C(18)—C(11)—C(12)—C(13)	−61.0 (6)
C(11)—C(12)—C(13)=C(14)	−21.8 (8)
C(12)—C(13)=C(14)—C(15)	12.7 (7)
C(13)=C(14)—C(15)—C(16)	69.5 (6)
C(14)—C(15)—C(16)—C(17)	−60.7 (6)
C(15)—C(16)—C(17)=C(18)	−22.1 (8)
C(16)—C(17)=C(18)—C(11)	13.0 (7)
C(17)=C(18)—C(11)—C(12)	69.9 (6)

Table 3. *Geometrical characteristics of hydrogen bonds (Å, °)*

	N—H···S	H···S	N—H···S
(Thiourea)₃-1,5-cyclooctadiene* (173 K)	3.366(3)–3.457(3)	2.44(4)–2.66(3)	151(3)–172(3)
Pure thiourea† (110 K)	3.348(4)–3.417(6)	2.354(8)–2.415(10)	169.5(5)–170.5(7)
Pure thiourea† (298 K)	3.394(4)–3.526(8)	2.397(6)–3.055(12)	133.7(7)–168.9(5)

* This work.
† Elcombe & Taylor (1968).

The structure was solved by direct methods and difference Fourier synthesis using *SHELXS*86 (Sheldrick, 1985). The parameters were refined with *SHELX*76 (Sheldrick, 1976).

References

Adams, R. D., Chen, G., Wu, W. & Jin, Y. (1990). *Inorg. Chem.* **29**, 4208–4214.
Anderson, A. G., Calabrese, J. C., Tam, W. & Williams, I. D. (1987). *Chem. Phys. Lett.* **134**, 392–396.
Bengen, M. F. (1940). DP Patent 12 438 18.
Chenite, A. & Brisse, F. (1991). *Macromolecules*, **24**, 2221–2225.
Chenite, A. & Brisse, F. (1992). *Macromolecules*, **25**, 776–782.
Chenite, A. & Brisse, F. (1993). *Macromolecules*, **26**, 3055–3061.
Cromer, D. T. & Mann, J. B. (1968). *Acta Cryst.* A**24**, 321–324.
Elcombe, M. M. & Taylor, J. C. (1968). *Acta Cryst.* A**24**, 410–420.
Fait, J. F. (1987). PhD thesis, Montana State Univ., USA.
Fetterly, L. C. (1964). In *Non-Stoichiometric Compounds*, edited by L. Mandelcorn. New York: Academic Press.
Gopal, R., Robertson, B. E. & Rutherford, J. S. (1989). *Acta Cryst.* C**45**, 257–259.
Hagan, M. (1962). In *Clathrate Inclusion Compounds.* New York: Rheinhold.
Harris, K. D. M. & Thomas, J. M. (1990). *J. Chem. Soc. Faraday Trans.* **86**, 1095–1101.
Lenné, H. U. (1954). *Acta Cryst.* **7**, 1–16.
Ounsworth, J. P. & Weiler, L. (1987). *J. Chem. Educ.* **64**, 568–572.
Raymond, S. & Brisse, F. (1995). *Can. J. Chem.* Submitted.
Schiessler, R. W. & Flitter, D. (1952). *J. Am. Chem. Soc.* **74**, 1720–1723.
Sheldrick, G. M. (1976). *SHELX76. Program for Crystal Structure Determination.* Univ. of Cambridge, England.
Sheldrick, G. M. (1985). *SHELXS86. Program for the Solution of Crystal Structures.* Univ. of Göttingen, Germany.
Smith, A. E. (1952). *Acta Cryst.* **5**, 224–235.

Stewart, R. F., Davidson, E. R. & Simpson, W. T. (1965). *J. Chem. Phys.* pp. 3175–3187.
Truter, M. R. (1966). *Acta Cryst.* **22**, 556–559.

1-Cyclohexyl-2-cyclohexylamino-5(4H)-imidazolone

Romana Anulewicz, Dariusz Fiertek and Jan Izdebski

Department of Chemistry, Warsaw University, ul. Pasteura 1, 02-093 Warsaw, Poland
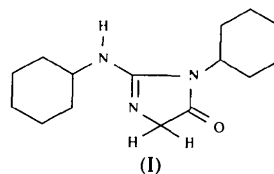
Abstract

The structure of the title compound, $C_{15}H_{25}N_3O$, a product of the reaction of glycine ethyl ester with N,N'-dicyclohexylcarbodiimide, was elucidated by X-ray diffraction. The imidazolone ring is planar and the two cyclohexyl rings adopt chair confirmations.

