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REACTIONS OF 4,5-DIHYDRO-5-METHYL-3H-SPIRO[BENZ-2-AZEPINE-3-CYCLOHEXANE] N-OXIDE WITH SOME NUCLEOPHILIC REAGENTS

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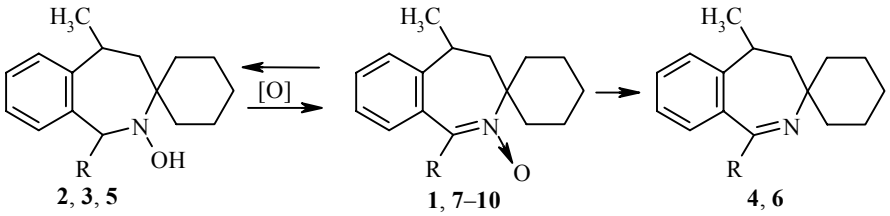
4,5-Dihydro-5-methyl-3H-spiro[benz-2-azepine-3-cyclohexane] N-oxide reacted with cyanide ion and isopropyl magnesium bromide to give the corresponding 1-cyano- and 1-isopropyl-4,5-dihydro-5-methyl-3H-spiro[benz-2-azepine-3-cyclohexane], but reaction with phenyl magnesium bromide, benzyl magnesium chloride, and nitromethane gave cyclic hydroxylamines: 1-substituted N-hydroxy-1,2,4,5-tetrahydro-5-methyl-3H-spiro[benz-2-azepine-3-cyclohexanes] which were oxidized to the corresponding nitrones.

Keywords: benz-2-azepines, cyclic hydroxylamines, cyclic nitrones, nucleophilic additions, oxidation.

The [3+2] dipolar cycloadditions have been well studied for a series of nitrones in connection with the wide synthetic possibilities of the isoxazolidines and isoxazolines formed in this way [2-5]. The reactions of these nitrones (especially cyclic compounds) with nucleophilic reagents have been studied considerably less [6,7]. The α-substituted hydroxylamines formed in this way can serve as starting materials for the synthesis of a series of derivatives of acids and heterocyclic compounds [8-10].

In the present work, the reactions of 4,5-dihydro-5-methyl-3H-spiro[benz-2-azepine-3-cyclohexane] N-oxide (**1**) [11] with organo-magnesium compounds, nitromethane, potassium cyanide, and acetone cyanohydrin have been studied for the first time.

The corresponding 1-substituted hydroxylamines of the spiro[benz-2-azepinecyclohexane] series **2** and **3** have been obtained in 42-62% yields from the reaction of the nitrone **1** with phenyl magnesium bromide and benzyl magnesium chloride:



1 R = H; **2**, **7** R = Ph; **3**, **10** R = CH_2Ph; **4**, **9** R = *i*-Pr; **5** R = CH_2NO_2; **6** R = CN; **8** R = PhCO

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Hydroxylamines **2** and **3** are crystalline compounds which are readily recrystallized from hexane without marked decomposition. 1-Isopropyl-4,5-dihydro-5-methyl-3H-spiro[benz-2-azepine-3-cyclohexane] (**4**) was produced in 81% yield in place of the expected hydroxylamine when nitrone **1** reacted with isopropyl magnesium bromide. The unpurified reaction mixture contained about 10% of N-hydroxy-1-isopropyl-1,2,4,5-tetrahydro-5-methyl-3H-spiro[benz-2-azepine-3-cyclohexane] according to the ^{1}H NMR spectrum.

The reaction of nitrone **1** with nitromethane in the presence of base [8] gave the nitromethyl-substituted hydroxylamine **5**. Reaction of nitrone **1** with potassium cyanide in acid medium [12] occurred with vigorous resinification of the reaction mixture from which no addition products were isolated. The reaction occurred more readily in weakly basic media at 20°C and was accompanied by dehydration of the initially formed addition product [6]. 1-Cyano-4,5-dihydro-5-methyl-3H-spiro[benz-2-azepine-3-cyclohexane] (**6**) was obtained in 29% yield. When acetone cyanhydrin was used as the source of cyanide ion in the presence of potassium hydroxide and DMSO the yield of compound (**6**) increased to 84%.

