

# 3-MCPD in Vegetable Oils

*...and 2-MCPD and Glycidol*



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# Edible Fats and Oils

Are subject to processing **But:**

- ▷ Improve:
  - Quality
  - Stability
  - Safety
- ▷ Through removing a very large portion of impurities

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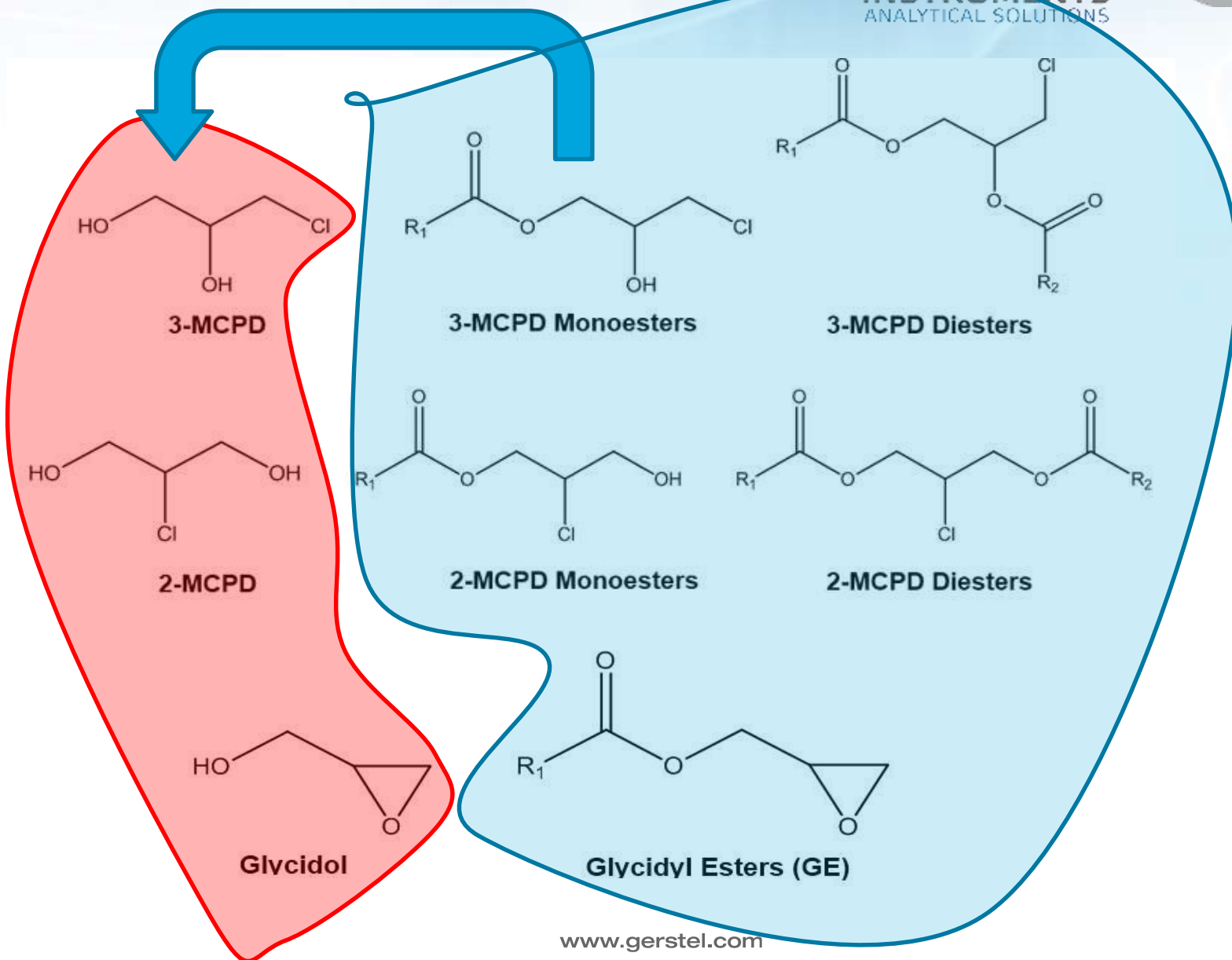
▷ Through removing a very large portion of impurities