Comment

It has been reported (Muramatsu, 1961) that glycine ethyl ester reacts with N,N'-dicyclohexylcarbodiimide to form 1-cyclohexyl-2-cyclohexylamino-5(4H)-imidazolone, (I). The product was studied by IR spectroscopy and the structure proposed on the basis of analogy with guanidine formation observed for related reactions (DeTar, Silverstein & Rogers, 1966).

(I)

The title compound was synthesized by the method of Merrifield, Gisin & Bach (1977) and identified by comparison of its melting point with that given by Muramatsu (1961). A drawing of the molecule showing displacement ellipsoids is presented in Fig. 1.

Atom C4 is involved in three C—N bonds [C4—N1 1.405 (3), C4—N2 1.288 (2) and C4—N3 1.348 (6) Å] which are different from the bond lengths observed for fully delocalized π bonds (d_{opt} = 1.334 Å; Krygowski, 1993). The distances are similar to the values expected for localized bonds (Allen *et al.*, 1987). This suggests low (if any) delocalization of π electrons in this region.

The title structure has an intramolecular hydrogen bond: N3···N2ⁱ 3.058 (8), H3N···N2ⁱ 2.23 (5) Å, N3—H3N···N2ⁱ 159 (4)° [symmetry code: (i) $y-\frac{1}{2}$, $x-\frac{1}{2}$, $z-\frac{1}{2}$]. Its energy is estimated as 2.8 kJ using the empirical formula of Krygowski *et al.* (1991).
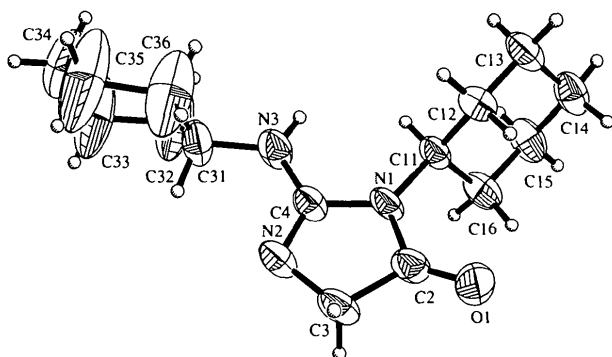
Fig. 1. View of the title molecule showing the atomic numbering system. Displacement ellipsoids are plotted at the 50% probability level.

Experimental

Crystal data

$C_{15}H_{25}N_3O$	Cu $K\alpha$ radiation
$M_r = 263.38$	$\lambda = 1.5418$ Å
Trigonal	Cell parameters from 25
$R3c$	reflections
$a = 17.158\ (2)$ Å	$\theta = 10–15°$
$\alpha = 116.66\ (2)°$	$\mu = 0.560$ mm^{-1}
$V = 2344.1\ (5)$ Å^3	$T = 293\ (2)$ K
$Z = 6$	Prism
$D_x = 1.119$ Mg m^{-3}	$0.33 \times 0.30 \times 0.28$ mm
	Colourless

Data collection

Kuma KM-4 diffractometer	$\theta_{max} = 70.07°$
$\omega/2\theta$ scans	$h = -20 \rightarrow 0$
Absorption correction:	$k = -8 \rightarrow 20$
none	$l = -9 \rightarrow 15$
4024 measured reflections	2 standard reflections
2705 independent reflections	monitored every 100
1688 observed reflections	reflections
$[I > 2\sigma(I)]$	intensity decay: <5%
$R_{int} = 0.0415$	

Refinement

Refinement on F^2	$(\Delta/\sigma)_{max} = -0.031$
$R(F) = 0.0493$	$\Delta\rho_{max} = 0.198$ e Å^{-3}
$wR(F^2) = 0.1482$	$\Delta\rho_{min} = -0.281$ e Å^{-3}
$S = 1.080$	Atomic scattering factors
2705 reflections	from *International Tables*
191 parameters	*for Crystallography* (1992,
$w = 1/[\sigma^2(F_o^2) + (0.0878P)^2$	Vol. C, Tables 4.2.6.8 and
$+ 0.0449P]$	6.1.1.4)
where $P = (F_o^2 + 2F_c^2)/3$	

