Supporting Information

Catalyst- and Additive-Free Sunlight-Induced Autoxidation of Aldehydes to Carboxylic Acids

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1. General Information

Chemicals were purchased from commercial suppliers and used as delivered. All solvents can be used directly without further drying and deoxygenation. Deuterated solvents were bought from Euriso-Top. All reactions were carried out under ambient atmosphere and monitored by thin layer chromatography (TLC). Components were visualized by treatment with aqueous potassium permanganate (KMnO₄) solution or by fluorescence quenching under UV light (254 nm). ¹H, ¹³C {¹H}, and ¹⁹F NMR spectra were recorded on a Bruker Avance-III-300, Bruker Avance-III-400 spectrometer. Chemical shifts are expressed as parts per million (ppm, δ) downfield of tetramethylsilane (TMS) and are referenced to CDCl₃ (7.26 / 77.0 ppm) and DMSO-d₆ (2.50 / 39.52 ppm) as internal standards. Data is reported as follows: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad, dd = doublet of doublets, td =triplet of doublets, ddd = doublet of doublet of doublets, dddd = doublet of doublet of doublet of doublets, dt = doublet of triplets,qd = quartet of doublets, dtd = doublet of triplet of doublets; constants are absolute values and J values are expressed in Hertz (Hz). Mass spectra (HRMS) was determined in the chemistry department of the University Heidelberg under the direction of Dr. J. Gross. Infrared Spectroscopy (IR) was processed on a FT-IR Vektor 22.

2. Reaction Equipment

The simple reaction equipment is shown in the pictures below. The pyrex reaction tube (open) was mounted on a magnetic stirrer and then placed under sunlight irradiation at room temperature. (Left: a 0.5 mmol scale reaction setup. Right: a 20 mmol scale reaction setup.)

A 10-mL pyrex reaction tube equipped with a magnetic stir bar was charged with 4-methoxybenzaldehyde $1a$ (60.8 µL, 0.5 mmol), 0.5 mL solvent was added. The reaction mixtures were placed under sunlight and stirred under open air at room temperature (22-27 °C). After 4 h, the solvent was removed under reduced pressure. The conversion rate of 4-methoxybenzaldehyde $1a$, the yield of carboxylic acid $2a$ and peroxycacid $3a$ were determined by comparing the integration of the $1H$ NMR resonance of $1a$, $2a$ and $3a$ with that of CH$_2$Br$_2$ (DMSO-$d_6$ as solvent). Conversion rates of $1a$ and yields of $2a$ and $3a$ are reported in Table S1.

**NOTE:** 1. More reaction information: weather (sunny); outdoor temperature (22-27 °C); humidity (50%); barometer (1015 mbar); time (from 11.20 AM to 15:20 PM); date (03.06.2022); place (Heidelberg, Germany).
2. After quenching with sat. aq. Na$_2$S$_2$O$_3$, tert-butanol, acetone and ethyl acetate all afforded carboxylic acid $2a$ in excellent isolated yields (> 99%).

**Table S1. Evaluation of Different Solvent**

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<th>$1a$ (%)</th>
<th>$2a$ (%)</th>
<th>$3a$ (%)</th>
<th>entry</th>
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H$_2$O: water; MeOH: methanol; EtOH: ethanol; tBuOH: tert-butanol; TAA: tert-amyl alcohol; EA: ethyl acetate; PE: petroleum ether; DCM: dichloromethane; CH$_3$Cl: chloroform; THF: etrahydrofuran; MeCN: acetonitrile; DMSO: dimethyl sulfoxide; DMF: dimethylformamide.

4. Control Experiments
4.1 The Effect of Light on the Autoxidation of Aldehydes

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

To three identical pyrex reaction tubes, 4-cyan-benzaldehyde 1aa (10 mmol) and acetone (10 mL) were added, respectively. The reaction mixtures were stirred under open air at room temperature. Expose the mixture in batches to dark (indoor temperature: 22 °C), natural light (avoid direct sunlight, outdoor temperature: 4-12 °C) and sunlight (outdoor temperature: 4-12 °C). Pipetted 10 µL of reaction solution into a NMR tube at a time and 0.5 mL CDCl₃ was added directly detected by ¹H NMR.

**Note:** More reaction information: weather(sunny); outdoor temperature (4-12 °C); humidity (41-59%); barometer (1006 mbar); time (from 10:30 AM to 18:30 PM); date (07.05.2022); place (Heidelberg, Germany). Natural light: the other side of the building on the same day, avoid direct sunlight. Dark: Indoors, avoid light, room temperature (22 °C).

![Figure S1](image)

**Figure S1** Autoxidation of 4-cyan-benzaldehyde 1aa in dark, natural light and sunlight

4.2 The Effect of Temperatures on the Autoxidation of Aldehydes

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

To a pyrex reaction tubes, 4-cyanobenzaldehyde 1aa (10 mmol) and acetone (10 mL) were added. Expose the mixture in batches sunlight (outdoor temperature: 15-23 °C) and stirred under open air. Pipetted 10 µL of reaction solution into a NMR tube at a time and 0.5 mL CDCl₃ was added directly detected by ¹H NMR. The obtained results are compared with figure S1C.

**Note:** More reaction information: weather(sunny); outdoor temperature (15-23 °C); humidity (41-59%); barometer (1006 mbar); time (from 10:30 AM to 18:30 PM); date (07.05.2022); place (Heidelberg, Germany).
4.3 The Effect of Oxygen on the Autoxidation of Aldehydes

To three identical pyrex reaction tubes, benzaldehyde 1b (20 mmol) and acetone (20 mL) were added, respectively. The reaction mixtures were stirred in sunlight at room temperature (2-12 °C) under 1 atm O₂, N₂ and open air. Pipetted 10 µL of reaction solution into a NMR tube at a time and 0.5 mL CDCl₃ was added directly detected by ¹H NMR.

Note: More reaction information: weather(sunny); outdoor temperature(2-12 °C); humidity (43-66%); barometer (1015 mbar); time (from 09:00 AM to 17:00 PM); date (28.02.2022); place (Heidelberg, Germany).

4.4 Autoxidation of Benzaldehyde 1b in the Dark

Figure S3 Autoxidation of benzaldehyde 1b under N₂, air and O₂
To a reaction tube, benzaldehyde 1b (20 mmol) and acetone (20 mL) were added. The reaction mixtures were stirred under open air in the dark at room temperature (indoor temperature: 22 °C). Pipetted 10 µL of reaction solution into a NMR tube at a time and 0.5 mL CDCl₃ was added directly detected by ¹H NMR. Within 72 h, the conversion of benzaldehyde 1b was 94%, which contained 89% of benzoic acid 2b and 5% of peroxybenzoic acid 3b.

**Figure S4** Autoxidation of benzaldehyde 1b in the dark

5. Synthetic Application of PhCHO/O₂/Sunlight Oxidation System

5.1 Preparation of Benzoperoxoic Acid (3b) Solution

![Chemical Reaction](image)

To a pyrex reaction tube, benzaldehyde 1b (20 mmol) and acetone (20 mL) were added. Evacuate the resulting solution and flush with O₂ several times, then was filled with oxygen with O₂ balloon. The reaction mixtures were placed under sunlight and stirred at room temperature. After 6 h, pipetted 20 µL of reaction solution into a NMR tube at a time and 0.5 mL CDCl₃ was added directly detected by ¹H NMR (Figure S5A). From this we obtained a solution of acetone peroxybenzoate 2b (0.75 M in acetone), which can be directly used in the later synthesis. It is worth mentioning that the solution was stored in the fridge at 2-8 °C. After three days, the mixture was detected by ¹H NMR, as shown in Figure S5B, the benzaldehyde was completely converted into carboxylic acid, and the content of peroxybenzoic acid was about 65%.

**Note:** More reaction information: weather(sunny); outdoor temperature (2-8 °C); humidity (56-61%); barometer (999 mbar); time (from 10:00 AM to 16:00 PM); date (03.03.2022); place (Heidelberg, Germany).

![Chemical Reaction](image)

**Figure S5** ¹H NMR of benzaldehyde under 1 atm O₂ in sunlight
5.2 Synthesis of Sulfonyldibenzene 5a

\[
\begin{align*}
\text{4a, 1.0 mmol} & \quad \text{3b (2.0 equiv)} \\
& \quad \text{acetone, rt, 1 h} \\
& \quad \text{5a, 100%}
\end{align*}
\]

Diphenylsulfane 4a (187 mg, 1.0 mmol) in a pyrex reaction tube, benzo peroxoic acid (2b) solution (2.7 mL, 2.0 mmol, 0.75 M in acetone) was added. The reaction was stirred at room temperature for 1 h. After completion of the reaction, products were detected by TLC. Then the mixture was quenched with 2 mL sat. aq. Na2S2O5. The aqueous layer was separated and extracted with 3 x 2 mL EtOAc, and the combined organic layers dried over Na2SO4, filtered, concentrated in vacuo. The residual mixture was purified by silica gel chromatography with PE/EA (10:1 to 2:1) as eluting solvent to afford the sulfonyldibenzene 5a as colorless solid (218 mg, 100%). Pectral data were in agreement with literature values.1

1H NMR (300 MHz, CDCl3) δ 8.02 – 7.82 (m, 1H), 7.65 – 7.41 (m, 2H).

