# The Reaction of Benzenesulfenanilides with Lewis Acids: Involvement of Radical Cation Intermediates.

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Abstract. The 4'-substituted N-methylbenzenesulfenanilides 1a-c react with Lewis acids, including BF<sub>3</sub>, AlCl<sub>3</sub> and GaCl<sub>3</sub>, to afford the radical cation intermediates 9a-c, some of which could be detected by e.p.r. spectroscopy. Thus, the 4'-methoxy substituted compound 1a gave the fairly persistent radical cation 9a. In contrast, the radical cation 9b, derived from the 4'-nitro substituted compound 4b, was not detected apparently because it decayed too rapidly forming the very stable radical cation 11b, intermediate in the formation of the rearranged sulfide 10b. The radical intermediates 9a-c react with cyclohexene to give the 1,2-adducts 4a-c, 5 and 6 which are believed to be formed from either the thiiranium ion 7b or the sulfurane 8a,c.

In recent years the reactions of benzenesulfenanilides (BSA) promoted by protic acids and Lewis acids have been the object of considerable interest<sup>1</sup>. In particular, it has been reported that boron trifluoride transforms 4'-nitrobenzenesulfenanilide (NBSA) into a highly reactive 'sulfenyl transfer' agent which can undergo electrophilic displacement at sulfur by several nucleophiles<sup>2</sup>. Thus, NBSA can be considered to be a phenylthio cation synthon and has been widely employed for this purpose in the 1,2-functionalization of alkenes and alkynes<sup>3</sup>.

We recently reported<sup>4</sup> that the reaction of NBSA with Lewis acids, such as BF<sub>3</sub> and AlCl<sub>3</sub>, leads to the formation of the thioarninyl radicals, ArNSPh (Ar = 4-NO<sub>2</sub>C<sub>6</sub>H<sub>4</sub>), and several lines of evidence suggested that these radicals were in rapid equilibrium with the 'sulfenyl transfer' species. In fact, when the Lewis acid promoted reaction of NBSA was examined, the persistent e.p.r. signal was found to be immediately quenched by the addition of nucleophiles, such as alkenes, alkynes or anisole and, if the thioarninyls radicals were scavenged with Bu<sub>3</sub>SnH, the electrophilic addition of NBSA to carbon-carbon double or triple bonds was inhibited.

We suggested that a radical cation formed by a electron transfer between NBSA and the Lewis acid was the 'sulfenyl transfer' agent. However, no direct evidence was obtained for this radical cation which, if formed, would be expected to loose the amino proton or the phenylthio group by reaction with a base or a nucleophile, respectively, rather rapidly. (Scheme1).

In order to obtain direct evidence for the radical cation nature of the 'sulfenyl transfer' intermediates involved in reactions between BSAs and Lewis acids, we have now undertaken a chemical and spectroscopic study on the reactivity of 4'-substituted N-methylbenzenesulfenanilides **1a-c**. In these cases, the potential radical cations are expected either to undergo a rapid sulfenyl transfer or, if sufficiently persistent, to be directly detectable by e.p.r. spectroscopy.



Scheme 1. Reagents : i, BF3·Et2O; ii, - H<sup>+</sup>; iii, + Nu<sup>-</sup>.

## **RESULTS AND DISCUSSION**

The 4'-methoxy-N-methylbenzenesulfenanilide 1a was reacted at room temperature in benzene solution with 1.5 molar equiv. of boron trifluoride-diethyl ether complex for 10 min.; the reaction mixture was neutralized with aqueous sodium carbonate and then chromatographed; diphenyl disulfide 2 was obtained in roughly quantitative yield, together with N-methyl-p-anisidine 3a (ca. 40%) and unidentifiable coloured, tarry products. Similar results were obtained when the reaction was carried out in acetonitrile under the same conditions. The formation of the disulfide 2 and aniline 3a was in agreement with the general behaviour exhibited by the BF<sub>3</sub>-promoted reactions of BSAs when carried out in the absence of nucleophilic reagents<sup>1</sup>. The mechanism of this reaction is still unknown.

