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Structure of (1*S*,3*R*,4*R*,8*S*,9*R*)-1-*tert*-Butyldiphenylsilyloxy-4-hydroxymethyl-2,5,7-trioxa-12-azatricyclo[7.3.1.$0^{3,8}$]tridecan-11-one, an Intermediate in the Synthesis of (+)-Sesbanimide A

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Abstract. $C_{26}H_{33}NO_6Si$, $M_r = 483{\cdot}6$, monoclinic, $P2_1$, $a = 15{\cdot}186\ (3)$, $b = 27{\cdot}498\ (4)$, $c = 14{\cdot}437\ (2)$ Å, $\beta = 115{\cdot}66\ (1)°$, $V = 5434\ (2)$ Å^3, $Z = 8$, $D_x = 1{\cdot}18$ g cm^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 10{\cdot}6$ cm^{-1}, $F(000) = 2064$, room temperature, final $R = 0{\cdot}084$ for 4774 observed reflections. The asymmetric unit contains four independent molecules. One of the four molecules shows a relative positioning of the substituents of the Si atom that differs from the other three. Two pairs of molecules are identically linked by hydrogen bridges.

Introduction. The compound (+)-sesbanimide A, which can be extracted from the seeds of *Sesbania drummondii*, is an active anti-leukemic alkaloid. The structure presented here is that of an intermediate in the synthesis of (+)-sesbanimide A as described by Vloon, van den Bos, Willard, Koomen & Pandit (1989); the X-ray crystallographic study confirms the proposed tricyclic nature of the title compound.

Experimental. A brick-shaped crystal (approximate dimensions 0·13 × 0·23 × 0·30 mm) was used for data collection on an Enraf–Nonius CAD-4 diffractometer with graphite-monochromated Cu $K\alpha$ radiation and θ–2θ scan. A total of 5720 unique reflections were measured within the range $-15 \le h \le 13$, $0 \le k \le 27$, $0 \le l \le 14$. Of these, 4774 were above the significance level of $2{\cdot}5\sigma(I)$. The maximum value of $(\sin\theta)/\lambda$ was 0·50 Å^{-1}. Two standard reflections (130, $\bar{1}11$) were measured hourly, no significant decrease in intensity was measured during the 66 h collecting time. Unit-cell parameters were refined by a least-squares fitting procedure using 23 reflections with $66 < 2\theta < 70°$. Corrections for Lorentz and polarization effects were applied. The structure was solved by direct methods using the *XTAL* system of crystallographic programs (Hall & Stewart, 1987). Structure factors were normalized using a profile scale and some 10 000 triplets and 6000 quartets for the strongest 999 reflections were included in a tangent phase extension and refinement using a magic cascade procedure. The Fourier map revealed some eighty atoms, mainly showing parts of the tricyclic systems. A subsequent iterative procedure, consisting of a least-squares refinement of the current model, an F_{obs} Fourier synthesis and a manual interpretation of the resulting peaks, completed the model.

The positions of the H atoms were calculated. Anisotropic block-diagonal least-squares refinement on F, keeping the H atoms fixed at their calculated positions with $U = 0{\cdot}06$ Å^2, converged to $R = 0{\cdot}084$, $wR = 0{\cdot}126$, $(\Delta/\sigma)_{max} = 0{\cdot}21$, origin defined by fixing y of C(1*A*). A weighting scheme $w = (6{\cdot}2 + F_{obs} + 0{\cdot}01F_{obs}^2)^{-1}$ was used. An empirical absorption correction was applied, with corrections in the range 0·53–1·22 (Walker & Stuart, 1983). The isotropic secondary-extinction coefficient refined to $2{\cdot}6\ (1) \times 10^{-2}$ (Zachariasen, 1968). A final difference Fourier map revealed a residual electron density between $-0{\cdot}3$ and $1{\cdot}2$ e Å^{-3}. Scattering factors were taken from Cromer & Mann (1968). Anomalous-dispersion

