

On-line polyethylene additives
analysis using a diode array uv /
visible spectrophotometer and
Charm Works

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Process Analysis and Automation

Process Analysis & Automation

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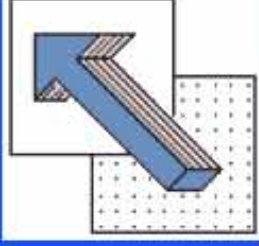
Process Analysis & Automation

- Process analysis systems
- Multivariate process analysis software
 - ◆ Charm Works
- Sonic product development
- Acoustic emission monitoring systems
- Laboratory automation

Applications - vertical markets

- Antioxidants in polyolefin melts
- Automation of standard tests
 - ◆ permanganate time
 - ◆ Hazen colour
- Batch processing
- Solvent purity
- Multicomponent stream composition

HAMILTON



**Custom Sensors
& Technology**

**PROCESSES &
ANALYSIS
AUTOMATION**

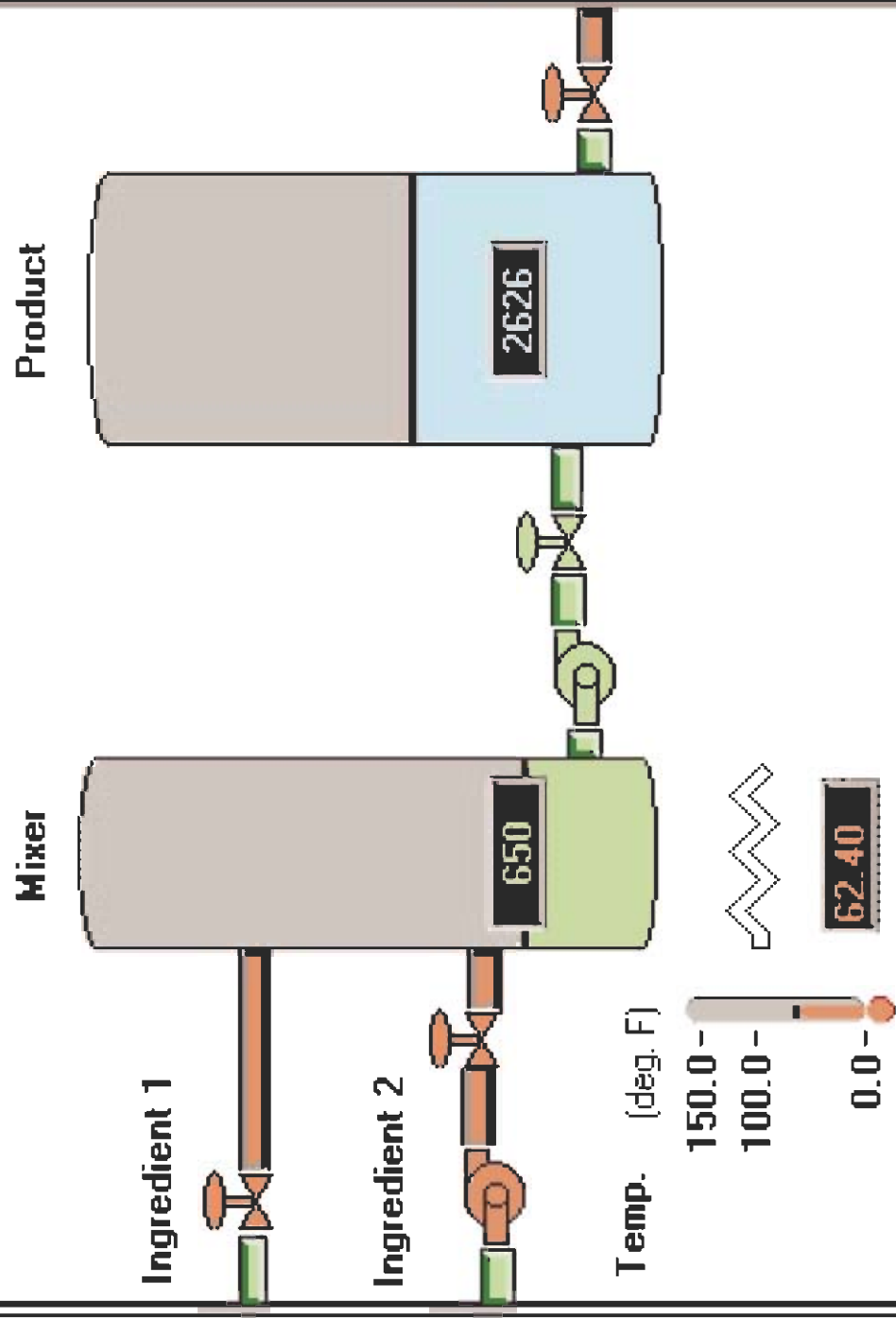


LabVIEW

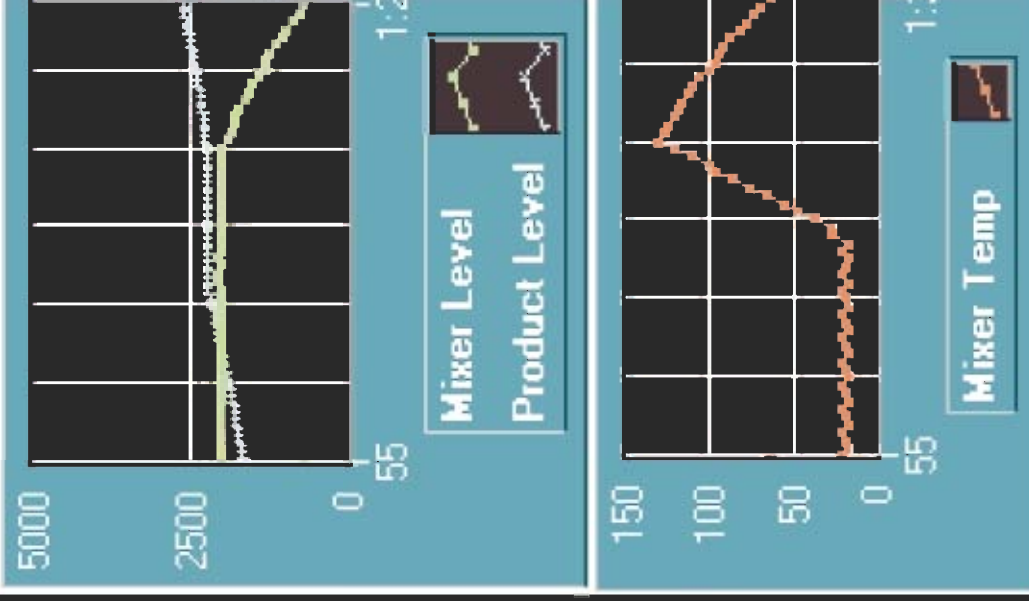
- Graphical programming environment
- Designed specifically for instrumentation
- Good graphical user interface
- High connectivity
- Charm Works add on toolkit provides chemometrics



Process



Operating Trends



RETURN [F4]

MORE INFO... [F5]

IN AUTO MODE

MANUAL [F3]

HELP [F1]

Charm Works

- Chemometrics for real time process analysis
- LabVIEW tool kit
- PLS, PCA, SIMCA
- Easy to use

