SUPPORTING INFORMATION

KIO₃-catalyzed selective oxidation of thiols to disulfides in water under ambient conditions

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Optimization via solid phase micro extraction (SPME) approach

The extractions were carried out using 1 cm PDMS fiber (100 µm of thickness) obtained from Supelco (Bellefonte, PA, USA), with a SPME holder (Supelco) for manual sampling. Capped vials of 10 mL containing 0.5 mL of UP water and magnetic stirrer (Dist, Florianópolis, Santa Catarina, Brazil) were using. The SPME optimization was performed using a gas chromatograph with a flame ionization detector GC-FID (Agilent, USA) equipped with Zebron ZB-5MS capillary column (30 m \times 0.25 mm \times 0.25 μ m, Torrance, CA, USA). The injection was performed in splitless mode, and the oven temperature program was adjusted to 60 °C (maintained for 1 min), increasing at 10 °C min⁻¹ to 260 °C (maintained for 2 min). The injector temperature was set at 260 °C. The fiber was subjected to a thermal desorption time of 5 min in GC injection port. No carryover effect was observed under these conditions. The reaction by-products were identified via comparison with reference standards. An univariate design was applied to optimize the extraction time (3 to 9 min) for the HS-SPME system (Table S1 and Figure S1). The reaction time (10 to 50 min) and the KIO₃ concentration (15 to 55%) were avaliable. A Doehlert design was applied to optimize the reaction conditions. The reaction time (10 to 50 min) and the KIO₃ concentration (15 to 55%) were available (Table S2 and Figure S2). Statistica 8.0 software was used for the data treatment in multivariate approaches.

$1a$ $H_2O, 25 °C$ $HS-SPME$ (extraction time)					
Entry ^[a]	Extraction time (min.)	Normalized peak area ± RSD			
1	3	54.0 ± 8.5			
2	5	73.1 ± 6.3			
3	7	97.0 ± 10.6			
4	9	100.0 ± 13.6			

Table S1. Thiophenol extraction time optimization by HS-SPME.

[a] Conditions: 1a (0.1 mmol), H₂O (0.5 mL), extraction time (minutes), 25 °C.

RSD: Relative Standard Deviation.



Figure S1. Thiophenol 1a extraction time optimization by HS-SPME.

	$\frac{\text{SH}}{1a} \frac{\text{H}_2\text{O}, \text{ cata}}{\text{time}}$	lyst S S	
Entry ^[a]	Catalyst (mol%)	Reaction time (min.)	% Yield ^[b]
1	-	30	-
2	I ₂ (20)	30	18
3	NaI (20)	30	12
4	KI (20)	30	15
5	NaIO ₃ (20)	30	100
6	KIO ₃ (20)	30	100
7	KIO ₃ (20)	10	62
8	KIO ₃ (15)	20	96
9	KIO ₃ (25)	20	99
10	KIO ₃ (20)	30	99
11	KIO ₃ (20)	30	99
12	KIO ₃ (20)	30	99
13	KIO ₃ (15)	40	90
14	KIO ₃ (25)	40	99
15	KIO ₃ (20)	50	99

 Table S2. Optimization of reaction conditions.

[a] Conditions: **1a** (0.1 mmol), catalyst (mol%), H₂O (0.5 mL), reaction time (minutes), temperature 25 °C. [b] % yield determined by HS-SPME. [c] extraction time by HS-SMPE (7 min).



Figure S2. Doehler planning for optimization of KIO₃ concentration and reaction time (Table S2, entry 7-15).





Figure S3. Response surface.

General remarks

Starting materials obtained from commercial suppliers were used unless otherwise stated. Column chromatography was performed using silica gel 60 (diameter 0.05 - 0.10 mm) Macherey-Nagel. Thin layer chromatography (TLC) was performed using Macherey-Nagel pre-coated TLC sheets ALUGRAM[®] Xtra SIL with layer of 0.20 mm. Visualization was achieved by UV fluorescence, iodine chamber and acidic vanillin.

General procedures for synthesis of disulfides

Procedure A:

In a 20 mL glass tube, thiophenol **1a** (1.0 mmol), potassium iodate (KIO₃; 20 mol%) and water (5 mL) were added. The reaction was stirred for 30 min at 25 °C. At the end of this period, the reaction was extracted with ethyl acetate (3 x 10 mL). The organic phase was dried over anhydrous Na₂SO₄, filtered through filter paper and the organic solvent removed in a rotary evaporator under reduced pressure at 40 °C. The crude product was purified by column chromatography on silica gel using an isocratic elution system (hexane). At the end of the isolation step, product **2a** was obtained as a white solid in quantitative yield.



