

and displacement-parameter restraints. Distance restraints were applied to refined hydroxy H atoms; other H atoms were constrained with a riding model and $U(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

For all compounds, data collection: *Nicolet P3 Software* (Nicolet XRD Corporation, 1980); cell refinement: *Nicolet P3 Software*; data reduction: *Nicolet P3 Software*; program(s) used to solve structures: *SHELXTL/PC* (Sheldrick, 1990); program(s) used to refine structures: *SHELXL93* (Sheldrick, 1993); molecular graphics: *SHELXTL/PC*; software used to prepare material for publication: *SHELXTL/PC*.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: CF1113). Services for accessing these data are described at the back of the journal.

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9a-Trimethylsilyloctahydroquinolizinium Picrate

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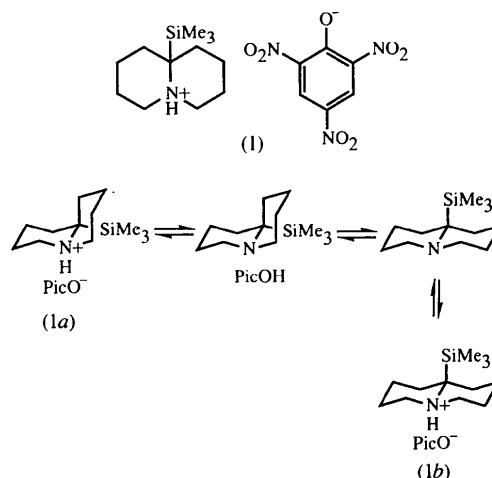
(Received 20 November 1996; accepted 22 April 1997)

Abstract

The title compound, $\text{C}_{12}\text{H}_{26}\text{NSi}^+\cdot\text{C}_6\text{H}_2\text{N}_3\text{O}_7^-$, exists in the solid state in a *cis*-decalin configuration, with the trimethylsilyl substituent at a bridgehead position. There exists a hydrogen bond between the ammonium N—H and the picrate O atom [N1—H1 0.90 (3), H \cdots O1, 2.03 (3) Å and N1—H1 \cdots O1 153 (2)°].

Comment

The title compound, (1), exists in solution as a *ca* 1:1 mixture of the *cis*-, (1a), and *trans*-decalin, (1b), configurations (White & Tham, 1997); these are interconvertible by a sequence of proton transfer to the picrate anion, nitrogen inversion, then protonation again (see scheme below). The structure of (1) was determined in order to obtain information on its preferred configuration in the solid state. It was then proposed to make use of solid-state ^{13}C NMR in order to assign the signals of the *cis*–*trans* mixture in solution.



The structure of (1), as depicted in Fig. 1, shows that it adopts a *cis*-decalin configuration in the solid state. The bridgehead trimethylsilyl substituent is in an equatorial position with respect to the ring defined by atoms N1, C6, C5, C4, C3 and C2, and axial to the ring defined by N1, C6, C7, C8, C9 and C10. As a result of the axial position of the trimethylsilyl substituent, one of the methyls (C13) lies across the ring, resulting in close contacts between the H atoms attached to C13 and the axial ring H atoms attached to C8 and C10 (H13A \cdots H8B 2.25 and H13B \cdots H10A 2.21 Å). The strain arising from the close contacts results in opening of the C13—Si—C6 bond angle to 118.2 (1)°, well past the preferred tetrahedral value, presumably to minimize these repulsive interactions. The Si—C6—C7 angle,

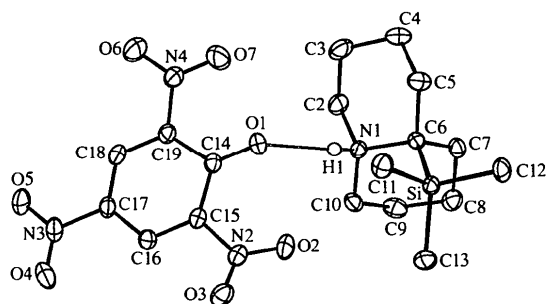


Fig. 1. ORTEP (Johnson, 1976) diagram of (1). Displacement ellipsoids are drawn at the 20% probability level.

113.8(1)°, also helps to minimize these non-bonded interactions. The bicyclic ammonium ion is hydrogen bonded to the picrate anion; H1···O1 2.03(3) Å and N1—H1···O1 153(2)°. This brings one of the *ortho*-nitro O atoms (O7) into close proximity to one of the methylene H atoms attached to C2; O7···H3A 2.45 Å, within the sum of the van der Waals radii (2.59 Å; Kitaigorodsky, 1973). There is high anisotropic thermal motion of the nitro O atoms; this is consistent with the expected 'soft' torsional motion about the C—N bonds.

