Synthesis and structure of 1-[ω-(3,3-dialkyldiaziridin-1-yl)alkyl]-3,3-dialkyldiaziridines

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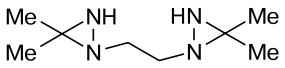
A method for the synthesis of 1-[ω-(3,3-dialkyldiaziridin-1-yl)alkyl]-3,3-dialkyldiaziridines based on the reaction of ketoxime *O*-arenesulfonates with alkylenediamines was developed. Diastrereomers (the racemic and *meso* forms) of 1-[2-(3,3-dimethyldiaziridin-1-yl)ethyl]-3,3-dimethyldiaziridine were isolated. The structure of the *meso* form was confirmed by X-ray diffraction analysis.

Key words: diaziridines, ketoxime *O*-arenesulfonates, 1-[ω-(3,3-dialkyldiaziridin-1-yl)alkyl]-3,3-dialkyldiaziridines, diastereomers, racemic form, *meso* form, epimerization, X-ray diffraction analysis.

Some diaziridines are known to possess neurotropic activity, which increases upon introduction of the second diaziridine ring into the molecule.[1—3] Recently, it has been reported[4,5] that 1-[2-(3,3-dimethyldiaziridin-1-yl)ethyl]-3,3-dimethyldiaziridine (**1a**) exhibits high antidepressant activity; however, neither method of its synthesis nor its characteristics were presented in the cited publications.

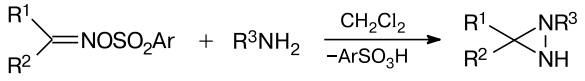


1a

Therefore, we developed a preparative route to compound **1a** and its analogs similar to the method for the synthesis of 1,3,3-trialkyldiaziridines **2** based on the reaction of ketoxime *O*-arenesulfonates **3** with primary aliphatic amines in aprotic organic solvents.[6] This reaction was carried out at 20 °C for ~150 h using excess amine for binding the arenesulfonic acid evolved during the reaction. The yields of 1,3,3-trialkyldiaziridines **2** were 50—80% (Scheme 1). The low reaction rate is apparently caused by the relatively low electrophilicity of the oxime carbon atom, while the use of higher temperature is impossible as the starting oxime esters are prone to the Beckmann rearrangement.

In the first stage of the study, 1-(2-aminoethyl)-3,3-dimethyldiaziridine (**4**) described in our previous work,[7] which has already contained one 3,3-dimethyldiaziridine fragment, was used as the amine for the preparation of compound **1a**. However, amines that did not react with the initial oxime esters **3**, namely, Et_2NH and Et_3N, rather than an excess of amine **4** were used as the bases for binding arenesulfonic acids. Amine **4** reacted with benzene- and *p*-toluenesulfonates of acetone oxime **3a**,**b** in CH_2Cl_2 for 150 h at 20 °C in the presence of Et_2NH or Et_3N to give the desired 1-[2-(3,3-dimethyldiaziridin-1-yl)ethyl]-3,3-dimethyldiaziridine (**1a**) (Scheme 2). How-

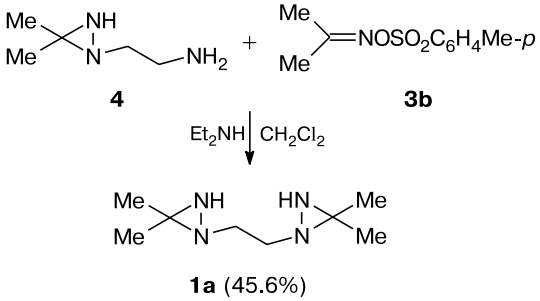
Scheme 1



3 **2**

$R^1 = R^2 = R^3 = Alk$

Scheme 2



4 **3b**

1a (45.6%)

* Dedicated to Academician V. A. Tartakovsky on his 75th birthday.

