

Stereochemistry of a Bis(trimethylsilyl)butene Derivative

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Abstract. (Z)-1-[2-Methyl-1,4-bis(trimethylsilyl)-1-buten-3-yl]-4-phenyl-1*H*-1,2,4-triazole-3,5(2*H*,4*H*)-dione, C₁₉H₃₁N₃O₂Si₂, *M_r* = 389.65, triclinic, *P* $\bar{1}$, *a* = 11.326 (6), *b* = 12.194 (5), *c* = 8.586 (2) Å, α = 100.13 (3), β = 89.49 (3), γ = 92.75 (4)°, *V* = 1166.0 (8) Å³, *Z* = 2, *D_x* = 1.109 g cm⁻³, λ (Mo *K* α) = 0.71069 Å, μ = 1.63 cm⁻¹, *F*(000) = 420, *T* = 293 K, *R* = 0.050, *wR* = 0.052 for 3908 unique observed reflections [*F* > 3 σ (*F*)]. The substituent of the investigated urazole compound has *Z* configuration at the double bond. All observed bond lengths are within the expected ranges.

Experimental. The urazole compound (1) was prepared by Adam & Schwarm (1988). Single crystals obtained from H₂O/CH₃OH solution. A plate-shaped crystal of dimensions 0.9 × 1.2 × 0.15 mm was used on a Syntex P3 diffractometer, Mo *K* α radiation, unit-cell dimen-

sions from 15 centred reflections (9 < θ < 13°). ω scan used for data collection of 4036 unique reflections of which 3908 were observed with *F* > 3 σ (*F*). According to the pre-scan intensity the ω -scan speed ranged from 0.5 to 29.3 °min⁻¹ and weak reflections were omitted. Absorption correction based on ψ scans of 11 reflections. Diffraction intensities were measured up to $\sin\theta/\lambda = 0.65$ Å⁻¹ in the index range *h* = 0→14, *k* = -15→15 and *l* = -11→11. One standard reflection (234) varied less than 2.5% over 95.5 h of data collection. Solved by direct phase determination, *E*_{min} = 1.7. Block-diagonal-matrix least-squares refinements minimized $\sum w(\Delta F)^2$; H-atom positions calculated geometrically and considered isotropically with *U* = 1.2 × *U* of bonded C, the position of the nitrogen-bonded H atom was found in difference Fourier maps and refined isotropically. Positions and thermal parameters of all other non-H atoms refined anisotropically giving 239

Table 1. Final atomic coordinates (× 10⁴) and equivalent isotropic thermal parameters (Å² × 10³) with their *e.s.d.*'s in parentheses

