

ORGANOSILICON COMPOUNDS. LXXVI.\*  
SYNTHESIS OF SOME SILYL  
SUBSTITUTED PHENYLDIMETHYLSILANES

F. MAREŠ, Z. PLZÁK and V. CHVALOVSKÝ

*Institute of Chemical Process Fundamentals,  
Czechoslovak Academy of Sciences, Prague-Suchdol*

Received June 15th, 1970

A synthesis of phenyldimethylsilanes substituted in *para* position by silyl groups with the Si—F, Si—Cl, Si—Br, Si—OCH<sub>3</sub> and Si—N(CH<sub>3</sub>)<sub>2</sub> bonds is described. It was found that the Si—Cl bond can be replaced by any of the mentioned bonds without affecting the Si—H bond in the same molecule.

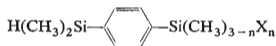
In our previous paper<sup>1</sup> we reported the synthesis of benzoic acids substituted in *para* position by silyl groups. These acids were aimed to be used for the evaluation of the  $\sigma$  constants for different silyl groups. Benzoic acids with the Si—OCH<sub>3</sub>, Si—Cl and Si—Br bonds could not be synthesized. We were compelled, therefore, to choose some other system which would allow us to investigate all the silyl groups of interest. The conditions were met with substituted phenyldimethylsilanes. In this paper we report on the synthesis of these compounds. Even if the benzene ring carries a functional group, the usual method for the formation of the silicon-phenyl bond is a reaction between substituted phenylmagnesium bromide and chlorosilanes.

For the preparation of our model compounds I—V, (*p*-bromophenyl)dimethylsilane should be the most convenient starting compound. In the case of phenylsilanes, however, the Si—H bond reacts<sup>2-4</sup> with the Grignard reagent. Gilman and coworkers<sup>4</sup> observed the reaction of diphenylsilane and phenylsilane with a Grignard agent in tetrahydrofuran. This result is in agreement with the fact that during the reaction of [*p*-(methyldichlorosilyl)phenyl]dimethylsilane (*Ic*) with an excess of methylmagnesium bromide both [*p*-(trimethylsilyl)phenyl]dimethylsilane (*Id*) and *p*-(trimethylsilyl)benzene were isolated in about equivalent yields. We, therefore, tried to prepare the Grignard reagent from (*p*-bromophenyl)dimethylsilane in the presence

\* Part LXXV: This Journal 36, 1682 (1971).

of an excess of chlorosilane assuming that arylmagnesium bromide formed would preferentially react with the silicon-chlorine bond and that in this way the Si—H bond would be protected. Unfortunately, during the reaction which was very slow even under reflux of the solvent, a transfer of chlorine and hydrogen from the Si—Cl and Si—H bonds took place. Therefore not only the expected product *Ia* but also *p*-bis(dimethylsilyl)benzene (*VI*) and *p*-bis(dimethylchlorosilyl)benzene (*VII*) were isolated. This interchange of hydrogen and chlorine is not affected by heating of [*p*-(dimethylchlorosilyl)phenyl]dimethylsilane (*Ia*) (it can be distilled without decomposition) and does not proceed during the formation of the Grignard agent without the presence of chlorosilanes in the reaction mixture or during the reaction of the prepared Grignard agent with chlorosilanes (compare the preparation of the compound *Ic* in Experimental). Chlorosilanes and silanes with the Si—H bond do not react with each other without a catalyst. The only component present in the reaction mixture in addition to chlorosilanes and silanes are magnesium halides. This leads us to assume that magnesium halides could catalyze the exchange of the Si—Cl and Si—H bonds. The reason why the Si—Cl and Si—H bonds are not exchanged for the Si—Br one can be visualized in the difference of the thermodynamic stability of these bonds.

