Supporting Information

Continuous-Flow Step-Economic Synthesis of Thiuram Disulfides via Visible-Light Photocatalytic Aerobic Oxidation

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General Information

Reagents

Carbon disulfide (CAS 75-15-0) was purchased from Energy Chemical (99%). Diethylamine (CAS 109-89-7) was purchased from Aladdin (99%). Dimethylamine (CAS 124-40-3) was purchased from Meryer (2

M in THF), Dibenzylamine (CAS 103-49-1) was purchased from Bidepharm (98.35%), Tetraethylthiuram disulfide (CAS 97-77-8) was purchased from Aladdin (97%). Tetramethylthiuram disulfide (CAS 137-26-8) was purchased from Macklin (97%). Tetrabenzylthiuram disulfide (CAS 10591-85-2) was purchased from Adamas-beta (98%). Eosin Y (CAS 152-75-0) was purchased from Macklin (AR 80%). Methylene Blue (CAS 61-73-4) was purchased from Adama-beta (>98%). Bromocresol Green sodium salt (CAS 62625-32-5) was purchased from Macklin, Bromocresol Purple (CAS 115-40-2) was purchased from General-Reagent. Rose Bengal sodium salt (CAS 632-69-9) was purchased from Shanghai Lingfeng Chemical Reagent Co., Ltd. Naphthalene (CAS 91-20-3) was purchased from Adama-beta (RG). Styrene (CAS 100-42-5) was purchased from Shanghai Lingfeng Chemical Reagent Co., Ltd. DMSO-d6 was purchased from CIL (99.9 atom% D). Ethanol (CAS 64-17-5) was purchased by Sinopharm Chemical Reagent Co., Ltd (AR, >99.7%). Methanol (CAS 67-56-1) was purchased from Sigma-Aldrich (HPLC grade, >99.9%). N-Ethylaniline (CAS 103-69-5) was purchased from Aladdin (99%). N-Methylaniline (CAS 100-61-8) was purchased from Aladdin. Dibutylamine (CAS 111-92-2) was purchased from Aladdin (99%). Piperidine (CAS 110-89-4) was purchased by Sinopharm Chemical Reagent Co., Ltd (AR, >99.5%). Dipropylamine (CAS 142-84-7) was purchased from Aladdin (99%). Morpholine (CAS 110-91-8) was purchased from Aladdin (AR 99%), Ethyl acetate (CAS 141-78-6) was purchased by Sinopharm Chemical Reagent Co., Ltd. (AR). Petroleum ether (CAS 8032-32-4) was purchased by Nanjing Chemical Co., Ltd. Magnesium sulfate (CAS 7487-88-9) was purchased from Aladdin (>98%). Diphenylamine (CAS 122-39-4) was purchased from Aladdin (AR >99%, GC). Hydrochloric acid (7647-01-0) was purchased by Nanjing Chemical Co., Ltd (AR 36%~38%). Diisobutylamine (CAS 110-96-3) was purchased from Aladdin (99%). Silica gel (CAS 112926-00-8) was purchased by Qingdao Haiwan Specialty Chemical Co., Ltd. Silicon Dioxide (CAS 7631-8-9) was purchased from General-Reagent (AR). 1,3-diphenylisobenzofuran (CAS 5471-63-6) was purchased from Bidepharm (95%).

Instruments

Magnetic stirrer (C-MAG HS 7) was manufactured by IKA®. Back Pressure Regulator (model# P-786, 75 psi) was manufactured by IDEX. Tubing sleeves (model# CT210, 1/16" to 1/16", model# CT102, 1/8" to 1/16") was manufactured by IDEX. T-micromixer was manufactured by IDEX (model# CT-108, 1/16"). Microchannel (material# PTFE, 1/16") was manufactured by Zhejiang Jiari Fluoroplastic Co., Ltd. Massflow controller (model# YJ-700C) was manufactured by Guangxi Nanning Kongxin Instrument Co., Ltd. Plunger pump (model# TBP-5010) was manufactured by Shanghai Tauto Biotech Co., Ltd. Green LED strip lights (model# 2835, 13 W) was manufactured by Jingzheng Lighting Technology Co., Ltd.

Chromatography Conditions

HPLC analyses were performed on a Wufeng LC100 system with SHISEIDO CAPCELL PAK ADME S5 column (S-5 μ m, 100 Å, 4.6 mm I.D. × 150 mm), at a detection wavelength of 254 nm and a flow rate of 0.5 mL/min, with water (pH = 3, adjusted with formic acid, as phase A) and methanol (as phase B) as the eluents. The isocratic eluents conditions were A:B = 1:9. Naphthalene was used as an internal standard.

NMR Spectra

¹H NMR spectra were collected on 400 MHz NMR spectrometers (Bruker AVANCE III). Chemical shifts

for protons are reported in parts per million (ppm) downfield and are referenced to residual protium in the NMR solvent (δ DMSO-d6 = 2.54). Date are presented as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, quint = quintet, m = multiplet), coupling constants in Hertz (Hz), integration.

FT-IR

FT-IR was manufactured by Tianjin Gangdong Sci. & Tech. Development Co., Ltd. (model# FTIR-650, Spectrum range: 4000–400 cm⁻¹).

Mass Spectra

MS spectra were collected on the electrospray ionization (ESI) MS detection mode (SHIMADZU LCMS-2020).

UV-Vis Spectra

UV-Vis spectroscopy was recorded on UV-Vis spectrometer (SHIMADZU model# UV-2550) in 10 mm quartz cuvettes. Scan wavelength range was 300 to 700 nm.

Batch Reaction Procedures

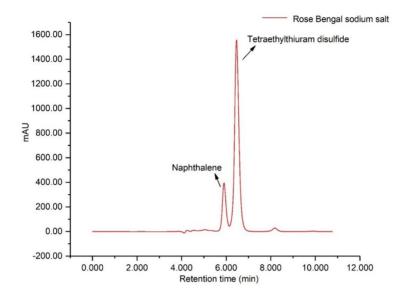
Procedure for Screening Light Sources in Batch

Carbon disulfide (66 μ L, 1.1 mmol), ethanol (2 mL) and Eosin Y (1 mg, 0.002 mmol) were added in Vial **1**. Diethylamine (103 μ L, 1 mmol) and ethanol (1 mL) were added in Vial **2**. The prepared solution in Vial **2** was added dropwise to Vial **1** upon stirring ([carbon disulfide] = 0.35 M, [dibenzylamine] = 0.32 M, [Eosin Y] = 0.00063 M). Vial **1** was continuously stirred on a magnetic stirrer (IKA[®]-C-MAG HS 7) at room temperature for 3.5 h with irradiation from LED strip lights with green, blue and white colors. The distance between the reaction mixture and the light source was approximately 2 cm. Upon completion of the reaction, a small fraction (100 μ L) of the resulting mixture was taken and diluted to 1.5 mL with ethanol, and added naphthalene (internal standard) for HPLC analysis.

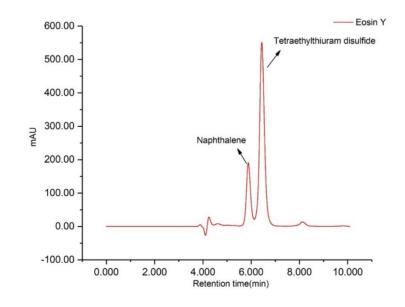
Procedure for the Photocatalytic Aerobic Oxidation of Thiols to Tetraethylthiuram Disulfide (2a) in Batch

Carbon disulfide ($66 \, \mu L$, $1.1 \, mmol$), ethanol ($2 \, mL$) and the photocatalyst were added in a round-bottom flask. The investigated photocatalysts include methylene blue ($1 \, mg$, $0.002 \, mmol$), bromocresol green ($1 \, mg$, $0.002 \, mmol$), bromocresol purple ($1 \, mg$, $0.002 \, mmol$), Eosin Y ($1 \, mg$, $0.002 \, mmol$), rose bengal sodium salt ($2 \, mg$, $0.002 \, mmol$). Then, diethylamine ($103 \, \mu L$, $1 \, mmol$) and ethanol ($1 \, mL$) were added in a vial. The prepared solution in the vial was added dropwise to the flask upon stirring ([carbon disulfide] = $0.35 \, M$, [diethylamine] = $0.32 \, M$, [photocatalyst] = $0.00063 \, M$). An oxygen balloon was attached to the flask and the mixture was continuously stirred on a magnetic stirrer (IKA®-C-MAG HS 7) at room temperature for $3.5 \, h$ with irradiation from a strip of $13 \, W$ green LED lights. The distance between the reaction mixture and the light source was approximately $2 \, cm$. Upon completion of the reaction, a small fraction ($100 \, \mu L$) of the resulting mixture was taken and diluted to $1.5 \, mL$ with ethanol, and added naphthalene (internal standard)

for HPLC analysis.



retention time	height	area	concentration
5.907	403.060	5060.542	15.707
6.465	1563.740	22583.791	74.560



retention time	height	area	concentration
5.882	207.000	2994.227	19.818
6.440	564.020	8860.539	58.647

Procedure for the Photocatalytic Aerobic Oxidation of Thiols to Dipentamethylenethiuram Disulfide (2f) in Batch

Carbon disulfide (66 μ L, 1.1 mmol), ethanol (2 mL) and Eosin Y (1 mg, 0.002 mmol) were added in Vial **1**. Piperidine (91 μ L, 1 mmol) and ethanol (1 mL) were added in Vial **2**. The prepared solution in Vial **2** was added dropwise to Vial **1** upon stirring ([carbon disulfide] = 0.35 M, [piperidine] = 0.32 M, [Eosin Y] = 0.00063 M). An oxygen balloon was attached to Vial **1** and the mixture was continuously stirred on a magnetic stirrer (IKA®-C-MAG HS 7) under irradiation from 13 W green LED strip lights at room temperature for 3.5 h. The distance between the reaction mixture and the light source was approximately 2 cm. Upon completion of the reaction, **2f** precipitated out as a reddish powder. After filtration and drying for 30 min at 50 °C, **2f** was isolated (0.122 g) with a yield of 76%.

