Automation of a Sample Preparation method for the analysis of Sodium benzoate in skin cream by RP-HPLC.

Application Note

Authors

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Abstract

A manual sample preparation workflow for the quantification of Sodium benzoate in skin cream was automated on an accroma® samplePrep system. The goal was to reduce extraction and overall sample preparation hands-on time without compromising on data quality. Sodium benzoate contents were determined using Reversed Phase HPLC, which was performed online (interfaced to accroma system). The reference standard and five samples of skin cream from the same containment were automatically prepared on the accroma system. The extraction was done by vertical shaking of accroTubes with 16mm steel balls as mixing aids. The measured Sodium benzoate contents of all five samples were within the manufacture's tolerances of 0.184 - 0.216%. The relative standard deviation of the five measurements were calculated to 0.82%. Extraction times could be reduced by 80% from 15 min (manually) to 3 min. The manual sample preparation hands-on time was estimated to be 20 min without considering documentation. The overall hands-on time could be reduced by 75% from 20 min to 5 min using the accroma system.

Introduction

Sodium benzoate is a preservative used in food (known as E211), pharmaceutical formulations and cosmetics to prevent bacteria and fungi from growing in the products. Contaminants may be introduced from environment or users. Therefore, it is important to assess content levels in cosmetics to ensure safety and quality during their shelf life. [1] HPLC analysis is widely used to determine quantities of Sodium benzoate. Sample preparation of cosmetic samples for analysis is extensively done manually using ultrasonication. Sample preparation methods using sonication often lack from reproducibility due to the introduction of uneven energy distribution and heat generation. It is also a manual and time-consuming process. [2]

Experimental

Chemicals and reagents

- Skin cream for face, body and hands
- Working standard Sodium benzoate (purity 100.0%)
- Water, HPLC grade
- Methanol, HPLC grade
- Orthophosphoric acid 85%

Instrumentation

accroma® samplePrep system

- Analytical balance Mettler-Toledo WMS 404C-L/11
- Shaker module
- Liquid- & Filtration module
- Agilent Online analysis interface
- accroLab software

Agilent 1260 Infinity II HPLC (400 bar)

- 1260 Infinity II Quat Pump VL (G7111A)
- 1260 Infinity II Vialsampler (G7129A) with Integrated Column Compartment and External Tray
- 1260 Infinity II Variable Wavelength Detector (G1314F)
- Chromatographic Data System (CDS): Empower 3

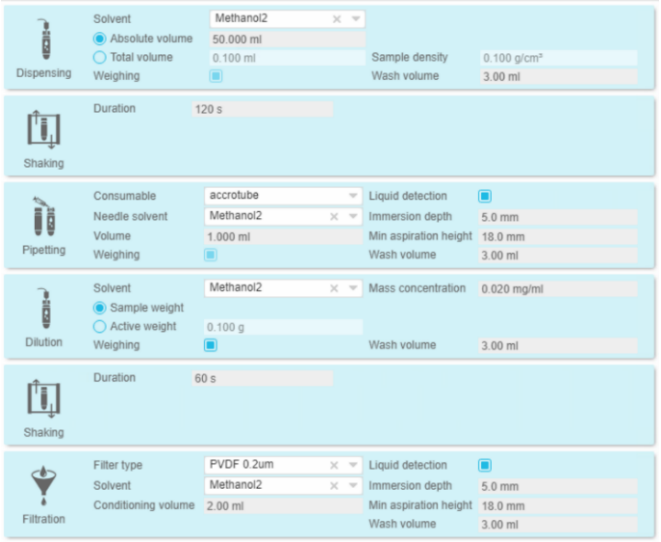
Procedure

Chromatography

Parameter	Value
Mobile Phase A	Methanol
Mobile Phase B	Water with 0.1% Phorphoric acid
Flow Rate	1.5 ml/min
Injection Volume	3 µl
Column Temperature	40◦C
UV Wavelength	271 nm
Gradient	**Time** **Min** **%A** **%B** 0.00 42.5 57.5 2.50 42.5 57.5
Column	ACE UltraCore Super C18, (100 x 4.6) mm, 2.6µm

Standard preparation

0.0516g of working standard (Purity: 100.0%) was accurately weighed and transferred into an accroTube. The working standard has then been prepared with the following workflow on the accroma sample prep system:



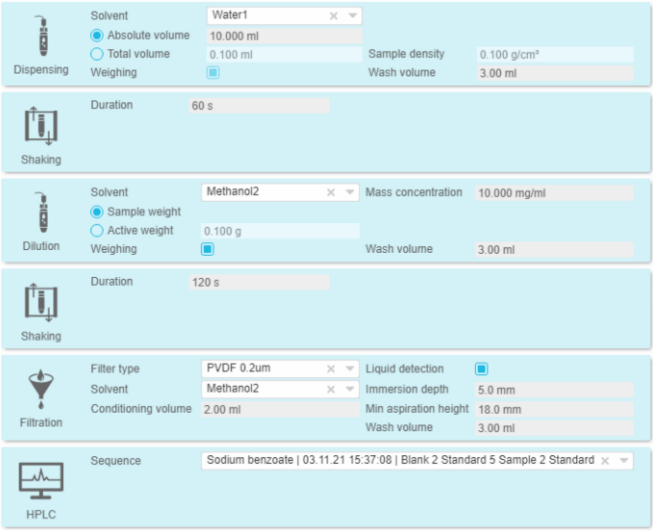
Standard preparation workflow on the accroma

Manual sample preparation method

Weigh (to the nearest 0.1mg) about 1g of sample into a 100ml volumetric flask. Add 10ml of water. Mix well using a vortex until the sample disperses completely and sonicate for about 10 minutes. Add 50ml of methanol. Mix well and sonicate for about 5 minutes. Cool to room temperature, dilute to volume with methanol and mix well. Filter the solution through a 0.20µm PVDF syringe filter.

Automated samplePrep method

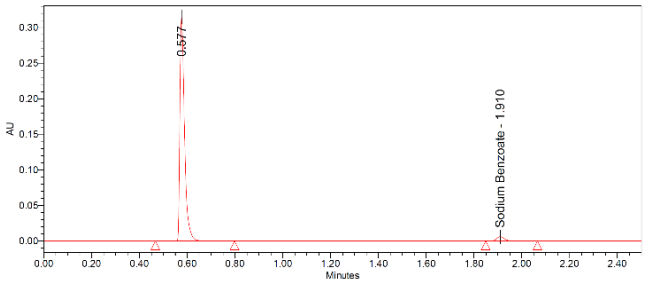
Compared to the extraction in the manual sample preparation, the extraction was performed by vertical shaking of accroTubes. About 1g of sample was weighed into an accroTube and a steel ball (16mm) was added. The closed accroTube was placed into the accroma® system. When setting up the sample, the net weight of the sample has been entered and workflow has been started.



Sample preparation workflow on the accroma

Results and discussion

The acquired chromatograms of the sample solutions showed peaks between 1.8 and 2.0 minutes. No non-expected peaks were identified comparing the working standard and the samples. The peak around 0.6 minutes is normal for the formulation of this sample. Therefore, it was assumed that no degradation was taking place during the dispersing and extraction process.



Chromatogram of sample solution prepared by the accroma®

A full comparison of the measured concentrations of the automated sample preparations are shown in the following table:

Experiment	Concentration	Target
Working standard	21.1 µg/ml	-
Sample 1	0.20062 %	0.184 % to 0.216 %
Sample 2	0.20196 %	
Sample 3	0.20502 %	
Sample 4	0.20149 %	
Sample 5	0.20183 %	
RSD	0.82 %	-

The measured concentrations are all within the expected range of 0.184% to 0.216%. In this sample preparation, the dilution method was used to achieve the target sample concentration as accurately as possible despite varying provenance. Thus, a relative standard deviation of 0.82% was achieved for the measured content.

Conclusion

Extraction times by the accroma® samplePrep system were shown to be significantly shorter than by the manual method of ultrasonication / vortex.

Parameter	Manual	accroma	Improvement
Recovery	100%	100%	-
Extraction time	15 min	3 min	80% reduction
Hands-on time	20 min	5 min	75% reduction

The accroma® system proved to be suitable for the automated sample preparation of semi solids such as skin creams. It provides standardization and can reduce variability. This was shown by very good reproducibility (relative standard deviation of 0.82%). The extraction time could be reduced by 80%. The overall sample preparation hands-on time could be reduced by 75% as the accroma® system can automate sample weighing, pipetting, extraction, filtration and transferring the vial into the HPLC. The system crosschecks every added volume gravimetrically which increases traceability and accuracy. It documents every possible parameter and step of the process. In conclusion, lab efficiency and compliance are significantly improved by using the accroma® samplePrep system.

References

[1] E. MacLeman., Why Preservatives Aren't so Bad: Sodium Benzoate, Article 10.09.20, **2020**, The Derm Review, https://thedermreview.com/sodium-benzoate/

[2] K. Zhang, J.W. Wong, Solvent-Based Extraction Techniques for the Determination of Pesticides in Food, Comprehensive Sampling and Sample Preparation, Volume 4, **2011**, Pages 245-261.

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