

NOTE

Synthesis and Characterization of Dimethoxy Diphenyl Silane

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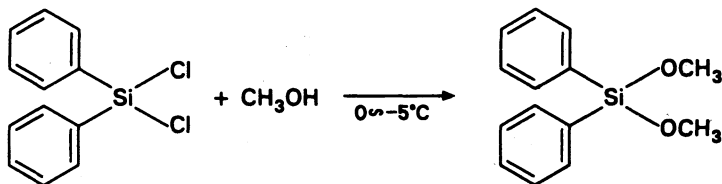
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Dimethoxy diphenyl silane was synthesized by the reaction of diphenyl dichlorine with dry methanol in dry benzene/pyridine at low temperature. It was characterized by ^1H NMR and IR.

Key Word: Dimethoxy diphenyl silane, Diphenyl dichlorine, Dry benzene/Pyridine.

Dimethoxydiphenyl silane is an important intermediate in preparing organo-metallic complexes^{1, 2}; it has been reported by many workers^{3–5}, and its applying field is to be developed farther. This experiment successfully synthesized the title compound using diphenyldichlorine silane reacting with dry methanol without using the literature method;⁶ not only did we get a high yield, but also it has a high purity.

Synthesis of the title compound was carried out under purified argon using Schlenk techniques. Methanol, benzene/pyridine were distilled before use. IR spectrum analysis was recorded on a Mattson Alpha Centauri FT-IR spectrophotometer (KBr crystal plate). ^1H NMR spectrum was recorded on DMX-500 Bio-NMR Lab, USTC.

Preparation of dimethoxy diphenyl silane

A 250 mL Schlenk flask, equipped with a pressure equalising dropping funnel, was charged with a solution of dichlorine diphenyl silane 10 mL (50 mmol) and dry methanol 30 mL in 20 mL benzene; the dropping funnel was charged with 25 mL benzene and 8 mL pyridine and this was added dropwise to the flask at

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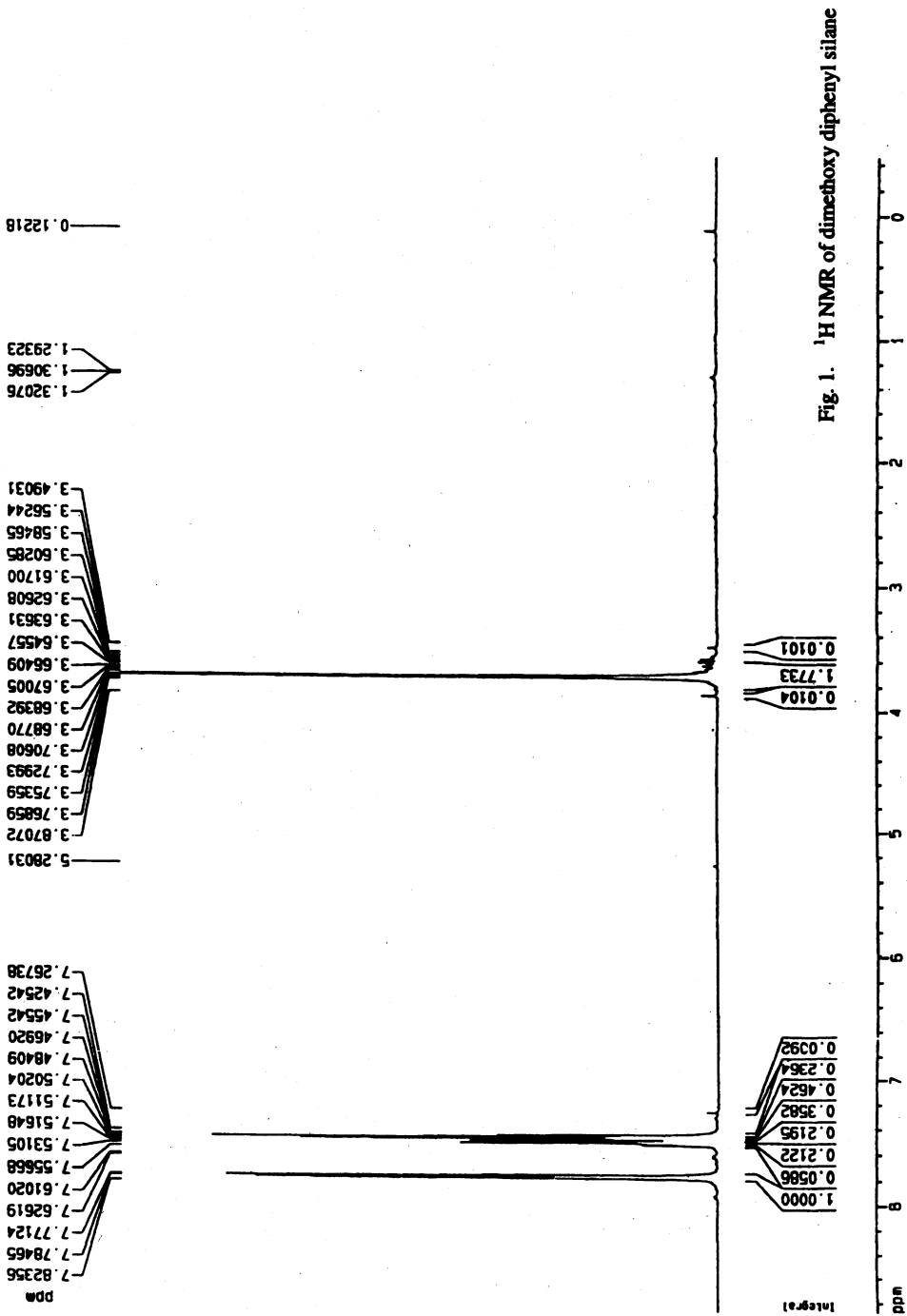


