Polyolefin Online Classification and Online SPC in LabWare LIMS

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Borealis
Borealis at a Glance

- Leading provider of chemical and innovative plastics solutions that create value for society
- More than 50 years of experience
- Unique Borstar® technology to develop polyolefin solutions that are tailored to customers’ needs
- 5,100 employees in over 20 countries
- Ownership 64% IPIC / 36% OMV
- Joint venture in Middle East and Asia: Borouge (Abu Dhabi)
PE and PP Producers 2011: Borealis/Borouge 8\textsuperscript{th} in World, Borealis 2\textsuperscript{nd} in Europe

Capacities 2011 based on ownership share %

**World**
(Capacities based on % ownership of units/effective sales of output)

**Western Europe**
(Capacities based on % ownership of units/effective sales of output)
Polyethylene and Polypropylene QC testing

- Melt flow rate
- Pellet size & shape
- Density
- Mechanical properties
- Contamination
Polyethylene and Polypropylene offline samples

1. Operator takes sample
2. Sample is taken to QC lab
3. QC testing

PRODUCTION BATCH
~ 20 – 500t

BATCH SAMPLE
~ 1 - 25kg

SAMPLE ALIQUOT
< 1kg
Product flow and quality control in a Borstar PO plant

Online Polymer Analysis (OLPA)

- NMR
- Rheometer
- Optical inspection NMR

Powder samples
- QC lab powder testing
  - Xylene solubles, Ethylene content...

Pellet samples
- QC lab pellet testing
  - Melt flow rate, Xylene solubles, Ethylene content, Mechanical properties, Optical inspection...
Example: Rheometer trend
Example: Film inspection
# Online Polymer Analysis (OLPA) vs Offline testing

<table>
<thead>
<tr>
<th>Process control &amp; trending</th>
<th>Offline testing</th>
<th>OLPA</th>
</tr>
</thead>
<tbody>
<tr>
<td>Low sample frequency</td>
<td>Continuous results (trends)</td>
<td></td>
</tr>
<tr>
<td>Sample preparation &amp; analysis take time (Density: 2 hours)</td>
<td>Immediate results</td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Online classification</th>
<th>Offline testing</th>
<th>OLPA</th>
</tr>
</thead>
<tbody>
<tr>
<td>Lot release delays (mechanical properties: 24h)</td>
<td>Real time lot release</td>
<td></td>
</tr>
<tr>
<td>Classification based on homogenised sample (a few kg)</td>
<td>Full final blend statistics</td>
<td></td>
</tr>
</tbody>
</table>
Automatic Online Classification (OLC)

- Fully automatic collection of OLPA measurement results (AspenTech InfoPlus.21)
- Automatic processing & statistics calculation (IP.21 OLC module)
- Integration into batch classification procedure in LabWare LIMS
  - Average
  - Minimum, maximum, standard deviation
  - Online coverage factor
- Installed by LabWare and AspenTech in 2007
- Replaces Excel sheets, VB macros, DCS printouts
OLC overview

- Instrument PC
- DCS
- Analysis data
- Batch details
- IP.21 plant server
- Analysis data
- Batch details
- Trends
- Batch statistics
- IP.21 OLPA server (OLC module)
- LabWare LIMS

<table>
<thead>
<tr>
<th>M230/2 [MFR 230°C/2.16kg]</th>
<th>MI230/2 OLC AVG</th>
<th>19.4</th>
</tr>
</thead>
<tbody>
<tr>
<td>MI230/2 OLC MAX</td>
<td>21.1</td>
<td></td>
</tr>
<tr>
<td>MI230/2 OLC STDEV</td>
<td>0.9466</td>
<td></td>
</tr>
<tr>
<td>MI230/2 OLC COVERAGE</td>
<td>0.913</td>
<td></td>
</tr>
</tbody>
</table>

- Instrument PC
- IP.21 OLPA server (OLC module)
- LabWare LIMS
OLC overview

IP.21 OLPA server (OLC module)

Datapoints

Statistics for LIMS

LabWare LIMS Background service

automatic file import

OLC sample

Event trigger OLC subroutine

Batch sample

Background service

Event trigger

OLC subroutine

Statistics for LIMS

Datapoints

IP.21 OLPA server (OLC module)

OLC overview

The diagram shows the process flow for OLC in LabWare LIMS. It involves several steps:

1. Datapoints are collected and sent to the IP.21 OLPA server (OLC module).
2.Automatic file import triggers the OLC sample.
3. Event trigger activates the OLC subroutine.
4. The LabWare LIMS Background service processes the data.

