

Synthesis and ring-opening metathesis polymerization of fullerene-containing α,ω -bis-norbornenes

**Yulia N. Biglova, Vladimir V. Mikheev, Seda A. Torosyan, Raisa Z. Biglova
and Mansur S. Miftakhov**

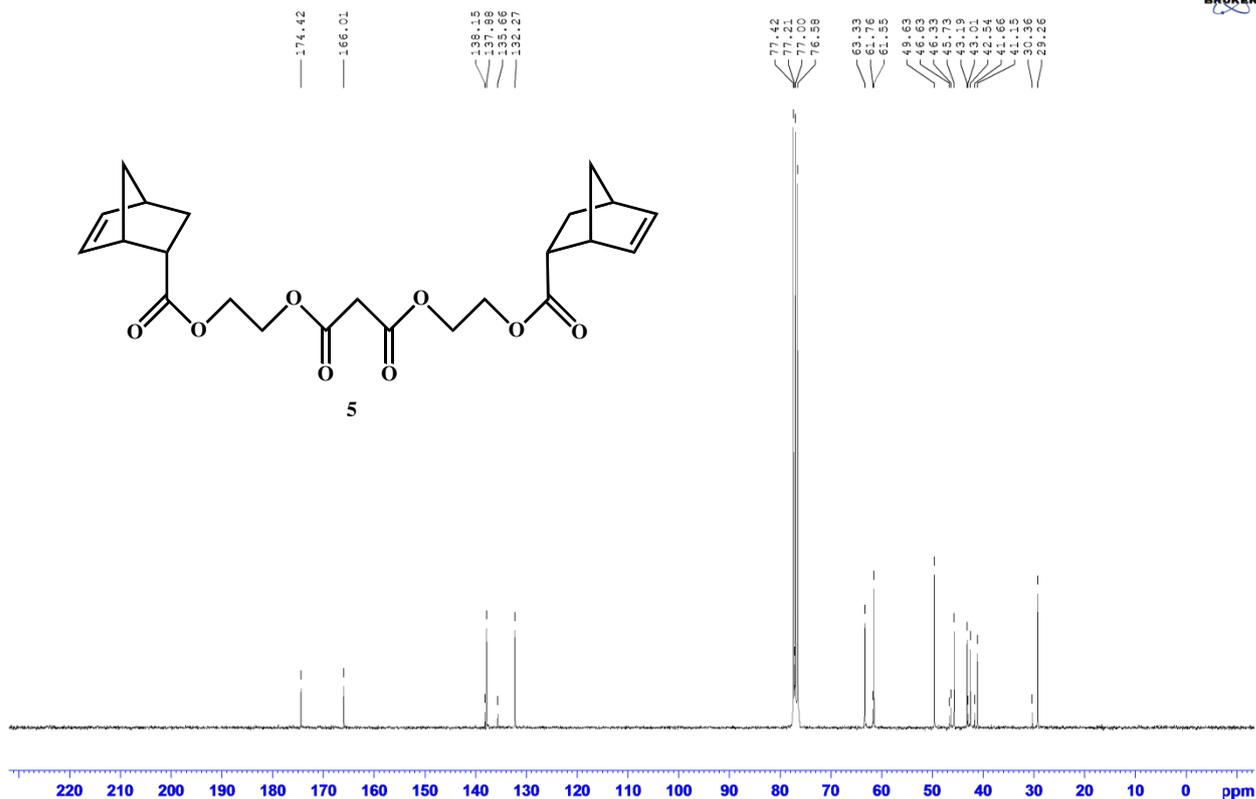
Experimental

IR spectra were obtained on an IR Prestige-21 Shimadzu spectrophotometer for samples in a thin layer. ^1H NMR spectra were recorded on a Bruker AM-300 spectrometer with an operating frequency of 300.13 MHz in solutions of CDCl_3 , TMS as internal standard. ^{13}C NMR spectra were recorded on a Bruker Avance-500 spectrometer with an operating frequency of 125.77 MHz. Mass spectra were obtained on MALDI Voyager-D STR TOF. Elemental analysis was performed on a Euro EA 3000 CHNS-analyzer. Molecular-weight characteristics were determined by GPC method on a liquid chromatograph "Waters AllianceTM GRC 2000 Systems".

