Supporting information

for

Elongated and substituted triazine-based tricarboxylic acid linkers for MOFs

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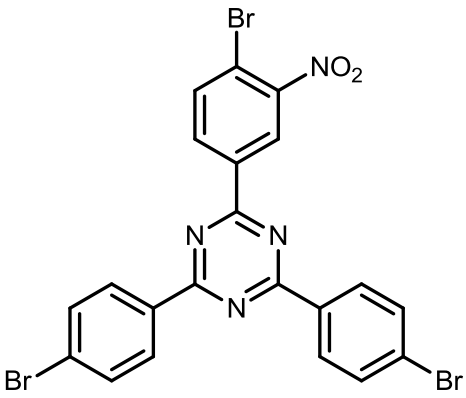
Experimental details

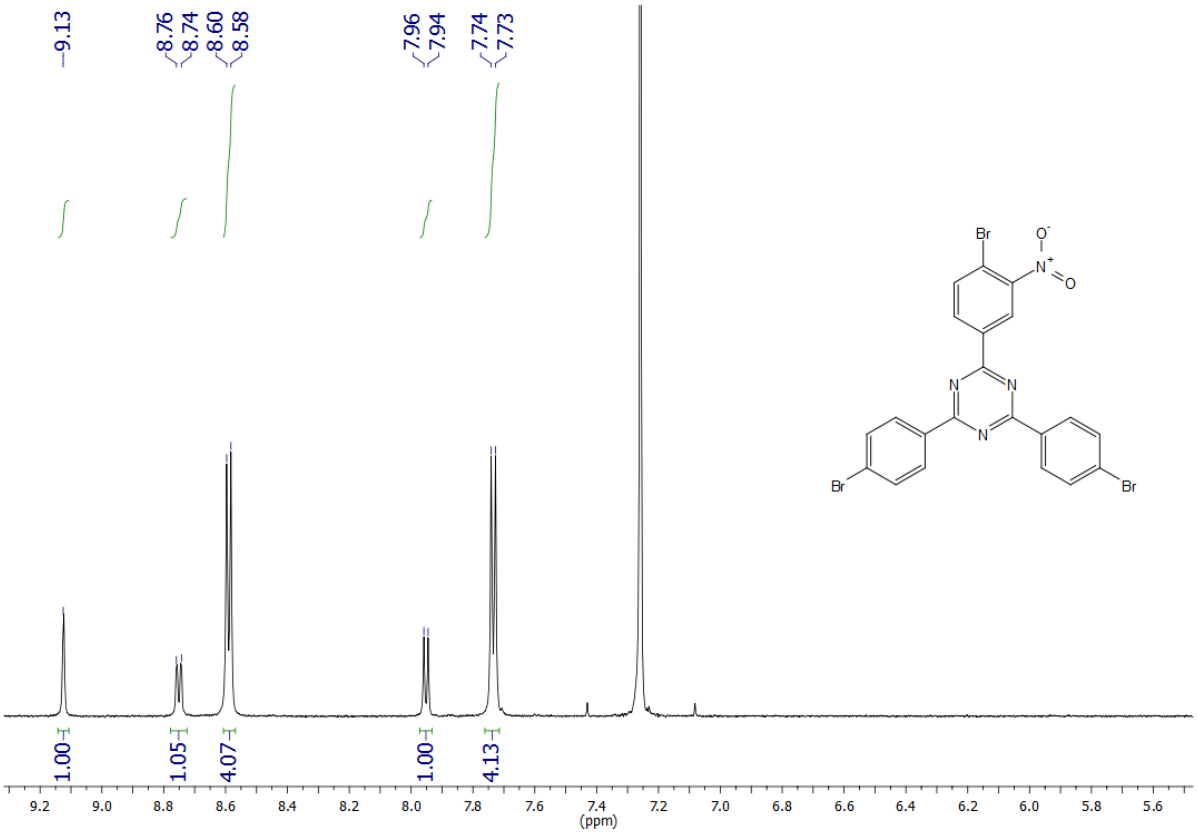
General remarks: Antimony(V) chloride (99%, Alfa Aesar), [1,1′-bis(diphenylphosphino)ferrocene]dichloro palladium(II) (99.9%, ABCR), 4-bromobenzoic acid (98 %, Sigma-Aldrich), 4-bromobenzonitrile (99%, Sigma-Aldrich), 3-hydroxybenzoic acid (97%, Sigma-Aldrich), 4-(methoxycarbonyl)phenylboronic acid (97%, ABCR), palladium on charcoal (10%, Alfa Aesar) and tetrakis(triphenylphosphine)palladium(0) (99%, ABCR) were purchased and used without further purification. Methyl 4'-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-1,1'-biphenyl-4-carboxylate (**18**) was synthesized as described.[1] Dry solvents were obtained with suitable desiccants. Other solvents were distilled before use. Column chromatography was carried out with silica gel (Macherey-Nagel, particle size 0.04–

0.063 mm). Melting points were measured with a Gallenkamp MPD350.BM2.5 instrument. NMR spectra were recorded with a Bruker AC 200, DRX 500 or Avance 600 instrument at 300 K. Assignments are supported by COSY, HSQC, and HMBC. In some cases, not all ^{13}C signals could be found in the 1D ^{13}C NMR spectra. In these cases, the listed chemical shifts have been extracted from the 2D spectra. Even when obtained by DEPT, the type of ^{13}C signal is always listed as singlet, doublet, etc. All chemical shifts are referenced to tetramethylsilane or the residual proton or carbon of the solvent. The NMR signals of atoms in the different arms of the triazines are assigned by using for instance Ar2 for atoms which are part of an benzene ring in the substituent at C-2 of the triazine. Aromatic rings within a biphenyl or terphenyl carry ’ or ’’ according to the biphenyl or terphenyl nomenclature. EI/CI mass spectra were recorded with a Finnigan MAT 8200, MAT 8230 or JEOL AccuTOF GCV 4G the latter one being used for HRMS as well. ESI mass spectra were recorded with an Applied Biosystems Mariner 5280. MALDI-TOF mass spectra were recorded with a Bruker-Daltronics Biflex III with Cl-CCA (4-chloro-α-cyano-cinnamic acid) as matrix. IR spectra were recorded with a Perkin-Elmer Spectrum 100 spectrometer equipped with a Golden Gate Diamond ATR unit A-531-G. Elemental analyses were carried out with a Euro EA 3000 Elemental Analyzer from Euro Vector.

2-(4-Bromo-3-nitrophenyl)-4,6-bis(4-bromophenyl)-1,3,5-triazine (3b)



Under nitrogen and at 0 °C, antimony(V) chloride (5.23 mL, 40.8 mmol) was added to a solution of crude 4-bromo-3-nitrobenzoyl chloride (**5b**, 8.86 g, max. 33.5 mmol) and *p*-bromobenzonitrile (**6**, 12.5 g, 68.7 mmol) in dry chloroform (120 mL). The mixture was stirred for 1 h at room temp. and for 16 h at reflux. A yellow solid formed which was filtered off and washed with chloroform. At 0 °C, aq. ammonia (390 mL, 28%) was added in portions to the residue and the suspension was stirred for 2 h at room temp. The solid changed its color from yellow to white. After filtration, the product was separated from insoluble side products by a Soxhlet extraction (chloroform, 48 h). Yield: 10.3 g (17.6 mmol, 53%). M. p.: >300 °C. ^{1}H NMR (500 MHz, $CDCl_3$, 323 K): δ = 9.11 (d, 1H, ^{4}J = 1.8 Hz, 2-Ar-*H*-2), 8.74 (dd, 1H, ^{3}J = 8.4 Hz, ^{4}J = 1.8 Hz, 2-Ar-*H*-6), 8.58 (d, 4H, ^{3}J = 8.5 Hz, 4,6-Ar-*H*-2,6), 7.98 (d, 1H, ^{3}J = 8.4 Hz, 2-Ar-*H*-5), 7.72 (d, 4H, ^{3}J = 8.5 Hz, 4,6-Ar-*H*-3,5) ppm. ^{13}C NMR (125 MHz, $CDCl_3$, 323 K): δ = 171.7 (s, triazine-*C*-4,6), 169.4 (s, triazine-*C*-2), 150.5 (s, 2-Ar-*C*-3), 136.7 (s, 2-Ar-*C*-4), 135.3 (d, 2-Ar-*C*-5), 134.4 (s, 4,6-Ar-*C*-4), 132.6 (d, 2-Ar-*C*-6), 132.3 (d, 4,6-Ar-*C*-3,5), 130.6 (d, 4,6-Ar-*C*-2,6), 128.4 (s, 4,6-Ar-*C*-1), 125.6 (d, 2-Ar-*C*-2), 118.8 (s, 2-Ar-*C*-1) ppm. MS (EI, 70 eV): *m/z* = 588/590/592/594 (34/100/90/33) $[M]^{+\cdot}$. MS (CI, isobutane): *m/z* = 589/591/593/595 (34/100/87/43) $[M + H]^{+}$. IR (ATR): $\tilde{\nu}$ = 3082 (aryl-H), 1592, 1509, 1486 (arom. C=C, arom. C=N), 1578 (NO_2), 1350 (C-N-val.), 806 (1,4-disubst. aryl, 1,2,4-trisubst. aryl) cm^{-1}. HRMS (EI): *m/z* = calcd. 589.8411; found 589.8391 (Δ 3.43 ppm). Elemental analysis ($C_{21}H_{11}Br_3N_4O_2$) (591.05): calcd. C 42.67 H 1.88 N 9.48; found C 42.67 H 1.77 N 9.07.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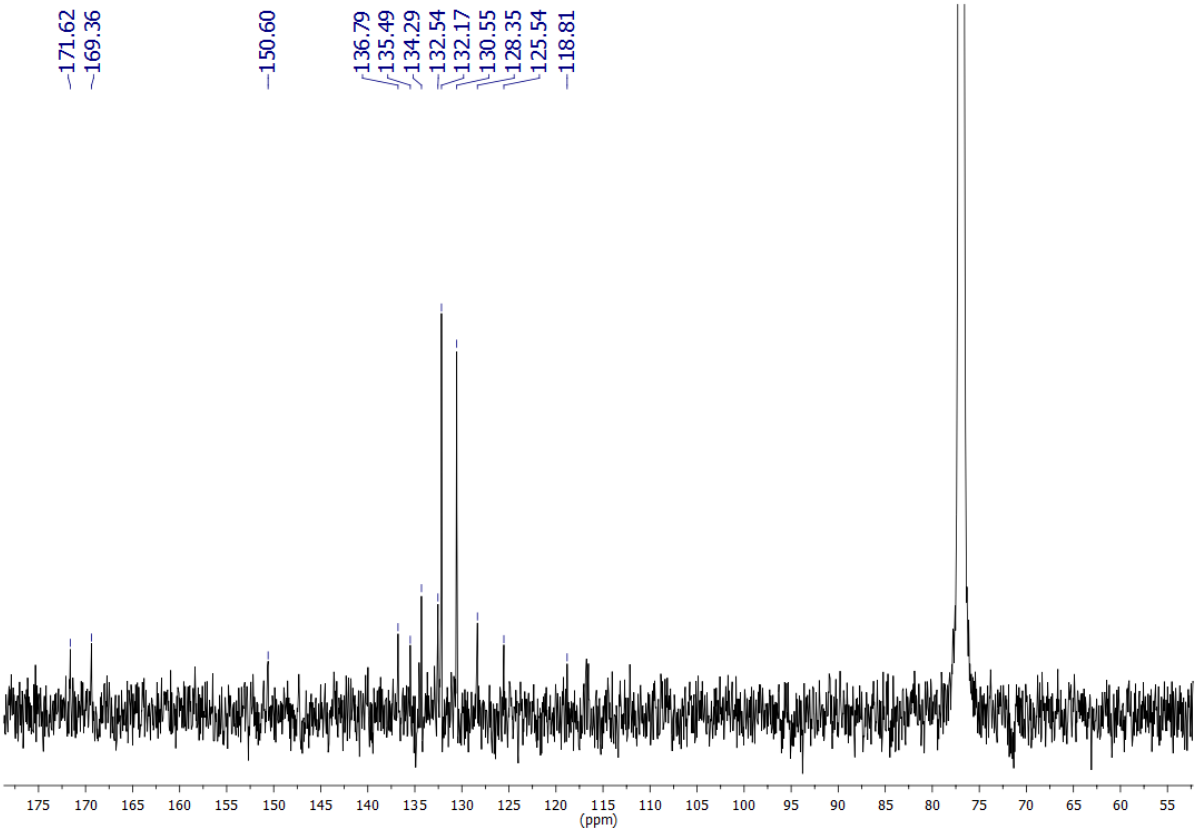
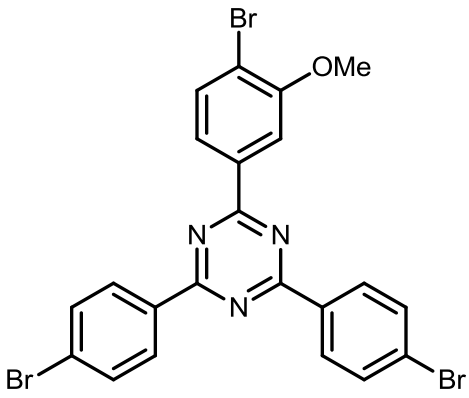
8.76
8.74
8.60
8.58
7.96
7.94
7.74
7.73
1.00
1.05
4.07
1.00
4.13
(ppm)
Br
O
N
O
N
N
N
Br
Br

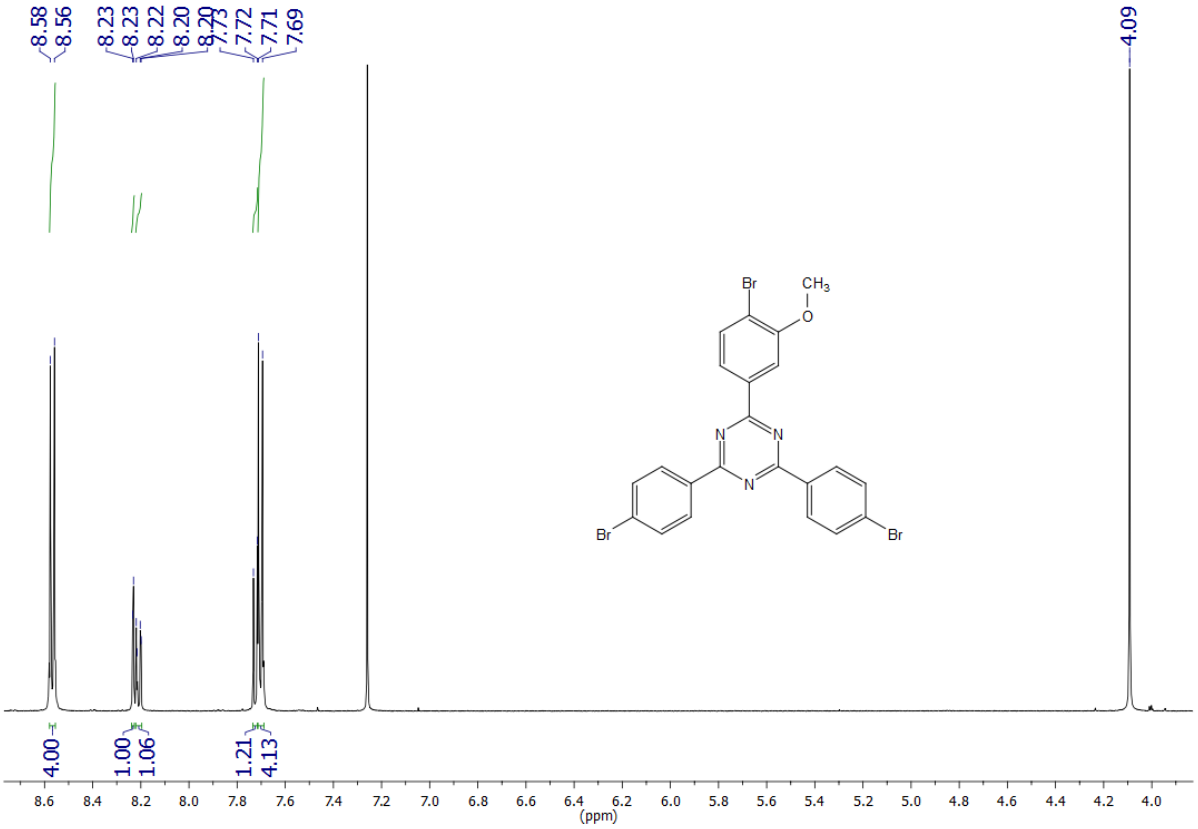

171.62
169.36
150.60
136.79
135.49
134.29
132.54
132.17
130.55
128.35
125.54
118.81
(ppm)

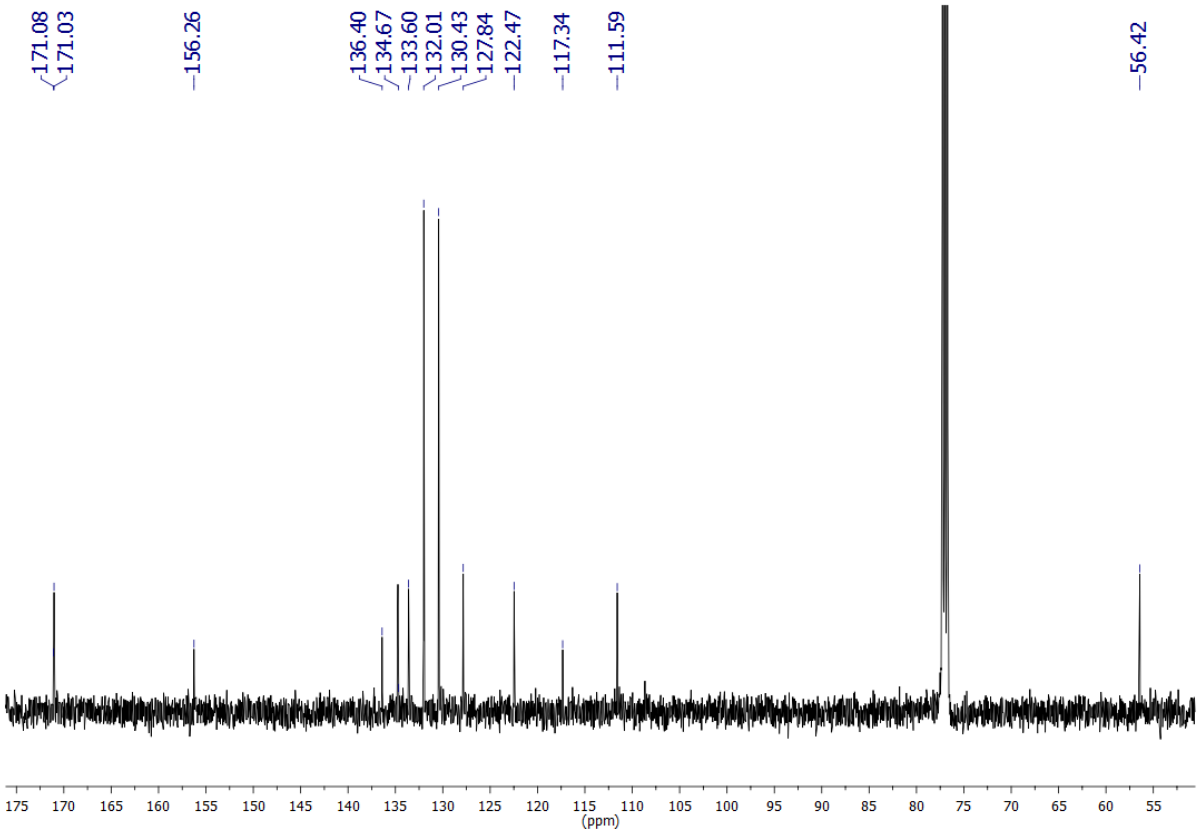
2-(4-Bromo-3-methoxyphenyl)-4,6-bis(4-bromophenyl)-1,3,5-triazine (3c)



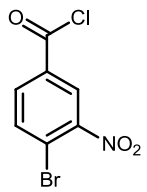
Under nitrogen, 4-bromo-3-methoxy-benzoyl chloride [**5c**, crude, from 1.00 g (4.01 mmol) of **14**] and *p*-bromobenzonitrile (**6**, 1.46 g, 8.02 mmol) were dissolved in dry chloroform (25 mL). At 0 °C, antimony(V) chloride (616 µL, 4.81 mmol) was added and the mixture was stirred for 1 h at room temp. followed by 16 h of reflux. The resulting red oxonium salt was filtered off and washed with chloroform. At 0 °C, aq. ammonia (50 mL, 28%) was added to the residue and the suspension was stirred for 2 h at room temp. The solid changed its color from yellow to white. After filtration, the product was filtered off and was extracted for 24 h with chloroform (Soxhlet). After evaporation of the solvent, the residue was recrystallized from a boiling mixture of chloroform and petrol ether (30–60 °C) yielding a colourless solid. Yield: 1.41 g (2.45 mmol, 61%). M. p.: 203 °C. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.57 (m_c (d), 4H, ^{3}J = 8.7 Hz, 4,6-Ar-*H*-2,6), 8.23 (d, 1H, ^{4}J = 1.8 Hz, 2-Ar-*H*-2), 8.21 (dd, 1H, ^{3}J = 8.2 Hz, ^{4}J = 1.8 Hz, 2-Ar-*H*-6), 7.72 (d, 1H, ^{3}J = 8.2 Hz, 2-Ar-*H*-5), 7.70 (m_c (d), 4H, ^{3}J = 8.7 Hz, 4,6-Ar-*H*-3,5), 4.09 (s, 3H, O*C*H_3) ppm. ^{13}C NMR (125 MHz, $CDCl_3$): δ = 171.1 (s, triazine-*C*-4,6), 171.0 (s, triazine-*C*-2), 156.3 (s, 2-Ar-*C*-3), 136.4 (s, 2-Ar-*C*-1), 134.7 (s, 4,6-Ar-*C*-1), 133.6 (d, 2-Ar-*C*-5), 132.0 (d, 4,6-Ar-*C*-3,5), 130.4 (d, 4,6-Ar-*C*-2,6), 127.8 (s, 4,6-Ar-*C*-4), 122.5 (d, 2-Ar-*C*-6), 117.3 (s, 2-Ar-*C*-4), 111.6 (d, 2-Ar-*C*-2), 56.4 (q, O*C*H_3) ppm. MS (EI, 70 eV): *m/z* = 572/574/576/578 (34/100/96/34) $[M]^{+\cdot}$, 180 (85) $[M - C_{15}H_{10}Br_2N_2O]^{+}$. IR (ATR): $\tilde{\nu}$ = 3033 (aryl-H), 1598, 1505, 1485 (arom. C=C, arom. C=N), 1414 (C-N-val.), 1251 (aryl-OCH_3), 831 (1,4-disubst. aryl, 1,2,4-trisubst. aryl) cm^{-1}. HRMS (EI): *m/z* = calcd. 578.8625; found 578.8608 (Δ 3.12 ppm).

Elemental analysis ($C_{22}H_{14}Br_3N_3O$) (576.08): calcd. C 45.87 H 2.45 N 7.29; found C 46.09 H 2.46 N 6.94.



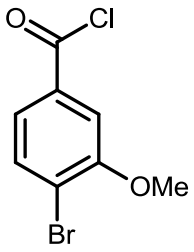

171.08
171.03
156.26
136.40
134.67
133.60
132.01
130.43
127.84
122.47
117.34
111.59
56.42
175 170 165 160 155 150 145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55
(ppm)

4-Bromo-3-nitrobenzoyl chloride (5b)



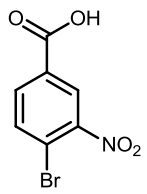
A solution of 4-bromo-3-nitrobenzoic acid (**9**, 4.00 g, 80.0 mmol) in thionyl chloride (24 mL) was heated to reflux for 2 h. Removal of excess thionyl chloride in vacuo yielded a yellow oil which was used without further purification. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.54 (d, 1H, ^{4}J = 2.2 Hz, Ar-*H*-2), 8.13 (dd, 1H, ^{3}J = 8.5 Hz, ^{4}J = 2.2 Hz, Ar-*H*-6), 7.94 (d, 1H, ^{3}J = 8.5 Hz, Ar-*H*-5) ppm. ^{13}C NMR (125 MHz, $CDCl_3$): δ = 166.0 (s, *C*OCl), 150.2 (s, Ar-*C*-3), 136.2 (d, Ar-*C*-6), 134.2 (d, Ar-*C*-5), 133.6 (s, Ar-*C*-1), 127.7 (d, Ar-*C*-2), 122.6 (s, Ar-*C*-4) ppm. MS (EI, 70 eV): *m/z* = 264/262 (14/11) $[M]^{+\cdot}$, 229/227 (96/100) $[M - Cl]^{+}$. MS (CI, isobutane): *m/z* = 265/263 (100/94) $[M + H]^{+}$, 229/227 (45/40) $[M - Cl]^{+}$.

4-Bromo-3-methoxy-benzoyl chloride (5c)



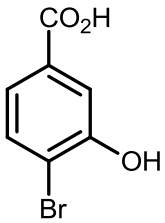
N,*N*-Dimethylformamide (10 µL) was added to a mixture of 4-bromo-3-methoxy-benzoic acid (**14**, 1.00 g, 4.01 mmol) and thionyl chloride (5 mL). After heating to reflux for 2 h, excess thionyl chloride was distilled off and the crude colourless solid was used without further purification. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.70 (d, 1H, ^{3}J = 8.3 Hz, Ar-*H*-5), 7.63 (dd, 1H, ^{3}J = 8.3 Hz, ^{4}J = 2.0 Hz, Ar-*H*-6), 7.54 (d, 1H, ^{4}J = 2.0 Hz, Ar-*H*-2), 3.97 (s, 3H, O*C*H_3) ppm. ^{13}C NMR (125 MHz, $CDCl_3$): δ = 167.7 (s, *C*OCl), 156.3 (s, Ar-*C*-3), 133.8 (d, Ar-*C*-5), 133.5 (s, Ar-*C*-1), 125.0 (d, Ar-*C*-6), 120.9 (s, Ar-*C*-4), 113.0 (d, Ar-*C*-2), 56.5 (q, O*C*H_3) ppm.

4-Bromo-3-nitrobenzoic acid (9)



p-Brombenzoic acid (**8**, 5.00 g, 24.9 mmol) was slowly added in portions to a cold mixture (0 °C) of conc. nitric acid (6.2 mL, 0.15 mmol) and conc. sulfuric acid (15.2 mL, 300 mmol). The addition was performed in such a way that the temperature did not exceed 5 °C. After stirring for 3 h at 0 °C and 2 h at room temp., the mixture was poured on ice-containing water. The resulting colourless solid was filtered off and washed extensively with water. Yield: 6.00 g (24.4 mmol, 98%) (ref. [2]: 96%), m. p. 202 °C. ^{1}H NMR (200 MHz, DMSO-d_6): δ = 8.46 – 8.42 (m, 1H, Ar-*H*-2), 8.09–8.05 (m, 2H, Ar-*H*-5,6) ppm. ^{13}C NMR (50 MHz, DMSO-d_6): δ = 165.0 (s, *C*O_2H), 149.7 (s, Ar-*C*-3), 135.4 (d, Ar-*C*-6), 132.4 (d, Ar-*C*-5), 131.7 (s, Ar-*C*-1), 125.9 (d, Ar-*C*-2), 118.0 (Ar-*C*-4) ppm. MS (EI, 70 eV): *m/z* = 245/247 (95/100) $[M]^{+\cdot}$. MS (CI, isobutane): *m/z* = 246/248 (100/99) $[M + H]^+$. IR (ATR): $\tilde{\nu}$ = 3082 (aryl-H), 2820 (br., OH), 1684 (C=O), 1595 (arom. C=C), 1530, 1303 (NO_2), 1034 (aryl-Br), 907, 809 (1,2,4-trisubst. aryl) cm^{-1}.