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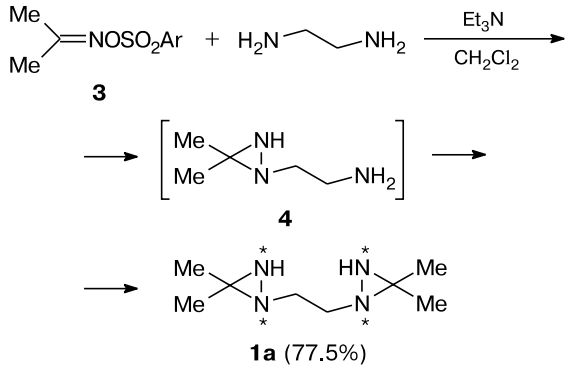
ever, the reaction did not proceed to completion under these conditions (TLC monitoring) and, hence, the reaction time was increased to 170 h. The lower rate of synthesis of bisdiaziridine **1a** according to Scheme 2 compared to the synthesis of 1,3,3-trialkyldiaziridines **2** (see Scheme 1) is probably due to the decrease in the nucleophilicity of the amino group in amine **4** caused by the electron-withdrawing effect of the diaziridine ring.

To develop a more efficient synthetic route to compound **1a**, we studied the reaction of acetone oxime *p*-toluenesulfonate **3b** with ethylenediamine (in 2 : 1 molar ratio). The reaction was carried out in CH_2Cl_2 with triethylamine as the base. The course of the reaction was monitored by TLC (disappearance of **3b**). During the study, amine **4** was isolated by preparative TLC and identified by comparison with a previously described sample.[7] It was found that at 20—22 °C, the reaction is even slower than the reaction according to Scheme 2 because the concentration of the intermediate 1-(2-aminoethyl)-3,3-dimethyldiaziridine **4** in the reaction mixture is relatively low at each particular time point. Therefore, for acceleration, the reaction was carried out at room temperature only during the first 24 h when the concentration of oxime ester **3b** in the reaction mixture was high. Then the temperature was raised to 25—30 °C. Thus, stirring at 20—22 °C for the first 24 h and at 25—30 °C for the subsequent 100—110 h are the conditions of choice for this reaction (Scheme 3).

Scheme 3



Ar = Ph (**a**), *p*-MeC_6H_4 (**b**)

After workup of the reaction mixture (see Experimental), compound **1a** was isolated in 77.5% yield. Similarly, compound **1a** was obtained from acetone oxime benzenesulfonate **3a**. According to ^{1}H NMR data, the product was a mixture of two diastereomers: the racemic form (1*S**, 2*S**, 1′*S**, 2′*S**) and the *meso* form (1*S**, 2*S**, 1′*R**, 2′*R**) in 2 : 3 ratio. The higher-melting diastereomer (m.p. 116—118 °C) was obtained by crystallization from acetone without stirring, while the lower-melting diastereomer (m.p. 72—74 °C) was isolated by crystallization from pentane of the remainder after the isolation of the higher-melting diastereomer concentrated to dryness.

The ^{1}H NMR spectra of both diastereomers (simulated and experimental ones) are shown in Figs 1 and 2. Note that the lower-melting diastereomer is easily converted in solution into the higher-melting one; therefore, the spectrum of the former always contains signals of the higher-melting compound. In order to determine the chemical shifts and the spin—spin coupling constants of the methylene protons of the NCH_2CH_2N group (AA′BB′ system), iteration calculations for the spectra of both diastereomers were carried out by the PANIC program (determination accuracy: spin—spin coupling constants 0.03 Hz, chemical shifts 0.01 ppm).

The assignment of the diastereomers to either racemic or *meso* form was based on the X-ray diffraction analysis of the high-melting isomer, which showed that this was the *meso* diastereomer (1*S**, 2*S**, 1′*R**, 2′*R**) (Fig. 3).