**Instrument
drivers**

**SPC
toolkit**

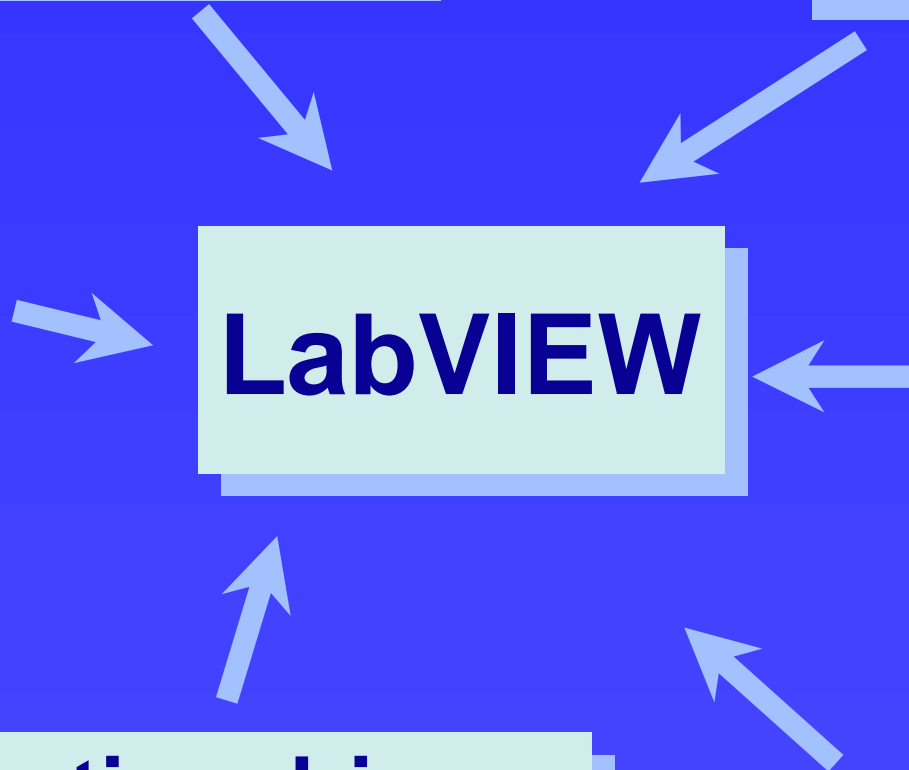
**Windows
DLL,
DDE, CINS**

LabVIEW

**SQL
toolkit**

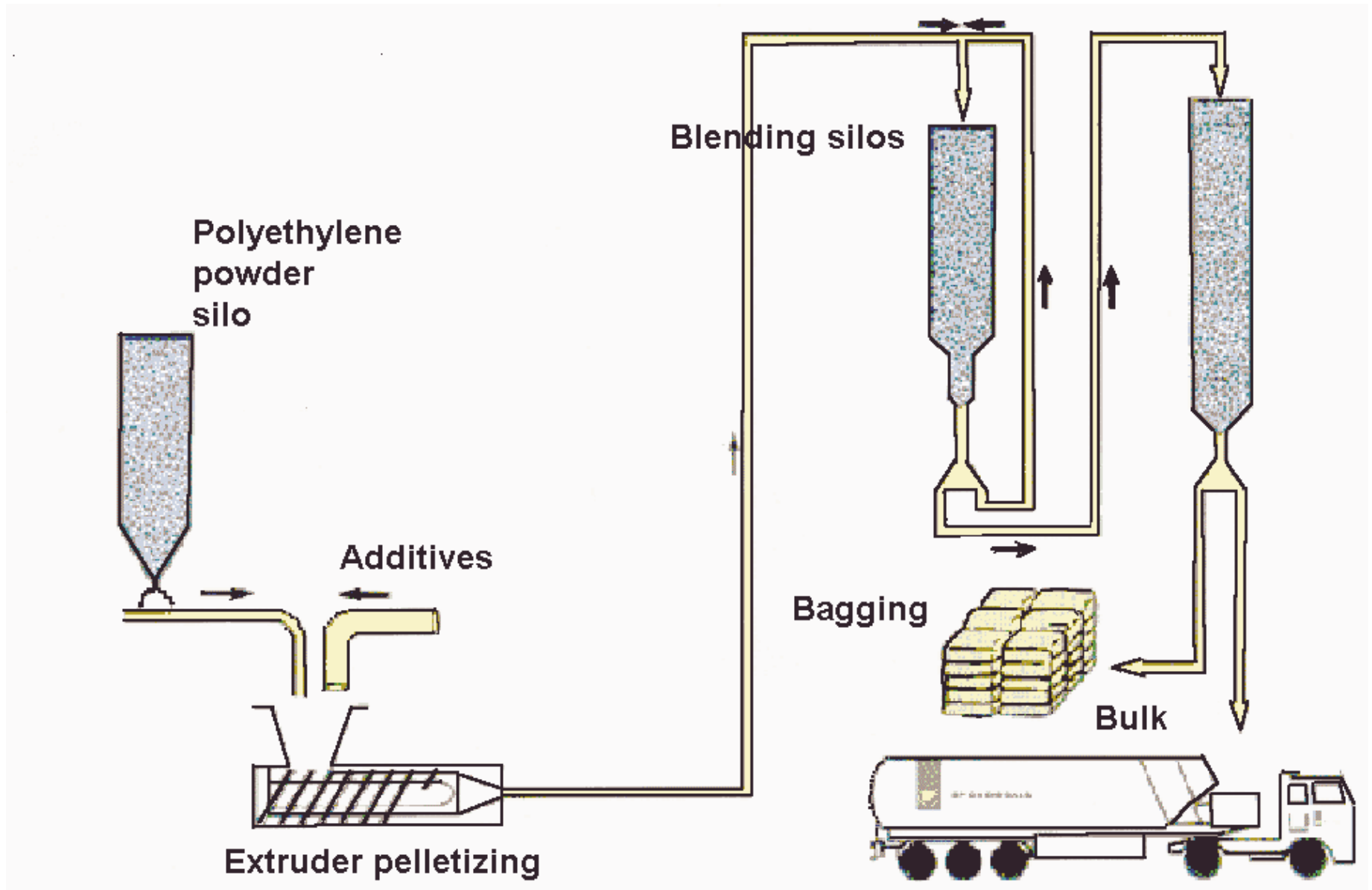
**Communication drivers
TCP/IP, serial, ModBus,
FieldBus**

Charm Works



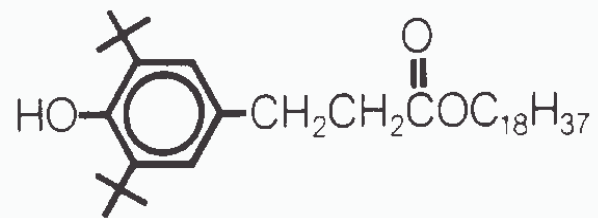
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Polyethylene manufacturing

