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Supporting Information

Ph₃P/ICH₂CH₂I-promoted reductive deoxygenation of alcohols

Wei-Ying Tang,^{a,e,#} Xing Zheng,^{a,b,#} Xu Yao,^a Jin-Hong Lin,^{*c,e} Qu-Tong Zheng,^{*d} and Ji-Chang Xiao^{*e}

^aInstitute of Pharmacy and Pharmacology, Hengyang Medicinal School, University of South China, Hengyang, Hunan, 421001, China.

^bDepartment of Pharmacy, Hunan Vocational College of Science and Technology, Third Zhongyi Shan Road, Changsha, Hunan, 410004, China.

^cDepartment of Chemistry, Innovative Drug Research Center, Shanghai University, 200444 Shanghai, China.

^dHunan University of Chinese Medicine, School of Pharmacy, Changsha, Hunan 410208, China.

^eKey Laboratory of Organofluorine Chemistry, Shanghai Institute of Organic Chemistry, University of Chinese Academy of Sciences, Chinese Academy of Sciences, Shanghai, 200032, China.

[#]These two authors contributed equally to this article.

Corresponding authors: jlin@sioc.ac.cn, jlin@shu.edu.cn (J.-H. Lin), <u>812756794@qq.com</u> (Q. -T. Zheng), jchxiao@sioc.ac.cn (J.-C. Xiao).

Contents

1.	General Information	2
2.	General Procedure for the reductive deoxygenation of alcohols	2
3.	¹ H, ¹⁹ F and ¹³ C NMR Spectral data of all the compounds	2
4.	Copies of ¹ H NMR, ¹⁹ F NMR and ¹³ C NMR	10
5.	Refenences	38

1. General Information

The ¹H, ¹³C and ¹⁹F NMR spectra were recorded on 400MHz NMR spectrometers (400 MHz for ¹H, 101MHz for ¹³C and 375 MHz for ¹⁹F respectively). Coupling constants (*J*) are reported in Hz. The following abbreviations were used to explain the multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, sept = septet. All reactions were monitored by TLC or ¹H NMR. Low-resolution mass spectrum (MS) was obtained on GC-MS (EI) or LC-MS (ESI), and high-resolution mass spectrometry (HRMS) data were measured on a Waters Premier GC-TOF MS instrument with an electron impact (EI) ionization mode, or on a Thermo Scientific Q Exactive HF Orbitrap-FTMS instrument with electrospray ionization (ESI) mode. Flash column chromatography was carried out using 300–400 mesh silica gel at medium pressure. Melting points were measured on a melting point apparatus. Unless otherwise noted, all reagents and solvents were obtained commercially and used without further purification. All reactions were performed in 25 mL sealed tube. All starting materials are commercially available, and were purchased and directly used without further purification.

2. General Procedure for the reductive deoxygenation of alcohols

Into a solution of alcohol **1** (0.5 mmol, 1.0 equiv) and Ph_3P (0.6 mmol, 157.4 mg, 1.2 equiv) in DMF (5 mL) in a 25 mL sealed tube was added ICH₂CH₂I (0.6 mmol, 169.1 mg, 1.2 equiv) under a N₂ atmosphere. After the reagents were completely dissolved, NaBH₄ (1.25 mmol, 2.5 equiv) was added. The tube was sealed and the resulting mixture was stirred at room temperature for 2 hours. Dichloromethane was added and the resulting solution was washed with water. The organic layer was removed by concentration under reduced pressure and the residue was subjected to flash column chromatography to give products.

3. ¹H, ¹⁹F and ¹³C NMR Spectral data of all the compounds





2a

Eluent: petroleum ether, white solid, 78 mg, 93% yield. ¹H NMR (400 MHz, Chloroform-d) δ 7.62 (d, *J* = 7.3 Hz, 2H), 7.54 (d, *J* = 8.0 Hz, 2H), 7.47 (t, *J* = 7.4 Hz, 2H), 7.36 (t, *J* = 7.3 Hz, 1H), 7.29 (d, *J* = 7.9 Hz, 2H), 2.44 (s, 3H). ¹³C NMR (101 MHz, Chloroform-d) δ 144.2, 138.4, 137.0, 129.5, 128.8, 127.03, 127.01, 21.2. Spectroscopic data are in agreement with those previously reported.¹

