La cohésion cristalline est assurée en dehors des contacts de van der Waals par l'intermédiaire d'une liaison hydrogène échangée entre H(3) et O(12) de deux molécules voisines (Tableau 2). L'angle de la liaison vaut 158 (10)°. L'arrangement obtenu forme des feuillets le long de l'axe z.

Les résultats sont comparables à ceux trouvés pour la structure de la benzoyl-6 dihydro-2,3 benzoxazole-1,3 one-2 (Mairesse, Boivin, Thomas, Bermann, Bonte & Lesieur, 1984). L'angle dièdre entre les plans benzothiazolinone et benzénique vaut 58 (1)° et il est égal à 54 (1)° entre les plans benzoxazolone et benzénique. Par contre, les deux structures diffèrent par leur arrangement tridimensionnel; en effet, les molécules sont assemblées par paires par un double système de liaison hydrogène dans le cas de la benzoyl-6 dihydro-2,3 benzoxazole-1,3 one-2.

Cette étude structurale a permis de déterminer sans équivoque la position du groupement acyle, la benzoylation du noyau aromatique de la benzothiazolinone s'effectue comme la benzoxazolinone, en *para* de l'atome d'azote, c'est à dire en position 6.

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Structure of Bis[bis(trimethylsilyl)methylene]methoxyphosphorane

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Abstract. $C_{15}H_{39}OPSi_4$, $M_r = 378.79$, monoclinic, $P2_1/n$, $a = 11.814 (6)$, $b = 15.931 (5)$, $c = 13.846 (9)$ Å, $\beta = 106.96 (8)°$, $V = 2492.6$ Å^3, $Z = 4$, $D_x = 1.01$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 0.298$ mm^{-1}, $F(000) = 832$, $T = 293$ K, $R = 0.054$ for 2663 significant reflections. The structure of the tricoordinate phosphorane contains two equivalent bis(trimethylsilyl)methylene moieties and a methoxy group. The phosphorus centre and three neighbouring atoms describe a plane with P—C bond lengths of 1.625 (4) and 1.646 (4) Å and a P—O bond length of 1.599 (3) Å, and a C—P—C angle of 134.0 (2)°.

Experimental. The title compound was prepared in 56% yield using the procedure described by Appel, Dunker, Gaitzsch & Gaitzsch (1984). Clear yellow plate-shaped crystals were grown from a saturated dichloromethane solution at room temperature (295 K). Crystal (irregular shaped fragment) 0.13 × 0.75 × 0.88 mm (Pyrex capillary); Enraf–Nonius CAD-4 diffractometer, graphite-monochromated Mo $K\alpha$ radiation. Lattice constants were obtained from 25 well centred reflections ($\theta > 19°$). Intensities were measured using an $\omega/2\theta$ scan (width 0.8 + 0.35tanθ) over a 2θ range of $2 \le 2\theta \le 50°$ (h,k,l: +14, +19, ±16). Three standard reflections monitored every hour showed no deviations greater than ±2% in intensity. Intensities of 4773 reflections were measured of which 2663 were considered observed [$F_o > 5\sigma(F_o)$]. No absorption correction was made. The structure was solved by direct methods *SHELXS* (Sheldrick, 1985) and refined by full-matrix least-squares procedure, *SHELX76* (Sheldrick, 1976). Scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV) and corrected for anomalous dispersion. All non-H atoms were refined anisotropically. The methyl H atoms were included as a rigid group with

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Table 1. *Final fractional coordinates for* POMe[C(SiMe₃)₂]₂

$$B_{eq} = (4/3)[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ab(\cos\gamma)B(1,2) + ac(\cos\beta)B(1,3) + bc(\cos\alpha)B(2,3)].$$

	x	y	z	$B_{eq}(\text{Å}^2)$
P	0·8939 (1)	0·38515 (7)	0·78248 (9)	2·76
Si(1)	0·6654 (1)	0·35992 (9)	0·6200 (1)	3·49
Si(2)	0·6808 (1)	0·34880 (9)	0·8578 (1)	3·84
Si(3)	1·1182 (1)	0·4250 (1)	0·7318 (1)	4·40
Si(4)	1·0058 (1)	0·23664 (9)	0·7104 (1)	3·97
O	0·9467 (1)	0·4546 (2)	0·8671 (2)	3·78
C(1)	0·7514 (4)	0·3654 (2)	0·7561 (3)	2·89
C(2)	0·7378 (5)	0·4202 (3)	0·5386 (4)	4·74
C(3)	0·5179 (4)	0·4088 (4)	0·6044 (5)	5·34
C(4)	0·6363 (5)	0·2513 (3)	0·5700 (4)	4·98
C(5)	0·5844 (6)	0·2542 (4)	0·8248 (5)	6·35
C(6)	0·7935 (5)	0·3282 (4)	0·9802 (4)	5·54
C(7)	0·5858 (5)	0·4370 (4)	0·8756 (5)	5·66
C(8)	0·9987 (4)	0·3510 (3)	0·7384 (3)	3·05
C(9)	1·1797 (6)	0·3937 (5)	0·6286 (5)	8·22
C(10)	1·2391 (5)	0·4301 (5)	0·8506 (5)	7·14
C(11)	1·0589 (6)	0·5335 (3)	0·6991 (6)	7·44
C(12)	0·9138 (5)	0·1725 (3)	0·7711 (4)	4·87
C(13)	1·1617 (5)	0·1998 (4)	0·7651 (5)	6·61
C(14)	0·9649 (6)	0·2150 (4)	0·5732 (4)	6·66
C(15)	0·8796 (5)	0·5281 (3)	0·8787 (5)	5·41

