Monatshefte für Chemie 125, 963–969 (1994)

Monatshefte für Chemie
Chemical Monthly

Synthesis and Stereochemistry of 1-Acyl-2-alkyl-1,2,3,4-tetrahydroquinoline-3,4-epoxides

M. Kratzel[1,*], **R. Hiessböck**[1], and **H. Völlenkle**[2]

[1] Institute of Pharmaceutical Chemistry, University of Vienna, A-1090 Vienna, Austria
[2] Institute of Mineralogy, Crystallography and Structural Chemistry, Technical University of Vienna, A-1060 Vienna, Austria

Summary. Analogous to the recently described synthesis of Reissert epoxides the treatment of 1-acyl-2-alkyl-1,2-dihydroquinolines with *m*-chloroperoxybenzoic acid gave diasteromeric pure epoxides which are stable in crystalline state, but reactive in solution versus nucleophiles. Acting as useful intermediates to stereocontrolled functionalized 1,2,3,4-tetrahydroquinolines an X-ray analysis was performed to confirm the relative stereochemistry of the epoxides.

Keywords. 2-Alkyl-1,2-dihydroquinolines; 1,2,3,4-Tetrahydroquinolin-3,4-epoxides; X-Ray analysis.

Synthese und Stereochemie von 1-Acyl-2-alkyl-1,2,3,4-tetrahydrochinolin-3,4-epoxiden

Zusammenfassung. Analog zu der kürzlich beschriebenen Synthese von Reissert-Epoxiden liefern 1-Acyl-2-alkyl-1,2-dihydrochinoline bei Behandlung mit *m*-Chlorperbenzoesäure diastereomerenreine Epoxide, die sich als Festsubstanzen außerordentlich stabil, in Lösung aber reaktiv gegenüber Nucleophilen erweisen. Da sie wertvolle Ausgangsverbindungen zu 1,2,3,4-substituierten Tetrahydrochinolinen mit definierter relativer Konfiguration darstellen, wurde die Stereochemie der Epoxide mittels Röntgenstrukturanalyse abgesichert.

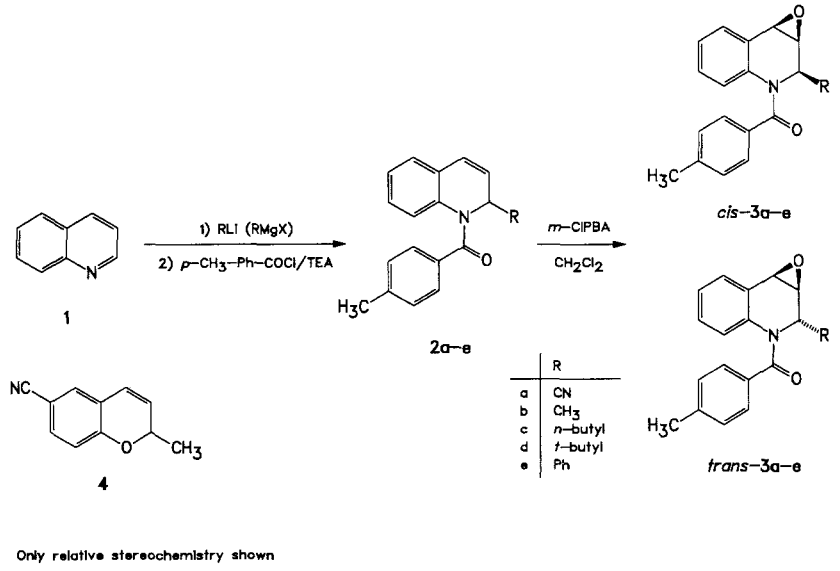
Introduction

Recently we have reported on the synthesis of Reissert epoxides (e.g. **3a**) as oxidation products of quinolines with well defined structure [1]. Furthermore, we have described that the epoxidation occurs stereospecifically yielding the racemate of only one diastereomer. Deduced from ^{1}H NMR measurements including n.O.e. experiments we believed that the oxirane ring and the 2-cyano substituent are related *cis*. The small $^3J_{2,3}$ coupling of 2.5 Hz was interpreted to be in accordance with a *trans* position of the epoxide oxygen and the proton on carbon 2 [2].