1,3-dichloro-2-methylbenzene (2b)



Eluent: petroleum ether, colorless transparent oil, 68 mg, 84% yield. ¹H NMR (400 MHz, Chloroform-d) δ 7.25 (d, J = 8.0 Hz, 2H), 7.03 (t, J = 8.0 Hz, 1H), 2.46 (s, 3H). ¹³C NMR (101 MHz, Chloroform-d) δ 135.5, 134.4, 127.7, 127.1, 17.4. Spectroscopic data are in agreement with those previously reported.²

1,3-dibromo-2-methylbenzene (2c)



Eluent: petroleum ether, colorless transparent oil, 100 mg, 80% yield. ¹H NMR (400 MHz, Chloroform-d) δ 7.48 (d, J = 8.0 Hz, 2H), 6.87 (t, J = 8.0 Hz, 1H), 2.56 (s, 3H). ¹³C NMR (101 MHz, Chloroform-d) δ 137.5, 131.8, 128.1, 125.4, 23.7. Spectroscopic data are in agreement with those previously reported.³

1-ethyl-4-iodobenzene (2d)



2d

Eluent: petroleum ether, white solid, 69 mg, 63% yield. ¹H NMR (400 MHz, Chloroform-d) δ 7.58 (d, *J* = 8.1 Hz, 2H), 6.94 (d, *J* = 7.9 Hz, 1H), 2.31 (s, 3H). ¹³C NMR (101 MHz, Chloroform-d) δ 137.4, 137.2, 131.2, 90.2 21.0. Spectroscopic data are in agreement with those previously reported.⁴

2-fluoro-5-methylbenzonitrile (2e)



Eluent: petroleum ether/EtOAc = 10/1, white solid, 51 mg, 75% yield. ¹H NMR (400 MHz, Chloroform-d) δ 7.40-7.36 (m, 2H), 7.09 (t, *J* = 8.6 Hz, 1H), 2.36 (s, 3H). ¹⁹F NMR (376 MHz, Chloroform-d) δ -111.78 – -111.83 (m, 1F). ¹³C NMR (101 MHz, Chloroform-d) δ 161.4 (d, *J* = 256.3 Hz), 135.5 (d, *J* = 7.8 Hz), 134.7 (d, *J* = 3.7 Hz), 133.4, 116.1 (d, *J* = 19.3 Hz), 114.1, 101.0 (d, *J* = 15.5 Hz), 20.4. Spectroscopic data are in agreement with those previously reported.⁵

methyl 4-methylbenzoate (2f)



Eluent: petroleum ether/EtOAc = 20/1, Yellow oil, 54 mg, 72% yield. ¹H NMR (400 MHz, Chloroform-d) δ 7.92 (d, *J* = 8.1 Hz, 2H), 7.21 (d, *J* = 8.0 Hz, 2H), 3.87 (s, 3H), 2.38 (s, 3H). ¹³C NMR (101 MHz, Chloroform-d) δ 167.1, 143.5, 129.6, 129.0, 127.4, 51.9, 21.6. Spectroscopic data are in agreement with those previously reported.⁶

methyl 3-methyl-5-nitrobenzoate (2g)



Eluent: petroleum ether/EtOAc = 10/1, white solid, 78 mg, 80% yield. Melting point: 77.7-78.6 °C. ¹H NMR (400 MHz, Chloroform-d) δ 8.63 (s, 1H), 8.20 (s, 1H), 8.16 (s, 1H), 3.96 (s, 3H), 2.51 (s, 3H). ¹³C NMR (101 MHz, Chloroform-d) δ 165.2, 148.3, 140.4, 135.9, 131.6, 127.8, 121.8, 52.7, 21.2. HRMS (FI) *m/z* [M]⁺ calcd for C₁₆H₁₆O 195.0532, found 195.0523. IR (KBr) (cm⁻¹) 3094, 3075, 2956, 2852, 1728, 1648, 1590, 1536, 1488, 1357, 1298, 1223, 913, 887, 738, 664.

