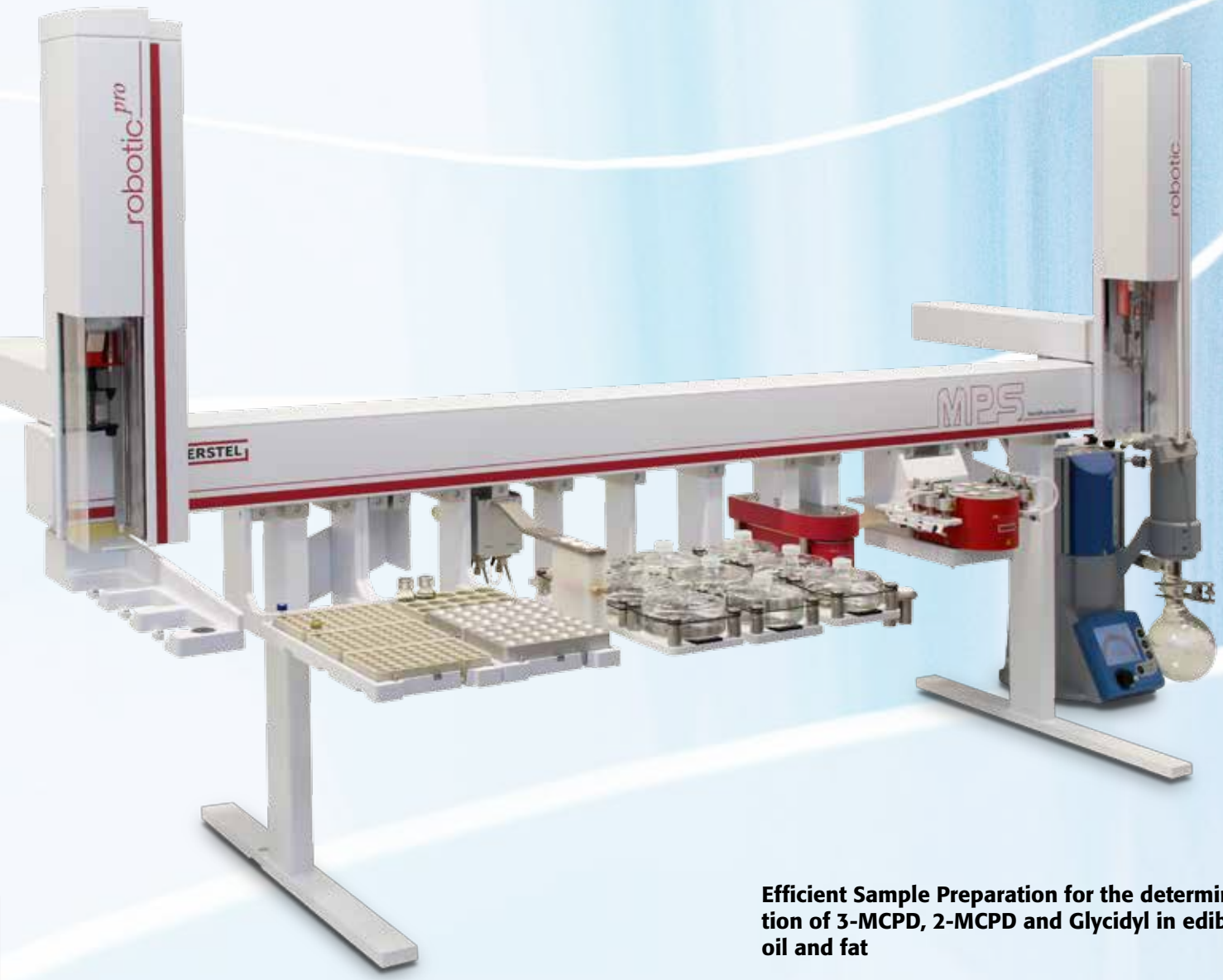
GERSTEL

Sample Prep Solution
3-MCPD



Efficient Sample Preparation for the determination of 3-MCPD, 2-MCPD and Glycidyl in edible oil and fat

