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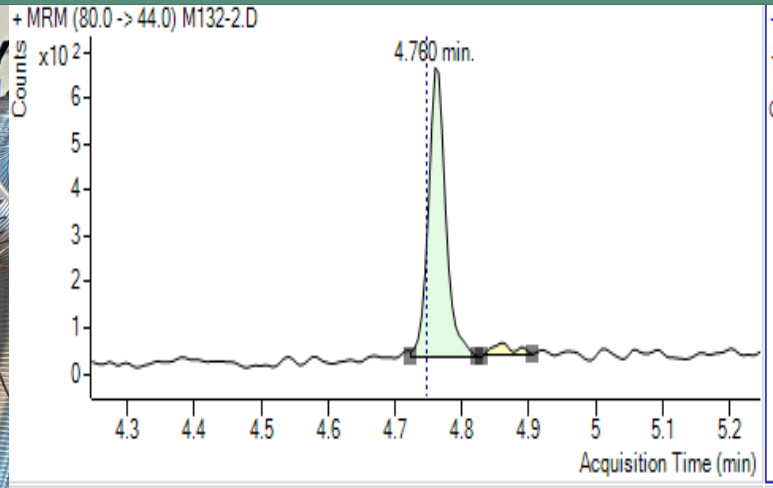
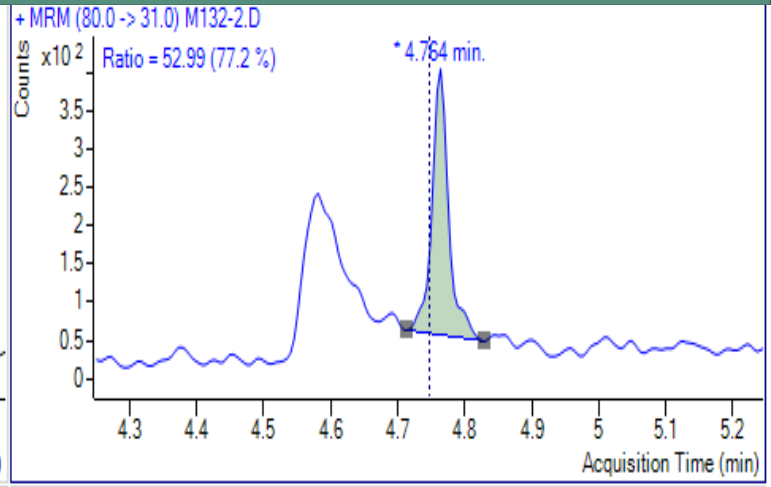
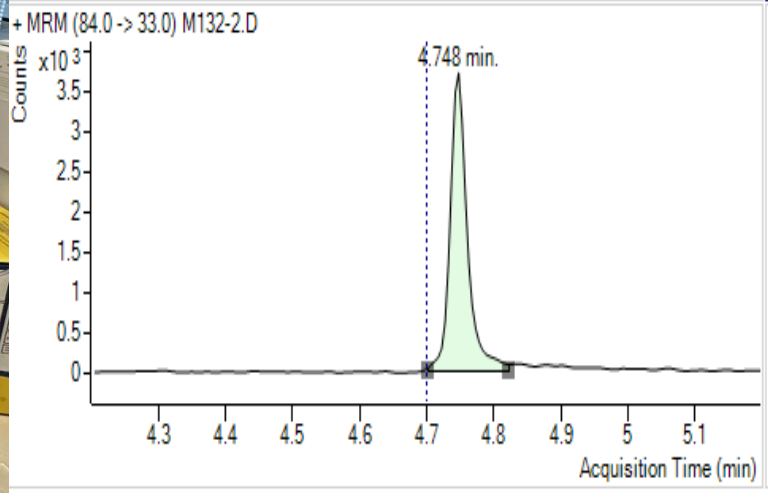
NORWEGIAN INSTITUTE OF BIOECONOMY RESEARCH

