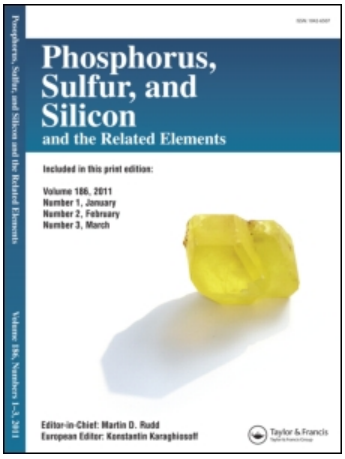

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SYNTHESIS AND CHARACTERIZATION OF (7,10-DIPHENYL FLUORANTHENE-8-)SILICON COMPOUNDS

Zonglin Liu[a]; Hongfang Lu[a]
[a] Department of Chemistry, Shandong University, Jinan, Shandong, P.R. China

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SYNTHESIS AND CHARACTERIZATION OF (7,10-DIPHENYL FLUORANTHENE-8-)SILICON COMPOUNDS

ZONGLIN LIU* and HONGFANG LU

Department of Chemistry, Shandong University, Jinan, Shandong, 250100 P.R. China

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(7,10-diphenylfluoranthene-8-)heptamethylcyclotetrasiloxane (DFHMCTS) and (7,10-diphenyl-fluoranthene-8-) triethoxysilane(DFTES) were synthesized via Diels-Alder reaction of 7,9-diphenyl-8H-cyclopentacena phthylene-8-one (DCPAO) with vinyl-heptamethylcyclotetrasiloxane (VHMCTS) and vinyl-triethoxysilane(VTES) respectively by using diphenylether as solvent. The structures of the two new products have been characterized by IR, UV, ^{1}HNMR and elememental analysis.

Keywords: (7,10-diphenylfluoranthene-8-)heptamethylcyclotetrasiloxane; (7,10-diphenylfluoranth-ene-8-)triethoxysilane; Diels-Alder reaction

INTRODUCTION

Some polysiloxanes containing polyphenylphenyl, condensed rings, (difuryldimethyl)phenyl or (diphenyl-dimethyl)phenyl have been synthesized and show good heat resistance.[1,2,3,4] The syntheses and characteristics of polysilanes containing substituted fluoranthene groups have also been described.[5] However, the low molecular weight organosilicon compounds containing diphenylfluoranthene group have not been reported. This article will report the synthesis and characterization of (7,10-diphenylfluoranthene-8-)triethoxysilane(DFTES) and (7,10-diphenyfluoranthene-8-)heptamethylcyclotetrasiloxne(DFHMCTS). We choose 7,9-diphenyl-8H-cyclopentacenaphthylen-8-one(DCPAO) as the diene, vinyltriethoxysilane (VTES) and vinyl-heptamethylcyclotetrasiloxane (VHMCTS) as

*Corresponding author.

the dienophiles to conduct Diels-Alder reaction respectively. As the experimental results we have obtained DFTES and DFHMCTS. At the same time we have characterized them with IR, UV, ^{1}HNMR and element analysis.

EXPERIMENT

General

Melting points were determined on an X_4 melting point apparatus and uncorrected. ^{1}HNMR spectra were recorded on a FX-90Q spectrometer in deuterochloroform ($CDCl_a$). IR spectra were measured in the region of 400–4000 cm^{-1} using potasium bromide disk method with a Nicolet-5DX spectrometer. UV spectra were examined on a UV-240 spectrophotometer with 0.4%(w/w) product solution in chloroform. Element analysis were performed by the institute of Element Organic Chemistry, Nankai University.

Materials

7,9-diphenyl-8H-cyclopentacenaphthylene-8-one(DCPAO) was prepared according to the procedure outlined in reference[6] and its mp determined was 288.5–290.5°C (liter.290°C). Vinyl-triethoxysilane was obtained from the Institute of Harbin Chemical Industry. Vinyl -heptamethylcyclotertrasiloxane (VHMCTS) was prepared according to the procedure outlined in reference [7] and distilled before used with a high efficiency fractionator (Perkin Elmer, USA) to obtain 54–56°C/8mmHg fraction. The thermometer was uncorrected.

Synthesis of (7,10-diphenylfluoranthene-8-)triethoxysilane (DFTES).