Fig. 1. ¹H NMR of dimethoxy diphenyl silane

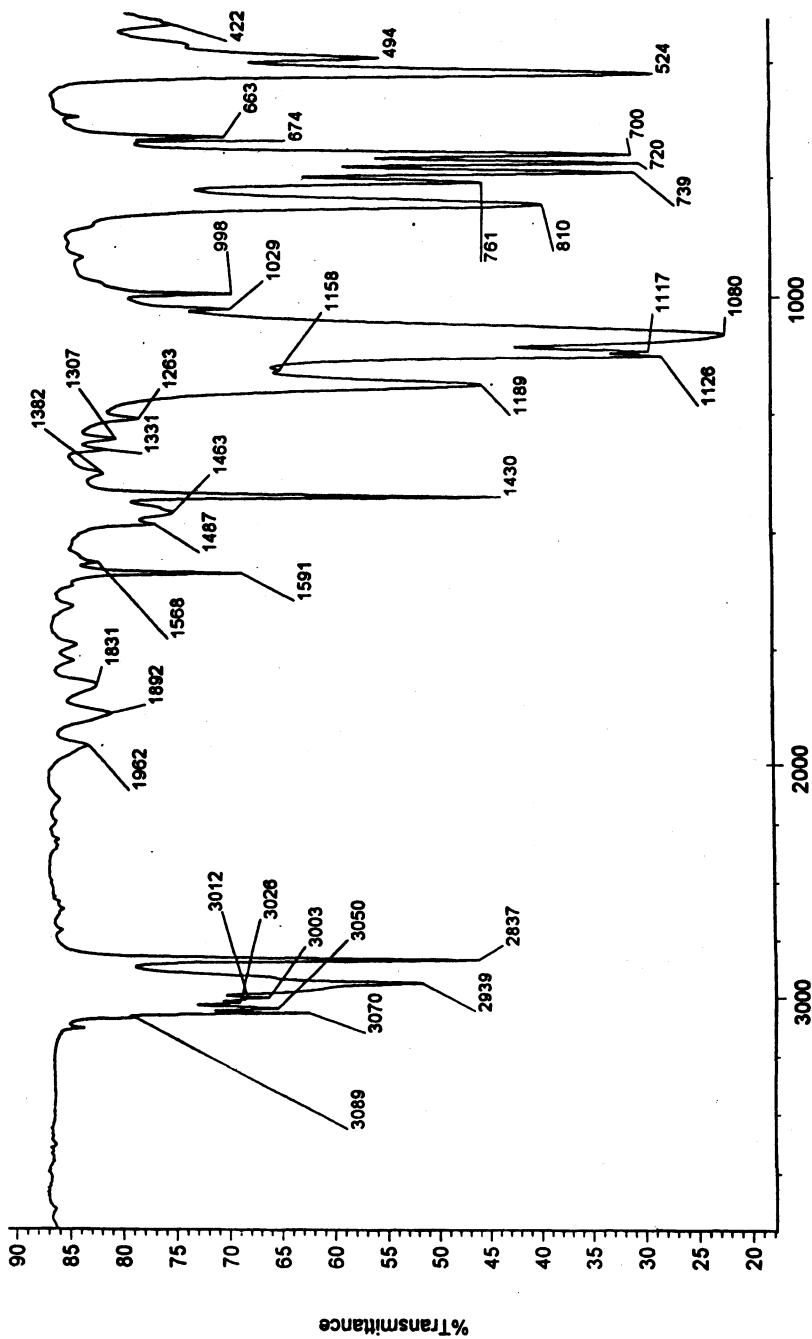


Fig. 2

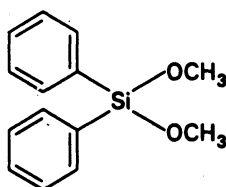
about 0–5°C while stirring; after 20 min, white pyridine hydrochloric acid was obtained. The mixture was allowed to warm up to room temperature, and refluxed at 70–80°C for 5 h. The mixture was carefully filtered at room temperature and the solvent was evaporated *in vacuo* using a rotary evaporator to yield 9.0 g (36.8 mmol, 71%). Vacuum distillation afforded the title compound as a colourless liquid in 64% yield. b.p. 105–108°C (0.2 mbar).

The NMR and IR spectra of dimethoxy diphenyl silane are given in Figs. 1 and 2 respectively.

$^1\text{H NMR}$: 7.77–7.82 (3H, aromatic), 7.42–7.52 (5H, aromatic), 7.61–7.63 (1H, aromatic), 7.53–7.56 (1H, aromatic), 3.56–3.87 (6H, 2CH₃) (Fig. 1).

IR (cm⁻¹): 3089, 3070, 3050, 3026, 3012, 3003, 2939, 2837, 1591, 1487, 1463, 1430, 1189, 1126, 1117, 1080, 1029, 998, 810, 761, 739, 720, 700, 674, 663, 524 and 494 (Fig. 2).