To minimize the formation of by-products the mixture of (*p*-bromophenyl)dimethylsilane and dimethyldichlorosilane was dropwise added to powdered magnesium in ether. This procedure enabled us to decrease the formation of the compounds with a higher content of the Si—Cl bonds, but the products with a higher content of the Si—H bonds were still formed in an appreciable amount (compare the preparation of *Ib* and *Ic*). This method is, however, not suitable for the synthesis of *Ic* because tetrachlorosilane reacts with powdered magnesium. We, therefore, had to prepare first *p*-(dimethylsilyl)phenylmagnesium bromide and then allowed it to react with an excess of tetrachlorosilane. Even though powdered magnesium in 200% excess was used and the Grignard reaction was started with ethyl bromide, the reac-



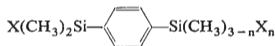
*Ia-d*,  $n = 0-3$   $\text{X} = \text{Cl}$

*IIa-c*,  $n = 1-3$   $\text{X} = \text{OCH}_3$

*IIIa-c*,  $n = 1-3$   $\text{X} = \text{Br}$

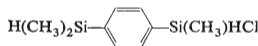
*IVa-c*,  $n = 1-3$   $\text{X} = \text{F}$

*Va-c*,  $n = 1-3$   $\text{X} = \text{N}(\text{CH}_3)_2$

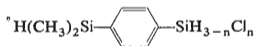


*VI*,  $n = 1$   $\text{X} = \text{H}$

*VII*,  $n = 1$   $\text{X} = \text{Cl}$



*VIII*



*IX*,  $n = 1$

$\text{X}$ ,  $n = 2$

tion never gave high yield of the expected Grignard agent. The yield after the reaction with silicon tetrachloride was only 26.8%. It is, however, important that besides undistillable residue, which formed a substantial part of the reaction mixture, the distillable portion was a mixture of the starting compounds (isolated 21.4%) and the expected product. This result would suggest that a short contact of the silanes with magnesium halides at room temperature does not lead to a redistribution of the Si—Cl and Si—H bonds.

The methoxy derivatives *Ia-c* were prepared from the chlorosilanes *Ia-c* by treatment with absolute methanol and pyridine in benzene according to the usual method. The methoxy derivatives were reacted with acetyl bromide, yielding corresponding bromosilanes *IIIa-c*. A similar selective exchange of the Si—OCH<sub>3</sub> bond for the Si—Cl one, without affecting the Si—H bond, was accomplished with benzoyl chloride<sup>5</sup>. With acetyl bromide the reaction proceeded under very mild conditions, being a reverse reaction at least in the case of dimethoxysilane *Iib*. After the exchange of one methoxy group *Iib* it was necessary to distil off the formed methyl acetate and add again a great excess of acetyl bromide. Only then the reaction could be completed. The Si—H bond did not react at all.

The tribromoderivative *IIIc* could not be obtained by the described method; apparently because of the steric hindrance. *p*-(Dimethylsilyl)phenylmagnesium bromide was reacted with tetrabromosilane to give, after purification of the crude material, only a very low yield of the product *IIIc* (95% purity).

Fluorosilanes are usually prepared by the reaction of methoxysilanes with boron trifluoride. This method cannot be applied in our case because a cleavage of the Si—H and Si—Ph bonds takes place. We, therefore, made use of the method of Müller and coworkers<sup>6</sup> who employed sodium phenylpentafluorosilicate as a fluorinating agent. The chlorosilanes *Ia-c* were mixed with equivalent of sodium phenylpentafluorosilicate in ether. In all cases the Si—H bond remained completely unchanged, in contrast to the results of Müller and coworkers<sup>6</sup>. It is necessary to stress that sodium pentafluorosilicate used was only of 95% purity. Therefore, in all experiments there was a slight excess of the Si—Cl bonds over the amount of the active fluorine. As a result the crude products were contaminated with the starting chlorosilanes. These can easily be removed by distillation.

The chlorosilanes *Ia-c* also served as starting compounds for preparation of dimethylaminoderivatives *Va-c*. Treatment of one equivalent of chlorosilane with two equivalents of dimethylamine in benzene or petrolether gave high yields of dimethylaminosilanes *Va-c* and the precipitate of dimethylamine hydrochloride. Only for complete exchange of all of the chlorine atoms in trichlorosilane *Id* a higher temperature and pressure had to be employed.

In summary the exchange of chlorine in chlorosilanes *Ia-c* for other functional groups can be made in good yields without affecting the Si—H bond.

## EXPERIMENTAL

(*p*-Bromophenyl)dimethylchlorosilane. *p*-Bromophenylmagnesium bromide prepared from magnesium (115 g, 4.58 gatom) and *p*-dibromobenzene (944 g, 4.0 mol) in 3 l of ether was dropwise added to a solution of dimethyldichlorosilane (1032 g, 8 mol) in 500 ml of ether. The salts formed were decanted and then continually extracted by ether for two days. Distillation afforded the product (650 g, 65%, b.p. 125–127°C/12 Torr, lit.<sup>7</sup> b.p. 133.5°C/18 Torr) containing 15% of dibromobenzene according to glc analysis.

