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Reaction of 2,3-Epoxyoctafluorobutane with 2-Aminobenzenethiol

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Abstract—The reaction of 2,3-epoxyoctafluorobutane with 2-aminobenzenethiol in *N*,*N*-dimethylacetamide gave 3-(2-aminophenylsulfanyl)-1,1,1,3,4,4,4-heptafluorobutane-2,2-diol. In the reaction of the same compounds in dioxane, 2,3-bis(trifluoromethyl)-3,4-dihydro-2*H*-1,4-benzothiazin-2-ol was formed as a result of primary attack by the amino group in 2-aminobenzenethiol on the epoxy ring. The same product was obtained by treatment with 2-aminobenzenethiol of 2,3-bis(trifluoromethyl)-2*H*-1,4-benzoxazin-2-ol which was synthesized from 2,3-epoxyoctafluorobutane and 2-aminophenol.

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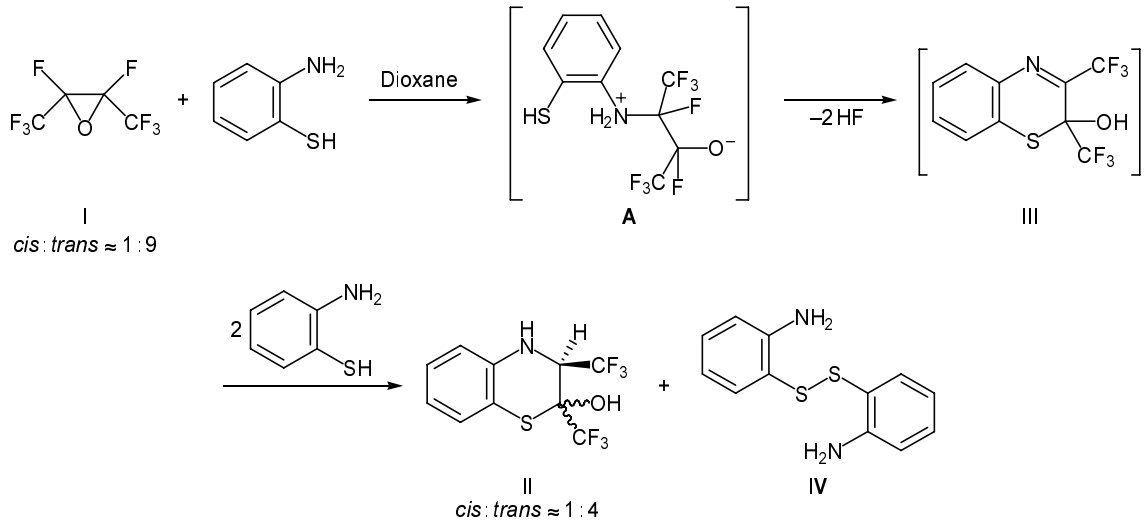
Compounds of the thiazine series are known to exhibit physiological activity and are used for the treatment of mental disorders, in particular schizophrenia [1]. At present, heterocyclic compounds containing perfluoroalkyl groups attract specific interest, for introduction of fluorinated substituents enhances the activity of medical agents and favors their prolonged action, thus improving their efficiency. Most known methods for the synthesis of perfluoroalkyl- and perfluoroaryl-containing thiazines are based on reactions of fluorinated precursors with 2-aminoethanethiol and 2-aminobenzenethiol; however, these methods are limited to a few examples. The reaction of ethyl pentafluorobenzoylpyruvate copper complex with 2-aminobenzenethiol hydrochloride was reported to give 3-pentafluorobenzoylmethylidene-3,4-dihydro-2*H*-1,4-benzothiazin-2-one [2]. 4-Ethoxy-1,1,1-trifluorobut-3-en-2-one reacted with 2-aminobenzenethiol, yielding 2-trifluoroacetyl-2*H*-1,4-benzothiazine, while in the reaction with 2-aminoethanethiol 5,6-dihydro-2-trifluoroacetyl-4*H*-1,4-tiazine was formed [3]. It is also known that perfluoroalkyl-substituted benzothiazines can be synthesized from terminal perfluoroolefin oxides. For example, Ishikawa and Sasaki [4] obtained 2-fluoro-2-trifluoromethyl-3,4-dihydro-2*H*-1,4-benzothiazin-3-one by reaction of 1,2-epoxyhexafluoropropane with 2-aminobenzenethiol in *N*,*N*-dimethylacetamide.

