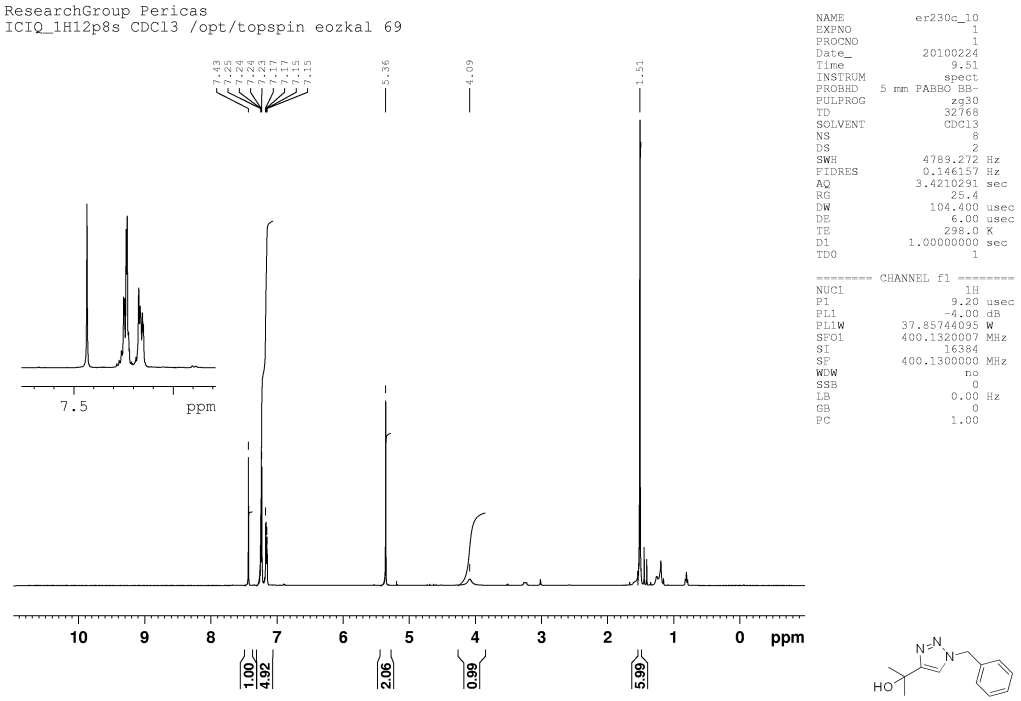


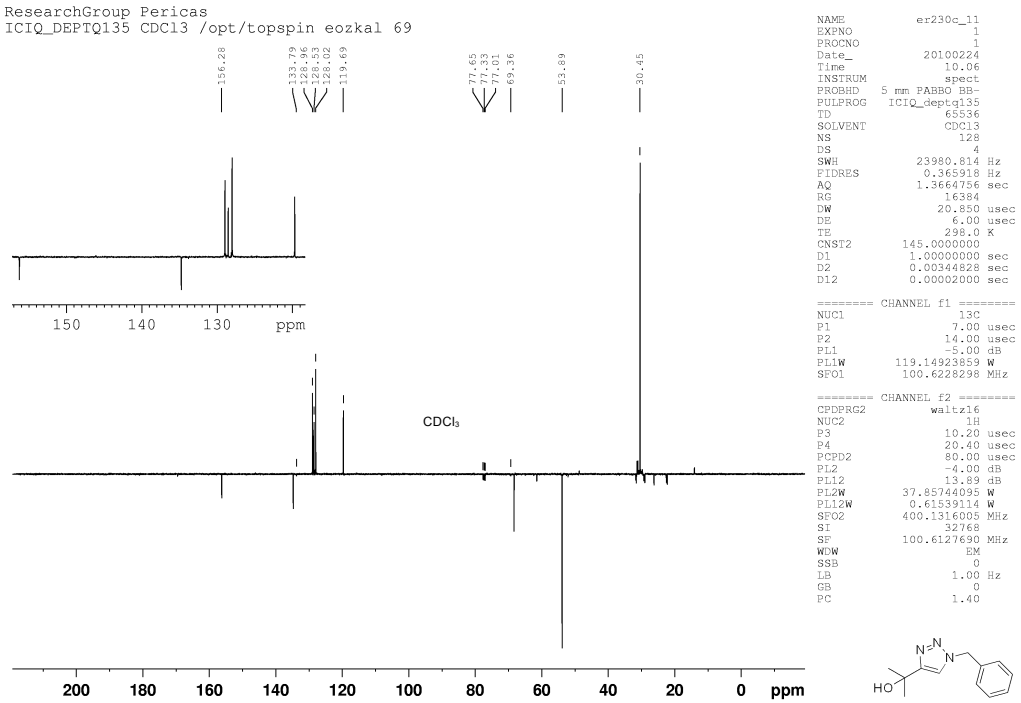

```
NAME            ersa12_11
EXPNO                   1
PROCNO                  1
Date_            20101224
Time                14.33
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG          zgpg30
TD                 65536
SOLVENT             DMSO
NS                   512
DS                     4
SWH          24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3632196 sec
RG                 46341
DW              20.800 usec
DE              10.00 usec
TE              298.1 K
D1            5.00000000 sec
D11           0.03000000 sec
TD0                    1

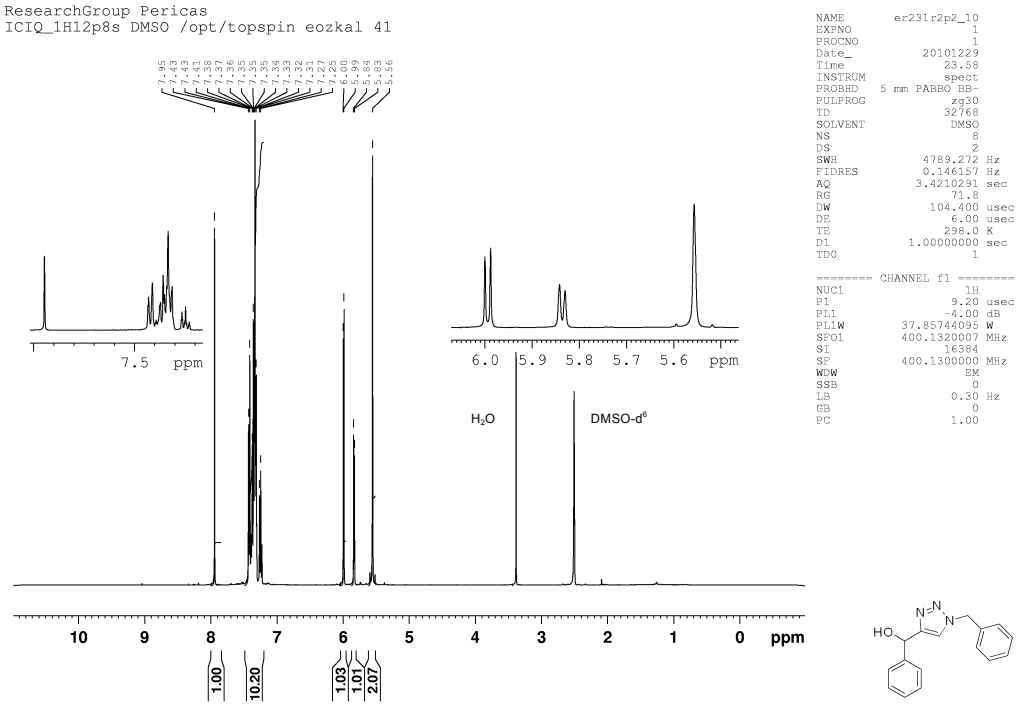
======== CHANNEL f1 ========
NUC1                  13C
P1               9.25 usec
PL1              -3.00 dB
PL1W        75.17808533 W
SFO1       100.6303736 MHz

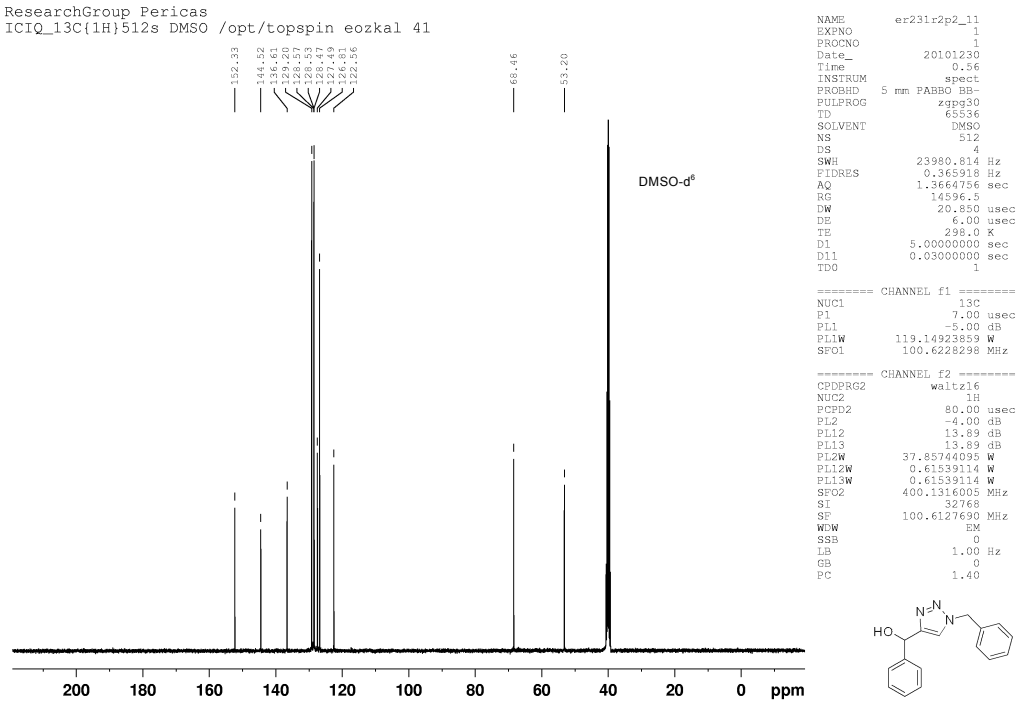
======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                   1H
PCPD2           100.00 usec
PL2              -3.00 dB
PL12             14.50 dB
PL13             14.50 dB
PL2W        30.07123375 W
PL12W        0.53475058 W
PL13W        0.53475058 W
SFO2       400.1616006 MHz
SI                 32768
SF         100.6203120 MHz
WDW                   EM
SSB                    0
LB              1.00 Hz
GB                     0
PC                  1.40
```

