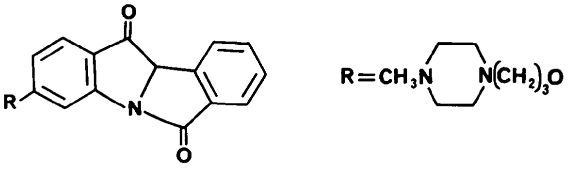
A difference in chromophore geometry of the present compound from that of ellipticines is that the isoindoloindole has a linear chromophore with the terminal six-membered rings parallel to each other, due to the two fused five-membered rings in the centre of the chromophore. Using standard techniques (Bennett, Sharples & Brown, 1982), the melting profile of DNA was found to be the same in the presence of 1(*d*) as in its absence, suggesting that the compound does not interact with DNA. This was confirmed by the finding that DNA has no effect on the light-absorption properties of 1(*d*). This lack of DNA interaction may be due to the projecting *N*-ethyl group of the isoindoloindole which may hamper the intercalation of the chromophore between the DNA base pairs. To investigate the geometrical feature of the molecule, empirical energetic calculations were used with the aid of molecular graphics using the program system *MOLEC* (Islam & Neidle, 1983). The self-complementary dimer of d(CpG) was adopted as a DNA model structure and the coordinates were taken from the known intercalating structure of d(CpG).proflavine (Shieh, Berman, Dabrow & Neidle, 1980). The coordinates of 1(*c*) were those determined in this work and those of a side chain of 1(*d*) were generated assuming a standard geometry. The d(CpG) geometry was held invariant throughout the study and the molecules 1(*c*) and 1(*d*) were restricted to rotational and translational movements. The non-bonded energy component was arbitrarily taken as the interaction energy between d(CpG) and isoindoloindoles in this approximate study. Full intercalation of the molecule into the docking site of d(CpG) exhibits high energy due to short distances between O(6Gl) and C(19) (typically 1·5–1·9 Å). It was found that a partial intercalation with some overlapping of DNA base and isoindoloindole chromophore planes is possible both for 1(*c*) and 1(*d*). The geometry of an energy minimum is shown in Fig. 2.* Therefore the total lack of interaction with DNA cannot be attributed to the *N*-ethyl groups. This was further confirmed by the DNA-binding study with a diketone (2), which was synthesized by hydrolysis of 1(*c*) in conc. HCl/$CH_3OH$. Again compound (2) was found to have no stabilizing effect on the thermal-denaturation profile of DNA.



(2)

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* Deposited.

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*Acta Cryst.* (1984). C**40**, 529–531

# Structure of 1,1′-Dimethylsilanediylbis(acetone phenylhydrazone), $C_{20}H_{28}N_4Si$

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**Abstract.** $M_r = 352·6$, monoclinic, $C2/c$, $a = 20·788\,(3)$, $b = 7·525\,(1)$, $c = 13·200\,(2)$ Å, $\beta = 102·62\,(1)°$, $U = 2015·0$ Å$^3$, $Z = 4$, $D_x = 1·162$ Mg m$^{-3}$, $\lambda(\text{Mo } K\alpha) = 0·71069$ Å, $\mu = 0·12$ mm$^{-1}$, $F(000) = 760$, $T = 291$ K, $R = 0·094$ for 971 observed reflections. The molecule, with a crystallographic twofold axis through Si, contains essentially single N–N and double N=C bonds in the hydrazone

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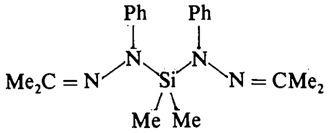
fragment; the electron lone pairs of the N atoms bonded to Si are conjugated with the substituent phenyl rings rather than with the N=C double bond.

**Introduction.** The compound was obtained as a side-product during the synthesis of silyl-substituted hydrazones, which are precursors to 1,2-diaza-3-sila-5-cyclopentenes containing $SiN_2C_2$ rings (Klingebiel & Werner, 1979; Klingebiel, Pohlmann & Werner, 1980). The crystal structure was determined after the nature of the compound had not been elucidated by spectroscopic methods.



