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INVESTIGATION OF THE STEREODIRECTION OF THE RITTER REACTION. CRYSTALLINE AND MOLECULAR STRUCTURES OF N-(2-PHENETHYL)-2,5-DIMETHYL-4-PHENYLPIPERID-4-OL AND N-(2-PHENETHYL)-2,5-DIMETHYL-4-PHENYL-4-ACETAMIDOPIPERIDINE

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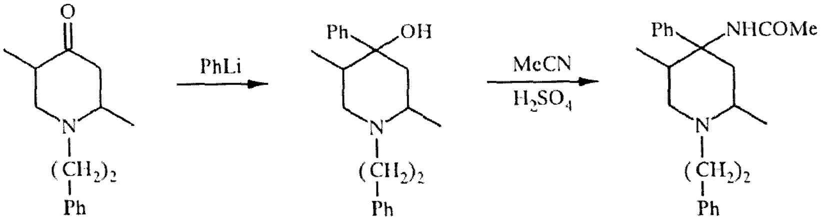
The stereodirection of the Ritter reaction of N-(2-phenethyl)-2,5-dimethyl-4-phenylpiperid-4-ol to the corresponding amide has been studied. X-ray analysis has shown that the reaction occurs with retention of the configuration about atom C_4 of the piperidine ring.

In continuation of a study of the application of the Ritter reaction to a series of piperidine derivatives [1] we have investigated the stereodirection of the reaction of N-(2-phenethyl)-2,5-dimethyl-4-phenylpiperid-4-ol to the corresponding amide.

Synthesis of N-(2-phenethyl-2,5-dimethyl-4-phenyl-4-acetamidopiperidine (III) was carried out according to the following scheme:



I II III

Treatment of N-(2-phenethyl)-2,5-dimethylpiperid-4-one (I) with phenyllithium gave the tertiary alcohol (II) which was readily converted using acetonitrile in concentrated sulfuric acid to give (III). According to chromatographic data II and III were obtained as a single isomer. The effect of N-substitution on the stereochemistry of the phenyllithium synthesis has been discussed before [2, 3]. From this work it could be supposed that alcohol II would be obtained as one isomer with an axial orientation of the OH group. This was confirmed by our X-ray structural investigation which showed that both the 4-hydroxy and the 4-acetamido groups in II and III had an axial orientation.

The structure of the individual parts of II and cation III with bond lengths are given in Figs. 1 and 2 and valence angles for II and III in Tables 1 and 2.

The notable difference in the corresponding geometrical parameters of II and III and their deviation from standard values [4] arose from the moderate quality of the monocrystals obtained and does not merit further discussion. X-ray analysis clearly proved structures II and III, i.e. resolving the problem in hand.

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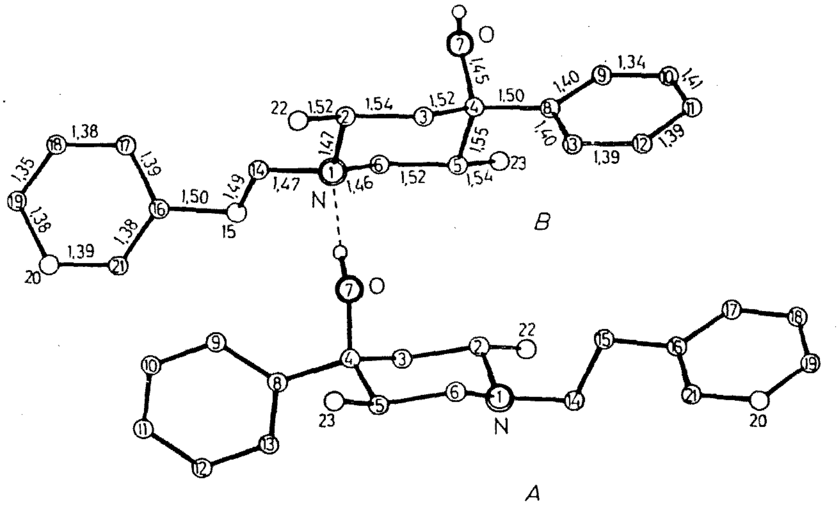

Fig. 1. Structure of the individual components of crystal II with bond lengths ($\sigma = 0.01$ Å).

