Regio- and Diastereoselective Ene Reaction of Triazolinedione with Vinylsilanes

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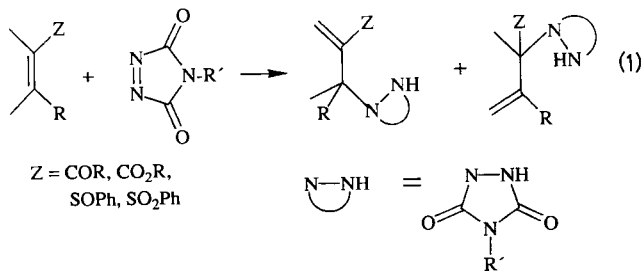
The ene reaction of 4-methyl-1,2,4-triazoline-3,5-dione (MTAD) with vinylsilanes **1** has been investigated. In all cases studied, only hydrogen abstraction geminal to the silyl group with formation of triazolidinediones **3** occurred, irrespective of the number and size of the substituents at the double bond. A mechanism with an aziridinium imide as intermediate is proposed to explain the observed regioselectivity and diastereoselectivity. For the former we invoke preferential cleavage of the C–N bond proximate to the silyl substituent on the aziridine ring, for the latter steric repulsion between the adjacent silyl and alkyl group controls the observed *E*-type stereochemistry. A new synthetic approach to the trisubstituted vinylsilane **1e** based on reductive lithiation of the corresponding vinyl sulfide is described.

The ene reaction of triazolinediones with olefins, although discovered more than twenty years ago[1], is still subject of intensive investigation[2]. Among other criteria, the regiochemical course of the ene reaction has been used to gain insight into the complex mechanism[3]. One important feature that has been recognized is the geminal selectivity[4], i.e. the predominant and in some cases even exclusive abstraction of allylic hydrogens geminal to functional groups such as ketones[5], esters[6], sulfoxides[7], and sulfones[4] (Eq. 1).

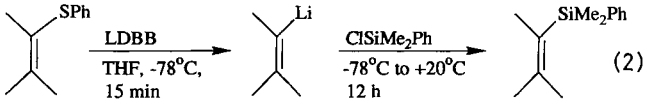


(1)

Recently[4], one example also was reported in which the trimethylsilyl group of a vinylsilane induced geminal selectivity in the reaction with 4-phenyl-1,2,4-triazoline-3,5-dione (PTAD). This urges us to present our results on this subject, which show that this selectivity is general for vinylsilanes, irrespective of substitution pattern or double bond geometry.

Results

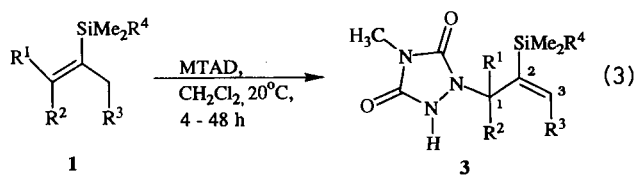
The required vinylsilanes **1a–d** were prepared by literature methods (cf. Experimental). For the trisubstituted derivative **1e** a novel synthetic concept was used, based on the recent finding[8] that cyclic vinyl sulfides are transformed to vinyllithium derivatives by reductive lithiation with lithium 4,4′-di-*tert*-butylbiphenylide (LDBB). Reaction of the acylic sulfide **2**[9] with LDBB (Eq. 2) afforded the corresponding vinyllithium compound, which was coupled with chlorodimethylphenylsilane to give **1e** in 66% yield. Modification of the original workup procedure allowed the preparation of **1e** on gram scale. This convenient and general synthesis of vinylsilanes[10] may also be extended to vinylstannanes[11].