3.80g (0.02mol) of VTES, 3.65g (0.01mol) of DCPAO and 45ml of diphenyl ether were introduced into a 100mL four-necked round bottom flask fitted with a mechanical stirrer, a reflux condensor connected to a drying tube of calcium chloride, a nitrogen inlet tube and a thermometer. The reaction mixture was heated under an atmosphere of dry nitrogen with stirring and appeared deep blue. While the blue mixture was refluxed for 3h, the colour changed into red. Stirring and heating were continued for 5h so that the reaction was completed. After evaporating the diphenyl ether under reduced pressure, the residual red liquid was chromatographed on an Al_2O_3 column using petroleum ether as the developing agent. Removing the volatiles, 3.01g of DFTES as an orange solid

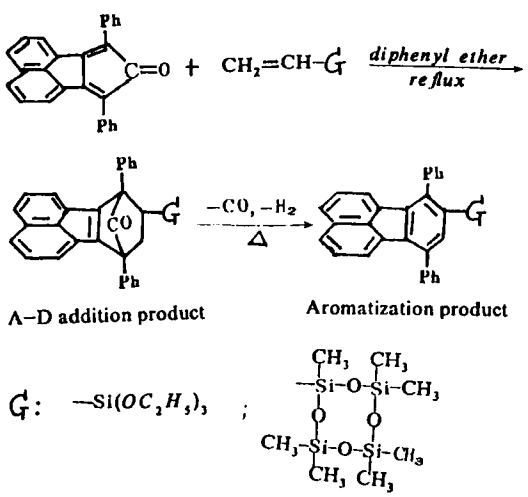
was obtained. The yield was 58.3%, mp 46.0–48.0°C. The main peaks in the IR(KBr) were 3055, 2984, 1589, 1485, 1431, 1237, 1103, 1078, 960, 826, 756cm^{-1}. ^{1}HNMR (δ, ppm): 1.02–1.20 [9H, t, Si(OCH_2CH_3)], 3.51–3.76 [6H, q, Si(OCH_2CH_3)], 6.40–7.80 [17H,m, (7,10-diphenylfluoranthene-8-)]. UV_{max} (nm): 250, 300, 380. Elemental Analysis, Calcd., For $C_{34}H_{32}O_3Si$: C, 79.03, H, 6.24; found: C, 79.87, H, 6.30.

Synthesis of (7,10-diphenylfluoranthene-8-)heptamethylcyclotetrasiloxane (DFHMCTS).

3.56 g (0.01 mol) of DCPAO, 3.08 g (0.01 mol) of VHMCTS and 30 ml of diphenyl ether were introduced into round bottom flask with the same equipment as DFTES. The synthetic procedure was the same as that of DFTES. The product obtained using chromatograph was 3.61 g tangerine colored solid. Recrystallization of the solid in refluxing ethanol with cooling afforded 3.42 g (yield 54.0%) yellow crystals, whose mp was 126 −127°C. The main peaks of IR(KBr) were 3052, 2961, 1430, 1260, 1073, 864, 804, 774, 701. ^{1}HNMR(δ, ppm): 0.05(21H,m,$SiCH_3$), 6.50–7.70[17H,m,(7,10-diphenyl fluoranthene-8-)]. UV_{max}(nm): 250, 285, 380. Elemental analysis, calcd. for $C_{35}H_{38}O_4Si_4$:C, 66.19,H,6.03;found: C,66.15,H,6.27.

RESULT AND DISCUSSION

Two new compounds above were prepared as follows:



In the literature[2], polysiloxanes with (7,10-diphenylfluoranthene-8-) group were synthesized via Diels-Alder reaction of polyvinyl siloxane with acecyclone using α-chloronaphthalene as the solvent. However, in the present paper, diphenyl ether was used to replace α-chloronaphthalene as the solvent the experimental results demonstrate that diphenylether is an efficient solvent for the Diels-Alder reaction of vinylsilane with 7,9-diphenyl-8H-cyclopentacenaphthylen-8-one, and that not only the Diels-Alder reactions can be carried out but also the product obtained can be continuously aromatized into (7,10-diphenylfluoranthene-8-) silicon compounds. Since the liquid polysiloxanes with (7,10-diphenylfluoranthene-8-) group possess good heat resistance and at the same time, some similar compounds, such as (2,3,4,5-tetraphenyl)phenyltriethoxysilane [8] and (2,5-dimethy-3,4-diphenyl)phenyl silicon compounds[9] can be used as cross linkers on room-curable silicone rubber or as monomers of polysiloxane, we can assume that (7,10-diphenylfluoranthene-8-)silicon compounds are important in silicon chemistry and macromolecular science.

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