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4-Perfluoroalkylbutoxybenzene derivatives as liquid crystalline organogelators based on phase-selective gelators

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General information

Instrument and equipment

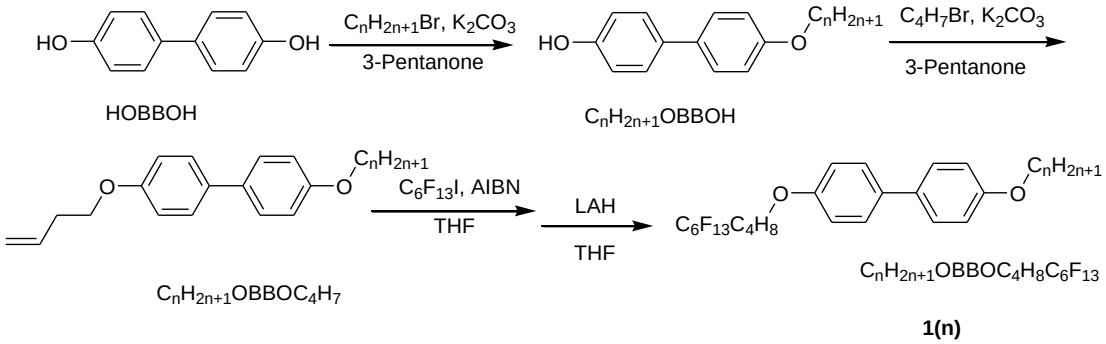
Melting point was obtained with a Yanaco MP-J3 micro melting point apparatus. Infrared spectra were recorded on a Shimadzu IR Prestige-21 spectrometer using KBr disc. ^{1}H NMR spectra were recorded with JMN-LA500 (500 MHz) spectrometer, where tetramethylsilane was used as an internal standard. High resolution mass spectra (HRMS) were recorded with a Waters LCT Premier™ XE. Liquid crystalline properties were determined using a Seiko SSC-5200 DSC, where indium (99.9%) was used as a calibration standard (mp = 56 °C, 28.4 J g^{-1}). The DSC thermogram was operated at a heating rate of 5 °C min^{-1}. The mesophases were characterized when samples were heated up to clear points and then observed at a coling rate of 5 °C min^{-1} using a Nikon POH polarizing microscope fitted with a Mettler thermo-control system (FP-900). High performance liquid chromatograms were recorded on a Shimadzu Prominence HPLC System. Scanning electron microscope (SEM) images were observed with a JEOL JSM-6510LA operating at 200 kV using an acceletating voltage of 100 kV. The accelerating voltage of SEM was 5-15 kV.

Materials

4,4’-Biphenol was purchased from wako Industries, Ltd. reagent chemicals; 4-bromo-1-butene, 1-bromopentane, 1-bromodecane, 4-pentyloxyphenol, 4-hexyloxyphenol, lithium aluminum hydride (LAH) and 2,2'-azobis(isobutyronitrile) (AIBN) were purchased from Tokyo Chemical Industry Co., Ltd. reagent chemicals; 1-Iodoperfluorohexane were purchased from Daikin Industries Ltd. reagent chemicals. Rape oil was purchased from Nisshin oillio group Ltd. Other reagents and solvents were obtained from general commercial sources.

Scheme S1 Synthesis scheme for **1(n)**-**4(5)**

Scheme S1-1(n) Synthesis route of compounds **1(n)**



1(n)

Preparation of C_nH_{2n+1}OBBOH

1-Bromoalkane (20 mmol) and potassium carbonate (4.14 g, 30 mmol) were added to a 3-pentanone solution (20 mL) of 4,4'-biphenol (3.72 g, 20 mmol) and stirred at 80 °C for one day. The precipitate of reaction mixture was removed by filtration, the filtrate was evaporated in *vacuo*, and the residue was purified by silica gel column chromatography, and recrystallization from CH_3OH gave pure products.

Physical date of C_5H_{11}OBBOH

Yield = 50%, colorless needles, mp = 166-167 °C, IR (KBr, cm^{-1}) ν = 3346.5, 2956.8, 2933.7, 2864.3, 1502.6, 1271.1, 1253.7, 823.6.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.53-7.37 (m, 4H), 7.02-6.92 (m, 2H), 6.92-6.83 (m, 2H), 4.78 (s, 1H), 3.99 (t, *J* = 6.6 Hz, 2H), 1.87-1.72 (m, 2H), 1.54-1.33 (m, 4H), 0.94 (t, *J* = 7.1 Hz, 3H) ppm.

Physical date of C_6H_{13}OBBOH

Yield = 45%, colorless needles, mp = 155-156 °C, IR (KBr, cm^{-1}) ν = 3346.5, 2955.0, 2933.7, 2870.1, 1608.6, 1502.6, 1230.0, 816.0.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.45-7.41 (m, 4H), 6.94 (d, *J* = 8.6 Hz, 2H), 6.87 (d, *J* = 8.6 Hz, 2H), 5.02 (s, 1H), 3.98 (t, *J* = 6.6 Hz, 2H), 1.82-1.78 (m, 2H), 1.48-1.45 (m, 2H), 1.38-1.32 (m, 4H), 0.91 (t, *J* = 7.2 Hz, 3H) ppm.

Preparation of C_nH_{2n+1}OBBOC$_4$H$_7$

4-Bromo-1-butene (1.35 g, 10 mmol) and potassium carbonate (2.07 g, 15 mmol) were added to a 3-pentanone solution (10 mL) of C_nH_{2n+1}OBBOH (10 mmol) and refluxed for 2 days. The precipitate of reaction mixture was removed by filtration, the filtrate was evaporated in *vacuo*, and the residue was purified by silica gel column chromatography, and recrystallization from CH_3OH gave pure products.

Physical date of C_5H_{11}OBBOC$_4$H$_7$

Yield = 30%, colorless needles, mp = 126-127 °C, IR (KBr, cm^{-1}) ν = 2958.8, 2933.7, 2873.9, 1606.7, 1500.6, 1275.0, 1248.0, 825.5.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.55- 7.38 (m, 4H), 7.02-6.87 (m, 4H), 5.93 (ddt, *J* = 17.2, 10.4, 6.7 Hz, 1H), 5.19 (d, *J* = 17.2 Hz, 1H), 5.12 (d, *J* = 10.3 Hz, 1H), 4.05 (t, *J* = 6.7 Hz, 2H), 3.98 (t, *J* = 6.5 Hz, 2H), 2.63-2.51 (m, 2H), 1.81 (dd, *J* = 10.7, 4.0 Hz, 2H), 1.54-1.34 (m, 4H), 0.94 (t, *J* = 7.2 Hz, 3H) ppm.

ESI-TOF-MS: m/z calcd for $C_{21}H_{26}O_2$, $[M+H]^+$: 311.2011, found: 311.2004.

Physical date of C_6H_{13}OBBOC$_4$H$_7$

Yield = 35%, colorless needles, mp = 117-128 °C, IR (KBr, cm^{-1}) ν = 2955.0, 2933.7, 1606.7, 1500.6, 1273.0, 1248.0, 825.5.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.45-7.33 (m, 4H), 6.94-6.84 (m, 4H), 5.85 (ddt, *J* = 17.0, 10.3, 6.7 Hz, 1H), 5.12 (d, *J* = 17.0 Hz, 1H), 5.06 (d, *J* = 10,3 Hz, 1H), 3.98 (t, *J* = 6.7 Hz, 2H), 3.91 (t, *J* = 6.6 Hz, 2H), 2.52-2.47 (m, 2H), 1.81-1.62 (m, 2H), 1.47-1.36 (m, 2H), 1.30-1.28 (m, 4H), 0.88 (t, *J* = 7.2 Hz,3H) ppm.

Preparation of $C_nH_{2n+1}OBBOC_4H_8C_6F_{13}$

1-Iodoperfluorohexane (1.35 g, 3 mmol) and AIBN (0.50 g, 3 mmol) were added to a THF solution (4 mL) of $C_nH_{2n+1}OBBOC_4H_7$ (3 mmol) and stirred at 70 °C under nitrogen for one day. The reaction was quenched with Na_2CO_3 (aq.), diluted with ethyl acetate and rinsed with water twice and then with brine. After the organic layer was dried using anhydrous magnesium sulphate, the solvent was evaporated in *vacuo*. The residue without any other refined was dissolve in THF (anhydrous) to the next step. The mixture with $LiAlH_4$ (1 *eq*.) stirred at room temperature for one day. The reaction quenched with NH_4Cl (aq.). The mixture was filtered and the filtrate was evaporated in *vacuo*. The residue was purified by silica gel column chromatography, and recrystallization from CH_3OH gave pure products.

Physical date of $C_5H_{11}OBBOC_4H_8C_6F_{13}$

Yield = 35%, colorless needles, IR (KBr, cm^{-1}) v = 2958.8, 2933.7, 2875.9, 1606.7, 1500.6, 1273.0, 1246.0, 1211.3, 1190.1, 1180.2, 1143.8, 1037.7, 823.6, 700.2.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.59-7.38 (m, 4H), 7.01-6.86 (m, 4H), 4.03 (t, *J* = 5.8 Hz, 2H), 3.98 (t, *J* = 6.6 Hz, 2H), 2.17 (m, 2H), 1.84 (m, 6H), 1.51-1.31 (m, 4H), 0.94 (t, *J* = 7.1 Hz, 3H) ppm.

ESI-TOF-MS: m/z calcd for $C_{27}H_{27}F_{13}O_2$, $[M+HCOO]^-$: 675.1780, found: 675.1789.