(*p*-Bromophenyl)dimethylsilane. (*p*-Bromophenyl)dimethylchlorosilane was reduced with lithium aluminium hydride (36 g, 0.95 mol) in 600 ml of ether. The reaction mixture was decomposed by pouring it onto a mixture of crushed ice and conc. hydrochloric acid. After the usual work-up the crude product was cooled to –30°C and the crystals of dibromobenzene were filtered off. Fractional distillation afforded 543 g (2.52 mol) of the product, b.p. 108–112°C/23 Torr, containing 5% of dibromobenzene.

*[p*-(Dimethylsilyl)phenyl]methylchlorosilanes Ia–d

*[p*-(Dimethylsilyl)phenyl]dimethylchlorosilane (Ib). A. To a mixture of ether (150 ml), powdered magnesium (24 g, 1 gatom) and dimethyldichlorosilane (258 g, 2 mol) a solution of (*p*-bromophenyl)dimethylsilane (90.6 g) in ether (450 ml) was dropwise added at such a speed that the reaction mixture was kept under reflux. After the ether and the unreacted dimethyldichlorosilane were distilled off the crude product was analyzed by glc. Four components were present; three of them in about equivalent amounts and the fourth one, having the same retention time as the starting compound, only as a trace. Fractional distillation afforded first only two fractions; the first one, *p*-bis(dimethylsilyl)benzene, b.p. 105 to 108°C/12 Torr (24.6 g) and the second one, b.p. 126–133°C/12 Torr (27 g), containing 17.36% of hydrolysable chlorine and according to glpc it was a mixture of two compounds. This mixture after fractional distillation on a 20 TP spinning band column gave [*p*-(dimethylsilyl)phenyl]dimethylchlorosilane, b.p. 78–79°C/1 Torr. For C<sub>10</sub>H<sub>17</sub>ClSi<sub>2</sub> (228.9) calculated: 15.49% Cl; found: 15.62% Cl. The further fraction contained two compounds. One compound crystallized off the mixture. This material was filtered off and recrystallized from petrolether. The NMR spectrum showed the presence of one type of methyl on silicon and *p*-substituted benzene ring. On the basis of the NMR and hydrolysable chlorine analysis the compound was assigned the structure of *p*-bis(dimethylchlorosilyl)benzene. For C<sub>10</sub>H<sub>16</sub>Cl<sub>2</sub>Si<sub>2</sub> (216.9) calculated: 26.94% Cl; found: 26.80% Cl.

B. In this experiment most of dimethyldichlorosilane (19 g, 1.7 mol) together with (*p*-bromophenyl)dimethylsilane (151 g, 0.7 mol) were dissolved in ether (250 ml) and, after starting the Grignard reaction with ethyl bromide, the solution was slowly added to a mixture of magnesium (34 g, 1.4 g atom) and dimethyldichlorosilane (13 g, 0.1 mol) in ether (150 ml). Next day the reaction mixture was worked-up in the same way as in the previous experiment, giving the starting compound (16.7 g) and [*p*-(dimethylchlorosilyl)phenyl]dimethylsilane (74.1 g). The product showed only one peak on glc. In this case *p*-bis(dimethylchlorosilyl)benzene was not detected in the reaction mixture.

*[p*-(Dimethylsilyl)phenyl]methylchlorosilane (Ic). Using the procedure B, the reaction of (*p*-bromophenyl)dimethylsilane (86 g, 0.4 mol), magnesium (19.2 g, 0.8 gatom), and methyltrichlorosilane (191.3 g, 1.2 mol) in ether (500 ml) yielded two fractions; the first one (15.2 g) boiling 30°C/15 Torr (15.2 g) and the second one boiling 30–120°C/0.5 Torr. The second fraction was fractionated on a 20 TP spinning band column and three portions were collected. The first one