Taking into account the synthetic possibilities of nitrones, we carried out oxidation of the synthesized hydroxylamines **2** and **3** and the imine **4** into the N-oxides of 1-substituted 4,5-dihydro-5-methyl-3H-spiro[benz-2-azepine-3-cyclohexanes] **7-10**. The oxidation was carried out with 30% hydrogen peroxide in boiling acetone in the presence of sodium tungstate. Under these conditions the benzyl group of compound **4** was oxidized to benzoyl. When the oxidation was carried out in boiling ethanol the benzyl group was not affected. The corresponding nitrone **9** was obtained in 67% yield.

The stretching frequency of the hydroxyl group appeared in the IR spectra of hydroxylamines **2**, **3**, **5** in the 3226-3542 cm^{-1} region. The IR spectra of the nitrones **7-10** are characterized by the presence of C=N and N→O stretching frequencies in the 1493-1530 and 1227-1247 cm^{-1} regions respectively. In the spectrum of the cyanimine **4** the imine stretching band appears at 1655 cm^{-1}. Stretching bands of the corresponding functional groups appear in the IR spectra of the functionally substituted compounds **5**, **6**, and **8**: 1560 and 1385 for NO_2, 2230 for CN, and 1660 cm^{-1} for CO.

Only one set of signals is observed in the ^{1}H NMR spectra of the hydroxylamines **2**, **3**, and **5** (Table 1) which indicates the absence of geometric isomers with respect to the substituent R on carbon atom $C_{(1)}$. It may be suggested that the bulky substituent occupies an equatorial position in compounds **2**, **3**, and **5**. In the spectrum of the mixture obtained from the reaction of compound **1** with isopropyl magnesium bromide, which contains about 10% of the corresponding hydroxylamine and imine **4**, only one doublet for proton 1-H was observed at 3.98 ppm and a vicinal coupling J = 9.5 Hz. Because of the closeness of the values of the geminal coupling of the protons of 1-R (CH_AH_B) and the vicinal coupling between 1-H and 1-R ($C\underline{H}_AH_B$), it is difficult to assign signals in the range 3.22-5.34 ppm in the spectra of compounds **3** and **5** to individual protons. The signals with two maximal geminal and vicinal coupling constants (J = 10.3-13.4 Hz) are assigned to the 1-R proton ($C\underline{H}_AH_B$). Of the four doublet signals with coupling constants (J = 10.3-10.7 and 3.4-3.9 Hz), the closest values of the chemical shifts, bearing in mind the distance of 1-H from nitro and phenyl substituents, should belong to proton 1-H: 4.68 ($J_{1,A}$ = 10.3, $J_{1,B}$ = 3.9) in compound **3** and 4.79 ($J_{1,A}$ = 10.7, $J_{1,B}$ = 3.4) in hydroxylamine **5**. Because the signals of protons 4-H$_a$ and 4-H$_e$ are masked by the complex multiplets of the cyclohexane fragment, it was not possible to measure their chemical shifts and coupling constants.