Table 2. *Bond distances* (Å) *and angles* (°) *for* POMe[C(SiMe₃)₂]₂

P—O	1·599 (3)	P—C(1)	1·646 (4)
P—C(8)	1·625 (4)	Si(1)—C(1)	1·864 (5)
Si(1)—C(2)	1·867 (5)	Si(1)—C(3)	1·863 (5)
Si(1)—C(4)	1·858 (5)	Si(2)—C(1)	1·852 (4)
Si(2)—C(5)	1·864 (5)	Si(2)—C(6)	1·853 (5)
Si(2)—C(7)	1·859 (5)	Si(3)—C(8)	1·862 (4)
Si(3)—C(9)	1·851 (6)	Si(3)—C(10)	1·838 (6)
Si(3)—C(11)	1·870 (5)	Si(4)—C(8)	1·870 (5)
Si(4)—C(12)	1·862 (5)	Si(4)—C(13)	1·870 (5)
Si(4)—C(14)	1·851 (5)	O—C(15)	1·449 (5)

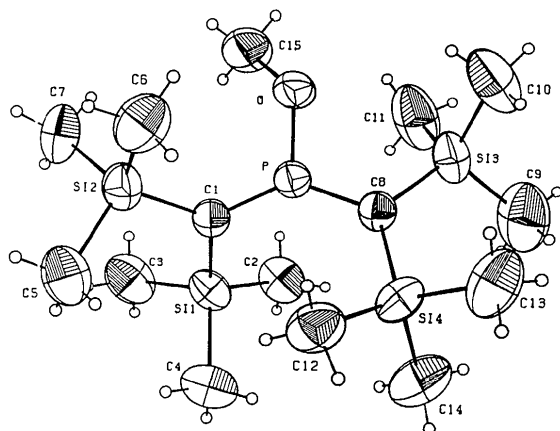| O—P—C(1)	116·7 (2)	O—P—C(8)	109·3 (2)
C(1)—P—C(8)	134·0 (2)	C(1)—Si(1)—C(2)	112·1 (2)
C(1)—Si(1)—C(3)	108·2 (2)	C(2)—Si(1)—C(3)	107·0 (3)
C(1)—Si(1)—C(4)	114·1 (2)	C(2)—Si(1)—C(4)	108·8 (3)
C(3)—Si(1)—C(4)	106·3 (3)	C(1)—Si(2)—C(5)	107·9 (2)
C(1)—Si(2)—C(6)	111·0 (2)	C(5)—Si(2)—C(6)	107·8 (3)
C(1)—Si(2)—C(7)	114·6 (2)	C(5)—Si(2)—C(7)	107·1 (3)
C(6)—Si(2)—C(7)	108·2 (3)	C(8)—Si(3)—C(9)	109·9 (3)
C(8)—Si(3)—C(10)	112·7 (3)	C(9)—Si(3)—C(10)	109·0 (3)
C(8)—Si(3)—C(11)	111·2 (2)	C(9)—Si(3)—C(11)	105·4 (3)
C(10)—Si(3)—C(11)	108·3 (3)	C(8)—Si(4)—C(12)	112·0 (2)
C(8)—Si(4)—C(13)	108·7 (3)	C(12)—Si(4)—C(13)	106·4 (3)
C(8)—Si(4)—C(14)	112·3 (3)	C(12)—Si(4)—C(14)	110·4 (3)
C(13)—Si(4)—C(14)	106·8 (3)	P—O—C(15)	122·1 (3)
P—C(1)—Si(1)	117·0 (2)	P—C(1)—Si(2)	121·1 (3)
Si(1)—C(1)—Si(2)	121·9 (2)	P—C(8)—Si(3)	118·6 (3)
P—C(8)—Si(4)	119·3 (2)	Si(3)—C(8)—Si(4)	121·7 (2)

Fig. 1. *ORTEP* (Johnson, 1976) diagram and atom-numbering scheme of title compound.

rotational freedom at the bonded C atom (C—H = 0·95 Å, $B = 5·52$ Å²). The final cycles of refinement of 229 parameters gave $R = 0·054$, $wR = 0·055$ and a goodness of fit of 0·81, $w - [\sigma(F_o)^2 + 0·00025F_o^2]^{-1}$. The largest feature in the final difference map was 0·4 e Å⁻³. The shift/e.s.d.'s in the final cycle is <0·01. Table 1 lists refined fractional coordinates,* and Table 2 lists bond lengths and angles. An *ORTEP* (Johnson, 1976) view of the molecular structure with the numbering scheme is shown in Fig. 1.

