Supporting Information

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Antioxidants and α-Glucosidase Inhibitors from

*Lactuca serriola* L.

Nouran H. Abdel Fatah¹, Yhiya Amen¹, Fatma M. Abdel Bar¹,², Ahmed F. Halim¹ and Hassan-Elrady A. Saad¹

¹Department of Pharmacognosy, Faculty of Pharmacy, Mansoura University, Mansoura 35516, Egypt
²Department of Pharmacognosy, Faculty of Pharmacy, Prince Sattam Bin Abdulaziz University, Al-Kharj 11942, Saudi Arabia

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1. Experimental and Chemistry

1.1. General experimental

Infra-red spectra were recorded on IR spectrophotometer (Perkin-Elmer 1430 ratio recording). HR-ESI mass spectra were determined using LC-TOF-MS (Shimadzu, Tokyo, Japan), HR-FAB-MS was determined using JEOL JMS 700 spectrophotometer (JEOL, Japan). NMR spectra was recorded on Bruker Ascend™ spectrometer (Bruker Daltonics, Bremen, Germany) at 400 MHz for $^1$H and 100 MHz for $^{13}$C, Bruker Advance DPX spectrometer (Bruker Daltonics Inc., MA, USA) at 600 MHz for $^1$H and 200 MHz for $^{13}$C and JEOL Eclipse NMR spectrometer at 500 MHz for $^1$H; 125 MHz for $^{13}$C where Chemical shifts were obtained I part per million (ppm) on the δ scale with reference to the TMS resonance. Thin layer chromatography was performed on precoated silica gel 60 GF254 (20× 20 cm, 0.2 mm thick) on aluminum sheets and precoated RP-C18 F254 plates (5 x 7.5 cm x 0.2 mm thick) on aluminium sheets (Merck Co., Darmstadt, Germany). Column chromatography was carried out using silica gel G 60-230 (Merck, Germany) packed by the wet method in the stated solvent, reversed phase chromatography using phase-bonded octadecylsilyl-silica gel (RP-C18, Merck, Germany), Sephadex LH 20 (Pharmacia Fine Chemicals, Sweden) and Diaion HP 20 (Mitsubishi Chemical Corporation, Japan). All the solvents used in column chromatography were purchased from El-Nasr Co. for Pharmaceutical Chemicals, Egypt. Azino-bis-(3-ethyl benzthiazoline-6-sulfonic acid) (ABTS), DPPH (2,2-diphenyl-1-picryl-hydrayl-hydrate), TPTZ (2,4,6-tripyridyl-S-triazine), Trolox (6-hydroxy-2,5,7,8-tetramethyl chromane 2-carboxylic acid), $\alpha$-glucosidase (Saccharomyces cerevisiae) and 3, 5 di-nitro salicylic acid (DNS) were obtained from sigma co., St. Louis, USA. Ascorbic acid (Cevarol®) tablets was obtained from Memphis Pharmaceutical Co., Cairo, Egypt. P-nitro-phenyl-$\alpha$-D-glucopyranoside (p-NPG), sodium carbonate (Na$_2$ CO$_3$), sodium dihydrogen phosphate, di-sodium hydrogen phosphate were purchased from Hi-Media, Mumbai, India. All the other chemicals and reagents used in the experiments were of analytical grade and were purchased from El-Nasr Co. for Pharmaceutical Chemicals, Egypt.

1.2. Extraction and isolation:

The air dried powdered aerial parts of Lactuca serriola at the flowering stage (1Kg) were extracted by maceration with methanol 90% (7 × 2 L). After removal of the solvent under reduced pressure, the extract (180 g; 18% yield) was partitioned by petroleum ether, methylene chloride, ethyl acetate, and finally by n-butanol. The pet. ether fraction (34.94 g) was separated by column chromatography (CC) on silica gel column (78 × 4.5 cm, 500 gm) using a stepwise gradient elution from 100% pet. ether to 100% EtOAc. The effluent was collected in 250 mL fractions into (Fr. 1-173). Fraction A (Fr. 26-29) eluted with pet. ether: EtOAc (92: 8) was left for crystallization to yield 1 (2.3 g). Fraction B (Fr. 39-44) eluted with pet. ether: EtOAc (90:10) was allowed to crystallize using CH$_2$Cl$_2$/MeOH (2:1) mix. to
yield 2 (120 mg). Fraction C (Fr. 83-86) eluted with pet. ether: EtOAc (82:18) was left for crystallization to give powder of 3 (12 mg). Fraction D (Fr. 130-140; 400 mg) eluted with pet. ether: EtOAc (60:40) was rechromatographed over silica gel column (40 × 1.5 cm) with CH₂Cl₂: EtOAc (88:12) to give 4 (26 mg). Fraction E (Fr. 167-169) eluted with pet. ether: EtOAC (10:90) was left for crystallization to give 5 (8 mg).

The methylene chloride fraction (10.42 g) was chromatographed using silica gel column (100 × 3.5 cm, 375 gm) by gradient elution from 100% CH₂Cl₂ to 100% MeOH. The effluent was collected in 100 mL fractions into (Fr. 1- 287). Fraction F (Fr. 101-116; 263.6 mg) eluted with CH₂Cl₂: MeOH (98:2) was applied repeatedly over silica gel column (40 × 1.5 cm) with CH₂Cl₂: EtOAc (90:10) to give 6 (12 mg). Fraction G (Fr. 122-141; 231 mg) eluted with CH₂Cl₂: MeOH (98:2) was rechromatographed over silica gel column (35 × 1.5 cm) in isocratic manner using methylene chloride (100%) and collecting 15 mL each fraction to elute 1 (subfraction 21-25) & 2 (subfraction 80-100). These subfractions were further purified over sephadex LH20 column (40 × 1 cm) with MeOH to give 8 (2 mg) & 9 (11 mg) respectively. Fraction H (Fr. 185-213) eluted with CH₂Cl₂: MeOH (97:3) was washed with CH₂Cl₂ and MeOH to yield powder of 7 (25 mg).

