

Quantitative NMR Foundation Meeting

Spectral Service



Round Robin Test: Edible and Krill Oil Analysis

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Lipid Analysis

Spectral Service



Questions:

How can we ensure the quality of our holistic control of edible oils?

Are preparation and analysis easy to handle?

How is the quality of the PL analysis?

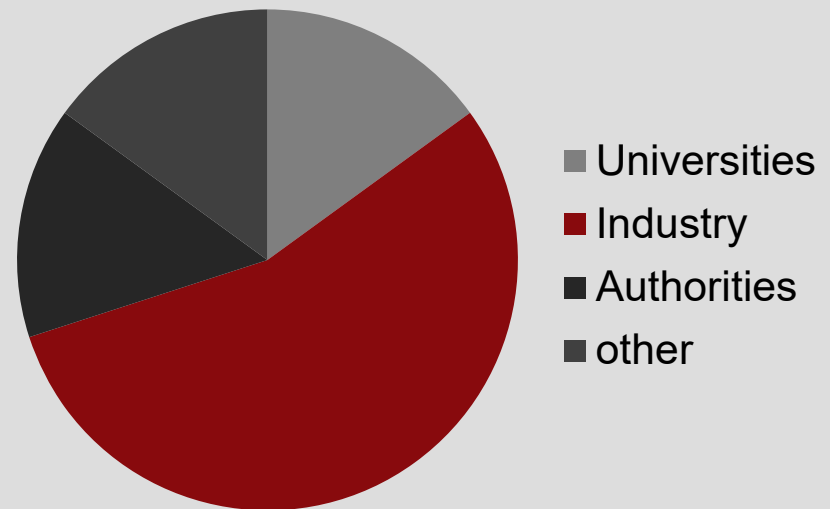
→ Round Robin Tests

Lipid Analysis

Spectral Service



20 Laboratories



Samples

5 Edible Oils

5 Krill Oils

Lipid Analysis

Spectral Service



Oil Analysis

Krill Analysis

Sample
Preparation

NMR Analysis

Spectra Evaluation

by Participant

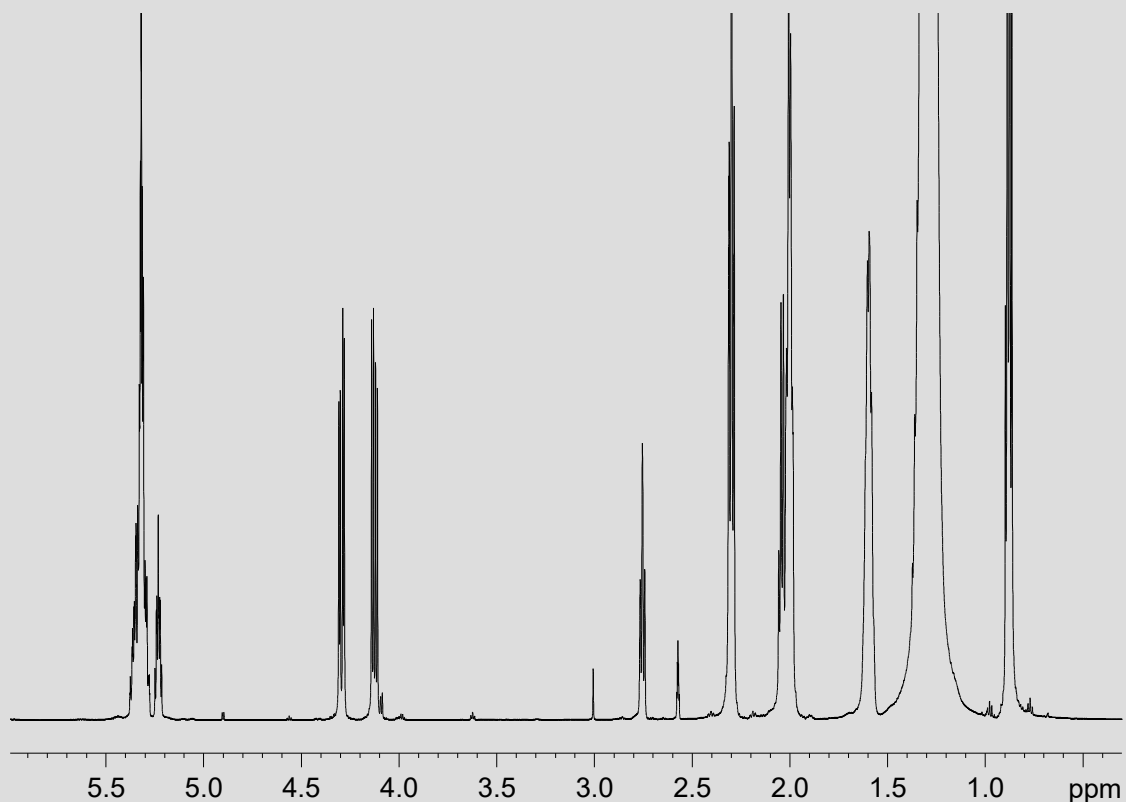
by Spectral Service



Analysis of Edible Oils

15 signals are integrated:

- FFA
- Peroxides
- Aldehydes
- Double Bonds
- Glycerin
- C18:3 and C18:2
- α -CH₂ and β -CH₂
- CH₂ long chain
- terminal CH₃

