Supporting Information for:

**Nitrate as a Redox Co-Catalyst for the Aerobic Pd-Catalyzed Oxygenation of Unactivated sp³-C–H Bonds**

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1. General Information

NMR spectra were obtained on a Varian Inova 500 (499.90 MHz for $^1$H; 125.70 MHz for $^{13}$C) or a Varian vnmrs 500 (499.90 MHz for $^1$H; 125.70 MHz for $^{13}$C) or a Varian MR400 (400.53 MHz for $^1$H; 100.71 MHz for $^{13}$C; 375.94 MHz for $^{19}$F) spectrometer. $^1$H NMR chemical shifts are reported in parts per million (ppm) relative to TMS, with the residual solvent peak used as an internal reference. The chloroform reference peak is set to 7.23 ppm for $^1$H and 77.23 ppm for $^{13}$C. In the case of benzene, the reference peak is set to 7.16 ppm. Multiplicities are reported as follows: singlet (s), doublet (d), doublet of doublets (dd), doublet of doublets of doublets (ddd), doublet of triplets (dt), triplet (t), quartet (q), quintet (quin), multiplet (m), and broad resonance (br). IR spectra were obtained on a Perkin-Elmer spectrum BX FT-IR spectrometer. Melting points were determined with a Mel-Temp 3.0 (Laboratory Devices Inc) and are uncorrected. HRMS data were obtained on a Micromass AutoSpec Ultima Magnetic Sector mass spectrometer.

2. Materials and Methods

Pd(OAc)$_2$ was obtained from Pressure Chemical and used as received. NaNO$_3$ was obtained from Fisher Scientific and used as received. 4 Å molecular sieves were dried in Precision Economy Oven at 200 ºC for 48 h. Solvents were obtained from Fisher Chemical and used without further purification. Flash chromatography was performed on EM Science silica gel 60 (0.040–0.063 mm particle size, 230–400 mesh) and thin layer chromatography was performed on Merck TLC plates pre-coated with silica gel 60 F254. HPLC purification of 13-15 was performed on a Varian ProStar 210 HPLC using Waters $\mu$-Porasil 10$\mu$m silica (19 x 300 mm) columns.
3. Synthesis and Characterization of Substrates 1, S3-S11

The oxime ether substrates 1, S3-S6 were prepared via oximation of the corresponding ketones according to a procedure previously reported by our group.\(^1\) \(^1\)H and \(^13\)C NMR spectral data for 1, S3, S5, and S6 matched that previously reported in the literature for these compounds.\(^1\)

![Image of chemical structures](image)

The pyridine substrates S7, S8 and S10 were prepared via Cu-catalyzed coupling between 2-bromopyridine derivatives and the corresponding alkyl magnesium chloride (purchased from Aldrich and used as received) using a procedure previously reported by our group.\(^2\) Substrate S9 was synthesized as described below using a procedure from a patent.\(^3\) Substrate S11 was obtained from Sigma-Aldrich and used as received. \(^1\)H and \(^13\)C NMR spectral data for S7-S10 matched that previously reported in the literature for these compounds.

![Image of chemical structures](image)

Substrate S4 was obtained via oximation of the corresponding ketone according to a literature procedure.\(^1\) It was obtained as a colorless oil in 80% isolated yield (R\(_f\) = 0.41 in 1:19 ethyl acetate/hexanes). \(^1\)H NMR (500 MHz, C\(_6\)D\(_6\)): \(\delta\) 3.78 (s, 3H), 2.59 (m, 2H), 1.61-1.65 (multiple peaks, 4H), 1.30 (m, 2H), 1.10 (s, 6H). \(^13\)C\({}^{1\text{H}}\) NMR (125 MHz, CDCl\(_3\)): \(\delta\) 165.08, 67.50, 60.98, 41.36, 37.24, 26.77, 26.20, 21.73, 21.23. HRMS EI (m/z): [M]+ calcd for C\(_9\)H\(_{17}\)NO predicted: 155.1310, measured: 155.1315. IR (thin film) 1625, 1050 cm\(^{-1}\).
4. Apparatus Set-Up and Optimization Table

General procedure for screening (Table 1 in manuscript)