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Table 1. *Fractional coordinates of the non-*H *atoms and equivalent isotropic thermal parameters*

$$U_{eq} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i . \mathbf{a}_j .$$

	x	y	z	U_{eq}(Å^2)
Molecule *A*				
C(1*A*)	0·626 (1)	0·0000	0·088 (1)	0·041 (8)
O(2*A*)	0·6023 (6)	0·0273 (3)	0·1567 (6)	0·039 (5)
C(3*A*)	0·669 (1)	0·0206 (5)	0·2630 (9)	0·036 (8)
C(4*A*)	0·632 (1)	0·0466 (5)	0·329 (1)	0·043 (9)
O(5*A*)	0·5489 (8)	0·0216 (4)	0·3297 (8)	0·061 (7)
C(6*A*)	0·573 (1)	−0·0265 (7)	0·361 (1)	0·06 (1)
O(7*A*)	0·5959 (8)	−0·0533 (4)	0·2928 (8)	0·057 (7)
C(8*A*)	0·682 (1)	−0·0340 (6)	0·289 (1)	0·046 (9)
C(9*A*)	0·699 (1)	−0·0645 (5)	0·211 (1)	0·048 (9)
C(10*A*)	0·800 (1)	−0·0541 (6)	0·212 (1)	0·049 (9)
C(11*A*)	0·807 (1)	−0·0062 (5)	0·167 (1)	0·038 (8)
N(12*A*)	0·7240 (7)	0·0149 (4)	0·0985 (8)	0·036 (6)
C(13*A*)	0·623 (1)	−0·0538 (5)	0·106 (1)	0·046 (9)
C(14*A*)	0·596 (1)	0·0961 (6)	0·292 (1)	0·05 (1)
O(15*A*)	0·6720 (9)	0·1243 (5)	0·285 (1)	0·081 (9)
O(16*A*)	0·8871 (6)	0·0130 (4)	0·1895 (7)	0·049 (6)
O(17*A*)	0·5610 (6)	0·0152 (4)	−0·0100 (6)	0·042 (5)
Si(18*A*)	0·4400 (3)	0·0215 (1)	−0·0629 (3)	0·039 (2)
C(19*A*)	0·378 (1)	−0·0174 (6)	0·000 (1)	0·046 (9)
C(20*A*)	0·340 (1)	−0·0623 (6)	−0·033 (1)	0·06 (1)
C(21*A*)	0·294 (1)	−0·0879 (7)	0·017 (2)	0·09 (1)
C(22*A*)	0·291 (1)	−0·0713 (7)	0·100 (2)	0·07 (1)
C(23*A*)	0·331 (1)	−0·0261 (8)	0·139 (1)	0·07 (1)
C(24*A*)	0·373 (1)	0·0011 (6)	0·088 (1)	0·05 (1)
C(25*A*)	0·415 (1)	0·0862 (5)	−0·045 (1)	0·05 (1)
C(26*A*)	0·490 (1)	0·1195 (7)	0·004 (1)	0·06 (1)
C(27*A*)	0·476 (2)	0·1688 (6)	0·015 (2)	0·07 (1)
C(28*A*)	0·382 (2)	0·1863 (6)	−0·021 (2)	0·10 (2)
C(29*A*)	0·303 (2)	0·1535 (9)	−0·071 (2)	0·12 (2)
C(30*A*)	0·321 (1)	0·1030 (8)	−0·080 (2)	0·09 (1)
C(31*A*)	0·403 (1)	0·0083 (7)	−0·202 (1)	0·06 (1)
C(32*A*)	0·446 (1)	0·0464 (8)	−0·248 (1)	0·07 (1)
C(33*A*)	0·292 (1)	0·0039 (9)	−0·264 (1)	0·08 (1)
C(34*A*)	0·444 (1)	−0·0426 (8)	−0·213 (1)	0·08 (1)
Molecule *B*				
C(1*B*)	1·0131 (9)	0·1344 (6)	0·283 (1)	0·045 (9)
O(2*B*)	1·0359 (7)	0·1022 (3)	0·3718 (7)	0·044 (5)
C(3*B*)	0·964 (1)	0·1066 (5)	0·415 (1)	0·045 (9)
C(4*B*)	1·002 (1)	0·0753 (6)	0·512 (1)	0·06 (1)
O(5*B*)	1·0863 (9)	0·0990 (4)	0·5916 (8)	0·063 (7)
C(6*B*)	1·063 (1)	0·1467 (7)	0·608 (1)	0·07 (1)
O(7*B*)	1·0405 (8)	0·1763 (4)	0·5235 (8)	0·059 (7)