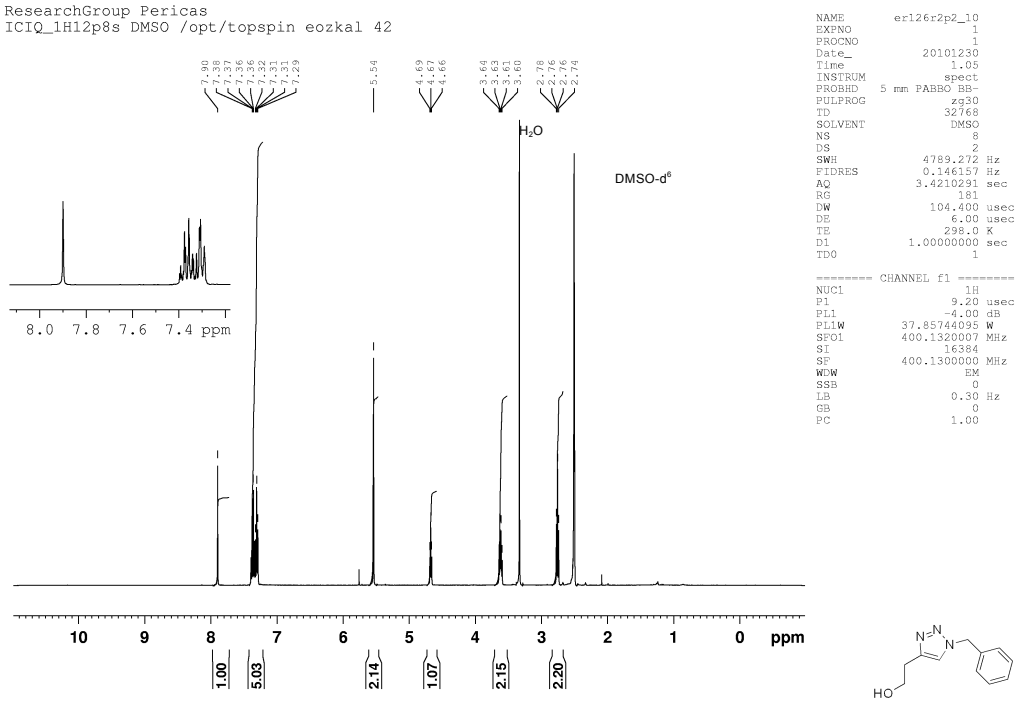


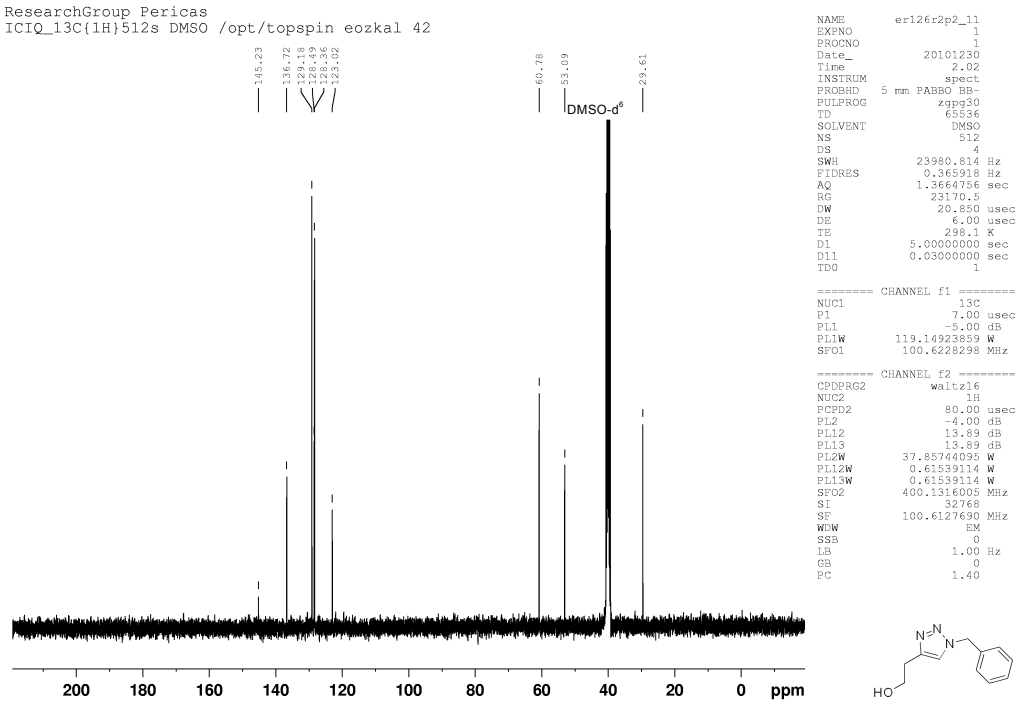

ResearchGroup Pericas
ICIQ_1H12p8s CDCl3 /opt/topspin eozkal 69
7.43
7.25
7.24
7.24
7.23
7.17
7.17
7.15
7.15
5.36
4.09
1.51
7.5
ppm
10 9 8 7 6 5 4 3 2 1 0 ppm
1.00
4.92
2.06
0.99
5.99
NAME er230c_10
EXPNO 1
PROCNO 1
Date_ 20100224
Time 9.51
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 2
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.42102291 sec
RG 25.4
DW 104.400 usec
DE 6.00 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 9.20 usec
PL1 -4.00 dB
PL1W 37.85744095 W
SFO1 400.1320007 MHz
SI 16384
SF 400.1300000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
HO


ResearchGroup Pericas
ICIQ_DEPTQ135 CDCl3 /opt/topspin eozkal 69
156.28
133.79
128.96
128.53
128.02
119.69
77.65
77.33
77.01
69.36
53.89
30.45
150 140 130 ppm
CDCl3
200 180 160 140 120 100 80 60 40 20 0 ppm
NAME er230c_11
EXPNO 1
PROCNO 1
Date_ 20100224
Time 10.06
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG ICIQ_deptq135
TD 65536
SOLVENT CDCl3
NS 128
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 16384
DW 20.850 usec
DE 6.00 usec
TE 298.0 K
CNST2 145.0000000
D1 1.00000000 sec
D2 0.00344828 sec
D12 0.00002000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 7.00 usec
P2 14.00 usec
PL1 -5.00 dB
PL1W 119.14923859 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
P3 10.20 usec
P4 20.40 usec
PCPD2 80.00 usec
PL2 -4.00 dB
PL12 13.89 dB
PL2W 37.85744095 W
PL12W 0.61539114 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
HO

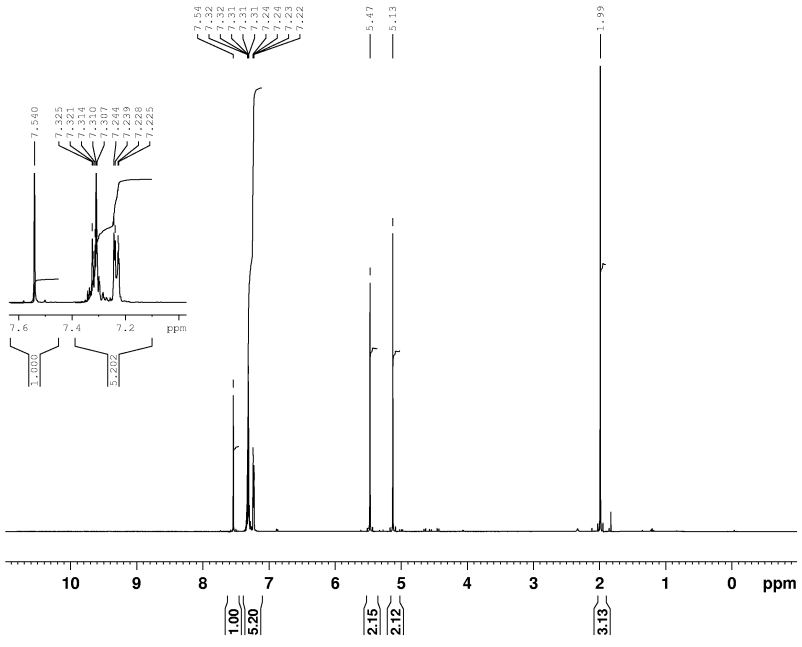

ResearchGroup Pericas
ICIQ_1H12p8s DMSO /opt/topspin eozkal 41
7.95 7.43 7.43 7.41 7.41 7.38 7.37 7.36 7.35 7.35 7.35 7.34 7.33 7.32 7.31 7.27 7.25 6.00 5.99 5.84 5.83 5.56
7.5 ppm
6.0 5.9 5.8 5.7 5.6 ppm
H2O
DMSO-d6
10 9 8 7 6 5 4 3 2 1 0 ppm
1.00 10.20 1.03 1.01 2.07
NAME er231r2p2_10
EXPNO 1
PROCNO 1
Date_ 20101229
Time 23.58
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 8
DS 2
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 71.8
DW 104.400 usec
DE 6.00 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 9.20 usec
PL1 -4.00 dB
PL1W 37.85744095 W
SFO1 400.1320007 MHz
SI 16384
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


ResearchGroup Pericas
ICIQ_13C{1H}512s DMSO /opt/topspin eozkal 41
152.33 144.52 136.61 129.20 128.57 128.53 128.47 127.49 126.81 122.56
68.46 53.20
DMSO-d6
200 180 160 140 120 100 80 60 40 20 0 ppm
NAME er231r2p2_11
EXPNO 1
PROCNO 1
Date_ 20101230
Time 0.56
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 512
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 14596.5
DW 20.850 usec
DE 6.00 usec
TE 298.0 K
D1 5.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 7.00 usec
PL1 -5.00 dB
PL1W 119.14923859 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -4.00 dB
PL12 13.89 dB
PL13 13.89 dB
PL2W 37.85744095 W
PL12W 0.61539114 W
PL13W 0.61539114 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