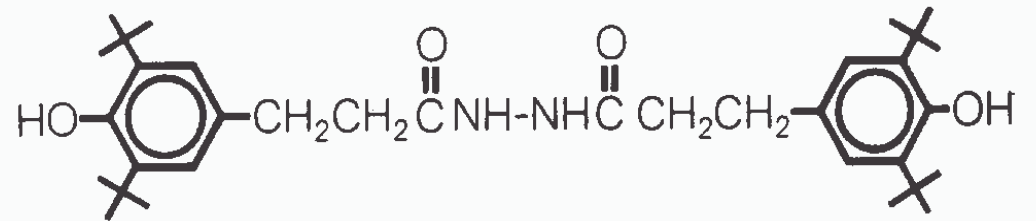


Antioxidants

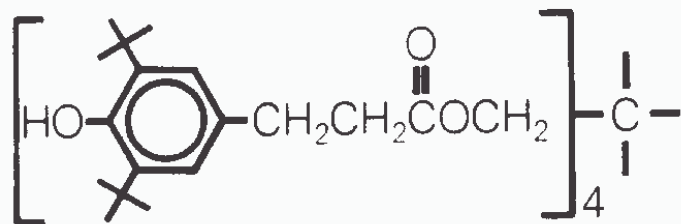
IRGANOX 1076



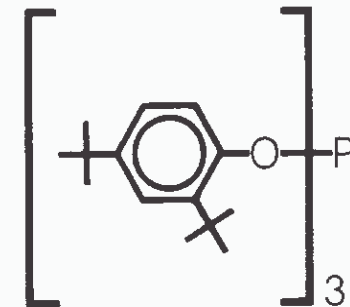
IRGANOX MD 1024



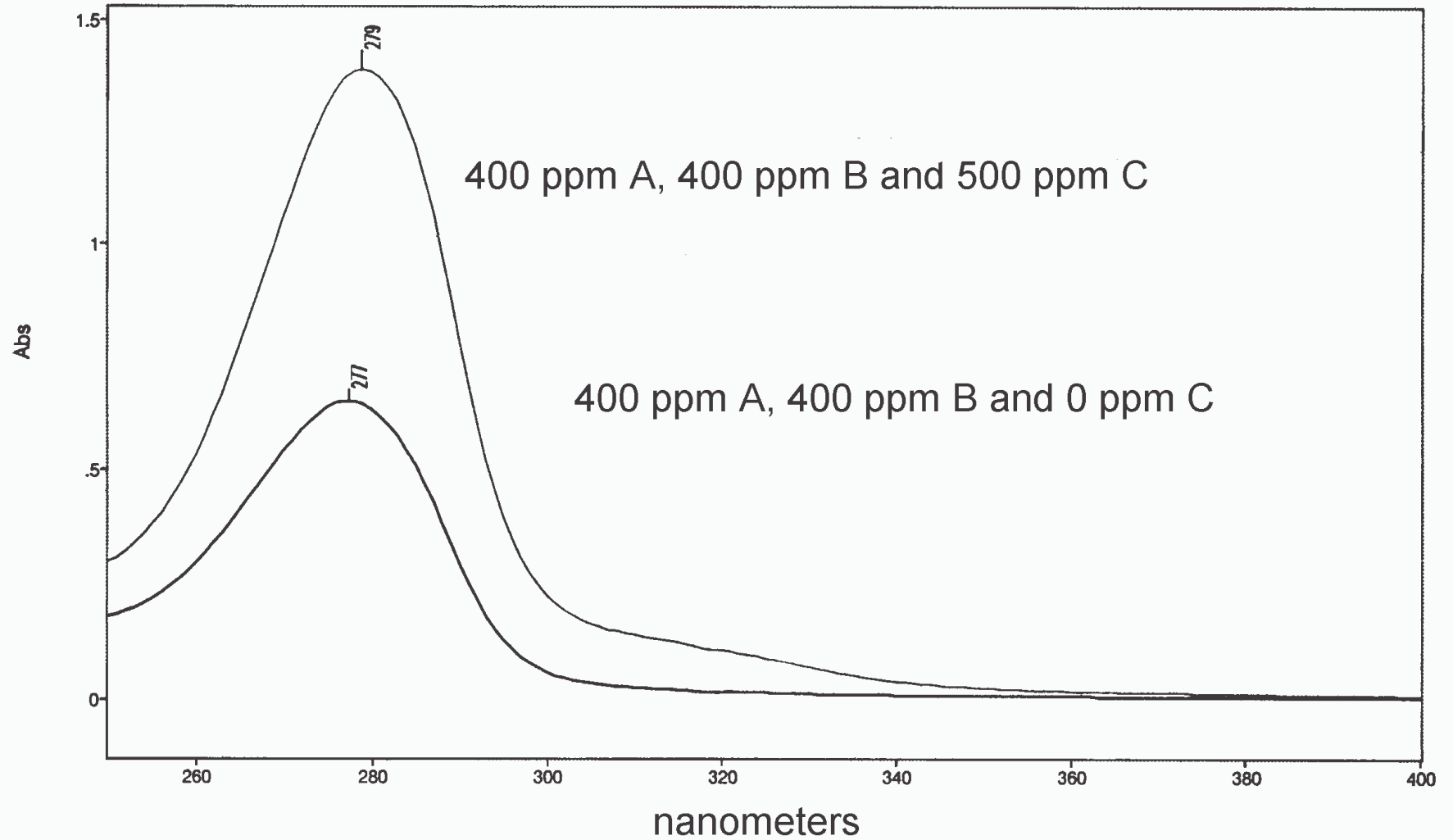
IRGANOX 1010

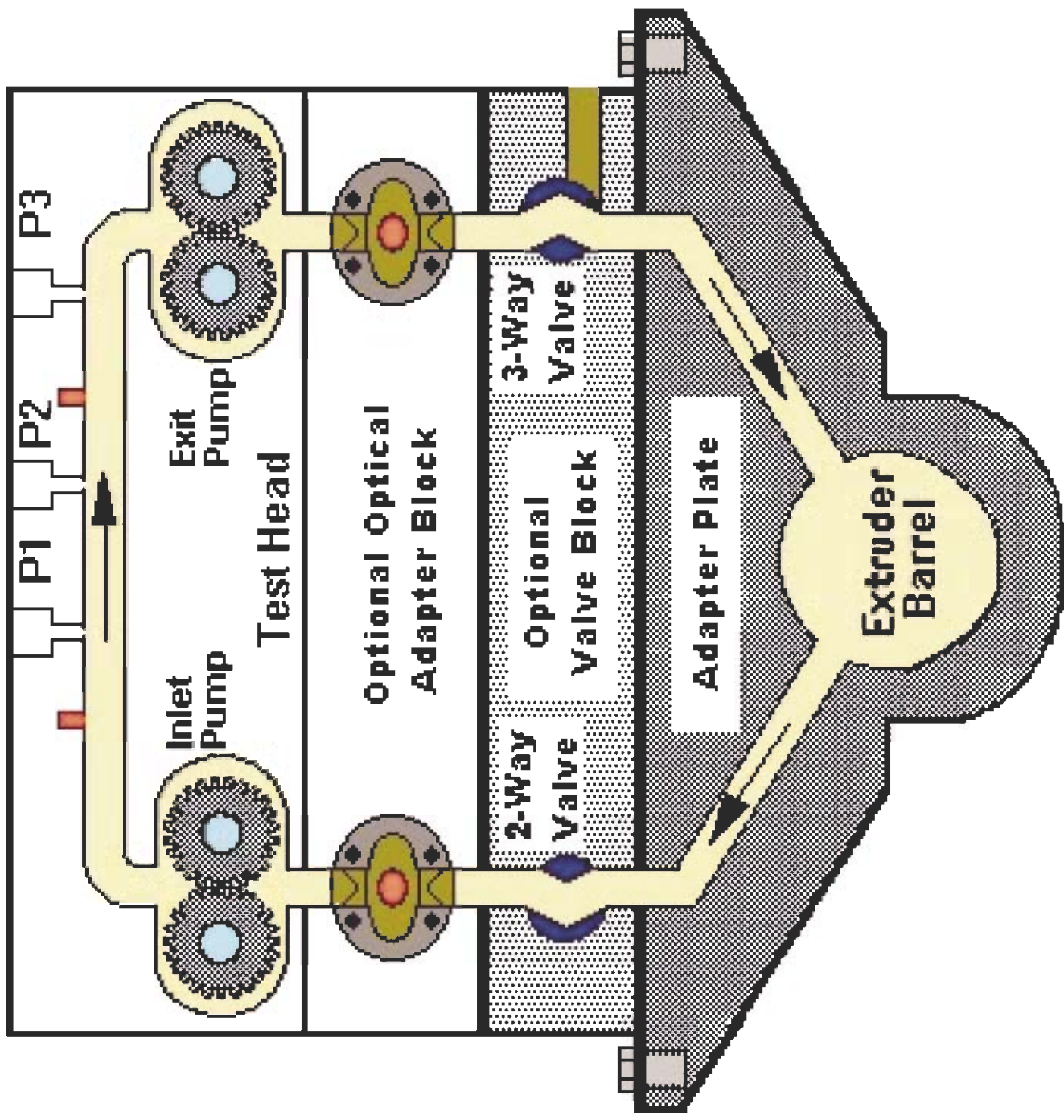


IRGAFOS 168



Additives Spectra

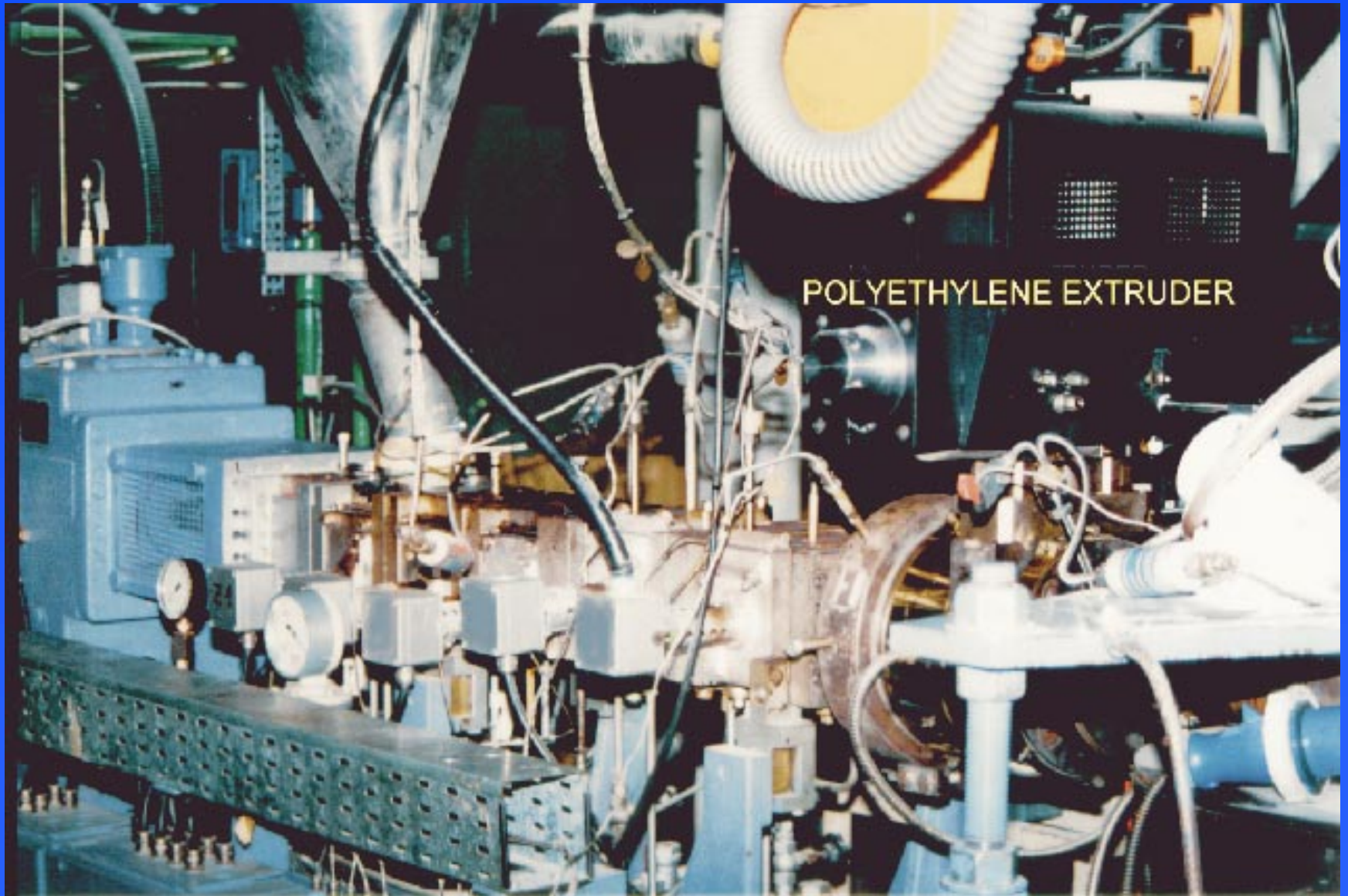




Rheometric PCR-320



