

Syntheses and molecular structures of 2-[1'-N-(alkyl)imino-2',2',2'-trifluoroethyl]phenols

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Abstract

N-(Alkyl)imino derivatives of trifluoroacetyl phenol of the general formula 1,2-C₆H₄[C(=NR)CF₃][OH] (R = H, **3a**; Me, **3b**; ⁱPr, **3c**; ^tPr, **3d**; CH₂CH₂NH₂, **3e**; CH₂CH₂CH₂NH₂, **3f**; CH₂CH₂CH₂CH₂NH₂, **3g**; CH₂CH₂OH, **3h**; CH₂CH₂NMe₂, **3i**) were synthesized from 2-trifluoroacetyl phenol and the corresponding primary amines RNH₂. The molecular structures of **3b** [monoclinic, P2₁/n, $a=6.641(3)$, $b=18.044(8)$, $c=7.716(3)$ Å, $\beta=99.95(4)$ °], **3d** [orthorhombic, P2₁2₁2₁, $a=7.784(2)$, $b=9.291(2)$, $c=15.942(4)$ Å] and **3i** [monoclinic, P2₁/n, $a=8.660(2)$, $b=18.135(2)$, $c=13.300(2)$ Å, $\beta=99.83(2)$ °] were determined. In the solid state, **3b**, **3d** and **3i** exist exclusively as *E* isomers with intermolecular hydrogen bridges, whereas according to the ¹H and ¹⁹F NMR spectra in chloroform solution, compounds **3b**, **3c** and **3d** isomerize to give a mixture of 66% *Z* and 34% *E* isomers.

Keywords: Syntheses; Crystal structures; [N-(Alkyl)imino trifluoroethyl]phenols; NMR spectroscopy; Mass spectrometry

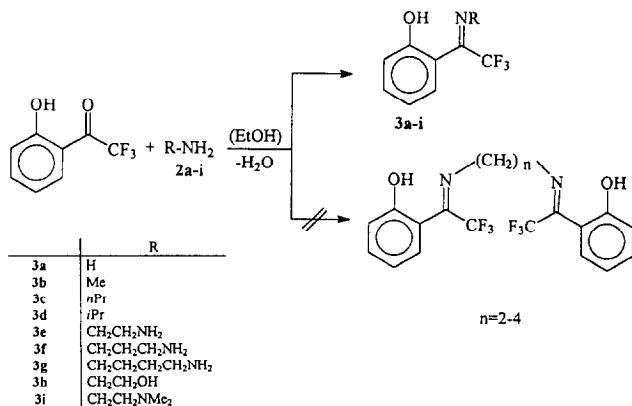
1. Introduction

For the synthesis of 2-trifluoroacetyl phenol (**1**), a copper(II) complex of the imino derivative, 2-(1'-imino-2',2',2'-trifluoromethyl)phenol [1] is required. Since the reactions of 2-trifluoroacetyl phenol and $\lambda^3\sigma^3$ P compounds turned out to yield mono, spiro and tricyclic phosphoranes [2–6], 2-[1'-N-(alkyl)imino-2',2',2'-trifluoromethyl]phenols attracted our attention [7]. In addition, *E/Z* isomerism and hydrogen-bridging properties were of considerable interest.

2. Results and discussion

Primary amines, RNH₂ (**a**: R = H; **b**: R = Me; **c**: R = ⁱPr; **d**: R = ^tPr; **e**: R = CH₂CH₂NH₂; **f**: R = CH₂CH₂CH₂NH₂; **g**: R = CH₂(CH₂)₂CH₂NH₂; **h**: R = CH₂CH₂OH; **i**: R = CH₂CH₂NMe₂) and 2-trifluoroacetyl phenol (**1**) in ethanol reacted to furnish the respective imines **3a–i** (Scheme 1). The water formed was removed completely together with ethanol by distillation. The new compounds were colourless solids but turned yellow in chloroform in the case of **3b**, **c** and **d**.