* Lists of structure factors, anisotropic thermal parameters calculated planes, and H-atom parameters and a unit-cell diagram have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53525 (10 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Related literature. Structural reports of related substituted bis[bis(trimethylsilyl)methylene]phosphoranes (Appel, Gaitzsch & Knoch, 1985; Appel, Gaitzsch, Dunker & Knoch, 1986) and an asymmetric bis(trimethylsilyl)methylene-diphenylmethylene-phenylphosphorane (Appel, Gaitzsch, Knoch & Lenz, 1986) have been reported. The P=C bond lengths and the C=P=C bond angles of the methoxy derivative reported here are typical.

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Structure of Diethyl 2,6-Dimethyl-4-[5-(4-methylphenyl)-2-furyl]-1,4-dihydropyridine-3,5-dicarboxylate

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(*Received* 10 *November* 1989; *accepted* 17 *September* 1990)

Abstract. $C_{24}H_{27}NO_5$, $M_r = 409.5$, orthorhombic, $Pna2_1$, $a = 12.843$ (4), $b = 17.768$ (4), $c = 9.960$ (3) Å, $V = 2272$ (1) Å^3, $Z = 4$, $D_m = 1.16$ (1), $D_x = 1.197$ Mg m^{-3}, λ(Cu $K\alpha$) = 1.54178 Å, $\mu = 0.65$ mm^{-1}, $F(000) = 872$, $T = 293$ K, final $R = 0.043$ for 1699 unique observed reflections. The substituted 1,4-dihydropyridine ring has a flat-boat conformation with the N(1) and C(4) atoms displaced by 0.173 (1) and 0.413 (2) Å, respectively, from the base of the boat. The phenyl group is in conjugation with the furan ring (dihedral angle 6.4°) and this π system is nearly perpendicular to the least-squares plane of the dihydropyridine ring. The molecules are linked by an intermolecular hydrogen bond between N(1)—H and O(9) $(x - \frac{1}{2}, \frac{1}{2} - y, z)$[N···O 2.970 (2) Å].

Experimental. Transparent needles, crystal size $0.1 \times 0.15 \times 0.5$ mm. D_m by flotation in bromoform/n-octane. Systematic absences: 0kl for $k + l$ odd and $h0l$ for h odd. Syntex $P2_1$ diffractometer, graphite-monochromated Cu $K\alpha$ radiation. Choice of unit-cell parameters verified by program *UB* (Sivý, Sivý & Koreň, 1987); refinement by least squares on the basis of 15 independent 2θ values in the range 35–52°. Intensity data ($h = 0$ to 15, $k = 0$ to 20, $l = 0$ to 11) by $\theta/2\theta$ scans, scan width 2° plus α-doublet

splitting, $2\theta_{max} = 130°$. Three standards (600, 040 and 002) measured every 100 reflections, no systematic fluctuation. 2059 unique reflections, 1699 with $I \geq 2\sigma(I)$ considered observed and included in the refinement. Data reduction (no absorption or extinction corrections) by using program *XP*21 (Pavelčik, 1987). Structure solved by direct methods using *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980) and *MAGEX* (Hull, Viterbo, Woolfson & Zhang, 1981) and refined by block-diagonal least squares with anisotropic thermal parameters for non-H atoms. C(18) disordered in two positions with occupancy factors fixed at 0.60 [C(181)] and 0.40 [C(182)], based on relative heights in a difference Fourier map. All H atoms, except those affected by disorder, located from maps and refined isotropically. In final cycle $R = 0.043$, $wR = 0.076$ for observed reflections only, $S = 1.55$, max. shift/e.s.d. 0.15, function minimized $\sum w(\Delta F)^2$, where $w = [\sigma^2(I) + (0.02F_o^2)^2]^{-1/2}$. Max. and min. heights in final $\Delta\rho$ synthesis 0.11 and -0.12 e Å^{-3}. Scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV). All calculations except *UB*, *XP*21, *MULTAN* and *MAGEX* performed with a local version of the *NRC* system (Ahmed, Hall, Pippy & Huber, 1973).