From $^1\text{H NMR}$, it is in agreement with the general formula Ph₂Si(OCH₃)₂, from IR spectra data, we can see that approximately 3000, 1591 and 1481 cm⁻¹



are the characteristic absorptions of phenyl; 2939 and 2837 cm⁻¹ also exhibit the characteristic absorption of methoxy group.

From the above discussion, the proposed structure for the title compound is:

REFERENCES

1. Iwao Ojima, Tetsuo Kogure, Mitsuru Nihonyanagi, Kono Hiromichi, Shinichi Inaba and Yoichiro Nagai, Sagami Chem Res. Cent. Sagamihara (Japan), *Chem. Lett.*, **5**, 501 (1973).
2. M. Cerny, *Collect. Czech. Chem. Commun. (Eng.)*, **40**, 26428-8 (1975).
3. Robert J.P. Corriu and Felix C.R. Larcher, *Hebd. Seances Acad. Sci. (Fr.), Ser. C*, **279**, 1077 (1974).
4. Robert C Antonen, Ger. Offen, 2, 106, 129 (C1. 08g) (19 Aug 1971); US Appl., 16 pp. 12 Feb. (1970).
5. K.D. Goyal, R.R. Gupta and R.L. Mital, *J. Phys. Chem.*, **76**, 1579 (1972).
6. Yoichiro Nagai, Iwao Ojima and Tetsuo Kogure, Japan Kokai 74 42, 615 (Cl, 16, B941) (22 Apr. 1974), Appl. 72 84, 094, 6 pp. (24 Aug 1972).