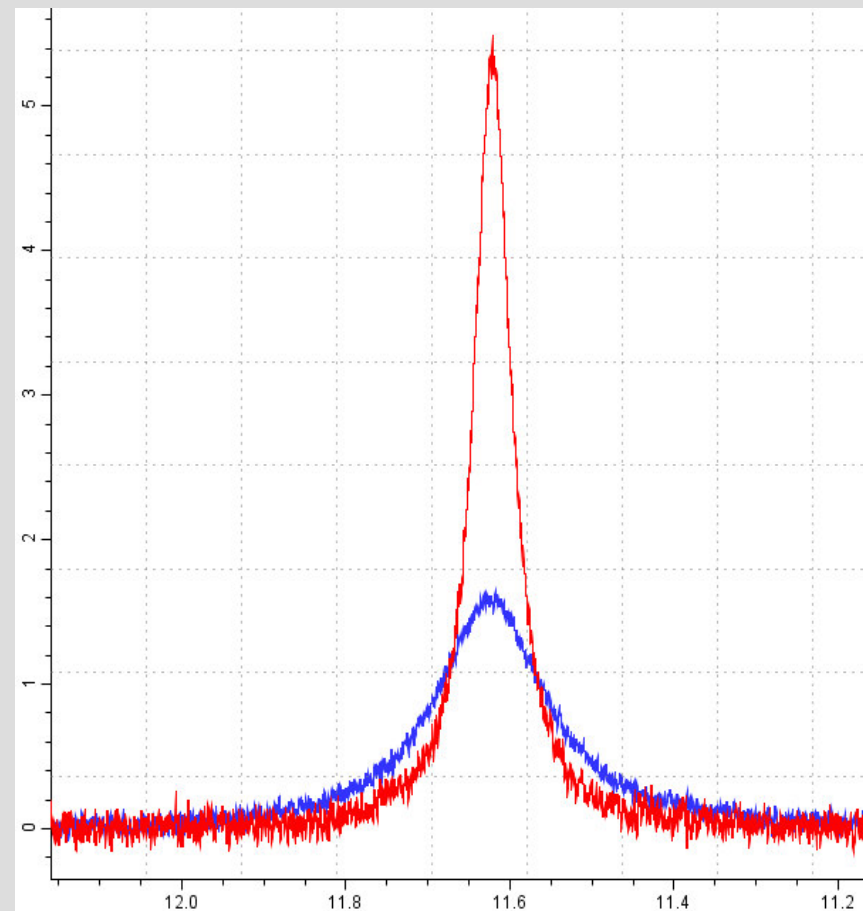


Analysis of Edible Oils: FFA

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