Procedure for the Photocatalytic Aerobic Oxidation of Thiols to Dimorpholinethiuram Disulfide (2g) in Batch (with EtOH)

Carbon disulfide (66 μ L, 1.1 mmol), ethanol (2 mL) and Eosin Y (1 mg, 0.002 mmol) were added in Vial **1**. Morpholine (87 μ L, 1 mmol) and ethanol (1 mL) were added in Vial **2**. The prepared solution in Vial **2** was added dropwise to Vial **1** upon stirring ([carbon disulfide] = 0.35 M, [Morpholine] = 0.32 M, [Eosin Y] = 0.00063 M). An oxygen balloon was attached to Vial **1** and the mixture was continuously stirred on a magnetic stirrer (IKA®-C-MAG HS 7) under irradiation from 13 W green LED strip lights at room temperature for 3.5 h. The distance between the reaction mixture and the light source was approximately 2 cm. Upon completion of the reaction, **2g** precipitated out as a white powder. After filtration and vacuum drying for 30 min at 50 °C, **2g** was isolated (0.103 g) with a yield of 64%. A small fraction (10 mg) of the product was taken for NMR and FT-IR analyses. ¹H NMR (400 MHz, CDCl₃) δ (ppm): 3.85 (t, 8H, J = 16 Hz), 4.31(s, 8H).

Procedure for the Photocatalytic Aerobic Oxidation of Thiols to Dimorpholinethiuram Disulfide (2g) in Batch (with MeOH)

Carbon disulfide (66 μ L, 1.1 mmol), <u>methanol</u> (2 mL) and Eosin Y (1 mg, 0.002 mmol) were added in Vial **1**. Morpholine (87 μ L, 1 mmol) and methanol (1 mL) were added in Vial **2**. The prepared solution in Vial **2** was added dropwise to Vial **1** upon stirring ([carbon disulfide] = 0.35 M, [morpholine] = 0.32 M, [Eosin Y] = 0.00063 M). An oxygen balloon was attached to Vial **1** and the mixture was continuously stirred on a magnetic stirrer (IKA®-C-MAG HS 7) under irradiation from 13 W green LED strip lights at room temperature for 3.5 h. The distance between the reaction mixture and the light source was approximately 2 cm. Upon completion of the reaction, **2g** precipitated out as a white powder. After filtration and drying for 30 min at 50 °C, **2g** was isolated (0.055 g) with a yield of 34%.

Procedure for the Photocatalytic Aerobic Oxidation of Thiols to Tetrabenzylthiuram Disulfide (2h) in Batch (with EtOH)

Carbon disulfide (66 µL, 1.1 mmol), ethanol (2 mL) and Eosin Y (1 mg, 0.002 mmol) were added in

Vial **1**. Dibenzylamine (192 μ L, 1 mmol) and ethanol (1 mL) were added in Vial **2**. The prepared solution in Vial **2** was added dropwise to Vial **1** upon stirring ([carbon disulfide] = 0.34 M, [dibenzylamine] = 0.31 M, [Eosin Y] = 0.00061 M). An oxygen balloon was attached to Vial **1** and the mixture was continuously stirred on a magnetic stirrer (IKA®-C-MAG HS 7) under irradiation from 13 W green LED strip lights at room temperature for 3.5 h. The distance between the reaction mixture and the light source was approximately 2 cm. Upon completion of the reaction, **2h** precipitated out as a white powder. After filtration and drying for 30 min at 50 °C, **2h** was isolated (0.166 g) with a yield of 61%. A small fraction (10 mg) of the product was taken for NMR and FT-IR analyses. ¹H NMR (400 MHz, CDCl₃) δ (ppm): 7.34 (m, 20H), 5.36 (s, 4H), 5.19 (s, 4H).

Procedure for the Photocatalytic Aerobic Oxidation of Thiols to Tetrabenzylthiuram Disulfide (2h) in Batch (with MeOH)

Carbon disulfide (66 μ L, 1.1 mmol), methanol (2 mL) and Eosin Y (1 mg, 0.002 mmol) were added in Vial **1**. Dibenzylamine (192 μ L, 1 mmol) and methanol (1 mL) were added in Vial **2**. The prepared solution in Vial **2** was added dropwise to Vial **1** upon stirring ([carbon disulfide] = 0.34 M, [dibenzylamine] = 0.31 M, [Eosin Y] = 0.00061 M). An oxygen balloon was attached to Vial **1** and the mixture was continuously stirred on a magnetic stirrer (IKA®-C-MAG HS 7) under irradiation from 13 W green LED strip lights at room temperature for 3.5 h. The distance between the reaction mixture and the light source was approximately 2 cm. Upon completion of the reaction, **2h** precipitated out as a white powder. After filtration and drying for 30 min at 50 °C, **2h** was isolated (0.142 g) with a yield of 52%.

Flow Reaction Procedures

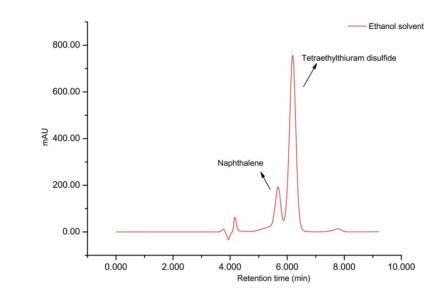
Procedure for the Photocatalytic Aerobic Oxidation of Thiols to Tetraethylthiuram Disulfide (2a) in Flow (Pressure Effect)

Carbon disulfide (265 μ L, 4.4 mmol), ethanol (8 mL) and Eosin Y (6 mg, 0.008 mmol) were added in a round-bottom flask. Diethylamine (410 μ L, 4 mmol) and ethanol (4 mL) were added in a vial. The prepared solution in the vial was added dropwise to the round-bottom flask upon stirring ([carbon disulfide] = 0.35 M, [diethylamine] = 0.32 M, [Eosin Y] = 0.00063 M). The mixture was continuously stirred on a magnetic stirrer (IKA®-C-MAG HS 7) at room temperature for 30 min. The solution in the flask was introduced into the microreactor set-up with a plunger pump (model# TBP-5010). The pump and the mass-flow controller were operated at a volume ratio of liquid/oxygen of 1:2. The retention time was 25 min. The liquid hold-up of microchannel was 10 mL. The microchannel was irradiated with green LED light strips (13 W). The back-pressure regulator was adjusted to 0.2 MPa, 0.3 MPa, 0.4 MPa, 0.5 Mpa and 0.6 MPa, and was placed in a water bath of 50 °C. Upon completion of the reaction, a small fraction (100 μ L) of the resulting mixture was taken and diluted to 1.5 mL with ethanol, and added naphthalene (internal standard) for HPLC analysis.

Procedure for the Photocatalytic Aerobic Oxidation of Thiols to Tetraethylthiuram Disulfide (2a) in Flow (Retention Time Effect)

Carbon disulfide (265 µL, 4.4 mmol), ethanol (8 mL) and Eosin Y (6 mg, 0.008 mmol) were added in a

round-bottom flask. Diethylamine (410 μ L, 4 mmol) and ethanol (4 mL) were added in a vial. The prepared solution in the vial was added dropwise to the flask upon stirring ([carbon disulfide] = 0.35 M, [diethylamine] = 0.32 M, [Eosin Y] = 0.00063 M). The mixture was continuously stirred on a magnetic stirrer (IKA®-C-MAG HS 7) at room temperature for 30 min. The solution in the flask was introduced into the microreactor set-up with a plunger pump (model# TBP-5010). The pump and the mass-flow controller were operated at a volume ratio of liquid/oxygen of 1:2. The <u>retention time</u> was adjusted to 1 min, 3 min, 5 min, 10 min, 20 min, 25 min and 30 min. The liquid hold-up of microchannel was 10 mL. The microchannel was irradiated with green LED light strips (13 W). A back-pressure regulator with 0.5 MPa was placed in a water bath of 50 °C. Upon completion of the reaction, a small fraction (100 μ L) of the resulting mixture was taken and diluted to 1.5 mL with ethanol, and added naphthalene (internal standard) for HPLC analysis.



retention time	height	area	concentration
5.680	212.090	4911.622	23.047
6.190	772.010	13550.590	63.584

Procedure for the Photocatalytic Aerobic Oxidation of Thiols to Tetraethylthiuram Disulfide (2a) in Flow (the Optimized Conditions)

Carbon disulfide (1060 μ L, 17.6 mmol), ethanol (32 mL) and Eosin Y (22 mg, 0.032 mmol) were added in a round-bottom flask (flask 1). To another flask (flask 2), diethylamine (1650 μ L, 16 mmol) and ethanol (16 mL) were added. The prepared solution in flask 2 was added dropwise to flask 1 upon stirring ([carbon disulfide] = 0.35 M, [diethylamine] = 0.32 M, [Eosin Y] = 0.00063 M). The mixture was continuously stirred on a magnetic stirrer (IKA®-C-MAG HS 7) at room temperature for 30 min. The solution was introduced into the microreactor set-up with a plunger pump (model# TBP-5010). The pump and the mass-flow controller were operated at a volume ratio of liquid/oxygen of 1:2. The retention time was 25 min. The liquid hold-up of microchannel was 10 mL. The microchannel was irradiated with green LED light strips (13 W). The back-pressure regulator was adjusted to 0.5 MPa, and was placed in a water bath of 50 °C. Upon completion of

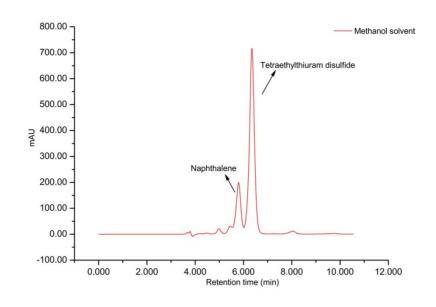
the reaction, a total of 36 mL reaction mixture was collected. Crude **2a** was obtained as a red crystal. After cold crystallization and filtration, **2a** was isolated as a red crystal. Washing with ethanol and drying for 30 min at 50 °C gave a colorless glassy crystal (1.590 g), with a yield of 94%. A small fraction (10 mg) of the products was taken for NMR, FT-IR and MS analyses. ¹H NMR (400 MHz, CDCl₃) δ (ppm): 4.03 (m, 8H), 1.30 (t, 6H, J = 8 Hz), 1.48 (t, 6H, J = 8 Hz).