We previously showed that five- and six-membered N,O,S-heterocycles can be synthesized by reactions of epoxy derivatives of internal perfluoroolefins with difunctional nucleophiles, such as ethylenediamine, 2-aminoethanol, thiourea, thiosemicarbazide, thiosemicarbazones derived from carbonyl compounds, *o*-phenylenediamine, and 2-aminophenol [5–8]. However, reactions of internal perfluoroolefin epoxides with difunctional nucleophiles having a thiol group were not studied.

With a view to build up a new type of benzothiazines possessing two perfluoroalkyl substituents, in the present work we examined the reaction of 2,3-epoxyoctafluorobutane (**I**) (*cis*/*trans* ratio ~1:9) [9] with 2-aminobenzenethiol. The reaction was carried out in aprotic solvents differing in their polarity (dioxane and *N*,*N*-dimethylacetamide) to estimate solvent effect on the direction of nucleophilic attack and heterocyclization process.

Unlike 1,2-epoxyhexafluoropropane which reacted with 2-aminobenzenethiol in *N*,*N*-dimethylacetamide (DMA) to give 2-fluoro-2-trifluoromethyl-3,4-dihydro-2*H*-1,4-benzothiazin-3-one as a result of primary nucleophilic attack by the SH group of 2-aminobenzenethiol [4], analogous reaction of internal epoxide, 2,3-epoxyoctafluorobutane (**I**) with both excess and insufficient amount of the nucleophile in dioxane at

Scheme 1.



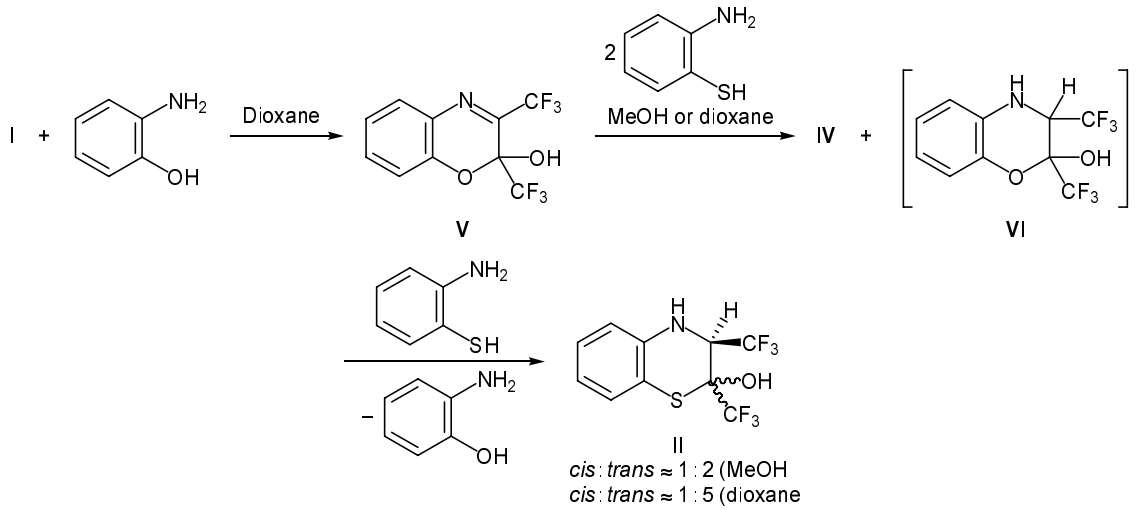
elevated temperature (sealed ampule, ~100°C) led to the formation of an unexpected product, 2,3-bis(trifluoromethyl)-3,4-dihydro-2*H*-1,4-benzothiazin-2-ol (**II**) (*cis*/*trans* ratio ~1:4; Scheme 1). Obviously, the reaction begins with attack by the amino group of 2-aminobenzenethiol at one carbon atom of the oxirane ring. A probable reason is that the SH group is solvated by dioxane to a stronger extent, as compared to the amino group; as a result, no charged S-centered nucleophile is present in the reaction mixture. The process is likely to involve intermediate formation of zwitterionic species **A** and 2,3-bis(trifluoromethyl)-2*H*-1,4-benzothiazin-2-ol (**III**); compound **III** possesses a C=N bond and is reduced to dihydrobenzothiazine **II** with 2-aminobenzenethiol, while the latter is oxidized to 2,2'-diaminodiphenyl disulfide (**IV**).

