Supporting Information for “An Efficient and Regioselective Synthesis of 2-Alkyl-2*H*-indazoles”

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¹H NMR (400 MHz, DMSO-*d*₆) spectra for 1-methyl-6-nitro-1*H*-indazole and 2-methyl-6-nitro-2*H*-indazole are attached. Both spectra matched literature data (see: Boyer, G.; Galy, J. P.; Barbe, J. *Heterocycles* **1995**, *41*, 487). In addition, data from ROESY experiments for 1-methyl-6-nitro-1*H*-indazole and 2-methyl-6-nitro-2*H*-indazole are attached.

Regiochemistry of the 1-methyl-6-nitro-1*H*-indazole was determined by observing a correlation between H-1 and H-7 and a lack of correlation between H-1 and H-3 from the ROESY NMR Spectroscopic experiment.

Regiochemistry of the 2-methyl-6-nitro-2*H*-indazole was determined by observing a correlation between H-1 and H-3 from the ROESY NMR Spectroscopic experiment.

M:Cheung u17337.80-1
STANDARD 1H OBSERVE

1H NMR (400 MHz, DMSO-d6)

FILE = /gw/nmr/6awork/nmr/2001/u17337/u17337.80-1_H1.fid
SOLVENT 1s DMSO
Date - Apr 26 2001
OBSERVE H1
FREQUENCY 299.963 MHz
SPECTRAL WIDTH 4799.9 Hz
ACQUISITION TIME 4.000 sec
RELAXATION DELAY 0.500 sec
PULSE WIDTH 7.0 usec
AMBIENT TEMPERATURE
NO. REPETITIONS 32
DOUBLE PRECISION ACQUISITION
DATA PROCESSING
GAUSSIAN APODIZATION 0.500 sec
FT SIZE 65536
TOTAL ACQUISITION TIME 2 minutes

1-methyl-6-nitro-1H-indazole

PPM	FREQUENCY	H
8.72	2615.1	
8.29	2485.3	
8.01	2403.9	
7.88	2385.1	
7.86	2386.7	
7.85	2384.8	
7.83	2377.9	
7.82	2376.0	
4.18	1255.0	
3.92	994.8	
2.50	749.0	
2.49	747.4	
2.49	745.6

M Cheung u17337.96-2
STANDARD 1H OBSERVE

¹H NMR (400 MHz, DMSO-d₆)

FILE = /gm/nmr/6awork/nmr/2001/u17337/u17337.96-2_H1.fid
SOLVENT is DMSO
Date - May 5 2001
OBSERVE H1
FREQUENCY 399.942 MHz
SPECTRAL WIDTH 5644.5 Hz
ACQUISITION TIME 2.135 sec
RELAXATION DELAY 0.100 sec
PULSE WIDTH 6.0 usec
AMBIENT TEMPERATURE
NO. REPETITIONS 64
DOUBLE PRECISION ACQUISITION
DATA PROCESSING
GAUSSIAN APODIZATION 0.500 sec
F1 SIZE 65536
TOTAL ACQUISITION TIME 2 minutes

2-methyl-6-nitro-2H-indazole

PPM	FREQUENCY	HEIGHT
8.58	3429.8	61.
7.85	3178.3	20.
7.92	3169.2	26.
7.79	3115.9	13.
7.78	3113.9	14.
7.77	3106.6	10.4
7.76	3104.8	11.
4.25	1700.2	164.
3.31	1325.5	31.
2.48	992.4	12.
2.48	990.7	16.
2.47	989.1	12.

M. Cheung u17337.80-1
STANDARD 1H OBSERVE

ROESY ¹H NMR (400 MHz)
DMSO-d₆

H-1

PULSE SEQUENCE roesy
FILE = /gw/nmr/5uwork/nmr/2001/u17337/u17337.80-1_roesy.fid
SOLVENT 1s DMSO
Date - May 7 2001
OBSERVE H1

FREQUENCY 399.941 MHz
SPECTRAL WIDTH 3987.6 Hz
2D SPECTRAL WIDTH 3587.6 Hz
ACQUISITION TIME 0.285 sec
RELAXATION DELAY 1.200 sec
MIXING TIME 0.250 sec
PULSE WIDTH 5.0 usec
FIRST PULSE WIDTH 15.0 usec
TEMPERATURE 25.0 deg. C.
NO. REPEATITIONS 8
NO. INCREMENTS 256 X2
DOUBLE PRECISION ACQUISITION
DATA PROCESSING
SINE BELL SQUARED 0.282 sec
FT SIZE 2048
F1 DATA PROCESSING
SINE BELL SQUARE 0.145 sec
SHIFTED BY -0.143 sec
FT SIZE 2048
TOTAL ACQUISITION TIME 32.5 hours

1-methyl-6-nitro-1H-indazole

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STANDARD 1H OBSERVE

ROESTY ¹H NMR (400 MHz)
DMSO-d₆

H-1

PULSE_SEQUENCE roesy
FILE = /gw/nmr/5uwork/nmr/2001/u17337.96-2_roesy.fid
SOLVENT is DMSO
Date - May 6 2001
OBSERVE H1
FREQUENCY 399.941 MHz
2D SPECTRAL WIDTH 3585.4 Hz
ACQUISITION TIME 0.286 sec
RELAXATION DELAY 1.200 sec
MIXING TIME 0.250 sec
PULSE WIDTH 5.0 usec
FIRST PULSE WIDTH 15.0 usec
TEMPERATURE 25.0 deg. C.
NO. REPETITIONS 8
NO. INCREMENTS 256 X2
DOUBLE PRECISION ACQUISITION
DATA PROCESSING
GAUSSIAN APODIZATION 0.129 sec
FT SIZE 2048
F1 DATA PROCESSING
GAUSSIAN APODIZATION 0.065 sec
FT SIZE 1024
TOTAL ACQUISITION TIME 32.5 hours

2-methyl-6-nitro-2H-indazole