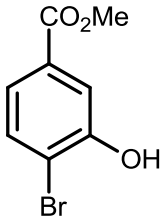
4-Bromo-3-hydroxybenzoic acid (11)



To a solution of 3-hydroxybenzoic acid (**10**, 15.0 g, 109 mmol) in a mixture of ethanol (60 mL) and acetic acid (30 mL), bromine (11.2 mL, 218 mmol) was added slowly and dropwise. After stirring for 30 min at room temp., aq. sodium thiosulfate solution (90 mL) was added and ethanol was removed in vacuo. The remaining aq. layer was extracted with ethyl acetate (5 x 200 mL) and the combined organic layer was dried with magnesium sulfate. After removal of the solvent, the crude product was recrystallized from boiling water and a colourless solid was obtained. Yield: 12.19 g (56.2 mmol, 52%) (ref.[3]:

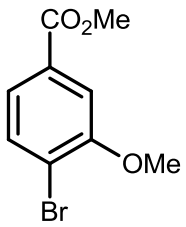
40%). M. p.: 225 – 227 °C. ^{1}H NMR (500 MHz, DMSO-d_6): δ = 13.04 (br. s, 1H, CO_2H), 10.62 (s, 1H, O*H*), 7.59 (d, 1H, ^{3}J = 8.3 Hz, Ar-*H*-5), 7.51 (d, 1H, ^{4}J = 2.0 Hz, Ar-*H*-2), 7.28 (dd, 1H, ^{3}J = 8.3 Hz, ^{4}J = 2.0 Hz, Ar-*H*-6) ppm. ^{13}C NMR (125 MHz, DMSO-d_6): δ = 167.2 (s, CO_2H), 154.6 (s, Ar-*C*-3), 133.5 (d, Ar-*C*-5), 131.8 (s, Ar-*C*-1), 121.6 (d, Ar-*C*-6), 117.1 (d, Ar-*C*-2), 115.0 (s, Ar-*C*-4) ppm. MS (EI, 70 eV): *m/z* = 216/218 (100/97) $[M]^{+\cdot}$, 199, 201 (73, 70) $[M - OH]^{+}$.

Methyl 4-bromo-3-hydroxybenzoate (12)



Conc. sulfuric acid (12 mL) was added to a suspension of 4-bromo-3-hydroxybenzoic acid (**11**, 12.2 g, 56.2 mmol) in methanol (120 mL). After heating to reflux for 16 h, the solution was neutralized with sat. aq. sodium bicarbonate solution and methanol was distilled off in vacuo. The remaining aq. phase was extracted with ethyl acetate (3 x 100 mL) and the combined organic layer was dried with magnesium sulfate. Filtration and removal of the solvent in vacuo yielded a colourless solid. Yield: 12.2 g (52.7 mmol, 94%) (ref.[4]: no yield given). M. p.: 124 °C (ref.[4]: 120 – 122 °C). ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.68 (d, 1H, ^{4}J = 2.0 Hz, Ar-*H*-2), 7.54 (d, 1H, ^{3}J = 8.3 Hz, Ar-*H*-5), 7.48 (dd, 1H, ^{3}J = 8.3 Hz, ^{4}J = 2.0, Ar-*H*-6), 5.75 (br. s, 1H, O*H*), 3.91 (s, 3H, CO_2CH_3) ppm. ^{13}C NMR (125 MHz, $CDCl_3$): δ = 166.2 (s, CO_2CH_3), 152.4 (s, Ar-*C*-3), 132.2 (d, Ar-*C*-5), 131.3 (s, Ar-*C*-1), 122.7 (d, Ar-*C*-6), 117.1 (d, Ar-*C*-2), 115.6 (s, Ar-*C*-4), 52.4 (q, CO_2CH_3) ppm. MS (EI, 70 eV): *m/z* = 230, 232 (63, 60) $[M]^{+\cdot}$.

Methyl 4-bromo-3-methoxy-benzoate (13)

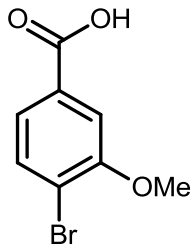


Method A: Potassium carbonate (4.56 g, 33.0 mmol) was added to a solution of methyl 4-bromo-3-hydroxybenzoate (**12**, 6.24 g, 27.0 mmol) in acetone (40 mL). Dimethyl sulfate (3.03 mL, 32.0 mmol) was added and the mixture was heated to reflux for 3 h. At room temp., deionized water was added (5 mL) and acetone was distilled off in vacuo. The remaining aq. phase was extracted with dichloromethane (3 x 100 mL), the combined organic layer was dried with magnesium sulfate, filtered and the solvent was removed in vacuo yielding a colourless solid. Yield: 6.59 g (26.9 mmol, >99%) (ref.[5]: 99%).

Method B: Potassium carbonate (2.54 g, 18.4 mmol) was added to a solution of 4-bromo-3-hydroxybenzoic acid (**11**, 1.00 g, 4.61 mmol) in *N*,*N*-dimethylformamide (10 mL). Methyl iodide (863 µL, 13.8 mmol) was added and the mixture was stirred for 16 h at room temp. Deionized water (50 mL) was added and the aq. phase was extracted with *tert*-butyl methyl ether (3 x 100 mL). The combined organic layer was washed with brine (100 mL) and dried with magnesium sulfate. After filtration, the solvent was evaporated in vacuo yielding a colourless solid. Yield: 938 mg (3.83 mmol, 83%) (ref.[3]: 91%).

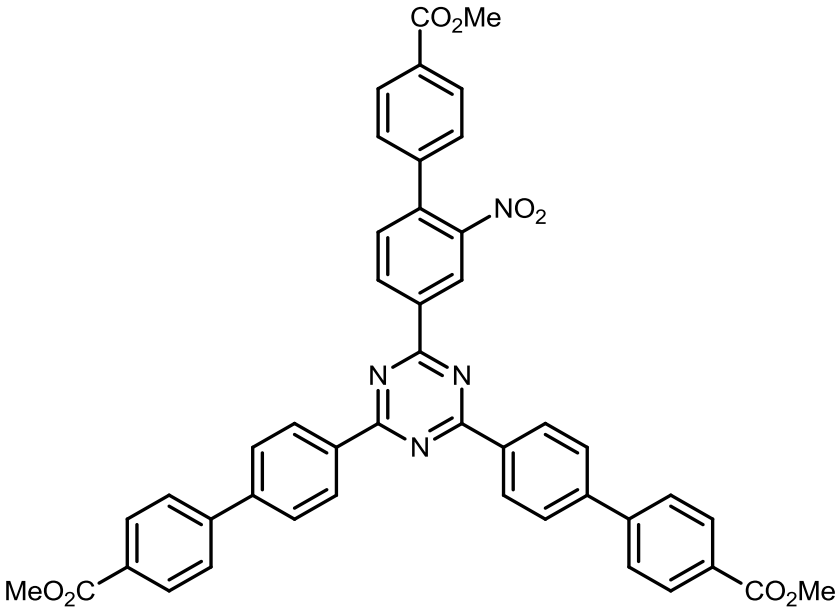
M. p. 53 °C. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.61 (d, 1H, ^{3}J = 8.2 Hz, Ar-*H*-5), 7.55 (d, 1H, ^{4}J = 1.8 Hz, Ar-*H*-2), 7.51 (dd, 1H, ^{3}J = 8.2 Hz, ^{4}J = 1.8 Hz, Ar-*H*-6), 3.95 (s, 3H, OCH_3), 3.92 (s, 3H, CO_2CH_3) ppm. ^{13}C NMR (125 MHz, $CDCl_3$): δ = 166.4 (s, CO_2Me), 155.9 (s, Ar-*C*-3), 133.3 (d, Ar-*C*-5), 130.6 (s, Ar-*C*-1), 122.9 (d, Ar-*C*-6), 117.5 (s, Ar-*C*-4), 112.4 (d, Ar-*C*-2), 56.4 (q, OCH_3), 52.4 (q, CO_2CH_3) ppm. HRMS (EI): *m/z* = calcd. 243.9735; found 243.9744 (Δ 4.01 ppm).

4-Bromo-3-methoxybenzoic acid (14)



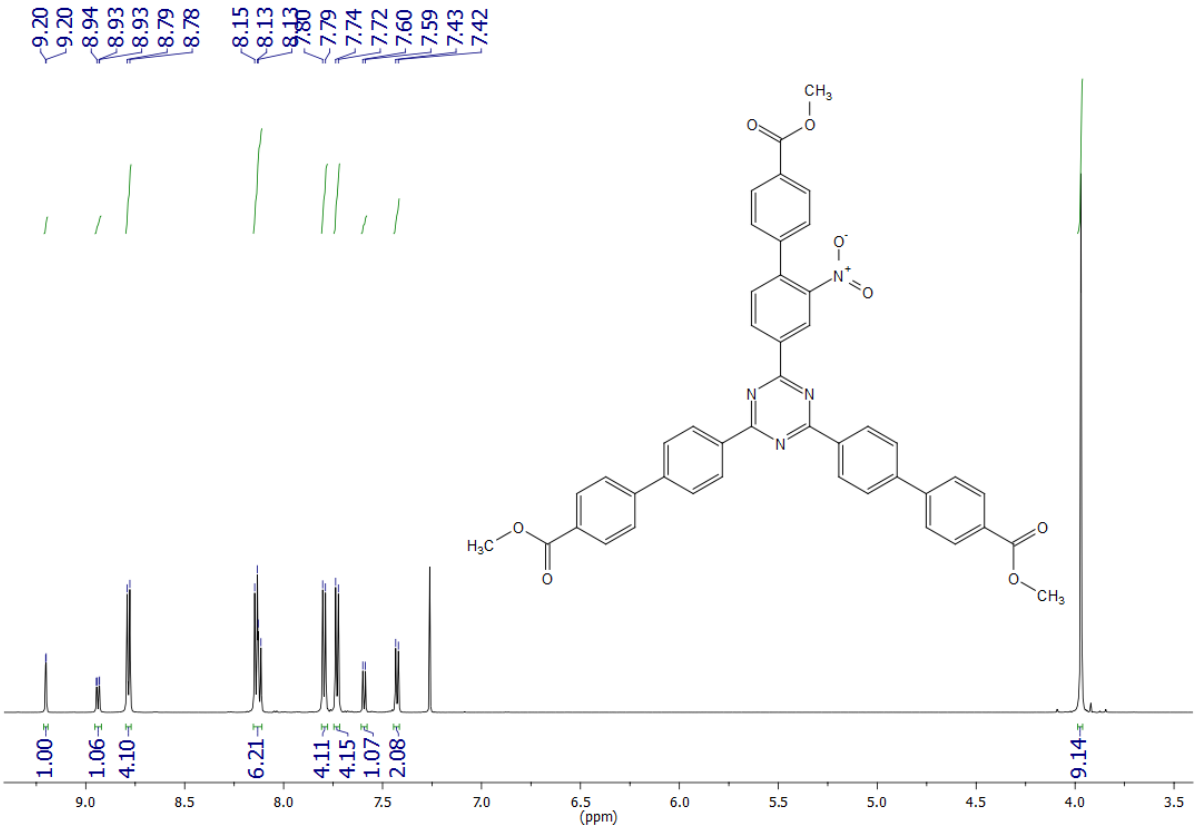
Aq. sodium hydroxide (2 M, 40 mL) was added to a solution of methyl 4-bromo-3-methoxy-benzoate (**13**, 6.52 g, 27.1 mmol) in methanol (200 mL) and the mixture was stirred for 6 h at room temp. Deionized water (100 mL) was added and methanol was distilled off in vacuo. The remaining aq. phase was washed with dichloromethane (3 x 100 mL) and acidified with hydrochloric acid (6 M). The precipitate was filtered off and was washed thoroughly with water yielding a colourless solid. Yield: 4.10 g (17.8 mmol, 66%) (ref[5]: 89%). M. p.: 220 °C. ^{1}H NMR (500 MHz, DMSO-d_6): δ = 13.21 (br. s, 1H, CO_2H), 7.71 (d, 1H, ^{3}J = 8.2 Hz, Ar-*H*-5), 7.54 (d, 1H, ^{4}J = 1.8 Hz, Ar-*H*-2), 7.46 (dd, 1H, ^{3}J = 8.2 Hz, ^{4}J = 1.8 Hz, Ar-*H*-6), 3.91 (s, 3H, OCH_3) ppm. ^{13}C NMR (125 MHz, DMSO-d_6): δ = 167.1 (s, CO_2H), 155.9 (s, Ar-*C*-3), 133.6 (d, Ar-*C*-5), 132.1 (s, Ar-*C*-1), 123.2 (d, Ar-*C*-6), 116.5 (s, Ar-*C*-4), 113.0 (d, Ar-*C*-2), 56.8 (q, OCH_3) ppm. MS (EI, 70 eV): *m/z* = 230/232 (100/98) $[M]^{+\cdot}$.

2,4-Bis[4'-(methoxycarbonyl)-biphenyl-4-yl]-6-[4'-(methoxycarbonyl)-2-nitro-biphenyl-4-yl)-1,3,5-triazine (16b)



Under nitrogen, 2-(4-bromo-3-nitrophenyl)-4,6-bis(4-bromophenyl)-1,3,5-triazine (**3b**, 3.00 g, 5.08 mmol), 4-methoxycarbonyl-phenylboronic acid (**15**, 4.10 g, 22.8 mmol), potassium phosphate (7.00 g, 33.0 mmol) and tetrakis(triphenylphosphine) palladium(0) (300 mg, 260 µmol) in a mixture of 1,4-dioxane (300 mL) and deionized water (20 mL) were heated to reflux for 48 h. Conversion was checked by TLC (silica gel, cyclohexane/ethyl acetate, 1:1, R_f = 0.69). After evaporation of the 1,4-dioxane in vacuo, chloroform (150 mL) was added and the organic layer was washed with deionized water (2 x 100 mL) and brine (50 mL). The organic layer was dried with magnesium sulfate. After filtration and evaporation of the solvent in vacuo, the crude product was dissolved in chloroform, activated charcoal was added, and after filtration, the product was recrystallized from chloroform yielding a colourless solid. Yield: 3.46 g (4.57 mmol, 90%). M. p.: 174 °C. ^{1}H NMR (500 MHz, $CDCl_3$,): δ = 9.22 (d, 1H, ^4J = 1.7 Hz, 6-Ar-*H*-3), 8.96 (dd, 1H, ^3J = 8.0 Hz, ^4J = 1.7 Hz, 6-Ar-*H*-5), 8.81 (m_c (d), 4H, ^3J = 8.6 Hz, 2,4-Ar-*H*-2,6), 8.15 (m_c (d), 4H, ^3J = 8.6 Hz, 2,4-Ar'-*H*-3,5), 8.13 (m_c (d), 2H, ^3J = 8.6 Hz, 6-Ar'-*H*-3,5), 7.81 (m_c (d), 4H, ^3J = 8.6 Hz, 2,4-Ar-*H*-3,5), 7.74 (m_c (d), 4H, ^3J = 8.6 Hz, 2,4-Ar'-*H*-2,6), 7.61 (d, 1H, ^3J = 8.0 Hz, 6-

Ar-*H*-6), 7.44 (m_c (d), 2H, ^3J = 8.6 Hz, 6-Ar'-*H*-2,6), 3.97 (s, 9H, OCH_3) ppm. ^{13}C NMR (125 MHz, $CDCl_3$): δ = 171.6 (s, triazine-*C*-2,4), 169.3 (s, triazine-*C*-6), 166.8 (s, 2,4-Ar'-*C*O_2Me), 166.5 (s, 6-Ar'-*C*O_2Me), 149.6 (s, 6-Ar-*C*-2), 144.4 (s, 2,4-Ar'-*C*-1), 144.3 (s, 2,4-Ar-*C*-4), 141.4 (s, 6-Ar'-*C*-1), 138.8 (s, 6-Ar-*C*-1), 137.3 (s, 6-Ar-*C*-4), 135.2 (s, 2,4-Ar-*C*-1), 132.2 (d, 6-Ar-*C*-5), 132.1 (d, 6-Ar-*C*-6), 130.4 (s, 6-Ar'-*C*-4), 130.3 (d, 2,4-Ar'-*C*-3,5), 130.1 (d, 2,4-Ar-*C*-3,5), 129.7 (s, 2,4-Ar'-*C*-4), 129.7 (d, 6-Ar'-*C*-2,6), 128.0 (d, 2,4-Ar'-*C*-2,6), 127.6 (6-Ar'-*C*-3,5), 127.2 (d, 2,4-Ar-*C*-2,6), 124.6 (d, 6-Ar-*C*-3), 52.3 (q, 6-Ar'-CO_2CH_3), 52.2 (q, 2,4-Ar'-CO_2CH_3) ppm. MS (MALDI, Cl-CCA): *m/z* = 757 $[M + H]^+$, 767 $[M + Na]^+$, 776 $[M + K]^+$. IR (ATR): $\tilde{\nu}$ = 3076, 3006 (aryl-H), 2951, 2895 (CH-val.), 2844 (OCH_3), 1717 (C=O), 1607, 1580, 1563, 1506 (arom. C=C, arom. C=N), 1535, 1314 (NO2), 1434 (CH-Def.), 1357 (C-N-val.), 1257, 1105 (CO_2Me), 813 (1,4-disubst. aryl, 1,2,4-trisubst. aryl) cm^{-1}. MS (EI, 70 eV): *m/z* (%) = 756 (65) $[M]^{+\cdot}$, 726 (100) $[M - CH_3O + H]^{+\cdot}$, 238 (61) $[(M - C_{15}H_{10}N_2O_4)/2]^+$, 206 (70) $[(M - C_{15}H_{10}N_2O_4)/2 - CH_3O]^+$, 178 (63) $[(M - C_{15}H_{10}N_2O_4)/2 - C_2H_3O]^+$. HRMS (EI): *m/z* = calcd. 756.2219; found 756.2191 (Δ 3.9 ppm). Elemental analysis ($C_{45}H_{32}N_4O_8$) (756.76): calcd. C 71.42 H 4.26 N 7.40; found C 71.54 H 4.16 N 7.32.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9.20
8.94
8.93
8.93
8.79
8.78
8.15
8.13
8.13
7.80
7.79
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7.72
7.60
7.59
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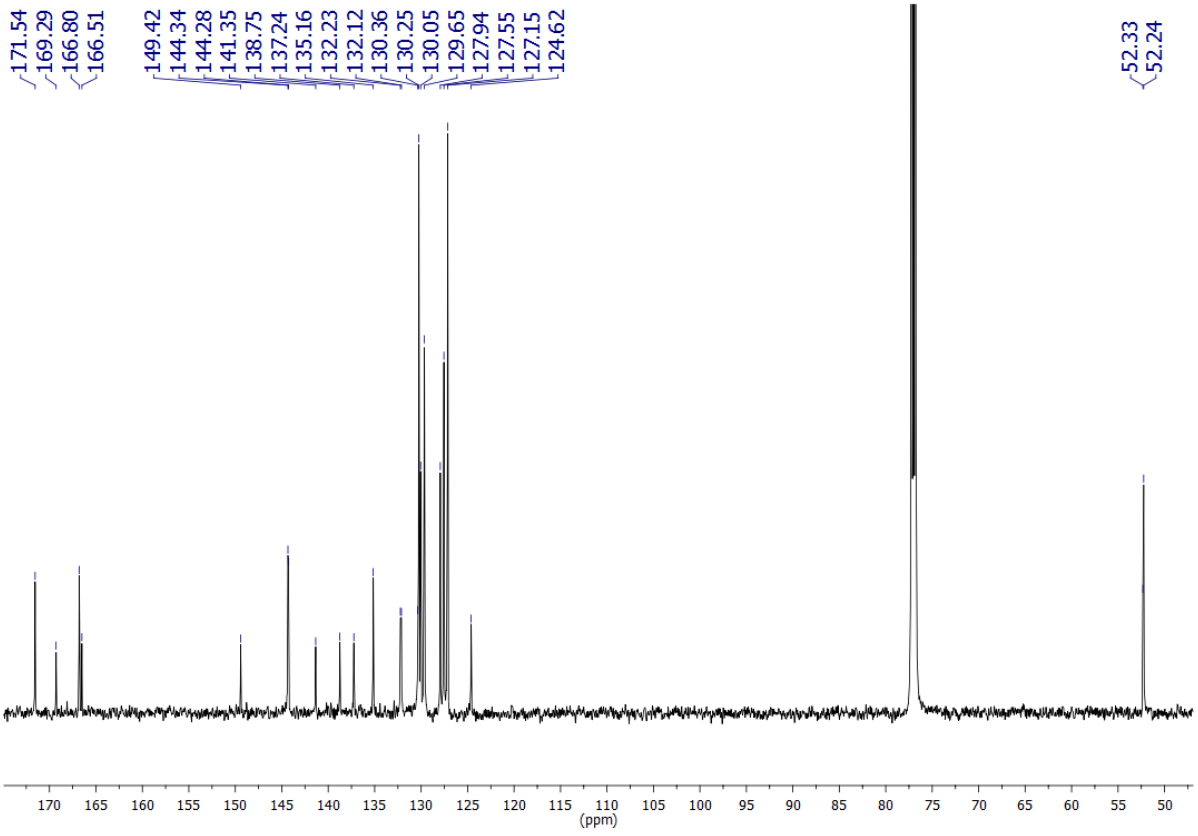
4.11
4.15
1.07
2.08
9.14
(ppm)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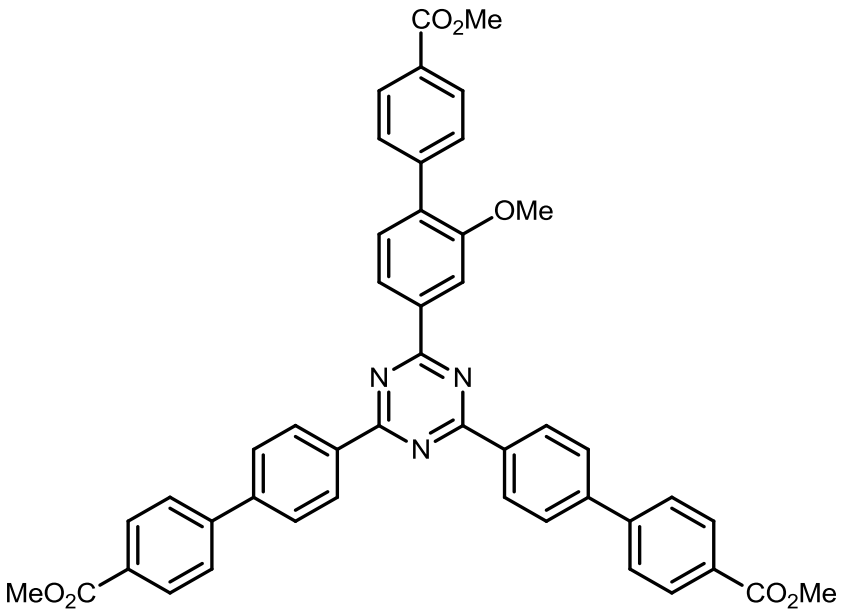
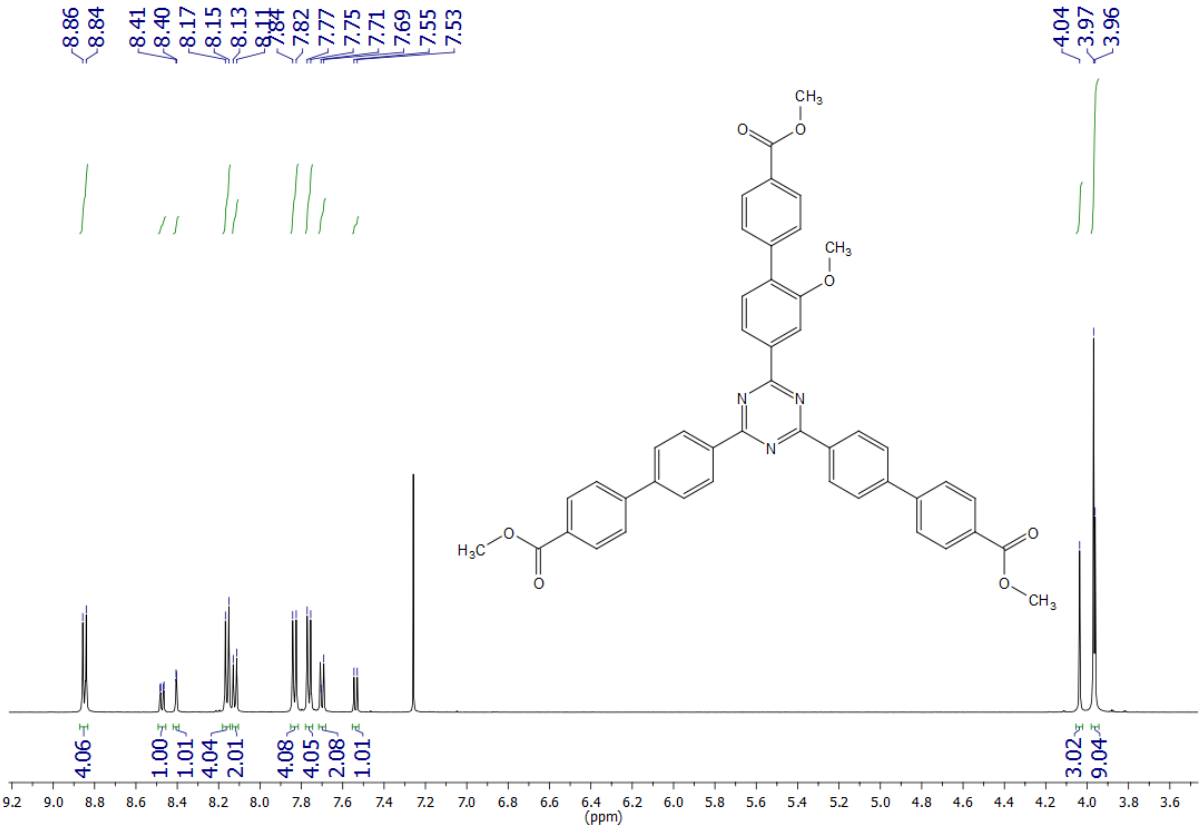
144.28
141.35
138.75
137.24
135.16
132.23
132.12
130.36
130.25
130.05
129.65
127.94
127.55
127.15
124.62
52.33
52.24
(ppm)

2,4-Bis[4'-(methoxycarbonyl)-biphenyl-4-yl]-6-[2-methoxy-4'-(methoxycarbonyl)-biphenyl-4-yl]-1,3,5-triazine (16c)