The *trans* isomer of **II** was isolated by column chromatography, followed by recrystallization, and its structure was established on the basis of its ^{1}H, ^{19}F, and ^{13}C NMR, IR, and mass spectra and elemental composition. The ^{19}F NMR spectrum of a solution of **II** in $CDCl_3$ contained a doublet of quartets at δ_F 92.6 ppm, which was assigned to the 3-CF_3 group ($^{3}J_{FH}$ = 6.9, $^{5}J_{FF}$ = 4.3 Hz), and a quartet at δ_F 83.3 ppm due to fluorine atoms in the 2-CF_3 group ($^{5}J_{FF}$ = 4.3 Hz). Splitting of the first of these signals into a doublet and the corresponding spin–spin coupling constant indicate that the trifluroromethyl group and hydrogen atom are attached to the same carbon atom, and the weak coupling between the CF_3 groups is typical of their *trans* arrangement with respect to each other [6]. In the ^{1}H NMR spectrum of *trans*-**II** ($CDCl_3$), we observed signals from protons in the fused benzene ring [δ, ppm: 6.76 d.d (1H, 5-H, J = 7.4, 1.1 Hz), 6.86 d.d.d (1H, 7-H, J = 8.1, 7.1, 1.1 Hz), 7.04–7.08 m (2H, 6-H, 8-H)] and those belonging to the OH and NH protons (δ 3.56 s and 4.45 br.s, respectively) and 3-H (δ 4.28 ppm, q, $^{3}J_{FH}$ = 6.9 Hz). The ^{19}F NMR spectrum ($CDCl_3$) of **II** before recrystallization contained an additional set of signals which were attributed to the *cis* isomer, δ_F, ppm: 90.7 q.d (3F, 3-CF_3, $^{5}J_{FF}$ = 9.6, $^{3}J_{FH}$ = 6.9 Hz), 86.4 q (3F, 2-CF_3, $^{5}J_{FF}$ = 9.6 Hz).

Comparison of our present results with those obtained previously [8] led us to conclude that the reaction of oxirane **I** with 2-aminobenzenethiol in dioxane occurs in a way similar to the reaction with 2-aminophenol: in both cases, the reaction direction is determined by primary nucleophilic attack by the amino group of the difunctional nucleophile. However, the reaction with 2-aminophenol yields 2,3-bis(trifluoromethyl)-2*H*-1,4-benzoxazin-2-ol (**V**) having a C=N bond, whereas analogous 2,3-bis(trifluoromethyl)-2*H*-1,4-benzothiazin-2-ol (**III**) is not the final product in the reaction of **I** with 2-aminobenzenethiol; As with diazenecarboxamides possessing an N=N bond [10], 2-aminobenzenethiol readily reduces the endocyclic C=N bond in **III** to give benzothiazine **II** and disulfide **IV** (Scheme 1).

To obtain an additional proof for the proposed reaction scheme we made an attempt to reduce the C=N bond in benzoxazine **V** [8] (as a structural analog of intermediate **III**) by the action of 2-aminobenzenethiol. In fact, the formation of 2,3-bis(trifluoromethyl)-3,4-dihydro-2*H*-1,4-benzoxazin-2-ol (**VI**) (Scheme 2) was detected by GC–MS analysis of the reaction mixture, but the major product was again benzothiazine **II**;

Scheme 2.



presumably, it was formed via subsequent nucleophilic replacement of the 2-aminophenol moiety in **VI** by 2-aminobenzenethiol fragment.