13C NMR (75 MHz, CDCl3) δ 141.51, 133.13, 129.22, 127.57.

5.3 Synthesis of Dibenzo[b,d]thiophene 5-oxide 5b

\[
\begin{align*}
\text{4b, 1.0 mmol} & \quad \text{3b (2.0 equiv)} \\
& \quad \text{DCM, overnight} \\
& \quad \text{5b, 100%}
\end{align*}
\]

To a solution of diphenylsulfane 4a (184 mg, 1.0 mmol) in 3.0 mL DCM was dropwised added benzoperoxoic acid (2b) solution (2.7 mL, 2.0 mmol, 0.75 M in acetone) at 0 °C. The mixture was warmed up to room temperature and stirred overnight. After completion of the reaction, products were detected by TLC. Then the mixture was quenched with 3 mL sat. aq. Na2S2O5. The aqueous layer was separated and extracted with 3 x 3 mL DCM, and the combined organic layers dried over Na2SO4, filtered, concentrated in vacuo. The residual mixture was purified by silica gel chromatography with PE/EA (2:1) as eluting solvent to afford the sulfonyldibenzene 5a as colorless solid (200 mg, 100%). Pectral data were in agreement with literature values.2

1H NMR (300 MHz, CDCl3) δ 8.00 – 7.86 (m, 1H), 7.75 (d, J = 7.5 Hz, 1H), 7.54 (td, J = 7.5, 1.2 Hz, 1H), 7.45 (td, J = 7.5, 1.1 Hz, 1H).

13C NMR (75 MHz, CDCl3) δ 144.93, 136.88, 132.39, 129.36, 127.30, 121.79.

5.4 Synthesis of Quinoline 1-oxide 5c

\[
\begin{align*}
\text{4c, 1.0 mmol} & \quad \text{3b (2.0 equiv)} \\
& \quad \text{DCM, overnight} \\
& \quad \text{5c, 100%}
\end{align*}
\]

To a solution of quinoline 4c (129 mg, 1.0 mmol) in 3.0 mL DCM was dropwised added benzoperoxoic acid (2b) solution (2.7 mL, 2.0 mmol, 0.75 M in acetone) at 0 °C. The mixture was warmed up to room temperature and stirred overnight. After completion of the reaction, products were detected by TLC. Then Ph3P (262 mg, 1.0 mmol) was added and stirred at room temperature for 2 h. The mixture was concentrated in vacuo and purified by silica gel chromatography with EA/MeOH (8:1) as eluting solvent to afford the quinoline 1-oxide 5c as colorless solid (145 mg, 100%). Pectral data were in agreement with literature values.3

1H NMR (300 MHz, CDCl3) δ 8.63 (s, 1H), 8.42 (s, 1H), 7.74 (s, 1H), 7.62 (s, 2H), 7.52 (s, 1H), 7.18 (s, 1H).

13C NMR (75 MHz, CDCl3) δ 141.22, 135.30, 130.22, 130.10, 128.46, 127.89, 125.67, 120.71, 119.40.
6. Experimental Procedures

6.1 General Procedure

General Procedure A: Without Chromatographic Purification and Quenching
To a pyrex reaction tube, aldehydes 1 (0.5 mmol) and acetone (0.5 mL) were added. The reaction mixtures were placed under sunlight and stirred under open air at room temperature. The reaction was monitored by thin layer chromatography (TLC). After completion of the reaction the mixture was concentrated in vacuo, to give the pure carboxylic acid 2 without chromatographic purification.

General Procedure B: Chromatographic Purification Free but With Quenching
To a pyrex reaction tube, aldehydes 1 (0.5 mmol) and acetone (0.5 mL) were added. The reaction mixtures were placed under sunlight and stirred under open air at room temperature. The reaction was monitored by thin layer chromatography (TLC). After completion of the reaction, the mixture was concentrated in vacuo and 2 mL EtOAc and 2 mL sat. aq. Na₂S₂O₃. The aqueous layer was separated and extracted with 3 x 2 mL EtOAc, and the combined organic layers dried over Na₂SO₄, filtered, concentrated in vacuo. The pure carboxylic acid 2 was obtained without chromatographic purification.

General Procedure C: With Chromatographic Purification but Without Quenching
To a pyrex reaction tube, aldehydes 1 (0.5 mmol) and acetone (0.5 mL) were added. The reaction mixtures were placed under sunlight and stirred under open air at room temperature. The reaction was monitored by thin layer chromatography (TLC). After completion of the reaction, the mixture was concentrated in vacuo, purified by column chromatography on silica gel to give the pure carboxylic acid 2.

General Procedure D: With Chromatographic Purification and Quenching
To a pyrex reaction tube, aldehydes 1 (0.5 mmol) and acetone (0.5 mL) were added. The reaction mixtures were placed under sunlight and stirred under open air at room temperature. The reaction was monitored by thin layer chromatography (TLC). After completion of the reaction, the mixture was concentrated in vacuo, 2 mL EtOAc 2 mL and sat. aq. Na₂S₂O₃ were added. The aqueous layer was separated and extracted with 3 x 2 mL EtOAc, and the combined organic layers dried over Na₂SO₄, filtered, concentrated in vacuo, purified by column chromatography on silica gel to give the pure carboxylic acid 2.

Note:
1) If the reaction solvent has evaporated, the solvent can be replenished in time (generally, the solvent is only replenished once), then continued to be irradiated in the sunlight.
2) Whether chromatographic purification is required depends on the complete conversion of aldehyde 1 or the production of by-products.
3) Generally, as long as the content of the corresponding peracetic acid produced is greater than that of the aldehyde, the conversion of the aldehyde can be promoted by vacuum concentration, and the excess peracid can be reduced to obtain the corresponding carboxylic acid.

6.2 General Procedure for Gram-scale Reaction

6.2.1 Gram-scale Synthesis of Benzoic Acid

To a 100-mL pyrex reaction tube, benzaldehyde 1b (20 mmol) and acetone (20 mL) were added. The reaction mixtures were placed under sunlight and stirred under open air at room temperature for 8 h. The reaction was detected by ¹H NMR and it showed that the conversion of benzaldehyde 1b was 63%, which contained 33% of...
benzoic acid 2b and 30% of peroxybenzoic acid 3b. Removed the reaction tube to the fume hood and the mixtures were stirred in the dark at room temperature for another 17 h. The reaction was detected by 1H NMR and it showed that the conversion of benzaldehyde 1b was 76%, which contained 56% of benzoic acid 2b and 20% of peroxybenzoic acid 3b. The next day, and the mixture continued to be placed under sunlight, stirred under open air at room temperature for 7 h. The reaction was detected by 1H NMR and it showed that the conversion of benzaldehyde 1b was 92%, which contained 64% of benzoic acid 2b and 28% of peroxybenzoic acid 3b (Figure 5B, step 1). Then the mixtures were concentrated in vacuo, detected by 1H NMR and it showed that the conversion of benzaldehyde 1b was 100%, which contained 85% of benzoic acid 2b and 15% of peroxybenzoic acid 3b (Figure 5B, step 2). Then 20 mL DCM and 20 mL sat. aq. Na2SO3 was added. The aqueous layer was separated and extracted with 3 x 20 mL DCM, and the combined organic layers dried over Na2SO4, filtered, concentrated in vacuo, without column chromatography on silica gel to give the benzoic acid 2b as colorless solid (2.39 g, 98%).

Note: More reaction information: place (Heidelberg, Germany).

First day, weather (sunny); outdoor temperature (4-10 °C); humidity (43-75%); barometer (1016 mbar); time (from 09:00 AM to 17:00 PM); date (27.02.2022).
In the dark, indoor temperature (22 °C); time (from 17:00 PM to 10:00 AM ); date (27.02.2022-28.02.2022).
The next day, weather (sunny); outdoor temperature (2-12 °C); humidity (43-66%); barometer (1015 mbar); time (from 10:00 AM to 17:00 PM); date (28.02.2022).

Figure 6. Gram-scale synthesis of PhCHO. Figure A, reaction process of gram-scale synthesis of PhCHO (black line: in the dark). Figure B, the proportion of products after different processes (step 1: after reaction; step 2: after concentration; step 3: after quenching). Figure C, 1H NMR spectra after reaction, concentration and quenching.

6.2.2 Gram-scale Synthesis of Octanoic Acid

To a 100-mL pyrex reaction tube, octanal 1am (20 mmol) and acetone (5 mL) were added. The reaction mixtures were placed under sunlight and stirred under open air at room temperature. The reaction was monitored by thin layer chromatography (TLC). The reaction was not completed on the first day (10 h, cov. 72% of 1am), removed the reaction tube to the fume hood. The next day, since the solvent basically evaporated on the first day, added 2 mL acetone, and the mixture continued to be placed under sunlight, stirred under open air at room temperature
for 10 h (cov. 93% of 1am). On the third day, added 2 mL acetone, placed under sunlight, stirred under open air at room temperature for 10 h (cov. 98% of 1am). Then the mixture was concentrated in vacuo to give the octanoic acid 2am as colorless oil (2.76 g, 96%).

