PAL SYSTEM
Ingenious sample handling

Application Note

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CHEMICAL

Automated pipetting and analysis of polymer suspensions for process control in chemical production

Pipetting suspensions for process control in chemical production

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Introduction

AkzoNoble is the producer of Expancel. Expancel microspheres (fig. 1) are small spherical thermoplastic particles. The microspheres consist of a polymer shell encapsulating a gas. When heated, the internal pressure from the gas increases and the thermoplastic shell softens, resulting in a dramatic increase of the volume of the microspheres. The gas remains inside the spheres.

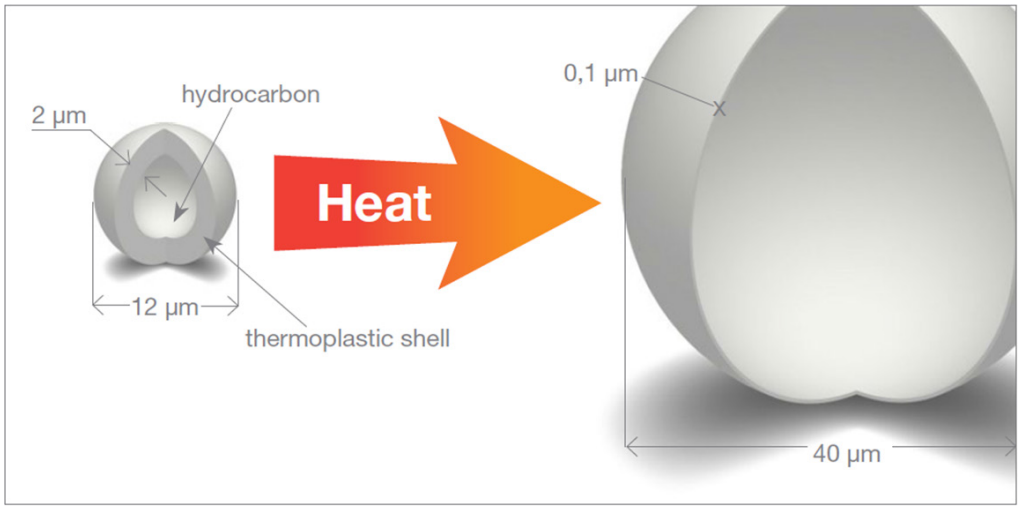


Fig 1: Enlargement of a heated Expancel microspehere

Process control

During the production of Expancel samples are regularly analysed for a number of critical parameters (fig. 3). So far this sample preparation has been a manual process. To increase productivity a semi-automated sample preparation has been evaluated. 100 mL samples are still taken manually at line. However, the processing and analysis of the samples should then be fully automated.
A critical point in the evaluation of the automated sample prep was to find out if pipetting can be used to reliably transfer defined amounts of the sample suspension (see cover photo).

