Application Note 284

Automated, high-sensitivity analysis of residual fumigants for food safety testing by multi-step enrichment–headspace–trap (MSE–HS–trap) with GC–MS

This study shows highly sensitive detection of residual fumigants in seeds and spices by MSE–HS–trap developed on Centri 90, an automated sample concentration platform, coupled with gas chromatography–mass spectrometry (GC–MS). The MDLs obtained are as low as 0.0018 mg/kg, well below regulatory limits imposed by EU Regulation 2015/868 (0.01–0.05 mg/kg). Excellent chromatographic performance is achieved, with linearity R^2 > 0.99 and relative standard deviations <10% for all compounds, indicating good reproducibility. Full automation with prep-ahead functionality provides high productivity with throughput of approximately 40 samples per system per day. A further enhancement in sensitivity when using single-ion-monitoring (SIM) is also demonstrated, with excellent linearity and reproducibility achieved on the same analytical system for ethylene oxide and 2-chloroethanol in seeds.

Introduction

Fumigants are volatile, poisonous chemicals sprayed as gases or mists into an enclosed space to kill pests such as insects, rodents, fungi and bacteria. They are widely used on foodstuffs (among other products) to prevent spoilage during shipping or long-term storage, and are subsequently allowed to dissipate, so that (in principle) there is no risk to consumers.[1]

However, residues may remain on the surface of foodstuffs that can have harmful effects when later ingested by consumers. This has led to regulatory bodies within the EU specifying maximum residue limits (MRLs) on the concentrations of residual fumigants that may be present in imported foodstuffs, to ensure consumer and food safety.[2] Excess levels above regulatory limits can lead to product recalls and rejects, such as the thousands of food products withdrawn from sale (at great cost to suppliers) since August 2020 due to the presence of alarming levels of ethylene oxide.[3]

There is therefore a need for a sensitive, robust and high-throughput extraction method for the detection of residual fumigants from foodstuffs. In this paper, we expand on previously published work, where we developed a method for determining ethylene oxide from sesame seeds.[4] This involved a multi-step enrichment–headspace–trap (MSE–HS–trap) method, shown in Figure 1, using the Centri extraction and enrichment platform, with subsequent detection by gas chromatography–mass spectrometry (GC–MS). The method detection limits (MDLs) achieved were well below regulatory limits.

Background to Centri 90

Centri 90 is an automated sample concentration instrument for the GC–MS analysis of VOCs/SVOCs in solids, liquids and gases. Owing to Markes' world-leading focusing trap technology, the system delivers outstanding sensitivity and enhanced productivity for routine static headspace, SPME and SPME Arrow applications.

Benefit from:

- Large-volume preconcentration for headspace analysis
- Selective trap purging to remove unwanted interferences resulting in improved chromatography
- Sample re-collection for repeat analysis without re-sampling, simplifying and expediting method development and providing a reliable means of data validation.

Award-winning Multi-Gas technology is incorporated into Centri 90. Independent certification for safe use with hydrogen carrier gas (as well as helium and nitrogen) enables faster chromatographic speeds while performance is maintained and cost-of-ownership lowered.

Key applications of Markes' Centri 90 platform include food safety testing for residual fumigants in seeds and spices, investigating authenticity of olive oil (a product prone to fraudulent adulteration), and monitoring VOCs in environmental air, water and soil.

For more on Centri, visit markes.com/sample-preparation-platforms

Markes International Ltd
T: +44 (0)1443 230935 **F:** +44 (0)1443 231531 **E:** enquiries@markes.com

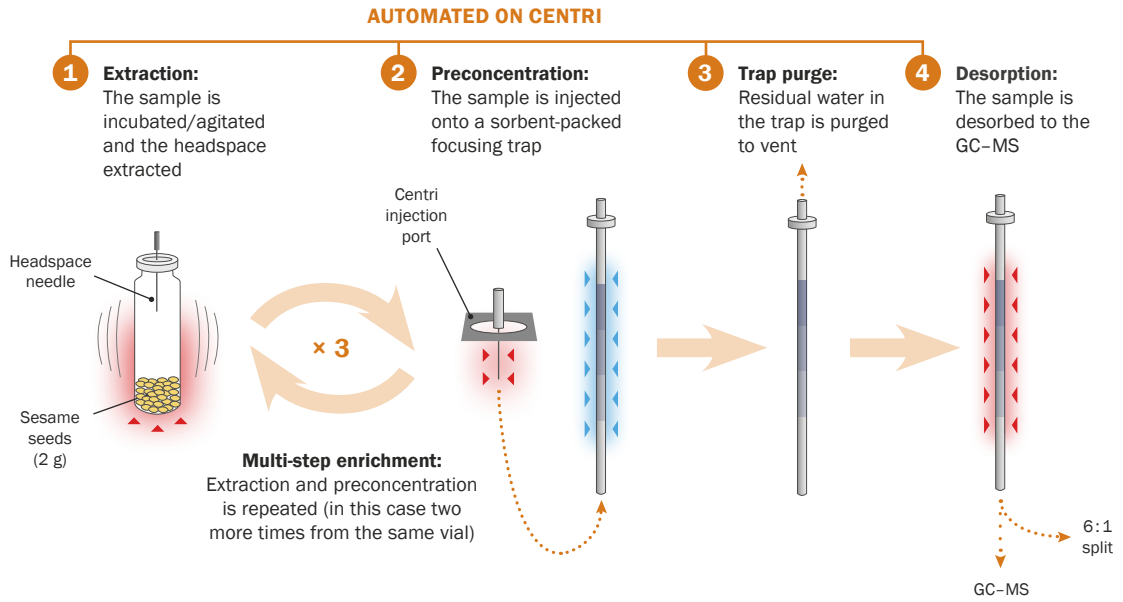

Figure 1: Headspace–trap with multi-step enrichment on the Centri platform.