The reaction of ^tBuNH₂ or (CF₃)₂CHNH₂ and **1** did not yield the expected products under the conditions applied.



Scheme 1.

Surprisingly enough, the diamines **2e–g** gave the mono-substituted compound only in their *E* configuration. In solution, an inversion to furnish the *Z* isomer was not observed. The condensation of a second molecule of ketone **1** did not take place (Scheme 1). Probably there would be a too close steric interaction of the two neighbouring phenol groups in an *E* isomeric arrangement. The decreased reactivity of the second H₂N moiety due to intermolecular interactions with phenolic HO groups could also prevent the disubstitution.

X-Ray investigations revealed that the imines **3b**, **3d** and **3i** prefer an *E* configuration (Figs. 1–3, Scheme 2). Compounds **3b** and **3d** showed long intermolecular hydrogen-

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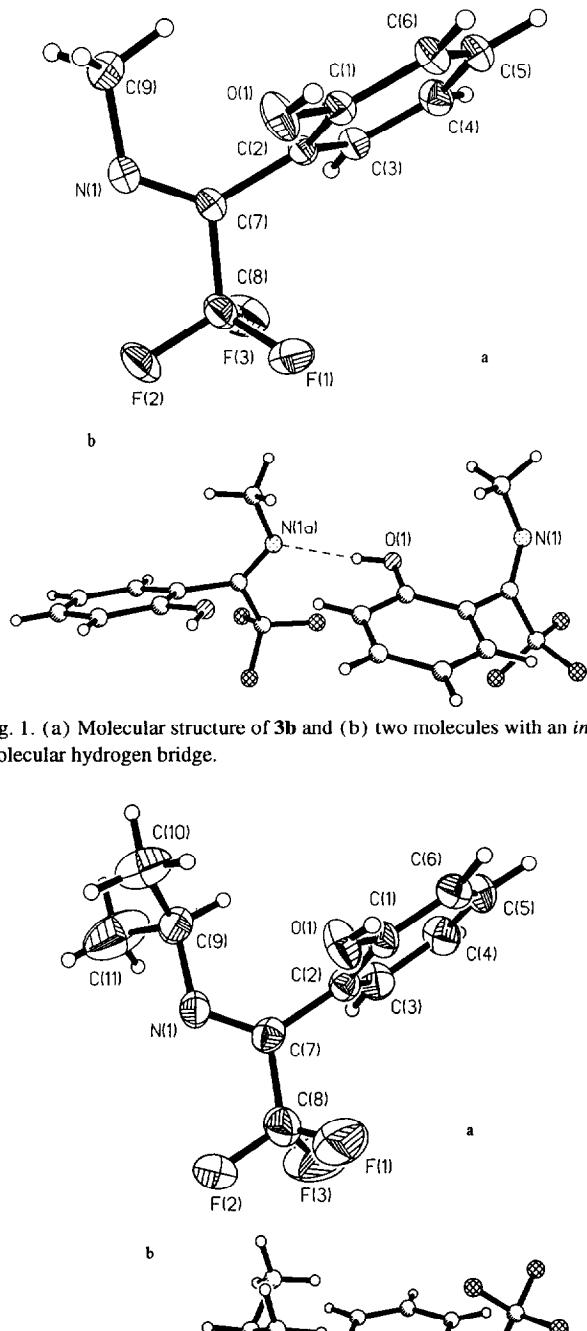


Fig. 1. (a) Molecular structure of **3b** and (b) two molecules with an *inter*-molecular hydrogen bridge.

for **3b**, 97.2(0.3) $^{\circ}$ for **3d** and 93.0(0.5) $^{\circ}$ for **3i**. The C=N bond distances were averaged to be 126.1 pm with a maximum deviation of 0.8 pm (Table 1).