Fig 2: A sample for the analysis of critical parameters is taken

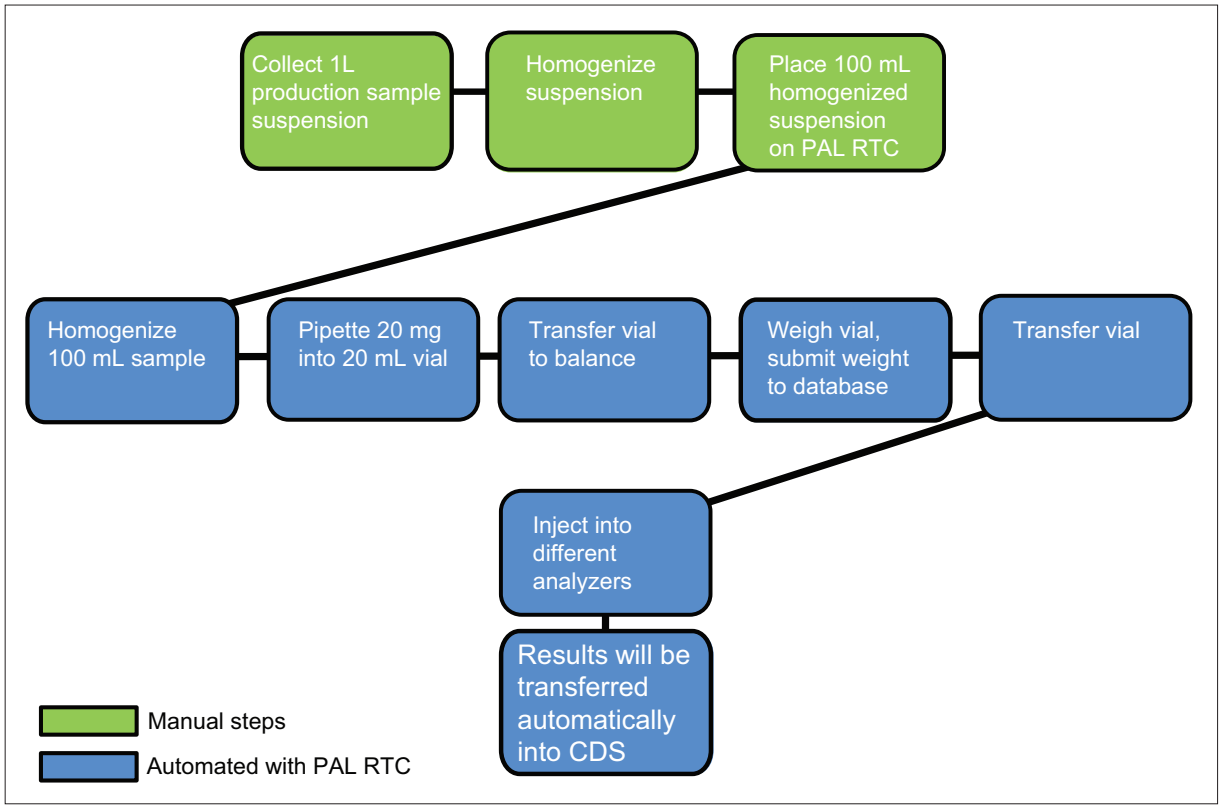


Fig. 3: Process steps for the at-line analysis of Expancel production batches

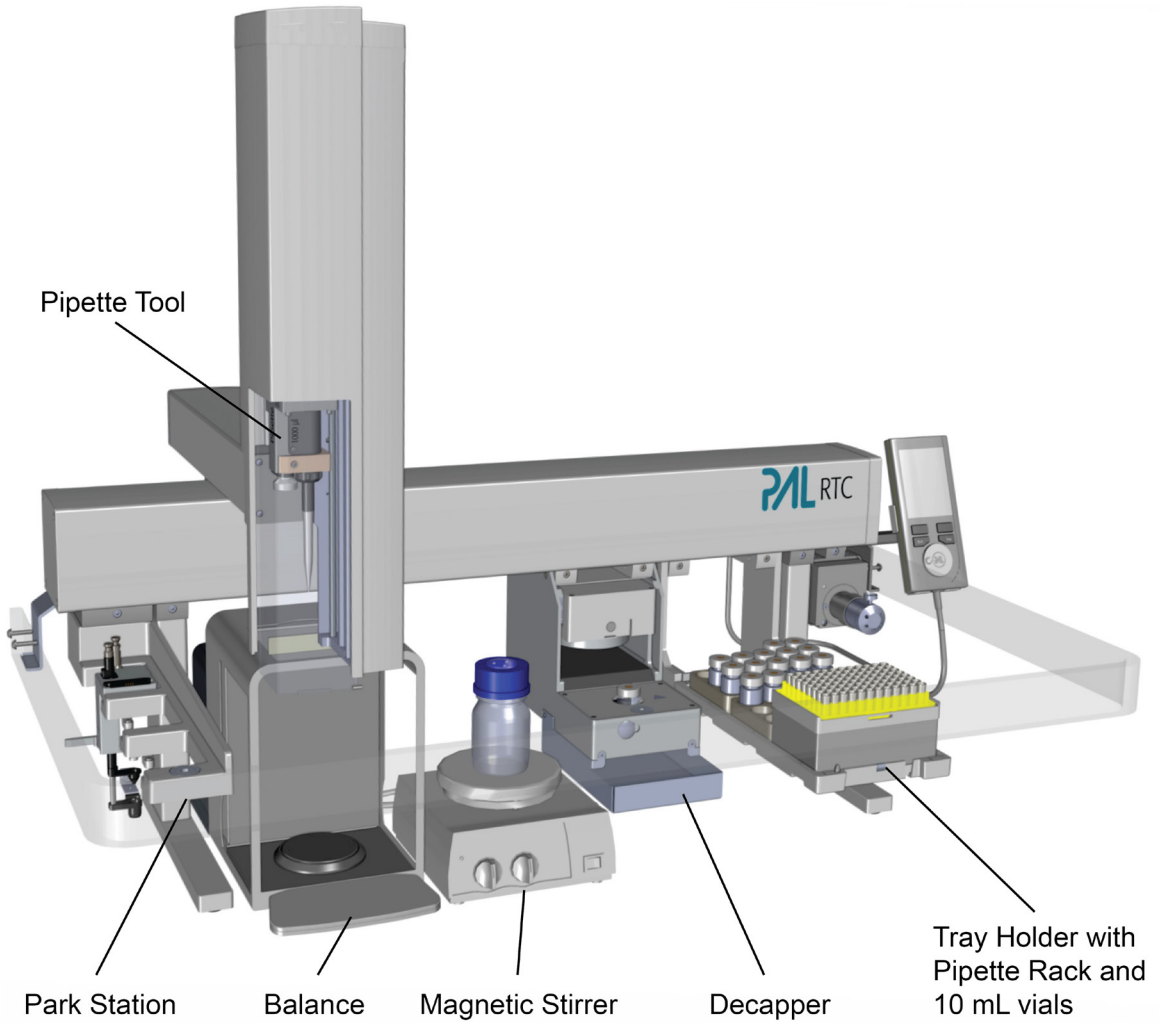
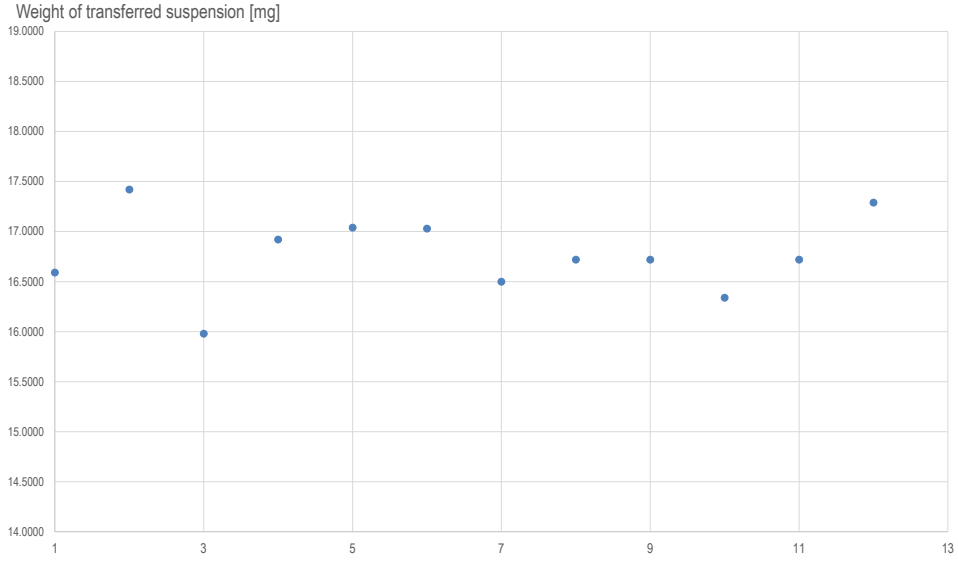


