LSC Cocktail Development and Evolution

Jock Thomson
Global Product Manager Cocktails & Vials
Development Process

- Customer need defined
- Concept for development
- Prototypes
- Evaluation
- Final products
**Alpha / Beta Cocktail**

**Requirements**

- Naphthalene compound present
- Good alpha/beta separation characteristics
- Compatible with mineral acids
- Accept a wide range of mineral acid concentrations
- No permeation through plastic vials
- Safer cocktail
  - High flash point
  - Biodegradable
  - Low toxicity
  - Low vapor pressure
Naphthalene compound present

- Presence of naphthalene enhances alpha-beta separation by converting a "fast" solvent to a "slow" solvent with respect to its fluorescence decay time.
- This enhances the difference between an alpha decay and a beta decay - alphas decay slower than betas.
- Previous cocktails were doped with naphthalene to produce this effect.
- DIN (di-isopropynaphthalene) was tried as an alternative solvent base.
Existing DIN based cocktails were evaluated for alpha/beta separation characteristics to establish the potential for using DIN.

Results were encouraging and confirmed that it would be possible to use DIN as the solvent.
Compatibility with mineral acids

This is a prerequisite since virtually all samples will be in either a hydrochloric acid or nitric acid medium. The cocktail must have good mineral acids holding capacity over a wide range of concentrations from 0.01M to >2M.
No permeation through plastic vials

Previous experience with DIN based cocktails indicates there will be no permeation. This allows more flexibility during sample preparation and counting.

Safety properties

The inherent characteristics of DIN allied with the known properties of surfactants ensures that the cocktail will be high flash point, biodegradable, and safer to use.
Cocktail Development

The first step was to devise formulations that would accept mineral acids.

The second step was to evaluate these for alpha/beta separation characteristics.

From this evaluation two factors emerged :-
1. The type of detergent significantly affects alpha/beta separation.
2. The overall concentration of detergents affects alpha/beta separation.
Cocktail Development

These two findings were crucial in deciding the final formulation of the cocktail. In effect a total of 25 different formulations were evaluated before deciding upon the final cocktail.

The concentration effect was further investigated and the results were published to allow users to customise mixtures for their own particular application.

The final cocktail was named Ultima Gold AB.
### Alpha / Beta Cocktail

<table>
<thead>
<tr>
<th>Type of Sample</th>
<th>Ultima Gold AB</th>
<th>85% (v/v) UG AB</th>
<th>75% (v/v) UG AB</th>
<th>50% (v/v) UG AB</th>
</tr>
</thead>
<tbody>
<tr>
<td>Water</td>
<td></td>
<td>85% (v/v) UG AB</td>
<td>75% (v/v) UG AB</td>
<td>50% (v/v) UG AB</td>
</tr>
<tr>
<td>Sample Uptake Range (mL)</td>
<td>0.2 - 10.0</td>
<td>0.2 - 10.0</td>
<td>0.2 - 2.25</td>
<td>0.2 - 0.5</td>
</tr>
<tr>
<td>Misclassification Range (%)</td>
<td>0.74-1.87</td>
<td>0.70-1.74</td>
<td>0.58-0.68</td>
<td>0.42-0.47</td>
</tr>
<tr>
<td>1 M HCl</td>
<td></td>
<td>85% (v/v) UG AB</td>
<td>75% (v/v) UG AB</td>
<td>50% (v/v) UG AB</td>
</tr>
<tr>
<td>Sample Uptake Range (mL)</td>
<td>0.2 - 5.5</td>
<td>0.2 - 2.0</td>
<td>0.2 - 1.25</td>
<td>0.2 - 0.4</td>
</tr>
<tr>
<td>Misclassification Range (%)</td>
<td>0.87-1.73</td>
<td>0.64-0.79</td>
<td>0.64-0.75</td>
<td>0.43-0.52</td>
</tr>
<tr>
<td>2 M HCl</td>
<td></td>
<td>85% (v/v) UG AB</td>
<td>75% (v/v) UG AB</td>
<td>50% (v/v) UG AB</td>
</tr>
<tr>
<td>Sample Uptake Range (mL)</td>
<td>0.2 - 2.25</td>
<td>0.2 - 1.15</td>
<td>0.2 - 1.0</td>
<td>0.2 - 0.3</td>
</tr>
<tr>
<td>Misclassification Range (%)</td>
<td>0.61-1.07</td>
<td>0.56-0.76</td>
<td>0.49-0.60</td>
<td>0.38-0.49</td>
</tr>
<tr>
<td>1 M HNO3</td>
<td></td>
<td>85% (v/v) UG AB</td>
<td>75% (v/v) UG AB</td>
<td>50% (v/v) UG AB</td>
</tr>
<tr>
<td>Sample Uptake Range (mL)</td>
<td>0.2 - 3.25</td>
<td>0.2 - 1.75</td>
<td>0.2 - 1.25</td>
<td>0.2 - 0.4</td>
</tr>
<tr>
<td>Misclassification Range (%)</td>
<td>0.75-3.85</td>
<td>0.66-1.51</td>
<td>0.55-0.98</td>
<td>0.48-0.53</td>
</tr>
<tr>
<td>2 M HNO3</td>
<td></td>
<td>85% (v/v) UG AB</td>
<td>75% (v/v) UG AB</td>
<td>50% (v/v) UG AB</td>
</tr>
<tr>
<td>Sample Uptake Range (mL)</td>
<td>0.2 - 2.25</td>
<td>0.2 - 1.5</td>
<td>0.2 - 1.0</td>
<td>0.2 - 0.3</td>
</tr>
<tr>
<td>Misclassification Range (%)</td>
<td>0.77-4.21</td>
<td>0.60-1.91</td>
<td>0.70-1.17</td>
<td>0.54-0.62</td>
</tr>
</tbody>
</table>

