PAPERS

A Simple and Efficient Synthesis of Lactone *N,N*-Dialkylhydrazones and their Isomeric *N*-(Dialkylamino)-lactams

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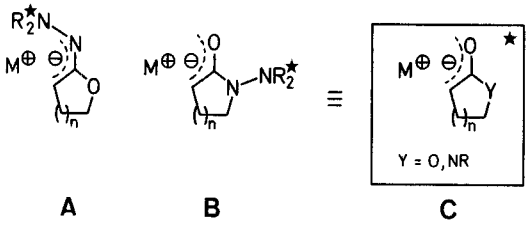
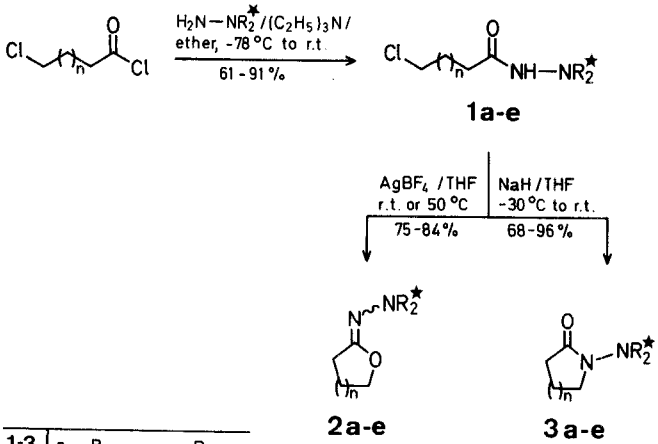
The hitherto unknown lactone *N,N*-dialkylhydrazones **2** and *N*-(dialkylamino)-lactams **3** have been synthesized in good yields by a simple two-step procedure involving silver tetrafluoroborate- or sodium hydride-promoted cyclization of ω-chloroalkanohydrazides **1**, which, in turn, were easily prepared from ω-chloroalkanoyl chlorides and hydrazines.

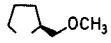
In continuation of our efforts to utilize chiral organometallic reagents in asymmetric synthesis[1], we envisaged the metallated lactone hydrazones **A** and *N*-aminolactams **B** as potential chiral equivalents of lactone (Y = O) and lactam (Y = NR) enolates **C**, as chirality can easily be introduced via the amino function.



A **B** **C**

However, a detailed computer search of the literature did not reveal any straightforward routes leading to lactone *N,N*-dialkylhydrazones **2** or their isomeric *N*-(dialkylamino)-lactams **3** (see Scheme **A**). Apparently, simple members of these classes of compounds are unknown, although methods to prepare the related lactone imines or tosyl- and arylhydrazones, such as the intramolecular cyclization of ω-chloroalkanamides[2,3,4] and aminations of *O*-ethylbutyrolactonium tetrafluoroborate[5] and ortho- or imidolactones[6,7,8], have been reported.



1-3	n	R	R
a	1	CH_3	CH_3
b	1	*i*-C_3H_7	*i*-C_3H_7
c	1	$-(CH_2)_4-$	
d	2	CH_3	CH_3
e	2		

Scheme **A**

In this paper, we first describe a general and convenient two-step synthesis of the title compounds in good overall yield, based on readily available starting materials. As depicted in the Scheme **A**, both the lactone hydrazones **2** and the *N*-aminolactams **3**, are obtained via the same ω-chloroalkanohydrazide intermediates **1**, simply by changing the cyclization conditions.

Our first attempts to prepare the lactone hydrazones **2** by treatment of ortholactones and imidolactones with *N,N*-dialkylhydrazines, a technique successfully applied in the case of tosylhydrazones[7,8], failed completely. Another approach, the mercury-assisted cyclization of ω-hydroxyalkanal *N,N*-dimethylhydrazones, also did not lead to the desired compounds **2**. Finally, in analogy to the previous work of Eschenmoser et al.[2] on ω-haloamide cyclizations, the silver tetrafluoroborate-promoted cyclization of ω-chloroalkanohydrazides **1** proved to be the only efficient route to "hydrazonolactones" of type **2**.

