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# 1-CHLORO-2,6-DINITRO-4-PERFLUOROALKYLTHIOBENZENES IN THE SYNTHESIS OF HETEROCYCLES

**S. M. Sipyagin[1], V. S. Enshov[1], S. A. Kashtanov[1], and J. S. Thrasher[2]**

*1-Chloro-2,6-dinitro-4-perfluoroalkylthiobenzenes was obtained in first time by perfluoroalkylation of bis(4-chloro-3,5-dinitrophenyl)disulfide in the presence of xenon bisperfluoroalkylcarboxylates. At the interaction of these compounds with potassium ethylxanthogenate only substitution of chlorine atom occurred. The reaction with sodium N,N-dimethyldithiocarbamate leads to the nucleophilic substitution of nitro group with formation of 1,3-benzodithiol-2-one, but on action of ethyl thioglycolate the intramolecular condensation occurs with formation of benzothiazoles N-oxides.*

**Keywords:** 1-ethylthio-2,6-dinitro-4-trifluoromethylthiobenzene, 2-ethoxycarbonyl-7-nitro-5-perfluoroalkylthiobenzothiazole N-oxide, 7-nitro-6-trifluoromethylthio-1,3-benzodithiol-2-one, 1-chloro-2,6-dinitro-4-perfluoroalkylthiobenzenes, heterocyclization, perfluoroalkylation of aromatic disulfides.

The replacement of the hydrogen atom in aromatic and heterocyclic systems by perfluoroalkyl groups may significantly affect the physical and biological properties of such molecules [1]. However, the introduction of "superlipophilic" perfluoroalkylthio groups $SR_F$ ($R_F$ = $CF_3$, $C_2F_5$, etc.), which have the highest lipophilicity indices [2], may substantially enhance the biological effect of the resultant compounds [3].

In light of our current scientific interests, we are developing a synthesis of new synthones containing both superlipophilic substituents and functional groups, which are capable of reacting with various nucleophilic agents. For example, this might involve aromatic and heterocyclic compounds activated by other electron-withdrawing substituents [4]. Such an approach, in our view, might lead to new compounds with potential biological activity.

In the present work, we describe heterocyclization involving two new 1-chloro-3,5-dinitro-4-perfluoroalkylthiobenzenes synthesized by the perfluoroalkylation of bis(4-chloro-3,5-dinitrophenyl) disulfide (**1**). This is the first report of the preparation of **1** by the reduction of the corresponding sulfonyl chloride by an HBr/PhOH mixture in acetic acid according to Lukashevich [5]. We have recently developed a method for the perfluoroalkylation of heterocyclic thiols and disulfides based on the thermolytic reactions of xenon(II) bis(perfluoroalkylcarboxylates) [6]. In the case of disulfide **1**, this method was extended to aromatic compounds. In general, the perfluoroalkylation of **1** may be initiated by electron transfer from the disulfide RSSR to the xenon bisperfluoroalkylcarboxylate molecule to give a radical–cation and radical–anion [7]. Then,

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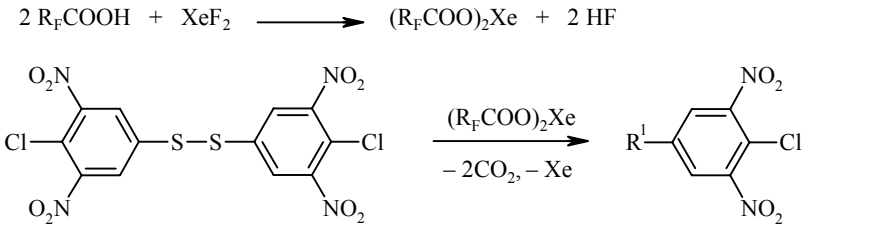
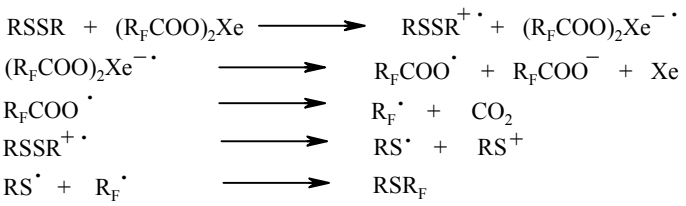
[1] Institute of Problems of Chemical Physics, Russian Academy of Sciences, 142432 Chernogolovka, Moscow Region, Russia; e-mail: sip@icp.ac.ru. [2] Department of Chemistry, The University of Alabama, Tuscaloosa, AL 35487-0336, USA; e-mail: fluorine@bama.ua.edu. Translated from Khimiya Geterotsiklicheskikh Soedinenii, No. 11, pp. 1559-1565. November, 2002. Original article submitted April 12, 2000.

