

Table 2. Selected geometric parameters (Å, °)

S—C(14)	1.755 (4)	S—C(15)	1.775 (4)
O(2)—C(2)	1.401 (4)	O(2)—C(11)	1.420 (5)
O(3)—C(2)	1.415 (3)	O(3)—C(4)	1.443 (4)
O(5)—C(4)	1.418 (3)	O(5)—C(6)	1.428 (4)
O(6)—C(6)	1.388 (4)	O(6)—C(13)	1.409 (4)
O(8)—C(14)	1.192 (4)	C(1)—C(2)	1.501 (5)
C(1)—C(9)	1.541 (4)	C(1)—C(10)	1.542 (5)
C(4)—C(9)	1.541 (4)	C(4)—C(12)	1.506 (5)
C(6)—C(7)	1.525 (4)	C(7)—C(8)	1.544 (4)
C(7)—C(10)	1.528 (5)	C(8)—C(9)	1.536 (5)
C(8)—C(14)	1.501 (4)		
C(14)—S—C(15)	101.7 (2)	C(2)—O(2)—C(11)	114.0 (2)
C(2)—O(3)—C(4)	110.6 (2)	C(4)—O(5)—C(6)	114.8 (2)
C(6)—O(6)—C(13)	114.0 (3)	C(2)—C(1)—C(9)	103.0 (3)
C(2)—C(1)—C(10)	115.5 (3)	C(9)—C(1)—C(10)	104.0 (3)
O(2)—C(2)—O(3)	111.6 (3)	O(2)—C(2)—C(1)	109.0 (2)
O(3)—C(2)—C(1)	105.3 (3)	O(3)—C(4)—O(5)	107.8 (2)
O(3)—C(4)—C(9)	106.2 (2)	O(5)—C(4)—C(9)	111.7 (3)
O(3)—C(4)—C(12)	109.3 (3)	O(5)—C(4)—C(12)	106.5 (2)
C(9)—C(4)—C(12)	115.1 (2)	O(5)—C(6)—O(6)	107.2 (2)
O(5)—C(6)—C(7)	111.5 (2)	O(6)—C(6)—C(7)	109.6 (3)
C(6)—C(7)—C(8)	110.1 (2)	C(6)—C(7)—C(10)	112.3 (3)
C(8)—C(7)—C(10)	100.6 (2)	C(7)—C(8)—C(9)	99.1 (3)
C(7)—C(8)—C(14)	119.6 (2)	C(9)—C(8)—C(14)	116.4 (3)
C(1)—C(9)—C(4)	102.8 (2)	C(1)—C(9)—C(8)	103.7 (3)
C(4)—C(9)—C(8)	110.5 (2)	C(1)—C(10)—C(7)	106.0 (3)
S—C(14)—O(8)	121.0 (3)	S—C(14)—C(8)	114.6 (2)
O(8)—C(14)—C(8)	124.2 (3)		

The structure of the title compound was solved by direct methods using *SHELXTL-Plus* (Sheldrick, 1991). All non-H atoms were located from successive Fourier maps. Anisotropic displacement parameters were used for all non-H atoms and fixed isotropic displacement parameters were used for H atoms, which were included using a riding model. The structure was refined with *SHELXTL-Plus XLS*. Analytical scattering factors for neutral atoms were used throughout the analysis.

We would like to thank the National Science Council of the Republic of China for financial support and Professor Hsien-Jen Wu for supplying the crystals.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AS1180). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1995). **C51**, 2145–2147