The EtOAc fraction (15.47 g) was loaded over silica gel column (90 × 3.5 cm, 300 gm) starting with pet. ether: EtOAc 80:20% then increasing gradient to 100% EtOAc to 100% MeOH. The effluent was collected in 250 mL fractions into (Fr. 1- 168). Fraction I (Fr. 10-14; 785.6 mg) eluted with pet. ether: EtOAc (60:40) was applied over silica gel column (38 × 1.5 cm) to elute (1'-3') subfractions with isocratic elution using CH₂Cl₂: EtOAc (93:7) and collecting 15 mL each fraction. Subfraction 1' (70-78) was further purified on sephadex LH20 column (22 × 1 cm) with MeOH (97:3) to yield 10 (11 mg). Sephadex LH20 column (20 × 1 cm) was used for subfraction 2'(80-90) purification to yield 11 (4 mg) with MeOH. Subfraction 3'(105-199) was rechromatographed over sephadex LH20 column (25 × 1 cm) with MeOH to yield 12 (3 mg) & 13 (5 mg) respectively. Fraction J (Fr. 83-90) was left for recrystallization from methanol to yield 15 (21 mg). The remaining part after crystallization was purified over sephadex LH20 column (36 × 1 cm) with CH₂Cl₂: MeOH (99:1) to yield 14 (7 mg).

The n-butanol fraction (15 g) was applied over Diaion HP 20 column (40 × 4.5 cm, 120 gm). The effluent was collected in 1000 mL fractions into (Fr. 1- 10). Fraction K (Fr. 3-4; 2.6 gm) eluted with H₂O: MeOH (70:30)& (60:40) was re-chromatographed over Diaion HP 20 column (50 × 1.5 cm, collecting 100 mL fractions). Fraction (20-39) eluted with H₂O: MeOH (93:17) was purified repeatedly over Sephadex LH20 column (38 × 1 cm) with MeOH to elute 16 (4mg), 17 (4.3 mg) respectively.
Flowchart 1: Phytochemical investigation of petroleum ether fraction of aerial part of *Lactuca serriola*
Flowchart 2: Phytochemical investigation of methylene chloride fraction of aerial part of *Lactuca serriola*
Flowchart 3: Phytochemical investigation of ethyl acetate fraction of aerial part of *Lactuca serriola*
Flowchart 4: Phytochemical investigation of n-butanol fraction of aerial part of *Lactuca serriola*
1.3. Biological Activity Assessments

1.3.1. Determination of α-Glucosidase Activity

α-Glucosidase inhibitory activity was carried out according to [1] with minor modifications. In a 96-well plate, reaction mixture containing 10 μL alpha-glucosidase (1 U/mL), 50 μL phosphate buffer (100 mM, pH = 6.8) and 20 μL of varying concentrations of extracts and compounds (1000 to 7.81 μg/mL) was preincubated at 37°C for 15 min. Then, 20 μL P-NPG (5 mM) was added as a substrate and incubated further at 37°C for 20 min. The reaction was stopped by adding 50 μL Na₂CO₃ (0.1 M). The absorbance of the released p-nitrophenol was measured using Multiplate Reader at 405 nm. Acarbose at various concentrations 1000 to 7.81 μg/mL was used as a standard. Wells without test substance was used as a control and each experiment was performed in triplicates. The results were expressed as percentage inhibition, which was calculated using the formula: 

\[
\text{Inhibitory activity (\%) = (1 - } \frac{A_{\text{test}}}{A_{\text{control}}} \times 100
\]

1.3.2. Determination of ABTS Radical Scavenging Activity

The assay was carried out according to [2] with minor modifications. The ABTS (2,2′-Azino-bis(3-ethylbenzothiazoline-6-sulfonic acid) radical cation (blue-dark green) was prepared by mixing equal volumes of ABTS stock solution (colorless; 7 mM in distilled H2O) and K₂S₂O₈ stock solution (potassium persulfate; 3.5 mM in distilled H2O). The mixture was allowed to stand in the dark at R.T. for 12-16 h until the reaction was complete. The ABTS⁺ solution was prepared by diluting the ABTS⁺ stock solution in pure EtOH to have an absorbance (A_{\text{control}}) of 0.7±0.02 at a wavelength of 734 nm and was equilibrated with an incubator at 30 °C. Free radical scavenging activity was assessed by mixing 1.5 mL of the blue-green ABTS⁺ solution with 10 μL of the extract/compound at various concentrations ranging from 10 to 60 μM (in distilled H2O, pure EtOH, or mixture of both of them). The change in absorbance at 734 nm was immediately monitored after 15 min (A_{\text{test}}). Ascorbic acid was used as a standard. The decrease in absorbance can represent % inhibition which is calculated as follow: 

\[
\% \text{ inhibition} = \frac{(A_{\text{control}} - A_{\text{test}})}{A_{\text{control}}} \times 100
\]

1.3.3. Determination of DPPH Radical Scavenging Activity

The DPPH (2,2-diphenyl-1-picryl-hydrazyl-hydrate) free radical assay was carried out according to the method of [3]. 100 μL of freshly prepared DPPH reagent (0.1% in methanol) were added to 100 μL of the sample/compound at various concentrations (dissolved in DMSO and diluted in methanol) in 96 wells plate (n=6), the reaction was incubated at room temperature for 30 min in dark. At the end of incubation time, the resulting reduction in DPPH color intensity was measured at 540 nm. Trolox was used as a standard. Data are represented as means ± SD according to the following equation:

\[
\% \text{ inhibition} = \frac{(A_{\text{control}} - A_{\text{test}})}{A_{\text{control}}} \times 100
\]

1.3.4. Determination of Ferric Reducing Antioxidant Power (FRAP)

The ferric reducing ability assay was carried out according to the method of [4] with minor modifications to be carried out in microplates. A freshly prepared TBTZ reagent (300 mM Acetate Buffer (PH=3.6), 10 mM TBTZ in 40 mM HCl, and 20 mM FeCl₃, in a ratio of 10:1:1 v/v/v, respectively). 190 μL from the freshly prepared TPTZ reagent were mixed with 10 μL of the extract/compound in 96 wells plate (n=6) where compound 10, 11, 14 at 1 mM, compound 12, 13, 15 at 0.166 mM, the MeOH and pet. ether extract at 2 mg/mL and the CH₂Cl₂, EtOAc and n-BuOH extract
at 0.333 mg/mL, the reaction was incubated at room temperature for 30 min in dark. At the end of incubation time the resulting blue color was measured at 593 nm. Data are represented as means ± SD. Torolox stock solution of 10 mg/mL in methanol was used as standard at various concentrations from 500 to 7.8 µg/mL.
### Table S1. $^1$H (400 MHz) and APT (100 MHz) data of compound 1 in CDCl₃.