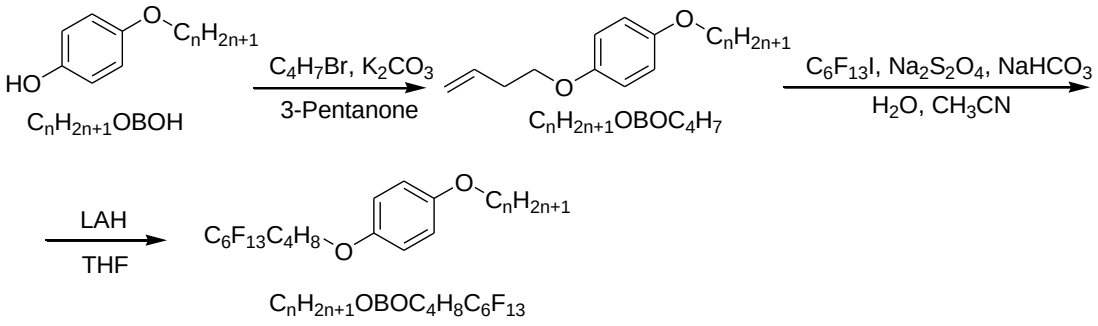
Physical date of $C_6H_{13}OBBOC_4H_8C_6F_{13}$

Yield = 40%, colorless needles, IR (KBr, cm^{-1}) v = 2931.8, 2875.9, 1606.7, 1500.6, 1273.0, 1247.9, 1190.1, 1143.8, 1041.6, 825.5, 700.2.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.48-4.73 (m, 4H), 7.02- 6.88 (m, 4H), 4.04 (t, *J* = 5.9 Hz, 2H), 3.99 (t, *J* = 6.6 Hz, 2H), 2.22-2.11 (m, 2H), 1.91-1.70 (m, 6H), 1.54-1.43 (m, 2H), 1.42-1.29 (m, 4H), 0.94 (m, *J* = 7.2 Hz, 3H) ppm.

ESI-TOF-MS: m/z calcd for $C_{28}H_{29}F_{13}O_2$, $[M+HCOO]^-$: 689.1937, found: 689.1940.

Scheme S1-2(n) Synthesis route of compounds **2(n)**



2(n)

Preparation of $C_nH_{2n+1}OBOC_4H_7$

4-Bromo-1-butene (5.40 g, 40 mmol) and potassium carbonate (5.52 g, 40 mmol) were added to a 3-pentanone solution (40 mL) of 4-pentyloxyphenol (or 4-hexyloxyphenol) (40 mmol) and the reaction mixture was refluxed for 2 days. The precipitate of reaction mixture was removed by filtration, the filtrate was evaporated in *vacuo*, and the residue was purified by silica gel column chromatography, and recrystallization from CH_3OH gave pure products.

Physical date of $C_5H_{11}OBOC_4H_7$

Yield = 40 %, a colorless oil.

^{1}HNMR (500 MHz, $CDCl_3$) δ = 6.82 (s, 4H), 5.90 (ddt, *J* = 17.1, 10.1, 6.6 Hz, 1H), 5.16 (d, *J* = 17.1 Hz, 1H), 5.10 (d, *J* = 10.1 Hz, 1H), 3.96 (t, *J* =6.6 Hz, 2H), 3.89 (t, *J* =6.4 Hz, 2H), 2.51 (m, 2H), 1.75 (m, 2H), 1.33-1.46 (m, 4H), 0.92 (t, *J* =6.9 Hz, 3H) ppm.

Physical date of $C_6H_{13}OBOC_4H_7$

Yield = 43 %, a colorless oil.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.07 (t, *J* = 5.6 Hz, 2H), 6.89-6.76 (m, 2H), 5.90 (ddt, *J* = 17.1, 10.1, 6.7 Hz, 1H), 5.16 (d, *J* = 17.1 Hz, 1H), 5.09 (d, *J* = 10.1 Hz, 1H), 3.99 (t, *J* = 6.7 Hz, 2H), 2.56-2.48 (m, 4H), 1.57 (t, *J* = 7.2 Hz, 2H), 1.33-1.27 (m, 6H), 0.87 (t, *J* = 7,2 Hz,3H) ppm.

Preparation of $C_nH_{2n+1}OBOC_4H_8C_6F_{13}$

1-Iodoperfluorohexane (4.80 g, 8.79 mmol), sodium hydrogen carbonate (0.70 g, 8.69 mmol) and hydrosulfite sodium (1.50 g, 8.79 mmol) were added to an acetonitrile (8.5 ml) and water (5.5 ml) solution of the compound ($C_nH_{2n+1}OBOC_4H_7$) (8.77 mmol) and stirred under shielded light for one night. The reaction solution was quenched with Na_2CO_3 (aq.), diluted with ethyl acetate and rinsed with water twice and then with brine. After the organic layer was dried using anhydrous magnesium sulphate, the solvent evaporated in *vacuo*. The residue without any other refined was dissolved in THF (anhydrous) to the next step. The mixture with $LiAlH_4$ (1 *eq*.) stirred at room temperature for one day. The reaction quenched with NH_4Cl (aq.). The mixture was filtered and the filtrate was evaporated in *vacuo*. The residue was purified by silica gel column chromatography, and recrystallization from CH_3OH gave pure products.

Physical date of $C_5H_{11}OBOC_4H_8C_6F_{13}$

Yield = 25 %, colorless needles, mp = 42-43°C, IR (KBr, cm^{-1}) ν = 2952.5, 2913.9, 2873.4, 1508.1, 1238.1, 1209.1, 1149.4.

^{1}HNMR (270 MHz, $CDCl_3$) δ = 6.82 (s, 4H), 3.92 (t, *J* = 7.0 Hz, 2H), 3.90 (t, *J* = 7.0 Hz, 2H), 2.15 (m, 2H), 1.70-1.90 (m, 6H), 1.30-1.45 (m, 4H), 0.93 (t, *J* = 7.0 Hz, 3H) ppm.

ESI-TOF-MS: m/z calcd for $C_{21}H_{23}F_{13}O_2$, $[M-H]^-$: 553.1412, found: 553.1413.

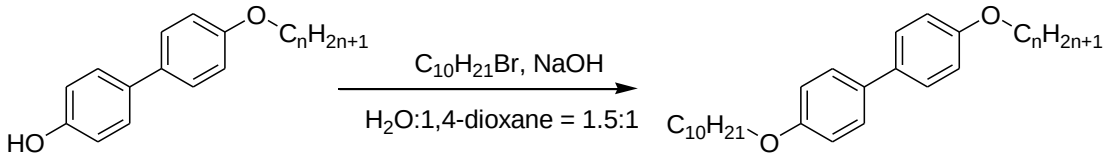
Physical date of $C_6H_{13}OBOC_4H_8C_6F_{13}$

Yield = 20%, a colorless oil.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.13-7.11 (m, 2H), 6.85-6.83 (m, 2H), 3.99 (t, *J* = 5.6 Hz, 2H), 2.68-2.51 (m, 2H), 2.20-2.18 (m, 2H), 1.87 (t, *J* = 10.6 Hz, 4H), 1.62 (d, *J* = 6.4 Hz, 2H), 1.34 (s, 6H), 0.92 (t, *J* = 6.6 Hz, 3H) ppm.

ESI-TOF-MS: m/z calcd for $C_{22}H_{25}F_{13}O_2$, $[M-H]^-$: 567.1569, found: 567.1575.

Scheme S1-3(n) Synthesis route of compounds **3(n)**



$C_nH_{2n+1}OBBOH$ $C_nH_{2n+1}OBBOC_{10}H_{21}$

3(n)

Preparation of $C_5H_{11}OBBOC_{10}H_{21}$

1,4-Dioxane of $C_nH_{2n+1}OBBOH$ (20 mmol) was dissolved in a solution of NaOH (1 mol/L, 20 mL), 40 mL water and 40 mL 1,4-dioxiane, then 1-bromodecane (20 mmol) was added and stirred at 70 °C for one day. The mixture was poured into 200 mL ice water. The precipitate of reaction mixture was removed by filtration, the filtrate was evaporated in *vacuo*, and the residue was purified by silica gel column chromatography, and recrystallization from CH_3OH gave pure products.

Physical date of $C_5H_{11}OBBOC_{10}H_{21}$

Yield = 65 %, colorless needles, mp = 107-108 °C, IR (KBr, cm^{-1}) ν = 2956.9, 2935.7, 2850.8, 1608.6, 1500.6, 1275.0, 1251.8, 1033.9, 825.5.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.45 (d, *J* = 8.6 Hz, 4H), 6.94 (d, *J* = 8.6 Hz, 4H), 3.98 (t, *J* = 6.6 Hz, 4H), 1.88-1.73 (m, 4H), 1.51-1.18 (m, 19H), 0.94 (t, *J* = 7.1 Hz, 3H), 0.88 (t, *J* = 6.8 Hz, 3H) ppm.

ESI-TOF-MS: m/z calcd for $C_{27}H_{40}O_2$, $[M+H]^+$: 397.3107, found: 397.3099.

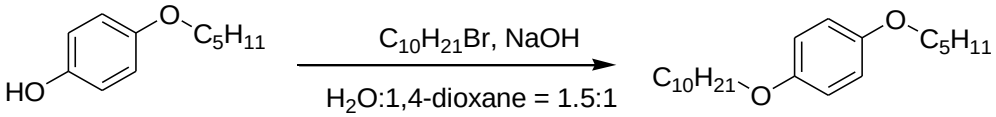
Physical date of $C_6H_{13}OBBOC_{10}H_{21}$

Yield = 70 %, colorless needles, IR (KBr, cm^{-1}) v = 2955.0, 2933.7, 2872.0, 2362.8, 1606.7, 1500.6, 1273.0, 1251.8, 1039.7, 825.5.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.45 (d, *J* = 8.6 Hz, 4H), 6.94 (d, *J* = 8.6 Hz, 4H), 3.98 (t, *J* = 6.6 Hz, 4H), 2.01-1.71 (m, 4H), 1.56-1.21 (m, 20H), 1.13-0.77 (m, 6H) ppm.

