Advanced Software Features for the LA-950

Ian Treviranus
ian.treviranus@horiba.com
www.horiba.com/us/particle
What we’ll talk about

- Measurement tools
- Data analysis tools
- Data verification tools
- Q&A
One-button Measurement

- Use the Navigator or Method Expert to create Sequence (.seq) files
  - Manual: Navigator
  - Auto: Method Expert
LA-950 Method Expert

- Unique guided method development
- Optimize parameters
- Choose the best refractive index
- Create “one button” SOPs
- Webinar TE004 for Method Expert
Collecting and Calculating

The LA-950 hardware collects scattered light data

The LA-950 software calculates the particle size distribution using that scattered light data

Both must be optimized to maximize data quality
LA-950 Method Expert

Welcome to the Method Expert for the HORIBA LA-950 particle size analyzer!

Think of the Method Expert as one big wizard to help you develop a robust method for running any sample with high accuracy and precision. The measurement process is divided into three sections containing simple, guided tests to help you generate great particle size data.

You'll find two special buttons throughout the Method Expert:
- Click 🕵️‍♂️ to discover more information about why a particular idea or test is important.
- Click ✅ on the results screen to read HORIBA's Expert Advice for choosing the best value for that particular test.

Start Measurement Wizard
Start Calculation Wizard
Start Automation Wizard
There are four important tests...

- Circulation
- Concentration
- Dispersion
- Duration
There are two important tests...

- **Real RI**
- **Imaginary RI**
LA-950 Method Expert

Why is the test important?
What does the test do?
How will the results be displayed?
What is the best value?

User selects up to 5 values for testing
LA-950 Method Expert

Method Expert guides user to prepare the LA-950 for each test

Results displayed in multiple formats: PSD, D50, R-parameter
LA-950 Method Expert

Welcome to the Automation Wizard!

The purpose of the Automation Wizard is to teach the LA-950 how to analyze a particular sample so that the user need only push a single button to collect a measurement.

A Condition and Sequence file will be created to automate the process and effectively create a standard operating procedure.

The entire measurement process can be separated into four sections: Preparation, Collection, Calculation, and Output.

- **Preparation** is everything that needs to be done before the sample is added to the analyzer. This includes identifying the sample, filling the analyzer with liquid, turning on the circulation pump, aligning the laser, and taking a good background blank.

- **Collection** is adding sample to the analyzer at the correct concentration and then measuring the scattered light data over time.

- **Calculation** refers to the refractive index of the sample material and number of iterations for the data to pass through the algorithm.

- **Output** consists of various ways to save, export, and print the measurement. The Condition and Sequence files are created here.
LA-950 Method Expert

Automation Wizard
Outputting/Reporting the Measurement

Section Purpose
The measurement has been collected and calculated and can now be saved, exported, and printed for reporting. The LA-950 was designed to meet a variety of customer preferences, so there are many ways to perform these tasks.

Once the reporting setup is finished, simply name the Condition and Sequence files used to run this method.

Step 4. Give this Expert Method a unique, descriptive name.
(This name is used as the output sequence file name)

Step 5. Input condition file name.

Step 6. Push save button.
This wizard is temporarily closed, and the sequence file and condition file are saved.

Save Sequence and Condition
Dry Measurement

- Method Expert currently wet only
- Use “Auto Measurement” for dry
- Webinar TE016: Optimizing Dry Powder Measurements
Navigator

- LA Navigator function creates Sequence programs to operate the LA-950
- Maximum flexibility
Navigator

Edit sequence

- Load
- Save

Command
- Condition Setting
- Counter clear
- Counter decrement
- Counter increment
- Export Data
- Layout Setting
- Memory ADD
- Memory CLR
- Memory clear
- Memory Div
- Memory Mul
- Memory Save
- Message
- MessageSound
- Print
- Save Data
- Wall End
- Wall Start

Put in Sequence

Add New Sequence

Clear all

Common Command

OK
Automatic Dilution

- Concentration control
- Adds dispersant, drains, repeat
- Is not possible without fill pump
What we’ll talk about

- Measurement tools
- Data analysis tools
- Data verification tools
- Q&A
Refractive Index

- Many, many resources on website
- Webinar TR009: Optimization of RI
Manually Changing RI
Manually Changing RI
Automated RI Computation

- Real part study
  - Need to fix imaginary part
  - Set up to 5 real parts
  - Software will compute all RI and display R parameter variation with RI selection

Step 1: Select measurement data for test
- Select Active Memory Data

Step 2: Choose RI for liquid dispersant
- 1.333

Step 3: Input RI imaginary component for test
- 0

Step 4: Input RI real component for test
- Test Value 1: 1.5
- Test Value 2: 1.6
- Test Value 3: 1.7
- Test Value 4: 1.8
- Test Value 5: 1.9

Step 5: Push "Execute ..." button.
This wizard is temporarily closed, and the test sequence is executed.