Ethylene oxide analysis

Lina Aarsbog and Hans Ragnar Norli – EURL-SRM worshop 2024







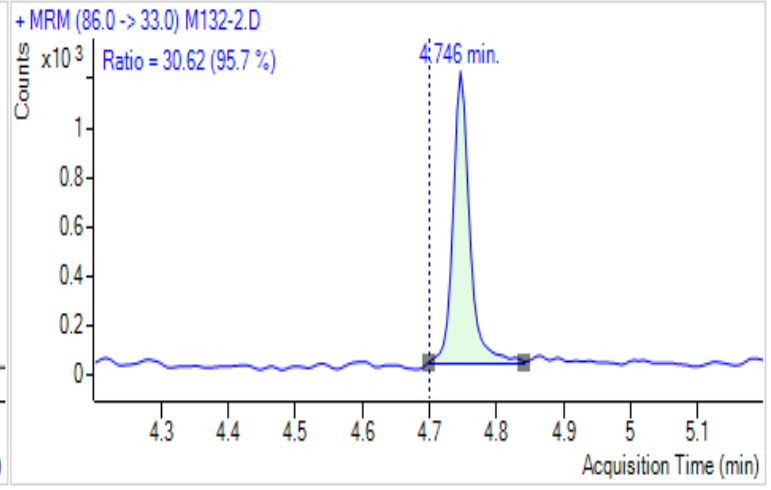


Foto: Erling Fløistad/NIBIO

Norwegian institution of bioeconomy research

- National reference laboratory for pesticides and plant toxins in food
- Target analysis of over 400 pesticides
- Analysing, sampling and reporting in connection with the Norwegian Food Safety Authority's monitoring program for pesticide residues in food

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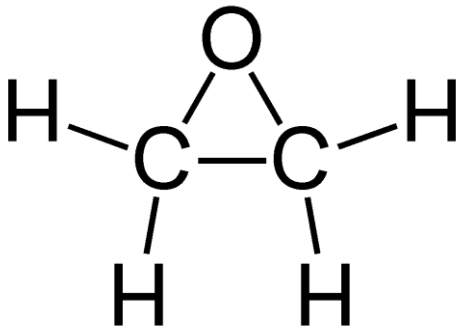
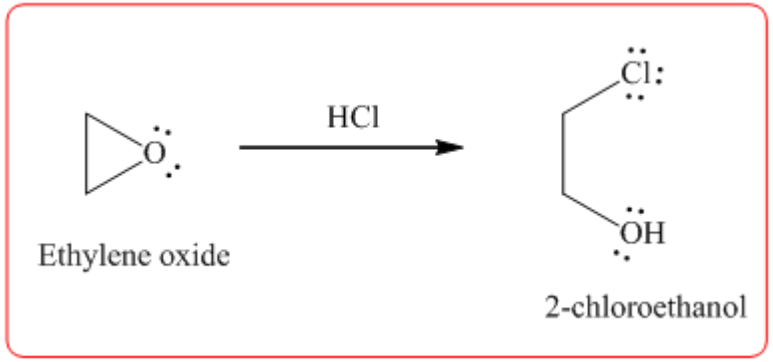
Ethylene oxide analysis - initiation



- Ethylene oxide «crisis» in 2021
- Initiated method development in 2022. Completed after summer in 2023.
- RD: Ethylene oxide (sum of ethylene oxide and 2-chloro-ethanol expressed as ethylene oxide)



Instrument method

GC	Agilent 8890
Injector	Agilent 7693A Automatic liquid sampler
MS/MS	Agilent 7010B
Column	J&W HP-VOC GC Column, 30 m, 0.20 mm, 1.10 µm, Part Number:19091R-303
Pre-column	2,5 m x 0,25mm methyl deactivated retention gap
Mobile phase/flow	Helium / 1,0 ml/min
Injection mode / volume	1:10 split / 1µl
Injection temp program	Start at 90°C and hold for 0.8 min, ramp with 12°C/s to 280°C, hold 10 min.
Liner	2 mm deactivated dimpled liner
Oven temp program	Start at 45 °C, hold 2 min, ramp with 0,83 °C/s to 220°C hold 0 min. Total run time: 5.5 min
Backflush	10 min at 280 °C, 4,4 void volumes backflushed.
MS parameters	Ionisation mode: EI Transfer line temperature: 280 °C; Ion source temperature: 230°C