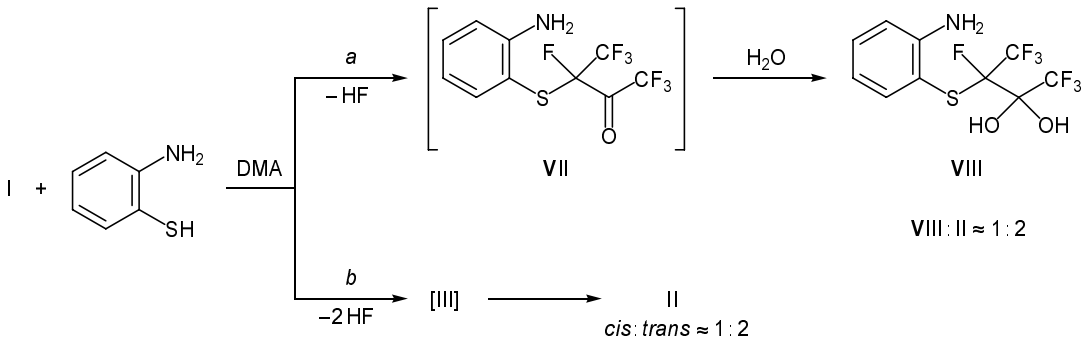
The reaction of oxirane **I** with 2-aminobenzenethiol in more polar *N*,*N*-dimethylacetamide was carried out under similar conditions (sealed ampule, ~100°C), but a tarry mixture of products was obtained which was difficult to separate. We found that the reaction gives mainly 1,1,1,3,4,4,4-heptafluoro-3-(2-aminophenylsulfanyl)-butan-2-one (**VII**) which was identified by ^{19}F and ^{1}H NMR spectroscopy and GC–MS analysis as hydrate **VIII** (Scheme 3, pathway *a*). As in the reaction of 1,2-epoxyhexafluoropropane with 2-aminobenzenethiol [4], compound **VII** is likely to be formed as a result of primary attack by the thiol group which in DMA solution gives rise to solvent-separated ion pair $-S^{-}\ H^{+}$. However, intramolecular cyclization of **VII** at the carbonyl group does not occur (cf. [4]); here, 2-aminobenzenethiol acts as a monofunctional nucleophile. Presumably, solvation of both the carbonyl group and the amino group in **VII** by strongly polar DMA hampers intramolecular nucleophilic attack.

Analysis of the reaction mixture by ^{19}F NMR spectroscopy showed that only a small part of initial oxirane **I** (~15–20%) reacts with 2-aminobenzenethiol in DMA along pathway *b* with formation of benzothiazine **II** (Scheme 3), in contrast to the reaction in dioxane (Scheme 1). Compound **III** was detected in the product mixture by GC–MS analysis; in combination with the above results, this confirms the proposed scheme for the formation of compound **II** [Schemes 1 and 3 (pathway *b*)]. Unidentified products obtained in the reaction of oxirane **I** with 2-aminobenzenethiol in DMA are likely to be oligomers formed from oxirane **I** due to the presence of fluoride ion in the reaction mixture [11].

It is interesting that oxirane **I** reacted with 2-aminobenzenethiol in DMA at a lower temperature (sealed ampule, ~40–50°C); however, under these conditions the yield of oligomeric products was greater.

We can conclude that the reaction direction of 2,3-epoxyoctafluorobutane with 2-aminobenzenethiol is determined by solvation of the SH and NH_2 groups in the difunctional nucleophile, depending on the sol-

Scheme 3.



vent polarity. The reaction in dioxane gives 2,3-bis(trifluoromethyl)-3,4-dihydro-2*H*-1,4-benzothiazin-2-ol in a high yield as a result of primary attack by the amino group of 2-aminobenzenethiol at the oxirane carbon atom, followed by reduction of the C=N bond in intermediate 2,3-bis(trifluoromethyl)-2*H*-1,4-benzothiazin-2-ol. In going from dioxane to more polar *N*,*N*-dimethylacetamide (which is capable of stabilizing ion pairs), primary attack by the SH group of the nucleophile becomes the main reaction pathway leading to 1,1,1,3,4,4,4-heptafluoro-3-(2-aminophenylsulfanyl)-butane-2,2-diol; under these conditions, 2,3-bis(trifluoromethyl)-3,4-dihydro-2*H*-1,4-benzothiazin-2-ol is formed as a minor product.