The diagram also includes a screenshot of a spreadsheet showing data points, indicating the format and type of data processed by the system.
OLC overview

Online Classification and Online SPC in LabWare LIMS

**IP.21 OLPA server (OLC module)**

**Datapoints**

**Statistics for LIMS**

Automatic file import

**OLC sample**

Event trigger OLC subroutine

**LabWare LIMS Background service**

**Batch sample**

---

<table>
<thead>
<tr>
<th>Field</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sample Template</td>
<td>sample.template</td>
</tr>
<tr>
<td>Sample Text ID</td>
<td>sample.text_id</td>
</tr>
<tr>
<td>Sample Batch Name</td>
<td>sample.batch_name</td>
</tr>
<tr>
<td>Sample Product</td>
<td>sample.product</td>
</tr>
<tr>
<td>Test</td>
<td>sample.test</td>
</tr>
<tr>
<td>Analysis</td>
<td>sample.analysis</td>
</tr>
<tr>
<td>Result Name</td>
<td>result.name</td>
</tr>
<tr>
<td>Result Result Type</td>
<td>result.result_type</td>
</tr>
</tbody>
</table>
| Data File                   | "\Sw07\3PPS\BatchData\2014-04\Sample\MFR\DataPoints.csv", "T"
| Mean                        | "35.3243", "N"
| Std                         | "1.33191", "N"
| Minimum                     | "33.1082", "N"
| Maximum                     | "37.0896", "N"
| Coverage                    | "1", "N"
| Coverage Limit              | "0.9", "N"
| Weighted Average            | "35.6386", "N"
| Weighted Standard Deviation | "1.32949", "N"
| Average Moving Range        | "0.0964534", "N"
| UCL                         | "35.7809", "N"
| LCL                         | "35.267", "N"
| CP                        | "19.491", "N"
| CPK                          | "17.4474", "N"
| Cpl                          | "21.5352", "N"
| Cpk                          | "17.4474", "N"
| LIMS ANALYSIS NAME          | "BTM66149", "T"
| LIMS ANALYSIS_TYPE          | "BTM", "T"
| LIMS COMPONENT              | "R23", "T"
| LIMS SIGNAL_TYPE            | "T"
| OLC Complete                | "Ok", "T"
OLC overview

IP.21 OLPA server (OLC module)

Datapoints
Statistics for LIMS

automatic file import

OLC sample

Event trigger OLC subroutine

Batch sample

LabWare LIMS Background service

OLC Result: MFR
Data File
Mean
Std
Minimum
Maximum
Coverage
Coverage limit
Weighted average
Weighted standard deviation
Average moving range

UCL
LCL
Cp
Cpk
Cpu
Cpl

LIMS_ANALYSIS_NAME
LIMS_ANALYSIS_TYPE
LIMS_COMPONENT
LIMS_INSTRUMENT
ANALYSIS_SIGNAL_TYPE
Complete

OLC overview

Online Classification and Online SPC in LabWare LIMS
OLC overview

IP.21 OLPA server (OLC module)

Datapoints

automatic file import

Statistics for LIMS

OLC sample

Event trigger OLC subroutine

LabWare LIMS Background service

Batch sample

OLC sample for MFR / MI230/2

Sample Number
Analysis
Version
Test Status
Date Received
Date Started
Date Reviewed
Reviewer
Date Completed
Stage
Test Number
X Sent Sap
X Total Points

OLC subroutine
LIMS batch sample
Online SPC in LabWare LIMS & AspenTech IP.21

LabWare LIMS
- LIMS sample: lab results (entered by operator)
- Online SPC settings (custom table)
  - Tolerance
  - Time offset
  - Time window

Event trigger subroutine
Lab result + test number + sampling time + limits

AspenTech InfoPlus.21
Online SPC Module
- Get average result from online tag
- Report if the online measurement is stable (calibration allowed)
- Report if the online measurement is within limits

Automatic import
online average / STDEV + info text (OK/NOK)
Online SPC in LabWare LIMS & AspenTech IP.21