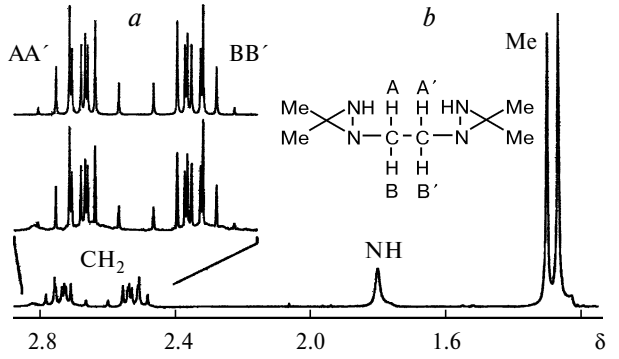


Fig. 1. ^{1}H NMR spectrum of higher-melting diastereomer: (*a*) simulation: δ(A) = δ(A′) = 2.73, δ(B) = δ(B′) = 2.52, ^3J(AA′) = ^3J(BB′) = 7.80 Hz, ^2J(AB) = ^2J(A′B′) = −12.30 Hz, ^3J(AB′) = ^3J(A′B) = 6.14 Hz; (*b*) experiment.

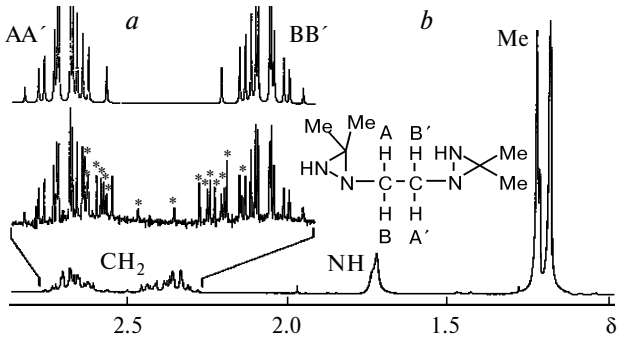


Fig. 2. ^{1}H NMR spectrum of lower-melting diastereomer: (the asterisk marks the signals from the higher-melting diastereomer): (*a*) simulation: δ(A) = δ(A′) = 2.70, δ(B) = δ(B′) = 2.36, ^3J(AA′) = 7.70 Hz, ^3J(A′B) = ^3J(AB′) = 6.47 Hz, ^2J(AB) = ^2J(A′B′) = −12.46 Hz, ^3J(BB′) = 5.20 Hz; (*b*) experiment.

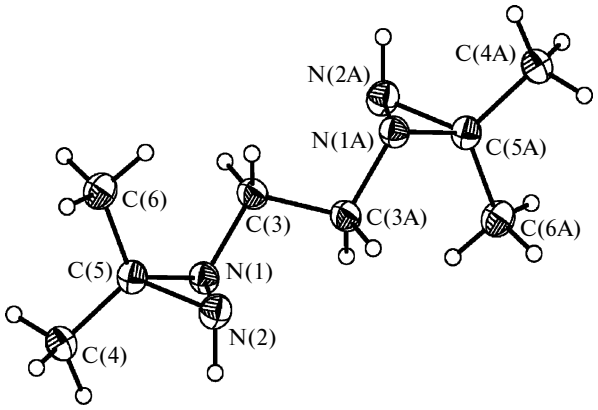
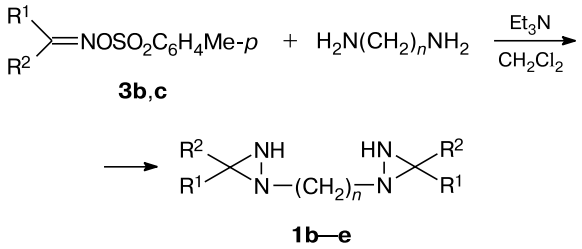


Fig. 3. General view of the molecule of *meso* form of compound **1a** with atoms represented by thermal ellipsoids ($p = 50\%$).

Scheme 4



$R^1 = R^2$ = Me (**1c**—**e**, **3b**); $R^1—R^2$ = —$(CH_2)_5$— (**1b**, **3c**)
n = 2 (**1b**), 3 (**1c**), 4 (**1d**), 5 (**1e**)

In the crystal, the molecule occupies a special position, namely, a symmetry center passing through the midpoint of the C(3)—C(3A) bond. The *N*-substituents are located in the *trans*-positions and the N(1)C(3)C(3A)N(3A) fragment has an antiperiplanar conformation. The bond lengths in molecule **1a** are close to the expected values. In particular, the N(1)—N(2) bond length (1.517(1) Å) is only slightly longer than these bonds in dimethyl ester and diamide of 1-methyldiaziridine-3,3-dicarboxylic acid (1.504 Å).[8] This bond elongation can be attributed to the difference between the inductive effects of substituents at C(5).