When the BF<sub>3</sub>-promoted reaction of **1a** was carried out in the presence of cyclohexene, work-up and subsequent column chromatography led to the isolation of the addition products **4a**, **5**, and **6** together with major amounts of **2** and **3a**. According to previous work<sup>1,3</sup>, the formation of these adducts can be inferred to an initial BF<sub>3</sub>-promoted sulfenyl transfer from **1a** to the alkene double bond and subsequent capture of the resulting electrophilic intermediate, **7a** or **8a**. Thus, nucleophilic attack by the aniline **3a** and by the disulfide **2** would eventually lead to the amino sulfide **4a** and the bis-sulfide **5**, respectively. The cyclohexanol **6** was probably formed under the hydrolytic conditions employed during work-up (see Scheme 2). However, the observed yield of adducts **4a** and **5** had a ratio of 20:80, which does not parallel the relative nucleophilicities of the aniline **3a** and the disulfide **2**. The overall yield of the **1**,2-adducts **4a**, **5**, and **6** (ca. 30%) is quite low when compared with related reactions involving other BSAs, which can afford up to 100% of addition products<sup>1,3</sup>. This suggested that the 'sulfenyl transfer' agent formed from **1a** has little tendency to undergo

nucleophilic displacement at sulfur, which is consistent with the expected stabilizing effect of the two electronreleasing substituents, i.e. the 4'-methoxy and N-methyl groups.

When the reaction of 1a with  $BF_3$  was carried out in acetonitrile directly in the cavity of the e.p.r. spectrometer, a strong signal of a persistent radical was detected. (Fig.1a) Analysis of the spectrum, characterized by several sets of coupling constants, and its computer simulation (Fig. 1b) served to identify it as arising from the radical cation 9a (see Table 1).



Fig. 1 (a) EPR spectrum of 9a in acetonitrile, recorded at room temperature. (b) Computer simulation.

The spectrum exhibited an alternating line width effect at room temperature, most likely due to two concomitant restricted rotations about the C(1)-N and C(4)-O bonds. The energy barriers, previously reported for related radical species<sup>5</sup>, lie in the range of 8-12 Kcal/mol.. To verify the assignment of the h.f.c. to radical **9a**, we synthetized the benzenesulfenanilide **1a**-d<sub>5</sub> with the deuterated phenylthio group. The BF<sub>3</sub>-promoted reaction of **1a**-d<sub>5</sub> gave a radical species which shown an e.p.r. spectrum with a h.f.c. pattern identical to that of the non-deuterated **9a**, which proved that there was no unpaired electron density on the phenylthio group.

Table 1. Hyperfine coupling constants of radical cation 9a (Gauss)



 $a^{a}a(H^{2,6}) = [a (H^{2}) + a (H^{6})]/2$ ; because of the alternating line width effect the exact coupling constants cannot be assigned.

The boron trifluoride-promoted reaction of 4'-nitro-N-methylbenzenesulfenanilide 1b gave results somewhat different from those obtained from 1a. The reactions carried out in benzene or acetonitrile gave, besides the disulfide 2 and the aniline 3b, the rearranged sulfide 10b, with higher yield in benzene than in acetonitrile. Moreover, in the former solvent the rate of disappearance of the anilide 1b was slower and noticeable amount (ca. 30%) of starting material was recovered after the usual work-up and column chromatography. This solvent effect can be explained by reasonable assumption that in the more polar solvent acetonitrile an ionic 'sulfenyl transfer intermediate is effectively stabilize, thus favouring its formation but disfavouring the electrophilic intramolecular *ortho*-sulfenylation leading to the rearranged sulfide 10b.



Scheme 2. Reagents and Conditions: i, +  $BF_3$ , -  $BF_3^2$ , room temperature; ii, cyclohexene; iii, - ArNMe; iv, + 3, -  $H^+$ ; v, + e<sup>-</sup>; vi, + 2, - 1; vii, +  $H_2O$ , - 3.

