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Direct Determination of Glycidyl Esters of Fatty Acids in Vegetable Oils by LC–MS

Michael R. Blumhorst · Padmesh Venkitasubramanian · Mark W. Collison

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Abstract An LC–MS method using a single quadrupole mass spectrometer was developed for direct analysis of glycidyl esters of fatty acids in vegetable oils. Without any sample clean-up, this method provided acceptable recovery of seven glycidyl esters, comparable results to a previously-published method utilizing two solid-phase extraction steps, and consistent detection parameters after greater than 200 injections without any cleaning operations performed. This method could readily be implemented as a screening assay for glycidyl esters in most oil laboratories.

Keywords Monochloropropanediol · 3-MCPD · MCPD · Glycidol · Glycidyl esters · LC–MS · Vegetable oil

Abbreviations

LC–MS	Liquid chromatography mass spectrometry
3-MCPD	3-Monochloro-1,2-propanediol
LC–TOFMS	Liquid chromatography time-of-flight mass spectrometry
LOD	Limit of detection
LOQ	Limit of quantitation
GC–MS	Gas chromatography mass spectrometry
HPLC	High performance liquid chromatography
SPE	Solid-phase extraction
RBWD	Refined, bleached, de-waxed, deodorized

M. R. Blumhorst · P. Venkitasubramanian · M. W. Collison (✉)
Research Division, Archer Daniels Midland Company, James R. Randall Research Center, 1001 N. Brush College Road, Decatur, IL 62521, USA
e-mail: Mark.Collison@adm.com

Introduction

The initial detection of glycidyl esters of fatty acids in vegetable oils was the result of research to investigate the origin of 3-monochloro-1,2-propanediol (3-MCPD) and 3-MCPD esters of fatty acids in these oils [1–4]. In their work directed at identifying the precursors to these compounds, Weißhaar and Perz [5] reported the existence of relatively high levels of glycidyl esters of fatty acids. In response to this finding, Masukawa et al. [6] performed a survey of commercial oils sold in Japan and reported that glycidyl esters were detected in every sample tested. Preliminary reports from these studies led the Federal Institute for Risk Assessment (BfR), a scientific agency of the Federal Republic of Germany, to state its opinion regarding the necessity for a method to quantify glycidyl esters in edible oils to provide reliable risk assessment [7].

Recent work by Haines et al. [8] casts doubt on the extent of 3-MCPD and 3-MCPD ester formation during oil processing. These researchers concluded that the full Weißhaar method [2] is not measuring 3-MCPD in vegetable oil, but that base methanolysis and subsequent introduction of sodium chloride is potentially converting a wide variety of compounds into 3-MCPD as an unintended by-product [8]. Using a direct LC-TOFMS method without sample derivatization or clean-up, they reported 3-MCPD monoesters were not detected in any deodorized oil samples, and the only commercial samples containing 3-MCPD diesters originated from palm oil. Glycidyl esters, however, were detected in a variety of vegetable oils with amounts correlated to the diglyceride content of the oil [8].

The presence of glycidyl esters of fatty acids in commercial oils is of concern; therefore, methods to provide accurate quantitation of these compounds are required. The direct LC-TOFMS method referenced above [8] has the

advantages of minimal sample preparation and high sensitivity (method LOD = 5 ng/g). However, time-of-flight mass spectrometers are not common instruments in most laboratories, and the sodium content of the mobile phase requires cleaning of the instrument on a daily basis and more extensive cleaning on a weekly basis. Masukawa et al. [6] developed and later refined [9] a method for quantifying glycidyl esters in edible oils using a single quadrupole mass spectrometer, an instrument that is more affordable and user-friendly, but sample preparation requires two solid-phase extraction and solvent evaporation steps which add significant costs for time and materials on a per sample basis.

Therefore, the objective of this research was to develop a fast, accurate and rugged screening method for glycidyl esters in edible oils using minimal sample preparation and a single quadrupole mass spectrometer.

Experimental Procedures

Reagents

HPLC grade methanol and acetone were purchased from Fisher Scientific (Pittsburgh, PA, USA). HPLC grade acetonitrile was from EMD Chemicals, Inc. (Gibbstown, NJ, USA).