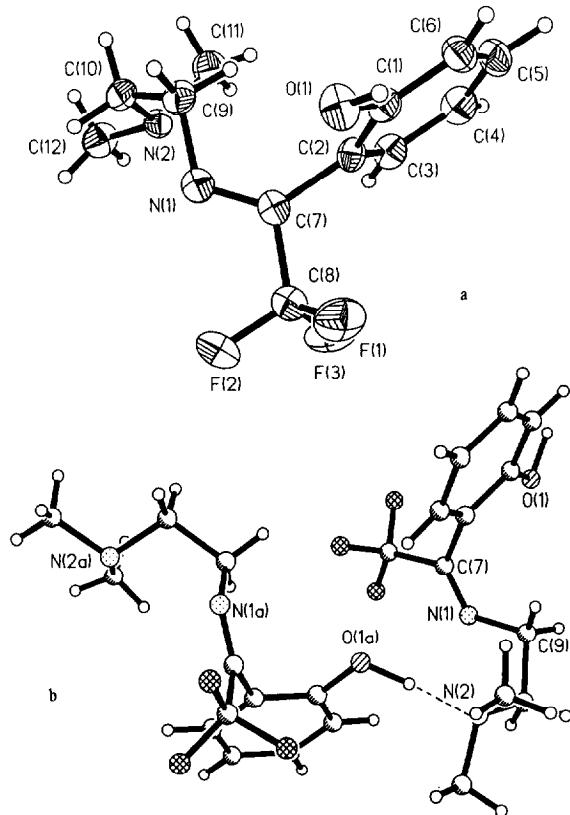


Fig. 3. (a) Molecular structure of **3i** and (b) two molecules with an *inter*-molecular hydrogen bridge.

Table 1
The main geometrical parameters of **3b**, **3d** and **3i**

	3b (R = Me)	3d (R = iPr)	3i (R = CH ₂ CH ₂ NMe ₂)
<i>Bond lengths (Å)</i>			
C(1)-O(1)	135.3(2)	135.5(5)	135.9(4)
C(1)-C(2)	139.9(2)	140.0(6)	140.0(4)
C(2)-C(7)	149.3(2)	151.6(6)	149.7(4)
C(7)-C(8)	152.3(2)	149.6(7)	152.1(4)
C(7)-N(1)	126.9(2)	125.9(6)	125.6(4)
N(1)-C(9)	145.7(2)	146.7(6)	146.2(3)
C(8)-F(1)	133.2(2)	133.9(6)	133.7(4)
C(8)-F(2)	132.5(2)	129.9(6)	131.9(4)
C(8)-F(3)	133.8(2)	133.6(7)	133.4(3)
<i>Bond angles (°)</i>			
C(2)-C(7)-N(1)	128.6(1)	127.3(4)	128.6(2)
C(2)-C(7)-C(8)	115.2(1)	115.6(4)	115.1(2)
C(8)-C(7)-N(1)	116.1(1)	117.0(4)	116.3(2)
C(1)-C(2)-C(3)	119.8(3)	121.0(4)	119.8(1)
C(1)-C(2)-C(7)	118.3(1)	115.9(4)	118.9(2)
C(7)-N(1)-C(9)	118.7(1)	121.8(4)	119.5(2)
<i>Torsion angles (°)</i>			
C(1)-C(2)-C(7)-C(8)	114.8(1)	93.0(5)	97.2(3)

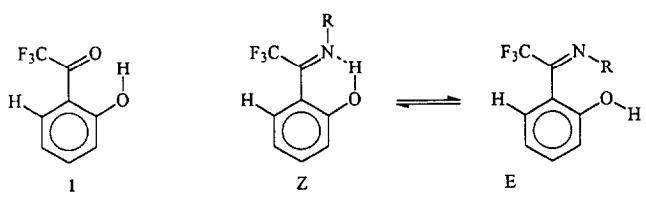
Fig. 2. (a) Molecular structure of **3d** and (b) two molecules with an *inter*-molecular hydrogen bridge.

bridge bonding, $-\text{O}-\text{H}\cdots\text{N}(\text{R})=\text{C}-$, of 198 pm. A much shorter distance, 185.3 pm, was found for the interaction $-\text{O}-\text{H}\cdots\text{N}(\text{Me}_2)\text{CH}_2-$ in compound **3i**. In all three compounds the aromatic ring plane is forced to be almost perpendicular with respect to the imine system. The dihedral angle C(1)-C(2)-C(7)-C(8) was found to be 114.8(0.1) $^{\circ}$