| H/C no. | $^1$H-NMR | APT | $^1$H-NMR* [5] | $^{13}$C-NMR* [5] |
|---------|-----------|-----|----------------|-------------------|
| 1       | 38.7      |     | 38.1           |                   |
| 2       | 27.4      |     | 27.4           |                   |
| 3       | 79.0      | 3.21 (dd, $J=5.2, 11.2$) | 79.1 |
| 3       | 38.9      |     | 38.7           |                   |
| 4       | 55.3      |     | 55.3           |                   |
| 5       | 18.3      |     | 18.3           |                   |
| 6       | 34.3      |     | 34.3           |                   |
| 7       | 40.8      |     | 40.9           |                   |
| 8       | 50.4      |     | 50.4           |                   |
| 9       | 37.1      |     | 37.2           |                   |
| 10      | 20.9      |     | 21.0           |                   |
| 11      | 25.1      |     | 25.2           |                   |
| 12      | 38.1      |     | 38.9           |                   |
| 13      | 42.8      |     | 42.9           |                   |
| 14      | 27.5      |     | 27.5           |                   |
| 15      | 35.6      |     | 35.6           |                   |
| 16      | 43.0      |     | 43.0           |                   |
| 17      | 48.3      |     | 48.0           |                   |
| 18      | 2.40 (1H, m) | 48.0 | 48.0 |
| 19      | 151.0     |     | 151.0          |                   |
| 20      | 29.9      |     | 29.9           |                   |
| 21      | 19.3      |     | 19.3           |                   |
| 22      | 40.0      |     | 40.0           |                   |
| 23      | 1.05 (3H, s) | 28.0 | 1.00 (s)       | 28.0 |
| 24      | 0.81 (3H, s) | 15.4 | 0.79 (s)       | 15.4 |
| 25      | 0.85 (3H, s) | 16.1 | 0.83 (s)       | 16.1 |
| 26      | 0.96 (3H, s) | 16.0 | 0.95 (s)       | 16.0 |
| 27      | 0.78 (3H, s) | 14.6 | 0.76 (s)       | 14.6 |
| 28      | 0.99 (3H, s) | 18.0 | 0.97 (s)       | 18.0 |
| 29      | 4.59 (1H, dd, $J=2.4, 1.6$) | 109.3 | 4.57 (s)       | 109.3 |
| 29      | 4.71 (1H, d, $J=2.4$) | 4.68 (s) |                   | |
| 30      | 1.70 (3H, s) | 19.3 | 1.68 (s)       | 19.3 |

* $^{13}$C-NMR (50 MHz), $^1$H-NMR (200 MHz) is measured in CDCl₃
Figure S1: $^1$H (400 MHz) data of compound 1 in CDCl$_3$

Figure S2: APT (100 MHz) data of compound 1 in CDCl$_3$
Figure S3: IR spectrum of compound 1 (KBr)
Compound 2:

![Compound 2](image)

Figure S4: IR spectrum of compound 2 (KBr)
Compound 3:

Table S2: $^1$H (400 MHz) and APT (100 MHz) data of compound 3 in CDCl$_3$

| H/C no. | $^1$H-NMR | APT | $^{13}$C-NMR [6] |
|---------|-----------|-----|------------------|
| 1       | 39.3      | 38.7 |
| 2       | 28.1      | 27.6 |
| 3       | 79.6      | 79.0 |
| 4       | 39.4      | 38.9 |
| 5       | 55.8      | 55.3 |
| 6       | 18.9      | 18.3 |
| 7       | 34.7      | 34.3 |
| 8       | 41.6      | 41.1 |
| 9       | 50.9      | 50.4 |
| 10      | 37.2      | 37.1 |
| 11      | 22.1      | 21.6 |
| 12      | 27.9      | 27.0 |
| 13      | 39.7      | 39.2 |
| 14      | 42.9      | 42.4 |
| 15      | 27.5      | 27.4 |
| 16      | 37.6      | 36.7 |
| 17      | 34.2      | 34.5 |
| 18      | 48.9      | 48.5 |
| 19      | 32.5      | 32.0 |
| 20      | 144.2     | 143.7|
| 21      | 121.3     | 120.7|
| 22      | 42.2      | 42.2 |
| 23      | 0.74 (3H, s) | 15.3 | 15.4 |
| 24      | 0.95 (3H, s) | 28.5 | 28.0 |
| 25      | 0.85 (3H, s) | 16.8 | 16.3 |
| 26      | 1.01 (3H, s) | 16.6 | 16.0 |
| 27      | 0.94 (3H, s) | 14.7 | 14.8 |
| 28      | 0.83 (3H, s) | 18.3 | 17.7 |
| 29      | 0.99 (3H, d, $J=6.4$) | 23.1 | 22.5 |
| 30      | 4.12 (1H, d, $J=13.2$) | 66.0 | 65.5 |
|         | 4.01 (1H, d, $J=12.4$) |     |     |

* $^{13}$C-NMR is measured in CDCl$_3$ at 100 MHz.
Figure S5: $^1$H (400 MHz) data of compound 3 in CDCl$_3$

Figure S6: APT (100 MHz) data of compound 3 in CDCl$_3$
Figure S7: HR-FAB-MS of compound 3
**Compound 4:**

![Chemical Structure](image)