**Note:** More reaction information: place (Heidelberg, Germany).

First day, weather (sunny); outdoor temperature (19-28 °C); humidity (56-74%); barometer (1002 mbar); time (from 09:00 AM to 19:00 PM); date (18.07.2021).

Second day, weather (partly sunny); outdoor temperature (21-26 °C); humidity (54-75%); barometer (1002 mbar); time (from 09:00 AM to 19:00 PM); date (19.07.2021).

Third day, weather (partly sunny); outdoor temperature (19-25 °C); humidity (48-78%); barometer (1002 mbar); time (from 09:00 AM to 19:00 PM); date (20.07.2021).

### 6.2.3 Gram-scale Synthesis of Aspirin

![Chemical structure](image)

**Step 1:** To a solution of 2-hydroxybenzaldehyde 1au (2.13 mL, 20 mmol) in 40 mL DCM was added pyridine (2.42 mL, 30 mmol) at 0 °C and the reaction was stirred at this temperature for 10 min. Then acetic anhydride (2.83 mL, 4.2 mmol) was slowly added and the mixture was warmed up to room temperature and stirred for 3 h. After completion of the reaction, the mixture was removed to ice bath, 1 M HCl was slowly added until pH = 5-6 flowed by adding 30 mL saturated brine and extracted with 30 mL EtOAc. The aqueous layer was separated and extracted with 2 x 30 mL EtOAc, and the combined organic layers dried over Na₂SO₄, filtered, concentrated in vacuo to give the 2-formylphenyl acetate 2au as colorless oil, which was directly used in the next step without chromatographic purification.

**Step 2:** To a 100-mL pyrex reaction tube, 2-formylphenyl acetate 2au and acetone (20 mL) were added. The reaction mixtures were placed under sunlight and stirred under open air at room temperature. The reaction was monitored by thin layer chromatography (TLC). The reaction was not completed on the first day (8 h), removed the reaction tube to the fume hood. The next day, since the solvent basically evaporated on the first day, added 10 mL acetone, and the mixture continued to be placed under sunlight, stirred under open air at room temperature for 8 h. On the third day, added 5 mL acetone, placed under sunlight, stirred under open air at room temperature for 9 h. Then the mixture was concentrated in vacuo, added 5 mL Et₂O sonicated, filtered, and washed with 5mL Et₂O, collected filter residue and added 5 mL Et₂O sonicated, filtered, and washed with 5 mL Et₂O, and the mother liquor is recovered and purified by column chromatography, collected all products, to obtain the Aspirin 2au as colorless solid (3.12 g, 95%).

**Note:** More reaction information: place (Heidelberg, Germany).

First day, weather (partly sunny); outdoor temperature (18-24 °C); humidity (40-68%); barometer (995 mbar); time (from 09:00 AM to 17:00 PM); date (29.07.2021).

Second day, weather (sunny); outdoor temperature (20-26 °C); humidity (41-68%); barometer (995 mbar); time (from 09:00 AM to 17:00 PM); date (30.07.2021).

Third day, weather (partly cloudy); outdoor temperature (19-24 °C); humidity (40-68%); barometer (995 mbar); time (from 09:00 AM to 18:00 PM); date (31.07.2021).

### 7. Spectral Data of Products

4-Methoxybenzoic acid (2a)
Prepared as described in general procedure D: 4-methoxybenzaldehyde 1a (0.5 mmol) in 0.5 mL acetone, sunlight, open air, room temperature, 2 h. The residue was purified by silica gel chromatography with DCM/MeOH 15:1 (v/v) as eluting solvent to afford 4-methoxybenzoic acid 2a as colorless solid (75.3 mg, 99%).

**Note:** More reaction information: weather (sunny); outdoor temperature (3-8 °C); humidity (42-58%); barometer (1002 mbar); time (from 10:30 AM to 12:30 PM); date (05.03.2022); place (Heidelberg, Germany).

1H NMR (300 MHz, DMSO) δ 12.61 (br, 1H), 7.90 (d, J = 8.8 Hz, 2H), 6.99 (d, J = 8.6 Hz, 2H), 3.80 (s, 3H).

13C NMR (75 MHz, DMSO) δ 167.15, 162.93, 131.44, 123.09, 113.85, 55.44.

Benzoic acid (2b)

Prepared as described in general procedure B: benzaldehyde 1b (0.5 mmol) in 0.5 mL acetone, sunlight, open air, room temperature, 4 h. To give benzoic acid 2b as colorless solid (60.9 mg, 99%).

**Note:** More reaction information: weather (sunny); outdoor temperature (3-8 °C); humidity (42-58%); barometer (1002 mbar); time (from 10:30 AM to 14:30 PM); date (05.03.2022); place (Heidelberg, Germany).

1H NMR (300 MHz, DMSO) δ 12.94 (br, 1H), 7.95 (dd, J = 5.2, 3.3 Hz, 2H), 7.64 – 7.56 (m, 1H), 7.48 (dd, J = 10.3, 4.6 Hz, 2H).

13C NMR (75 MHz, DMSO) δ 167.40, 132.88, 130.85, 129.33, 128.59.

2-Methoxybenzoic acid (2c)

Prepared as described in general procedure B: 2-methoxybenzaldehyde 1c (0.5 mmol) in 0.5 mL acetone, sunlight, open air, room temperature, 6 h. To give 2-methoxybenzoic acid 2c as colorless solid (73.0 mg, 96%).

**Note:** More reaction information: weather (sunny); outdoor temperature (19-27 °C); humidity (44-88%); barometer (1002 mbar); time (from 08:30 AM to 14:30 PM); date (11.06.2022); place (Heidelberg, Germany).

1H NMR (300 MHz, DMSO) δ 12.59 (br, 1H), 7.65 (dd, J = 7.6, 1.8 Hz, 1H), 7.48 (dd, J = 8.5, 7.4, 1.8 Hz, 1H), 7.09 (d, J = 8.1 Hz, 1H), 6.98 (td, J = 7.5, 0.9 Hz, 1H), 3.80 (s, 3H).

13C NMR (75 MHz, DMSO) δ 167.46, 158.16, 133.11, 130.74, 121.38, 120.09, 112.46, 55.74.

2,4-Dimethoxybenzoic acid (2d)

Prepared as described in general procedure D: 2,4-dimethoxybenzaldehyde 1d (0.5 mmol) in 0.5 mL acetone, sunlight, open air, room temperature, 4 h. The residue was purified by silica gel chromatography with DCM/MeOH 15:1 (v/v) as eluting solvent to afford 2,4-dimethoxybenzoic acid 2d as colorless solid (74.0 mg, 81%).

**Note:** More reaction information: weather (sunny); outdoor temperature (3-8 °C); humidity (42-58%); barometer (1002 mbar); time (from 10:30 AM to 14:30 PM); date (05.03.2022); place (Heidelberg, Germany).

1H NMR (300 MHz, DMSO) δ 12.16 (br, 1H), 7.71 (d, J = 8.6 Hz, 1H), 6.62 – 6.53 (m, 2H), 3.81 (s, 6H).

13C NMR (75 MHz, DMSO) δ 166.52, 163.77, 160.71, 133.36, 112.71, 110.18, 98.92, 55.79, 55.51.

4-Methylbenzoic acid (2e)
Prepared as described in general procedure D: 4-methylbenzaldehyde 1e (0.5 mmol) in 0.5 mL acetone, sunlight, open air, room temperature, 4 h. The residue was purified by silica gel chromatography with DCM/MeOH 15:1 (v/v) as eluting solvent to afford 4-methylbenzoic acid 2e as colorless solid (64.2 mg, 94%).

**Note:** More reaction information: weather (sunny); outdoor temperature (3-8 °C); humidity (42-58%); barometer (1002 mbar); time (from 10:30 AM to 14:30 PM); date (05.03.2022); place (Heidelberg, Germany).

**1H NMR** (300 MHz, DMSO) δ 12.79 (br, 1H), 7.84 (d, J = 8.1 Hz, 2H), 7.27 (d, J = 7.5 Hz, 2H), 2.34 (s, 3H).

**13C NMR** (75 MHz, DMSO) δ 167.41, 143.06, 129.41, 129.16, 128.13, 21.16.

3-Methylbenzoic acid (2f)

Prepared as described in general procedure D: 3-methylbenzaldehyde 1f (0.5 mmol) in 0.5 mL acetone, sunlight, open air, room temperature, 8 h. The residue was purified by silica gel chromatography with DCM/MeOH 15:1 (v/v) as eluting solvent to afford 3-methylbenzoic acid 2f as colorless solid (61.5 mg, 90%).

**Note:** More reaction information: weather (partly sunny); outdoor temperature (27-33 °C); humidity (38-57%); barometer (995 mbar); time (from 09:00 AM to 17:00 PM); date (17.06.2021); place (Heidelberg, Germany).