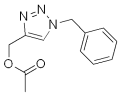


1H experiment

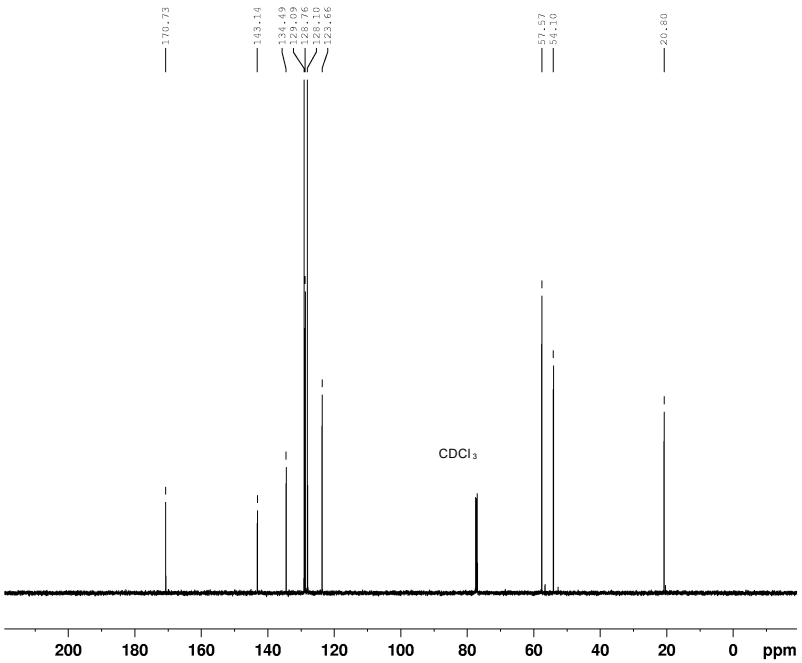



```
NAME            er264p_1_1
EXPNO                    1
PROCNO                   1
Date_             20100505
Time                 11.26
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG               zg30
TD                   32768
SOLVENT              CDCl3
NS                       8
DS                       2
SWH               6009.615 Hz
FIDRES            0.183399 Hz
AQ                2.7264309 sec
RG                    28.5
DW                 83.200 usec
DE                 10.00 usec
TE                  298.0 K
D1                1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                  13.80 usec
PL1                  3.00 dB
PL1W            12.58925438 W
SFO1           500.1325007 MHz
SI                   16384
SF             500.1300000 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```



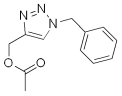
13C{1H} experiment

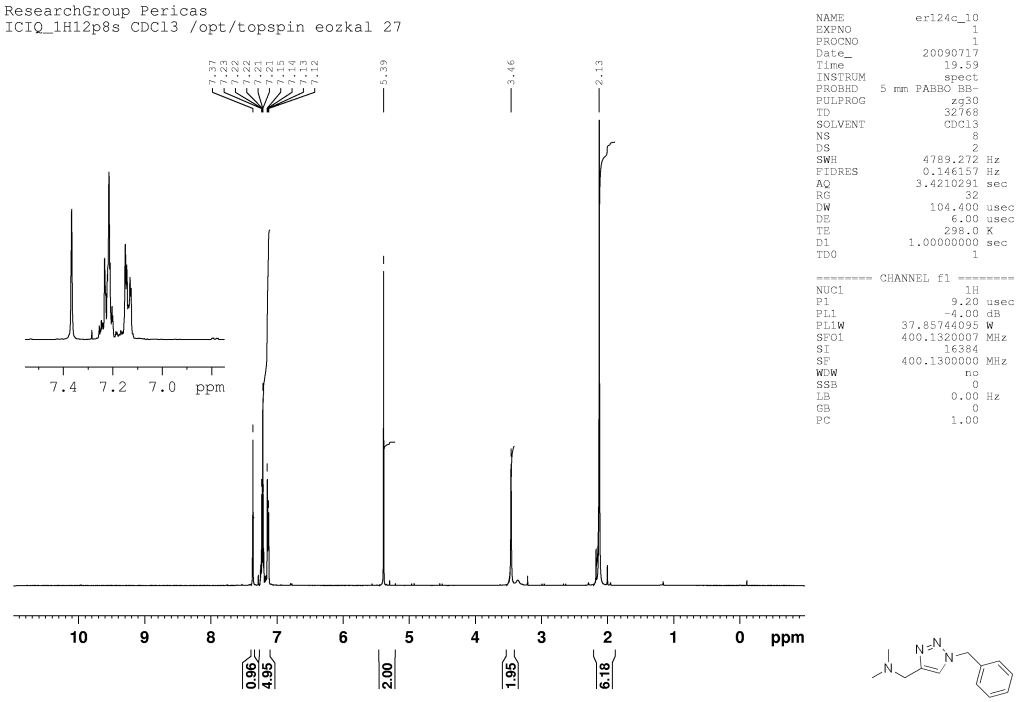


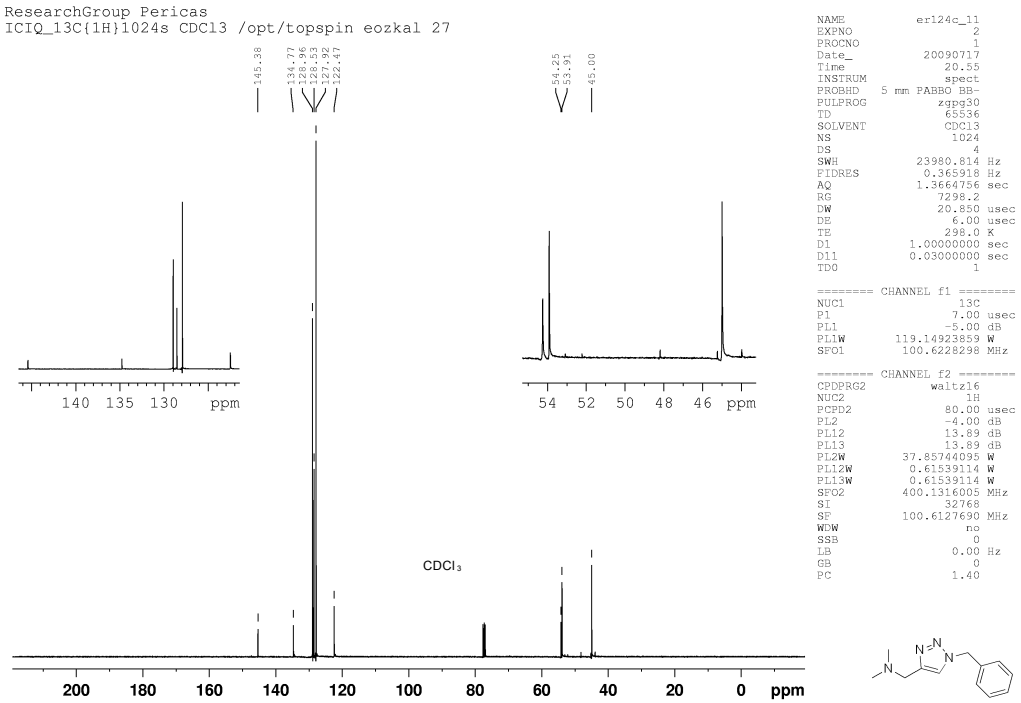

```
NAME            er264p_2_1
EXPNO                    1
PROCNO                   1
Date_             20100505
Time                 11.33
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                   65536
SOLVENT              CDCl3
NS                      55
DS                       4
SWH              30303.031 Hz
FIDRES            0.462388 Hz
AQ                1.0814105 sec
RG                    1820
DW                 16.500 usec
DE                 10.00 usec
TE                  298.1 K
D1                1.00000000 sec
D11               0.03000000 sec
TD0                      1

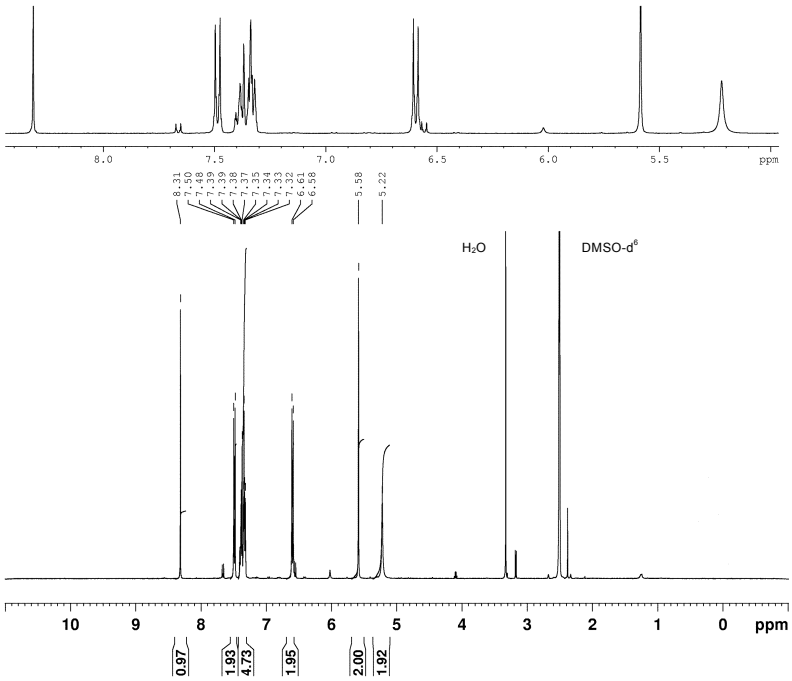
======== CHANNEL f1 ========
NUC1                   13C
P1                  8.50 usec
PL1                  0.00 dB
PL1W            75.35659027 W
SFO1           125.7703648 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                    1H
PCPD2               80.00 usec
PL2                  3.00 dB
PL12                18.14 dB
PL13                20.39 dB
PL2W            12.58925438 W
PL12W            0.38547841 W
PL13W            0.22961490 W
SFO2           500.1320005 MHz
SI                   32768
SF             125.7577890 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```



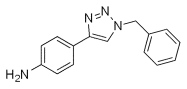

ResearchGroup Pericas
ICIQ_1H12p8s CDCl3 /opt/topspin eozkal 27
7.37
7.23
7.22
7.22
7.21
7.21
7.15
7.14
7.13
7.12
5.39
3.46
2.13
7.4 7.2 7.0 ppm
10 9 8 7 6 5 4 3 2 1 0 ppm
0.96
4.95
2.00
1.95
6.18
NAME er124c_10
EXPNO 1
PROCNO 1
Date_ 20090717
Time 19.59
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 2
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 32
DW 104.400 usec
DE 6.00 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 9.20 usec
PL1 -4.00 dB
PL1W 37.85744095 W
SFO1 400.1320007 MHz
SI 16384
SF 400.1300000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