Procedure B: (For substrates that have limited solubility in water)

In a 20 mL glass tube, corresponding thiol (1.0 mmol), potassium iodate (KIO₃; 20 mol%), Triton-X100 (0.5 ML) and water (5 mL) were added. The reaction was stirred for 120 min at 25 °C. At the end of this period, the reaction was extracted with ethyl acetate (3 x 10 mL). The organic phase was dried over anhydrous Na_2SO_4 , filtered through filter paper and the organic solvent removed in a rotary evaporator under reduced pressure at 40 °C. The crude product was purified by column chromatography on silica gel using an isocratic elution system (hexane). At the end of the isolation step, desired product was obtained.



Gram-Scale Procedure: In a 100 mL glass baloom, corresponding thiol (5.0 mmol or 10.0 mmol), and water (25 mL for 5.0 mmol scale or 50 mL for 10 mmol scale) were added. The reaction was stirred for 30 min at 25 °C. At the end of this period, the reaction was extracted with ethyl acetate (3 x 30 mL). The organic phase was dried over anhydrous Na₂SO₄, filtered through filter paper and the organic solvent removed in a rotary evaporator under reduced pressure at 40 °C. The crude product was purified by column chromatography on silica gel using an isocratic elution system (hexane). At the end of the isolation step, product **2a** was obtained as a white solid in respective yield.

Characterization data products

General considerations: The melting points were taken on a MQAPF-301 melting point apparatus, uncorrected. ¹H and ¹³C NMR spectra were recorded on Varian NMR AS 400 spectrometer and Brucker NMR AC 200, with the samples dissolved in CDCl₃ or DMSO- d_6 . Chemical shifts are informed in ppm downfield from the signal of TMS, used as internal standard, and the coupling constants (*J*) are expressed in Hertz (Hz).



Diphenyl disulfide (2a):¹ Obtained as white solid (104.6 mg, 96%); Purified using hexane as a eluent; mp: 60-62 °C, Rf = 0.8 (hexane); ¹H NMR (400 MHz, Chloroform-*d*) δ 7.49 (dd, J = 7.4, 1.9 Hz, 4H), 7.33 – 7.24 (m, 4H), 7.25 – 7.16 (m, 2H). ¹³C NMR (100 MHz, CDCl₃) δ 136.9, 129.0, 127.4, 127.1.



1,2-di-p-tolyldisulfane (2b):¹ Obtained as white solid (113,2 mg, 92%); Purified using hexane as a eluent; mp: 48-50 °C, Rf = 0.8 (hexane); ¹H NMR (400 MHz, Chloroform-*d*) δ 7.37 (d, J = 8.2 Hz, 1H), 7.08 (d, J = 8.1 Hz, 1H), 2.29 (s, 1H). ¹³C NMR (100 MHz, CDCl₃) δ 137.5, 133.8, 129.7, 128.4, 21.0.



1,2-di-o-tolyldisulfane (2c):¹ Obtained as yellow oil (108.2 mg, 88%); Purified using hexane as a eluent; Rf = 0.6 in (hexane); ¹H NMR (200 MHz, Chloroform-*d*) δ 7.71 – 7.44 (m, 2H), 7.13 (d, J = 2.8 Hz, 6H), 2.42 (s, 6H). ¹³C NMR (50 MHz, CDCl₃) δ 137.4, 135.4, 130.3, 128.7, 127.3, 126.7, 20.0.



1,2-bis(4-methoxyphenyl)disulfane (2d):¹ Obtained as white solid (125.3 mg, 90%); Purified using hexane/ethyl acetate (95:5), Mp: 34-35 °C, Rf = 0.4 (hexane); ¹H NMR (200 MHz, Chloroform-*d*) δ 7.39 (d, J = 8.9 Hz, 4H), 6.83 (d, J = 8.8 Hz, 4H), 3.79 (s, 6H). ¹³C NMR (50 MHz, CDCl₃) δ 205.6, 160.0, 132.6, 128.5, 114.6, 55.3.