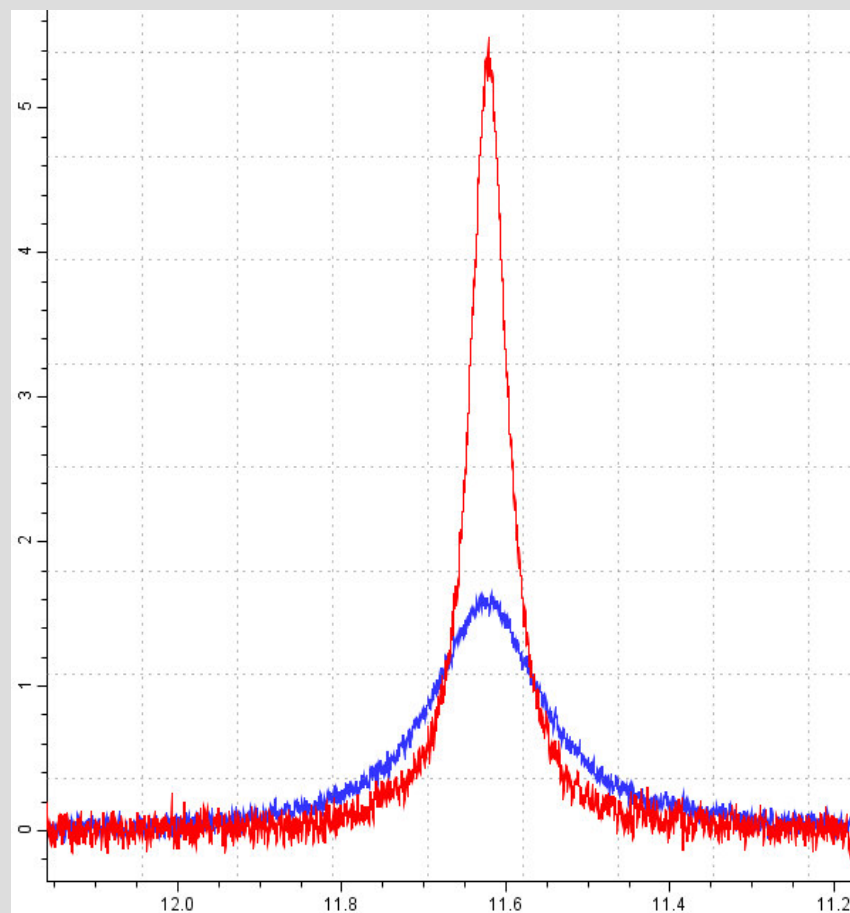
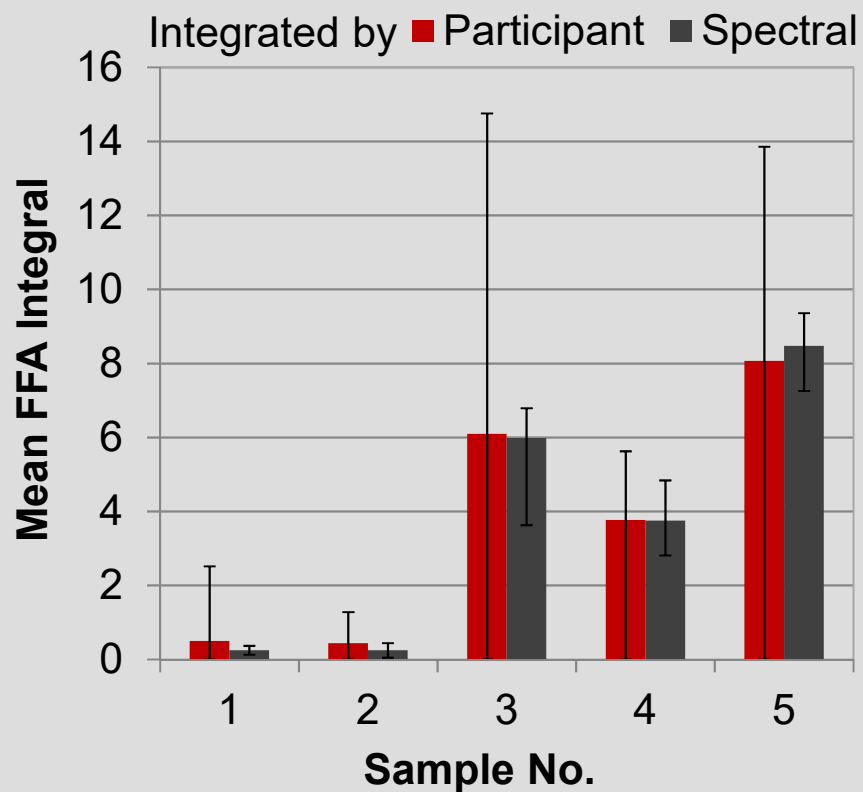