Due to the great reactivity of the Reissert epoxides (e.g. **3a**) versus N-nucleophiles [3], which is caused by the cyano group acting as nucleofugal, we planned to synthesize stabilized analogues. At first we chose an approach to derivatives with an alkyl substituent instead of the cyano group.

Results and Discussion

Thus, our synthetic strategy based upon the synthesis of the Reissert epoxides: Quinoline (**1**) was treated with alkyllithiums or alkyl Grignard reagents, respectively, yielding 2-alkyl substituted 1,2-dihydroquinolines [4] which were converted by an appropriate acid chloride to the 2-alkyl Reissert analogues **2b–e**. Oxidation of **2b–e** with *m*-chloroperoxybenzoic acid led to epoxides **3b–e** which were formed diastereoselectively in good to excellent yields. Analyzing the ^{1}H NMR spectrum of **3b**, the $^3J_{2,3}$ and $^3J_{3,4}$ coupling constants were almost identical to the values of **3a** that an analogous relative stereochemistry could be deduced.



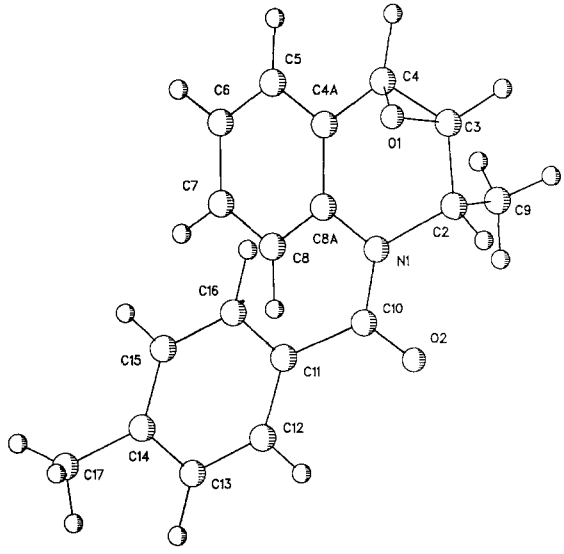


Fig. 1. Perspective view and atomic numbering of the X-ray structure of **3b** [9]

Table 1. Atomic coordinates and thermal parameters of **3b** [Å^2] with standard deviations ( )

	x	*y*	*z*	*U*11	*U*22	*U*33	*U*12	*U*13	*U*23
C2	0.8196(4)	0.037(1)	1.0334(4)	0.036(3)	0.050(4)	0.047(4)	0.004(3)	0.004(3)	−0.007(3)
C3	0.8212(6)	−0.193(1)	1.0653(5)	0.066(5)	0.040(4)	0.066(5)	0.010(4)	0.011(4)	−0.007(4)
C4	0.7530(5)	−0.262(1)	1.1174(5)	0.054(4)	0.050(4)	0.056(4)	0.003(4)	−0.005(3)	0.004(4)
C4A	0.6834(5)	−0.095(1)	1.1365(4)	0.039(3)	0.050(4)	0.036(3)	−0.012(3)	0.001(2)	−0.001(3)
C5	0.6355(5)	−0.121(1)	1.2028(5)	0.060(4)	0.070(5)	0.048(4)	−0.005(4)	0.008(3)	0.007(4)
C6	0.5740(5)	0.035(1)	1.2215(5)	0.054(4)	0.096(6)	0.034(4)	−0.009(4)	0.009(3)	0.006(4)
C7	0.5639(5)	0.228(1)	1.1778(4)	0.042(3)	0.083(5)	0.037(4)	0.007(4)	0.005(3)	0.002(4)
C8	0.6126(4)	0.259(1)	1.1120(4)	0.037(3)	0.057(4)	0.039(3)	−0.003(3)	0.003(2)	−0.005(3)
C8A	0.6705(4)	0.094(1)	1.0901(4)	0.035(3)	0.044(4)	0.029(3)	−0.006(3)	0.000(2)	0.003(3)
C9	0.8849(5)	0.183(1)	1.0907(5)	0.042(4)	0.052(4)	0.074(5)	−0.001(3)	−0.002(3)	−0.002(4)
C10	0.6886(4)	0.206(1)	0.9494(4)	0.045(3)	0.038(3)	0.035(3)	−0.005(3)	0.009(3)	0.001(3)
C11	0.5848(4)	0.227(1)	0.9253(4)	0.034(3)	0.035(4)	0.029(3)	−0.002(3)	0.008(2)	−0.000(3)
C12	0.5487(4)	0.407(1)	0.8831(4)	0.038(3)	0.038(4)	0.045(4)	−0.003(3)	0.005(3)	0.006(3)
C13	0.4544(5)	0.417(1)	0.8516(5)	0.055(4)	0.047(4)	0.048(4)	0.005(4)	0.010(3)	0.010(3)
C14	0.3957(4)	0.240(1)	0.8608(4)	0.040(3)	0.045(4)	0.037(3)	0.005(3)	0.008(3)	−0.004(3)
C15	0.4320(5)	0.059(1)	0.9025(4)	0.037(3)	0.047(4)	0.039(3)	−0.007(3)	0.006(2)	−0.001(3)
C16	0.5260(4)	0.052(1)	0.9355(4)	0.041(3)	0.041(4)	0.041(3)	−0.008(3)	0.003(3)	−0.000(3)
C17	0.2932(5)	0.245(1)	0.8256(5)	0.044(4)	0.086(5)	0.060(4)	−0.008(4)	0.001(3)	0.002(4)
N1	0.7217(4)	0.1194(8)	1.0224(4)	0.041(3)	0.045(3)	0.034(3)	0.004(2)	0.006(2)	0.004(2)
O1	0.7463(4)	−0.3295(7)	1.0333(4)	0.071(3)	0.042(3)	0.078(3)	−0.005(2)	0.003(2)	−0.012(3)
O2	0.7400	0.2585(7)	0.9000	0.043(2)	0.059(3)	0.048(2)	−0.011(2)	0.012(2)	0.007(2)