The extruder



Objectives

- Analyse three anti-oxidant additives concentration in real time
- On-line in the melt (up to 250°C)
- Manage all of the functions in a single, integrated, easily maintainable application

Basic approach

- Spectroscopy (proven off-line)
- Probes and optical fibres
- Software environment (connectivity)
- PLS prediction model (1 off-line / 2 on-line)
- Reliable engineering (essential)
 - ◆ opto-isolation (analog + digital)
 - ◆ watchdog

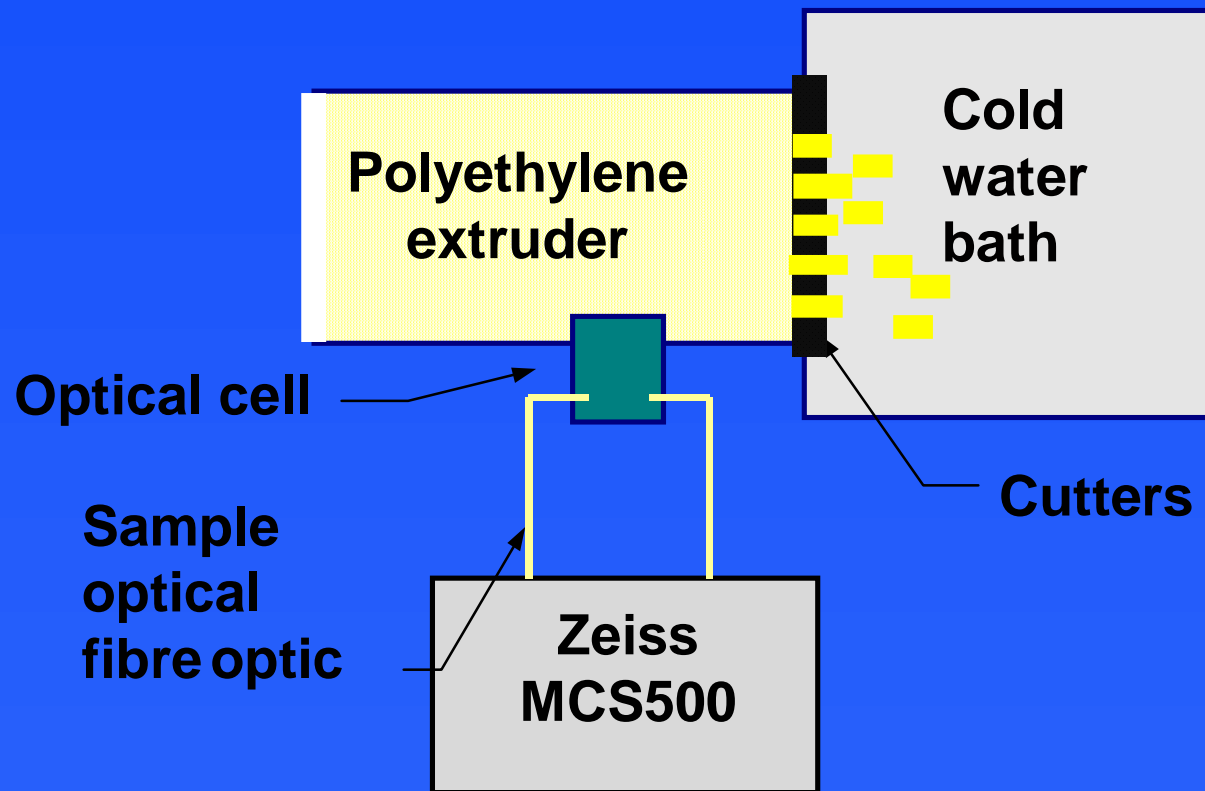
Technology

- In-line probes (melt) - SpectrAlliance
- Fibre optics as interface
(UV Quartz grade 600 μ m)
- Zeiss MCS500, PDA spectrometer
- National Instruments LabVIEW system
- PLS algorithm (Charm Works)