TABLE I  
Preparation of [*p*-(Dimethylsilyl)phenyl]methylmethoxysilanes *Ila-c*

Com- pound	Starting chlorosilane mol	Methanol mol	Pyridine mol	B.p., °C/Torr	$n_D^{20}$	Yield %	Formula (m.w.)	Calculated/Found	
								% C	% H
<i>Ila</i>	<i>Ib</i> (0.0299)	0.036	0.037	72-3/0.5	1.4937	96.5	C <sub>11</sub> H <sub>20</sub> OSi <sub>2</sub> (224.4)	58.86 58.82	8.98 9.05
<i>Ilb</i>	<i>Ic</i> (0.0727)	0.165	0.165	71/0.5	1.4872	83.6	C <sub>11</sub> H <sub>20</sub> O <sub>2</sub> Si <sub>2</sub> (240.5)	54.94 54.90	8.38 8.37
<i>Ilc</i>	<i>Id</i> (0.0167)	0.06	0.06	109/2	1.4818	84.1	C <sub>11</sub> H <sub>20</sub> O <sub>3</sub> Si <sub>2</sub> (256.5)	51.52 51.37	7.86 7.89

TABLE II  
Analytical Data and Physical Properties of [*p*-(Dimethylsilyl)phenyl]methyl(dimethylamino)silanes *Ia-e*

Compound	B.p., °C/Torr	$n_D^{20}$	M.w.	Calculated/Found	
				% C	% H
<i>Ia</i>	100/0.5	1.5012	237.3	60.68 00.35	9.77 9.57
<i>Ib</i>	106/0.5	1.5034	266.3	58.58 58.24	0.84 9.76
<i>Ic</i>	102/0.3	1.5058	295.4	56.87 56.75	10.23 10.41

(b.p. 55—60°C/2 Torr, 23 g, no hydrolyzable chlorine) was identified by glpc as the starting compound. The second one (b.p. 60—90°C/2 Torr) contained [*p*-(methylchlorosilyl)phenyl]dimethylsilane VIII as the main compound and [*p*-(dimethylsilyl)phenyl]methylchlorosilane (*c*), as shown by glc and the hydrolyzable chlorine analysis. The third portion (b.p. 99°C/2 Torr, 19.1 g) gave on glc only one peak with the retention time identical with that of [*p*-(dimethylsilyl)phenyl]methylchlorosilane. For  $C_9H_{14}Cl_2Si_2$  (249.31) calculated: 28.45% Cl; found 28.50% Cl.

[*p*-(Dimethylsilyl)phenyl]trichlorosilane Id. A. Using procedure B described in the case of [*p*-(dimethylsilyl)phenyl]dimethylchlorosilane *Ib*, (*p*-bromophenyl)dimethylsilane (86 g, 0.4 mol), magnesium (29 g, 0.82 gatom) and tetrachlorosilane (204 g, 1.2 mol) afforded two fractions, as shown by glc. The first one contained traces of the starting compound and another silane to which, on the basis of the retention time in glc and of the analysis of hydrolyzable chlorine, the structure of [*p*-(dimethylsilyl)phenyl]chlorosilane IX was assigned: calculated 17.65% Cl; found 16.94% Cl. The second fraction (b.p. 80 to 95°C/1 Torr, 27.5 g) contained, according to glc, two compounds. The rectification on a 20 TP spinning band column did not yield two pure products. The purest fraction of a b.p. 83—84°C : 0.7—0.8 Torr (6.05 g) contained about 5% of the lower boiling compound. On the basis of the quantitative glc analysis, elemental analysis and IR spectra of all portions we could show that the lower boiling compound is [*p*-(dimethylsilyl)phenyl]dichlorosilane (*X*) and the higher boiling one is [*p*-(dimethylsilyl)phenyl]trichlorosilane (*Id*).

B. First the Grignard reagent was prepared from (*p*-bromophenyl)dimethylsilane (48.2 g, 0.223 mol) and magnesium (6.43 g, 0.268 gatom) in 200 ml of ether. The reaction was initiated by ethyl bromide. The resulting solution was added into a solution of tetrachlorosilane (95 g, 0.669 mol) in 100 ml of ether. After distilling off the solvents, the residue was analyzed by glc showing the presence of the starting compound and the expected product. A rectification afforded a fraction b.p. 81—82°C/0.5 Torr (16.1 g) which consisted of one peak on glc. For  $C_8H_{11}Cl_3Si_2$  (269.7) calculated: 35.63% C, 4.11% H, 39.44% Cl; found: 35.92% C, 4.00% H, 39.53% Cl.