▷ Formation of process contaminants

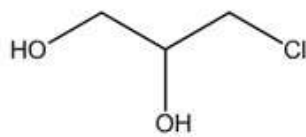
▷ Can result in a significant health risk



# What's the «real» problem?

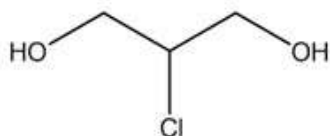


# 3-MCPD, 2-MCPD, Glycidol and their esters



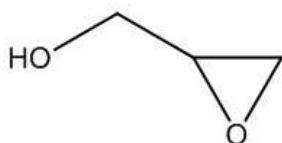
**3-MCPD**

International Agency for Cancer Research (IACR)  
-> Group 2B: possibly carcinogen to humans



**2-MCPD**

Only limited data yet, but some (partially unpublished)  
data suggest high toxicity level



**Glycidol**

International Agency for Cancer Research (IACR)  
-> Group 2A: probably carcinogen to humans  
Tolerable daily intake: as low as reasonably achievable

# Validated Indirect Methods

*for 3-MCPD-, 2-MCPD- and Glycidol Esters*

## Direct

### ▷ Using LC/MS

- High amount of different

EU-Commission Recommendation 2014/661/EU

"...it is recommended to use the American Oil Chemists' Society standard methods"

- Poor standard availability

## Indirect

### ▷ Cleavage of all esters, derivatization. GC/MS

- Favorable method since the human body also cleaves the esters

# Validated Indirect Methods for 3-MCPD-, 2-MCPD- and Glycidol Esters

## ▷ AOCS Cd 29a-13

- *slow acid esters cleavage, liquid-liquid extraction, derivatization with*

Slow = derivatization at +40°C for 16 hours

*assay.*

## ▷ AOCS Cd 29b-13

- *slow alkaline esters cleavage, liquid-liquid extraction, derivatization with*

Slow = derivatization at -25°C for at least 16 hours

*assays.*

## ▷ AOCS Cd 29c-13 / ISO 18363-1

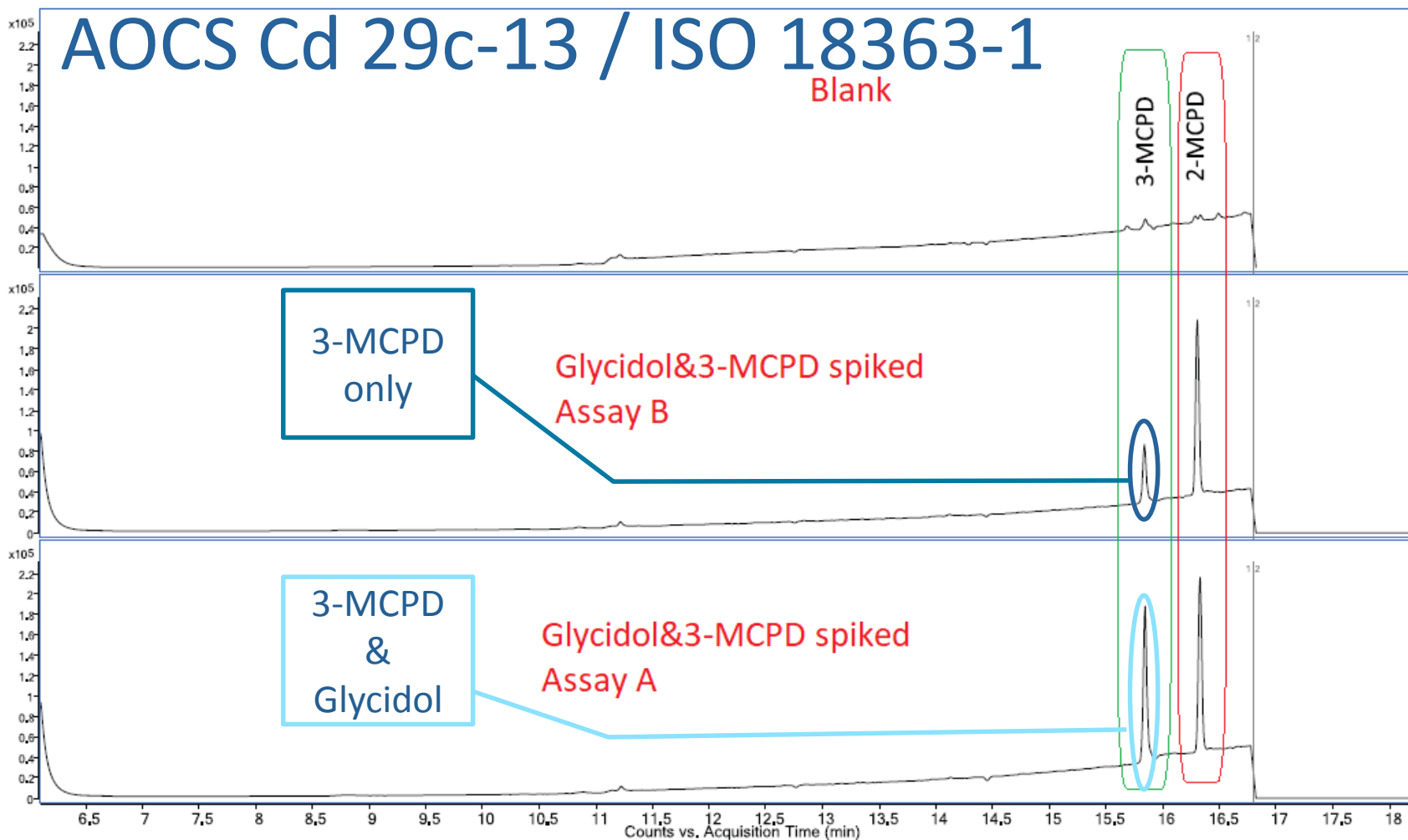
- *fast alkaline esters cleavage, liquid-liquid extraction, derivatization with phenylboronic acid. Measures 2-MCPD and 3-MCPD in two assays. Glycidol calculated from difference between both assays.*