When the BF<sub>3</sub>-promoted reaction of 1b was carried out in benzene in the presence of cyclohexene, we observed the rapid disappearance of starting anilide and the concomitant exclusive formation of the adduct 4b in quantitative yield, at the expense of the disulfide 2. Both findings were showing that reaction of the anilide 1b with boron trifluoride is reversable and that the 'sulfenyl transfer' agent is a highly reactive species, as would be expected in view of the electron-withdrawing 4'-nitro-substituent. In agreement with this observed high reactivity, experiments performed directly in the e.p.r. spectrometer cavity did not show any trace of the

radical 9b. Instead, we detected a very stable radical cation, 11b, (Fig. 2a and 3a) which reasonably arose from the initially formed 9b by intramolecular attack of the electrophilic sulfur at a *ortho*-position of the anilino ring. Actually, the radical cation 11b can be considered the precursor of the sulfide 10b, obtained by 1,3-proton transfer and concomitant N-S bond fission. (see Scheme 2).

In repeated experiments, reactions of the anilides 1a,b were also carried out with both AlCl<sub>3</sub> and GaCl<sub>3</sub>, yielding just the same radical species that were obtained in the BF<sub>3</sub>-promoted reactions.



Fig. 2 (a) EPR spectrum recorded in acetonitrile at room temperature of the radical cation 11b. (b) Computer simulation.

Table 2. Hyperfine coupling constants of radical cation 11a (Gauss)<sup>a</sup>



<sup>a)</sup> In brackets the coupling constants of the corresponding radical with the deuterated phenylthio group are reported. <sup>b)</sup>These coupling constants can not unambigously assigned to these positions. <sup>c)</sup> $a(H^2)$  and  $a(H^6)$  can be exchanged.



Fig. 3 EPR spectra recorded in acetonitrile at room temperature of (a) radical cation 11b and (b) radical cation 11b-d<sub>5</sub>.

The identification of **11b** is supported by its e.p.r. spectroscopic parameters (Table 2) and the relative computer simulation. (Fig. 2b) In addition, the corresponding benzenesulfenanilide  $1b-d_5$  with the deuterated phenylthio group was synthetized, and on reaction with BF<sub>3</sub> gave a radical cation having a spectrum with a different hyperfine pattern, as would be expected (Fig 3b). The deuterium coupling constants were not measurable as they were smaller than spectral line width.

The chemical reactivity of the parent N-methylbenzenesulfenanilide 1c was intermediate, between that of 1a and 1b, as expected. In particular, the BF<sub>3</sub>-promoted reaction in acetonitrile led to the formation of the disulfide 2 and the aniline 3c, whereas the same reaction carried out in benzene afforded the rearranged sulfide 12 together with 2 and 3,c. Furthermore, when the reaction was performed in the presence of cyclohexene, the 1,2-adducts 4c, 5, 6, and 13 wefe isolated in good overall yield (>80%), which indicates a fairly high reactivity for this 'sulfenyl transfer' agent. The adducts 4c and 13 are easily explained as arising from attack of the anilines 3c and 12, respectively, on the electrophilic intermediate obtained by sulfenyl group transfer to the carbon-carbon double bond. The pattern of the 1,2-adducts obtained from the anilide 1c resembles the pattern obtained with 1a, but is quite different from that exhibited by 1b. This behaviour would suggest the involvement of different reaction intermediates. Thus, we claim that the BF<sub>3</sub>-promoted reaction of the anilide

1b with cyclohexene might actually lead to the thiiranium ion intermediate 7b which rapidly undergoes nucleophilic attack at carbon atom to give the adduct 4b. By contrast, the anilides 4a and 4c might afford mainly the sulfurane intermediates 8a and 8c which can undergo both nucleophilic attack by the disulfide 2 or, after surviving the reaction environment, are hydrolized during work-up to form the cyclohexanol 6. The involvement of sulfurane intermediates is generally accepted in addition of sulferyl halides to alkenes.<sup>6</sup>

In agreement with the rather high reactivity exhibited by the 'sulfenyl transfer' agent formed from 1c, the e.p.r experiments showed the initial formation of a non-persistent radical-cation, possibly 9c, which rapidly decayed. Other persistent radical species were also detected, which appeared to have been formed from byproducts.