(2)

2 **1e**

For the reaction of the silanes with 4-methyl-1,2,4-triazoline-3,5-dione (MTAD), equimolar amounts of the reactants were mixed in dichloromethane and stirred at room temperature until the red color of MTAD had disappeared (Table 1). Removal of the solvent gave the crystalline 1,2,4-triazolidine-3,5-diones (urazoles) **3a–e** quantitatively; no other products could be detected in the ^{1}H-NMR spectrum of the crude reaction mixture (Eq. 3). Only the isomers which result from hydrogen abstraction geminal to the R_3Si group were obtained in each case, as is easily deduced from the ^{1}H- and ^{13}C-NMR spectra. For urazole **3c** only one double bond diastereomer was observed, whose structure was assigned the *E* configuration by NOE experiments. Irradiation of the $SiMe_3$ protons resulted in strong enhancement (15%) of the vinyl proton. Moreover, irradiation of the proton next to the urazole ring (R^2) exhibited an NO effect (4%) on the allylic methyl group (R^3) and vice versa (5%).



(3)

1 **3**

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Table 1. Reaction of vinylsilanes **1a–e** with MTAD

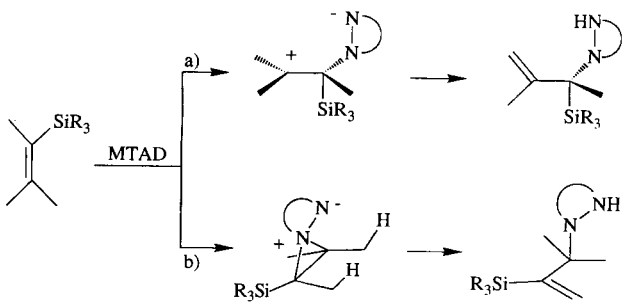
	Vinylsilane **1** R^1	R^2	R^3	R^4	Reaction time [h]	Yield of **3** (%)
a[a]	H	Me	H	Ph	18	99
	Me	H	H	Ph		
b	*n*Bu	H	H	Me	24	99
c	*n*Bu	H	Me	Me	4	97
d	H	$-[CH_2]_3-$		Me	48	98
e	Me	Me	H	Ph	24	97

[a] A mixture of diastereomers ($E:Z$ = 34:66) was employed.

Discussion

These results clearly show the profound effect of the trialkylsilyl group on the regio- and diastereoselectivity of the ene reaction of MTAD. The regiocontrol is not influenced by the number or size of the substituents on the double bond nor by its stereochemistry, which is quite in contrast to α,β-unsaturated ketones, for which only α,β-dialkyl-substituted derivatives show geminal selectivity[6]. A 1,4-dipolar intermediate (Scheme 1, path a), which has been invoked to explain the products of the reaction of PTAD with allyltrimethylsilane[12], can be excluded in the present case. Because of the well-established ability of silicon to stabilize β-cations[13], the most stable 1,4-dipole should be the one depicted in Scheme 1. The latter would, however, after proton abstraction result in the regioisomer which is not observed. The absence of 1,4-dipoles has recently been concluded on the basis of the analogous regioselectivity of the ene reaction for carbonyl enophiles with vinylsilanes[14].

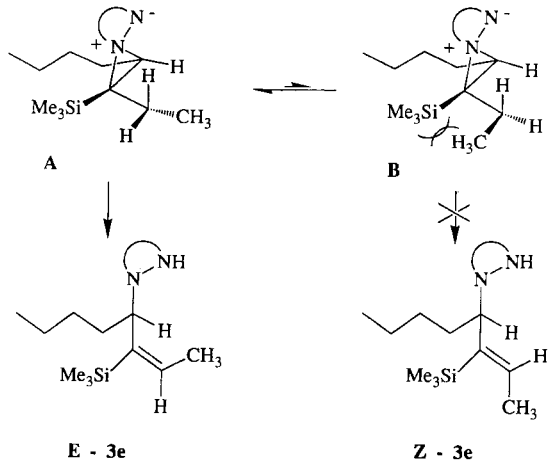
Scheme 1



Alternatively, an aziridinium imide (Scheme 1, path b) may operate as an intermediate, for which kinetic[3c] and direct spectral[15] evidence has been obtained. Since it is known that nucleophilic opening of silylaziridinium ions proceeds by rupture of the C–N bond proximate to silicon[16], the aziridinium imide of vinylsilanes should also cleave at the C–N bond adjacent to the SiR_3 group and thereby lead to the observed regioisomer. In analogy to the observed ring opening of epoxysilanes[17], the propensity of cleaving the C–N bond next to the silicon atom can be explained in terms of the antibonding interaction between the C–Si σ bond orbital and the occupied urazole orbital. In view of the higher orbital energy for the C–Si bond, these interactions are more effective than for an alkyl-substituted aziridinium imide and hence the observed regioselectivity is high. A similar theoretical analysis has been offered to explain the regioselectivity observed in the related ene reaction of singlet oxygen with vinylsilanes[17].