C(8*B*)	0·951 (1)	0·1586 (6)	0·437 (1)	0·06 (1)
C(9*B*)	0·933 (1)	0·1938 (5)	0·348 (1)	0·045 (9)
C(10*B*)	0·834 (1)	0·1843 (6)	0·254 (1)	0·05 (1)
C(11*B*)	0·831 (1)	0·1387 (5)	0·192 (1)	0·044 (9)
N(12*B*)	0·9196 (8)	0·1206 (4)	0·2028 (8)	0·043 (7)
C(13*B*)	1·016 (1)	0·1874 (5)	0·313 (1)	0·046 (9)
C(14*B*)	1·039 (1)	0·0268 (6)	0·500 (1)	0·06 (1)
O(15*B*)	0·963 (1)	0·0008 (5)	0·4165 (8)	0·079 (8)
O(16*B*)	0·7541 (6)	0·1206 (4)	0·1364 (8)	0·055 (6)
O(17*B*)	1·0825 (6)	0·1226 (3)	0·2484 (6)	0·040 (5)
Si(18*B*)	1·2036 (3)	0·1170 (2)	0·3085 (3)	0·043 (2)
C(19*B*)	1·231 (1)	0·0534 (6)	0·348 (1)	0·053 (9)
C(20*B*)	1·325 (1)	0·0381 (8)	0·412 (1)	0·08 (1)
C(21*B*)	1·347 (2)	−0·0116 (8)	0·438 (2)	0·09 (1)
C(22*B*)	1·273 (2)	−0·0450 (7)	0·398 (2)	0·10 (2)
C(23*B*)	1·181 (2)	−0·0326 (6)	0·338 (2)	0·09 (1)
C(24*B*)	1·159 (1)	0·0148 (7)	0·312 (1)	0·07 (1)
C(25*B*)	1·264 (1)	0·1555 (6)	0·427 (1)	0·06 (1)
C(26*B*)	1·263 (1)	0·1391 (7)	0·519 (1)	0·07 (1)
C(27*B*)	1·308 (1)	0·167 (1)	0·609 (2)	0·10 (2)
C(28*B*)	1·363 (1)	0·2111 (9)	0·606 (2)	0·10 (2)
C(29*B*)	1·367 (1)	0·2249 (8)	0·520 (2)	0·09 (1)
C(30*B*)	1·317 (1)	0·1982 (6)	0·428 (1)	0·07 (1)
C(31*B*)	1·237 (1)	0·1316 (6)	0·201 (1)	0·06 (1)
C(32*B*)	1·204 (1)	0·1833 (7)	0·157 (1)	0·07 (1)
C(33*B*)	1·348 (1)	0·1273 (8)	0·236 (2)	0·08 (1)
C(34*B*)	1·188 (1)	0·0944 (7)	0·114 (1)	0·07 (1)
Molecule *C*				
C(1*C*)	0·056 (1)	0·4655 (5)	0·255 (1)	0·046 (9)
O(2*C*)	0·1108 (6)	0·4585 (3)	0·1964 (6)	0·038 (5)
C(3*C*)	0·089 (1)	0·4948 (6)	0·116 (1)	0·045 (9)
C(4*C*)	0·145 (1)	0·4785 (6)	0·054 (1)	0·05 (1)
O(5*C*)	0·1049 (9)	0·4358 (4)	−0·0046 (7)	0·066 (7)
C(6*C*)	0·004 (1)	0·4419 (8)	−0·072 (1)	0·08 (1)
O(7*C*)	−0·0488 (8)	0·4499 (4)	−0·0126 (8)	0·068 (7)
C(8*C*)	−0·021 (1)	0·4953 (6)	0·043 (1)	0·06 (1)
C(9*C*)	−0·080 (1)	0·4994 (7)	0·107 (1)	0·06 (1)
C(10*C*)	−0·066 (1)	0·5489 (6)	0·159 (1)	0·05 (1)
C(11*C*)	0·029 (1)	0·5546 (5)	0·251 (1)	0·049 (9)
N(12*C*)	0·0775 (9)	0·5135 (4)	0·3002 (8)	0·043 (7)
C(13*C*)	−0·051 (1)	0·4584 (6)	0·189 (1)	0·05 (1)
C(14*C*)	0·254 (1)	0·4682 (6)	0·124 (1)	0·05 (1)
O(15*C*)	0·2949 (8)	0·5064 (5)	0·1933 (9)	0·077 (8)
O(16*C*)	0·0620 (7)	0·5951 (4)	0·2843 (8)	0·053 (6)
O(17*C*)	0·0944 (7)	0·4320 (3)	0·3353 (7)	0·046 (6)
Si(18*C*)	0·1220 (3)	0·3742 (1)	0·3337 (3)	0·045 (2)
C(19*C*)	0·245 (1)	0·3696 (6)	0·331 (1)	0·050 (9)
C(20*C*)	0·275 (1)	0·3257 (7)	0·308 (1)	0·06 (1)