Scale-up Experiment for the Photocatalytic Aerobic Oxidation of Thiols to Tetraethylthiuram Disulfide (2a) in Flow

Carbon disulfide (5980 μ L, 99 mmol), ethanol (180 mL) and Eosin Y (126 mg, 0.18 mmol) were added in a round-bottom flask (flask 1). To another flask (flask 2), diethylamine (9270 μ L, 90 mmol) and ethanol (90 mL) were added. The prepared solution in flask 2 was added dropwise to flask 1 upon stirring ([carbon disulfide] = 0.35 M, [diethylamine] = 0.32 M, [Eosin Y] = 0.00063 M). The solution was continuously stirred on a magnetic stirrer (IKA®-C-MAG HS 7) at room temperature for 30 min. The solution was introduced into the microreactor set-up with a plunger pump (model# TBP-5010). The pump and the mass-flow controller were operated at a volume ratio of liquid/oxygen of 1:2. The retention time was 25 min. The liquid hold-up of microchannel was 10 mL. The microchannel was irradiated with green LED light strips (13 W). The back-pressure regulator was adjusted to 0.5 MPa, and was placed in a water bath of 50 °C. Upon completion of the reaction, a total of 250 mL reaction mixture was collected in 25 h. Crude 2a was obtained as a red crystal. After cold crystallization and filtration, 2a was isolated as a red crystal. Washing with ethanol and drying at 50 °C for 30 min gave a colorless glassy crystal (11.240 g), with a yield of 96%.

Procedure for the Photocatalytic Aerobic Oxidation of Thiols to Tetraethylthiuram Disulfide (2a) in Flow (with MeOH, Retention Time Effect)

Carbon disulfide (265 μ L, 4.4 mmol), methanol (8 mL) and Eosin Y (6 mg, 0.008 mmol) were added in a round-bottom flask. Diethylamine (410 μ L, 4 mmol) and methanol (4 mL) were added in a vial. The prepared solution in the vial was added dropwise to the flask upon stirring ([carbon disulfide] = 0.35 M, [diethylamine] = 0.32 M, [Eosin Y] = 0.00063 M). The mixture was continuously stirred on a magnetic stirrer (IKA®-C-MAG HS 7) at room temperature for 30 min. The solution was introduced into the microreactor set-up with a plunger pump (model# TBP-5010). The pump and the mass-flow controller were operated at a volume ratio of liquid/oxygen of 1:2. The retention time was adjusted to 10 min, 15 min, 20 min, 25 min and 30 min. The liquid hold-up of microchannel was 10 mL. The microchannel was irradiated with green LED light strips (13 W). The back-pressure regulator with 0.5 MPa was placed in a water bath of 50 °C. Upon completion of the reaction, a small fraction (100 μ L) of the resulting mixture was taken and diluted to 1.5 mL with ethanol, and added naphthalene (internal standard) for HPLC analysis.



retention time	height	area	concentration
5.798	205.050	2946.570	18.300
6.348	720.750	10860.107	67.448

Procedure for the Photocatalytic Aerobic Oxidation of Thiols to Tetraethylthiuram Disulfide (2a) in Flow (with MeOH, Optimized Retention Time)

Carbon disulfide (1060 μ L, 17.6 mmol), methanol (32 mL) and Eosin Y (22 mg, 0.032 mmol) were added in a round-bottom flask (flask 1). To another flask (flask 2), diethylamine (1650 μ L, 16 mmol) and methanol (16 mL) were added. The prepared solution in flask 2 was added dropwise to flask 1 upon stirring ([carbon disulfide] = 0.35 M, [diethylamine] = 0.32 M, [Eosin Y] = 0.00063 M). The mixture was continuously stirred on a magnetic stirrer (IKA®-C-MAG HS 7) at room temperature for 30 min. The solution was introduced into the microreactor set-up with a plunger pump (model# TBP-5010). The pump and the mass-flow controller were operated at a volume ratio of liquid/oxygen of 1:2. The retention time was 20 min. The liquid hold-up of microchannel was 10 mL. The microchannel was irradiated with green LED light strips (13 W). The back-pressure regulator was adjusted to 0.5 MPa, and was placed in a water bath of 50 °C. Upon completion of the reaction, a total of 12 mL reaction mixture was collected. Crude 2a was obtained as a red crystal. After cold crystallization and filtration, 2a was isolated as a red crystal. Washing with methanol and drying for 30 min at 50 °C gave a colorless glassy crystal (0.505 g), with a yield of 90%.

Procedure for the Photocatalytic Aerobic Oxidation of Thiols to Tetramethylthiuram Disulfide (2b) in Flow (with EtOH)

Carbon disulfide (1060 μ L, 17.6 mmol), <u>ethanol</u> (32 mL) and Eosin Y (22 mg, 0.032 mmol) were added in a round-bottom flask (flask 1). To another flask (flask 2), dimethylamine (8 mL, 16 mmol) and ethanol (16 mL) were added. The prepared solution in flask 2 was added dropwise to flask 1 upon stirring ([carbon

disulfide] = 0.31 M, [dimethylamine] = 0.28 M, [Eosin Y] = 0.00056 M). The mixture was continuously stirred on a magnetic stirrer (IKA®-C-MAG HS 7) at room temperature for 30 min. The solution was introduced into the microreactor set-up with a plunger pump (model# TBP-5010). The pump and the mass-flow controller were operated at a volume ratio of liquid/oxygen of 1:2. The retention time was 5 min. The liquid hold-up of microchannel was 10 mL. The microchannel was irradiated with green LED light strips (13 W). The back-pressure regulator was adjusted to 0.5 MPa, and was placed in a water bath of 50 °C. Upon completion of the reaction, a total of 33 mL reaction mixture was collected, and **2b** was obtained as a red powder. Evaporation of solvent by rotary evaporator and vacuum drying for 30 min at 50 °C gave **2b** as a red powder (0.585 g), with a yield of 53%. A small fraction (10 mg) of **2b** was taken for NMR, FT-IR and MS analyses. 1 H NMR (400 MHz, CDCl₃) δ (ppm): 3.61 (s, 6H), 3.64 (s, 6H).

Procedure for the Photocatalytic Aerobic Oxidation of Thiols to Tetramethylthiuram Disulfide (2b) in Flow (with MeOH)

Carbon disulfide (1060 μ L, 17.6 mmol), methanol (32 mL) and Eosin Y (22 mg, 0.032 mmol) were added in a round-bottom flask (flask 1). To another flask (flask 2), dimethylamine (8 mL, 16 mmol) and methanol (16 mL) were added. The prepared solution in flask 2 was added dropwise to flask 1 upon stirring ([carbon disulfide] = 0.31 M, [dimethylamine] = 0.28 M, [Eosin Y] = 0.00056 M). The mixture was continuously stirred on a magnetic stirrer (IKA®-C-MAG HS 7) at room temperature for 30 min. The solution was introduced into the microreactor set-up with a plunger pump (model# TBP-5010). The pump and the mass-flow controller were operated at a volume ratio of liquid/oxygen of 1:2. The retention time was 6.5 min. The liquid hold-up of microchannel was 10 mL. The microchannel was irradiated with green LED light strips (13 W). The back-pressure regulator was adjusted to 0.5 MPa, and was placed in a water bath of 50 °C. Upon completion of the reaction, 7.5 mL of the resulting mixture was collected, and 2b was obtained as a red powder. Evaporation of solvent by rotary evaporator and vacuum drying for 30 min at 50 °C gave 2b as a reddish powder (0.108 g), with a yield of 43%.

Procedure for the Photocatalytic Aerobic Oxidation of Thiols to Tetrapropylthiuram Disulfide (2c) in Flow (with EtOH)

Carbon disulfide (396 μ L, 6.6 mmol), ethanol (12 mL) and Eosin Y (8 mg, 0.012 mmol) were added in a round-bottom flask. Dipropylamine (822 μ L, 6 mmol) and ethanol (6 mL) were added to a vial. The prepared solution in the vial was added dropwise to the flask upon stirring ([carbon disulfide] = 0.34 M, [dipropylamine] = 0.31 M, [Eosin Y] = 0.00062 M). The mixture was continuously stirred on a magnetic stirrer (IKA®-C-MAG HS 7) at room temperature for 30 min. The solution was introduced into the microreactor set-up with a plunger pump (model# TBP-5010). The pump and the mass-flow controller were operated at a volume ratio of liquid/oxygen of 1:2. The retention time was 25 min. The liquid hold-up of microchannel was 10 mL. The microchannel was irradiated with green LED light strips (13 W). The back-pressure regulator was adjusted to 0.5 MPa, and was placed in a water bath of 50 °C. Upon completion of the reaction, 5 mL of the resulting mixture was collected. The resulting mixture was washed with H₂O (3 × 5 mL) and dried over MgSO₄. The solvent was removed under vacuum, and **2c** was obtained as a reddish powder (0.176 g) with a yield of 64%. A small fraction (10 mg) of the product was taken for NMR, FT-IR

and MS analyses. ¹H NMR (400 MHz, CDCl₃) δ (ppm): 0.93 (t, 6H, J = 6 Hz), 1.03 (t, 6H, J = 8 Hz), 1.79 (t, 4H, J = 6 Hz), 1.94 (t, 4H, J = 8 Hz), 3.90 (m, 8H).

Procedure for the Photocatalytic Aerobic Oxidation of Thiols to Tetrapropylthiuram Disulfide (2c) in Flow (with MeOH)

Carbon disulfide (396 μ L, 6.6 mmol), methanol (12 mL) and Eosin Y (8 mg, 0.012 mmol) were added in a round-bottom flask. Dipropyllamine (822 μ L, 6 mmol) and methanol (6 mL) were added to a Vial. The prepared solution in the vial was added dropwise to the flask upon stirring ([carbon disulfide] = 0.34 M, [dipropyllamine] = 0.31 M, [Eosin Y] = 0.00062 M). The mixture was continuously stirred on a magnetic stirrer (IKA®-C-MAG HS 7) at room temperature for 30 min. The solution was introduced into the microreactor set-up with a plunger pump (model# TBP-5010). The pump and the mass-flow controller were operated at a volume ratio of liquid/oxygen of 1:2. The retention time was 20 min. The liquid hold-up of microchannel was 10 mL. The microchannel was irradiated with green LED light strips (13 W). The back-pressure regulator was adjusted to 0.5 MPa, and was placed in a water bath of 50 °C. Upon completion of the reaction, 5 mL of the resulting mixture was collected. The mixture was washed with H₂O (3 × 5 mL) and dried over MgSO₄. The solvent was removed under vacuum, and **2c** was obtained as a reddish powder (0.189 g) with a yield of 69%.

