Influence of Redox and Lewis Acid Functions of SbCl$_3$-Rich Molten Salt Catalysts on the Reactions of Coal Model Compounds

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Abstract

Recent studies of the chemistry of coal model compounds in SbCl$_3$-based molten salt catalysts have shown that both the redox and Lewis acid functions of these media can play principal roles in catalyzing reactions for organic substrates. These two catalytic functions are discussed in relation to two reaction types that have been recently discovered in highly purified, anhydrous SbCl$_3$-rich melts. The redox function of the melt is shown to play the dominant catalytic role in a transfer hydrogenation reaction for oxidizable arenes that are selectively hydrogenated by tetralin at only 80°C. The arene radical cation is found to serve as the key organic reactive intermediate. The Lewis acid function of the melt is shown to catalyze selective transalkylation chemistry for the $\alpha,\omega$-diphenylalkanes, C$_6$H$_5$(CH$_2$)$_n$C$_6$H$_5$ [$n = 1-4$] with the catalyst generating a benzylic carbocation as the key reactive intermediate.

Introduction

Molten SbCl$_3$ is one of the metal halide melts that has been reported to be an effective, selective catalyst for hydrocracking coal (1). From our extensive fundamental studies of the chemistry of coal model compounds (e.g. aromatics, hydroaromatics, and $\alpha,\omega$-diphenylalkanes) (2-7), the SbCl$_3$-based molten salt catalysts have exhibited substantial flexibility particularly with regard to tuning the strength of the catalytic functions. This ability to alter the strength of the catalytic functions in a reproducible manner results in a catalyst system in which not only reaction rates can be controlled but also product selectivities.

Earlier research concentrated on the chemistry of individual polycyclic aromatic hydrocarbons (arenes) in the SbCl$_3$-based melts (2-4). These arenes were activated by the redox function of the catalytic medium, and arene radical cations were found to be key intermediates. Tuning the strength of the redox function of the melt was found to dramatically alter reaction rates and product selectivities.
In the present description of the melt catalyzed transfer hydrogenation reaction, the versatility of these SbCl$_3$-based melts is further illustrated by the ability to activate one organic substrate (a polycyclic arene) in the melt in the presence of a second unactivated substrate (tetralin) and observe the resulting reaction.

The $\alpha,\omega$-diphenylalkanes, on the other hand, are not capable of being oxidized to radical cations in the SbCl$_3$-based melts, and the transalkylation chemistry observed results from activation by the Lewis acid function of the melt. Tuning the strength of this catalytic function leads not only to a control over reaction rates, but also provides a valuable means for the investigation and discovery of a novel mechanism for this important organic reaction.

### Catalytic Functions of SbCl$_3$

Molecular SbCl$_3$ is a notoriously weak Lewis acid. However, in the molten state SbCl$_3$ is weakly ionized, as modeled in eq. 1, with

\[
\text{SbCl}_3 \rightleftharpoons \text{SbCl}_2^+ (\text{solv.}) + \text{Cl}^- (\text{solv.})
\]

A reported ionization constant, $K_i$, at 99°C ranging from $3 \times 10^{-11}$ to $2 \times 10^{-8}$ M$^2$ (8,9). The modest Lewis acid function of the melt resides in the chloride deficient antimony cation, SbCl$_2^+$. As indicated in eq. 1 the ions are not present in the melt as discrete entities, but instead are strongly solvated to neutral SbCl$_3$ molecules (or chains) through chlorine bridge bonds as indicated from Raman and electrical conductivity studies (10,11). However, for the purposes of discussing the organic chemistry, it will be sufficient to use SbCl$_2^+$ to model the chloride deficient antimony species that is acting as the Lewis acid in the melts.

The Lewis acidity of the melt is tuned by controlling the concentration of SbCl$_2^+$ in the melts by adding chloride donors or acceptors. Basic melts are formed by adding a few mol % of a strong chloride donor such as CsCl (eq. 2), while acidic melts are generated by adding a strong chloride acceptor such as AlCl$_3$ (eq. 3). The degree to which the Lewis acidity can be altered is indicated from the calculation that in an SbCl$_3$-10 mol %
The concentration of SbCl$_2^+$ in a neat SbCl$_3$ melt is increased by a factor of $10^4 - 10^5$ compared to that in a melt containing 3 mol % AlCl$_3$. We have also found that SbCl$_3$ melts have a redox function in which Sb$^{3+}$ can act as an oxidant. The concomitant reduction of Sb$^{3+}$ in the melt liberates chloride and, thus, the oxidizing power of the melt will depend on the melt acidity. The effect of added chloride donor or acceptor on the formal potential of the Sb$^{3+}$/Sb couple was measured by electrochemical methods. As expected, the addition of a few mol % of a strong chloride donor (KCl) decreased the oxidizing power of SbCl$_3$, while the addition of a few mol % of a strong chloride acceptor (AlCl$_3$) increased its oxidizing power. In going from an SbCl$_3$-5 mol % KCl melt to an SbCl$_3$-5 mol % AlCl$_3$ melt, the formal potential of the Sb$^{3+}$/Sb couple and the resulting oxidizing strength can be tuned over ~0.5 V.

