Tetrahedron Vol. 49, No. 10, pp. 2115-2122, 1993
Printed in Great Britain

0040-4020/93 $6.00+.00

Substituent Effects on the Ring-chain Tautomerism of 1,3-Oxazines

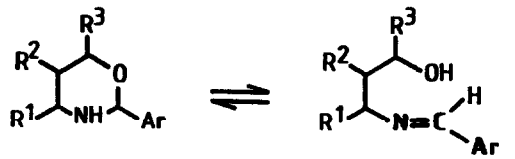
Ferenc Fülöp,[a,b*] László Lázár,[a] Gábor Bernáth[a], Reijo Sillanpää[b] and Kalevi Pihlaja[b*]

[a]Institute of Pharmaceutical Chemistry, Albert Szent-Györgyi Medical University, H-6701 Szeged, POB 121, Hungary;
[b]Department of Chemistry, University of Turku, SF-20500 Turku, Finland

(*Received in UK* 10 *November* 1992)

Abstract: *The aryl substituents at positions 4 and 6 do not exert observable electronic effects on the ring-chain tautomeric ratios of 2,4- or 2,6-diarylsubstituted-tetrahydro-1,3-oxazines. On the other hand, the electronic effect of the 2-aryl substituent is marked: in all eight series studied, the equilibria can be described by the equation* $\log K = \rho\sigma^+ + \log K_0$, *where* σ^+ *is the Hammett-Brown constant of the 2-aryl substituents, and* ρ *is a constant characteristic of the ring system (=0.75±0.05).*

The syntheses and derivatizations of 1,3-aminoalcohols are of current interest from both pharmacological and chemical respects.[1,2] From 1,3-aminoalcohols, tautomeric mixtures of 1,3-oxazines and the corresponding open-chain Schiff bases can be obtained with oxo compounds.[3] The tautomeric character of the products can be utilized in different transformations, *e.g.* the development of aminoalcohol prodrugs,[4] *N*-substitution methods[5,6] and enantioselective syntheses.[7,8] Because of their tautomeric character, the mixtures can be used as aldehyde or ketone sources.[9,10] As a result of a comparative study of differently substituted 2-aryltetrahydro-1,3-oxazines, we recently reported[11] that the ring-chain tautomeric process (**1A**⇌**1B**) can be described by equation (1):



1A **1B**

$$\log K = \rho\sigma^+ + \log K_0 \qquad (1)$$

where K=[ring]/[chain] and σ^+ is the Hammett-Brown constant of the 2-aryl substituents. It was also proved that ρ is a constant characteristic of the ring system. The ρ value depends on the nature of the solvent and the temperature.[11,12] For 1,3-oxazines in $CDCl_3$ solution at room temperature, it is 0.76±0.04. With the aid of equation (1) and the known ρ values, constant σ^+ has been determined for 3- and 4-pyridyl substituents.[13]

Since data were available only for the effects of 2-aryl substituents on the tautomerism of 1,3-oxazines, our present aim was to study the effects of different aromatic substituents at positions 4 (beside the nitrogen) and 6 (beside the oxygen).

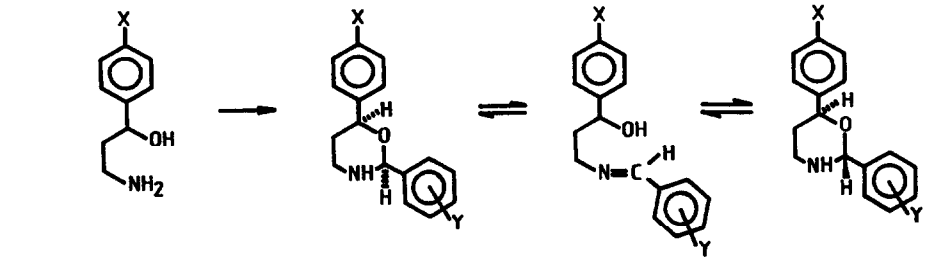
Dedicated to Professor **Jenő Kóbor** (Chemical Department, Pedagogical Training College, Szeged, Hungary) on the occasion of his 70th birthday.