The exclusive formation of the *E* isomer of urazole **3c** can be understood by comparing the two possible conformations **A** and **B** (Scheme 2). Steric interaction of the methyl group is less severe in **A** than in **B**, so that **A** is preferred to the extent that only **E–3c** is formed.

Scheme 2



Besides its mechanistic implications, the profound effect of the trialkylsilyl group on the regio- and stereochemistry of the ene reaction of MTAD could also be of synthetic interest. The readily prepared urazoles **3** may serve as novel building blocks for syntheses, because they contain both the versatile vinylsilane function[18] and the urazole moiety, the latter constituting a potential precursor to an amino group[19].

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Experimental

Melting points: Büchi 535. – IR: Perkin-Elmer, 1420. – ^{1}H NMR: Bruker AC 250 (250 MHz), $CDCl_3$ (δ = 7.26) as internal standard. – ^{13}C NMR: Bruker AC 250 (63 MHz), $CDCl_3$ (δ = 77.0) as internal standard. – All solvents were purified by standard literature methods; 4-methyl-1,2,4-triazoline-3,5-dione (MTAD)[20], 4,4′-di-*tert*-butylbiphenyl[21], (1,2-dimethyl-1-propenylthio)benzene (**2**)[9] and silane **1d**[22] were prepared by following the published procedures. Silane **1b** was obtained by hydromagnesation of 1-hexynyltrimethylsilane, followed by coupling with methyl iodide according to the literature procedure[23] and purified by column chromatography on silica gel with petroleum ether as eluent. Silane **1c**[24] was synthesized analogously by employing ethyl iodide. The isomeric purities of **1b, c** were determined by ^{1}H-NMR spectroscopy to be >95%. Silane **1a**[25] ($E:Z$ = 34:66) was prepared by coupling of commercial 2-bromo-2-butene with chlorodimethylphenylsilane by following the published procedure[26] and purified by column chromatography.

(1,2-Dimethyl-1-propenyl)dimethylphenylsilane (**1e**): An oven-dried flask was charged under argon with 6.66 g (25.0 mmol) of 4,4′-di-

tert-butylbiphenyl and 65 ml of THF (freshly distilled from potassium). 190 mg (27.5 mmol) of lithium wire was washed free from mineral oil with petroleum ether and cut directly into the flask under a rapid stream of argon. After 5 min a deep blue-green color developed, and the mixture was stirred at 0°C for 5 h. After cooling to −78°C, 2.00 g (11.2 mmol) of **2** was added in 15 min. The red-colored mixture was stirred for additional 15 min at −78°C, treated with 4.27 g (25.0 mmol) of chlorodimethylphenylsilane, and allowed to reach room temp. overnight. 50 ml of *tert*-butyl methyl ether and 20 ml of 10% aqu. NaOH solution were added and the phases separated. The aqueous phase was extracted with *tert*-butyl methyl ether (3 × 20 ml), and the combined organic phases were washed with 10% aqu. NaOH solution (3 × 20 ml), with water (10 ml), and finally with satd. aqu. NaCl solution (10 ml). After removal of the solvent (ca. 20°C/20 Torr), the residue was repeatedly suspended in ethanol, the insoluble di-*tert*-butylbiphenyl removed by filtration, washed with ethanol, and the filtrate concentrated to result in an oily residue. Final purification by column chromatography (silica gel, petroleum ether) afforded 1.51 g (66%) of **1e**. – IR (neat): $\tilde{\nu}$ = 3070 cm^{-1} (m), 3000 (sh), 2960 (s), 2920 (s), 2860 (sh), 1620 (m), 1430 (s), 1250 (s), 1110 (s), 845 (sh), 830 (s), 810 (m), 775 (m), 730 (w), 705 (s), 665 (m). – ^{1}H NMR (250 MHz, $CDCl_3$): δ = 0.40 (s, 6H, $SiCH_3$), 1.74 (m, 6H), 1.80 (br. s, 3H), 7.32–7.40 (m, 3H, arom. H), 7.55–7.60 (m, 2H, arom. H). – ^{13}C NMR (63 MHz, $CDCl_3$): δ = −0.4 (q, $SiCH_3$), 18.4 (q), 21.3 (q), 25.9 (q), 124.8 (s), 127.9 (d), 128.5 (d), 133.8 (d), 140.6 (s), 144.4 (s).