Procedure for the Photocatalytic Aerobic Oxidation of Thiols to Tetrabutylthiuram Disulfide (2d) in Flow (with EtOH)

Carbon disulfide (396 μ L, 6.6 mmol), ethanol (12 mL) and Eosin Y (8 mg, 0.012 mmol) were added in a round-bottom flask. Dibutylamine (1011 μ L, 6 mmol) and ethanol (6 mL) were added to a vial. The prepared solution in the vial was added dropwise to the flask upon stirring ([carbon disulfide] = 0.34 M, [dibutylamine] = 0.31 M, [Eosin Y] = 0.00062 M). The mixture was continuously stirred on a magnetic stirrer (IKA®-C-MAG HS 7) at room temperature for 30 min. The solution was introduced into the microreactor set-up with a plunger pump (model# TBP-5010). The pump and the mass-flow controller were operated at a volume ratio of liquid/oxygen of 1:2. The retention time was 25 min. The liquid hold-up of microchannel was 10 mL. The microchannel was irradiated with green LED light strips (13 W). The back-pressure regulator was adjusted to 0.5 MPa, and was placed in a water bath of 50 °C. Upon completion of the reaction, 5 mL of the resulting mixture was collected. Crude 2d was purified by column chromatography over silica gel. The solvent was removed under vacuum, and 2d was obtained as a yellowish liquid (0.190 g) with a yield of 60%. A small fraction (10 mg) of the product was taken for NMR, FT-IR and MS analyses. 1 H NMR (400 MHz, CDCl₃) δ (ppm): 0.94 (t, 6H, J = 8 Hz), 1.01 (d, 6H, J = 8 Hz), 3.94 (m, 8H), 1.89 (m, 4H), 1.73 (m, 4H), 1.45 (m, 4H), 1.35 (m, 4H).

Procedure for the Photocatalytic Aerobic Oxidation of Thiols to Tetrabutylthiuram Disulfide (2d) in Flow (with MeOH)

Carbon disulfide (396 μ L, 6.6 mmol), methanol (12 mL) and Eosin Y (8 mg, 0.012 mmol) were added in a round-bottom flask. Dibutylamine (1011 μ L, 6 mmol) and methanol (6 mL) were added to a vial. The prepared solution in the vial was added dropwise to the flask upon stirring ([carbon disulfide] = 0.34 M,

[dibutylamine] = 0.31 M, [Eosin Y] = 0.00062 M). The mixture was continuously stirred on a magnetic stirrer (IKA®-C-MAG HS 7) at room temperature for 30 min. The solution was introduced into the microreactor set-up with a plunger pump (model# TBP-5010). The pump and the mass-flow controller were operated at a volume ratio of liquid/oxygen of 1:2. The retention time was 20 min. The liquid hold-up of microchannel was 10 mL. The microchannel was irradiated with green LED light strips (13 W). The back-pressure regulator was adjusted to 0.5 MPa, and was placed in a water bath of 50 °C. Upon completion of the reaction, 5 mL of the resulting mixture was collected. Crude **2d** was purified by column chromatography over silica gel. The solvent was removed under vacuum, and **2d** was obtained as a yellowish liquid (0.228 g) with a yield of 72%.

Procedure for the Photocatalytic Aerobic Oxidation of Thiols to Tetraisobutylthiuram Disulfide (2e) in Flow (with EtOH)

Carbon disulfide (396 μ L, 6.6 mmol), ethanol (12 mL) and Eosin Y (8 mg, 0.012 mmol) were added in a round-bottom flask. Diisobutylamine (1047 μ L, 6 mmol) and ethanol (6 mL) were added to a vial. The prepared solution in the vial was added dropwise to the flask upon stirring ([carbon disulfide] = 0.34 M, [diisobutylamine] = 0.31 M, [Eosin Y] = 0.00062 M). The mixture was continuously stirred on a magnetic stirrer (IKA®-C-MAG HS 7) at room temperature for 30 min. The solution was introduced into the microreactor set-up with a plunger pump (model# TBP-5010). The pump and the mass-flow controller were operated at a volume ratio of liquid/oxygen of 1:2. The retention time was 25 min. The liquid hold-up of microchannel was 10 mL. The microchannel was irradiated with green LED light strips (13 W). The back-pressure regulator was adjusted to 0.5 MPa, and was placed in a water bath of 50 °C. Upon completion of the reaction, 5 mL of the resulting mixture was collected. Crude **2e** was purified by column chromatography over silica gel. The solvent was removed under vacuum, and **2e** was obtained as a yellowish powder (0.123 g) with a yield of 39%. A small fraction (10 mg) of the product was taken for NMR, FT-IR and MS analyses. 1 H NMR (400 MHz, CDCl₃) $\bar{\delta}$ (ppm): 0.93 (d, 12H, J = 8 Hz), 1.04 (d, 12H, J = 8 Hz), 3.85 (t, 8H, J = 8 Hz), 2.50 (m, 4H).

Procedure for the Photocatalytic Aerobic Oxidation of Thiols to Tetraisobutylthiuram Disulfide (2e) in Flow (with MeOH)

Carbon disulfide (396 μ L, 6.6 mmol), methanol (12 mL) and Eosin Y (8 mg, 0.012 mmol) were added in a round-bottom flask. Diisobutylamine (1047 μ L, 6 mmol) and methanol (6 mL) were added to a vial. The prepared solution in the vial was added dropwise to the flask upon stirring ([carbon disulfide] = 0.34 M, [diisobutylamine] = 0.31 M, [Eosin Y] = 0.00062 M). The mixture was continuously stirred on a magnetic stirrer (IKA®-C-MAG HS 7) at room temperature for 30 min. The solution was introduced into the microreactor set-up with a plunger pump (model# TBP-5010). The pump and the mass-flow controller were operated at a volume ratio of liquid/oxygen of 1:2. The retention time was 20 min. The liquid hold-up of microchannel was 10 mL. The microchannel was irradiated with green LED light strips (13 W). The back-pressure regulator was adjusted to 0.5 MPa, and was placed in a water bath of 50 °C. Upon completion of the reaction, 5 mL of the resulting mixture was collected. Crude **2e** was purified by column chromatography over silica gel. The solvent was removed under vacuum, and **2e** was obtained as a yellowish powder (0.146).

g) with a yield of 46%.

Procedure for the Photocatalytic Aerobic Oxidation of Thiols to Dipentamethylenethiuram Disulfide (2f) in Flow

Carbon disulfide (396 µL, 6.6 mmol), methanol (12 mL) and Eosin Y (8 mg, 0.012 mmol) were added in a round-bottom flask. Piperidine (547 µL, 6 mmol) and methanol (6 mL) were added to a vial. The prepared solution in the vial was added dropwise to the flask upon stirring ([carbon disulfide] = 0.35 M, [piperidine] = 0.32 M, [Eosin Y] = 0.00063 M). The mixture was continuously stirred on a magnetic stirrer (IKA®-C-MAG HS 7) at room temperature for 30 min. The solution was introduced into the microreactor set-up with a plunger pump (model# TBP-5010). The pump and the mass-flow controller were operated at a volume ratio of liquid/oxygen of 1:2. The retention time was 20 min. The liquid hold-up of microchannel was 10 mL. The microchannel was irradiated with green LED light strips (13 W). The back-pressure regulator was adjusted to 0.5 MPa, and was placed in a water bath of 50 °C. Upon completion of the reaction, 5 mL of the resulting mixture was collected. The mixture was condensed under vacuum and the residue was dissolved in CH₂CI₂. The solution was washed with 0.2 M HCl (3 × 5 mL) and dried over MqSO₄. The solvent was removed under vacuum to give a reddish powder 2f (0.168 g), with a yield of 66%. A small fraction (10 mg) of the product was taken for NMR, FT-IR and MS analyses. ¹H NMR (400 MHz, CDCl₃) δ (ppm): 1.77 (s, 12H), 4.22 (s, 8H).

Procedure for the Photocatalytic Aerobic Oxidation of Thiols to *N,N'*-Dimethyldiphenylthiuram Disulfide (2i) in Flow (with EtOH)

Carbon disulfide (396 μ L, 6.6 mmol), ethanol (12 mL) and Eosin Y (8 mg, 0.012 mmol) were added in a round-bottom flask. *N*-Methylaniline (650 μ L, 6 mmol) and ethanol (6 mL) were added to a vial. The prepared solution in the vial was added dropwise to the flask upon stirring ([carbon disulfide] = 0.35 M, [*N*-methylaniline] = 0.32 M, [Eosin Y] = 0.00063 M). The mixture was continuously stirred on a magnetic stirrer (IKA®-C-MAG HS 7) at room temperature for 30 min. The solution was introduced into the microreactor set-up with a plunger pump (model# TBP-5010). The pump and the mass-flow controller were operated at a volume ratio of liquid/oxygen of 1:2. The retention time was 25 min. The liquid hold-up of microchannel was 10 mL. The microchannel was irradiated with green LED light strips (13 W). The back-pressure regulator was adjusted to 0.5 MPa, and was placed in a water bath of 50 °C. Upon completion of the reaction, 5 mL of the resulting mixture was collected. The resulting mixture was condensed under vacuum and the residue was dissolved in CH_2Cl_2 . The solution was washed with 0.2 M HCl (3 × 5 mL) and dried over MgSO₄. The solvent was removed under vacuum to give a reddish powder **2i** (0.033 g), with a yield of 12%. A small fraction (10 mg) of the product was taken for NMR, FT-IR and MS analyses. ¹H NMR (400 MHz, CDCl₃) δ (ppm): 3.82 (s, 6H), 7.48 (m, 10H).

Procedure for the Photocatalytic Aerobic Oxidation of Thiols to *N,N'*-Dimethyldiphenylthiuram Disulfide (2i) in Flow (with MeOH)

Carbon disulfide (396 μ L, 6.6 mmol), <u>methanol</u> (12 mL) and Eosin Y (8 mg, 0.012 mmol) were added in a round-bottom flask. *N*-Methylaniline (650 μ L, 6 mmol) and methanol (6 mL) were added to a vial. The

prepared solution in the vial was added dropwise to the flask upon stirring ([carbon disulfide] = 0.35 M, [N-methylaniline] = 0.32 M, [Eosin Y] = 0.00063 M). The mixture was continuously stirred on a magnetic stirrer (IKA®-C-MAG HS 7) at room temperature for 30 min. The solution was introduced into the microreactor set-up with a plunger pump (model# TBP-5010). The pump and the mass-flow controller were operated at a volume ratio of liquid/oxygen of 1:2. The retention time was 20 min. The liquid hold-up of microchannel was 10 mL. The microchannel was irradiated with green LED light strips (13 W). The back-pressure regulator was adjusted to 0.5 MPa, and was placed in a water bath of 50 °C. Upon completion of the reaction, 5 mL of the resulting mixture was collected. The mixture was condensed under vacuum and the residue was dissolved in CH_2CI_2 . The solution was washed with 0.2 M HCI (3 × 5 mL) and dried over MgSO₄. The solvent was removed under vacuum to give a reddish powder **2i** (0.043 g), with a yield of 15%.