Thus, reaction of equimolar amounts of the ω-chloroalkanoyl chlorides, *N,N*-dialkylhydrazines, and triethylamine in ether at −78 °C to room temperature affords, after a nonaqueous workup, the ω-chloroalkanohydrazides **1a–e** as colorless solids in 61–91% yield. These compounds are best stored under *n*-pentane in a refrigerator; upon heating or melting decomposition occurs. The 5-chloropentanohydrazides **1d, e** were found to be more stable than their lower homologs **1a–c**. As has been reported for other acid hydrazides[9,10], the ω-chloroderivatives **1a–e** exist as mixtures of (*E*/*Z*)-isomers, as seen from their ^{1}H-N.M.R. spectra. The analytical and spectroscopic data for compounds **1** are summarized in Tables 1 and 4.

In the second step, the ω-chloro hydrazides **1** are treated with silver tetrafluoroborate in tetrahydrofuran. As one equivalent is consumed by complexation with the hydrazino function, two equivalents of the silver salt are required to reach optimum cyclization yields. Whereas dihydro-3*H*-furan-2-one *N,N*-dialkylhydrazones **2a–c** (n = 1) are formed at room temperature, the corresponding tetrahydro-2*H*-pyran-2-one derivatives **2d, e** (n = 2) require heating at 50 °C for 2.5 h for formation.

Table 1. ω-Chloroalkanohydrazides **1a–e** prepared

Product	Yield [%]	m.p.[a] [°C]	Molecular formula[b]	M.S. (70 eV)[c] *m/e* (M^+)
1a	70	48.2–48.4°	$C_6H_{13}ClN_2O$ (164.6)	calc. 166.0687; 164.0716 found 166.0681; 164.0709
1b	75	57.4–58.2°	$C_{10}H_{21}ClN_2O$ (220.8)	calc. 222.1313; 220.1342 found 222.1381; 220.1329
1c	61	84.0–84.4°	$C_8H_{15}ClN_2O$ (190.7)	calc. 192.0843; 190.0873 found 192.0843; 190.0873
1d	78	37.0°	$C_7H_{15}ClN_2O$ (178.6)	calc. 180.0843; 178.0873 found 180.0835; 178.0869
1e[d]	91	48.0°	$C_{11}H_{21}ClN_2O_2$ (248.8)	calc. 250.1262; 248.1291 found 250.1268; 248.1294

[a] Melting points are uncorrected.
[b] Satisfactory microanalyses obtained: C ±0.16, H ±0.17, N ±0.15.
[c] Recorded on a Kratos MS 50 spectrometer, A.E.I., Manchester, U.K.
[d] $[\alpha]_D^{20}$: −36.6° (*c* 2.43, benzene); optical rotation of (*E*/*Z*)-mixture.

To avoid light-induced reduction processes of the silver salts, the reactions are carried out under exclusion of light (aluminium foil wrapped reaction flask). Furthermore, anhydrous reaction conditions must be maintained during the whole procedure to prevent any hydrolysis of the products **2** to the corresponding γ-butyro- or δ-valerolactones, which are difficult to separate from the desired products. After removal of tetrafluoroboric acid in vacuo, all silver compounds are converted into silver iodide, and the product lactone hydrazones **2** are isolated as colorless liquids and purified by distillation (Table 2).

Table 2. Dihydro-3*H*-furan-2-one (**2a-c**) and Tetrahydro-2*H*-pyran-2-one (**2d, e**) *N,N*-Dialkylhydrazones prepared

Product	Yield [%]	b.p.[a] [°C]/torr	Molecular formula[b]	M.S. (70 eV) *m/e* (M^+)
2a	75	75–100°/8	$C_6H_{12}N_2O$ (128.2)	calc. 128.0949 found 128.0953
2b	75	80–100°/5	$C_{10}H_{20}N_2O$ (184.3)	calc. 184.1575 found 184.1580
2c	84	110–120°/5	$C_8H_{14}N_2O$ (154.2)	calc. 154.1106 found 154.1108
2d	75	60–70°/0.6	$C_7H_{14}N_2O$ (142.2)	calc. 142.1106 found 142.1105
2e[c]	83	100°/0.001	$C_{11}H_{20}N_2O_2$ (212.3)	calc. 212.1524 found 212.1527

[a] Boiling points (bath temperatures) are uncorrected (Kugelrohr distillation).
[b] Satisfactory microanalyses obtained: C ±0.39, H ±0.11, N ±0.24; exception: **2a**, N −0.53.
[c] $[\alpha]_D^{20}$: +62.2° (*c* 1.4, benzene); optical rotation of (*E*/*Z*)-mixture.