Fresh solutions of **3b**, **3c** and **3d** at ambient temperature showed one signal in the ^{19}F NMR spectrum attributed to the *E* isomer (Table 2). After a few minutes the solution turned yellow and a second signal appeared at lower field probably due to a conversion into the *Z* isomer (Scheme 2). After 20 min a constant ratio $Z/E = 66:34$ was obtained. In the *Z* isomers, intramolecular hydrogen bridges should be present as in the starting compound **1**, which was also yellow and showed a J_{FCCCH} coupling of 2.2 Hz with a considerable 'through-space' contribution [4]. For this rearrangement, the aromatic ring should be in the same plane (dihedral angle $\sim 0^\circ$) as the imine system, so that the 'through-space' coupling contributions for J_{FCCCH} could be larger. The same argument may apply for the increase in the value of J_{FCCNCH} . The conversion of the *E* isomers of **3e–i** was not observed, probably because of steric reasons or strong intermolecular hydrogen bridges $-\text{O}(\text{H})\cdots\text{N}(\text{R}_2)\text{CH}_2-$ ($\text{R} = \text{H}, \text{Me}$) or $-\text{O}(\text{H})\cdots\text{O}(\text{H})\text{CH}_2-$ (**3h**).

3. Experimental details

The appropriate precautions in handling moisture and oxygen-sensitive compounds were observed throughout this



Scheme 2.

work. Elemental analysis: Mikroanalytische Laboratorium Beller, Göttingen. MS: MAT 8222 spectrometer (EI, electron energy 70 eV). NMR: AC 80, operating at 80.13 MHz (H , internal standard TMS) and 75.39 MHz (^{19}F , internal standard CCl_3F). Compound **1** was synthesized using a literature procedure [1].

3.1. General method (see Table 3)

Compound **1** was dissolved in 20 ml of ethanol and the appropriate amount of ammonia or amine added. After removing all volatiles in vacuo, the imines were recrystallized from n-hexane.

Table 2

^1H and ^{19}F NMR data for compounds **3a–i** (J -values are given in Hz)

Compound	^1H chemical shifts ^a (ppm)			^{19}F chemical shifts ^a (ppm)	
	CH_3 ($^3J_{\text{HH}}$)	CH_2	C_6H_4	CF_3 ($^nJ_{\text{HNCCF}}$; $^5J_{\text{HCCCCF}}$)	
3a ($\text{R} = \text{H}$) ^b	—	—	6.7–7.6	−73.4 (2.3, $n = 4$; 1.5)	
3b ($\text{R} = \text{Me}$) ^c	(<i>Z</i>) 3.6	—	6.6–7.6	−63.1 (4.0, $n = 5$; 2.6)	
	(<i>E</i>) 3.2	—	6.6–7.6	−75.7 (1.9, $n = 5$; 0.8)	
3c ($\text{R} = ^{\text{n}}\text{Pr}$) ^c	(<i>Z</i>) 1.1 ^d (7.2)	1.8, 3.9 ^d	6.6–7.6	−63.0 (3.0, $n = 5$; —)	
	(<i>E</i>) 1.1 ^d (7.2)	1.8, 3.9 ^d	6.6–7.6	−75.1 (1.8, $n = 5$; 0.6)	
3d ($\text{R} = ^{\text{i}}\text{Pr}$) ^{c,e}	(<i>Z</i>) 1.1 (5.2)	—	6.5–7.5	−63.1 (2.3, $n = 6$; —)	
	(<i>E</i>) 1.3 (6.2)	—	6.5–7.5	−75.9 (—)	
3e [$\text{R} = (\text{CH}_2)_2\text{NH}_2$] ^f	—	3.1	6.5–7.5	−84.0	
3f [$\text{R} = (\text{CH}_2)_3\text{NH}_2$] ^g	—	1.2, 2.0, 3.0 ^h	6.8–7.4	−87.9	
3g [$\text{R} = (\text{CH}_2)_4\text{NH}_2$]	—	1.6, 2.9	6.7–7.6	−83.5	
3h [$\text{R} = (\text{CH}_2)_2\text{OH}$] ⁱ	—	3.3 ^j , 4.0 ^j	6.7–7.5	−85.0	
3i [$\text{R} = (\text{CH}_2)_2\text{Me}_2$] ^k	2.2	3.5	6.7–7.4	−76.2	