**1H NMR** (300 MHz, CDCl3) δ 11.71 (br, 1H), 7.95-7.93 (m, 2H), 7.50 – 7.33 (m, 2H), 2.43 (s, 3H).

**13C NMR** (75 MHz, CDCl3) δ 172.67, 138.28, 134.58, 130.69, 129.24, 128.35, 127.36, 21.21.

4-Ethylbenzoic acid (2g)

Prepared as described in general procedure D: 4-ethylbenzaldehyde 1g (0.5 mmol) in 0.5 mL acetone, sunlight, open air, room temperature, 9 h. The residue was purified by silica gel chromatography with DCM/MeOH 15:1 (v/v) as eluting solvent to afford 4-ethylbenzoic acid 2g as colorless solid (68.9 mg, 93%).

**Note:** More reaction information: weather (partly sunny); outdoor temperature (27-33 °C); humidity (38-57%); barometer (995 mbar); time (from 09:00 AM to 18:00 PM); date (17.06.2021); place (Heidelberg, Germany).

**1H NMR** (300 MHz, CDCl3) δ 12.03 (br, 1H), 8.06 (d, J = 8.2 Hz, 2H), 7.31 (d, J = 8.2 Hz, 2H), 2.74 (q, J = 7.6 Hz, 2H), 1.28 (t, J = 7.6 Hz, 3H).

**13C NMR** (75 MHz, CDCl3) δ 172.61, 150.79, 130.36, 127.99, 126.82, 29.01, 15.12.

4-(tert-Butyl)benzoic acid (2h)

Prepared as described in general procedure D: 4-(tert-butyl)benzaldehyde 1h (0.5 mmol) in 0.5 mL acetone, sunlight, open air, room temperature, 9 h. The residue was purified by silica gel chromatography with DCM/MeOH 15:1 (v/v) as eluting solvent to afford 4-(tert-butyl)benzoic acid 2h as colorless solid (86.2 mg, 97%).

**Note:** More reaction information: weather (sunny); outdoor temperature (19-29 °C); humidity (44-88%); barometer (1002 mbar); time (from 08:30 AM to 17:30 PM); date (11.06.2022); place (Heidelberg, Germany).

**1H NMR** (300 MHz, DMSO) δ 12.77 (br, 1H), 7.88 (d, J = 8.2 Hz, 2H), 7.47 (d, J = 8.1 Hz, 2H), 1.28 (t, J = 7.6 Hz, 3H).

**13C NMR** (75 MHz, DMSO) δ 167.32, 155.78, 129.26, 128.13, 125.33, 34.75, 30.86.

2,4,6-Trimethylbenzoic acid (2i)

Prepared as described in general procedure C: 2,4,6-trimethylbenzaldehyde 1i (0.5 mmol) in 0.5 mL acetone, sunlight, open air, room temperature, 9 h. The residue was purified by silica gel chromatography with DCM/MeOH 15:1 (v/v) as eluting solvent to afford 2,4,6-trimethylbenzoic acid 2i as colorless solid (67.3 mg, 82%).

**Note:** More reaction information: weather (sunny); outdoor temperature (19-29 °C); humidity (44-88%); barometer (1002 mbar); time (from 08:30 AM to 17:30 PM); date (11.06.2022); place (Heidelberg, Germany).

**1H NMR** (300 MHz, DMSO) δ 12.77 (br, 1H), 7.88 (d, J = 8.2 Hz, 2H), 7.47 (d, J = 8.1 Hz, 2H), 1.26 (s, 9H).

**13C NMR** (75 MHz, DMSO) δ 167.32, 155.78, 129.26, 128.13, 125.33, 34.75, 30.86.
Note: More reaction information: weather (partly sunny); outdoor temperature (27-33 °C); humidity (38-57%); barometer (995 mbar); time (from 09:00 AM to 18:00 PM); date (17.06.2021); place (Heidelberg, Germany).

$^1$H NMR (300 MHz, CDCl$_3$) δ 10.37 (br, 1H), 6.90 (s, 2H), 2.44 (s, 6H), 2.31 (s, 3H).

$^{13}$C NMR (75 MHz, CDCl$_3$) δ 175.76, 140.02, 136.09, 129.32, 128.75, 21.09, 20.27.

2,3,4,5,6-Pentamethylbenzoic acid (2j)

Prepared as described in general procedure D: 2,3,4,5,6-pentamethylbenzaldehyde 1j (0.5 mmol) in 0.5 mL acetone, sunlight, open air, room temperature, 10 h. The residue was purified by silica gel chromatography with DCM/MeOH 15:1 (v/v) as eluting solvent to afford 2,3,4,5,6-pentamethylbenzoic acid 2j as colorless solid (86.0 mg, 90%).

Note: More reaction information: weather (sunny); outdoor temperature (16-24 °C); humidity (42-73%); barometer (1009 mbar); time (from 08:30 AM to 18:30 PM); date (13.06.2021); place (Heidelberg, Germany).

$^1$H NMR (300 MHz, CDCl$_3$) δ 10.21 (br, 1H), 2.32 (s, 6H), 2.25 (s, 3H), 2.23 (s, 6H).

$^{13}$C NMR (75 MHz, CDCl$_3$) δ 177.14, 136.42, 132.83, 132.06, 128.87, 17.64, 16.72, 16.09.

4-(Methoxycarbonyl)benzoic acid (2k)

Prepared as described in general procedure B: methyl 4-formylbenzoate 1k (0.5 mmol) in 0.5 mL acetone, sunlight, open air, room temperature, 4 h. To give 4-(methoxycarbonyl)benzoic acid 2k as light yellow solid (84.4 mg, 94%).

Note: More reaction information: weather (sunny); outdoor temperature (3-8 °C); humidity (42-58%); barometer (1002 mbar); time (from 10:30 AM to 14:30 PM); date (05.03.2022); place (Heidelberg, Germany).

$^1$H NMR (300 MHz, DMSO) δ 11.70 (br, 1H), 8.01 (s, 4H), 3.84 (s, 3H).

$^{13}$C NMR (75 MHz, DMSO) δ 166.64, 165.64, 134.86, 133.19, 129.59, 129.35, 52.42.

3-(Methoxycarbonyl)benzoic acid (2l)

Prepared as described in general procedure B: methyl 3-formylbenzoate 1l (0.5 mmol) in 0.5 mL acetone, sunlight, open air, room temperature, 10 h. To give 3-(methoxycarbonyl)benzoic acid 2l as light yellow solid (88.6 mg, 98%).

Note: More reaction information: weather (sunny); outdoor temperature (16-24 °C); humidity (42-73%); barometer (1009 mbar); time (from 08:30 AM to 18:30 PM); date (13.06.2021); place (Heidelberg, Germany).

$^1$H NMR (300 MHz, DMSO) δ 10.92 (br, 1H), 8.44 (s, 1H), 8.23 – 8.02 (m, 2H), 7.60 (t, $J = 6.7$ Hz, 1H), 7.60 (t, $J = 6.7$ Hz, 1H), 3.85 (s, 3H).

$^{13}$C NMR (75 MHz, DMSO) δ 166.54, 165.58, 133.77, 133.20, 131.41, 130.41, 130.07, 129.87, 129.28, 52.39.

4-Acetamidobenzoic acid (2m)

Prepared as described in general procedure D: N-(4-formylphenyl)acetamide 1m (0.5 mmol) in 0.5 mL acetone, sunlight, open air, room temperature. The reaction was monitored by thin layer chromatography (TLC). The
reaction was not completed on the first day (9 h), continued to react in the sun on the next day (9 h). After completion of the reaction, the mixture was quenched with 2 mL sat. aq. Na$_2$S$_2$O$_3$ and extracted with 2 mL EtOAc. The aqueous layer was separated and extracted with 3 x 2 mL EtOAc, and the combined organic layers dried over Na$_2$SO$_4$, filtered, concentrated in vacuo, the residue was purified by silica gel chromatography with DCM/MeOH 15:1 (v/v) as eluting solvent to afford 4-acetamidobenzoic acid 2m as light yellow solid (71.6 mg, 80%).

**Note:** More reaction information: place (Heidelberg, Germany).

First day, weather (partly sunny); outdoor temperature (27-33 °C); humidity (44-60%); barometer (999 mbar); time (from 10:30AM to 19:30 PM); date (19.06.2021).

Next day, weather (partly cloudy); outdoor temperature (24-29 °C); humidity (42-69%); barometer (992 mbar); time (from 10:00AM to 19:00 PM); date (20.06.2021).

$^1$H NMR (300 MHz, DMSO) δ 12.54 (br, 1H), 10.52 (s, 1H), 7.86 (d, $J = 8.5$ Hz, 2H), 7.73 (d, $J = 8.6$ Hz, 2H), 2.09 (s, 3H).

$^{13}$C NMR (75 MHz, DMSO) δ 169.03, 167.08, 143.49, 130.35, 124.98, 118.25, 24.18.