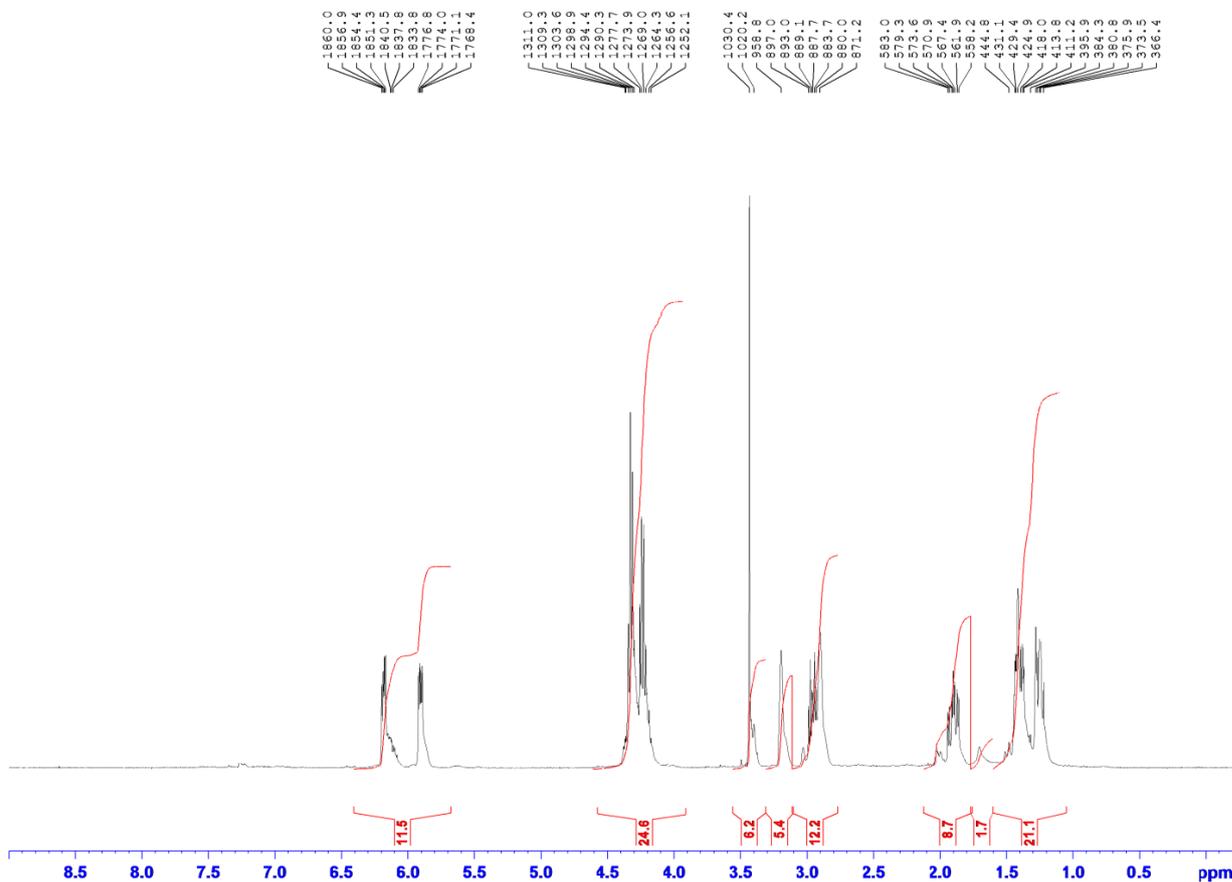
The reaction was monitored by TLC on Sorbfil plates (Russia) with detection of substances by burning or wetting plates alkaline solution of potassium permanganate. The products were isolated by column chromatography on silica gel (30-60 g of the adsorbent per 1g of the substance), light petroleum–ethyl acetate, methylene chloride, toluene were used as eluents.