EXPERIMENTAL

The ^{1}H, $^{13}C-\{^{1}H\}$, and ^{19}F NMR spectra were recorded on a Bruker DRX-400 spectrometer at 400, 100, and 376 MHz, respectively; the chemical shifts were measured relative to tetramethylsilane (^{1}H, ^{13}C) or hexafluorobenzene (^{19}F) as internal reference; $CDCl_3$ was used as solvent. The mass spectra (electron impact, 70 eV) were obtained on a Fisions GC–MS system (MD 800 detector; HP-5 capillary column, 25 m × 0.25 mm, film thickness 0.25 μm; carrier gas helium. The IR spectra (400–4000 cm^{-1}) were recorded from samples dispersed in mineral oil on a Perkin–Elmer Spectrum One FT-IR instrument. The elemental compositions were determined using a Perkin–Elmer 2400 analyzer. Thin-layer chromatography was performed on Silufol UV-254 plates, and silica gel L (100–250 μm) was used for column chromatography. 2,3-Epoxyoctafluorobutane (**I**) was synthesized according to the procedure described in [9]. The product ratios were determined from signal intensities in the ^{19}F NMR spectra.

Reaction of 2,3-epoxyoctafluorobutane (I) with 2-aminobenzenethiol. *a*. A glass ampule was charged with 1.2 g (5.56 mmol) of compound **I**, 2.8 g (22.4 mmol) of 2-aminobenzenethiol, and 10 ml of dioxane, and the ampule was sealed and heated for 23 h on a boiling water bath with intermittent shaking. When the reaction was complete, the ampule was cooled to –70°C and opened, the precipitate of 2-aminobenzenethiol hydrofluoride was filtered off, the filtrate was poured into 200 ml of ice water, and the precipitate was filtered off and dried at room temperature. According to the TLC (chloroform–methanol, 10:0.5) and ^{19}F and ^{1}H NMR data, the product (3.4 g) contained benzothiazine **II** (R_f 0.36, *cis*/*trans* ratio ~1:4) and disulfide **IV** (R_f 0.74). It was extracted with hot hexane, the extract was dried over $MgSO_4$ and evaporated, and the solid residue (containing mainly compound **II** with an impurity of disulfide **IV**) was subjected to column chromatography on silica gel using chloroform–methanol (10:0.5) as eluent. Compound **II** was additionally recrystallized first from hexane and then from hexane–benzene (10:0.5); disulfide **IV** was recrystallized from hexane–chloroform (10:1). We thus isolated 0.55 g (33%) of the *trans* isomer of **II** as colorless crystals and disulfide **IV** as yellow crystals.

2,3-Bis(trifluoromethyl)-3,4-dihydro-2*H*-1,4-benzothiazin-2-ol (II). *trans* Isomer. mp 71–73°C. IR spectrum, ν, cm^{-1}: 1570 (C=C); 2640, 2710, 3320, 3360, 3410 (NH, OH). ^{1}H NMR spectrum, δ, ppm: 3.56 s (1H, OH), 4.28 q (1H, 3-H, $^{3}J_{HF}$ = 6.9 Hz), 4.45 br.s (1H, NH), 6.76 d.d (1H, 5-H, J = 7.4, 1.1 Hz), 6.86 d.d.d (1H, 7-H, J = 8.1, 7.1, 1.1 Hz), 7.04–7.08 m (2H, 6-H, 8-H). ^{19}F NMR spectrum, δ_F, ppm: 83.3 q (3F, 2-CF_3, $^{5}J_{FF}$ = 4.3 Hz), 92.6 d.q (3F, 3-CF_3, $^{3}J_{HF}$ = 6.9, $^{5}J_{FF}$ = 4.3 Hz). ^{13}C NMR spectrum, δ_C, ppm: 56.5 q (C^3, $^{2}J_{CF}$ = 29.3 Hz), 80.2 q (C^2, $^{2}J_{CF}$ = 31.6 Hz), 115.7 s (C^{8a}), 116.7 s (C^5), 121.4 s (C^7), 123.4 q (CF_3, $^{1}J_{CF}$ = 285.9 Hz), 123.5 q (CF_3, $^{1}J_{CF}$ = 284.8 Hz), 126.6 s and 126.8 s (C^6, C^8), 137.0 s (C^{4a}). Mass spectrum, *m*/*z* (I_{rel}, %): 304 (7.7) $[M+1]^+$, 303 (88.1) $[M]^+$, 234 (18.4) $[M - CF_3]^+$, 216 (10.4) $[M - CF_3 - H_2O]^+$, 207 (8.5) $[M - C_6H_4 - HF]^+$, 206 (100) $[M - CF_3CHNH]^+$, 204 (8.4), 184 (11.0) $[M - C_2F_5]^+$, 174 (10.4) $[M - CF_3CSO]^+$, 166 (13.2), 165 (14.3) $[M - 2CF_3]^+$, 150 (14.2), 146 (15.1) $[M - 2CF_3 - H_2O - H]^+$, 142 (8.9) $[M - C_6H_4NH_2 - CF_3]^+$, 137 (9.6), 136 (84.3) $[M - 2CF_3 - COH]^+$, 109 (27.0), 108 (7.9) $[C_6H_4S]^+$, 104 (16.4) $[C_6H_4NHCH]^+$, 96 (10.5), $[CF_3CNH]^+$, 93 (8.8) $[C_6H_5NH_2]^+$, 77 (10.1) $[C_6H_5]^+$, 69 (29.7) $[CF_3]^+$. Found, %: C 39.6; H 2.3; F 37.7; N 4.3; S 10.2. $C_{10}H_7F_6NOS$. Calculated, %: C 39.6; H 2.3; F 37.6; N 4.6; S 10.6.