Automated system for ISO 18363-1, AOCS Cd 29c-13, and DGF C-VI 18 (10)

High stability and lowest limits of determination achieved using automated evaporation step

Sample Prep Solution

Sample Prep Solution 3-MCPD

During edible oil refining processes, 2-MCPD, 3-MCPD and glycidyl fatty acid esters can be generated, resulting in a contaminated product. The GERSTEL 3-MCPD Sample Prep Solution enables automated determination of these potentially health relevant contaminants supporting the methods ISO 18363-1, AOCS Cd 29c-13, and DGF C-VI 18 (10).

In fats and oils, glycerol is contained in the form of fatty acid esters (triglycerides). Since many oils are not suitable for consumption and are not stable in storage in their native, untreated form, they are refined to remove unwanted substances. The refining process involves a deodorization step, in which the oil is heated with hot steam to between 200 and 230 °C under vacuum to remove unwanted odor and flavor active substances along with other unwanted substances. When chloride is present, however, the heat treatment can result in the substitution of a fatty acid chain by a chloride atom forming 2-MCPD- and 3-MCPD fatty acid esters, respectively. Under these conditions, glycidyl fatty acid esters can also be formed. These contaminants are classified as potential health risks.

For the determination of 3-MCPD, the fatty acid esters, as well as glycidyl fatty acid esters, the German Society for Fat Sciences (DGF) recommends the unified DGF C-VI 18 (10) method, based on a complex sequence of sample preparation steps combined with GC/MS determination. The DFG C-VI 18(10) method is similar to the ISO 18363-1 and AOCS Cd 29c-13 methods, which are practically identical. The 3-MCPD Sample Prep Solution developed by GERSTEL automates the reliable indirect DGF method one to one using reduced volumes. If required, 2-MCPD can be determined as well.

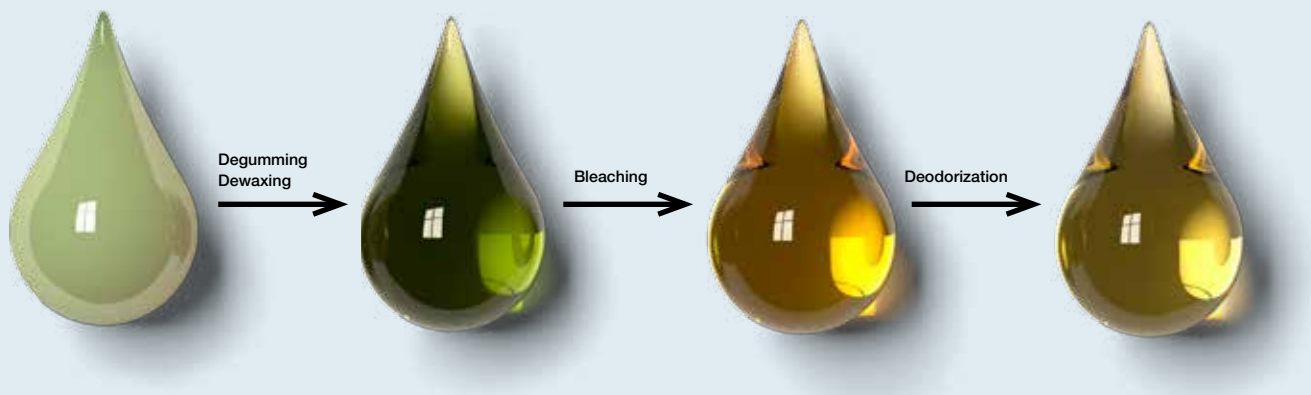
The GERSTEL MultiPurpose Sampler (MPS robotic) Dual Head version, automates all steps including liquid handling, liquid-liquid extraction, evaporative concentration of extracts, change to a GC compatible solvent and derivatization of the analytes. If the MPS is integrated with a GC/MS system, the entire process including GC/MS analysis is automated and automatically optimized for highest productivity and throughput.