A Diaza-cyclo-tetradecadiene-Bis(pyrido-silafuran) Ring System

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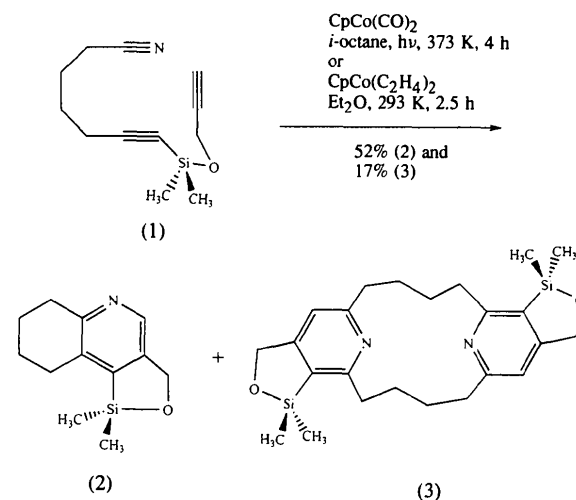
(Received 1 February 1995; accepted 11 April 1995)

Abstract

The crystal structure of 8,8,20,20-tetramethyl-9,21-dioxo-25,26-diaza-8,20-disilapentacyclo[16.6.1.1^{6,13}.0^{7,11}.0^{19,23}]-hexacosia-1(25),6,11,13(26),18,23-hexaene, C₂₄H₃₄N₂O₂-Si₂, has been determined at low temperature. The molecule lies on a crystallographic inversion centre.

Comment

In connection with our current work on the cobalt-catalysed intramolecular [2+2+2] cycloaddition of diyne-nitriles to obtain substituted pyridines (Groth, Eckenberg & Huhn, 1993; Huhn, 1994), the cyclization of the diyne-nitrile (1) yielded 52% of the furylquinoline (2) and 17% of the dimeric title compound (3). The relative amount of the dimeric was found to be independent of the reaction temperature and the cobalt catalyst.



The crystal structure analysis was performed in order to establish the position of the pyridine N atom within the aromatic ring. All bond lengths and angles are in

the expected range. The ring system adopts an almost flat conformation (mean deviation from least-squares plane 0.0112 Å).

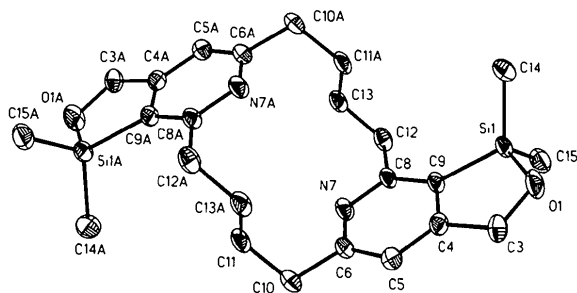


Fig. 1. *SHELXTL-Plus* drawing (Sheldrick, 1994) of the title compound. Displacement ellipsoids are shown at 50% probability. The H atoms are omitted for clarity.

Experimental

The title compound was crystallized by slowly diffusing pentane into a saturated solution of (3) in diethyl ether at room temperature.

Crystal data

C₂₄H₃₄N₂O₂Si₂

M_r = 438.71

Triclinic

P $\bar{1}$

a = 6.225 (2) Å

b = 9.369 (2) Å

c = 10.794 (2) Å

α = 87.54 (2)°

β = 73.81 (2)°

γ = 83.70 (2)°

V = 600.9 (3) Å³

Z = 1

D_x = 1.212 Mg m⁻³

Mo *K*α radiation

λ = 0.71073 Å

Cell parameters from 50

reflections

θ = 10–12.5°

μ = 0.170 mm⁻¹

T = 193 (2) K

Colourless

0.4 × 0.4 × 0.3 mm

Block

Data collection

Stoe Siemens Huber four-circle diffractometer

Profile data from 2 θ / ω scans

Absorption correction: none

3911 measured reflections

2730 independent reflections

2424 observed reflections

[*I* > 2σ(*I*)]