Sample	Mean	SD	SD [%]
Oil 1	0.39	0.33	84.56
Oil 2	0.28	0.31	78.10
Oil 3	6.29	0.89	14.08
Oil 4	4.18	0.77	18.43
Oil 5	8.92	1.55	17.32



Analysis of Edible Oils: FFA

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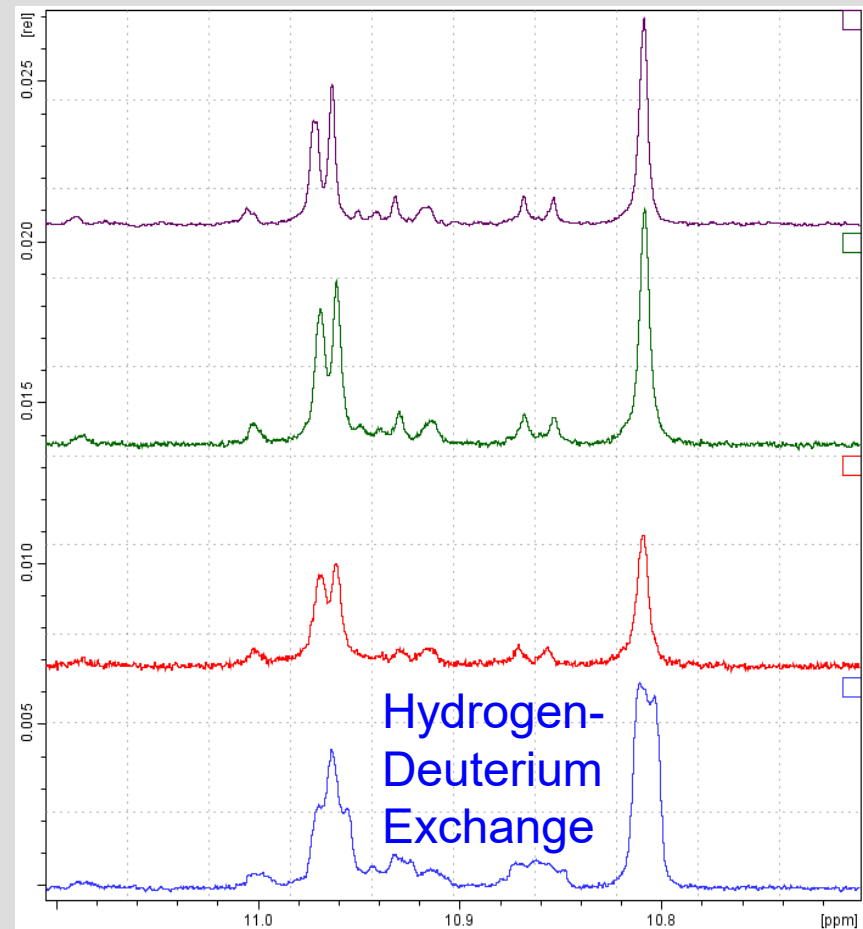