Standards

Glycidyl stearate was purchased from TCI America (Portland, OR, USA). Glycidyl linolenate, glycidyl linoleate, glycidyl oleate and glycidyl palmitate were purchased from Wako Pure Chemical Industries (Tokyo, Japan).

Glycidyl esters of lauric acid, myristic acid and fully-deuterated (*d*31) palmitic acid were synthesized individually using a two-step chemical procedure in which the allyl ester was the initial product followed by conversion to the glycidyl ester. Deuterated palmitic acid was included in these experiments as an internal standard. A detailed description of the synthesis is provided elsewhere [8]. Briefly, allyl alcohol, toluene and Amberlyst 15 were refluxed with the respective fatty acid in an oil bath for 24 h. The reaction mixture was diluted with 20 mL hexane and filtered to remove Amberlyst 15. The reaction mixture was washed with water and saturated sodium chloride, and the organic layer was dried over anhydrous Na_2SO_4 and concentrated in vacuo to yield a colorless oil that solidified on cooling (allyl ester). This material was mixed with dichloromethane and cooled in an ice bath for 5–10 min before *meta*-chloroperbenzoic acid was added in small amounts. After the addition was complete, the reaction mixture was stirred and allowed to slowly warm to room temperature over a 24 h period. The progress of the reaction was monitored by TLC and ^{1}H NMR. After the reaction was complete, the reaction mixture was diluted with 20 mL hexane and washed with aqueous sodium bisulfite (2% w/w), aqueous sodium bicarbonate (10% w/w), water and aqueous saturated sodium chloride. The organic layer was dried over anhydrous Na_2SO_4 and concentrated in vacuo to yield a colorless oil. The oil was purified on a silica gel column (60–200 mesh, 100 mL bed volume) using gradient elution of ethyl acetate (0–20% v/v) in hexane. The fractions containing glycidyl esters were concentrated, and each product yielded a colorless oil that solidified on cooling. Final products were characterized using ^{1}H NMR and ^{13}C NMR, and purity was determined using GCMS.

Internal Standard Solution Preparation

Deuterated glycidyl palmitate was diluted with acetone to a target concentration of 200 ng/mL. This internal standard solution was used in the direct LC–MS method for sample and calibration standard preparation.

Calibration Standard Solution Preparation

Mixed standards containing each of the seven glycidyl esters were prepared by dilution with *d*31-glycidyl palmitate internal standard solution in acetone. Individual glycidyl ester concentrations ranged from 10 to 400 ng/mL and were corrected for purity.

Samples

Samples included refined, bleached, de-waxed, deodorized corn oil (RBWD Corn); refined, bleached, de-waxed, deodorized canola oil (RBWD Canola); refined, bleached, deodorized mid-oleic sunflower oil (RBD sunflower); refined, bleached soy oil (RB Soy); refined, bleached, deodorized palm oil (RBD Palm); and palm kernel oil.

Sample Preparation for Direct LC–MS Method

An aliquot of oil ($\sim$0.25 g) was accurately weighed into a glass centrifuge tube, and 5 mL of the internal standard solution in acetone were added. If necessary, samples were placed in a heating block set at 65 °C after addition of the internal standard solution to melt the oil and ensure sample homogeneity. Sample amounts were increased for reproducibility assays, but the sample:solvent ratio was maintained at 1:20 (1.25 g diluted with 25 mL internal standard solution). Sample extracts were assayed directly using LC–MS without further clean-up. Sample fortification, when performed, utilized the same final volume (5 mL), internal

standard concentration and solvent. Target concentrations for sample fortification were 1×, 2× and 4× the LOQ of the respective analyte.

Sample Preparation for Double Solid-Phase Extraction Method

The double SPE method of Masukawa et al. [6] was included for comparison purposes. Briefly, 0.1 g oil plus 4 mL acetonitrile containing *d*31-glycidyl palmitate internal standard were stirred for 10 min. Samples were centrifuged and transferred to previously-washed Sep-Pak Vac RC 18 cartridges. The sample solvent was eluted and discarded. Cartridges were washed with 2 × 2 mL acetonitrile, and these fractions were combined and evaporated to dryness using N_2. The dried residue was dissolved in 2 mL chloroform and transferred to chloroform-washed Sep-Pak Vac RC Silica cartridges. The sample solvent was eluted and collected. Silica cartridges were washed with 3 × 2 mL chloroform, and all chloroform fractions were combined and evaporated to dryness using N_2. Samples were dissolved in 2 mL methanol/isopropanol (1/1 v/v) and assayed using the LC–MS parameters described in the following section.