Results and discussion

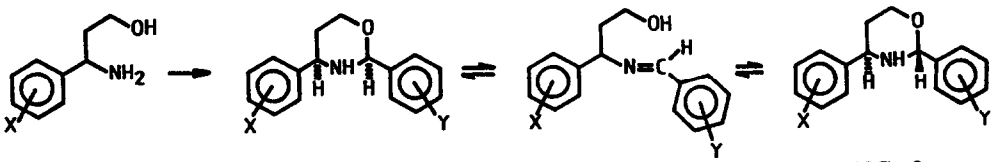
The 3-amino-1-arylpropanols **2** and **3** were prepared by Claisen condensation of the corresponding ethyl benzoates with acetonitrile, followed by lithium aluminium hydride reduction.[14,15] 3-Amino-1-(*p*-methoxyphenyl)-propanol **4** was prepared by Mannich reaction of *p*-methoxyacetophenone and benzylamine,[16] followed by sodium borohydride reduction and catalytic debenzylation.

The 3-amino-3-arylpropanols **5-9** were prepared by lithium aluminium hydride reduction of the β-aminoacids obtained from substituted benzaldehydes via malonic acid/ammonium acetate treatment.[17]

Condensations of 1,3-aminoalcohols **2-9** with six differently substituted aromatic aldehydes took place in nearly quantitative yield at room temperature in ethanol. In $CDCl_3$ solution, all of the products participate in a three-component tautomeric mixture (Scheme 1) involving two ring and one open-chain form.



X=H:	**2**	**10Aa-f**	**10Ba-f**	**10Ca-f**
Me:	**3**	**11Aa-f**	**11Ba-f**	**11Ca-f**
OMe:	**4**	**12Aa-f**	**12Ba-f**	**12Ca-f**



X=*m*Cl:	**5**	**13Aa-f**	**13Ba-f**	**13Ca-f**
*p*Cl:	**6**	**14Aa-f**	**14Ba-f**	**14Ca-f**
H:	**7**	**15Aa-f**	**15Ba-f**	**15Ca-f**
*p*Me:	**8**	**16Aa-f**	**16Ba-f**	**16Ca-f**
*p*OMe:	**9**	**17Aa-f**	**17Ba-f**	**17Ca-f**

Y=*p*NO_2: **a**; *m*Cl: **b**; H: **c**; *p*Me: **d**; *p*OMe: **e**; *p*NMe_2: **f**

Scheme 1

The tautomeric ratios were determined by integration of the corresponding well-separated lines: mainly those of the O-C*H*(Ar)-N (ring) and N=C*H* (chain) singlets in the regions 5.3-5.6 and 8.2-8.4 ppm, respectively. The chemical shifts of the above protons and the sums of the ring percentages are given in Tables 2 and 3. The different 2-aryl substituents have only slight effects on the above chemical shifts, but practically no effect was observed for 4- and 6-aryl substituents. As expected, in both series

one of the ring forms, **A**, having the two aryl substituents *equatorially* oriented, predominates, while the content of the minor ring form **C** is constantly only about 2-4% of that of the major ring form. The predominance of the chair conformations for differently substituted 1,3-oxazines was established earlier (see *e.g.* lit. 18-20).

In the tautomeric mixture, the major ring form has an almost perfect chair conformation. The structure of **10a** was determined in the solid state by means of X-ray diffraction. The ORTEP perspective view is shown in Fig. 1. The solid-state result corresponds to that of the earlier solid-state NMR study, where it was established that the chain form is preferred in the solid state for those cases where less than 80% of the ring form is found in $CDCl_3$ solution.[21]