Analysis of the intermolecular contacts in **1a** has shown that the molecules in the crystal are combined by weak hydrogen bonds (N(2)—H(2)...N(1) [$-x+2, -y, -z+1$], (N(2)...N(1) 3.393(2) Å, H(2N)...N(1) 2.52 Å, N(2)H(2N)N(1) 159°) to form double chains arranged along the crystallographic axis *a*.

The epimerization conditions of the obtained diastereomers were found using ^{1}H NMR spectroscopy. The process was monitored by measuring the variation of integral intensities of the methyl group signals, whose chemical shifts differ by 0.012 ppm for the two diastereomers. Each diastereomer was refluxed in a number of solvents ($CHCl_3$, CCl_4). Every 15 min samples was taken and, after evaporation of the solvent, analyzed by ^{1}H NMR. It was found that epimerization proceeds to an equilibrium, which is attained after refluxing in chloroform (5 h) or after refluxing in CCl_4 (3 h), and the *meso* to racemic form ratio in equilibrium is 3 : 2.

The method developed for the synthesis of 1-[2-(3,3-dimethyldiaziridin-1-yl)ethyl]-3,3-dimethyldiaziridine (**1a**) was extended to the preparation of other 1-[ω-(3,3-dialkyldiaziridin-1-yl)alkyl]-3,3-dialkyldiaziridines **1**. A number of diamines: ethylenediamine, 1,3-diaminopropane, 1,4-diaminobutane, and 1,5-diaminopentane, were made to react with ketoxime *O*-*p*-toluenesulfonates **3b**,**c** under conditions found for bisdiaziridine **1a**. In all cases, the corresponding 1-[ω-(3,3-dialkyldiaziridin-1-yl)alkyl]-3,3-dialkyldiaziridines **1b**—**e** were obtained in 47—61% yields (Scheme 4, Table 1). According to ^{1}H NMR spectra, freshly isolated compounds **1b** and **1c** were also diastereomer mixtures; how-

Table 1. Yields and some physicochemical characteristics of the synthesized compounds

Compound	Yield (%)	M.p./°C [B.p./°C] (p/Torr)	R_f*	Found / Calculated (%)			Molecular formula
				C	H	N	
1a	77.5**	116—118 (*meso* form)	0.59	56.61 / 56.44	10.73 / 10.66	32.58 / 32.91	$C_8H_{18}N_4$
		72—74 (racemic form)	0.47				
1b	34.2	135—137	0.67	68.21 / 68.14	10.61 / 10.67	21.15 / 21.19	$C_{15}H_{28}N_4$
1c	69.8	Oil	0.55	58.71 / 58.66	10.70 / 10.94	30.56 / 30.40	$C_9H_{20}N_4$
1d	61.0	82—83	0.54	60.73 / 60.57	11.08 / 11.18	28.19 / 28.25	$C_{10}H_{22}N_4$
1e	47.3	[108—112] (1.5)	0.63	62.35 / 62.22	11.20 / 11.39	26.45 / 26.39	$C_{11}H_{24}N_4$

* Eluent: MeOH : 25% NH_3 = 10 : 1, visualization with I_2 vapor.
** Yield of diastereomer mixture from which the *meso* (yield 46.3%) and racemic (yield 27.2%) forms were isolated by crystallization.