*R*_{int} = 0.0401

θ_{\max} = 27.60°

h = -7 → 8

k = -12 → 12

l = -12 → 14

3 standard reflections

monitored every 90

reflections

intensity decay: none

Refinement

Refinement on *F*²

R(*F*) = 0.0563

wR(*F*²) = 0.1609

S = 1.098

2730 reflections

138 parameters

H atoms riding

$w = 1/[\sigma^2(F_o^2) + (0.1035P)^2 + 0.1919P]$

where $P = (F_o^2 + 2F_c^2)/3$

(Δ/σ)_{max} < 0.001

$\Delta\rho_{\max} = 0.710 \text{ e } \text{Å}^{-3}$

$\Delta\rho_{\min} = -0.714 \text{ e } \text{Å}^{-3}$

Extinction correction: none

Atomic scattering factors

from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)

$$U_{eq} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^*$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{eq}
Si1	0.40179 (8)	0.83198 (5)	0.38385 (5)	0.0260 (2)
O1	0.3920 (3)	1.00008 (15)	0.32825 (14)	0.0374 (4)
C3	0.3187 (3)	1.0203 (2)	0.2144 (2)	0.0305 (4)
C4	0.2750 (3)	0.8773 (2)	0.1717 (2)	0.0252 (4)
C5	0.2083 (3)	0.8568 (2)	0.0625 (2)	0.0274 (4)
C6	0.1790 (3)	0.7178 (2)	0.0346 (2)	0.0271 (4)
N7	0.2068 (3)	0.6050 (2)	0.11094 (15)	0.0282 (3)
C8	0.2714 (3)	0.6257 (2)	0.2168 (2)	0.0239 (4)
C9	0.3096 (3)	0.7614 (2)	0.2512 (2)	0.0231 (4)
C10	0.1181 (3)	0.6849 (2)	-0.0863 (2)	0.0330 (4)
C11	0.2897 (3)	0.5727 (2)	-0.1687 (2)	0.0292 (4)
C12	0.3035 (3)	0.4930 (2)	0.2963 (2)	0.0297 (4)
C13	0.4756 (3)	0.3781 (2)	0.2185 (2)	0.0281 (4)
C14	0.6896 (3)	0.7666 (3)	0.3898 (2)	0.0417 (5)
C15	0.2022 (4)	0.8204 (2)	0.5458 (2)	0.0371 (5)

Table 2. Selected geometric parameters (Å, °)

Si1—O1	1.662 (2)	C6—N7	1.339 (2)
Si1—C14	1.846 (2)	C6—C10	1.509 (2)
Si1—C15	1.848 (2)	N7—C8	1.341 (2)
Si1—C9	1.854 (2)	C8—C9	1.403 (2)
O1—C3	1.424 (2)	C8—C12	1.507 (2)
C3—C4	1.511 (2)	C10—C11	1.530 (3)
C4—C5	1.382 (3)	C11—C13 ⁱ	1.525 (3)
C4—C9	1.391 (2)	C12—C13	1.528 (3)
C5—C6	1.391 (2)		
O1—Si1—C14	110.60 (10)	N7—C6—C10	115.8 (2)
O1—Si1—C15	110.18 (9)	C5—C6—C10	121.5 (2)
C14—Si1—C15	110.25 (11)	C6—N7—C8	118.9 (2)
O1—Si1—C9	93.74 (7)	N7—C8—C9	122.5 (2)
C14—Si1—C9	115.90 (9)	N7—C8—C12	115.65 (15)
C15—Si1—C9	114.99 (9)	C9—C8—C12	121.8 (2)
C3—O1—Si1	114.80 (11)	C4—C9—C8	117.5 (2)
O1—C3—C4	109.47 (15)	C4—C9—Si1	107.21 (12)
C5—C4—C9	120.3 (2)	C8—C9—Si1	135.33 (14)
C5—C4—C3	124.9 (2)	C6—C10—C11	111.92 (15)
C9—C4—C3	114.8 (2)	C13 ⁱ —C11—C10	113.33 (15)
C4—C5—C6	118.2 (2)	C8—C12—C13	112.40 (15)
N7—C6—C5	122.6 (2)	C11 ⁱ —C13—C12	112.94 (14)

Symmetry code: (i) 1 - *x*, 1 - *y*, -*z*.

Data were collected using the learnt-profile method (Clegg, 1981). The H atoms were included in calculated positions with isotropic displacement parameters set to 1.2 times (1.5 times for methyl groups) the displacement parameter of the atom to which they are attached.