The evaporation step ensures that the required limits of determination can be reached using a single quadropole mass spectrometer (MSD) for most matrices. In addition, excess derivatization reagent is removed for improved GC/MS system stability.
The PrepAhead function ensures maximum productivity and parallel processing of individual tasks. When performing differential determination of 3-MCPD and glycidol, 24 samples can be processed in 24 hours, based on 48 GC/MS analysis runs.

The GERSTEL 3-MCPD Sample Prep Solution supports the following standard methods: **ISO 18363-1, AOCS Cd 29c-13, and DGF C-VI 18 (10).**

Sample Prep Solution

To prepare edible oils for consumption, a refining process is frequently required. During this process, 3-MCPD-, 2-MCPD-, and glycidyl fatty acid esters can be formed. A reduction in their levels is typically achieved by optimizing the refining process conditions.



Manual

- Weigh a 100 mg sample into a vial
- Fill a second vial with sodium sulfate drying agent (drying vial) - optional

Automated by the MPS:

- Add MTBE to the sample
- Add ISTD solution and mix, or melt and mix (solids)
- Add MeOH/NaOH mixture
- Agitate and incubate
- Add acidic NaCl solution (Assay A)
- Add acidic NaBr solution (Assay B)
- Add n-hexane for matrix extraction
- Agitate and incubate
- Discard hexane phase
- Repeat extraction with n-hexane twice
- Perform multiple analyte extractions using MTBE/Ethylacetate 3:2 (v/v), transfer the organic phases to the drying vial
- Add phenylboronic acid solution
- Evaporate to dryness and derivatize in the mVAP at 50 °C and subambient pressure
- Take up the derivatives in isooctane
- Introduction to GC/MS(/MS) if integrated with sampler.

One manual step is required followed by the long list of steps shown to the left prescribed in the unified DGF C-VI 18 (10) method. These are all performed automatically by the GERSTEL MultiPurpose Sampler (MPS). Depending on the instrument configuration, introduction of the prepared extract to the GC/MS system can be included.

MAESTRO Scheduler: Instant overview of total process timing for a batch of samples including each process step and GC/MS analysis for easy laboratory workflow planning. In 24 hours, 24 samples can be analyzed using the differential method, equal to 48 GC/MS analysis runs.

GERSTEL quickMIX

quickMix enables extremely fast and efficient mixing and extraction of samples as part of an automated sample preparation process. The mixing power is comparable to that of a vortex mixer making quickMIX highly suitable for extraction of oil samples. In quickMIX, samples are placed in special trays that hold up to 6 samples, depending on the vial size, for simultaneous, batchwise agitation.

GERSTEL mVAP

GERSTEL mVAP performs evaporative concentration of up to 6 samples in parallel. Vacuum level, temperature and agitation speed are user defined and can be optimized for the analytes in question. mVAP makes it possible to reach lower limits of determination. When analyzing for 3-MCPD and associated compounds in edible oils and fats, separation of excess derivatization reagent also helps to keep the GC/MS system stable, resulting in improved long term stability and accuracy. Depending on the type of oil analyzed, a concentration step enables single quadrupole mass spectrometers to reach the required limits of detection.

Application details...

GERSTEL AppNote 191 [1] offers a detailed overview of the performance of the GERSTEL 3-MCPD Sample Prep Solution. Among other things, it is demonstrated that results achieved using the automated sample preparation procedure correlate well with results from established manual sample preparation procedures. The usefulness for different types of oil is discussed, and linearity and repeatability shown.

All results demonstrated the suitability of the automated Sample Prep Solution for routine analysis.

[1] Lucas, D.; Hoffmann, A.; Gil, C. 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) GERSTEL AppNote No. **191**, *2017*