PAL SYSTEM
Ingenious sample handling

Application Note

FOOD SAFETY ENVIRONMENTAL CHEMICAL

Weighing samples automatically: Gravimetric dilutions of highly viscous samples

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Short summary

This application note describes the automated gravimetric dilution of (viscous) samples.

Introduction

Dosing highly viscous samples is challenging. Such samples are frequently handled in the food, pharma or the oil sector. Normally it takes a dilution step before they can be analyzed. Since volumetric dosing is temperature-dependent the best way to accurately dilute the sample is weighing solvent and compound. A weighing step is applied to allow dilution to a known mass concentration.
The goal of the gravimetric sample preparation shown here is to dilute a highly viscous sample to a predefined mass concentration.
The CHRONOS software (http://www.axel-semrau.de/en/Software/Software+Solutions/Chronos-p-966.html) allows to automate the gravimetric dilution procedure. It controls both the PAL System sampler as well as a Mettler-Toledo balance. The built-in capability to perform the required calculations for the dilution greatly simplifies the whole process.

System setup

Dual Head PAL RTC-RSI, 120 cm
- 1mL Headspace Tool on the RSI head
- 1mL gastight Syringe 8mm with custom needle (FN 0,72 Gauge 22)
- Syringe Tools for 10, 100 und 1000 µL
- Parkstation
- Two wash stations
- Agitator Module, with adapter for 10 mL vials
- Vortex Mixer Module (optional)
- Tray Holder, VT 15
- Multiple Headspace Extraction Tool (recommended)
- LC Injection Valve (optional)

Mettler-Toledo balance XP205
Option to minimize electrostatic charging is recommended.

Results

Dilution methods are easily realized on PAL Sytems thanks to the Robotic Tool Change (s. application note http://www.palsystem.com/index.php?id=191). However, to handle highly viscous samples or even samples that are solid at room temperature some obstacles have to be overcome. Firstly samples have to be heated to reduce viscosity or to melt them (e.g. long chain ethers in cosmectic formulations) to be able to aspirate them with a syringe. Secondly it is crucial that there are no "cold spots" in the system because they may lead to the sample blocking the system. Therefore a heated Headspace Tool is being used. The standard syringe in this tool has been replaced by a custom one with a wider bore needle. To prevent clogging the needle has to be pre-heated by completely inserting it into a heated sample or destination vial for a defined time.

During the process each dilution vial is weighed four times: empty, after addition of the substance to be diluted, after a first and a second addition of the dilution solvent. With the first addition of solvent its density at the prevailing room temperature is determined. This value is required to calculate the volume of solvent necessary to achieve the desired dilution factor of the sample with the second addition.

Table 1 shows the results from diluting samples of semi-solid crude oil. The volume weighed was 200 µL each. Dilution solvent was hexane. The too low amount of the sample marked with an asterisk resulted from an almost empty sample vial. Even in this case the correct dilution factor has been achieved.

Table 2 shows the results from diluting motor oil. The volume weighed was 200 µL each. Dilution solvent was hexane.

Fig.1 Dual Head PAL with Mettler-Toledo balance

No.	Sample volume [µL]	Net weight sample [mg]	Net weight solvent [mg]	Dilution nominal value	Dilution actual value
1	200	165,3	165,0	1:2	1,998
2	200	167,2	164,2	1:2	1,982
3	200	167,7	169,8	1:2	2,013
4	200	166,8	167,3	1:2	2,003
5	200	167,5	164,9	1:2	1,984
6	200	168,4	165,9	1:2	1,985
7	200	167,8	167,3	1:2	1,997
8	200	171,2	170,1	1:2	1,994
9	200	167,1	165,2	1:2	1,989
10	200	169,3	172,1	1:2	2,017
11	200	171,8	168,4	1:2	1,980
12	200	166,3	167,4	1:2	2,007
13	200	92,4(*)	92,1	1:2	1,997
Mean value		168,0			1,996
Standard deviation		1,9			0,012
Rel. Standard deviation		1,13%			0,61%

Table 1

No.	Sample volume [µL]	Net weight sample [mg]	Net weight solvent [mg]	Dilution nominal value	Dilution actual value
1	200	170.8	171.0	1:2	2.001
2	200	169.0	168.3	1:2	1.996
3	200	170.0	171.0	1:2	2.006
4	200	168.6	168.2	1:2	1.998
5	200	169.6	169.5	1:2	1.999
6	200	168.3	166.7	1:2	1.990
7	200	169.0	169.1	1:2	2.001
8	200	168.8	169.3	1:2	2.003
9	200	166.9	166.8	1:2	1.999
10	200	171.3	174.6	1:2	2.019
11	200	172.0	171.7	1:2	1.998
12	200	164.1	164.7	1:2	2.004
Mean value		169.0			2.001
Standard deviation		2.1			0.007
Rel. Standard deviation		1.2%			0.3%

Table 2

Summary

The automated method described reliably and accurately dilutes highly viscous compounds, based on gravimetric measurements. Even solid samples, as long as meltable, can be handled.
The samples require heated sample storage and transfer. Aspirating and dispensing flow rates were optimized in order to achieve precise results.

CHRONOS is an open platform for device control that allows to dynamically respond to input values. This enables innovative solutions increasing the productivity of laboratories.

See the Balance PAL in action at Analytica 2016, May 10-13, Munich, Germany, booth A2.306, Axel Semrau GmbH. If you cannot make it to Analytica a video featuring this method is available: https://www.youtube.com/watch?v=qNr9P5jcv9s

Legal Statements

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