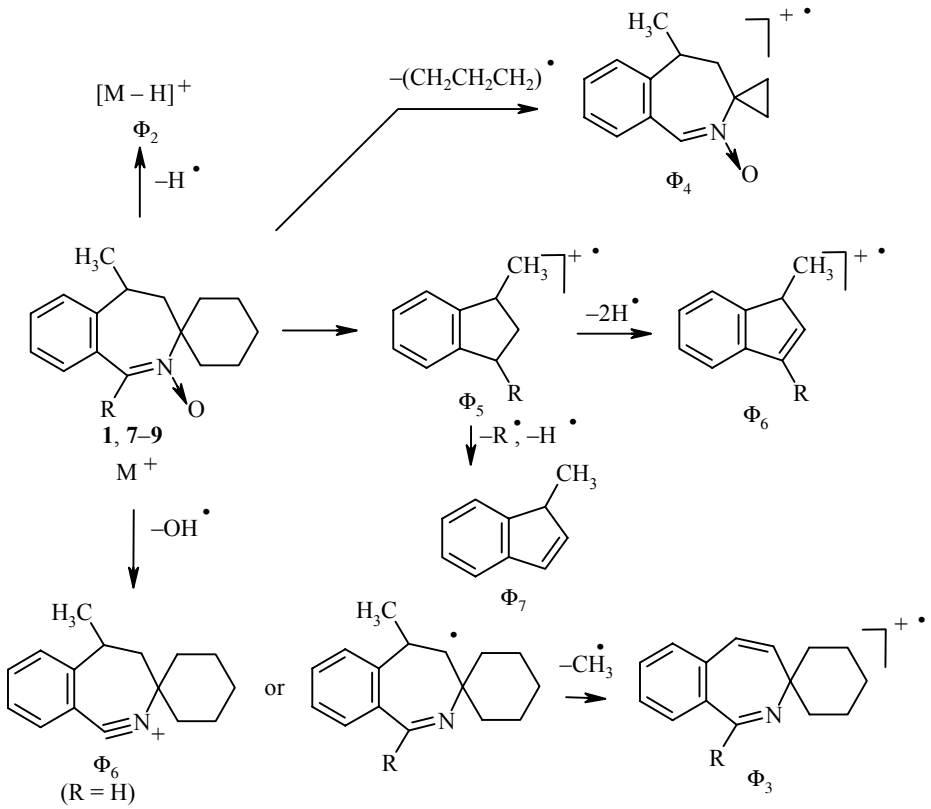
The presence of large axial-axial coupling constants between protons 5-H and 4-H$_a$ (J = 11.0-14.0 Hz) in the spectra of imine **6** and the nitrones **8-10** indicates that proton 5-H has an axial orientation and consequently that the 5-CH_3 methyl group has equatorial disposition which has been confirmed by X-ray crystallography and ^{1}H NMR spectroscopy on the example of 8-nitro-1,2,4,5-tetrahydro-1,5-dimethyl-3H-spiro[benz-2-azepine-3-cyclohexane].*

* To be published in a separate paper.

TABLE 1. ^{1}H NMR Spectra of Compounds **2-10** in $CDCl_3$ Solutions

Com-pound	Chemical shifts, δ, ppm (multiplicity), coupling constants , *J*, Hz								
	1-H	2-OH	4-H$_a$	4-H$_e$	5-H	5-CH$_3$	H-cyclohex.	6-H-9-H	1-R
2	5.68 (s)	4.12 (br. s)	1.3-2.0		3.59 (m)	1.24 (d) ^3J = 7.3	1.2-2.0 (m)	6.6-7.8	6.6-7.8
3	4.68 (dd) $^3J_{1,A}$ = 10.3 $^3J_{1,B}$ = 3.9	3.56 (br. s)	1.2-1.9		3.24 (m)	1.37 (d) ^3J = 7.0	0.8-2.0 (m)	7.1-7.6	3.42 (A) (dd) ^2J = 13.4 $^3J_{1,A}$ = 10.3; 3.22 (B) (dd), ^2J = 13.4 $^3J_{1,B}$ = 3.9; 7.1-7.6 (Ph) (m)
4	—	—	1.80 (dd) ^2J = 13.7 $^3J_{4a,5}$ = 13.1	2.36 (dd) ^2J = 13.7 $^3J_{4e,5}$ = 5.8	3.10 (m) $^3J_{4a,5}$= 13.1, $^3J_{4e,5}$ = 5.8 $^3J_{5,Me}$ = 7.0	1.29 (d) ^3J = 7.0	1.1-2.0 (m)	7.1-7.45	1.08 (CH_3) (d) ^3J = 7.0; 1.40 (CH_3) (d) ^3J = 7.0; 3.77 (CH) (m) ^3J = 7.0
5	4.79 (dd) $^3J_{1,A}$ = 10.7, $^3J_{1,B}$= 3.4	3.99 (br. s)	1.2-1.9		3.26 (m)	1.40 (d) ^3J = 7.0	1.2-1.9 (m)	7.1-7.4 (6H...8H) (m) 6.92 (9-H) (d)	5.21 (A) (t) ^2J = $^3J_{1,A}$ = 10.7; 5.34 (B) (dd), ^2J = 10.7 $^3J_{1,B}$ = 3.4
6	—	—	1.85 (dd) ^2J = 14.3 $^3J_{4a,5}$ = 11.0	2.18 (dd) ^2J = 14.3 $^3J_{4e,5}$ = 4.0	2.98 (m) $^3J_{4a,5}$ = 11.0 $^3J_{4e,5}$ = 4.0 $^3J_{5,Me}$ = 7.0	1.33 (d) ^3J = 7.0	1.1-2.0 (m)	7.29 (6-H) (dd) ^3J = 8.0 ^4J = 2.0; 7.35-7.42 (7-H, 8-H) (td), ^3J = 8.0, ^4J = 2.0; 7.74 (9-H) (dd) ^3J = 8.0 ^4J = 2.0	—
7	—	—	1.98 (t) ^2J = $^3J_{4a,5}$ = 13.4	2.48 (dd) ^2J = 13.4 $^3J_{4e,5}$ = 6.0	3.39 (m) $^3J_{4a,5}$ = 13.4, $^3J_{4e,5}$ = 6.0 $^3J_{5,Me}$ = 6.7	1.33 (d) ^3J = 6.7	1.1-2.5 (m)	7.1-7.4 (6-H, 8-H) (m); 6.83 (9-H) (d)	7.1-7.4 (*m*-, *p*-Ph) (m); 7.59 (*o*-Ph) (d)
8	—	—	2.14 (dd) ^2J = 14.0 $^3J_{4a,5}$ = 11.7	2.51 (dd) ^2J = 14.0 $^3J_{4e,5}$ = 4.9	3.45 (m) $^3J_{4a,5}$ = 11.7, $^3J_{4e,5}$ = 4.9 $^3J_{5,Me}$ = 7.0	1.45 (d) ^3J = 7.0	1.2-2.4 (m)	7.0-7.7 (m)	7.0-7.7 (*m*-, *p*-Ph) (m); 7.97 (*o*-Ph) (m)
9	—	—	1.80 (t) ^2J = $^3J_{4a,5}$ = 13.7	2.36 (dd) ^2J = 13.7 $^3J_{4e,5}$ = 5.8	3.10 (m) $^3J_{4a,5}$ = 13.7, $^3J_{4e,5}$ = 5.8 $^3J_{5,Me}$ = 6.7	1.29 (d) ^3J = 6.7	0.8-2.5 (m)	7.1-7.4 (m)	1.08 (CH_3) (d) ^3J = 7.0; 1.40 (CH_3) (d) ^3J = 7.0; 3.78 (CH) (m) ^3J = 7.0
10	—	—	1.94 (t) ^2J = $^3J_{4a,5}$ = 13.7	2.34 (dd) ^2J = 14.0 $^3J_{4e,5}$ = 5.5	2.95 (m) $^3J_{4a,5}$ = 14.0, $^3J_{4e,5}$ = 5.5, $^3J_{5,Me}$ = 6.7	1.21 (d) ^3J = 6.7	1.0-2.5 (m)	7.1-7.4 (m)	4.32 (A) (d) ^2J = 14.0; 4.17 (B) (d) ^2J = 14.0