**Table S3:** $^1$H (600 MHz) and APT (150 MHz) data of compound 4 in CDCl$_3$, CD$_3$OD mixture

| H/C no. | $^1$H-NMR       | APT  | $^1$H-NMR* [7] | $^{13}$C-NMR* [7] |
|---------|-----------------|------|----------------|------------------|
| 1       |                 | 175.7|                |                  |
| 2       | 2.35 (2H, $t$, $J$=7.8) | 35.1 | 2.34 (3H, $t$) | 34.2             |
| 3       | 1.62 (2H, $m$)  | 26.1 | 1.62 (2H, $m$) | 24.6             |
| 4-13    | 1.28 (26H, $m$) | 30.2-30.8 | 1.24-1.28 (28H, $m$) | 29.1-29.7 |
| 14      |                 | 33.1 |                | 29.1-29.7        |
| 15      |                 | 23.8 |                | 29.1-29.7        |
| 16      | 0.89 (3H, $t$, $J$=7.2) | 14.6 | 0.87 (3H, $t$) | 14.1             |
| 1'      | 4.08 (1H, $dd$, $J$= 11.4, 6.0) | 64.1 | 4.13 (1H, $dd$) | 63.3             |
| 2'      | 4.16 (1H, $dd$, $J$= 11.4, 4.8) | 64.1 | 4.19 (1H, $dd$) | 63.3             |
| 3'      | 3.83 (1H, $m$)  | 71.2 | 3.92 (1H, $dd$) | 70.3             |
| 3       | 3.56 (1H, $t$, $J$=4.8) | 66.6 | 3.59 (1H, $dd$) | 65.2             |
| 3'      | 3.67 (1H, $dd$, $J$=10.8, 4.8) | 66.6 | 3.69 (1H, $dd$) | 65.2             |

* $^{13}$C and $^1$H-NMR are measured in CDCl$_3$
Figure S8: $^1$H (600 MHz) data of compound 4 in CDCl₃, CD₃OD mixture

Figure S9: $^{13}$C NMR (APT, 150 MHz) data of compound 4 in CDCl₃, CD₃OD mixture
Figure S10: GC-MS of fatty acid methyl ester of compound 4
Table S4: $^1$H (600 MHz) and APT (150MHz) data of compound 5 in CDCl$_3$

| H/C no. | $^1$H-NMR | APT | $^{13}$C-NMR* [8] |
|---------|-----------|-----|------------------|
| 1       | 1a 3.74 (1H, $dd$, $J$= 12.0, 4.8) | 61.0 | 61.3 |
|         | 1b 3.81 (1H, $dd$, $J$= 12.0, 4.2) |     |      |
| 2       | 4.09 (1H, $m$) | 51.5 | 51.8 |
| 3       | 3.53 (1H, $m$) | 75.6 | 72.4 |
| 4       | 3.54 (1H, $m$) | 72.3 | 72.2 |
| 5       | 5a 1.41 (1H, $m$) | 32.5 | 34.7 |
|         | 5b 1.71 (1H, $m$) |     |      |
| 6       | 1.58 (1H, $m$) | 25.8 | 25.4 |
| 7-12    | 1.26- 1.31 | 29.1-29.6 | 29.5-32.1 |
| 13      | 1.97 (2H, $m$) | 32.5 | 33.1 |
| 14      | 5.40 (2H, $t$, $J$=5.4) | 129.8 | 130.0 |
| 15      | 5.40 (2H, $t$, $J$=5.4) | 130.8 | 132.1 |
| 16      | 1.97 (2H, $m$) | 32.5 | 32.8 |
| 17      | 1.29 | 22.6 | 26.1 |
| 18      | 0.88 (6H, $t$, $J$= 6.6) | 13.9 | 14.2 |
| 1'      | ...... | 175.7 | 176.1 |
| 2'      | 4.04 (1H, $dd$, $J$= 8.4, 3.6 ) | 72.2 | 75.5 |
| 3'      | 1.80 (1H, $m$) | 34.3 | 32.8 |
|         | 1.58 (1H, $m$) |     |      |
| 4'      | 1.41 (1H, $m$) | 25.2 | 22.9 |
| 5'-23'  | 1.26- 1.31 | 29.1-31.8 | 29.5-32.1 |
| 24'     | 0.88 (6H, $t$, $J$= 6.6) | 13.9 | 14.2 |

* $^{13}$C-NMR is measured in CDCl$_3$ at 100 MHz.

Figure S11: Selected HMBC correlations of compound 5
Figure S12: $^1$H (600 MHz) data of compound 5 in CDCl$_3$.

Figure S13: APT (150MHz) data of compound 5 in CDCl$_3$.
Figure S14: HSQC experiment of compound 5 in CDCl₃

Figure S15: HMBC experiment of compound 5 in CDCl₃
Figure S16: Negative HR-ESI-TOF-MS spectrum of compound 5
Table S5: $^1$H (500 MHz) and $^{13}$C NMR (125MHz) data of compound 6 in C$_5$D$_5$N

| H/C no. | $^1$H-NMR | $^{13}$C-NMR | $^1$H-NMR*[9] | $^{13}$C-NMR*[9] |
|---------|------------|--------------|---------------|-----------------|
| 1       | 133.8      |              | 133.0         |                 |
| 2       | 194.7      |              | 194.1         |                 |
| 3       | 6.97 (1H, d, J=1) | 133.3 | 6.32 (d, J=1.08) | 132.0 |
| 4       | 175.2      |              | 174.8         |                 |
| 5       | 3.80 (1H, d, J=10.5) | 48.7 | 3.89 (d, J=10.80) | 47.5 |
| 6       | 3.71 (1H, t, J=10.5) | 81.2 | 3.96 (t, J= 10.80) | 80.3 |
| 7       | 3.46 (1H, tt, J=10.0, 3.0) | 54.5 | 3.52 (tt, J=10.260) | 52.9 |
| 8       | 5.04 (1H, dt, J=10.5, 1.5 ) | 69.9 | 4.88 (dt, J=10.50, 1.90) | 69.2 |
| 9       | 2.79 (1H, t, J=13.0) | 44.0 | 2.87 (dd, J=13.30, 10) | 43.4 |
|         | 2.44 (1H, dd, J=13.0, 2.2) |             | 2.30 (dd, J=13.30, 2) |        |
| 10      | 144.9      |              | 144.6         |                 |
| 11      | 137.1      |              | 136.5         |                 |
| 12      | 168.5      |              | 168.1         |                 |
| 13      | 5.51 (1H, d, J=3.0) | 121.3 | 5.90 (d, J=3.20) | 120.9 |
|         | 6.15 (1H, d, J=3.0) |              | 5.38 (d, J=3.00) |           |
| 14      | 2.46 (3H, s) | 20.9 | 2.08 (s) | 20.6 |
| 15      | 4.72 (H, d, J= 18.5) | 62.4 | 4.70 (ddd, J=18.80, 5.80, 1.90) | 61.3 |
|         | 5.28 (H, d, J=18.5) |              | 4.29 (ddd, J=19.10, 5.60, 1.50) |       |
| 1       | 171.2      |              | 170.7         |                 |
| 2'      | 3.83 (2H)$^a$ | 40.8 | 3.70 (d, 15.50) | 39.7 |
|         |             |              | 3.66 (d, 15.50) |           |
| 3'      | 124.5      |              | 123.8         |                 |
| 4'      | 7.39 (2H)$^a$ | 131.1 | 7.11 (dd, J=6.60, 1.50) | 130.4 |
| 5'      | 7.20 (2H, d, J=6, 1.5) | 116.5 | 6.74 (dd, J=6.60, 1.50) | 115.3 |
| 6'      | 158.3      |              | 156.4         |                 |
| 7'      | 7.20 (2H, d, J=6.0, 1.5) | 116.5 | 6.74 (dd, J=6.60, 1.50) | 115.3 |
| 8'      | 7.39 (2H)$^a$ | 131.1 | 7.11 (dd, J=6.60, 1.50) | 130.4 |