Typical percentage misclassification using a Tri-Carb 2550TR/AB and Time-Resolved Pulse Decay Analysis. Sample uptake per 10 mL cocktail at 20 °C
Low Level Tritium Cocktail

Requirements

- High water loading capacity
- Accept different types of water
  - Distilled water; Deionised water; Tap water
  - River water; Rain water; Sea water
- High capacity for urine
- High Tritium counting efficiency
- Low background level
- No permeation through plastic vials
- Desirable safety characteristics
Low Level Tritium Cocktail

Requirements

High water loading capacity
Accept different types of water
   Distilled water; Deionised water; Tap water
   River water; Rain water; Sea water
High capacity for urine
High Tritium counting efficiency
Low background level
No permeation through plastic vials
Desirable safety characteristics
Low Level Tritium Cocktail

High water loading capacity

Detection and quantitation of low levels of Tritium necessitate a particularly high loading capacity to ensure a high degree of accuracy in the analysis. Cocktail/water mixture must be stable with respect to time and temperature for low level counting as the count times can be >500 minutes and counting temperatures can be as low as 14°C.

Accept different types of water

The radioactive species of interest can be in water from a variety of sources and therefore the cocktail must be able to accommodate this sample diversity.
Low Level Tritium Cocktail

High capacity for urine

High sample acceptance is desirable for Health Physics Departments who regularly monitor urine samples as part of their occupational health programmes.

High $^3$H efficiency : Low background : High figure of merit

In LSC the signal-to-background ratio is expressed as the square of the detection efficiency divided by the background ($E^2/B$) and is usually termed the Figure of Merit.

Fundamentally the highest efficiency with the lowest background produces the highest sensitivity.
No permeation through plastic vials

Previous experience with DIN based cocktails indicates there will be no permeation. This allows more flexibility during sample preparation and increased performance during the normally long periods of counting. Lowest backgrounds are obtained with PE vials which are not contaminated with $^{40}$K as in glass vials.

Safety properties

The inherent characteristics of DIN allied with the known properties of surfactants ensures that the cocktail will be high flash point, biodegradable, and safer to use.
Cocktail development

The target was well defined in that different types of water should be accepted at high loading (up to 12mL in 10mL cocktail at all temperatures between 12°C and 20°C).

Ultima Gold AB was used as a starting point as this cocktail could hold 10mL water at 20°C but not at lower temperatures. A series of subtle yet significant changes were made to the formulation and the performance criteria were met.

A few formulations were evaluated in a low level LSC before deciding upon a final cocktail. The final cocktail was named Ultima Gold LLT.
Low Level Tritium Cocktail

Cocktail development

During this evaluation certain factors emerged:

1. With DIN based cocktails lower backgrounds can be obtained when counting at low temperatures. This is most probably due to an increase in viscosity.