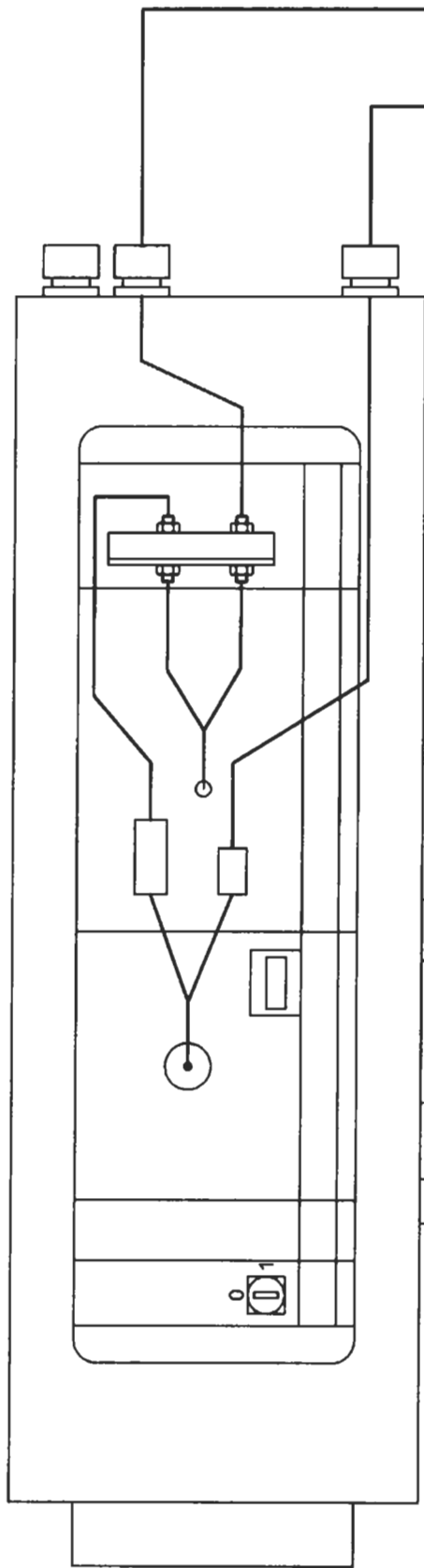
Zeiss Spectrometer - MCS500



Monitoring system

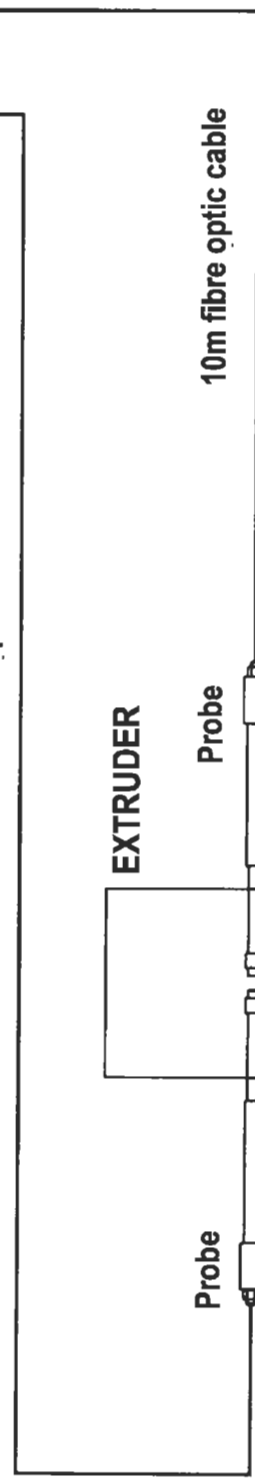


SPECTROPHOTOMETER

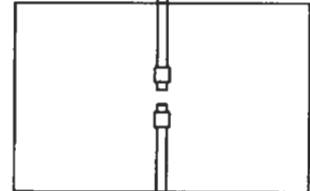


TO PC

10m fibre optic cable



EXTRUDER



Probe

Probe

10m fibre optic cable

Spectrometer functions

- Control and data transfer via RS-422
- Choose averaging time and spectral range
- Initiate scans and down load data
- Monitor reference spectrum (alarm on 60 % of initial integrated value for 370-400nm)

Communications

- Serial communications with site VAX computer
 - ◆ incorporation of PLS predicted data into site database
- Digital alarms to local enunciator panel
- Digital inputs
 - ◆ reset alarms
 - ◆ rheometer fault

Additional I/O

■ Digital outputs

- ◆ analyser fault
- ◆ bad prediction (high Mahalanobis distance)
- ◆ low additive concentration alarm
- ◆ low melt flow or extruder stopped
- ◆ PC Watchdog

■ Digital inputs

- ◆ sample temperature alarm
- ◆ reset low flow

Error detection / correction

- Monitor reference spectrum (alarm on 60 % of initial integrated value for 370-400nm)
- Monitor prediction quality statistics to warn of calibration problems
- To detect flow problems in the rheometer by signal noise
- Correct for spectral offsets