ESI-TOF-MS: m/z calcd for $C_{28}H_{42}O_2$, $[M+H]^+$: 411.3263, found: 411.3252.

Scheme S1-4(5) Synthesis route of compound **4(5)**



$C_5H_{11}OBOH$ $C_5H_{11}OBOC_{10}H_{21}$

4(5)

Preparation of $C_5H_{11}OBOC_{10}H_{21}$

1,4-Dioxane of $C_5H_{11}OBOH$ (20 mmol) was dissolved in a solution of NaOH (1 mol/L, 20 mL), 40 mL water and 40 mL 1,4-dioxiane, then 1-bromodecane (20 mmol) was added and stirred at 70 °C for one day. The reaction solution was poured into 200 mL ice water, diluted with ethyl acetate and rinsed with water twice and then with brine. After the organic layer was dried using anhydrous magnesium sulphate, the solvent was evaporated in *vacuo*, and refined by silica gel column chromatography to obtain a compound, and recrystallization from CH_3OH gave pure products.

Physical date of $C_5H_{11}OBBOC_{10}H_{21}$

Yield = 25 %, colorless needles, mp = 46-47 °C, IR (KBr, cm^{-1}) v = 2956.9, 2935.7, 2850.8, 1512.2, 1475.5, 1290.4, 1242.2, 1030.0, 825.5, 771.5.

^{1}H NMR (500 MHz, $CDCl_3$) δ 6.82 (s, 4H), 3.91-3.88 (m, 4H), 1.77-1.73 (m, 4H), 1.52-1.27 (m, 18H), 0.93 (t, *J* = 7.2 Hz, 3H), 0.88 (t, *J* = 6.8 Hz, 3H) ppm.

ESI-TOF-MS: m/z calcd for $C_{21}H_{36}O_2$, $[M+NH_4]^+$: 338.3059, found: 338.3065.

Figure S1 Polarized photomicrographs of compounds **1(6)** and **3(6)** on cooling process

Figure S1-1(6) Polarized photomicrographs of compound **1(6)** on cooling process at: (a) 120 °C, (b) 110 °C, (c) & (d) 100 °C, (e) & (f) 85 °C.

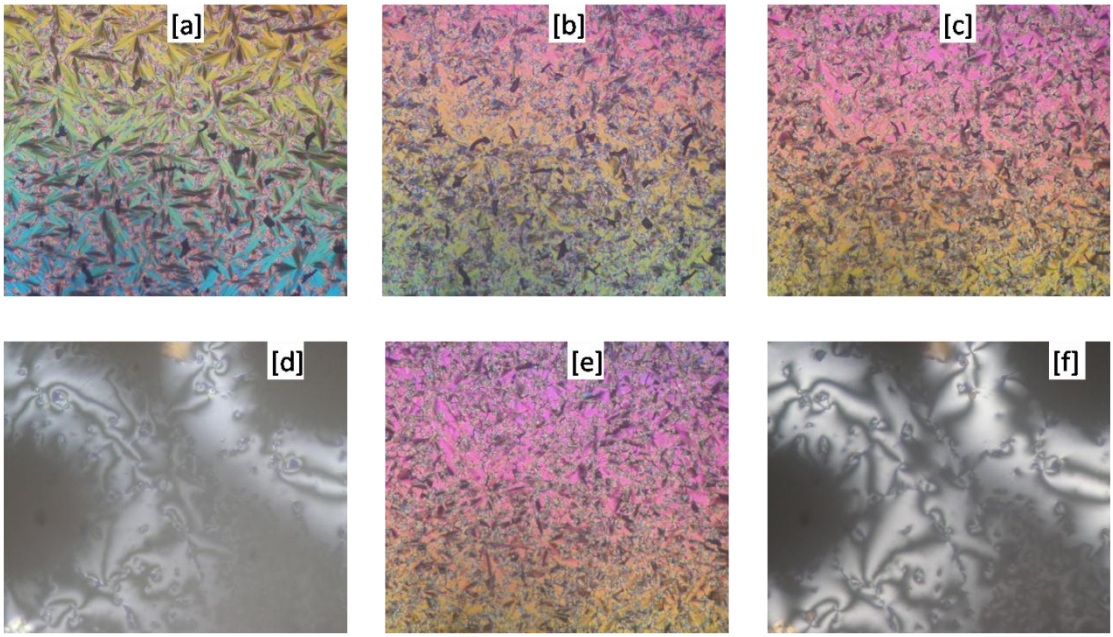


Figure S1-3(6) Polarized photomicrographs of compound **3(6)** on cooling process at: (a) 108 °C, (b) 107 °C.

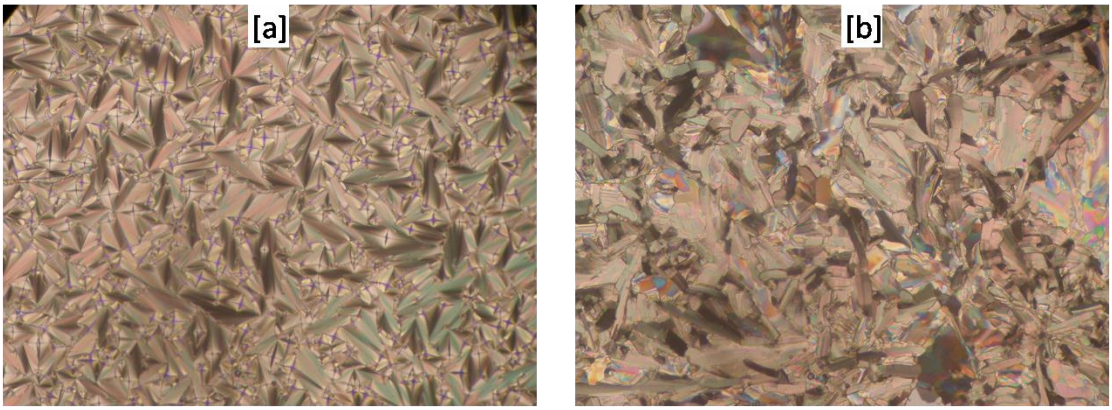


Table S1. The transition temperature (°C) and the associated enthalpies [kJ mol^{-1}] of compounds during the first scanning process[a]

Compound	Heating/Cooling process	Transition temperature (°C) and ΔH [kJ mol^{-1}]
1(5)	H	Cryst. 87[11.7] SmX 97[2.3] SmC 108[2.3] SmA(1) 112[-][b] SmA(2) 163[11.2] Iso
	C	Cryst. 79[4.1] SmX 94[2.4] SmC 105[2.3] SmA(1) 110[-][b] SmA(2) 160[11.3] Iso
1(6)	H	Cryst. 93[13.0] Sm C 108[2.7] SmA(1) 118[0.5] SmA(2) 159[10.4] Iso
	C	Cryst. 79[10.1] SmX 86[1.7] SmC 106[2.8] SmA(1) 115[0.5] SmA(2) 156[10.7] Iso
3(6)	H	Cryst. 111[25.3] Iso
	C	Cryst. 106[22.6] SmA 108[7.2] Iso

[a] Peak temperatures from DSC at a rate of 5 °C min^{-1}; [b] The enthalpy change was contained in Sm A(1)→Sm A(2); abbreviations: Cryst = crystalline solid; SmX = unidentified smectic phase; SmC = smectic C phase; SmA = smectic A phase; Iso = isotropic liquid.

Figure S2 Differential scanning calorimetry during the first process for compounds **1(n)** and **3(6)**

Figure S2-1(5) Differential scanning calorimetry during the first process for compound **1(5)**

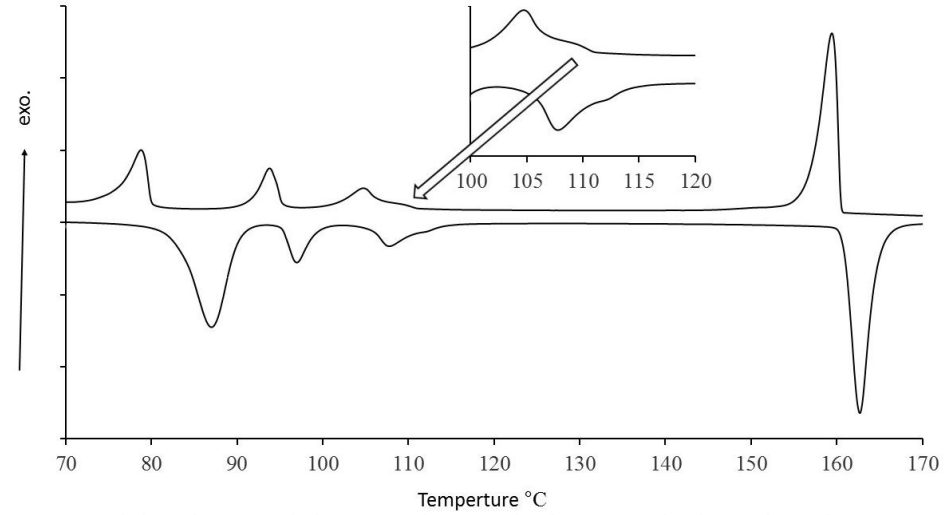


Figure S2-1(6) Differential scanning calorimetry during the first process for compound **1(6)**