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

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

	x	y	z	U_{eq}
N1	0.1626 (3)	0.2728 (2)	0.7415 (2)	0.0417 (6)
C2	0.2453 (3)	0.4137 (3)	0.8226 (3)	0.0472 (7)
O1	0.2191 (3)	0.4428 (3)	0.7720 (3)	0.0668 (7)
C3	0.3715 (2)	0.5180 (2)	0.98431 (15)	0.0519 (8)
N2	0.3478 (2)	0.4253 (2)	0.98449 (15)	0.0476 (6)
C4	0.2280 (2)	0.2896 (2)	0.84543 (15)	0.0411 (6)
C11	0.0300 (2)	0.1344 (2)	0.57589 (15)	0.0410 (7)
C12	-0.1028 (3)	0.0877 (4)	0.5060 (3)	0.0483 (7)
C13	-0.2391 (4)	-0.0569 (4)	0.3354 (4)	0.0578 (9)
C14	-0.2028 (4)	-0.0405 (4)	0.2732 (4)	0.0586 (9)
C15	-0.0709 (4)	0.0062 (4)	0.3433 (4)	0.0613 (9)
C16	0.0667 (4)	0.1493 (4)	0.5142 (3)	0.0535 (8)
N3	0.1634 (3)	0.1677 (3)	0.7964 (3)	0.0542 (7)
C31	0.2190 (4)	0.1799 (4)	0.9013 (4)	0.0605 (9)
C32	0.2150 (10)	0.0805 (10)	0.8543 (9)	0.124 (2)
C33	0.2781 (13)	0.0963 (13)	0.9675 (12)	0.149 (3)
C34	0.1876 (11)	0.0535 (10)	0.9730 (11)	0.134 (2)
C35	0.1876 (18)	0.1513 (19)	1.0189 (17)	0.195 (4)
C36	0.1400 (13)	0.1527 (14)	0.9189 (13)	0.148 (3)

Table 2. *Selected geometric parameters* (Å, °)

N1—C2	1.388 (4)	C13—C14	1.522 (12)
N1—C4	1.405 (3)	C14—C15	1.509 (10)
N1—C11	1.478 (2)	C15—C16	1.525 (6)
C2—O1	1.203 (9)	N3—C31	1.461 (10)
C2—C3	1.505 (5)	C31—C36	1.44 (3)
C3—N2	1.456 (5)	C31—C32	1.48 (3)
N2—C4	1.288 (2)	C32—C33	1.55 (3)
C4—N3	1.348 (6)	C33—C34	1.46 (3)
C11—C16	1.509 (8)	C34—C35	1.50 (4)
C11—C12	1.523 (7)	C35—C36	1.55 (4)
C12—C13	1.525 (6)		
C2—N1—C4	107.3 (3)	C11—C12—C13	110.5 (3)
C2—N1—C11	124.1 (3)	C14—C13—C12	111.1 (3)
C4—N1—C11	128.6 (2)	C15—C14—C13	111.0 (6)
O1—C2—N1	126.6 (3)	C14—C15—C16	112.0 (3)
O1—C2—C3	128.8 (3)	C11—C16—C15	111.2 (3)
N1—C2—C3	104.6 (3)	C4—N3—C31	121.0 (3)
N2—C3—C2	106.8 (2)	C36—C31—N3	112.2 (8)
C4—N2—C3	105.7 (3)	C36—C31—C32	112.9 (13)
N2—C4—N3	124.7 (2)	N3—C31—C32	109.4 (7)
N2—C4—N1	115.5 (1)	C31—C32—C33	109.7 (14)
N3—C4—N1	119.8 (2)	C34—C33—C32	110.3 (15)
N1—C11—C16	111.6 (2)	C33—C34—C35	110.0 (2)
N1—C11—C12	111.3 (2)	C34—C35—C36	112.0 (2)
C16—C11—C12	112.1 (2)	C31—C36—C35	113.0 (18)

Initially, we found the unit-cell parameters in the monoclinic system [a = 18.014 (5), b = 29.204 (8), c = 9.525 (4) Å, β = 110.66 (3)°], but could not solve the structure, so all the parameters were transformed to the trigonal cell. The structure was solved by direct methods and refined by full-matrix least-squares calculations. The H atoms were placed in calculated positions and refined with isotropic displacement parameters. The N3 H atom was obtained from a $\Delta\rho$ map and refined with an isotropic displacement parameter. Programs used were *SHELX*76 (Sheldrick, 1976), *SHELXS*86 (Sheldrick, 1990) and *SHELXL*93 (Sheldrick, 1993).

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–S19.

DeTar, D. F., Silverstein, R. & Rogers, F. F. Jr (1966). *J. Am. Chem. Soc.* **88**, 1024–1030.

Krygowski, T. M. (1993). *J. Chem. Inf. Comput. Sci.* **33**, 70–78.

Krygowski, T. M., Kalinowski, M. K., Turowska-Tyrk, I., Hiberty, P. C., Milart, P., Silvestro, A., Topsom, R. D. & Daehne, S. (1991). *Struct. Chem.* **2**, 71–80.

Merrifield, R. B., Gisin, B. F. & Bach, A. N. (1977). *J. Org. Chem.* **42**, 1291–1295.