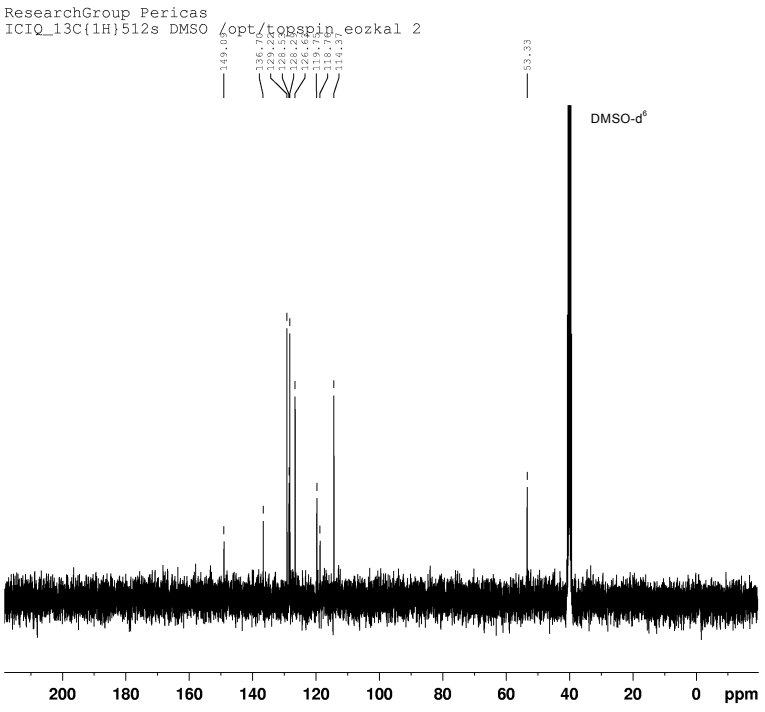

ResearchGroup Pericas
ICIQ_13C{1H}1024s CDCl3 /opt/topspin eozkal 27
145.38
134.77
128.96
128.53
127.92
122.47
54.25
53.91
45.00
140 135 130 ppm
54 52 50 48 46 ppm
CDCl3
200 180 160 140 120 100 80 60 40 20 0 ppm
NAME er124c_11
EXPNO 2
PROCNO 1
Date_ 20090717
Time 20.55
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 7298.2
DW 20.850 usec
DE 6.00 usec
TE 298.0 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 7.00 usec
PL1 -5.00 dB
PL1W 119.14923859 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -4.00 dB
PL12 13.89 dB
PL13 13.89 dB
PL2W 37.85744095 W
PL12W 0.61539114 W
PL13W 0.61539114 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127690 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40




```
NAME            er413c_10
EXPNO                   1
PROCNO                  1
Date_            20101223
Time                10.54
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG              zg30
TD                  32768
SOLVENT              DMSO
NS                      8
DS                      2
SWH              4807.692 Hz
FIDRES           0.146719 Hz
AQ               3.4080260 sec
RG                  322.5
DW                104.000 usec
DE                 10.00 usec
TE                 297.9 K
D1              1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  13.00 usec
PL1                 -3.00 dB
PL1W         30.07123375 W
SFO1         400.1620008 MHz
SI                  16384
SF           400.1600000 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```

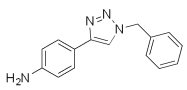


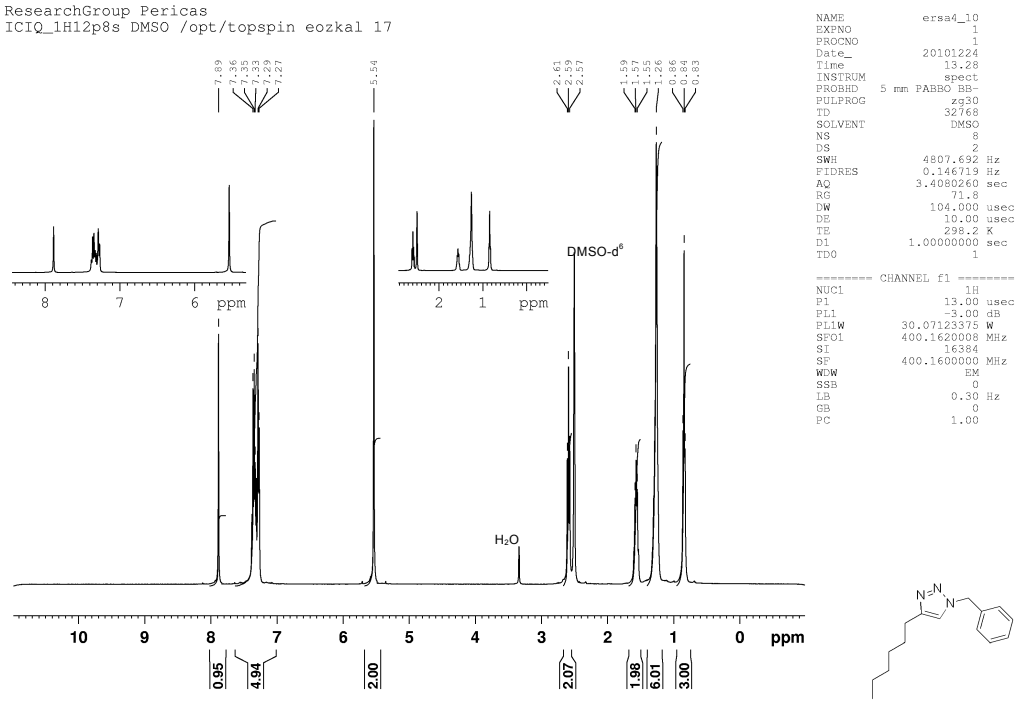



```
NAME            er413c_11
EXPNO                   1
PROCNO                  1
Date_            20101223
Time                11.18
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG            zgpg30
TD                  65536
SOLVENT              DMSO
NS                    512
DS                      4
SWH             24038.461 Hz
FIDRES           0.366798 Hz
AQ               1.3632196 sec
RG                  46341
DW                 20.800 usec
DE                 10.00 usec
TE                 298.0 K
D1              1.00000000 sec
D11             0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                  9.25 usec
PL1                 -3.00 dB
PL1W         75.17808533 W
SFO1         100.6303736 MHz

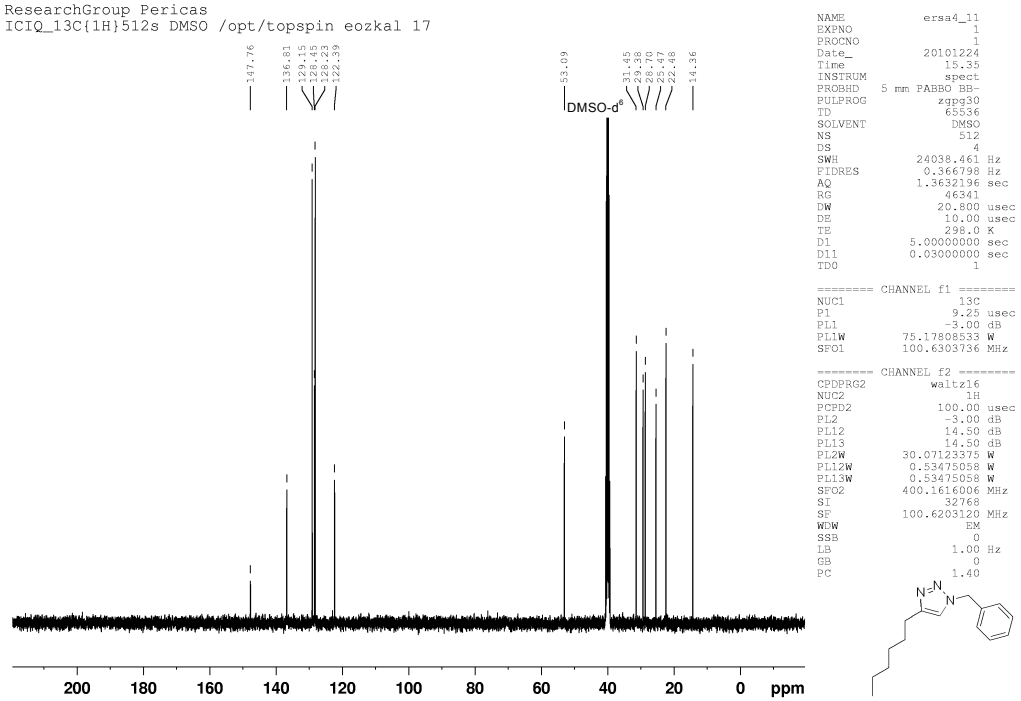
======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                   1H
PCPD2              100.00 usec
PL2                 -3.00 dB
PL12                14.50 dB
PL13                14.50 dB
PL2W         30.07123375 W
PL12W         0.53475058 W
PL13W         0.53475058 W
SFO2         400.1616006 MHz
SI                  32768
SF           100.6203120 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40
```






```
NAME                ersa4_10
EXPNO                      1
PROCNO                     1
Date_               20101224
Time                   13.28
INSTRUM                spect
PROBHD    5 mm PABBO BB-
PULPROG                zg30
TD                    32768
SOLVENT                DMSO
NS                        8
DS                        2
SWH                4807.692 Hz
FIDRES             0.146719 Hz
AQ                 3.4080260 sec
RG                     71.8
DW                   104.000 usec
DE                    10.00 usec
TE                    298.2 K
D1                 1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                     1H
P1                    13.00 usec
PL1                   -3.00 dB
PL1W          30.07123375 W
SFO1          400.1620008 MHz
SI                    16384
SF            400.1600000 MHz
WDW                      EM
SSB                       0
LB                     0.30 Hz
GB                        0
PC                     1.00
```

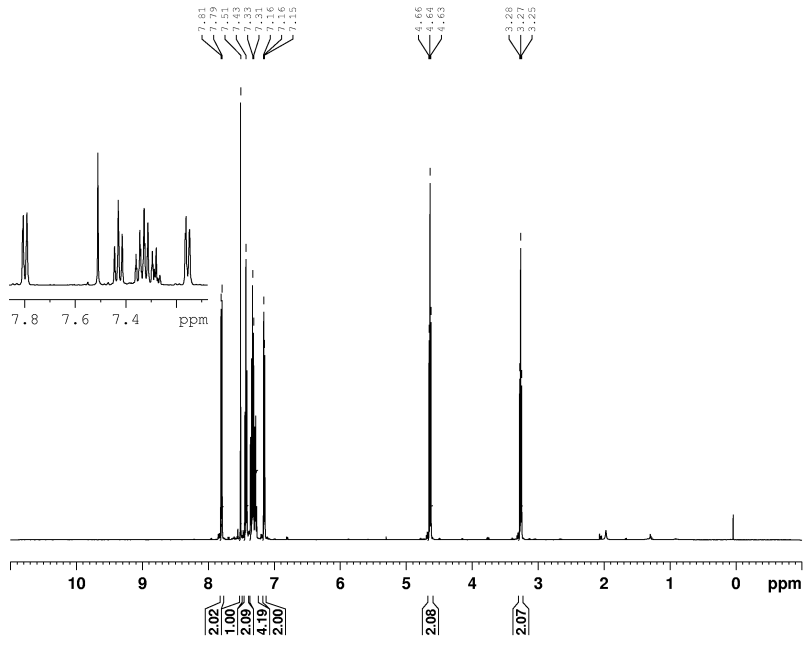