We have reported that antimony has lower oxidation states between 3+ and the metal, the nature of these species depending on the melt acidity. Thus, the single electron oxidation of organic compounds by Sb$^{3+}$ can take place homogeneously in the melt with the soluble lower oxidation state species acting as electron carriers. When SbCl$_3$ is reduced in significant quantities, Sb metal may be isolated from the reaction products upon hydrolysis of the melt as a consequence of a disproportionation reaction of the lower oxidation state species.

Redox Catalyzed Transfer Hydrogenation

Certain arenes can be oxidized to radical cations by Sb$^{3+}$ in SbCl$_3$-based melts with the degree of oxidation depending on melt acidity. This oxidation process has been studied for many arenes by ESR spectroscopy and, for the case of perylene, by UV-VIS spectroscopy. If no other substrates are present to react with the arene radical cations, they will react with themselves; or perhaps with unoxidized arene, leading to condensed products with new aryl-aryl bonds. These radical cation reactions produced some unusual hydrogen transfer chemistry in the melts, which we have previously reported.
We have initiated a study of the chemistry of these arene radical cations with other organic substrates that are not reactive, alone, in the melt. In this current investigation, tetralin (1,2,3,4-tetrahydronaphthalene) was chosen as the second reagent. Tetralin is a well known hydrogen donor for thermally generated neutral radicals, and we wished to see if tetralin could react with arene radical cations.

The arenes that were examined cover a wide range of oxidizability in the melt, and they are shown in Fig. 1 along with the corresponding anodic $E_{1/2}$ values measured in a basic SbCl$_3$ melt at 99°C (16). The oxidative $E_{1/2}$ of phenanthrene lies beyond the anodic limit of the melt (Sb$^{3+}$ + Sb$^{5+}$), but it has been estimated from the excellent correlation with arene $E_{1/2}$ values measured in acetonitrile (17). Of these arenes, phenanthrene is the most difficult to oxidize while naphthacene is the most easily oxidized.

Phenanthrene and pyrene are not capable of being oxidized to radical cations by neat SbCl$_3$. Equimolar solutions of either of these arenes with tetralin in SbCl$_3$ produces no reaction even at temperatures up to 150°C. The 200 MHz $^1$H NMR of these melts gives well-resolved spectra of the molecular components with no indication of any complexation of the organics with the solvent.

The more oxidizable arenes, anthracene, perylene, and naphthacene, are capable of being oxidized to radical cations in neat SbCl$_3$, as we have previously observed in dilute solutions by ESR (14). Anthracene is observed by NMR to react readily with tetralin at 80°C. Analysis of the product mixture following hydrolysis of the melt shows that a very selective transfer hydrogenation reaction has taken place as shown in Fig. 2 with 9,10-dihydroanthracene (DHA) the only hydroanthracene produced. The anthracene radical cation is apparently efficiently trapped by the tetralin, and no bianthracenes are detected that would result from coupling reactions of the radical cation (2,4). Product analysis further shows that no naphthalene is generated, and that the only other major products are 1,2,3,4,5,6,7,8-octahydro-1,2-binaphthalene (1) and 2-(1,2,3,4-tetrahydro-1-naphthyl)anthracene (2). The yield of DHA, 1, and 2 depend on the initial concentration of tetralin present, and this dependence is illustrated in Table 1. Anthracene conversions are in the range of 35 to 45% after the 30 min reaction period at 80°C. The yield of DHA is maximized at a tetralin/anthracene ratio of 2.0. Note that it is critical to maintain SbCl$_3$ as a concentrated melt in order to have catalytic activity. As the last entry in the table indicates, if tetralin is present in large excess such that it is actually the solvent and SbCl$_3$ is present as a homogeneous catalyst (with the same SbCl$_3$/anthracene ratios), no reaction occurs. The $1/2$ product ratio is also found to increase with increasing tetralin/anthracene ratio. The formation of these particular compounds and the dependence of their yields on the initial
tetralin/anthracene ratio will be discussed later in relation to the proposed mechanism.