Bis(2-acryloyloxyethyl) malonate **4**. Pyridine (3.47 ml, 43.06 mmol) of and dropwise slowly 3 g (21.28 mmol) of malonyl dichloride **2** were added to a solution of 5 g (43.06 mmol) of 2-hydroxyethyl acrylate **3** in 20 ml of dichloromethane at 0 °C. The reaction mixture was stirred at room temperature for 24 h until complete consumption of the starting acrylate **3** (TLC monitoring). The reaction mass was washed with aqueous HCl, dried with MgSO_4 , the solvent was evaporated. The residue was purified by column chromatography on silica gel (light petroleum–ethyl acetate, 2:1), 4.85 g (76%) of product **4** was obtained. IR, cm^{-1} : 810, 870, 975, 984, 1044, 1077, 1151, 1190, 1271, 1291, 1298, 1334, 1371, 1410, 1445, 1729, 1736, 1755, 2961. ^1H NMR, δ , ppm: 3.40 s (2H, CH_2), 4.30 br.s (8 H, 4 CH_2O), 5.85 dd (2H, $J = 1.5$ and 10.5 Hz, $\text{CH}=\text{}$), 6.10 dd (2 H, $J = 10.48$ and 17.28 Hz $\text{CH}_2=\text{}$), 6.42 dd (2 H, $J = 0.7$ and 12.0 Hz, $\text{CH}_2=\text{}$). ^{13}C NMR: 41.11 (CH_2), 61.90 and 63.10 (4 CH_2O), 127.88 and 131.50 (2 $\text{CH}_2=\text{CH}$), 165.77 and 166.04 (4 CO_2). Found, %: C 52.00; H 5.30. Calc. for $\text{C}_{13}\text{H}_{16}\text{O}_8$, %: C 52.00; H 5.37.

