Copper(I)-catalyzed cycloaddition of silver acetylides and azides: Incorporation of volatile acetylenes into the triazole core

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N-Phenyl-2-(4-phenyl-1H-1,2,3-triazol-1-yl) acetamide (3)

Electronic Supplementary Material (ESI) for Organic and Biomolecular Chemistry
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Std carbon

Sample: ilaria_59
File: /home/5jw/OMD/OMD/OMD/Iliea/59_12CNMR.fid

Pulse Sequence: z2pul
Solvent: DMSO
Temp. 25.0 C / 298.1 K
Operator: z2p
File: ilaria_59_12CNMR

Pulse 90.0 degrees
Acq. time 1.500 sec
Width 301660.7 Hz
T2S_repetition
RESRVRY 10.1: 0.2756588 MHz
DEOUPLE H2: 0.9992690 MHz
Power 52 dB
Continuous on
Data processing
Line broadening 1.0 Hz
FT size 102472
Total time 25 min, 8 sec
1-Benzyl-4-phenyl-1H-1,2,3-triazole (4)
Sample: 1hla1e_52
Sample ID: 1hla1e_52_01
File: /home/mikolajp/t2i-y_data/bio/2701/1hla1e_52_Carbon-Std-51.fld

Pulse Sequence: x3pe7
Solvent: deso
Temp: 295 C / 298.1 K
Operator: t2i-y
File: 1hla1e_52_Carbon-Std-51
INova-550 "ChemSolv"

Pulse 45.6 degrees
Acq. time 1.500 sec
Width 180.888 Hz
128 repetitions
Observations 120, 225.606438 MHz
DECOUPLE H1, 495.898029 MHz
Power 62 dB
Continuously on
MALT-10 modulated
Data shown line broadening 1.0 Hz
FT times 130872
Total time 8 min, 46 sec
1-Benzyl-4-butyl-1H-1,2,3-triazole (5)

**NMR Spectra**

- **Sample**: 1liria_81
- **File**: /home/walkeup2jw-ty_data10/0ct18/1liria_81_Proton-Std-01.fld

**Pulse Sequence**: szpul

- **Solvent**: depeo
- **Temp**: 25.0 °C / 298.1 K
- **Operator**: kp-ty
- **File**: ilaria_81_Proton-Std-01

**Pulse 45.0 degrees**

- **Time**: 4.080 sec
- **Width**: 295.2 Hz
- **NMR-500 CChemStation**

**NMR Data**

- **Observed**: H2, 499.8077404 MHz
- **Data Processing**: FT size 101372
- **Total time 2 min, 10 sec
4-Ethyl-1-benzyl-1H-1,2,3-triazole (6)
1-Benzyl-4-(2-methoxynaphthalen-7-yl)-1H-1,2,3-triazole (7)
Std carbon

Sample: tc-2-138-Cl
Filter: Hg
Pulse Sequence: 61p91p
Solvent: DMSO
Temp: 30.0 °C / 303.1 K
Chemical Shift: 6.0 ppm
Spin System: Molecule
Relax, delay: 2.000 sec
Pulse 60.0 degrees
N1: 1001.0 mHz
N2: 100.0 mHz
529 repetitions
Resolving 305.5502866 MHz
Resolving: 11.3099.7412189 MHz
Power 40 dB
Continuously on
WALTZ-16 modulated
With PRECISION
Sweep width: 10.0 kHz
FT Vials 10
Total time 4 hr, 50 min, 25 sec
1-Benzyl-4-(2-methoxyphenyl)-1H-1,2,3-triazole (8)
2-(4-Butyl-1H-1,2,3-triazol-1-yl)-N-phenyl acetamide (9)

Std proton
Sample: larin_77
file: vp
Pulse Sequence: ecpul
Solvent: dmo
Temp: 25.0 °C / 298.1 K
Determination
INWR-569 "ibi000"
Pulse 45.0 degrees
Mix time 0.008 sec
MININ 4893.2 Hz
32 repetitions
OVERLAP RESC 1.58820442 MHz
DATA PROCESSING
FT echo 123244
Total time 2 min, 8 sec
2-(4-Ethyl-1H-1,2,3-triazol-1-yl)-N-phenylacetamide (10)
4-Phenyl-1-octyl-1H-1,2,3-triazole (11)

Sample: ST-173
Sample ID: ST-173_91
File: /home/michaelj/spj/st-173/dataset/Y2-1820_Symmetry_Std-02.fid

Pulse Sequence: s2pq1
Solvent: d6-py
Temperature: 298.1 K
Operator: h.r.
File: Y2-1820_Symmetry_Std-02
INova-500 "Chemacs"