4-Hydroxybenzoic acid (2n)

Prepared as described in general procedure D: 4-hydroxybenzaldehyde 1n (0.5 mmol) in 0.5 mL acetone, sunlight, open air, room temperature. The reaction was monitored by thin layer chromatography (TLC). The reaction was not completed on the first day (9 h), continued to react in the sun on the next day (9 h). After completion of the reaction, the mixture was quenched with 2 mL sat. aq. Na$_2$S$_2$O$_3$ and extracted with 2 mL EtOAc. The aqueous layer was separated and extracted with 3 x 2 mL EtOAc, and the combined organic layers dried over Na$_2$SO$_4$, filtered, concentrated in vacuo, the residue was purified by silica gel chromatography with DCM/MeOH 10:1 (v/v) as eluting solvent to afford 4-hydroxybenzoic acid 2n as light yellow solid (52.1 mg, 75%).

**Note:** More reaction information: place (Heidelberg, Germany).

First day, weather (partly sunny); outdoor temperature (27-33 °C); humidity (44-60%); barometer (999 mbar); time (from 10:30AM to 19:30 PM); date (19.06.2021).

Next day, weather (partly cloudy); outdoor temperature (24-29 °C); humidity (42-69%); barometer (992 mbar); time (from 10:00AM to 19:00 PM); date (20.06.2021).

$^1$H NMR (300 MHz, DMSO) δ 10.47 (br, 1H), 7.78 (d, $J = 8.6$ Hz, 2H), 6.81 (d, $J = 8.6$ Hz, 2H), 3.34 (br, 1H).

$^{13}$C NMR (75 MHz, DMSO) δ 167.24, 161.64, 131.57, 121.45, 115.16.

4-Phenoxybenzoic acid (2o)

Prepared as described in general procedure C: 4-phenoxybenzaldehyde 1o (0.5 mmol) in 0.5 mL acetone, sunlight, open air, room temperature, 10 h. The residue was purified by silica gel chromatography with DCM/MeOH 15:1 (v/v) as eluting solvent to afford 4-phenoxybenzoic acid 2o as colorless solid (85.3 mg, 80%).

**Note:** More reaction information: weather (partly sunny); outdoor temperature (27-33 °C); humidity (38-57%); barometer (995 mbar); time (from 09:00 AM to 19:00 PM); date (17.06.2021); place (Heidelberg, Germany).

$^1$H NMR (300 MHz, CDCl$_3$) δ 11.57 (br, 1H), 7.94 – 7.83 (m, 1H), 7.79 – 7.68 (m, 1H), 7.50 – 7.34 (m, 3H), 7.28 (ddd, $J = 7.9$, 2.3, 0.7 Hz, 1H), 7.17 (t, $J = 7.4$ Hz, 1H), 7.12 – 6.99 (m, 2H).

$^{13}$C NMR (75 MHz, CDCl$_3$) δ 171.83, 157.62, 156.48, 131.00, 129.94, 129.86, 124.82, 124.08, 123.89, 119.88, 119.20.

4-Fluorobenzoic acid (2p)

Prepared as described in general procedure B: 4-fluorobenzaldehyde 1p (0.5 mmol) in 0.5 mL acetone, sunlight, open air, room temperature, 2 h. To give 4-fluorobenzoic acid 2p as colorless solid (68.4 mg, 98%).

**Note:** More reaction information: weather (partly sunny); outdoor temperature (27-33 °C); humidity (38-57%); barometer (995 mbar); time (from 09:00 AM to 19:00 PM); date (17.06.2021); place (Heidelberg, Germany).

$^1$H NMR (300 MHz, CDCl$_3$) δ 11.57 (br, 1H), 7.94 – 7.83 (m, 1H), 7.79 – 7.68 (m, 1H), 7.50 – 7.34 (m, 3H), 7.28 (ddd, $J = 7.9$, 2.3, 0.7 Hz, 1H), 7.17 (t, $J = 7.4$ Hz, 1H), 7.12 – 6.99 (m, 2H).

$^{13}$C NMR (75 MHz, CDCl$_3$) δ 171.83, 157.62, 156.48, 131.00, 129.94, 129.86, 124.82, 124.08, 123.89, 119.88, 119.20.
**Note:** More reaction information: weather (sunny); outdoor temperature (3-8 °C); humidity (42-58%); barometer (1002 mbar); time (from 10:30 AM to 12:30 PM); date (05.03.2022); place (Heidelberg, Germany).

1H NMR (300 MHz, DMSO) δ 12.54 (br, 1H), 7.99 (dd, J = 8.5, 5.7 Hz, 2H), 7.28 (t, J = 8.1 Hz, 2H).

13C NMR (75 MHz, DMSO) δ 166.47, 165.00 (d, J = 250.6 Hz), 132.16 (d, J = 9.5 Hz), 127.44 (d, J = 2.8 Hz), 115.63 (d, J = 21.9 Hz).

19F NMR (283 MHz, DMSO) δ -106.93.

2-Fluorobenzoic acid (2q)

Prepared as described in general procedure A: 2-fluorobenzaldehyde 1q (0.5 mmol) in 0.5 mL acetone, sunlight, open air, room temperature, 5 h. To give 2-fluorobenzoic acid 2q as colorless solid (69.7 mg, 99%).

**Note:** More reaction information: weather (partly cloudy); outdoor temperature (22-31 °C); humidity (38-61%); barometer (995 mbar); time (from 09:30 AM to 14:30 PM); date (16.06.2021); place (Heidelberg, Germany).

1H NMR (300 MHz, CDCl₃) δ 11.87 (br, 1H), 8.04 (td, J = 7.6, 1.7 Hz, 1H), 7.67 – 7.45 (m, 1H), 7.24 (t, J = 7.6 Hz, 1H), 7.17 (dd, J = 10.6, 8.8 Hz, 1H).

13C NMR (75 MHz, CDCl₃) δ 170.00 (d, J = 3.3 Hz), 162.62 (d, J = 262.2 Hz), 135.62 (d, J = 9.2 Hz), 132.72, 124.07 (d, J = 4.0 Hz), 117.52 (d, J = 8.9 Hz), 117.13 (d, J = 22.2 Hz).

19F NMR (283 MHz, CDCl₃) δ -108.06.

2,4,6-Trifluorobenzoic acid (2r)

Prepared as described in general procedure A: 2,4,6-trifluorobenzaldehyde 1r (0.5 mmol) in 0.5 mL acetone, sunlight, open air, room temperature, 7 h. To give 2,4,6-trifluorobenzoic acid 2r as colorless solid (86.6 mg, 98%).

**Note:** More reaction information: weather (mostly cloudy); outdoor temperature (20-27 °C); humidity (48-80%); barometer (1002 mbar); time (from 08:30 AM to 15:30 PM); date (12.06.2021); place (Heidelberg, Germany).

1H NMR (300 MHz, DMSO) δ 13.55 (br, 1H), 7.24 (s, 2H).

13C NMR (75 MHz, DMSO) δ 163.35 (dt, J = 251.0, 15.9 Hz), 161.62, 160.41 (ddd, J = 253.7, 15.9, 9.7 Hz), 109.04 (dd, J = 19.8, 15.3 Hz), 101.53 (t, J = 26.8 Hz).

19F NMR (283 MHz, DMSO) δ -103.63 (d, J = 6.9 Hz), -108.32 (d, J = 5.7 Hz).

2,3,4,5,6-Pentafluorobenzoic acid (2s)

Prepared as described in general procedure A: 2,3,4,5,6-pentafluorobenzaldehyde 1s (0.5 mmol) in 0.5 mL acetone, sunlight, open air, room temperature, 9 h. To give 2,3,4,5,6-pentafluorobenzoic acid 2s as colorless solid (106.4 mg, 99%).

**Note:** More reaction information: weather (partly cloudy); outdoor temperature (22-32 °C); humidity (38-61%); barometer (995 mbar); time (from 09:30 AM to 18:30 PM); date (16.06.2021); place (Heidelberg, Germany).

1H NMR (300 MHz, CDCl₃) δ 11.37 (br, 1H).

13C NMR (101 MHz, CDCl₃) δ 164.64, 146.29 (dtt, J = 16.4, 10.1, 4.2 Hz), 144.14 (dtt, J = 261.7, 13.3, 5.0 Hz), 137.89 (dddd, J = 15.1, 12.7, 7.1, 3.9 Hz), 106.67 (td, J = 14.0, 4.0 Hz).

19F NMR (283 MHz, CDCl₃) δ -135.17 – -139.85 (m), -146.11 (tt, J = 20.8, 6.1 Hz), -158.43 – -161.43 (m).

4-Chlorobenzoic acid (2t)

Prepared as described in general procedure A: 2,3,4,5,6-pentafluorobenzaldehyde 1s (0.5 mmol) in 0.5 mL acetone, sunlight, open air, room temperature, 9 h. To give 2,3,4,5,6-pentafluorobenzoic acid 2s as colorless solid (106.4 mg, 99%).

**Note:** More reaction information: weather (partly cloudy); outdoor temperature (22-32 °C); humidity (38-61%); barometer (995 mbar); time (from 09:30 AM to 18:30 PM); date (16.06.2021); place (Heidelberg, Germany).

1H NMR (300 MHz, CDCl₃) δ 11.37 (br, 1H).