$C_{13}H_{20}Si$ (204.4) Calcd. C 76.39 H 9.86
Found C 76.78 H 10.26

General Procedure for the Reaction of MTAD with Vinylsilanes **1a–e**: A solution of 1.0 equiv. of MTAD in CH_2Cl_2 was added to a solution of the silane **1a–e** in CH_2Cl_2 and stirred at ca. 20°C until the red color of MTAD had disappeared. Evaporation of the solvent at ca. 20°C/20 Torr gave the urazoles **3a–e** in quantitative yield. Analytical samples were prepared by recrystallisation.

1-[2-(Dimethylphenylsilyl)-1-methyl-2-propenyl]-4-methyl-1,2,4-triazolidine-3,5-dion (**3a**): According to the general procedure 301 mg (99%) of **3a** was obtained by starting from 190 mg (1.00 mmol) of **1a** and 113 mg (1.00 mmol) of MTAD in 20 ml of CH_2Cl_2. Recrystallisation from ether gave colorless cubes, m.p. 73–74°C. – IR (KBr): $\tilde{\nu}$ = 3180 cm^{-1} (br, m), 3090 (w), 3080 (sh), 1760 (m), 1690 (s), 1490 (s), 1260 (m), 1255 (m), 945 (m), 850 (m), 830 (s), 790 (m), 705 (m). – ^{1}H NMR (250 MHz, $CDCl_3$): δ = 0.42, 0.48 (s, 6H, $SiCH_3$), 1.25 (d, *J* = 6.6 Hz, 3H, CH_3), 2.93 (s, 3H, NCH_3), 4.93 (qt, *J* = 6.6/1.7 Hz, 1H, 1-H), 5.71 (t, *J* = 1.6 Hz, 1H, 3-H), 5.92 (t, *J* = 1.7 Hz, 1H, 3-H), 6.10 (br. s, 1H, NH), 7.28–7.48 (m, 5H, arom. H). – ^{13}C NMR (63 MHz, $CDCl_3$): δ = −4.3, −3.0 (q, $SiMe_2$), 14.7 (q), 25.1 (q, NCH_3), 54.1 (d, C-1), 128.1, 129.6, 133.3 (d, arom. C), 128.7 (t, C-3), 137.3 (s, arom. C), 148.2 (s, C-2), 153.4, 155.0 (s, C=O). – MS (70 eV): *m/z* (%) = 303 (0.01) [M^+], 249 (3), 226 (5), 225 (27) [$M^+ - C_6H_6$], 210 (10), 171 (4), 137 (5), 136 (14), 135 (100) [Me_2PhSi^+], 107 (6), 105 (5).

$C_{15}H_{21}N_3O_2Si$ (303.4) Calcd. C 59.37 H 6.98 N 13.85
Found C 59.30 H 7.24 N 14.05