1,2-bis(2-methoxyphenyl)disulfane (**2e**):¹ Obtained as white solid (130,8 mg, 94%); Purified using hexane/ethyl acetate (95:5), Mp: 117-119 °C, Rf = 0.3 (hexane); ¹H NMR (400 MHz, CDCl₃) δ 7.51 (dd, J = 7.7, 1.7 Hz, 2H), 7.15 (td, J = 7.6, 1.7 Hz, 2H), 6.87 (dd, J = 7.6, 1.2 Hz, 2H), 6.81 (dd, J = 8.1, 1.2 Hz, 2H), 2.42 (s, 6H). ¹³C NMR (100 MHz, CDCl₃) δ 156.4, 127.6, 127.4, 124.3, 121.2, 110.4.



1,2-bis(4-chlorophenyl)disulfane (2f):¹ Obtained as pallid yellow solid (114.1 mg, 80%); Purified using hexane as a eluent, Mp: 65-66 °C, Rf = 0.6 (hexane); ¹H NMR (400 MHz, CDCl₃) δ 7.38 (d, J = 8.7 Hz, 4H), 7.25 (d, J = 8.7 Hz, 4H). ¹³C NMR (100 MHz, CDCl₃) δ 135.0, 133.5, 129.2, 129.1, 55.7.



1,2-bis(2-chlorophenyl)disulfane (2g):² Obtained as yellow solid (112.0 mg, 78%); Obtained as white solid (108.2 mg, 88%); Purified using hexane as a eluent; mp: 80-82°C ¹H NMR (400 MHz, CDCl₃) δ 7.53 (dd, *J* = 7.8, 1.8 Hz, 2H), 7.33 (dd, *J* = 7.7, 1.6 Hz, 2H), 7.23 – 7.07 (m, 4H). ³C NMR (100 MHz, CDCl₃) δ 134.2, 131.7, 129.6, 127.7, 127.6, 127.5, 127.1.



1,2-bis(3-chlorophenyl)disulfane (2h):¹ Obtained as white solid (137.8 mg, 98%); Purified using hexane as a eluent; mp: 68-70 °C ¹H NMR (400 MHz, CDCl₃) δ 7.46 (m, 2H), 7.32 (dq, *J* = 7.2, 1.8 Hz, 2H), 7.26 – 7.14 (m, 4H). ¹³C NMR (100 MHz, CDCl₃) δ 138.3, 135.0, 130.1, 127.5, 126.9, 125.2.



1,2-bis(4-bromophenyl)disulfane(2i):¹ Obtained as pallid yellow solid (163.6 mg, 87%); Purified using hexane as a eluent, Mp: 92-95 °C, Rf = 0.5 (hexane); ¹H NMR (400 MHz, Chloroform-*d*) δ 7.41 (d, J = 8.6 Hz, 4H), 7.32 (d, J = 8.6 Hz, 4H). ¹³C NMR (100 MHz, CDCl₃) δ 135.7, 132.2, 129.3, 121.5.



1,2-bis(4-fluorophenyl)disulfane (**2j**):² Obtained as yellow oil (119.5 mg, 94%); Purified using hexane as a eluent, Rf = 0.6 in hexane; ¹H NMR (200 MHz, Chloroformd) δ 7.43 (dd, J = 8.6, 5.2 Hz, 4H), 6.99 (t, J = 8.6 Hz, 4H). ¹³C NMR (50 MHz, Chloroform-d) δ 162.6 (d, ¹J = 248.2 Hz), 132.2 (d, ⁴J = 2.7 Hz), 131.2 (d, ³J = 8.2 Hz), 116.2 (d, ²J = 22.1 Hz).



1,2-bis(3-(trifluoromethyl)phenyl)disulfane (2k):³ Obtained as yellow oil (130.1 mg, 74%); Purified using hexane as a eluent, Rf = 0.6 in hexane; ¹H NMR (400 MHz, CDCl₃) δ 7.82 (d, J = 8.1 Hz, 2H), 7.62 (d, J = 7.8 Hz, 2H), 7.46 (t, J = 7.7 Hz, 2H), 7.30 (t, J = 7.7 Hz, 2H). ¹³C NMR (100 MHz, CDCl₃) δ 135.39, 132.51, 129.46, 128.5 (q, J = 32.7 Hz), 127.2, 126.0 (q, J = 8.2 Hz), 123.8 (q, J = 272.7 Hz).