$^a$ Peaks J values cannot be determined due to peaks deformity.

*13C and $^1$H-NMR are measured in DMSO-$_d_6$ at 100 and 400 MHz respectively.
Figure S17: $^1$H (500 MHz) data of compound 6 in C$_5$D$_5$N

Figure S18: $^{13}$C NMR (125MHz) data of compound 6 in C$_5$D$_5$N
Figure S19: IR spectrum of compound 6 (KBr)
Compound 7:

\[
\begin{align*}
\text{HO} & \quad \text{HO} \\
\text{H} & \quad \text{H} \\
\text{O} & \quad \text{HO} \\
\text{H} & \quad \text{H} \\
\text{O} & \quad \text{HO} \\
\text{H} & \quad \text{H} \\
\text{O} & \quad \text{HO} \\
\text{H} & \\
\text{H} & \quad \text{H} \\
\text{OH} & \quad \text{HO} \\
\text{H} & \quad \\
\end{align*}
\]

**Figure S20:** IR spectrum of compound 7 (KBr)

**Figure S21:** QTOF-HR-ESI-MS analysis of compound 7 (positive mode)
**Compound 8:**

![Molecular structure of compound 8](image)

**Table S6:** $^1$H (500 MHz) and $^{13}$C NMR (125MHz) data of compound 8 in CD$_3$OD

| H/C no. | $^1$H-NMR       | $^{13}$C-NMR | $^1$H-NMR* [10] | $^{13}$C-NMR* [10] |
|---------|----------------|--------------|----------------|-------------------|
| 1       | ……………       | 127.6        |                 |                   |
| 2       | 7.41 (2H, $d$, $J$ = 8.5) | 130.8       | 7.44 (2H, $d$, $J$ = 8.6) | 131.1             |
| 3       | 6.78 (2H, $d$, $J$ = 8.5) | 116.7       | 6.79 (2H, $d$, $J$ = 8.6) | 116.8             |
| 4       | ……………       | 160.8        |                 |                   |
| 5       | 6.78 (2H, $d$, $J$ = 8.5) | 116.7       | 6.79 (2H, $d$, $J$ = 8.6) | 116.8             |
| 6       | 7.41 (2H, $d$, $J$ = 8.5) | 130.8       | 7.44 (2H, $d$, $J$ = 8.6) | 131.8             |
| 7       | 7.51 (1H, $d$, $J$ = 16.0) | 145.2       | 7.58 (1H, $d$, $J$ = 15.9) | 146.5             |
| 8       | 6.27 (1H, $d$, $J$ = 16.0) | 116.0       | 6.28 (1H, $d$, $J$ = 15.9) | 115.8             |
| 9       | ND             | ND           |                 |                   |

$^1$H and $^{13}$C-NMR are measured in CD$_3$OD at 500 and 125 MHz respectively.
ND: Not detected.
Figure S22: $^1$H (500 MHz) data of compound 8 in CD$_3$OD

Figure S23: $^{13}$C NMR (125 MHz) data of compound 8 in CD$_3$OD
Figure S24: QTOF-HR-ESI-MS analysis (negative mode) of compound 8
## Table S7: \(^1\)H (500 MHz) and \(^{13}\)C NMR (125MHz) data of compound 9 in CD\(_3\)OD

| H/C no. | \(^1\)H-NMR       | \(^{13}\)C-NMR | \(^1\)H-NMR* [11] | \(^{13}\)C-NMR* [11] |
|--------|-------------------|----------------|-------------------|-------------------|
| 1      | 133.1             |                | 132.3             |                  |
| 2      | 197.3             |                | 195.8             |                  |
| 3      | 6.39 (1H, \(d, J=1.0\)) | 133.6        | 6.44 (br. s)            | 133.1             |
| 4      | 176.5             |                | 173.4             |                  |
| 5      | 3.71 (\(d, J=9.0\)) | 49.7          | 3.56 (\(d, J=10\)) | 49.2             |
| 6      | 3.67 (\(dd, J=10, 1.5\)) | 82.4          | 3.66 (\(dd, J=10,10\)) | 81.2             |
| 7      | 2.15 \(^a\)      | 62.2           | 2.15 (\(add, J=12, 10, 9.6\)) | 61.4             |
| 8      | 3.69 \(^a\)      | 69.9           | 3.74 (\(m\))         | 68.0             |
| 9      | 2.40 (\(dd, J=14.0, 2.0\)) | 50.0          | 2.40 (\(dd, J=13.6, 2\)) | 49.3             |
|        | 2.79 (\(dd, J=14.0, 11.0\)) | 2.76 (\(dd, J=13.6, 10.8\)) |                  |                  |
| 10     | 149.9             |                | 148.3             |                  |
| 11     | 2.63 (\(q, J=7.0\)) | 42.5          | 2.59 (\(dq, J=12, 7.2\)) | 41.7             |
| 12     | 179.9             |                | 178.6             |                  |
| 13     | 1.36 (\(d, J=7.0\)) | 15.8          | 1.43 (\(d, J=7.2\)) | 15.3             |
| 14     | 2.41 (\(s\))    | 21.8           | 2.45 (br. s)            | 21.8             |
| 15     | 4.41 (\(dd, J=18.5, 1.0\)) | 63.1          | 4.43 (\(d, J=17.6\)) | 62.2             |
|        | 4.82 (\(dd, J=18.5, 1.0\)) | 4.48 (\(d, J=17.6\)) |                  |                  |

\(^a\) Peaks multiplicity is not clear due to overlapping with other peaks.