1-[1-Butyl-2-(trimethylsilyl)-2-propenyl]-4-methyl-1,2,4-triazolidine-3,5-dione (**3b**): According to the general procedure 282 mg (99%) of **3b** was obtained by starting from 170 mg (1.00 mmol) of **1b** and 113 mg (1.00 mmol) of MTAD in 10 ml of CH_2Cl_2. Recrystallisation from water/methanol gave a colorless powder, m.p. 90–91°C. – IR (KBr): $\tilde{\nu}$ = 3140–3300 cm^{-1} (m), 2980 (w), 1775 (m), 1700 (s), 1490 (m), 1260 (sh), 1255 (m), 940 (w), 860 (m), 765 (w). – ^{1}H NMR (250 MHz, $CDCl_3$): δ = 0.10 (s, 9H, $SiCH_3$), 0.87 (t, *J* = 7.0 Hz, 3H, CH_3), 1.05–1.40 (m, 4H), 1.76 (q, *J* = 7.5 Hz, 2H), 3.07 (s, 3H, NCH_3), 4.79 (t, *J* = 7.5 Hz, 1H, 1-H), 5.65, 5.88 (m, 2H, 3-H), 8.32 (br. s, 1H, NH). – ^{13}C NMR (63 MHz, $CDCl_3$): δ = 1.6 (q, $SiCH_3$), 17.1 (q), 25.6 (t), 28.4 (q, NCH_3), 31.5 (t), 33.1 (t), 60.5 (d, C-1), 130.7 (t, C-3), 151.8 (s, C-2), 156.0, 158.1 (s, C=O). – MS (70 eV): *m/z* (%) = 283 (0.4) [M^+], 187 (12), 172 (4), 115 (3), 95 (2), 75 (5), 74 (9), 73 (100) [$SiMe_3^+$], 59 (6), 58 (4), 45 (9), 43 (7).

$C_{13}H_{25}N_3O_2Si$ (283.4) Calcd. C 55.09 H 8.89 N 14.82
Found C 55.30 H 9.21 N 14.81

(E)-1-[1-Butyl-2-(trimethylsilyl)-2-butenyl]-4-methyl-1,2,4-triazolidine-3,5-dione (**3c**): According to the general procedure 290 mg (97%) of **3c** was obtained by starting from 184 mg (1.00 mmol) of **1c** and 113 mg (1.00 mmol) of MTAD in 30 ml of CH_2Cl_2. Recrystallisation from water/methanol gave colorless needles, m.p. 108–109°C. – IR (KBr): $\tilde{\nu}$ = 3200–3320 cm^{-1} (w), 2990 (w), 2960 (sh), 1785 (m), 1715 (s), 1505 (m), 1260 (w), 860 (m), 845 (m), 770 (w). – ^{1}H NMR (250 MHz, $CDCl_3$): δ = 0.06 (s, 9H, $SiCH_3$), 0.89 (t, *J* = 7.0 Hz, 3H, CH_3), 1.15–1.40 (m, 4H), 1.55–1.70 (m, 1H), 1.82 (d, *J* = 6.8 Hz, 3H, =CH–CH_3), 1.85–2.05 (m, 1H), 3.03 (s, 3H, NCH_3), 5.02 (t, *J* = 7.8 Hz, 1H, 1-H), 6.13 (q, *J* = 6.8 Hz, 1H, 3-H), 9.00 (br. s, 1H, NH). – ^{13}C NMR (63 MHz, $CDCl_3$): δ = 0.3 (q, $SiCH_3$), 13.9 (q), 15.4 (q), 22.0 (t), 24.9 (q, NCH_3), 28.6 (t), 31.6 (t), 57.2 (d, C-1), 139.1 (s, C-2), 140.9 (d, C-3), 152.8, 155.5 (s, C=O). – MS (70 eV): *m/z* (%) = 297 (0.07) [M^+], 187 (11), 183 (6), 172 (7), 127 (3), 109 (5), 99 (3), 75 (6), 74 (13), 73 (100) [$SiMe_3^+$], 59 (7), 50 (9).

$C_{14}H_{27}N_3O_2Si$ (297.5) Calcd. C 56.53 H 9.14 N 14.13
Found C 56.30 H 9.49 N 14.30