1,2-bis(**4-nitrophenyl**)**disulfane**(**2l**): Obtained as yellow solid (87.9 mg, 57%); Purified using hexane/ethyl acetate (50:50), Mp:182-185°C, Rf = 0.4; ¹H NMR (400 MHz, Chloroform-*d*) δ 8.20 (d, J = 9.1 Hz, 4H), 7.62 (d, J = 9.1 Hz, 4H). ¹³C NMR (100 MHz, CDCl₃) δ 147.0, 144.0, 126.4, 124.4.



4,4'-disulfanediyldianiline (2m):¹ Obtained as white solid (57.1 mg, 46%); Purified using hexane/ethyl acetate (50:50); mp: 108-109 °C ¹H NMR (200 MHz, Chloroformd) δ 7.25 (d, *J* = 8.4 Hz, 1H), 6.57 (d, *J* = 8.4 Hz, 4H), 3.76 (s, 1H). ¹³C NMR (50 MHz, CDCl3) δ 147.1, 133.9, 125.6, 115.3.



2,2'-disulfanediyldianiline (**2n**):² Obtained as yellow solid (79.4 mg, 64%); Purified using hexane/ethyl acetate (50:50); mp: 92-94 °C ¹H NMR (400 MHz, CDCl₃) δ 7.18 (dd, *J* = 8.2, 6.9 Hz, 4H), 6.80 – 6.67 (m, 2H), 6.61 (tt, *J* = 7.3, 1.3 Hz, 2H), 4.36 (s, 4H). ¹³C NMR (100 MHz, CDCl₃) δ 148.6, 136.7, 131.5, 118.6, 118.1, 115.2.



1,2-di(pyridin-2-yl)disulfane (20):² Obtained as white solid (90.3 mg, 82%); Purified using hexane/ethyl acetate (1:1), Mp: 52 °C, Rf = 0.4; ¹H NMR (400 MHz, Chloroformd) $\delta 8.50 - 8.42$ (m, 2H), 7.65 - 7.50 (m, 4H), 7.11 (td, J = 5.2, 3.0 Hz, 2H). ¹³C NMR (100 MHz, CDCl₃) δ 158.8, 149.4, 137.3, 121.0, 119.5.



1,2-di(pyrimidin-2-yl)disulfane(2p):⁴ Obtained as white solid (77.4 mg, 70%); Purified using hexane/ethyl acetate (95:5); mp: 108-109 °C ¹H NMR (200 MHz, Chloroform-*d*) δ 8.59 (d, *J* = 4.9 Hz, 4H), 7.11 (t, *J* = 4.8 Hz, 2H).¹³C NMR (50 MHz, CDCl₃) δ 169.5, 157.8, 118.1.



1,2-di(thiophen-2-yl)disulfane (2q):⁵ Obtained as yellow oil (2w = 69.1 mg, 30%; 2wⁱ = 188.9 mg = 82%) ¹H NMR (400 MHz, CDCl₃) δ 7.46 (dd, *J* = 5.3, 1.5 Hz, 2H), 7.13 (dd, *J* = 3.7, 1.4 Hz, 2H), 6.98 (dd, *J* = 5.5, 3.5 Hz, 2H). ¹³C NMR (100 MHz, CDCl₃) δ 135.6, 132.2, 127.7(2C).



1,2-bis(4,5-dihydrothiazol-2-yl)disulfane (2r):⁶ Obtained as yellow oil (2r = 70.8 mg = 60%; ¹H NMR (400 MHz, Chloroform-*d*) δ 4.70 (t, *J* = 7.8 Hz, 1H), 4.00 (t, *J* = 8.3

Hz, 1H), 3.40 (t, J = 7.8 Hz, 1H), 3.31 (t, J = 8.3 Hz, 1H). ¹³C NMR (100 MHz, Chloroform-d) δ 58.7, 56.9, 36.3, 28.7.



1,2-di(naphthalen-2-yl)disulfane (2s):⁴ Obtained as white solid (2x = traces; $2x^{i} = 242.0$ mg, 76%); Purified using hexane/ethyl acetate (95:5), Mp: 140-142 °C, Rf = 0.7 (hexane); ¹H NMR (200 MHz,) δ 7.98 (s, 1H), 7.84 – 7.55 (m, 8H), 7.53 – 7.37 (m, 4H). ¹³C NMR (50 MHz, CDCl₃) δ 134.3, 133.5, 132.5, 129.0, 127.8, 127.5, 126.7, 126.6, 126.2, 125.7.