Procedure for the Photocatalytic Aerobic Oxidation of Thiols to Diethyldiphenylthiuram Disulfide (2j) in Flow (with EtOH)

Carbon disulfide (396 μ L, 6.6 mmol), ethanol (12 mL) and Eosin Y (8 mg, 0.012 mmol) were added in a round-bottom flask. *N*-Ethylaniline (755 μ L, 6 mmol) and ethanol (6 mL) were added to a vial. The prepared solution in the vial was added dropwise to the flask upon stirring ([carbon disulfide] = 0.34 M, [*N*-ethylaniline] = 0.31 M, [Eosin Y] = 0.00063 M). The mixture was continuously stirred on a magnetic stirrer (IKA®-C-MAG HS 7) at room temperature for 30 min. The solution was introduced into the microreactor set-up with a plunger pump (model# TBP-5010). The pump and the mass-flow controller were operated at a volume ratio of liquid/oxygen of 1:2. The retention time was 25 min. The liquid hold-up of microchannel was 10 mL. The microchannel was irradiated with green LED light strips (13 W). The back-pressure regulator was adjusted to 0.5 MPa, and was placed in a water bath of 50 °C. Upon completion of the reaction, 5 mL of the resulting mixture was collected. The mixture was condensed under vacuum and the residue was dissolved in CH₂Cl₂. The solution was washed with 0.2 M HCl (3 × 5 mL) and dried over MgSO₄. The solvent was removed under vacuum to give a reddish powder **2j** (0.026 g), with a yield of 9%. A small fraction (10 mg) of the product was taken for NMR and FT-IR analyses. ¹H NMR (400 MHz, CDCl₃) δ (ppm): 1.28 (t, 6H, J = 8 Hz), 4.35 (q, 4H, J = 8 Hz), 7.45 (m,10H).

Procedure for the Photocatalytic Aerobic Oxidation of Thiols to Diethyldiphenylthiuram Disulfide (2j) in Flow (with MeOH)

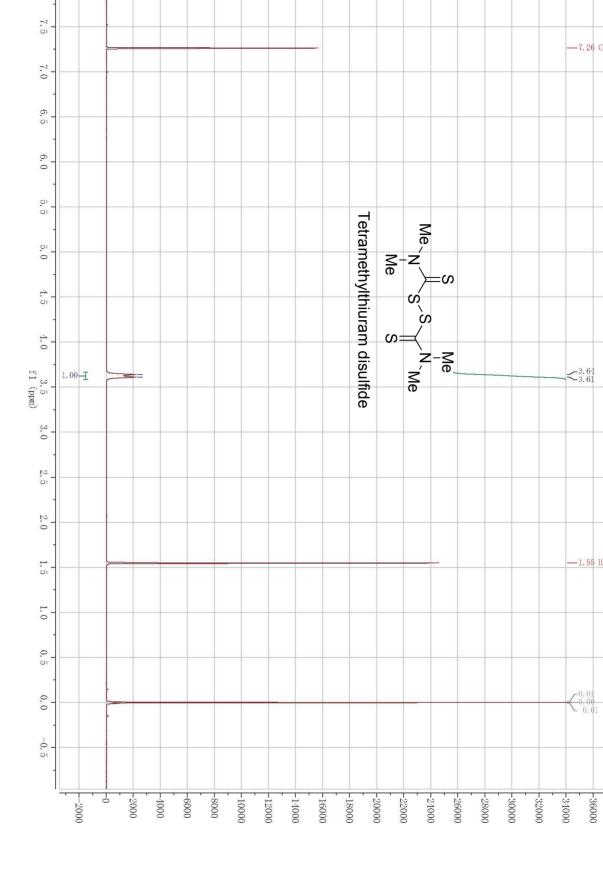
Carbon disulfide (396 μ L, 6.6 mmol), methanol (12 mL) and Eosin Y (8 mg, 0.012 mmol) were added in a round-bottom flask. *N*-Ethylaniline (755 μ L, 6 mmol) and methanol (6 mL) were added to a vial. The prepared solution in the vial was added dropwise to the flask upon stirring ([carbon disulfide] = 0.367 M, [*N*-ethylaniline] = 0.333 M, [Eosin Y] = 0.000667 M). The mixture was continuously stirred on a magnetic stirrer (IKA®-C-MAG HS 7) at room temperature for 30 min. The solution was introduced into the microreactor set-up with a plunger pump (model# TBP-5010). The pump and the mass-flow controller were operated at a volume ratio of liquid/oxygen of 1:2. The retention time was 20 min. The liquid hold-up of microchannel was 10 mL. The microchannel was irradiated with green LED light strips (13 W). The back-pressure regulator was adjusted to 0.5 MPa, and was placed in a water bath of 50 °C. Upon completion of the reaction, 5 mL of the resulting mixture was collected. The mixture was condensed under vacuum and the

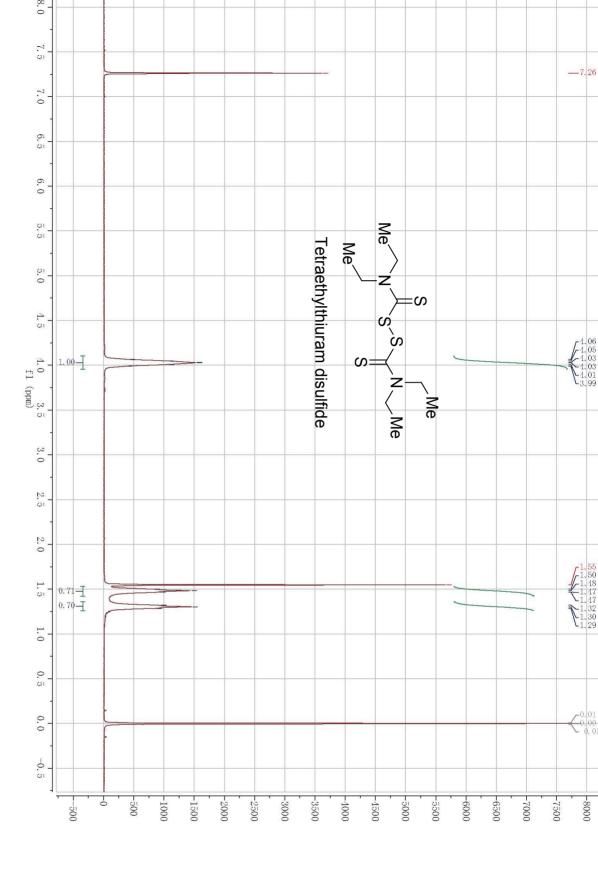
residue was dissolved in CH_2CI_2 . The solution was washed with 0.2 M HCl (3 × 5 mL) and dried over MgSO₄. The solvent was removed under vacuum to give a reddish powder **2j** (0.065 g), with a yield of 21%.

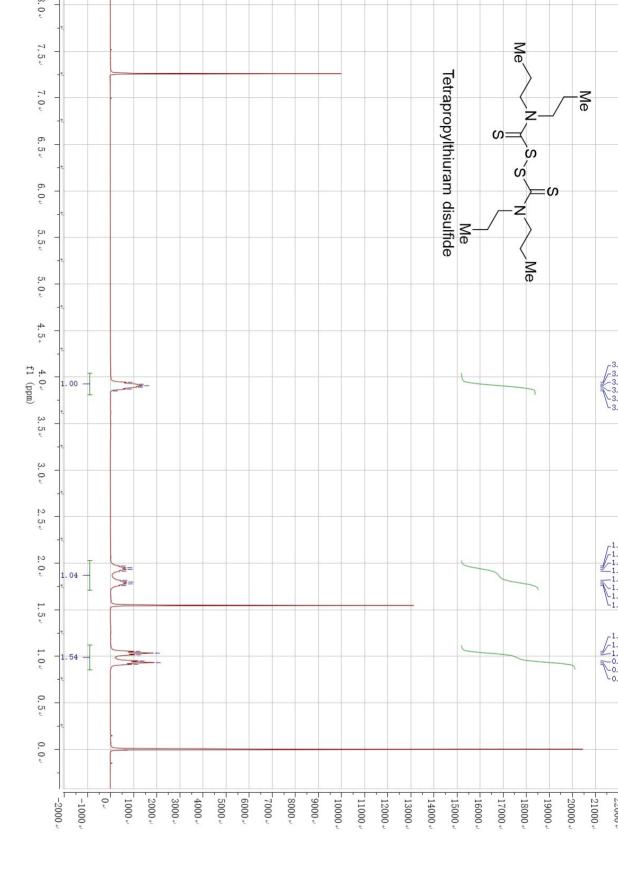
Singlet Oxygen (1O2) Trapping Experiment

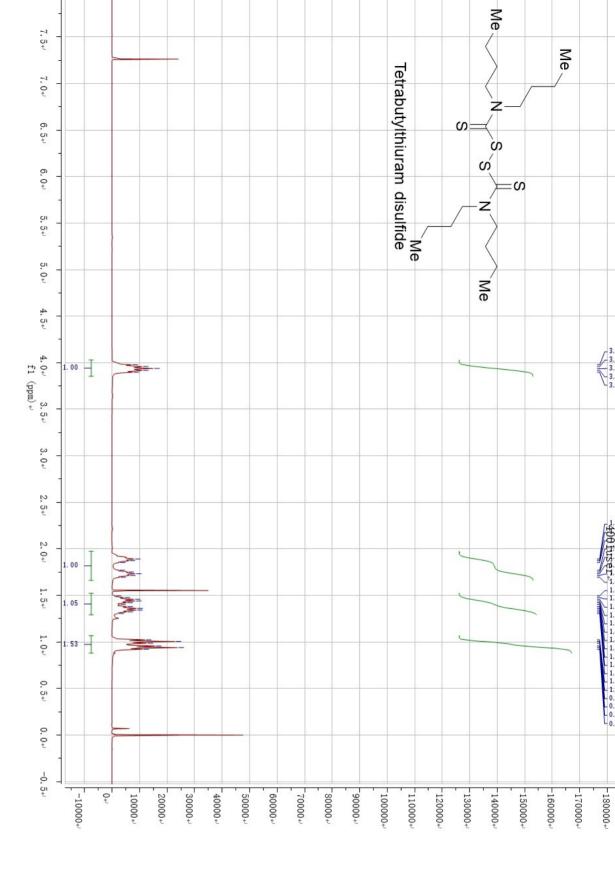
Eosin Y (14 mg, 0.02 mmol), 1,3-diphenylisobenzofuran (DPBF, 5 mg, 0.02 mmol) and ethanol (3 mL) were added in a vial upon stirring on a magnetic stirrer (IKA®-C-MAG HS 7) under irradiation from 13 W green LED strip lights at room temperature for 3.5 h ([Eosin Y] = 0.0066 M, [DPBF] = 0.0066 M). An oxygen balloon was attached to the vial. The distance between the reaction mixture and the light source was approximately 2 cm. A small fraction (100 μ L) of the reaction mixture was taken for UV-Vis (SHIMADZU #UV-2550) analyses at reaction times of 0 min, 60 min, 120 min and 210 min.