# Validated Indirect Methods for 3-MCPD-, 2-MCPD- and Glycidol Esters

## AOCS Cd 29c-13 / ISO 18363-1

Blank



# AOCS Cd 29c-13

## workflow

- ▷ Weigh sample in Vial
- ▷ Add MTBE
- ▷ Add internal standard solution
- ▷ Add MeOH/NaOH solution
- ▷ Shake and incubate for 10 min at room temperature
- ▷ Quenching (acidic NaCl solution for assay A, acidic NaBr “chlorine-free” solution for assay B)
- ▷ Add n-Hexane
- ▷ Shake and incubate for 10 mins at room temperature
- ▷ Discard hexane phase
- ▷ Add n-Hexane 2<sup>nd</sup> time
- ▷ Shake and incubate 2<sup>nd</sup> time for 10 mins at room temperature
- ▷ Discard hexane phase
- ▷ Extract analytes with more polar solvent (e.g. MTBE/Ethyl acetate)
- ▷ Transfer into vial pre-filled with NaSO<sub>4</sub>
- ▷ Extract analytes 2<sup>nd</sup> time with more polar solvent (e.g. MTBE/Ethyl acetate)
- ▷ Transfer into vial pre-filled with NaSO<sub>4</sub>
- ▷ Extract analytes 3<sup>rd</sup> time with more polar solvent (e.g. MTBE/Ethyl acetate)
- ▷ Transfer into vial pre-filled with NaSO<sub>4</sub>
- ▷ Add phenyl boronic acid (derivatization agent)
- ▷ Derivatize and vaporize to dryness at 40°C
- ▷ Redissolve residue with isooctane
- ▷ Inject isooctane solution

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# 3-MCPD Workstation

srainstruments.com



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# GERSTEL *quick*MIX

- ▷ Highly efficient mixing
  - Unique horizontal and vertical movements of the *quick*MIX ensure highly efficient mixing independent of sample volume
  - Emulgation of oil/water phases in seconds



