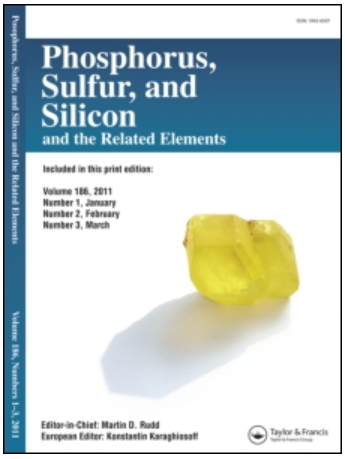

Phosphorus, Sulfur, and Silicon and the Related Elements
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713618290

SYNTHESIS OF ORGANOSILANE CONTAINING FUSED AROMATIC RINGS BY DIELS-ALDER REACTION

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To cite this Article Qiu, Huayu , Feng, Shengyu , Chen, Jianmei and Du, Zuodong(1990) 'SYNTHESIS OF ORGANOSILANE CONTAINING FUSED AROMATIC RINGS BY DIELS-ALDER REACTION', Phosphorus, Sulfur, and Silicon and the Related Elements, 47: 3, 325 — 326
To link to this Article: DOI: 10.1080/10426509008037985
URL: http://dx.doi.org/10.1080/10426509008037985

Phosphorus, Sulfur, and Silicon, 1990, Vol. 47, pp. 325–326
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SYNTHESIS OF ORGANOSILANE CONTAINING FUSED AROMATIC RINGS BY DIELS–ALDER REACTION

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(Received March 23, 1989; Revised June 6, 1989)

Organosilane I containing substituted fluoranthene group has been synthesized by Diels–Alder reaction of vinylmethyldichlorosilane with acecyclone. The structure of I has been determined by elemental analysis, ^{1}H-NMR and IR.

Key words: Fused ring; organosilane; vinylmethyldichlorosilane; Diels–Alder reaction; acecyclone; high-boiling point solvent.

In recent years, some research works on the syntheses of polyphenylphenylsilanes and bis(polyphenylphenyl)silanes have been done by our research group.[1–4] These kinds of compounds have good heat-resistant qualities. They can be used as the high temperature stationary phase of gas chromatograph and additives of heat-vulcanized silicone rubber.[1] Some of them can be used to prepare heat resistant paints.[2] Polysilanes containing the polyphenylphenyl groups may be used for semiconductivity material after contacting with electronic acceptors.[5] The polyphenylphenylsilanes have been synthesized by the Diels–Alder reaction of dienophiles, such as vinyl or phenylethynylsilanes with tetraphenylcyclopentadienone(tetracyclone). The fused ring on the *C*-face is a better substituent than a polyphenylphenyl group.[6] However, no organosilane containing fused rings has been prepared hitherto. In this paper, we describe the synthesis of organosilane containing fused ring, methyl(7,10-diphenylfluoranthene-8-)dichlorosilane I, the first substituted fluoranthene organosilane derivative to be reported.

The organodichlorosilane I could be obtained by Diels–Alder reaction of vinylmethyldichlorosilane with 7,9-diphenyl-8H-cyclopentacenaphthylen-8-one (acecyclone) II[7] in toluene (method A) or α-chloronaphthalene (method B) (see Scheme 1).

Compound I is moisture-sensitive, but is indefinitely stable in an inert atmosphere. It can be hydrolyzed to form organosilanediol.[2] The new compound I give satisfactory elemental analyses.[8] The ^{1}H-NMR spectrum (90 MHz, 298 K, C_6D_6) of I shows only two singlets at about 0.58 ppm (b, 3H, Me) and 7.20 ppm (vb, 17 H, Ph and fused ring). The IR spectrum (KBr pellet) of I shows absorptions attributed to Si—Cl, Si—Me and Si—R (R = Ph or fused ring) bonds.

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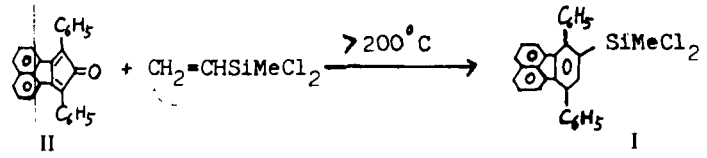
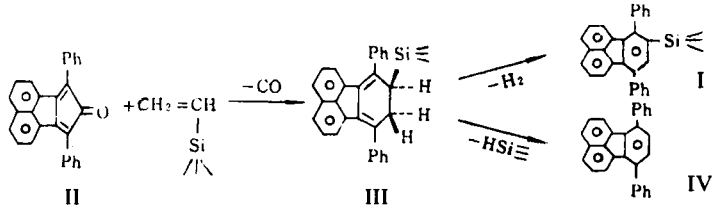


SCHEME 1

The yield of I is lower in method A than in method B. This because the reaction of a vinylsilane with acecyclone is complicated by the fact that the intermediate dihydrofluoranthene III can aromatize to produce I by loss of hydrogen or to produce 7,10-diphenylfluoranthene IV by loss of silane moiety (see Scheme 2).[6]



SCHEME 2

Further studies on the syntheses and properties of other members of this new class of organosilanes are in progress.

EXPERIMENTAL PROCEDURE

Method A: acecyclone II (3.56 g, 0.01 mol) and vinylmethyldichlorosilane (2.82 g, 0.02 mol) were dissolved in 40 mL of toluene and were sealed in an evacuated glass tube at 0°C under reduced pressure. This mixture was heated at 230°C for 24 h until the black color was disappeared. The tube was cooled to room temperature and opened, evaporation of the volatile materials and recrystallization of the residue from toluene under nitrogen afforded 3.32 g (70%, m.p.[9] 161–163°C) of I as a purple black solid.

Method B: acecyclone (3.56 g, 0.01 mol) and vinylmethyldichlorosilane (2.82 g, 0.02 mol) were dissolved in 50 mL of α-chloronaphthalene and were added to the round-bottomed flask. This mixture was heated slowly and then refluxed for 18 h until the colour of the mixture was changed from black to purple black. After this, the mixture was cooled to room temperature, the volatile materials were removed under reduced pressure. The residue was recrystallized from toluene under nitrogen and 4.30 g (90%, m.p.[9] 161–163°C) of I was obtained.

REFERENCES AND NOTES

1. Zuodong Du, Jianhua Chen, Baochuan Shi and Hao Wang, *Polym. Commun.* (China) 174, 1981.
2. Zuodong Du, Jianhua Chen and Baochuan Shi, ibid., 110, 1983.
3. Jianhua Chen, Baochuan Shi and Zuodong Du, *Chem. J. of Chinese Univ.* (China) **7**, 807, 1986.
4. Jianhua Chen, Shengyu Feng and Zuodong Du, ibid., **7**, 1150, 1986.
5. Zuodong Du, Jianhua Chen and Shengyu Feng, *Acta. Polym. Sinica* (China) 81, 1988.
6. Zuodong Du and Shang Yin, ibid., 422, 1988.
7. Synthesis of acecyclone. See: W. Dilthey, I. ter Horst and W. Schommer, *J. Prakt. Chem.* **143**, 189, 1935. Acecyclone was recrystallized from toluene before use in D–A reaction.
8. Calcd. for $C_{29}H_{20}Cl_2Si$: C, 74.51; H, 4.28; Cl, 15.20; Si, 6.00. Found: C, 74.49; H, 4.29; Cl, 15.20; Si, 6.06.
9. The melting point is uncorrected.