LC–MS Conditions

The LC–MS analysis was adapted from the procedure of Masukawa et al. [6]. A Shimadzu Series 20 gradient LC system and Shimadzu LCMS2020 single quadrupole mass spectrometer equipped with LabSolutions software were used for analysis of glycidyl esters. The autosampler was maintained at 40 °C to ensure sample solubility, and the injection volume was 5 µL. HPLC separation was performed using a YMC-Pack ODS-AM C18 column, 120 Å pore size, 150 × 3 mm, and 3 µm particle size. The column was maintained at 60 °C. HPLC mobile phase A was prepared by mixing 425 mL methanol, 425 mL acetonitrile and 150 mL water. HPLC mobile phase B was acetone. The mobile phase program was 2% B held for 9.5 min after injection, stepped to 6% B from 9.5 to 14.0 min, and stepped again to 15% B from 14.0 to 20.0 min. A 60% B wash for 5 min was used to elute low polarity compounds, and the column was returned to 2% B and equilibrated 9 min. HPLC flow rate was 0.6 mL/min.

Atmospheric pressure chemical ionization (APCI) was used in positive ion mode. The interface temperature was 450 °C; desolvation line temperature was 300 °C; heating block temperature was 300 °C; nebulizing gas flow was 2.5 L/min; and drying gas flow was 5 L/min. The interface voltage was 4.5 kV, and the Q-array RF voltage was 40 V. The desolvation line voltage and Q-array DC voltage were set to 0. The list of analytes, their formulae, mass/charge values used for selected ion monitoring (SIM), Limits of Detection (LOD) and Limits of Quantitation (LOQ) are shown in Table 1. Instrument LOQ was based on the lowest detected concentration with ±20% accuracy and coefficient of variation less than 5%. Instrument limit of detection was based on a signal:noise value greater than 3.

Results and Discussion

Three experiments were performed in order to validate the usefulness of this method: (1) replicate analyses of each oil sample to evaluate method reproducibility; (2) fortification and recovery of four oils at three levels; and (3) comparison of sample results to those obtained using the method of Masukawa et al. [6].

Method Reproducibility

Preliminary results with the YMC ODS-AM column and mobile phase program listed above showed potential as a direct method because analytes were generally free of

Table 1 List of analytes, formulae, mass/charge values for selected ion monitoring, limits of detection and limits of quantitation

Analyte	Formula	*m/z* $[M + H]^+$	Instrument LOD (ng/mL)	Method LOD (ng/g)	Instrument LOQ (ng/mL)	Method LOQ (ng/g)
Glycidyl laurate	$C_{15}H_{28}O_3$	257.2	2	40	10	200
Glycidyl myristate	$C_{17}H_{32}O_3$	285.2	2	40	10	200
Glycidyl palmitate	$C_{19}H_{36}O_3$	313.3	5	100	10	200
Glycidyl stearate	$C_{21}H_{40}O_3$	341.3	8	160	30	600
Glycidyl oleate	$C_{21}H_{38}O_3$	339.3	6	120	15	300
Glycidyl linoleate	$C_{21}H_{36}O_3$	337.3	3	60	10	200
Glycidyl linolenate	$C_{21}H_{34}O_3$	335.2	4	80	15	300
*d*31 Glycidyl palmitate internal standard	$C_{19}H_5d_{31}O_3$	344.4	NA	NA	NA	NA

NA not applicable

interfering peaks in the oils tested. In order to test method reproducibility, RBWD corn, RBWD canola, RBD mid-oleic sunflower, RB soy, RBD palm and palm kernel oil samples were prepared in larger volumes (1.25 g oil + 25 mL *d*31-glycidyl palmitate in acetone), and each sample was analyzed fourteen times (one injection from each of fourteen vials to prevent solvent evaporation). These analyses were performed in succession without any cleaning or maintenance operations performed on the LC–MS system. Detected levels of glycidyl esters were reproducible (Table 2). Fifteen different analyte/matrix combinations were detected at levels above the Limit of Quantitation, and all of these mean values had coefficients of variation less than 10%. Masukawa et al. [6] indicated their double SPE method was developed because analyte resolution and sensitivity decreased in their LC–MS system following multiple injections of oils dissolved directly in solvent. However, we did not observe any deterioration of system performance during the method validation experiments described here, even after greater than 200 sample injections.