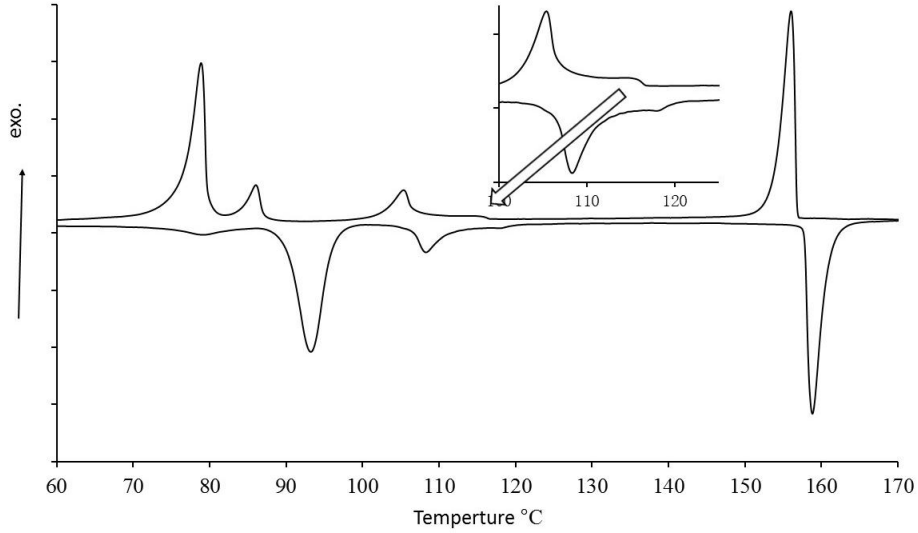


Figure S2-3(6) Differential scanning calorimetry during the first process for compound **3(6)**

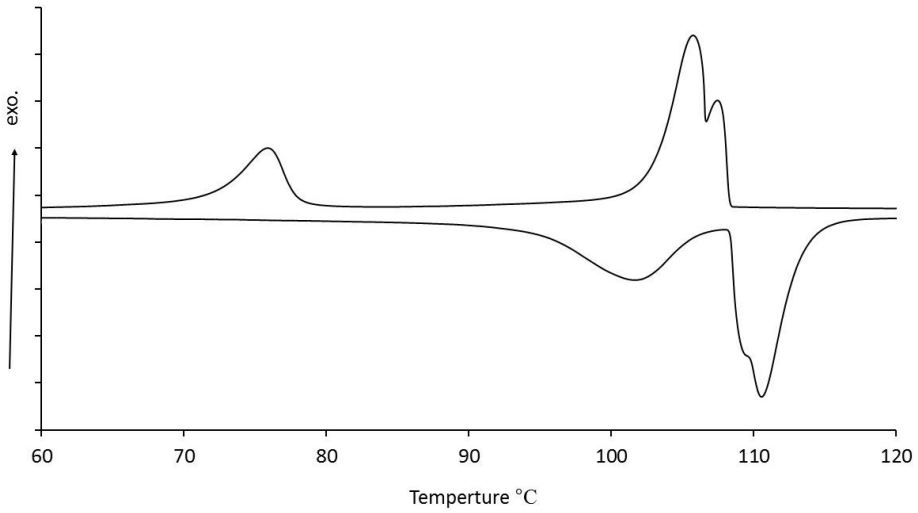


Table S2 Gelation test and critical gel concentration (CGC) of compounds **1(n)** in solvents[a]

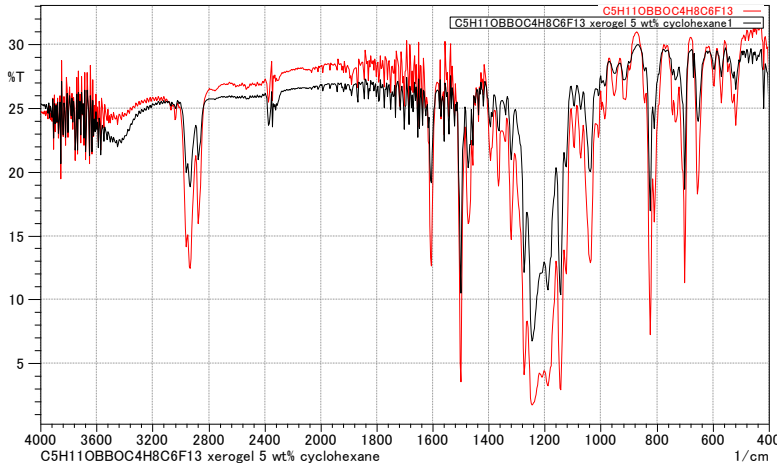
Solvents	**1(5)**/wt%	**1(6)**/wt%
Cyclohexane	G(4.0)	G(4.0)
Octane	G(3.0)	G(2.0)
THF	S(5.0)	S(5.0)
Toluene	S(5.0)	S(5.0)
Methanol	Ins(5.0)	Ins(5.0)
1-Octanol	G(0.7)	G(0.6)
DMF	G(2.0)	G(1.0)
DMSO	G(2.0)	Ins(5.0)
PC[b]	G(0.6)	Ins(5.0)
GBL[c]	G(0.5)	G(0.5)

[a] G = gelation, S = soluble, Ins = insoluble; [b] PC = propylene carbonate; [c] GBL = butyrolactone.

Figure S3-1(n) Infrared spectra of the xerogels of compounds **1(n)**

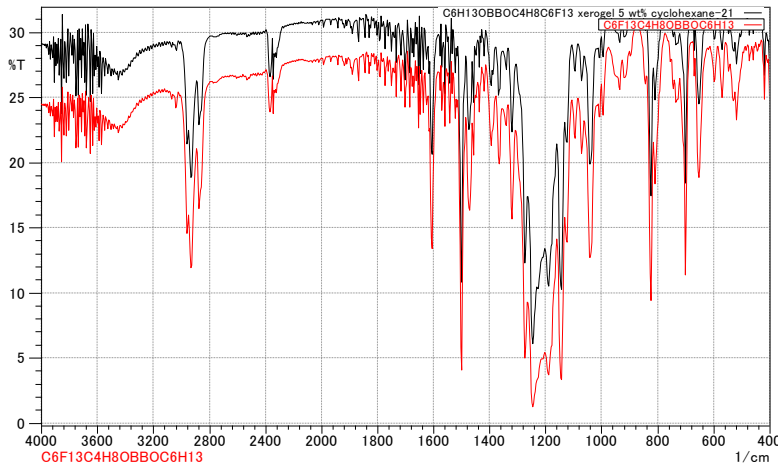
in cyclohexane compared with compounds **1(n)**

Figure S3-1(5) Infrared spectra of the xerogels of compound **1(5)** compared with compound **1(5)**



Red one is compound **1(5)**, black one is xerogel of compound **1(5)** at 5 wt% in cyclohexane.

Figure S3-1(6) Infrared spectra of the xerogels of compound **1(6)** compared with compound **1(6)**



Red one is compound **1(6)**, black one is xerogel of compound **1(6)** at 5 wt% in cyclohexane.

Figure S4 1H NMR spectra of compound **1(5)** in *d*-DMSO

Figure S4-1 At different temperatures

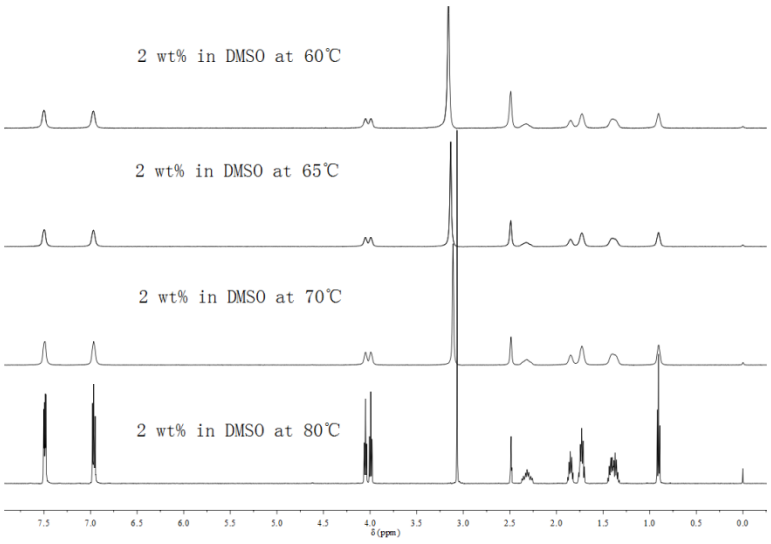


Figure S4-2 At different concentration

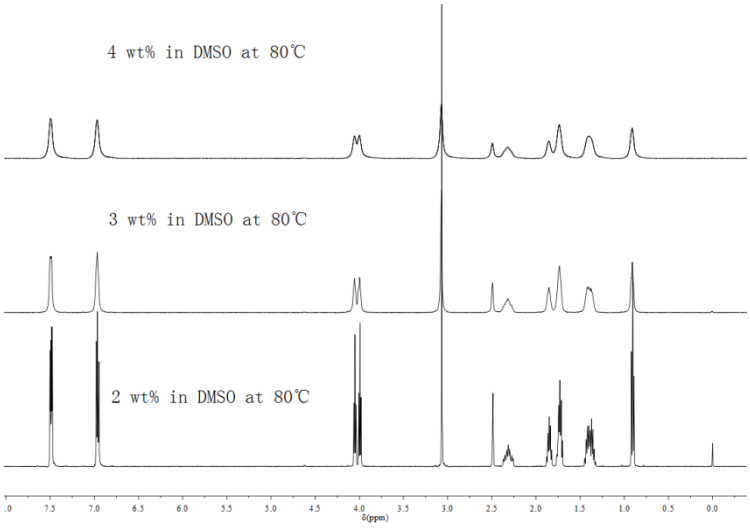


Table S3 Gelation test in aniline and oil using compounds **1(n)** [a]

Solvents	**1(5)**/wt%	**1(6)**/wt%
Aniline	G(1.0)	G(1.0)
Benzylamine	G(1.0)	G(1.0)
β-phenylethlamine	G(1.0)	G(1.0)
Piperidine	G(1.0)	G(1.0)
HMPA[b]	S(5.0)	G(5.0)
N-ethyldiisopropy amine	G(5.0)	S(5.0)
Synthetic lubricant	G(1.0)	G(1.0)
Mineral oil	G(1.0)	G(1.0)
Lamp oil	G(5.0)	G(5.0)
Polyolefin	G(1.0)	G(1.0)
Rape oil	G(1.0)	G(1.0)

[a] G = gelation, S = soluble, [b]HMPA = hexamethylphosphoric triamide.