the radical–anion decomposes to give a perfluoroalkyl radical, perfluoroalkanoate, carbon dioxide, and xenon, while the radical–cation may decompose to give $RS^{\cdot}$ and $RS^{+}$. The recombination of $RS^{\cdot}$ and $R_F^{\cdot}$ leads to the expected products **2a** or **2b**.




**1** **2a,b**

**2 a** $R^1 = SCF_3$, **b** $R^1 = SC_2F_5$




R = 1-chloro-2,6-dinitrophenyl; $R_F = C_nF_{2n+1}$ (when $n$ = 1–2)

The greatest yield of **2a** (70%) was achieved when the disulfide–perfluoroalkylcarboxylic acid–xenon difluoride ratio was 1:5:4. When smaller amounts of the perfluorocarboxylic acid and $XeF_2$ were used, some of disulfide **1** remains unreacted in the reaction mixture. A significant effect of the solvents (trifluoroacetic acid, perfluoropropionic acid, methylene chloride, and acetonitrile) on the yields of **2a** and **2b** was found. The best results were obtained when the perfluoroalkylation was carried out in methylene chloride, in which disulfide **1** has high solubility. Products **2a** and **2b** are light brown or yellow crystalline compounds.

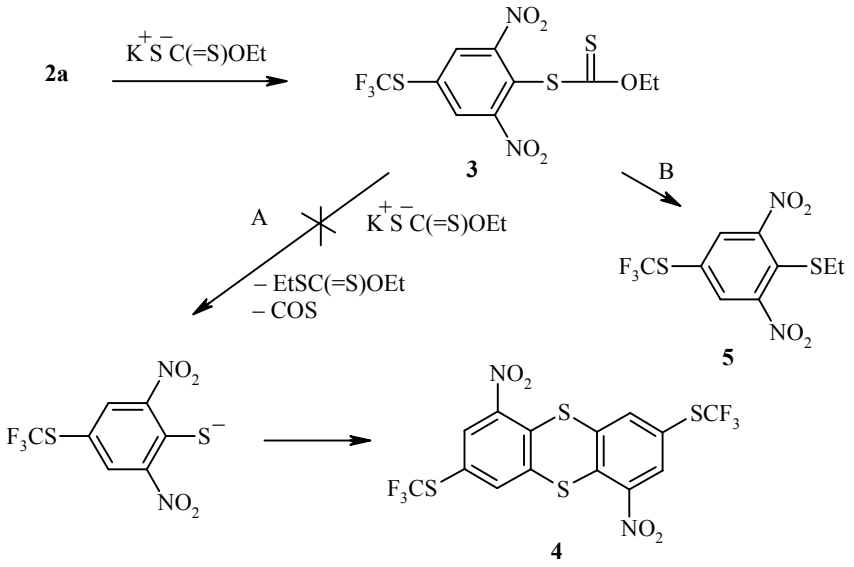
The $^{19}F$ NMR spectrum of **2a** shows a singlet for the $CF_3S$ group at -41.62 ppm. The quartet at -90.88 ppm and triplet at -82.74 ppm confirm the presence of a $C_2F_5S$ fragment in **2b**. The $^{13}C$ NMR spectrum of **2a** has five signals. The quartet with $^1J_{C–F}$ = 310 Hz is assigned to the $CF_3$ group. The triplet at 126.6 ppm ($^3J_{C–SCF_3}$ = 2.7 Hz) is assigned to the carbon atom directly attached to the $SCF_3$ fragment, while the triplet at 134.2 ppm ($^4J_{C–SCF_3}$ = 1.1 Hz) is ascribed to $C_{(4)}$ and $C_{(5)}$. The singlet at 149.8 ppm belongs to the carbon atom bound to the chlorine atom.