Pd(OAc)$_2$ (2.2 mg, 0.0025 mmol, 0.05 equiv) and the appropriate redox co-catalyst were weighed into a 25 mL Schlenk flask. The solids were dissolved in Ac$_2$O (0.2 mL). Substrate 1 (36 mg, 0.2 mmol, 1 equiv) was added to the Schlenk flask as a solution in AcOH (1.4 mL). The top of the Schlenk flask was sealed with a rubber septum. The flask was charged with O$_2$ through the side arm using the setup shown in Figure S1. The flask was evacuated (aspirator vacuum) and backfilled with oxygen (1 atm) three times through the side arm to achieve 1 atm of O$_2$. The flask was sealed at the side arm with a glass stopcock and then heated at the desired temperature in an oil bath for 18 h. The reaction was then cooled to room temperature, and 1 M nitrobenzene in CDCl$_3$ (50 μL, 0.5 mmol, 0.25 equiv) was added as a standard. The crude reaction mixtures were analyzed by $^1$H NMR spectroscopy using nitrobenzene as an internal standard.

Figure S1. Schematic setup for filling reaction vessels with 1 atm of O$_2$.

The air-free reaction (Table 1, entry 6) was set up using the same setup as shown in Figure S1 but with N$_2$ instead of O$_2$.

The reactions under an air atmosphere (Table 1, entries 1-5) were set up in air and sealed directly.
5. Synthesis and Characterization of Products 2-12

Standard Procedure

Pd(OAc)$_2$ (5.6 mg, 0.025 mmol, 0.05 equiv) and NaNO$_3$ (10.6 mg, 0.125 mmol, 0.25 equiv) were weighed into a 50 mL resealable Schlenk flask. Substrate (0.5 mmol, 1 equiv) was added to the Schlenk flask as a solution in AcOH/Ac$_2$O (AcOH : Ac$_2$O = 7 : 1; overall concentration = 0.125 M in substrate). The Schlenk flask was charged with 1 atm of O$_2$ using the method described above (see Figure S1). The flask was heated at 110 °C in an oil bath for 18 h (unless otherwise noted). The reaction was cooled to rt, filtered through a plug of Celite, and then concentrated under reduced pressure to remove the solvent. The resulting residue was neutralized with a saturated solution of NaHCO$_3$, and the organic products were extracted into either diethyl ether or ethyl acetate. The organic extracts were washed with brine, combined, and then dried over MgSO$_4$. The products were purified by column chromatography on silica gel.

This reaction was conducted with substrate 1 according to the standard procedure. Product 2 was obtained as a pale yellow oil (95 mg, 80% yield, R$_f$ = 0.25 in 1:9 ethyl acetate/hexanes). Spectral data [$^1$H NMR (CDCl$_3$) and $^{13}$C($^1$H) NMR (CDCl$_3$)] matched that reported in the literature for this compound.$^1$

This reaction was conducted with substrate S3 according to the standard procedure. Product 3 was obtained as a colorless oil (126 mg, 83% yield, R$_f$ = 0.25 in 1:9 ethyl acetate/hexanes). Spectral data [$^1$H NMR (CDCl$_3$) and $^{13}$C($^1$H) NMR (CDCl$_3$)] matched that reported in the literature for this compound.$^1$

This reaction was conducted with substrate S4 according to the standard procedure, except using 50 mol % of NaNO$_3$ (0.25 mmol, 0.5 equiv) at 100 °C. Product 4 was obtained as a pale yellow oil (81 mg, 60% yield, R$_f$ = 0.25 in 1:4 ethyl acetate/hexanes). $^1$H NMR (500 MHz, C$_6$D$_6$): $\delta$ 4.64 (d, $J = 11.0$ Hz, 2H), 4.19 (d, $J = 11.0$ Hz, 2H), 3.70 (s, 3H), 2.51 (t, $J = 6.0$ Hz, 2H), 1.65 (two peaks, s, 6H), 1.42 (t, $J = 6.5$ Hz, 2H), 1.20 (m, 2H), 1.13 (m, 2H). $^{13}$C($^1$H) NMR (125 MHz, C$_6$D$_6$): $\delta$ 169.97, 157.98, 64.90, 61.37, 57.24, 55.03, 38.16, 33.08, 33.05, 31.80, 31.78, 28.08, 27.65, 27.39, 26.55, 25.22, 21.75.
44.16, 32.37, 25.32, 21.66, 21.06, 20.32. HRMS electrospray (m/z): [M+H]⁺ calcd for C_{13}H_{22}NO₅ predicted: 272.1492, measured: 272.1491. IR (thin film) 1739 cm⁻¹.