In this study, we now investigate the efficacy of the same MSE–HS–trap method for five other fumigants, and demonstrate excellent analytical performance both from sesame seeds and other food matrices. We also further expand method sensitivity by adopting SIM detection, thus allowing, for example, the possibility of analysing even lower sample quantities. It is important to remember that the method requires no extraction solvent, making it more cost-effective and safer for users than other methods, and eliminating the disposal of environmentally hazardous waste.

MSE–HS–trap exploits the use of the focusing trap of Centri 90 to significantly enhance the sensitivity of traditional headspace analyses. With this technique, headspace extracted from the sample vial is injected not to the GC inlet directly, but instead to an electrically-cooled trap containing multiple sorbent beds that retain a wide range of volatile organic compounds (VOCs). The trap is subsequently purged and then rapidly heated (>100°C/s) in a reverse flow of carrier gas (backflush) so that analytes are transferred to the GC–MS for separation and detection.

The HS–trap methodology has three major advantages over direct headspace:

1. **Improved detection limits:** Typically, direct headspace is limited to ~1 mL headspace injection volume. During injection, a large split is often required to achieve good peak shape, yet by doing so, this further limits the amount of headspace being sent to the analytical system for separation and detection. Since Centri separates the HS injection from the start of the GC analysis and analytes are transferred to the focusing trap instead, this allows much larger headspace volumes (up to 5 mL, and (using MSE) multiples thereof) to be transferred to the focusing trap, with excess nitrogen or air allowed to pass through to vent while the sorbents retain compounds of interest.
2. **Improved water management:** Purging of the trap prior to desorption can help to reduce or remove residual moisture (coming from the original sample and lab air) that could reach the GC column, thereby improving chromatography and extending column and detector lifetime.
3. **Enhanced chromatography:** The narrow-bore trap design of Centri 90 combined with rapid, backflush desorption during GC injection means that analytes reach the analytical column in a tight band of vapour, enhancing peak shape.

The simultaneous analysis of a range of fumigant compounds in foodstuffs demands high throughput and full automation. MSE–HS–trap on Centri 90 provides both, with all elements of sample preparation (from sample incubation to trap desorption) being fully automated. Here, the daily throughput was calculated at approximately 40 samples per system per day. To demonstrate the applicability of the method to other sample types, a variety of food matrices (seeds, spices and ground pepper) were tested.

Experimental

The EU Reference Laboratories' document (SANTE 11312/2021) describing "method validation and analytical quality control requirements for pesticide residue analysis in food and feed" was used as guidance material to evaluate the methodology described here.[5]

Target analytes:
Five fumigants were assessed in this application, and are detailed in Table 1. Hazardous properties specified in safety data sheets (SDS) are also highlighted, showing that most fumigants are either highly toxic or carcinogenic, or both, and are a serious cause for concern to human health if detected above the MRL. Deuterated 1,2-dichloroethane (1,2-dichloroethane-d_4, quantification ion m/z 65) was used as an internal standard.

Compound	Formula	B.p. (°C)	Quant ion (m/z)	Hazardous properties	EU MRL (mg/kg)
Bromomethane	CH_3Br	3.6	94	Highly toxic	0.01[a]
Bromoethane	C_2H_5Br	38	108	Toxic	0.01[a]
Carbon tetrachloride	CCl_4	77	117	Highly toxic	0.01[b]
1,2-Dichloroethane	$C_2H_4Cl_2$	83	62	Carcinogen	0.01[c]
1,2-Dibromoethane	$C_2H_4Br_2$	131	107	Highly toxic, carcinogen	0.01[c]

Table 1: Target analyte information with EU MRLs shown, and regulation number denoted with superscript. [a] Regulation (EC) No. 1005/2009. [b] Regulation (EU) 2021/155. [c] Regulation (EU) 2012/649.

The MRLs are typically specified by sample type; for example, ground pepper has an MRL of 0.02 mg/kg for 1,2-dichloroethane and 1,2-dibromoethane. A lower, 'blanket' MRL of 0.01 mg/kg is applied where no actual MRL is specified. Therefore, this value was tested across all matrices for each compound (Table 1).

Analytical standards:
Solution A: Bromomethane stock solution was acquired at 2000 µg/mL in methanol.

Solution B: All remaining target analytes were acquired as pure liquid certified reference standards. These were subsequently combined into a single stock solution containing each analyte at a concentration of 2000 µg/mL in dichloromethane (DCM).

Solution C: Stock solutions A and B were then combined with DCM in a 1:1:2 ratio, respectively, to create a stock solution of all target analytes at 500 µg/mL. Serial dilution of stock solution C was then used to create working solutions at 10, 25, 50, 100, 200 and 300 µg/mL in DCM. When 1 µL of each is spiked onto 2 g sample matrix, the calibration to assess linearity equates to 0.005–0.15 mg/kg.