To a mixture of 2-(4-bromo-3-methoxyphenyl)-4,6-bis(4-bromophenyl)-1,3,5-triazine (**3c**, 100 mg, 174 µmol), 4-methoxycarbonyl-phenylboronic acid (**15**, 130 mg, 783 µmol) and potassium phosphate (250 mg, 1.18 mmol) in 1,4-dioxane (10 mL) and deionized water (1 mL) under nitrogen, tetrakis(triphenylphosphine)palladium(0) (19 mg, 16 µmol) was added. The mixture was stirred for 48 h at 100 °C, the solvent was evaporated in vacuo, the residue was dissolved in chloroform (50 ml) and washed with deionized water (3 x 25 mL) and brine (25 mL). The organic layer was dried with magnesium sulfate and heated after activated charcoal had been added. After filtration through celite, the solvent was evaporated in vacuo and the crude product was recrystallized from a boiling mixture of toluene and *n*-heptane. A colourless solid was obtained. Yield: 94 mg (127 µmol, 73%). M. p.: 203 °C. ^{1}H NMR (500 MHz, $CDCl_{3,}$): δ = 8.85 (d, 4H, ^{3}J = 8.5 Hz, 2,4-Ar-*H*-2,6), 8.47 (dd, 1H, ^{3}J = 7.9 Hz, ^{4}J = 1.5 Hz, 6-Ar-*H*-5), 8.40 (d, 1H, ^{3}J = 1.5 Hz, 6-Ar-*H*-3), 8.16 (d, 4H, ^{3}J = 8.5 Hz, 2,4-Ar'-*H*-3,5), 8.12 (d, 2H, ^{3}J = 8.5 Hz, 6-Ar'-*H*-3,5), 7.83 (d, 4H, ^{3}J = 8.5 Hz, 2,4-Ar-*H*-3,5), 7.76 (d, 4H, ^{3}J = 8.5 Hz, 2,4-Ar'-*H*-2,6), 7.70 (d, 2H, ^{3}J = 8.5 Hz, 6-Ar'-*H*-2,6), 7.54 (d, 1H, ^{3}J = 7.9 Hz, 6-Ar-*H*-6), 4.04 (s, 3H, OCH_3), 3.97 (s, 6H, 2,4-Ar'-CO_2CH_3), 3.96 (s,

3H, 6-Ar'-CO_2CH_3) ppm. ^{13}C NMR (125 MHz, $CDCl_3$): δ = 171.2 (s, triazine-*C*-2,4), 171.2 (s, triazine-*C*-6), 167.0 (s, 2,4-Ar'-*C*O_2Me), 166.9 (s, 6-Ar'-*C*O_2Me), 156.8 (s, 6-Ar-*C*-2), 144.6 (s, 2,4-Ar'-*C*-1), 144.0 (s, 2,4-Ar-*C*-4), 142.6 (s, 6-Ar'-*C*-1), 137.2 (s, 6-Ar-*C*-4), 135.8 (s, 2,4-Ar-*C*-1), 133.8 (s, 6-Ar-*C*-1), 131.0 (s, 6-Ar-*C*-6), 130.2 (d, 2,4-Ar'-*C*-3,5), 129.6 (d, 2,4-Ar-*C*-2,6), 129.6 (d, 6-Ar'-*C*-2,6), 129.5 (s, 2,4-Ar'-*C*-4), 129.4 (d, 6-Ar'-*C*-3,5), 129.0 (s, 6-Ar'-*C*-4), 127.5 (d, 2,4-Ar-*C*-3,5), 127.2 (d, 2,4-Ar'-*C*-2,6), 121.9 (d, 6-Ar-*C*-5), 111.4 (d, 6-Ar-*C*-3), 55.8 (q, OCH_3), 52.2 (q, 2,4-Ar'-CO_2CH_3), 52.1 (q, 6-Ar'-CO_2CH_3) ppm. MS (EI, 70 eV): *m/z* = 741 (100) $[M]^{+\cdot}$, 238 (69) $[M - C_{31}H_{23}N_2O_5]^+$. IR (ATR): $\tilde{\nu}$ = 3005 (aryl-H), 2954, 2878, 2840 (CH-val.), 1715 (C=O), 1607, 1582, 1508, 1419 (arom. C=C, arom. C=N), 1434 (CH-Def.), 1360 (C-N-val.), 1273 (aryl-OCH_3), 812 (1,4-disubst. aryl, 1,3,4-trisubst. aryl) cm^{-1}. HRMS (EI): *m/z* = calcd. 741.2475; found 741.2465 (Δ 1.42 ppm). Elemental analysis ($C_{46}H_{35}N_3O_7$) (741.79): calcd. C 74.48 H 4.76 N 5.66; found C 74.59 H 4.93 N 5.69.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8.41
8.40
8.17
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8.13
7.82
7.77
7.75
7.71
7.69
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7.53
4.04
3.97
3.96
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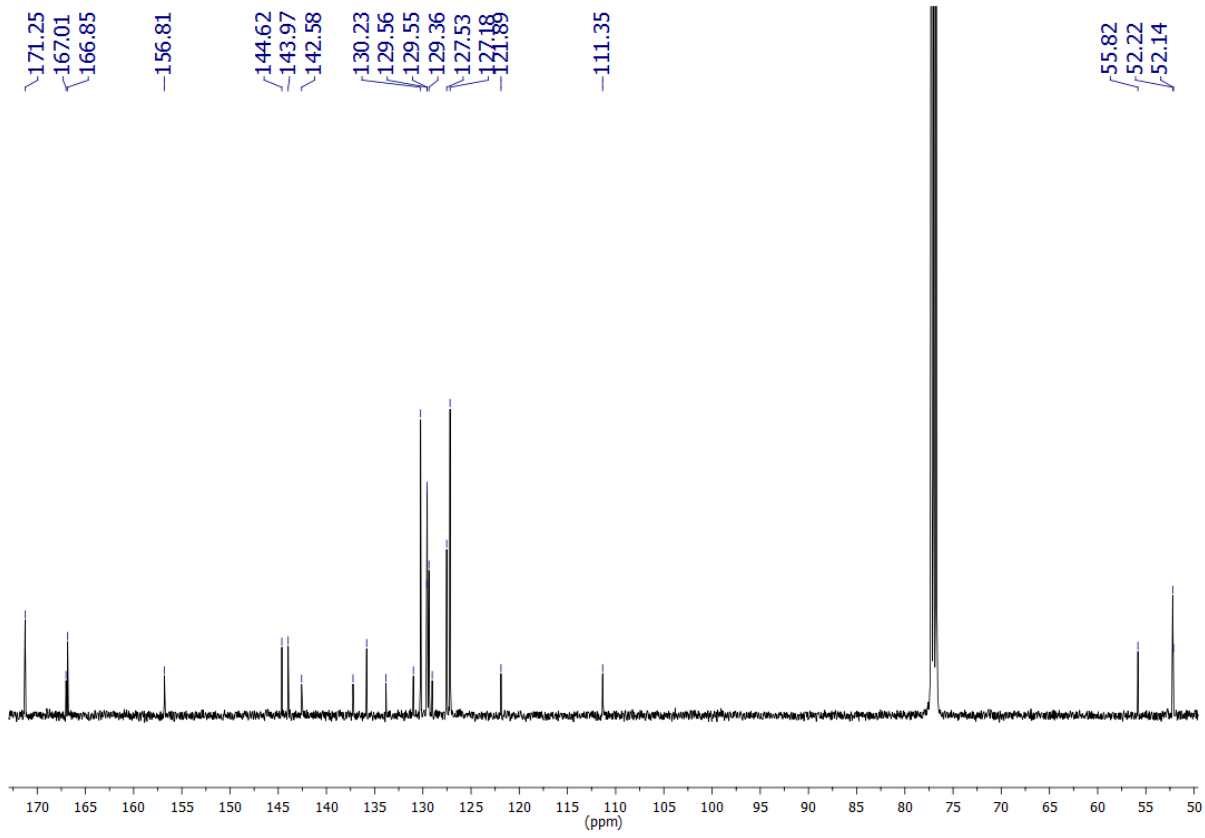
4.05
2.08
1.01
3.02
9.04
(ppm)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167.01
166.85
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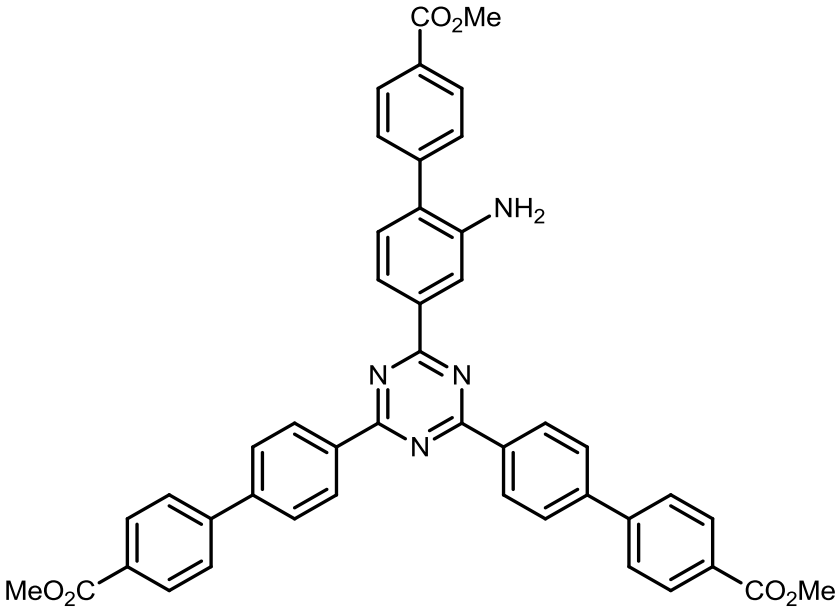
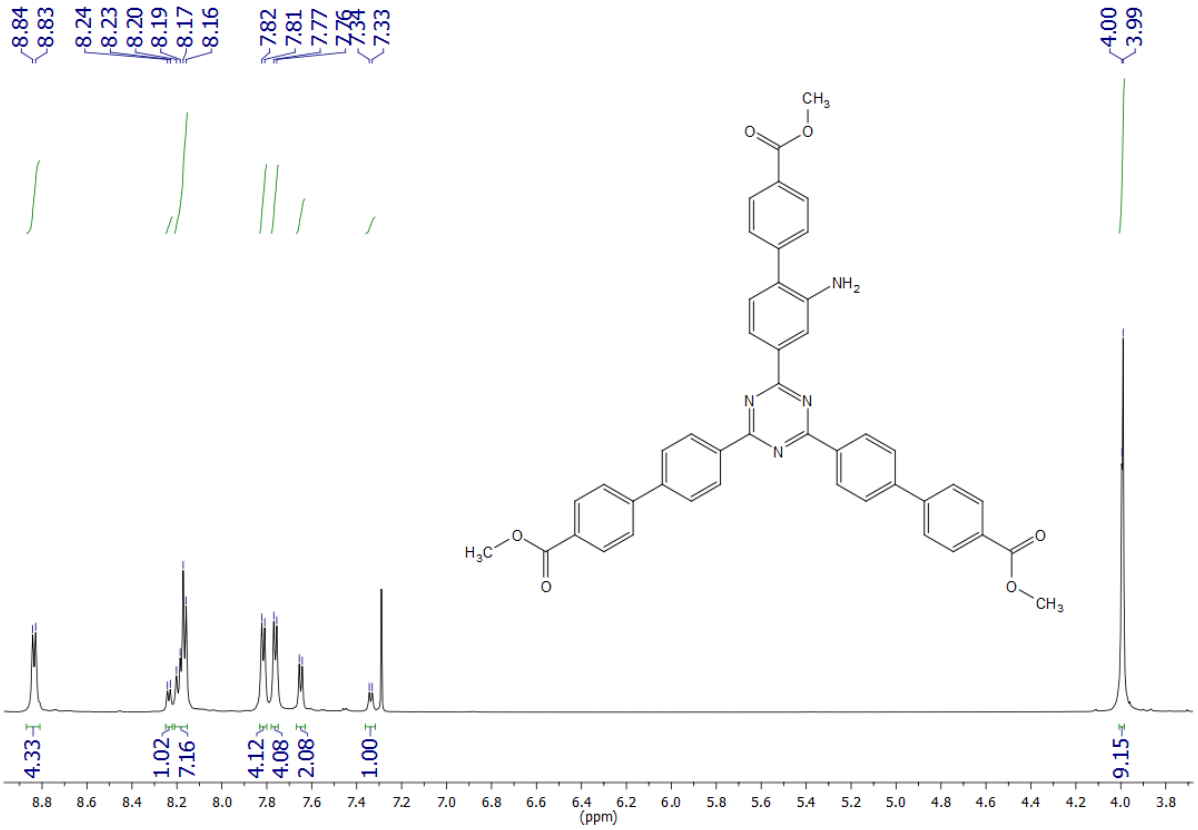
144.62
143.97
142.58
130.23
129.56
129.55
129.36
127.53
111.35
55.82
52.22
52.14
(ppm)

2-[2-Amino-4'-(methoxycarbonyl)-biphenyl-4-yl]-4,6-bis[4'-(methoxycarbonyl)-biphenyl-4-yl]-1,3,5-triazine (16d)



An autoclave was filled with 2-[4'-(methoxycarbonyl)-2-nitro-biphenyl-4-yl]-4,6-bis[4'-(methoxycarbonyl)-biphenyl-4-yl]-1,3,5-triazine (**16b**, 100 mg, 132 µmol) and palladium on charcoal (45 mg, 10%) in dichloromethane (5 mL). The apparatus was evacuated five times and refilled with hydrogen (5 bar). After stirring under a 5 bar hydrogen atmosphere for 5 d at room temp., the mixture was filtered through celite and the solvent was evaporated in vacuo. The crude product was recrystallized from a boiling mixture of toluene and *n*-heptane yielding a yellow solid. Yield: 77 mg (0.11 mmol, 77%). M. p.: 225 °C. ^{1}H NMR (600 MHz, $CDCl_3$): δ = 8.85 (d, 4H, ^{3}J = 8.3 Hz, 4,6-Ar-*H*-3,5), 8.25 (dd, 1H, ^{3}J = 8.0 Hz, ^{4}J = 1.3 Hz, 2-Ar-*H*-5), 8.20 (br. s, 1H, 2-Ar-*H*-3), 8.17 (d, 2H, ^{3}J = 8.2 Hz, 2-Ar'-*H*-3,5), 8.16 (d, 4H, ^{3}J = 8.4 Hz, 4,6-Ar'-*H*-3,5), 7.83 (d, 4H, ^{3}J = 8.4 Hz, 4,6-Ar'-*H*-2,6), 7.70 (d, 4H, ^{3}J = 8.3 Hz, 4,6-Ar-*H*-2,6), 7.64 (d, 2H, ^{3}J = 8.2 Hz, 2-Ar-*H*-2,6), 7.34 (d, 1H, ^{3}J = 8.0 Hz, 2-Ar-*H*-6), 3.97 (s, 3H, 2-Ar'-CO_2CH_3), 3.96 (s, 6H, 4,6-Ar'-CO_2CH_3) ppm. ^{13}C NMR (150 MHz, $CDCl_3$): δ = 171.4 (s, triazine-*C*-2), 171.1 (s, triazine-*C*-4,6), 166.9 (s, 4,6-Ar'-CO_2Me), 166.8 (s, 2-Ar'-CO_2Me), 144.6 (s, 2-Ar-*C*-2), 143.8 (s, 2-Ar'-*C*-1), 143.7 (s, 4,6-Ar-*C*-1), 143.7 (s, 4,6-Ar'-*C*-1), 136.8 (s, 2-Ar-*C*-4), 135.9 (s, 4,6-Ar-*C*-4),

130.9 (d, 2-Ar-*C*-6), 130.7 (d, 4,6-Ar'-*C*-3,5), 130.5 (s, 4,6-Ar'-*C*-4), 130.2 (d, 2-Ar'-*C*-3,5), 130.0 (s, 2-Ar-*C*-1), 129.5 (d, 4,6-Ar-*C*-3,5), 129.3 (s, 2-Ar-*C*-4), 128.9 (d, 2-Ar'-*C*-2,6), 127.4 (d, 4,6-Ar-*C*-2,6), 127.2 (d, 4,6-Ar'-*C*-2,6), 119.6 (d, 2-Ar-*C*-5), 116.1 (d, 2-Ar-*C*-3), 52.2 (q, 2-Ar'-CO_2CH_3), 52.2 (q, 4,6-Ar'-CO_2CH_3) ppm. MS (EI, 70 eV): *m/z* = 726 (100) $[M]^{+\cdot}$. IR (ATR): $\tilde{\nu}$ = 3350 (NH_2), 3001 (aryl-H), 2954, 2846 (CH–val.), 1715 (C=O), 1607, 1582, 1507, 1419 (arom. C=C, arom. C=N), 1434 (CH-Def.) 1360 (C-N-val.), 812 (1,4-disubst. aryl, 1,3,4-trisubst. aryl) cm^{-1}. HRMS (EI): *m/z* = calcd. 726.2478; found 726.2465 (Δ 1.78 ppm). Elemental analysis ($C_{45}H_{34}N_4O_6$) (726.77): calcd. C 74.73 H 7.71 N 4.72; ($C_{45}H_{34}N_4O_6 \cdot 1.3H_2O \cdot 0.5C_7H_8$) (796.26): calcd. C 73.16 H 5.14 N 7.04; found C 73.27 H 5.46 N 7.35.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8.83
8.24
8.23
8.20
8.19
8.17
8.16
7.82
7.81
7.77
7.34
7.33
4.00
3.99
CH3
O
O
NH2
N
N
N
H3C
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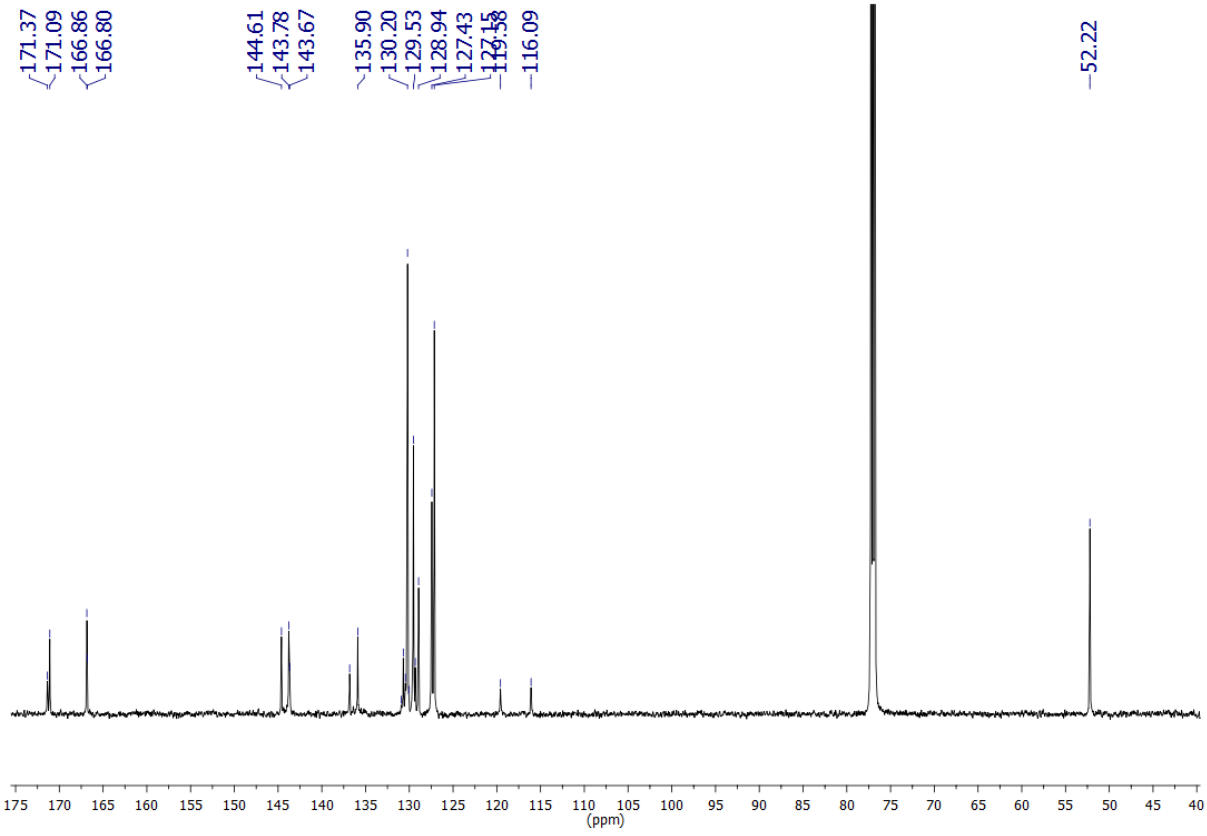
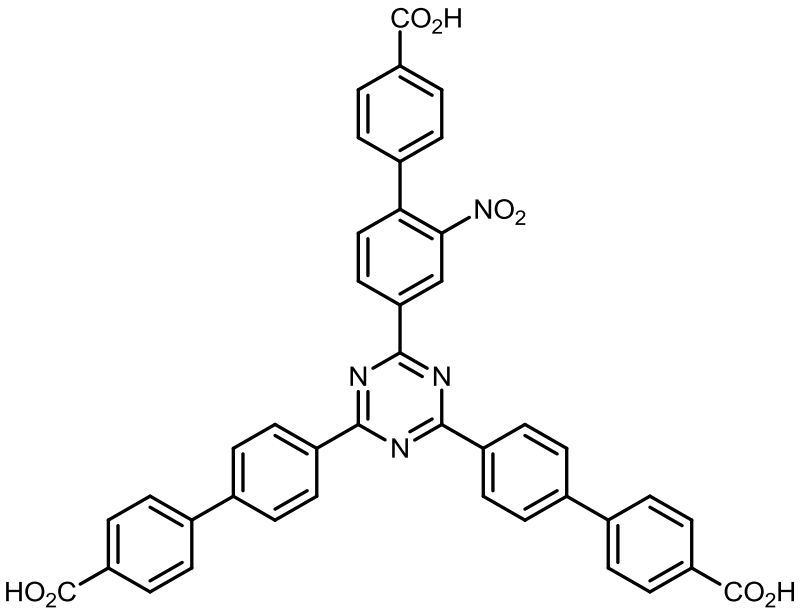
4.12
4.08
2.08
1.00
9.15
(ppm)

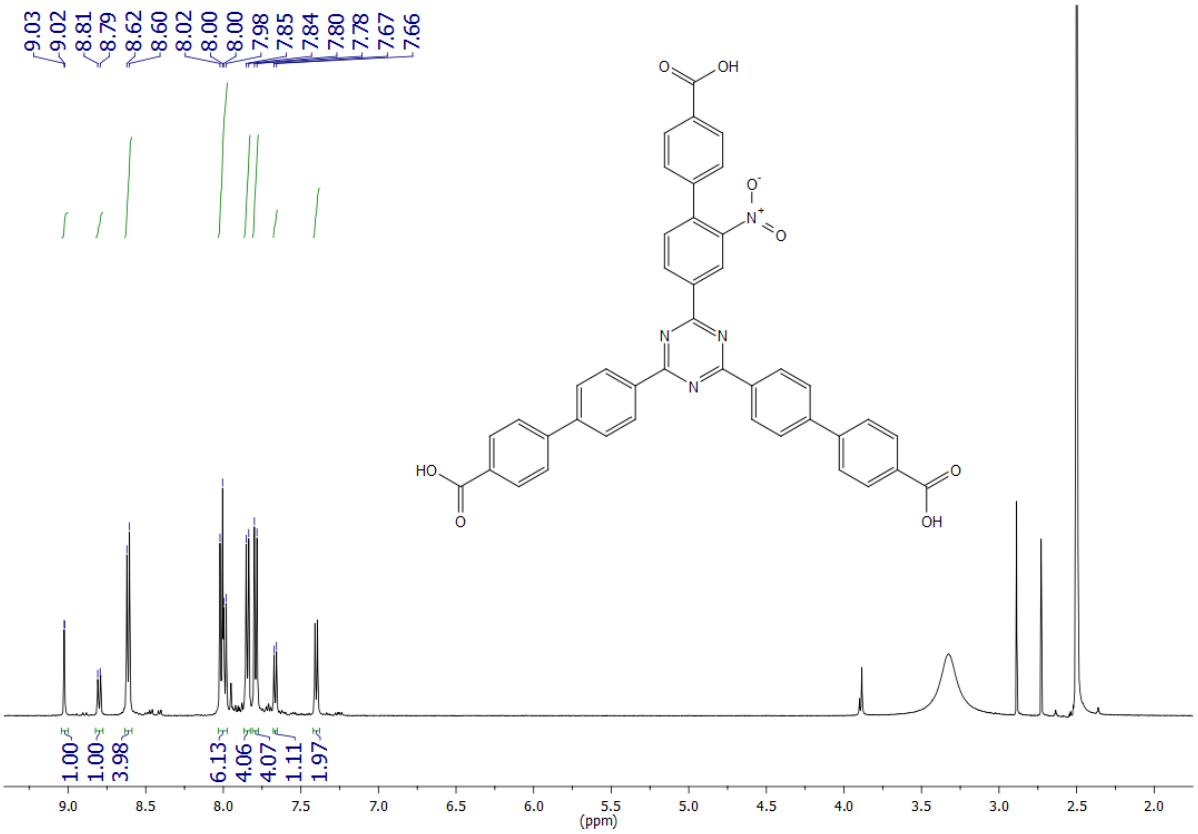

171.37
171.09
166.86
166.80
144.61
143.78
143.67
135.90
130.20
129.53
128.94
127.43
116.09
52.22
(ppm)

2,4-Bis(4'-carboxy-biphenyl-4-yl)-6-(4'-carboxy-2-nitro-biphenyl-4-yl)-1,3,5-triazine (17b)