Figure S5 Photograph of gel formed at amine solution using compounds **1(n)** as gelators

A: aniline, B: benzylamine, C: ß-phenylethylamine, D: HMPA.

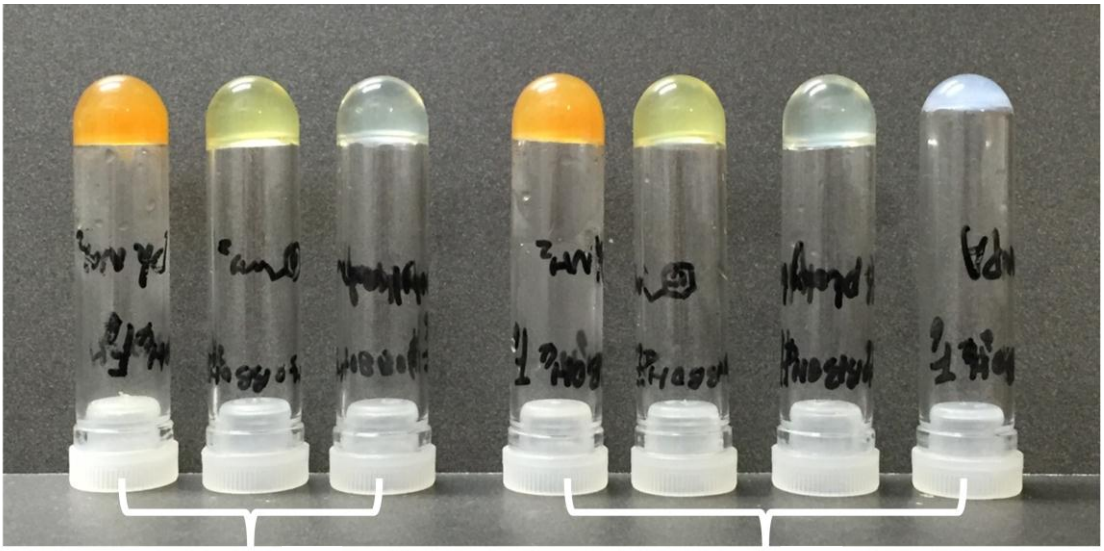


Figure S6-1(n) Photograph of gel formed at oil solution using compounds **1(n)** as gelators

A: synthetic lubricant, B: mineral oil, C: lamp oil, D: polyolefin, E: rape oil.

Figure S6-1(5) Photograph of gel formed at oil solution using compound **1(5)**

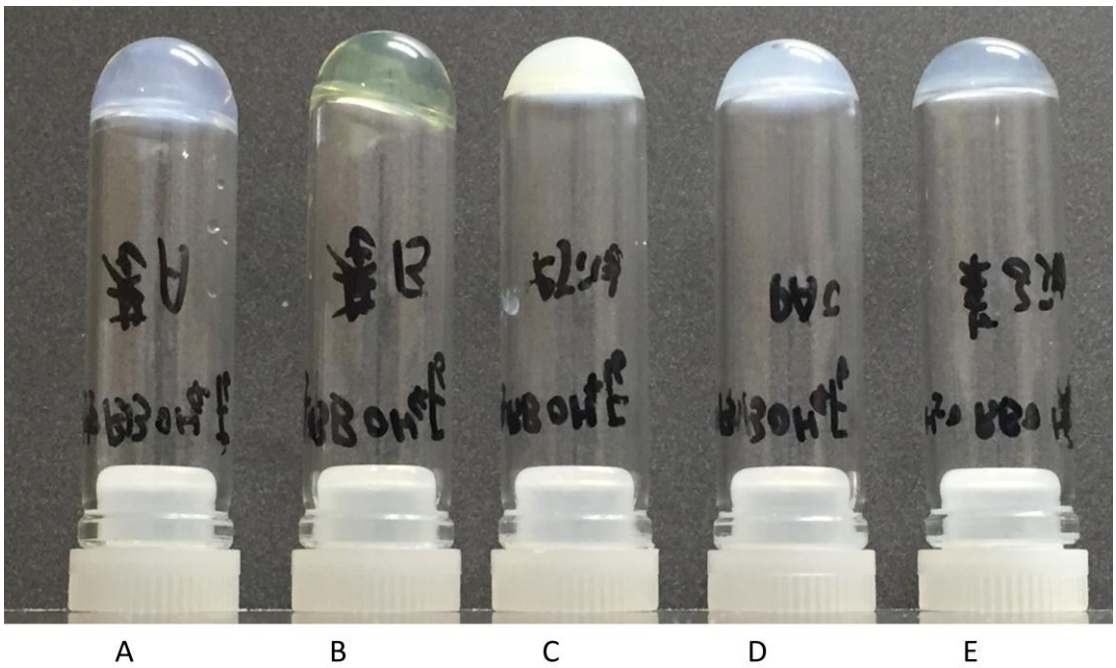


Figure S6-1(6) Photograph of gel formed at oil solution using compound **1(6)** as gelator

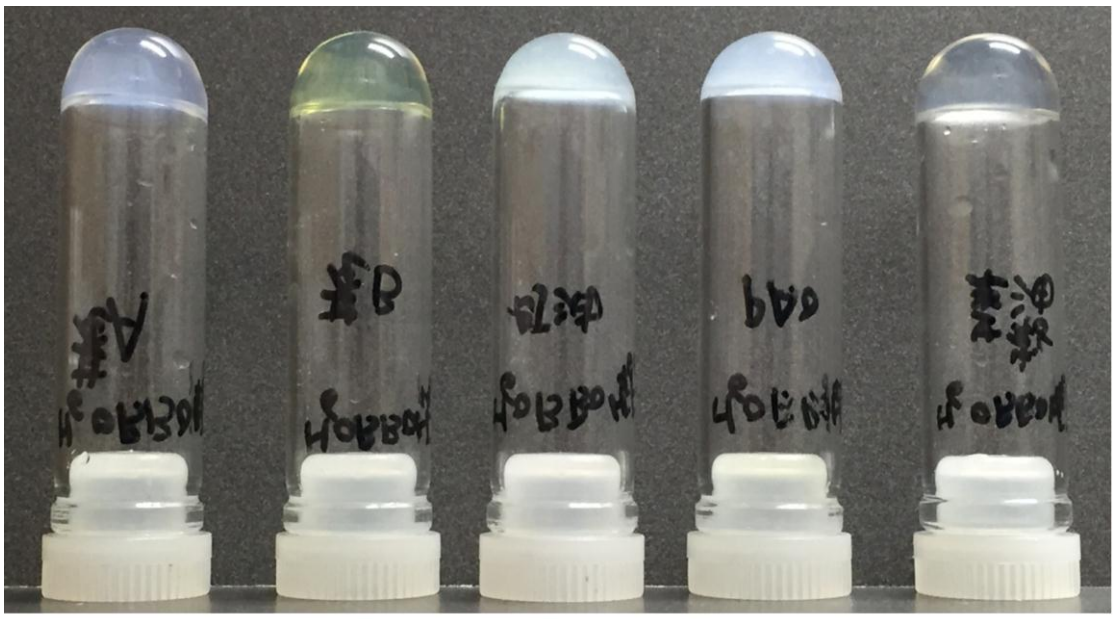


Figure S7 Photograph of gel formed at biphasic mixture using compounds **1(n)** as gelators

A: aniline, B: benzylamine, C: β-phenylethlamine.

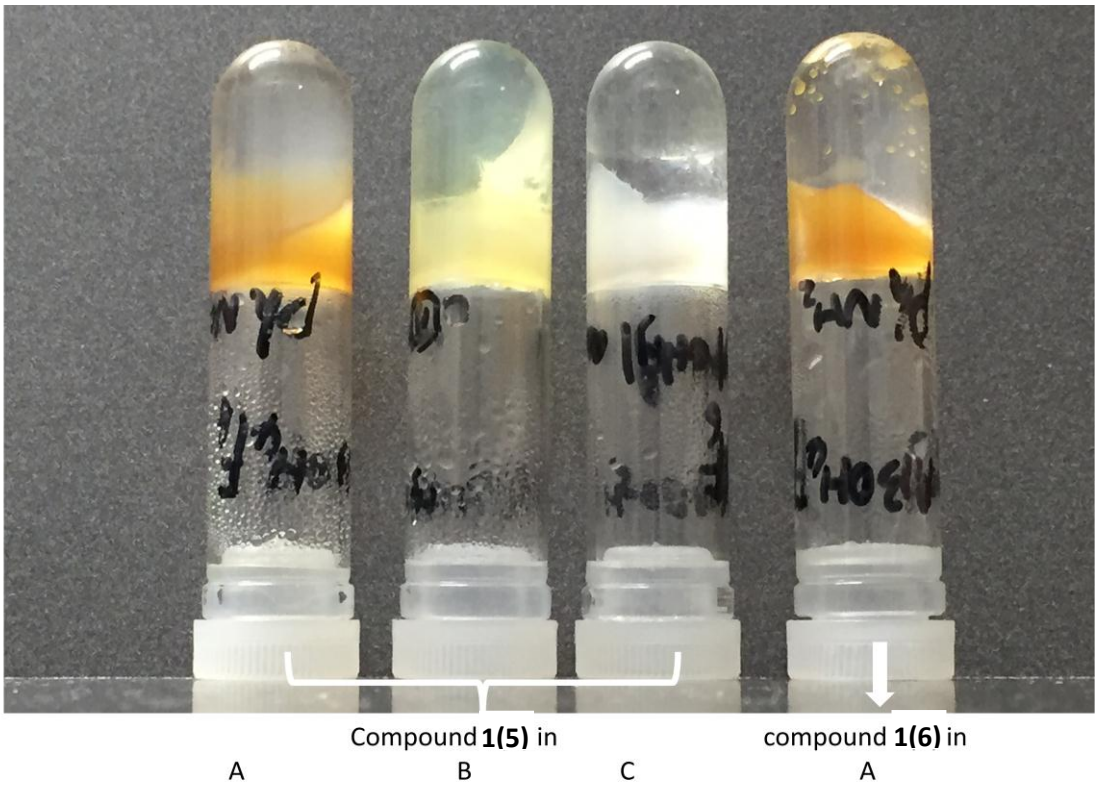


Figure S8-1(n) Photograph of gel formed at saturated solution using compounds **1(n)** as gelators

Saturated solution: NaCl and $NaHCO_3$

A: synthetic lubricant, B: mineral oil, C: lamp oil, D: polyolefin, E: rape oil.