```
NAME                ersa4_11
EXPNO                      1
PROCNO                     1
Date_               20101224
Time                   15.35
INSTRUM                spect
PROBHD    5 mm PABBO BB-
PULPROG             zgpg30
TD                    65536
SOLVENT                DMSO
NS                      512
DS                        4
SWH               24038.461 Hz
FIDRES             0.366798 Hz
AQ                 1.3632196 sec
RG                     46341
DW                    20.800 usec
DE                    10.00 usec
TE                    298.0 K
D1                 5.00000000 sec
D11                0.03000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                     13C
P1                     9.25 usec
PL1                   -3.00 dB
PL1W          75.17808533 W
SFO1          100.6303736 MHz

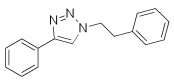
======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                     1H
PCPD2                100.00 usec
PL2                   -3.00 dB
PL12                  14.50 dB
PL13                  14.50 dB
PL2W          30.07123375 W
PL12W          0.53475058 W
PL13W          0.53475058 W
SFO2          400.1616006 MHz
SI                    32768
SF            100.6203120 MHz
WDW                      EM
SSB                       0
LB                     1.00 Hz
GB                        0
PC                     1.40
```

1H experiment

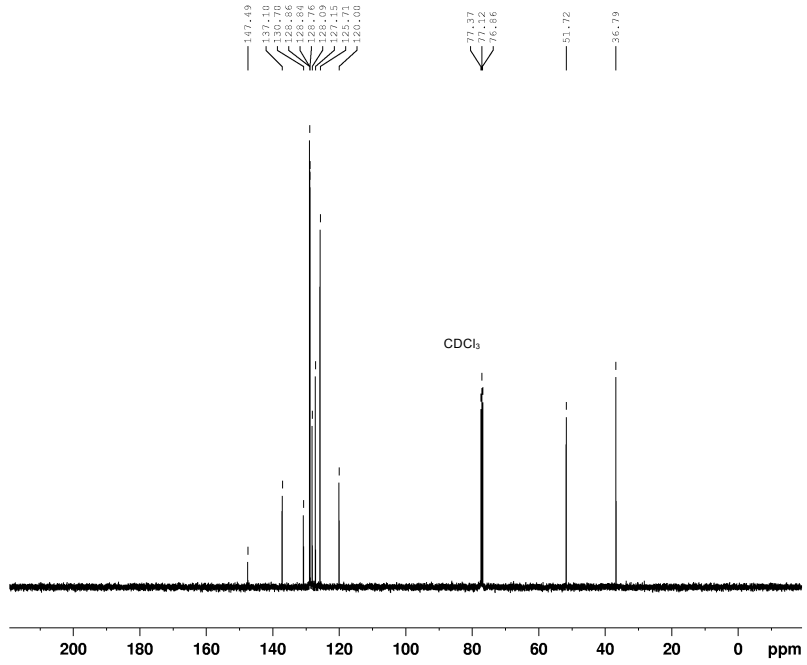



```
NAME          er262c_1_1.topspin
EXPNO                          1
PROCNO                         1
Date_                   20100505
Time                       11.11
INSTRUM                    spect
PROBHD    5 mm PABBO BB-
PULPROG                     zg30
TD                         32768
SOLVENT                    CDCl3
NS                             6
DS                             2
SWH                     6009.615 Hz
FIDRES                  0.183399 Hz
AQ                      2.7264309 sec
RG                          50.8
DW                        83.200 usec
DE                        10.00 usec
TE                        298.1 K
D1                      1.00000000 sec
TD0                            1

======== CHANNEL f1 ========
NUC1                          1H
P1                         13.80 usec
PL1                         3.00 dB
PL1W                12.58925438 W
SFO1                500.1325007 MHz
SI                         16384
SF                  500.1300000 MHz
WDW                           EM
SSB                            0
LB                          0.30 Hz
GB                             0
PC                          1.00
```



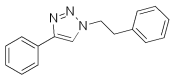
13C {1H} experiment, 1024 scans

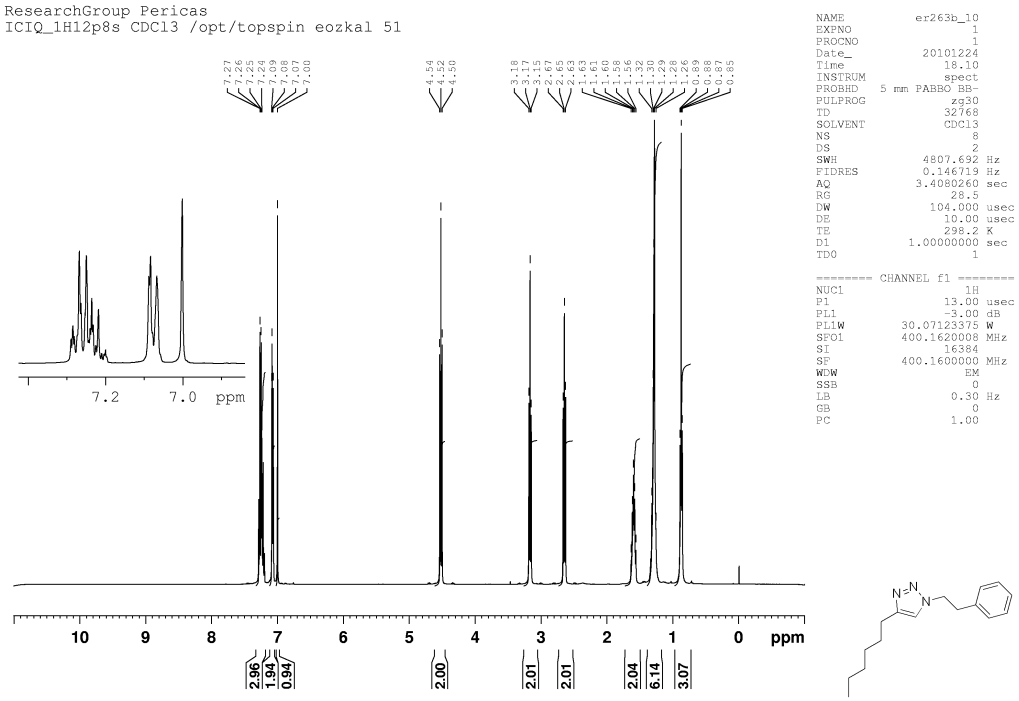


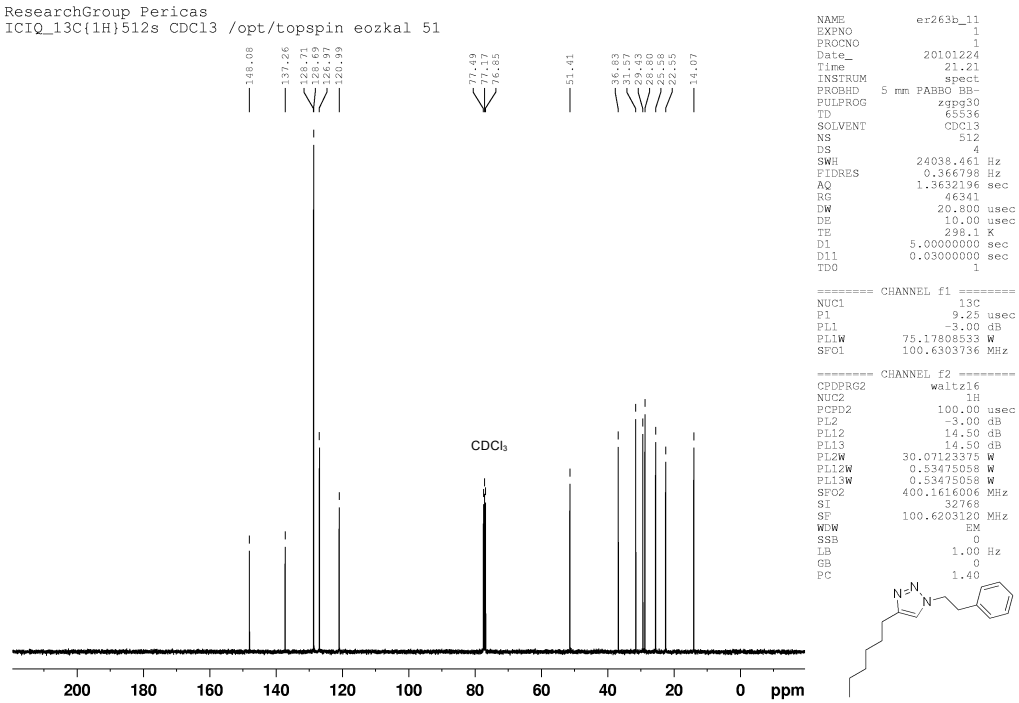

```
NAME          er262c_2_1
EXPNO                          1
PROCNO                         1
Date_                   20100505
Time                       11.14
INSTRUM                    spect
PROBHD    5 mm PABBO BB-
PULPROG                   zgpg30
TD                         65536
SOLVENT                    CDCl3
NS                            64
DS                             4
SWH                    30303.031 Hz
FIDRES                  0.462388 Hz
AQ                      1.0814105 sec
RG                          1820
DW                        16.500 usec
DE                        10.00 usec
TE                        298.1 K
D1                      1.00000000 sec
D11                     0.03000000 sec
TD0                            1