The chlorine atom and one of the nitro groups in 1-chloro-2,6-dinitro-4-trifluoromtehylbenzene are good leaving groups in reactions with nucleophilic agents, while two-step intramolecular substitution reactions may occur in the presence of bifunctional sulfur-containing reagents such as potassium ethyl xanthate or sodium N,N-dimethyldithiocarbamate to give condensed heterocycles [8, 9].

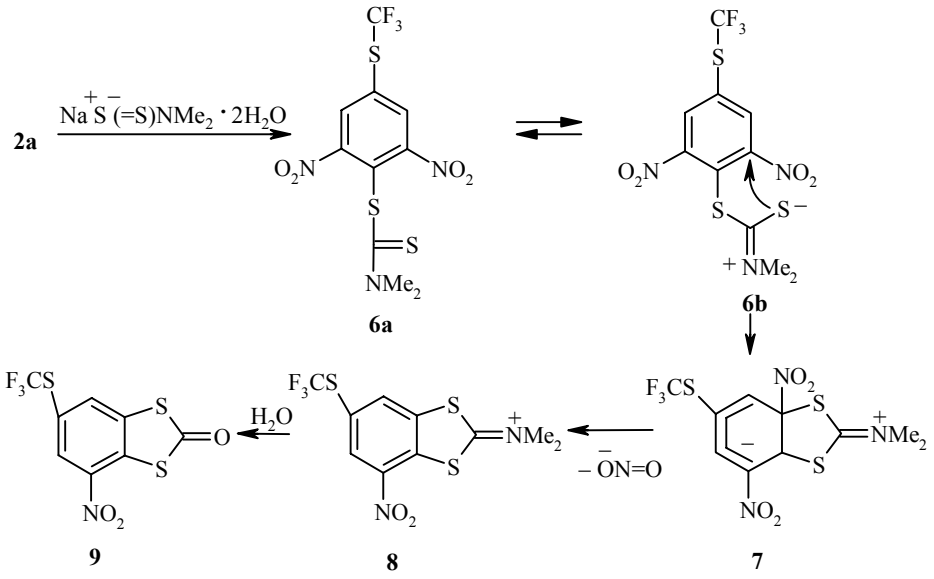
Thus, the formation of thianthrene **4** through pathway A would be expected in the reaction of **2a** with potassium ethyl xanthate (Scheme 1). However, in fact, pathway B is realized, involving initial attack of the chlorine atom in synthone **2a** by the ethyl xanthate anion to give intermediate **3**, which then converts to ethylthio derivative **5** through loss of a COS molecule.

An attempt to synthesize a 1,3-benzdithiol-2-one derivative in the reaction of dinitrochlorobenzene **2a** with sodium N,N-dimethyldithiocarbamate proved more successful. We are the first to report the preparation of a 1,3-benzodithiol-2-one containing a $CF_3S$ group **9** in 40% yield and thereby expand the scope of this general method for the synthesis of this important class of organic compounds, which are precursors to tetrathiafulvenes. The formation of 1,3-benzodithiol-2-one **9** may be seen as the result of initial replacement of

Scheme 1

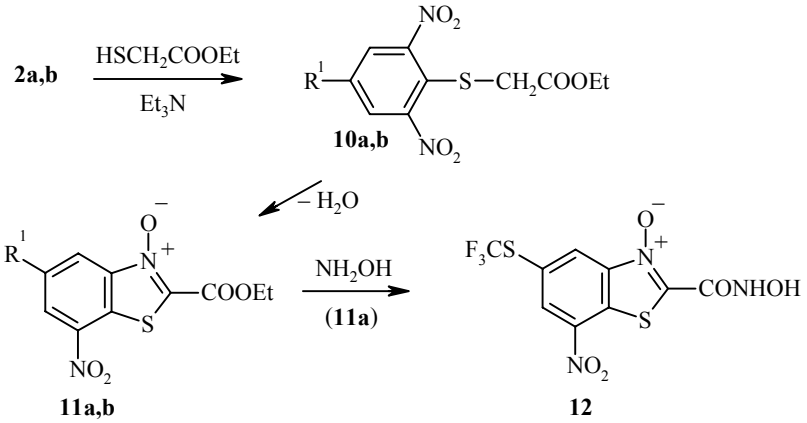


the chlorine atom in synthone **2a** by the N,N-dimethyldithiocarbamate fragment, subsequent loss of one of the nitro groups at a nitrite ion from σ-complex **7** as the result of intramolecular attack of the negatively charged sulfur atom of mesomeric form **6b**, and, finally, hydrolysis of iminium intermediate **8** in the presence of water molecules found in the initial sample of sodium N,N-dimethyldithiocarbamate:



On the other hand, an radical–ion mechanism may be proposed for the intramolecular cyclization of intermediate **6a** by analogy with our previous findings [10]. The presence of the 1,3-dithiol-2-one fragment in **9** is indicated by the singlet for the carbonyl carbon atom in the $^{13}C$ NMR spectrum at 189.0 ppm, the IR bands at 1704 and 1666 $cm^{-1}$ characteristic for the C=O bond, and the major mass spectral decomposition pathway with loss of CO by the molecular ion [11].

Another possibility for the use of **2a** and **2b** for the synthesis of heterocyclic compounds may be seen in the reaction with ethyl thioglycolate [12], leading to derivatives of benzothiazole N-oxide **11**. In this case, the chlorine atom is replaced by the ethyl thioglycolate fragment (intermediates **10a** and **10b**) and subsequent intramolecular condensation with a nitro group leading to heterocyclization. The conversion of ethyl ester **11a** into hydroxamic acid **12** provides additional information on the reactivity of these heterocyclic compounds:




**2, 10, 11** **a** $R^1 = SCF_3$, **b** $R^1 = SC_2F_5$

The $^{13}C$ NMR spectrum of the benzothiazole fragment in **11a** shows seven signals. The two strongest signals at 132.4 and 132.7 ppm belong to $C_{(4)}$ and $C_{(6)}$ bound to protons. The signals for $C_{(5)}$ and $C_{(7)}$ attached to the $NO_2$ and $SCF_3$ groups in the benzene ring are seen at 142.4 and 123.4 ppm, respectively, and are rather similar to the values for the analogous atoms in **9**. The thiazole ring carbon atoms are seen as singlets at 126.1, 137.4, and 147.0 ppm and are ascribed to $C_{(2)}$, $C_{(8)}$, and $C_{(9)}$, respectively. The ethoxycarbonyl fragment is seen as a carbonyl carbon at 156.5 ppm, while a strong IR band is observed for the carbonyl group at 1740 $cm^{-1}$. The N–O fragment is indicated in the finding of strong $[M - O]^+$ ion peaks in the mass spectra, typical for the decomposition of the molecular ions of heterocyclic N-oxides [13].

## EXPERIMENTAL

Column chromatography was carried out on Merck silica gel 60, 230-400 mesh. Thin-layer chromatography was carried out on Merck silica gel 60 $F_{254}$. The $^1H$, $^{13}C$, and $^{19}F$ NMR spectra were taken on a Bruker AM-360 spectrometer at 360 MHz or AM-500 spectrometer at 500 MHz using TMS or $CFCl_3$ as the internal standard. The IR spectra were taken on Biorad FTS-40 FT-IR and Specord M-80 spectrometers. The GC/MS analysis was carried out on a Hewlett–Packard 5890 GC/MS unit at 70 eV using a 30-m capillary column packed with HP1 oil. The high-resolution mass spectra were taken on a VG Autospec mass spectrometer. The measurement precision was ±0.002 dalton.

**Bis(4-chloro-3,5-dinitrophenyl) Disulfide (1).** This starting aromatic disulfide was obtained in a three-step synthesis. The potassium salt of 4-chloro-3,5-dinitrobenzenesulfonic acid is obtained in the first step from chlorobenzene. In the second step, this salt is converted to the corresponding sulfonyl chloride according to Ullmann [15]. In the third step, sulfonyl chloride (20 g, 66.4 mol) is dissolved with stirring in glacial acetic acid (340 ml) saturated with 35 g of gaseous HBr. Then, phenol (6.9 g) is added to the reaction mixture and carefully heated to 55-60°C. An exothermal reaction ensued. The reaction mixture was stirred for 26 h at 60°C and a light yellow precipitate formed. After cooling, the precipitate was filtered off, washed with acetic acid and ethanol, and dried to give 14 g (90%) **1**. The precipitate was recrystallized from glacial acetic acid to give 13.3 g of yellow crystals; mp 188-189°C (acetic acid). IR spectrum (vaseline oil), ν, $cm^{-1}$: 3063 (C–H), 1536, 1351, 1278,