Lithium hydroxide monohydrate (5.12 g, 122 mol) was added to 2-[4'-(methoxycarbonyl)-2-nitro-biphenyl-4-yl]-4,6-bis[4'-(methoxycarbonyl)-biphenyl-4-yl]-1,3,5-triazine (**16b**, 3.30 g, 4.36 mmol) in a mixture of tetrahydrofuran (430 mL) and deionized water (45 mL). After stirring for 48 h at room temp., the solvent was evaporated in vacuo, the yellow residue was dissolved in water and acidified with hydrochloric acid (3 M). The yellow precipitate was filtered off and washed with deionized water and chloroform. Yield: 2.99 g (4.18 mmol, 96%). M. p.: >300 °C. ^{1}H NMR (500 MHz, DMSO-d_6): δ = 8.99 (d, 1H, ^{4}J = 1.4 Hz, 6-Ar-*H*-2), 8.77 (dd, 1H, ^{3}J = 7.9 Hz, ^{4}J = 1.4 Hz, 6-Ar-*H*-6), 8.58 (d, 4H, ^{3}J = 8.3 Hz, 2,4-Ar-*H*-2,6), 8.00 (d, 4H, ^{3}J = 8.3 Hz, 2,4-Ar'-*H*-3,5), 7.98 (d, 2H, ^{3}J = 8.3 Hz, 6-Ar'-*H*-3,5), 7.82 (d, 4H, ^{3}J = 8.3 Hz, 2,4-Ar-*H*-3,5), 7.77 (d, 4H, ^{3}J = 8.3 Hz, 2,4-Ar'-*H*-2,6), 7.64 (d, 1H, ^{3}J = 7.9 Hz, 6-Ar-*H*-5), 7.38 (d, 2H, ^{3}J = 8.3 Hz, 6-Ar'-*H*-2,6) ppm. ^{13}C NMR (125 MHz, DMSO-d_6): δ = 171.1 (s, triazine-*C*-2,4), 169.1 (s, triazine-*C*-6), 167.4 (s, 2,4-Ar-*C*O_2H), 167.2 (s, 6-Ar-*C*O_2H), 149.2 (d, 6-Ar-*C*-3), 143.7 (s, 2,4-Ar'-*C*-1), 143.4 (s, 2,4-Ar-*C*-4), 141.1 (s, 6-Ar'-*C*-1), 138.4 (s, 6-Ar-*C*-4), 136.7 (s, 6-Ar-*C*-1), 134.9 (s, 2,4-Ar-*C*-1), 132.9 (s, 6-Ar'-*C*-4), 132.7 (d, 6-Ar-*C*-6), 131.3 (s, 2,4-Ar'-*C*-4), 130.8 (d, 6-Ar-*C*-5), 130.4 (d, 2,4-Ar'-*C*-3,5), 130.0 (d, 2,4-Ar-*C*-3,5), 129.7 (d, 6-Ar'-

C-2,6), 129.0 (d, 2,4-Ar-*C*-2,6), 127.7 (6-Ar'-*C*-3,5), 127.4 (d, 2,4-Ar'-*C*-2,6), 124.3 (s, 6-Ar-*C*-2) ppm. MS (MALDI, Cl-CCA): *m/z* = 715 $[M + H]^+$. IR (ATR): $\tilde{\nu}$ = 3400 (br., OH), 3005 (aryl-H), 1714 (C=O), 1607, 1582, 1506, 1419 (arom. C=C, arom. C=N), 1510, 1316 (NO_2), 1359 (C-N–val.), 813 (1,4-disubst. aryl, 1,3,4-trisubst. aryl) cm^{-1}. Elemental analysis ($C_{42}H_{26}N_4O_8$) (714.69): calcd. C 70.58 H 3.67 N 7.84; ($C_{42}H_{26}N_4O_8 \cdot 1.35H_2O$) (738.99): calcd. C 68.69 H 3.91 N 7.58; found C 68.69 H 4.35 N 7.36.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8.00
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7.84
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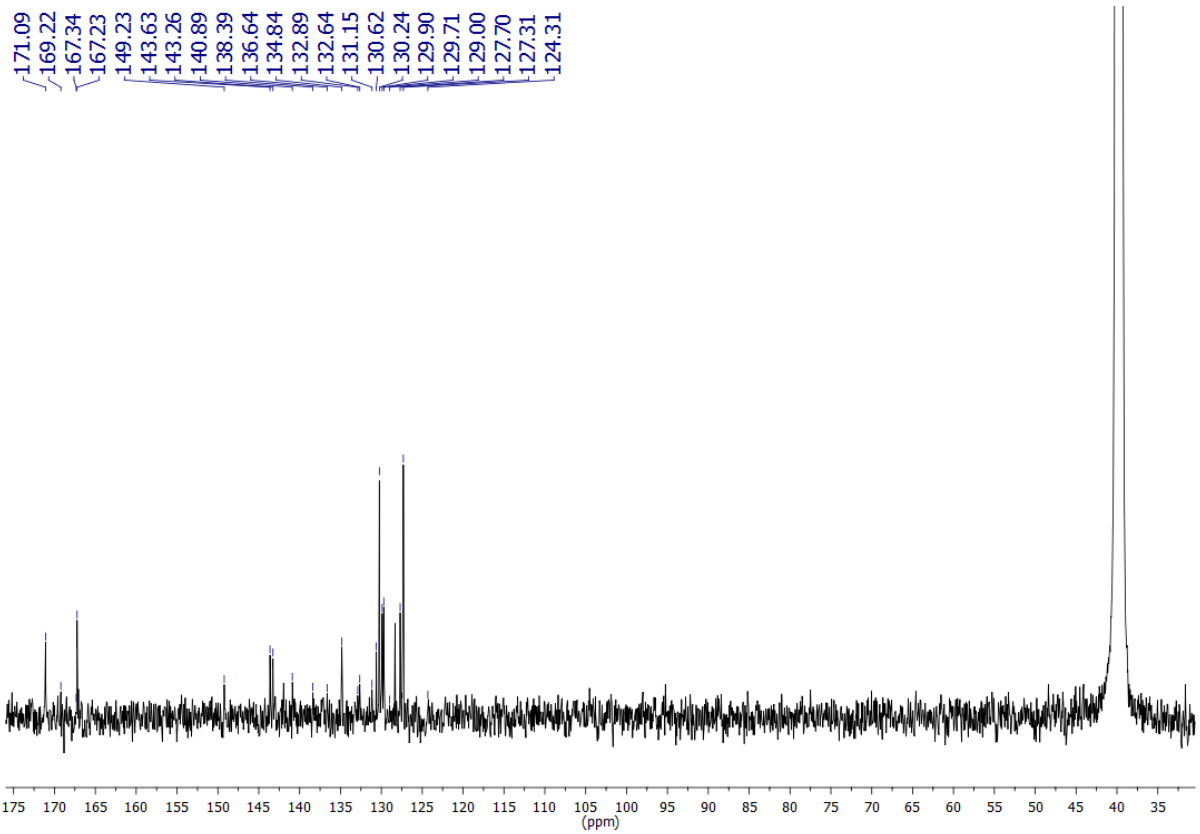
6.13
4.06
4.07
1.11
1.97
(ppm)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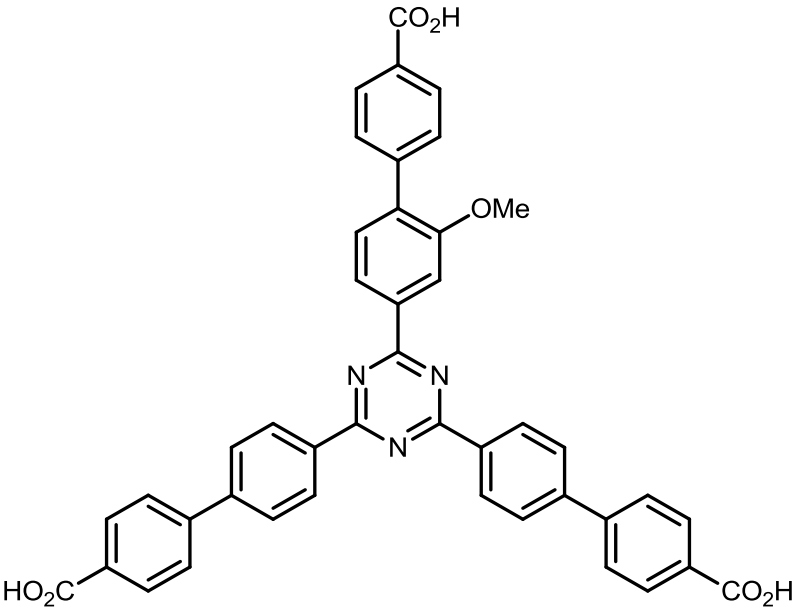
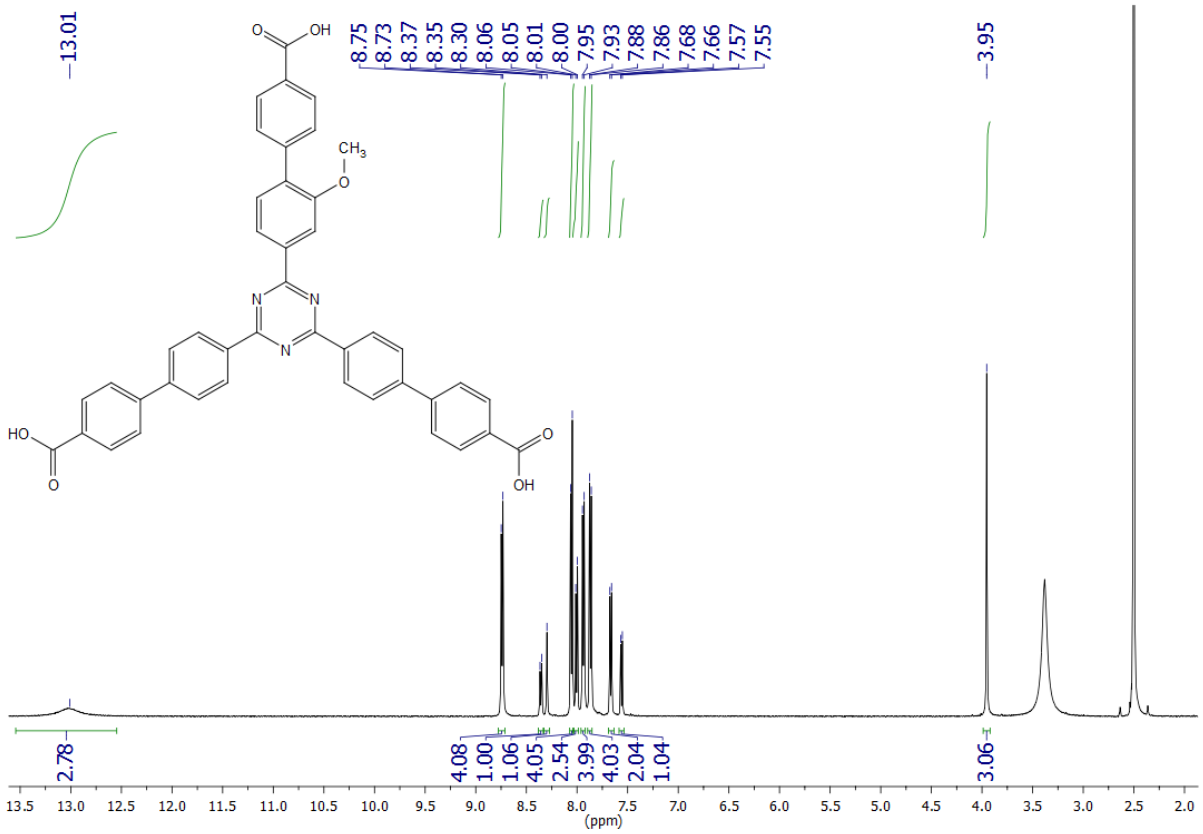
134.84
132.89
132.64
131.15
130.62
130.24
129.90
129.71
129.00
127.70
127.31
124.31
(ppm)

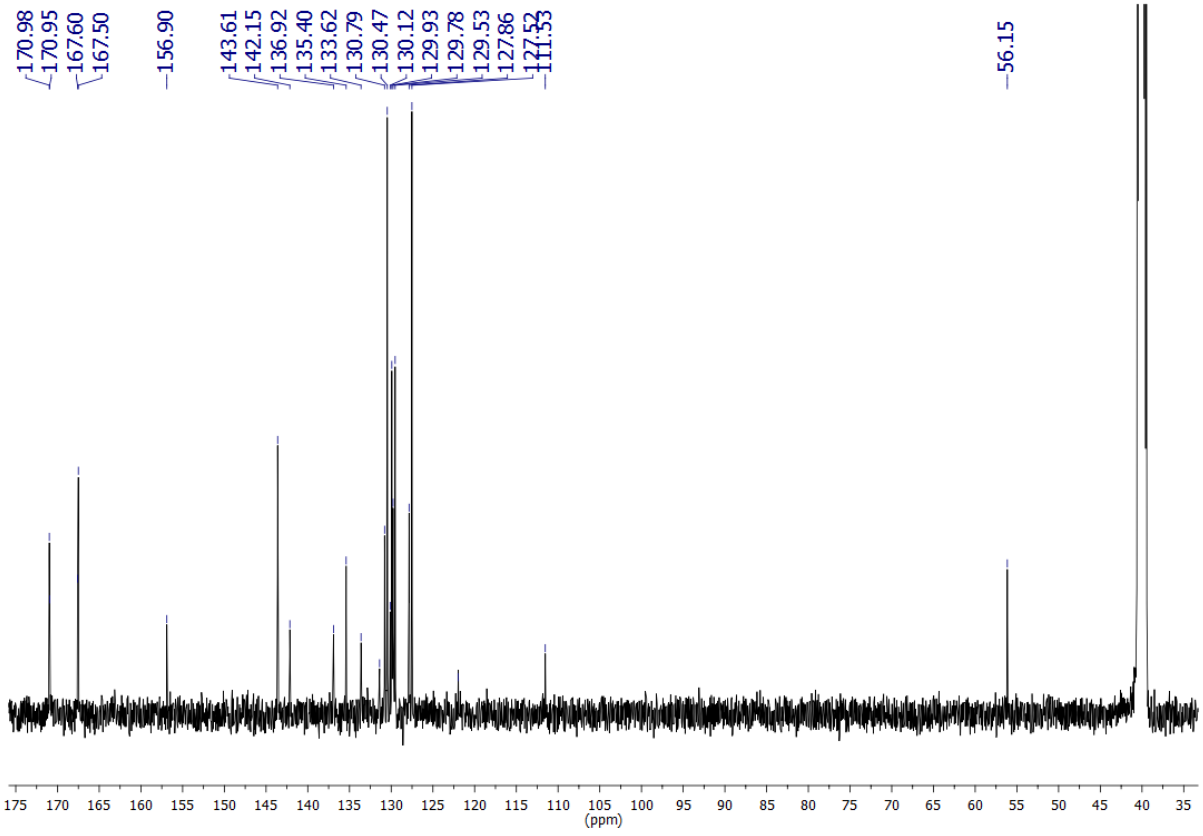
2,4-Bis(4’-carboxy-biphenyl-4-yl)-6-(4’-carboxy-2-methoxy-biphenyl-4-yl]-1,3,5-triazine (17c)



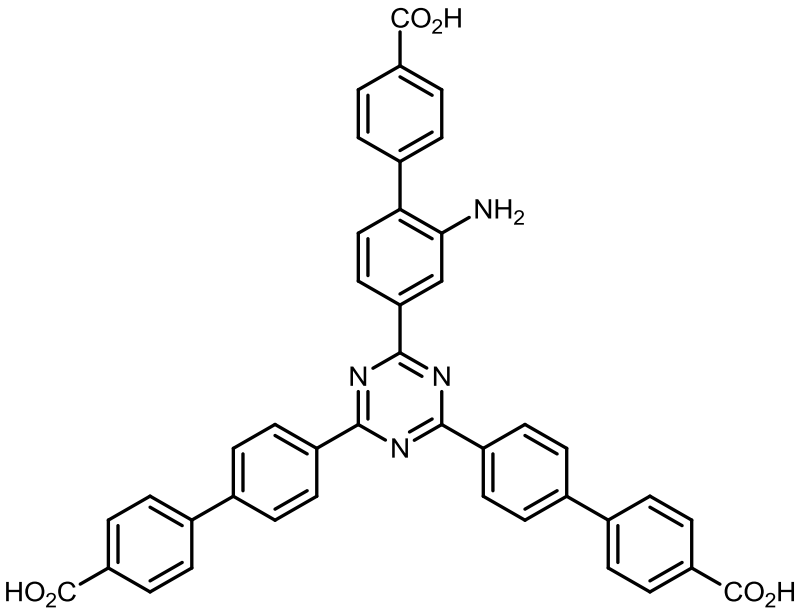
A suspension of 2,4-bis[4’-(methoxy-carbonyl)biphenyl-4-yl]-6-[2-methoxy-4’-(methoxycarbonyl)-biphenyl-4-yl]-1,3,5-triazine (**16c**, 50 mg, 67.5 µmol) and lithium hydroxide monohydrate (101 mg, 2.41 mmol) in a mixture of tetrahydrofuran (10 mL) and deionized water (1.5 mL) was stirred at 60 °C for 24 h. After removal of the tetrahydrofuran in vacuo, the aq. layer was diluted slightly and acidified with hydrochloric acid (3 M). The precipitate was filtered off and washed thoroughly with deionized water and chloroform. Yield: 47 mg (67 µmol, >99%). M. p.: >300 mg. ^{1}H NMR (500 MHz, DMSO-d_6): δ = 13.01 (br. s, 3H, CO_2H), 8.74 (d, 4H, ^{3}J = 8.4 Hz, 2,4-Ar-*H*-3,5), 8.36 (m_c (dd), 1H, J = 7.9 Hz, ^{4}J = 1.2 Hz, 6-Ar-*H*-5), 8.29 (m_c (d), 1H, ^{4}J = 1.2 Hz, 6-Ar-*H*-3), 8.05 (d, 4H, ^{3}J = 8.4 Hz, 2,4-Ar’-*H*-3,5), 8.00 (d, 2H, ^{3}J = 8.4 Hz, 6-Ar’-*H*-3,5), 7.94 (d, 4H, ^{3}J = 8.4 Hz, 2,4-Ar-*H*-2,6), 7.86 (d, 4H, ^{3}J = 8.4 Hz, 2,4-Ar’-*H*-2,6), 7.67 (d, 2H, ^{3}J = 8.4 Hz, 6-Ar’-*H*-2,6), 7.56 (d, 1H, ^{3}J = 7.9 Hz, 6-Ar-*H*-6), 3.95 (s, 3H, OCH_3) ppm. ^{13}C NMR (125 MHz, DMSO-d_6): δ = 171.0 (s, triazine-*C*-2,4), 170.9 (s, triazine-*C*-6), 167.6 (s, 6-Ar’-CO_2H), 167.5 (s, 2,4-Ar’-CO_2H), 156.9 (s, 6-Ar-*C*-2), 143.6 (br. s, 2,4-Ar-*C*-1, 2,4-Ar’-*C*-1), 142.1 (s, 6-Ar’-*C*-1), 136.9 (s, 6-Ar-*C*-4), 135.4 (s, 2,4-Ar-*C*-4), 133.6 (s, 6-Ar-*C*-1), 131.7 (d, 6-Ar-*C*-6), 130.8 (s, 2,4-Ar’-*C*-4), 130.5 (d, 2,4-Ar’-*C*-

3,5), 130.1 (s, 6-Ar'-*C*-4), 129.9 (d, 6-Ar'-*C*-2,6), 129.8 (d, 2,4-Ar-*C*-3,5), 129.5 (d, 6-Ar'-*C*-3,5), 127.9 (d, 2,4-Ar-*C*-2,6), 127.5 (d, 2,4-Ar'-*C*-2,6), 121.9 (d, 6-Ar-*C*-5), 111.5 (d, 6-Ar-*C*-3), 56.1 (q, O*C*H_3) ppm. MS (MALDI, Cl-CCA): *m/z* = 700 $[M + H]^+$. IR (ATR): $\tilde{\nu}$ = 3100 (br., OH), 1684 (C=O), 1605, 1581, 1505, 1417 (arom. C=C, arom. C=N), 1357 (C-N-val.), 1231 (aryl-OCH_3), 813 (1,4-disubst. aryl, 1,3,4-trisubst. aryl) cm^{-1}. Elemental analysis ($C_{43}H_{29}N_3O_7$) (699.70): calcd. C 73.81 H 4.18 N 6.01; ($C_{43}H_{29}N_3O_7 \cdot 0.4H_2O \cdot 0.25CHCl_3$) (738.99): calcd. C 70.51 H 4.11 N 5.70; found C 70.47 H 4.11 N 5.73.


13.01
8.75
8.73
8.37
8.35
8.30
8.06
8.05
8.01
8.00
7.95
7.93
7.88
7.86
7.68
7.66
7.57
7.55
3.95
2.78
4.08
1.00
1.06
4.05
2.54
3.99
4.03
2.04
1.04
3.06
(ppm)

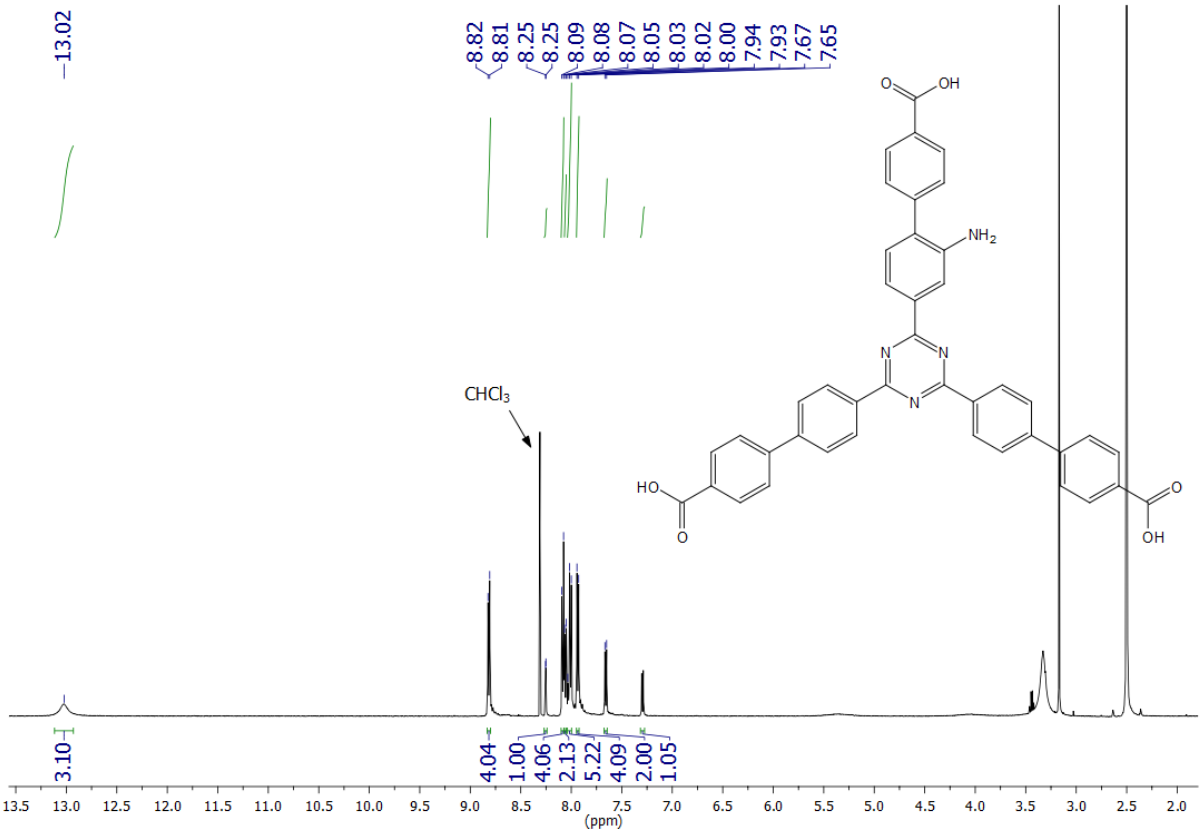

170.98
170.95
167.60
167.50
156.90
143.61
142.15
136.92
135.40
133.62
130.79
130.47
130.12
129.93
129.78
129.53
127.86
56.15
(ppm)

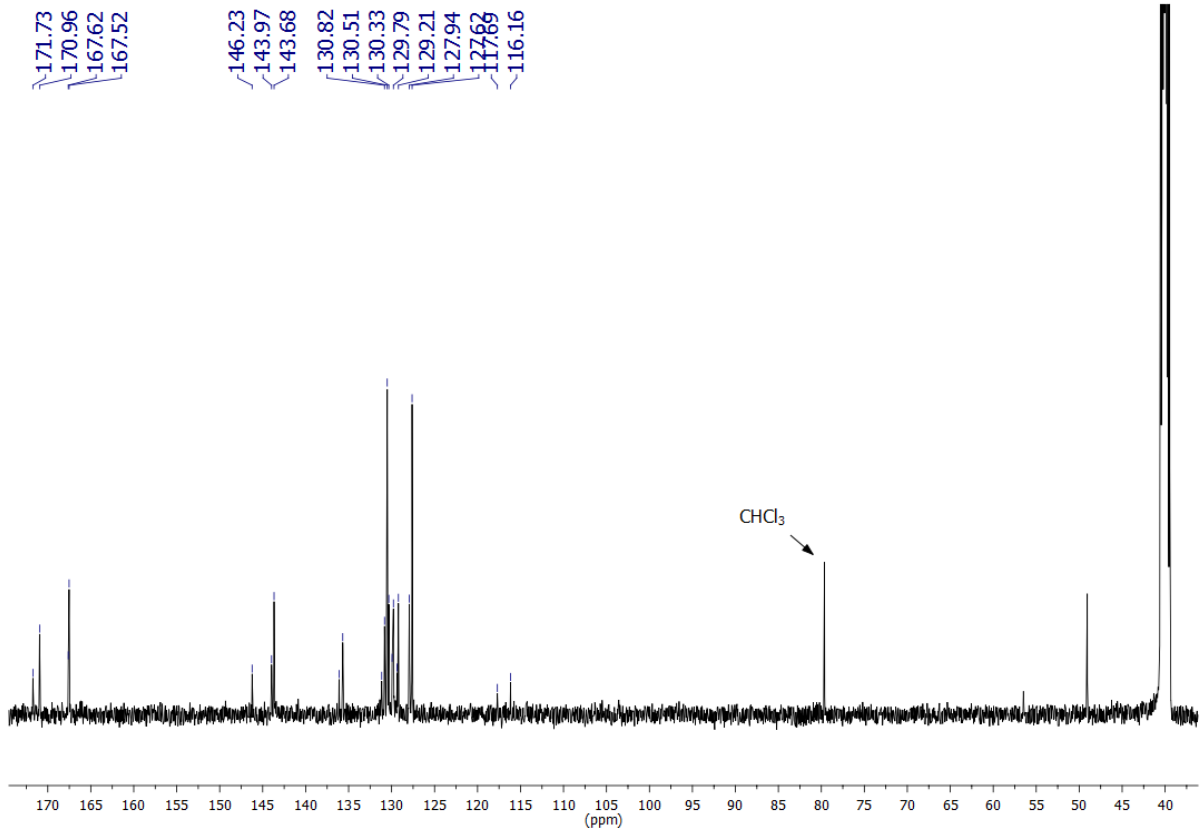
2-(2-Amino-4'-carboxy-biphenyl-4-yl)-4,6-bis(4'-carboxy-biphenyl-4-yl)-1,3,5-triazine (17d)