1,2-dibenzyldisulfane (2t):¹ Obtained as colourless solid (108.4 mg, 88%); Purified using hexane/ethyl acetate (95:5), Mp: 68-69 °C, Rf = 0.8; ¹H NMR (400 MHz, Chloroform-*d*) δ 7.40 – 7.02 (m, 2H), 3.58 (s, 1H). ¹³C NMR (100 MHz, CDCl₃) δ 137.3, 129.4, 128.4, 127.4, 43.2.



1,2-bis(4-chlorobenzyl)disulfane (2u):² Obtained as pallid yellow solid (143.4 mg, 91%); Purified using hexane/ethyl acetate (95:5); mp: 52-54 °C ¹H NMR (400 MHz, CDCl₃) δ 7.33 – 7.20 (m, 4H), 7.20 – 7.10 (m, 4H), 3.55 (s, 4H). ¹³C NMR (100 MHz, CDCl₃) δ 135.7, 133.3, 130.6, 128.6, 42.3.



1,1'-disulfanediylbis(propan-2-ol) (**2v**):⁷ Obtained a colorless oil (72.0 mg, 79%); Purified using hexane/ethyl acetate (70:30), Rf= 0.27; ¹H NMR (400 MHz, Chloroform*d*) δ 4.08 (dqd, J = 8.0, 4.0, 1.8 Hz, 2H), 3.01 (s, 1H), 2.87 (ddd, J = 14.0, 10.3, 3.9 Hz, 3H), 2.73 (td, J = 13.3, 8.1 Hz, 3H), 1.29 (d, J = 6.3 Hz, 6H). ¹³C NMR (100 MHz, CDCl₃) δ , 66.1, 66.0, 47.8, 47.5, 22.0.



3,3'-disulfanediyldipropionic acid (2w):² Obtained as white solid (111.4 mg, 53%); Purified using hexane/ethyl acetate (70:30); mp= 153-155°C; ¹H NMR (200 MHz, CDCl₃) δ 12.42 (s, 2H), 3.08 – 2.86 (m, 4H), 2.68 (t, *J* = 6.8 Hz, 4H). ¹³C NMR (50 MHz, CDCl₃) δ 172.7, 33.6, 33.0.



1,2-dithiane-4,5-diol (2x):¹ Obtained as white solid (68.8 mg, 91%); Purified using hexane/ethyl acetate (70:30); mp: 128-130°C; ¹H NMR (200 MHz, DMSO- d_6) δ 5.22 (d, J = 3.7 Hz, 2H), 3.35 (dd, J = 8.9, 3.9 Hz, 2H), 3.12 – 2.62 (m, 4H). ¹³C NMR (50 MHz, DMSO) δ 73.2, 40.2.



1,2-dicyclohexyldisulfane (2y):¹ Obtained as light yellow oil (202.7 mg, 88%) ¹H NMR (400 MHz, CDCl₃) δ 3.40 – 0.64 (m, 12H). ¹³C NMR (100 MHz, CDCl₃) 49.9; 32,8; 26.0; 25.6.



1-(dodecyldisulfanyl)dodecane (**2z**):¹ Obtained as colorless oil (200.0 mg, 99%).¹H NMR (400 MHz, CDCl₃) δ 2.51 (qd, J = 7.6, 1.9 Hz, 4H), 1.66 – 1.56 (m, 4H), 1.45 – 1.20 (m, 10H), 0.88 (t, J = 6.8 Hz, 6H). ¹³C NMR (100 MHz, CDCl₃) 34.0, 31.9, 29.6 (2C), 29.5, 29.4, 29.3, 29.0, 28.3, 24.5, 22.6, 14.0.



1-(4-methoxyphenyl)-2-phenyldisulfane: Obtained as yellow oil. (47.2 mg, 38%); Purified using hexane/ethyl acetate (95:5); ¹H NMR (200 MHz, Chloroform-*d*) δ 7.55 – 7.36 (m, 4H), 7.39 – 7.05 (m, 3H), 6.81 (d, *J* = 8.8 Hz, 2H), 3.76 (s, 3H). ¹³C NMR (50 MHz, CDCl₃) δ 159.8, 137.4, 132.6, 131.7, 129.0, 128.2, 127.2, 114.7, 55.3.



