A Fully Automated Method For MCPD – and GE- Esters And The Importance of Glass Quality Of the used Autosampler Vials

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ABSTRACT AND INTRODUCTION

Monochloropropanediol (MCPD) and glycidyl esters (GE) are the central point of attention of the food industry. Compounds are unwanted by-products of oil refining, and predominately occur in palm oil, and great care should be taken in processing the oils. Nonetheless, any oil can contain MCPD and GE when it is not processed with the utmost care. In the oil market, MCPD and GE occur as esters, but they are analysed after transesterification and derivatization in order to report total MCPD and GE contents. In this poster an automated method will be presented based on a method from AOCS Cd29-13 or the so-called Zwagerman method showing excellent recoveries. One of the critical steps in the automation is the choice of autosampler vials. Vials with lower glass quality will have an effect on recovery as the esters tend to adsorb to the free silanol groups on the glass walls.

The importance of glass quality Today’s vials in Europe are normally made of 1st hydrolytic class glass, according to all Pharmacopeias. But there are differences in the glass qualities meeting the requirements and provide a significant better low adsorption every Pharmacopeia. Especially for MCPD when they are put on the automation system before they are derivatized, the glass is a key factor for success. In this is the most critical step, since at this point in the process, polarity of analytes play a central role. During this step it is key to make sure that they do not stick on the glass wall in order to reach the necessary detection limits.

The IMPORTANCE OF GLASS QUALITY

Figure 1. Chromatol GOLD grade inert vials

MATERIALS AND METHODS

For the automated sample preparation a Thermo Scientific™ TriPak™ RSH autosampler was used, with modified heated tray and programing by SampleQ. All samples were analysed using a Thermo Scientific™ TRACE™ 1310 gas chromatography linked with a connect quadrupole detector and selective detector able to perform method. Detection was performed with a Thermo Scientific™ 1522™ 5000 MS/MS detector.

In the automation method the complete sample preparation is done in addition, the calibration curve is made, the GC sample is being spiked and prepared and a blank oil is also prepared.

Data reviewing was performed by the Thermo Scientific™ ChromaQuest™ software and the complete control of blank, QC sample, calculations and all classic controls of GC-MS such as injection time, 1D recovery and ion ratios are being maintained automatically.

For separating the compounds a TriGold TG-SBMS GC Column, 30 m, ID 0.25 mm x 0.25 mm was used. Linked to a 2 m internal standard 5%Phen.

The GC and the MS method are described in the tables below.

The automated sample preparation process is described in the schematics.

Sample preparation for the analyst is limited to weighing in 100-150 mg of oil and the final injection volume is 2 μl.

RESULTS AND DISCUSSION

For determining the detection and quantification limits (LOD/LLOQ) extra virgin olive oil was prepared 10 times with the MCPD-ratio for determining the blank levels and standard deviations. The same was of course subsequently spiked with reference material 5 times at concentrations between 1 and 50 ppb. Each level was therefor prepared and analyzed 5 times and compared to the blank extra virgin olive oil.

For 3-MCPD the LOD was determined at 10 μg/kg and the LLOQ at 15 μg/kg, glycidol LOD was at 10 μg/kg and LLOQ 41 μg/kg. Finally the 3-MCPD LLOD was 2.7 μg/kg and LLOQ 6.8 μg/kg.

Figure 5. Chromatogram of all analytes at 250 ppb

Figure 6. Chromatogram of both transitions of 3 MCPD and the calibration curve from 1 to 50 ppb

CALCULATION

All data reviewing, calculations and reporting are performed using ChromaQuest software. Also the weight of the sample is included so the results are immediately reported as concentration in the samples, not in the extracts. Quality checks such as recovery, QC control, blank values, calibration-curves and ion ratios are included in the report section of Chromeleon for fast data reviewing without compromising the quality of the results.

CONCLUSIONS

The approach for MCPD and GE analysis enables

- Limiting the manual labor during the entire sample analysis
- Analyzing a full scope of the MCPDe and GE in one single injection
- Internal standard
- Short analysis time
- ME, MCPDe and 3-MCPDe are measured
- High productivity: 40 samples per day from prep-to-ready to 30 samples per 4hrs in offline mode
- Automated calibration curves and addition of internal standards and automated QC control
- Automated calculation, data evaluation and LRS5 export
- Complete sequencing, data reviewing and reporting in one single report

REFERENCES

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