

FEDIOL recommendations on the methodology to be used for the analysis of 3-MCPD esters and glycidol esters in oils and fats

A number of studies are currently ongoing that aim at developing analytical methods for the determination of 3-MCPD esters and glycidol esters. This issue is being addressed by industry, authorities and academia.

The oils and fats sector is highly concerned about this area of research, primarily because the reliability of the analytical method is key when defining strategies to mitigate the presence of 3-MCPD-esters and/or glycidol esters during refining. FEDIOL is, therefore, highly committed to address this problem. In that respect, the FEDIOL analytical experts have come together to share their expertise and agree on a common approach.

As a result, FEDIOL prepared recommendations on the criteria considered critical to obtain workable and reliable analytical methods for the determination of 3-MCPD esters and glycidol esters.

FEDIOL experts recommend in first instance to have separate analytical methods for 3-MCPD esters and glycidol esters. Both analytical methods should be specific to the compounds to be analysed.

1. ANALYSIS OF 3-MCPD ESTERS IN OILS AND FATS

- **Recommended method: Indirect method**

At present two approaches exist to analyse 3-MCPD esters: a direct and an indirect methods.

For the direct method an analytical standard for each of the esters present is needed and the sum of all esters is then measured. Considering the diversity of fatty acids and possibility to esterify any of the two hydroxyl groups of 3-MCPD, a high number of different molecules are expected to be formed. Consequently, FEDIOL believes that the direct method is not widely applicable and recommends the use of an indirect method.

In the indirect method all 3-MCPD esters are converted into one compound i.e. free 3-MCPD. Indirect methods comprise several basic steps i.e. use of an internal standard, hydrolysis/methanolysis, neutralisation & salting out, derivatisation and GC/MS analysis.

For each of these steps several options can be envisaged leading to significant variations in the analytical results. FEDIOL experts assessed the critical parameters and made the following recommendations:

- **Recommended parameters**

Parameters	FEDIOL recommendations	Comments
Internal Standard	Deuterated esterified internal standard (e.g. 3-MCPD dipalmitate) rather than free 3-MCPD	As the vast majority of 3-MCPD exist in the bound form (esters) in oils/fats it makes sense to use a 3-MCPD ester as an I.S., as it undergoes similar degree of hydrolysis (see point below)
Hydrolysis method	Acidic or Alkali hydrolyses	The main difference between the 2 methods is the time of hydrolysis, <u>fast</u> for alkali and <u>slow</u> for acidic hydrolysis. It should also be noted that the time of alkali hydrolysis can have a slight impact on the results. A too fast hydrolysis can result in over estimation, whereas a too slow hydrolysis can lead to 3-MCPD esters decomposition (50% degraded within 10 mins – the internal standard will decompose at the same speed). The prerequisite for the use of alkali hydrolysis is to have a back-flash system in your GC, as the GC system becomes rapidly dirty.
Salting out agent	Na₂SO₄ or (NH₄)₂SO₄	Even though NaCl is not a problem in case of acidic hydrolysis there is probably no benefit in using it. Sulphate is therefore preferred.
Derivatisation agent	PBA or HFBI	There is probably no significant difference in the benefit between the two agents. PBA is more commonly used.

2. ANALYSIS OF GLYCIDOL ESTERS IN OILS AND FATS

Recommended method: Direct method

Based on the fatty acid profile of the oil/fat, only five or six different glycidol esters are usually present in the product, which makes the application of the direct method more feasible.

In addition, glycidyl stearate is commercially available for quantification. Among the currently existing methods LC/MS serves as a good method for the direct determination of glycidol esters.