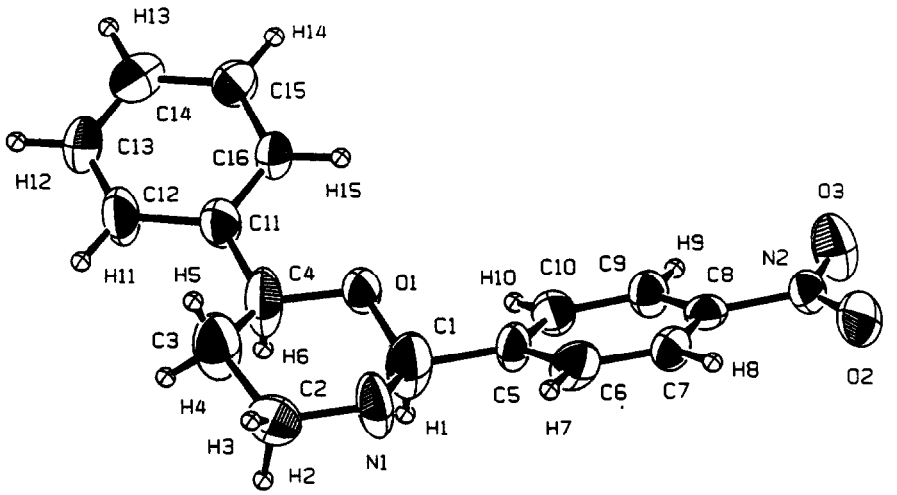


Figure 1. ORTEP perspective diagram of compound **10a**

The data of linear regression analysis according to equation (1), which shows the effect of 2-aryl substituents, are given in Table 1. The slopes and plots correspond practically within experimental error to those determined earlier for other 1,3-oxazine derivatives. To analyse the effects of aromatic substituents at position 4 or 6, the ring-chain data on the parent 2-aryl-substituted-1,3-oxazines, taken from the literature, are also given in Table 1. As Table 1 shows, the contributions to the ring-chain tautomerism of the different aryl substituents in either position 6 (compounds **10-12**) or position 4 (compounds **13-17**) are seen to be practically constant.

Table 1. Linear regression analysis data on compounds **10-17**

	10	**11**	**12**	**13**	**14**	**15**	**16**	**17**	*
no. of points	6	6	6	6	6	6	6	6	7
slope	0.86	0.75	0.82	0.69	0.70	0.71	0.70	0.74	0.74
intercept	-0.04	-0.01	0.02	0.41	0.40	0.43	0.41	0.39	-0.15
SE of the slope[a]	±0.04	±0.03	±0.03	±0.04	±0.03	±0.05	±0.03	±0.03	±0.06
SE of the intercept[a]	±0.03	±0.03	±0.02	±0.03	±0.03	±0.04	±0.03	±0.02	±0.05
correlation coefficient	0.996	0.996	0.998	0.994	0.995	0.992	0.996	0.997	0.998
c (see text)	0.11	0.14	0.17	0.56	0.55	0.58	0.56	0.54	

*Data on the parent 2-aryltetrahydro-1,3-oxazines.[11] [a]SE = standard error.

The tautomerism of the products of *N*-(2-hydroxyalkyl)hydrazones with oxo compounds was studied earlier, where the equilibrium mixture contained 1,3,4-oxadiazine and the corresponding open-chain hydrazone. In these compounds, a clear tendency was found as concerns the different aromatic substituents on N-4 (which corresponds to C-4 in 1,3-oxazines) and the ring-chain tautomeric mixture: electron-withdrawing substituents increased the percentage of the ring form, while electron-donating substituents increased the percentage of the open-chain form.[3,22,23]

From this comparison, it can be concluded that in the 1,3,4-oxadiazine ring system the electronic character of the N-4 substituent plays an important role in the tautomeric process, while in the 1,3-oxazines the electronic character of the C-4 and C-6 substituents is not of importance as concerns the tautomeric ratios. The difference can be explained by the fact that in the 1,3,4-oxadiazines the hydrazine moiety has a common electron system, while in the 1,3-oxazines the C-4 and C-6 substituents are isolated electronically from *N*-3.

The sum of the steric and electronic effects (c) of the substituents at positions 4, 5 and 6 was earlier introduced and defined as the difference of the intercepts of the substituted derivatives and the unsubstituted parent compound (-0.15).[11] Accordingly, the steric and electronic contribution of the aryl substituent at position 6 is about 0.15, while that of the substituent at position 4 is about 0.55 (Table 1). This means that aryl substituents at both positions increase the contribution of the ring forms; the effect is relatively low for 6-substituted derivatives, but stronger for 4-substituted-1,3-oxazines.