1129, 1054, 914, 883, 722. $^1$H NMR spectrum ($CDCl_3$), δ, ppm: 8.09 (s). Mass spectrum, *m/z* (*I*, %): 466 ($[M]^+$, 73), 234 ($[M/2 + 1]^+$, 100), 233 ($[M/2]^+$, 23), 218 (8), 188 (31). Found: *m/z* 465.883 $[M]^+$. $C_{12}H_4Cl_2N_4O_8S_2$. Calculated: M 465.885.

**Perfluoroalkylation of Bis(4-chloro-3,5-dinitrophenyl) Disulfide (1) (General Method).** A sample of disulfide **1** (4.0 g, 8.57 mmol) was added with stirring to a mixture of $XeF_2$ and perfluoroalkylcarboxylic acid in $CH_2Cl_2$ (15-20 ml) at -30°C (the disulfide–$XeF_2$–perfluoroalkylcarboxylic acid mole ratio was 1:4:5). Stirring was continued and the mixture was gradually let warm to room temperature. The end of the reaction was defined as the cessation of gas evolution. The reaction mixture was washed with saturated aqueous $NaHCO_3$. The organic layer was separated and dried over $Na_2SO_4$. After removal of the solvent in vacuum, the residue was subjected to chromatography on a silica gel column using 1:2 hexane–benzene as the eluent.

**1-Chloro-2,6-dinitro-4-trifluoromethylthiobenzene (2a)** was obtained in 70% yield as light yellow crystals; mp 35-37°C. $^1$H NMR spectrum ($CDCl_3$), δ, ppm: 8.27 (s). $^{19}$F NMR spectrum, δ, ppm: -41.62 (s). $^{13}$C NMR spectrum, δ, ppm, *J* (Hz): 123.6, 126.6 (q, $^3J_{C–SCF_3}$ = 2.7); 128.3 (q, $J_{C–F}$ = 31.0.1); 134.2 (q, $^4J_{C–SCF_3}$ = 1.1); 149.8. Mass spectrum, *m/z* (*I*, %): 302 ($[M]^+$, 100), 283 ($[M - F]^+$, 5), 256 ($[M - NO_2]^+$, 1), 233 ($[M - CF_3]^+$, 2), 210 ($[M - 2NO_2 - CF_3]^+$, 16), 69 $[CF_3]^+$, 9). Found: *m/z* 301.937 $[M]^+$. $C_7H_2ClF_3N_2O_4S$. Calculated: M 301.938.

**1-Chloro-2,6-dinitro-4-pentafluoroethylthiobenzene (2b)** was obtained in 66% yield as yellow crystals; mp 65-67°C. $^1$H NMR spectrum ($CDCl_3$), δ, ppm, *J* (Hz): 8.27 (s). $^{19}$F NMR spectrum, δ, ppm, *J* (Hz): -90.88 (2F, q, $J_{F–F}$ = 3.3); -82.74 (3F, t, $J_{F–F}$ = 3.3). Mass spectrum, *m/z* (*I*, %): 352 ($[M]^+$, 100), 316 ($[M - HCl]^+$, 1), 283 ($[M - CF_3]^+$, 6), 237 ($[M - CF_3 - NO_2]^+$, 2), 233 ($[M - C_2F_5]^+$, 4), 191 (2), 187 (1), 106 (20). Found: *m/z* 351.934 $[M]^+$. $C_8H_2ClF_5N_2O_4S$. Calculated: M 351.934.

**Reaction of 1-Chloro-2,6-dinitro-4-trifluoromethylthiobenzene (2a) with Potassium Ethyl Xanthate.** A sample of **2a** (2.48 mmol) was added in a single portion to a stirred solution of potassium ethyl xanthate (0.44 g, 2.75 mol) in DMF (3 ml). The reaction mixture was heated to 80°C and maintained at this temperature with stirring for 10 h and then at room temperature for an additional 5 h. The solvent was removed in vacuum. The residue was washed with 10% hydrochloric acid and extracted with chloroform. The organic layer was removed and dried over $Na_2SO_4$. The solvent was evaporated. The residue was subjected to chromatography on a silica gel column using 1:2 benzene–hexane as the eluent).