C(21*C*)	0·370 (2)	0·3215 (9)	0·308 (2)	0·10 (2)
C(22*C*)	0·428 (1)	0·363 (1)	0·330 (2)	0·10 (2)
C(23*C*)	0·399 (1)	0·4081 (9)	0·360 (1)	0·09 (1)
C(24*C*)	0·309 (1)	0·4103 (6)	0·360 (1)	0·06 (1)
C(25*C*)	0·027 (1)	0·3408 (5)	0·220 (1)	0·053 (9)
C(26*C*)	−0·058 (1)	0·3245 (6)	0·216 (1)	0·06 (1)
C(27*C*)	−0·131 (2)	0·3035 (7)	0·128 (2)	0·10 (2)
C(28*C*)	−0·115 (2)	0·2950 (7)	0·048 (2)	0·12 (2)
C(29*C*)	−0·035 (2)	0·312 (1)	0·046 (2)	0·11 (2)
C(30*C*)	0·041 (2)	0·3352 (8)	0·132 (2)	0·09 (1)
C(31*C*)	0·132 (1)	0·3494 (6)	0·461 (1)	0·06 (1)
C(32*C*)	0·060 (2)	0·3717 (8)	0·492 (2)	0·10 (2)
C(33*C*)	0·125 (1)	0·2954 (6)	0·460 (1)	0·06 (1)
C(34*C*)	0·237 (2)	0·3640 (9)	0·546 (1)	0·11 (2)
Molecule *D*				
C(1*D*)	0·288 (1)	0·6676 (5)	0·369 (1)	0·040 (8)
O(2*D*)	0·2370 (6)	0·6726 (3)	0·2591 (7)	0·038 (5)
C(3*D*)	0·262 (1)	0·6374 (5)	0·204 (1)	0·046 (9)
C(4*D*)	0·210 (1)	0·6512 (6)	0·089 (1)	0·051 (9)
O(5*D*)	0·2543 (8)	0·6917 (4)	0·0677 (9)	0·066 (8)
C(6*D*)	0·353 (2)	0·6832 (7)	0·096 (2)	0·07 (1)
O(7*D*)	0·4057 (8)	0·6778 (4)	0·206 (1)	0·072 (8)
C(8*D*)	0·372 (1)	0·6342 (6)	0·235 (1)	0·05 (1)
C(9*D*)	0·430 (1)	0·6321 (6)	0·352 (1)	0·06 (1)
C(10*D*)	0·414 (1)	0·5823 (6)	0·395 (1)	0·06 (1)
C(11*D*)	0·316 (1)	0·5775 (6)	0·401 (1)	0·049 (9)
N(12*D*)	0·2655 (8)	0·6191 (4)	0·3970 (9)	0·043 (7)
C(13*D*)	0·398 (1)	0·6736 (6)	0·402 (1)	0·06 (1)
C(14*D*)	0·103 (1)	0·6618 (6)	0·053 (1)	0·06 (1)
O(15*D*)	0·0535 (8)	0·6236 (5)	0·0800 (9)	0·076 (8)
O(16*D*)	0·2816 (7)	0·5376 (3)	0·4041 (8)	0·055 (6)
O(17*D*)	0·2471 (7)	0·7010 (3)	0·4086 (7)	0·050 (6)
Si(18*D*)	0·2425 (3)	0·7609 (2)	0·4077 (3)	0·049 (3)
C(19*D*)	0·283 (1)	0·7787 (7)	0·545 (1)	0·07 (1)
C(20*D*)	0·338 (2)	0·742 (1)	0·623 (1)	0·13 (2)
C(21*D*)	0·367 (3)	0·755 (1)	0·727 (2)	0·17 (3)
C(22*D*)	0·352 (3)	0·800 (1)	0·753 (2)	0·18 (3)
C(23*D*)	0·303 (3)	0·834 (1)	0·679 (3)	0·17 (3)
C(24*D*)	0·272 (2)	0·8231 (9)	0·579 (2)	0·12 (2)
C(25*D*)	0·323 (1)	0·7914 (5)	0·358 (1)	0·042 (9)
C(26*D*)	0·400 (1)	0·8200 (6)	0·422 (1)	0·06 (1)
C(27*D*)	0·460 (1)	0·8440 (7)	0·387 (1)	0·08 (1)
C(28*D*)	0·446 (1)	0·8407 (6)	0·288 (2)	0·07 (1)
C(29*D*)	0·369 (2)	0·8129 (7)	0·224 (1)	0·08 (1)
C(30*D*)	0·309 (1)	0·7867 (7)	0·257 (1)	0·06 (1)
C(31*D*)	0·111 (1)	0·7804 (6)	0·329 (1)	0·06 (1)
C(32*D*)	0·066 (2)	0·753 (1)	0·227 (1)	0·10 (2)
C(33*D*)	0·053 (2)	0·768 (1)	0·393 (2)	0·10 (2)
C(34*D*)	0·100 (2)	0·8324 (8)	0·307 (2)	0·11 (2)