PLS2 modelling

■ X block

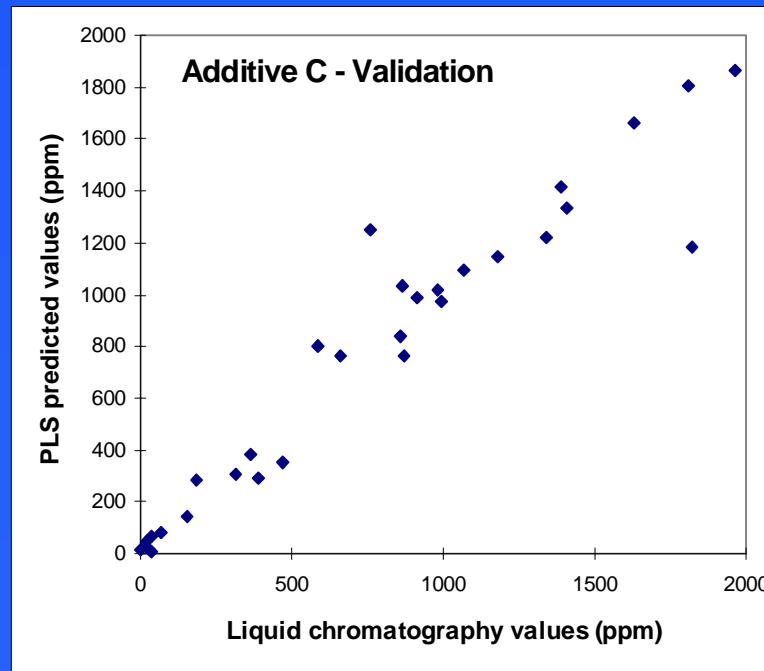
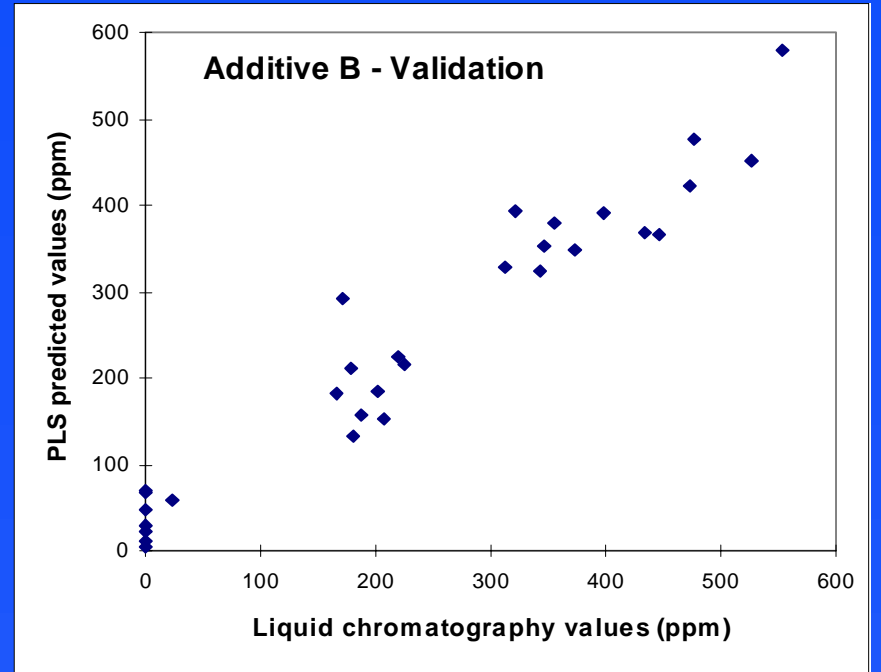
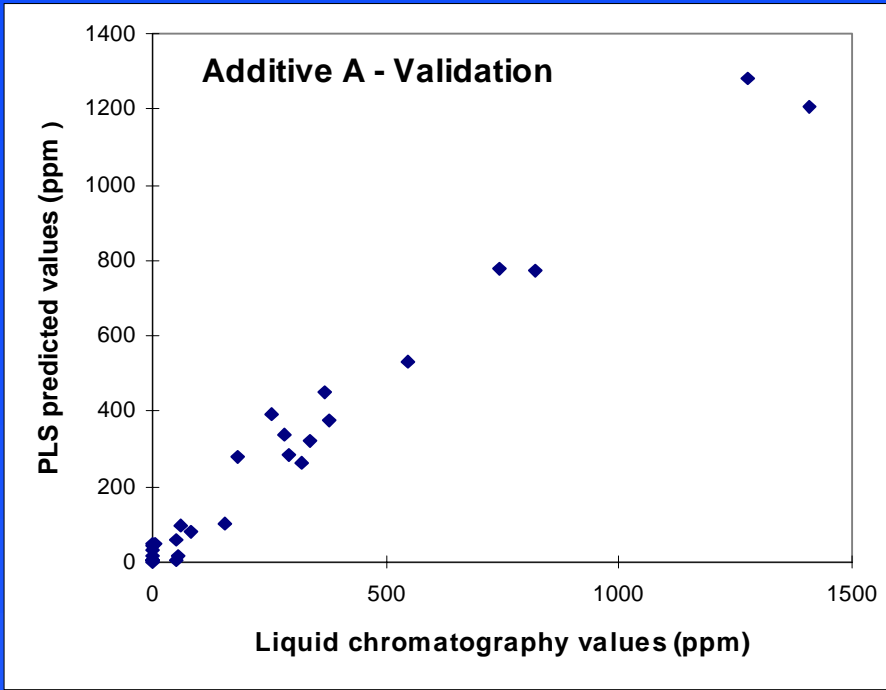
- ◆ spectra 243 - 400 nm (180 data points)
- ◆ mean centred scaling

■ Y block

- ◆ LC data (concentration of A, B and C)

■ 20 samples used initially to build model

■ Model extended on-line



Conclusions

- The Zeiss MCS500 spectrometer has produced good spectral results for polyethylene/polypropylene additives
- PLS modelling has proved effective for analysing 3 non-resolvable anti-oxidant additives
- LabVIEW has enabled a fully integrated process analysis system to be developed in a short period of time at low cost

New developments

■ Charm Works 99

- ◆ automatic baseline correction
- ◆ calibration free prediction

■ New applications

- ◆ batch processes
- ◆ reacting systems
- ◆ NIR