3M™ Antistatic Additives
Ionic Liquids and Solids
February 2016
3M™ Antistatic Additives Overview

• Our Mission
• Fluorochemicals and Anions
• Products
• Performance
• Highlights
3M™ Antistatic Additives

Our Mission

To solve the toughest antistatic control problems using 3M’s unique fluorochemical synthesis and manufacturing capability.

Safety
- Reduce electrostatic discharge events
- Spark control

Cleanliness
- Reduce particles
- Reduce dust

Product Protection

Process Control
- Increase assembling efficiency
- Improve positioning process
- Increase control of toner liquid
- Increase easiness of peeling
Fluorochemical Capabilities

- 3M is a world leader in fluorine chemistry and electrochemical fluorination
- 50+ years manufacturing experience
- High purity fluorinated electrolyte salts and fluids manufacturing
  - Cordova, IL and Cottage Grove, MN (USA)
  - Antwerp, Belgium
- Extensive capabilities in fluorinated anion synthesis
  - Leverages existing ECF capabilities
- 15+ years experience making fluorinated ionic liquids and antistatic additives (lab and factory scale)
- Research labs in USA (St. Paul), Japan, Korea and China
Fluorinated Anions Derived from ECF

\[ \text{R}_f\text{SO}_2\text{F} \rightarrow \text{NH}_3 \rightarrow \text{SO}_2\text{R}_f \text{ imides} \]

\[ \rightarrow \text{H}_2\text{O} \rightarrow \text{OSO}_2\text{R}_f \text{ sulfonates} \]

\[ \rightarrow \text{CH}_3\text{MgCl} \rightarrow \text{SO}_2\text{R}_f \text{ methides} \]

Highly delocalized negative charge
3M™ Antistatic Additives
General Properties

- High ionic conductivity
  - Good electrolytes and antistats
- Lipophilic
  - Good solubility in organic solvents, monomers and polymers
  - Good optical clarity and low haze
  - Low affinity for water
- Low water content
  - Compatible with water sensitive monomers (e.g. urethanes)
  - Minimize bubble formation during high temperature processing

- Low to no vapor pressure
  - Non-flammable
  - No contribution to outgassing
  - No contribution to VOCs
- Stability
  - Thermal → Can process at high temperatures
  - Chemical → Improved pot life; little to no affect on polymer
  - Hydrolytic → No evidence of hydrolysis or HF formation in presence of water
  - Redox
  - Stable to melt processing
3M™ Antistatic Additives
Competitive Advantage

Adhesive
- Optical Clear
- Bonding Force
- Hydrophobic
- Thermal stability
- Independent to humidity

Protection / Optical Film
- No Migration
- Graft into Polymer
- No Leaching
- Static Decay Time

Coating
- Hydrophobic
- Thermal Stability
- Graft into Polymer

Packaging
- Optical Clear
- Hydrophobic
- Thermal Stability

Transportation Carrier Tape/Tray
- Graft in to Polymer
- Easier to Compound
- Thermal Stability
- No Leaching

Urethane Roller
- Static Decay Time
- Pot Life
- Durable
- Thermal Stability

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3M™ Antistatic Additives
Product Portfolio

Commercial Ionic Liquid Antistats

3M™ Ionic Liquid Antistat FC-4400
- (n-Bu)₃MeN⁺·N(SO₂CF₃)₂
- Salt of quaternary ammonium cation and fluorinated imide anion

3M™ Ionic Liquid Antistat FC-5000
- US Only
- R₄N⁺·N(SO₂CF₃)₂
- Salt of quaternary ammonium cation and fluorinated imide anion
- Single primary alcohol group on quaternary ammonium improves polymer compatibility and can be grafted into select polymer networks (e.g. urethanes, epoxies)

3M™ Ionic Liquid Antistat FC-5000i*
- OUS version of 3M™ Ionic Liquid Antistat FC-5000