They are hygrosopic, easily hydrolyzed, and oxidized; for example, chromatography on silica gel leads to rapid decomposition. On heating or under basic conditions, the six-membered lactone hydrazones **2d** and **e** polymerize more readily than their five-membered analogs **2a–c**.

As is evident from their ^{1}H- and ^{13}C-N.M.R. spectroscopic data, these new lactone hydrazones **2** exist as (*E*/*Z*)-mixtures of isomers (Table 4). In agreement with previous ^{13}C-N.M.R. spectroscopic investigations on

Table 3. 1-Dialkylaminopyrrolidin-2-ones (**3a-c**) and 1-Dialkylaminopiperidin-2-ones (**3d, e**) prepared

Product	Yield [%]	b.p.[a] [°C]/torr	Molecular formula[b]	M.S. (70 eV) *m/e* (M^+)
3a	80	75°/8	$C_6H_{12}N_2O$ (128.2)	calc. 128.0949 found 128.0953
3b	96	120°/2	$C_{10}H_{20}N_2O$ (184.3)	calc. 184.1575 found 184.1571
3c	96	100°/1	$C_8H_{14}N_2O$ (154.2)	calc. 154.1106 found 154.1105
3d	68	100°/15	$C_7H_{14}N_2O$ (142.2)	calc. 142.1106 found 142.1110
3e[c]	82	100–105°/0.07	$C_{11}H_{20}N_2O_2$ (212.3)	calc. 212.1524 found 212.1521

[a] Boiling points (bath temperatures) are uncorrected (Kugelrohr distillation).
[b] Satisfactory microanalyses obtained: C ±0.35, H ±0.36, N ±0.09; exception: **3a**, C −1.1; extremely hygroscopic.
[c] $[\alpha]_D^{20}$: −23.6° (*c* 1.08, benzene).

Table 4. Spectral Data of Compounds **1–3**

Compound	I.R. (film)[a] ν [cm^{-1}]	^{1}H-N.M.R. ($CDCl_3$/TMS_{int})[b] δ [ppm]
1a	3230 (NH); 3080, 3010, 2980, 2880 (CH); 2840, 2800 (NCH_3); 1670 (CO); 1570, 1550, 1450, 1320, 1180, 1030	1.9–2.8 [m, 4H, CH_2CO, (*E*/*Z*), CH_2]; 2.5, 2.55 [2s, 6H, $N(CH_3)_2$, (*E*/*Z*)]; 3.6 (2t, 2H, *J* = 6.0 Hz, CH_2Cl, (*E*/*Z*)]; 6.35, 6.5 [2br. s, 1H, NH, (*E*/*Z*)]
1b	3270, 3200, 3100 (NH); 2980, 2950, 2890 (CH); 1670 (CO); 1550, 1420, 1390, 1190, 1150	0.95–1.25 [2d, 12H, *J* = 6.0 Hz, CH_3, (*E*/*Z*)]; 1.95–2.85 [m, 4H, CH_2, CH_2CO, (*E*/*Z*)]; 3.1, 3.15 [2sept, 2H, *J* = 6.0 Hz, NCH, (*E*/*Z*)]; 3.6 [t, 2H, *J* = 6.0 Hz, CH_2Cl]; 6.1, 6.3 [2br s, 1H, NH, (*E*/*Z*)]
1c	3170, 3060 (NH); 2980, 2940 (CH); 2820 (NCH_2); 1660 (CO); 1420, 1320, 980, 790	1.4–1.95 [m, 4H, ring-CH_2]; 2.1 [quint, 2H, *J* = 6.0 Hz, CH_2]; 2.25–3.15 [m, 6H, CH_2CO, (*E*/*Z*), NCH_2]; 3.55, 3.57 [2t, 2H, *J* = 6.0 Hz, CH_2Cl, (*E*/*Z*)]; 6.9, 7.1 [2br. s, 1H, NH, (*E*/*Z*)]
1d	3220 (NH); 3060 (CH_2Cl); 3000, 2960, 2870 (CH); 2830, 2790 (NCH_3); 1670 (CO); 1550, 1450, 1170, 1020	1.7–2.8 [m, 6H, CH_2, CH_2CO, (*E*/*Z*)]; 2.55, 2.65 [2s, 6H, $N(CH_3)_2$, (*E*/*Z*)]; 3.45–3.7 [m, 2H, CH_2Cl]; 7.1, 7.8 [2br. s, 1H, NH, (*E*/*Z*)]
1e	3220 (NH); 3050 (CH_2Cl); 2960, 2940, 2880, 2840 (CH); 1670 (CO); 1550, 1460, 1130, 1100	1.5–3.7 [m, 17H, CH_2, CH_2CO (*E*/*Z*), NCH_2, NCH, CH_2O, CH_2Cl]; 3.3 [s, 3H, OCH_3]; 6.85, 7.2 [2br. s, 1H, NH, (*E*/*Z*)]