**Experimental.** Crystal 0·2 × 0·2 × 0·5 mm, mounted in capillary. Stoe–Siemens AED diffractometer. Unit-cell parameters from $2\theta$ values of 20 reflections ($20 < 2\theta < 25°$). 2001 reflections with $2\theta < 50°$ and $k, l \geq 0$. Profile analysis (Clegg, 1981). No significant intensity variation for three standard reflections. No absorption correction. $R_{\rm int} = 0{\cdot}037$ (*hk*0 reflections only). 1768 unique reflections, 971 with $F > 3\sigma(F)$. Multi-solution direct methods. Blocked-cascade refinement on $F$. $w^{-1} = \sigma^2(F)$. H atoms constrained to give C—H = 0·96 Å, methyl H—C—H = 109·5°, phenyl H on C—C—C external bisectors, $U(\rm H) = 1{\cdot}2U_{\rm eq}(C)$. Anisotropic thermal parameters for non-H atoms. No extinction correction. Scattering factors from *International Tables for X-ray Crystallography* (1974). 123 parameters. $R = 0{\cdot}094$, $wR = 0{\cdot}050$. Slope of normal probability plot 1·26. Max. $\Delta/\sigma$ 0·013, mean 0·003. Largest peak in final difference map 0·44 e Å$^{-3}$, largest hole −0·36 e Å$^{-3}$. Programs used: *SHELXTL* (Sheldrick, 1978), diffractometer-control program by the author.

Table 1. *Atomic coordinates* (×10⁴) *and equivalent isotropic thermal parameters* (Å² ×10⁴)

$$U_{\rm eq} = \tfrac{1}{3}\textstyle\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

| | *x* | *y* | *z* | $U_{\rm eq}$ |
|---|---|---|---|---|
| Si | 5000 | 5010 (4) | 2500 | 375 (8) |
| N(1) | 5709 (2) | 3713 (6) | 2928 (3) | 382 (17) |
| C(1) | 5112 (3) | 6397 (8) | 1391 (4) | 505 (25) |
| N(2) | 6029 (2) | 3722 (6) | 4021 (3) | 416 (18) |
| C(2) | 6431 (2) | 4964 (10) | 4300 (3) | 406 (20) |
| C(3) | 6614 (3) | 6439 (8) | 3646 (4) | 638 (29) |
| C(4) | 6770 (3) | 5046 (10) | 5438 (3) | 566 (23) |
| C(5) | 5954 (3) | 2399 (7) | 2361 (4) | 337 (21) |
| C(6) | 5597 (2) | 1891 (7) | 1368 (4) | 416 (23) |
| C(7) | 5858 (3) | 683 (7) | 779 (4) | 477 (25) |
| C(8) | 6462 (3) | −93 (9) | 1149 (4) | 527 (25) |
| C(9) | 6812 (3) | 352 (8) | 2142 (4) | 543 (26) |
| C(10) | 6556 (3) | 1574 (8) | 2729 (4) | 484 (25) |

Table 2. *Bond lengths* (Å) *and angles* (°)

| | | | |
|---|---|---|---|
| Si—N(1) | 1·755 (4) | Si—C(1) | 1·853 (6) |
| N(1)—N(2) | 1·451 (5) | N(1)—C(5) | 1·400 (7) |
| N(2)—C(2) | 1·253 (7) | C(2)—C(3) | 1·506 (9) |
| C(2)—C(4) | 1·514 (6) | C(5)—C(6) | 1·411 (7) |
| C(5)—C(10) | 1·388 (8) | C(6)—C(7) | 1·381 (8) |
| C(7)—C(8) | 1·373 (8) | C(8)—C(9) | 1·395 (7) |
| C(9)—C(10) | 1·381 (9) | | |
| N(1)—Si—C(1) | 109·0 (2) | N(1)—Si—N(1)′ | 112·4 (2) |
| N(1)—Si—C(1)′ | 107·5 (2) | C(1)—Si—C(1)′ | 111·5 (4) |
| Si—N(1)—N(2) | 119·2 (3) | Si—N(1)—C(5) | 126·7 (3) |
| N(2)—N(1)—C(5) | 113·2 (4) | N(1)—N(2)—C(2) | 115·3 (4) |
| N(2)—C(2)—C(3) | 128·1 (4) | N(2)—C(2)—C(4) | 117·3 (5) |
| C(3)—C(2)—C(4) | 114·7 (5) | N(1)—C(5)—C(6) | 120·6 (5) |
| N(1)—C(5)—C(10) | 122·3 (4) | C(6)—C(5)—C(10) | 117·0 (5) |
| C(5)—C(6)—C(7) | 120·7 (5) | C(6)—C(7)—C(8) | 121·3 (5) |
| C(7)—C(8)—C(9) | 118·8 (6) | C(8)—C(9)—C(10) | 120·1 (5) |
| C(5)—C(10)—C(9) | 122·0 (5) | | |

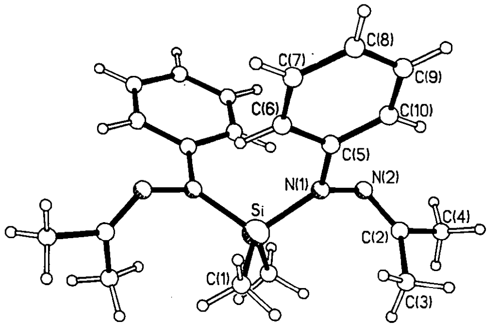


Fig. 1. The molecular structure, showing the atom-numbering scheme.