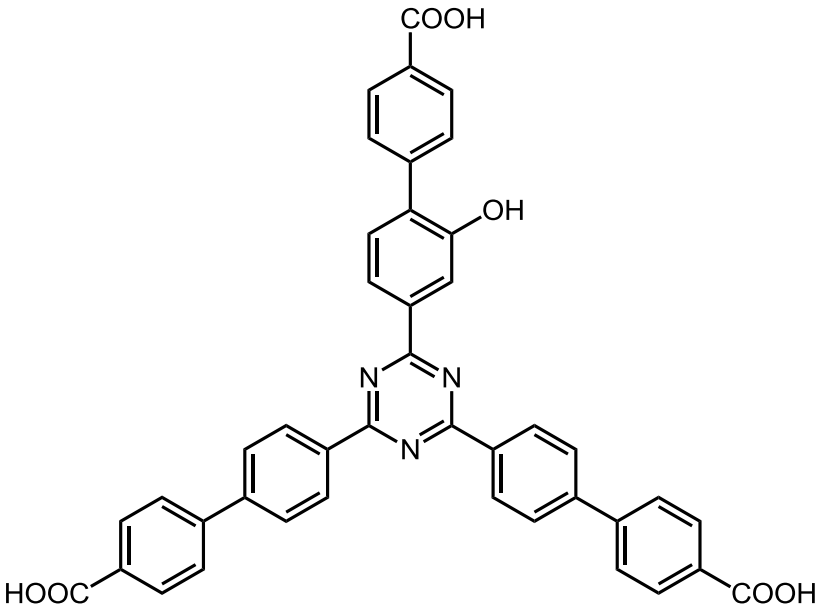
Lithium hydroxide mono-hydrate (101 mg, 2.41 mmol) and 2-[2-amino-4'-carboxy-biphenyl-4-yl]-4,6-bis[4'-carboxy-biphenyl-4-yl]-1,3,5-triazine (**16d**, 50 mg, 69 µmol) in a mixture of tetrahydrofuran (430 mL) and deionized water (45 mL) were stirred at 60 °C for 48 h. After evaporation of the solvent in vacuo, the yellow residue was dissolved in deionized water and acidified with hydrochloric acid (3 M). The yellow precipitate was filtered off and washed with deionized water and chloroform. Yield: 47 mg (68 µmol, >99%). M. p.: >300 °C. ^{1}H NMR (500 MHz, DMSO-d_6): δ = 13.02 (br. s, 3H, CO_2H), 8.81 (d, 4H, ^{3}J = 8.5 Hz, 4,6-Ar-*H*-3,5), 8.25 (d, 1H, ^{4}J = 1.6 Hz, 2-Ar-*H*-3), 8.08 (d, 4H, ^{3}J = 8.5 Hz, 4,6-Ar'-*H*-3,5), 8.05 (d, 2H, ^{3}J = 8.4 Hz, 2-Ar'-*H*-3,5), 8.03* (dd, 1H, ^{4}J = 1.6 Hz, 2-Ar-*H*-5), 8.01 (d, 4H, ^{3}J = 8.5 Hz, 4,6-Ar'-*H*-2,6), 7.93 (d, 4H, ^{3}J = 8.5 Hz, 4,6-Ar-*H*-2,6), 7.66 (d, 2H, ^{3}J = 8.4 Hz, 2-Ar'-*H*-2,6), 7.29 (d, 1H, ^{3}J = 8.0 Hz, 2-Ar-*H*-6) ppm. *signal overlaps with adjacent one unabling the determination of all coupling constants. ^{13}C NMR (125 MHz, DMSO-d_6): δ = 171.1 (s, triazine-*C*-2), 171.0 (s, triazine-*C*-4,6), 167.6 (s, 2-Ar'-CO_2H), 167.5 (s, 4,6-Ar'-CO_2H), 146.2 (s, 2-Ar-*C*-2), 144.0 (s, 2-Ar'-*C*-1), 143.7 (s, 4,6-Ar-*C*-1), 143.7 (s, 4,6-Ar'-*C*-1), 136.1 (s, 2-Ar-*C*-4), 135.7 (s, 4,6-Ar-*C*-4), 131.2 (d, 2-Ar-*C*-6), 130.8 (s, 4,6-Ar'-*C*-4), 130.5 (d, 4,6-Ar'-*C*-3,5), 130.3 (d, 2-

Ar'-*C*-3,5), 129.9 (s, 2-Ar'-*C*-4), 129.8 (d, 4,6-Ar-*C*-3,5), 129.4 (s, 2-Ar-*C*-1), 129.2 (d, 2-Ar'-*C*-2,6), 127.9 (d, 4,6-Ar-*C*-2,6), 127.6 (d, 4,6-Ar'-*C*-2,6), 117.7 (d, 2-Ar-*C*-5), 116.2 (d, 2-Ar-*C*-3) ppm. MS (MALDI, Cl-CCA): *m/z* = 686 $[M + H]^+$. IR (ATR): $\tilde{\nu}$ = 3100 (br., OH, NH_2), 1684 (C=O), 1606, 1581, 1507, 1419 (arom. C=C, arom. C=N), 1561 (NH_2), 1434 (CH-Def.) 1360 (C-N-val.), 810 (1,4-disubst. aryl, 1,3,4-trisubst. aryl) cm^{-1}. Elemental analysis ($C_{42}H_{18}N_4O_6$) (684.69): calcd. C 73.68 H 4.12 N 8.18; ($C_{42}H_{18}N_4O_6 \cdot 0.4CHCl_3 \cdot 0.2H_2O$) (753.89): calcd. C 69.19 H 3.94 N 7.61; found C 69.41 H 3.91 N 7.31.


13.02
8.82
8.81
8.25
8.25
8.09
8.08
8.07
8.05
8.03
8.02
8.00
7.94
7.93
7.67
7.65
CHCl3
NH2
OH
HO
O
N
3.10
4.04
1.00
4.06
2.13
5.22
4.09
2.00
1.05
(ppm)

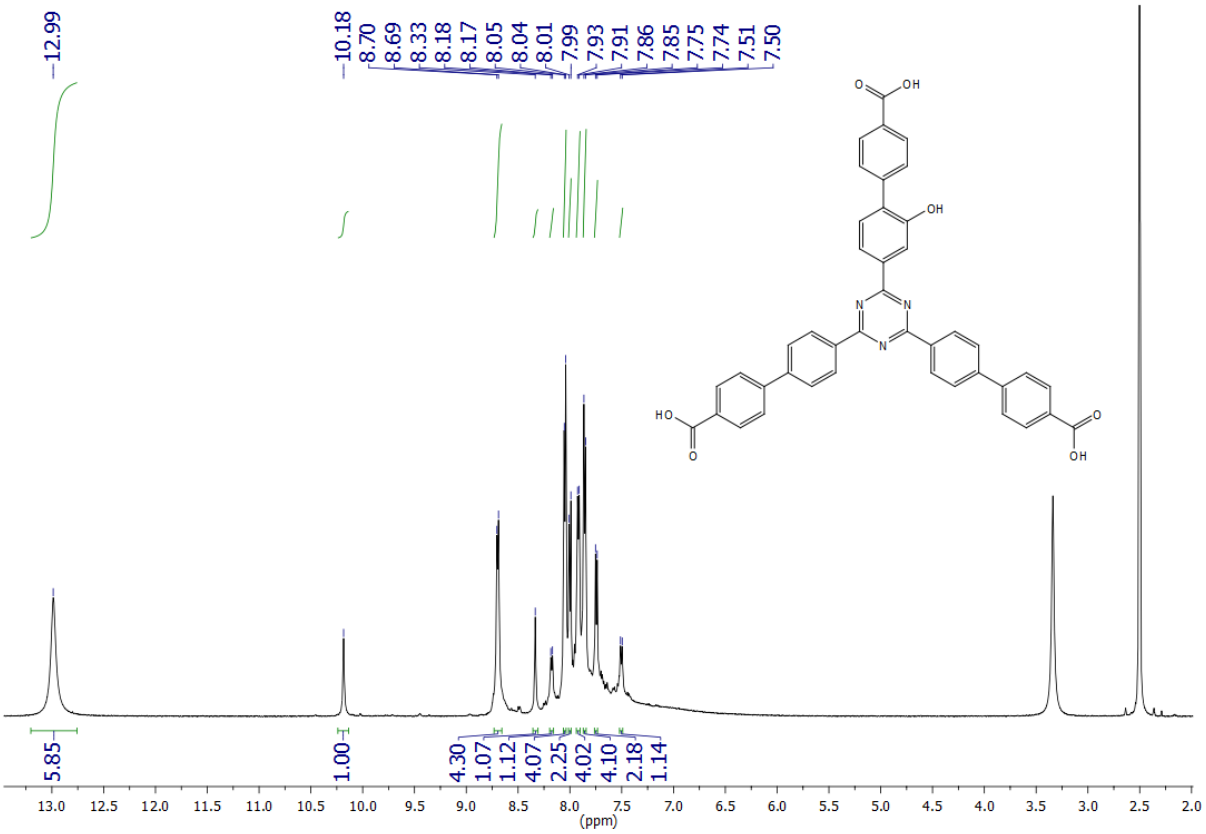

171.73
170.96
167.62
167.52
146.23
143.97
143.68
130.82
130.51
130.33
129.79
129.21
127.94
116.16
CHCl3
(ppm)

2,4-Bis(4'-carboxy-biphenyl-4-yl)-6-(4'-carboxy-2-hydroxy-biphenyl-4-yl)-1,3,5-triazine (17e)



2,4-Bis[4'-(methoxy-carbonyl)-biphenyl-4-yl]-6-[2-methoxy-4'-(methoxycarbonyl)-biphenyl-4-yl]-1,3,5-triazine (**16c**, 300 mg, 400 µmol) and pyridinium hydrochloride (3.60 g, 31.2 mmol) were mixed and stirred for 12 h at 200 °C. After cooling to room temp., sodium hydroxide (2 M) was added to the solution until pH 14 was reached. After stirring for 12 h at 120 °C, the reaction mixture was acidified with conc. hydrochloric acid and the precipitate was filtered off. Washing with water and drying gave a dark brown solid. Yield: 143 mg (209 µmol, 52%). M. p.: > 300 °C. ^{1}H NMR (500 MHz, DMSO-d_6): δ = 12.99 (br. s, 3H, COO*H*), 10.18 (s, 1H, O*H*), 8.70 (d, 4H, ^{3}J = 7.6 Hz, 2,4-Ar-*H*-3,5), 8.33 (s, 1H, 6-Ar-*H*-3), 8.18 (d, ^{3}J = 7.8 Hz, 1H, 6-Ar-*H*-5), 8.05 (d, ^{3}J = 8.0 Hz, 4H, 2,4-Ar'-*H*-3,5), 8.00 (d, ^{3}J = 8.2 Hz, 2H, 6-Ar'-*H*-3,5), 7.92 (d, ^{3}J = 7.6 Hz, 4H, 2,4-Ar-*H*-2,6), 7.86 (d, ^{3}J = 8.0 Hz, 4H, 2,4-Ar'-*H*-2,6), 7.74 (d, ^{3}J = 8.2 Hz, 2H, 6-Ar'-*H*-2,6), 7.50 (d, ^{3}J = 7.8 Hz, 1H, 6-Ar-*H*-6) ppm. ^{13}C NMR (125 MHz, DMSO-d_6): δ = 171.1 (s, triazine-*C*-6), 170.9 (s, triazine-*C*-2,4), 167.7 (s, 6-Ar'-*C*OOH), 167.5 (s, 2,4-Ar'-*C*OOH), 155.3 (s, 6-Ar-*C*-2), 143.6 (s, 2,4-Ar-*C*-1, 2,4-Ar'-*C*-1), 142.6 (s, 6-Ar'-*C*-1), 136.6 (s, 6-Ar-*C*-4), 135.5 (s, 2,4-Ar-*C*-4), 131.4 (s, 6-Ar-*C*-1), 131.2 (d, 6-Ar-*C*-6), 130.8 (s, 2,4-Ar'-*C*-4), 130.4 (d, 2,4-Ar'-*C*-3,5), 129.8 (d, 6-Ar'-*C*-2,6), 129.7 (d, 2,4-

Ar-*C*-3,5), 129.7 (s, 6-Ar'-*C*-4), 129.5 (d, 6-Ar'-*C*-3,5), 127.8 (d, 2,4-Ar-*C*-2,6), 127.5 (d, 2,4-Ar'-*C*-2,6), 120.5 (d, 6-Ar-*C*-5), 116.5 (d, 6-Ar-*C*-3) ppm. MS (MALDI, Cl-CCA): *m/z* = 686 $[M + H]^+$. IR (ATR): $\tilde{\nu}$ = 3050 (OH), 1681 (C=O), 1606, 1580, 1562, 1504 (arom. C=C, arom. C=N), 1418 (CH-Def.), 1360 (O-H-Def.), 1238, 1175, 1101 (C-O), 852, 813, 772 (1,4-disubst. aryl, 1,2,4-trisubst. aryl) cm^{-1}. Elemental analysis ($C_{42}H_{27}N_3O_7$) (685.18): calcd. C 73.57 H 3.97 N 6.13; ($C_{42}H_{27}N_3O_7 \cdot H_2O$) (703.20): calcd. C 71.69 H 4.15 N 5.97; found C 71.47 H 3.95 N 5.91.


12.99
10.18
8.70
8.69
8.33
8.18
8.17
8.05
8.04
8.01
7.99
7.93
7.91
7.86
7.85
7.75
7.74
7.51
7.50
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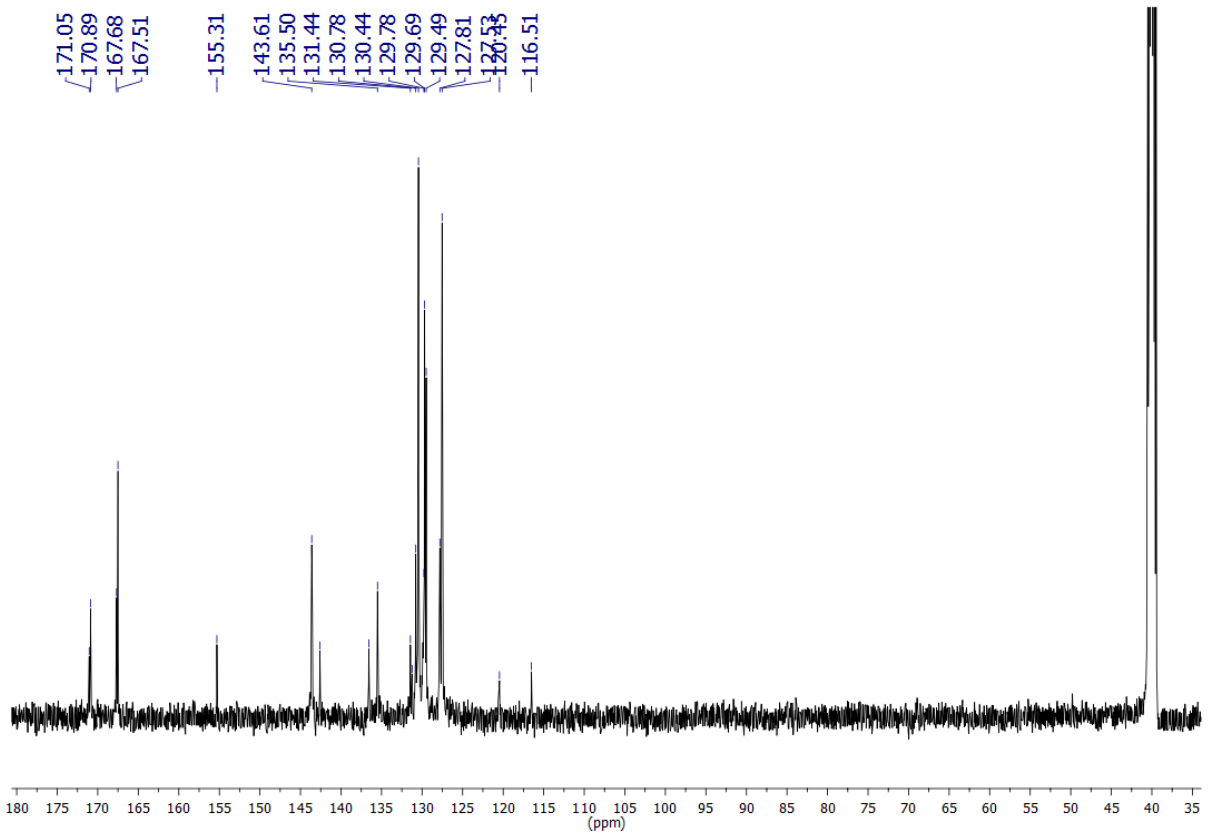
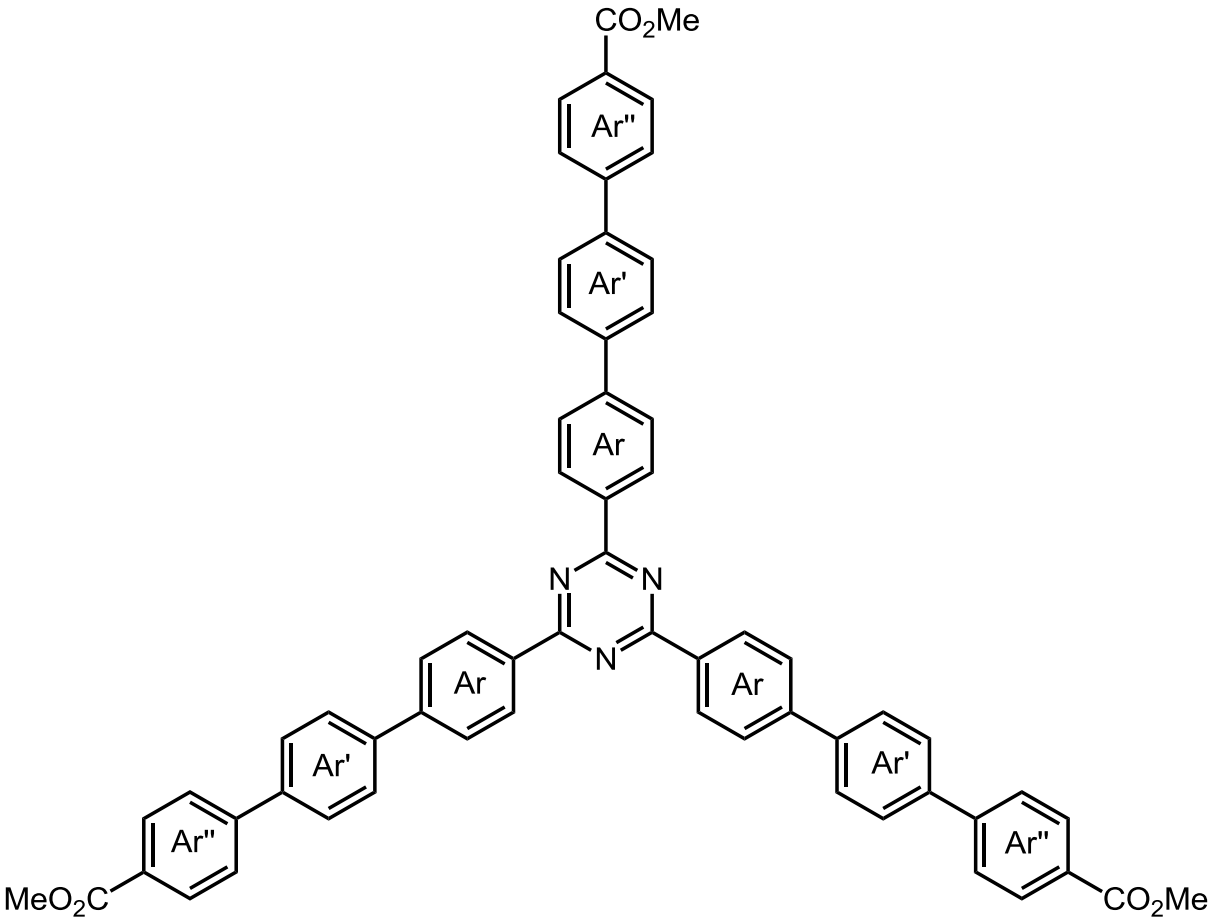
1.07
1.12
4.07
2.25
4.02
4.10
2.18
1.14
(ppm)

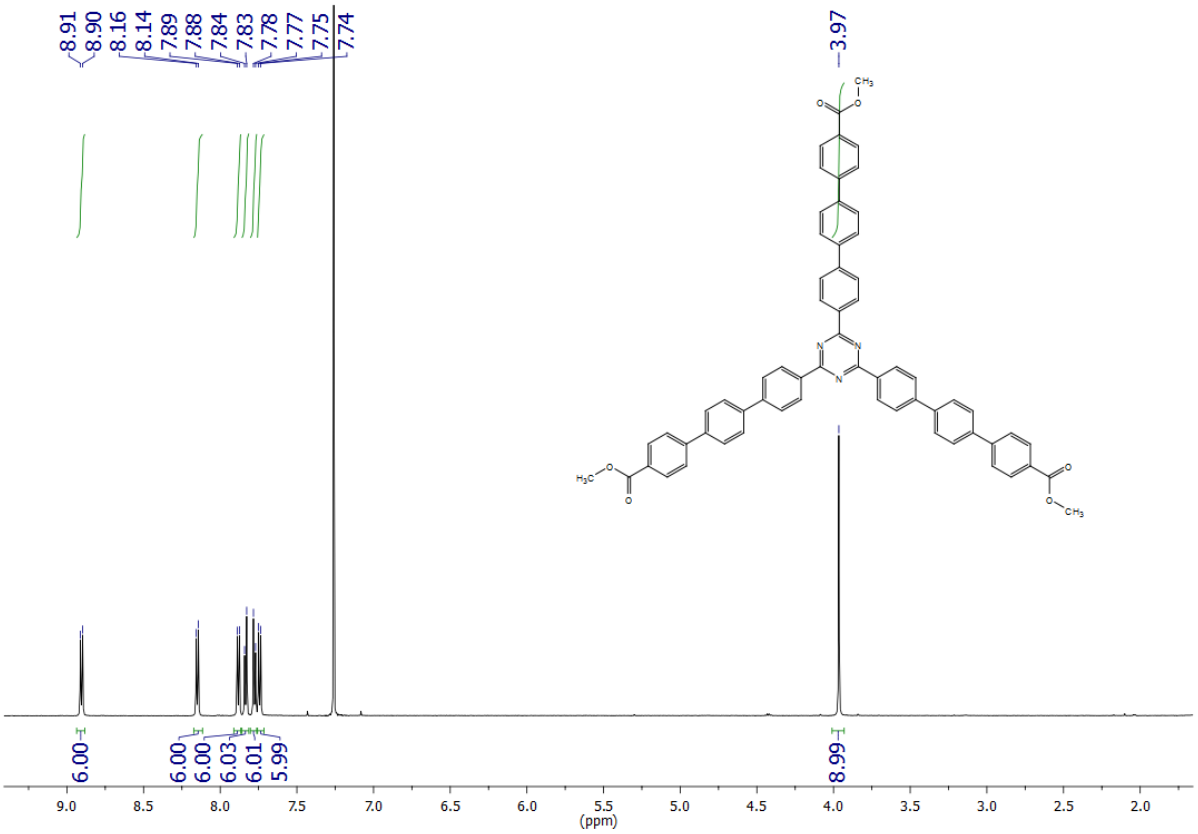

171.05
170.89
167.68
167.51
155.31
143.61
135.50
131.44
130.78
130.44
129.78
129.69
129.49
127.81
116.51
(ppm)

2,4,6-Tris[4''-(methoxycarbonyl)-1,1':4',1''-terphenyl-4-yl]-1,3,5-triazine (19a)



A suspension of 2,4,6-tris(4-bromophenyl)-1,3,5-triazine (**3a**, 413 mg, 758 µmol), methyl 4'-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-1,1'-biphenyl-4-carboxylate (**18**, 1.00 g, 2.96 mmol)**,** potassium phosphate (1.44 g, 6.82 mmol) and tetrakis(triphenylphosphine)palladium(0) (79 mg, 68 µmol) in a mixture of 1,4-dioxane (70 mL) and deionized water (15 mL) was stirred under nitrogen for 2 d at 120 °C. Deionized water (50 mL) was added to the reaction mixture. The precipitate was filtered off and washed with water. The crude product was recrystallized from chloroform. A colourless solid was obtained. Yield: 174 mg (185 µmol, 24%). M. p.: > 300 °C. ^{1}H NMR (600 MHz, $CDCl_3$,): δ = 8.90 (d, ^{3}J = 8.3 Hz, 6H, Ar-*H*-3,5), 8.15 (d, ^{3}J = 8.3 Hz, 6H, Ar''-*H*-3,5), 7.88 (d, ^{3}J = 8.3 Hz, 6H, Ar-*H*-2,6), 7.83 (d, ^{3}J = 8.3 Hz,

6H, Ar'-*H*-2,6), 7.78 (d, ^{3}J = 8.3 Hz, 6H, Ar'-*H*-3,5), 7.74 (d, ^{3}J = 8.3 Hz, 6H, Ar''-*H*-2,6), 3.97 (s, 9H, $CO_2C\mathit{H}_3$) ppm. ^{13}C NMR (150 MHz, $CDCl_3$): δ = 171.4 (s, triazine-*C*-2,4,6), 167.0 (s, $\mathit{C}O_2CH_3$), 145.0 (s, Ar''-*C*-1), 144.4 (s, Ar-*C*-1), 140.1 (s, Ar'-*C*-1), 139.6 (s, Ar'-*C*-4), 135.5 (s, Ar-*C*-4), 130.2 (d, Ar''-*C*-3,5), 129.6 (d, Ar-*C*-3,5), 129.1 (s, Ar''-*C*-4), 127.8 (d, Ar'-*C*-2,3,5,6), 127.3 (d, Ar-*C*-2,6), 127.0 (s, Ar''-*C*-2,6), 52.2 (q, $\mathit{C}H_3$) ppm. MS (MALDI, Cl-CCA): *m/z* = 940 $[M + H]^+$. IR (ATR): $\tilde{\nu}$ = 2950 (CH-val.), 1716 (C=O), 1606, 1581, 1552, 1505 (arom. C=C, arom. C=N), 1434 (CH-Def.), 1361 (C-N-val.), 1272, 1184, 1108 (C-O), 808, 773 (1,4-disubst. aryl) cm^{-1}. Elemental analysis ($C_{63}H_{45}N_3O_6$) (939.33): calcd. C 80.49 H 4.82 N 4.47; found C 80.15 H 4.74 N 4.27.