2-phenyl-3-(phenylthio)imidazo[1,2-a]pyridine che(4a):⁸ Obtained as white solid (116.4 mg, 77%); Purified using hexane/ethyl acetate (70:30); mp: 77-79 °C ¹H NMR (200 MHz, CDCl₃) δ 8.33 – 8.08 (m, 3H), 7.73 (dt, *J* = 9.0, 1.1 Hz, 1H), 7.62 – 7.03 (m, 8H), 7.04 – 6.93 (m, 1H), 6.85 (td, *J* = 6.8, 1.2 Hz, 1H). ¹³C NMR (50 MHz, CDCl₃) δ 151.4, 147.1, 135.1, 133.3, 129.4, 128.5, 128.3, 126.6, 126.0, 125.5, 124.4, 117.6, 113.0, 106.3.



2-phenyl-3-(p-tolylthio)imidazo[1,2-*a***]pyridine (4b):⁸** Obtained as white solid (129.7 mg, 85%); Purified using hexane/ethyl acetate (70:30); mp: 110-112 °C ¹H NMR (200 MHz, CDCl₃) δ 8.21 (dddt, *J* = 11.2, 8.6, 6.4, 2.1 Hz, 3H), 7.72 (ddd, *J* = 9.0, 2.8, 1.7 Hz, 1H), 7.54 – 7.10 (m, 5H), 7.06 – 6.75 (m, 4H), 2.24 (s, 3H).¹³C NMR (50 MHz, CDCl₃) δ 151.1, 146.9, 135.9, 133.3, 131.4, 130.1, 129.5, 128.3, 126.8, 126.5, 125.7, 124.4, 117.5, 112.9, 106.8, 20.8.



3-((4-chlorophenyl)thio)-2-phenylimidazo[1,2-a]pyridine (4c):⁸ Obtained as white solid (50.5mg, 30%); Purified using hexane/ethyl acetate (95:5); mp: 158-160 °C ¹H NMR (200 MHz, CDCl₃) δ 8.31 – 8.08 (m, 3H), 7.74 (dt, *J* = 9.0, 1.2 Hz, 1H), 7.55 – 7.35 (m, 5H), 7.24 – 7.09 (m, 2H), 7.00 – 6.81 (m, 2H). ¹³C NMR (50 MHz, CDCl₃) δ 151.6, 147.2, 133.7, 133.1, 132.0, 129.6, 128.7, 128.4, 128.3, 126.8, 124.3, 117.8, 113.2, 105.7.

Selected Spectra



¹H RMN (top) ¹³C NMR (bottom) CDCl₃ spectra of compound 2a.



 1H RMN (top) ^{13}C NMR (bottom) CDCl₃ spectra of compound $\mathbf{2b}$



¹H RMN (top) ¹³C NMR (bottom) CDCl₃ spectra of compound **2c**.





¹H RMN (top) ¹³C NMR (bottom) CDCl₃ spectra of compound **2e**.



 ^1H RMN (top) ^{13}C NMR (bottom) CDCl_3 spectra of compound 2f.











¹H RMN (top) ¹³C NMR (bottom) CDCl₃ spectra of compound **2j**.



 $^1\!H$ RMN (top) $^{13}\!C$ NMR (bottom) CDCl₃ spectra of compound 2k.



 1 H RMN (top) 13 C NMR (bottom) CDCl₃ spectra of compound **2**l.



¹H RMN (top) ¹³C NMR (bottom) CDCl₃ spectra of compound **2m**.



¹H RMN (top) ¹³C NMR (bottom) CDCl₃ spectra of compound 2n.



 1 H RMN (top) 13 C NMR (bottom) CDCl₃ spectra of compound **20**.





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¹H RMN (top) ¹³C NMR (bottom) CDCl₃ spectra of compound **2r**.



¹H RMN (top) ¹³C NMR (bottom) CDCl₃ spectra of compound 2s.



 1 H RMN (top) 13 C NMR (bottom) CDCl₃ spectra of compound **2t**.



¹H RMN (top) ¹³C NMR (bottom) CDCl₃ spectra of compound **2u**.



¹H RMN (top) ¹³C NMR (bottom) CDCl₃ spectra of compound 2v.



¹H RMN (top) ¹³C NMR (bottom) CDCl₃ spectra of compound 2w.

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 1H RMN (top) ^{13}C NMR (bottom) CDCl3 spectra of compound 2y.



¹H RMN (top) ¹³C NMR (bottom) CDCl₃ spectra of compound **2z**.



¹H RMN (top) ¹³C NMR (bottom) CDCl₃ spectra of compound **3a**.



¹H RMN (top) ¹³C NMR (bottom) CDCl₃ spectra of compound **4a**.







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