4-Methyl-1-[2-(trimethylsilyl)-2-cyclohexen-1-yl]-1,2,4-triazolidine-3,5-dione (**3d**): According to the general procedure 261 mg (98%) of **3d** was obtained by starting from 154 mg (1.00 mmol) of **1d** and 113 mg (1.00 mmol) of MTAD in 25 ml of CH_2Cl_2. Recrystallisation from CH_2Cl_2/petroleum ether gave colorless needles, m.p. 144–145°C. – IR (KBr): $\tilde{\nu}$ = 3180–3300 cm^{-1} (w), 2950 (w), 1760 (m), 1680 (s), 1475 (s), 1245 (m), 1030 (w), 860 (w), 830 (m), 765 (w). – ^{1}H NMR (250 MHz, $CDCl_3$): δ = 0.03 (s, 9H, $SiCH_3$), 1.50–2.20 (m, 6H), 3.06 (s, 3H, NCH_3), 4.83 (m, 1H, 1-H), 6.40 (m, 1H, 3-H), 7.60 (br. s, 1H, NH). – ^{13}C NMR (63 MHz, $CDCl_3$): δ = −1.6 (q, $SiCH_3$), 19.1 (t), 25.1 (q, NCH_3), 26.1 (t), 27.3 (t), 52.4 (d, C-1), 134.8 (s, C-2), 144.6 (d, C-3), 152.6, 154.3 (s, C=O). – MS (70 eV): *m/z* (%) = 267 (0.7) [M^+], 172 (6), 153 (17), 115 (3), 79 (13), 74 (9), 73 (100) [$SiMe_3^+$], 59 (10), 58 (4), 45 (7).

$C_{12}H_{21}N_3O_2Si$ (267.4) Calcd. C 53.90 H 7.91 N 15.71
Found C 53.38 H 8.01 N 15.87

1-[2-(Dimethylphenylsilyl)-1,1-dimethyl-2-propenyl]-4-methyl-1,2,4-triazolidine-3,5-dione (**3e**): According to the general procedure 154 mg (97%) of **3e** was obtained by starting from 102 mg (0.500 mmol) of **1e** and 56.0 mg (0.500 mmol) of MTAD in 10 ml of CH_2Cl_2. Recrystallisation from CH_2Cl_2/petroleum ether gave colorless cubes, m.p. 93–94°C. – IR (KBr): $\tilde{\nu}$ = 3200–3260 cm^{-1} (m), 1760 (m), 1690 (s), 1480 (m), 1255 (w), 1110 (w), 940 (w), 840 (w), 830 (m), 785 (m). – ^{1}H NMR (250 MHz, $CDCl_3$): δ = 0.48 (s, 6H, $SiCH_3$), 1.50 (s, 6H, CH_3), 2.92 (s, 3H, NCH_3), 5.67 (d, *J* = 0.9 Hz, 1H, 3-H), 5.91 (d, *J* = 0.9 Hz, 1H, 3-H), 7.25–7.35 (m, 3H, arom. H), 7.45–7.53 (m, 2H, arom. H), 7.85 (br. s, 1H, NH). – ^{13}C NMR (63 MHz, $CDCl_3$): δ = 1.0 (q, $SiCH_3$), 26.8 (q), 28.3 (q, NCH_3), 67.8 (s, C-1), 129.7 (t, C-3), 129.9 (d), 131.2 (d), 135.7 (d), 140.1 (s), 150.0 (s, C-2), 155.6, 156.5 (s, C=O). – MS (70 eV): *m/z* (%) = 302 (0.2) [$M^+ - CH_3$], 249 (7), 239 (4) [$M^+ - C_6H_6$], 224 (2) [$M^+ - CH_3 - C_6H_6$], 204 (4), 203 (21) [$M^+ - HN_2(CO)_2N$ –

CH_3], 137 (6), 136 (14) [$MePhSiOH^+$], 135 [Me_2PhSi^+], 107 (5), 105 (5).

$C_{16}H_{23}N_3O_2Si$ (317.5) Calcd. C 60.54 H 7.30 N 13.27
Found C 60.51 H 7.36 N 13.19

CAS Registry Numbers

(*E*)-**1a**: 65119-04-2 / (*Z*)-**1a**: 116487-96-8 / **1b**: 77113-46-3 / **1c**: 137041-32-8 / **1d**: 17874-17-8 / **1e**: 35802-49-4 / **2**: 17414-02-7 / **3a**: 137041-33-9 / **3b**: 137041-34-0 / **3c**: 137041-35-1 / **3d**: 137041-36-2 / **3e**: 137041-37-3 / DBB: 1625-91-8 / MTAD: 13274-43-6 / $ClSiMe_2Ph$: 768-33-2

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