Table 2. IR and ^{1}H and ^{13}C NMR data for the synthesized compounds

Compound	IR, ν/cm^{-1}	δ, *J*/Hz ($CDCl_3$)	
		^{1}H NMR	^{13}C NMR
1a	656, 760, 808, 828, 976, 1004, 1072, 1104, 1128, 1212, 1276, 1300, 1320, 1352, 1384, 1448, 1464, 2864, 2932, 2964, 2996, 3008, 3212	*Meso* form*: 1.25, 1.28 (both s, 6 H each, Me); 1.79 (br.s, 2 H, NH); 2.52, 2.73 (both m, ΔD = 54.7 Hz)	*Meso* form: 17.35 (Me); 28.16 (Me); 53.21 (NCH_2); 56.90 (C_{cycl})
		Racemic form**: 1.17, 1.25 (both s, 6 H each, Me); 1.71 (br.s, 2 H, NH); 2.36, 2.70 (both m, Δν = 78.0 Hz)	Racemic form: 17.42 (Me); 28.34 (Me); 53.11 (NCH_2); 57.17 (C_{cycl})
1b***	656, 828, 872, 1040, 1132, 1208, 1248, 1280, 1292, 1308, 1336, 1408, 1436, 1460, 2852, 2920, 2936, 2988, 3188	1.25—1.75 (m, 20 H, $(CH_2)_5$); 1.85 (br.s, 2 H, NH); 2.65, 2.95 (both m, 2 H each, NCH_a, NCH_b, $NCH_{a'}$, $NCH_{b'}$, ^2J = −10.9, ^3J = 6.8, Δν = 81.9 Hz)	24.83, 25.03, 25.48 ($CH_2\underline{C}H_2\underline{C}H_2\underline{C}H_2CH_2$); 28.11, 38.85 ($\underline{C}H_2CH_2CH_2CH_2\underline{C}H_2$); 52.42 ($NCH_2$); 61.45 ($C_{cycl}$)
1c***	724, 764, 820, 972, 1104, 1132, 1196, 1264, 1308, 1352, 1388, 1460, 1668, 1712, 2876, 2932, 2956, 3004, 3216, 3388	1.34, 1.38 (both s, 6 H each, Me); 1.82 (br.s, 2 H, NH); 1.86 (m, 2 H, $CH_2C\underline{H}_2CH_2$, ^3J = 4.7); 2.48 (m, 2 H, NCH_a, NCH_b, ^2J = −11.5, ^3J = 4.7, Δν = 48.3 Hz); 2.69 (m, 2 H, $NCH_{a'}$, $NCH_{b'}$, ^2J = −13.8, ^3J = 4.7, Δν = 48.3 Hz)	17.03 (Me); 28.07 (Me); 29.00 ($CH_2\underline{C}H_2CH_2$); 51.39 (NCH_2); 56.83 (C_{cycl})
1d***	616, 676, 744, 768, 832, 936, 980, 1000, 1020, 1084, 1108, 1136, 1188, 1240, 1280, 1320, 1356, 1384, 1460, 1480, 2872, 2912, 2940, 2996, 3104, 3192	1.16, 1.23 (both s, 6 H each. Me); 1.50 (br.s, 4 H, $CH_2C\underline{H}_2C\underline{H}_2CH_2$); 1.70 (br.s, 2 H, NH); 2.25, 2.42 (both m, 2 H each, NCH_2, Δν = 36.9 Hz)	16.94 (Me); 27.04 ($CH_2\underline{C}H_2\underline{C}H_2CH_2$); 28.11 (Me); 53.36 ($NCH_2$); 56.57 ($C_{cycl}$)
1e***	820, 972, 1004, 1132, 1180, 1312, 1352, 1388, 1460, 1540, 1660, 2864, 2936, 3004, 3228	1.25, 1.32 (both s, 6 H each, Me); 1.38 (m, 2 H, $CH_2CH_2C\underline{H}_2CH_2CH_2$, ^3J = 7.8); 1.56 (m, 4 H, $CH_2C\underline{H}_2CH_2C\underline{H}_2CH_2$, ^3J = 7.8); 1,80 (br. s, 2 H, NH); 2.30, 2.52 (both m, 2 H each, NCH_2, ^2J = −13.7, ^3J = 8.6, Δν = 58.6 Hz)	17.00 (Me); 25.14 ($CH_2CH_2\underline{C}H_2CH_2CH_2$); 28.01 (Me); 29.12 ($CH_2\underline{C}H_2CH_2\underline{C}H_2CH_2$); 53.32 ($NCH_2$); 56.78 ($C_{cycl}$)

* The full spectrum is shown in Fig. 1.
** The full spectrum is shown in Fig. 2.
*** One diastereomer.

ever, only one diastereomer was obtained after purification in both cases (Table 2). The second diastereomers were not isolated from the mother liquors. No diastereomers were detected by NMR in either crude or recrystallized or distilled compounds **1d**,**e**, which is apparently due to the increase in the length of the alkyl chain between the diaziridine rings. The yields and spectral characteristics of the resulting compounds **1a**—**e** are summarized in Tables 1 and 2.