Table 2. Calculated atomic coordinates of hydrogen atoms of **3b**

	x	*y*	*z*
H21	0.844	0.041	0.975
H31	0.890	−0.269	1.071
H41	0.776	−0.395	1.161
H51	0.647	−0.265	1.240
H61	0.533	0.007	1.271
H71	0.518	0.353	1.195
H81	0.606	0.409	1.078
H91	0.883	0.349	1.069
H92	0.950	0.110	1.083
H93	0.876	0.183	1.155
H121	0.593	0.542	0.874
H131	0.427	0.562	0.820
H151	0.388	−0.078	0.910
H161	0.553	−0.090	0.969
H171	0.297	0.412	0.805
H172	0.244	0.234	0.868
H173	0.272	0.141	0.774

The formation of a defined diastereomer in the epoxidation step is a really remarkable fact, all the more as in the comparable epoxidation of the nor-methyl cromakalim precursors **4** the diastereomeric epoxides are generated in nearly equal amounts [5].

In the meantime, we have synthesized several epoxides with alkyl substituents adjacent to the nitrogen, including the bulky 2-*tert.*-butyl derivative **3d**. All compounds **2b–e** gave epoxides in high yields. Hence, there is much evidence that the 3,4-double bond is attacked by the peracid from the rear yielding the *trans* products **3b–e**. To get definite clarification we performed an X-ray analysis of **3b** which finally revealed that the substituents are related *trans*, indeed (Fig. 1; the atom coordinates and thermal factors are summarized in Tables 1 and 2). This conforms with the suggested reactivity but conflicts with the supposed relative stereochemistry, previously deduced from the cyano congener (and derivatives of) **3a**. Particularly, it is fundamental to know the relative stereochemistry of the epoxides with regard to the synthesis of variable substituted 1,2,3,4-tetrahydroquinolines by oxirane ring opening which are also characterized by a well defined relative stereochemistry [6].

Experimental Part

Melting points: Kofler hot stage apparatus (uncorrected). ^{1}H NMR: Bruker AC 80, Varian UNITY-plus 300, solvent: $CDCl_3$, TMS as the internal reference. ^{13}C NMR: Bruker AC 80, Varian UNITYplus 300, solvent: $CDCl_3$, chemical shifts are given in ppm related to the resonance of $CDCl_3$ (77.0 ppm) (* indicates peaks with double intensity, # means peaks with triple intensity). IR: Perkin Elmer 298. MS: Hewlett Packard 5890A/5970B-GC/MSD instrument, the mass spectra of compounds **2d** and **3d** were obtained on a Finnigan MAT 8230 (70 eV). Elemental analyses: Mikroanalytisches Laboratorium, Institute of Physical Chemistry, University of Vienna.

