Supplementary Material :

Synthesis and Characterization of Novel Substituted Quinone Derivatives from the reactions of p-chloranil with N-, S-, O- Nuclephiles

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EXPERIMENTAL :

Material and Methods

Melting points were determined on a Buchi B-540 melting point apparatus and uncorrected. Microanalyses were performed on a Thermo Finnigan Flash EA 1112 Series elemental analyser. Infrared (IR) spectra were recorded in KBr pellets in Nujol mulls or fon a Perkin Elmer Precisely Spectrum One FTIR spectrometer. Fluorescence Spectra were run on a VARIAN Cary Eclipse Fluorescence Spectrophotometer. ¹H-NMR and ¹³C-NMR spectra were recorded in CDCl₃ on a Varian UNITY INOVA spectrometer operating at 500 MHz. Mass spectra were obtained on a Thermo Finnigan LCQ Advantage MAX LC/MS/MS spectrometer using ion-trap mass analyzer for ESI source.

Products were isolated by column chromatography on Silica gel (Fluka silica gel 60, particle size 63-200 µm). TLC plates silica 60F₂₅₄ (Merck, Darmstadt) and detection was carried out with ultraviolet light (254 nm). All reagents and solvents were of reagent-grade, obtained from commercial suppliers and used without further purification.

General procedure 1

p-Chloranil and thiol were stirred in chloroform as solvent (40mL). Triethylamine (1 mL) was added to reaction mixture slowly without heating, the mixture was stirred for 12 h. The color of the solution quickly changed, and the extent of the reaction was monitored by TLC. Chloroform (30 mL) was added to the reaction mixture. The organic layer was separated, wash with water (4 x 30 mL), and dried with Na₂SO₄. After the solvent was evaporated, the residue was purified by column chromatography on silica gel.

General procedure 2

Potassium carbonate (1.52 g) was dissolved in acetonitrile (30mL) and into the resulting solution, firstly *p*-chloranil and alcohol were added slowly in small portions. Without heating, the mixture was stirred for 12 h. The colour of the solution quickly changed, and the extent of the reaction was monitored by TLC. Chloroform (30 mL) was added to the reaction mixture. The organic layer was separated, wash with water (4 x 30 mL), and dried with Na₂SO₄. After the solvent was evaporated, the residue was purified by column chromatography on silica gel.

General procedure 3

Sodium carbonate (1.52 g) was dissolved in ethanol (30mL) and into the resulting solution, firstly *p*-chloranil and thiol were added slowly in small portions. Without heating, the mixture was stirred for 8 h. The colour of the solution quickly changed, and the extent of the reaction was monitored by TLC. Chloroform (30 mL) was added to the reaction mixture. The organic layer was separated, wash with water (4 x 30 mL), and dried with Na₂SO₄. After the solvent was evaporated, the residue was purified by column chromatography on silica gel.

General procedure 4

p-Chloranil and thiol were stirred in methanol as solvent (40mL). Triethylamine (1 mL) was added to reaction mixture slowly without heating, the mixture was stirred for 12 h. The color of the solution quickly changed, and the extent of the reaction was monitored by TLC. Chloroform (30 mL) was added to the reaction mixture. The organic layer was separated, wash with water (4 x 30 mL), and dried with Na₂SO₄. After the solvent was evaporated, the residue was purified by column chromatography on silica gel.

3-ethoxy-2,5,6-tris(octylthio)-1,4-benzoquinone (3a) :

Compound **3a** was synthesized by the reaction of *p*-chloranil (**1**) (0.5 g, 2.034 mmol) with *n*-octanethiol (**2a**) (0.47 ml, 4.066 mmol) according to the general procedure 3.

Dark brown oil; Yield: 0.303g. (25%); *R*_f = 0,520[Petroleum Ether/CHCl₃(1:1)]; IR (film,): ν 2926 (C-H), 1580 (C=C), 1653 (C=O) cm⁻¹; ¹H-NMR: δ 0.76-0.84 (m, 9H, -CH₃), 1.19-1.35 (m, 3H, -OCH₂CH₃), 1.47-1.54 (m, 36H, -CH₂), 2.93-3.08 (m, 6H, -SCH₂), 4.25 (m, -, -OCH₂);

^{13}C -NMR: δ 13.05, 14.77 (-CH₃), 21.61, 27.68, 27.69, 27.72, 28.10, 28.12, 28.14, 29.18, 29.45, 29.49, 30.77 (-CH₂), 39.30, 39.33 (-SCH₂), 68.73 (-OCH₂), 130.65, 141.30, 146.14, 155.65 (C_{arom}), 173.64, 177.69 (C=O); MS (+ESI): m/z 585 (M+H)⁺; C₃₂H₅₆O₃S₃ (M = 584.98 g/mol); Calcd.: C 65.70, H 9.65, S 16.44. Found: C 65.40, H 9.32, S 17.61.