cis Isomer **II**. ^{19}F NMR spectrum, δ_F, ppm: 86.4 q (3F, 2-CF_3, $^{5}J_{FF}$ = 9.6 Hz), 90.7 q.d (3F, 3-CF_3, $^{5}J_{FF}$ = 9.6, $^{3}J_{FH}$ = 6.9 Hz).

2,2'-Diaminodiphenyl disulfide (IV). mp 90–91°C; published data [12]: mp 91.5–92.5°C. IR spectrum, ν, cm^{-1}: 1555, 1570, 1600, 1610 (C=C, NH); 3165, 3190, 3285, 3365 (NH). ^{1}H NMR spectrum, δ, ppm: 4.28 br.s (4H, NH_2), 6.58 m (2H, CH), 6.71 m (2H, CH), 7.15 m (4H, CH). Found, %: C 58.2; H 4.8;

N 11.2; S 26.0. $C_{12}H_{12}N_2S_2$. Calculated, %: C 58.1; H 4.8; N 11.3; S 25.8.

b. The reaction was carried out as described above in *a* with 5.2 g (24.07 mmol) of compound **I** and 3.0 g (24 mmol) of 2-aminobenzenethiol in 5 ml of *N*,*N*-dimethylacetamide. The mixture was poured into ice water (200 ml), and the bottom (organic) layer was separated, washed with water, and dried (~40°C). According to the IR, ^{19}F and ^{1}H NMR, and GC–MS data, the tarry residue, 7.0 g, was a mixture of compound **VIII** and benzothiazine **II** (*cis*/*trans* ratio ~1:2) at a ratio of ~4:1 and a small amount of unidentified products.

1,1,1,3,4,4,4-Heptafluoro-3-(2-aminophenylsulfanyl)butane-2,2-diol (VIII). IR spectrum, ν, cm^{-1}: 1586 (C=C); 1610 (NH); 3070, 3180, 3390 br, 3480 sh (NH, OH). ^{1}H NMR spectrum, δ, ppm: 3.8 br.s (4H, NH_2, OH), 6.6 m (1H, H_{arom}), 6.7 m (1H, H_{arom}), 7.2 m (2H, H_{arom}). ^{19}F NMR spectrum, δ_F, ppm: 22.5 q.q (1F, $CF_3C\mathbf{F}$, $^3J_{FF} = {}^4J_{FF} = 11.2$ Hz), 84.5 d.q [3F, $CF_3C(OH)_2$, $^4J_{FF} = 11.2$, $^5J_{FF} = 9.5$ Hz], 90.1 d.q (3F, $C\mathbf{F}_3CFS$, $^3J_{FF} = 11.2$, $^5J_{FF} = 9.5$ Hz). Mass spectrum, *m*/*z* (I_{rel}, %): 322 (8.2) $[M - OH]^+$, 321 (89.8) $[M - H_2O]^+$, 252 (38.7) $[M - H_2O - CF_3]^+$, 235 (10.5) $[M - OH - H_2O - CF_3]^+$, 234 (11.0), 233 (7.2), 232 (54.7) $[M - H_2O - HF - CF_3]^+$, 205 (9.4), 204 (100) $[M - H_2O - HF - CF_3CO]^+$, 189 (6.1), 188 (40.2), 185 (9.0), 184 (61.7), 172 (14.5), 168 (5.5), 162 (8.6), 154 (14.7) $[C_6H_4(NH)SCF]^+$, 151 (5.5), 150 (12.9), 135 (6.7) $[C_6H_4(NH)SC]^+$, 124 (8.9) $[C_6H_4(S)NH_2]^+$, 123 (6.8), 122 (7.7), 120 (27.3) $[C_6H_4SC]^+$, 109 (12.4), 108 (8.3) $[C_6H_4S]^+$, 102 (7.2), 96 (19.0), 95 (9.6), 93 (6.3), 92 (22.7) $[C_6H_4NH_2]^+$, 91 (7.7), 80 (6.7), 77 (14.7) $[C_6H_5]^+$, 70 (7.6), 69 (40.4) $[CF_3]^+$.