Analysis of Edible Oils: Peroxide

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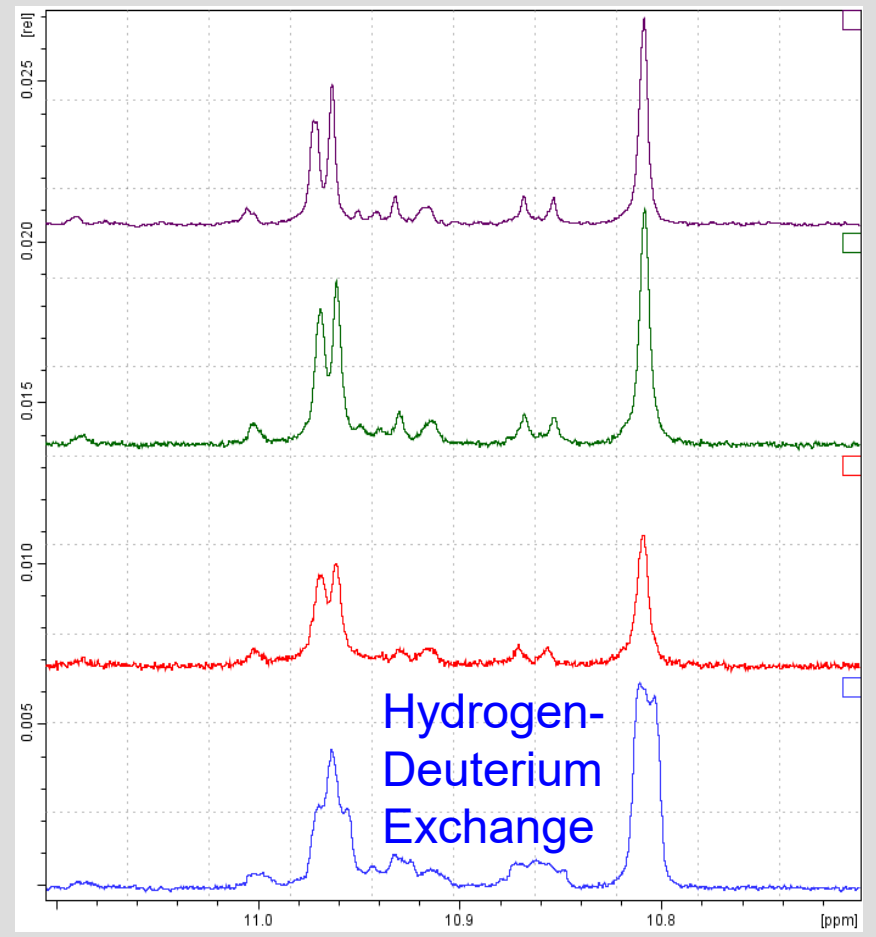
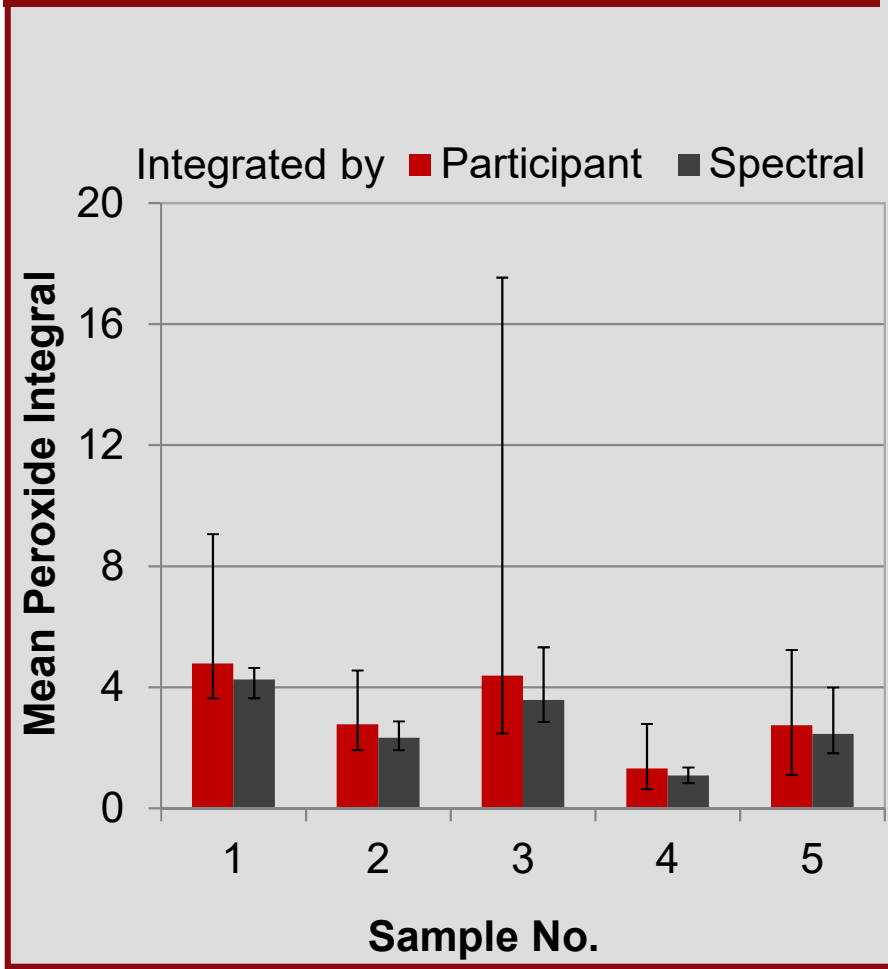