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# GERSTEL *m*VAP

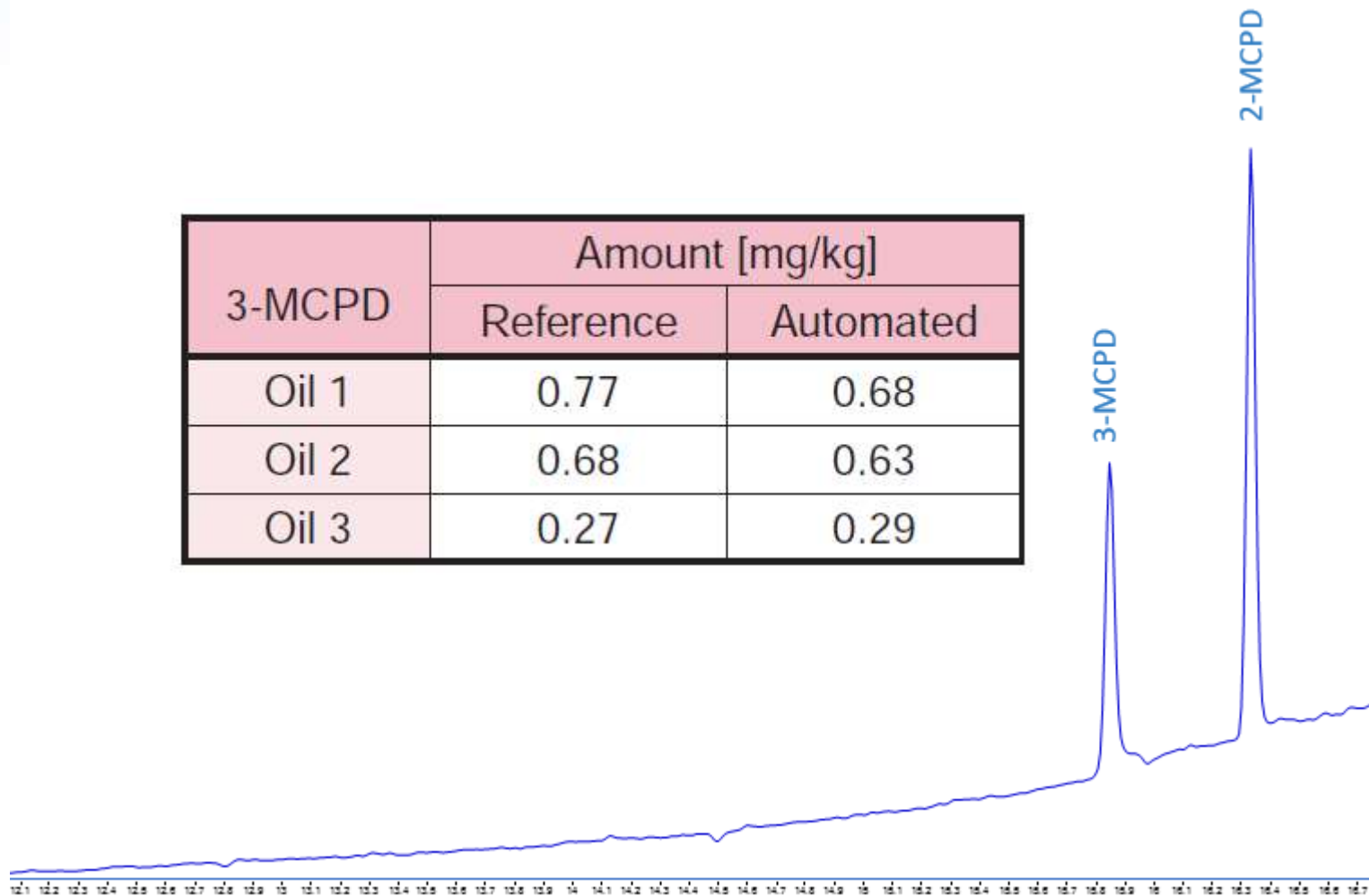
Evaporative concentration  
option for the MPS





# Accuracy

3-MCPD	Amount [mg/kg]	
	Reference	Automated
Oil 1	0.77	0.68
Oil 2	0.68	0.63
Oil 3	0.27	0.29