1-methyl-4-(methylsulfonyl)-benzene (2h)



Eluent: petroleum ether/EtOAc = 3/1, white solid, 81 mg, 95% yield. ¹H NMR (400 MHz, Chloroform-d) δ 7.81 (d, J = 8.1 Hz, 2H), 7.35 (d, J = 7.9 Hz, 2H), 3.02 (s, 3H), 2.44 (s, 3H). ¹³C NMR (101 MHz, Chloroform-d) δ 144.7, 137.7, 130.0, 127.4, 44.6, 21.6. Spectroscopic data are in agreement with those previously reported.⁷

N-(p-tolyl)acetamide (2i)



4

Eluent: petroleum ether/EtOAc = 1/1, white solid, 69 mg, 92% yield. ¹H NMR (400 MHz, Chloroform-d) δ 7.80 (s, 1H), 7.37 (d, *J* = 8.3 Hz, 2H), 7.09 (d, *J* = 8.1 Hz, 2H), 2.30 (s, 3H), 2.13 (s, 3H). ¹³C NMR (101 MHz, Chloroform-d) δ 168.7, 135.4, 133.9, 129.4, 120.2, 24.4, 20.9. Spectroscopic data are in agreement with those previously reported.⁸

4,4,5,5-tetramethyl-2-(p-tolyl)-1,3,2-dioxaborolane (2j)



Eluent: petroleum ether/EtOAc = 20/1, white solid, 92 mg, 84% yield. ¹H NMR (400 MHz, Chloroform-d) δ 7.72 (d, *J* = 7.8 Hz, 2H), 7.20 (d, *J* = 7.7 Hz, 2H), 2.38 (s, 3H), 1.35 (s, 12H). ¹³C NMR (101 MHz, Chloroform-d) δ 141.4, 134.8, 128.5, 83.6, 24.9, 21.7. Spectroscopic data are in agreement with those previously reported.⁹

1-(tert-butyl)-4-methylbenzene (2k)



Eluent: petroleum ether, colorless transparent oil, 53 mg, 71% yield. ¹H NMR (400 MHz, Chloroform-d) δ 7.32 (d, *J* = 8.2 Hz, 2H), 7.15 (d, *J* = 8.0 Hz, 2H), 2.35 (s, 3H), 1.34 (s, 9H). ¹³C NMR (101 MHz, Chloroform-d) δ 148.2, 134.8, 128.8, 125.2, 34.3, 31.4, 20.9. Spectroscopic data are in agreement with those previously reported.¹⁰

1,3-dimethoxy-5-methylbenzene (2l)



Eluent: petroleum ether/EtOAc = 20/1, colorless transparent oil, 67 mg, 88% yield. ¹H NMR (400 MHz, Chloroform-d) δ 6.37 (s, 2H), 6.32 (s, 1H), 3.80 (s, 6H), 2.34 (s, 3H). ¹³C NMR (101 MHz, Chloroform-d) δ 160.8, 140.2, 107.1, 97.6, 55.2, 21.8. Spectroscopic data are in agreement with those previously reported.¹¹

1-methyl-3-phenoxybenzene (2m)



2m

Eluent: pentane, yellow oil, 67 mg, 73% yield. ¹H NMR (400 MHz, Chloroform-d) δ 7.33 (t, *J* = 8.0 Hz, 2H), 7.21 (t, *J* = 7.8 Hz, 1H), 7.09 (t, *J* = 7.4 Hz, 1H), 7.01 (d, *J* = 7.6 Hz, 2H), 6.92 (d, *J* = 7.5 Hz, 1H), 6.86-6.82 (m, 2H), 2.35 (s, 3H). ¹³C NMR (101 MHz, Chloroform-d) δ 157.2, 157.4, 139.9, 129.7, 129.4, 124.0, 123.1, 119.6, 118.9, 115.9, 21.4. Spectroscopic data are in agreement with those previously reported.¹²

1-cyclopropyl-4-methylbenzene (2n)



Eluent: pentane, colorless transparent oil, 46 mg, 70% yield. ¹H NMR (400 MHz, Chloroform-d) δ 7.09 (d, *J* = 7.9 Hz, 2H), 7.00 (d, *J* = 8.0 Hz, 2H), 2.33 (s, 3H), 1.92-1.85 (m, 1H), 0.98-0.88 (m, 2H), 0.70-0.66 (m, 2H). ¹³C NMR (101 MHz, Chloroform-d) δ 140.9, 134.8, 129.0, 125.6, 21.0, 15.0, 8.9. Spectroscopic data are in agreement with those previously reported.¹³

2-methylbenzo[b]thiophene (2o)