Sample	Mean	SD	SD [%]
Oil 1	4.55	0.75	16.40
Oil 2	2.68	0.56	20.88
Oil 3	3.68	0.84	22.67
Oil 4	1.24	0.39	31.96
Oil 5	2.60	0.79	30.54



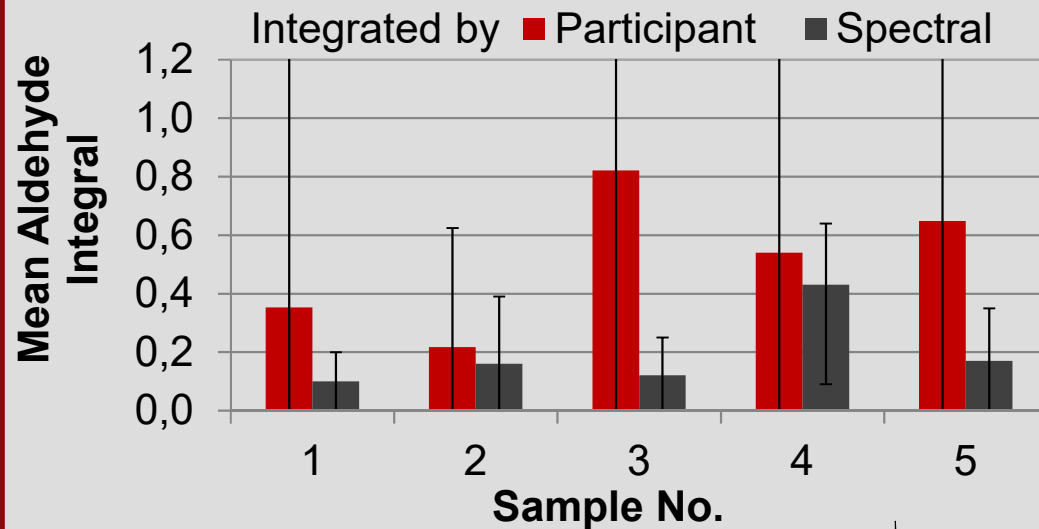


Analysis of Edible Oils: Peroxide

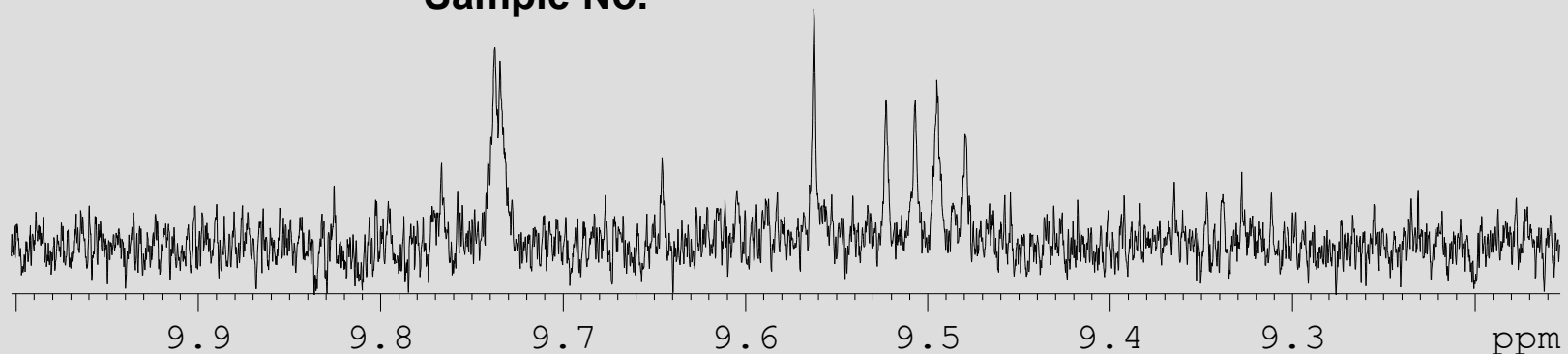