\(^1\)H and \(^{13}\)C-NMR are measured in CDCl\(_3\) at 400 and 100 MHz respectively.
Figure S25: \(^1\)H (500 MHz) data of compound 9 in CD\textsubscript{3}OD

Figure S26: \(^{13}\)C NMR (125 MHz) data of compound 9 in CD\textsubscript{3}OD

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Figure S27: IR spectrum of compound 9 (KBr)
### Compound 10:

![Chemical structure of compound 10](image)

#### Table S8: $^1$H (400 MHz) and $^{13}$C NMR (100MHz) data of compound 10 in CD$_3$OD

| H/C no. | $^1$H-NMR | $^{13}$C-NMR | $^1$H-NMR* [10] | $^{13}$C-NMR* [10] |
|---------|------------|--------------|-----------------|-------------------|
| 1       |            | 123.8        |                 |                   |
| 2       | 7.12 (2H, d, J=8.4) | 130.0        | 7.87 (2H, d, J = 8.8 Hz) | 133.0             |
| 3       | 6.76 (2H, d, J=8.0)  | 114.8        | 6.80 (2H, d, J = 8.8 Hz) | 116.0             |
| 4       |            | 156.0        |                 |                   |
| 5       | 6.76 (2H, d, J=8.0)  | 114.8        | 6.80 (2H, d, J = 8.8 Hz) | 116.0             |
| 6       | 7.12 (2H, d, J=8.4)  | 130.0        | 7.87 (2H, d, J = 8.8 Hz) | 133.0             |
| 7       | ND         | ND           |                 |                   |

*$^1$H and $^{13}$C-NMR are measured in CD$_3$OD at 500 and 125 MHz respectively. ND: Not detected.*
Figure S28: $^1$H (400 MHz) data of compound 10 in CD$_3$OD

Figure S29: $^{13}$C NMR (100MHz) data of compound 10 in CD$_3$OD
Figure S30: QTOF-HR-ESI-MS analysis (negative mode) of compound 10
Compound 11:

Table S9: $^1$H (500 MHz) and $^{13}$C NMR (125MHz) data of compound 11 in CD$_3$OD

| H/C no. | $^1$H-NMR  | $^{13}$C-NMR | $^{13}$C-NMR* [12] |
|--------|------------|--------------|---------------------|
| 1      |            | 123.7        |                     |
| 2      | 7.42 (H, s)| 117.7        | 117.8               |
| 3      |            | 146.0        | 146.0               |
| 4      |            | 151.4        | 151.3               |
| 5      | 6.78 (H, d, $J$=8.5) | 115.7    | 115.8               |
| 6      | 7.40 (H, bd. s) | 123.8    | 123.8               |
| 7      | ND         | ND           | 170.6               |

*$^1$H and $^{13}$C-NMR are measured in CD$_3$OD at 300 and 75.5 MHz respectively.
ND: Not detected.
Figure S31: $^1$H (500 MHz) of compound 11 in CD$_3$OD

Figure S32: $^{13}$C NMR (125MHz) data of compound 11 in CD$_3$OD
Figure S33: QTOF-HR-ESI-MS analysis (negative mode) of compound 11
Compound 12:

![Chemical Structure of Compound 12]

| H/C no. | $^1$H-NMR | $^{13}$C-NMR | $^1$H-NMR$^*$ [13] | $^{13}$C-NMR$^{**}$ [14] |
|---------|-----------|--------------|---------------------|---------------------|
| 1       | 148.9     | 146.8        |                     |                     |
| 2       | 138.0     | 136.6        |                     |                     |
| 3       | 178.2     | 176.6        |                     |                     |
| 4       | 163.3     | 162.3        |                     |                     |
| 5       |           |              |                     |                     |
| 6       | 6.18 (1H, $d$, $J$= 1.5) | 100.1 | 6.15 (s) | 99.2 |
| 7       | 166.4     | 164.9        |                     |                     |
| 8       | 6.39 (1H, $d$, $J$=1.5) | 95.3 | 6.35 (s) | 94.4 |
| 9       | 161.4     | 157.7        |                     |                     |
| 10      | 105.3     | 104.1        |                     |                     |
| 1'      | 124.5     | 123.3        |                     |                     |
| 2',6'   | 8.07 (2H, $d$, $J$= 9.0) | 131.5 | 8.05 (2H, $d$, $J$= 8.5) | 125.9 |
| 3', 5'  | 6.89 (2H, $d$, $J$= 9.0) | 117.1 | 6.88 (2H, $d$, $J$= 8.5) | 116.3 |
| 4'      | 159.1     | 160.1        |                     |                     |

* $^1$H-NMR is measured in CD$_3$OD at 400 MHz.

** $^{13}$C-NMR is measured in CD$_3$OD at 125 MHz.

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Figure S34: $^1$H (500 MHz) data of compound 12 in CD$_3$OD

Figure S35: $^{13}$C NMR (125MHz) data of compound 12 in CD$_3$OD
Figure S36: QTOF-HR-ESI-MS analysis (negative mode) of compound 12
**Compound 13:**

```

| H/C no. | ¹H-NMR       | ¹H-NMR* [15] |
|---------|--------------|--------------|
| 1       |              |              |
| 2       |              |              |
| 3       |              |              |
| 4       |              |              |
| 5       |              |              |
| 6       | 6.17 (1H, d, J=1.5) | 6.18 (d, J=2.03) |
| 7       | 6.38 (1H, d, J=2.0)  | 6.40 (d, J=2.03) |
| 8       | 6.38 (1H, d, J=2.0)  | 6.40 (d, J=2.03) |
| 9       | 6.38 (1H, d, J=2.0)  | 6.40 (d, J=2.03) |
| 10      | 7.73 (1H, d, J=2.0)  | 7.68 (d, J= 2.2) |
| 1'      | 7.62 (1H, d, J=8.0)  | 6.88 (d, J= 8.47) |
| 2'      |              |              |
| 3'      |              |              |
| 4'      |              |              |
| 5'      |              |              |
| 6'      |              |              |

*¹H-NMR is measured in DMSO-d6 at 600 MHz*
```
Figure S37: $^1$H (400 MHz) data of compound 13 in CD$_3$OD