8.91
8.90
8.16
8.14
7.89
7.88
7.84
7.83
7.78
7.77
7.75
7.74
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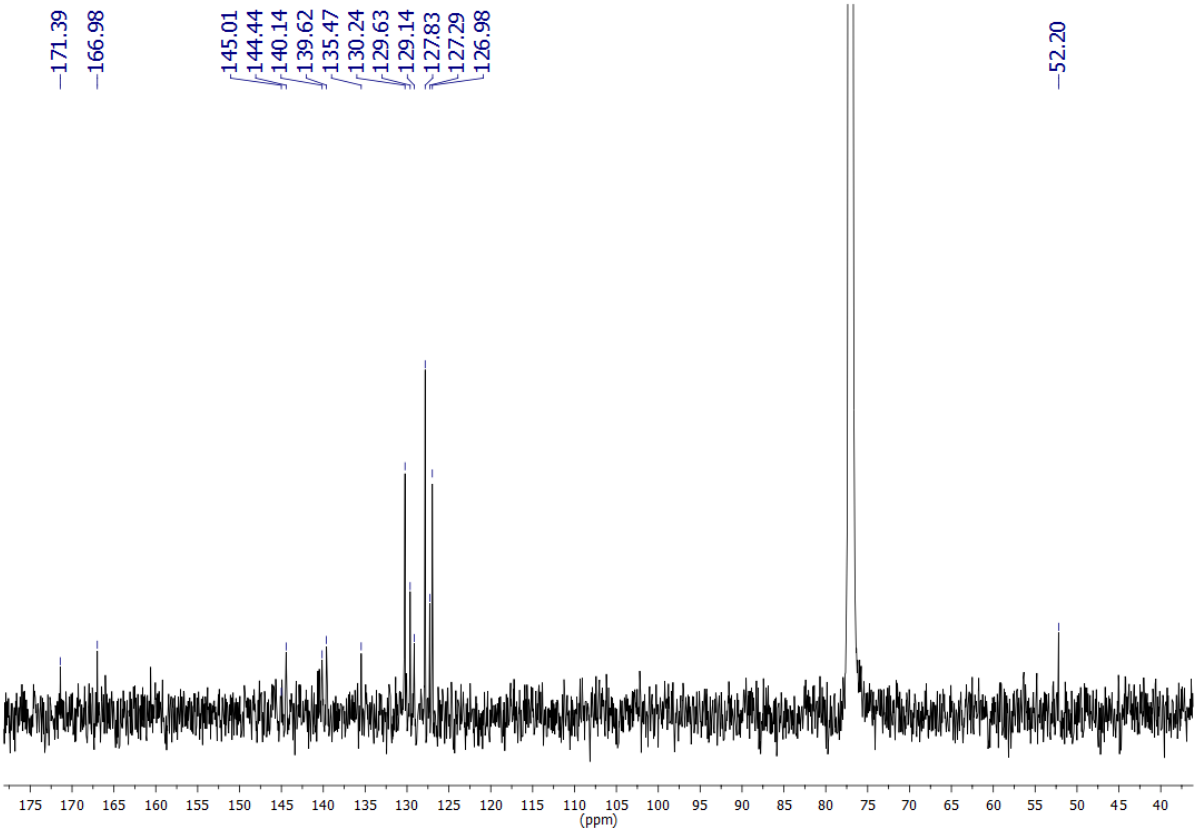
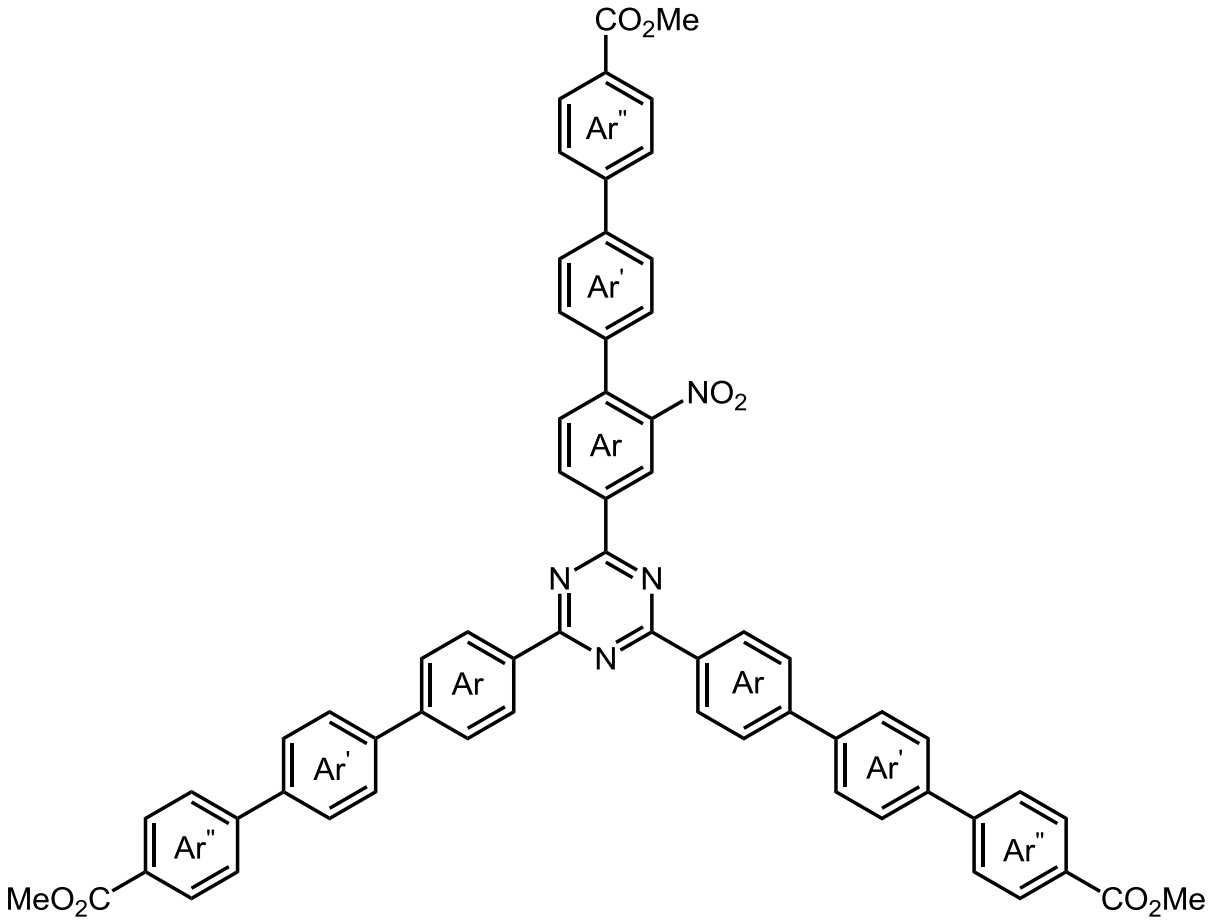
6.00
6.00
6.03
6.01
5.99
8.99
(ppm)

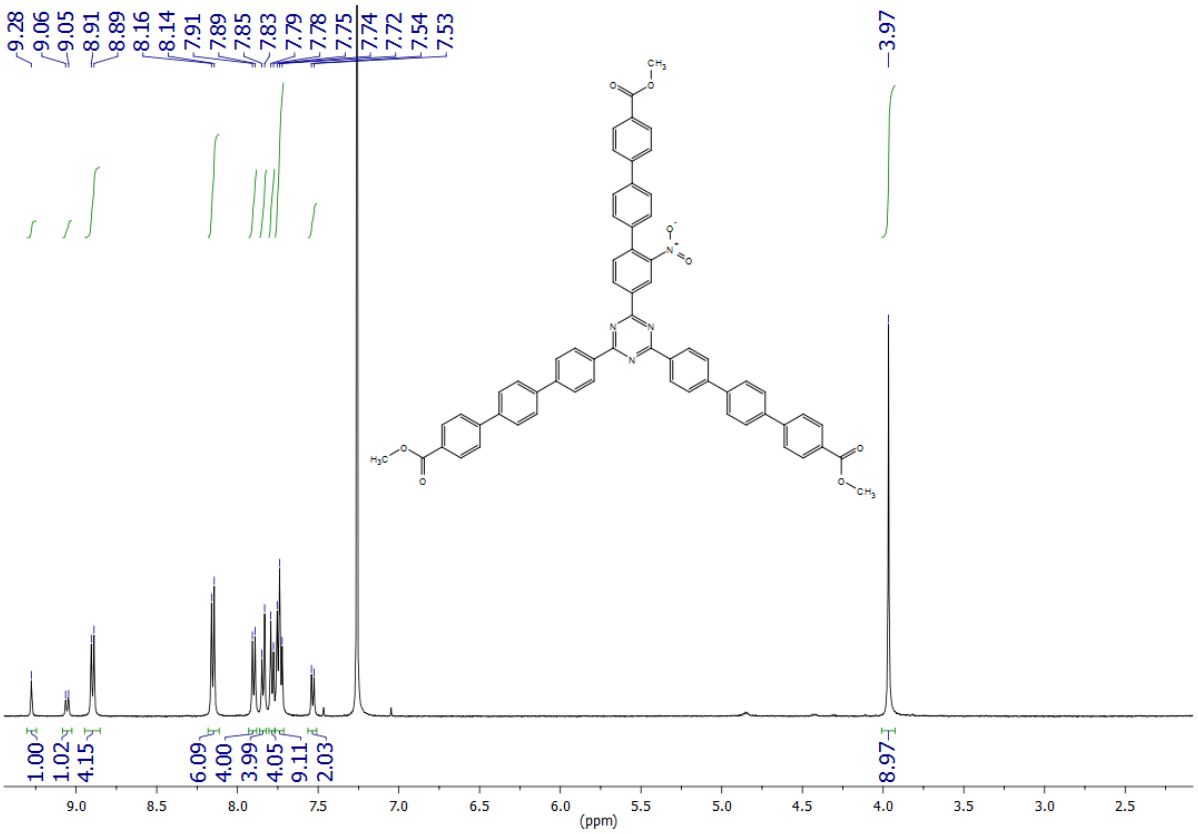

171.39
166.98
145.01
144.44
140.14
139.62
135.47
130.24
129.63
129.14
127.83
127.29
126.98
52.20
(ppm)

2-[4’’-(Methoxycarbonyl)-2-nitro-1,1’:4’,1’’-terphenyl-4-yl]-4,6-bis[4’’-(methoxycarbonyl)-1,1’:4’,1’’-terphenyl-4-yl]-1,3,5-triazine (19b)



A mixture of 2-(4-bromo-3-nitrophenyl)-4,6-bis(4-bromophenyl)-1,3,5-triazine (**3b**, 500 mg, 846 µmol), methyl 4’-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-1,1’-biphenyl-4-carboxylate (**18**, 1.12 g, 3.30 mmol), potassium phosphate (1.62 g, 7.61 mmol) and [1,1′-bis(diphenylphosphino)ferrocene]dichloropalladium(II) (62.0 mg, 84.6 µmol) in a mixture of 1,4-dioxane (100 mL) and deionized water (10 mL) was stirred under nitrogen for 3 d at 120 °C. The solvent was evaporated in vacuo, the residue was dissolved in chloroform (2 L) and washed with deionized water (3 x 250 mL) and brine (250 mL). The organic layer was evaporated in vacuo until 1 L was left. Charcoal (5 g) was added and the solution was heated to 80 °C. The hot suspension was filtered through celite and the colourless filtrate was evaporated in vacuo. After

the addition ethyl acetate (50 mL), the undissolved solid was filtrated and dried to obtain the slightly yellow product. Yield: 132 mg (134 µmol, 16%). M. p.: > 300 °C. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 9.28 (d, ^{4}J = 1.4 Hz, 1H, 2-Ar-*H*-3), 9.06 (dd, ^{3}J = 8.1 Hz, ^{4}J = 1.4 Hz, 1H, 2-Ar-*H*-5), 8.90 (d, ^{3}J = 8.5 Hz, 4H, 4,6-Ar-*H*-3,5), 8.15 (d, ^{3}J = 8.5 Hz, 6H, 2,4,6-Ar''-*H*-3,5), 7.90 (d, ^{3}J = 8.5 Hz, 4H, 4,6-Ar-*H*-2,6), 7.84 (d, ^{3}J = 8.5 Hz, 4H, 4,6-Ar'-*H*-2,6), 7.78 (d, ^{3}J = 8.5 Hz, 4H, 4,6-Ar'-*H*-3,5), 7.77 – 7.70 (m, 9H, 2,4,6-Ar''-*H*-2,6, 2-Ar-*H*-6, 2-Ar'-*H*-3,5), 7.53 (d, ^{3}J = 8.3 Hz, 2H, 2-Ar'-*H*-2,6), 3.97 (s, 9H, CO_2CH_3) ppm. ^{13}C NMR (125 MHz, $CDCl_3$): δ = 171.5 (s, triazine-*C*-4,6), 169.5 (s, triazine-*C*-2), 166.9 (s, CO_2CH_3), 149.9 (s, 2-Ar-*C*-2), 144.9 (s, 4,6-Ar-*C*-1), 144.8 (s, 2,4,6-Ar''-*C*-1), 140.5 (s, 2-Ar'-*C*-4), 139.8 (s, 4,6-Ar'-*C*-1), 139.7 (s, 4,6-Ar'-*C*-4), 139.1 (s, 2-Ar-*C*-1), 137.6 (s, 2-Ar-*C*-4), 136.5 (s, 2-Ar'-*C*-1), 134.9 (s, 4,6-Ar-*C*-4), 132.4 (d, 2-Ar-*C*-6), 132.0 (d, 2-Ar-*C*-5), 130.2 (d, 2,4,6-Ar''-*C*-3,5), 129.7 (d, 4,6-Ar-*C*-3,5), 129.1 (s, 2,4,6-Ar''-*C*-4), 128.4 (d, 2-Ar'-*C*-2,6), 127.9 (d, 4,6-Ar'-*C*-3,5), 127.8 (d, 4,6-Ar'-*C*-2,6), 127.4 (d, 4,6-Ar-*C*-2,6), 127.1 (d, 2-Ar'-*C*-3,5), 126.9 (d, 2,4,6-Ar''-*C*-2,6), 124.6 (d, 2-Ar-*C*-3), 52.1 (q, CO_2CH_3) ppm. MS (MALDI, Cl-CCA): *m/z* = 986 $[M + H]^+$. IR (ATR): $\tilde{\nu}$ = 2952 (CH-val.), 1718 (C=O), 1606, 1572 (arom. C=C, arom. C=N), 1508 (asymm. N=O), 1400, 1433 (CH-def.), 1357 (symm. N=O), 1274, 1184, 1108 (C-O), 810, 771 (1,4-disubst. aryl, 1,2,4-trisubst. aryl) cm^{-1}. Elemental analysis ($C_{63}H_{44}N_4O_8$) (985.05): calcd. C 76.82 H 4.50 N 5.69; found C 77.04 H 4.83 N 5.38.


9.28
9.06
9.05
8.91
8.89
8.16
8.14
7.91
7.89
7.85
7.83
7.79
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7.75
7.74
7.72
7.54
7.53
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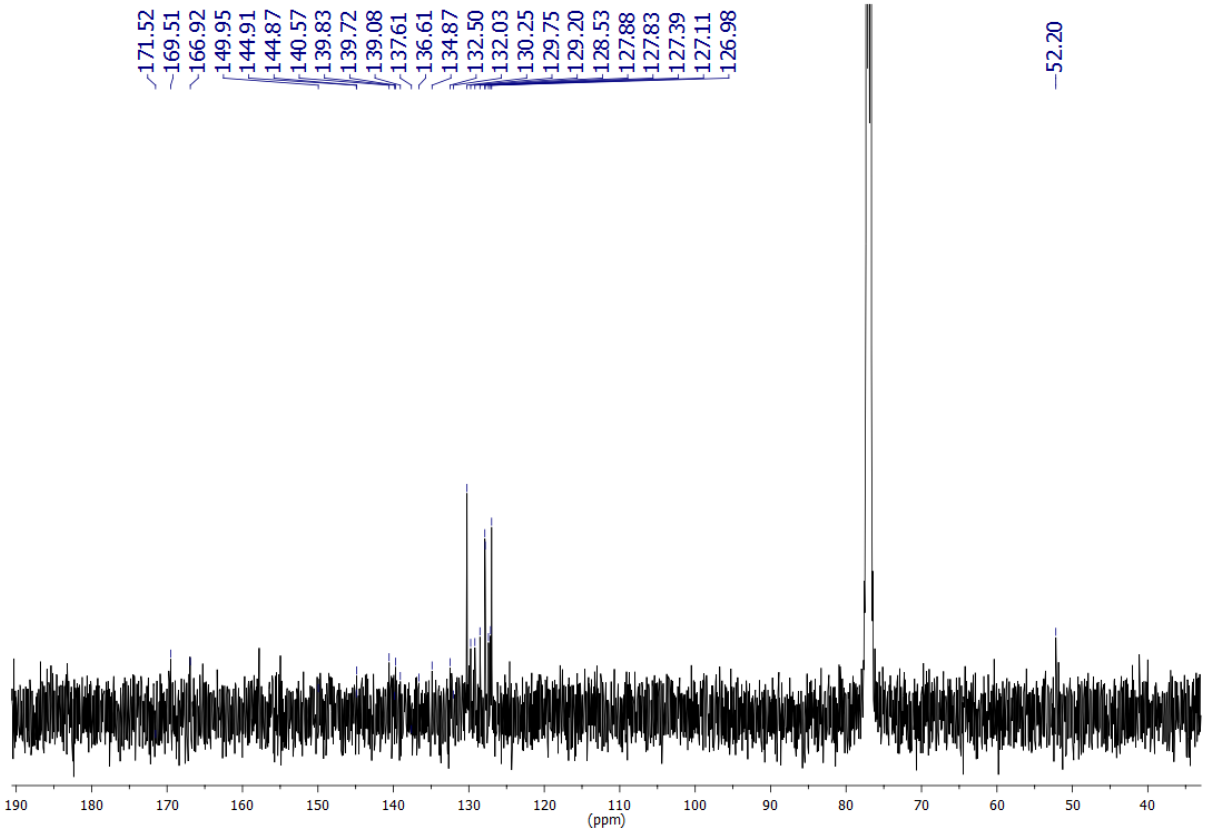
3.99
4.05
9.11
2.03
8.97
(ppm)


171.52
169.51
166.92
149.95
144.91
144.87
140.57
139.83
139.72
139.08
137.61
136.61
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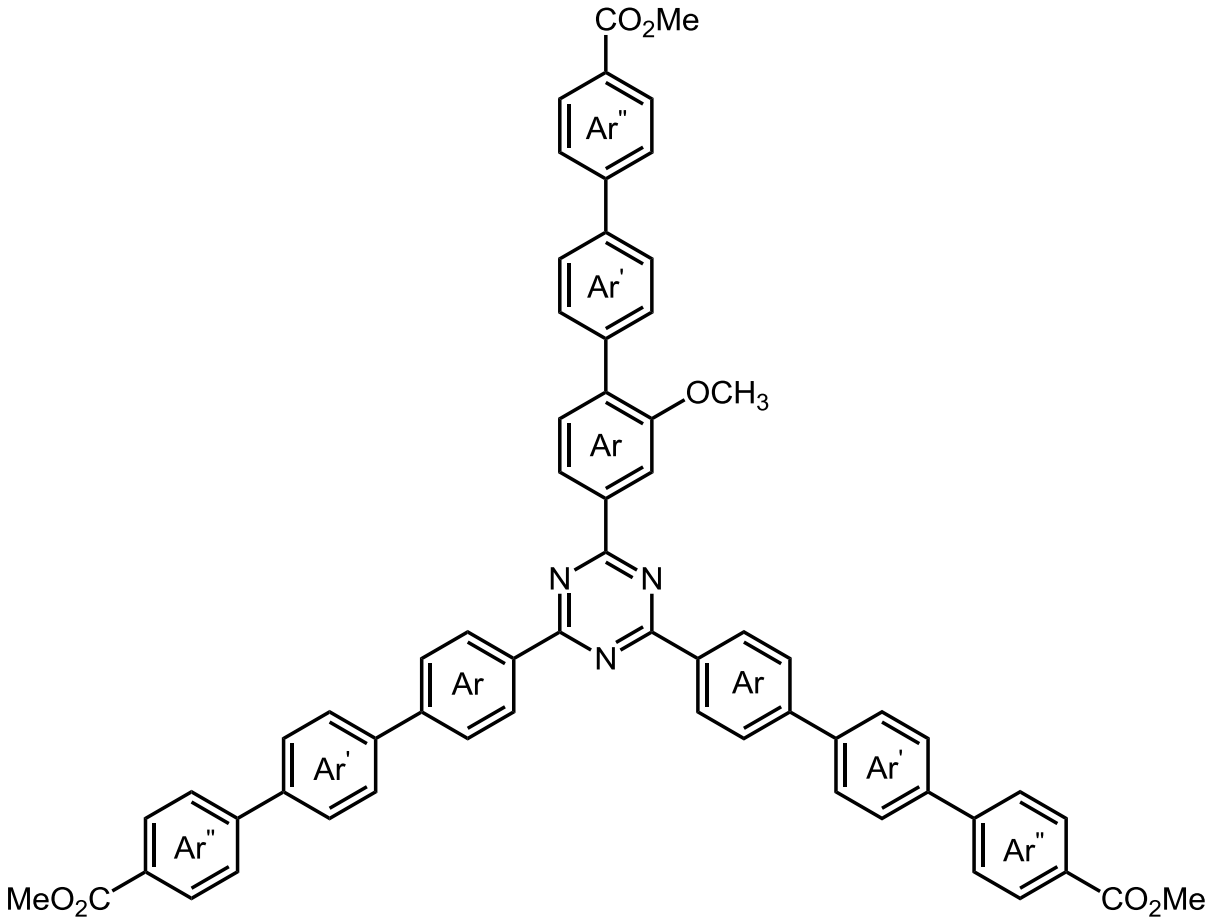
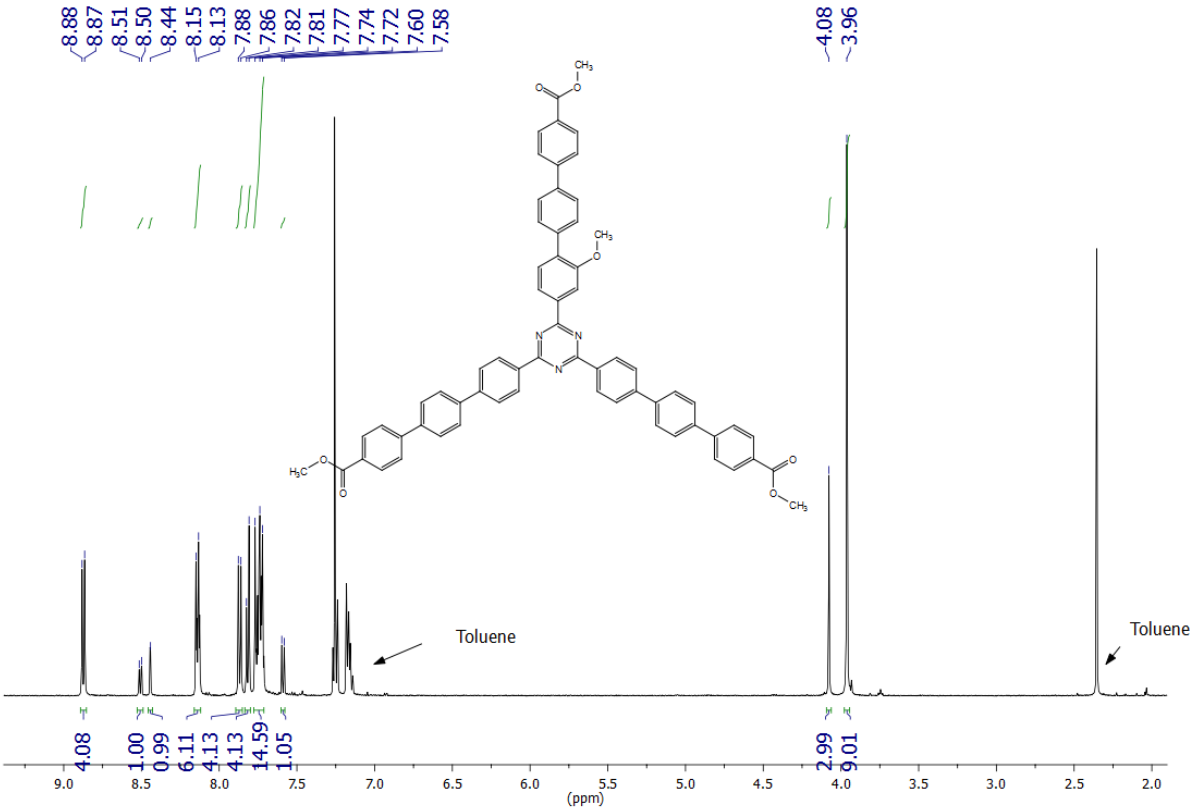
132.50
132.03
130.25
129.75
129.20
128.53
127.88
127.83
127.39
127.11
126.98
52.20
(ppm)

2,4-Bis[4''-(methoxycarbonyl)-1,1':4',1''-terphenyl-4-yl]-6-[2-methoxy-4''-(methoxycarbonyl)-1,1':4',1''-terphenyl-4-yl]-1,3,5-triazine (19c)



A mixture of 2-(4-bromo-3-methoxyphenyl)-4,6-bis(4-bromophenyl)-1,3,5-triazine (**3c**, 102 mg, 180 µmol), methyl 4'-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-1,1'-biphenyl-4-carboxylate (**18**, 269 mg, 796 µmol), potassium phosphate (259 mg, 1.22 mmol) and tetrakis(triphenylphosphine)palladium(0) (19.4 mg, 17.0 µmol) in a mixture of 1,4-dioxane (15 mL) and deionized water (1 mL) was stirred under nitrogen for 3 d at 100 °C. The solvent was evaporated in vacuo, the residue was dissolved in chloroform (50 mL) and washed with deionized water (3 x 25 mL) and brine (25 mL). The organic layer was dried with magnesium sulfate and the solvent was evaporated in vacuo. The crude product was recrystallized from a mixture of toluene and *n*-heptane. A colourless solid was obtained. Yield: 140 mg (144 µmol,