In summary, we have shown that the N-methylbenzenesulphenanilides 1a-c react with Lewis acids to give radical cation intermediates 9a-c which, depending on the effect of the 4'-substituent, may survive long enough to be directly detected by e.s.r. spectroscopy.

The overall findings obtained in this and in our earlier work<sup>4</sup> indicate that BSAs reacts with Lewis acids through an initial mono-electron transfer process, leading to conclude that these reactions can not be strictly considered as acid-base Lewis type reactions. However, we wish to point out that a SET process can also account for an acid-base type reaction, if pairing of radical ions occurs before their escape from the solvent cage, leading to the acid-base zwitterionic complex.

#### EXPERIMENTAL SECTION

The N- methylbenzenesulfenanilides 1a, 1b<sup>7</sup>, and 1c were prepared by reaction of benzenesulfenyl chloride with the appropriate anilines 3a, 3b, and 3c, respectively, in 70-90% yield. *N-Methyl-4'methoxybenzenesulfenanilide* 1a had m.p. 66-68 °C;  $\delta_H$  3.43 (3H, s), 3.77 (3H, s), and 6.7-7.3 (9H, m) (Found: M<sup>+</sup>, 245.0880, C<sub>14</sub>H<sub>15</sub>NOS requires M, 245.0874); m/z 245 (25), 137 (10), 136 (100), and 121 (11). *N-Methylbenzenesulfenanilide* 1c had m.p. 57-58 °C;  $\delta_H$  3.40 (3H, s) and 6.9-7.4 (10H, m) (Found: M<sup>+</sup>, 215.0762, C<sub>13</sub>H<sub>13</sub>NOS requires M, 215.0769); m/z 215 (100), 109 (10), 106 (90), 105 (23), 104 (13), 79 (15), and 77 (43). The deuterated sulfenanilides 1a-d<sub>5</sub> [ $\delta_H$  3.43 (3H, s), 3.77 (3H,s), 6.8 (2H, A part of a A<sub>2</sub>B<sub>2</sub> system, J 9 Hz) and 7.2 (2H, B part of a A<sub>2</sub>B<sub>2</sub> system, J 9 Hz); m/z 250 (M<sup>+</sup>, 26) and 136 (100)] and 1b-d5 [ $\delta_H$  3.8 (3H, s), 7.2 (2H, A part of a A<sub>2</sub>B<sub>2</sub> system, J 9 Hz), and 8.2 (2H, B part of a A<sub>2</sub>B<sub>2</sub> system, J 9 Hz); m/z 265 (M<sup>+</sup>, 100), 115 (24), 114 (35), and 105 (40) were similarly prepared from deuterated sulfenyl chloride<sup>8</sup>. Reaction products such as the disulfide 2, the anilines 3a-c, the bis-sulfide  $5^9$ , the cyclohexanol  $6^9$ , and the aminosulfide  $10b^7$  were each identified by spectral comparison with authentic specimens. The homogeneity of the hitherto unknown adducts 4b-c and 13 and aminosulfide 12 was confirmed by TLC analysis.

EPR spectra were recorded on Varian E-104 X-band spectrometer, with 100 KHz modulation. <sup>1</sup>H NMR spectra were measured on Varian 200 (200 MHz) spectrometer, and are for CDCl<sub>3</sub> solutions with SiMe<sub>4</sub> as internal standard. Mass spectra were determined by the electron impsct method on VG 7070 instrument. Column chromatography was carried out on Merck silica gel (0.040-0.063 particle size) by gradual elution with light petroleum (b. p. 40-70 °C)/diethyl ether.