Pulse 45.0 degrees
Acq. time 4.000 sec
Width 2.000 Hz
Scale 1000 Hz
Resolution 11.62027476 MHz
Data Processing
Fit size 23840
Total time 2 min, 18 sec
pad=10 run with findz0 before acquisition
Sample: BY-173-13C
Sample ID: BY-173-13C_01
File: /home/walker/2D-by_data16/Movi16/BY-173-13C_Carbon-Std-01.fid
Pulse Sequence: eipul
Solvent: deoa
Temp. 25.0 C / 298.1 K
Operator: jwry
File: BY-173-13C_Carbon-Std-01
INova-500 "ChemSSR"
Pulse 45.0 degrees
Acq. Time 1.990 sec
Vl/30 28200.0 Hz
A. 0.1 Hz
OBSERVE CH2 122.448866 Hz
DECOUPLE NO, 499.992434 Hz
Power 42 dB
Continuity on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT 4198 102972
Total time 8 min, 48 sec
4-Ethyl-1-octyl-1H-1,2,3-triazole (12)

pad=10 run with findz2 before acquisition
Sample: SY-181-columned
Sample ID: SY-181-columned_01
File: /home/m1/mypj/sy-sy_detail/Nov18/SY-181-columned_proton-std-01.rdf
Pulse Sequence: z2pol
Solvent: cdc53
Temp: 25.0 °C / 298.1 K
Operator: sjry
File: SY-181-columned_proton-std-01
INNOVA-500 "cheatsheet"
Pulse 45.8 degrees
Acq. time 4.050 sec
WIDTH 7955.2 Hz
32 repetitions
OBSERVE 64, 499.6053764 MHz
DATA PROCESSING
Total time 2 min, 18 sec

pad=10 run with findz2 before acquisition
Sample: SY-181-columned
Sample ID: SY-181-columned_02
File: /home/m1/mypj/sy-sy_detail/Nov18/SY-181-columned_carbon-std-01.rdf
Pulse Sequence: z2pol
Solvent: cdc53
Temp: 25.0 °C / 298.1 K
Operator: sjry
File: SY-181-columned_carbon-std-01
INNOVA-500 "cheatsheet"
Pulse 45.8 degrees
Acq. time 4.050 sec
WIDTH 28428.8 Hz
32 repetitions
OBSERVE 128, 105.5459882 MHz
DECAY 128, 499.5879899 MHz
Power 40 dB
Continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT sided 131,872
Total time 5 min, 48 sec
2-(4-(2-Methoxyphenyl)-1H-1,2,3-triazol-1-yl)-(N-(4-biphenyl))acetamide (13)
4-Ethyl-1-((naphthalen-6-yl)methyl)-1H-1,2,3-triazole (14)

Sample: SY-187-columned
Sample ID: SY-187-columned_61
File: /home/walkup/jw-vy/Details/SY-187-columned_Proton-Std-01.txt

Pulse Sequence: 2pul
Solvent: ccc3
Temp: 298.1 K
Operator: jw-vy
File: SY-441-columned_Proton-Std-01
INova-500 *chems*00*

Pulse 45.0 degrees
Acq. time 4.000 sec
Width 7685.2 Hz
32 repetitions
FID 128 scan
Set 0.85375 Hz
Q 8 data
FT 4 scan 12800
Total time 2 min, 8 sec
1-(4-Methoxybenzyl)-4-ethyl-1H-1,2,3-triazole (15)
1,4-Bis-((4-phenyl-1H-1,2,3-triazol-1-yl)methyl)benzene (16)
Sample: te-2-139-ppt
File: sp
Pulse Sequence: es2pu1
Solvent: DMSO
Temp. 298.1 K / 323.1 K
Operator: h+b
INova-450 "Chester"

Relax. delay 1.500 sec
Pulse 45.0 degrees
Acq. time 1.501 sec
6547 acquisitions
Observ. 513.710553266 MHz
DECOUPLE: H2, 400.1341188 MHz
Power 64 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
All the spectra
Total time 8 hr, 21 min, 37 sec
1,4-Bis-((4-butyl-1H-1,2,3-triazol-1-yl)methyl)benzene (17)
2-(4-Methyl-1H-1,2,3-triazol-1-yl)-N-phenylacetamide (18)

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1-(4-Methoxybenzyl)-4-butyl-1H-1,2,3-triazole (19)

Sample: SY-185-1
Sample ID: SY-185-1_01
File: /home/walshop/5x3/sy_data19/Delig/SY-185-1_Proton-Std-01.fld

Pulse Sequence: zgpol
Solvent: cdcl3
Temperature: 298.1 K
Dimensions: 1024 x 1024
File: SY-185-1_Proton-Std-01
INNOVA-500 "VBRM595"

Pulse 90.8 degrees
Acq. time 4.000 sec
Width 799.2 Hz
22 repetitions
Detective 2H
Pulse spacing 0.016666666666666664
FT size 131072
Total time 2 min, 8 sec
Sample: SY-195-1
Sample ID: SY-195-1.02
File: /home/valtis/ESI-ly_data18/Dec18/SY-195-1_Carbon-Std-01.fid

Pulse Sequence: sump
Solvent: dcmd
Temp: 25.0 °C / 298.1 K

Files: SY-195-1_Carbon-Std-01
INVSQ=500 "chem500"

Pulse 65.0 degrees
Acq. time 1.000 sec
Width 28000.0 Hz
512 repetitions
OESENCE 13.1, 125.6476832 MHz
DECoupled H1, 608.6076893 MHz
Power 42 dB
Phase on
VTLD=amplitude
DATA PROCESSING
Line Broadening 1.0 Hz
FT size 104896
Total time 8 min, 37 sec