13C NMR (101 MHz, CDCl₃) δ 164.64, 146.29 (dtt, J = 16.4, 10.1, 4.2 Hz), 144.14 (dtt, J = 261.7, 13.3, 5.0 Hz), 137.89 (dddd, J = 15.1, 12.7, 7.1, 3.9 Hz), 106.67 (td, J = 14.0, 4.0 Hz).

19F NMR (283 MHz, CDCl₃) δ -135.17 – -139.85 (m), -146.11 (tt, J = 20.8, 6.1 Hz), -158.43 – -161.43 (m).

4-Chlorobenzoic acid (2t)
Prepared as described in general procedure B: 4-chlorobenzaldehyde 1t (0.5 mmol) in 0.5 mL acetone, sunlight, open air, room temperature, 4 h. To give 4-chlorobenzoic acid 2t as colorless solid (73.9 mg, 95%).

**Note:** More reaction information: weather (sunny); outdoor temperature (3-8 °C); humidity (42-58%); barometer (1002 mbar); time (from 10:30 AM to 14:30 PM); date (05.03.2022); place (Heidelberg, Germany).

$^1$H NMR (300 MHz, DMSO) δ 10.69 (br, 1H), 7.92 (d, $J = 8.1$ Hz, 2H), 7.52 (d, $J = 6.8$ Hz, 2H).

$^{13}$C NMR (75 MHz, DMSO) δ 166.54, 137.88, 131.18, 129.70, 128.74.

3-Bromobenzoic acid (2u)$^{18}$

Prepared as described in general procedure B: 3-bromobenzaldehyde 1u (0.5 mmol) in 0.5 mL acetone, sunlight, open air, room temperature, 6 h. To give 3-bromobenzoic acid 2u as colorless solid (97.4 mg, 97%).

**Note:** More reaction information: weather (sunny); outdoor temperature (3-8 °C); humidity (41-58%); barometer (1002 mbar); time (from 10:30 AM to 16:30 PM); date (05.03.2022); place (Heidelberg, Germany).

$^1$H NMR (300 MHz, DMSO) δ 13.29 (br, 1H), 8.02 (t, $J = 1.7$ Hz, 1H), 7.91 (d, $J = 7.7$ Hz, 1H), 7.79 (d, $J = 8.0$ Hz, 1H), 7.50 – 7.40 (m, 1H).

$^{13}$C NMR (75 MHz, DMSO) δ 166.00, 135.58, 133.11, 131.80, 130.87, 128.29, 121.75.

2-Bromobenzoic acid (2v)$^{18}$

Prepared as described in general procedure C: 2-bromobenzaldehyde 1v (0.5 mmol) in 0.5 mL acetone, sunlight, open air, room temperature, 9 h. The residue was purified by silica gel chromatography with DCM/MeOH 15:1 (v/v) as eluting solvent to give 2-bromobenzoic acid 2v as colorless solid (91.9 mg, 94%).

**Note:** More reaction information: weather (sunny); outdoor temperature (16-24 °C); humidity (42-73%); barometer (1009 mbar); time (from 08:30 AM to 17:30 PM); date (13.06.2021); place (Heidelberg, Germany).

$^1$H NMR (300 MHz, DMSO) δ 11.67 (br, 1H), 7.79 – 7.63 (m, 2H), 7.56 – 7.33 (m, 2H).

$^{13}$C NMR (75 MHz, DMSO) δ 167.42, 133.81, 132.54, 130.66, 127.73, 120.02.

3,5-Dibromobenzoic acid (2w)$^{19}$

Prepared as described in general procedure D: 3,5-dibromobenzaldehyde 1w (0.5 mmol) in 0.5 mL acetone, sunlight, open air, room temperature, 6 h. The residue was purified by silica gel chromatography with DCM/MeOH 15:1 (v/v) as eluting solvent to afford 3,5-dibromobenzoic acid 2w as light yellow solid (111.7 mg, 80%).

**Note:** More reaction information: weather (sunny); outdoor temperature (16-22 °C); humidity (47-73%); barometer (1009 mbar); time (from 08:30 AM to 14:30 PM); date (13.06.2021); place (Heidelberg, Germany).

$^1$H NMR (300 MHz, DMSO) δ 13.53 (br, 1H), 8.07 – 7.89 (m, 3H).

$^{13}$C NMR (75 MHz, DMSO) δ 164.74, 133.81, 132.54, 130.66, 127.73, 120.02.

5-Bromo-2-methoxybenzoic acid (2x)$^{20}$

Prepared as described in general procedure D: 3,5-dibromobenzaldehyde 1w (0.5 mmol) in 0.5 mL acetone, sunlight, open air, room temperature, 6 h. The residue was purified by silica gel chromatography with DCM/MeOH 15:1 (v/v) as eluting solvent to afford 3,5-dibromobenzoic acid 2w as light yellow solid (111.7 mg, 80%).

**Note:** More reaction information: weather (sunny); outdoor temperature (16-22 °C); humidity (47-73%); barometer (1009 mbar); time (from 08:30 AM to 14:30 PM); date (13.06.2021); place (Heidelberg, Germany).

$^1$H NMR (300 MHz, DMSO) δ 13.53 (br, 1H), 8.07 – 7.89 (m, 3H).

$^{13}$C NMR (75 MHz, DMSO) δ 164.74, 137.42, 134.69, 130.97, 122.69.
Prepared as described in general procedure D: 5-bromo-2-methoxybenzaldehyde 1x (0.5 mmol) in 0.5 mL acetone, sunlight, open air, room temperature, 10 h. The residue was purified by silica gel chromatography with DCM/MeOH 15:1 (v/v) as eluting solvent to afford 5-bromo-2-methoxybenzoic acid 2x as light yellow solid (108.4 mg, 94%).

**Note:** More reaction information: weather (partly sunny); outdoor temperature (27-33 °C); humidity (38-57%); barometer (995 mbar); time (from 09:00 AM to 19:00 PM); date (17.06.2021); place (Heidelberg, Germany).

1H NMR (300 MHz, CDCl$_3$) δ 10.20 (br, 1H), 8.14 (d, $J = 2.6$ Hz, 1H), 7.60 (dd, $J = 8.9$, 2.6 Hz, 1H), 6.93 (d, $J = 8.9$ Hz, 1H), 4.01 (s, 3H).

13C NMR (75 MHz, CDCl$_3$) δ 165.32, 157.38, 137.49, 135.67, 119.16, 113.93, 113.69, 56.85.

4-(Trifluoromethyl)benzoic acid (2y)

Prepared as described in general procedure C: 4-(trifluoromethyl)benzaldehyde 1y (0.5 mmol) in 0.5 mL acetone, sunlight, open air, room temperature, 4 h. The residue was purified by silica gel chromatography with DCM/MeOH 15:1 (v/v) as eluting solvent to afford 4-(trifluoromethyl)benzoic acid 2y as colorless solid (88.2 mg, 93%).

**Note:** More reaction information: weather (sunny); outdoor temperature (3-8 °C); humidity (41-58%); barometer (1002 mbar); time (from 10:30 AM to 16:30 PM); date (05.03.2022); place (Heidelberg, Germany).

1H NMR (300 MHz, DMSO) δ 13.43 (br, 1H), 8.11 (d, $J = 8.0$ Hz, 2H), 7.91 – 7.73 (m, 2H).

13C NMR (75 MHz, DMSO) δ 166.29, 134.68, 132.62 (q, $J = 32.0$ Hz), 130.14 , 125.52 (d, $J = 3.6$ Hz), 123.86(q, $J = 270.75$).

19F NMR (283 MHz, DMSO) δ -56.59 – -68.84 (m).

3,5-Bis(trifluoromethyl)benzoic acid (2z)

Prepared as described in general procedure C: 3,5-bis(trifluoromethyl)benzaldehyde 1z (0.5 mmol) in 0.5 mL acetone, sunlight, open air, room temperature, 10 h. The residue was purified by silica gel chromatography with DCM/MeOH 15:1 (v/v) as eluting solvent to afford 3,5-bis(trifluoromethyl)benzoic acid 2z as colorless solid (122.1 mg, 95%).

**Note:** More reaction information: weather (sunny); outdoor temperature (16-24 °C); humidity (42-73%); barometer (1009 mbar); time (from 08:30 AM to 18:30 PM); date (13.06.2021); place (Heidelberg, Germany).

1H NMR (400 MHz, DMSO) δ 12.89 (br, 1H), 8.34 (s, 2H), 8.19 (s, 1H).

13C NMR (101 MHz, DMSO) δ 164.68, 133.73, 130.93 (q, $J = 33.5$ Hz), 129.46, 125.98, 122.92 (q, $J = 272.8$ Hz).

19F NMR (283 MHz, DMSO) δ -50.60 – -78.53 (m).

4-Cyanobenzoic acid (2aa)

Prepared as described in general procedure D: 4-cyanobenzaldehyde 1aa (0.5 mmol) in 0.5 mL acetone, sunlight, open air, room temperature, 5 h. The residue was purified by silica gel chromatography with DCM/MeOH 15:1 (v/v) as eluting solvent to afford 4-cyanobenzoic acid 2aa as colorless solid (71.5 mg, 97%).