Data collection: *DIF4* (Stoe & Cie, 1988a). Cell refinement: *DIF4*. Data reduction: *REDU4* (Stoe & Cie, 1988b). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *SHELXTL-Plus* (Sheldrick, 1994). Software used to prepare material for publication: *SHELXL93*.

We are grateful to the Deutsche Forschungsgemeinschaft and the Fonds der Chemischen Industrie for financial support.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: JZ1048). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1995). **C51**, 2147–2148

2-Hydroxymethyl-1-pentylbenzimidazole Hemi(1,4-dioxane) Solvate

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(Received 8 February 1995; accepted 18 May 1995)

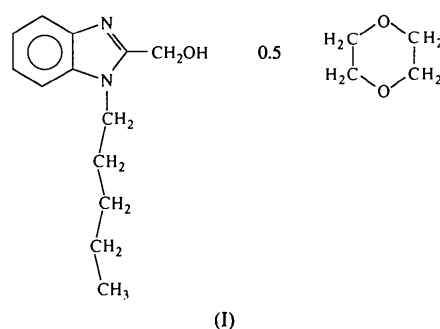
Abstract

2-Hydroxymethyl-1-pentylbenzimidazole serves as a simple model for the active site of hydrolytic metalloenzymes. The crystal structure shows that this compound crystallizes with 1,4-dioxane molecules: two benzimidazole units for one solvent molecule, $C_{13}H_{18}N_2O \cdot 0.5C_4H_8O_2$. The benzimidazole rings are hydrogen bonded ($O-H \cdots N$) into dimers.

Comment

2-Hydroxymethyl-1-pentylbenzimidazole was found to be active under its complexed form in the transesterification reaction of co-complexing activated esters (e.g. 4-nitrophenyl-5-alkoxypicolinic esters; Faivre, 1993) in cetyltrimethylammonium bromide (CTAB) micellar solutions. Indeed, this compound is capable, through its chelating sites, of forming active complexes

with bivalent metal ions (e.g. Zn^{2+}) in the presence of non-ionic or cationic surfactants. Unlike the 5(6)-alkyl-chain substituted homologues which give, whatever the surfactant concentration, an active complex with only a 2/1 stoichiometry, 2-hydroxymethyl-1-pentylbenzimidazole forms two active bidentate complexes simultaneously with 1/1 and 2/1 stoichiometry types at low surfactant concentrations (slightly above the cmc). At higher surfactant concentrations, these complexes evolve to only an active 2/1 complex type (Faivre, Brembilla, Roizard & Lochon, 1991). Therefore, the interpretation of the complexation mode as a function of the surfactant concentration initially prompted us to determine the crystal structure of this ligand (I) by X-ray crystallography.



The experimental intramolecular parameters (bond lengths and angles) of the heterocyclic ring are in good agreement with those determined for the non-substituted 2-hydroxymethylbenzimidazole (Aubry, Brembilla, Faivre & Lochon, 1995).

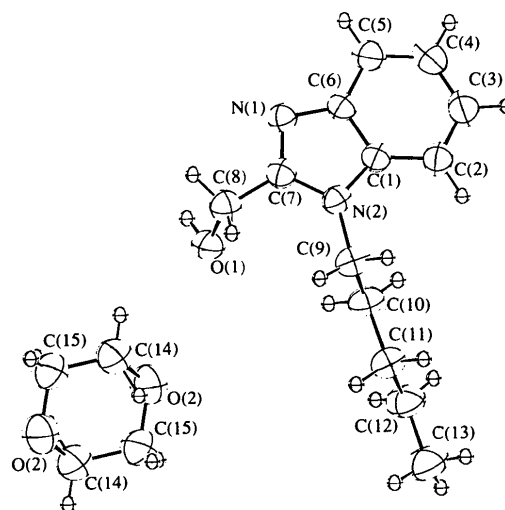


Fig. 1. ORTEP (Johnson, 1965) drawing of the title molecule with the atom-numbering scheme. Ellipsoids are plotted at the 50% probability level.