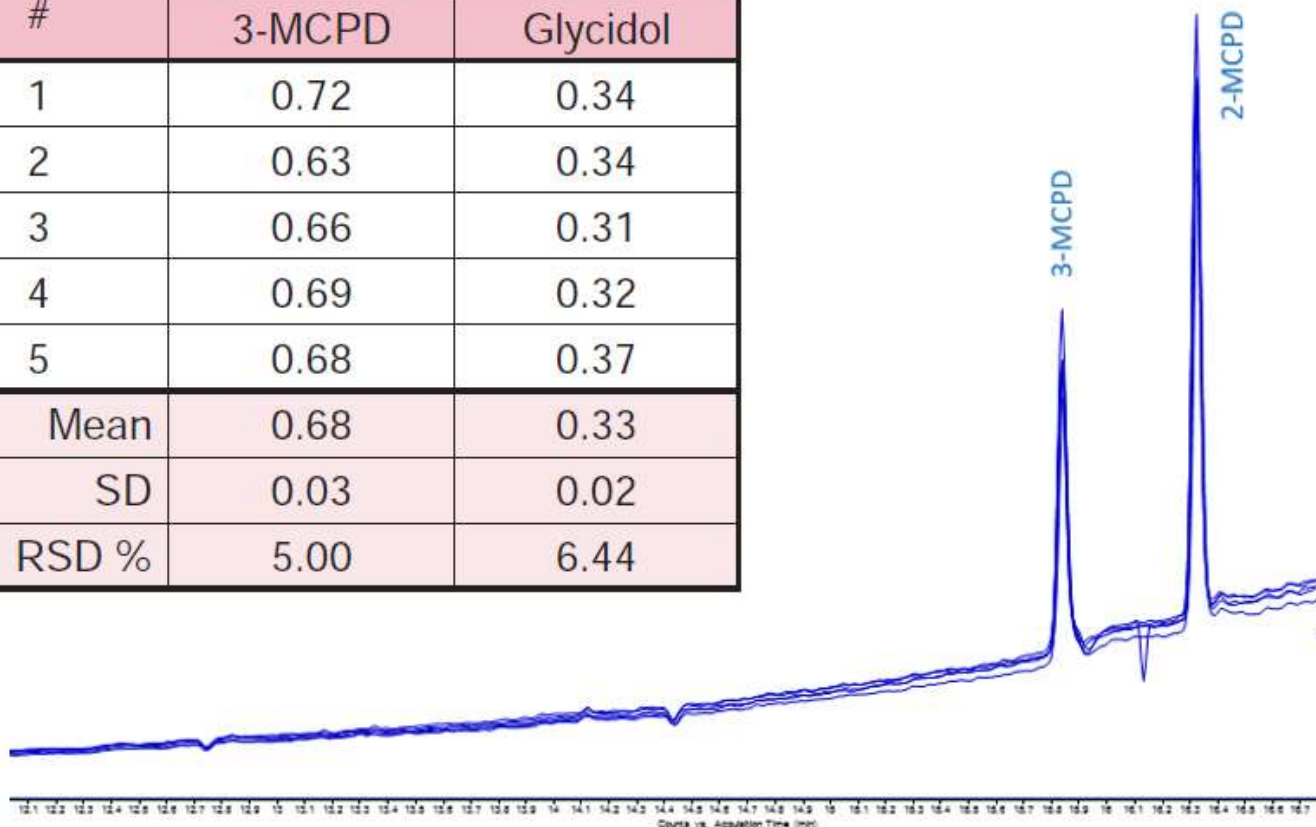


GERSTEL Application Note No. **191**, 2017

# Repeatability

(*n=5 samples*).