2. Lower backgrounds can be obtained at the expense of some efficiency by using optimized window settings.

3. The cocktail was found to be very suitable for use with urine samples.

4. Since the cocktail was developed from Ultima Gold AB, it was found to be equally suitable for alpha/beta work.
Low Level Tritium Cocktail

The graph shows the mL Water Uptake per 10 mL of Ultima Gold LLT for different types of water and temperatures:

- **Sea Water**:
  - 20 °C: Black bar
  - 18 °C: Blue bar
  - 16 °C: White bar
  - 14 °C: Light blue bar
  - 12 °C: Dark grey bar

- **Deionized, Distilled and Rain Water**:
  - 20 °C: Black bar
  - 18 °C: Blue bar
  - 16 °C: White bar
  - 14 °C: Light blue bar
  - 12 °C: Dark grey bar

The graph indicates lower water uptake at lower temperatures for all types of water.
## Low Level Tritium Cocktail

<table>
<thead>
<tr>
<th>Water to Cocktail Ratio</th>
<th>Optimized Windows (0.4-4.5keV)</th>
<th>Bkg (CPM)</th>
<th>E²/B</th>
<th>E²V²/B</th>
<th>MDA Bq/L⁻¹</th>
</tr>
</thead>
<tbody>
<tr>
<td>8 mL:12 mL</td>
<td></td>
<td>24.6%</td>
<td>1.15</td>
<td>526</td>
<td>33680</td>
</tr>
<tr>
<td>10 mL:10 mL</td>
<td></td>
<td>21.2%</td>
<td>0.05</td>
<td>405</td>
<td>40490</td>
</tr>
<tr>
<td>11 mL:9 mL</td>
<td></td>
<td>18.1%</td>
<td>0.95</td>
<td>345</td>
<td>41730</td>
</tr>
</tbody>
</table>

Tri-Carb 2770TR/SL (operated at 15 °C) Low Level Count Mode, all samples in duplicate, 500 minute count time.
Low Level Tritium Cocktail

Detection Limit
MDA vs. CPM Background

[100pCi = 3.7Bq]
Useful Tips

Water samples

✓ Sometimes researchers distill water or collect ground water and then add acid to stabilize the water samples.
✓ The addition of even small amounts of acid can have a serious affect on sample capacity.
✓ It is recommended that the cocktail be re-evaluated with this modified sample to ensure that the mixture is still homogeneous.
✓ This is especially important if plastic vials are being used, as they are not transparent.
✓ Additionally carry out the evaluation in glass vials at the counting temperature to be sure of stability over the count period.
### Cocktail Performance with Mineral Acids