Guidance requires a multi-level calibration of three or more data points, with the lowest calibration level (LCL) equal to or lower than the reporting limit (RL). Here, the LCL is 0.005 mg/kg, which is two times lower than the smallest RL of 0.01 mg/kg.

The internal standard, 1,2-dichloroethane-d_4, was acquired as a pure liquid reference standard. A 2000 µg/mL stock solution was produced by dilution in DCM, and from this a 200 µg/mL working solution in DCM was created by further dilution. The internal standard (1 µL) was added to each sample vial during preparation, equating to 0.1 mg/kg in concentration, and therefore can also be referred to as a procedural internal standard.

Analyte handling:
Some target analytes are extremely volatile and can readily evaporate, so great care must be taken to prevent evaporative loss, which would otherwise introduce errors in calculating concentrations. Pure standards, stock and working solutions were wrapped in a sealing film and stored at –20°C when not in use. When in use they were opened for as short a time as possible and returned to the freezer immediately upon completion of the work. All glassware was chilled prior to work commencing, and laboratory air temperature was controlled at 20°C. New stock solutions were prepared every 14 days, and working solutions were never used more than three times (guidance from SANTE section F).

Matrix-matching:
The background produced by the sample (or matrix) must be considered when developing a new method for analysing target compounds in foodstuffs. This is because matrix effects can lead to poor quantification for various reasons, such as analyte interference, where a co-elution may occur and resulting in an over-reporting of the resultant target analyte concentration.

Matrix effects are a regular occurrence in GC methods, so combatting any issues before extraction is necessary and should always be assessed at the earliest stage when developing a method. A common approach is to matrix-match, where calibration samples are prepared using clean, blank matrix that is the same as the samples for investigation. Blanks of this clean matrix are analysed to determine the background prior to analysis of the samples in question, and method alterations can be made at this point.

Including internal standards can also compensate for matrix effects. For example, the internal standard (ISTD) can compensate for any changes between the extraction of different vials in a sequence and allow the raw data to be corrected. Using ISTDs is key to enabling accurate reporting of results. In this application, 1,2-dichloroethane-d_4 was used as an internal standard at a constant concentration of 0.1 mg/kg. For each matrix analysed, these measures have been implemented for accurate reporting.

For quantitative methods, representative matrices are a requirement for validation of both single- and multi-residue methods, and so this approach has been used for this investigation.

Sample preparation:
The food matrices analysed (sesame seeds, ground black pepper and crushed chilli flakes) were acquired from a local supermarket. Each matrix (2 g) was weighed into a 20 mL crimp-top vial, then 1,2-dichloroethane-d_4 internal standard solution (1 µL, 200 µg/mL) and the target analyte working solutions (1 µL, varying concentrations) were added with a liquid syringe. Vials were immediately sealed by crimping to reduce evaporative loss. The final concentration of target compounds on matrix was therefore equivalent to 0.005–0.15 mg/kg; the internal standard concentration was at 0.1 mg/kg.

Additional replicate samples were prepared for analysis at the stated concentrations to determine the following:

- Recoveries at a midpoint level, 0.1 mg/kg, n = 7
- Within-laboratory reproducibility (RSD_{wR}), 0.1 mg/kg, n = 7
- Method detection limits (MDLs), 0.0125 mg/kg, n = 8

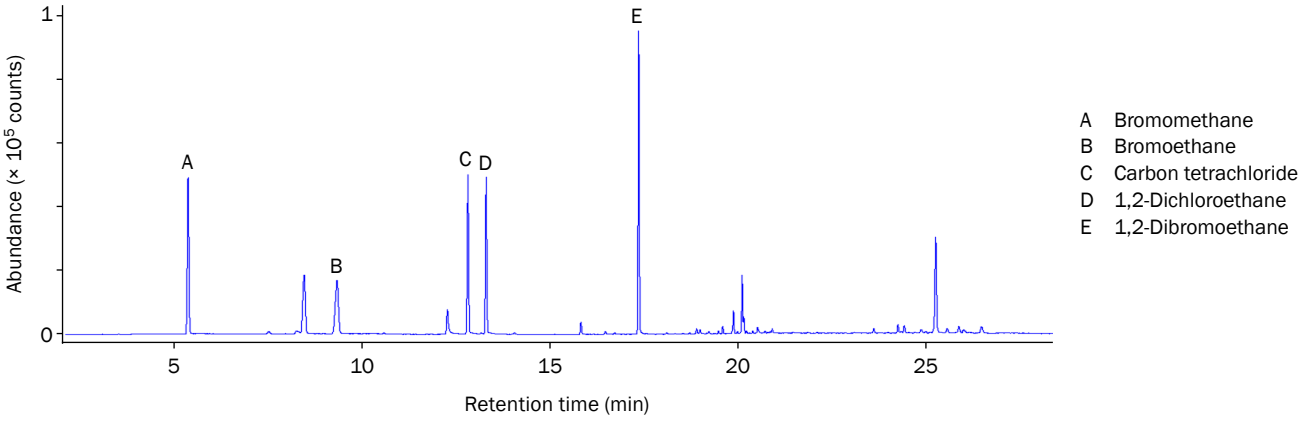

Figure 2: Extracted-ion chromatogram (merged EIC of m/z 94, 108, 31, 117, 62 and 107) for all target compounds, spiked at 0.05 mg/kg on sesame seeds.