Preparation of 2-Alkyl-1,2-dihydroquinolines – General Procedure

According to [4].

*Preparation of 1-Acyl-2-alkyl-1,2-dihydroquinolines (**2b–e**) – General Procedure*

To a stirred solution of 2-alkyl-1,2-dihydroquinoline (9.5 mmol) in dichloromethane (20 ml), triethylamine (10 mmol, 1.39 ml) was added, followed by a solution of *p*-toluoyl chloride (9.0 mmol, 1.19 ml) in dichloromethane (10 ml) under ice cooling. Then, the reaction mixture was stirred at room temperature over night. Finally, the organic layer was washed twice with diluted hydrochloric acid, twice with saturated sodium bicarbonate solution, and twice with brine. The organic layer was separated, dried over anhydrous sodium sulphate and evaporated in vacuo.

2-Methyl-1-(p-toluoyl)-1,2-dihydroquinoline (**2b**)

Prepared by acylation of 2-methyl-1,2-dihydroquinoline (9.5 mmol, 1.38 g). Colorless crystals from methanol/acetone, m.p. 132–133 °C, additional purification of the mother liquor by column chromatography on silica gel (petroleum ether/diethylether = 1 + 1), total yield: 1.78 g **2b** (75%). ^{1}H NMR (80 MHz): δ (ppm) = 7.40–6.68 (m, 9H, arom. H, 4-H), 6.12 (dd, 1H, J = 5.5 Hz, J = 9.5 Hz, 3-H), 5.28 (m, 1H, 2-H), 2.30 (s, 3H, 4′-CH_3), 1.20 (d, 3H, J = 7.0 Hz, 2-CH_3). ^{13}C NMR (20.12 MHz): δ (ppm) = 169.6 (Ar–**C**O–N), 140.4 (4′-C), 135.2 (8a–C), 132.8 (1′-C), 126.9 (4a–C), 131.7, 129.0, 128.5, 126.5, 126.0, 125.7, 124.4, 123.9 (arom. **C**–H, 3-C, 4-C), 48.8 (2-C), 21.3 (4′-CH_3), 17.5 (2-CH_3). IR (KBr): 1650 cm^{-1} (ν_{amide}). MS (*m/z*): 263 (M^+). Anal. calcd. for $C_{18}H_{17}NO$: C 82.10, H 6.51, N 5.32. Found: C 82.23, H 6.48, N 5.34.

2-n-Butyl-1-(p-toluoyl)-1,2-dihydroquinoline (**2c**)

Prepared by acylation of 2-*n*-butyl-1,2-dihydroquinoline (9.5 mmol, 1.78 g). Colorless crystals from diethylether, m.p. 77–78 °C, additional purification of the mother liquor by column chromatography on silica gel (petroleum ether/diethylether = 1 + 1), total yield: 2.05 g **2c** (71%). ^{1}H NMR (80 MHz): δ (ppm) = 7.40–6.45 (m, 9H, arom. H, 4-H), 6.19 (dd, 1H, J = 5.5 Hz, J = 10.0 Hz, 3-H), 5.21 (m, 1H, 2-H), 2.31 (s, 3H, 4′-CH_3), 1.68–1.10, 1.05–0.68 (m, 9H, *n*-butyl-H). ^{13}C NMR (75.43 MHz): δ (ppm) = 169.8 (Ar–**C**O–N), 140.2 (4′-C), 135.9 (8a–C), 133.0 (1′-C), 127.6 (4a–C), 131.3, 130.5, 129.5, 129.0, 128.5, 126.5, 126.0, 125.8, 124.6, 124.3 (arom. **C**–H, 3-C, 4-C), 52.5 (2-C), 32.2, 27.6, 22.5 (butyl-1″-C, -2″-C, and -3″-C), 21.3 (4′-CH_3), 13.9 (2-CH_3). IR (KBr): 1640 cm^{-1} (ν_{amide}). MS (*m/z*): 305 (M^+). Anal. calcd. for $C_{21}H_{23}NO$: C 82.59, H 7.59, N 4.59. Found: C 82.81, H 7.78, N 4.50.