3-ethoxy-2,5,6-tris(2-ethyl-hexanethio)-1,4-benzoquinone (3b) :

Compound **3b** was synthesized by the reaction of (0.5 g, 2.034 mmol) p-chloranil (**1**) with 2-ethylhexanethiol (**2b**) (0.594 mL, 4.060 mmol) according to the general procedure 3.

Dark brown oil; Yield: 0.113 g (11%). R_f =0.70 [CHCl₃]. IR (film): ν 2959, 2928, 2872, 2858 (C-H), 1655 (C=O), 1580 (C=C) cm⁻¹; ^1H -NMR: δ = 0.80 (s, 21H, -CH₃), 2.70-3.20 (m, 6H, -SCH₂), 1.19- 1.38 (m, 32H, -CH₂-CH₂), 4.25-4.50 (m, 2H, -OCH₂); ^{13}C -NMR: δ = 13.06, 14.86, 19.59 (-CH₃), 21.96, 24.33, 27.74, 31.11, 36.19 (-CH₂, -CH), 39.25, 39.33 (-SCH₂), 68.67 (-OCH₂), 155.79, 148.2, 147.03 (C_{arom}), 173.86, 177.63 (C=O); MS (+ESI): m/z 585 (M+H)⁺; C₃₂H₅₆O₃S₃ (M = 584.98 g/mol); Calcd.: C 65.70, H 9.65, S 16.44. Found: C 65.55, H 9.54, S 17.33.

2,6-bis(4-chlorobenzylthio)-3,5-diethoxy-1,4-benzoquinone (4e) and 2,3:5,6-tetra(4-chlorobenzylthio)-1,4-benzoquinone (5e) :

Compounds **4e** and **5e** were synthesized by the reaction of p-chloranil (**1**) (0.5 g, 2.034 mmol) with 4-chlorobenzyl mercaptan (**2e**) (0.53 ml, 4.066 mmol) according to the general procedure 3.

(4e): Brown oil; Yield: 0.106g. (10%); R_f = 0.468 [Petroleum Ether/CHCl₃(1:1)]; IR(film): ν 3078 (C-H_{arom}), 2980 (C-H_{aliph}), 1553 (C=C), 1654 (C=O) cm⁻¹; ^1H -NMR: δ 1.17-1.24 (t, J =6.35, 6H, -CH₃), 4.10 (m, 4H, SCH₂), 4.17 (m, 4H, -OCH₂), 7.02-7.17 (m, 8H, H_{arom}); ^{13}C -NMR: δ 14.68 (-CH₃), 36.84-37.27 (-SCH₂), 69.12 (-OCH₂), 127.70, 127.72, 127.79, 128.10, 129.20, 129.24, 129.32, 129.49, 132.22, 134.60, 140.79, 145.58, 156.58 (CH_{arom}, C_{arom}), 173.76, 177.35 (C=O); MS (+ESI): m/z 508 (M)⁺; C₂₄H₂₂Cl₂O₄S₂ (M = 509.47 g/mol); Calcd.: C 56.68, H 4.35, S 12.59. Found: C 56.88, H 3.96, S 9.71.

(5e): Dark brown oil; Yield: 0.558g. (36%); R_f = 0.540 [Petroleum Ether/CHCl₃(1:1)]; IR(film): ν 3027 (C-H_{arom}), 2919 (C-H_{aliph}), 1547 (C=C), 1659 (C=O) cm⁻¹; ^1H -NMR : δ 3.78-4.34 (m, 8H, -SCH₂), 6.52-7.39 (m, 16H, H_{arom}); ^{13}C -NMR : δ 36.95-37.67 (-SCH₂), 127.54, 127.67, 127.77, 127.81, 127.86, 129.10, 129.16, 129.22, 129.25, 129.51, 129.63, 132.40, 132.51, 132.54, 132.63, 134.18, 134.32, 134.49, 134.63, 144.51, 145.05

(CH_{arom}, C_{arom}), 175.18 (C=O); MS (+ESI): m/z 734 (M)⁺; C₃₄H₂₄Cl₄O₂S₄ (M = 734.63 g/mol); Calcd.: C 55.59, H 3.29, S 17.46. Found: C 55.35, H 3.40, S 15.69.

