Analysis of Ethylene Carbonate and Ethyl Methyl Carbonate by GC/FID on a Thermo Scientific TraceGOLD TG-35MS Column

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Abstract
Ethylene carbonate (EC) and ethyl methyl carbonate (EMC) are used as electrolytes in lithium-ion batteries. As such the quantitative analysis of these compounds is important to battery manufacturers. Reproducible peak areas and excellent peak shape for EC and EMC across multiple chromatographic runs were obtained on a TraceGOLD™ TG-35MS capillary GC column, an intermediate polarity 35% diphenyl/65% dimethyl polysiloxane phase.

Introduction
Ethylene carbonate (EC) and ethyl methyl carbonate (EMC) are used as electrolytes in lithium-ion batteries. Therefore the quantitative analysis of these compounds is important to battery manufacturers. Ethylene carbonate is also used as plasticizer, and as a precursor to vinylene carbonate, which is used in polymers and in organic synthesis. Sharp, symmetrical peak shapes are desirable for accurate and reproducible quantitative data.

Experimental Details

Key Words
- TG-35MS
- Ethylene carbonate
- Ethyl methyl carbonate

Chemicals and Reagents
- Ethylene carbonate (EC); ethyl methyl carbonate (EMC)

Sample Handling Equipment
- Vials and closures: 2 mL clear vial and Si/PTFE seal 60180-599

Separation Conditions
- Instrumentation: Thermo Scientific Focus GC with TriPlus Autosampler
- Column: TraceGold TG-35MS, 30 m x 0.25 mm x 0.25 µm 26094-1420
- Septum: Thermo Scientific BTO 17 mm septa 31303211
- Liner: split/Splitless Liner with Siltek deactivation 45372121
- Column ferrules: graphite ferrules to fit 0.25 mm id columns 29553488
- Injection syringe: 10 µL, 50 mm Syringe 36500525

Carrier gas: helium
- Column flow: 1.0 mL/min (constant flow mode)
- Oven temperature: 100 °C (2.5 minute hold)-200 °C (4 minute hold) at 30 °C /minute

Injectors:
- Injector type: split
- Injector mode: split, split ratio: 70:1
- Split flow: 70 mL/minute
- Injector temperature: 200 °C

FID parameters:
- Temperature: 250 °C
- Air flow: 350mL/minute
- Hydrogen flow: 35 mL/minute
- Nitrogen makeup flow: 30 mL/minute
Solutions
Sample preparation: ECM/EC (70:30) diluted 2.2mg/1mL CH2Cl2

Data Processing
Software: ChromQuest

Results
Sharp, symmetrical peak shapes for EC and EMC, desirable for accurate and reproducible quantitative data, were obtained across multiple chromatographic runs on a TraceGOLD TG-35MS capillary GC column, an intermediate polarity 35% diphenyl/65% dimethyl polysiloxane phase. The peak area ratios obtained with five consecutive runs of the ECM/EC (70:30) sample was reproducible (see table below).

| Ethyl Methyl Carbonate (ECM)/Ethylene Carbonate (EC)-70:30 |
|-----------------|-----------------|-----------------|
| Run Number     | Peak Area ECM   | Peak Area EC    | EC/ECM |
| 1              | 6579920         | 2035819         | 0.308399 |
| 2              | 5923342         | 1847084         | 0.311831 |
| 3              | 6339728         | 1983810         | 0.312917 |
| 4              | 6512267         | 2026708         | 0.311214 |
| 5              | 669894          | 2093681         | 0.312539 |

Conclusions
Narrow, symmetrical peak shapes for EC and EMC were obtained.

Peak areas for EC and EMC consistent from run to run.

Figure 1: FID chromatogram of Teledyne ethylene carbonate sample on a TG-35MS column

<table>
<thead>
<tr>
<th>Peak</th>
<th>Teledyne Ethylene Carbonate Sample</th>
<th>Rt/min</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Ethyl methyl carbonate (ECM)</td>
<td>2.38</td>
</tr>
<tr>
<td>2</td>
<td>Ethylene carbonate (EC)</td>
<td>4.33</td>
</tr>
</tbody>
</table>

Table 1: Teledyne ethylene carbonate sample-Component retention times