^a Highfield shifts allocated negative signs (TMS and CCl_3F used as standards).

^b $\delta_{\text{H}} = 10.3$ (OH); 13.8 (NH) ppm; only one set of signals found.

^c No resonance for the phenolic OH group found.

^d Signals for *E* and *Z* isomer overlapping.

^e $\delta_{\text{H}} = 3.5$ (CH) ppm for *E* and *Z* isomer.

^f $\delta_{\text{H}} = 2.5$ (NH₂), 11.4 (OH) ppm.

^g $\delta_{\text{H}} = 11.4$ (OH) ppm.

^h 4H, CH_2 and NH₂.

ⁱ No signal found for OH.

^j Multiplets, centred at value given.

^k $\delta_{\text{H}} = 10.1$ (OH) ppm.

Table 3

Experimental details for the preparation of compounds **3a–i**

Compound	Reactants	[g (mmol)]	M.p. (°C)	Yield [g (%)]
3a ^a	1 NH ₃	3.3 (17.4) 0.5 (23.5)	25	2.8 (86)
3b	1 H ₂ NCH ₃	2.5 (13.2) 0.4 (15.0)	88	1.9 (73)
3c	1 H ₂ N(CH ₂) ₂ CH ₃	2.2 (11.5) 0.7 (12)	125	1.8 (68)
3d	1 H ₂ NCH(CH ₃) ₂	1.9 (10) 0.6 (11)	128	2.0 (86)
3e	1 H ₂ N(CH ₂) ₂ NH ₂	1.9 (10) 0.6 (10)	85	1.9 (81)
3f	1 H ₂ N(CH ₂) ₃ NH ₂	2.8 (15) 1.0 (15)	91	2.6 (70)
3g	1 H ₂ N(CH ₂) ₄ NH ₂	1.9 (10) 0.9 (10)	121	2.4 (90)
3h	1 H ₂ N(CH ₂) ₂ OH	1.9 (10) 0.6 (10)	87	1.8 (77)
3i	1 H ₂ N(CH ₂) ₂ NMe ₂	3.0 (16) 1.5 (16)	114	3.8 (91)

^aB.p. 81/12 mmHg, orange solid.

3.2. Crystal structure analysis of **3b**, **3d** and **3i** (see Table 4)

The main crystallographic data are listed in Table 5.