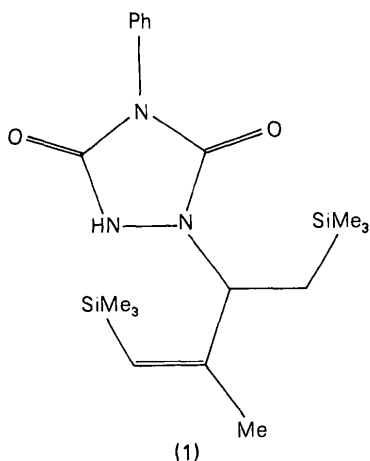
$$U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{eq}
Si(1)	6190 (1)	7162 (1)	1361 (1)	63 (1)
Si(2)	2040 (1)	9725 (1)	2701 (1)	67 (1)
N(1)	3660 (2)	6552 (2)	3128 (2)	52 (1)
N(2)	4108 (2)	5911 (2)	4184 (2)	58 (1)
C(3)	3386 (2)	4994 (2)	4181 (3)	52 (1)
N(4)	2382 (2)	5156 (2)	3329 (2)	49 (1)
C(5)	2534 (2)	6145 (2)	2719 (3)	50 (1)
C(6)	4038 (2)	7742 (2)	3235 (3)	49 (1)
C(7)	3483 (2)	8481 (2)	4626 (3)	55 (1)
C(8)	2717 (2)	9236 (2)	4427 (3)	64 (1)
C(9)	5386 (2)	7861 (2)	3175 (3)	54 (1)
C(11)	5405 (3)	7385 (3)	-451 (4)	93 (1)
C(12)	7693 (3)	7848 (3)	1478 (4)	103 (2)
C(13)	6325 (3)	5643 (3)	1296 (4)	83 (1)
C(21)	453 (4)	9258 (4)	2594 (6)	146 (2)
C(22)	2177 (3)	11258 (3)	3166 (4)	95 (1)
C(23)	2709 (4)	9265 (3)	743 (4)	121 (2)
O(30)	3573 (1)	4186 (1)	4793 (2)	67 (1)
C(41)	1328 (2)	4440 (2)	3174 (3)	51 (1)
C(42)	924 (2)	3973 (2)	4445 (3)	60 (1)
C(43)	-99 (2)	3289 (2)	4270 (4)	76 (1)
C(44)	-704 (3)	3092 (3)	2866 (4)	91 (1)
C(45)	-305 (3)	3567 (3)	1614 (4)	92 (1)
C(46)	721 (2)	4239 (2)	1749 (3)	70 (1)
O(50)	1826 (1)	6562 (1)	1974 (2)	64 (1)
C(70)	3887 (3)	8348 (3)	6263 (3)	85 (1)

Table 2. Selected interatomic distances (Å) and angles (°)

Si(1)–C(9)	188.8 (2)	Si(1)–C(11)	186.5 (3)
Si(1)–C(12)	185.6 (3)	Si(1)–C(13)	185.7 (3)
Si(2)–C(8)	187.5 (3)	Si(2)–C(21)	185.7 (4)
Si(2)–C(22)	184.1 (3)	Si(2)–C(23)	184.5 (4)
N(1)–N(2)	140.9 (3)	N(1)–C(5)	137.1 (3)
N(1)–C(6)	148.0 (3)	N(2)–H(2)	103.3 (2)
N(2)–C(3)	135.3 (3)	C(3)–N(4)	139.8 (3)
C(3)–O(30)	122.3 (3)	N(4)–C(5)	139.9 (3)
N(4)–C(41)	143.8 (3)	C(5)–O(50)	121.4 (3)
C(6)–C(7)	151.6 (3)	C(6)–C(9)	152.8 (3)
C(7)–C(8)	132.7 (4)	C(7)–C(70)	151.9 (4)
C(9)–Si(1)–C(11)	109.5 (1)	C(9)–Si(1)–C(12)	105.5 (1)
C(11)–Si(1)–C(12)	110.8 (2)	C(9)–Si(1)–C(13)	113.1 (1)
C(11)–Si(1)–C(13)	109.2 (1)	C(12)–Si(1)–C(13)	108.7 (2)
C(8)–Si(2)–C(21)	108.3 (2)	C(8)–Si(2)–C(22)	105.3 (1)
C(21)–Si(2)–C(22)	109.7 (2)	C(8)–Si(2)–C(23)	116.9 (2)
C(21)–Si(2)–C(23)	108.2 (2)	C(22)–Si(2)–C(23)	108.4 (2)
N(2)–N(1)–C(5)	107.9 (2)	N(2)–N(1)–C(6)	120.8 (2)
C(5)–N(1)–C(6)	122.9 (2)	N(1)–N(2)–H(2)	118.7 (2)
N(1)–N(2)–C(3)	109.1 (2)	H(2)–N(2)–C(3)	117.2 (2)
N(2)–C(3)–N(4)	105.9 (2)	N(2)–C(3)–O(30)	127.1 (2)
N(4)–C(3)–O(30)	127.0 (2)	C(3)–N(4)–C(5)	109.9 (2)
C(3)–N(4)–C(41)	124.8 (2)	C(5)–N(4)–C(41)	125.2 (2)
N(1)–C(5)–N(4)	105.8 (2)	N(1)–C(5)–O(50)	126.6 (2)
N(4)–C(5)–O(50)	127.6 (2)	N(1)–C(6)–C(7)	112.5 (2)
N(1)–C(6)–C(9)	109.7 (2)	C(7)–C(6)–C(9)	114.4 (2)
C(6)–C(7)–C(8)	121.8 (2)	C(6)–C(7)–C(70)	116.5 (2)
C(8)–C(7)–C(70)	121.6 (2)	Si(2)–C(8)–C(7)	136.2 (2)
Si(1)–C(9)–C(6)	119.3 (1)	N(4)–C(41)–C(42)	119.9 (2)
N(4)–C(41)–C(46)	119.2 (2)		