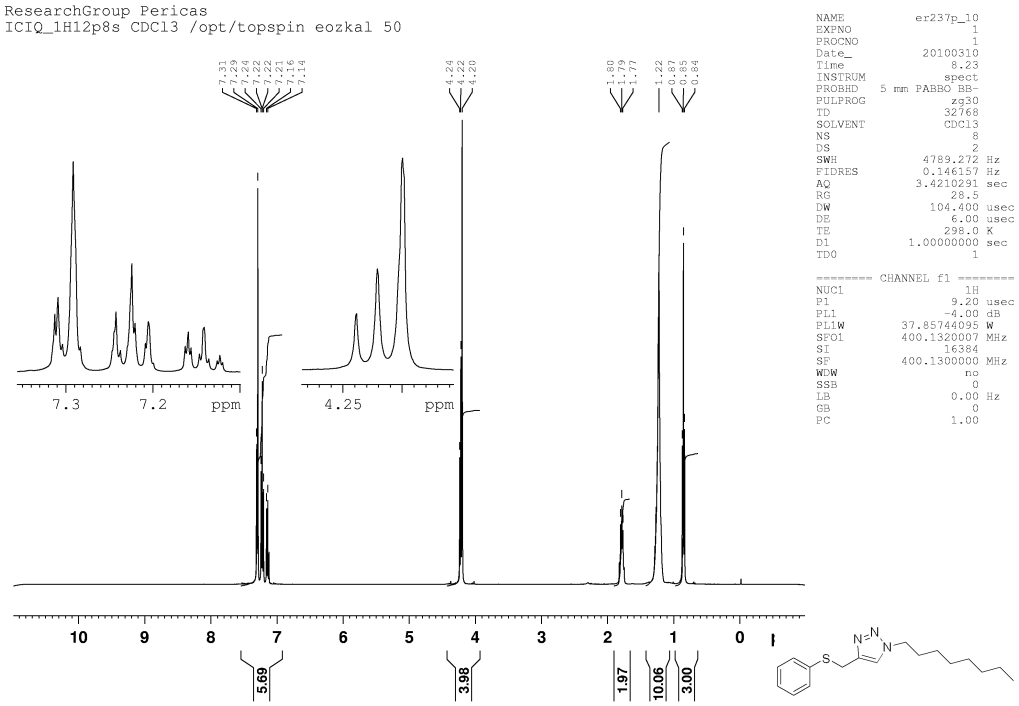
======== CHANNEL f1 ========
NUC1                          13C
P1                          8.50 usec
PL1                         0.00 dB
PL1W                75.35659027 W
SFO1                125.7703648 MHz