Figure S38: QTOF-HR-ESI-MS analysis (negative mode) of compound 13
Table S12: \(^1\)H (500 MHz) and \(^{13}\)C NMR (125MHz) data of compound 14 in CD\(_3\)OD

| H/C no. | \(^1\)H-NMR          | \(^{13}\)C-NMR | \(^1\)H-NMR * [16] | \(^{13}\)C-NMR*[16] |
|---------|-----------------------|-----------------|--------------------|---------------------|
| 1       | 6.56 (1H, \(t, J=8.4\) Hz) | 152.1           | 6.24 (1H, br. \(t, J=8\) Hz) | 150.1 |
| 2       | 3.54-4.38 (\(m\))     | 33.6            | 3.90-4.90 (\(m\))    | 32.9 |
| 3       | 80.7                  |                 |                    | 79.8 |
| 4       | 137.7                 |                 |                    | 136.8 |
| 5       | 5.02 (1H, br. \(d, J= 10.0\) HZ) | 128.0           | 5.04 (1H, br. \(d, J=10\) Hz) | 127.0 |
| 6       | 3.54-4.38 (\(m\))     | 81.9            | 3.90-4.90 (\(m\))    | 80.2 |
| 7       | 50.5                  |                 |                    | 49.5 |
| 8       | 23.2                  |                 |                    | 22.4 |
| 9       | 26.1                  |                 |                    | 25.5 |
| 10      | 147.0                 |                 |                    | 145.6 |
| 11      | 42.4                  |                 |                    | 41.3 |
| 12      | 181.4                 |                 |                    | 178.5 |
| 13      | 0.97 (3H, \(d, J=7.2\) HZ) | 12.9            | 1.18 (3H, \(d, J=7\) Hz) | 12.8 |
| 14      | 9.31 (1H, br \(s\))  | 197.8           | 9.49 (1H, br \(s\))  | 195.6 |
| 15      | 1.86 (3H, br \(s\))  | 11.3            | 2.14 (3H, br \(s\))  | 11.2 |
| 1'      | 4.12 (1H, \(d, J=7.7\)) | 102.0           |                    | 101.9 |
| 2'      | 75.1                  |                 |                    | 74.9 |
| 3'      | 78.0                  |                 |                    | 78.2 |
| 4'      | 71.8                  |                 |                    | 71.6 |
| 5'      | 78.0                  |                 |                    | 78.2 |
| 6'      | 62.8                  |                 |                    | 62.7 |

*\(^1\)H and \(^{13}\)C-NMR are measured in C\(_5\)D\(_5\)N at 400 and 90 MHz respectively.
Figure S39: $^1$H (500 MHz) data of compound 14 in CD$_3$OD

Figure S40: $^{13}$C NMR (125MHz) data of compound 14 in CD$_3$OD
Figure S41: IR spectrum of compound 14 (KBr)
### Table 13: $^1$H (500 MHz) and $^{13}$C NMR (125MHz) data of compound 15 in DMSO-$d_6$

| H/C no. | $^1$H-NMR | $^{13}$C-NMR | $^1$H-NMR $^*$ [17] | $^{13}$C-NMR $^**$ [18] |
|---------|-----------|--------------|----------------------|------------------------|
| 2       |           | 164.5        |                      | 163.6                  |
| 3       | 6.75 (1H, s) | 103.2        | 6.61 (1H, s)         | 103.7                  |
| 4       |           | 181.9        |                      | 182.5                  |
| 5       |           | 161.1        |                      | 161.8                  |
| 6       | 6.43 (1H, d, $J=2.0$ Hz) | 99.5        | 6.42 (1H, d, $J=2.3$ Hz) | 100.2                  |
| 7       |           | 162.9        |                      | 165.2                  |
| 8       | 6.78 (1H, d, $J=2.0$ Hz) | 94.7        | 6.90 (1H, d, $J=2.3$ Hz) | 95.4                   |
| 9       |           | 156.9        |                      | 157.6                  |
| 10      |           | 105.3        |                      | 106.0                  |
| 1$'$    |           | 121.3        |                      | 121.8                  |
| 2$'$    | 7.41 (1H, s) | 113.3        | 7.43 (1H, d, $J=2.3$ Hz) | 114.1                  |
| 3$'$    |           | 145.8        |                      | 146.6                  |
| 4$'$    |           | 149.9        |                      | 151.0                  |
| 5$'$    | 6.90 (1H,d, $J=8.5$ Hz) | 116.0        | 6.92 (1H, d, $J=8.4$ Hz) | 116.7                  |
| 6$'$    | 7.43 (1H, $dd$, $J=8.0, 2.0$ Hz) | 119.2        | 7.40 (1H, d, $J=2.3, 8.4$ Hz) | 119.9                  |
| 1$''$   | 5.08 (1H, d, $J=7.0$Hz) | 99.8        | 5.08 (1H, d, $J=7.65$ Hz) | 100.6                  |
| 2$''$   | 3.42-3.70 | 73.1         | 3.5-4.1              | 73.8                   |
| 3$''$   |           | 76.4         |                      | 77.1                   |
| 4$''$   |           | 69.5         |                      | 70.3                   |
| 5$''$   |           | 77.1         |                      | 77.8                   |
| 6$''$   |           | 60.6         |                      | 61.3                   |

$^*$$^1$H-NMR is measured in DMSO-$d_6$ at 500 MHz.