**1-Ethylthio-2,6-dinitro-4-trifluoromethylthiobenzene (7b)** was obtained in 28% yield as orange crystals; mp 50-52°C. $^1$H NMR spectrum, δ, ppm, *J* (Hz): 1.26 (3H, t, *J* = 7.5, $CH_3$); 2.94 (2H, q, *J* = 7.5, $CH_2$); 8.12 (2H, s, Ar). $^{19}$F NMR spectrum: -41.63 (s). Mass spectrum, *m/z* (*I*, %): 328 ($[M]^+$, 54), 300 ($[M - C_2H_4]^+$, 32), 283 ($[M - C_2H_4 - OH]^+$, 67), 270 ($[M - C_2H_4 - NO]^+$, 73), 223 ($[M - C_2H_4 - HNO_2]^+$, 26), 206 ($[M - C_2H_4 - 2HNO_2]^+$, 100), 176 (82). Found: *m/z* 327.978 $[M]^+$. $C_9H_7F_3N_2O_4S_2$. Calculated: M 327.980.

**Reaction of 1-Chloro-2,6-dinitro-4-trifluoromethylthiobenzene (2a) with Sodium N,N-Dimethyldithiocarbamate.** A solution of sodium N,N-dimethyldithiocarbamate (0.5 g) in DMSO (6 ml) was added dropwise to a solution of **2a** (1 g, 2.8 mmol) in DMSO (6 ml) stirred at room temperature. Stirring was continued for 24 h. Then, water (150 ml) was added and the mixture was extracted with three 75-ml chloroform portions. The combined extracts were washed thrice with water and dried over $Na_2SO_4$. The solvent was evaporated and the residue was subjected to chromatography on a silica gel column with 1:1 benzene–hexane as the eluent.

**4-Nitro-6-trifluoromethylthio-1,3-benzodithiol-2-one (9)** was obtained in the above reaction as yellow crystals; mp 93-94°C. Yield of **9** 0.33 g (40%). IR spectrum ($CHCl_3$), ν, $cm^{-1}$: 1704, 1666 (C=O), 1542, 1339 (N–O), 1173, 1146, 1105 ($CF_3$). $^1$H NMR spectrum ($CDCl_3$), δ, ppm, *J* (Hz): 8.11 (d, *J* = 1.25, Ar); 8.62 (d, *J* = 1.25, Ar). $^{19}$F NMR spectrum, δ, ppm: -42.07 (s, $SCF_3$). $^{13}$C NMR spectrum, δ, ppm, *J* (Hz): 124.8 (q, $J_{C(5)–SCF_3}$ = 2.4, $C_{(5)}$); 129.4 (q, $J_{C–F}$ = 308, $CF_3$); 130.9, 135.4 ($C_{(4)}$, $C_{(6)}$); 134.2, 138.2 ($C_{(1)}$, $C_{(2)}$); 143.6 (br. s, $C_{(3)}$); 189.0 (s, C=O). Mass spectrum, *m/z* (*I*, %): 368 ($[M]^+$, 100), 294 ($[M - F]^+$, 2), 285 ($[M - CO]^+$, 57), 239 ($[M - CO - NO_2]^+$, 4), 216 (3), 209 (3), 170 (11). Found: *m/z* 312.915 $[M]^+$. $C_8H_2F_3NO_3S_3$. Calculated: M 312.914.

**Reactions of 1-Chloro-2,6-dinitro-4-perfluoroalkylthiobenzenes (2a,b) with Ethyl Thioglycolate (General Method).** A sample of triethylamine (1.2 ml, 8.4 mmol) was added with stirring to a mixture of 2,6-dinitro-4-perfluoroalkylchlorobenzene **2a** or **2b** (7.6 mmol) and ethyl thioglycolate (0.9 g, 7.6 mmol) in ethanol with ice cooling. The reaction solution was stirred at room temperature for 2 h. The precipitate formed was filtered off, washed with water and then ethanol, and dried.