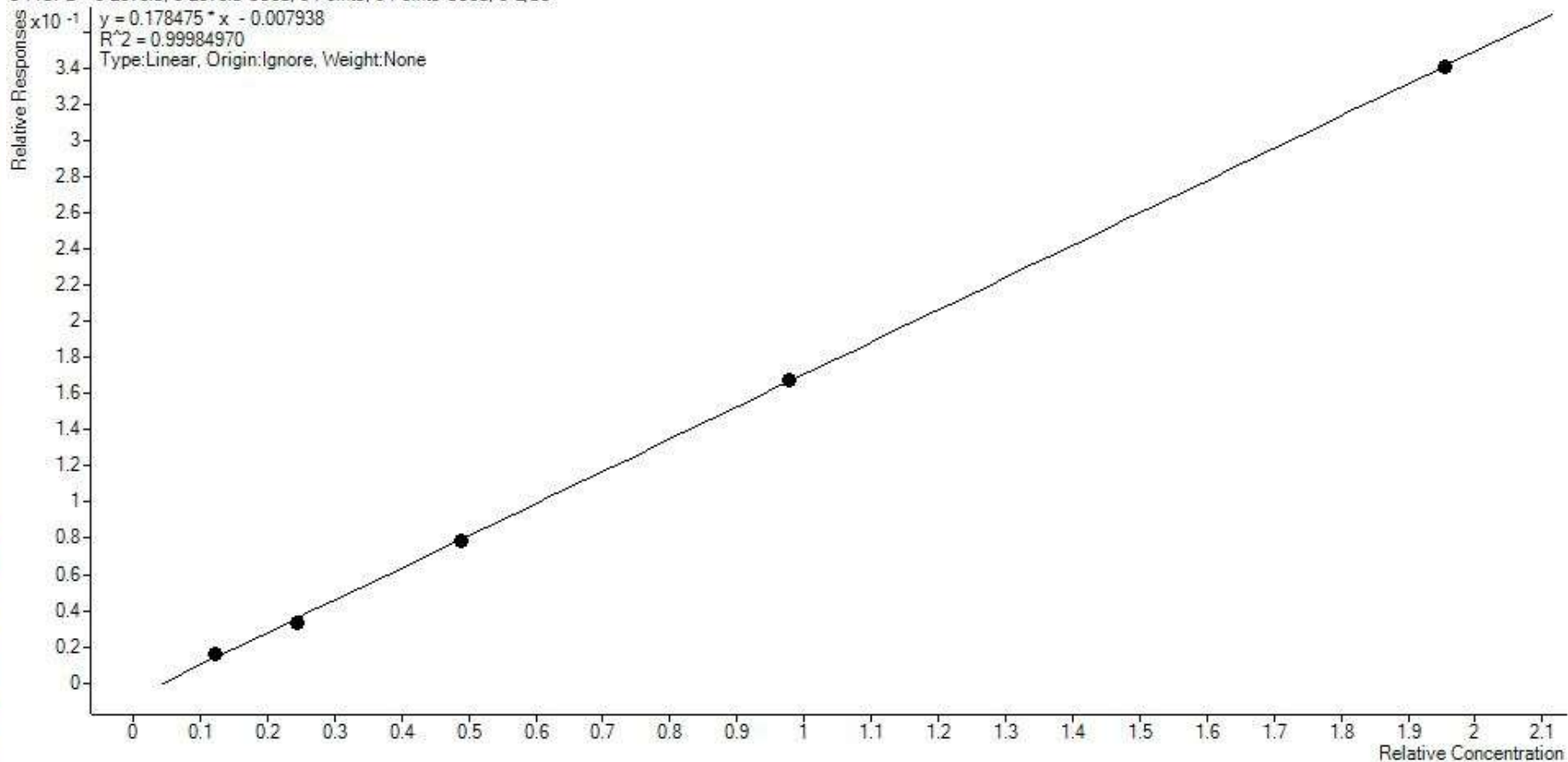
#	Amount [mg/kg]	
	3-MCPD	Glycidol
1	0.72	0.34
2	0.63	0.34
3	0.66	0.31
4	0.69	0.32
5	0.68	0.37
Mean	0.68	0.33
SD	0.03	0.02
RSD %	5.00	6.44



# Linearity 3-MCPD (0.1 – 2.0 mg/kg)

3-MCPD - 5 Levels, 5 Levels Used, 5 Points, 5 Points Used, 0 QCs

Relative Responses  
 $y = 0.178475 \cdot x - 0.007938$   
 $R^2 = 0.99984970$   
Type: Linear, Origin: Ignore, Weight: None