**Note:** More reaction information: weather (sunny); outdoor temperature (3-8 °C); humidity (41-58%); barometer (1002 mbar); time (from 10:30 AM to 15:30 PM); date (05.03.2022); place (Heidelberg, Germany).

1H NMR (300 MHz, DMSO) δ 13.32 (br, 1H), 8.05 (d, $J = 8.2$ Hz, 2H), 7.92 (d, $J = 8.2$ Hz, 2H).
\[^{13}\text{C}\ \text{NMR}\ (75\ \text{MHz, DMSO})\ \delta 166.13, 134.95, 132.67, 129.98, 118.24, 115.13.\]

\[\text{[1,1'-Biphenyl]-2-carboxylic acid (2ab)}\]

Prepared as described in general procedure D: [1,1'-biphenyl]-2-carbaldehyde 1ab (0.5 mmol) in 0.5 mL acetone, sunlight, open air, room temperature, 9 h. The residue was purified by silica gel chromatography with DCM/MeOH 15:1 (v/v) as eluting solvent to afford [1,1'-biphenyl]-2-carboxylic acid 2ab as colorless solid (43.6 mg, 44%).

\[^{1}\text{H}\ \text{NMR}\ (300\ \text{MHz, DMSO})\ \delta 12.75\ \text{br, 1H}, 7.73\ (d, \ J = 7.4\ Hz, 1H), 7.57\ (t, \ J = 7.3\ Hz, 1H), 7.50 - 7.27\ (m, 7H).\]

\[^{13}\text{C}\ \text{NMR}\ (75\ \text{MHz, DMSO})\ \delta 169.67, 140.95, 140.88, 132.34, 130.84, 130.48, 129.07, 128.30, 128.11, 127.26, 127.16.\]

\[^{1,1'}\text{Biphenyl}-4-carboxylic acid (2ac)}\]

Prepared as described in general procedure D: [1,1'-biphenyl]-4-carbaldehyde 1ac (0.5 mmol) in 0.5 mL acetone, sunlight, open air, room temperature, 9 h. The residue was purified by silica gel chromatography with DCM/MeOH 15:1 (v/v) as eluting solvent to afford [1,1'-biphenyl]-4-carboxylic acid 2ac as colorless solid (83.5 mg, 84%).

\[^{1}\text{H}\ \text{NMR}\ (300\ \text{MHz, DMSO})\ \delta 13.00\ \text{br, 1H}, 8.04\ (d, \ J = 8.2\ Hz, 2H), 7.77\ (d, \ J = 8.1\ Hz, 2H), 7.70\ (d, \ J = 7.6\ Hz, 2H), 7.47\ (t, \ J = 7.4\ Hz, 2H), 7.43 - 7.36\ (m, 1H).\]

\[^{13}\text{C}\ \text{NMR}\ (75\ \text{MHz, DMSO})\ \delta 167.24, 144.37, 139.09, 130.40, 127.04, 126.99, 126.69, 126.83.\]

\[\text{1-Naphthoic acid (2ad)}\]

Prepared as described in general procedure D: 1-naphthaldehyde 1ad (0.5 mmol) in 0.5 mL acetone, sunlight, open air, room temperature, 9 h. The residue was purified by silica gel chromatography with DCM/MeOH 15:1 (v/v) as eluting solvent to afford 1-naphthoic acid 2ad as colorless solid (47.8 mg, 56%).

\[^{1}\text{H}\ \text{NMR}\ (300\ \text{MHz, DMSO})\ \delta 8.89\ (d, \ J = 8.4\ Hz, 1H), 8.15\ (t, \ J = 8.6\ Hz, 2H), 7.99\ (d, \ J = 7.9\ Hz, 1H), 7.47\ (t, \ J = 7.4\ Hz, 2H), 7.43 - 7.36\ (m, 1H).\]

\[^{13}\text{C}\ \text{NMR}\ (75\ \text{MHz, DMSO})\ \delta 168.74, 144.32, 144.02, 132.57, 131.57, 130.47, 129.92, 128.62, 127.83, 127.50, 125.98, 125.76, 125.67, 124.19.\]

\[\text{Nicotinic acid (2ae)}\]

Prepared as described in general procedure A: nicotinaldehyde 1ae (0.5 mmol) in 0.5 mL acetone, sunlight, open air, room temperature, 9 h. After completion of the reaction, the mixture was concentrated in vacuo, added 5 mL DCM, sonicated, filtered to obtain nicotinic acid 2ae as light yellow solid (58.7 mg, 95%).
**Note:** More reaction information: weather (partly cloudy); outdoor temperature (22-32 °C); humidity (38-61%); barometer (995 mbar); time (from 09:30 AM to 18:30 PM); date (16.06.2021); place (Heidelberg, Germany).

1H NMR (300 MHz, DMSO) δ 12.31 (br, 1H), 9.07 (s, 1H), 8.76 (s, 1H), 8.25 (d, J = 5.9 Hz, 1H), 7.51 (s, 1H).

13C NMR (75 MHz, DMSO) δ 166.35, 153.27, 150.29, 137.04, 126.68, 123.83.

6-Methoxynicotinic acid (2af)

Prepared as described in general procedure C: 6-methoxynicotinaldehyde 1af (0.5 mmol) in 0.5 mL acetone, sunlight, open air, room temperature, 11 h. The residue was purified by silica gel chromatography with DCM/MeOH 10:1 (v/v) as eluting solvent to afford 6-methoxynicotinic acid 2af as colorless solid (47.8 mg, 56%).

**Note:** More reaction information: weather (sunny); outdoor temperature (16-24 °C); humidity (42-73%); barometer (1009 mbar); time (from 08:30 AM to 19:30 PM); date (13.06.2021); place (Heidelberg, Germany).

1H NMR (300 MHz, DMSO) δ 13.00 (br, 1H), 8.71 (s, 1H), 8.11 (dd, J = 8.6, 2.3 Hz, 1H), 6.94 – 6.72 (m, 1H), 3.90 (s, 3H).

13C NMR (75 MHz, DMSO) δ 166.22, 149.56, 139.85, 120.43, 110.52, 53.83.

5-Methylthiophene-2-carboxylic acid (2ag)

Prepared as described in general procedure C: 5-methylthiophene-2-carbaldehyde 1ag (0.5 mmol) in 0.5 mL acetone, sunlight, open air, room temperature, 10 h. The residue was purified by silica gel chromatography with DCM/MeOH 10:1 (v/v) as eluting solvent to afford 5-methylthiophene-2-carboxylic acid 2ag as dark solid (16.4 mg, 23%).

**Note:** More reaction information: weather (sunny); outdoor temperature (18-24 °C); humidity (42-66%); barometer (1009 mbar); time (from 09:30 AM to 19:30 PM); date (13.06.2021); place (Heidelberg, Germany).

1H NMR (300 MHz, CDCl3) δ 10.44 (br, 1H), 7.70 (d, J = 3.7 Hz, 1H), 6.80 (dd, J = 3.7, 0.9 Hz, 1H), 2.54 (s, 3H).

13C NMR (75 MHz, CDCl3) δ 167.72, 149.85, 135.50, 130.15, 126.73, 15.85.

Benzo[b]thiophene-2-carboxylic acid (2ah)

Prepared as described in general procedure C: benzo[b]thiophene-2-carbaldehyde 1ah (0.5 mmol) in 0.5 mL acetone, sunlight, open air, room temperature, 7 h. The residue was purified by silica gel chromatography with DCM/MeOH 10:1 (v/v) as eluting solvent to afford benzo[b]thiophene-2-carboxylic acid 2ah as dark solid (21.0 mg, 15%).

**Note:** More reaction information: weather (mostly cloudy); outdoor temperature (20-27 °C); humidity (48-80%); barometer (1002 mbar); time (from 08:30 AM to 15:30 PM); date (12.06.2021); place (Heidelberg, Germany).

1H NMR (300 MHz, DMSO) δ 13.28 (br, 1H), 8.11 (s, 1H), 8.08 – 7.97 (m, 2H), 7.56 – 7.40 (m, 2H).

13C NMR (75 MHz, DMSO) δ 163.57, 141.32, 138.75, 134.93, 130.17, 126.99, 125.73, 125.05, 122.97.

1-Methyl-1H-pyrazole-4-carboxylic acid (2ai)

Prepared as described in general procedure C: 1-methyl-1H-pyrazole-4-carbaldehyde 1ai (0.5 mmol) in 0.5 mL acetone, sunlight, open air, room temperature, 9 h. The residue was purified by silica gel chromatography with DCM/MeOH 20:1 (v/v) as eluting solvent to afford 1-methyl-1H-pyrazole-4-carboxylic acid 2ai as dark solid (26.0 mg, 41%).

**Note:** More reaction information: weather (sunny); outdoor temperature (16-24 °C); humidity (42-73%); barometer (1009 mbar); time (from 08:30 AM to 17:30 PM); date (13.06.2021); place (Heidelberg, Germany).
**5-Methyl-2-phenylhex-2-enoic acid (2aj)**

Prepared as described in general procedure D: 5-methyl-2-phenylhex-2-enal 1aj (0.5 mmol) in 0.5 mL acetone, sunlight, open air, room temperature, 9 h. The residue was purified by silica gel chromatography with DCM/MeOH 15:1 (v/v) as eluting solvent to afford 5-methyl-2-phenylhex-2-enoic acid 2aj as colorless solid (51.1 mg, 54%).