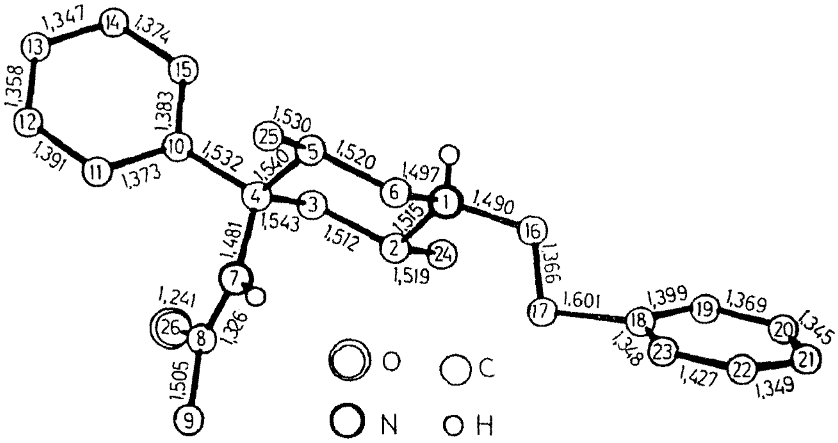

Fig. 2. Structure of cation II with bond lengths ($\sigma = 0.005$ to 0.007 Å).

In structure II the configurations of the chiral centers of the central piperidine ring of both individual molecules A and B are the same. The piperidine heterocycle has a chair conformation in A and B. There is a displacement of atoms N_1 and C_4 from the mean square plane of atoms C_2, C_3, C_5, and C_6 of -0.677 and 0.585 Å in molecule A and -0.618 and 0.692 Å in B. In both molecules, all of the substituents at positions 1, 2, 4, and 5 except the hydroxyl group occupy an equatorial position. The displacements of C_{14}, C_{22}, C_8, and C_{23} from the mean plane of the piperidine ring are 0.095, -0.134, -0.574, and 0.329 Å in molecule A and 0.333, -0.176, -0.281, and 0.287 Å in B respectively. The displacements of the hydroxyl O_7 atom from the plane of the ring in A and B are 1.588 and 1.666 Å showing that the OH group occupies an axial position in both individual crystalline molecules.

In structure II the conformations of the individual molecules A and B are somewhat different. Thus the dihedral angle between the mean plane of the piperidine ring and the 4-phenyl ring in A is 86.8° in A but $-59.6°$ in B. Even greater differences are seen in the dihedral angles between the mean piperidine plane and the Ph ring of the 1-phenethyl substituent. In A this is 26.7° whereas it is $-104.7°$ in B. Differences are also seen in the conformation of the 1-phenethyl substituent relative to the piperidine ring. In both molecules this substituent has an extended conformation (torsional angles for $N_1C_{14}-C_{15}H_{16}$ being 173(1) and 178(1)° in A and B. However, its orientation relative to the piperidine ring differs (torsional angles for $C_2N_1-C_{14}C_{15}$ in A and B being $-64(1)$ and 168(1)° respectively).