Thus, we developed a general and facile method for the synthesis of 1-[ω-(3,3-dialkyldiaziridin-1-yl)alkyl]-3,3-dialkyldiaziridines **1a**—**e** based on the reaction of ketoxime arenesulfonates with alkylenediamines in 2 : 1 molar ratio in the presence of Et_3N in aprotic organic solvents. According to ^{1}H NMR data, bisdiaziridines **1a**—**c** are formed as mixtures of racemic and *meso* diastereomers. For compound **1a**, both diastereomers were isolated and the structure of the *meso* form was determined by X-ray diffraction analysis.

Experimental

IR spectra were recorded on a UR-20 spectrometer in KBr pellets (for crystalline compounds) and in thin films (for oils); NMR spectra were recorded on Bruker WM-250 (250 MHz (^{1}H)), Bruker AM-300 (300 (^{1}H) and 75.5 MHz (^{13}C)), and Bruker AC-200 spectrometers (200 MHz (^{1}H)); the chemical shifts are referred to Me_4Si. The reaction mixtures were monitored and the product purity was checked by TLC on Silufol UV_{254} plates, which were visualized under UV light or in I_2 vapor. The melting points were determined on a GALLENKAMP Sanyo device. The X-ray diffraction study of the *meso* diastereomer of compound **1a** ($C_8H_{18}N_4$) was carried out at 120 K on a Smart 1000 CCD three-circle automated diffractometer (MoKα, graphite monochromator, ω-scanning, 2θ < 58°). At 120 K, the crystals are monoclinic: a = 6.3714(8), b = 5.8403(8), c = 13.104(2) Å, β = 100.500(5)°, V = 479.45(11) Å^3, space group $P2_1/n$, Z = 2 (Z' = 0.5), M = 170.26, d_{calc} = 1.179 g cm^{-3}, μ = 0.76 cm^{-1}, F(000) = 188. Out of the total of 4999 measured reflections (R_{int} = 0.0272), 1245 independent reflections were used in the subsequent calcu-

lations. The structure of **1a** was solved by the direct method and refined by the least-squares method in the full-matrix anisotropic approximation. The hydrogen atoms were located from difference Fourier electron density syntheses and refined in the isotropic approximation. The final *R*-factors were $R = 0.0476$ for 5805 reflections with $I > 2\sigma(1)$, $wR_2 = 0.0928$, GOOF = 0.928 for all measured reflections. All calculations were carried out using the SHELXTL PLUS 5 software.

Preparation of 3,3-dimethyl-1-[2-(3,3-dimethyldiaziridin-1-yl)ethyl]diaziridine (1a) from 1-(2-aminoethyl)-3,3-dimethyldiaziridine (4). 1-(2-Aminoethyl)-3,3-dimethyldiaziridine (**4**) (3.5 g, 0.03 mol) prepared by a reported procedure[7] and Et_2NH (2.2 g, 0.03 mol) were added to a solution of acetone oxime *p*-toluenesulfonate **3b** (6.8 g, 0.03 mol), prepared by a known procedure,[9] in CH_2Cl_2 (10 mL). The reaction mixture was stirred for 170 h at 18—20 °C (TLC monitoring). The precipitated salt was filtered off, the solvent was evaporated on a rotary evaporator under vacuum of a water jet pump at a bath temperature of ≤40 °C. Then CH_2Cl_2 (30 mL) and finely ground potassium carbonate (5 g) were added, and the mixture was stirred for 5 h. The precipitate was filtered off and washed with CH_2Cl_2 (30 mL) and the solvent was evaporated on a rotary evaporator at a bath temperature of ≤40 °C to give 2.4 g (45.6%) of compound **1a** as a diastereomer mixture.