[*p*-(Dimethylsilyl)phenyl]trimethylsilane (Ia). Into a solution of methylmagnesium chloride, prepared from magnesium (4 g, 0.166 gatom) and methyl chloride in 100 ml of ether, (*p*-(dimethylsilyl)phenyl)methylchlorosilane (14.05 g, 0.0636 mol) was added. The reaction mixture was allowed to stand overnight and the next day worked up. Distillation afforded the fraction of b.p. 60—62°C/0.5 Torr (11.3 g), which partially crystallized. It contained two compounds (glc analysis). The mixture was cooled down and the crystals (4.5 g) filtered off and recrystallized several times from methanol. A compound of m.p. 91°C was obtained. This corresponds to bis-(*p*-trimethylsilyl)benzene<sup>8</sup>. A part of the liquid product was purified by glc and the portion of the shorter retention time was then submitted to a fractional distillation. The first fraction gave only one peak on glc. For  $C_{11}H_{20}Si_2$  (208.5) calculated: 63.38% C, 3.61% H; found 63.24% C, 9.88% H.

#### [*p*-(Dimethylsilyl)phenyl]methylmethoxysilanes IIa—c

A solution of dry methanol and dry pyridine in benzene was added to a solution of [*p*-(dimethylsilyl)phenyl]methylchlorosilane in benzene. The formed salts were filtered off and the solution evaporated in vacuum. Distillation yielded pure compounds. Molar ratios of the compounds used, yields, elemental analysis and physical properties are summarized in Table I.

*[p-(Dimethylsilyl)phenyl]methylbromosilanes IIIa—c*

*[p-(Dimethylsilyl)phenyl]dimethylbromosilane (IIIa)*. The compound *Ila* (10 g, 0.0411 mol) was mixed with acetyl bromide (5.70 g, 0.12 mol) under nitrogen and the mixture was heated 16 hours to 60°C and then allowed to stand 25 hours at room temperature. The volatile portions were distilled off in vacuum. A distillation afforded two fractions b.p. 92–93°C/1 Torr (3.6 g) and 94–96°C/1 Torr (4.0 g) which both — according to the content of the hydrolysable bromine — were proved to be the pure compound *IIIa*. For  $C_{10}H_{17}BrSi_2$  (273.3) calculated: 29.24% Br; found: 29.56% Br.

*[p-(Dimethylsilyl)phenyl]methyl dibromosilane (IIIb)*. A mixture of *[p-(dimethylsilyl)phenyl]methyl*dimethoxysilane (11.2 g, 46.6 mmol) and acetyl bromide (12 g, 98 mmol) was heated to 60°C for 12 hours. The reaction mixture was stripped off acetyl bromide and methyl acetate in vacuum and the rest was distilled. The obtained product contained only 33.21% Br which corresponds to *[p-(dimethylsilyl)phenyl]methyl*methoxybromosilane. The product was therefore mixed with 4 ml of acetyl bromide. After 36 hours standing at room temperature the content of bromine increased only to 33.63%. The silane was therefore dissolved in 25 ml of acetyl bromide and the mixture allowed to stand overnight. Distillation afforded a compound of b.p. 118°C/1.5 Torr (13 g) containing 49.25% of bromine. Distillation on a column yielded the pure silane *IIIb*. B.p. 92–95°C/0.4–0.5 Torr. For  $C_9H_{14}Br_2Si_2$  (338.2) calculated: 47.26% Br; found: 47.06% Br.

*[p-(Dimethylsilyl)phenyl]tribromosilane (IIIc)*. The Grignard reagent, prepared from *p*-bromophenyl)dimethylsilane (21.5 g, 0.1 mol) and magnesium (2.92 g, 0.12 gatom) in 100 ml of ether, was dropwise added to a solution of tetrabromosilane (105 g, 0.3 mol) in 50 ml of ether. The mixture was allowed to stand overnight and then refluxed for three hours. The ether was gradually exchanged for toluene (50 ml). The salts were filtered off. Distillation of the product afforded a fraction of a b.p. 142–147°C/2.5 Torr. For  $C_8H_{11}Br_3Si_2$  (403.1) calculated: 59.40% Br, 0.250% Si—H; found: 57.60% Br, 0.235% Si—H.