When (*R*)-phenylglycinol was reacted with benzaldehyde or *p*-anisaldehyde, the product proved to be an 83:17 or 93:7 mixture of the corresponding Schiff base and 2-aryl-4-phenyl-oxazolidine, respectively.[2] As compared to the parent 2-arylsubstituted-oxazolidines, the difference in log K is 0.41 for the 2-phenyl and 0.40 for the 2-(4-methoxyphenyl) derivative. This means that the contribution (c) of the 4-phenyl group is very similar for oxazolidines and 1,3-oxazines.

EXPERIMENTAL

^{1}H NMR spectra were recorded on a JEOL GX 400 MHz FT spectrometer in $CDCl_3$ solution at ambient temperature, using TMS as internal standard.

Melting points were determined on a Kofler micro melting point apparatus and are not corrected.

3-Amino-1-phenyl-1-propanol (**2**), 3-amino-1-(*p*-tolyl)-1-propanol (**3**) and 3-amino-3-aryl-1-propanols (**2-9**) were prepared by known procedures.[14,15,17]

3-Benzylamino-1-(p-methoxyphenyl)-1-propanol

15.29 g (0.05 mol) *N*-Benzyl-2-(*p*-methoxybenzoyl)-ethylamine[16] was dissolved in 200 ml methanol, and 7.57 g (0.2 mol) $NaBH_4$ was added in small portions to the stirred and ice-cooled solution. After addition, the mixture was stirred at ambient temperature for 3 hrs and then evaporated. The residue was dissolved in water (200 ml), acidified with HCl, then made alkaline with 10% NaOH and extracted with chloroform (3x100 ml). After drying (Na_2SO_4) and evaporation the oily residue crystallized on treatment with diethyl ether. Yield 11.2 g (83 %), mp 73-75 °C (diisopropyl ether). The compound was used for the further experiment without purification.

Table 2. Physical data on compounds **10-13**

Compound	Mp (°C) Solvent[b]	Formula	Ring[a] (%)	δ N-C*H*-O (s) ring[c]	ring[d]	δ N=C*H*- (s) chain
10a	94-96 (D)	$C_{16}H_{16}N_2O_3$	82	5.43	5.70	8.28
10b	oil	$C_{16}H_{16}ClNO$	69	5.33	5.52	8.19
10c	oil	$C_{16}H_{17}NO$	41	5.38	5.67	8.25
10d	oil	$C_{17}H_{19}NO$	33	5.36	5.60	8.22
10e	oil	$C_{17}H_{19}NO_2$	19	5.33	5.63	8.17
10f	91-92 (H)	$C_{18}H_{22}N_2O$	2.9	5.30	5.61	8.08
11a	114-116 (D)	$C_{17}H_{18}N_2O_3$	79	5.42	5.70	8.34
11b	72-73 (H)	$C_{17}H_{18}ClNO$	67	5.33	5.68	8.20
11c	oil	$C_{17}H_{19}NO$	49	5.39	5.70	8.27
11d	64-65 (H)	$C_{18}H_{21}NO$	32	5.34	5.61	8.22
11e	77-78 (H)	$C_{18}H_{21}NO_2$	24	5.31	5.65	8.16
11f	oil	$C_{19}H_{24}N_2O$	4.5	5.28	5.63	8.10
12a	87-88 (H)	$C_{17}H_{18}N_2O_4$	83	5.42	5.72	8.35
12b	oil	$C_{17}H_{18}ClNO_2$	71	5.32	5.63	8.19
12c	oil	$C_{17}H_{19}NO_2$	46	5.36	5.64	8.25
12d	69-70 (H-E)	$C_{18}H_{21}NO_2$	37	5.33	5.66	8.20
12e	68-69 (H-E)	$C_{18}H_{21}NO_3$	21	5.31	5.60	8.16
12f	oil	$C_{19}H_{24}N_2O_2$	4.1	5.30	5.67	8.08

[a]The sum of the major and minor ring forms. [b]Recrystallization solvents: D: diisopropyl ether; H: *n*-hexane; H-E: *n*-hexane - EtOH. [c]Major ring form. [d]Minor ring form.