Experimental

Crystals of (1) (422–425 K) were grown from ethanol/ether.

Crystal data

C₁₂H₂₆NSi⁺·C₆H₂N₃O₇⁻

M_r = 440.52

Monoclinic

*P*2₁/*c*

a = 11.871(1) Å

b = 21.586(2) Å

c = 8.484(1) Å

β = 102.58(1)°

V = 2121.8(4) Å³

Z = 1

D_x = 1.379 Mg m⁻³

D_m not measured

Mo Kα radiation

λ = 0.71069 Å

Cell parameters from 25 reflections

θ = 12–15°

μ = 0.158 mm⁻¹

T = 293(2) K

Thick plate

0.55 × 0.52 × 0.17 mm

Yellow

Data collection

Enraf–Nonius CAD-4S diffractometer

ω/2θ scans

Absorption correction:

Gaussian (SHELX76; Sheldrick, 1976)

T_{min} = 0.91, *T_{max}* = 0.97

4771 measured reflections

3712 independent reflections

3121 reflections with

I > 2σ(*I*)

R_{int} = 0.025

θ_{max} = 24.96°

h = -14 → 14

k = -25 → 1

l = -1 → 10

3 standard reflections

frequency: 120 min

intensity decay: 0.6%

Refinement

Refinement on *F*²

R[*F*² > 2σ(*F*²)] = 0.050

wR(*F*²) = 0.139

S = 1.034

3712 reflections

384 parameters

All H atoms refined

w = 1/[σ²(*F_o*²) + (0.0791*P*)² + 0.8915*P*]

where *P* = (*F_o*² + 2*F_c*²)/3

(Δ/σ)_{max} = -0.001

Δρ_{max} = 0.422 e Å⁻³

Δρ_{min} = -0.269 e Å⁻³

Extinction correction:

SHELXL93 (Sheldrick, 1993)

Extinction coefficient:

0.0034 (13)

Scattering factors from

International Tables for Crystallography (Vol. C)

C12—Si—C6	105.3 (1)	C4—C5—C6	116.0 (2)
C11—Si—C6	107.8 (1)	C7—C6—N1	107.9 (2)
C13—Si—C6	118.2 (1)	C7—C6—C5	110.2 (2)
C10—N1—C2	109.7 (2)	N1—C6—C5	108.4 (2)
C10—N1—C6	112.4 (2)	C7—C6—Si	113.77 (15)
C2—N1—C6	114.3 (2)	N1—C6—Si	112.16 (13)
C10—N1—H1	107.6 (16)	C5—C6—Si	104.27 (15)
C2—N1—H1	102.7 (16)	C8—C7—C6	114.6 (2)
C6—N1—H1	109.6 (16)	C7—C8—C9	110.6 (2)
C3—C2—N1	111.3 (2)	C10—C9—C8	111.8 (2)
C2—C3—C4	111.0 (2)	N1—C10—C9	111.7 (2)
C3—C4—C5	109.3 (2)		

The structure was solved by direct methods using SHELXS86 (Sheldrick, 1990). Refinement was performed using SHELXL-93 using anisotropic displacement parameters for all non-H atoms and isotropic displacement parameters for the H atoms. The figure was drawn using ORTEPII (Johnson, 1976) and tables prepared using SHELXL93. All calculations were carried out on a VAXstation 4000VLC computer system.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: SX1022). Services for accessing these data are described at the back of the journal.

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A Putrescine Triamide from *Lilium regale*

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Abstract

The asymmetric unit of the crystals of the synthetic equivalent of the terrestrial natural product (±)-(E)-3-(4-hydroxyphenyl)-N-[4-(3-methyl-2,5-dioxo-1-pyrrolidinyl)butyl]-2-propenamamide, C₁₈H₂₂N₂O₄, contains two independent but structurally similar molecules. The cinnamide, putrescine and succinimide regions of the molecule are each planar but lie almost perpendicular to the adjacent moiety. Hydrogen bonds link the mol-

Table 1. Selected geometric parameters (Å, °)

Si—C12	1.856 (3)	C2—C3	1.501 (4)
Si—C11	1.860 (3)	C3—C4	1.505 (5)
Si—C13	1.862 (3)	C4—C5	1.511 (4)
Si—C6	1.947 (2)	C5—C6	1.548 (3)
N1—C10	1.506 (3)	C6—C7	1.534 (3)
N1—C2	1.512 (3)	C7—C8	1.508 (4)
N1—C6	1.544 (3)	C8—C9	1.511 (4)
N1—H1	0.90 (3)	C9—C10	1.507 (4)