Instrument method

	Precursor	Product	CE
Ethylene oxide	44	28	5
Ethylene oxide	44	29	5
Ethylene oxide-D4	48	16	40
Ethylene oxide-D4	48	30	5
2-chloro ethanol	86	33	5
2-chloro ethanol	84	33	5
2-chloro ethanol-D4	80	31	5
2- chloro ethanol-D4	80	44	0

Standard	Provider
Etylene oxide	Sigma
Ethylene oxide-D4	Gifted to us
2-chloro ethanol	Sigma
2-chloro ethanol-D4	HPC

Photo: Lina Aarsbog/NIBIO

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Extraction

- Extraction method: QuEChERS-method with citrate buffer and dispersive clean up with PSA/C18/MgSO4
- Modification: Addition of 5% water in the acetonitrile for dry products and ceramic homogenizers before liquid addition.

Photo: Lina Aarsbog/NIBIO

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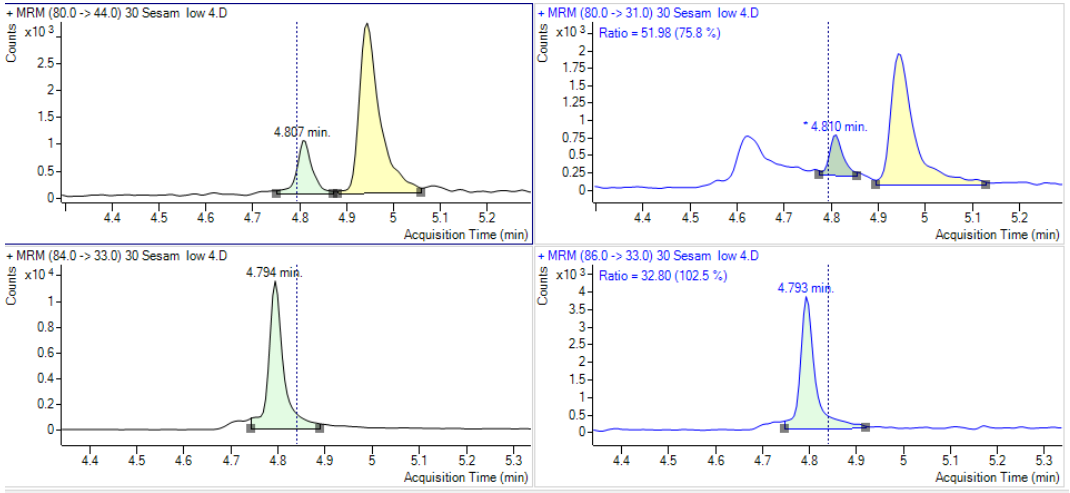
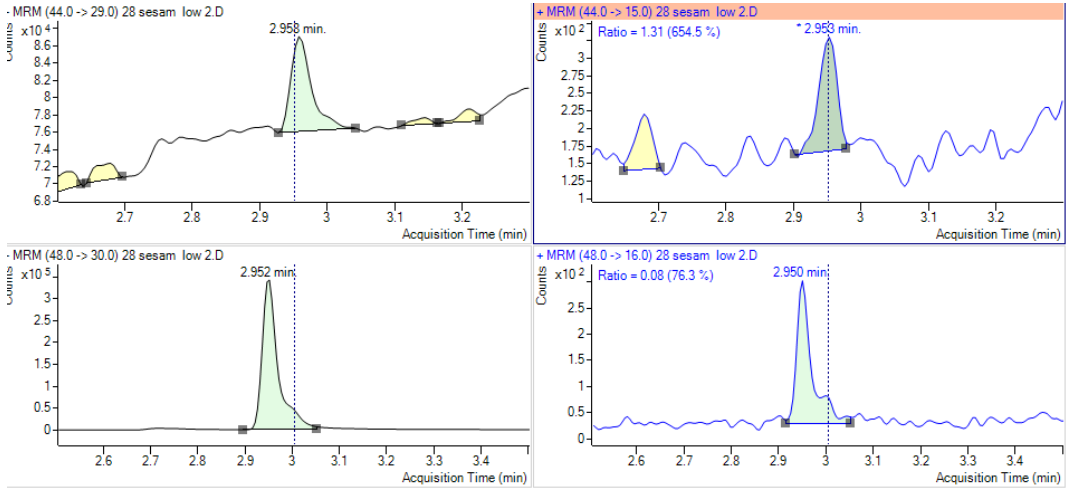
Validation