This reaction was conducted with substrate S₅ according to the standard procedure, except using 20 mol % of Pd(OAc)₂ (0.1 mmol, 0.2 equiv) at 100 °C. Product 5 was obtained as a pale yellow oil (51 mg, 51% yield, Rᵢ = 0.25 in 3:7 diethyl ether/pentanes). Spectral data ['¹H NMR (CDCl₃) and '³C{¹H} NMR (CDCl₃)] matched that reported in the literature for this compound.¹

This reaction was conducted with substrate S₆ according to the standard procedure, except using 1 equiv of NaNO₃ (0.5 mmol, 1 equiv) under air. Product 6 was obtained as a yellow oil in (49 mg, 41% yield, Rᵢ = 0.25 in 1:4 ethyl acetate/hexanes). Spectral data ['¹H NMR (CDCl₃) and '³C{¹H} NMR (CDCl₃)] matched that reported in the literature for this compound.¹

This reaction was conducted with substrate S₇ according to the standard procedure, except that 4 Å molecular sieves (270 mg) were added. Product 7 was obtained as a colorless oil (122 mg, 79% yield, Rᵢ = 0.2 in 3:7 ethyl acetate/hexanes). Spectral data ['¹H NMR (CDCl₃) and '³C{¹H} NMR (CDCl₃)] that reported in the literature for this compound.¹

This reaction was conducted with substrate S₈ according to the standard procedure, except using 50 mol % of NaNO₃ (0.25 mmol, 0.5 equiv) along with 4 Å molecular sieves (270 mg). Product 8 was obtained as a colorless oil in (147 mg, 78% yield, Rᵢ = 0.2 in 1:9 ethyl acetate/hexanes). '¹H NMR (400 MHz, CDCl₃): δ 8.74 (d, J = 4.8 Hz, 1H), 7.44 (s, 1H), 7.41 (d, J = 4.8 Hz, 1H), 4.52 (s, 6H), 1.96 (s, 9H). '³C{¹H} (125 MHz) NMR
This reaction was conducted with substrate S9 according to the standard procedure, except using 50 mol % of NaNO₃ (0.25 mmol, 0.5 equiv) along with 4 Å molecular sieves (270 mg). Product 9 was obtained as a colorless oil (149 mg, 77% yield, Rₐ = 0.2 in 3:7 ethyl acetate/hexanes). ¹H NMR (400 MHz, CDCl₃): δ 8.41 (d, J = 5.2 Hz, 1H), 7.45 (d, J = 2.0 Hz, 1H), 7.39 (dd, J = 5.2, 2.0 Hz, 1H), 4.52 (s, 6H), 2.02 (s, 9H). ¹³C{¹H} NMR (100 MHz, CDCl₃): δ 170.53, 159.75, 150.04, 133.12, 125.81, 125.09, 64.00, 48.01, 20.77. HRMS electrospray (m/z): [M+H]+ calcd for C₁₅H₁₈F₃NO₆ predicted: 388.0390, measured: 388.0396. IR (thin film) 1739 cm⁻¹.

This reaction was conducted with substrate S10 according to the standard procedure, except using 1 equiv of NaNO₃ (0.5 mmol, 1 equiv) along with 4 Å molecular sieves (270 mg). Product 10 was obtained as a colorless oil (110 mg, 68% yield, Rₐ = 0.2 in 3:7 ethyl acetate/hexanes). ¹H NMR (400 MHz, CDCl₃): δ 7.50 (app t, J = 7.6 Hz, 1H), 7.01 (d, J = 7.6 Hz, 1H), 6.99 (d, J = 7.6 Hz, 1H), 4.51 (s, 6H), 2.47 (s, 3H), 2.47 (s, 9H). ¹³C{¹H} NMR (100 MHz, CDCl₃): δ 170.88, 158.33, 157.39, 136.50, 121.92, 118.39, 64.79, 47.92, 24.73, 21.00. HRMS electrospray (m/z): [M+H]+ calcd for C₁₆H₂₂NO₆ predicted: 324.1442, measured: 324.1446. IR (thin film) 1734 cm⁻¹.