Sample extraction and preconcentration:
Instrument: Centri 90 (Markes International)

Headspace–trap:
Incubation: 70°C (10 min) with agitation at 300 rpm
Extraction volume: 5 mL
Injection: 200°C (2 min)
Enrichment: Three extractions from the same sample vial, with a 3 min sampling delay between repeat extraction (total 15 mL extracted for analysis)
Flow path: 180°C
Focusing trap: U-T23ETO-2S
Purge flow: 20 mL/min for 1 min
Trap low: –30°C
Trap high: 250°C (3 min)
Split flow: 10 mL/min (6:1 split)

GC:
Column: MEGA®-624, 60 m × 0.25 mm i.d. × 1.4 µm film thickness
Column flow: 2 mL/min
Oven ramp: 30°C (6 min), 10°C/min to 100°C (1 min), 20°C/min to 230°C (8 min)

MS:
MS transfer line: 230°C
MS source: 250°C
Scan range: m/z 27–300

Results and discussion

Sesame seeds

For the preliminary analyses, sesame seeds were used as the benchmark prior to testing other matrices for the additional fumigants, since a method for extracting ethylene oxide from this matrix was previously developed and demonstrated.[4] Seeds were prepared as described above and sample vials were transferred immediately to the Centri 90 for automated, unattended analysis, ideal for rapidly analysing large batches of samples in situations where time-to-result is crucial.

Figure 2 shows the merged extracted ion chromatogram (EIC) using the quantification ion masses (Table 1) of the target analytes added to sesame seeds at 0.05 mg/kg. All compounds are readily apparent, with excellent peak shape resulting from both analyte refocusing on the trap and the subsequent fast desorption to the analytical column in a narrow band.

From these analyses we were able to determine excellent linearities of $R^2 > 0.997$ for the six-point calibrations for all target compounds, using internal standard correction to adjust for any variability (Figure 3). Signal-to-noise ratios were all ≥3 and seven replicates for each target compound provided RSDs below 7%.

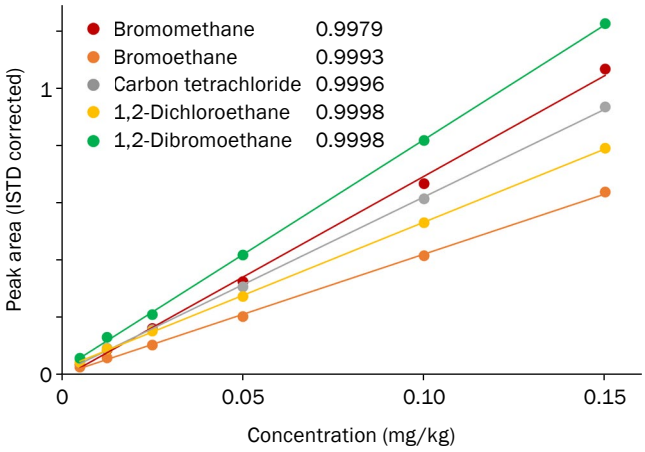

Figure 3: Calibration curves of all fumigants tested in sesame seeds, from 0.005–0.15 mg/kg, with good linearity ($R^2 > 0.997$).

Method detection limits (MDLs) were calculated from eight replicates (n = 8) of fumigants spiked onto seeds at 0.0125 mg/kg. The peak areas obtained were calculated to mg/kg concentrations and standard deviations determined. Next, these values were multiplied by the Student's t-test statistic for seven degrees of freedom (n – 1) at the 99% confidence interval, which is 2.9980.[6] This method is recommended by the US Environmental Protection Agency,

and has previously been used to determine MDLs when developing the ethylene oxide method.[4,7]

Table 2 shows the MDLs calculated for each fumigant. As shown, all MDLs are below the 0.005 mg/kg LOQ and well below 0.01 mg/kg, the lowest MRL for any of the fumigants investigated (Table 1). This demonstrates that MSE–HS–trap with GC–MS surpasses the sensitivity required for compliant detection of fumigants from sesame seeds.

Compound	Calculated MDL (mg/kg)
Bromomethane	0.0018
Bromoethane	0.0018
Carbon tetrachloride	0.0021
1,2-Dichloroethane	0.0033
1,2-Dibromoethane	0.0031

Table 2: Method detection limits (MDLs) for fumigants in sesame seeds.

Ground black pepper and chilli flakes

To demonstrate the applicability of this developed method to other sample matrices, ground black pepper and crushed chilli flakes were also tested. These matrices posed a greater analytical challenge than sesame seeds, in the form of their strong aromas originating from a complex range of volatile organic compounds (VOCs) they emit. Some of these may be present in high concentrations, and could interfere with fumigant analysis if they happened to co-elute with the target analytes. This emphasises why matrix-matching and using internal standards to correct this issue is vital for analysis of fumigants. The analytical system must also be capable of evaluating samples over a wide concentration range without any significant hardware changes.

Overall aroma profiles

Figure 4 shows the total ion chromatograms (TICs) for ground pepper (green) and chilli flakes (purple). This time window reveals a range of organoleptic compounds that contribute to the overall aroma profiles, with variability in the intensity of common peaks seen between the two sample types. Higher abundances of sabinene, α-pinene and 3-carene were noted in the ground pepper, providing woody, spicy and citrus notes, which correlate to typical aromas identified in pepper samples.[8] These compounds were also detected in the chilli flakes but were less abundant. Acetic acid was more intense in the chilli profile and is usually identified in this sample type, providing pungent, sharp and sour notes. Two compounds, 2-methylbutanal and 3-methylbutanal, were also more abundant in chilli flakes, contributing roasted notes.[9] Both compounds are common products of Maillard reactions, and so suggest this product has undergone some form of heating process during production.