corrections for Si and O from *International Tables for X-ray Crystallography* (1974). The absolute configuration, known from the synthesis in which only optically pure reagents were used, could not be verified by least-squares refinement. All calculations were performed with *XRAY*76 (Stewart, Machin, Dickinson, Ammon, Heck & Flack, 1976), unless stated otherwise.

Table 2. *Interatomic distances* (Å), *bond angles* (°) *and intermolecular distances between A and B and between C and D* (Å)

	Molecule *A*	Molecule *B*	Molecule *C*	Molecule *D*
C(1)—O(2)	1·41 (2)	1·46 (2)	1·44 (2)	1·44 (2)
C(1)—N(12)	1·48 (2)	1·44 (1)	1·45 (2)	1·48 (2)
C(1)—C(13)	1·51 (2)	1·51 (2)	1·49 (2)	1·54 (2)
C(1)—O(17)	1·40 (1)	1·39 (2)	1·40 (2)	1·36 (2)
O(2)—C(3)	1·44 (1)	1·48 (2)	1·45 (2)	1·41 (2)
C(3)—C(4)	1·49 (2)	1·53 (2)	1·54 (3)	1·54 (2)
C(3)—C(8)	1·54 (2)	1·50 (2)	1·55 (2)	1·53 (2)
C(4)—O(5)	1·44 (2)	1·45 (2)	1·43 (2)	1·40 (2)
C(4)—C(14)	1·48 (2)	1·48 (3)	1·54 (2)	1·50 (2)
O(5)—C(6)	1·39 (2)	1·40 (2)	1·42 (2)	1·39 (3)
C(6)—O(7)	1·38 (2)	1·38 (2)	1·43 (3)	1·44 (2)
O(7)—C(8)	1·44 (2)	1·48 (2)	1·44 (2)	1·44 (2)
C(8)—C(9)	1·52 (2)	1·54 (2)	1·54 (3)	1·53 (2)
C(9)—C(10)	1·55 (3)	1·55 (2)	1·52 (3)	1·57 (3)
C(9)—C(13)	1·48 (2)	1·56 (3)	1·55 (2)	1·54 (3)
C(10)—C(11)	1·49 (2)	1·53 (2)	1·49 (2)	1·52 (3)
C(11)—N(12)	1·35 (2)	1·38 (2)	1·36 (2)	1·37 (2)
C(11)—O(16)	1·23 (2)	1·21 (1)	1·23 (2)	1·23 (2)
C(14)—O(15)	1·43 (2)	1·45 (2)	1·40 (2)	1·44 (2)
O(17)—Si(18)	1·665 (9)	1·666 (9)	1·65 (1)	1·65 (1)
Si(18)—C(19)	1·89 (2)	1·83 (2)	1·89 (2)	1·86 (2)
Si(18)—C(25)	1·86 (2)	1·88 (2)	1·89 (1)	1·86 (2)
Si(18)—C(31)	1·87 (2)	1·88 (2)	1·90 (2)	1·90 (2)
C(19)—C(20)	1·36 (2)	1·38 (2)	1·39 (3)	1·49 (3)
C(19)—C(24)	1·41 (3)	1·45 (2)	1·42 (2)	1·35 (3)
C(20)—C(21)	1·39 (4)	1·42 (3)	1·44 (4)	1·42 (4)
C(21)—C(22)	1·31 (4)	1·37 (3)	1·39 (4)	1·34 (6)
C(22)—C(23)	1·39 (3)	1·33 (3)	1·44 (4)	1·37 (5)
C(23)—C(24)	1·38 (3)	1·36 (3)	1·38 (3)	1·35 (5)
C(25)—C(26)	1·39 (2)	1·40 (3)	1·35 (3)	1·38 (2)
C(25)—C(30)	1·38 (3)	1·41 (2)	1·37 (3)	1·40 (2)
C(26)—C(27)	1·39 (2)	1·40 (3)	1·39 (3)	1·37 (3)
C(27)—C(28)	1·38 (3)	1·48 (4)	1·29 (5)	1·36 (3)
C(28)—C(29)	1·42 (3)	1·32 (4)	1·33 (4)	1·36 (3)
C(29)—C(30)	1·43 (3)	1·42 (3)	1·43 (3)	1·39 (3)
C(31)—C(32)	1·53 (3)	1·55 (2)	1·48 (4)	1·52 (3)
C(31)—C(33)	1·54 (2)	1·53 (3)	1·49 (2)	1·56 (4)
C(31)—C(34)	1·57 (3)	1·54 (2)	1·58 (3)	1·46 (3)
O(2)—C(1)—N(12)	109 (1)	109 (1)	108 (1)	108 (1)
O(2)—C(1)—C(13)	111 (1)	112 (1)	111 (1)	109 (1)
O(2)—C(1)—O(17)	106 (1)	104 (1)	105 (1)	106 (1)
N(12)—C(1)—C(13)	111 (1)	112 (1)	112 (1)	111 (1)
N(12)—C(1)—O(17)	105 (1)	106 (1)	107 (1)	107 (1)
C(13)—C(1)—O(17)	114 (1)	113 (1)	113 (1)	115 (1)