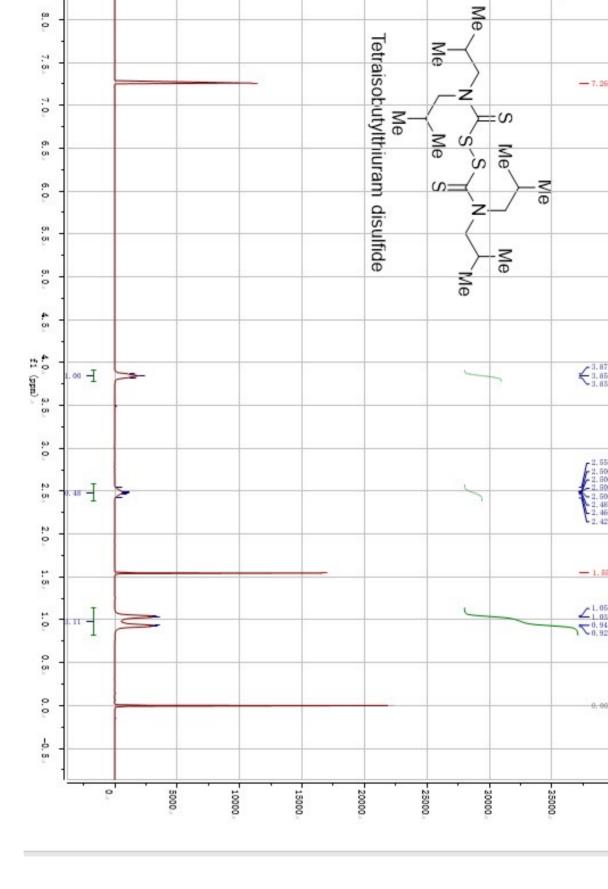
NMR Spectra (see next page)