Fig. 4: Out-line of hardware set up for automation

Fig.5 PAL RTC setup for automated weighing of pipetted samples

Application Note: Weighing samples automatically: Gravimetric dilutions of highly viscous samples



Sample	Weight Tara [mg]	Weight Gross [mg]	Weight Net [mg]
1	17.64339	17.65998	16.5900
2	17.54938	17.5668	17.4200
3	17.67467	17.69065	15.9800
4	17.58607	17.60299	16.9200
5	17.60297	17.62001	17.0400
6	17.61459	17.63162	17.0300
7	17.69568	17.71218	16.5000
8	17.53305	17.54977	16.7200
9	17.45578	17.4725	16.7200
10	17.53061	17.54695	16.3400
11	17.70394	17.72066	16.7200
12	17.70634	17.72363	17.2900
Average			**16.77250**
SD			**0.40200**
%RSD			**2.40**

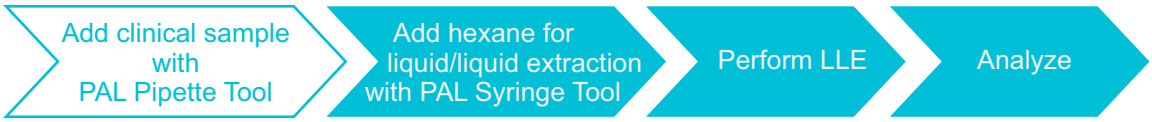
Fig 6: Typical results for a particular production batch

PAL Pipette Tool – Essential for sample preparation

Pipetting liquids with tips is a common practice in many applications. The most important feature is probably the fact that carryover is zero by design because the used tip is discarded after every sample transfer. Moreover, functionalized tips (e.g. for desalting or protein capture) allow for effective sample work-up.