81%). M. p.: > 300 °C. ^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.87 (d, ^{3}J = 8.4 Hz, 4H, 2,4-Ar-*H*-3,5), 8.51 (dd, ^{3}J = 7.9 Hz, ^{4}J = 1.5 Hz, 1H, 6-Ar-*H*-5), 8.44 (d, ^{4}J = 1.5 Hz, 1H, *6*-Ar-*H*-3), 8.14 (d, ^{3}J = 8.4 Hz, 4H, 2,4-Ar''-*H*-3,5), 8.13 (d, ^{3}J = 8.4 Hz, 2H, 6-Ar''-*H*-3,5), 7.87 (d, ^{3}J = 8.4 Hz, 4H, 2,4-Ar-*H*-2,6), 7.82 (d, ^{3}J = 8.4 Hz, 4H, 2,4-Ar'-*H*-2,6), 7.78 – 7.71 (m, 14H, 2,4,6-Ar'-*H*-3,5, 6-Ar'-*H*-2,6, 2,4,6-Ar''-*H*-2,6), 7.59 (d, ^{3}J = 7.9 Hz, 1H, 6-Ar-*H*-6), 4.08 (s, 3H, OCH_3), 3.962 (s, 6H, 2,4-Ar''-$CO_2C H_3$), 3.960 (s, 3H, 6-Ar''-$CO_2C H_3$) ppm. ^{13}C NMR (125 MHz, $CDCl_3$): δ = 171.32 (s, triazine-*C*-2,4), 171.29 (s, triazine-*C*-6), 167.0 (s, 6-Ar''-CO_2CH_3), 166.9 (s, 2,4-Ar''-CO_2CH_3), 156.9 (s, 6-Ar-*C*-2), 145.3 (s, 6-Ar''-*C*-1), 144.9 (s, 2,4-Ar''-*C*-1), 144.4 (s, 2,4-Ar''-*C*-4), 140.1 (s, 2,4-Ar-*C*-1), 139.6 (s, 2,4-Ar'-*C*-4), 139.0 (s, 2,4-Ar'-*C*-1), 136.9 (s, 6-Ar'-*C*-1), 135.8 (s, 6-Ar-*C*-4), 135.3 (s, 2,4-Ar-*C*-4), 134.2 (s, 6-Ar-*C*-1), 130.9 (d, 6-Ar-*C*-6), 130.21 (d, 2,4-Ar''-*C*-3,5), 130.16 (d, 6-Ar''-*C*-3,5), 130.1 (d, 2,4-Ar-*C*-3,5), 129.0 (s, 6-Ar''-*C*-4), 128.2 (d, 6-Ar'-*C*-2,6), 127.8 (d, 2,4-Ar'-*C*-3,5), 127.76 (d, 2,4-Ar'-*C*-2,6), 127.2 (d, 2,4-Ar-*C*-2,6), 127.0 (d, 6-Ar'-*C*-3,5), 126.9 (d, 2,4,6-Ar''-*C*-2,6, 6-Ar'-*C*-4), 122.0 (d, 6-Ar-*C*-5), 111.3 (d, 6-Ar-*C*-3), 55.9 (q, O*C*H_3), 52.2 (q, 2,4-Ar''-CO_2CH_3), 52.1 (q, 6-Ar''-CO_2CH_3) ppm. MS (MALDI, Cl-CCA): *m/z* = 971 $[M + H]^+$. IR (ATR): $\tilde{\nu}$ = 3030, 3001 (aryl-H), 2949, 2842 (CH-val.), 1715 (C=O), 1605, 1582, 1505, 1403 (arom. C=C, arom. C=N), 1434 (CH-Def.), 1360 (C-N-val.), 1272 (Aryl-OCH_3), 807 (1,4-disubst. aryl, 1,2,4-trisubst. aryl) cm^{-1}. Elemental analysis ($C_{64}H_{47}N_3O_7$) (970.07): calcd. C 79.24 H 4.88 N 4.33; found C 79.10 H 5.11 N 4.02.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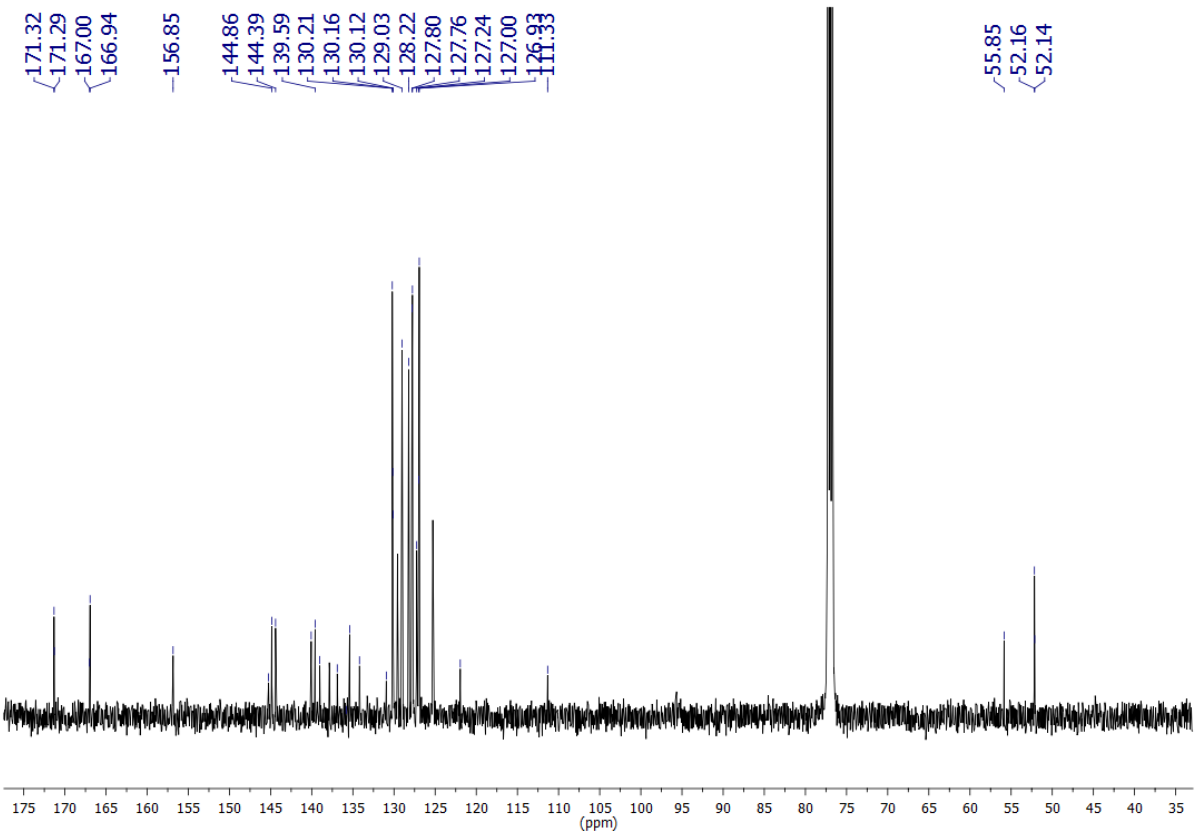
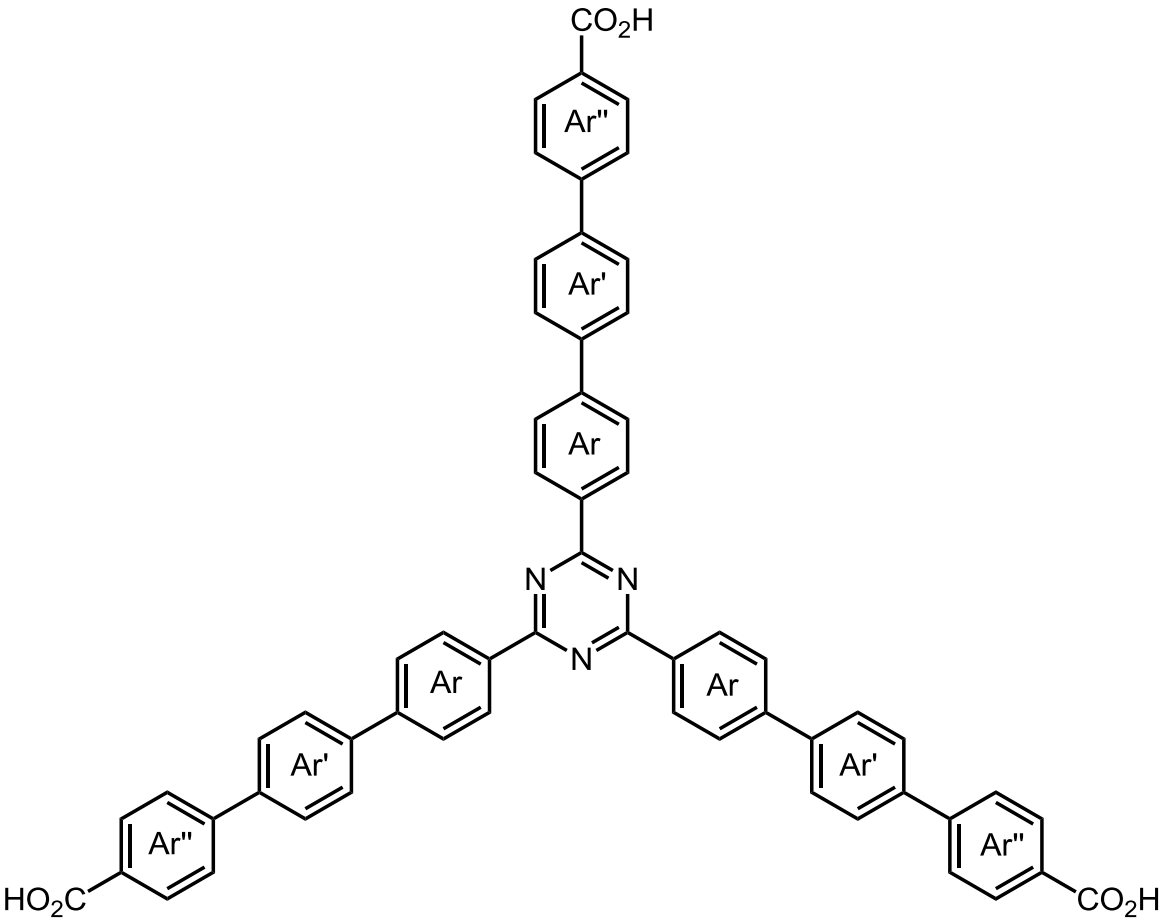
8.87
8.51
8.50
8.44
8.15
8.13
7.88
7.86
7.82
7.81
7.77
7.74
7.72
7.60
7.58
4.08
3.96
Toluene
Toluene
4.08
1.00
0.99
6.11
4.13
4.13
14.59
1.05
2.99
9.01
(ppm)

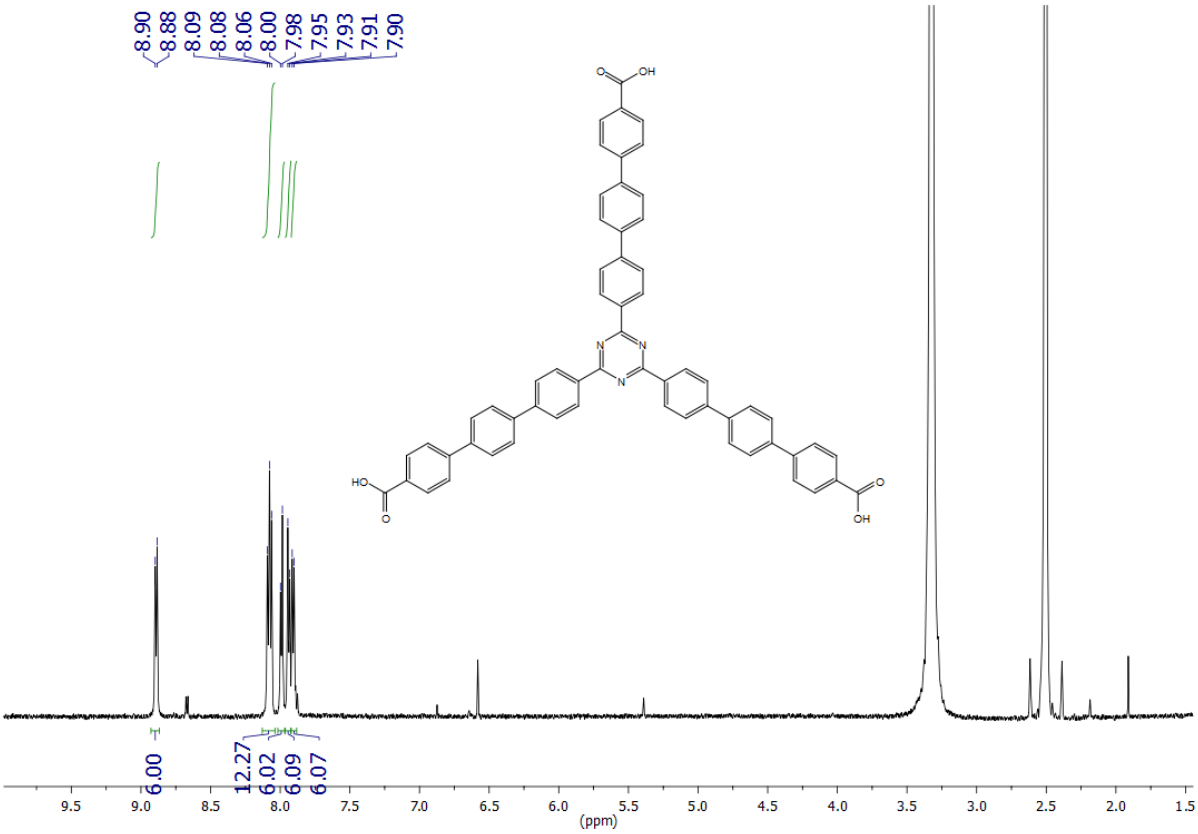

171.32
171.29
167.00
166.94
156.85
144.86
144.39
139.59
130.21
130.16
130.12
129.03
128.22
127.80
127.76
127.24
127.00
126.93
111.33
55.85
52.16
52.14
(ppm)

2,4,6-Tris(4''-carboxy-1,1':4',1''-terphenyl-4-yl)-1,3,5-triazine (20a)



Lithium hydroxide monohydrate (733 mg, 17.5 mmol) was added to 2,4,6-tris[4''-(methoxycarbonyl)-1,1':4',1''-terphenyl-4-yl]-1,3,5-triazine (**19a**, 587 mg, 624 µmol) in a mixture of tetrahydrofuran (100 mL) and deionized water (10 mL). After stirring for 24 h at 120 °C, the solvent was evaporated in vacuo and enough sodium hydroxide (2 M) was added to the residue to dissolved it. Then it was acidified with conc. hydrochloric acid. The precipitate was filtered off and washed with deionized water and chloroform. A white solid was obtained. Yield: 555 mg (618 µmol, 99%). M. p.: > 300 °C. ^{1}H NMR (600 MHz, DMSO-d_6): δ = 8.89 (d, 6H, ^{3}J = 8.1 Hz, Ar-*H*-3,5), 8.10 – 8.05 (m, 12H, Ar''-*H*-3,5, Ar-*H*-2,6), 7.99 (d, ^{3}J = 8.3 Hz, 6H, Ar'-*H*-2,6), 7.94 (d, ^{3}J = 8.3 Hz, 6H, Ar'-*H*-3,5), 7.91 (d, ^{3}J = 7.9 Hz, 6H, Ar''-*H*-2,6) ppm. ^{13}C NMR (150 MHz, DMSO-d_6): δ = 171.2 (s, triazine-*C*-2,4,6), 167.6 (s, *C*OOH), 144.1 (s, Ar-*C*-1),

143.8 (s, Ar''-*C*-1), 139.3 (s, 6-Ar'-*C*-4), 139.2 (s, Ar'-*C*-1), 135.0 (s, Ar-*C*-4), 130.6 (s, Ar''-*C*-4),130.5 (d, Ar''-*C*-3,5), 129.9 (d, Ar-*C*-3,5), 128.1 (d, Ar'-*C*-2,3,5,6), 127.6 (d, Ar-*C*-2,6), 127.2 (s, Ar''-*C*-2,6) ppm. MS (MALDI, Cl-CCA): *m/z* = 898 $[M + H]^+$. IR (ATR): $\tilde{\nu}$ = 1683 (C=O), 1605, 1580, 1504 (arom. C=C, arom. C=N), 1404 (CH-Def.), 1359 (C-N-val.), 1283, 1182 (C-O), 808, 773 (1,4-disubst. aryl) cm^{-1}. Elemental analysis ($C_{60}H_{39}N_3O_6$) (897.28): calcd. C 80.25 H 4.38 N 4.68; ($C_{60}H_{39}N_3O_6 \cdot 0.66$ H_2O) (909.29): calcd. C 79.19 H 4.47 N 4.62; found C 78.97 H 4.57 N 4.66.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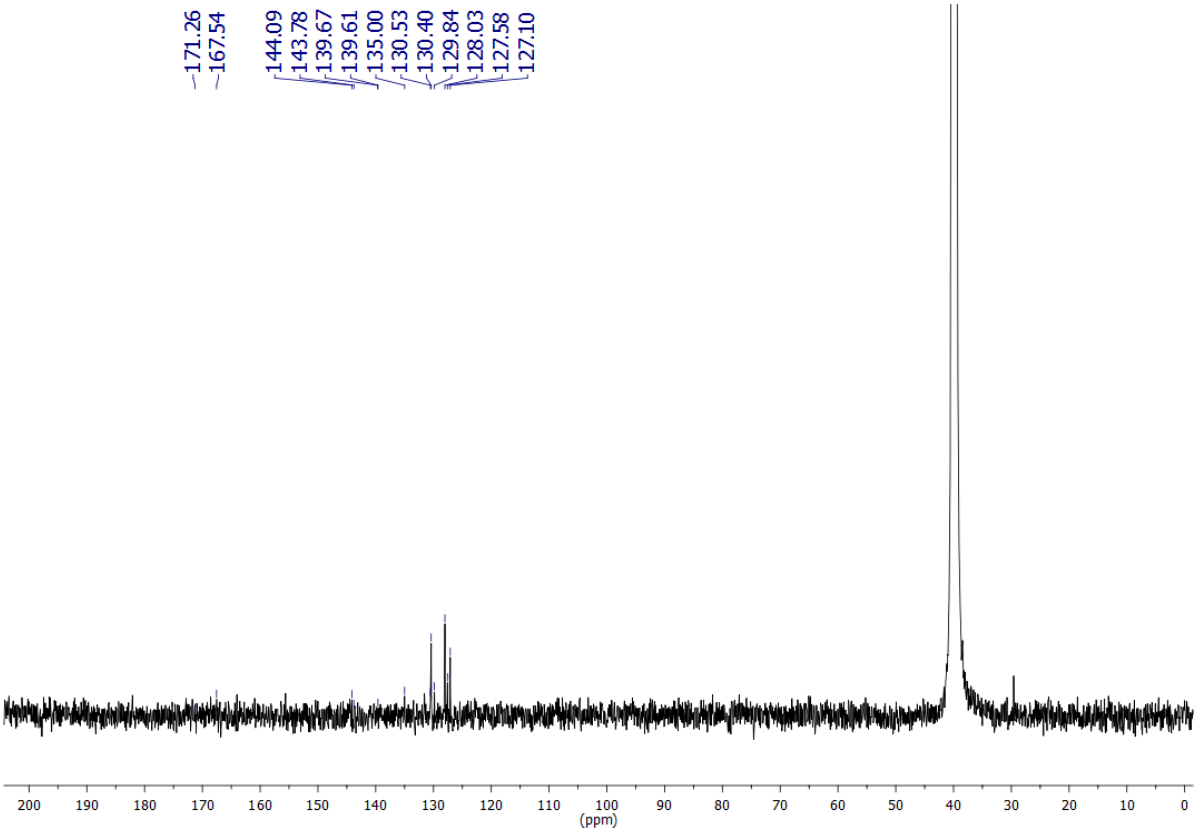
8.09
8.08
8.06
8.00
7.98
7.95
7.93
7.91
7.90
6.00
12.27
6.02
6.09
6.07
(ppm)


171.26
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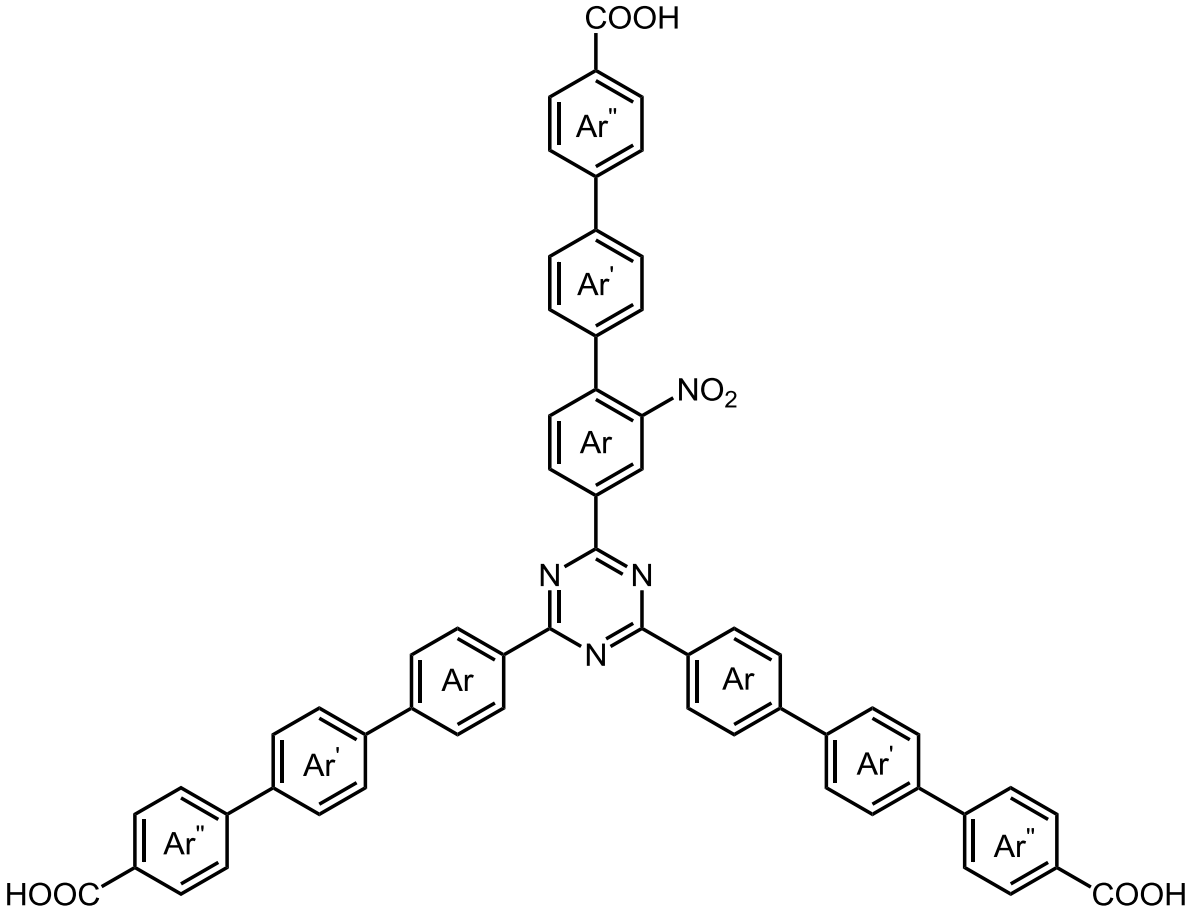
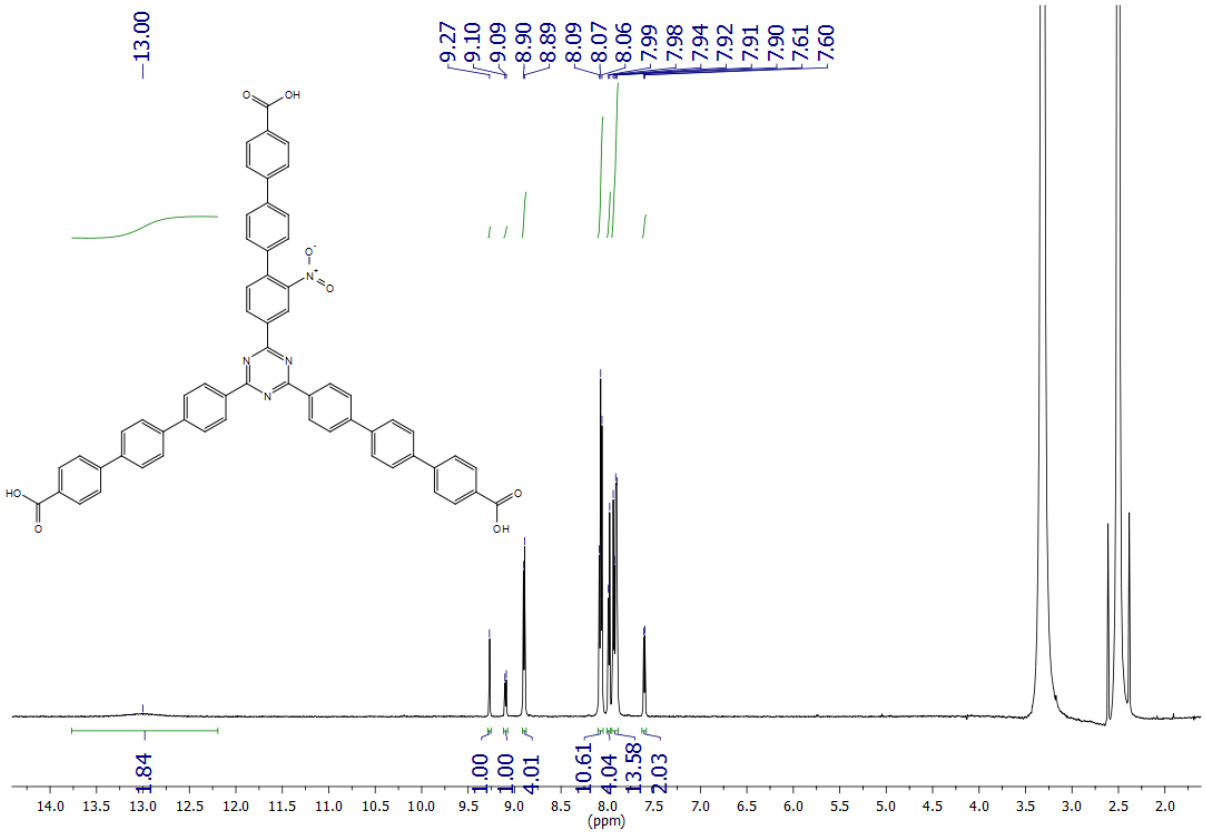
144.09
143.78
139.67
139.61
135.00
130.53
130.40
129.84
128.03
127.58
127.10
(ppm)

2-(4’’-Carboxy-2-nitro-1,1’,4’,1’’-terphenyl-4-yl)-4,6-bis(4’’-carboxy-1,1’:4’,1’’-terphenyl-4-yl)-1,3,5-triazine (20b)



To a mixture of 1,4-dioxane (10 mL) and sodium hydroxide (6 M, 50 mL) was added 2-[4’’-(methoxycarbonyl)-2-nitro-1,1’:4’,1’’-terphenyl-4-yl]-4,6-bis[4’’-(methoxycarbonyl)-1,1’:4’,1’’-terphenyl-4-yl]-1,3,5-triazine (**19b**, 93 mg, 94 µmol). After stirring for 24 h at 120 °C the solvent was evaporated in vacuo. The residue was dissolved in 10 mL water and acidified with conc. hydrochloric acid. The precipitate was filtered off and washed with deionized water. A yellow solid was obtained. Yield: 70 mg (74 µmol, 79%). M. p.: > 300 °C. ^{1}H NMR (600 MHz, DMSO-d_6): δ = 13.0 (br.s, 3H, COO*H*), 9.27 (d, ^{4}J = 1.3 Hz, 1H, 2-Ar-*H*-2), 9.10 (d, ^{3}J = 8.0 Hz, 1H, 2-Ar-*H*-5), 8.90 (d, ^{3}J = 8.3 Hz, 4H, 4,6-Ar-*H*-3,5), 8.12 – 8.04 (m, 10H, 2,4,6-Ar’’-*H*-3,5, 4,6-Ar-*H*-2,6), 7.98 (d, ^{3}J = 8.3 Hz, 4H, 4,6-Ar’-*H*-2,6), 7.96 –