C(1)—O(2)—C(3)	114 (1)	112 (1)	113 (1)	114 (1)
O(2)—C(3)—C(4)	110 (1)	106 (1)	106 (1)	108 (1)
O(2)—C(3)—C(8)	110 (1)	111 (1)	111 (1)	114 (1)
C(4)—C(3)—C(8)	111 (1)	112 (1)	108 (1)	109 (2)
C(3)—C(4)—O(5)	111 (1)	109 (1)	112 (1)	111 (1)
C(3)—C(4)—C(14)	113 (1)	114 (2)	113 (1)	113 (2)
O(5)—C(4)—C(14)	105 (1)	105 (1)	107 (1)	109 (1)
C(4)—O(5)—C(6)	110 (1)	111 (1)	112 (1)	111 (1)
O(5)—C(6)—O(7)	113 (2)	113 (1)	109 (1)	111 (2)
C(6)—O(7)—C(8)	110 (1)	110 (1)	111 (1)	108 (1)
C(3)—C(8)—O(7)	110 (1)	110 (1)	110 (1)	110 (1)
C(3)—C(8)—C(9)	114 (1)	114 (2)	109 (1)	112 (2)
O(7)—C(8)—C(9)	106 (1)	105 (1)	107 (1)	104 (1)
C(8)—C(9)—C(10)	112 (1)	112 (1)	112 (2)	111 (1)
C(8)—C(9)—C(13)	110 (1)	109 (1)	111 (1)	110 (1)
C(10)—C(9)—C(13)	108 (1)	109 (1)	110 (1)	109 (2)
C(9)—C(10)—C(11)	114 (1)	114 (1)	114 (1)	115 (1)
C(10)—C(11)—N(12)	119 (1)	117 (1)	118 (1)	118 (1)
C(10)—C(11)—O(16)	121 (1)	120 (1)	121 (1)	122 (1)
N(12)—C(11)—O(16)	120 (1)	123 (1)	121 (1)	120 (2)
C(1)—N(12)—C(11)	122 (1)	124 (1)	124 (1)	125 (1)
C(1)—C(13)—C(9)	108 (1)	105 (1)	105 (1)	107 (1)
C(4)—C(14)—O(15)	110 (1)	110 (1)	110 (1)	112 (1)
C(1)—O(17)—Si(18)	131 (1)	132·1 (8)	130·0 (9)	134 (1)
O(17)—Si(18)—C(19)	113·5 (5)	107·6 (6)	108·9 (7)	105·2 (7)
O(17)—Si(18)—C(25)	106·6 (6)	114·6 (6)	112·3 (6)	114·9 (7)
O(17)—Si(18)—C(31)	103·4 (7)	100·6 (6)	104·3 (7)	108·5 (6)
C(19)—Si(18)—C(25)	107·6 (8)	107·5 (7)	109·8 (8)	108·6 (8)
C(19)—Si(18)—C(31)	114·9 (7)	110·5 (9)	109·0 (7)	109·5 (9)
C(25)—Si(18)—C(31)	110·6 (8)	115·7 (8)	112·3 (8)	110·0 (8)
Si(18)—C(19)—C(20)	125 (2)	122 (1)	120 (1)	116 (2)
Si(18)—C(19)—C(24)	118 (1)	124 (1)	120 (1)	126 (2)
C(20)—C(19)—C(24)	117 (2)	114 (2)	120 (2)	117 (2)
C(19)—C(20)—C(21)	121 (2)	122 (2)	120 (2)	116 (3)
C(20)—C(21)—C(22)	122 (2)	118 (2)	118 (2)	122 (3)
C(21)—C(22)—C(23)	120 (2)	123 (2)	122 (2)	121 (3)
C(22)—C(23)—C(24)	120 (2)	119 (2)	118 (2)	120 (3)
C(19)—C(24)—C(23)	120 (2)	123 (1)	121 (2)	124 (2)
Si(18)—C(25)—C(26)	122 (1)	118 (1)	124 (2)	121 (1)
Si(18)—C(25)—C(30)	121 (1)	122 (2)	120 (1)	122 (1)
C(26)—C(25)—C(30)	117 (2)	120 (2)	117 (2)	117 (2)
C(25)—C(26)—C(27)	125 (2)	120 (2)	123 (2)	122 (2)
C(26)—C(27)—C(28)	119 (2)	118 (2)	120 (2)	122 (2)
C(27)—C(28)—C(29)	119 (2)	121 (2)	119 (2)	116 (2)
C(28)—C(29)—C(30)	121 (2)	120 (2)	122 (3)	124 (2)
C(25)—C(30)—C(29)	120 (2)	121 (2)	118 (2)	119 (1)
Si(18)—C(31)—C(32)	110 (1)	112 (1)	112 (1)	110 (1)
Si(18)—C(31)—C(33)	113 (1)	112 (1)	112 (1)	108 (1)
Si(18)—C(31)—C(34)	110 (1)	109 (1)	107 (1)	113 (1)
C(32)—C(31)—C(33)	112 (1)	109 (2)	111 (2)	109 (2)
C(32)—C(31)—C(34)	107 (2)	108 (1)	107 (2)	108 (2)
C(33)—C(31)—C(34)	105 (1)	107 (2)	108 (1)	108 (2)