2-t-Butyl-(p-toluoyl)-1,2-dihydroquinoline (**2d**)

Prepared by acylation of 2-*t*-butyl-1,2-dihydroquinoline (9.5 mmol, 1.78 g). Colorless crystals from diethylether, m.p. 131–133 °C, additional purification by column chromatography (petroleum ether/diethylether = 1 + 1), total yield: 1.30 g **2d** (45%). ^{1}H NMR (80 MHz): δ (ppm) = 7.44–6.04 (m, 10H, arom. H, 3-H, 4-H), 5.12 (d, 1H, J = 5.5 Hz, 2-H), 2.31 (s, 3H, 4′-CH_3), 0.89 (s, 9H, *t*-butyl-CH_3). ^{13}C NMR (20.12 MHz): δ (ppm) = 170.4 (Ar–**C**O–N), 139.7, 139.6, 138.0, 133.4 (4a–C, 8a–C, 1′-C, 4′-C) 130.6, 129.5, 129.1, 128.9, 128.4, 126.6, 126.0, 125.7, 125.6, 124.6 (arom. **C**–H, 3-C, 4-C), 60.2 (2-C), 37.9 (–**C**–(CH_3)$_3$), 26.2 (C–(**C**H$_3$)$_3$), 21.3 (4′-CH_3). IR (KBr): 1645 cm^{-1} (ν_{amide}). MS (*m/z*): 248 (M^+ – *t*-butyl). Anal. calcd. for $C_{21}H_{23}NO$: C 82.59, H 7.59, N 4.59. Found: C 82.32, H 7.78, N 4.54.

2-Phenyl-1-(p-toluoyl)-1,2-dihydroquinoline (**2e**)

Prepared by acylation of 2-phenyl-1,2-dihydroquinoline (9.5 mmol, 1.97 g). Colorless crystals from diethylether, m.p. 144–145 °C, additional purification by column chromatography (petroleum

ether/diethylether = 2 + 1), total yield: 1.70 g **2e** (58%). ^{1}H NMR (80 MHz): δ (ppm) = 7.74–6.64 (m, 13H, arom. H), 6.50–6.27 (m, 3H, 2-H, 3-H, and 4-H), 2.30 (s, 3H, 4′-CH_3). ^{13}C NMR (75.43 MHz): δ (ppm) = 170.0 (Ar–CO–N), 140.6, 138.8, 136.0, 132.6, 127.4 (4a-C, 8a-C, 1′-C, 4′-C, 1″-C), 129.5, 129.2, 128.6, 128.4, 127.7, 127.5, 126.9, 126.1, 126.0, 125.3, 124.7 (remaining arom. C), 55.1 (2-C), 21.4 (4′-CH_3). IR (KBr): 1650 cm^{-1} (ν_{amide}). MS (*m/z*): 325 (M^+). Anal. calcd. for $C_{23}H_{19}NO$: C 84.89, H 5.88, N 4.30. Found: C 84.61, H 6.05, N 4.21.

Preparation of 1-Acyl-2-alkyl-1,2,3,4-tetrahydroquinolin-3,4-epoxides – General Procedure

1-Acyl-2-alkyl-1,2-dihydroquinoline **2b–e** (1 mmol) and *m*-chloroperoxybenzoic acid (1.5 mmol) were stirred in methylene chloride (20 ml) at room temperature until complete conversion was indicated by TLC (2–3 h). Then the reaction mixture was treated with 2*N* sodium carbonate solution (10 min). Separation of the organic phase, additional twice extraction of the aqueous layer with methylene chloride, washing the combined organic fractions with 2*N* sodium carbonate solution, afforded, after drying over anhydrous sodium sulphate and evaporation, **3b–e**.