The new PAL Pipette Tool significantly enhances the PAL System sample prep platform. In combination with the complete range of tools and modules the PAL Pipette Tool facilitates seamless lab workflows, especially in combination with the PAL DeCapper Module:

Only the PAL RTC can switch automatically between pipettes and positive displacement syringes. Pipettes are free from carryover by design. Chemically inert glass syringes are best when highest accuracy and compatibility with organic solvents is required.



Application areas:
- Serial dilutions
- Micro SPE in a tip
- Protein digestion in a tip
- Reformatting of samples
- Pipetting of suspensions (e.g. magnetic beads) and emulsions

Highlights:
- Carryover free sample handling
- Complete workflows in combination with PAL Tool and Modules
- Direct injection from the Pipette Tool into HPLC valves (see fig. 1)
- Glass syringe (positive displacement) and Pipette Tool on one system
- Tested PAL Consumables like pipette tips and racks enhance the performance further

Technical data, ordering information:

0-200 µL P/N PAL3-TH-Pip-200
0-1000 µL P/N PAL3-TH-Pip-1000

PAL3 Firmware: 2.4.x or later

Detailed specifications are available.

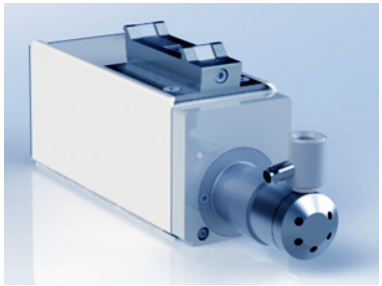


Fig. 7: Wash module and adapter for LC-injection port with Pipette Tool (PN PAL3-WashPip)

Fig. 8: Pipette Tool placed in Park Station

http://www.palsystem.com/index.php?id=284

Pipette Tool, Gravimetric Specifications

Repeatability, accuracy and linearity, single dispense, 200 µL Pipette Tool

n=7

Dispense Volume	Repeatability	Accuracy	Linearity
20 µL	RSD ≤ 1.5 %	≤ 2 %	
100 µL	RSD ≤ 0.5 %	≤ 1 %	$R^2 \geq 0.999$
200 µL	RSD ≤ 0.5 %	≤ 1 %	

Conditions:
Pipette Tool 200 µL, measured with two different tools
Liquid: water, 23 °C, density 0.997538 g/mL
Balance used: Precisa, XB 120A (d=0.1 mg)
PAL System 200 µL tips

Liquid Class Parameter	Liquid Class: Water (Single Dispense)
Airgap, front [µL]	0
Airgap, rear [µL]	100.0
Aspirate delay [ms]	300
Aspirate flow rate [µL/s]	100.0
Correction factor	1.04
Dispense delay [ms]	100
Dispense flow rate [µL/s]	100.0
Volume offset [µL]	1.6
Volume, front [µL]	0
Volume, rear [µL]	0

PAL Sample Control Method:
Aspirate Dispense with Liquid Class_Rev1

Repeatability, accuracy and linearity, single dispense, 1000 µL Pipette Tool

n=7

Dispense Volume	Repeatability	Accuracy	Linearity
100 µL	RSD ≤ 0.5 %	≤ 0.5 %	
500 µL	RSD ≤ 0.3 %	≤ 0.5 %	$R^2 \geq 0.999$
1000 µL	RSD ≤ 0.3 %	≤ 0.5 %	

Conditions:
Pipette Tool 1000 µL, measured with two different tools
Liquid: water, 23 °C, density 0.997538 g/mL
Balance used: Precisa, XB 120A (d=0.1 mg)
PAL System 1000 µL tips

Liquid Class Parameter	Liquid Class: Water (Single Dispense)
Airgap, front [µL]	0
Airgap, rear [µL]	20.0
Aspirate delay [ms]	300
Aspirate flow rate [µL/s]	100.0
Correction factor	1.026
Dispense delay [ms]	100
Dispense flow rate [µL/s]	500.0
Volume offset [µL]	5.9
Volume, front [µL]	0
Volume, rear [µL]	0

PAL Sample Control Method:
Aspirate Dispense with Liquid Class_Rev1

Conclusions:

- The PAL RTC with Pipette Tool allowed for the precise pipetting of the Expancel suspensions.
- The precision of the pipetting step has been assessed by automatic weighing.
- Batch to batch variations showed RSDs 1 - 4%.
- The automation of the sample preparation allows for a significant increase in productivity since sample preparation during the night or over weekends is now possible when no skilled lab personnel is available.

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Imprint

Date of print: 04.2018

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