This reaction was conducted with substrate S11 according to the standard procedure, except using 1 equiv of NaNO₃ (0.5 mmol, 1 equiv) along with air as the oxidant. Product 11 was obtained as a colorless oil (80 mg, 80% yield, Rₐ = 0.25 in 1:9 ethyl acetate/hexanes). Spectral data [¹H NMR (CDCl₃) and ¹³C{¹H} NMR (CDCl₃)] matched that reported in the literature for this compound.⁴
This reaction was conducted with substrate S7 according to the standard procedure, except that 4 Å molecular sieves (270 mg) were added and EtCO$_2$H/(EtCO)$_2$O was used as the solvent. Product 12 was obtained as a colorless oil (141 mg, 80% yield, $R_f = 0.32$ in 3:7 ethyl acetate/hexanes). $^1$H NMR (500 MHz, CDCl$_3$): $\delta$ 8.58 (m, 1H), 7.66 (m, 1H), 7.27 (m, 1H), 7.19 (m, 1H), 4.55 (d, $J = 2.0$ Hz, 6H), 2.26 (dq, $J = 2.0$, 8.0 Hz, 6H), 1.05 (dt, $J = 2.0$, 8.0 Hz, 9H). $^{13}$C($^1$H) NMR (125 MHz, CDCl$_3$): $\delta$ 173.93, 158.21, 149.25, 136.14, 122.22, 121.42, 64.18, 48.13, 27.44, 9.00. HRMS electrospray (m/z): [M+H]$^+$ calcd for C$_{18}$H$_{26}$NO$_6$ predicted: 352.1755, measured: 352.1755. IR (thin film) 1737 cm$^{-1}$. 

Electronic Supplementary Material (ESI) for Chemical Science
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Under air, Pd(OAc)$_2$ (5.6 mg, 0.025 mmol, 0.05 equiv), NaCl (14.6 mg, 0.25 mmol, 10 equiv) and NaNO$_3$ (2.1 mg, 0.025 mmol, 0.10 equiv) were weighed into a 20 mL scintillation vial. Substrate S7 (68 mg, 0.5 mmol, 1 equiv) was added as a 0.125 M solution in AcOH. The vial was sealed with a Teflon-lined cap then heated at 80 °C for 24 h. The reaction was cooled to room temperature, filtered through a plug of Celite, and then concentrated under reduced pressure to remove the solvent. The resulting residue was neutralized with a saturated solution of NaHCO$_3$, and the organic products were extracted into ether. NMR analysis of the crude reaction mixture showed 80% conversion of the starting material, and 67% total yield of the mono-, di-, and tri-chlorinated products. The remaining 13% of the material was a mixture of the mono-, di-, and triacetoxylated products. The organic extracts were washed with brine, combined, and then dried over MgSO$_4$. A clean mixture of the three chlorinated products 13-15 was obtained by silica gel column chromatography (all three chlorinated products elute together with $R_f = 0.20$ in 1:9 ethyl acetate/hexanes). The yields reported below are based on the proportion of each product in the material isolated by column chromatography. Pure samples of 13-15 for HRMS and NMR analysis were obtained after by purification by HPLC (97:3% hexanes/ethyl acetate, 20 mL/min, Waters µ-porasil 19.1 mm). Using this solvent system, the products elute in the following order: 15 then 14 then 13.

Product 13 was obtained as a colorless oil (11% yield). Peak #3 from HPLC purification. $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 8.56 (dd, $J = 4.6, 1.4$ Hz, 1H), 7.62 (app td, $J = 7.6, 1.4$ Hz, 1H), 7.30 (d, $J = 8.0$ Hz, 1H), 7.11 (dd, $J = 7.6, 4.6$ Hz, 1H), 3.84 (s, 2H), 1.43 (s, 6H). $^{13}$C($^1$H) NMR (100 MHz, CDCl$_3$): $\delta$ 164.94, 149.11, 136.55, 121.62, 120.47, 55.61, 42.67, 25.97. HRMS electrospray (m/z): [M+H]$^+$ calcd for C$_9$H$_{13}$ClN predicted: 170.0731, measured: 170.0730.

Product 14 was obtained as a colorless oil (45% yield). Peak #2 from HPLC purification. $^1$H NMR (500 MHz, CDCl$_3$): $\delta$ 8.55 (td, $J = 3.6, 1.2$ Hz, 1H), 7.64 (app td, $J = 6.0, 1.2$ Hz, 1H), 7.28 (d, $J = 6.4$ Hz, 1H), 7.14 (dd, $J = 6.0, 3.6$ Hz, 1H), 3.96 (app q, $J = 8.9$ Hz, 4H), 1.51 (s, 3H). $^{13}$C($^1$H) NMR (125 MHz, CDCl$_3$): $\delta$ 161.36, 149.37, 136.64, 122.32, 121.29, 51.25, 47.42, 21.67. HRMS electrospray (m/z): [M+H]$^+$ calcd for C$_9$H$_{12}$Cl$_2$N predicted: 204.0341, measured: 204.0341.
Product 15 was obtained as a colorless oil (12% yield). Peak #1 from HPLC purification. 

