Electronic supplementary information

HYDROLYTIC POLYCONDENSATION OF MONOSODIUMOXY(METHYL)(DIETHOXY)SILANE UNDER CONDITIONS OF AN EXCESS OF WATER

A. A. Nesterkina,[a] M. A. Obrezkova,*[b] and A. M. Muzafarov[a,b]

[a] *Enikolopov Institute of Synthetic Polymeric Materials, Russian Academy of Sciences, ul. Profsoyuznaya 70, Moscow, 117393 Russia*
[b] *Nesmeyanov Institute of Organoelement Compounds, Russian Academy of Sciences, ul. Vavilova 28, str. 1, Moscow, 119334 Russia*

Experimental section

General remarks

Sodium hydroxide (reagent grade, Spektrkhim), sodium sulfate (OOO Komponent-Reaktiv, Russia), chloro(dimethyl)vinyl silane (98%, ABCR, Germany) were purchased from commercial sources and used without additional purification. The work was concerned with triethoxy(methyl)silane (98%, OOO Silan, Russia), pyridine (reagent grade, Spektrkhim), and organic solvents, including toluene, ethanol, propan-2-ol, and butan-1-ol. All reagents were processed according to the standard methods [1, 2]. Triethoxy(methyl)silane was distilled prior to syntheses. The organic solvents and pyridine were dried by refluxing over CaH_2 and then distilled under an argon atmosphere. The synthesis of sodiumoxy(methyl)(diethoxy)silane was carried out according to the published procedure [3].

Analysis and general methodology

The GPC analysis was performed on a chromatographic system including an LC-10ADvp high-pressure pump (Shimadzu, Japan), a Smartline RI 2300 refractive index detector (KNAUER, Germany), and a JETSTREAM 2 PLUS thermostat (KNAUER, Germany). The temperature was 40 ± 0.1 °C, the eluent was toluene + 2% of THF, the flow rate was 1.0 mL/min. The columns in use were Phenogel 5 μm columns (300 × 7.8 mm, Phenomenex, USA) with the pore sizes of 10000 Å. The column calibration was performed relative to the Agilent polystyrene standards (USA). The chromatograms were processed and the molecular weight characteristics were calculated using MultiChrom software for Windows version 1.6. (Ampersend, Russia)

The ^{1}H NMR spectra were recorded on a Bruker WP 250 SY spectrometer (Germany). The solvent was $CDCl_3$. The spectra were processed with the ACD LABS program package.

Syntheses

General method for the synthesis of polysodiumoxy(methyl)siloxane. A 30% solution of sodiumoxy(methyl)(diethoxy)silane in a dry solvent (ethanol, propanol-2-ol, or butan-1-ol) was placed into a single-necked round-bottom flask equipped with a dropping funnel. Then an excess of water (from 25% to 100%) was added dropwise upon stirring. After the addition of water (5 h), the reaction mixture gelled. The solvent was removed using an oil pump (1 Torr).

Polysodiumoxy(methyl)siloxane was blocked using the method described in Ref. [4]. ^{1}H NMR ($CDCl_3$): δ H: 5.69–6.21 (m, 3H, $SiCH=CH_2$), 0.05–0.17 (m, 9H, $SiCH_3$) ppm.

References

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