**Discussion.** Atomic coordinates are given in Table 1,* bond lengths and angles in Table 2. The molecular structure is shown in Fig. 1. The Si atom lies on a crystallographic twofold axis.

A search for the fragment Si—N—N=C in the data files of the Cambridge Crystallographic Data Centre indicates that no other such silylhydrazones have been crystallographically investigated. Bond lengths and angles show that the N—N and N=C bonds are essentially single and double, respectively. This contrasts with the geometry of the Si—N—N=C—C ring in various diazasilacyclopentenes (Clegg, Haase, Hesse, Klingebiel & Sheldrick, 1982; Clegg, Graalmann, Haase, Klingebiel, Sheldrick, Werner, Henkel & Krebs, 1983; Clegg, 1983; Graalmann, Hesse, Klingebiel, Clegg, Haase & Sheldrick, 1984), where there is some conjugation within the ring, giving shorter N—N (1·386–1·425 Å) and longer N=C (1·276–1·297 Å) bonds. Lack of such conjugation in the silylhydrazone is also demonstrated by the approximate orthogonality [dihedral angle 79·7 (7)°] of the coordination planes of N(1) and C(2) [deviation 0·085 (4) Å for N(1) from the

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39031 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

plane of Si, N(2), C(5); deviation 0·010 (6) Å for C(2) from the plane of N(2), C(3), C(4)]. Some conjugation of the lone pair of N(1) with the phenyl ring is, however, evident in the short N(1)—C(5) bond and the near coplanarity of the phenyl ring [r.m.s. deviation 0·009 (6) Å] with the coordination plane of N(1) [dihedral angle 11·9 (8)°].

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# Structure of 6-Kestose Monohydrate,* $C_{18}H_{31}O_{16}.H_2O$

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**Abstract.** $M_r = 521{\cdot}45$, orthorhombic, $P2_12_12_1$, $a = 8{\cdot}718\ (2)$, $b = 11{\cdot}317\ (2)$, $c = 23{\cdot}386\ (3)$ Å, $U = 2307{\cdot}3\ (7)$ Å$^3$, $Z = 4$, $D_x = 1{\cdot}501$ Mg m$^{-3}$, $\lambda(\mathrm{Cu}\ K\alpha) = 1{\cdot}54178$ Å, $\mu = 1{\cdot}13$ mm$^{-1}$, $F(000) = 1108$, $T = 298$ K. Final $R = 0{\cdot}039$ for 1381 observed reflections. Molecules are associated in the crystal by an extended network of hydrogen bonds where most O atoms are both donors and acceptors. Moreover, there are four bifurcated intramolecular hydrogen bonds. Ring conformation is discussed in terms of puckering-coordinate analysis and the conformation of the glucose–fructose linkage of the sucrose subunit in terms of the potential-energy map calculated in the atom–atom potential approximation.

**Introduction.** Kestoses are non-reducing trisaccharides present in several plant extracts which are synthetized by enzymatic action of invertase on sucrose (Gross, 1962). The sucrose:sucrose-fructosyltransferase isolated from plants or yeast invertase is able to transfer fructose from a donor sucrose molecule to one of the primary alcoholic groups of an acceptor sucrose molecule giving kestoses and glucose. Three different kestoses are reported, that is 1-kestose, 6-kestose and neo-kestose. They can be written, respectively, as $1^F$-fructosyl-sucrose, $6^F$-fructosyl-sucrose, $6^G$-fructosyl-sucrose, where the suffix F or G indicates that the bond links the fructose or glucose part of sucrose. 1- and 6-kestose are believed to act as the smallest possible nuclei for the synthesis of polyfructosanes which can be considered kestoses extended by progressive addition of other fructose units (Binkley, Horton & Bhacca, 1969; Lewis, Cyr & Smith, 1967) by action of the fructan:fructan-fructosyltransferase. The polysaccharide inulin, one of the main reserve carbohydrates of plants, derives from 1-kestose while 6-kestose gives the polyfructosan levan, which is mainly produced in particular non-physiological conditions, for instance in frost-damaged sugar-beets.

At present only the crystal-structure determination of 1-kestose has been reported (Jeffrey & Park, 1972) and that of 6-kestose is reported here. Apart from the intrinsic interest in the conformational aspects of these sugars the present crystal-structure determination has been undertaken in the frame of a wider research

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* *O*-α-D-Glucopyranosyl-(1→2)-*O*-β-D-fructofuranosyl-(6→2)-β-D-fructofuranoside monohydrate.