

Automated Analysis of MCPD and Glycidol in food using GC/MS

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Mit uns stimmt die Chemie ...

"What is efficient cargo handling?"



- The laboratory needs to analyse the oil first
- And release the product as "good" or "MCPD free"
- Every waiting hour costs \$\$

Every hour that a ship is parked at the harbour it costs ~25.000 - 100.000 € depending on its size







"What is efficient cargo handling?"



- There are two options available
 - Sent samples out to be analysed
 - Have your own lab analyse the samples
- In both cases, depending on the method that is used, a result can take up to 24h or even more
- After a few hours of waiting in the harbour, a new MCPD-Analyzer can be bought





All official AOCS methods automated



Trivial name	Components	Automation using PAL3 sampler	Official Method Name	Comment
DGF-Method	3 MCPD,GE (as difference), 2MCPD	100 % 160 cm DHR PAL RSI/RTC	AOCS Cd 29c-13 DGF C-VI 18 (10)	 Optional TQ for higher sample throughput ~ 36 samples/day
Kuhlmann "3in1" SGS "3in1"	3MCPD,GE, 2MCPD	90 % 160 cm DHR PAL RSI/RTC with manual step	AOCS Cd 29b-13	 Manual step involves placing the tray in the fridge @ -22 °C for 16 h.
Unilever-Method	3MCPD,GE , 2MCPD	100 % 160 cm DHR PAL RSI/RTC with CooledStack & Centrifuge	AOCS Cd 29a-13	 16 h @ 40 °C May require ultrasonic bath
Zwagerman- Overman-Method	3 MCPD, GE, 2MCPD	100 % 160 cm DHR PAL RSI/RTC	-	 Requires ¹³C-Marked standard A Triple-Quadrupole is required for correct GE quantification

- Other methods require ASE for sample preparation
- Optimization of AOCS Cd 29c-13 in cooperation with the Institut Kirchhoff Berlin
- For higher sample throughput and longevity



DGF Fast & Clean workflow





Sample preparation following the official AOCS/DGF method.

Workbench configuration



Dual Head system with Bruker GC/MS



Full solution with Bruker GC/MS due to its robust MS-Source

Other GC/MS-Analyzer are possible



Advantage of TQ-MS



Single Quadrupole

Triple Quadrupole

S/N 7608



Axel Semrau[®]

High sensitivity



3-MCPD at 0.05 mg/kg



Investigating Carryover



No Carryover visible after highly contaminated samples

High sensitivity?



3-MCPD; (+) 196.0 > 147.0; Ohne Internen Standard.xms; Filtered (Cps 3-MCPD; 147.0 (TIC); Blindöl_A.xms; Filtered The internal Standard 12.5 contains ~ 0.01 % 3-**MCPD** => Reducing the 10.0amount of internal Standard will increase sensitivity 7.5 1 2 5.0 2.5-0.0 7.75 minutes 7.00 7.25 7.50 6 50 6.75

> 90 % of the background are from the internal Standard

Analyzer longevity





Clean Technology physical and chemical







Very good reproducibility at different concentration levels

3-MCPD at 1 mg/kg





Linearity



Calibration: 0,05 - 0,5 mg/kg

Calibration: 0,5 - 5 mg/kg



 Quantification can also be done using only the internal standard, as written in the official AOCS norm



Repeatability and Recovery of *DGF F&C* for 3-MCPD and 2-MCPD on consecutive days



Part A

Part B	
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	Recovery %	Reproduci bility %
3-MCPD part A	91.6	7.7
3-MCPD part B	101.9	8.8
2-MCPD part B	116.2	8.9

Results with DGF F&C



Real samples vs. round robin results

FAPAS: Manually prepared samples from a round robin test

Processed according to AOCS Cd 29a-13, AOCS Cd 29b-13 or AOCS Cd 29c-13



Validation with FAPAS Reference Oil

*Using a transformation factor of 1.



Comparison of manual sample handling according to AOCS Cd 29b-13 with the automated *DGF F&C*



*Using a transformation factor of 1.

DGF F&C for different matrices



Comparison of manual sample handling according to AOCS Cd 29c-13 with the automated sample preparation *DGF fast* & *clean* in different matrices



Efficient Overlapping with CHRONOS





AOCS Cd 29a-13

Efficient Overlapping with CHRONOS



DGF F&C



Advanced Automation Features

No Calibration needed for quantification

Automated addition of spiked samples at the LOQ to monitor recovery and LOQ/LODs

With every sequence, the transformation factor for glycidol can be checked

Control samples can be analysed as a control chart with every sequence

Routinely monitoring of blank concentration

Accuracy, precision, productivity





Summary MCPDs



Accuracy and precision are comparable to manual results.

36 samples can be processed in 24 hours (Part A & B).

The automated sample preparation delivers "clean" samples to ensure detector longevity.

Offline or online approach possible.

Coupling to a GC Triple Quadrupole allows low limits of detection.

The modular PAL system platform allows to adapt the system to many methods, e.g. Unilever, SGS 3in 1 or Zwagerman.

Accuracy, precision, productivity



Contact for further information







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Thank you for your attention

Special thanks to



Questions?