Supporting Information

Highly Efficient Ruthenium-catalyzed Transfer Hydrogenation/Hydrogenation of 1,3- Cycloalkanediones to 1,3-Cycloalkanediols using Controlled Microwave Heating

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S2	General Experimental
S3-S10	Copies of ^1H and ^{13}C NMR spectra

Experimental

General. ^1H (400 or 300 MHz) and ^{13}C NMR (100 or 75 MHz) spectra were recorded in chloroform- d_1 or pyridine- d_5 . Chemical shifts (δ) are reported in ppm, using residual solvent as internal standard (CDCl_3 , ^1H δ 7.26, ^{13}C δ 77.0 or Pyridine- d_5 , ^1H δ 7.22, ^{13}C δ 123.87), and coupling constants (J) are given in hertz. Microwave heating was performed in a Smith Creator (Biotage AB, Uppsala, Sweden). The temperature was measured by an external IR sensor and kept constant during the reactions. Unless otherwise noted, all materials were obtained from commercial suppliers and used without further purification. Toluene was distilled from sodium. Ruthenium catalysts **3-5** and **7-8**ⁱ as well as diones **1c**,ⁱⁱ and **1e**ⁱⁱⁱ were synthesized according to literature procedures.

ⁱ Paetzold, J.; Baeckvall, J. E., *J. Am. Chem. Soc.* **2005**, 127, 17620-17621

ⁱⁱ Jacobson, B. M.; Soteropoulos, P.; Bahadori, S., *J. Org. Chem.* **1988**, 53, 3247-55

ⁱⁱⁱ Ragan, J. A.; Makowski, T. W.; Am Ende, D. J.; Clifford, P. J.; Young, G. R.; Conrad, A. K.; Eisenbeis, S. A., *Org. Process Res. Devel.* **1998**, 2, 379-381.

c1s-2c

Pulse Sequence: s2pul

Solvent: CDCl3

Temp. 25.0 C / 298.1 K

File: km1c126_fr13-24

INOVA-500 "nmr6"

Relax. delay 1.000 sec
Pulse 41.6 degrees
Acq. time 2.733 sec
Width 5955.2 Hz
16 repetitions
OBSERVE H1, 393.9356487 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 65536
Total time 1 min, 2 sec

cis-2c

cis-2c

Pulse Sequence: s2pul

Solvent: CDCl3

Temp. 25.0 C / 298.1 K

File: km1c126_cis_13C

INOVA-500 "nmr6"

Relax. delay 1.000 sec

Pulse 48.9 degrees

Acq. time 1.199 sec

Width 25000.0 Hz

758 repetitions

OBSERVE C13, 100.563885 MHz

DECOUPLE H1, 399.9376154 MHz

Power 42 dB

continuously on

VALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 42 min, 5 sec

cis-2c

trans-2c

Pulse Sequence: s2pul

Solvent: CDCl3

Temp. 25.0 C / 298.1 K

File: km1c126_fr27-41

INOVA-500 "nmr5"

Relax. delay 1.000 sec

Pulse 41.6 degrees

Acq. time 2.733 sec

Width 5955.2 Hz

16 repetitions

OBSERVE H1, 399.9356489 MHz

DATA PROCESSING

Line broadening 0.1 Hz

FT size 65536

Total time 1 min, 2 sec

trans-2c

trans-2c

Pulse Sequence: s2pul

Solvent: CDCl3

Temp. 25.0 C / 298.1 K

Mercury-400BB "nuc4"

Relax. delay 1.000 sec

Pulse 48.9 degrees

Acq. time 1.199 sec

Width 25000.0 Hz

406 repetitions

OBSERVE C13, 100.5638993 MHz

DECOUPLE H1, 399.9376154 MHz

Power 42 dB

continuously on

VALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 42 min, 5 sec

trans-2c

2d

Pulse Sequence: s2pul
Solvent: pyridine
Temp. 25.0 C / 298.1 K
File: 5-me-cyklohexandiol
INOVA-500 "nmr6"

Relax. delay 1.000 sec
Pulse 57.3 degrees
Acq. time 1.995 sec
Width 4506.5 Hz
16 repetitions
OBSERVE H1, 299.9463069 MHz
DATA PROCESSING
Line broadening 0.1 Hz
FT size 32768
Total time 0 min, 49 sec

2d

Pulse Sequence: s2pul
Solvent: pyridine
Temp. 25.0 C / 298.1 K
File: 5-me-cyhexandiolbland_13C
INOVA-500 "nmr6"

Relax. delay 1.000 sec
Pulse 67.0 degrees
Acq. time 1.815 sec
Width 18761.7 Hz
531 repetitions
OBSERVE C13, 75.4214582 MHz
DECOUPLE H1, 299.9475106 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 53 min, 4 sec

2d

cis-2e

Pulse Sequence: s2pu1

Solvent: CDCl3

Temp. 25.0 C / 298.1 K

File: kmld003_b

INOVA-500 "nmr6"

Relax. delay 1.000 sec

Pulse 57.3 degrees

Acq. time 1.995 sec

Width 4506.5 Hz

16 repetitions

OBSERVE H1, 299.9464278 MHz

DATA PROCESSING

Line broadening 0.1 Hz

FT size 32768

Total time 0 min, 49 sec

cis-2e

cis-2e

Pulse Sequence: s2pul

Solvent: CDCl₃

Temp. 25.0 C / 298.1 K

File: kmld003_cis_13C

INOVA-500 "nmr6"

Relax. delay 1.000 sec

Pulse 67.0 degrees

Acq. time 1.815 sec

Width 18761.7 Hz

272 repetitions

OBSERVE C13, 75.4215895 MHz

DECOUPLE H1, 299.9479605 MHz

Power 38 dB

Continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 131072

Total time 53 min, 4 sec

cis-2e