Table 4

Crystal and data reduction parameters for compounds **3b**, **3d** and **3i**

	3b	3d	3i
Formula	C ₉ H ₈ F ₃ NO	C ₁₁ H ₁₂ F ₃ NO	C ₁₂ H ₁₅ F ₃ N ₂ O
Formula weight	203.2	231.2	260.3
Crystal size (mm)	1.0 × 0.8 × 0.7	0.3 × 0.4 × 0.2	0.6 × 0.5 × 0.5
F(000)	416	480	544
Crystal system	monoclinic	orthorhombic	monoclinic
Space group	P2 ₁ /n	P2 ₁ 2 ₁ 2 ₁	P2 ₁ /n
a (Å)	6.641(3)	7.784(2)	8.660(2)
b (Å)	18.044(8)	9.291(2)	11.135(2)
c (Å)	7.716(3)	15.942(4)	13.300(2)
β (°)	99.95(4)	—	99.83(2)
V (Å ³)	910.6(7)	1153.1(5)	1263.6(3)
Z	4	4	4
D _{calc} (g cm ⁻³)	1.482	1.332	1.368
μ (cm ⁻¹)	1.38	1.18	1.18
Index ranges	−8 ≤ h ≤ 8 −14 ≤ k ≤ 23 −10 ≤ l ≤ 9	0 ≤ h ≤ 9 0 ≤ k ≤ 11 0 ≤ l ≤ 18	−5 ≤ h ≤ 11 −14 ≤ k ≤ 14 −17 ≤ l ≤ 17
Temperature (K)	153	153	153
θ _{max} (°) for data	55	50	55
Reflections collected	4774	1246	3284
Independent reflections	2099 (<i>R</i> _{int} = 1.77%)	1203 (<i>R</i> _{int} = 0.89%)	2890 (<i>R</i> _{int} = 1.98%)
Observed reflections	1918 [F > 4.0σ(F)]	958 [F > 4.0σ(F)]	1503 [F > 4.0σ(F)]
Diffractometer used	Siemens P4 m/V		
Radiation	Mo Kα (λ = 0.71073 Å)		
Monochromator	Highly oriented graphite crystal		
Background measurement	Stationary crystal and stationary counter at beginning and end of scan, each for 25% total scan time		
Standard reflections	3 measured every 197 reflections		
Hydrogen atoms	Riding model, fixed isotopic U		

3.3. Analytical data

2-[1'-Imino-2',2',2'-trifluoroethyl]phenol (3a**):** MS (30 °C) *m/e* (%): 189 (M⁺, 100); 120 (M⁺ − CF₃, 97); 102 (C₇H₄N⁺, 38); 92 (C₇H₈⁺, 25) and other fragments. Analysis: C₈H₆F₃NO (189.14): found C, 50.68; H, 3.30; F, 30.0%. Calc.: C, 50.80; H, 3.20; F, 30.13%.

2-[1'-N-Methylimino-2',2',2'-trifluoroethyl]phenol (3b**):** MS (60 °C) *m/e* (%): 203 (M⁺, 36); 134 (M⁺ − CF₃, 100); 119 (C₇H₅NO⁺, 10), 77 (C₆H₅⁺, 6); 69 (CF₃⁺, 5); 42 (CH₂=N=CH₂, 20) and other fragments. Analysis: C₉H₈F₃NO (203.23): Found: C, 53.26; H, 4.11; F, 28.2%. Calc.: C, 53.19; H, 3.97; F, 28.04%.

2-[1'-N-Propylimino-2',2',2'-trifluoroethyl]phenol (3c**):** MS (80 °C) *m/e* (%): 231 (M⁺, 61); 202 (M⁺ − C₂H₅, 15); 162, (M⁺ − CF₃, 32); 120 (HOC₆H₄CNH⁺, 100) and other fragments. Analysis: C₁₁H₁₂F₃NO (231.22): Found: C, 57.31; H, 5.34; F, 24.7%. Calc.: C, 57.14; H, 5.23; F, 24.65%.

2-[1'-N-Isopropylimino-2',2',2'-trifluoroethyl]-phenol (3d**):** MS (50 °C) *m/e* (%): 231 (M⁺, 42); 189 (M⁺ − C₃H₆, 13); 162 (M⁺ − CF₃, 10); 120 (HOC₆H₄CNH⁺, 100); 102 (C₆H₅CN⁺, 8); 42 (C₃H₆⁺, 7) and other fragments. Analysis: C₁₁H₁₂F₃NO (231.22): Found: C, 57.34; H, 5.34; F, 24.7%. Calc.: C, 57.14; H, 5.23; F, 24.65%.