Eluent: petroleum ether/EtOAc = 100/1, white solid, 59 mg, 79% yield. ¹H NMR (400 MHz, Chloroform-d) δ 7.77 (d, J = 7.9 Hz, 1H), 7.67 (d, J = 7.8 Hz, 1H), 7.35-7.25 (m, 2H), 6.99 (s, 1H), 2.61 (s, 3H). ¹³C NMR (101 MHz, Chloroform-d) δ 140.9, 140.5, 139.7, 124.1, 123.4, 122.6, 122.0, 121.6, 16.2. Spectroscopic data are in agreement with those previously reported.¹⁴

5-methylbenzo[d][1,3]dioxole (2p)



Eluent: petroleum ether/EtOAc = 20/1, colorless transparent oil, 48 mg, 71% yield. ¹H NMR (400 MHz, Chloroform-d) δ 6.75-6.64 (m, 3H), 5.93 (s, 2H), 2.31 (s, 3H). ¹³C NMR (101 MHz, Chloroform-d) δ 147.5, 145.3, 131.5, 121.5, 109.6, 108.0, 100.7, 21.2. Spectroscopic data are in agreement with those previously reported.¹⁵

1-(p-Tolyl)-1H-pyrazole (2q)



Eluent: petroleum ether/EtOAc = 10/1, white solid, 76 mg, 96% yield. ¹H NMR (400 MHz, Chloroform-d) δ 7.85 (d,

J = 2.2 Hz, 1H), 7.69 (d, J = 1.8 Hz, 1H), 7.55 (d, J = 8.3 Hz, 2H), 7.22 (d, J = 7.8 Hz, 2H), 6.42 (t, J = 2.2 Hz, 1H), 2.36 (s, 3H). ¹³C NMR (101 MHz, Chloroform-d) δ 140.8, 138.0, 136.2, 129.9, 126.7, 119.2, 107.3, 20.9. Spectroscopic data are in agreement with those previously reported.¹⁶

9-methylanthracene (2r)



Eluent: pentane, yellow solid, 72 mg, 75% yield. ¹H NMR (400 MHz, Chloroform-d) δ 8.33-8.27 (m, 3H), 8.00 (d, *J* = 7.8 Hz, 2H), 7.53-7.45 (m, 4H), 3.10 (s, 3H). ¹³C NMR (101 MHz, Chloroform-d) δ 131.5, 130.2, 129.1, 125.34, 125.28, 124.8, 124.7, 14.0. Spectroscopic data are in agreement with those previously reported.¹⁷

N-(4-(4-fluorophenyl)-6-isopropyl-5-methylpyrimidin-2-yl)-N-methylmethanesulfonamide (2s)



Eluent: petroleum ether/EtOAc = 10/1, white solid, 139 mg, 82% yield. ¹H NMR (400 MHz, Chloroform-d) δ 7.56-7.52 (m, 2H), 7.14-7.09 (m, 2H), 3.52 (s, 3H), 3.48 (s, 3H), 3.28 (sept, *J* = 6.5 Hz, 1H), 2.25 (s, 3H), 1.28 (d, *J* = 6.7 Hz, 6H). ¹⁹F NMR (376 MHz, Chloroform-d) δ -111.96 – -112.04 (m, 1F). ¹³C NMR (101 MHz, Chloroform-d) δ 175.4, 164.7, 163.1 (d, *J* = 249 Hz), 156.8, 134.8 (d, *J* = 3.3 Hz), 131.3 (d, *J* = 8.5 Hz), 118.7, 115.2 (d, *J* = 21.7 Hz), 42.3, 33.2, 31.9, 21.3, 14.3. Spectroscopic data are in agreement with those previously reported.¹⁸

2-ethylnaphthalene (2t)



Eluent: petroleum ether, colorless transparent oil, 66 mg, 84% yield. ¹H NMR (400 MHz, Chloroform-d) δ 7.87-7.81 (m, 3H), 7.68 (s, 1H), 7.52-7.44 (m, 2H), 7.41 (d, *J* = 8.4 Hz, 1H), 2.87 (q, *J* = 7.6 Hz, 2H), 1.39 (t, *J* = 7.6 Hz, 3H). ¹³C NMR (101 MHz, Chloroform-d) δ 141.8, 133.8, 132.0, 127.8, 127.6, 127.5, 127.1, 125.9, 125.6, 125.0, 29.1, 15.6. Spectroscopic data are in agreement with those previously reported.⁸