TABLE 1. Valence Angles τ (deg.) in Structure II

Angle	τ (σ)	
	molecule A	molecule B
$C_{(2)}-N_{(1)}-C_{(6)}$	111,1(4)	109,9(5)
$C_{(2)}-N_{(1)}-C_{(14)}$	116,9(5)	110,0(5)
$C_{(6)}-N_{(1)}-C_{(14)}$	110,1(5)	110,2(6)
$N_{(1)}-C_{(2)}-C_{(3)}$	108,3(5)	109,8(5)
$N_{(1)}-C_{(2)}-C_{(22)}$	112,2(5)	112,4(6)
$C_{(3)}-C_{(2)}-C_{(22)}$	110,3(5)	110,7(6)
$C_{(2)}-C_{(3)}-C_{(4)}$	114,9(5)	114,8(5)
$C_{(3)}-C_{(4)}-C_{(5)}$	110,0(5)	106,0(5)
$C_{(3)}-C_{(4)}-O_{(7)}$	107,1(5)	110,2(5)
$C_{(3)}-C_{(4)}-C_{(8)}$	113,9(6)	114,2(6)
$C_{(5)}-C_{(4)}-O_{(7)}$	105,2(5)	105,3(5)
$C_{(5)}-C_{(4)}-C_{(8)}$	109,1(5)	112,2(5)
$O_{(7)}-C_{(4)}-C_{(8)}$	111,3(5)	108,7(5)
$C_{(4)}-C_{(5)}-C_{(6)}$	112,0(5)	111,0(5)
$C_{(4)}-C_{(5)}-C_{(23)}$	112,7(6)	114,4(5)
$C_{(6)}-C_{(5)}-C_{(23)}$	112,2(6)	107,4(6)
$N_{(1)}-C_{(6)}-C_{(5)}$	113,0(5)	113,4(6)
$C_{(4)}-C_{(8)}-C_{(9)}$	123,0(6)	122,8(6)
$C_{(4)}-C_{(8)}-C_{(13)}$	120,2(6)	121,1(6)
$C_{(9)}-C_{(8)}-C_{(13)}$	116,9(7)	116,1(7)
$C_{(8)}-C_{(9)}-C_{(10)}$	122,2(8)	123,6(8)
$C_{(9)}-C_{(10)}-C_{(11)}$	119,4(9)	121,6(9)
$C_{(10)}-C_{(11)}-C_{(12)}$	119,9(9)	116,7(9)
$C_{(11)}-C_{(12)}-C_{(13)}$	120,1(9)	120,2(8)
$C_{(8)}-C_{(13)}-C_{(12)}$	121,3(8)	121,6(7)
$N_{(1)}-C_{(14)}-C_{(15)}$	116,9(6)	118,4(7)
$C_{(14)}-C_{(15)}-C_{(16)}$	115,6(7)	114,0(8)
$C_{(15)}-C_{(16)}-C_{(17)}$	123,6(8)	120,9(8)
$C_{(15)}-C_{(16)}-C_{(21)}$	120,1(8)	121,5(9)
$C_{(17)}-C_{(16)}-C_{(21)}$	116,3(8)	117,6(9)
$C_{(16)}-C_{(17)}-C_{(18)}$	122,3(9)	119,4(9)
$C_{(17)}-C_{(18)}-C_{(19)}$	120,5(9)	123(1)
$C_{(18)}-C_{(19)}-C_{(20)}$	120,6(9)	117(1)
$C_{(19)}-C_{(20)}-C_{(21)}$	119,8(9)	120(1)
$C_{(16)}-C_{(21)}-C_{(20)}$	120,5(8)	123(1)

In crystalline II the active H atoms of both molecules take part in intermolecular hydrogen bonding. II forms a chain along [001] through formation of the bonds $O_{7A}-H\cdots N_{1B}$($O\cdots N$ 2.993(7), O−H 1.07, $H\cdots O$ 1.94 Å, $O-H\cdots N$ 167°) and $O_{7B}-H\cdots N_{1A}$ (0.5 − x, y, −0.5 + z) ($O\cdots N$ 2.886(6), O−H 0.82, $H\cdots N$ 2.12 Å, $O-H\cdots N$ 155°).

In structure III the piperidine heterocycle also has a chair conformation (the displacement of N_1 and C_4 from the mean plane of C_2, C_3, C_5, and C_6 being 0.665 and 0.700 Å. The displacements of C_{16}, C_{24}, C_{10}, N_7, and C_{25} from the mean plane are −0.124, 0.236, 0.425, −1.697, and −0.151 Å respectively, i.e. the 1-phenethyl, 2-Me, 4-Ph, and 5-Me occupy equatorial positions and the 4-acetyl substituent an axial position relative to the piperidine ring.