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 8.60 (dd, $J = 4.4, 0.8$ Hz, 1H), 7.64 (m, 1H), 7.28-7.21 (multiple peaks, 2H), 4.18 (s, 6H). 

$^{13}$C($^1$H) NMR (100 MHz, CDCl$_3$): $\delta$ 157.44, 149.72, 136.48, 122.92, 122.44, 52.15, 47.01. HRMS electrospray (m/z): [M+H]$^+$ calcd for C$_9$H$_7$Cl$_3$N predicted: 237.9952, measured: 237.9952.
7. Reaction in the Presence of BHT

Pd(OAc)$_2$ (2.2 mg, 0.01 mmol, 0.05 equiv), NaNO$_3$ (4.3 mg, 0.05 mmol, 0.25 equiv), and BHT (11 mg, 0.05 mmol, 0.25 equiv) were weighed into a 25 mL Schlenk flask (14/20 top opening with glass side arm that can be sealed with a glass stopcock) containing a stir bar. Ac$_2$O (0.2 mL) was added. A solution of substrate 1 (36 mg, 0.2 mmol, 1 equiv) in AcOH (1.4 mL) was then added to the Schlenk flask. The top of the Schlenk flask was sealed with a rubber septum. The flask was charged with O$_2$ through the side arm using the set-up shown in Figure S1. The flask was evacuated (aspirator vacuum) and backfilled with oxygen (1 atm) three times to achieve 1 atm of O$_2$. The flask was sealed at the side arm with a glass stopcock and then heated at 110 °C in an oil bath for 30 min. The reaction was cooled to room temperature, and 1 M nitrobenzene in CDCl$_3$ (50 μL, 0.5 mmol, 0.25 equiv) was added as a standard. The crude reaction mixture was analyzed by $^1$H NMR spectroscopy. The yield of 2,6-di-tert-butyl-4-methyl-4-nitrosocyclohexa-2,5-dienone$^5$ in the crude reaction mixture was determined by integration of a diagnostic singlet associated with this product (at 6.6 ppm) versus the internal standard.
8. Reaction with $^{16}\text{O}_2$ vs $^{18}\text{O}_2$

Pd(OAc)$_2$ (2.2 mg, 0.001 mmol, 0.05 equiv) and NaNO$_3$ (4.3 mg, 0.05 mmol, 0.25 equiv) were weighed into a 25 mL resealable Schlenk flask. A solution of substrate 1 (36 mg, 0.2 mmol, 1 equiv) in AcOH (1.4 mL) was then added to the Schlenk flask. The top of the Schlenk flask was sealed with a rubber septum. The Schlenk flask was evacuated and refilled with N$_2$ twice using the set-up shown in Figure S1. The flask was then evacuated a third time and sealed at the side arm by a glass stopcock. A balloon filled with either O$_2$ or $^{18}\text{O}_2$ (99% Aldrich) was inserted through the rubber septum using a needle in order to allow 1 atm of dioxygen to enter the reaction vessel. The flask was then heated at 110 °C in an oil bath for 18 h. The reaction was cooled to rt, diluted with ethyl acetate, and neutralized with a saturated solution of NaHCO$_3$. The organic layer was washed saturated NaHCO$_3$ (X 2) and brine then dried over MgSO$_4$. The products were purified by column chromatography on silica gel (1:9 ethyl acetate/hexanes). The isolated products were submitted for HRMS and analyzed by ESI.
HRMS data for the molecular ion region: reaction with $^{16}$O$_2$

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HRMS data for the molecular ion region: reaction with $^{18}$O$_2$

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Using the published deconvolution mass spreadsheet it was determined that 5% of product in the reaction with $^{18}$O$_2$ contained a $^{18}$O in product 2.
9. References

3. NOVARTIS, AG; Caravatti, G.; Fairhurst, R. A.; Furet, P; Guagano, V.; Imbach, P. WO2010/29082; (2010); (A1) English.
Sample Name:

Data Collected on:

Archive directory:

Sample directory:

Fidfile: 11kja139-C

Pulse Sequence: CARRR (82ps1)

Solvent: cdcl3

Data collected on: Jan 30 2012

Operator: kmowers

Relax. delay 0.100 sec

Pulse 45.0 degrees

Acq. time 2.086 sec

Width 31421.8 Hz

480 repetitions

OBSERVE C13, 125.710246 MHz

DECOUPLE 81, 499.9007536 MHz

Power 32 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 131072

Total time 37 min
1H NMR spectrum of compound 4.