# Linearity Glycidol (0.1 – 2.0 mg/kg)

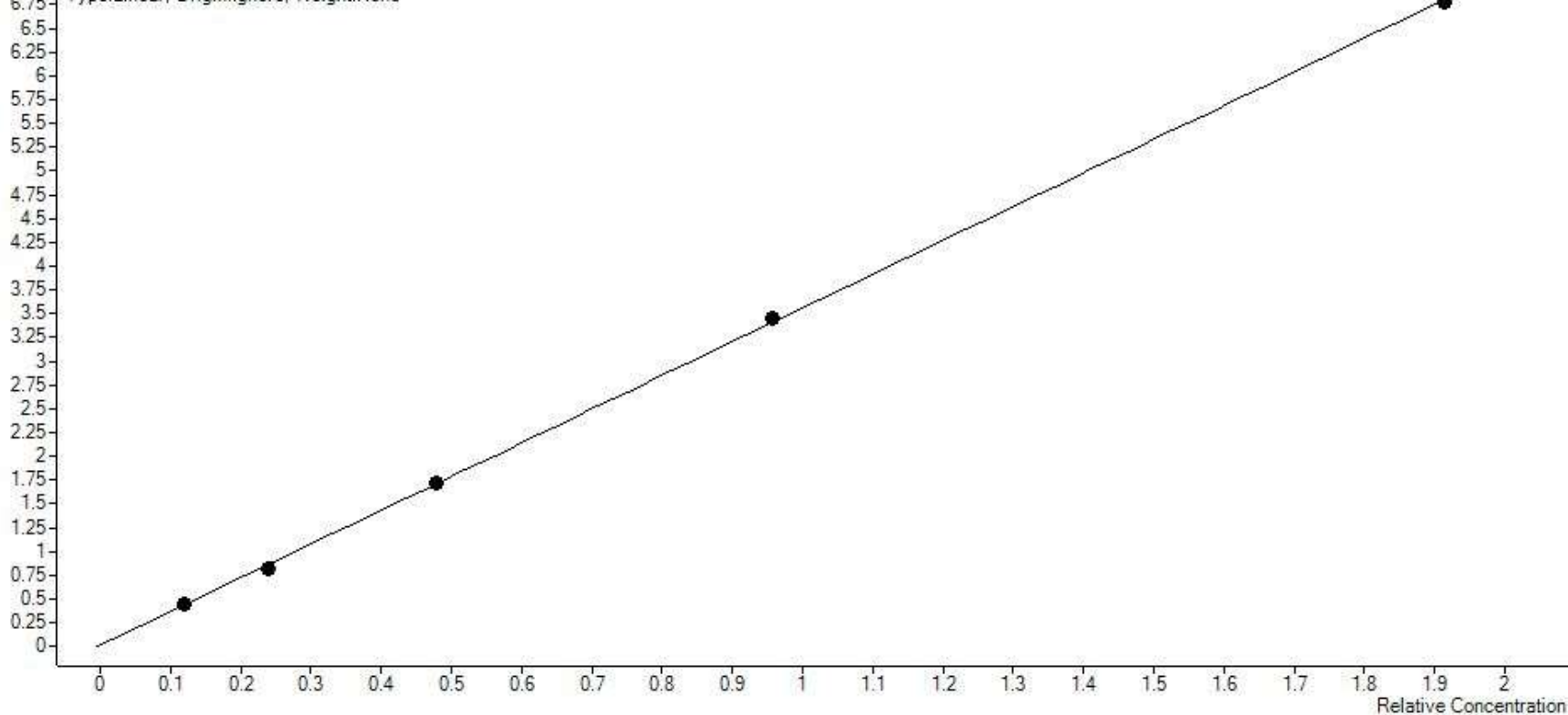
3-MCPD - 5 Levels, 5 Levels Used, 5 Points, 5 Points Used, 0 QCs

Relative Responses  
 $\times 10^{-1}$

$$y = 0.354540 \cdot x + 0.001574$$

$$R^2 = 0.99981942$$

Type: Linear, Origin: Ignore, Weight: None



GERSTEL Application Note No. **191**, 2017

# Final Remarks

## Gerstel 3&2-MCDP & Glycidol solution

### Allows to:

- ▷ Automate the whole sample preparation

*The only manual step required is the injection*

- ▷ Use a single quantitative vial

*Thanks to the integrated vial filling by the <sup>m</sup>VAP*

- ▷ Have a sample preparation station

*Lot of sample preparations eventually needed*

- ▷ Reduce the occurrence of random errors

*No manual intervention required*

**More than 20 completely automated analysis per day with high reliable results**

# Aknowledgments

Fully Automated Determination of 3-MCPD and Glycidol in Edible Oils by GC/MS Based on the Commonly Used Methods ISO 18363-1, AOCS Cd 29c-13, and DGF C-VI 18 (10)

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