In the cation III the orientation of 4-Ph and the Ph group of the 1-phenethyl substituent relative to the mean piperidine plane is close to that seen in molecule A for structure II. The dihedral angles between the mean planes of the phenyl rings in position 4 and in the 1-phenethyl substituent and the mean plane of the pyridine ring are 83 and 27°. The extended conformation of the 1 phenethyl substituent (torsional angle $N_1C_{16}-C_{17}C_{18}$ −173°) and its orientation with respect to the piperidine ring are close to those found in molecule A of structure II (torsional angle $C_2N_1-C_{16}C_{17}$ −55°).

In crystal III all three active H atoms take part in formation of intermolecular hydrogen bonds. Cation−anion pairs are formed through bonding $O_{S2}-H_S\cdots O_{26}$ ($O\cdots O\cdots O$ 2.560(4), O−H 0.83(4), $H\cdots O$ 1.74(4) Å, $O-H\cdots O$ 176(4)°) which form a lattice parallel to (100) through the bonds $N_1-H_1\cdots O_{S3}$ (x, 1 + y, z) ($N\cdots O$ 2.736(4), N−H 0.84(4), $H\cdots O$ 1.90(4) Å, $N-H\cdots O$ 172(3)° and $N_7-H_7\cdots O_{S1}$ (x, 1-y, 0.5 + z) ($N\cdots O$ 2.94)(4), N−H 0.87(3), $H\cdots O$ 2.08(3) Å, $N-H\cdots O$ 170(3)°).

TABLE 2. Valence Angles τ (deg.) in Structure III

Angle	τ (σ)	Angle	τ (σ)
$O_{(S1)}-S-O_{(S2)}$	106,1(2)	$C_{(4)}-N_{(7)}-C_{(8)}$	125,4(3)
$O_{(S1)}-S-O_{(S3)}$	111,8(2)	$N_{(7)}-C_{(8)}-C_{(9)}$	116,5(3)
$O_{(S1)}-S-O_{(S4)}$	112,1(2)	$N_{(7)}-C_{(8)}-O_{(26)}$	122,0(3)
$O_{(S2)}-S-O_{(S3)}$	102,7(2)	$C_{(9)}-C_{(8)}-O_{(26)}$	121,5(3)
$O_{(S2)}-S-O_{(S4)}$	107,9(2)	$C_{(4)}-C_{(10)}-C_{(11)}$	122,8(3)
$O_{(S3)}-S-O_{(S4)}$	115,2(2)	$C_{(4)}-C_{(10)}-C_{(15)}$	119,8(3)
$C_{(2)}-N_{(1)}-C_{(6)}$	111,0(3)	$C_{(11)}-C_{(10)}-C_{(15)}$	117,3(3)
$C_{(2)}-N_{(1)}-C_{(16)}$	117,0(3)	$C_{(10)}-C_{(11)}-C_{(12)}$	120,3(4)
$C_{(6)}-N_{(1)}-C_{(16)}$	111,3(3)	$C_{(11)}-C_{(12)}-C_{(13)}$	121,4(5)
$N_{(1)}-C_{(2)}-C_{(3)}$	108,4(3)	$C_{(12)}-C_{(13)}-C_{(14)}$	118,6(5)
$N_{(1)}-C_{(2)}-C_{(24)}$	112,1(3)	$C_{(13)}-C_{(14)}-C_{(15)}$	121,2(5)
$C_{(3)}-C_{(2)}-C_{(24)}$	109,5(3)	$C_{(10)}-C_{(15)}-C_{(14)}$	121,3(4)
$C_{(2)}-C_{(3)}-C_{(4)}$	114,8(3)	$N_{(1)}-C_{(16)}-C_{(17)}$	117,6(5)
$C_{(3)}-C_{(4)}-C_{(5)}$	106,5(3)	$C_{(16)}-C_{(17)}-C_{(18)}$	106,8(5)
$C_{(3)}-C_{(4)}-N_{(7)}$	109,8(3)	$C_{(17)}-C_{(18)}-C_{(19)}$	120,4(6)
$C_{(3)}-C_{(4)}-C_{(10)}$	110,7(3)	$C_{(17)}-C_{(18)}-C_{(23)}$	119,6(6)
$C_{(5)}-C_{(4)}-N_{(7)}$	108,5(3)	$C_{(19)}-C_{(18)}-C_{(23)}$	119,5(7)
$C_{(5)}-C_{(4)}-C_{(10)}$	109,7(3)	$C_{(18)}-C_{(19)}-C_{(20)}$	121,0(6)
$N_{(7)}-C_{(4)}-C_{(10)}$	111,4(3)	$C_{(19)}-C_{(20)}-C_{(21)}$	120,1(5)
$C_{(4)}-C_{(5)}-C_{(6)}$	111,3(3)	$C_{(20)}-C_{(21)}-C_{(22)}$	119,9(5)
$C_{(4)}-C_{(5)}-C_{(25)}$	114,8(3)	$C_{(21)}-C_{(22)}-C_{(23)}$	121,4(6)
$C_{(6)}-C_{(5)}-C_{(25)}$	109,5(3)	$C_{(18)}-C_{(23)}-C_{(22)}$	117,8(7)
$N_{(1)}-C_{(6)}-C_{(5)}$	112,7(3)		