Fortification and Recovery

Since the method was shown to be reproducible, the next experiment was to evaluate analyte recovery. Oil matrices included in this experiment were RBWD canola, RB soy, RBD palm and palm kernel oil. Target fortification levels were 1, 2 and 4 times the analyte LOQ, and three replicates of each fortification level were included. Sample chromatograms of a mixed standard and canola samples are presented in Fig. 1.

Recovery values ranged from 82.7% (glycidyl stearate fortified at LOQ in palm kernel oil) to 147.5% (glycidyl oleate fortified at LOQ in palm oil) and are presented in Table 3. Mean recovery values were 80–120% for 76 of the 84 analyte/sample combinations, and standard deviation values support the precision requirements of this method. Three analyte recovery values were greater than 120% because of the measurement error associated with intrinsic sample values being significantly greater than fortified levels. These values include glycidyl oleate and glycidyl linoleate fortified at the LOQ in RBD palm oil,

Table 2 Reproducibility of direct LC–MS method in six oil samples

Sample	Glycidyl laurate	Glycidyl myristate	Glycidyl palmitate	Glycidyl stearate	Glycidyl oleate	Glycidyl linoleate	Glycidyl linolenate
	Sample conc. (ng/g)						
LOQ	200	200	200	600	300	200	300
RBWD corn							
Mean	<LOQ	<LOQ	<LOQ	<LOQ	429	950	<LOQ
Std dev					41	33	
CV					9.4%	3.5%	
RBWD canola							
Mean	<LOQ	<LOQ	<LOQ	<LOQ	1,284	658	<LOQ
Std dev					51	24	
CV					4.0%	3.6%	
RBD mid oleic sunflower							
Mean	<LOQ	<LOQ	<LOQ	<LOQ	549	474	<LOQ
Std dev					43	21	
CV					7.8%	4.4%	
RB soy							
Mean	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	280	<LOQ
Std dev						18	
CV						6.4%	
RBD palm							
Mean	<LOQ	<LOQ	2,638	<LOQ	4,440	1,439	<LOQ
Std dev			76		150	37	
CV			2.9%		3.4%	2.5%	
Palm kernel							
Mean	578	308	338	<LOQ	904	569	<LOQ
Std dev	49	14	20		35	22	
CV	8.4%	4.5%	6.0%		3.8%	3.8%	