Figure S8-1(5) Photograph of gel formed at saturated solution using compound **1(5)**

Figure S8-1(6) Photograph of gel formed at saturated solution using compound **1(6)** as gelator.

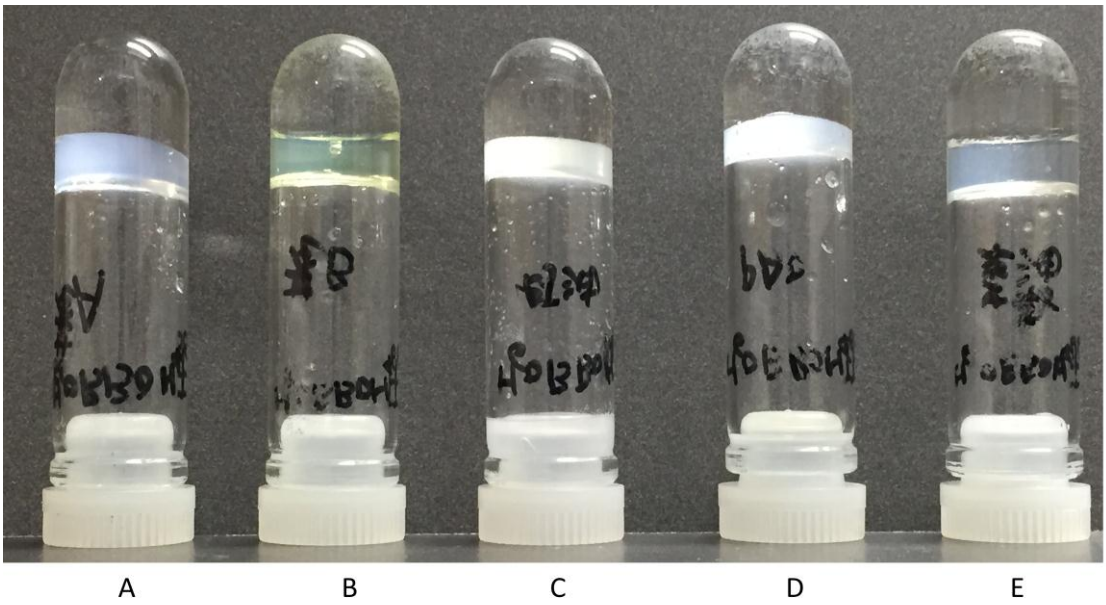
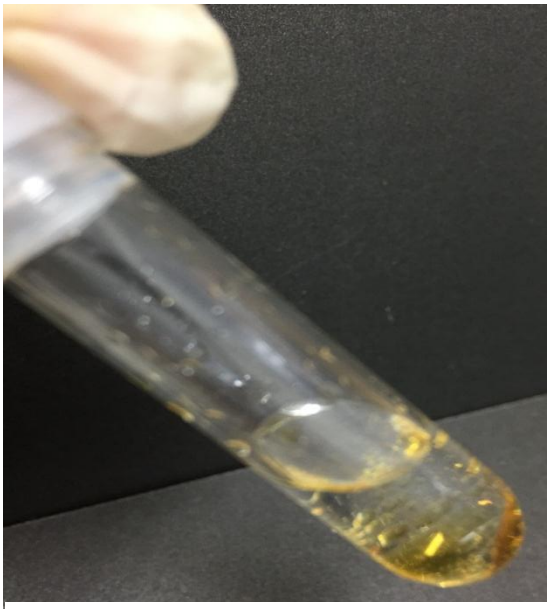
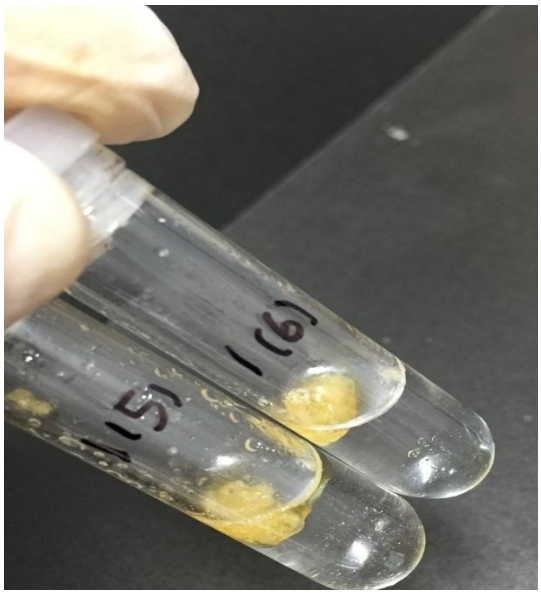


Figure S9 Photograph of gel formed at biphasic systems (Aniline/ water = 1:10) using the toluene solution of compounds **1(n)** as gelators



biphasic systems (Aniline/ water = 1:10)

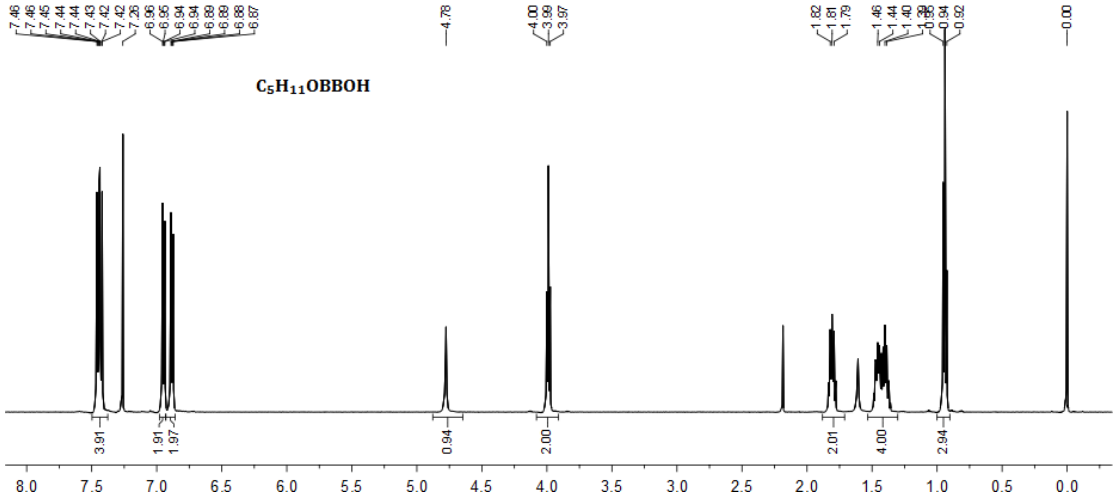


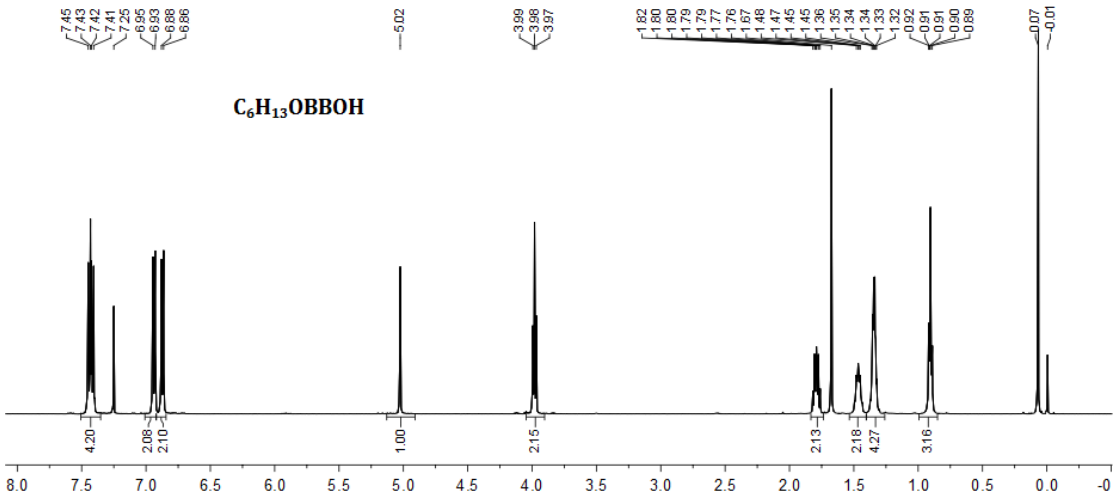
Photograph of gel

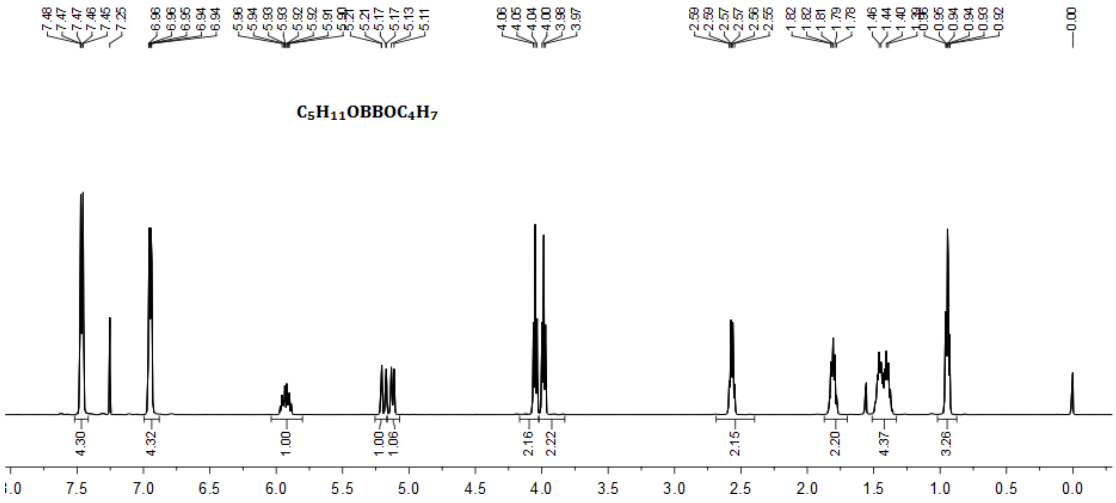
1(5): gel formed by toluene solution of compound **1(5)**;