Sample Name: 1H3586-H

Data Collected on: vin.chen.lsa.umich.edu-17Nov4300
Archive directory: Sample directory:
Fidfile: 1H3586-H

Pulse Sequence: DRX103 (a2ps1)
Solvent: c6d6
Data collected on: Nov 8 2011

Operator: kmcowers

Relax. delay 0.500 sec
Pulse 44.9 degrees
Acq. time 3.500 sec
Width 7998.4 Hz
15 repetitions

OBSERVE 499.9042987 MHz
DATA PROCESSING
Line broadening 0.3 Hz
PT n/a 6500x
Total time 1 min 12 sec
11k386-C

Sample Name:

Data Collected on:

Site: chem.lsa.umich.edu

Archive directory:

Sample directory:

Fidfile: 11k386-C

Pulse Sequence: CARBon (s2pm)

Solvent: c6d6

Data collected on: Nov 8 2011

Operator: kmowers

Relax. delay 0.100 sec

Pulse 45.0 degrees

Acq. time 2.086 sec

Width 3142.1 Hz

720 repetitions

OBSERVE C13, 125.703559 Hz

DECOUPLE H1, 499.9007982 MHz

Power 32 dB

continuously on

WALZ-14 modulated

DATA PROCESSING

Lins broadening 1.0 Hz

FT size 131072

Total time 25 min

S18
Electronic Supplementary Material (ESI) for Chemical Science

Sample Name:

Data Collected on:

Acquisition: wavenumbers
Solvent: c6d6
Data collected on: Oct 11 2011

Operator: kmowers

Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 3.500 sec
Width 6410.3 Hz
16 repetitions
OBSEIVE E1, 399.5337144 MHz
DATA PROCESSING
Linewidth 0.3 Hz
FT size 4096x4096
Total time 1 min 12 sec

S19
Sample Name: N

Data Collected on: 12/02/2011
Archive directory:
Sample directory:
Fidfile: 11kjs3129N
Pulse Sequence: 2D
Solvent: cdc13
Data collected on: Dec 6 2011

Operator: kmowers

Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 3.500 sec
Width 6410.3 Hz
16 repetitions

Observe 51.599.537144 MHz

Data Processing
Line broadening 0.3 Hz
PT value 0.000
Total time 1 min 12 sec
11kys140-C

Sample Name:  

Data Collected on:  

Archive directory:  

Sample directory:  

Fidfile: 11kys140-C  

Pulse Sequence: CARRINGTON 62pm1  

Solvent: d6-CDCl3  

Data collected on: Jan 30 2012  

Operator: kmowers  

Relax. delay 0.100 sec  

Pulse 45.0 degrees  

Acq. time 2.569 sec  

Width 25510.2 Hz  

368 repetitions  

OBSERVE 13, 100.4603638 MHz  

DECouple H1, 399.5728256 MHz  

Power 42 dB  

Continuously on  

WALCE-14 modulated  

DATA PROCESSING  

Lines broadening 1.0 Hz  

FT size 131072  

Total time 15 min
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10kysls2-N
Sample Name:

Data Collected on:

archive.chemistry.harvard.edu

Archive directory:

Sample directory:

Fidfile: 10kysls2-N

Pulse Sequence: PROTON (a2pml)
Solvent: dddd
Data collected on: Aug 22 2011

Operator: kmowers

Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 3.500 sec
Width 6410.3 Hz

16 repetitions

OBSERVE 8L 599.5397144 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT Alias 44.999 Hz
Total time 1 min 12 sec

S24
Sample Name:

Data Collected on:

gs.chem.ias.edu-vnmrse600

Sample directory:

Fidfile: 10k5s182-C

Pulse Sequence: CARBON (e2psie)
Solvent: cdcl3
Data collected on: Aug 22 2011

Operator: kmowers

Relax. delay 0.100 sec
Pulse 90.0 degrees
Acq. time 2.569 sec

Width 25510.2 Hz

1424 repetitions

OBSEIVE C11, 100.6283248 MHz
DECOSPE H1, 399.5371321 MHz
Power 39 dB
continuously on
WALCE-16 modulated