Table 4. (continued)

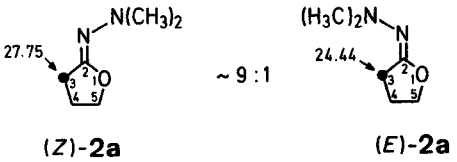
Compound	I.R. (film)[a] ν [cm^{-1}]	^{1}H-N.M.R. ($CDCl_3$/TMS_{int})[b] δ [ppm]
2a[c]	3000, 2970, 2910, 2870 (CH); 2830, 2790 (NCH_3); 1680 (CN); 1470, 1250, 1190, 1050, 1000	1.9–2.3 [2quint, 2H, J = 6.0 Hz, CH_2, (*E*/*Z*)]; 2.4–2.95 [m, 2H, CH_2CN, (*E*/*Z*)]; 2.4, 2.5 [2s, 6H, $N(CH_3)_2$, (*E*/*Z*)]; 4.2, 4.3 [2t, 2H, J = 6.0 Hz, CH_2O, (*E*/*Z*)]
2b	3000, 2950, 2920, 2890 (CH); 1680 (CN); 1470, 1380, 1220, 1060	0.85–1.1 [2d, 12H, J = 6.0 Hz, CH_3, (*E*/*Z*)]; 1.8–2.25 [2quint, 2H, J = 6.0 Hz, CH_2, (*E*/*Z*)]; 2.5–2.8 [2t, 2H, J = 6.0 Hz, CH_2CN, (*E*/*Z*)]; 3.0, 3.05 [2sept, 2H, J = 6.0 Hz, NCH, (*E*/*Z*)]; 4.15, 4.25 [2t, 2H, J = 6.0 Hz, CH_2O, (*E*/*Z*)]
2c	2970, 2820 (CH); 1670 (CN); 1460, 1380, 1190, 1140, 1040	1.65–2.4 [m, 6H, ring-CH_2, CH_2]; 2.45–2.1 [m, 6H, CH_2CN, (*E*/*Z*), NCH_2]; 4.2, 4.3 [2t, 2H, J = 6.0 Hz, CH_2O, (*E*/*Z*)]
2d	2960, 2860 (CH); 2820, 2780 (NCH_3); 1650 (CN); 1470, 1450, 1350, 1230, 1160, 1080, 1060, 1020	1.75–1.95 [m, 4H, CH_2]; 2.3–2.65 [m, 8H, CH_2CN, $N(CH_3)_2$, (*E*/*Z*)]; 4.1–4.3 [m, 2H, CH_2O, (*E*/*Z*)]
2e	2960, 2880, 2840 (CH); 1650 (CN); 1460, 1350, 1280, 1230, 1140, 1070, 1050	1.5–2.15 [m, 8H, CH_2, ring-CH_2]; 2.25–3.75 [m, 7H, CH_2CN, NCH_2, NCH, CH_2O]; 3.3 [s, 3H, OCH_3]; 3.95–4.25 [m, 2H, ring-CH_2O]
3a	2960, 2940, 2860 (CH); 2810, 2770 (NCH_3); 1690 (CO); 1450, 1390, 1270, 1030, 1010	1.8–2.7 [m, 4H, CH_2, CH_2CO]; 2.6 [s, 6H, $N(CH_3)_2$]; 3.45 [t, 2H, NCH_2]
3b	2970, 2930, 2880 (CH); 1700 (CO); 1460, 1380, 1270, 1190, 1170, 1120	0.9–1.25 [m, 12H, CH_3]; 1.75–2.5 [m, 4H, CH_2, CH_2CO]; 3.2–3.7 [m, 4H, NCH_2, NCH]
3c	2990, 2890 (CH); 1700 (CO); 1470, 1400, 1280	1.7–2.45 [m, 8H, CH_2, CH_2CO]; 2.85–3.15 [m, 4H, NCH_2]; 3.4 [t, 2H, J = 6.0 Hz, CH_2NCO]
3d	2950, 2890 (CH); 2820, 2790 (NCH_3); 1660 (CO); 1450, 1410, 1350, 1330, 1310, 1160	1.55–2.0 [m, 4H, CH_2]; 2.35 [t, 2H, J = 6.0 Hz, CH_2CO]; 2.65 [s, 6H, $N(CH_3)_2$]; 3.35 [t, 2H, J = 6.0 Hz, NCH_2]
3e	2940, 2870, 2830 (CH); 1650 (CO); 1460, 1410, 1350, 1330, 1310, 1200, 1110	1.3–2.5 [m, 10H, CH_2, CH_2CO]; 2.95–4.1 [m, 7H, NCH_2, NCH, CH_2O, CH_2NCO]; 3.3 [s, 3H, OCH_3]