Despite the many compounds present in these matrices, target fumigant compounds carbon tetrachloride, 1,2-dichloroethane and 1,2-dibromoethane (at 0.05 mg/kg) could still be easily identified in this time window by using extracted ion chromatograms (Figure 5), with excellent peak shape observed.

It is worth noting that although the fumigant peaks identified have an intensity about 1000 times lower than the highly abundant aroma-active compounds, this single analysis demonstrates how both high and low concentration compounds are confidently and automatically detected and identified using this method, without any manual sample preparation or intervention.

Ground black pepper

Blank ground black pepper was prepared as stated, with fumigants at six concentration levels ranging from 0.005 to 0.15 mg/kg. As shown in Figure 6, excellent linearity was observed, with R^2 values all in excess of 0.992.

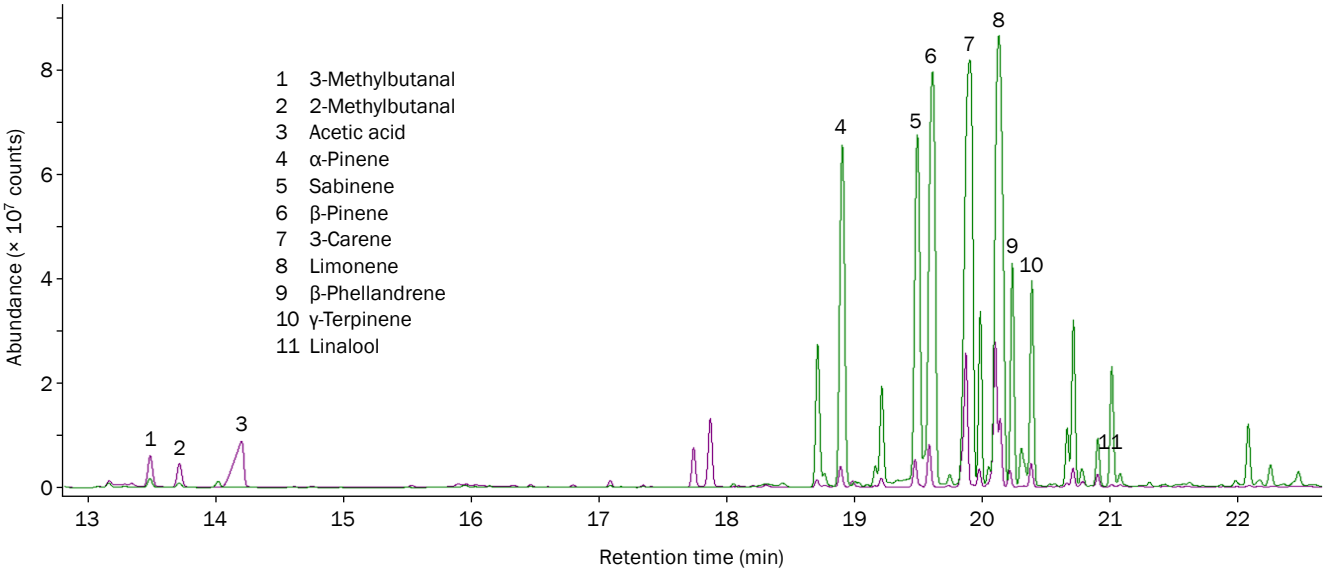

Figure 4: TICs of ground pepper (green) and chilli flakes (purple) showing key aroma compounds identified among a range of other matrix-derived compounds.

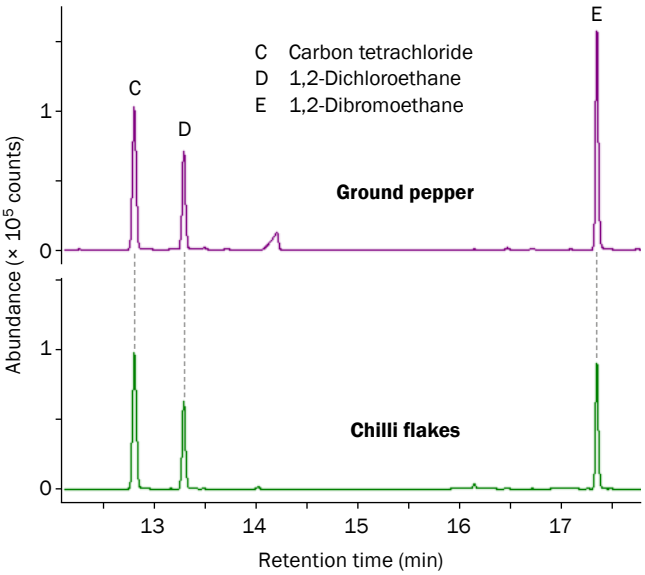

Figure 5: A merged EIC (m/z 62, 107, 117) of three trace-level fumigants, successfully extracted at 0.05 mg/kg in both the ground pepper and chilli flake samples.