BF3-Promoted Reaction of N-Methylbenzenesulfenanilides 1a-c. General Procedure. To a solution of the benzenesulfenanilide 1a-c (2 mmol.) in the appropriate solvent (20 cm<sup>3</sup>) was added boron trifluoridediethyl ether complex, ca. 47% BF3 (0.38 cm<sup>3</sup>, 3 mmol.) under vigorous stirring at room temperature. After being stirred for 10 min., the reaction mixture was treated with 10% aqueous sodium carbonate and extracted with diethyl ether. The organic layer was then separated, the excess solvent removed and the residue chromatographed.

Reaction of the Anilides 1a-c in acetonitrile. (a) N-Methyl-4'-methoxybenzenesulfenanilide 1a gave (i) diphenyl disulfide 2 (0.97 mmol., 97%); (ii) N-methyl-p-anisidine 3a (0.8 mmol., 40%); and (iii) tarry material.

(b) N-Methyl-4'-nitrobenzenesulfenanilide 1b gave (i) diphenyl disulfide 2 (0.84 mmol., 84%); (ii) N-methyl-2-(phenylthio)-4-nitroaniline 10b (0.4 mmol., 7%); and (iii) N-methyl-4-nitroaniline 3b (1.30 mmol., 65%).

(c) N-Methylbenzenesulfenanilide 1c gave (i) diphenyl disulfide 2 (0.98 mmol., 98%); (ii) N-methylaniline 3c (1.0 mmol., 50%); and (iii) tarry meterial.

Reaction of the Anilides 1a-c in benzene. (a) N-Methyl-4'-methoxybenzenesulfenanilide 1a gave (i) diphenyl disulfide 2 (1.0 mmol., 100%); (ii) N-methyl-p-anisidine 3a (1.0 mmol., 50%); and (iii) tarry material. (b) N-Methyl-4'-nitrobenzenesulfenanilide 1b gave (i) diphenyl disulfide 2 (0.44 mmol., 63%); (ii) unreacted anilide 1b (0.6 mmol., 30%); (iii) N-methyl-2-(phenylthio)-4-nitroaniline 10a (0.18 mmol., 26%); and (iv) N-methyl-4-nitroaniline 3b (0.25 mmol., 36%).

(c) N-Methylbenzenesulfenanilide 1c gave (i) diphenyl disulfide 2 (0.7 mmol., 70%); (ii) N-methyl-4-(phenylthio)aniline 12 (0.60 mmol., 30%), thick oil;  $\delta_H 2.83$  (3H, s), 3.7 (1H, br s), 6.60 (2H, A part of a  $A_2B_2$  system, J 9 Hz), 7.2 (5H, m), and 7.40 (2H, B part of a  $A_2B_2$  system, J 9 Hz) (Found: M<sup>+</sup>, 215.0775.  $C_{13}H_{13}NS$  requires M, 215.0769); m/z 215 (100), 214 (12), 200 (11), 183 (12) and 138 (16); (iii) N-methylaniline 3c (0.90 mmol., 45%); and (iv) tarry material.

Reaction of the anilides 1a-c in benzene/cyclohexene 9:1. (a) N-Methyl-4'-methoxybenzenesulfenanilide 1a gave (i) diphenyl disulfide 2 (0.67 mmol., 67%); (ii) 1,2-bis-(phenylthio) cyclohexane 5 (0.20 mmol.), 20%); (iii) 2-(phenylthio)cyclohexanol 6 (0.12 mmol., 12%); (iv) trans-1-[N-methyl-N-(4methoxyphenyl)]amino-2-(phenylthio)- cyclohexane 4a (0.06 mmol., 6%), solid product which was not recrystalized;  $\delta_{\rm H}$  1.2-2.2 (8H, m), 2.70 (3H, s), 3.28 (1H, dt, J<sub>t</sub> 11.2, J<sub>d</sub> 3.7 Hz), 3.47 (1H, dt, J<sub>t</sub> 11.2, J<sub>d</sub> 3.1 Hz), 6.85 (4H, m), 7.2-7.5 (5H, m) (Found: M<sup>+</sup>, 327.1660. C<sub>20</sub>H<sub>25</sub>NOS requires M, 327.1657); m/z 327 (90), 218 (30), 191 (10), 177 (13), 176 (100), 150 (20), and 81 (18); (v) N-methyl-p-anisidine 3a (0.90 mmol., 45%); and (vi) tarry material.