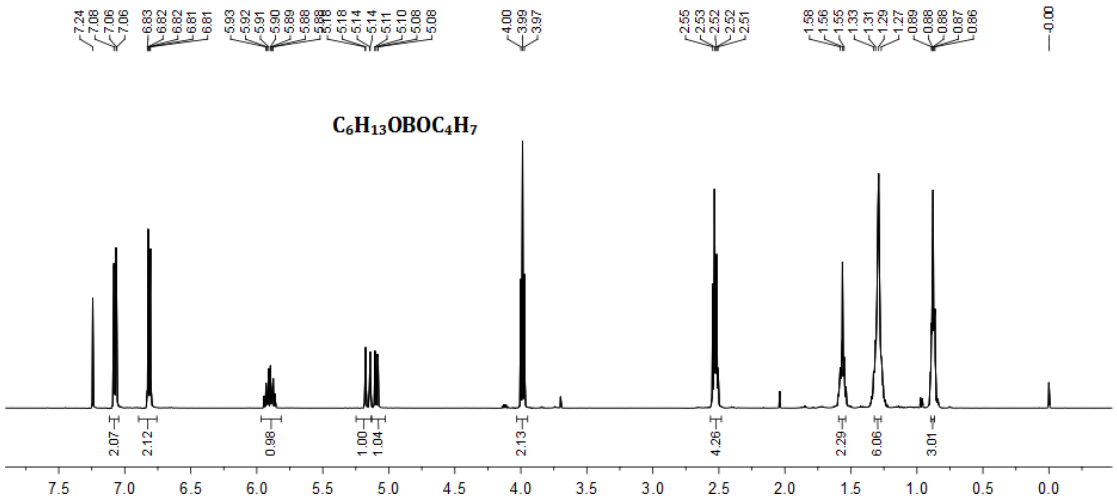
1(6): gel formed by toluene solution of compound **1(6)**.

^{1}H NMR Spectra





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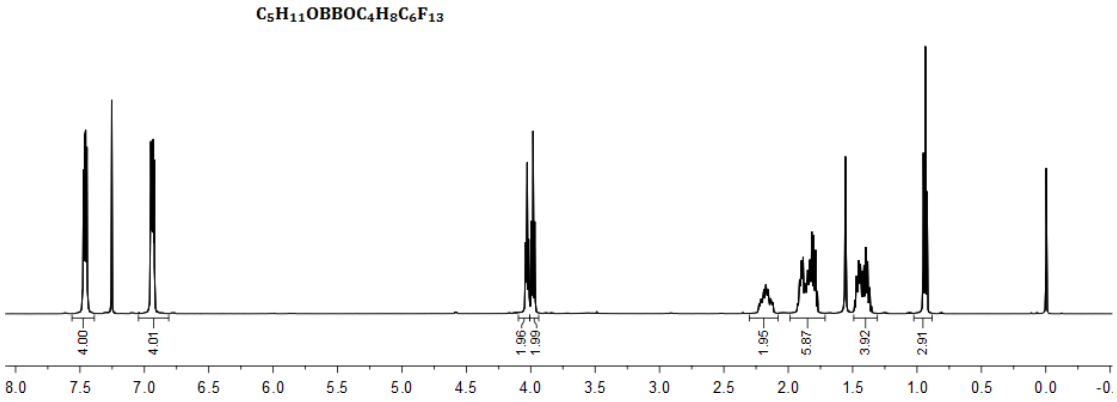
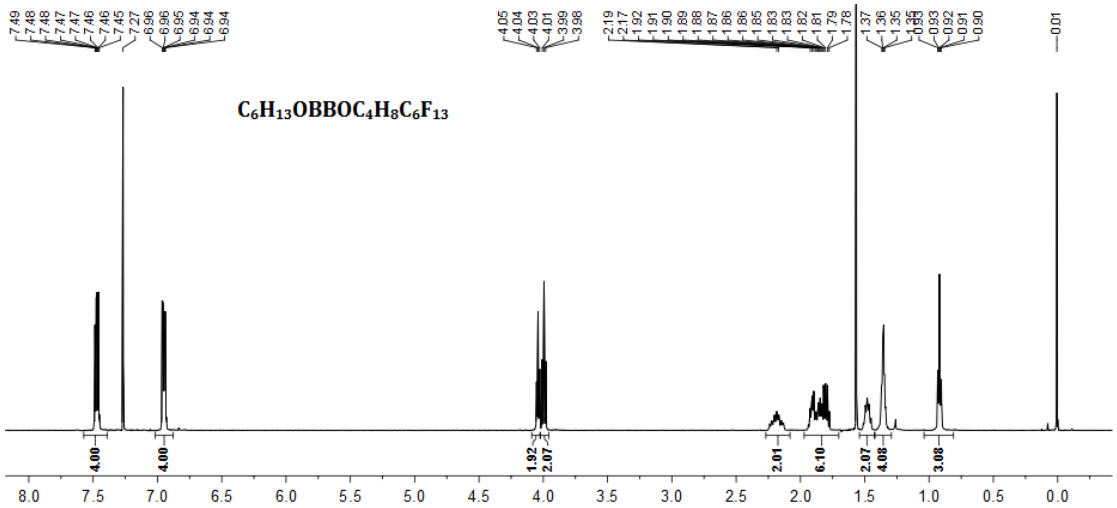
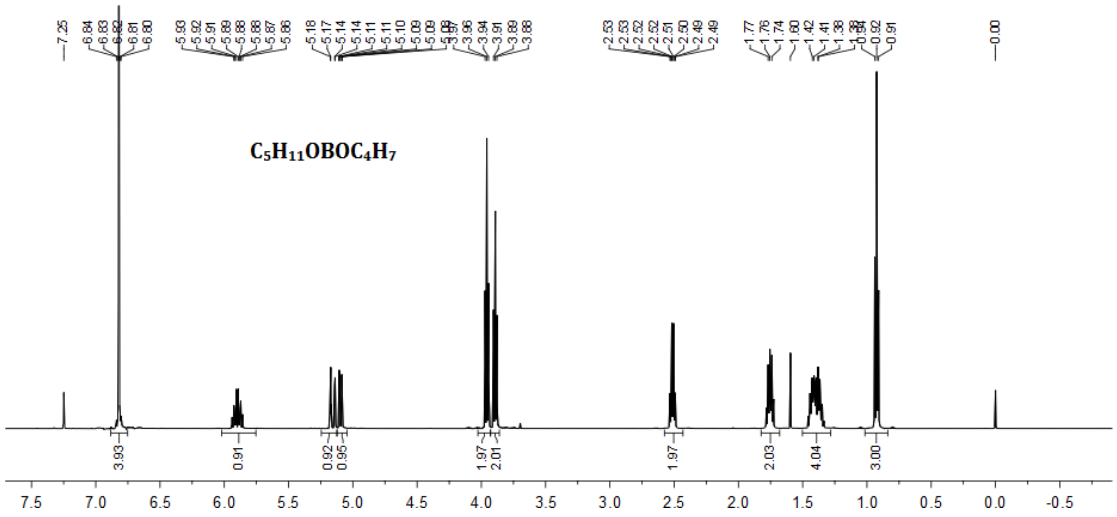