Mean recovery values at each spiking level must generally fall in the range of 70–120% to demonstrate a robust, quantitative analytical method. In this study, each of the fumigants provided excellent recoveries from data produced at 0.1 mg/kg (Table 3). Within-laboratory reproducibility (RSD_{wR}) should be from a minimum of five replicates and "derived from on-going method validation". Here, this was assessed with seven replicates at 0.1 mg/kg on the ground pepper samples, and reproducibility was excellent, with relative standard deviation (RSD) values below 5.4% in all cases (Table 3).

Chilli flakes

Excellent linearity was also observed for the analysis of target fumigants in chilli flakes, demonstrated in Figure 7, with R^2 values above 0.994 for the same concentration range 0.005–0.15 mg/kg. RSD values were slightly higher in the chilli results compared to the ground pepper, but were still below 9% for all target compounds. Recoveries were in accordance with the guidelines, but bromomethane did demonstrate more variability here, with lower recovery values compared to the other fumigants analysed (Table 4). The high volatility of bromomethane may have played a role here, therefore fresh standards and rapid capping of vials is highly advisable.

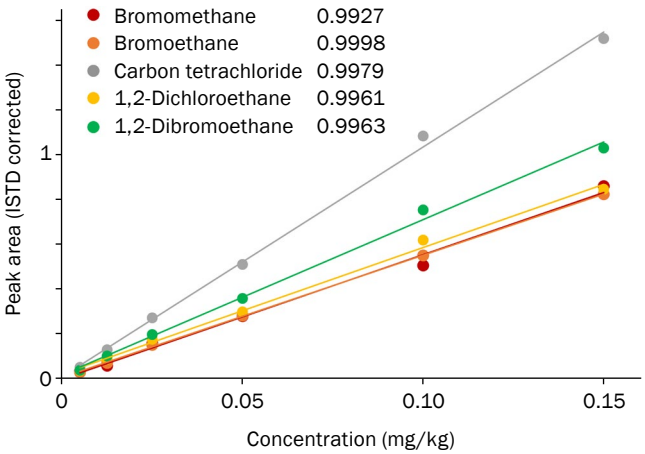

Figure 6: Calibration curves for all fumigants tested in the ground pepper samples from 0.005–0.15 mg/kg, with good linearity (R^2 > 0.992).

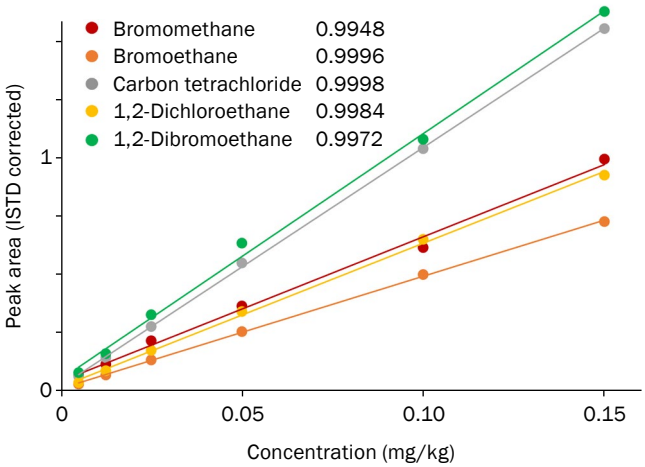

Figure 7: Calibration curves for all fumigants tested in the chilli flake samples from 0.005–0.15 mg/kg, with good linearity (R^2 > 0.994).

Compound	Recovery (%)	Reproducibility (%)
Bromomethane	85–114	4.85
Bromoethane	89–108	2.16
Carbon tetrachloride	97–111	4.50
1,2-Dichloroethane	94–113	5.35
1,2-Dibromoethane	102–119	4.99

Table 3: Recovery and reproducibility for all fumigants tested in the ground pepper samples at 0.1 mg/kg (n = 7).

Compound	Recovery (%)	Reproducibility (%)
Bromomethane	72–101	5.06
Bromoethane	91–114	5.88
Carbon tetrachloride	89–113	6.58
1,2-Dichloroethane	90–110	8.27
1,2-Dibromoethane	94–120	8.92

Table 4: Recovery and reproducibility for all fumigants tested in the chilli flake samples at 0.1 mg/kg (n = 7).

The results from both ground pepper and chilli flakes indicate that the method developed on Centri 90 is robust and reproducible when applied to a variety of sample types that undergo fumigation. Emphasis must be placed on taking due care and attention during sample preparation to minimise evaporative loss of these highly volatile target compounds prior to analysis. Matrix-matching is also an important step in sample preparation, to ensure any variability or interferences from the sample matrix are accounted for.

Extending sensitivity for ethylene oxide analysis using SIM mode

We previously reported a newly developed method for simultaneous analysis of ethylene oxide (EtO) and 2-chloroethanol (2-CE) in sesame seeds.[4] Since publication of the application note, regulatory bodies responsible for setting testing limits have implemented more stringent maximum residue limits of 0.01 mg/kg, five times lower than the previous MRL (0.05 mg/kg), and in-line with those stated for other residual fumigants like those evaluated above.

As a result, the developed method on Centri (used above) was evaluated again here, but this time with the single-quadrupole mass spectrometer operating in single ion monitoring (SIM) mode. This requires a simple change to the MS settings that allow only compound-specific ions to be filtered and detected in the defined retention windows where each compound elutes, and so provides an enhancement in sensitivity for the required trace-level analysis.