2,3:5,6-tetrakis(4-tertbutylphenylthio)-1,4-benzoquinone (5f) :

Compound **5f** was synthesized by the reaction of p-chloranil (**1**) (0.5 g, 2.034 mmol) with 4-tert-butyl thiophenol (**2f**) (0.68 ml, 4.066 mmol) according to the general procedure 3.

Black powder; Yield: 0.764g. (%49.21); m.p.=162-164⁰C; R_f=0,528[Petroleum Ether/CHCl₃(1:1)]; IR(KBr): ν 3078 (C-H_{arom}), 2965 (C-H_{aliph}), 1595 (C=C), 1671 (C=O) cm⁻¹; ¹H-NMR : δ 1.21 (s, 36H, -CH₃), 7.06-7.16 (m, 16H, H_{arom}); ¹³C-NMR : δ 31.51 (-CH₃), 126.35-129.49, 131.40, 146.69, 151.32 (CH_{arom}, C_{arom}), 175.33 (C=O); MS (-ESI): m/z 765 (M+H)⁺; C₄₆H₅₂O₂S₄ (M = 765.16 g/mol); Calcd.: C 72.21, H 6.85, S 16.76. Found: C 72.65, H 6.37, S 15.33.

2,3:5,6-tetrakis(2,5-dichlorophenylthio)-1,4-benzoquinone (5g):

Compound **5g** was synthesized by the reaction of p-chloranil (**1**) (0.5 g, 2.034 mmol) with 2,5-dichlorobenzenethiol (**2f**) (1.10 g, 6.066 mmol) according to the general procedure 3.

Orange powder; Yield: 0.310 g (20%); m.p. 201-202 °C; R_f = 0.70 [Petroleum Ether/CHCl₃(1:1)]; IR (KBr): ν 3084,40 (C_{arom}), 2922,89 ve 2851,64 (CH), 1670,10 (C=O), 1566.8 (C=C) cm⁻¹; ¹H-NMR : δ = 7.10 -7.64 (m, 12H, H_{arom}); ¹³C-NMR : δ = 125.88-127.75, 130.13 - 133.47, 143.86 (CH_{arom}, C_{arom}), 171.78 (C=O); MS (-ESI): m/z 816 (M)⁺. C₃₀H₁₂Cl₈O₂S₄ (M = 816.30 g/mol); Calcd.: C 44.14, H 1.48, S 15.71. Found: C 44.10, H 1.54, S 16.42.

3-Chloro-2,5,6 tris(2-ethyl-hexanethio)- 1,4-benzoquinone (6b) :

Compound **6b** was synthesized by the reaction of p-chloranil (**1**) (0.5 g, 2.034 mmol) with 2-thylhexanethiol (**2b**) (0.59 mL, 4.066 mmol) according to the general procedure 2.

Dark red oil; Yield: 0.070 g (6%). R_f = 0.62 [CHCl₃] IR (KBr): ν 2959, 2928, 2872, 2858 (C-H), 1671 (C=O), 1514 (C=C) cm⁻¹; ¹H-NMR : δ = 0.75-0.85 (m, 18H, -CH₃), 1.12-1.47 (m, 33H, -CH₂, -CH), 2.76 (d, J=5.97, 2H, -SCH₂), 3.12 (d, J=6.34, 4H, -SCH₂); ¹³C-NMR : δ = 13.10 (-CH₃), 21.89, 24.25, 27.63, 31.01, 37.14 (-CH₂-CH₂), 39.16, 39.22 (-SCH₂), 175.06, 167.08 (C=O); MS (+ESI): m/z 575 (M+H)⁺; C₃₀H₅₁ClO₂S₃ (M = 575.37 g/mol); Calcd.: C 62.62, H 8.93, S 16.72. Found: C 62.56, H 9.30, S 18.40.

3-Chloro-2,5,6-tris(ethylthio)-1,4-benzoquinone (6c) :

Compound **6c** was synthesized by the reaction of p-chloranil (**1**) (0.5 g, 2.034 mmol) with ethanethiol (**2c**) (0.30 mL, 4.056 mmol) according to the general procedure 1.