Execute Test Sequence >>
Automated RI Computation

Calculation Optimization
Real Refractive Index Wizard -Result-

Graph Summary | Distributions | D90,D50,D10 | R Parameter

Distribution Graph

D90, D50, D10
D90
D50
D10

R Parameter

R Parameter

Explore the future
Automotive Test Systems | Process & Environmental | Medical | Semiconductor | Scientific

© 2012 HORIBA, Ltd. All rights reserved.
Automated RI Computation

Step 6. Choose the best value
Please select the parameter listed below which provides the best particle size distribution.
If you would like assistance choosing the best value, please click the help symbol to the right.

Real RI: 1.50 - 0.00i
Automated RI Computation

- Imaginary part study
  - Need to fix real part
  - Set up to 5 imaginary parts
  - Software will compute all RI and display R parameter variation with RI selection

Step 1: Select measurement data for test
- Select Active Memory Data
- Select Data File

Step 2: Choose RI for liquid dispersant
- 1.33
- Open List

Step 3: Input RI real component for test
- 2.75

Step 4: Input RI imaginary component for test
- Test Value 1: 0.01
- Test Value 2: 0.1
- Test Value 3: 0.5
- Test Value 4: 0.7
- Test Value 5: 1

Step 5: Push "Execute ..." button.
This wizard is temporarily closed, and the test sequence is executed.

Execute Test Sequence >>
Automated RI Computation

- Imaginary study

Graph Summary | Distributions | D90, D50, D10 | R Parameter

![Graphs showing data distribution and R parameter values.](image)
Error Calculations

\[ \chi^2 = \sum \left\{ \frac{1}{\sigma_i^2} \left[ y_i - y(x_i) \right]^2 \right\} \]

\[ R = \frac{1}{N} \sum_{i=1}^{N} \left\{ \frac{1}{y(x_i)} \left| y_i - y(x_i) \right| \right\} \]

- \( y_i \): The measured scattered light at each channel (i) of the detector.
- \( y(x_i) \): The calculated scattered light at each channel (i) of the detector based on the chosen refractive index kernel and reported particle size distribution.
- \( \sigma_i \): The standard deviation of the scattered light intensity at each channel (i) of the detector. A larger \( \sigma_i \) indicates lower reliability of the signal on a given detector.
- \( N \): The number of detectors used for the calculation.

Figure 3

Emulsion Sample

Chi Square : 0.002295
R Parameter : 4.0698E-002

Chi Square (%)

Red Transmission %
Multimodal Report

- Hard to use full-distribution metrics to describe multimodal results

Graph Type | D(0.1) | D(0.5) | D(0.9)  
---|---|---|---
     | 0.06163(μm) | 0.35642(μm) | 0.64072(μm)  