2,3-Bis(trifluoromethyl)-2*H*-1,4-benzothiazin-2-ol (III). Mass spectrum, *m*/*z* (I_{rel}, %): 301 (3.1) $[M]^+$, 205 (9.3) $[M - CF_3CNH]^+$, 204 (100) $[M - CF_3CO]^+$, 185 (8.8) $[M - CF_3COF]$, 184 (69.4), 162 (5.6) $[M - H - 2CF_3]^+$, 135 (21.6) $[M - 2CF_3 - CO]^+$, 134 (7.1) $[M - 2CF_3 - COH]^+$, 108 (15.3) $[C_6H_4S]^+$, 102 (12.9) $[C_6H_4NC]^+$, 69 (31.2) $[CF_3]^+$.

Reaction of 2,3-bis(trifluoromethyl)-2*H*-1,4-benzoxazin-2-ol (V) with 2-aminobenzenethiol. *a*. A mixture of 0.3 g (1.05 mmol) of compound **V** and 0.4 g (3.2 mmol) of 2-aminobenzenethiol in 5 ml of methanol was placed in an ampule, and the ampule was sealed and heated for 36 h on a boiling water bath with intermittent shaking. The ampule was cooled and opened, the mixture was poured into 100 ml of water, and the bottom (organic) layer was separated and dried at ~40°C. The solid material thus isolated was extracted with chloroform, and the undissolved material was dried at room temperature and recrystallized from aqueous methanol to obtain 0.1 g (87%) of 2-aminophenol as light yellow crystals with mp 172–173°C; published data [13]: mp 174°C. The extract was dried over $MgSO_4$ and evaporated. According to the ^{19}F and ^{1}H NMR and GC–MS data, the solid residue, 0.58 g, contained 2,3-bis(trifluoromethyl)-3,4-dihydro-2*H*-1,4-benzothiazin-2-ol (**II**, *cis*/*trans* ratio ~1:2), disulfide **IV**, traces of **VI**, and unidentified products.

2,3-Bis(trifluoromethyl)-3,4-dihydro-2*H*-1,4-benzoxazin-2-ol (VI). Mass spectrum, *m*/*z* (I_{rel}, %): 287 (36.4) $[M]^+$, 286 (6.4) $[M - H]^+$, 218 (17.3) $[M - CF_3]^+$, 190 (100) $[M - CF_3CHNH]^+$, 170 (8.2) $[M - CF_3 - HF - CO]^+$, 162 (17.3), 150 (6.4), 149 (22.3) $[M - 2CF_3]^+$, 136 (18.3) $[M - CFCH - CF_3]^+$, 120 (53.9) $[M - 2CF_3 - COH]^+$, 104 (14.5) $[C_6H_4NHCH]^+$, 97 (11.8) $[CF_3CHNH]^+$, 95 (10.0) $[CF_3CN]^+$, 91 (18.3) $[C_6H_4NH]^+$, 79 (11.8), 69 (42.7) $[CF_3]^+$, 52 (20.0), 50 (11.8).

b. The reaction was performed as described above in *a* with 0.2 g (0.7 mmol) of compound **V** and 0.27 g (2.16 mmol) of 2-aminobenzenethiol in 6 ml of dioxane. We isolated 0.065 g (86%) of 2-aminophenol (mp 172°C) and 0.3 g of a mixture of compound **II** (*cis*/*trans* ratio ~1:5), disulfide **IV**, and a small amount of unidentified products (according to the ^{1}H and ^{19}F NMR and GC–MS data).

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