variables. $R = 0.050$, $wR = 0.052$, $S = 4.20$, where $w^{-1} = \sigma^2(F)$. Final $(\Delta/\sigma)_{\max} = 0.23$, $\Delta\rho_{\max} = 0.3$ and $\Delta\rho_{\min} = -0.3 \text{ e } \text{\AA}^{-3}$ in final difference Fourier map. Atomic scattering factors taken from *SHELXTL* program (Sheldrick, 1985). Table 1 gives the atom parameters and Table 2 selected bond distances and angles. Fig. 1 shows the molecule with the atom labelling.*



* Lists of all bond lengths and angles, atomic coordinates of H atoms, anisotropic thermal parameters, as well as those of structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51770 (30 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

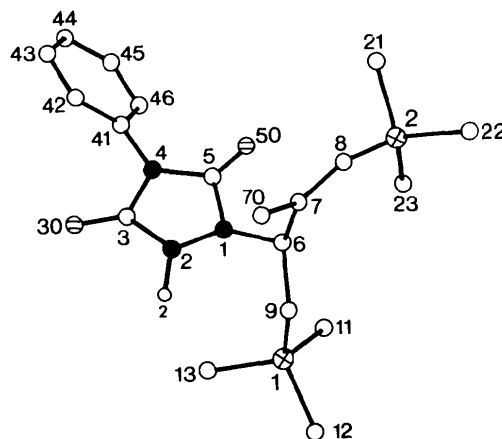


Fig. 1. A view of the molecule with the numbering scheme corresponding to Tables 1 and 2. White, black, lined, and crossed circles represent C, N, O, and Si atoms, respectively.

Related literature. (1) was synthesized by reaction of (*Z*)-2-methyl-1,4-bis(trimethylsilyl)-2-butene with 4-phenyl-1*H*-1,2,4-triazole-3,5-(2*H*,4*H*)-dione (Adam & Schwarm, 1988). Mechanistical studies of related reactions have also been reported by Dubac & Laporterie (1987).

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Structure du Diméthylamino-4 Ethoxycarbonyl-4 Phényl-2 Thia-1 Aza-3 Butadiène

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Abstract. Ethyl dimethylamino(phenylthiocarbonylimino)acetate, $\text{C}_{13}\text{H}_{16}\text{N}_2\text{O}_2\text{S}$, $M_r = 264.4$, orthorhombic, $P2_12_12_1$, $a = 7.920$ (4), $b = 10.813$ (3), $c = 16.007$ (4) Å, $U = 1370.7$ (9) Å³, $Z = 4$, $D_x = 1.28 \text{ g cm}^{-3}$, $\lambda(\text{Mo K}\alpha) = 0.71069$ Å, $\mu = 2.2 \text{ cm}^{-1}$, $F(000) = 560$, $T = 296 \text{ K}$, final $R = 0.050$ for 758

observations. The X-ray structure allows the reactivity of this compound to be explained.

Partie expérimentale. L'intérêt présenté par les réactions de cycloadditions 4 + 2 effectuées à partir du dérivé thia-1 aza-3 butadiène (I) (Tea, Pradère, Quiniou