Dark brown oil; Yield: 0.175g. (27%); $R_f = 0.505$ [Petroleum Ether/ CHCl_3 (1:1)]; IR(KBr): ν 2965 (C-H), 1547 (C=C), 1659 (C=O) cm^{-1} ; $^1\text{H-NMR}$: δ 1.24 (t, $J=6.35$, 9H, $-\text{CH}_3$), 3.05-3.22 (m, 6H, $-\text{SCH}_2$); $^{13}\text{C-NMR}$: δ 14.30-14.44 ($-\text{CH}_3$), 27.23-28.34 ($-\text{SCH}_2$), 135.94, 143.58, 144.36, 146.58 (C_{arom}), 170.24, 174.25 (C=O); MS (+ESI): m/z 323 ($\text{M}+\text{H}$) $^+$; $\text{C}_{12}\text{H}_{15}\text{ClO}_2\text{S}_3$ (M = 322.89 g/mol); Calcd.: C 44.64, H 4.68, S 29.79. Found: C 44.11, H 5.26, S 27.95.

3-Chloro-2,5,6-tris(propylthio)-1,4-benzoquinone (6d) :

Compound **6d** was synthesized by the reaction of p-chloranil (**1**) (0.5 g, 2.034 mmol) with n-propanethiol (**2d**) (0.68 mL, 4.066 mmol) according to the general procedure 1.

Dark brown oil; Yield: 0.174g. (23%); $R_f = 0.606$ [Petroleum Ether/ CHCl_3 (1:1)]; IR(film): ν 2962 (C-H), 1548 (C=C), 1660 (C=O) cm^{-1} ; $^1\text{H-NMR}$: δ 0.94 (t, $J=7.32$, 9H, $-\text{CH}_3$), 1.57-1.61 (m, 6H, $-\text{CH}_2$), 2.99-3.14 (m, 6H, $-\text{SCH}_2$); $^{13}\text{C-NMR}$: δ 12.20-12.26 ($-\text{CH}_3$), 22.77-22.97 ($-\text{CH}_2$), 34.65-35.92 ($-\text{SCH}_2$), 136.20, 143.82, 144.90, 146.84 (C_{arom}), 170.22, 174.29 (C=O); MS (+ESI): m/z 364 (M) $^+$; $\text{C}_{15}\text{H}_{21}\text{ClO}_2\text{S}_3$ (M = 364.97 g/mol); Calcd.: C 49.36, H 5.80, S 26.36. Found: C 49.62, H 6.44, S 24.79.

3,6-Dichloro-2,5-bis(6-methoxypiridine-3-amino)-1,4-benzoquinone (8h) :

Compound **8h** was synthesized by the reaction of p-chloranil (**1**) (0.5 g, 2.034 mmol) with 5-amino-2-methoxypiridine (**7h**) (0.504 g, 4.066 mmol) according to the general procedure 1.

Red solid; Yield: 0.120 g (% 14.25); mp: 248-249 $^{\circ}\text{C}$; $R_f = 0.40$ [Chloroform/Petroleum ether(1:1)]. IR (KBr): ν 3291.07 (N-H), 3059.63 (CH_{arom}), 2920.32 (C-H), 1689 (C=O), 1569 (C=C), 1203 (C-O) cm^{-1} ; $^1\text{H-NMR}$: δ = 3.07 (s, 6H, $-\text{OCH}_3$); 3.83 (m, 2H, $-\text{NH}$), 7.50 (d, $J=6.35$, 2H, H_{arom}), 7.65 (d, $J=6.35$, 2H, H_{arom}); $^{13}\text{C-NMR}$: δ = 68.57($-\text{OCH}_3$), 119.1, 128.2, 126.70, 143.1, 166.74 (CH_{arom} , C_{arom}), 182.5 (C=O); MS (-ESI): m/z 421 ($\text{M}+\text{H}$) $^+$; $\text{C}_{18}\text{H}_{14}\text{Cl}_2\text{N}_4\text{O}_4$ (M= 421.23 g/mol); Calcd.: C 51.32, H 3.35, N 13.30. Found: C 50.44, H 3.50, N 13.45.

3-[5-chloro-2-methoxy-4-(2-methoxycarbonyl-ethylamino)-3,6-dioxo-cyclohexa-1,4-dienylamino]-propionic acid methyl ester (9i)

Compound **9i** was synthesized by the reaction of p-chloranil (**1**) (0.5 g, 2.034 mmol) with Beta-Alanine methyl hydrochloride (**7i**) (0.570 g, 4.066 mmol) according to the general procedure 4.