EXPERIMENTAL

TLC was carried out on Polygram Sil G/UV-254 plates using methanol–chloroform (1:5).

N-(2-Phenethyl)-2,5-dimethyl-4-phenylpiperid-4-ol (II). A solution of I (13.8 g, 0.06 mole) in absolute ether (100 ml) was added dropwise with stirring to a solution of phenyllithium which had been prepared by treating bromobenzene (17.0 g, 0.1 mole) with lithium (1.4 g, 0.2 g-atom) in absolute ether (300 ml). The product was stirred for 5 h at 30°C and then basified by addition of aqueous potassium hydroxide. The ether layer was separated, washed with water, and dried with magnesium sulfate. After evaporation of solvent the product was distilled *in vacuo* to give II (12.0 g, 64.2%) with bp 208-210°C/1 mm which crystallized on standing (mp 89-90°C, R_f 0.31).

N-(2-Phenethyl-2,5-dimethyl-4-phenyl-4-acetamidopiperidine (III). Concentrated sulfuric acid (5 ml) was added with stirring to a mixture of II (7.0 g, 0.02 mole) and acetonitrile (5 g, 0.1 mole) so that the temperature did not exceed 60°C. The product was stirred for 16 h at room temperature and poured onto ice (20 g), neutralized with ammonium hydroxide, extracted with chloroform, and dried (magnesium sulfate). Evaporation of solvent gave amide III (4.6 g, 58%) as white crystals with mp 158-159°C and R_f 0.62.

The monocrystals were of low quality but none the less suitable for x-ray investigation since the basic problem was that of determining the molecular stereochemistry and conformation. They were obtained by slow evaporation of an ethanol solution of II (as the base) or III (as the bisulfate).

X-ray analyses of both structures were made on a four circle automatic Hilger-Watts diffractometer (λ MoK_α, graphite monochromator).

Crystals of II are rhombic: a = 18.237(2), b = 11.306(1), c = 18.095(2) Å, V = 3731.1(8) $Å^3$, M = 303.4, d_{calc} = 1.102 g/cm^3, Z = 8 [$C_{21}H_{27}NO$], space group $Pca2_1$, two independent molecules in the overall state.

Crystals of the bisulfate III are monoclinic: a = 40.358(3), b = 9.288(1), c = 13.401(1) Å, β = 108.62(1)°, V = 4760.4(6) $Å^3$, M = 448.6, d_{calc} = 1.251 g/cm^3, Z = 8 [$C_{23}H_{31}NO$]$XHSO_4$, space group C2/c.