2-[1'-N-(2"-Aminoethyl)imino-2',2',2'-trifluoroethyl]-phenol (3e**):** MS (50 °C) *m/e* (%): 232 (M⁺, 62); 203

Table 5
Coordinates ($\times 10^4$) for non-hydrogen atoms and their equivalent isotopic temperature factors B_{eq} ($\text{\AA}^2 \times 10^{-1}$)

Compound	Atom	<i>x</i>	<i>y</i>	<i>z</i>	B_{eq} (\AA^2)
3b	C(1)	2381(2)	3306(1)	6516(2)	20(1)
	C(2)	1696(2)	3876(1)	5329(2)	18(1)
	C(3)	2481(2)	4590(1)	5637(2)	22(1)
	C(4)	3916(2)	4742(1)	7131(2)	26(1)
	C(5)	4585(2)	4176(1)	8298(2)	27(1)
	C(6)	3848(2)	3462(1)	8004(2)	24(1)
	C(7)	203(2)	3688(1)	3712(2)	19(1)
	C(8)	991(2)	3787(1)	1986(2)	27(1)
	C(9)	-2513(2)	3360(1)	5169(2)	27(1)
	O(1)	1589(2)	2622(1)	6140(1)	28(1)
	N(1)	-1595(2)	3432(1)	3593(1)	21(1)
	F(1)	2840(1)	3487(1)	2074(1)	45(1)
	F(2)	-212(2)	3486(1)	619(1)	45(1)
	F(3)	1193(2)	4506(1)	1627(1)	48(1)
3d	C(1)	-919(6)	3404(5)	3324(3)	30(1)
	C(2)	-1620(6)	4419(5)	3876(3)	30(1)
	C(3)	-2654(7)	4003(5)	4529(3)	41(2)
	C(4)	-3031(7)	2537(5)	4632(3)	43(2)
	C(5)	-2322(6)	1549(5)	4103(3)	37(2)
	C(6)	-1300(6)	1964(5)	3445(3)	33(2)
	C(7)	-1145(6)	5975(5)	3708(3)	32(1)
	C(8)	384(8)	6526(6)	4177(3)	48(2)
	C(9)	-3446(6)	6434(5)	2764(3)	39(1)
	C(10)	-3101(7)	6207(7)	1845(3)	62(2)
	C(11)	-4709(9)	7622(9)	2908(4)	95(3)
	O(1)	59(5)	3938(3)	2694(2)	39(1)
	N(1)	-1863(5)	6831(4)	3206(2)	32(1)
	F(1)	83(6)	6489(5)	5004(2)	98(2)
	F(2)	871(5)	7823(3)	3988(2)	89(2)
	F(3)	1754(4)	5675(4)	4082(3)	85(1)
3i	C(1)	1072(3)	3476(3)	2966(2)	27(1)
	C(2)	1600(3)	2297(2)	2886(2)	24(1)
	C(3)	1538(3)	1487(3)	3672(2)	31(1)
	C(4)	929(3)	1837(3)	4521(2)	37(1)
	C(5)	390(3)	3000(3)	4592(2)	38(1)
	C(6)	476(3)	3816(3)	3825(2)	33(1)
	C(7)	2228(3)	1937(2)	1949(2)	24(1)
	C(8)	1063(3)	1303(3)	1137(2)	31(1)
	C(9)	4763(3)	2700(3)	2493(2)	32(1)
	C(10)	6288(3)	2014(3)	2638(2)	31(1)
	C(11)	6194(4)	979(3)	4233(2)	41(1)
	C(12)	7493(3)	76(3)	2976(3)	39(1)
	O(1)	1166(3)	4243(2)	2184(2)	36(1)
	N(1)	3584(3)	2084(2)	1754(2)	28(1)
	N(2)	6182(3)	845(2)	3133(2)	28(1)
	F(1)	573(2)	271(2)	1488(1)	52(1)
	F(2)	1629(2)	1049(2)	302(1)	53(1)
	F(3)	-216(2)	1973(2)	858(1)	49(1)

($M^+ - H_2C=NH_2$, 32); 163 ($M^+ - CF_3$, 100); 134 [$HOC_6H_4CN(CH_3)^+$, 68]; 120 ($HOC_6H_4CNH^+$, 20); 44 ($H_2C=NH-CH_3^+$, 48); 30 ($H_2C=NH_2^+$, 60) and other frag-

ments. Analysis: $C_{10}H_{11}F_3N_2O$ (232.20): Found C, 51.78%; H, 4.77%; F, 24.70%. Calc.: C, 51.73%; H, 4.77%; F, 24.54%.