Molecular ions corresponding to the molecular formula were observed in the mass spectra of spiro[benz-2-azepine-3-cyclohexanes]. The decomposition of the molecular ions of the nitrones **1**, **8**, and **9** occur by three basic routes: hydroxyl elimination, splitting of the cyclohexane ring, and splitting of the benzazepine ring (scheme, Table 2).



The fragmentation of the M^+ ions for compounds **8** and **9** is characterized by the elimination of the benzoyl and isopropyl groups respectively from C_1. The fragment ions with *m/z* 242 have intensities of 45 and 66% respectively. The ion $[M\text{-}OH]^+$ (Φ_1) in the second stage of the decomposition eliminates $CH_3^{\cdot}$ to give fragment Φ_3. Decomposition of the benzazepine fragment of the molecule leads to the formation of the cation

TABLE 2. Intensities of Characteristic Ions in the Mass Spectra of Nitrones **1**, **7-9** and the Cyano-substituted Benz-2-azepine **6**

Compound	Ions (intensities, %)						
	M^+	Φ_1	Φ_2	Φ_3	Φ_4	Φ_5	Φ_6
1	243 (21)	226 (100)	—	211 (9)	201 (7)	132 (100)	130 (50)
7	319 (32)	302 (100)	319 (6)	287 (10)	277 (20)	208 (60)	206 (60)
8	347 (18)	330 (57)	—	315 (5)	305 (18)	236 (10)	234 (9)
9	285 (55)	268 (100)	284 (8)	253 (10)	243 (20)	174 (55)	172 (35)
6	252 (36)	—	251 (18)	—	210 (77)	157 (31)	155 (82)