4-ethyl-1,1'-biphenyl (2u)



Eluent: petroleum ether, white solid, 64 mg, 70% yield. ¹H NMR (400 MHz, Chloroform-d) δ 7.66-7.63 (m, 2H), 7.61-7.57 (m, 2H), 7.49 (t, *J* = 7.6 Hz, 2H), 7.41-7.33 (m, 3H), 2.76 (q, *J* = 7.6 Hz, 2H), 1.35 (t, *J* = 7.6 Hz, 3H). ¹³C NMR (101 MHz, Chloroform-d) δ 143.4, 141.3, 138.7, 128.8, 128.3, 127.13, 127.06, 127.0, 28.6, 15.6. Spectroscopic data are in agreement with those previously reported.¹⁹

Propoxybenzene (2v)



Eluent: petroleum ether/EtOAc = 100/1, colorless transparent oil, 27 mg, 40% yield. ¹H NMR (400 MHz, Chloroform-d) δ 7.30 (t, *J* = 7.7 Hz, 2H), 6.97-6.92 (m, 3H), 3.94 (t, *J* = 6.6 Hz, 2H), 1.86-1.79 (m, 2H), 1.07 (t, *J* = 7.4 Hz, 3H). ¹³C NMR (101 MHz, Chloroform-d) δ 159.2, 129.4, 120.5, 114.5, 69.4, 22.6, 10.6. Spectroscopic data are in agreement with those previously reported.²⁰

methyl 4-butylbenzoate (2w)



Eluent: petroleum ether/EtOAc = 50/1, colorless transparent oil, 53 mg, 55% yield. ¹H NMR (400 MHz, Chloroform-d) δ 7.94 (d, *J* = 8.0 Hz, 2H), 7.23 (d, *J* = 8.0 Hz, 2H), 3.89 (s, 3H), 2.65 (t, *J* = 7.8 Hz, 2H), 1.64-1.57 (m, 2H), 1.39-1,30 (m, 2H), 0.92 (t, *J* = 7.3 Hz, 3H). ¹³C NMR (101 MHz, Chloroform-d) δ 167.2, 148.5, 129.6, 128.4, 127.6, 51.9, 35.7, 33.3, 22.3, 13.9. Spectroscopic data are in agreement with those previously reported.²¹

1-methoxy-4-propylbenzene (2x)



Eluent: pentane, colorless transparent oil, 34 mg, 46% yield. ¹H NMR (400 MHz, Chloroform-d) δ 7.13 (d, *J* = 8.6 Hz, 2H), 6.86 (d, *J* = 8.6 Hz, 2H), 3.82 (s, 3H), 2.57 (t, *J* = 7.7 Hz, 2H), 1.70-1.60 (m, 2H), 0.97 (t, *J* = 7.3 Hz, 3H). ¹³C NMR (101 MHz, Chloroform-d) δ 157.7, 134.8, 129.3, 113.7, 55.2, 37.2, 24.8, 13.8. Spectroscopic data are in agreement with those previously reported.²²

2-decyl-5,6-dimethoxy-3-methylcyclohexa-2,5-diene-1,4-dione (2y)



Eluent: petroleum ether/EtOAc = 5/1, yellow oi, 92 mg, 57% yield. ¹H NMR (400 MHz, Chloroform-d) δ 3.98 (s, 3H), 3.97 (s, 3H), 2.44 (t, *J* = 7.4 Hz, 2H), 2.00 (s, 3H), 1.39-1.25 (m, 16H), 0.87 (t, *J* = 6.8 Hz, 3H). ¹³C NMR (101 MHz, Chloroform-d) δ 184.7, 184.1, 144.30, 144.28, 143.1, 138.6, 61.1, 31.9, 29.8, 29.6, 29.5, 29.4, 29.3, 28.7, 26.4, 22.7, 14.1, 11.9. HRMS (FI) *m/z* [M]⁺ calcd for C₁₉H₃₀O₄ 322.2144, found 322.2145. IR (KBr) (cm⁻¹) 2926, 2854, 1651, 1610, 1451, 1265, 1219, 772, 673.