7.88 (m, 13H, 4,6-Ar'-*H*-3,5, 2,4,6-Ar''-*H*-2,6, 2-Ar-*H*-6, 2-Ar'-*H*-3,5), 7.60 (d, ^{3}J = 8.1 Hz, 2H, 2-Ar'-*H*-2,6) ppm. ^{13}C NMR (150 MHz, DMSO-d_6): δ = 170.9 (s, triazine-*C*-4,6), 169.0 (s, triazine-*C*-2), 167.2 (s, *C*O_2H), 149.5 (s, 2-Ar-*C*-2), 143.9 (s, 4,6-Ar-*C*-1), 143.3 (s, 2,4,6-Ar''-*C*-1), 139.3 (s, 2-Ar'-*C*-4), 138.8 (s, 4,6-Ar'-*C*-1), 138.7 (s, 4,6-Ar'-*C*-4), 138.0 (s, 2-Ar-*C*-1), 137.5 (s, 2-Ar-*C*-4), 136.3 (s, 2-Ar'-*C*-1), 134.1 (s, 4,6-Ar-*C*-4), 133.7 (d, 2-Ar-*C*-6), 132.6 (d, 2-Ar-*C*-5), 130.4 (d, 2,4,6-Ar''-*C*-3,5), 130.0 (d, 4,6-Ar-*C*-3,5), 129.8 (s, 2,4,6-Ar''-*C*-4), 129.0 (d, 2-Ar'-*C*-2,6), 128.0 (d, 4,6-Ar'-*C*-2,6), 127.9 (d, 4,6-Ar'-*C*-3,5), 127.6 (d, 4,6-Ar-*C*-2,6), 127.3 (d, 2-Ar'-*C*-3,5), 127.3 (d, 2,4,6-Ar''-*C*-2,6), 124.4 (d, 2-Ar-*C*-3) ppm. MS (MALDI, Cl-CCA): *m/z* = 944 $[M + H]^+$. IR (ATR): $\tilde{\nu}$ = 3031 (aryl-H), 1684 (C=O), 1605, 1571 (arom. C=C, arom. C=N), 1506 (asymm. N=O), 1424, 1401 (CH-Def.), 1356 (symm. N=O), 1272, 1182, 1110 (C-O), 811, 773 (1,4-disubst. aryl, 1,2,4-trisubst. aryl) cm^{-1}. Elemental analysis ($C_{60}H_{38}N_4O_8$) (942.97): calcd. C 76.42 H 4.06 N 5.94; ($C_{60}H_{38}N_4O_8 \cdot 0.5\ H_2O$) (951.96): calcd. C 75.70 H 4.13 N 5.89; found C 75.43 H 4.06 N 5.87.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7.98
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7.91
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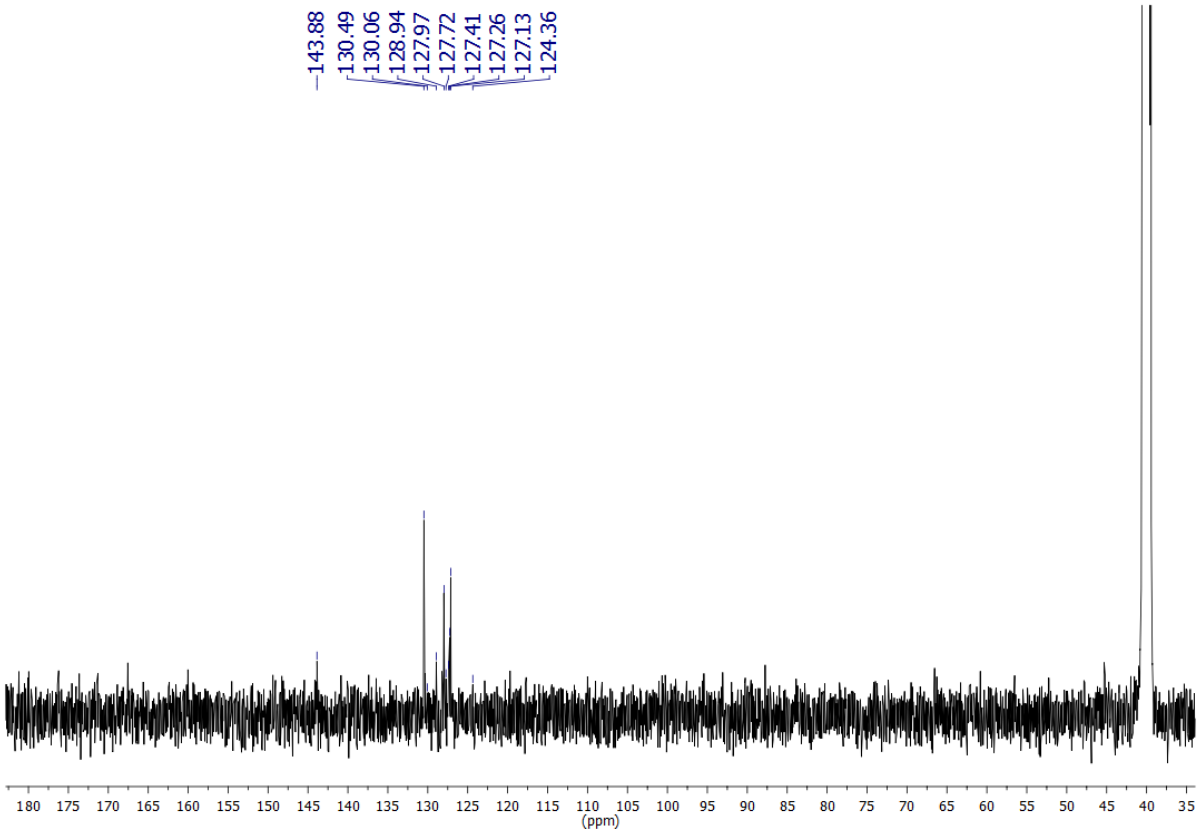
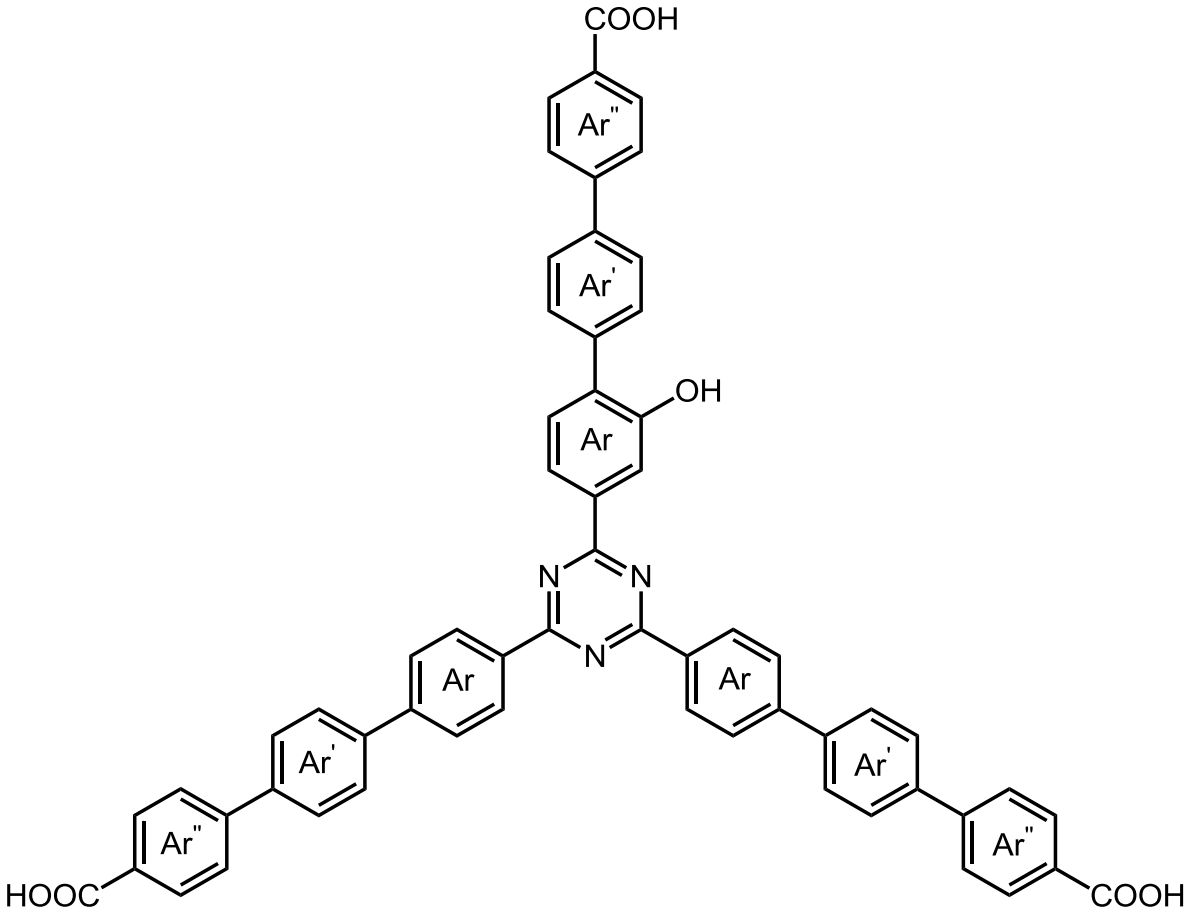
4.01
10.61
4.04
13.58
2.03
(ppm)

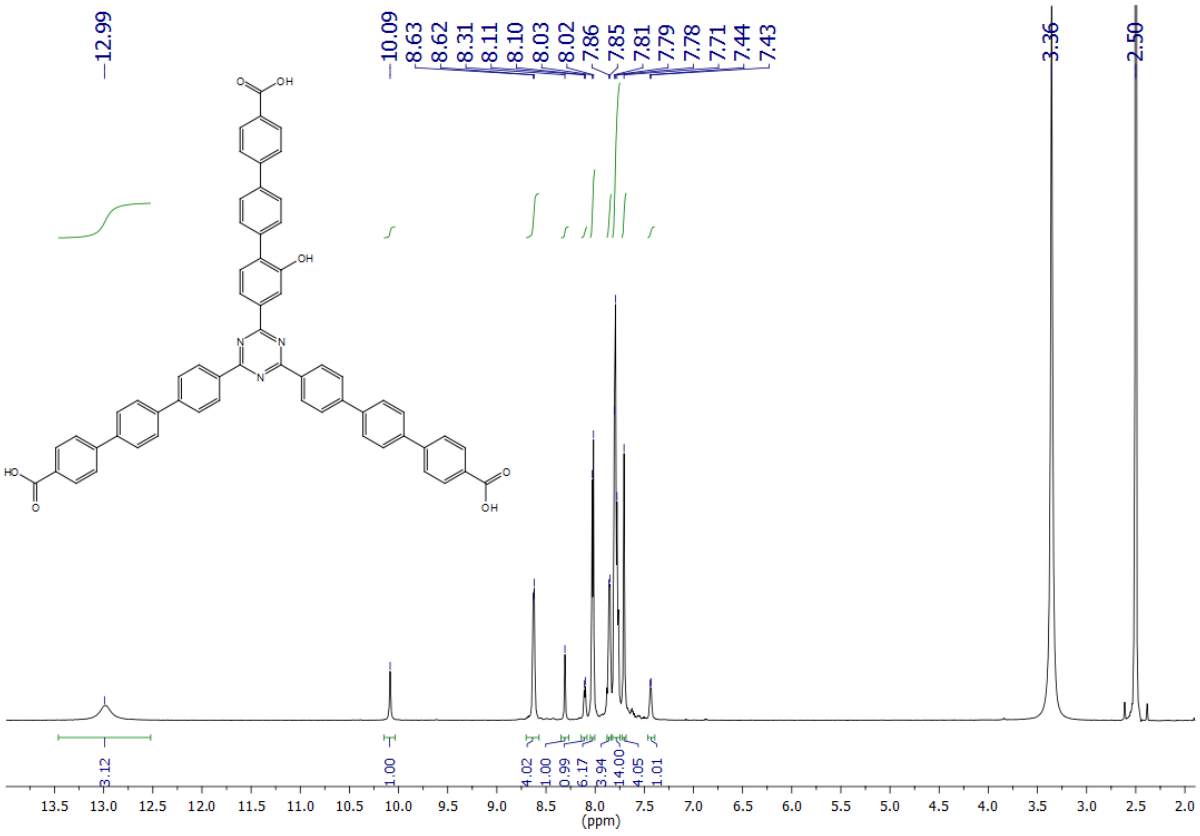

143.88
130.49
130.06
128.94
127.97
127.72
127.41
127.26
127.13
124.36
180 175 170 165 160 155 150 145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35
(ppm)

2-(4’’-Carboxy-2-hydroxy-1,1’:4’,1’’-terphenyl-4-yl)-4,6-bis(4’’-carboxy-1,1’:4’,1’’-terphenyl-4-yl)-1,3,5-triazine (20e)



2,4-Bis[4’’-(methoxycarbonyl)-(1,1’:4’,1’’-terphenyl)-4-yl]-6-[2-methoxy-4’’-(methoxycarbonyl)-(1,1’:4’,1’’-terphenyl)-4-yl]-1,3,5-triazine (**19c**, 109 mg, 113 µmol) and pyridinium hydrochloride (1.80 g, 15.6 mmol) were mixed and stirred for 12 h at 200 °C. After cooling to room temp., sodium hydroxide (2 M) was added to the solution until pH 14 was reached. The reaction mixture was stirred for 12 h at 120 °C before it was acidified with conc. hydrochloric acid and the resulting precipitate was filtered off. Washing with water and drying gave a dark red solid. Yield: 64 mg (70.0 µmol, 62%). M. p.: > 300 °C. ^{1}H NMR (600 MHz, DMSO-d_6): δ = 12.99 (br.s, 3H, COO*H*), 10.09 (s, 1H, O*H*), 8.63 (d, ^{3}J = 7.7 Hz, 4H, 4,6-Ar-*H*-3,5), 8.31 (s, 1H, *2*-Ar-

H-3), 8.11 (d, ^{3}J = 7.6 Hz, 1H, 2-Ar-*H*-5), 8.06 - 7.99 (m, 6H, 4,6-Ar''-*H*-3,5, 2-Ar''-*H*-3,5), 7.86 (d, ^{3}J = 7.7 Hz, 4H, 4,6-Ar-*H*-2,6), 7.82 - 7.75 (m, 14H, 2-Ar''-*H*-2,6, 4,6-Ar'-2,3,5,6, 4,6-Ar''-2,6), 7.74 – 7.68 (m, 4H, 2-Ar'-*H*-2,3,5,6), 7.44 (d, ^{3}J = 7.6 Hz, 1H, 2-Ar-*H*-6) ppm. ^{13}C NMR (150 MHz, DMSO-d_6): δ = 170.5 (s, triazine-*C*-2), 170.4 (s, triazine-*C*-4,6), 167.2 (s, 2-Ar''-*C*OOH), 167.1 (s, 4,6-Ar''-*C*OOH), 154.8 (s, 2-Ar-*C*-2), 143.9 (s, 2-Ar''-*C*-1), 143.4 (s, 4,6-Ar-*C*-1), 143.3 (s, 4,6-Ar''-*C*-1), 138.8 (s, 4,6-Ar'-*C*-1), 138.6 (s, 4,6-Ar'-*C*-4), 137.6 (s, 2-Ar'-*C*-4), 137.5 (s, 2-Ar'-*C*-1), 135.7 (s, 2-Ar-*C*-4), 134.6 (s, 4,6-Ar-*C*-4), 131.4 (s, 2-Ar''-*C*-4), 131.2 (s, 2-Ar-*C*-1), 130.4 (d, 2-Ar-*C*-6), 130.0 (d, 2-Ar''-*C*-3,5, 4,6-Ar''-*C*-3,5), 129.8 (d, 2-Ar'-*C*-2,6), 129.6 (s, 4,6-Ar''-*C*-4), 129.1 (d, 4,6-Ar-*C*-3,5), 127.5 (d, 2-Ar''-*C*-2,6, 4,6-Ar''-*C*-2,6), 126.8 (d, 4,6-Ar-*C*-2,6), 126.6 (d, 4,6-Ar'-*C*-2,3,5,6), 126.4 (d, 2-Ar'-*C*-3,5), 120.0 (d, 2-Ar-*C*-5), 115.9 (d, 2-Ar-*C*-3) ppm. MS (MALDI, Cl-CCA): *m/z* = 914 $[M + H]^+$. IR (ATR): $\tilde{\nu}$ = 2925 (O-H), 1682 (C=O), 1605, 1572, 1504 (arom. C=C, arom. C=N), 1407 (CH-Def.), 1361 (O-H-Def.), 1251, 1180, 1100 (C-O), 808, 773 (1,4-disubst. aryl, 1,2,4-trisubst. aryl) cm^{-1}. Elemental analysis ($C_{60}H_{39}N_3O_7$) (913.28): calcd. C 78.35 H 4.30 N 4.60; ($C_{60}H_{39}N_3O_7 \cdot 2.5\ H_2O$) (958.31): calcd. C 75.14 H 4.62 N 4.38; found C 75.23 H 4.29 N 4.35.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7.86
7.85
7.81
7.79
7.78
7.71
7.44
7.43
3.36
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4.02
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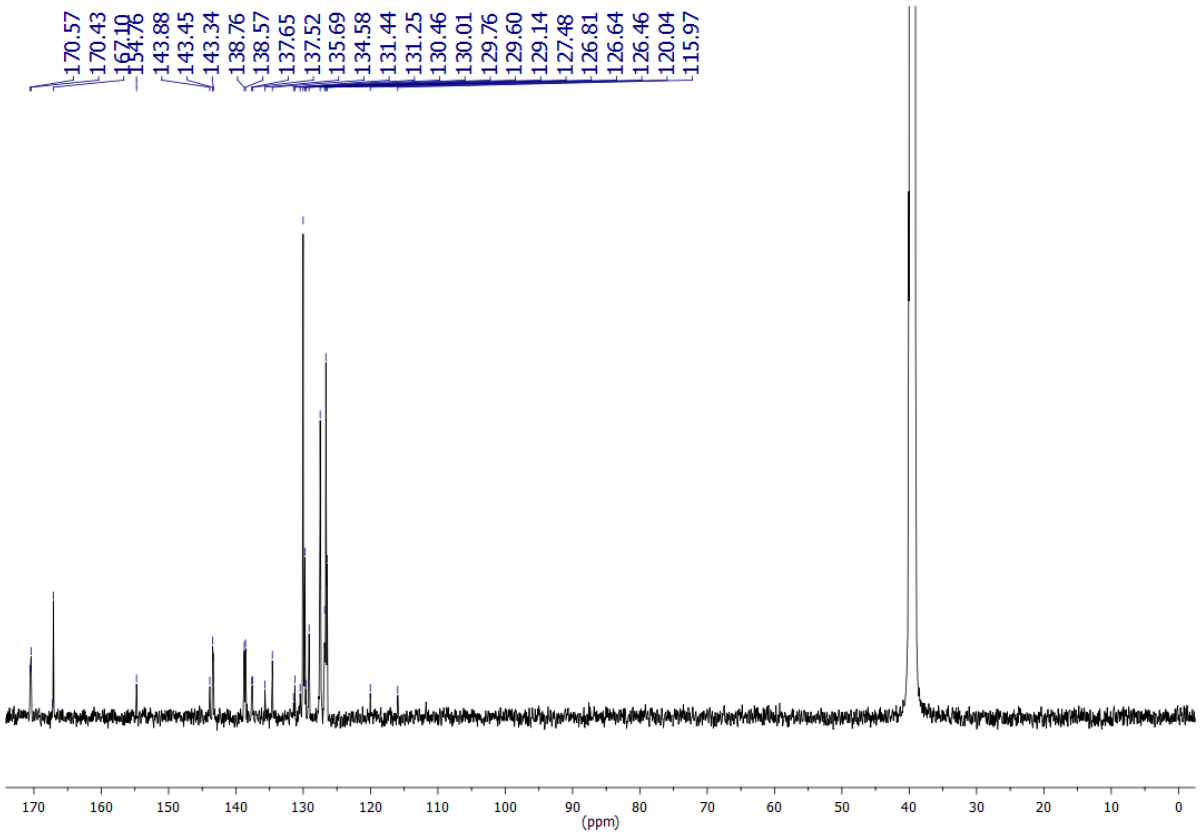
6.17
3.94
14.00
4.05
1.01
(ppm)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137.52
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131.44
131.25
130.46
130.01
129.76
129.60
129.14
127.48
126.81
126.64
126.46
120.04
115.97
(ppm)

Calculations

BTB (benzene-1,3,5-tribenzoic acid)

The BTB molecule was optimized using Gaussian® 09 [6] with B3LYP//6-31G*.

Energy: -935705.0864025

C	-1.32248	-0.43370	-0.01257
C	-1.05177	0.94191	-0.01132
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C	1.34177	0.44074	-0.00996
C	1.03767	-0.92784	-0.01051
C	-0.28902	-1.38108	-0.01175
H	0.50783	2.42534	-0.00981
H	1.84625	-1.65211	-0.01107
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TATB (1,3,5-triazine-2,4,6,-tribenzoic acid)

The BTB molecule was optimized using Gaussian® 09 [7] with B3LYP//6-31G*.

Energy: -965912.4624218

N	-0.31172	1.33316	0.00010
C	0.95712	0.89757	0.00012
N	1.31008	-0.39655	0.00015
C	0.29846	-1.27761	0.00013
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C	-1.25600	0.38039	0.00011
C	0.63612	-2.72290	0.00008
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C	2.29624	-4.49078	0.00035
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C	-0.06509	-5.04180	-0.00038
C	-0.38269	-3.68972	-0.00030
H	-1.41640	-3.36476	-0.00054
H	2.75925	-2.38714	0.00070
H	3.33293	-4.80800	0.00062
H	-0.84159	-5.79938	-0.00068
C	-2.67650	0.81061	0.00006
C	-3.00440	2.17634	-0.00005
C	-3.70617	-0.14360	0.00014
C	-5.03766	0.25700	0.00011
C	-5.35924	1.62159	-0.00001
C	-4.33413	2.57740	-0.00009
H	-2.20618	2.90914	-0.00012
H	-4.60177	3.62872	-0.00017
H	-3.44761	-1.19591	0.00024
H	-5.83068	-0.48225	0.00017
C	2.04007	1.91251	0.00008
C	3.38673	1.51348	0.00001
C	4.39900	2.46441	-0.00006
C	4.08397	3.83013	-0.00004
C	2.74146	4.23409	0.00003
C	1.72867	3.28138	0.00009
H	0.68806	3.58361	0.00013
H	2.49780	5.29051	0.00004
H	3.62219	0.45578	-0.00001
H	5.44324	2.17042	-0.00013
C	5.20817	4.80548	-0.00013
O	6.38544	4.50696	-0.00027

O	4.79028	6.09682	-0.00006
C	1.55839	-6.91272	-0.00014
O	0.71162	-7.78338	-0.00051
H	5.60203	6.63716	-0.00015
C	-6.76624	2.10688	-0.00004
O	-7.09701	3.27549	-0.00015
O	-7.67503	1.09874	0.00004
H	-8.54915	1.53098	0.00000
O	2.88583	-7.19581	0.00027
H	2.94847	-8.16894	0.00017

1\1\GINC-ACID10\Freq\RB3LYP\6-31G(d)\C24H15N3O6\WINKLER\13-Sep-2016\0\\#N Geom=AllCheck Guess=TCheck SCRF=Check GenChk RB3LYP/6-31G(d) Freq\\Title\\0,1\N,-1.1401903877,0.7577732869,0.0144617401\C,0.0826720638,1.3094158965,0.0143672534\N,1.2261866429,0.608207938,0.0139165625\C,1.092513972,-0.7266172174,0.0135180922\N,-0.0865112887,-1.3663533129,0.0135674073\C,-1.1756840739,-0.5831834696,0.014062193\C,2.3284296408,-1.5484450986,0.012919009\C,3.5896133783,-0.9318426151,0.0131400316\C,4.7488770009,-1.6995803864,0.0125841997\C,4.662984125,-3.0989243532,0.0117735747\C,3.4057109758,-3.7183505391,0.0115475627\C,2.2482616759,-2.9506941969,0.0121261525\H,1.2724924368,-3.4219046119,0.0119727392\H,3.6454627214,0.150323436,0.0137592061\H,5.7215124362,-1.2206940972,0.012772477\H,3.3587886788,-4.8021754951,0.010916616\C,-2.5053637508,-1.2426068299,0.0141250296\C,-3.6796776068,-0.4720637117,0.0145208426\C,-2.6020892048,-2.6431064378,0.0137975795\C,-3.8466351731,-3.2631476504,0.0138667694\C,-5.0155556488,-2.4891041075,0.0142592757\C,-4.9232500296,-1.0905654,0.0145853889\H,-3.5999529373,0.6085848988,0.0147695643\H,-5.8383092278,-0.5078432348,0.0148957946\H,-1.6929648434,-3.2327680685,0.0135025055\H,-3.9181455502,-4.344924074,0.0136126733\C,0.1766135797,2.7906499402,0.0147616084\C,1.4311330907,3.4222270386,0.0145904612\C,1.5174236803,4.8084132122,0.014930077\C,0.3525028282,5.5877730851,0.015457089\C,-0.9023745657,4.9626204397,0.0156335346\C,-0.9878215806,3.574781762,0.0152833484\H,-1.9530418665,3.0822856712,0.015399103\H,-1.803450182,5.5654879992,0.0160356863\H,2.3271184832,2.8128174366,0.0141816237\H,2.4796510128,5.3094018123,0.0147898087\C,0.5036221955,7.0684049549,0.0157950049\O,1.5658812784,7.6572063633,0.0155856007\O,-0.6853021335,7.7231128823,0.0163394529\C,5.8700371511,-3.9696517089,0.0111345348\O,5.849362635,-5.1839991961,0.0103906983\H,-0.463908874,8.6727906505,0.0164876452\C,-6.3731260866,-3.0991589712,0.0143503532\O,-7.4145149093,-2.4742097096,0.0146787463\O,-6.3448330076,-4.4561548059,0.0140134191\H,-7.2777858606,-4.7398863844,0.0141027738\O,7.0311450898,-3.2667551767,0.0114906088\H,7.7432820157,-

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0.00000260,0.00007686,-0.00003255,0.,0.00001262,0.00001439,0.00000048,-
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0.00001838,-0.00001027,-0.00000091,-0.00002190,0.00001204,0.00000054,-
0.00003098,-0.00000558,0.00000018,0.00001719,-
0.00001763,0.00000017,0.00002672,0.00000670,-0.00000014,0.00000039,-
0.00002213,-0.00000167,0.00005539,-0.00004358,0.00000023,0.00001476,-
0.00000225,-0.00000061,-
0.00006018,0.00004409,0.00000082,0.00001565,0.00002186,-0.00000060,-
0.00000924,0.00001076,-0.00000059,0.00001851,0.00000030,-0.00000023,-
0.00000273,-0.00000195,0.00000047,-0.00001248,-0.00001121,0.00000025,-
0.00001886,-0.00000060,-0.00000026,-0.00001040,0.00005847,0.00000026,-
0.00000879,-0.00000477,-0.00000014,0.00005071,0.00000827,-
0.00000016,0.00003468,-0.00003969,-0.00000092,0.00001917,0.00001209,-
0.00000011,-0.00007143,0.00000342,0.00000040,0.00004538,-0.00003481,-
0.00000053,-0.00001756,-0.00000938,-
0.00000011,0.00001399,0.00001188,0.00000014,-0.00000978,-0.00000392,-
0.00000025,-0.00000333,-0.00001219,0.00000363,-0.00002659,0.00001497,-
0.00000160,0.00001000,0.00002067,-0.00000060,0.00007494,-0.00000592,-
0.00000263,-0.00000588,-0.00000225,0.00000091,0.00000612,-0.00001053,-
0.00000030,-0.00003242,-0.00002074,0.00000122,-0.00000529,-0.00000465,-
0.00000047,0.00003165,-0.00000130,-0.00000029,-0.00000233,-0.00000542,-
0.00000006,-0.00004189,0.00002520,0.00000114,0.00001079,0.00000074,-
0.00000013\\\@

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