D10  D50  D90

Are these meaningful?
Multimodal Report
Multimodal Report

- Deconvolute distribution into components
Multimodal Report

Statistics for each distribution mode

<table>
<thead>
<tr>
<th>Summary</th>
<th>Frequency</th>
<th>Statistics</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sample Distribution</td>
<td>Distribution 1</td>
<td>Distribution 2</td>
</tr>
<tr>
<td>Display</td>
<td>✓ Display</td>
<td>✓ Display</td>
</tr>
<tr>
<td>D50 : 0.36(μm)</td>
<td>D50 : 0.11(μm)</td>
<td>D50 : 0.83(μm)</td>
</tr>
<tr>
<td>D10 : 0.06(μm)</td>
<td>D10 : 0.04(μm)</td>
<td>D10 : 0.27(μm)</td>
</tr>
<tr>
<td>D90 : 0.64(μm)</td>
<td>D90 : 0.11(μm)</td>
<td>D90 : 0.69(μm)</td>
</tr>
<tr>
<td>Average : 0.36(μm)</td>
<td>Average : 0.07(μm)</td>
<td>Average : 0.46(μm)</td>
</tr>
<tr>
<td>Mode : 0.42(μm)</td>
<td>Mode : 0.07(μm)</td>
<td>Mode : 0.42(μm)</td>
</tr>
<tr>
<td>STD Dev. : 0.23(μm)</td>
<td>STD Dev. : 0.03(μm)</td>
<td>STD Dev. : 0.17(μm)</td>
</tr>
<tr>
<td>Span : NoValue</td>
<td>Span : NoValue</td>
<td>Span : NoValue</td>
</tr>
<tr>
<td>Area Ratio:</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Sample:Dist1 : 3.72</td>
<td>Dist1:Sample : 0.27</td>
<td>Dist1:Sample : 0.72</td>
</tr>
<tr>
<td>Sample:Dist2 : 1.39</td>
<td>Dist1:Dist2 : 0.37</td>
<td>Dist1:Dist1 : 2.57</td>
</tr>
<tr>
<td>Sample:Dist3 : ...</td>
<td>Dist1:Dist3 : ....-</td>
<td>Dist2:Dist3 : ....-</td>
</tr>
</tbody>
</table>

Better understanding of entire distribution
Intensity Graph

- Diffraction analyzer measures light scattering pattern, algorithm transforms this into a particle size distribution.
Size affects intensity

- **LARGE PARTICLE:**
  - Low angle scatter
  - Large signal

- **SMALL PARTICLE:**
  - High Angle Scatter
  - Small Signal
Intensity Graph
Intensity Graph

- One way to use the Intensity Graph
  - Two results, one good and one bad

![Graph with data points and labels showing diameter (µm) and transmittance (R) values.](image-url)
Intensity Graph

- Not Passing
- Passing
- Low Angle Large Particle
- High Angle Small Particle
Blank Check

- Need to explain difference in scattering
- Try other tools, i.e. Blank Check
Blank Check

Validation Result: NG

Sample Measurement Date: 2011-12-14
Standard Measurement Date: 2007-05-11

CH Intensity Validation

<table>
<thead>
<tr>
<th></th>
<th>Sample Value</th>
<th>Standard Value</th>
<th>UCL (%)</th>
<th>LCL (%)</th>
<th>Distance(%)</th>
<th>Deviation</th>
</tr>
</thead>
<tbody>
<tr>
<td>1CH</td>
<td>0.140044</td>
<td>0.0451122</td>
<td>100</td>
<td>-99.99</td>
<td>210.436</td>
<td>NG</td>
</tr>
<tr>
<td>2CH</td>
<td>0.0518867</td>
<td>0.0379509</td>
<td>100</td>
<td>-99.99</td>
<td>36.7205</td>
<td>OK</td>
</tr>
<tr>
<td>3CH</td>
<td>0.0184333</td>
<td>0.0281972</td>
<td>100</td>
<td>-99.99</td>
<td>-34.6272</td>
<td>OK</td>
</tr>
<tr>
<td>4CH</td>
<td>0.0102854</td>
<td>0.0100466</td>
<td>100</td>
<td>-99.99</td>
<td>2.37669</td>
<td>OK</td>
</tr>
<tr>
<td>5CH</td>
<td>0.0166478</td>
<td>0.0112805</td>
<td>100</td>
<td>-99.99</td>
<td>47.5802</td>
<td>OK</td>
</tr>
</tbody>
</table>
Custom Calculations
What we’ll talk about

■ Measurement tools
■ Data analysis tools
■ Data verification tools
■ Q&A
Automate COV Calculation

- Coefficient of Variation indicates precision of multiple measurements
- ISO 13320 and USP <429> make recommendations using COV

<table>
<thead>
<tr>
<th>File Name</th>
<th>Sample Name</th>
<th>D(v,0.1)</th>
<th>D(v,0.5)</th>
<th>D(v,0.9)</th>
</tr>
</thead>
<tbody>
<tr>
<td>200811061138068.NGB</td>
<td>Zircoa Slurry</td>
<td>0.065</td>
<td>0.107</td>
<td>0.185</td>
</tr>
<tr>
<td>200811061140069.NGB</td>
<td>Zircoa Slurry</td>
<td>0.071</td>
<td>0.145</td>
<td>11.896</td>
</tr>
<tr>
<td>200811061144070.NGB</td>
<td>Zircoa Slurry</td>
<td>0.069</td>
<td>0.129</td>
<td>3.838</td>
</tr>
</tbody>
</table>