Commercial Salts

3M™ Battery Electrolyte HQ-115
- Li⁺·N(SO₂CF₃)₂
- Salt of lithium cation and fluorinated imide anion
- Available in three product grades
  - 3M™ Battery Electrolyte HQ-115 (standard grade): <5000 ppm water
  - 3M™ Battery Electrolyte HQ-115U: <200 ppm water
  - 3M™ Ionic Liquid Precursor HQ-115IL: 20% water solution

Experimental Materials
3M can synthesize a variety of materials using toolkit of fluorinated anions and organic cations

* Referred to as FC-5000 for rest of the presentation
3M™ Battery Electrolyte HQ-115 (Antistatic Salt)

### Properties and Values

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Appearance</td>
<td>White powder</td>
</tr>
<tr>
<td>Melting Point</td>
<td>236°C</td>
</tr>
<tr>
<td>Thermal Decomposition Trigger Temperature</td>
<td>~330°C</td>
</tr>
</tbody>
</table>

### Advantages

- Effective in a variety of polymer hosts
  - Adhesives (e.g. acrylate based PSA)
  - Coatings (e.g. UV cured acrylates)
  - Urethanes

- Solid
- 100% active material – no solvent
- Hydrophilic; works in aqueous formulations
- Precursor for manufacture of ionic liquids
  - Available as 3M™ Ionic Liquid Precursor HQ-115IL: 80% concentrate in water
- Developed and used as Li battery electrolyte
3M™ Ionic Liquid Antistat FC-4400

**Effective in a variety of polymer hosts**
- Adhesives (e.g. acrylate based PSA)
- Coatings (e.g. UV cured acrylates)
- Urethanes
- Thermoplastic and thermoset polymers

**Advantages**
- Ionic liquid – no solids handling required
- 100% active material – no solvent
- Low reactivity to polymers at high temperatures
- Good cleanliness
  - Low metal and halogen ion levels
  - No outgassing or particle sloughing
- Hydrophobic – low H₂O content and solubility
- Stable performance over wide humidity range

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Appearance</td>
<td>Clear, colorless liquid or white crystalline solid</td>
</tr>
<tr>
<td>Melting Point</td>
<td>27.5°C</td>
</tr>
<tr>
<td>Thermal Decomposition Trigger Temperature</td>
<td>~340°C</td>
</tr>
<tr>
<td>Density @ 25°C</td>
<td>1.3 g/mL</td>
</tr>
<tr>
<td>Viscosity @ 25°C</td>
<td>499 cps (supercooled)</td>
</tr>
<tr>
<td>pH (Typical)</td>
<td>5 (neutral)</td>
</tr>
</tbody>
</table>
3M™ Ionic Liquid Antistat FC-5000

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Appearance</td>
<td>Clear, colorless liquid</td>
</tr>
<tr>
<td>Melting Point</td>
<td>&lt; -50°C</td>
</tr>
<tr>
<td>Thermal Decomposition Trigger Temperature</td>
<td>~320°C</td>
</tr>
<tr>
<td>Density @ 25°C</td>
<td>1.3 g/mL</td>
</tr>
<tr>
<td>Viscosity @ 25°C</td>
<td>251 cps</td>
</tr>
<tr>
<td>pH (Typical)</td>
<td>5 (neutral)</td>
</tr>
</tbody>
</table>

Effective in a variety of polymer hosts
- Adhesives (e.g. acrylate based PSA)
- Coatings (e.g. UV cured acrylates)
- Urethanes
- Thermoplastic and thermoset polymers

Advantages
- Wide liquid range – no solids handling
- 100% active material – no solvent
- Single pendant alcohol group
  - Graft in to some polymer networks
  - Improved solubility in some polymers
- Good cleanliness
  - Low metal and halogen ion levels
  - No outgassing or particle sloughing
- Hydrophobic – low H₂O content and solubility
- Stable performance over wide humidity range
## 3M™ Antistatic Additives
### Product Comparison