Using $\theta/2\theta$ scanning, the intensities of 1580 independent reflections for II ($\theta \leq 26°$) and 3168 for III ($\theta \leq 28°$) were measured. The structures were solved by a direct method using the MULTAN program and refined in a full matrix least squares anisotropic approximation for nonhydrogen atoms. The positions of the hydrogen atoms in II were calculated geometrically with the exception of the HMe and OH groups. In structure II the contribution of the H atoms with fixed posi-

TABLE 3. Coordinates of Non-Hydrogen Atoms ($\times 10^4$; for H atoms of the OH group $\times 10^3$) for Structure II and Equivalent Thermal Factor Parameters, Å^2

Atom	*x/a*	*y/b*	*z/c*	B_{iso}^{eq}
		Molecule A		
N(1)	1951(3)	6747(4)	4219	4,0(2)
C(2)	2299(3)	5933(6)	3696(3)	4,0(2)
C(3)	1890(3)	4747(6)	3729(3)	4,2(2)
C(4)	1068(3)	4826(6)	3595(4)	4,4(2)
C(5)	736(3)	5831(6)	4066(4)	4,2(2)
C(6)	1192(4)	6943(6)	4031(4)	4,7(2)
O(7)	966(2)	5200(4)	2826(2)	5,2(1)
C(8)	666(4)	3709(6)	3751(4)	4,9(2)
C(9)	202(4)	3149(7)	3225(5)	6,4(3)
C(10)	-191(4)	2179(8)	3388(6)	8,5(4)
C(11)	-105(5)	1638(9)	4090(7)	10,3(4)
C(12)	361(5)	2128(8)	4603(6)	8,3(3)
C(13)	748(4)	3145(7)	4435(5)	6,3(3)
C(14)	2322(4)	7886(6)	4347(4)	5,4(2)
C(15)	2382(5)	8702(6)	3704(6)	7,6(3)
C(16)	2677(5)	9901(7)	3871(5)	6,8(3)
C(17)	3315(4)	10328(8)	3586(6)	7,6(3)
C(18)	3571(5)	11445(7)	3746(7)	8,6(3)
C(19)	3190(5)	12165(7)	4202(7)	8,7(4)
C(20)	2561(5)	11810(7)	4498(6)	7,5(3)
C(21)	2295(5)	10657(8)	4351(6)	8,1(3)
C(22)	3108(4)	5753(6)	3861(4)	5,0(2)
C(23)	-71(4)	6064(7)	3886(4)	6,0(2)
		Molecule B		
N(1)	1812(3)	3912(5)	1659(3)	5,0(2)
C(2)	1629(3)	4699(3)	1042(4)	4,5(2)
C(3)	2014(3)	5895(6)	1144(4)	4,3(2)
C(4)	2841(3)	5830(6)	1181(4)	4,5(2)
C(5)	3022(3)	4950(6)	1821(3)	4,4(2)
C(6)	2619(3)	3779(6)	1715(6)	5,1(2)
O(7)	3126(2)	5286(4)	525(2)	4,8(1)
C(8)	3210(4)	7009(6)	1284(4)	4,9(2)
C(9)	3916(4)	7237(8)	1041(5)	6,8(3)
C(10)	4254(5)	8275(8)	1131(6)	8,5(3)
C(11)	3892(5)	9253(7)	1436(6)	8,2(3)
C(12)	3178(5)	9055(7)	1712(6)	7,6(3)
C(13)	2849(4)	7946(6)	1635(5)	5,9(3)
C(14)	1467(4)	2756(7)	1551(5)	6,3(3)
C(15)	1697(5)	1762(8)	2033(6)	7,8(3)
C(16)	1287(5)	626(7)	1893(6)	7,3(3)
C(17)	1310(5)	67(9)	1186(7)	9,3(4)
C(18)	953(6)	-1011(8)	1086(8)	11,0(4)
C(19)	573(6)	-1546(8)	1629(9)	12,8(6)
C(20)	561(6)	-982(9)	2338(8)	11,2(5)
C(21)	909(5)	82(9)	2433(7)	9,6(4)
C(22)	811(4)	4867(7)	955(4)	5,6(2)
C(23)	3843(4)	4667(7)	1908(5)	6,1(2)