radical of 3-substituted 1-methylindane Φ_5. Further fragmentation of this ion is characterized by consecutive elimination of either two hydrogen atoms or hydrogen and the substituent at C_3 which leads to the indanes Φ_6 and Φ_7 respectively. In distinction from 1,2,4,5-tetrahydro-5-methyl-3H-spiro[benz-2-azepine-3-cyclohexane] [13] and the N-hydroxy derivatives **2**, **3**, and **5**, where splitting of the cyclohexane ring in M^+ ions is accompanied by the elimination of a methyl, ethyl, or propyl group, a C_3H_6 group is eliminated in the fragmentation of compounds **1**, and **7-9** with the formation of a quite intense fragmentation ion Φ_4. Elimination of the C_3H_6 particle is also characteristic of fragmentation of the M^+ ion of the cyano substituted compound **6**.

Fragmentation of the M^+ of the N-hydroxy substituted benzazepines **2**, **3**, and **5** is characterized by elimination of the hydroxyl and benzyl or nitromethyl radicals from $C_{(1)}$. Fragmentation of the benzazepine part of the molecule in compounds **2**, **3**, and **5** also leads to the formation of the cation radical of 3R-1-methylindane, which dissociates analogously to the process described above for the nitrones.

EXPERIMENTAL

IR spectra of KBr disks were recorded with a UR-20 spectrometer. Mass spectra were recorded with a Varian MAT 112 with direct injection of the samples into the ion source with an ionizing voltage of 70 eV. ^{1}H NMR spectra of about 2% solutions of the compounds synthesized in $CDCl_3$ were recorded with a Bruker WP-200 machine at 30°C. Chemical shifts were measured relative to TMS as internal standard. TLC was carried out on Silufol UV-254 plates with 1:2 ethyl acetate–hexane as eluent, and development with iodine vapor. Silica gel L 100/250 was used for column chromatography with 1:5 ethyl acetate–hexane as eluent. Melting points were determined in glass capillaries and were not corrected.

N-Hydroxy-5-methyl-1-phenyl-1,2,4,5-tetrahydro-3H-spiro[benz-2-azepine-3-cyclohexane] (2). Nitrone **1** (1.0 g, 4 mmol) was added in portions over 10 min to a solution of phenyl magnesium bromide prepared from Mg (0.38 g, 16 mmol) and bromobenzene (1.29 g, 8 mmol) in absolute ether (25 ml). The mixture was boiled for 1 h (monitored by TLC), poured onto ice, and treated with saturated ammonium chloride solution. It was extracted with ether (3 × 30 ml), and dried over magnesium sulfate. The residue (1.13 g) after evaporating the ether was recrystallized from hexane to give hydroxylamine **2** (0.55 g, 42%) as white crystals; mp 105-106°C, R_f 0.60. IR spectrum: 3236 cm^{-1} (OH). Mass spectrum, *m/z* (I_{rel}, %): 321 (17, M^+), 304 (78), 207 (24), 130 (18), 114 (100), 103 (8), 91 (30), 77 (12). Found, %: C 82.0; H 8.1; N 4.7. $C_{23}H_{29}NO$. Calculated, %: C 82.2; H 8.4; N 4.4.

1-Benzyl-N-hydroxy-5-methyl-1,2,4,5-tetrahydro-3H-spiro[benz-2-azepine-3-cyclohexane] (3). Hydroxylamine **3** (3.44 g, 62%) was obtained as white crystals; mp 125-128°C, R_f 0.74, by the above method from the interaction of nitrone **1** (4 g, 16 mmol) with benzyl magnesium chloride, synthesized from Mg (1.52 g, 66 mmol) and benzyl chloride (4.19 g, 33 mmol). IR spectrum: 3542 cm^{-1} (OH). Mass spectrum, *m/z* (I_{rel}, %): 335 (15, M^+), 318 (10), 244 (100), 211 (7), 131 (28), 130 (20), 114 (35), 103 (8), 91 (57), 77 (10). Found, %: C 82.7; H 8.4; N 4.0. $C_{22}H_{27}NO$. Calculated, %: C 82.4; H 8.7; N 4.2.