Preparation of 3,3-dimethyl-1-[2-(3,3-dimethyldiaziridin-1-yl)ethyl]diaziridine (1a) from acetone oxime esters 3a,b. Ethylenediamine (6.0 g, 0.1 mol) and Et_3N (30.0 g, 0.3 mol) were added at a temperature of 10—15 °C to a solution of acetone oxime benzenesulfonate **3a** (42.6 g, 0.2 mol) or *p*-toluenesulfonate **3b** (45.4 g, 0.2 mol), obtained by known procedures,[9,10] in CH_2Cl_2 (40 mL). The reaction mixture was stirred for 24 h at 20—22 °C and for 100—110 h at 25—30 °C until the initial oxime ester was completely consumed (TLC monitoring: R_f (**3b**) = 0.56, R_f (**3a**) = 0.5, methanol as the eluent, UV visualization). Then CH_2Cl_2 (80 mL), and finely ground potassium carbonate (30 g) were added, and the mixture was stirred for 5 h. The precipitate was filtered off and washed with CH_2Cl_2 (50 mL). Solvent evaporation on a rotary evaporator at a bath temperature of ≤40 °C gave a crystallizing residue, which completely solidified after 1.5—2 h at 20—22 °C. After crystallization from acetone and fast cooling with stirring, the precipitated crystals were collected on a filter to give 15.4 g (77.5%) of compound **1a** as a diastereomer mixture. Crystallization from acetone with slow cooling without stirring gave 8.0 g (39.3%) of a diastereomer with m.p. 116—118 °C. The acetone mother liquor was evaporated on a rotary evaporator at a bath temperature of ≤40 °C and crystallized from pentane. The precipitate representing pure higher-melting diastereomer (1.2 g, 5.9%) was collected on a filter; the overall yield was 9.2 g (59.7% of the total yield of compound **1a**). The pentane solution was evaporated on a rotary evaporator without heating until crystallization started. The mixture was cooled to 0 °C and the precipitated crystals were collected on a filter and washed with cold pentane to give 5.4 g (35.1% of the total yield of compound **1a**) of the diastereomer with m.p. 72—74 °C. The physicochemical and spectral characteristics of both diastereomers are presented in Tables 1 and 2.

3,3-Dialkyl-1-[ω-(3,3-dialkyldiaziridin-1-yl)alkyl]diaziridines 1 (general procedure). The corresponding alkylenebisamine (0.1 mol) and Et_3N (30.0 g, 0.3 mol) were added at 10—15 °C to a solution of ketoxime *p*-toluenesulfonate **3b** or **3c** (0.2 mol) prepared by known methods[9,10] in CH_2Cl_2 (40 mL). The reaction mixture was stirred for 24 h at a temperature of 20—22 °C and for 90—120 h at 25—30 °C until the initial oxime ester was completely consumed (TLC monitoring). Then the mixture was worked-up as described above. The solvent was evaporated on a rotary evaporator at a bath temperature of ≤40 °C.

1-[2-(1,2-Diazaspiro[2.5]oct-1-yl)ethyl]-1,2-diazaspiro[2.5]octane (1b). Evaporation of CH_2Cl_2 on a rotary evaporator gave compound **1b** as a crystallizing oil (according to ^1H NMR, as a mixture of two diastereomers), which solidified after 1.5—2 h at 20—22 °C. Crystallization from acetone gave 8.2 g (34.2%) of compound **1b** containing only one diastereomer.

3,3-Dimethyl-1-[3-(3,3-dimethyldiaziridin-1-yl)propyl]diaziridine (1c). Evaporation of CH_2Cl_2 on a rotary evaporator gave compound **1c** as an undistillable liquid. Dissolution in CH_2Cl_2 (15 mL) and precipitation with pentane gave compound **1c** (12.8 g, 69.8%) as a liquid containing one diastereomer according to ^1H NMR.

3,3-Dimethyl-1-[4-(3,3-dimethyldiaziridin-1-yl)butyl]diaziridine (1d). Crystallization from acetone gave compound **1d** (12.1 g, 61.0%) as a single diastereomer.

3,3-Dimethyl-1-[5-(3,3-dimethyldiaziridin-1-yl)pentyl]diaziridine (1e). Vacuum distillation (oil vacuum pump) gave compound **1e** (10.0 g, 47.3%) as a single diastereomer.

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