<table>
<thead>
<tr>
<th>Sample</th>
<th>Ultima Gold</th>
<th>Ultima Gold XR</th>
<th>Ultima Gold AB</th>
<th>Ultima Gold LLT</th>
<th>Opti-Fluor</th>
<th>Emulsifier Safe/</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.1M HCl</td>
<td>6.5mL</td>
<td>7.0mL</td>
<td>10.0mL</td>
<td>10.0mL</td>
<td>4.0mL</td>
<td>2.7mL</td>
</tr>
<tr>
<td>1.0M HCl</td>
<td>0.5mL</td>
<td>2.5mL</td>
<td>5.5mL</td>
<td>5.0mL</td>
<td>0.5mL</td>
<td>3.0mL</td>
</tr>
<tr>
<td>2.0M HCl</td>
<td>None</td>
<td>1.0mL</td>
<td>2.25mL</td>
<td>3.0mL</td>
<td>None</td>
<td>4.5mL</td>
</tr>
<tr>
<td>5.0M HCl</td>
<td>None</td>
<td>&lt;0.5mL</td>
<td>2.0mL</td>
<td>1.5mL</td>
<td>None</td>
<td>0.5mL</td>
</tr>
<tr>
<td>1.0M HNO₃</td>
<td>None</td>
<td>2.5mL</td>
<td>3.25mL</td>
<td>3.5mL</td>
<td>0.75mL</td>
<td>3.5mL</td>
</tr>
<tr>
<td>2.0M HNO₃</td>
<td>0.5mL</td>
<td>2.0mL</td>
<td>2.25mL</td>
<td>2.5mL</td>
<td>0.75mL</td>
<td>3.5mL</td>
</tr>
<tr>
<td>3.0M HNO₃</td>
<td>None</td>
<td>1.0mL</td>
<td>2.0mL</td>
<td>2.25mL</td>
<td>0.5mL</td>
<td>1.0mL</td>
</tr>
<tr>
<td>1.0M H₂SO₄</td>
<td>None</td>
<td>0.25mL</td>
<td>6.5mL</td>
<td>7.0mL</td>
<td>None</td>
<td>2.0mL</td>
</tr>
<tr>
<td>2.0M H₂SO₄</td>
<td>None</td>
<td>None</td>
<td>4.0mL</td>
<td>4.0mL</td>
<td>None</td>
<td>2.75mL</td>
</tr>
<tr>
<td>1.0M HClO₄</td>
<td>2.0mL</td>
<td>2.0mL</td>
<td>2.25mL</td>
<td>2.25mL</td>
<td>1.5mL</td>
<td>1.0mL</td>
</tr>
<tr>
<td>2.0M HClO₄</td>
<td>1.5mL</td>
<td>1.5mL</td>
<td>2.0mL</td>
<td>2.5mL</td>
<td>1.0mL</td>
<td>0.75mL</td>
</tr>
<tr>
<td>1.0M H₃PO₄</td>
<td>None</td>
<td>1.5mL</td>
<td>0.5-10.0mL</td>
<td>0.5-10.0mL</td>
<td>0.5-1.5mL</td>
<td>3.0mL</td>
</tr>
<tr>
<td>2.0M H₃PO₄</td>
<td>None</td>
<td>0.5mL</td>
<td>0.5-4.0mL</td>
<td>0.5-6.0mL</td>
<td>0.5-1.0mL</td>
<td>3.0mL</td>
</tr>
</tbody>
</table>
## Cocktails for Urine

<table>
<thead>
<tr>
<th>Cocktail</th>
<th>Type</th>
<th>Max. capacity at 20°C</th>
<th>Comments</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ultima Gold</td>
<td>Safer</td>
<td>8.0mL</td>
<td>Slight wispy precipitate appears after 24 hours at &gt;2.0mL sample.</td>
</tr>
<tr>
<td>Ultima Gold XR</td>
<td>Safer</td>
<td>10.0mL</td>
<td>Slight wispy precipitate appears after 24 hours at &gt;3.0mL sample.</td>
</tr>
<tr>
<td>Ultima Gold AB</td>
<td>Safer</td>
<td>10.0mL</td>
<td>Slight wispy precipitate appears after 24 hours at &gt;2.0mL sample.</td>
</tr>
<tr>
<td>Ultima Gold LLT</td>
<td>Safer</td>
<td>10.0mL</td>
<td>Slight wispy precipitate appears after 24 hours at &gt;2.0mL sample.</td>
</tr>
<tr>
<td>Opti-Fluor</td>
<td>Safer</td>
<td>9.0mL</td>
<td>Slight wispy precipitate appears after 24 hours at &gt;3.0mL sample.</td>
</tr>
<tr>
<td>Emulsifier Safe</td>
<td>Safer</td>
<td>2.5mL</td>
<td>Slight wispy precipitate appears after 24 hours at &gt;3.0mL sample.</td>
</tr>
<tr>
<td>Pico-Fluor MI</td>
<td>Classical</td>
<td>4.0mL</td>
<td>Remains clear up to 4.0mL sample</td>
</tr>
<tr>
<td>Hionic-Fluor</td>
<td>Classical</td>
<td>1.5mL</td>
<td>&gt;2.0mL sample forms a gel</td>
</tr>
<tr>
<td>Insta-Gel Plus</td>
<td>Classical</td>
<td>1.0mL</td>
<td></td>
</tr>
<tr>
<td>Pico-Fluor 40</td>
<td>Classical</td>
<td>3.0mL</td>
<td>Slight wispy precipitate appears after 24 hours at &gt;3.0mL sample.</td>
</tr>
</tbody>
</table>
PerkinElmer Cocktails

The Mercedes Benzene of Cocktails