Naphthacene also undergoes the transfer hydrogenation reaction with tetralin in SbCl₃. The reaction is even more rapid than that for anthracene with a 97% naphthacene conversion after a 30 min reaction period at 80°C. This reaction is also very selective as shown in Fig. 3 with 5,12-dihydronaphthacene the only hydronaphthacene formed. The byproducts are analogous to those produced in the anthracene reaction with the tetralyl-substituted naphthacene (3) product being a mixture of two isomers as indicated.

Perylene is also partially oxidized to a radical cation by Sb³⁺ in the melt. However, the radical cation is very persistent and undergoes no further reaction even in the presence of tetralin and at temperatures up to 130°C. This results in a ¹H NMR spectrum of the melt that exhibits signals due only to the tetralin, while the perylene signals are not observed because of rapid electron exchange between the perylene radical cation and unoxidized perylene.

The proposed mechanism for the catalytic transfer hydrogenation of arenes by tetralin in SbCl₃ is shown in Fig. 4 using anthracene as an example. The first step is the oxidation of the arene to the radical cation. Phenanthrene and pyrene do not undergo this electron transfer step and, thus, do not react. Perylene on the other hand undergoes the electron transfer reaction, but as described earlier the resulting radical cation is very persistent and undergoes no further reaction. Anthracene and naphthacene are also oxidized in the melt, and the radical cations react with tetralin by hydride transfer to the cationic center of the radical cation rather than hydrogen transfer to the radical center. The resulting 1-tetralyl cation, 5, then undergoes competitive electrophilic attack on unreacted tetralin and arene as shown in the third and fourth steps. The formation of the particular positional isomers shown is consistent with the known Friedel-Crafts alkylation chemistry of tetralin (18) and anthracene (19) when sterically bulky electrophiles are employed. Since the reaction is catalytic in SbCl₃, there must be a reoxidation step as shown in the last two equations. The detailed nature of the reoxidation step is not known, and it has been combined with the hydrogen transfer step that lead to the formation of the hydroarene.

Altering the oxidizing power of the melt has dramatic effects on the transfer hydrogenation reaction. If anthracene and tetralin (1:2 mole ratio) are reacted in a weakly oxidizing SbCl₃ - 8 mol % KCl melt, the reaction rate is substantially reduced with only a 2% DHA yield (rather than 39%) after 30 min at 80°C. On the other hand, if the reaction is run in the highly oxidizing SbCl₃-10 mol % AlCl₃ melt, not only are the rates enhanced but the product selectivity is completely altered. The transfer hydrogenation reaction is no longer observed and, instead, coupling reactions of the radical cations are
observed exclusively, which produce condensed anthracenes in a reaction that has been previously characterized (4).

The transfer hydrogenation chemistry has produced the first evidence that solution phase hydrocarbon radical cations can react by hydride abstraction. The research also points out the versatility of the SbCl$_3$-based molten salt catalyst resulting from the ability to reproducibly tune the strength of the redox function, which leads to control over reaction rates and product selectivities. Furthermore, the facility with which arene radical cations can be produced in the presence of a second unactivated organic substrate should allow for substantial advancement in the understanding of reactions of arene radical cations.

**Lewis Acid Catalyzed Transalkylation (7)**

The α,ω-diphenylalkanes C$_6$H$_5$(CH$_2$)$_n$C$_6$H$_5$ [n = 1-4] have first ionization potentials from photoelectron spectroscopy that are comparable to that of toluene (20). The high oxidation potentials of these compounds, compared to those of polycyclic arenes, places them beyond the range of the oxidizing power of SbCl$_3$ even in acidic melts. However, these compounds react readily in SbCl$_3$-rich melts in a reaction that is catalyzed by the Lewis acid function of the melt.

Diphenylmethane (DPM) is the most reactive of the diphenylalkanes with about a 60% conversion in SbCl$_3$ after 2 hrs at 130°C. The overall reaction is a transalkylation (Fig. 5) that produces, at low conversions (<10%), only benzene and the o-, m-, and p-isomers of benzyl diphenylmethane. At higher conversions, larger polybenzyl oligomers are observed.

1,2-Diphenylethane (DPE) is much less reactive than DPM. At 130°C in SbCl$_3$, the DPE reaction rate is measured to be $1 \times 10^3$ slower than that of DPM. However, rapid reaction rates may be obtained if the more acidic SbCl$_3$-10 mol % AlCl$_3$ melt is employed as catalyst. At 100°C, nearly 60% of the DPE reacts within the first 10 min and produces the same product mixture (Fig. 6) as observed in neat SbCl$_3$. Again a transalkylation reaction has occurred producing benzene, the o-, m-, and p-isomers of (2-phenylethyl)-1,2-diphenylethane, and also the higher oligomers at higher conversions. The observed cleavage of the benzyl sp$^2$-sp$^3$ bond for DPE is very selective, and no products are detected resulting from the cleavage of the central sp$^3$-sp$^3$ bond.

