UDC 546.287 + 543.422.8

1,1,5,5-TETRAMETHYL-3,3,7,7-TETRAPHENYLCYCLOTETRASILOXANE AND 1,1,3,3-TETRAMETHYL-5,5,7,7-TETRAPHENYLCYCLOTETRASILO XANE

K. A. Andrianov, S. E. Akuskina, and L. N. Guniava

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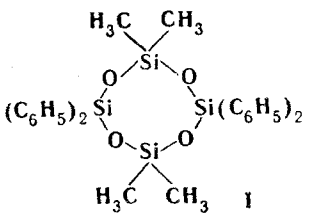
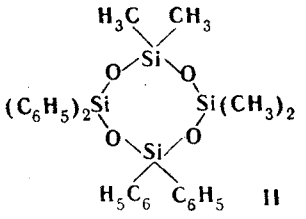
1,1,5,5-Tetramethyl-3m3,7,7-tetraphenylcyclotetrasiloxane (I) and 1,1,3,3-tetramethyl-5,5,7,7-tetraphenylcyclotetrasiloxane (II) are not described in the literature. Vacuum-distillation of the products of



I



II

joint hydrolysis of dimethyldichlorosilane and diphenyldichlorosilane gave a cut bp 230–245° C (1.5 mm), from which were crystallized out successively two isomers of tetramethyltetraphenylcyclotetrasiloxane, which were crystalline compounds with mps 72 and 126° C after recrystallization from MeOH. Their elementary compositions were identical. Isomer mp 72° C: Found: C 61.45; H 5.81; Si 20.57%. Cl 61.85; H 5.90; Si 20.69%. $C_{28}H_{32}O_4Si_4$: C 61.86; H 5.92; Si 20.61%. The two isomers had identical IR spectra at 1429, 1130, 725 cm^{-1} (C_6H_5), 1410, 1259, 800–814 cm^{-1} (CH_3), and 1080–1020 cm^{-1} (Si–O–Si).

X-ray analysis* reveals that the isomers have different crystal structures. The crystals of tetramethyltetraphenylcyclotetrasiloxane mp 72° C were colorless prisms. The elementary cell parameters were a-12.62± 0.06 Å, b-26.19 ± ± 0.01 Å, c-9.13± 0.01 Å, β = 96.48°. The crystals belonged to the monoclinic system, and the space group was P2/n. Number of molecules per cell z = 4, d_{found} 1.15, d_{calc} 1.17 v = 3582 A [3].

The crystals of tetramethyltetraphenylcyclotetrasiloxane, mp 126°C, had the following elementary cell parameters: a–11.05 Å, b–8.33 Å, c–17.47 Å. They belonged to the triclinic system and had angles: α = 146°48′, β = 118°58′, γ = 59°0.8′. The space group was P1. Number of molecules per cell 1. d_{found} 1.18°, d_{calc} 1.20, v = 750 Å [3]. The crystals have a center of symmetry which coincides with the center of symmetry of the ring molecule.

This gives grounds for assigning the symmetric structure I to the isomer mp 126°C.

The crystals mp 72° C lack a center of symmetry, and probably the ring molecule has the unsymmetric structure II.

22 June 1965

Institute of Heteroorganic Compounds, AS USSR

UDC 547.895 + 542.953

SYNTHESIS AND ACIDOCHROME CONDENSATION OF N-BENZYLOYL-1,2,3,4-TETRAHYDROQUINOLINE

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Khimiya Geterotsiklicheskikh Soedinenii, Vol. 2, No. 6, pp. 944–945, 1966

Acidchrome condensation of N-substituted amides of hydroxycarboxylic acids has been studied mainly with aromatic compounds [1].

It is now shown that the reaction can also be extended to heterocyclic compounds. Thus reaction of 1,2,3,4-tetrahydroquinoline (1) with the acid chloride of monoethyloxalate gave N-ethoxalyl-1,2,3,4-tetrahydroquinoline (II), and

* The X-ray analysis was carried out by Yu. T. Struchkov's team in the X-ray analysis laboratory of this Institute.