¹³C NMR

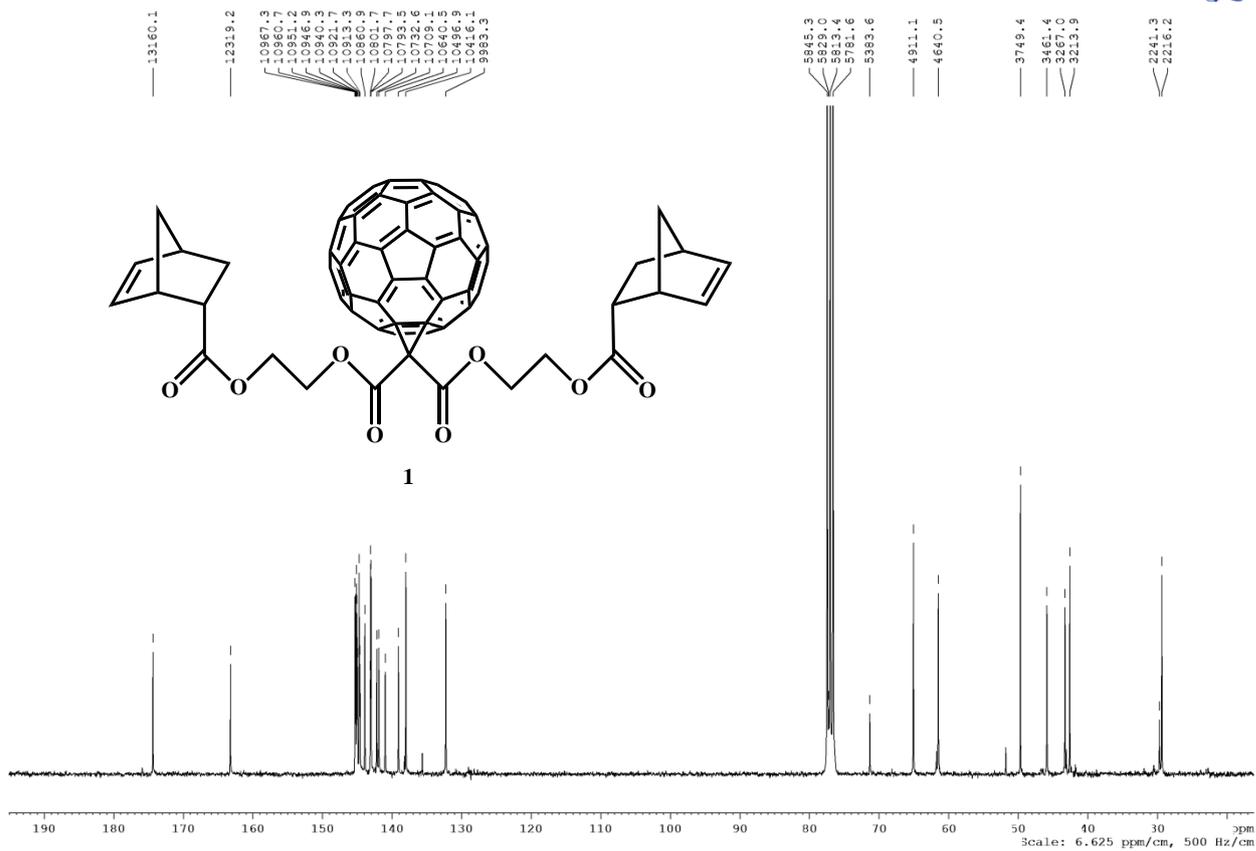


¹H NMR



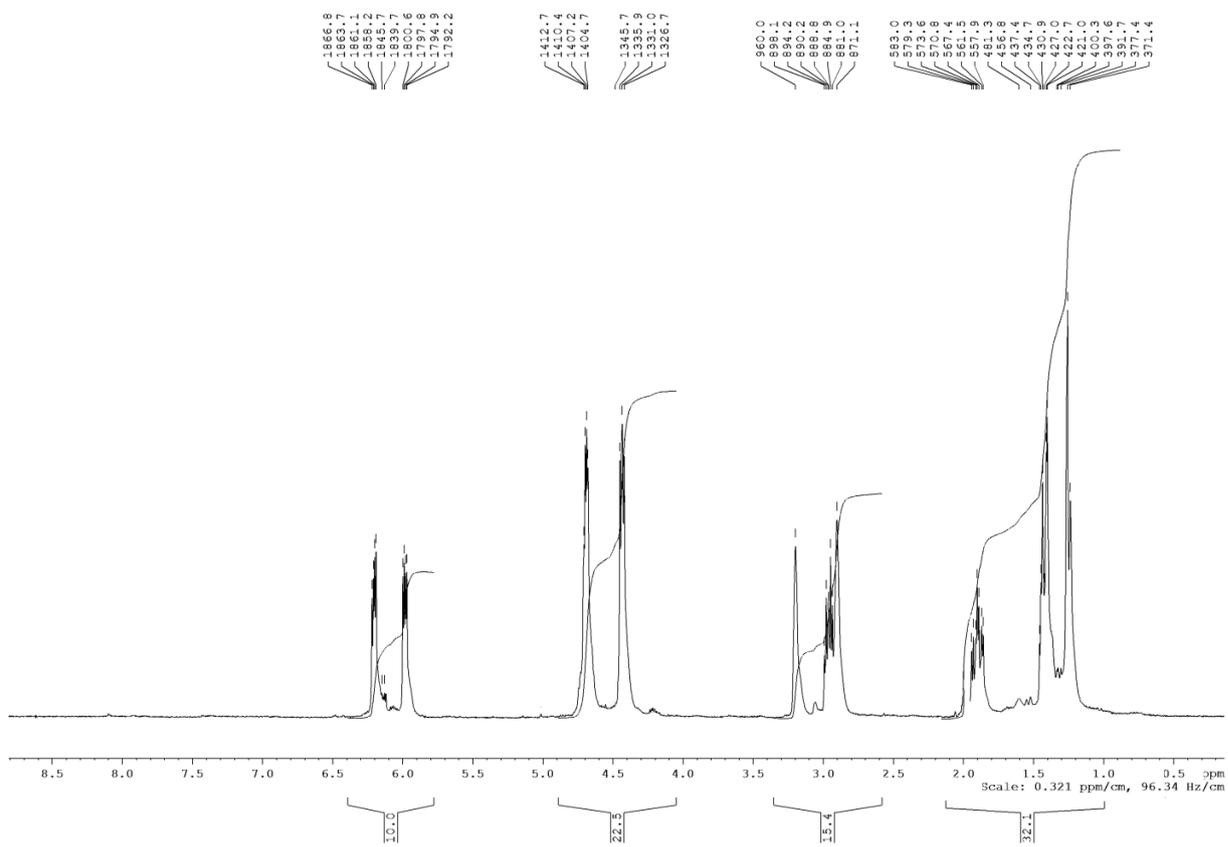
¹³C NMR

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¹H NMR

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^1H NMR

Copolymer **8**