2-[1'-*N*-(3"-Aminopropyl)imino-2',2',2'-trifluoroethyl]phenol (**3f**): MS (50 °C) *m/e* (%): 246 (M^+ , 58); 216 ($M^+ - H_2C=NH_2$, 32); 177 ($M^+ - CF_3$, 100); 134 [$HOC_6H_4CN(CH_3)$, 40]; 120 ($HOC_6H_4CNH^+$, 42); 30 ($H_2C=NH_2$, 48) and other fragments. Analysis: $C_{11}H_{13}F_3N_2O$ (246.23): Found: C, 53.79%; H, 5.25%; F, 23.0%. Calc.: C, 53.66%; H, 5.32%; F, 24.15%.

2-[1'-*N*-(3"-Aminobutyl)imino-2',2',2'-trifluoroethyl]phenol (**3g**): MS (80 °C) *m/e* (%): 260 (M^+ , 30); 191 ($M^+ - CF_3$, 20); 120 ($HOC_6H_4CNH^+$, 20); 70 ($C_4H_8N^+$, 70); 43 ($C_2H_5N^+$, 38); 30 (CH_4N^+ , 100) and other fragments. Analysis: $C_{12}H_{15}F_3N_2O$ (260.26): Found: C, 55.43%; H, 5.81%; F, 22.00%. Calc.: C, 55.38%; H, 5.81%; F, 21.90%.

2-[1'-*N*-(Ethyl-2"-ol)imino-2',2',2'-trifluoroethyl]phenol (**3h**): MS (50 °C) *m/e* (%): 233 (M^+ , 70); 202 ($M^+ - CH_2OH$, 30); 164 ($M^+ - CF_3$, 100); 121 ($HOC_6H_4N=CH_2$, 70); 44 ($H_2C=CHOH^+$, 60) and other fragments. Analysis: $C_{10}H_{10}F_3NO_2$ (233.12): Found C, 51.15%; H, 4.32%; F, 24.5%. Calc.: C, 51.51%; H, 4.32%; F, 24.44%.

2-[1'-*N*-(2"-Dimethylamino)ethyl]imino-2',2',2'-trifluoroethyl]phenol (**3i**): MS (60 °C) *m/e* (%): 260 (M^+ , 95); 120 ($HOC_6H_4CNH^+$, 42); 102 [$M^+ - H_2N=N(CH_3)_2$, 25]; 77 ($C_6H_5^+$, 30); 71 [$H_3CCH=N(CH_3)_2$, 68]; 58 [$H_2N=N(CH_3)_2$, 100]; 42 ($H_2C=N=CH_2$, 98); 30 ($H_2C=NH_2$, 90) and other fragments. Analysis: $C_{12}H_{15}F_3N_2O$ (260.26): Found C, 55.30%; H, 5.86%; F, 21.80%. Calc.: C, 55.38%; H, 5.81%; F, 21.90%.

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References

- [1] S. Matsumoto, H. Kobayashi and K. Ueno, *Bull. Chem. Soc. Jpn.*, **42** (1969) 960.
- [2] R.D. Hund and G.-V. Röschenthaler, *Phosphorus, Sulfur, Silicon*, **62** (1991) 65.
- [3] R.D. Hund and G.-V. Röschenthaler, *Phosphorus, Sulfur, Silicon*, **66** (1992) 301.
- [4] R.D. Hund, U. Behrens and G.V. Röschenthaler, *Phosphorus, Sulfur, Silicon*, **69** (1992) 119.
- [5] R.D. Hund and G.-V. Röschenthaler, *Phosphorus, Sulfur, Silicon*, **73** (1992) 99.
- [6] F.U. Seifert and G.-V. Röschenthaler, *Z. Naturforsch., Teil b*, **48** (1993) 169.
- [7] R.-M. Schoth and G.-V. Röschenthaler, *Phosphorus, Sulfur, Silicon*, **103** (1995) 235.