1,3-Diphenylpropane (DPP) and 1,4-diphenylbutane (DPB) react at very similar rates compared to DPE and require the SbCl$_3$-10 mol % AlCl$_3$ melt for rapid reaction rates. However, neither DPP or DPB undergoes an intermolecular transalkylation reaction. Instead, as shown in Fig. 7, they exclusively undergo an analogous intramolecular reaction to produce benzene and either indan (from DPP) or tetralin.
These extremely selective reactions have been monitored by in situ $^1$H NMR spectroscopy to near quantitative conversions of the DPP and DPB, and the only products observed are those described in Fig. 7. As before, only cleavage of the benzylic sp$^2$-sp$^3$ bond of these diphenylalkanes is observed.

The transalkylation chemistry of alkylbenzenes has been extensively studied and, normally, protons are invoked as key catalytic agents. In fact the reaction is typically performed in strong protic acid media such as AlBr$_3$-HBr, GaBr$_3$-HBr, and BF$_3$-HF (21-25). We have examined the least reactive of the alkylbenzenes, toluene, and found it to undergo typical transalkylation chemistry (Fig. 8) in the aprotic SbCl$_3$-10% AlCl$_3$ melt, although at a much slower rate ($\sim 4 \times 10^2$ slower than DPE in SbCl$_3$-10% AlCl$_3$ at 100°C).

The transalkylation chemistry for the diphenylalkanes and toluene in aprotic SbCl$_3$-based melts can be explained by a mechanism in which the rate determining step involves the abstraction of a benzylic hydride by the Lewis acid species in the melt, SbCl$_2^+$ (in its solvated form), as shown for the diphenylalkanes in eq. 6.

$$\text{PhCH}_2\text{(CH}_2\text{)}_x\text{Ph + SbCl}_2^+ \rightarrow \text{PhCH(}\text{CH}_2\text{)}_x\text{Ph + HSbCl}_2$$  \hspace{1cm} (6)

The relative reaction rates in SbCl$_3$ melts,

$$\text{PhCH}_2\text{Ph} \gg \text{Ph(CH}_2\text{)}_2\text{Ph} \sim \text{Ph(CH}_2\text{)}_3\text{Ph} \sim \text{Ph(CH}_2\text{)}_4\text{Ph} \gg \text{PhCH}_3,$$

are exactly those predicted based on the stabilities of the corresponding benzylic cations, namely

$$\text{PhCH}_2\text{Ph} \gg \text{PhCH(}\text{CH}_2\text{)}_2\text{Ph} \sim \text{PhCH(}\text{CH}_2\text{)}_3\text{Ph} \gg \text{PhCH}_3^+$$

Additional evidence supporting eq. 6 as the key mechanistic step includes:

1. For DPM the initial reaction rate has a first order dependence on the initial DPM concentration, which was varied from 0.028 to 1.02 M.

2. For DPE the rate in SbCl$_3$-10% AlCl$_3$ at 100°C is greater than that in SbCl$_3$ at 130°C by $1 \times 10^4$. The rate enhancement at a common temperature, e.g. 100°C, should be greater than $10^4$. This rate enhancement corresponds roughly to the calculated increase in SbCl$_2^+$ concentration of $10^4$-$10^5$.

3. We have also previously reported stoichiometric hydride abstractions for 9,10-dihydroanthracene and 5,12-dihydronaphthacene in
SbCl$_3$-10% AlCl$_3$ melts. For these substrates, the benzylic cation formed is stable in the melt and can be characterized by $^1$H NMR (5).

Once the benzylic cation is formed in the rate determining step, the product distributions can be readily understood as shown for the case of DPP in Fig. 9. For DPM and DPE stable cyclic products cannot be produced, and the incipient benzylic cation instead undergoes an electrophilic attack on unreacted starting material resulting in the intermolecular transalkylation chemistry observed.

The Lewis acid function of SbCl$_3$ melts is capable, therefore, of catalyzing a selective bond cleavage reaction for the $\alpha,\omega$-diphenylalkanes by generating a benzylic carbenium ion as the key organic reactive intermediate. The ability to tune the strength of this Lewis acid function allows for control of the reaction rates and provides kinetic information concerning the role of the catalyst.