3-Amino-1-(p-methoxyphenyl)-1-propanol (**4**)

A solution of 10.85 g (0.04 mol) 3-benzylamino-1-(*p*-methoxyphenyl)-1-propanol in 200 ml methanol was hydrogenated at ambient temperature and normal pressure in the presence of 3.0 g 10% Pd/C catalyst. When the hydrogen uptake had finished (1 hrs), the catalyst was filtered off and the solution was evaporated. The oily residue was crystallized on treatment with diethyl ether. Yield 6.3 g (82 %), mp 123-125 °C
(ethyl acetate). Formula (mw) $C_{11}H_{15}NO_2$ (193.24), analysis (calcd/found) C 68.37/68.53, H 7.82/7.61, N 7.25/7.40%.

General procedure for the preparation of 2,4- or 2,6-diarylsubstituted-3,4,5,6-tetrahydro-2H-1,3-oxazines **10a-f - 17a-f**

Aminoalcohol **2-9** (5 mmol) and the corresponding substituted benzaldehyde (5 mmol) were dissolved in 10 ml ethanol and left to stand for 30 min. The solvent was then evaporated off and the products were crystallized. If an oily product was formed, the evaporation was repeated after the addition of benzene. Products were dried in a vacuum desiccator, and analysed by ^1H NMR spectroscopy.

Table 3. Physical data on compounds **13-17**

Compound	Mp (°C) Solvent[b]	Formula	Ring[a] (%)	δ N-C*H*-O (s) ring[c]	δ N-C*H*-O (s) ring[d]	δ N=C*H*- (s) chain
13a	oil	$C_{16}H_{15}ClN_2O_3$	91	5.38	5.54	8.41
13b	oil	$C_{16}H_{15}Cl_2NO$	84	5.27	5.46	8.25
13c	oil	$C_{16}H_{16}ClNO$	72	5.30	5.51	8.28
13d	oil	$C_{17}H_{18}ClNO$	56	5.25	5.49	8.23
13e	oil	$C_{17}H_{18}ClNO_2$	38	5.26	5.49	8.21
13f	oil	$C_{18}H_{21}ClN_2O$	17	5.23	5.50	8.11
14a	93-94 (H)	$C_{16}H_{15}N_2O_3$	91	5.38	5.53	8.40
14b	oil	$C_{16}H_{15}Cl_2NO$	84	5.25	5.43	8.22
14c	oil	$C_{16}H_{16}ClNO$	68	5.23	5.41	8.25
14d	oil	$C_{17}H_{18}ClNO$	56	5.21	5.40	8.26
14e	oil	$C_{17}H_{18}ClNO_2$	44	5.26	5.47	8.19
14f	oil	$C_{18}H_{21}ClN_2O$	15	5.23	5.48	8.10
15a	90-91 (H)	$C_{16}H_{16}N_2O_3$	92	5.40	5.60	8.39
15b	oil	$C_{16}H_{16}ClNO$	84	5.30	5.52	8.24
15c	oil	$C_{16}H_{17}NO$	67	5.32	5.56	8.26
15d	oil	$C_{17}H_{19}NO$	59	5.28	5.54	8.21
15e	oil	$C_{17}H_{19}NO_2$	49	5.29	5.54	8.19
15f	oil	$C_{18}H_{22}N_2O$	14	5.22	5.51	8.06
16a	108-110 (H)	$C_{17}H_{18}N_2O_3$	91	5.39	5.39	8.37
16b	oil	$C_{17}H_{18}ClNO$	84	5.29	5.52	8.21
16c	oil	$C_{17}H_{19}NO$	72	5.32	5.56	8.24
16d	oil	$C_{18}H_{21}NO$	58	5.27	5.55	8.19
16e	oil	$C_{18}H_{21}NO_2$	38	5.25	5.51	8.15
16f	oil	$C_{19}H_{24}N_2O$	16	5.23	5.54	8.07
17a	84-86 (H)	$C_{17}H_{18}N_2O_4$	91	5.39	5.38	5.35
17b	oil	$C_{17}H_{18}ClNO_2$	83	5.28	5.51	8.21
17c	oil	$C_{17}H_{19}NO_2$	70	5.52	5.52	8.21
17d	oil	$C_{18}H_{21}NO_2$	55	5.27	5.52	8.19
17e	oil	$C_{18}H_{21}NO_3$	44	5.25	5.52	8.15
17f	oil	$C_{19}H_{24}N_2O_2$	12	5.24	5.52	8.07

[a]The sum of the major and minor ring forms. [b]Recrystallization solvent: H: *n*-hexane; [c]Major ring form. [d]Minor ring form.