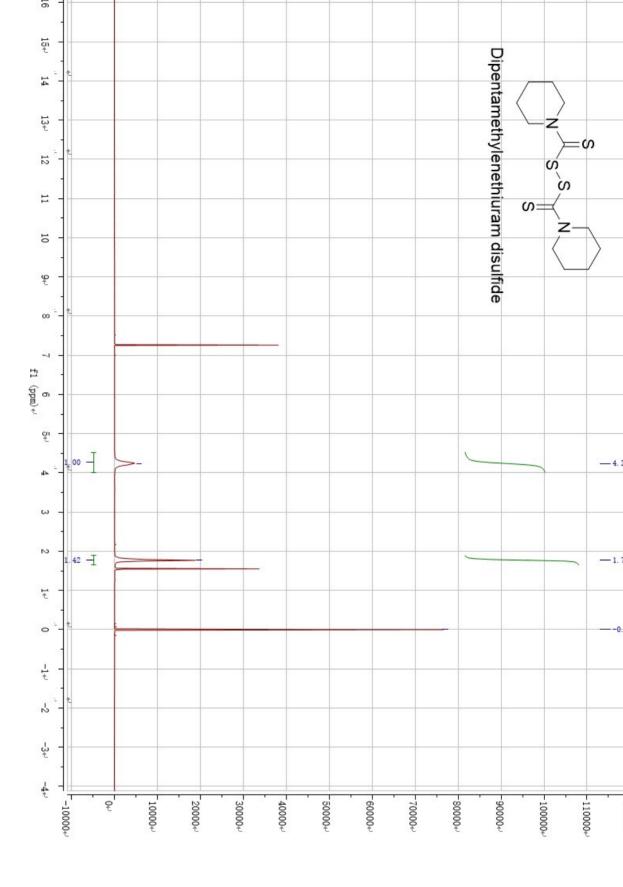


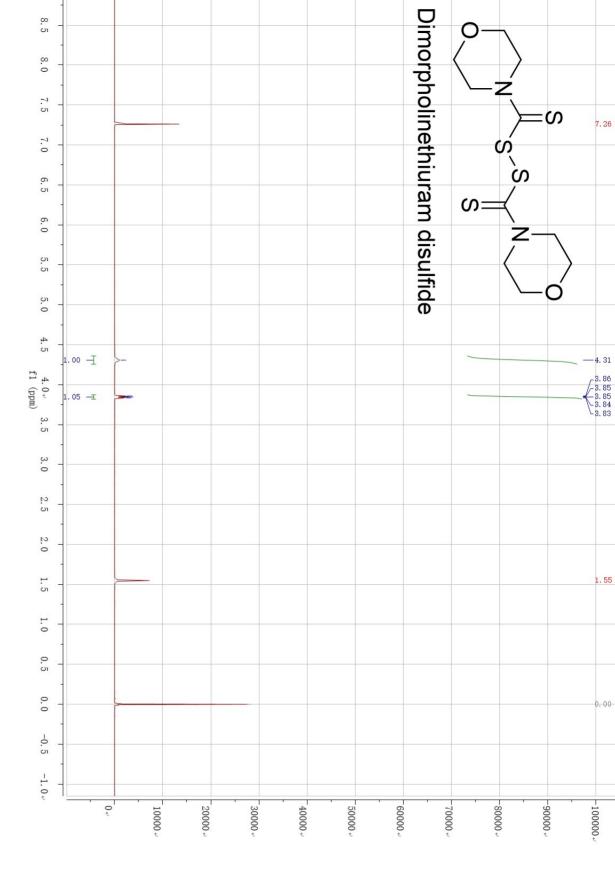


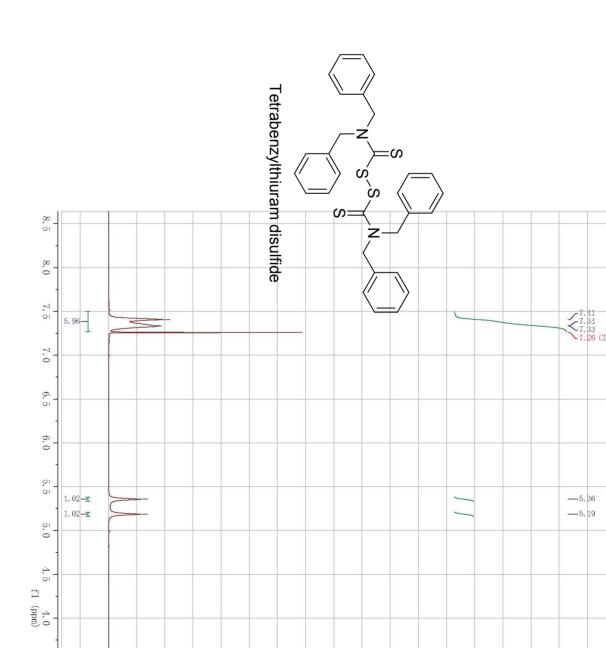


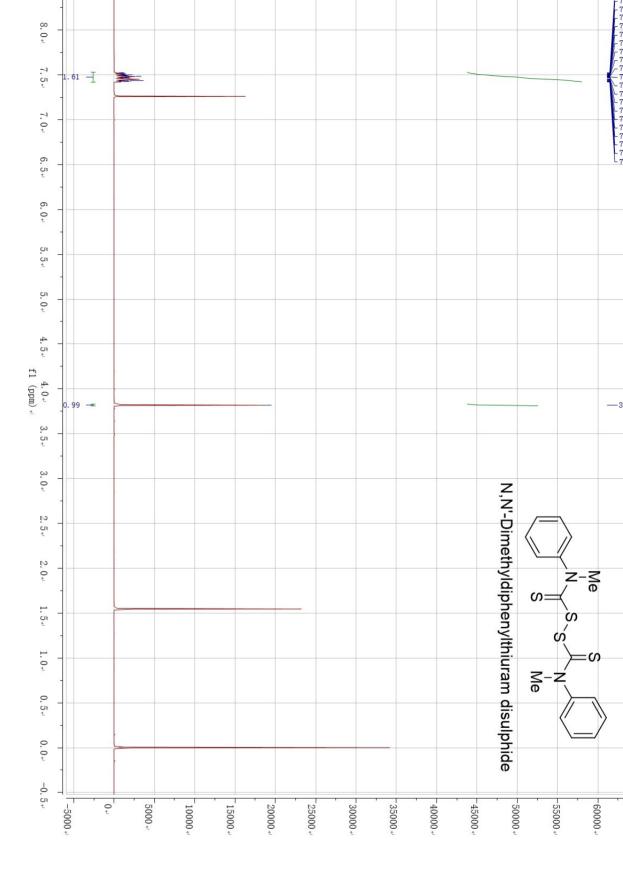


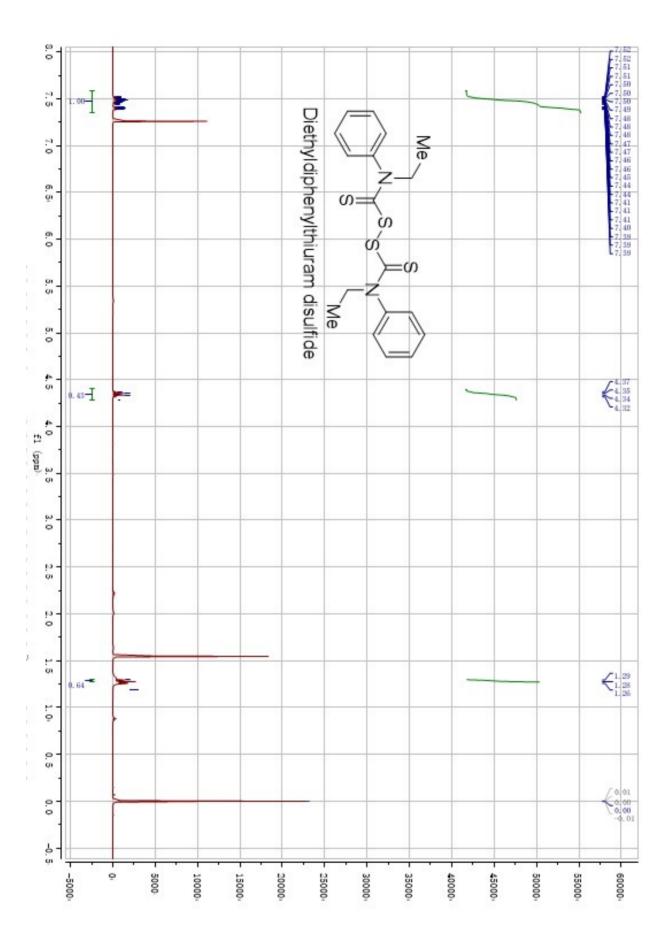








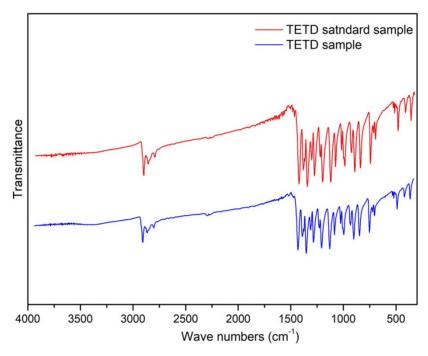




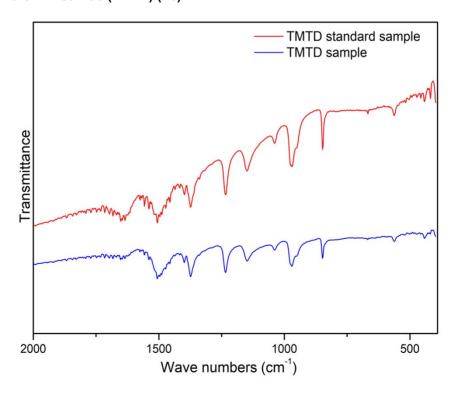
FT-IR

Characteristic wave number (cm $^{-1}$) for R-S-S-R: 705~570 & 520~500 (weak); for Ar-S-S-Ar: 500~430 (weak ~ medium).

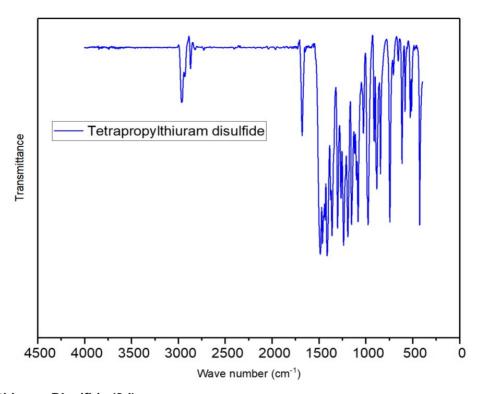
Tetraethylthiuram Disulfide (TETD) (2a)



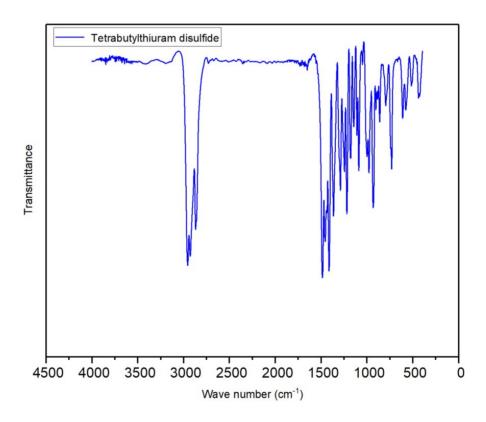
Tetramethylthiuram Disulfide (TMTD) (2b)



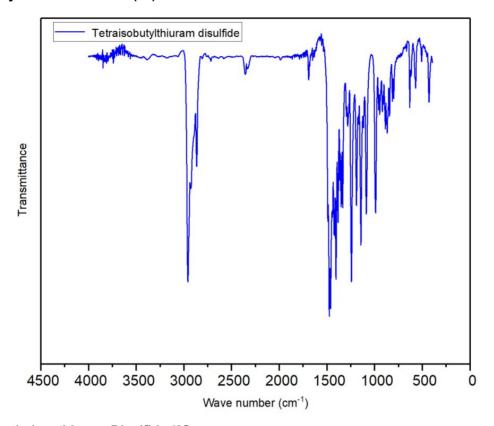
Tetrapropylthiuram Disulfide (2c):



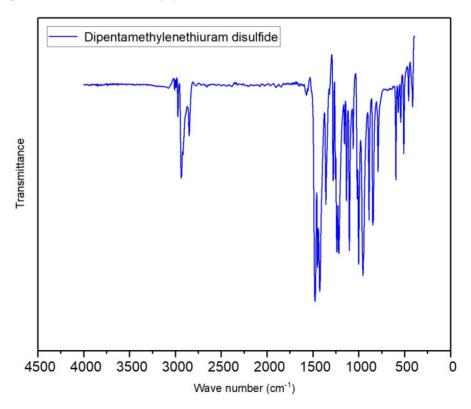
Tetrabutylthiuram Disulfide (2d):



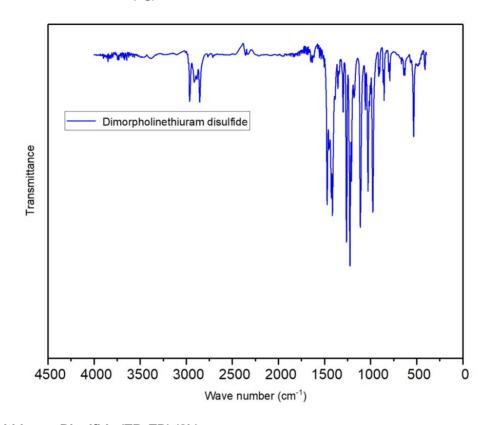
Tetraisobutylthiuram Disulfide (2e):



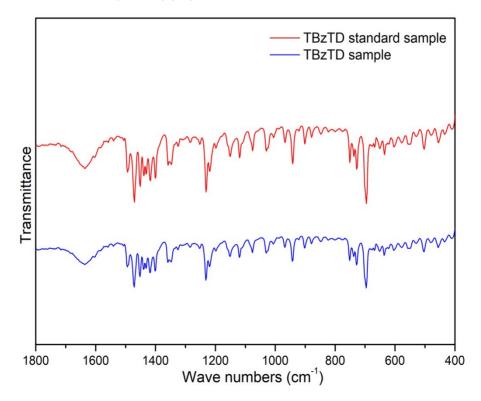
Dipentamethylenethiuram Disulfide (2f):



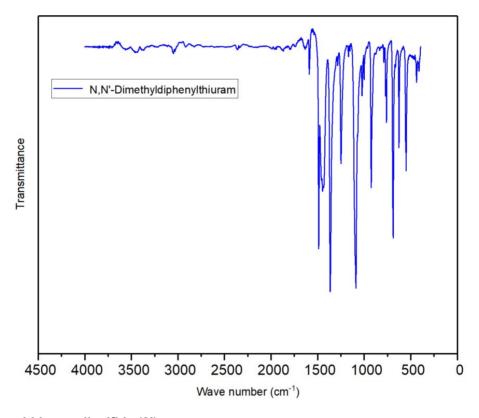
Dimorpholinethiuram Disulfide (2g):



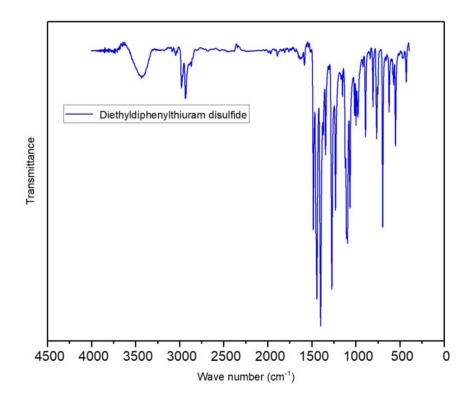
Tetrabenzylthiuram Disulfide (TBzTD) (2h):



N,N'-Dimethyldiphenylthiuram (2i):



Diethyldiphenylthiuram disulfide (2j):



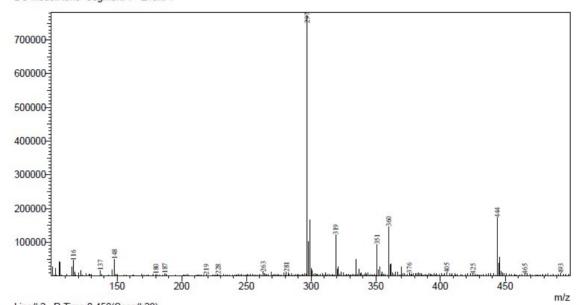
Mass spectra

Tetraethylthiuram Disulfide (2a): exact mass= 296.0

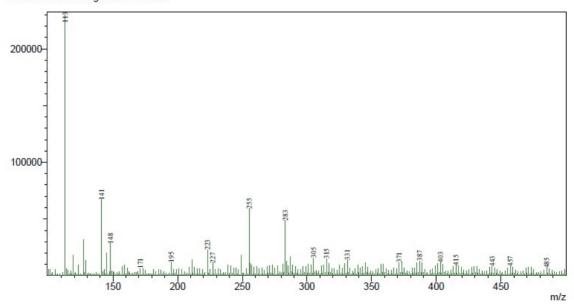
==== Shimadzu LabSolutions Data Report ====

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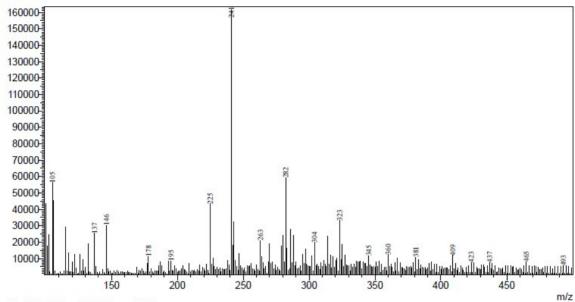