- Planned according to SANTE
- Matrices: Wheat, sesame seeds, peppercorn, and salad
- EUPT-SRM 16
- Calibration in solution: 0,005-1,0 µg/ml
- Target LOQ: 0,01 mg/kg for 2-chloro ethanol and 0,02 mg/kg for ethylene oxide



2-chloro ethanol at 0,01 mg/kg



Ethylene oxide at 0,02 mg/kg

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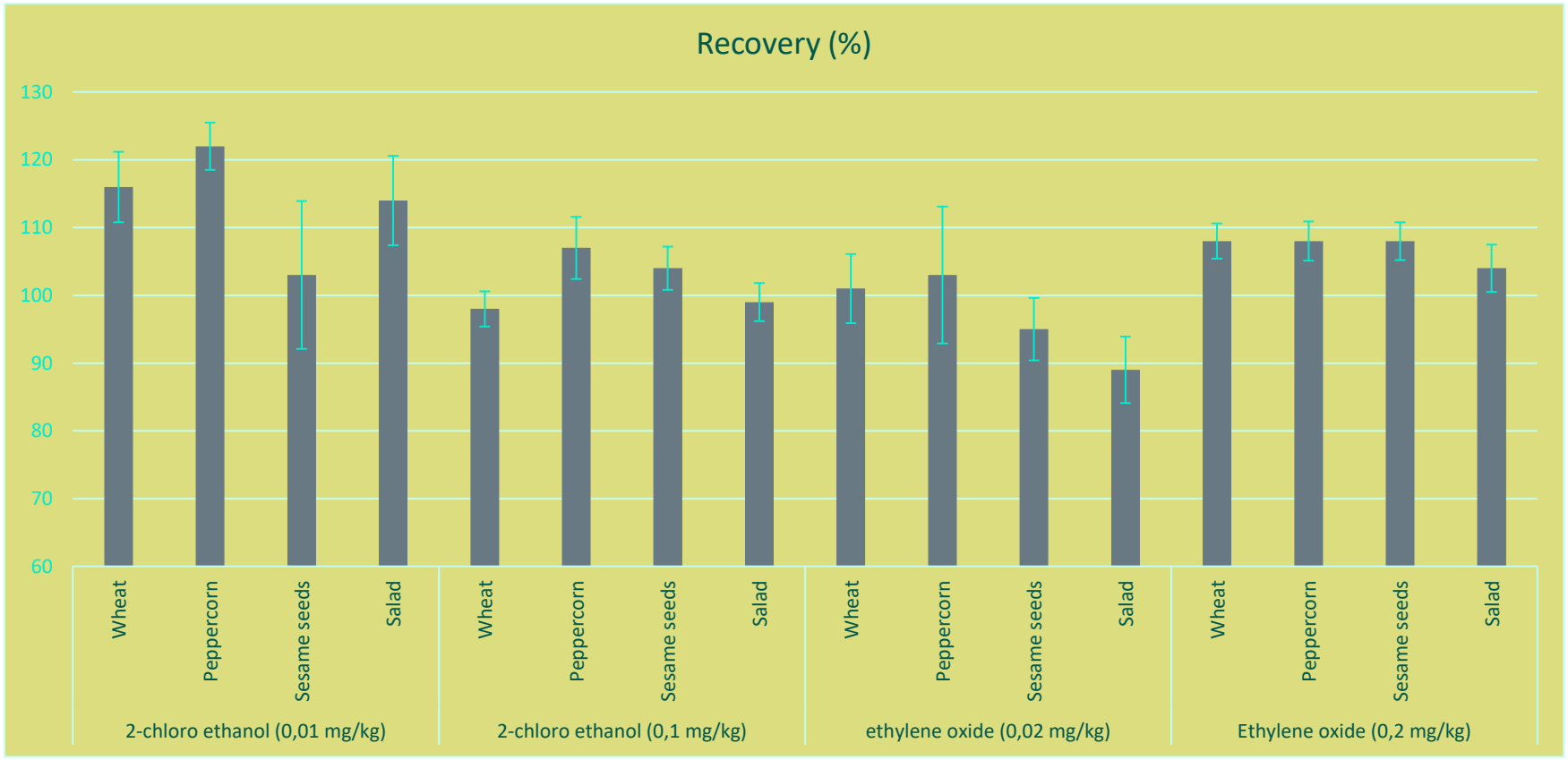
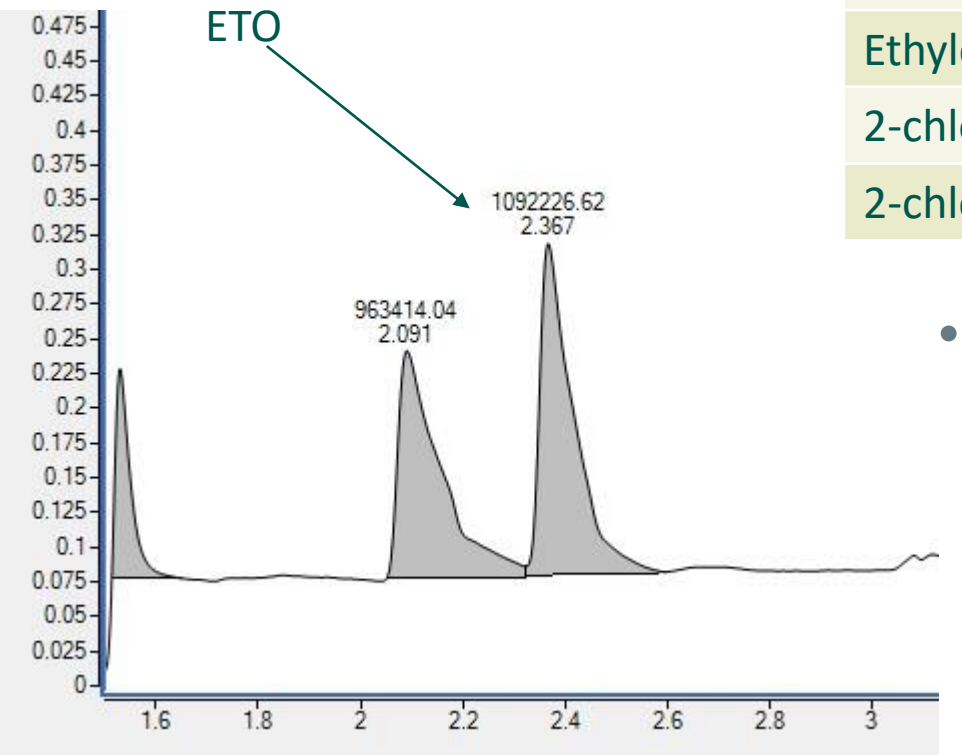
Validation - results



Validation - results



Matrix effects				
	Wheat	Peppercorn	Sesame seeds	Salat
Ethylene oxide	-1 %	-2 %	-7 %	9 %
Ethylene oxide-D4	2 %	4 %	-4 %	12 %
2-chloro ethanol	2 %	-23 %	1 %	0 %
2-chloro ethanol-D4	2 %	-23 %	4 %	0 %

- EUPT-SRM 16 on sesame seeds
  - Assigned value: 4,59 mg/kg
  - Our result: 4,27 mg/kg
  - Calculated z-score: -0,23

Application

- Ethylene oxide included in MACP in 2023: Dry beans, rice and rye
  - 1 exceedance of MRL in dry beans from Turkey
- Import control (1793) in 2023: Okra and supplement ingredients
- MACP 2024: Wheat and baby food of porridge
- NACP 2024: Peppercorn
- Import control (1793) in 2024: Okra, sesame seeds and ingredients

Photo: Silje Kvist Simonsen/NIBIO

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Summary – our experience

- Method based on method from the observation report from EURL-SRM
- No major surprises during set up and validation of the method
- Fast analysis and extraction time

Photo: Siri Svendgård-Stokke/NIBIO

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