TABLE 4. Coordinates of Atoms* ($\times 10^4$; $\times 10^5$ for S atom; $\times 10^3$ for H atoms) for Structure III and Equivalent Thermal Parameter Factors (B_{iso} for H atoms), $Å^2$

Atom	*x/a*	*y/b*	*z/c*	B_{iso}^{eq}
S	13330(3)	22275(10)	9702(7)	3,8
$O_{(S1)}$	1017(1)	2378(4)	100(2)	6,7
$O_{(S2)}$	1234(1)	2312(3)	1982(2)	4,7
$O_{(S3)}$	1477(1)	798(3)	1028(2)	6,6
$O_{(S4)}$	1570(1)	3352(4)	1012(3)	9,5
$N_{(1)}$	1536(1)	9124(3)	2756(2)	3,8
$C_{(2)}$	1503(1)	7600(4)	2333(2)	3,8
$C_{(3)}$	1139(1)	7407(4)	1565(2)	3,4
$C_{(4)}$	840(1)	7680(3)	2030(2)	3,1
$C_{(5)}$	890(1)	9224(3)	2407(2)	3,2
$C_{(6)}$	1256(1)	9443(4)	3237(2)	3,4
$N_{(7)}$	871(1)	6669(3)	2910(2)	3,2
$C_{(8)}$	896(1)	5249(4)	2862(3)	3,7
$C_{(9)}$	938(1)	4433(4)	3865(3)	5,3
$C_{(10)}$	481(1)	7548(4)	1175(2)	3,4
$C_{(11)}$	213(1)	6758(4)	1308(3)	5,1
$C_{(12)}$	-110(1)	6731(6)	523(4)	7,2
$C_{(13)}$	-166(1)	7456(6)	-396(4)	6,8
$C_{(14)}$	99(1)	8230(6)	-532(3)	7,3
$C_{(15)}$	420(1)	8289(5)	237(3)	5,7
$C_{(16)}$	1886(1)	9579(6)	3462(3)	7,1
$C_{(17)}$	2040(2)	8754(7)	4331(5)	8,8
$C_{(18)}$	2386(1)	9588(10)	5007(4)	12,3
$C_{(19)}$	2688(1)	9547(10)	4707(4)	10,7
$C_{(20)}$	2997(1)	10110(6)	5342(4)	7,1
$C_{(21)}$	3013(1)	10725(6)	6266(4)	7,3
$C_{(22)}$	2722(2)	10830(9)	6549(5)	11,8
$C_{(23)}$	2401(1)	10180(9)	5939(4)	11,0
$C_{(24)}$	1770(1)	7274(5)	1784(4)	5,8
$C_{(25)}$	620(1)	9703(4)	2987(3)	4,3
$O_{(26)}$	883(1)	4619(3)	2033(2)	4,9
$H_{(N1)}$	150(1)	961(3)	220(2)	4,0
$H_{(N7)}$	89(1)	702(4)	353(2)	3,8
$H_{(5)}$	111(1)	304(4)	203(3)	6,2

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*Active H coordinates only given.

tions and isotropic ($B_{iso} = 7.5$ $Å^2$) thermal parameters were refined in the calculation of F_{calc}. In III the position of all the hydrogen atoms was localized directly and included in the refinement in the isotropic approximation. The final difference factors were $R = 0.062$ and $R_w = 0.065$ for II (1543 reflections with $F^2 \geq 4\sigma$) and $R = 0.069$ and $R_w = 0.081$ for III (3168 reflections with $F^2 \geq 4.5\sigma$). All crystallographic calculations were made on an Eclipse S/200 computer using the INEXTL program [5].

Coordinates for the nonhydrogen atoms of II and III are given in Tables 3 and 4.

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