Electronic supplementary information

SYNTHESIS OF LOW-MOLECULAR-WEIGHT OLIGODIMETHYLSILOXANES IN AN ACTIVE MEDIUM

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General method for the condensation of DEDMS and HMDS in acetic acid upon addition of CTMS as a catalyst

A stirred mixture of DEDMS, HMDS, anhydrous acetic acid, and CTMS, taken at the specified ratio, was refluxed until complete conversion of ethoxy groups according to the results of ^{1}H NMR spectroscopic analysis of the reaction mixture samples. Then a three-fold excess of aq. sodium bicarbonate relative to the acid was added for neutralization. The liquid part was decanted and distilled. The resulting fractions were analyzed using GLC and IR spectroscopy. The yields of low-molecular-weight oligodimethylsiloxanes ranged from 71% to 99% depending on the reaction conditions.

IR (CCl_4, ν/cm^{-1}): 2959(m), 2902(s), 1442(s), 1413(s), 1256(s), 1087, 1032 (Si–O–Si).

^{1}H NMR ($CDCl_3$, 250 MHz): δ 0.035–0.135 ppm (Si-CH_3).

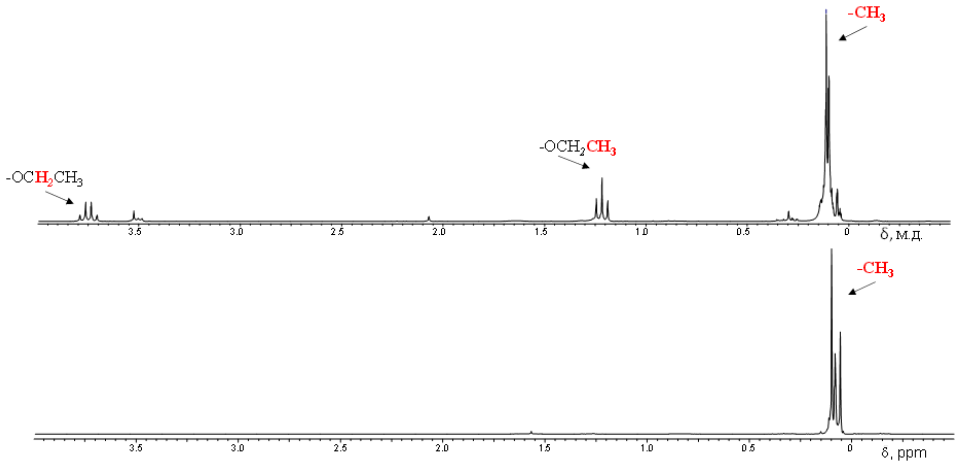

Figure S1. ^{1}H NMR spectra of the reaction mixture in 5 (top) and 9.5 (bottom) h after the reaction beginning for product 2 (Table 1, entry 2).

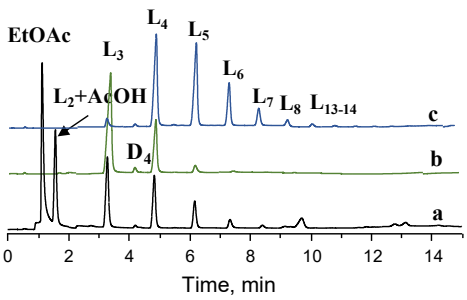

Figure S2. GLC curves of the reaction mixture before distillation (***a***), fractions of oligodimethylsiloxanes with boiling points ranging within 130–300 °C (***b***), and distillation bottoms (***c***). D_4 is $[Me_2SiO]_4$, L_n is Me_3SiO-$[Me_2SiO]_{(n-2)}SiMe_3$.

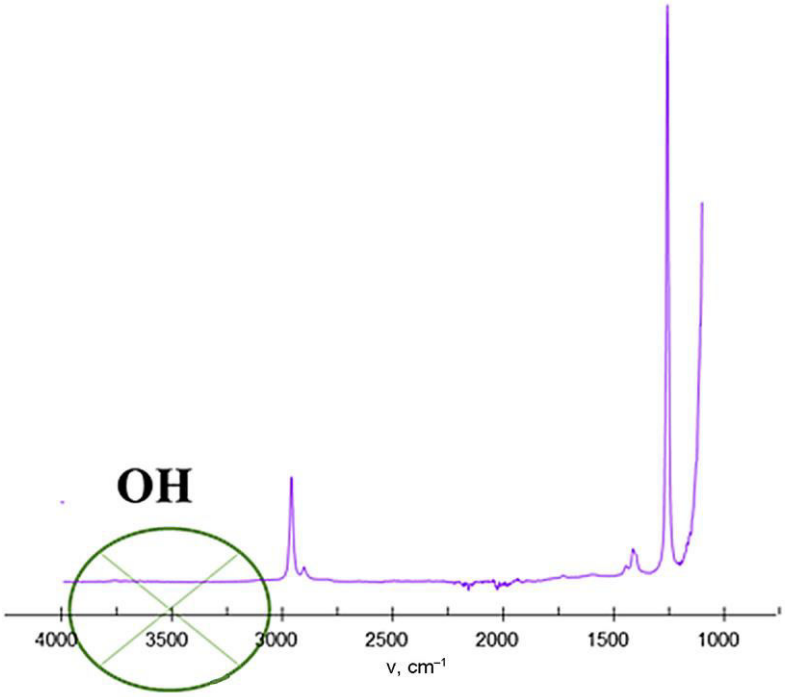

Figure S3. IR spectrum of the selected oligodimethylsiloxane.