Fourteen injections of each oil from a single sample preparation

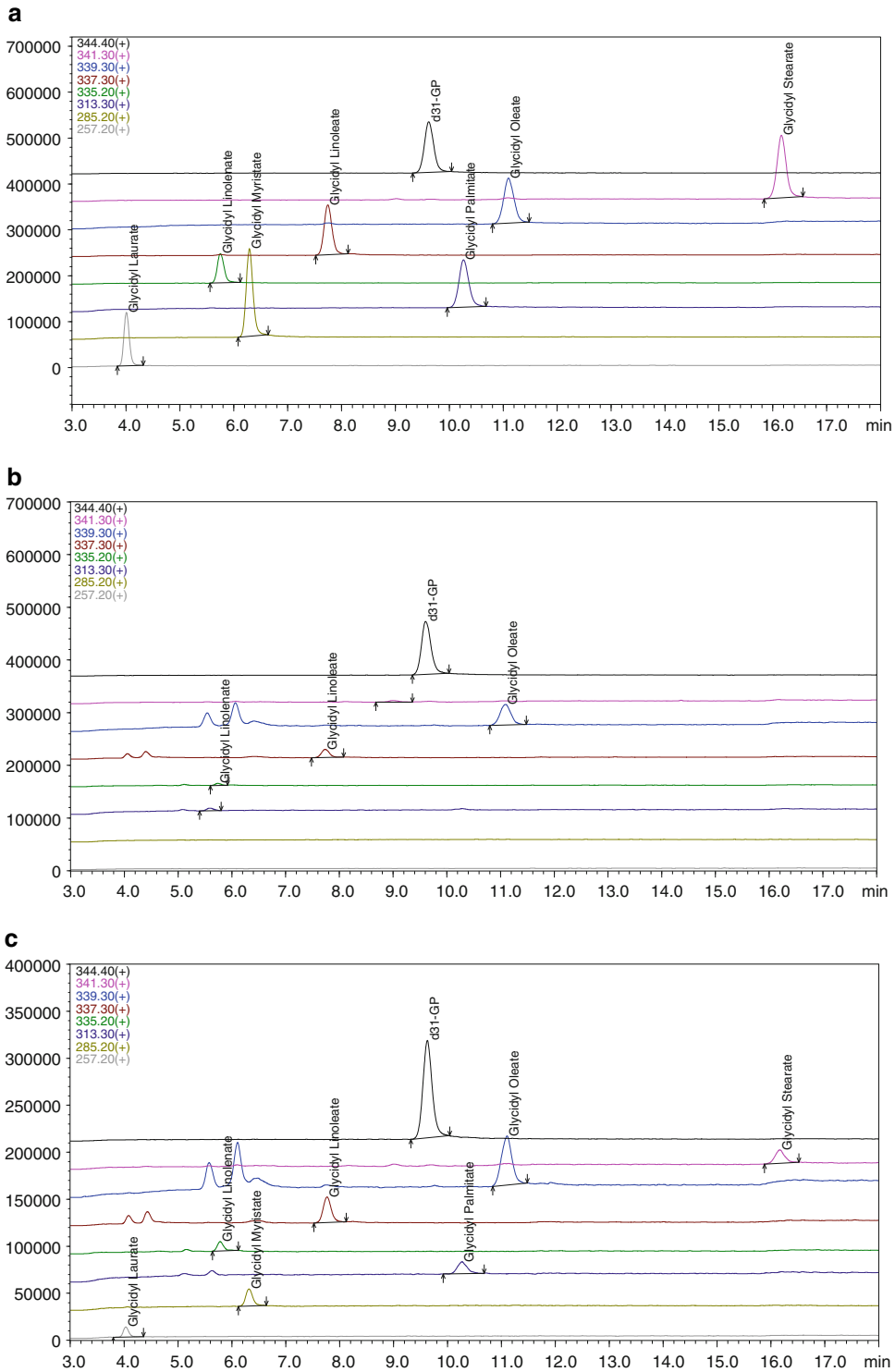

Fig. 1 LC–MS selected ion monitoring chromatograms of seven glycidyl esters plus internal standard (*d*31-GP, *d*31 glycidyl palmitate). **a** Mixed standard, **b** canola sample, **c** canola sample spiked at LOQ

and glycidyl oleate fortified at the LOQ in palm kernel oil. Although several mean analyte recovery values were greater than desired, all values were greater than 80% which indicates the method is free of interfering compounds that suppress glycidyl ester ionization. Therefore, false negative results using this method are unlikely.

Comparison of Sample Results to Previously Published Method

The last experiment performed was to compare results obtained with the direct LC–MS method to results obtained using the double SPE method of Masukawa et al. [6]. The only modifications made to the double SPE method were to