$C_5H_{11}OBBOC_4H_8C_6F_{13}$


$C_6H_{13}OBBOC_4H_8C_6F_{13}$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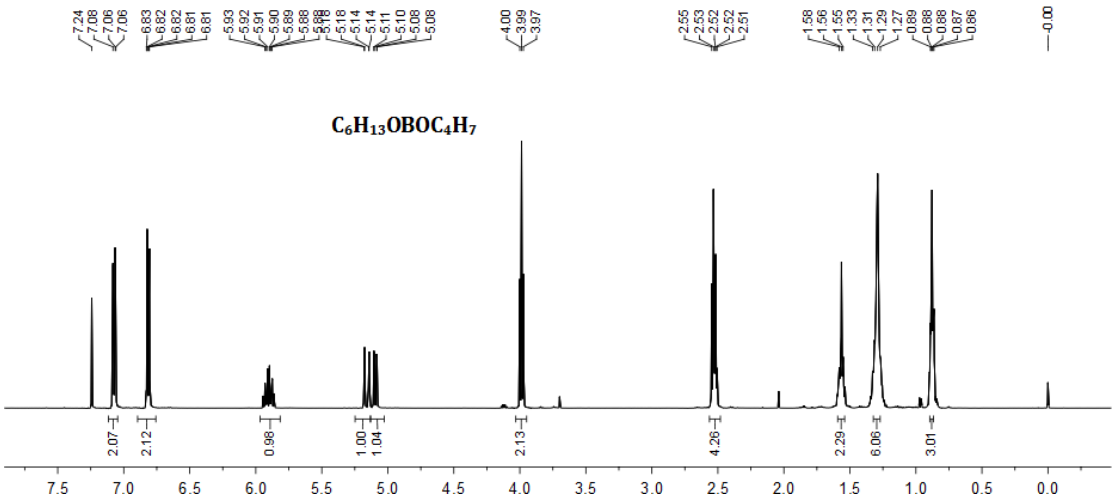
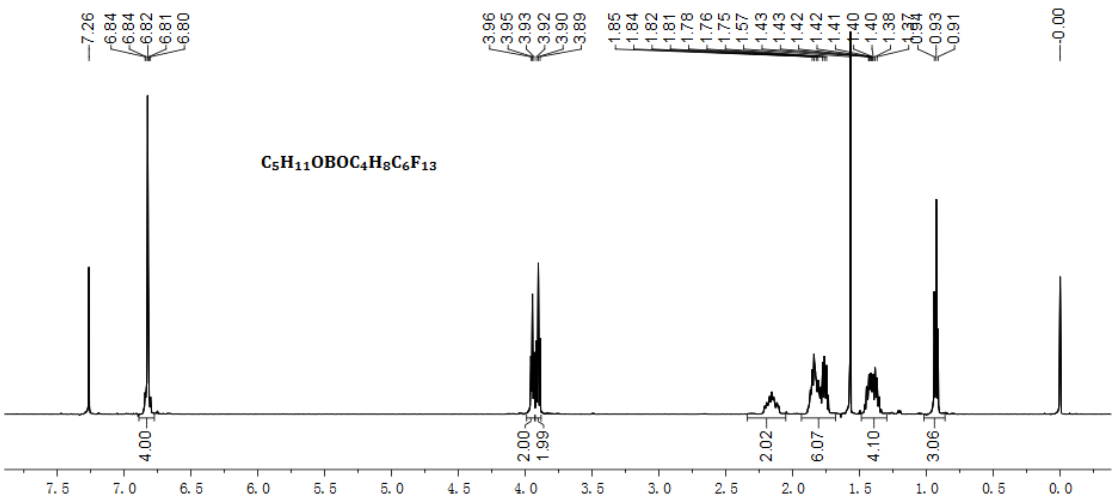
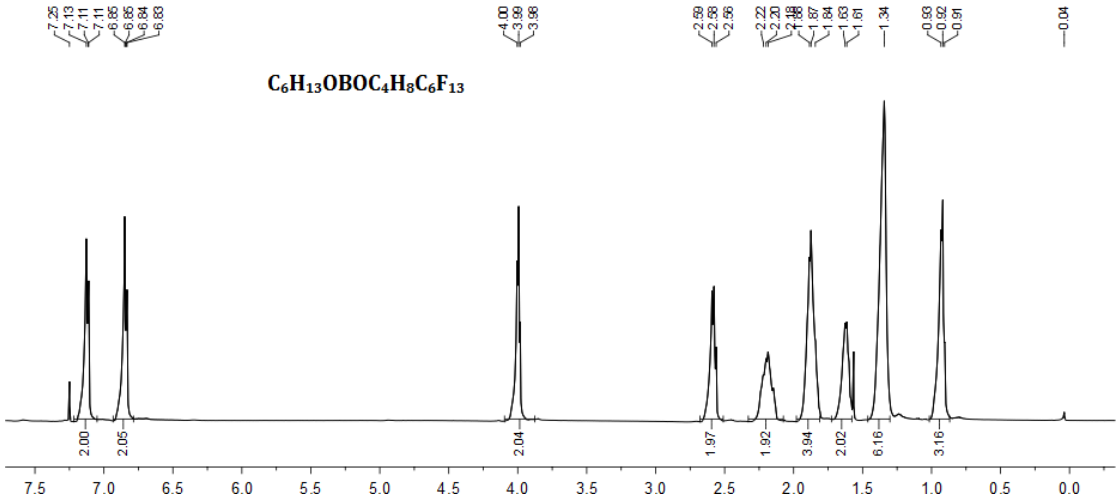
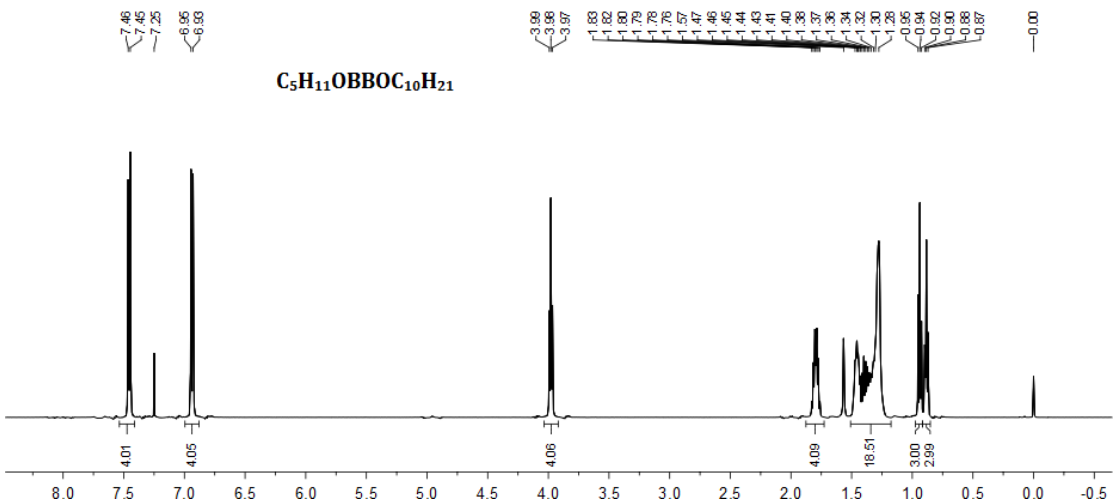

C6H13OBOC4H7

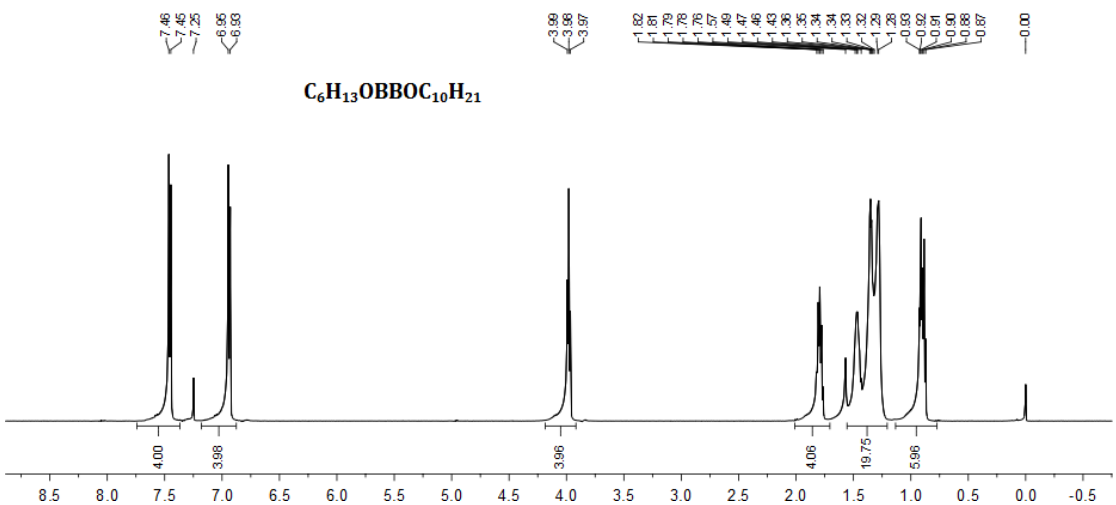

C5H11OBOC4H8C6F13



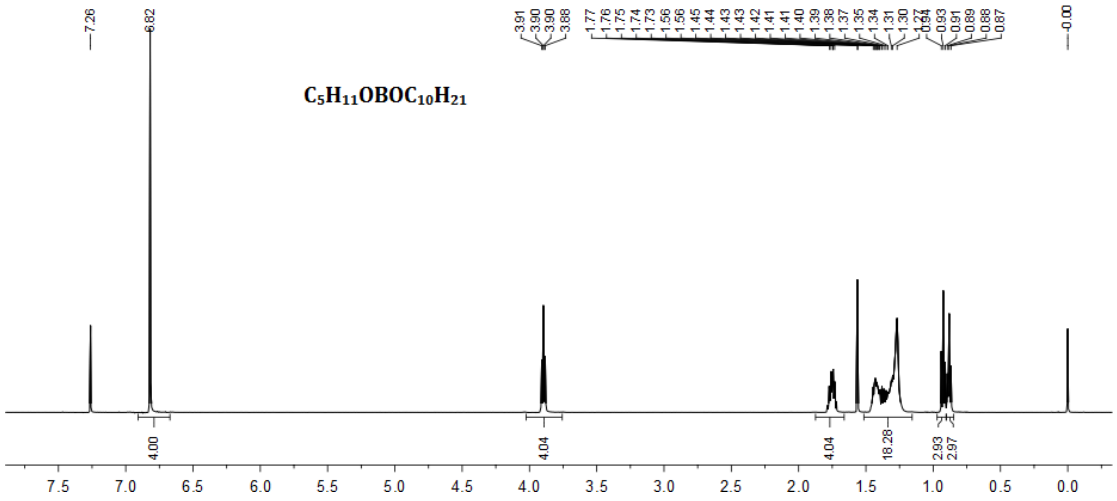
$C_6H_{13}OBOC_4H_8C_6F_{13}$



$C_5H_{11}OBBOC_{10}H_{21}$



$C_6H_{13}OBBOC_{10}H_{21}$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