1-Isopropyl-5-methyl-4,5-dihydro-3H-spiro(benz-2-azepine-3-cyclohexane] (4). Compound **4** (1.01 g, 87%) was obtained as a yellow viscous oil, R_f 0.91, after chromatography on silica gel, by an analogous method from nitrone **1** (1 g, 4 mmol) and isopropyl magnesium bromide, synthesized from Mg (0.37 g, 16 mmol) and isopropyl bromide (0.98 g, 8 mmol). IR spectrum: 1637 (C=N), 1348, and 1387 cm^{-1} (*i*-Pr). Found, %: C 84.4; H 10.2; N 5.0. M^+ 269. $C_{19}H_{27}N$. Calculated, %: C 84.8; H 10.1; N 5.2. M 269.

N-Hydroxy-5-methyl-1-nitromethyl-1,2,4,5-tetrahydro-3H-spiro[benz-2-azepine-3-cyclohexane] (5). Nitrone **1** (0.5 g, 2 mmol) followed by a mixture of DMSO (1.87 g, 24 mmol) and nitromethane (0.61 g, 10 mmol) in ethanol (10 ml) were added to a solution of sodium ethoxide (6 mmol) in ethanol (10 ml). The mixture was kept at 20°C for 24 h and then at 50°C for 4 h. It was poured into water and extracted with chloroform. The residue (0.54 g) was chromatographed on a silica gel column (30 × 2 cm). Hydroxylamine **5** (0.14 g, 23%) and the initial nitrone (0.06 g, 12%) were isolated. Compound **5**, white crystals; mp 108-110°C

(hexane), R_f 0.71. IR spectrum: 3512 (OH), 1560 and 1385 cm^{-1} (NO_2). Mass spectrum, *m/z* (I_{rel}, %): 304 (2, M^+), 286 (7), 244 (12), 190 (34), 130 (100), 115 (30), 103 (15), 91 (27), 77 (40). Found, %: C 67.4; H 8.1; N 9.5. $C_{17}H_{24}N_2O_3$. Calculated, %: C 67.1; H 7.9; N 9.2.

1-Cyano-4,5-dihydro-5-methyl-3H-spiro(benz-2-azepine-3-cyclohexane] (6). A. A solution of potassium cyanide (0.26 g, 4 mmol) in water (2 ml) was added gradually to a solution of nitrone **1** (0.5 g, 2 mmol) in THF (15 ml). The mixture was kept for a day at 20°C. After removal of the THF in vacuum, water was added (50 ml) and the mixture was extracted with ether (4 × 20 ml). The extract was dried over magnesium sulfate, the ether was evaporated, and the residue (0.43 g) was crystallized from hexane to give starting nitrone **1** (0.1 g, 20%). The mother liquor was chromatographed on silica gel to give compound **6** (0.15 g, 30%) as a viscous light yellow oil, R_f 0.81. IR spectrum: 2230 (C=N), 1655 (C=N) cm^{-1}. Mass spectrum, *m/z* (I_{rel}, %): 252 (36, M^+), 251 (18), 237 (30), 223 (23), 212 (14), 210 (27), 209 (21), 199 (22), 198 (51), 197 (36), 196 (14), 195 (20), 184 (19), 158 (14), 157 (31), 156 (100), 155 (82), 154 (33), 144 (36), 142 (36), 140 (62), 130 (30), 129 (83), 128 (71), 127 (41), 115 (79), 103 (29), 77 (41). Found, %: C 81.2; H 7.7; N 10.9. $C_{17}H_{20}N_2$. Calculated, %: C 81.0; H 7.9; N 11.1.

B. A solution of nitrone **1** (0.5 g, 2 mmol), potassium hydroxide (0.22 g, 4 mmol), DMSO (1.25 g, 16 mmol), and acetone cyanohydrin (0.51 g, 6 mmol) in ethanol (20 ml) was heated at 50°C for 1 h, then poured into water (50 ml) and extracted with ether (3 × 20 ml). The residue (0.53 g) after evaporation of the ether was chromatographed on a silica gel column (10 × 1 cm) to give compound **6** (0.42 g, 84%) identical in chromatographic mobility and spectroscopic characteristics with a sample made by method **A**.