Table 3 Glycidyl ester recovery from four oil samples

Sample/fortification level	Glycidyl laurate	Glycidyl myristate	Glycidyl palmitate	Glycidyl stearate	Glycidyl oleate	Glycidyl linoleate	Glycidyl linolenate
	Sample conc./recovery						
LOQ (ng/g)	200	200	200	600	300	200	300
RBWD canola							
Sample conc. (ng/g)	<LOQ	<LOQ	<LOQ	<LOQ	1,284	658	<LOQ
1× LOQ							
Mean (%)	116.2	120.3	94.6	87.3	111.3	105.5	100.2
Std dev (%)	2.4	2.3	1.4	3.9	21.1	11.7	11.9
2× LOQ							
Mean (%)	107.1	104.8	97.4	96.5	102.0	96.0	106.2
Std dev (%)	3.0	4.8	3.9	5.8	3.6	2.2	1.3
4× LOQ							
Mean (%)	103.9	102.3	92.1	101.6	97.8	98.2	103.9
Std dev (%)	3.1	1.5	2.5	3.6	4.6	3.7	5.2
RB soy							
Sample conc. (ng/g)	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	280	<LOQ
1× LOQ							
Mean (%)	119.5	114.1	96.0	87.9	95.6	97.2	125.8
Std dev (%)	0.4	2.5	9.6	8.4	4.4	4.2	6.4
2× LOQ							
Mean (%)	112.1	105.7	94.4	96.6	99.9	101.6	121.0
Std dev (%)	6.4	4.0	3.3	2.8	3.0	3.0	7.0
4× LOQ							
Mean (%)	103.1	103.1	91.1	93.6	94.5	96.8	112.7
Std dev (%)	6.4	1.4	2.1	1.7	2.8	5.7	3.5
RBD palm							
Sample conc. (ng/g)	<LOQ	<LOQ	2,638	<LOQ	4,440	1,439	<LOQ
1× LOQ							
Mean (%)	93.1	102.7	118.5	102.5	147.5	126.0	109.3
Std dev (%)	4.6	5.9	21.3	2.0	27.9	10.5	2.5
2× LOQ							
Mean (%)	103.5	104.2	108.7	100.4	97.5	115.3	112.1
Std dev (%)	1.8	2.5	6.1	5.1	26.3	3.5	2.5
4× LOQ							
Mean (%)	105.6	105.1	100.0	108.4	104.1	118.7	114.3
Std dev (%)	2.0	0.4	0.6	5.2	8.9	3.2	4.4
Palm kernel							
Sample conc. (ng/g)	578	308	338	<LOQ	904	569	<LOQ
1× LOQ							
Mean (%)	97.6	104.2	107.7	82.7	128.5	109.3	93.5
Std dev (%)	27.3	8.0	13.7	8.7	16.8	2.0	2.3
2× LOQ							
Mean (%)	141.5	110.9	103.3	87.3	116.1	105.6	107.3
Std dev (%)	3.8	6.5	6.4	2.4	2.6	3.4	2.6
4× LOQ							
Mean (%)	139.3	111.3	100.2	92.4	110.3	104.2	113.7
Std dev (%)	1.3	2.5	4.2	3.2	3.7	2.0	3.0

Three replicates of each fortification level were included

Table 4 Comparison of glycidyl ester measurements in five oils using direct LC–MS method and double SPE method

Sample/method	Glycidyl laurate	Glycidyl myristate	Glycidyl palmitate	Glycidyl stearate	Glycidyl oleate	Glycidyl linoleate	Glycidyl linolenate
	Sample conc. (ng/g)						
LOQ (ng/g)	200	200	200	600	300	200	300
RBWD corn							
Direct LC–MS							
Mean	<LOQ	<LOQ	240	<LOQ	504	1,069	<LOQ
Std dev			5		17	12	
CV			2.0%		3.3%	1.1%	
Double SPE							
Mean	<LOQ	<LOQ	212	<LOQ	565	960	<LOQ
Std dev			22		19	71	
CV			10.1%		3.3%	7.4%	
RBWD Canola							
Direct LC–MS							
Mean	<LOQ	<LOQ	<LOQ	<LOQ	1,320	643	<LOQ
Std dev					42	26	
CV					3.2%	4.0%	
Double SPE							
Mean	<LOQ	<LOQ	<LOQ	<LOQ	1,598	477	<LOQ
Std dev					71	37	
CV					4.5%	7.7%	
RBD mid oleic sunflower							
Direct LC–MS							
Mean	<LOQ	<LOQ	<LOQ	<LOQ	579	461	<LOQ
Std dev					24	22	
CV					4.2%	4.7%	
Double SPE							
Mean	<LOQ	<LOQ	<LOQ	<LOQ	641	326	<LOQ
Std dev					33	21	
CV					5.1%	6.5%	
RB soy							
Direct LC–MS							
Mean	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	262	<LOQ
Std dev						19	
CV						7.3%	
Double SPE							
Mean	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
Std dev							
CV							
RBD palm							
Direct LC–MS							
Mean	<LOQ	<LOQ	2870	<LOQ	5,641	1,963	<LOQ
Std dev			89		261	75	
CV			3.1%		4.6%	3.8%	
Double SPE							
Mean	<LOQ	<LOQ	1905	<LOQ	4,374	1,015	<LOQ
Std dev			131		304	108	
CV			6.9%		7.0%	10.6%	