Brown solid; Yield 0.510 g (68%); m.p: 167-168 °C; R_f =0.60 [CHCl_3]; IR (KBr): ν 3242.53 (-N-H), 2957.78 (C-H), 1665.28 (C=O), 1581.79 (C=C) cm^{-1} ; $^1\text{H-NMR}$: δ = 1.97 (s, 2H, -NH), 2.65 (t, J = 6.35, 4H, -C=O-CH₂), 3.68 (t, J = 6.35, 4H, -N-CH₂), 4.09 (q, J = 6.84, 9H, -O-CH₃); $^{13}\text{C-NMR}$: δ = 34.29, 38.9 (-N-CH₂), 59.95 (-OCH₃), 123.8, 167.73 (C_{arom}), 177.85 ppm (C=O); MS (-ESI): m/z (%) 377.0 [M-H]⁻ (100); C₁₅H₁₉ClN₂O₇ (M = 374.77 g/mol); Calcd.: C 48.07, H 5.11, N 7.47. Found: C 48.15, H 6.09, N 7.56.

2,3,4,5,9,10,11,12-Oktahidro-1,6,8,13-tetratia-benzo[1,2;4,5]disiklooktan-7,14-dion (11)

Compound **11** was synthesized by the reaction of p-chloranil (**1**) (0.5 g, 2.034 mmol) with 1,4-butanedithiol (**10**) (0.490 mL, 4.066 mmol) according to the general procedure 1.

Brown solid; Yield: 0,085 g (12%); m.p: 114-115 °C; R_f = 0.530 [CHCl_3]. IR (KBr): ν 2921.14, 2849.98 (C-H), 1648.11 (C=O), 1491.58 (C=C) cm^{-1} ; $^1\text{H-NMR}$: δ = 1.97 – 2.00 (m, 8H, -CH₂), 3.43– 3.44 (m, 8H, -SCH₂); $^{13}\text{C-NMR}$: δ = 27.03, 35.33 (-S-CH₂, -CH₂), 144.71 (C_{arom}), 177.29 (C=O); MS (-ESI): m/z 344 (M)⁺; C₁₄H₁₆O₂S₄ (M_w = 344.54 g/mol); Calcd.: C 48.80, H 4.68, S 37.23. Found: C 48.54, H 4.93, S 35.50.

10,11,22,23 Tetrakloro 2,7,14,19-tetratia-trisiklo[18,4,0,0^{8,13}]

tetrakosa-1(20),8(13),10,22-tetraen-9,12,21,24-tetraone (12)

Compound **12** was synthesized by the reaction of p-chloranil (**1**) (0.5 g, 2.034 mmol) with 1,4-butanedithiol (**10**) (0.250 g, 2.045 mmol) according to the general procedure 1.

Brown solid; Yield: 0.069 g (6%); m.p: 128-129 °C; R_f =0.420 [CHCl_3]; IR (KBr): ν 2959.25, 2928.02, 2858.68 (C-H), 1655.63 (C=O), 1540.43 (C=C) cm^{-1} ; $^1\text{H-NMR}$: δ = 2.60-3.5 (m, 8H, -SCH₂), 1.60-2.01 (m, 8H, -CH₂); $^{13}\text{C-NMR}$: δ = 28.34, 28.67, 29.04, 29.63 (-SCH₂), 124.2, 153.1 (C_{arom}), 178.1 (C=O); MS (-ESI): m/z 589 (M)⁻; C₂₀H₁₆Cl₄O₄S₄ (M = 590.41 g/mol); Calcd.: C 40.69, H 2.73, S 21.72. Found: C 40.50, H 2.93, S 24.32.

3,6-Dichloro-2,5 bis(1-(4-nitrophenyl)piperazinyl)-1,4-benzoquinone (**14**) :

Compound **14** was synthesized by the reaction of p-chloranil (**1**) (0.5 g, 2.034 mmol) with 1-(4-nitrophenyl)piperazine (**13**) (0.420 g, 4.066 mmol) according to the general procedure 1.