Sesame seeds were a priority foodstuff for testing, and therefore uncontaminated samples were obtained from a local supermarket and prepared as described:

- 2 g sample accurately weighed in 20 mL vials.
- 1,2-Dichloroethane-d_4 internal standard solution introduced (1 µL, 200 µg/mL).

The target analyte working solutions, ranging from 10 to 500 µg/mL (1 µL) were prepared (as described in Application Note 281) and introduced to the sample vials, which were rapidly crimp-capped to prevent evaporative analyte losses The resulting calibration range equated to 0.005–0.25 mg/kg, covering the new MRL of 0.01 mg/kg.

All above-mentioned method parameters were kept the same, with the exception that the MS was set to detect both a quantifier ion and qualifier ion, respectively, for each compound:

- Ethylene oxide: m/z 29 and 44 (time window 2)
- 2-Chloroethanol: m/z 31 and 44 (time window 4)
- 1,2-Dichloroethane-d_4: m/z 65 and 102 (time window 3).

When operating the mass spectrometer in SIM mode, the ratio between the intensities produced by the quantifier and qualifier ion peaks selected for each compound is experimentally determined by analysis of known standards.[11] The ratio is then assessed during data processing of sample batches, to provide further validation and confirmation of the peak detected (this is sometimes a requirement of standard methods).

The first step was to perform eight-point calibrations for EtO and 2-CE. Compared to results previously reported, the lower calibration point was extended down to 0.005 mg/kg, while keeping the range the same (up to 0.250 mg/kg).

Exceptional chromatographic performance, particularly for highly volatile EtO, was observed for all points. Controlled sub-ambient trap cooling enabled efficient retention of this challenging compound during headspace enrichment. An example chromatogram showing simultaneous analysis of EtO and 2-CE at the 0.05 mg/kg spiking level is shown in Figure 8. Subsequent backflush desorption of analytes retained on the trap to the GC in a narrow band provided superior, symmetrical peak shape, even at low levels, further enhancing the sensitivity achieved for both EtO (Figure 9) and 2-CE.

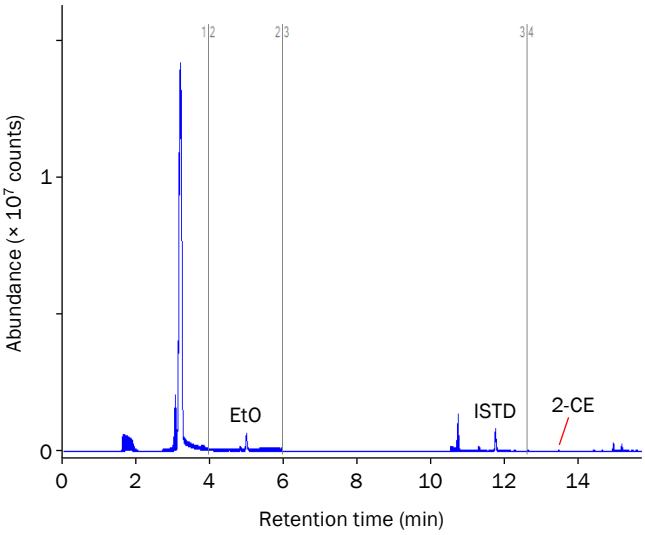

Figure 8: Example chromatogram of sesame seeds spiked with 0.05 mg/kg EtO and 2-CE (and 1,2-dichloroethane-d_4, ISTD), analysed using SIM mode on the mass spectrometer.

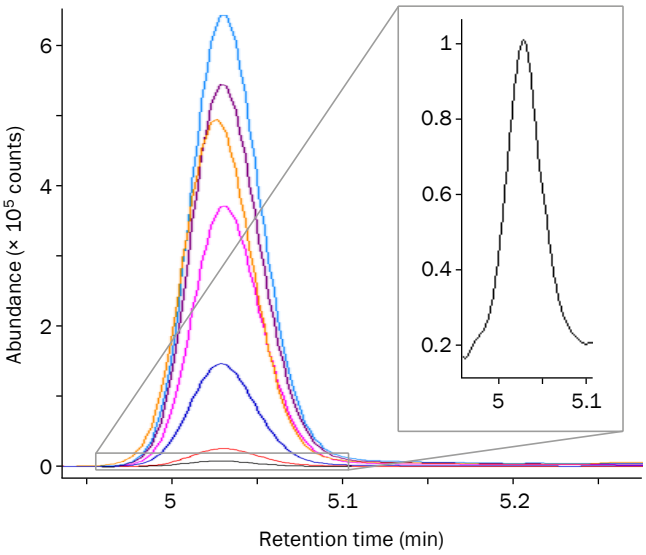

Figure 9: Peak shape of EtO across the entire calibration range. Inset shows peak shape at the lowest calibration point (0.005 mg/kg).

Excellent linearity was achieved, with R^2 values >0.999 (Figure 10), and relative standard deviations (n = 5) reported as <10% for both compounds, demonstrating good reproducibility. Recovery values evaluated with seven replicates of a mid-range calibration point at 0.05 mg/kg provided averaged values of 109% and 102% for EtO and 2-CE, respectively, both falling within acceptable limits.