**Note:** More reaction information: weather (sunny); outdoor temperature (16-24 °C); humidity (42-73%); barometer (1009 mbar); time (from 08:30 AM to 17:30 PM); date (13.06.2021); place (Heidelberg, Germany).

**1H NMR (300 MHz, CDCl_3) δ 7.42 – 7.31 (m, 3H), 7.25 (d, J = 10.2 Hz, 1H), 7.19 (dd, J = 7.7, 6.0 Hz, 2H), 2.02 (t, J = 7.2 Hz, 2H), 1.86 – 1.65 (m, 1H), 0.88 (d, J = 6.6 Hz, 6H).**

**13C NMR (75 MHz, CDCl_3) δ 171.96, 147.04, 134.85, 133.60, 129.77, 128.00, 127.53, 38.56, 28.34, 22.42.**

**HRMS (El) calcd for C_{13}H_{16}O_2 [M]⁺: 204.11448, found: 204.11356.**

**IR (Reflection): v = 3072, 3059, 2958, 1679, 1479, 1433, 1290, 1180, 1102, 1072, 1026, 999, 916, 781, 748, 713, 691, 617.**

**M.p. (amorphous) 89.8-93.7 °C**

### 3-Phenylbutanoic acid (2ak)

Prepared as described in general procedure B: 3-phenylbutanal 1ak (0.5 mmol) in 0.5 mL acetone, sunlight, open air, room temperature, 7 h. To give 3-phenylbutanoic acid 2ak as colorless solid (79.2 mg, 97%).

**Note:** More reaction information: weather (sunny); outdoor temperature (3-8 °C); humidity (41-58%); barometer (1002 mbar); time (from 10:30 AM to 17:30 PM); date (05.03.2022); place (Heidelberg, Germany).

**1H NMR (300 MHz, CDCl_3) δ 10.99 (br, 1H), 7.42 – 7.34 (m, 2H), 7.33 – 7.25 (m, 3H), 3.48 – 3.15 (m, 1H), 2.70 (qd, J = 15.5, 7.5 Hz, 2H), 1.40 (d, J = 7.0 Hz, 3H).**

**13C NMR (75 MHz, CDCl_3) δ 178.88, 145.37, 128.52, 126.65, 126.46, 42.56, 36.08, 21.79.**

### Pentanoic acid (2al)

Prepared as described in general procedure A: pentanal 1al (0.5 mmol) in 0.5 mL acetone, sunlight, open air, room temperature, 9 h. To give pentanoic acid 2al as colorless solid (49.6 mg, 97%).

**Note:** More reaction information: weather (partly sunny); outdoor temperature (27-33 °C); humidity (44-60%); barometer (999 mbar); time (from 10:30AM to 19:30 PM); date (19.06.2021); place (Heidelberg, Germany).

**1H NMR (300 MHz, CDCl_3) δ 9.85 (br, 1H), 2.35 (t, J = 7.5 Hz, 2H), 1.69 – 1.55 (m, 2H), 1.37 (dq, J = 14.4, 7.3 Hz, 2H), 0.92 (d, J = 7.3 Hz, 3H).**

**13C NMR (75 MHz, CDCl_3) δ 180.36, 33.78, 26.70, 22.15, 13.64.**

### Octanoic acid (2am)

Prepared as described in general procedure A: octanal 1am (0.5 mmol) in 0.5 mL acetone, sunlight, open air, room temperature, 10 h. To give octanoic acid 2am as colorless solid (70.3 mg, 98%).

**Note:** More reaction information: weather (sunny); outdoor temperature (3-8 °C); humidity (41-58%); barometer (1002 mbar); time (from 10:30 AM to 17:30 PM); date (05.03.2022); place (Heidelberg, Germany).

**1H NMR (300 MHz, CDCl_3) δ 11.00 (br, 1H), 2.34 (t, J = 7.5 Hz, 2H), 1.70 – 1.53 (m, 2H), 1.39 – 1.21 (m, 8H), 0.88 (dd, J = 9.2, 4.3 Hz, 3H).**
13C NMR (75 MHz, CDCl₃) δ 180.56, 34.11, 31.61, 28.99, 28.87, 24.65, 22.56, 13.99.

Prepared as described in general procedure A: cyclohexanecarbaldehyde 1an (0.5 mmol) in 0.5 mL acetone, sunlight, open air, room temperature, 9 h. To give cyclohexanecarboxylic acid 2an as colorless solid (57.1 mg, 89%).

**Note:** More reaction information: weather (partly cloudy); outdoor temperature (22-32 °C); humidity (38-61%); barometer (995 mbar); time (from 09:30 AM to 18:30 PM); date (16.06.2021); place (Heidelberg, Germany).

1H NMR (300 MHz, CDCl₃) δ 10.77 (br, 1H), 2.32 (tt, J = 11.1, 3.6 Hz, 1H), 1.99 – 1.85 (m, 2H), 1.75 (dd, J = 9.4, 3.3 Hz, 2H), 1.64 (dd, J = 7.5, 3.1 Hz, 1H), 1.53 – 1.37 (m, 2H), 1.36 – 1.17 (m, 3H).

13C NMR (75 MHz, CDCl₃) δ 182.76, 42.91, 28.72, 25.65, 25.29.

2-Methylpentanoic acid (2ao)

Prepared as described in general procedure A: 2-methylpentanal 1ao (0.5 mmol) in 0.5 mL acetone, sunlight, open air, room temperature, 4 h. To give 2-methylpentanoic acid 2ao as colorless solid (57.1 mg, 89%).

**Note:** More reaction information: weather (sunny); outdoor temperature (3-8 °C); humidity (42-58%); barometer (1002 mbar); time (from 10:30 AM to 14:30 PM); date (05.03.2022); place (Heidelberg, Germany).

1H NMR (300 MHz, CDCl₃) δ 10.59 (br, 1H), 2.58 – 2.38 (m, 1H), 1.75 – 1.61 (m, 1H), 1.46 – 1.26 (m, 3H), 1.16 (t, J = 6.9 Hz, 3H), 0.91 (t, J = 7.1 Hz, 3H).

13C NMR (75 MHz, CDCl₃) δ 183.60, 39.14, 35.64, 20.29, 16.75, 13.88.

1-(tert-Butoxycarbonyl)piperidine-4-carboxylic acid (2ap)

Prepared as described in general procedure C: tert-buty 4-formylpiperidine-1-carboxylate 1ap (0.5 mmol) in 0.5 mL acetone, sunlight, open air, room temperature. The reaction was monitored by thin layer chromatography (TLC). The reaction was not completed on the first day (10 h), continued to react in the sun on the next day (10 h). After completion of the reaction, concentrated in vacuo, the residue was purified by silica gel chromatography with DCM/MeOH 15:1 (v/v) as eluting solvent to afford 1-(tert-butoxycarbonyl)piperidine-4-carboxylic acid 2ap as colorless solid (88.4 mg, 77%).

**Note:** More reaction information: weather (sunny); outdoor temperature (19-28 °C); humidity (56-79%); barometer (1002 mbar); time (from 09:00 AM to 19:00 PM); date (18.07.2021); place (Heidelberg, Germany).

1H NMR (300 MHz, CDCl₃) δ 10.41 (br, 1H), 3.95 (d, J = 12.0 Hz, 2H), 2.80 (t, J = 11.7 Hz, 2H), 2.42 (dd, J = 12.6, 9.0 Hz, 1H), 1.84 (d, J = 11.5 Hz, 2H), 1.68 – 1.50 (m, 2H), 1.39 (s, 9H).

13C NMR (75 MHz, CDCl₃) δ 179.36, 154.72, 79.76, 40.63, 28.26, 27.60.

2-Acetoxybenzoic acid (2au)

Prepared as described in general procedure B: 2-formylphenyl acetate 1av (0.5 mmol) in 0.5 mL acetone, sunlight, open air, room temperature, 8 h. To give 2-acetoxybenzoic acid 2av as colorless solid (87.7 mg, 97%).

**Note:** More reaction information: weather (partly sunny); outdoor temperature (18-24 °C); humidity (40-68%).
barometer (995 mbar); time (from 09:00 AM to 17:00 PM); date (29.07.2021); place (Heidelberg, Germany).

$^1$H NMR (300 MHz, CDCl$_3$) δ 11.03 (br, 1H), 8.12 (d, $J = 7.3$ Hz, 1H), 7.62 (t, $J = 7.1$ Hz, 1H), 7.35 (t, $J = 7.2$ Hz, 1H), 7.14 (d, $J = 7.9$ Hz, 1H), 2.34 (s, 3H).

$^{13}$C NMR (75 MHz, CDCl$_3$) δ 170.16, 169.81, 151.19, 134.85, 132.47, 126.13, 123.95, 122.22, 20.95.

Notes and References

21. $^1$H NMR and $^{13}$C NMR data were consistent with that for material purchased from Sigma-Aldrich.

NMR Spectra

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