3,4-Epoxy-2-methyl-1-(p-toluoyl)-1,2,3,4-tetrahydroquinoline (**3b**)

Colorless crystals from diethylether, m.p. 152–155 °C, additional purification of the mother liquor by column chromatography on silica gel (petroleum ether/diethylether = 1 + 2), total yield: 210 mg **3b** (75%). ^{1}H NMR (80 MHz): δ (ppm) = 7.51–6.82 (m, 7H, arom. H), 6.60–6.40 (m, 1H, arom. H), 5.32 (dq, 1H, J = 2.1 Hz, J = 6.8 Hz, 2-H), 3.95 (d, 1H, J = 4.4 Hz, 4-H), 3.79 (dd, 1H, J = 2.1 Hz, J = 4.4 Hz, 3-H), 2.29 (s, 3H, 4′-CH_3), 1.13 (d, 3H, J = 6.8 Hz, 2-CH_3). ^{13}C NMR (20.12 MHz): δ (ppm) = 170.5 (Ar–CO–N), 140.2, 135.9, 132.8, 129.1, 128.8*, 128.3*, 128.1, 126.8, 125.3, 124.5 (C arom.), 61.8 (4-C), 50.5 (3-C), 45.3 (2-C), 21.1 (4′-CH_3), 13.9 (2-CH_3). IR (KBr): 1650 cm^{-1} (ν_{amide}). MS (*m/z*): 279 (M^+). Anal. calcd. for $C_{18}H_{17}NO_2$: C 77.39, H 6.13, N 5.01. Found C 77.15, H 6.29, N 5.07.

2-n-Butyl-3,4-epoxy-1-(p-toluoyl)-1,2,3,4-tetrahydroquinoline (**3c**)

Colorless crystals from diethylether, m.p. 112–113 °C, additional purification of the mother liquor by column chromatography on silica gel (petroleum ether/diethylether = 1 + 1), total yield: 250 mg **3c** (78%). ^{1}H NMR (80 MHz): δ (ppm) = 7.55–6.82 (m, 7H, arom. H), 6.60–6.40 (m, 1H, arom. H), 5.40–5.13 (m, 1H, 2-H), 3.95 (d, 1H, J = 4.4 Hz, 4-H), 3.84 (dd, 1H, J = 2.4 Hz, J = 4.4 Hz, 3-H), 2.27 (s, 3H, 4′-CH_3), 1.65–1.05 (m, 6H, butyl–CH_2–), 1.05–0.70 (m, 3H, butyl–CH_3). ^{13}C NMR (20.12 MHz): δ (ppm) = 170.8 (Ar–CO–N), 139.9, 136.3, 135.0, 129.0, 128.8*, 128.3*, 128.2, 127.0, 125.8, 124.7 (arom. C), 61.4 (4-C), 50.6 (3-C), 49.3 (2-C), 28.4, 27.4, 22.3 (butyl–CH_2), 21.2 (4′-CH_3). 13.7 (butyl–CH_3). IR (KBr): 1635 (ν_{amide}). MS (*m/z*): 321 (M^+). Anal. calcd. for $C_{21}H_{23}NO_2$: C 78.47, H 7.21, N 4.35. Found C 78.23, H 7.12, N 4.24.

2-t-Butyl-3,4-epoxy-1-(p-toluoyl)-1,2,3,4-tetrahydroquinoline (**3d**)

Colorless crystals from diethylether, m.p. 177–180 °C, yield: 255 mg (79%). ^{1}H NMR (80 MHz): δ (ppm) = 7.48–6.80 (m, 7H, arom. H), 6.53–6.35 (m, 1H, arom. H), 5.08 (m, 1H, 2-H), 4.03 (s, 2H, 3-H, 4-H), 2.27 (s 3H, 4′-CH_3), 0.93 (s, 9H, *t*-butyl–CH_3); ^{1}H NMR (300 MHz, section from 5.5 to 3.5 ppm): δ (ppm) = 5.07 (br.s, 1H, 2-H), 4.02 (dd, 1H, J = 2.0 Hz, J = 4.2 Hz, 3-H), 4.01 (d, 1H, J = 4.2 Hz, 4-H). ^{13}C NMR (75.43 MHz): δ (ppm) = 171.5 (Ar–CO–N), 139.8, 138.5, 133.4, 129.0, 128.9*, 128.5, 128.4*, 127.1, 126.8, 124.8 (arom.C), 59.6 (4-C), 57.0 (3-C), 51.8 (2-C), 35.2 (–C–$(CH_3)_3$), 27.8# (–C–$(CH_3)_3$), 21.3 (4′-CH_3). IR (KBr): 1645 cm^{-1} (ν_{amide}). MS (*m/z*): 321 (M^+). Anal. calcd. for $C_{21}H_{23}NO_2$: C 78.47, H 7.21, N 4.36. Found C 78.23, H 7.31, N 4.29.