<table>
<thead>
<tr>
<th>Sample Number</th>
<th>Analysis</th>
<th>Version</th>
<th>Test Status</th>
<th>Date Received</th>
<th>Date Started</th>
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<tbody>
<tr>
<td>3794176</td>
<td>BTM14580</td>
<td>1</td>
<td>In Progress</td>
<td>01.06.2014 01:30:30</td>
<td>01.06.2014 01:55:34</td>
</tr>
</tbody>
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**BTM14580 [Ethylene content] / 1**

| C2 Total | ✔️ 4.0% |
| C2 / AM | ✔️ |
| C2 Total Online | ✔️ 3.8% |
| C2 Total CAL flag | ✔️ OK |
| C2 Total OLC flag | ✔️ OK |
| C2 Total Online STDEV | ✔️ 0.0671% |
| C2 Total On/Offline Diff. | ✔️ -5.0% |

**Offline lab result**

**Online SPC results + Calculated result**
Presenting online results using Enhanced Visual Workflows

Online data (.txt file)

LIMS data (SPC samples)
Presenting online results using Enhanced Visual Workflows
Presenting online results using Enhanced Visual Workflows

OLC Result: MFR
Mean: 42.1301
Std: 0.619023
Minimum: 40.8924
Maximum: 42.3615
Coverage: 1
Coverage limit: 0.9
Weighted average: 42.1425
Weighted standard deviation: 0.613064
Average moving range: 0.136657
UCL: 42.4936
LCL: 41.7666
Cp: 13.7571
CpL: 21.6524
Cpl: 5.86082
Cpk: 5.86082

MT230/2 (g/10min)

<table>
<thead>
<tr>
<th>period start</th>
<th>period end</th>
<th>material qty</th>
<th>result</th>
<th>data valid</th>
<th>environment</th>
<th>outlier</th>
<th>online spc</th>
<th>spc sample</th>
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</thead>
<tbody>
<tr>
<td>03/02/2013 14:21:52</td>
<td>03/02/2013 14:26:52</td>
<td>2.99227</td>
<td>43.1563</td>
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<td>2.98851</td>
<td>43.115</td>
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<td>03/02/2013 14:31:52</td>
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<td>3.00295</td>
<td>43.2775</td>
<td>OK</td>
<td>OK</td>
<td>OK</td>
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<tr>
<td>03/02/2013 14:36:52</td>
<td>03/02/2013 14:41:52</td>
<td>2.99656</td>
<td>43.225</td>
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<tr>
<td>03/02/2013 14:41:52</td>
<td>03/02/2013 14:46:52</td>
<td>2.98837</td>
<td>43.0754</td>
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<td>03/02/2013 14:51:52</td>
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<td>2.98926</td>
<td>42.8162</td>
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<td>03/02/2013 14:56:52</td>
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<td>03/02/2013 15:01:52</td>
<td>03/02/2013 15:06:52</td>
<td>3.00501</td>
<td>42.7557</td>
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<tr>
<td>03/02/2013 15:06:52</td>
<td>03/02/2013 15:11:52</td>
<td>3.00695</td>
<td>42.8529</td>
<td>OK</td>
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<tr>
<td>03/02/2013 15:11:52</td>
<td>03/02/2013 15:16:52</td>
<td>3.00695</td>
<td>42.8529</td>
<td>OK</td>
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<td></td>
</tr>
<tr>
<td>03/02/2013 15:16:52</td>
<td>03/02/2013 15:21:52</td>
<td>3.00501</td>
<td>42.7557</td>
<td>OK</td>
<td>OK</td>
<td>OK</td>
<td>OK</td>
<td></td>
</tr>
</tbody>
</table>

Chart Legend:
- Online
- Online NOC
- Online SPC sample
- OLC details
LabWare LIMS Online classification experiences

- LabWare LIMS flexible enough to support complicated Online Classification procedure

- Online quality data structured and presented in an organised way (SPC, Offline and Online batch data)

- Extracted online quality statistics in LIMS are supporting product consistency improvement projects

- Availability of LIMS has become more important (24/7 uptime)

- Amount of results in the LIMS database has increased significantly
  - >100 extra results in LIMS per batch
  - 30% of the results table consists of OLC statistics results

- Regular performance tuning required (subroutine optimisation, background services, indexing)
Results created per year

• 36.5 Million total results since 2007
• 14.4 Million OLC statistics results
Thank you

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