[a] Recorded on a Perkin Elmer Acculab 4 spectrophotometer; **1a–e** (KBr).

[b] Recorded on a Varian EM-390 spectrometer.

[c] ^{13}C-N.M.R. ($CDCl_3$/TMS): δ = 161.3 (C-2); 71.4 [C-5, (*Z*)]; 69.2 [C-5, (*E*)]; 47.9 [C—N, (*E*)]; 47.1 [C—N, (*Z*)]; 27.5 [C-3, (*Z*)]; 24.4 [C-3, (*E*)]; 23.0 [C-4, (*Z*)]; 22.9 ppm [C-4, (*E*)]; recorded on a Bruker WP 80 spectrometer.

iminolactones[11] and dimethylhydrazones[12], the major isomer is assigned the (*Z*)-configuration, having the dialkylamino group *syn*-oriented towards the smaller oxygen atom. For instance, the ^{13}C-N.M.R. spectrum of **2** indicates a (*Z*/*E*)-ratio of ~ 9:1; the α-carbon *syn* to the hydrazone group, (*E*)-isomer, appears at higher field (Scheme **B**).



(*Z*)-**2a** (*E*)-**2a**

Scheme **B**

In contrast to the silver tetrafluoroborate-promoted cyclization, deprotonation of the ω-chloroalkanohydrazides **1** with sodium hydride in tetrahydrofuran at −30 °C and stirring at room temperature leads to intramolecular alkylation at nitrogen and thus, the *N*-(dialkylamino)-lactams **3** are formed as colorless, hygroscopic liquids in 68–96% yield (Scheme **A** and Table 3).

The ambidoselective intramolecular cyclizations may simply be interpreted using the HSAB-principle. In the case of lactone hydrazone formation, the hard oxygen of the ambident acid hydrazide moiety attacks the hard cationic center of the silver-activated chloride, whereas the aminolactams are formed by reaction of the soft nitrogen end of the hydrazide anion with the relatively soft, unactivated halide.

In summary, the cyclization procedures described in this paper constitute the first simple and efficient routes to lactone *N*,*N*-dialkylhydrazones **2** and *N*-(dialkylamino)-lactams **3**. Apparently, a wide variety of hydrazines can be transformed into their five or six-membered lactam or lactone derivatives by reaction with readily available ω-chloroalkanoyl chlorides in good overall yields. Of special interest are the chiral, enantiomerically pure compounds **2e** and **3e**, which should be useful for asymmetric synthesis in the way mentioned above. Such electrophilic substitutions with compounds **2** and **3** will be reported in due course.