3,4-Epoxy-2-phenyl-1-(p-toluoyl)-1,2,3,4-tetrahydroquinoline (**3e**)

Colorless crystals from diethylether, m.p. 156–158 °C, additional purification of the mother liquor by column chromatography on silica gel (petroleum ether/diethylether = 1 + 1), total yield: 300 mg **3e** (88%). ^{1}H NMR (300 MHz): δ (ppm) = 7.46 (d, 1H, J = 7.2 Hz, arom. H), 7.34–7.19 (m, 7H, arom. H), 7.09–6.87 (m, 4H, arom. H), 6.39 (d, 1H, J = 8.1 Hz, arom. H), 6.32 (s, 1H, 2-H), 4.17, 4.16 (s, s, each 1H, 3-H, 4-H), 2.28 (s, 3H, 4'-CH_3). ^{13}C NMR (75.43 MHz): δ (ppm) = 170.0 (Ar–CO–N), 140.6, 136.8, 136.6, 132.7, 129.3, 129.2*, 128.7, 128.6*, 128.5*, 128.1, 127.9*, 127.2, 125.6, 124.8 (arom. C), 61.7 (4-C), 52.8 (3-C), 51.6 (2-C), 21.4 (4'-CH_3). IR (KBr): 1645 cm^{-1} (ν_{amide}). MS (m/z): 341 (M^+). Anal. calcd. for $C_{23}H_{19}NO_2$: C 80.91, H 5.60, N 4.10. Found C 80.61, H 5.68, N 4.01.

Crystallography of **3b**

Crystal data: $C_{18}H_{17}NO_2$, M_r = 279.34, crystal dimensions: 0.33 × 0.27 × 0.09 mm, monoclinic, space group: Cc (no. 9), a = 14.462(3), b = 6.144(1), c = 16.534(3), β = 97.77(2), V = 1455.6(5) Å^3, Z = 4, D_x = 1.275 g/cm^3.

Intensity data were collected at room temperature with a Philips PW1100 four-circle diffractometer by using Mo–K_α radiation (graphite monochromator). Throughout data collection 1133 independent reflections [806 with $I > 2\sigma(I)$] were obtained up to Θ = 26°. The structure was solved by direct method (MULTAN 78 [7]), and refined by least-squares procedures (SHELX 76 [8]). All non-hydrogen atoms were refined with anisotropic thermal parameters. The two hydrogens of the oxirane ring were refined as independent atoms, the remaining hydrogens were calculated under the presupposition of ideal geometry. The refinement results in the final atom coordinates and thermal parameters, listed in Tables 1 and 2, and the R-values R = 0.076 and R_w = 0.047 [$w = 1/\sigma^2(F)$] [10].

References

[1] Kratzel M., Hiessböck R. (1993) Monatsh. Chem., in press
[2] Günther H. (1983) NMR-Spektroskopie. Thieme, Stuttgart, p. 110.
[3] Kratzel M., Hiessböck R., Synth. Commun., in press
[4] Goldstein S. W., Dambek P. J. (1989) Synthesis: 221
[5] Buckle D. R., Eggleston D. S., Houge-Frydrych C. S.V., Pinto I. L., Readshaw S. A., Smith D. G., Webster R. A. B. (1991) J. Chem. Soc. Perkin Trans. I: 2763
[6] Kratzel M., Hiessböck R., Heterocycles, accepted
[7] Main P. (1978) MULTAN 78, System of Computer Programs for Automatic Solution of Crystal Structures from X-ray Diffraction Data. University of York, Great Britain
[8] Sheldrick G. M. (1976) SHELX 76, Program for Crystal Structure Determination. University of Cambridge, Great Britain
[9] Kratzel M. (1988) CHEM-CAD and ALCHPLOT, PC-Programs for 2D-Plots of Chemical Structures and 3D-Plots from Crystal Structures and ALCHEMY Data. Universität Wien, Österreich
[10] Additional material to the structure determination can be ordered from Fachinformationszentrum Energie-Physik-Mathematik, D-76344 Eggenstein-Leopoldshafen 2, Federal Republic of Germany, referring to the deposition no. CSD-57794, the names of the authors and the citation of the paper

Received November 3, 1993. Accepted December 18, 1993