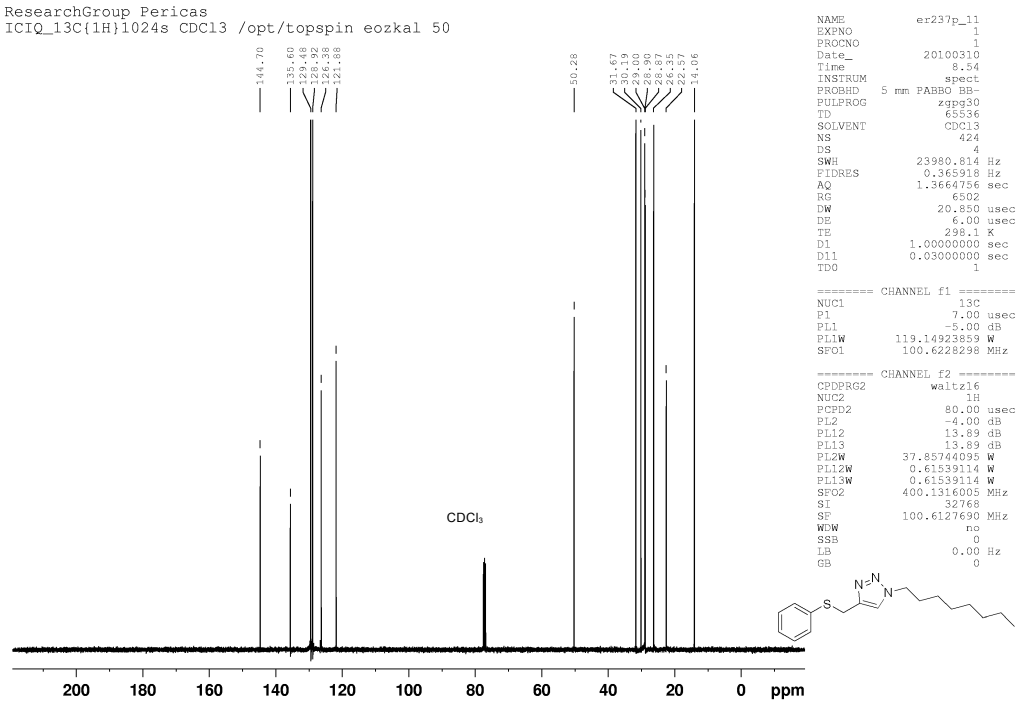
======== CHANNEL f2 ========
CPDPRG2                 waltz16
NUC2                          1H
PCPD2                      80.00 usec
PL2                         3.00 dB
PL12                       18.14 dB
PL13                       20.39 dB
PL2W                12.58925438 W
PL12W                0.38547841 W
PL13W                0.22961490 W
SFO2                500.1320005 MHz
SI                         32768
SF                  125.7577890 MHz
WDW                           EM
SSB                            0
LB                          1.00 Hz
GB                             0
PC                          1.40
```

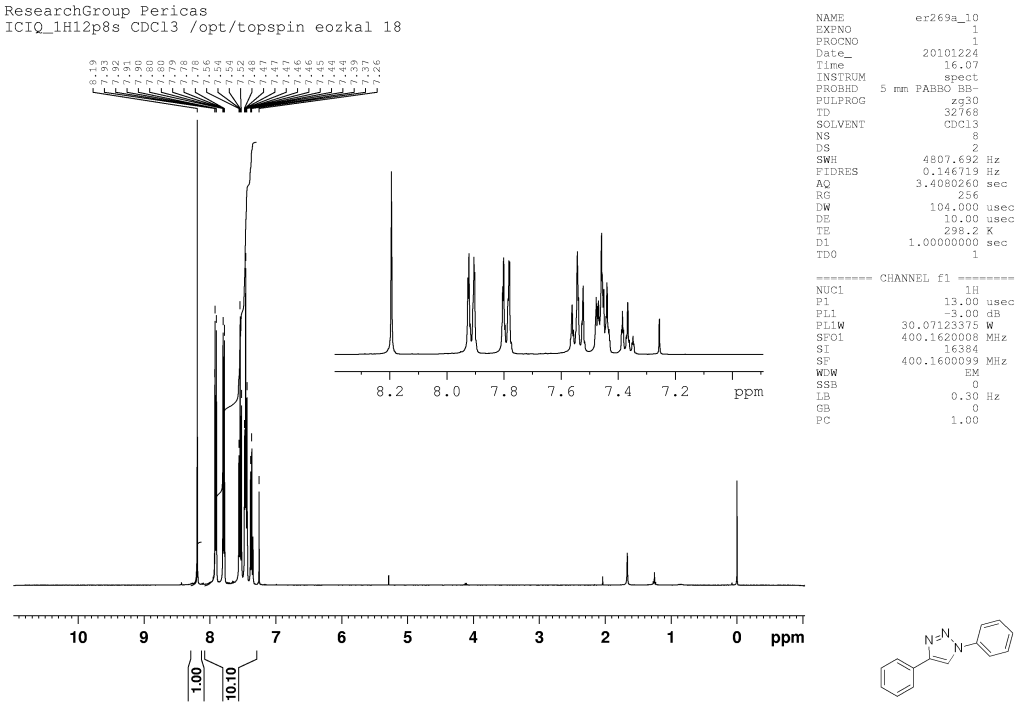


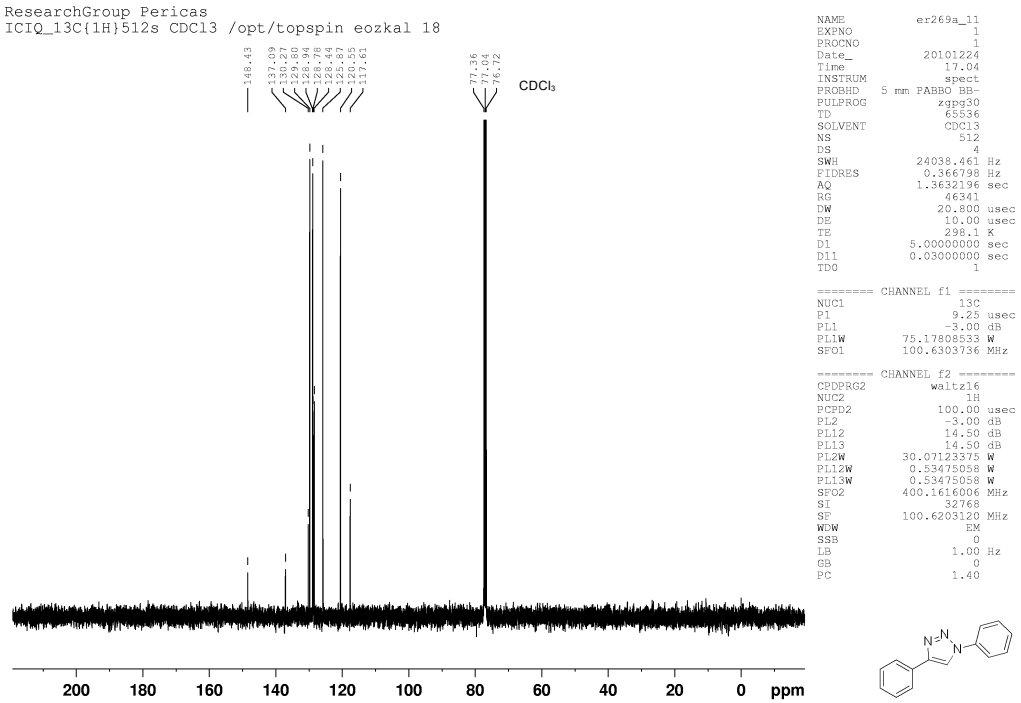


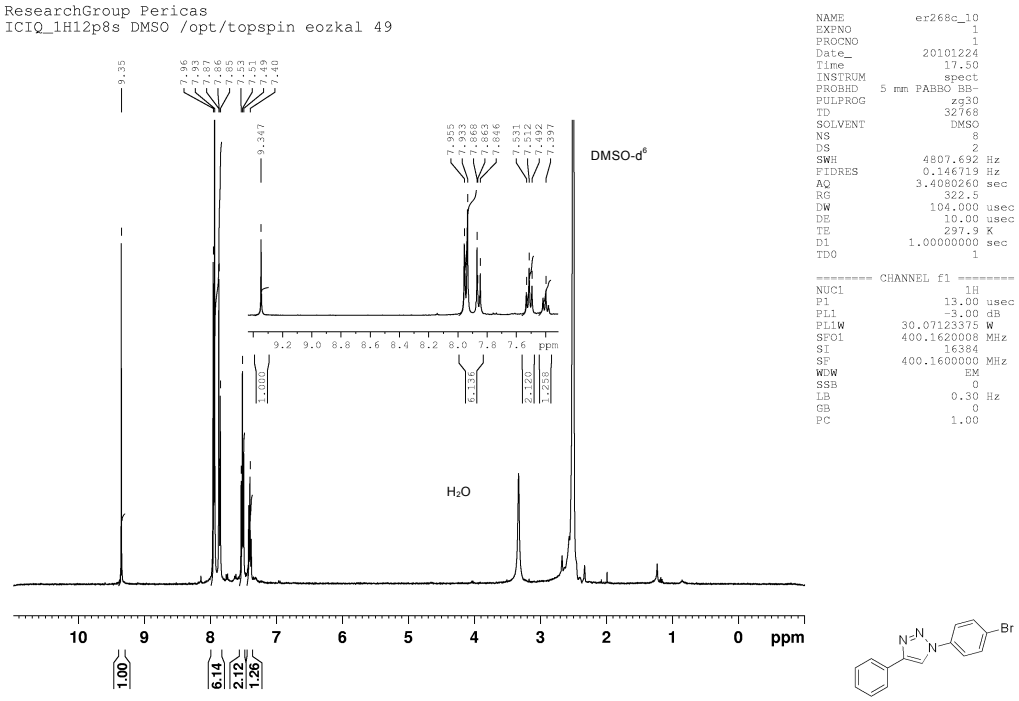




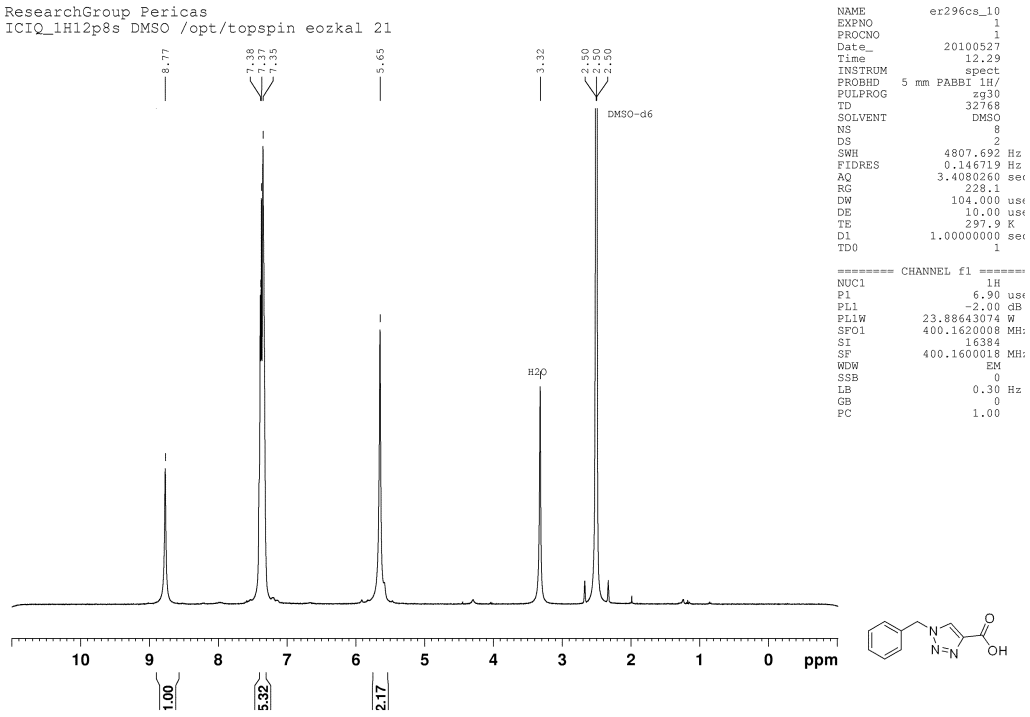


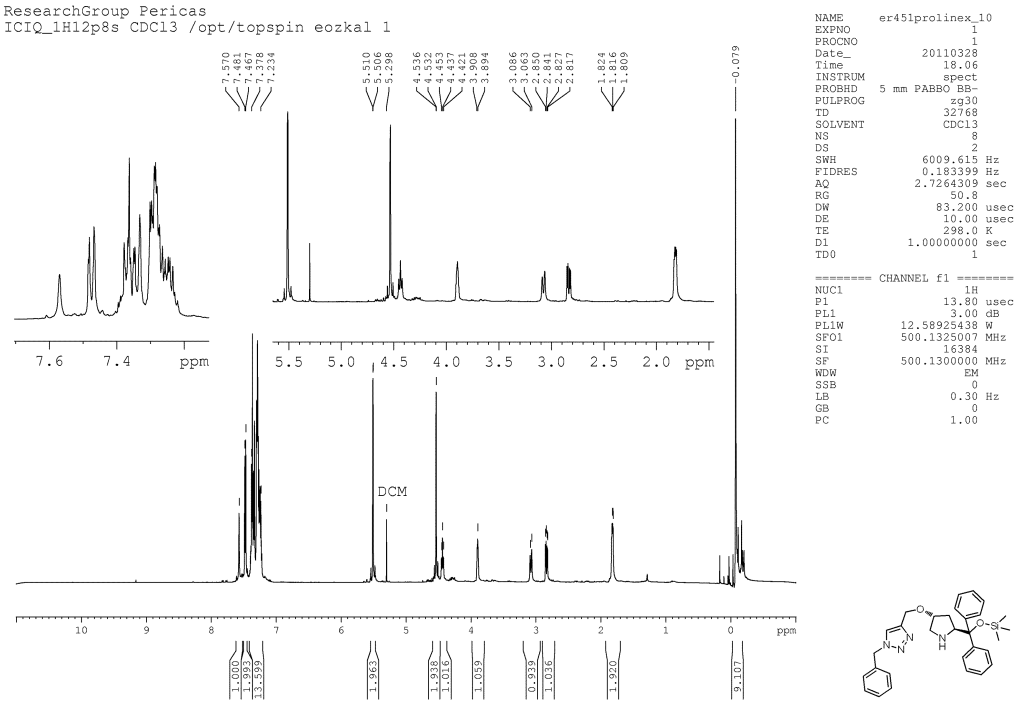

ResearchGroup Pericas
ICIQ_1H12p8s CDCl3 /opt/topspin eozkal 18
8.19 7.93 7.92 7.91 7.90 7.80 7.80 7.79 7.78 7.78 7.56 7.54 7.54 7.52 7.46 7.47 7.47 7.46 7.46 7.45 7.44 7.39 7.37 7.26
8.2 8.0 7.8 7.6 7.4 7.2 ppm
10 9 8 7 6 5 4 3 2 1 0 ppm
1.00
10.10
NAME er269a_10
EXPNO 1
PROCNO 1
Date_ 20101224
Time 16.07
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 2
SWH 4807.692 Hz
FIDRES 0.146719 Hz
AQ 3.4080260 sec
RG 256
DW 104.000 usec
DE 10.00 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.00 usec
PL1 -3.00 dB
PL1W 30.07123375 W
SFO1 400.1620008 MHz
SI 16384
SF 400.1600099 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

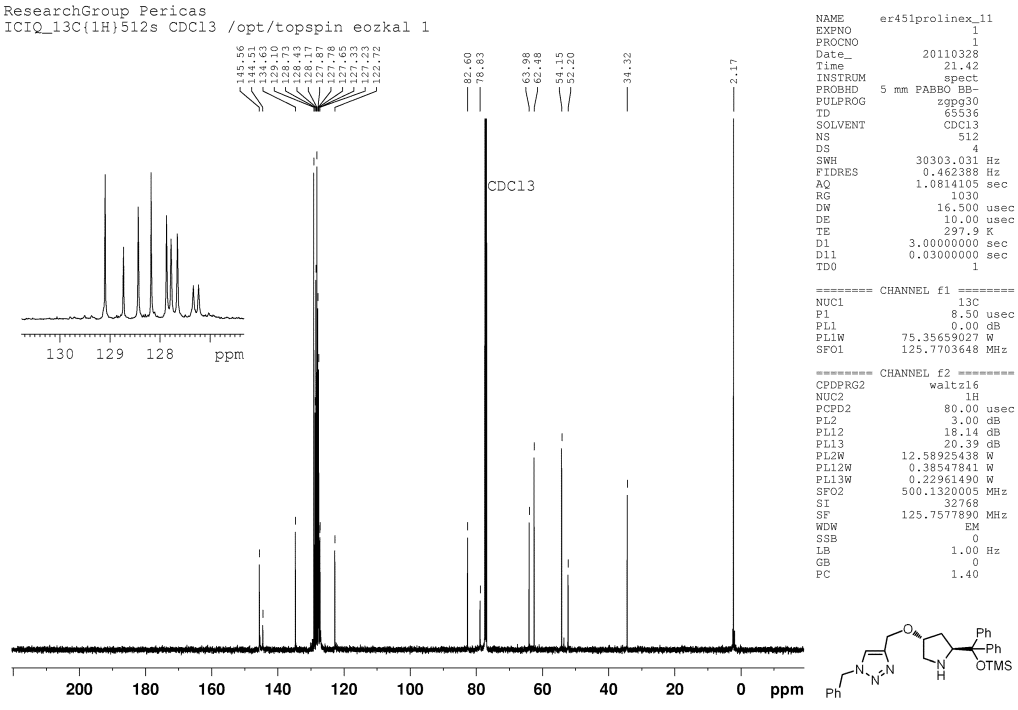

ResearchGroup Pericas
ICIQ_13C{1H}512s CDCl3 /opt/topspin eozkal 18
148.43 137.09 130.27 129.60 128.94 128.78 128.44 125.87 120.65 117.61
77.36 77.04 76.72
CDCl3
200 180 160 140 120 100 80 60 40 20 0 ppm
NAME er269a_11
EXPNO 1
PROCNO 1
Date_ 20101224
Time 17.04
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3632196 sec
RG 46341
DW 20.800 usec
DE 10.00 usec
TE 298.1 K
D1 5.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.25 usec
PL1 -3.00 dB
PL1W 75.17808533 W
SFO1 100.6303736 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -3.00 dB
PL12 14.50 dB
PL13 14.50 dB
PL2W 30.07123375 W
PL12W 0.53475058 W
PL13W 0.53475058 W
SFO2 400.1616006 MHz
SI 32768
SF 100.6203120 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

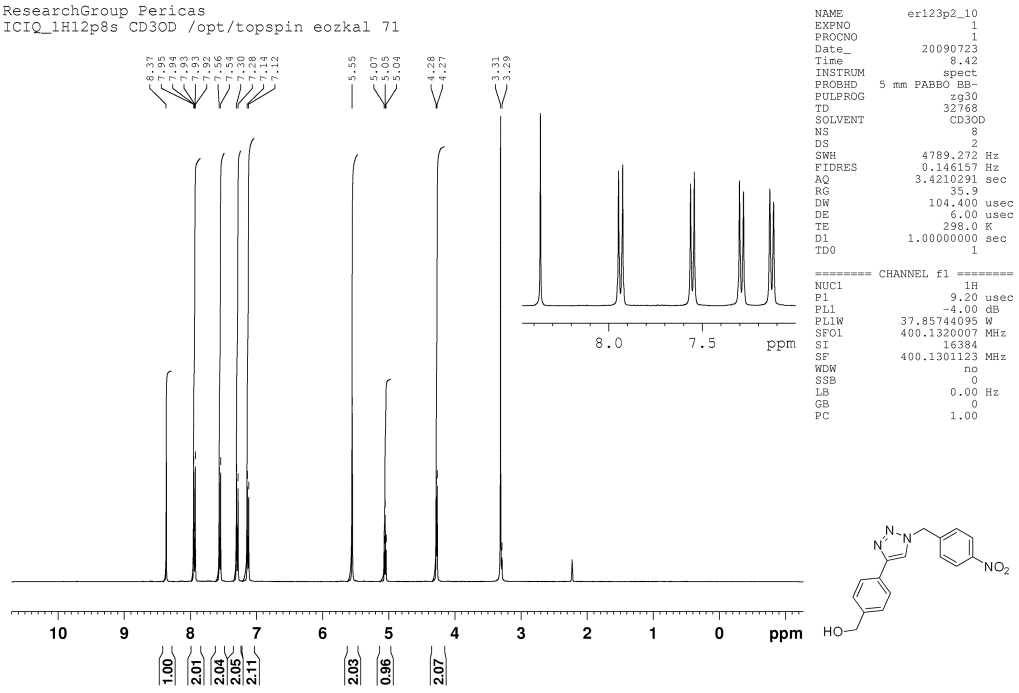


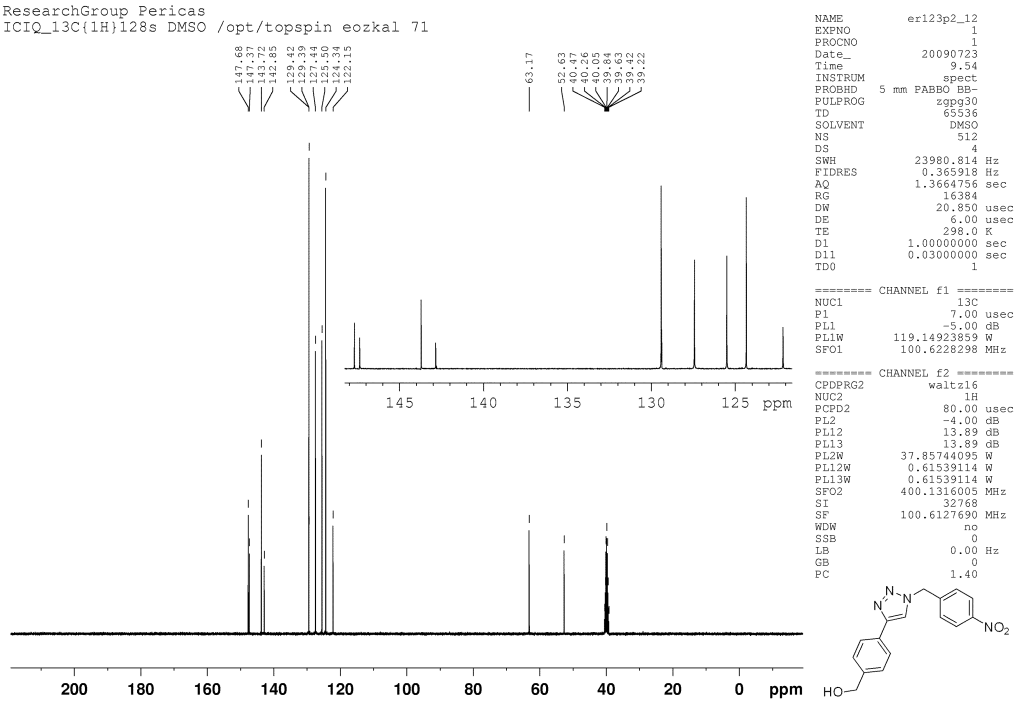
NAME	er268c_10
EXPNO	1
PROCNO	1
Date_	20101224
Time	17.50
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	32768
SOLVENT	DMSO
NS	8
DS	2
SWH	4807.692 Hz
FIDRES	0.146719 Hz
AQ	3.4080260 sec
RG	322.5
DW	104.000 usec
DE	10.00 usec
TE	297.9 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	13.00 usec
PL1	-3.00 dB
PL1W	30.07123375 W
SFO1	400.1620008 MHz
SI	16384
SF	400.1600000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00




ResearchGroup Pericas
ICIQ_1H12p8s CDCl3 /opt/topspin eozkal 1
7.570
7.561
7.467
7.378
7.234
5.510
5.506
5.298
4.536
4.532
4.453
4.437
4.271
3.908
3.894
3.086
3.063
2.850
2.841
2.827
2.817
1.824
1.816
1.809
0.079
DCM
7.6
7.4
ppm
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
ppm
10
9
8
7
6
5
4
3
2
1
0
ppm
1.000
1.993
13.599
1.963
1.938
1.016
1.059
0.939
1.036
1.920
9.107
NAME er451prolinex_10
EXPNO 1
PROCNO 1
Date_ 20110328
Time 18.06
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 2
SWH 6009.615 Hz
FIDRES 0.183399 Hz
AQ 2.7264309 sec
RG 50.8
DW 83.200 usec
DE 10.00 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.80 usec