*[p-(Dimethylsilyl)phenyl]methylfluorosilanes IVa—c*

*[p-(Dimethylsilyl)phenyl]dimethylfluorosilanes (IVa)*. *[p-(Dimethylsilyl)phenyl]dimethyl*chlorosilane (*Ib*) (10 g, 43.7 mmol) was added to the equivalent amount of finely powdered sodium phenylpentafluorosilicate (5.8 g, 26 mmol) in such an amount of ether to assure good stirring. After one hour of stirring the product was distilled off the salts. The crude product, containing (glc analysis) traces of phenyltrifluorosilane and 15% of the starting chlorosilane was distilled on a column. A fraction b.p. 94°C/10 Torr gave only one peak on glc. For  $C_{10}H_{13}FS_2$  (212.4) calculated: 56.54% C, 8.07% H, 8.9% F; found: 56.80% C, 8.22% H, 8.85% F.

*[p-(Dimethylsilyl)phenyl]methyl difluorosilane IVb*. Analogously as above, a mixture of *[p-(dimethylsilyl)phenyl]methyl*dichlorosilane (*Ic*) (8.42 g, 33.8 mmol) and sodium phenylpentafluorosilicate (8.34 g, 37.4 mmol) in 30 ml of ether yielded after distillation 10.3 g of a crude product which contained a substantial amount of phenyltrifluorosilane. Distillation on a column gave the product of a b.p. 88°C/26 Torr. The structure was confirmed by NMR. The purity was checked by glc. For  $C_9H_{14}F_2S_2$  (216.4) calculated: 49.95% C, 6.52% H; found: 50.04% C, 6.72% H.

*[p-(Dimethylsilyl)phenyl]trifluorosilane (IVc)*. A mixture of *[p-(dimethylsilyl)phenyl]trichloro*-silane (*Id*) (4.0 g, 14.8 mmol), sodium phenylpentafluorosilicate (5.71 g, 21.5 mmol) and ether (20 ml) was stirred for two hours. A distillation yielded the crude product (5.5 g) which contained phenyltrifluorosilane. It was, therefore, purified by distillation on a column, giving the pure product (1.3 g, one peak on glc), b.p. 85°C/45 Torr. For  $C_8H_{11}F_3Si_2$  (220.36) calculated: 43.61% C, 5.03% H; found: 43.63% C, 5.21% H.

*[p-(Dimethylsilyl)phenyl]methyl(dimethylamino)silanes Va—e*

In nitrogen atmosphere, 2.3 equivalents of 2M solution of dimethylamine in dry benzene were added to one equivalent of chlorosilane *Ib—d* (14 200 mmol) in dry benzene. The formed salts were filtered off, the solvent evaporated in vacuum and the crude product was purified by distillation. The pure products were obtained in about 60% yield. The structure of the compounds was confirmed by IR spectra and NMR spectra.

*[p-(Dimethylsilyl)phenyl]tris(dimethylamino)silane (Ve)*. To 30 ml (20.5 g, 0.45 mol) of dimethylamine in 100 ml of cyclohexane were added 12 g (0.044 mol) of *p*-dimethylsilylphenyltrichlorosilane in 30 ml of cyclohexane. Then the precipitated dimethylamino hydrochloride was filtered off. After removal of cyclohexane, the distillation of the residue afforded fraction boiling 108°C/0.5 Torr and containing 12.34% of chlorine. Nine grams of this compound was heated with 30 ml of cyclohexane and 15 ml of dimethylamine in a sealed ampule to 100°C for 6 hours. The usual work-up of the reaction mixture gave the pure product.

The elemental analysis and physico-chemical properties of other *p*-dimethylsilylphenylmethyl dimethylaminosilanes prepared in similar fashion are given in Table II.

## REFERENCES

1. Mareš F., Neudörfel P., Plzák Z., Chvalovský V.: This Journal 35, 2324 (1970).
2. Harvey M. C., Nebergall W. H., Peake Y. S.: J. Am. Chem. Soc. 79, 1437 (1957).
3. Gilman H., Zuech E. A.: J. Am. Chem. Soc. 79, 4560 (1957).
4. Gilman H., Zuech E. A.: J. Am. Chem. Soc. 81, 5925 (1959).
5. Okawara R., Sikigama M.: J. Chem. Soc. Japan, Ind. Chem. Sect. 58, 805 (1955); Chem. Abstr. 50, 11939 (1956).
6. Müller R., Dathe C., Frey H. J.: Ber. 99, 1614 (1966).
7. Lewis D. W., Gainer G. C.: J. Am. Chem. Soc. 74, 2931 (1942).
8. Petrov A. D., Černyšev E. A., Li Guan-Lian: Dokl. Akad. Nauk SSSR 132, 1099 (1960).

Translated by J. Hetflejš.