X-Ray Crystallographic Data

Crystal data for **10a**: $C_{16}H_{16}N_2O_3$, M_r=284.31, triclinic, space group P1, a=8.715(2), b=12.599(2), c=7.188(3) Å, α=95.23(2) β=112.37(2), γ=77.23(2), Z=2, V=711.7(7) Å^3, D_c=1.327 g/cm^3, μ(moK_α)=0.87 cm^{-1}, F(000)=300, T=296(1) K, colourless prisms, crystal dimensions 0.15x0.20x0.35 mm.

Table 4. Positional parameters[a] and B(eq) for compound **10a**

Atom	x	y	z	B(eq)
O(1)	0.6400(7)	0.1351(4)	0.4643(7)	6.0(3)
O(2)	0.0815(8)	0.6222(5)	-0.001(1)	8.2(4)
O(3)	0.1967(8)	0.5357(5)	-0.202(1)	8.7(4)
N(1)	0.597(1)	0.2389(6)	0.734(1)	10.2(6)
N(2)	0.1839(9)	0.5484(6)	-0.038(1)	5.5(4)
C(1)	0.602(2)	0.2332(8)	0.571(2)	12.6(8)
C(2)	0.712(1)	0.1538(8)	0.875(1)	7.7(9)
C(3)	0.760(2)	0.0471(9)	0.773(2)	10.9(8)
C(4)	0.756(2)	0.0498(9)	0.596(2)	14.1(9)
C(5)	0.492(1)	0.3167(6)	0.411(1)	6.2(5)
C(6)	0.363(1)	0.3947(7)	0.439(1)	6.6(5)
C(7)	0.264(1)	0.4716(6)	0.296(1)	5.8(5)
C(8)	0.294(1)	0.4694(6)	0.122(1)	4.3(4)
C(9)	0.423(1)	0.3949(7)	0.090(1)	5.3(5)
C(10)	0.521(1)	0.3196(6)	0.236(1)	6.0(5)
C(11)	0.789(1)	-0.0497(7)	0.474(1)	6.9(5)
C(12)	0.892(1)	-0.1445(9)	0.570(1)	10.3(7)
C(13)	0.922(1)	-0.2372(8)	0.460(2)	8.2(6)
C(14)	0.854(1)	-0.2352(7)	0.256(2)	6.5(5)
C(15)	0.753(1)	-0.1418(7)	0.160(1)	5.6(5)
C(16)	0.720(1)	-0.0499(6)	0.268(1)	5.2(4)
H(1)	0.7068	0.2561	0.6047	14.8
H(2)	0.8120	0.1795	0.9541	9.2
H(3)	0.6577	0.1395	0.9596	9.2
H(4)	0.8759	0.0166	0.8545	13.1
H(5)	0.6900	0.0010	0.7741	13.1
H(6)	0.8573	0.0743	0.6232	16.4
H(7)	0.3431	0.3948	0.5598	7.9
H(8)	0.1772	0.5253	0.3174	7.0
H(9)	0.4433	0.3957	-0.0301	6.4
H(10)	0.6111	0.2681	0.2158	7.2
H(11)	0.9421	-0.1461	0.7129	12.3
H(12)	0.9909	-0.3023	0.5271	9.9
H(13)	0.8772	-0.2982	0.1798	7.8
H(14)	0.7045	-0.1403	0.0175	6.8
H(15)	0.6484	0.0142	0.1989	6.2

[a]For numbering, see Fig.1.

Data Collection, Analysis and Refinement. A Rigaku AFC5S diffractometer, graphite-monochromated MoK$_\alpha$, λ =0.71069 Å, ω-2θ scan mode with an ω scan rate of 8.0° min^{-1} and a scan width of (1.42+0.30tanθ), weak reflections were [F< 10 σ(F)] were rescanned up to two times, 2516 unique reflections measured, $2\theta_{max}$=50°. The data were corrected for Lorentz and polarization effects. The structure was solved by direct methods and refined with the 980 data [I >3 σ(I)] to an R value of 0.079 (R_w=0.097, sigma weights); non-hydrogen atoms anisotropic, hydrogen atoms in calculated positions with fixed isotropic temperature factors. The maximum/minimum residual electron density was 0.36/-0.30e/Å^3. All calculations were performed with TEXSAN-89 software[24] using a VAXSTATION 3520 computer. Figures were drawn with ORTEP[25]. The final atomic positional coordinates, temperature parameters, bond lengths and angles have been deposited with the Cambridge Crystallographic Data Centre, Lensfield Rd., Cambridge, CB2 1EW, UK.