DATA PROCESSING

Lines broadening 1.0 Hz
FT size 131072
Total time 1 hr, 3 min
AKS-102-f15-17
Sample Name:

Data Collected on:
Te-vmax300
Archive directory:

Sample directory:

Fid file: AKS-102-f15-17-hv-C
Pulse Sequence: CARRON (+2pm)
Solvent: odd13
Data collected on: Apr 17 2012

Operator: akubota

Relax. delay 0.100 sec
Pulse 40.0 degrees
Acq. time 1.049 sec
Width 31250.0 Hz
1600 repetitions
OBSERVE C13, 125.705288 MHz
DECOUPLE 31, 500.0986704 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
List broadening 1.0 Hz
FT size 65536
Total time 30 min
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S28

Sample Name:

Data Collected on:

Archive directory:

Sample directory:

Fidfile: 11k5377-la

Pulse Sequence: SQXN (e2ps1)
Solvent: ddd13
Data collected on: Nov 5 2011

Operator: kmowers

Relax. delay 0.500 sec
Pulse arg. 0 degrees
Acq. time 3.500 sec
Width 6410.3 Hz
16 repetitions
OBSERVE SS 399.5337144 MHz
DATA PROCESSING
Line broadening 0.3 Hz
PT size 8888
Total time 1 min 12 sec
13

Sample Name:

Data Collected on:
- wk_chem.lda.unc.edu-vm00x00
- Archive directory:
- Sample directory:
- Fidfile: 11k577-1C

Pulse Sequence: CARBON (x2pm)
Solvent: cdcl3
Data collected on: Nov 5 2011

Operator: kmowers

Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 2.569 sec
Width 25510.2 Hz
1151 repetitions

OBSERVE Cl3, 100.462334 MHz
DECOUPLE H1, 399.535712 MHz
Power 42 dB
continuously on
WALTZ-16 modulated

DATA PROCESSING
Linb broadening 1.0 Hz
FT size 131072
Total time 51 min

ppm
160 140 120 100 80 60 40 20

S29
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![Chemical Structure](image)

**Sample Name:**

**Data Collected on:**
Su.chem.lsa.umich.edu-Ionova300

**Archive directory:**
Sample directory:

**Fidfile:** 11k340-2C

**Pulse Sequence:** CARRON (a3pm1)
**Solvent:** cdcl3
**Data collected on:** Oct 7 2011

**Operator:** kmowers

- **Relax. delay:** 0.100 sec
- **Pulse angle:** 45.0 degrees
- **Acq. time:** 2.086 sec
- **Width:** 31421.8 Hz
- **832 repetitions**
- **Observe:** C15, 125.7010246 MHz
- **Decoupled:** H1, 499.9087536 MHz
- **Power:** 32 dB
- **Continuous on**
- **WALTZ-16 modulated**

**DATA PROCESSING**
- **Lines broadening:** 1.0 Hz
- **FT size:** 131072
- **Total time:** 30 min
11ks577-3a

Sample Name:

Data Collected on:
- 01-chem.LSA.edu-vnmr-600
- 01-chem.LSA.edu-vnmr-600

Archive directory:

Sample directory:

Fidfile: 11ks577-3a

Pulses Sequence: PROTON (a2pu)
Solvent: cdcl3
Data collected on: Nov 5 2011

Operator: kmowers

 Relax delay 0.500 sec
 Pulse 45.0 degrees
 Acq. time 3.500 sec
 Width 6410.3 Hz
 16 repetitions
 OBSERVE 51, 399.5337144 MHz
 DATA PROCESSING
 Line broadening 0.3 Hz
 FT axis 66.8
 Total time 1 min 12 sec
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Sample Name: 11k3s77-3C

Data Collected on:
- 11k3s77-3C
- 11k3s77-3C

Pulse Sequence: CARNON (e2pul)
Solvent: cdd3
Data collected on: Nov 5 2011

Operator: khmowers

Relax. delay 0.100 sec
Pulse 45.0 degrees
Acq. time 2.569 sec
Width 25510.2 Hz
560 repetitions

OBSERVE 13C, 100.628324 MHz
DECOUPLE 1H, 399.559731 MHz
Power 2 dB
continuously on
WALTZ-16 modulated

DATA PROCESSING

Lins broadening 1.0 Hz
FT size 131072
Total time 24 min