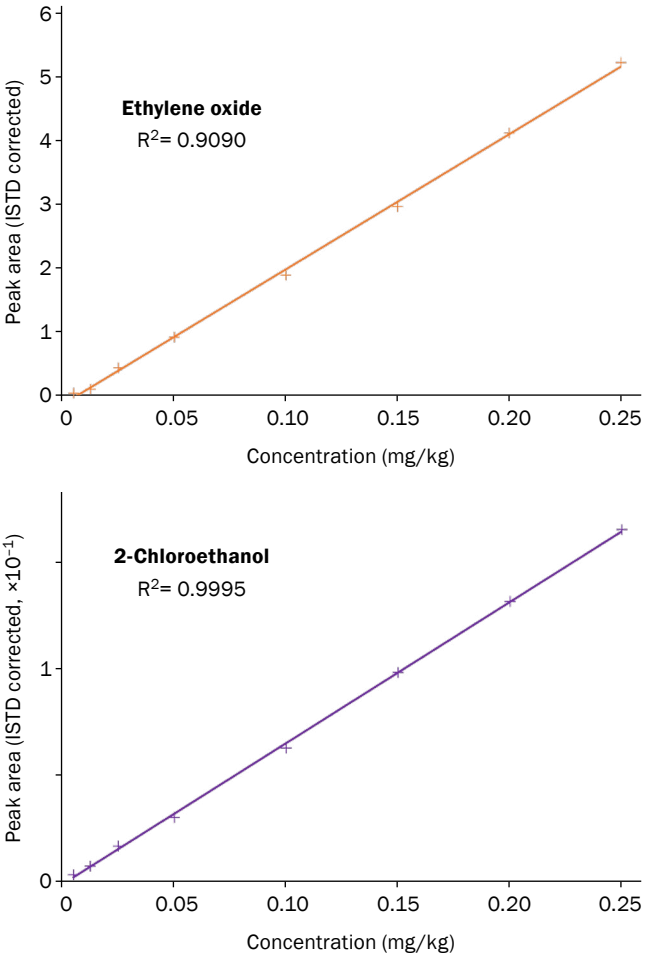

Figure 10: Calibration curves for ethylene oxide (top, m/z 29) and 2-chloroethanol (bottom, m/z 31) in sesame seeds, demonstrating excellent linearity.

It is important (once again) to note the use of an in-vial internal standard and matrix-matching, so that any variation in extraction is accounted for, and peak responses can be corrected to provide accurate results.

Here, ion ratios were all within the acceptable thresholds set in the quantitation methods (Figure 11), thus validating the developed method for sensitive detection of EtO and 2-CE in sesame seeds.

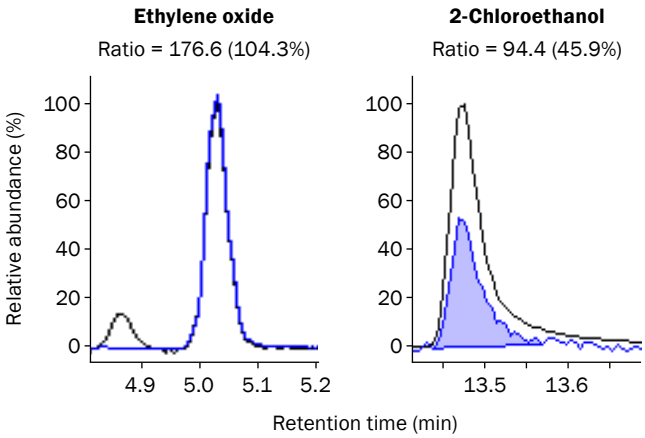

Figure 11: Example of peaks used for determining ion ratios for EtO (left, m/z ions 29 and 44) and 2-CE (right, m/z ions 31 and 44) in sesame seeds.

Conclusions

A multi-step enrichment–headspace–trap (MSE–HS–trap) method is reported for the detection of residual fumigant compounds ethylene oxide (EtO) and 2-chloroethanol (2-CE) in sesame seeds at concentrations down to 0.05 mg/kg. However, since the use of EtO is banned in many countries, we also optimised the method to rapidly monitor a range of other alternative fumigants.

Performed using the HS–trap capability on the new Centri 90 platform, the method exploits the backflushed, cryogen-free, multi-sorbent trap to concentrate a wide volatility range of analytes in a single GC run, improve chromatographic performance particularly for these challenging compounds, and extend sensitivity to reach lower limits being imposed by regulatory bodies.

Large-volume preconcentration was facilitated by the trap, enabling 5 mL injection volumes (compared to typical 1 mL volumes of conventional HS systems), and then multiples of 5 mL, giving a total extracted volume of 15 mL from the same sample vial, analysed in a single run. As well as improving sensitivity, this method also proved to be robust, quantitative and reproducible, with detection limits well below maximum residue limits (MRLs) imposed by governing bodies such as the EU.

Next, MSE–HS–trap was applied to a variety of foodstuffs increasingly flagged by the EU's Rapid Alert System for Food and Feed (RASFF) network. Excellent results continued to be observed, indicating the method is broadly applicable across not only a wide range of analytes but also different matrices, such as ground pepper and chilli flakes. Sensitivity enhancements were also seen when applying this method and operating the mass spectrometer in SIM mode for one of the most challenging of compounds – ethylene oxide – in sesame seeds.

After quick and easy sample preparation by an operator, and without the use of harmful solvents, fully automated analysis with prep-ahead functionality enabled more than 40 samples to be analysed per system in one day, ideal for large sample batches in high-productivity laboratories.

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Applications were performed under the stated analytical conditions. Operation under different conditions, or with incompatible sample matrices, may impact the performance shown.