N(12*A*)—O(16*B*)	2·96 (2)	N(12*C*)—O(16*D*)	2·88 (1)
O(15*A*)—O(16*B*)	2·91 (2)	O(15*C*)—O(16*D*)	3·25 (2)
O(16*A*)—N(12*B*)	2·99 (2)	O(16*C*)—N(12*D*)	2·88 (1)
O(16*A*)—O(15*B*)	2·99 (1)	O(16*C*)—O(15*D*)	3·00 (2)

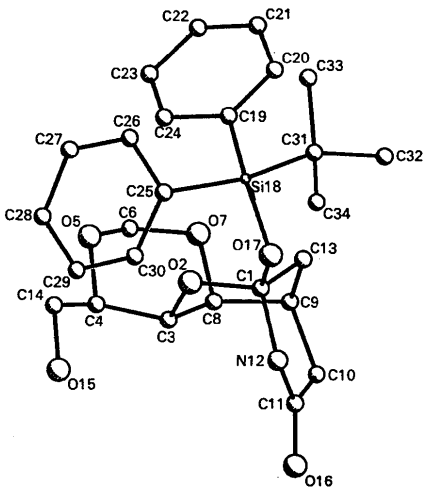

Fig. 1. The molecular structure of the title compound (molecule *A*), showing the numbering scheme.

