$\frac{2-[N,N-Bis(trimethylsily1)]amino-4,5-benzo-1,3,2-dioxaphospholane (I)}{g catechol chlorophosphite was added dropwise with stirring to an ethereal solution of 18.3 g of the sodium salt of hexamethyldisilazane cooled to -20°C. The mixture was warmed to ~20°C and maintained for 12 h. After separation of the precipitate and removal of the solvent, distillation gave 16.3 g (55%) (I), bp 78°C (0.05 mm), np²⁰ 1.5190, d₄²⁰ 1.0601, ³¹P 174 ppm. Found, %: C 48.53; H 7.33; P 10.14; Si 18.32. C₁₂H₂₂NO₂PSi₂. Calculated, %: C 48.16; H 7.35; P 10.36; Si 18.69.$

CONCLUSION

The reaction of 2-[N,N-bis(trimethylsily1)]amino-4,5-benzo-1,3,2-dioxaphospholane with acetyl and benzoyl chlorides leads to the formation of silicon-containing diazadiphosphetidines.

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SYNTHESIS OF NEW ORGANOSILICON AND ORGANOGERMANIUM DERIVATIVES OF DIALLYLISOCYANURATE AND CYANURIC ACID

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In a continuation of work on the synthesis and investigation of the properties of heteroorganic symm-triazines [1] and in a search for new biologically active compounds and inhibitors of the nitrification of nitrogen fertilizers, we are the first to report the preparation of a series of derivatives of diallylisocyanurate and cyanuric acid containing disilanyl, disiloxane or carbofunctional organogermanium fragments. Thus, the heterogeneous reactions of the silver salt of diallylisocyanurate with pentamethylchlorodisilane or β trialylgermylpropionyl chloride in ether or benzene solution in a sealed ampule at ~20°C over 70-80 h with rapid stirring (method A) gave high yields of the corresponding pentamethyldisilanyldiallylisocyanurate (I), β -(triethylgermyl)- (II) and β -(tributylgermyl)propionyldiallylisocyanurates (III).

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The use of 1,2-dichlorotetramethyldisilane or 1,3-dichlorotetramethyldisiloxane as the starting chlorine-containing heteroorganic compound led under similar conditions to the formation of bis(diallylisocyanurate) derivatives (IV) and (V).



The cesium and rubidium salts of diallylisocyanurate may be used for the preparation of organosilicon compounds (I), (IV) and (V) but the yields of the desired compounds are reduced. The replacement of diallylisocyanurate salts by trimethylsilyldiallylisocyanurate similarly to our previous procedure [2] is a convenient preparation method. In this case, the reagents are heated at 100-160°C for 1 h with gradual evacuation from 760 to 1 mm Hg (method B). The yields of (I)-(V) are 80-98%.

Tris(pentamethyldisilanyl)cyanurate (VI) was also obtained for the first time from the trisilver salt of cyanuric acid or tris(trimethylsilyl)cyanurate and pentamethylchlorodisilane using methods A and B.



 $M = Ag, Me_3Si.$

Products (I)-(VI) are colorless, viscous or immobile liquids which were identified by elemental analysis and IR spectroscopy. Organosilicon symm-triazines (I), (IV) and (V) by analogy with reported derivatives [1, 2] may be classified as ammelide heterocycles containing fragments with Si-O bonds. Their IR spectra have bands (ν , cm⁻¹): 3080 (allyl), 2940, 2880 (alkyl CH), 1690-1670 (C=O), 1450, 1430, 1410, 780 (ring vibrations of C-N bonds), 1260-1240, 840-820 (Me₃Si). In addition, the IR spectrum of (V) has a band for the SiOSi fragment at 1090 cm⁻¹. The cyanurate structure of (VI) was unequivocally indicated by the lack of bands for CO groups and the presence of band (ν , cm⁻¹): 2950, 2890 (Alk CH), 1540, 1400, 820 (vibrations of the C-N bonds of the cyanurate ring), 1245, 830 (Me₃Si), 1130 (SiOC). The IR spectra of (II) and (III) indicate their isocyanurate structure and contain the following major bands (ν , cm⁻¹): 3080 (allyl), 2980-2840 (Alk CH), 1800-1790 (acyl C=O), 1720-1690 (ring C=O), 1450-1440, 1410-1400, 760-755 (C-N bonds or the isocyanurate-type ring), 1340, 1250-1210 (δ -CH₂, CH₃), 1010-990, 965-960, 700 (Et₃Ge, Bu₃Ge), 580, 540 (GeC). Products (I) and (IV)-(VI) are very vigorously hydrolyzed by atmospheric moisture, while acyltriazines (II) and (III) are significantly more stable toward hydrolysis.