**Experimental**

The reactions described above were studied by $^1$H NMR spectroscopy at 200 MHz and by product analysis following quench and separation procedures. Detailed experimental procedures have been previously reported (6,7). Both NMR and large scale experiments were performed with highly purified reagents in tubes that were sealed on a vacuum line under purified argon. Material transfers were performed in a controlled atmosphere drybox whose argon atmosphere was continuously purified and monitored for moisture and oxygen content, which amounted to <1 ppm each. Products were analyzed by GC, GC-MS, HPLC, and, where applicable, by $^1$H and $^{13}$C NMR. Quantitative results were obtained by GC using internal standards and by HPLC separation with subsequent analysis by UV spectroscopy.

**Acknowledgement**

We appreciate the assistance of L. L. Brown and I. B. Rubin with the NMR measurements and M. V. Buchanan with the GC-MS measurements. This research was sponsored by the Division of Chemical Sciences, Office of Basic Energy Sciences, U.S. Department of Energy, under Contract W-7405-eng-26 with the Union Carbide Corporation.

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Table 1. Influence of Tetralin Concentration on the Anthracene-Tetralin-SbCl₃ Reaction

| Anthracene (mmol) | Tetralin (mmol) | SbCl₃ (mmol) | DHA Yield (%) | 1/2 Yield (mole ratio) |
|-------------------|-----------------|--------------|---------------|------------------------|
| 1.40              | 0               | 34.2         | —             | —                      |
| 1.40              | 0.70            | 34.2         | 20            | 0.15                   |
| 1.40              | 1.40            | 34.2         | 30            | 0.50                   |
| 1.40              | 2.80            | 34.2         | 39            | 1.5                    |
| 1.40              | 5.60            | 34.2         | 38            | 3.5                    |
| 0.39              | 37.8            | 9.52         | —             | —                      |

Reactions were run at 80° for 30 min.

Based on original anthracene (±2%).

![Figure 1. Anodic E₁/₂ values for selected arenes in a basic SbCl₃ melt at 99°C. Value for phenanthrene (0.9 V) is estimated.](image_url)
Figure 2. Transfer hydrogenation reaction for anthracene with tetralin in molten SbCl$_3$.

\[
\text{anthracene} + \text{tetralin} \xrightarrow{\text{SbCl}_3, 100^\circ} \text{product} + \text{product} + \text{product}
\]

Figure 3. Transfer hydrogenation reaction for naphthacene with tetralin in molten SbCl$_3$.

\[
\text{naphthacene} + \text{tetralin} \xrightarrow{\text{SbCl}_3} \text{product} + \text{product} + \text{product}
\]
Figure 4. Proposed redox mechanism for the transfer hydrogenation reaction of an arene catalyzed by molten SbCl$_3$ (illustrated for anthracene).

$$
2 \text{O} + \frac{2}{3} \text{SbCl}_3 \rightleftharpoons 2 \text{O}^+ + \frac{2}{3} \text{Sb}^0 + 2 \text{Cl}^- \\

2 \text{O}^+ + 2 \text{O} \rightarrow 2 \text{O} + 2 \text{O}^+ \\
\text{5} + \text{OC} \rightarrow \text{H} \text{OC} \\
\text{5} + \text{OC} \rightarrow \text{H} \text{OC} \\
\text{4} + \text{6} + \frac{1}{3} \text{Sb}^0 + \text{Cl}^- \rightarrow \text{O} + \text{4} + \frac{1}{3} \text{SbCl}_3 \\
\text{4} + \text{7} + \frac{1}{3} \text{Sb}^0 + \text{Cl}^- \rightarrow \text{O} + 2 + \frac{1}{3} \text{SbCl}_3
$$

Figure 5. Transalkylation reaction for DPM in molten SbCl$_3$.

$$
\text{Ph}_2\text{C} \xrightarrow{\text{SbCl}_3, 100-130^\circ} \text{Ph} + \text{PhCHC}_9\text{H}_{11} + \text{PhCHC}_9\text{H}_{11} + \text{PhC}_9\text{H}_{11}
$$
Figure 6. Transalkylation reaction for DPE in molten SbCl$_3$-10% AlCl$_3$.

Figure 7. Intramolecular transalkylation reactions for DPP and DPB in molten SbCl$_3$-10% AlCl$_3$.

Figure 8. Transalkylation reaction for toluene in molten SbCl$_3$-10% AlCl$_3$.
Figure 9. Proposed mechanism for the SbCl$_3$-AlCl$_3$ catalyzed intramolecular transalkylation reaction for DPP following the initial hydride abstraction by SbCl$_2^+$. 

\[ H + H \rightarrow H^+ + H \]