Average 0.068 0.127 5.306
Std. Dev. 0.003 0.019 5.992
CV (%) 4.471 15.023 112.921
ISO 13320-1 (20.0, 15.0, 20.0) PASSED FAILED FAILED
Automate COV Calculation
### Automate COV Calculation

#### Summary Report

<table>
<thead>
<tr>
<th>File Name</th>
<th>Sample Name</th>
<th>D((v, 0.1))</th>
<th>D((v, 0.5))</th>
<th>D((v, 0.9))</th>
</tr>
</thead>
<tbody>
<tr>
<td>200811061138068.NGB</td>
<td>Zircoa Slurry</td>
<td>0.065</td>
<td>0.107</td>
<td>0.185</td>
</tr>
<tr>
<td>200811061140069.NGB</td>
<td>Zircoa Slurry</td>
<td>0.071</td>
<td>0.145</td>
<td>11.896</td>
</tr>
<tr>
<td>200811061144070.NGB</td>
<td>Zircoa Slurry</td>
<td>0.069</td>
<td>0.129</td>
<td>3.838</td>
</tr>
<tr>
<td><strong>Average</strong></td>
<td></td>
<td><strong>0.068</strong></td>
<td><strong>0.127</strong></td>
<td><strong>5.306</strong></td>
</tr>
<tr>
<td><strong>Std. Dev.</strong></td>
<td></td>
<td><strong>0.003</strong></td>
<td><strong>0.019</strong></td>
<td><strong>5.992</strong></td>
</tr>
<tr>
<td><strong>CV (%)</strong></td>
<td></td>
<td><strong>4.471</strong></td>
<td><strong>15.023</strong></td>
<td><strong>112.921</strong></td>
</tr>
<tr>
<td>ISO 13320-1 (20.0, 15.0, 20.0)</td>
<td></td>
<td>PASSED</td>
<td>FAILED</td>
<td>FAILED</td>
</tr>
</tbody>
</table>
Result Verification

Verification Setting

Parameter: Median Size
Specification: ISO 13320-1
Standard Value: 50 [mm]
Tolerance: ± 5 [mm]
Certified range of values:
- D(v, 0.5) > 10μm: ± 0 [μm] ± 0 %
- D(v, 0.5) < 10μm: ± 0 [μm] ± 0 %

Result Display Setting

Pass:
- Color: [Blue]
- Text: OK

Fail:
- Color: [Red]
- Text: NG

Distribution Graph

- Mean Size: 0.18408 [μm]
- Variance: 1.89988E-3 [μm²]
- Median Size: 0.17730 [μm]
- Mode Size: 0.1649 [μm]
- Std.Dev.: 0.17730 [μm]
- Chi Square: 3.7379E-1
- R Parameter: 4.162519

Diameter on Cumulative %

- (2) 10.00 % [0.1345 [μm]]
- (9) 5.00 % [0.2450 [μm]]
- (1) 85.00 % [100.0000 %]
- (5) 21.20 % [100.0000 %]
- (4) 30.00 % [100.0000 %]
- (2) 600.00 % [100.0000 %]
- (3) 425.00 % [100.0000 %]
- (1) 106.0 % [100.0000 %]
- (3) 75.00 % [100.0000 %]
- (9) 5.00 % [100.0000 %]
- (10) 30.00 % [100.0000 %]

Cumulative % on Diameter

- 1.00 % [0.1345 [μm]]
- 2.00 % [0.2450 [μm]]
- 10.00 % [100.0000 %]
- 30.00 % [100.0000 %]

Verification Data

- andy1
  - Data Name: 200809181026014
  - Transmittance (R): 88.3 [%]
  - Median Size: 0.17730 [μm]
  - R Parameter: 0.373795
- andy1
  - Data Name: 200809181026014
  - Transmittance (R): 81.1 [%]
  - Median Size: 9.35329 [μm]
  - R Parameter: 0.069234
What we’ll talk about

- Measurement tools
- Data analysis tools
- Data verification tools
- Q&A
Thank you

Danke

Большое спасибо

Grazie

谢谢

Gracias

 obrigado

धन्यवाद

Ashk

Σας ευχαριστούμε

Tacka dig

감사합니다

오늘로 가시기

Explore the future

Automotive Test Systems | Process & Environmental | Medical | Semiconductor | Scientific

© 2012 HORIBA, Ltd. All rights reserved.