ω-Chloroalkanohydrazides 1a–e; General Procedure:
In a 100 ml-flask fitted with gas inlet and rubber septum, the *N*,*N*-dialkylhydrazine (20 mmol; dried over calcium hydride) and triethylamine (20 mmol) are dissolved in absolute diethyl ether (50 ml) under argon. The mixture is cooled to −78 °C and the ω-chloroalkanoyl chloride (20 mmol) is introduced with stirring in one portion, causing salt precipitation. Effective stirring is continued overnight, while the temperature is allowed to rise to 10 °C. The solvent is evaporated *in vacuo* and the salts are extracted with diethyl ether (3 × 100 ml). The salts are filtered off, and the combined ether extracts are concentrated *in vacuo* without heating to give the solid ω-chloroalkanohydrazides **1**. After crystallization from warm petroleum ether (30–40 °C), colorless, fluffy needles are obtained. Prolonged heating or boiling should be avoided, because this results in decomposition to oily products. The solid ω-chloroalkanohydrazides **1** are dried under vacuum over potassium hydroxide and are best stored under pentane in a refrigerator (Tables 1 and 4).

Lactone *N*,*N*-Dialkylhydrazones 2a–e; General Procedure:
In a flame-dried flask fitted with gas inlet and rubber septum is placed silver tetrafluoroborate (≥ 98%; 2 g, 10 mmol) under argon. The flask is shielded from light with aluminium foil and dry tetrahydrofuran (15 ml) is introduced via a syringe. After cooling the mixture to 0 °C, a solution of the ω-chloroalkanohydrazide **1** (5 mmol) in dry tetrahydrofuran (7 ml) is added and the mixture is stirred at room temperature for 2 h. In the case of the six-membered ring lactone hydrazones **2a** and **e**, the hydrazide is introduced at room temperature and the mixture is stirred at 50 °C for 2.5 h. Complete conversion of the ω-chloroalkanohydrazides may be checked by T.L.C. (Al_2O_3, basic, ethyl acetate, visualizing with ethanolic phosphomolybdic acid)[13]. To remove tetrafluoroboric acid formed under the

reaction conditions, about 10 ml of the solvent are evaporated *in vacuo* and then replaced again, followed by triethylamine (1.4 ml, 10 mmol) and stirring for 20 min at room temperature. Then a solution of dry potassium iodide (1.66 g, 10 mmol) in absolute methanol (30 ml) is added, which causes silver iodide precipitation. The solvent is removed and the remaining salts are extracted immediately with dry diethyl ether (4 × 80 ml). The salts are collected by filtration over celite, the ether extracts are dried with magnesium sulfate, and the solvent is evaporated *in vacuo* at 30 °C to yield the crude lactone hydrazones **2**, which are purified by Kugelrohr distillation. The hygroscopic lactone hydrazones **2** can be stored over molecular sieves (4Å) under argon in a refrigerator (Tables 2 and 4).

N-(Dialkylamino)-lactams 3a–e; General Procedure:
In a flame-dried flask fitted with gas inlet and rubber septum are placed sodium hydride (0.132 g, 5.5 mmol) and absolute tetrahydrofuran (10 ml). The suspension is cooled to −30 °C and a solution of the ω-chloroalkanohydrazide **1** (5 mmol) in tetrahydrofuran (7 ml) is introduced via a syringe. The cooling bath is removed and hydrogen evolves. Butyrolactam derivatives **3** (n = 1) are formed within 1 h at room temperature, whereas the preparation of valerolactam derivatives **3** (n = 2) requires 5 h at room temperature. The reaction can be monitored by T.L.C. (Al_2O_3, basic, ethyl acetate). The mixture is diluted with ether (50 ml), hydrolyzed with a few drops of water, dried with magnesium sulfate, and concentrated *in vacuo* at 30 °C. Kugelrohr distillation affords the pure products **3** as hygroscopic, colorless liquids, which can be stored over molecular sieves (4Å) in a refrigerator (Tables 3 and 4).

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