**2-Ethoxycarbonyl-7-nitro-5-trifluoromethylthiobenzothiazole N-Oxide (11a)** was obtained in 75% yield in the above reaction as orange crystals; mp 134-136°C. IR spectrum (vaseline oil), ν, $cm^{-1}$: 1740 (C=O). $^1$H NMR spectrum ($CDCl_3$), δ, ppm, *J* (Hz): 1.46 (3H, t, *J* = 7.2, $CH_3$); 4.53 (2H, q, *J* = 7.2, $CH_2$); 8.84 (2H, s, Ar). $^{19}$F NMR spectrum, δ, ppm: -42.03 (s). $^{13}$C NMR spectrum, δ, ppm, *J* (Hz): 14.1 (Me), 63.3 ($CH_2$); 125.7 ($C_{(5)}$); 126.1 ($C_{(2)}$); 128.6 (q, $J_{C–F}$ = 310.0, $CF_3$); 132.4, 132.7 ($C_{(4)}$, $C_{(6)}$); 137.4 ($C_{(8)}$); 142.4 ($C_{(7)}$); 147.0 ($C_{(9)}$); 159.5 (C=O). Mass spectrum, *m/z* (*I*, %): 368 ($[M]^+$, 6), 352 ($[M - O]^+$, 39), 333 ($[M - O - F]^+$, 14), 322 ($[M - NO_2]^+$, 307 ($[M - O - EtO]^+$, 39), 280 ($[M - O - EtO - HCN]^+$, 100), 250 (20), 234 (27), 222 (13). Found: *m/z* 367.976 $[M]^+$. $C_{11}H_7F_3N_2O_5S_2$. Calculated: M 367.975.

**2-Ethoxycarbonyl-7-nitro-5-pentafluoroethylthiobenzothiazole N-Oxide (11b)** was obtained in 66.7% yield in the above reaction as orange crystals; mp 145-147°C. IR spectrum (vaseline oil), ν, $cm^{-1}$: 1740 (C=O). $^1$H NMR spectrum ($CDCl_3$), δ, ppm, *J* (Hz): 1.48 (3H, t, *J* = 7.2, $CH_3$); 4.56 (2H, q, *J* = 7.2, $CH_2$); 8.85 (1H, s, Ar); 8.88 (1H, d, *J* = 1.7, Ar). $^{19}$F NMR spectrum, δ, ppm: -91.15 (2F, s, $CF_2$); -82.62 (3F, s, $CF_3$). Mass spectrum, *m/z* (*I*, %): 418 ($[M]^+$), 20), 402 ($[M - O]^+$, 55), 386 (15), 372 ($[M - NO_2]^+$, 17), 357 ($[M - O - EtO]^+$, 48), 346 ($[M - EtO - HCN]^+$, 28), 330 ($[M - O - EtO - HCN]^+$, 100), 311 (14), 284 (25). Found: *m/z* 417.973 $[M]^+$. $C_{12}H_7F_5N_2O_5S_2$. Calculated: M 417.972.

**7-Nitro-5-trifluoromethylthiobenzothiazolyl-2-hydroxamic Acid N-Oxide (12).** A mixture of hydroxylamine hydrochloride (0.11 g, 1.63 mol) and sodium hydroxide (0.13 g) in methanol was added with stirring to a solution of **9b** (0.5 g, 1.36 mmol) in methanol. Stirring was continued for about 5 h. The solvent was evaporated and the residue was dissolved in water. The solution was acidified by adding hydrochloric acid. The precipitate formed was filtered off, washed with water and then methanol, and dried to give **10** as orange crystals in 51% yield; mp 229-231°C. IR spectrum (vaseline oil), ν, $cm^{-1}$: 3450, 3150 (OH, NH), 1650 (C=O). $^1$H NMR spectrum (DMSO-$d_6$), δ, ppm: 8.54 (1H, s, Ar); 8.53 (1H, s, Ar); 11.83 (1H, br. s, NH). $^{19}$F NMR spectrum, δ, ppm: -42.45. Mass spectrum, *m/z* (*I*, %): 355 ($[M]^+$, 2), 339 ($[M - O]^+$, 3), 323 (12), 305 (14), 295 (15), 280 (53), 275 (27), 250 (35), 234 (29), 206 (16). Found: *m/z* 354.955 $[M]^+$. $C_9H_4F_3N_3O_5S_2$. Calculated: M 354.954.

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