EXPERIMENTAL

The IR spectra were taken neat on a Perkin-Elmer 577 spectrophotometer between CsI windows. <u>Method A</u>. A 1:1 mixture of 3.82 g silver salt of diallylisocyanurate, 2.01 g pentamethylchlorodisilane and 30 ml dry ether was stirred for 72 h in a sealed ampul at ~20°C. Filtration gave 2.15 g of a mixture of AgCl and unreacted starting salt. The solvent was distilled off and the residue was distilled in vacuum to give 2.46 g (60%) (I) as a colorless viscous liquid, bp 145-146°C (3 mm), nD^{20} 1.4925. Found, %: C 49.33; H 7.97; N 11.98; Si 17.03. $C_{14}H_{25}N_{3}O_{3}Si_{2}$. Calculated, %: C 49.52; H 7.42; N 12.37; Si 16.54.

<u>Method B.</u> A 1:1.5 mixture of 2.21 g trimethylsilyldiallylisocyanurate and 1.20 g 1,3dichlorotetramethyldisiloxane was heated in a distillation flask for 10 min at 100-125°C, 1 h at 140°C, distilling out the Me₃SiCl formed. The residue was heated for 30 min at 200°C (3 mm) for the complete removal of Me₃SiCl and excess $CIMe_2SiOSiMe_2Cl$ to yield 2.12 g (98%) (V) as a colorless, sticky liquid, nD^{20} 1.5120. Found, %: 48.16; H 6.13; N 15.24; Si 10.35. $C_{22}H_{32}N_6O_7Si_2$. Calculated, %: C 48.16; H 5.88; N 15.31; Si 10.24. Analogously, methods A and B were used to prepare (II) in 72% yield, (III) in 97% yield, (IV) in 68% yield, and (VI) in 75% yield. The nD^{20} values were 1.5030 for (II), 1.4945 for (III), 1.5300 for (IV), and 1.4732 for (VI). For (VI): bp 157-158°C (3 mm), d_4²⁰ 0.9463, MR 154.22 (calculated 155.70 with a correction for the isocyanurate ring [3]).

CONCLUSION

The reactions of the silver, cesium or rubidium salts of diallylisocyanurate and of trimethylsilyldiallylisocyanurate with pentamethylchlorodisilane, 1,2-dichlorotetramethyldisiloxane and β -(trialkylgermyl)propionyl chlorides leads to the corresponding organosilicon and organogermanium triazine derivatives. The reaction of pentamethylchlorodisilane with the trisilver salt of cyanuric acid or tris(trimethyl-silyl)cyanurate leads to the formation of tris(pentamethyldisilanyl)cyanurate.

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REACTION OF 1-ARYLSULFAMIDO-2-CHLOROETHANE WITH AMINES

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1-Arylsulfamido-2-chloroethanes are readily obtained by the homolytic and heterolytic addition of N,N-dichloroarylsrylsulfamides to unsaturated compounds [1, 2]. We have established a difference in the behavior of $HN(SO_2Ar)CH_2CHC1R$ (R = H, Ph, CH_2C1 , CN) and $R(ArSO_2 \cdot NH)CHCH_2C1$ (R = Ph, CCl_3) compounds and $HN(SO_2Ar)CH_2CHC1Ccl_3$ compounds toward the action of alkali: the former two types react to form aziridines [2-5], while the latter give dehydrochlorination products [6]. We undertook a study of the action of amines on these compounds since 1-arylsulfamido-2-aminoethanes may be obtained with potential biological activity [7, 8].

2-Chloro-2-phenyl-1-(p-chlorophenylsulfamido)ethane (I) and 1-chloro-2-phenyl-2-(pchlorophenylsulfamido)ethane (II), which are adducts of styrene with N,N-dichloro-p-chlorobenzenesulfamide (DCB) obtained under homolytic and heterolytic conditions, respectively [2] and the adducts of 1,1,3- [5] and 1,1,1-trichloropropenes [6] with N,N-dichlorobenzenesulfamide and DCB were taken as starting reagents. Aniline and diethylamine which differ in their basicities were used in these reactions.

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