<table>
<thead>
<tr>
<th>Property</th>
<th>3M™ Battery Electrolyte</th>
<th>3M™ Ionic Liquid Antistat</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>HQ-115</td>
<td>FC-4400</td>
</tr>
<tr>
<td>General Description</td>
<td>Li salt of imide</td>
<td>(n-Bu)₃MeN⁺ salt of imide</td>
</tr>
<tr>
<td>Thermal Decomposition Trigger Temperature</td>
<td>330°C</td>
<td>340°C</td>
</tr>
<tr>
<td>Melting Point</td>
<td>236°C</td>
<td>27.5°C</td>
</tr>
<tr>
<td>Hydrophobic / Hydrophilic</td>
<td>Hydrophilic</td>
<td>Hydrophobic</td>
</tr>
<tr>
<td>Inertness</td>
<td>Good</td>
<td>Excellent</td>
</tr>
<tr>
<td>Metals / Halogen Ions</td>
<td>High Li</td>
<td>Low</td>
</tr>
<tr>
<td>Graft in to Polymer</td>
<td>No</td>
<td>No</td>
</tr>
</tbody>
</table>

* Polymer dependent

![Imide Structure](image)
3M™ Antistatic Additives
General Comments

- Typical effective loading levels
  - 1 to 5% by weight
- Performance is dependent on:
  - Polymer type
  - Polymer properties (e.g. molecular weight, T_g)
  - Preparation method
- Best place to test performance is on the customer’s line
- Can run small scale evaluation at 3M lab

- Antistatic additives processing
  - Extruded with polymer (hot melt)
  - Cast from polymer solution (solvent cast)
  - Dissolve in solvent free monomer solution
    - (100% solids thermal or photo cure)
- Extrusion processing
  - Injected mid barrel
  - Added at feed chute
  - Pre-mixed with resin pellets / powder
  - Masterbatch
## 3M™ Antistatic Additives
### Performance Summary

<table>
<thead>
<tr>
<th>Polymer</th>
<th>3M™ Battery Electrolyte</th>
<th>3M™ Ionic Liquid Antistat</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>HQ-115</td>
<td>FC-4400</td>
</tr>
<tr>
<td>Urethanes</td>
<td>Yes</td>
<td>Yes (W)</td>
</tr>
<tr>
<td>Adhesives</td>
<td>Yes</td>
<td>Yes</td>
</tr>
<tr>
<td>Coatings (e.g. acrylates)</td>
<td>Yes</td>
<td>Yes</td>
</tr>
<tr>
<td>Epoxies</td>
<td>-</td>
<td>Yes</td>
</tr>
<tr>
<td>PVDF</td>
<td>-</td>
<td>Yes (W)</td>
</tr>
<tr>
<td>PVC</td>
<td>-</td>
<td>Yes (W)</td>
</tr>
<tr>
<td>PET / PEN</td>
<td>-</td>
<td>Yes (N)</td>
</tr>
<tr>
<td>PETG</td>
<td>-</td>
<td>Yes (N)</td>
</tr>
<tr>
<td>PC</td>
<td>No</td>
<td>Yes (N)</td>
</tr>
<tr>
<td>Silicone (functional)</td>
<td>-</td>
<td>In some cases</td>
</tr>
<tr>
<td>PMMA / CAB / CAP</td>
<td>-</td>
<td>No</td>
</tr>
</tbody>
</table>

General results observed to date. Performance will depend on specific polymer and sample preparation. W – resistant to water washing. N – not resistant to water washing.
3M™ Antistatic Additives
Product Highlights

- Platform of products to meet customer requirements
- Excellent solubility in polar organic solvents and various polymer hosts
- Good optical clarity and low haze
- High ionic conductivity in various polymers
- Excellent thermal, chemical, hydrolytic and redox stability
- Low to no vapor pressure – no outgassing or flammability
- 100% active material – no solvents

- Cleanliness – low halogen and metal levels in 3M™ Ionic Liquid Antistats FC-4400 and FC-5000
- Graft-able – 3M™ Ionic Liquid Antistat FC-5000 can be reacted into certain polymer networks (e.g. urethanes, epoxies)
- Stable performance over wide humidity range for 3M™ Ionic Liquid Antistats FC-4400 and FC-5000
- Effective at low loading levels
  - Minimal impact on polymer physical properties
  - Cost effective
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