Muramatsu, I. (1961). *Nippon Kagaku Zasshi*, **82**, 83; *Chem. Abstr.* (1962), **56**, 10273g.

Sheldrick, G. M. (1976). *SHELX76. Program for Crystal Structure Determination.* Univ. of Cambridge, England.

Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.

Sheldrick, G. M. (1993). *Program for the Refinement of Crystal Structures.* Univ. of Göttingen, Germany.

[Si1—Si2—Si3—C23 = −177.9 (1)°] conformation, with the two aromatic systems pointing away from each other.

The angle between the best planes through the pyrene and phenyl atoms is 56.2 (1)°. The maximum deviation from the best plane through the pyrene atoms is 0.030 (3) Å (for atom C1) and through the phenyl atoms the maximum deviation is 0.004 (3) Å. This structure is comparable to the recently reported structure of 1,3-bis(1-pyrenyl)hexamethyltrisilane (Van Meervelt, 1993), where the angle between the pyrene ring systems is 71° with a −anticlinal, +synclinal conformation for the trisilane chain.

1-[4-(Dimethylamino)phenyl]-3-(1-pyrenyl)hexamethyltrisilane

Luc Van Meervelt

Laboratorium voor Macromoleculaire Structuurchemie, KU Leuven, Celestijnenlaan 200F, B-3001 Leuven, Belgium
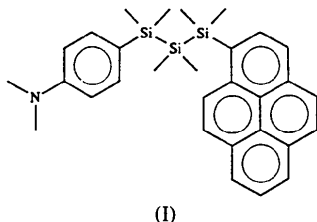
Abstract

The trisilane chain C1—Si1—Si2—Si3—C23 of the title compound, 1-[4-(dimethylamino)phenyl]-1,1,2,2,3,3-hexamethyl-3-(1-pyrenyl)trisilane, $C_{30}H_{37}NSi_3$, adopts a +synclinal, −antiperiplanar conformation. The two aromatic systems make an angle of 56.2 (1)° with each other.

Comment

Donor–acceptor substituted oligosilanes can be used as model systems for the study of through-bond interactions. The structure determination of the title compound, (I), was undertaken in order to establish the mutual orientation of the two aromatic systems, pyrene and phenyl, and to determine the conformation of the trisilane chain.

(I)

The molecular structure with atomic labelling is depicted in Fig. 1. The trisilane chain adopts a +synclinal [C1—Si1—Si2—Si3 = 48.2 (2)°], −antiperiplanar
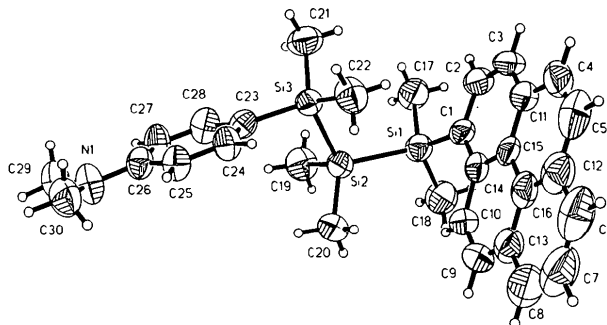

Fig. 1. View (*SHELXTL/PC*; Sheldrick 1990) of the title molecule with atomic labelling. Displacement ellipsoids are shown at 50% probability levels and H atoms are drawn as small circles of arbitrary size.

Experimental

Crystal data

$C_{30}H_{37}NSi_3$	Cu $K\alpha$ radiation
$M_r = 495.88$	$\lambda = 1.54178$ Å
Monoclinic	Cell parameters from 25
$P2_1/n$	reflections
$a = 7.8980 \ (10)$ Å	$\theta = 7$–23°
$b = 26.463 \ (4)$ Å	$\mu = 1.626$ mm^{-1}
$c = 14.099 \ (2)$ Å	$T = 289 \ (2)$ K
$\beta = 99.69 \ (2)°$	Block
$V = 2904.7 \ (7)$ Å^3	$0.20 \times 0.20 \times 0.10$ mm
$Z = 4$	Yellow
$D_x = 1.134$ Mg m^{-3}	Crystal source: grown from cyclohexane

Data collection

Siemens *P4/PC* diffractometer	$R_{int} = 0.0520$
θ–2θ scans	$\theta_{max} = 50.41°$
Absorption correction:	$h = -1 \rightarrow 7$
empirical	$k = -1 \rightarrow 26$
$T_{min} = 0.479$, $T_{max} = 0.991$	$l = -14 \rightarrow 14$
	3 standard reflections
4081 measured reflections	monitored every 100
3030 independent reflections	reflections
1827 observed reflections	intensity decay: none
$[I > 2\sigma(I)]$	