PL1 3.00 dB
PL1W 12.58925438 W
SFO1 500.1325007 MHz
SI 16384
SF 500.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


ResearchGroup Pericas
ICIQ_13C{1H}512s CDCl3 /opt/topspin eozkal 1
145.56
144.51
134.63
129.10
128.73
128.43
128.17
127.87
127.78
127.65
127.33
127.23
122.72
82.60
78.83
63.98
62.48
54.15
52.20
34.32
2.17
CDCl3
130
129
128
ppm
200
180
160
140
120
100
80
60
40
20
0
ppm
NAME er451prolinex_11
EXPNO 1
PROCNO 1
Date_ 20110328
Time 21.42
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0814105 sec
RG 1030
DW 16.500 usec
DE 10.00 usec
TE 297.9 K
D1 3.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 8.50 usec
PL1 0.00 dB
PL1W 75.35659027 W
SFO1 125.7703648 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 3.00 dB
PL12 18.14 dB
PL13 20.39 dB
PL2W 12.58925438 W
PL12W 0.38547841 W
PL13W 0.22961490 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


ResearchGroup Pericas
ICIQ_1H12p8s CD3OD /opt/topspin eozkal 71
8.37
7.95
7.94
7.93
7.92
7.56
7.54
7.30
7.28
7.13
7.12
5.55
5.07
5.05
5.04
4.28
4.27
3.31
3.29
8.0
7.5
ppm
10
9
8
7
6
5
4
3
2
1
0
ppm
1.00
2.01
2.04
2.05
2.11
2.03
0.96
2.07
NAME er123p2_10
EXPNO 1
PROCNO 1
Date_ 20090723
Time 8.42
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 32768
SOLVENT CD3OD
NS 8
DS 2
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 35.9
DW 104.400 usec
DE 6.00 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 9.20 usec
PL1 -4.00 dB
PL1W 37.85744095 W
SFO1 400.1320007 MHz
SI 16384
SF 400.1301123 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
HO
NO2


ResearchGroup Pericas
ICIQ_13C{1H}128s DMSO /opt/topspin eozkal 71
147.68
143.77
143.72
142.85
129.42
129.39
127.44
125.50
124.34
122.15
63.17
52.63
40.47
40.26
40.05
39.84
39.63
39.42
39.22
145
140
135
130
125
ppm
200
180
160
140
120
100
80
60
40
20
0
ppm
NAME er123p2_12
EXPNO 1
PROCNO 1
Date_ 20090723
Time 9.54
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 512
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 16384
DW 20.850 usec
DE 6.00 usec
TE 298.0 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 7.00 usec
PL1 -5.00 dB
PL1W 119.14923859 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -4.00 dB
PL12 13.89 dB
PL13 13.89 dB
PL2W 37.85744095 W
PL12W 0.61539114 W
PL13W 0.61539114 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127690 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40
HO
NO2

V. References

[1]Adapted from: (a) A. J. Brenner, E. D. Harris, *Anal*. *Biochem*. 1995, **226**, 80. (b) A. J. Brenner, E. D. Harris, *Anal*. *Biochem*. 1995, **230**, 360 (erratum).

[2] H. Sharghi, R. Khalifeh, M. M. Doroodmand, *Adv. Synth. Catal.* 2009, **351**, 207.

[3] Q. Zhang, J. M. Tacaks, *Org. Lett.* 2008 **10**, 545.

[4] P. Appukkuttan, W. Dehaen, V. V. Fokin, E. Van der Eycken, *Org. Lett.* 2004 **6**, 4223.

[5] S. Chassaing, S.; A. S. S. Sido, A. Alix, M. Kumarraja, P. Pale, J. Sommer, *Chem. Eur. J.* 2008, **14**, 6713.

[6] Z. Gonda, Z. Novák, *Dalton Trans.* 2010, **39**, 726.

[7] L. S. Campbell-Verduyn, L. Mirfeizi, R. A. Dierckx, P. A. Elsinga, B. L. Feringa, *Chem. Commun.* 2009, 2139.

[8] S. Özçubukçu, E. Ozkal, C. Jimeno, M. A. Pericàs, *Org*. *Lett*. 2009, **11**, 4680.

[9] K. Lőrincz, P. Kele, Z. Novák, *Synthesis* 2009, 3527.

[10] F. Alonso, Y. Moglie, G. Radivoy, G.; Yus, M. *Eur. J. Org. Chem.* **2010**, 1875.

[11] W. Feng, F. Hua, J. Yuyang, Z. Yufen, *Green Chem.* 2008, **10**, 452.