Three replicates of each method were included

Table 5 Comparison of glycidyl ester measurements in RBWD canola oil using direct LC–MS method with different sample dilution ratios

Sample dilution	Glycidyl laurate	Glycidyl myristate	Glycidyl palmitate	Glycidyl stearate	Glycidyl oleate	Glycidyl linoleate	Glycidyl linolenate
	Sample conc. (ng/g)						
Sample:solvent = 1:20							
LOQ (ng/g)	200	200	200	600	300	200	300
Mean	<LOQ	<LOQ	<LOQ	<LOQ	1,320	643	<LOQ
Std dev					43	26	
CV					3.2%	4.0%	
Sample:solvent = 1:16							
LOQ (ng/g)	160	160	160	480	240	160	240
Mean	<LOQ	<LOQ	<LOQ	<LOQ	1,383	648	<LOQ
Std dev					42	18	
CV					3.0%	2.7%	
Sample:solvent = 1:8							
LOQ (ng/g)	80	80	80	240	120	80	120
Mean	<LOQ	<LOQ	86.8	<LOQ	1,347	683	<LOQ
Std dev			4.1		42	25	
CV			4.7%		4.5%	3.7%	

Three replicates of each sample dilution were included

include *d*31-glycidyl palmitate as an internal standard, and the final volume after silica SPE clean-up was 2 mL instead of 1 mL.

Detected glycidyl ester levels between the two methods were similar (Table 4). Both methods provided acceptable coefficients of variation, and neither method consistently outperformed the other with respect to precision. Glycidyl linoleate in RB soy oil was the only analyte detected at levels above the LOQ using one method but not the other (262 ng/g using direct LC–MS method). Sample preparation using the method of Masukawa et al. [6] requires a minimum of 2 h for a single sample with the possibility of 15–20 samples per workday depending on the level of analytical skill. In contrast, the direct LC–MS method described here requires only 5 min of preparation time per sample and provides comparable results to the double SPE method.

Instrument differences notwithstanding, the original double SPE method [6] will provide greater sensitivity than the direct LC–MS method based on sample preparation—the double SPE method used 0.1 g sample with a final volume of 1 mL versus 0.25 g sample with a final volume of 5 mL for the direct LC–MS method. The 5 mL final volume was selected to ensure sample dissolution and homogeneity of the "hard" oils, but dilution ratios may be modified for the "light" oils. Sample dilution ratios for the direct LC–MS method were evaluated in RBWD canola oil using the same sample mass (0.25 g) but varying the final volume (2, 4 and 5 mL) to provide sample:solvent ratios of 1:8, 1:16 and 1:20. Glycidyl oleate and glycidyl linoleate values were consistent regardless of the sample:solvent ratio utilized (Table 5). Decreasing the solution volume from 5 to 2 mL also allowed glycidyl palmitate to be quantified in this sample. Therefore, method sensitivity may be modified for selected samples, as necessary, but the long-term effect on method ruggedness was not evaluated.

Conclusions

The direct LC–MS method provides acceptable sensitivity, reproducibility, accuracy and precision to be used as a screening method to quantify glycidyl esters of fatty acids in processed oil samples. The possibility exists that other oil samples not tested in this work could interfere with glycidyl ester quantitation, but the direct LC–MS method is free of interfering compounds that suppress analyte ionization. If glycidyl esters are detected in sample matrices at levels of concern, these samples could be reassayed using one of the double SPE methods [6, 9] to confirm analyte levels. There are a number of different single quadrupole mass spectrometers on the market today, each with their own set of ionization source parameters that can be optimized, so direct transfer of this method to instruments from other manufacturers may not be straightforward. However, the system utilized for this work proved to be extremely durable, especially in light of the fact that no cleaning or maintenance operations were performed during the course of these experiments.

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