Dark green solid; Yield: 0.256 g (22%); m.p.: 122-123 °C; R_f = 0.740 [CHCl_3] IR (KBr): ν 2928, 2872.40 (C-H), 1645 (C=O), 1552 (C=C); $^1\text{H-NMR}$: δ = 2.88 (m, 8H, N-CH₂), 3.86 (m, 8H, N-CH₂), 6.84 (m, 4H, -H_{arom}), 8.12 (m, 4H, -H_{arom}), $^{13}\text{C-NMR}$: δ = 47.47, 49.81 (-N-CH₂-), 115.5–122.84 (C_{arom}), 126.28 (=C-Cl), 147.49 (=C-NO₂), 155.01 (=C-N-), 177.32 ve 176.10 ppm (C=O); MS (+ESI): m/z 587 (M+H); C₂₆H₂₄Cl₂N₆O₆ (M = 587.41 g/mol); Calcd.: C 53.16, H 4.12, N 14.31. Found: C 53.04, H 4.25, S 15.34.

2-(4-chlorobenzylthio)-3,5,6-tripropylthio-1,4-benzoquinone (**16a**) :

Compound **16a** was synthesized by the reaction of 2-chloro-3,5,6-tripropylthio-1,4-benzoquinone (**6d**) (0.3 g (0.823 mmol) with 4-chlorobenzyl mercaptan (**15e**) (0.11 ml, 0.826 mmol) according to the general procedure 1.

Dark brown oil; Yield: 0.347g. (%57.39); R_f = 0,530[Petroleum Ether/ CHCl_3 (2:1)]; IR(film.): ν 3027 (C-H_{arom}), 2962 (C-H_{aliph}), 1573 (C=C), 1651 (C=O) cm^{-1} ; $^1\text{H-NMR}$: δ 0.94 (t, J =7.32, 9H, -CH₃), 1.39-1.56 (m, 6H, -CH₂), 2.81-3.00 (m, 6H, -SCH₂), 4.05-4.20 (m, 2H, -SCH₂), 7.00-7.21 (m, 4H, H_{arom}); $^{13}\text{C-NMR}$: δ 12.23-12.28 (-CH₃), 22.61-22.97, 35.13, 35.53, 36.84 (-SCH₂, -CH₂), 127.71, 127.78, 129.32, 129.34, 132.44, 142.40, 145.08, 145.54, 147.56 (CH_{arom}, C_{arom}), 173.23, 173.43 (C=O); MS (+ESI): m/z 488 (M+2H)⁺; C₂₂H₂₇ClO₂S₄ (M = 487.16 g/mol); Calcd.: C 54.24, H 5.59, S 26.33. Found: C 54.86, H 4.55, S 27.30.

2-(4-tertbutylphenylthio)-3,5,6-tripropylthio-1,4-benzoquinone (**16b**) :

Compound **16b** was synthesized by the reaction of 2-chloro-3,5,6-tripropylthio-1,4-benzoquinone (**6d**) (0.3 g (0.823 mmol) with 4-tertbutyl thiophenol (**15f**) (0.14 ml (0.819 mmol) according to the general procedure 1.

Dark brown oil; Yield: 0.380g. (%62.85); R_f = 0,610[Petroleum Ether/ CHCl_3 (2:1)]; IR(film): ν 3027 (C-H_{arom}), 2962 (C-H_{aliph}), 1594 (C=C), 1661 (C=O) cm^{-1} ; $^1\text{H-NMR}$: δ 0.77-0.97 (m, 9H, -CH₃), 1.50-1.59 (m, 6H, -CH₂), 2.57-3.03 (t, J =7.32, 6H, -SCH₂), 7.19-7.31 (m, 4H, H_{arom}); $^{13}\text{C-NMR}$: δ 12.16-12.30 (-CH₃), 22.55-22.99 (CH₂), 34.96-36.33 (-SCH₂), 125.11, 125.17, 128.66, 128.77, 128.81, 143.95, 145.08, 147.25, 150.23 (CH_{arom}, C_{arom}), 172.60,

173.30 (C=O); MS (+ESI): m/z 495 (M+H)⁺; C₂₅H₃₄O₂S₄ (M_w = 494.80 g/mol); Calcd.: C 60.69, H 6.93, S 25.92. Found: C 60.21, H 6.97, S 27.60.

Compound 3a :

Compound 3b :

Compounds 4e and 5e :

Compound 5f :

Compound 5g :

Compound 6b :

Compound 6c :

Compound 6d :

Compound 8h :

Compound 9i :

Compound 11 :

Compound 12 :

Compound 14 :

Compound 16a :

Compound 16b: