FACILE ROUTE TO 1-ALKOXY-3-PYRROLIN-2-ONES BY CYCLIC SULPHINYLATION OF N-ALKOXY-2-VINYLGLYCOLAMIDES

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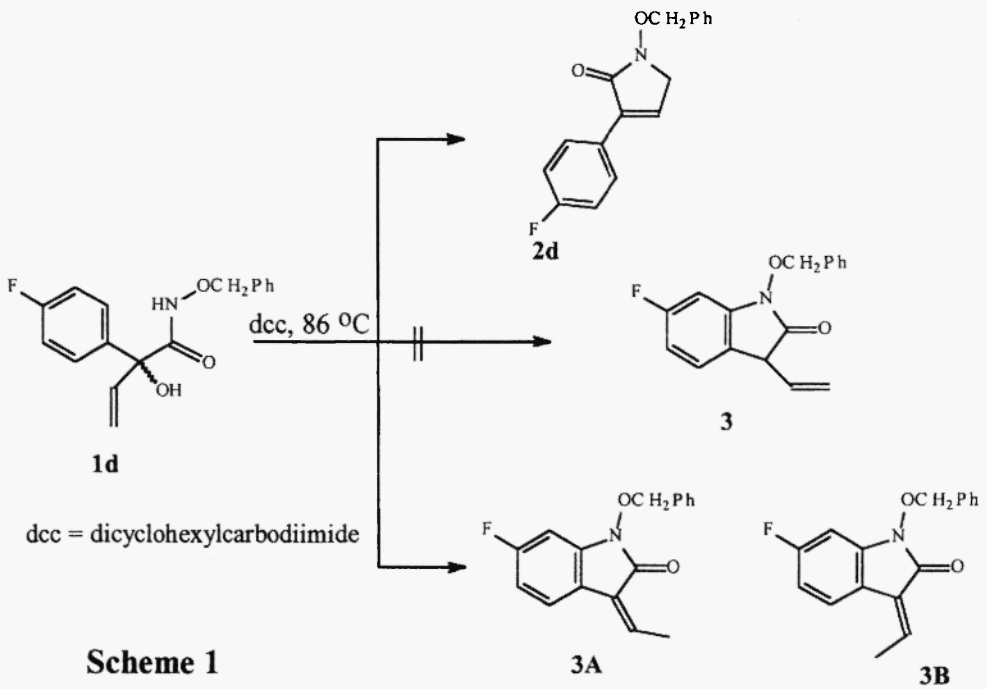
Abstract:

The cyclic sulphinylation of 2-arylated N-alkoxy-2-vinylglycolamides **1a-g** leads to 3-arylated 1-alkoxy-3-pyrrolin-2-ones **2a-g** via the short-lived 1,2,3-oxathiazolidin-4-one 2-oxides **4** in good yields. The synthesis of the 3-methylated 1-benzyloxy-3-pyrrolin-2-one 2**h** could be accomplished by thermolytic extrusion of sulfur dioxide from **4h**.

Introduction:

The dicyclohexylcarbodiimide mediated cyclodehydration of 2-arylsubstituted N-alkoxyglycolamides has been shown to offer an easy access to 1-alkoxy-indolin-2-ones (1) and their thieno analogs (2).

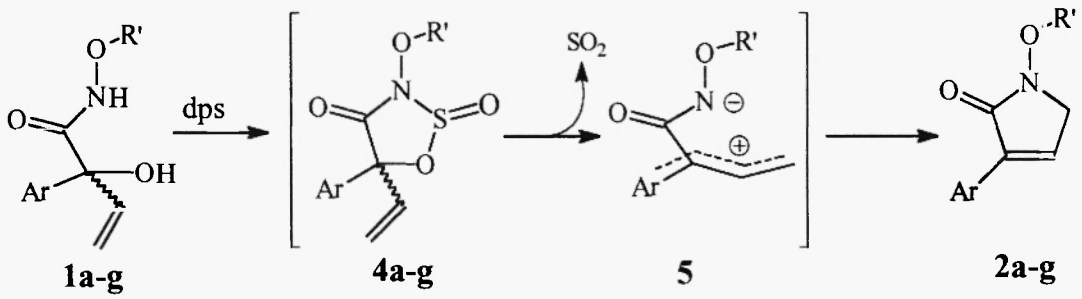


Scheme 1

Contrary to expectation we recently found (3), that the corresponding heterocyclization of the 2-vinylsubstituted N-alkoxyglycolamide **1d** did not afford the desired 3-vinylsubstituted 1-alkoxy-indolin-2-one **3** but rather the 1-alkoxy-3-pyrrolin-2-one **2d** as the major product besides small amounts of the isomeric 3-ethyliden-indolin-2-ones **3A** and **3B** (Scheme 1).
Further studies on a series of various 2-arylated N-alkoxy-2-vinylglycolamides (**1a-g**) revealed this 3-pyrrolin-2-one forming process to be of more general scope in comparison with the very few reported (4) synthetic methods for 1-alkoxy-3-pyrrolin-2-ones, but unfortunately the yields of pure **2** were not satisfactory due to losses during chromatographic separation.
Looking for a more practical approach to the title compounds we expected the 1,2,3-oxathiazolidin-4-one 2-oxides **4** to undergo a facile rearrangement to the desired heterocyclic system **2** on elimination of sulfur dioxide via a dipolar intermediate **5** according to literature (1,3).
As exemplified below, this route (Scheme 2) which parallels the well known ring contracting extrusion of SO_2 from 1,3,2,4-dioxathiazinan-5-one 2-oxides to give 1,2-oxazetidin-3-ones (5), does indeed afford a rapid entry into the family of 3-substituted 1-alkoxy-3-pyrrolin-2-ones.

Results and Discussion

When **1a-g** were allowed to react with freshly prepared 2,2-dipyridyl sulfite (6) in dichloromethane the solution soon became lightly red coloured. By running an IR-spectra immediately after having combined the reactants a short-lived absorption band at 1740 cm^{-1}, attributable to the cyclic sulfurous ester amides **4** could be observed, which disappeared in favour of a new band at 1690 cm^{-1}, indicating the formation of the 3-pyrrolin-2-one nucleus.



1a-g **4a-g** **5** **2a-g**

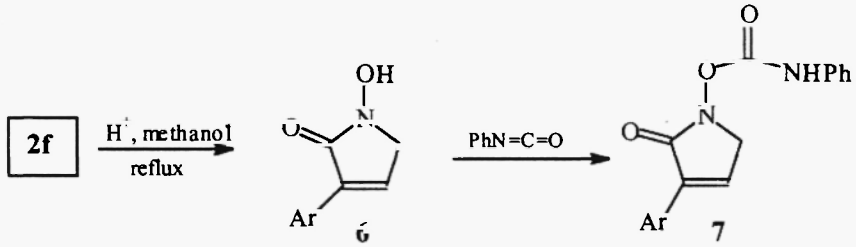
dps = 2,2'-dipyridyl sulfite

Scheme 2

1,2	Ar	R'
a	phenyl	ethoxymethoxy
b	phenyl	t-butyl
c	4-Cl-phenyl	benzyl
d	4-F-phenyl	benzyl
e	4-methoxyphenyl	methyl
f	4-cyclohexylphenyl	ethoxymethoxy
g	2-thienyl	methyl

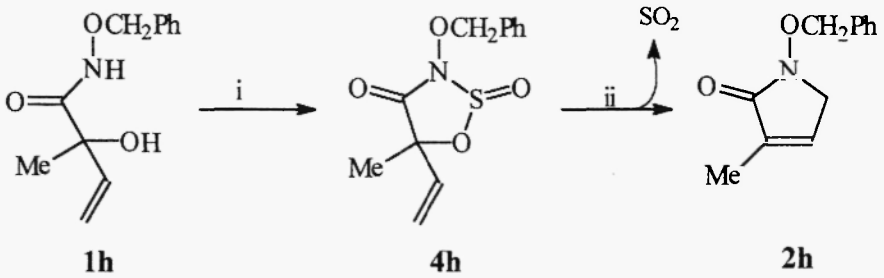
After stirring for 20 min at room temperature diethyl ether was added and the reaction mixture extracted with hydrochloric acid, followed by sodium bicarbonate. Treatment of the organic layer af-

forded a pale yellow solution, which upon evaporation furnished the 1-alkoxy-3-aryl-3-pyrrolin-2-ones **2a-g** as crystalline compounds in yields of 54-71 %.
As exemplified by **2f** the acetalic ethoxymethoxy group could be successfully cleaved by acidic hydrolysis in methanol yielding smoothly the 1-hydroxy-3-pyrrolin-2-one **6** which upon treatment with phenylisocyanate gave the 1-phenylcarbamoyloxy derivative **7** in 90 % yield.



Scheme 3

In contrast to the short-lived **4a-g** the corresponding 5-methyl-5-vinylsubstituted 1,2,3-oxathiazolidin-4-one 2-oxid **4h,** obtained from the sulphinylation of **1h** could be isolated in 62% yield as an oily mixture of diastereomers which did not eliminate sulfur dioxide spontaneously and could be stored at ambient temperature for several months.



i: 2,2'-dipyridyl sulfite; ii: boiling carbon tetrachloride, molecular sieves

Scheme 4

The complete transformation of **4h** required prolonged heating (7 days) in carbon tetrachloride in the presence of molecular sieves and furnished **2h** in 66% yield as a colourless oil.

Conclusion:

In conclusion, we here present an efficient synthetic method for 3-substituted 1-alkoxy-3-pyrrolin-2-ones **2** which reflects not only the versatility of N-alkoxy-glycolamides as building blocks for a variety of heterocyclic systems (1-3, 7) but also contributes to the well established chemistry of cyclic sulfites (8). Results of our ongoing studies toward the chemistry of the ring system **2** will be reported in due course.

Acknowledgement:
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Experimental:

Melting points were taken in open capillary tubes and are uncorrected. The IR-Spectra were recorded on a Philips Unicam SP 3 200. The ^{1}H-NMR-Spectra were recorded on a Bruker AC 250 P spectrometer with TMS as the internal reference. Chromatographic separations were performed on ICN Silica 100-200, active, 60 Å, 10 x 2 cm ∅.

Starting materials:
The N-alkoxy-2-aryl-2-vinylglycolamides **1a-g** were prepared by the reaction of the appropriate N-alkoxy α-oxoarylacetamide (9) with vinylmagnesium bromide according to literature (1b).

N-Ethoxymethoxy-2-hydroxy-2-phenyl-3-butenamide **(1a)**: 68%; mp 58 °C (toluene/petrolether); IR (KBr): 3280 (OH), 3180 (NH), 1670 (CO) cm^{-1}. ^{1}H-NMR ($CDCl_3$) δ = 1.09 (t, 3H, CH_3), 3.67 (q, 2H, OCH_2), 4.76 (s, 2H, OCH_2O), 5.24 (dd,1H, J=10.2, 2.0 Hz, =C*H*H), 5.40 (dd, 1H, J=17.4, 2.0 Hz, =C*H*H), 6.46 (dd, 1H, J=17, 10Hz, =C*H*), 6.24 (s, OH), 7.24-7.56 (5 aromatic H),11.11(s,NH).Anal.Calcd. for $C_{13}H_{17}NO_4$: C, 62.14; H, 6.82; N, 5.57. Found C, 62.44; H, 6.84; N, 5.74.

N-tert-Butoxy-2-hydroxy-2-phenyl-3-butenamide **(1b)**: 68%; mp 107 °C (diethyl ether/ petrolether). IR (KBr): 3420 (OH), 3240 (NH), 1660 (CO) cm^{-1}. ^{1}H-NMR (DMSO-d_6) δ = 1.15 (s, 9H, CH_3), 5.25 (dd, 1H, J=10.2, 2.0 Hz, =C*H*H), 5.41 (dd, 1H, J=17.4, 2.0 Hz, =C*H*H), 6.50 (dd, 1H, J=10.2, 17.4 Hz, =C*H*), 6.18 (s, OH), 7.24-7.51 (m, 5 aromatic H), 10.37 (s, NH). Anal. Calcd. for $C_{14}H_{19}NO_3$: C, 67.45; H, 7.68; N, 5.62. Found: C, 67.60; H, 7.68; N, 5.81.

N-Benzyloxy-2-hydroxy-2-(4-chlorophenyl)-3-butenamide **(1c)**: 62%; mp 109 °C (diethyl ether/ petrolether). IR (KBr): 3350 (OH), 3250 (NH), 1660 (CO) cm^{-1}. ^{1}H-NMR ($CDCl_3$) δ = 3.42 (s, OH), 4.83 (s, 2H, OCH_2), 5.37 (dd, 1H, J=11.3, 2.0 Hz, =C*H*H), 5.44 (dd, 1H, J=18.3, 2.0 Hz, C*H*H), 6.35 (dd, 1H, J=11.3, 18.3 Hz, =C*H*), 7.25-7.40 (m, 9 aromatic H), 9.00 (s, NH). Anal. Calcd. for $C_{17}H_{16}ClNO_3$: C, 64.26; H, 5.08; N, 4.41; Cl, 11.16. Found: C, 64.22; H, 5.20; N, 4.36; Cl, 10.99.

2-Hydroxy-N-methoxy-2-(4-methoxyphenyl)-3-butenamide **(1e)**: 62%; mp 105 °C (diethyl ether/petrolether). IR (KBr): 3330 (OH), 3250 (NH), 1655 (CO) cm^{-1}. ^{1}H-NMR ($CDCl_3$) δ = 3.20 (s, OH), 3.75 (s, 3H, OCH_3), 3.80 (s, 3H, OCH_3), 5.40 (dd, 1H, J= 10.2, 2.0 Hz, =C*H*H), 5.51 (dd, 1H, J=17.4, 2.0 Hz, =C*H*H), 6.40 (dd, 1H, J=10.2, 17.4 Hz, =C*H*), 6.85-6.92 (m, 2 aromatic H), 7.35-7.43 (m, 2 aromatic H), 9.08 (s, NH). Anal. Calcd. for $C_{12}H_{15}NO_4$: C, 60.75;
H, 6.37; N, 5.90. Found: C, 60.57; H 6.34; N, 6.07

2-(4-Cyclohexylphenyl)-N-ethoxymethoxy-2-hydroxy-3-butenamide **(1f)**: 63%; mp 94 °C (diethyl ether/petrolether). IR (KBr) 3300 (OH, NH), 1680 (CO) cm^{-1}. ^{1}H-NMR (DMSO-d_6) δ = 1.08 (s, 3H, CH_3), 1.12-1.90 (m, 10 H, CH_2), 3.65 (q, 2H, OCH_2), 4.75 (s, 2H, OCH_2O), 5.20 (dd, 1H, J= 10.2, 2.0 Hz, =C*H*H), 5.40 (dd, 1H, J=17.4, 2.0 Hz, =C*H*H), 6.42 (dd, 1H, J= 2.0, 17.4 Hz, =C*H*),

6.10 (s, OH), 7.09-7.41 (m, 4 aromatic H), 11.02 (s, NH). Anal. Calcd. for $C_{19}H_{27}NO_4$: C, 68.44; H, 8.16; N, 4.20. Found: C, 68.16; H, 8.14; N, 4.25.

2-Hydroxy-N-methoxy-2-(2-thienyl)-3-butenamide **(1g)**: 66%; mp 100 °C (diethyl ether/ petrolether). IR (KBr): 3380 (OH), 3240 (NH), 1675 (CO) cm^{-1}. ^{1}H-NMR ($CDCl_3$) δ = 3.68 (s, OH), 3.76 (s, 3H, OCH_3), 5.49 (dd, 1H, J=10.2, 2.0 Hz, =C***H***H), 5.55 (dd, 1H, J=17.4, 2.0 Hz, =C***H***H), 6.41 (dd, 1H, J=10.2, 17.4 Hz, =C***H***), 6.95-7.33 (m, 3 aromatic H), 9.14 (s, NH). Anal. Calcd. for $C_9H_{11}NO_3S$: C, 50.69; H, 5.20; N, 6.57; S, 15.04. Found: C, 50.68; H, 5.20; N, 6.53; S, 14.89.

N-Benzyloxy-2-hydroxy-2-methyl-3-butenamide **(1h)**: Pyruvoyl chloride (20 mmoles) (10) was reacted with benzyloxyamine according to (10) to give **N-benzyloxy-α-oxopropanamide** in 34 % yield; mp. 85 °C (toluene/petrolether). IR (KBr) 3180 (NH), 1720, 1670 (CO) cm^{-1}. ^{1}H-NMR (DMSO-d_6) δ = 2.33 (s, 3H, CH_3), 4.91 (s, 2H, OCH_2), 7.16-7.63 (m, 5 aromatic H), 11.80 (s, NH). Anal. Calcd. for $C_{10}H_{11}NO_3$: C, 62.16; H, 5.74; N, 7.25. Found: C, 62.42; H, 5.78; N, 7.35. To a stirred solution of N-benzyloxy-α-oxopropanamide (10 mmoles) in anhydrous diethyl ether were added 40 ml of a 1M vinylmagnesium bromide solution (Aldrich). After stirring at ambient temperature for 60 min the mixture was cooled in an ice-bath and dropwise treated with dilute hydrochloric acid. The organic layer was separated and the aqueous layer extracted twice with 30 ml diethyl ether. The combined organic layers were dried over $MgSO_4$, evaporated in vacuo and the residue chromatographed. After elution with 200 ml dichloromethane (rejected) **1h** was obtained by elution with 300 ml dichloromethane + diethyl ether (5:1) as a colourless oil which solidified on standing in the refrigerator: 39 %; mp 74 °C. IR (KBr): 3250 (OH, NH), 1660 (CO) cm^{-1}. ^{1}H-NMR (DMSO-d_6) δ = 1.33 (s, 3H, CH_3), 4.77 (s, 2H, OCH_2), 5.06 (dd, 1H, J=10.2, 2.0 Hz, =C***H***H), 5.55 (dd, 1H, J=17.4, 2.0 Hz, =C***H***H), 6.01 (dd, 1H, J= 10.2, 17.4 Hz, =C***H***), 5.55 (s, OH), 7.28-7.43 (m, 5 aromatic H), 11.02 (s, NH). Anal. Calcd. for $C_{12}H_{15}NO_3$: C, 65.14; H, 6.83; N, 6.33. Found: C, 65.32; H, 6.82; N, 6.39.

General procedure for the conversion of **1a-g** to the 1-alkoxy-3-aryl-3-pyrrolin-2-ones **2a-g**:

To a stirred solution of 2-pyridone (40 mmoles) and triethylamine (40 mmoles) in 30 ml of anhydrous dichloromethane was added dropwise at 0 °C a solution of thionyl chloride (20 mmoles) in 10 ml anhydrous dichloromethane. After stirring for 5 minutes at ambient temperature the corresponding glycolamide **1** was added and the mixture stirred for 20 minutes. 100 ml of diethyl ether were added and the reaction mixture extracted thrice with 10 ml 3 M hydrochloric acid, followed by 10 ml of saturated sodium bicarbonate solution. The organic layer was dried over $MgSO_4$, decolourized with charcoal and evaporated in vacuo. The remaining residues were recrystallized from the given solvents to afford analytically pure **2a-g.**

1-Ethoxymethoxy-3-phenyl-3-pyrrolin-2-one **(2a)**: 62%; mp 60 °C (diethyl ether/petrolether). IR (KBr): 1700 (CO) cm^{-1}. ^{1}H-NMR ($CDCl_3$): δ = 1.28 (t, 3H, CH_3), 3.86 (q, 2H, OCH_2), 4.29 (d, 2H, J= 2.0 Hz, H-5), 7.11 (t, 1H, J= 2.0 Hz, H-4), 5.10 (s, 2H, OCH_2O) 7.34-7.43 (m, 3 aromatic H), 7.78-7.85 (m, 2 aromatic H). Anal. Calcd. for $C_{13}H_{15}NO_3$: C, 66.65; H, 6.54; N, 6.06. Found: C, 66.68; H, 6.50; N, 6.21.

1-tert-Butoxy-3-phenyl-3-pyrrolin-2-one **(2b):** 63%; mp 86 °C (diethyl ether/petrolether). IR

(KBr): 1690 (CO) cm^{-1}. ^{1}H-NMR ($CDCl_3$) δ = 1.37 (s, 9H, CH_3), 4.17 (d, 2H, J=2.0 Hz, H-5), 7.00 (t, 1H, J=2.0 Hz, H-4), 7.70-7,85 (m, 2 aromatic H). Anal.Calcd. for $C_{14}H_{17}NO_2$: C, 72.70; H, 7.41; N, 6.06. Found: C, 72.58; H, 7.40; N, 6.33.

1-Benzyloxy-3-(4-chlorophenyl)-3-pyrrolin-2-one **(2c)**: 58%; mp 127 °C (dichloromethane/ petrolether). IR (KBr): 1670 (CO) cm^{-1}. ^{1}H-NMR ($CDCl_3$): 3.89 (d, 2H, J=2.1 Hz, H-5), 6.98 (t, 1H, J=2.1 Hz, H-4), 5.11 (s, 2H, OCH_2), 7.30-7.88 (m, 9 aromatic H). Anal. Calcd for $C_{17}H_{14}ClNO_2$: C, 68.12; H, 4.71; N, 4.67; Cl, 11.83. Found: C, 67.93; H, 4.71; N, 5.05; Cl, 11.53.

1-Benzyloxy-3-(4-fluorophenyl)-3-pyrrolin-2-one **(2d)**: 63%; mp 111 °C, Lit (3) mp 111 °C.

1-Methoxy-3-(4-methoxyphenyl)-3-pyrrolin-2-one **(2e)**: 71%; mp 114 °C (diethyl ether/petrolether). IR (KBr): 1675 (CO) cm^{-1}. ^{1}H-NMR ($CDCl_3$): δ = 3.83 (s, 3H, OCH_3), 3.92 (s, 3H, OCH_3), 4.21 (d, 2H, J=2.0 Hz, H-5), 6.98 (t, 1H, J=2.0 Hz, H-4), 6.90-7.00 (m, 2 aromatic H), 7.78-7.88 (2 aromatic H). Anal. Calcd. for $C_{12}H_{13}NO_3$: C, 65.74; H, 5.98; N, 6.39. Found: C, 65.43; H, 5.93; N, 6.55.

3-(4-Cyclohexylphenyl)-1-ethoxymethoxy-3-pyrrolin-2-one **(2f)**: 64%; mp.105 °C (diethyl ether/ petrolether). IR (KBr): 1690 (CO) cm^{-1}. ^{1}H-NMR ($CDCl_3$): δ = 1.15-1.93 (m, 13H, CH_3, CH_2), 2.31-2.70 (m, 1H, CH), 3.86 (q, 2H, OCH_2), 4.27 (d, 2H, J=2.0 Hz, H-5), 7.05 (t, 1H, J=2.0 Hz, H-4), 7.17-7.27 (m, 2 aromatic H), 5.10 (s, 2H, OCH_2O), 7.70-7.80 (m, 2 aromatic H). Anal. Calcd. for $C_{19}H_{25}NO_3$: C, 72.35; H, 7.99; N, 4.44. Found: C, 72.27; H, 7.91; N, 4.71.

1-Methoxy-3-(2-thienyl)-3-pyrrolin-2-one **(2g)**: 54%; mp 95 °C (diethyl ether/petrolether). IR (KBr): 1690 (CO) cm^{-1}. ^{1}H-NMR ($CDCl_3$): δ = 3.93 (s, 3H, OCH_3), 4.24 (d, 2H, J=2.1 Hz, H-5), 6.95 (t, 1H, J=2.1 Hz, H-4), 7.05-7.77 (m, 3 aromatic H). Anal. Calcd. for $C_9H_9NO_2S$: C, 55.37; H, 4.65; N, 7.17; S, 16.42. Found: C, 55.19; H, 4.73; N, 7.06; S, 16.27.

3-Benzyloxy-5-methyl-5-vinyl-1,2,3-oxathiazolidin-4-one 2-oxide **(4h)**: **1h** (10 mmol) was reacted with 2,2'-dipyridyl sulfite according to the general procedure for **2a-g**. The oily residue was chromatographed on silica gel. Elution with dichloromethane afforded 62 % **4h** as a colourless oil. IR (neat): 1740 (CO), 1200 (SO) cm^{-1}. Anal.Calcd. for $C_{12}H_{13}NO_4S$: C, 53.92; H, 4.90; N, 5.24; S, 11.99. Found: C, 53.90; H, 5.09 N, 5.45; S, 12.21.

1-Benzyloxy-3-methyl-3-pyrrolin-2-one **(2h)**: A solution of **4h** (1 mmol) in 5 ml tetrachloromethane was refluxed over 3g molecular sieves (0.4 nm) for 7 days. After evaporation in vacuo the residue was chromatographed by centifugal chromatography (Chromatotron, Model 8924, Harrison Reserarch, Palo Alto, CA, U.S.A., silica gel 60 PF 254, 2 mm, Merck Darmstadt, 8ml/min). Elution with n-hexane/diethyl ether (3:1) afforded 69% **2h** as a colourless oil. IR (neat): 1695 (CO) cm^{-1}. ^{1}H-NMR ($CDCl_3$): δ = 1.87 (m, 3H, CH_3), 3.68-3.72 (m, 2H, CH_2), 5.02 (s, 2H, OCH_2), 6.45-6.49 (m, 1H, CH=), 7.33-7.44 (m, 5 aromatic H). Anal. Calcd. for $C_{12}H_{13}NO_2$: C, 70.92; H, 6.45; N, 6.89. Found: C, 69.75; H, 6.27; N, 6.46.

3-(4-Cyclohexylphenyl)-1-hydroxy-3-pyrrolin-2-one **(6)**. A solution of **2g** (1 mmol) in 8 ml methanol was refluxed for 16 h over Lewatit SPC 108 (Bayer AG). After evaporation in vacuo the residue was dissolved in 10 ml dichloromethane and washed with 1M hydrochloric acid. The organic layer

was dried over $MgSO_4$ and evaporated in vacuo. Yield 86%; mp 200-202 °C (dichloromethane/petrolether). IR (KBr): 3300-2200 (OH), 1665 (CO) cm^{-1}. ^{1}H-NMR ($CDCl_3$): δ = 1.19-1.90 (m, 10H, CH_2), 2.30-2.70 (m, 1H, CH), 4.28 (d, 2H, J=2.1 Hz, H-5), 6.97 (t, 1H, J=2.1 Hz, H-4), 7.16-7.26 (m, 2 aromatic H), 7.65-7.75 (m, 2 aromatic H). Anal. Calcd. for $C_{16}H_{19}NO_2$: C, 74.68; H, 7.44; N, 5.44. Found: C, 74.61; H, 7.41; N, 5.44.

***3-(4-Cyclohexylphenyl)-1-phenylcarbamoyloxy-3-pyrrolin-2-one* (7)**: **6** (1mmol) was reacted in 10 ml anhydrous dichloromethane with phenylisocyanate (1 mmol) and a catalytic amount of 4-dimethylaminopyridine at 0 °C for 30 min, followed by the addition of petrolether. Yield 90%; mp 161 °C (dichloromethane/petrolether). IR (KBr): 3270 (NH), 1780, 1685 (CO) cm^{-1}. ^{1}H-NMR ($CDCl_3$) δ = 1.15-1.95 (m, 10H, CH_2), 2.52 (m, 1H, CH), 4.39 (d, 2H, J=2.1 Hz, H-5), 7.17 (t, 1H, J=2.1 Hz, H-4), 7.05-7.46 (m, 7 aromatic H), 7.68-7.78 (m, 2 aromatic H), 8.08 (s, NH). Anal. Calcd. for $C_{23}H_{24}N_2O_3$: C, 73.38; H, 6.43; N, 7.44. Found: C, 73.28; H, 6.34; N, 7.45.

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