Acknowledgements. The authors wish to express their gratitude to the National Scientific Research Grant, Hungary (T4466), and to the Research Council for Natural Sciences, the Academy of Finland, for financial support.

REFERENCES

1. Alberola, A.; Alvarez, A. M.; Andrés, C.; Gonzáles, A.; Pedrosa, R. *Synthesis* **1990**. 153.
2. Higashama, H.; Inone, H.; Takahashi, H. *Tetrahedron Letters* **1992**, *33*, 235.
3. Haack, T.; Mutter, M. *Tetrahedron Letters* **1992**, *33*, 1589.
4. Johansen, M.; Bundgaard, H. *J. Pharm. Sci.* **1983**, *72*, 1294.
5. Saavedra, J. E. *J. Org. Chem.* **1985**, *50*, 2271.
6. Fülöp, F.; Huber, I.; Bernáth, G.; Hönig, H.; Seufer-Wasserthal, P. *Synthesis* **1991**, 43.
7. Alberola, A.; Andrés, C.; Pedrosa, R. *Synlett.* **1990**, 763.
8. Eliel, E. L.; He, X. C. *J. Org. Chem.* **1990**, *55*, 2114.
9. Singh, H.; Sarin, R.; Singh, K. *Heterocycles* **1986**, *24*, 3039.
10. Singh, H.; Singh, K. *Tetrahedron* **1989**, *45*, 3967; *Ind. J. Chem.* **1989**, *28B*, 802.
11. Fülöp, F.; Pihlaja, K.; Mattinen, J.; Bernáth, G. *J. Org. Chem.* **1987**, *52*, 3281; *Tetrahedron* **1987**, *43*, 1863.
12. Parkkinen, A.; Fülöp, F.; Pihlaja, K. *Acta Chem. Scand.* **1990**, *44*, 364; *Tetrahedron* **1991**, *47*, 2229.
13. Fülöp, F.; Dahlquist, M.; Pihlaja, K. *Acta Chem. Scand.* **1991**, *45*, 273.
14. Long, R. S. *J. Am. Chem. Soc.* **1947**, *69*, 990.
15. Shih, Y. E.; Wang, J. S.; Lin, L. K. *Heterocycles* **1986**, *24*, 1599.
16. Nobles, L. W.; Burckhalter, J. H. *J. Am. Pharm. Ass. Ed.* **1958**, *47*, 77.
17. Shih, Y. E.; Wang. J. S.; Chen, C. T. *Heterocycles* **1978**, *9*, 1277.
18. Katritzky, A. R.; Baker, V. J.; Brito-Palma, F. M. S. *J. Chem. Soc. Perkin Trans 2.* **1980**, 1739.
19. Pihlaja, K.; Nurmi, T.; Kettunen, M. L. Private communication.
20. Samitov, Yu. Yu.; Danilova, O. I.; Unkovsky, B. B.; Boiko, I. P. *Magn. Reson. Chem.* **1986**, *24*, 480.
21. Riddell, F. G.; Arumugam, S.; Fülöp, F.; Bernáth, G. *Tetrahedron* **1992**, *48*, 4979.
22. Milman, B. L.; Potekhin, A. A. *Khim. Get. Soed.* **1973**, 920.
23. Potekhin, A. A.; Safronov, A. O. *Zh. Org. Khim.* **1981**, *17*, 379.
24. *TEXSAN: Single Crystal Structure Analysis Software*, Version 5.0, Molecular Structure Corporation, The Woodlands, Texas, 1989.
25. Johnson, C. K. "*ORTEP-II, A Fortran Thermal-ellipsoid Plot Program for Crystal Structure Illustrations*. Report ORNAL-5138", Oak Ridge National Laboratory, Tenn., 1976.