(E)-1-(benzyloxy)-3-(prop-1-en-1-yl)benzene (2z)



2z

Eluent: pentane, yellow oil, 91 mg, 81% yield. ¹H NMR (400 MHz, Chloroform-d) δ 7.50-7.24 (m, 6H), 7.03-6.86 (m, 3H), 6.43 (dd, *J* = 15.7, 1.7 Hz, 1H), 6.33-6.24 (m, 1H), 5.10 (s, 2H), 1.93 (dd, *J* = 6.5, 1.6 Hz, 3H). ¹³C NMR (101 MHz, Chloroform-d) δ 159.1, 139.6, 137.2, 131.0, 129.6, 128.6, 128.0, 127.6, 126.2, 118.9, 113.3, 112.4, 70.0, 18.6. HRMS (FI) *m/z* [M]⁺ calcd for C₁₆H₁₆O 224.1201, found 224.1193. IR (KBr) (cm⁻¹) 3064, 3026, 2912, 1655, 1596, 1488, 1438, 1219, 1156, 962, 771, 695.

methyl 4-(prop-1-yn-1-yl)benzoate (2aa)



Eluent: petroleum ether/EtOAc = 20/1, colorless transparent oil, 71 mg, 82% yield. ¹H NMR (400 MHz, Chloroform-d) δ 7.93 (d, *J* = 8.1 Hz, 2H), 7.42 (d, *J* = 8.3 Hz, 2H), 3.88 (s, 3H), 2.05 (s, 3H). ¹³C NMR (101 MHz, Chloroform-d) δ 166.6, 131.4, 129.4, 128.87, 128.86, 89.3, 79.3, 52.1, 4.4. Spectroscopic data are in agreement with those previously reported.²³

4. Copies of ¹ H NMR, ¹⁹F NMR and ¹³C NMR



¹H NMR (400 MHz, CDCl₃) of 2a

$^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, CDCl_3) of 2a







 $^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, CDCl₃) of 2b



¹H NMR (400 MHz, CDCl₃) of 2c



 $^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, CDCl_3) of 2c







¹H NMR (400 MHz, CDCl₃) of 2e



$^{19}\mathsf{F}$ NMR (400 MHz, CDCl₃) of 2e



$^{13}\text{C}\{^{1}\text{H}\}$ NMR (101 MHz, CDCl_3) of 2e



¹H NMR (400 MHz, CDCl₃) of $\mathbf{2f}$







¹H NMR (400 MHz, CDCl₃) of 2g







¹H NMR (400 MHz, CDCl₃) of $\mathbf{2h}$





¹H NMR (400 MHz, CDCl₃) of 2i







 ^1H NMR (400 MHz, CDCl₃) of 2j



$^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, CDCl_3) of 2j



^1H NMR (400 MHz, CDCl₃) of 2k





 ^1H NMR (400 MHz, CDCl₃) of 2I



$^{13}\text{C}\{^{1}\text{H}\}$ NMR (101 MHz, CDCl_3) of 2I



^1H NMR (400 MHz, CDCl₃) of 2m



$^{13}\text{C}\{^{1}\text{H}\}$ NMR (101 MHz, CDCl_3) of 2m



¹H NMR (400 MHz, CDCl₃) of 2n







¹H NMR (400 MHz, CDCl₃) of 2o





¹H NMR (400 MHz, CDCl₃) of **2p**





¹H NMR (400 MHz, CDCl₃) of 2q





¹H NMR (400 MHz, CDCl₃) of 2r





¹H NMR (400 MHz, CDCl₃) of 2s



 $^{19}\mathsf{F}$ NMR (400 MHz, CDCl₃) of **2e**



 $^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, CDCl₃) of 2s



¹H NMR (400 MHz, CDCl₃) of **2t**



 $^{13}\text{C}\{^{1}\text{H}\}$ NMR (101 MHz, CDCl_3) of 2t



¹H NMR (400 MHz, CDCl₃) of **2u**



$^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, CDCl_3) of 2u











$^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, CDCl3) of 2w





 $^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, CDCl_3) of 2x





 $^{13}\text{C}\{^{1}\text{H}\}$ NMR (101 MHz, CDCl₃) of 2y







 $^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, CDCl₃) of 2z





 $^{13}\text{C}\{^1\text{H}\}$ NMR (101 MHz, CDCl3) of 2aa



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