$^{**}$$^{13}$C-NMR is measured in DMSO-$d_6$ at 75 MHz.
Figure S42: $^1$H (500 MHz) data of compound 15 in DMSO-$d_6$

Figure S43: $^{13}$C NMR (125MHz) data of compound 15 in DMSO-$d_6$
Figure S44: QTOF-HR-ESI-MS analysis (negative mode) of compound 15
### Table S14: $^1$H (400 MHz) and $^{13}$C NMR (100 MHz) data of compound 16 in CD$_3$OD

| H/C no. | $^1$H-NMR | DEPTQ | $^1$H-NMR*[16] | $^{13}$C-NMR*[16] |
|---------|-----------|-------|----------------|------------------|
| 1       | 5.00$^a$  | 127.3 | 5.10 (1H, dd, $J$=13.4 Hz) | 127.4            |
| 2       | 2.34$^a$  | 31.9  |                | 32.8             |
| 3       | 4.44 (1H, t)$^b$ | 82.6  | 4.58 (1H, dd, $J$= 10, 7 Hz) | 83.3             |
| 4       | 140.0     |       |                | 140.6            |
| 5       | 4.95$^a$  | 127.1 | 4.94 (1H, br. $d$, $J$= 10 Hz) | 126.7            |
| 6       | 4.69$^a$  | 81.2  | 4.86 (1H, t, $J$= 10 Hz)  | 80.8             |
| 7       | 1.65$^a$  | 54.1  |                | 54.5             |
| 8       | 1.63$^a$  | 27.9  |                | 29.0             |
| 9       | 1.74$^a$  | 35.6  |                | 36.8             |
| 10      | 2.70 (1H, dd, $J$=7.5)$^b$ |      |                | 142.1            |
| 11      | 2.29$^a$  | 41.9  |                | 42.3             |
| 12      | 180.0     |       |                | 178.4            |
| 13      | 1.12 (3H, $d$, $J$=6.8 Hz) | 12.0  | 1.26 (3H, $d$, $J$=7 Hz)  | 13.3             |
| 14      | 3.73$^a$  | 57.4  | 4.19 (1H, br. $d$, $J$= 13 Hz) | 58.6             |
|         | 4.13$^a$  |       | 4.63 (IH, br. $d$, $J$= 13 Hz) |               |
| 15      | 1.57 (3H, br. $s$) | 10.3  | 1.90 (3H, br. $s$)  | 11.6             |
| 1'      | 4.11 (1H, $d$, $J$= 7.2) | 101.1 |                | 102.5            |
| 2'      | 3.11-3.79 | 73.8  |                | 75.0             |
| 3'      | 76.6      |       |                | 78.2             |
| 4'      | 70.4      |       |                | 71.7             |
| 5'      | 76.6      |       |                | 78.1             |
| 6'      | 61.4      |       |                | 62.8             |

$^a$ Peaks multiplicity is not clear due to overlapping with other peaks.

$^b$ Peaks $J$ values cannot be determined due to peaks deformity.

$^*$$^1$H and $^{13}$C-NMR are measured in C$_5$D$_5$N at 400 and 90 MHz respectively.
Figure S45: $^1$H (400 MHz) data of compound 16 in CD$_3$OD

Figure S46: DEPTQ (100 MHz) data of compound 16 in CD$_3$OD

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Figure S47: HSQC spectrum of compound 16 in CD$_3$OD

Figure S48: IR spectrum of compound 16 (KBr)
## Compound 17

The structure of compound 17 is shown in the image.

### Table S15: $^1$H (400 MHz) and DEPTQ (100 MHz) data of compound 17 in CD$_3$OD

| H/C no. | $^1$H-NMR       | DEPTQ | Type | $^1$H-NMR* [11] | $^{13}$C-NMR* [11] |
|---------|-----------------|-------|------|-----------------|-------------------|
| 1       | 132.1           | Q     |      | 132.1           |                   |
| 2       | 196.1           | Q     |      | 196.1           |                   |
| 3       | 6.56 (1H, br. s) | 133.3 | CH   | 6.57 (1H, br. s) | 134.4             |
| 4       | 170.5           | Q     |      | 169.4           |                   |
| 5       | 3.74$^b$         | 48.6  | CH   | 3.61 (d, J=10)  | 49.3              |
| 6       | 3.76$^b$         | 81.2  | CH   | 3.65 (dd, J=10, 10) | 81.4             |
| 7       | 2.18 (1H, dd, J=10.0)$^a$ | 60.8  | CH   | 2.15 (ddd, J=12, 10, 9.6) | 61.3 |
| 8       | 6.57 (1H, m)    | 68.6  | CH   | 3.81 (m)        | 69.0              |
| 9       | 2.39 (1H, dd, J=11.2,1.6) | 48.3  | CH$_2$ | 2.42 (dd, J=13.6,2.0) | 49.4 |
|         | 2.81 (1H, dd, J=10.8)$^a$ |      |      | 2.78 (dd, J=13.6, 10.8) |                   |
| 10      | 148.7           | Q     |      | 149.3           |                   |
| 11      | 2.65 (dd, J=5.6)$^a$ | 41.1  | CH   | 2.59 (dq, J=12, 7.2) | 41.8 |
| 12      | 178.7           | Q     |      | 178.9           |                   |
| 13      | 1.40 (1H, d, J=6.8) | 14.4  | CH$_3$ | 1.43 (d, J=7.2) | 15.3             |
| 14      | 2.44 (1H, s)    | 20.4  | CH$_3$ | 2.44 (br. s)    | 21.9              |
| 15      | 4.76$^b$        | 68.2  | CH$_2$ | 4.76 (d, J=17.2), | 68.7 |
|         | 4.81$^b$        |       |      | 4.85 (d, J=17.2) |                   |
| 1'      | 4.40 (d, J=7.6)  | 102.7 | CH   | 4.40 (d, 8.0)   | 102.8             |
| 2'      | 3.26 (1H, m)    | 73.7  | CH   | 3.32 m          | 73.8              |
| 3'      | 3.38 (1H, m)    | 76.6  | CH   | 3.45 m          | 76.4              |
| 4'      | 3.32 (1H, m)    | 70.2  | CH   | 3.36 m          | 70.3              |
| 5'      | 3.38 (1H, m)    | 76.6  | CH   | 3.44 m          | 76.7              |
| 6'      | 3.70$^b$        | 61.3  | CH$_2$ | 3.76 (dd, 12.0, 4.8), | 61.8 |
|         | 3.91 (1H, J=11.6) |      |      | 3.89 (dd, 12.0, 2.0) |                   |

$^b$ Peaks multiplicity is not clear due to overlapping with other peaks.

$^a$ Peaks $J$ values cannot be determined due to peaks deformity.

$^*$ $^1$H and $^{13}$C-NMR are measured in CD$_3$OD at 400 and 100 MHz respectively.
Figure S49: $^1$H (400 MHz) data of compound 17 in CD$_3$OD

Figure S50: DEPTQ (100 MHz) data of compound 17 in CD$_3$OD

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Figure S51: HSQC spectrum of compound 17 in CD$_3$OD

Figure S52: IR spectrum of compound 17 (KBr)
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