(b) N-Methyl-4'-nitrobenzenesulfenanilide 1b gave trans-1-[N-methyl-N-(4-nitrophenyl)] amino-2-(phenyl-thio)cyclohexane 4b (1.90 mmol., 95%), m. p. 144-145 °C;  $\delta_{\rm H}$  1.2-2.2 (8H, m), 2.76 (3H, s), 3.3 (1H, m), 3.7 (1H, m), 6.7 (2H, d, J 9 Hz), 7.3 (5H, m), and 8.2 (2H, d, J 9Hz) (Found: M<sup>+</sup>, 342.1408. C<sub>19</sub>H<sub>22</sub>N<sub>2</sub>O<sub>2</sub>S requires M, 342.1402); m/z 342 (55), 325 (13), 233 (36), 191 (100), and 165 (10).

(c) N-Methylbenzenesulfenanilide 1c gave (i) 1,2-bis(phenylthio)cyclohexane 5 (0.03 mmol., 3%); (ii) trans- I-(N-methyl-N-phenyl)amino-2-(phenylthio)cyclohexane 4c (0.58 mmol., 29%); m. p. 68-70 °C;  $\delta_{\rm H}$  1.2-2.2 (8H, m), 2.70 (3H, s), 3.25 (1H, dt, J<sub>t</sub> 11, J<sub>d</sub> 3 Hz), 3.60 (1H, dt, J<sub>t</sub> 11, J<sub>d</sub> 3 Hz), 6.65-6.85 (2H, m), and 7.15-7.35 (8H,m) (Found: M<sup>+</sup>, 297.1545. C<sub>19</sub>H<sub>3</sub>NS requires M, 297.1551); m/z 297 (50), 188 (34), 146 (100), 120 (15), 77 (14); (iii) trans-I-[N-methyl-N-(4-phenylthio)phenyl]amino-2-(phenylthio)cyclohexane 13 (0.20 $mmol., 20%); thick oil; <math>\delta_{\rm H}$  1.2-2.2 (8H, m), 2.70 (3H, s), 3.20 (1H, dt, J<sub>t</sub> 11, J<sub>d</sub> 4 Hz), 3.60 (1H, dt, J<sub>t</sub> 11, J<sub>d</sub> 4 Hz), 6.65 (2H, d, J 9Hz), 7.0-7.3 (10H, m), and 7.40 (2H, d, J 9 Hz) (Found: M<sup>+</sup>, 405.1590. C<sub>25</sub> H<sub>27</sub>NS<sub>2</sub> requires M, 405.1585); m/z 405 (100), 296 (17), 254 (56), 228 (13) 191 (13) and 144 (25); (iv) N-methyl-4-(phenylthio)aniline 12 (0.06 mmol., 3%); (v) 2-(phenylthio)cyclohexanol 6 (0.60 mmol., 30%); and (vi) Nmethylaniline 3c (0.62 mmol., 62%).

EPR Experiments. The experiments were run matching the conditions in the current preparative reaction. A 'U' sample tube, to keep separate the two reactants, was used and the acetonitrile solution of  $BF_3$ ·Et<sub>2</sub>O was deoxygenated with the freeze-thaw technique in a vacuum line. The reagents were mixed directly in the EPR sample tube, at room temperature, and then introduced in the spectrometer cavity. The g-values for the radical species were determined by comparison with the g-value (2.0037) of DPPH.

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