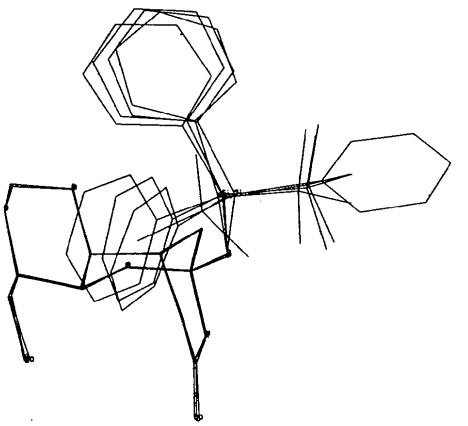

Fig. 2. Matching C(1) up to C(13) for the four molecules.

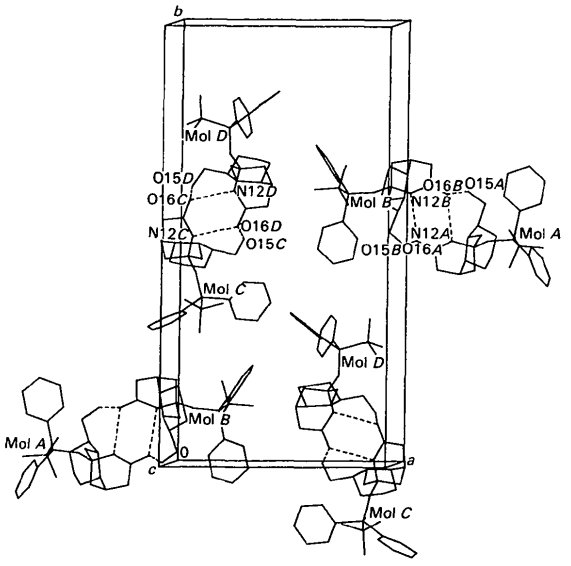

Fig. 3. The unit-cell contents, showing the hydrogen bridges between *A* and *B* and between *C* and *D*.

Discussion. In the tables the four molecules are referred to as *A*, *B*, *C* and *D*. Final positional parameters for the non-H atoms are listed in Table 1,* bond lengths and bond angles in Table 2. A *PLUTO* (Motherwell & Clegg, 1978) drawing of molecule *A* is given in Fig. 1.

Matching of the molecules shows that the conformations of the tricyclic systems of all four molecules are very similar [r.m.s.d. = 0·04 Å for atoms C1 up to C13 for any pair of molecules (Fig. 2)], but that for molecule *D* the relative position of the ligands of the Si atom differs from that of the other three. The dihedral angles between the planes N12, C1, Si18 and O17, C1, Si18 are 19, 20, 26 and 0° in *A*–*D* respectively. Both molecules *A* and *B* and molecules *C* and *D* are linked by hydrogen bridges between N12 and O16 and between O15 and O16 as is shown in Table 2 and in Fig. 3.

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53024 (31 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of 4-{2-[1-Methyl-4(1*H*)-pyridylidene]ethylidene}cyclohexa-2,5-dien-1-one Trihydrate

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Abstract. $C_{14}H_{13}NO.3H_2O$, $M_r = 265{\cdot}31$, monoclinic, $P2_1/c$, $a = 17{\cdot}305\ (1)$, $b = 7{\cdot}3128\ (9)$, $c = 11{\cdot}7955\ (9)$ Å, $\beta = 110{\cdot}294\ (7)°$, $V = 1400{\cdot}0\ (3)$ Å^3, $Z = 4$, $D_x = 1{\cdot}26$ g cm^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 7{\cdot}23$ cm^{-1}, $F(000) = 568$, room temperature, final $R = 0{\cdot}048$ for 1655 observed reflections. The molecule is essentially planar, the maximum deviation of an atom from the least-squares plane through the

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