5-Methyl-1-phenyl-4,5-dihydro-3H-spiro[benz-2-azepine-3-cyclohexane] N-Oxide (7). A solution of compound **2** (2.45 g, 7.6 mmol), sodium tungstate dihydrate (0.1 g, 0.3 mmol), and 30% hydrogen peroxide (4.5 ml, 45.6 mmol) in acetone (10 ml) was boiled for 8 h (monitored by TLC). After evaporation of the acetone, water (50 ml) was added to the residue and the mixture was extracted with chloroform. The extract was washed with water and dried over magnesium sulfate. After removal of the chloroform, the residue was crystallized from a mixture of hexane and ethyl acetate to give compound **7** (1.14 g, 55%) as white crystals; mp 126-128°C, R_f 0.2. IR spectrum: 1493 (C=N), 1247 cm^{-1} (N→O). Mass spectrum, *m/z* (I_{rel}, %): 319 (32, M^+), 318 (6), 302 (100), 287 (10), 277 (20), 260 (35), 224 (25), 208 (60), 207 (62), 206 (60), 199 (32), 191 (10), 190 (8), 178 (20), 165 (23), 131 (20), 130 (18), 129 (18), 128 (20), 115 (20), 103 (20), 91 (30), 77 (41). Found, %: C 82.6; H 8.0; N 4.5. $C_{22}H_{25}NO$. Calculated, %: C 82.8; H 7.8; N 4.4.

1-Benzoyl-5-methyl-4,5-dihydro-3H-spiro[benz-2-azepine-3-cyclohexane] N-Oxide (8) was obtained analogously to compound **7** from hydroxylamine **3** (0.5 g, 1.5 mmol). Compound **8** (0.41 g, 79%), white crystals; mp 135-137°C, R_f 0.27. IR spectrum: 1666 (C=O), 1530 (C=N), 1247 cm^{-1} (N→O). Mass spectrum, *m/z* (I_{rel}, %): 347 (18, M^+), 330 (57), 315 (5), 305 (18), 288 (5), 276 (4), 242 (43), 236 (10), 235 (12), 234 (9), 226 (35), 225 (25), 224 (30), 131 (20), 130 (40), 129 (22), 116 (13), 115 (13), 105 (100), 95 (88), 78 (50), 77 (50). Found, %: C 79.4; H 7.2; N 4.1. $C_{23}H_{25}NO_2$. Calculated, %: C 79.5; H 7.2; N 4.0.

1-Isopropyl-5-methyl-4,5-dihydro-3H-spiro[benz-2-azepine-3-cyclohexane] N-Oxide (9) was obtained analogously to compound **8** from imine **4** (1.95 g, 6.8 mmol). Yield 1.14 g (59%), a viscous light yellow which crystallized on prolonged standing; mp 68-70°C. IR spectrum: 1505 (C=N), 1230 cm^{-1} (N→O). Mass spectrum, *m/z* (I_{rel}, %): 285 (55, M^+), 284 (8), 268 (100), 253 (10), 252 (12), 243 (20), 242 (65), 226 (32), 199 (32), 190 (40), 174 (55), 173 (30), 172 (35), 161 (25), 160 (20), 158 (40), 157 (38), 146 (30), 144 (30), 143 (30), 131 (40), 130 (78), 129 (40), 128 (40), 115 (38), 103 (28), 91 (30), 77 (32), 43 (50). Found, %: C 79.6; H 9.3; N 5.1. $C_{19}H_{27}NO$. Calculated, %: C 80.0; H 9.5; N 4.9.

1-Benzyl-5-methyl-4,5-dihydro-3H-spiro[benz-2-azepine-3-cyclohexane] N-Oxide (10). A solution of hydroxylamine **3** (1.0 g, 3 mmol), 30% hydrogen peroxide (3 ml, 30 mmol), and sodium tungstate dihydrate (40 mg, 0.12 mmol) in ethanol (15 ml) was boiled for 2 h. The ethanol was evaporated, water (50 ml) was added, and the mixture was extracted with chloroform. The extract was dried over magnesium sulfate, the chloroform was evaporated, and the residue was crystallized from a mixture of hexane and ethyl acetate to give

compound **10** (0.67 g, 67%) as white crystals; mp 143-145°C, R_f 0.25. IR spectrum: 1519 (C=N), 1227 cm^{-1} (N→O). Mass spectrum, *m/z* (I_{rel}, %): 333 (2), 317 (12), 275 (26), 226 (64), 220 (22), 130 (73), 115 (28), 103 (30), 91 (100), 77 (24). Found, %: C 82.6; H 8.3; N 4.5. M^+ 333. $C_{23}H_{27}NO$. Calculated, %: C 82.9; H 8.1; N 4.2. M 333.

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