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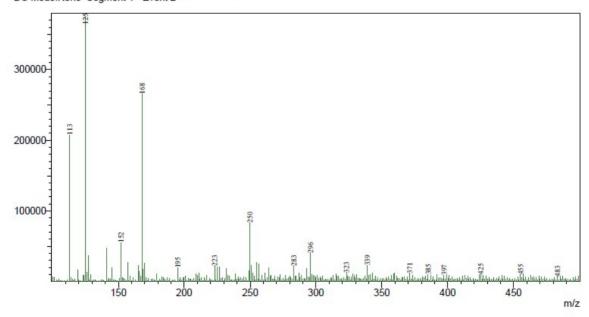


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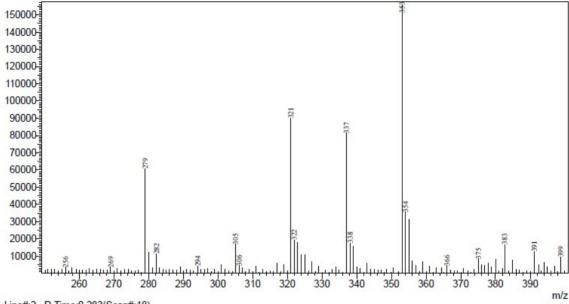


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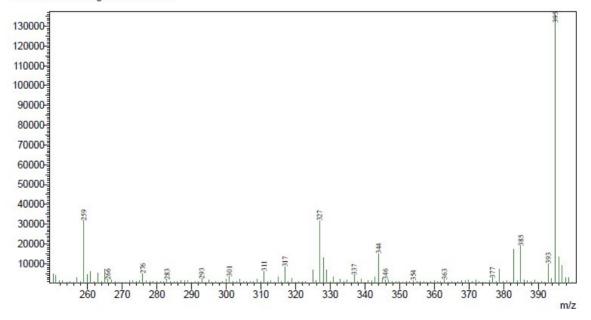


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BG Mode:None Segment 1 - Event 1

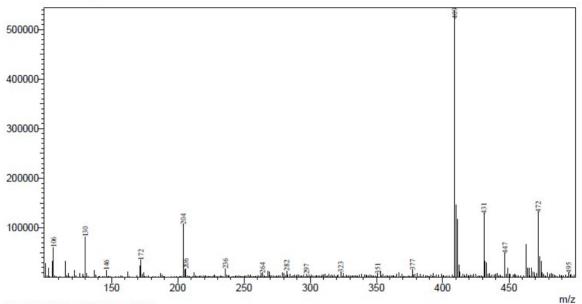


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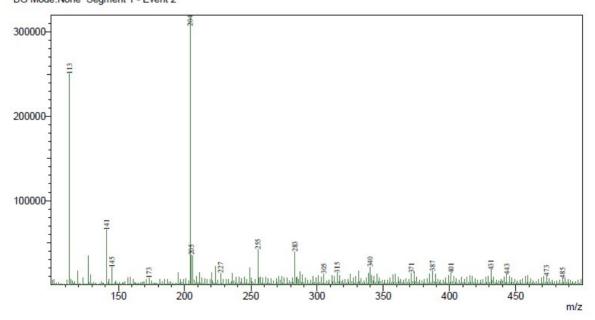


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BG Mode:None Segment 1 - Event 1

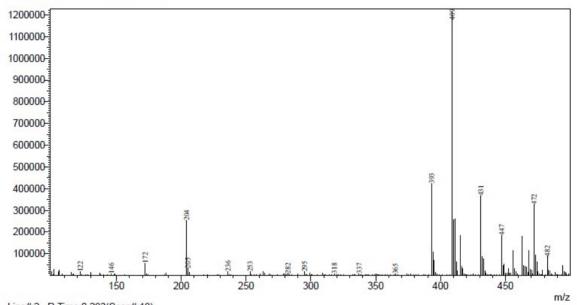


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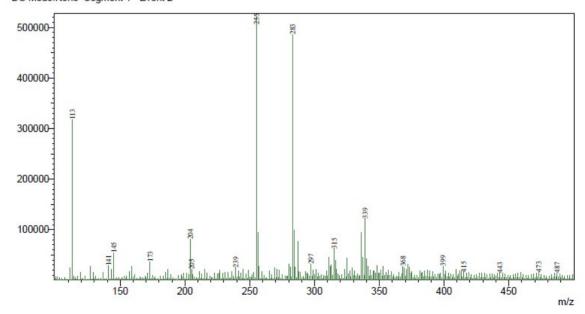


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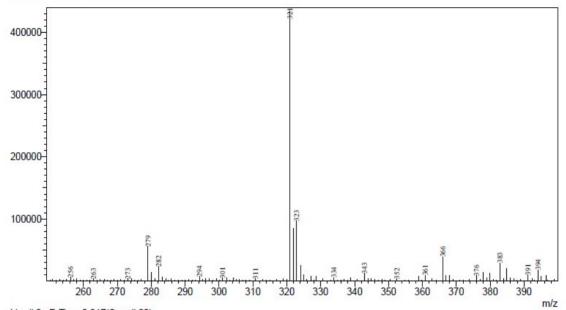


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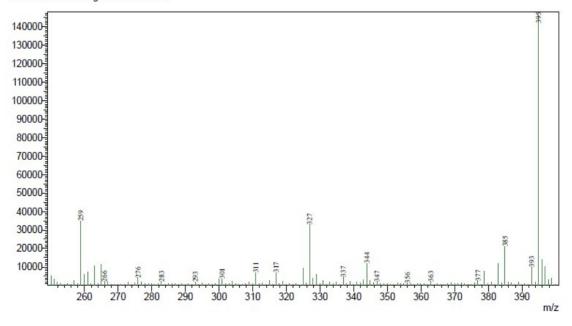


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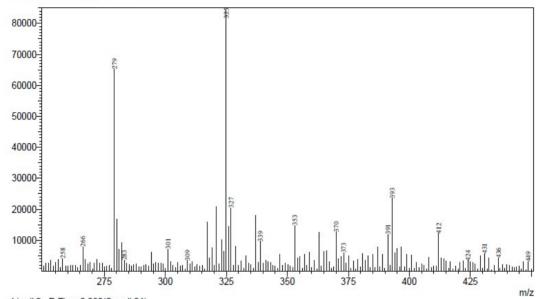


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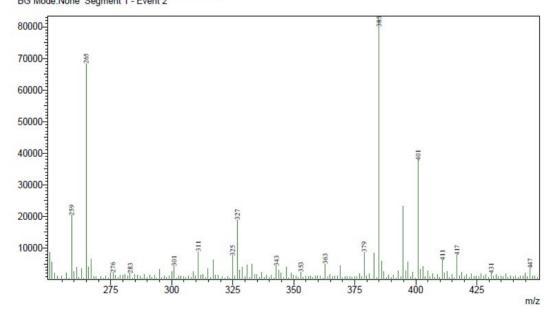


<Spectrum>

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BG Mode:None Segment 1 - Event 1

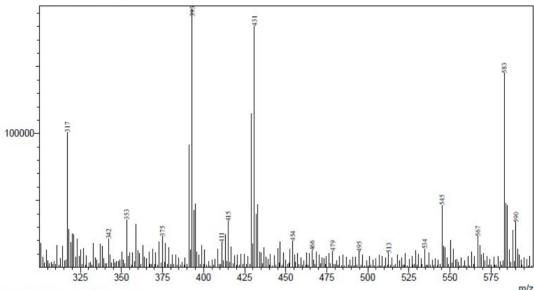


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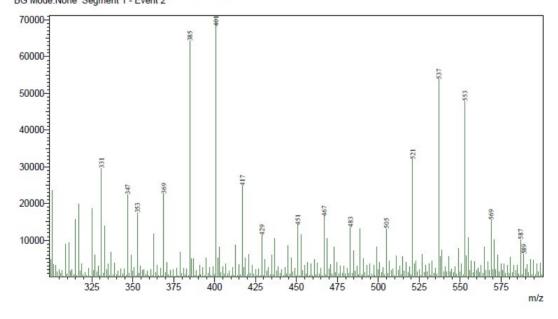


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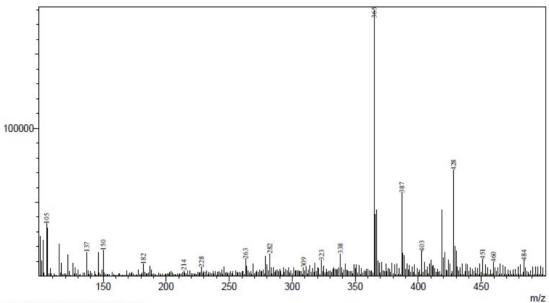


Line#:2 R.Time:0.250(Scan#:16) MassPeaks:301 RawMode:Single 0.250(16) BasePeak:401(70457) BG Mode:None Segment 1 - Event 2

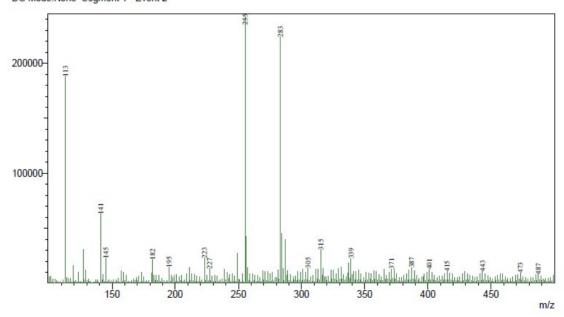


<Spectrum>

Line#:1 R.Time:0.400(Scan#:25)
MassPeaks:401
RawMode:Single 0.400(25) BasePeak:365(180094)
BG Mode:None Segment 1 - Event 1

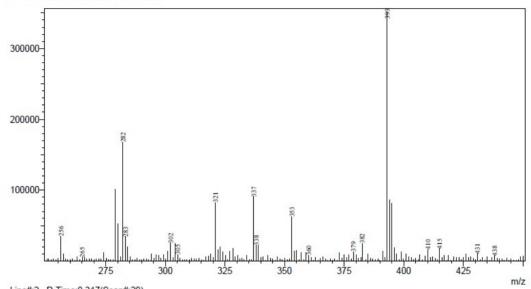


Line#:2 R.Time:0.416(Scan#:26) MassPeaks:400 RawMode:Single 0.416(26) BasePeak:255(242490) BG Mode:None Segment 1 - Event 2



<Spectrum>

Line#:1 R.Time:0.300(Scan#:19)
MassPeaks:201
RawMode:Single 0.300(19) BasePeak:393(352550)
BG Mode:None Segment 1 - Event 1



Line#:2 R.Time:0.317(Scan#:20) MassPeaks:201 RawMode:Single 0.317(20) BasePeak:395(155966) BG Mode:None Segment 1 - Event 2