Analysis of Edible Oils: Aldehyde



$SD [\%]_{\min} = 56.24$
 $SD [\%]_{\max} = 131.90$



Analysis of Edible Oils: other

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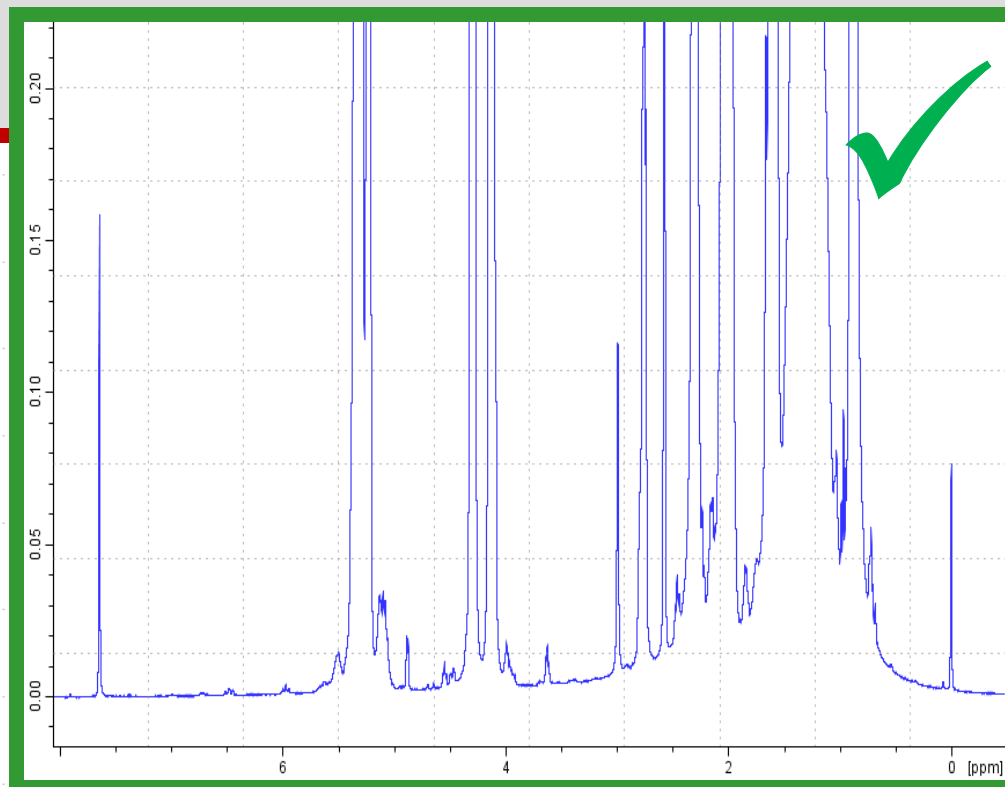
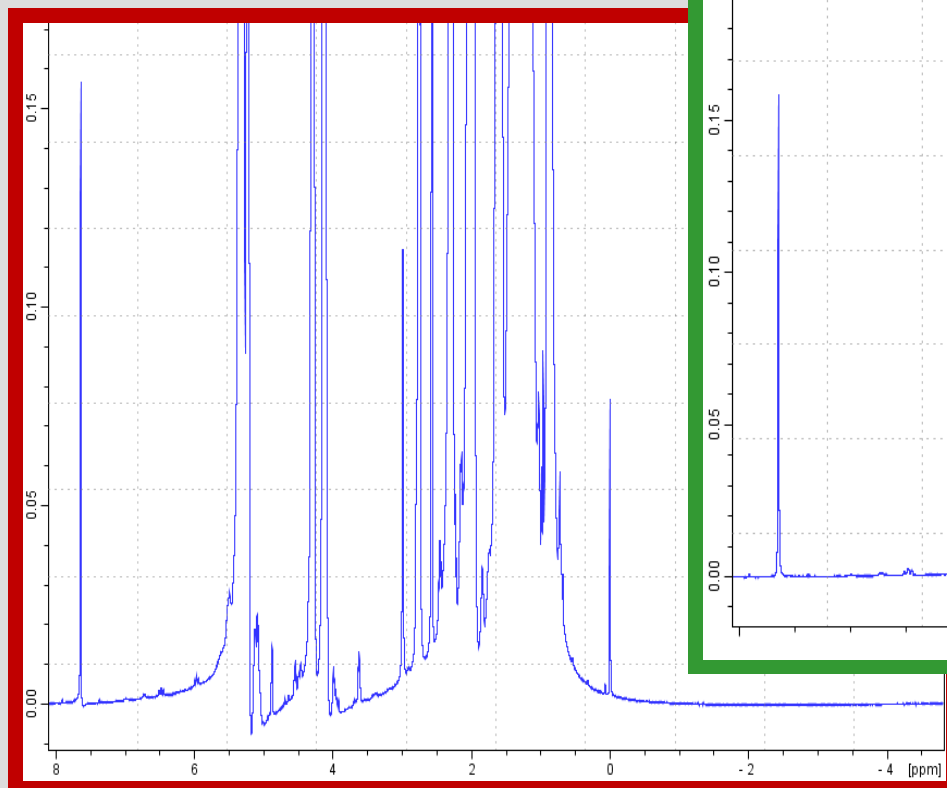
	SD_{\min} [%]	SD_{\max} [%]
Double Bonds	0.78	3.51
Glycerin	1.00	5.25
C18:3	44.37	441.36
C18:2	34.33	51.59
β -CH ₂	1.73	2.32
CH ₂ long chain	0.91	2.17
Terminal CH ₃	1.98	7.92

Troubleshooting

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Phase correction

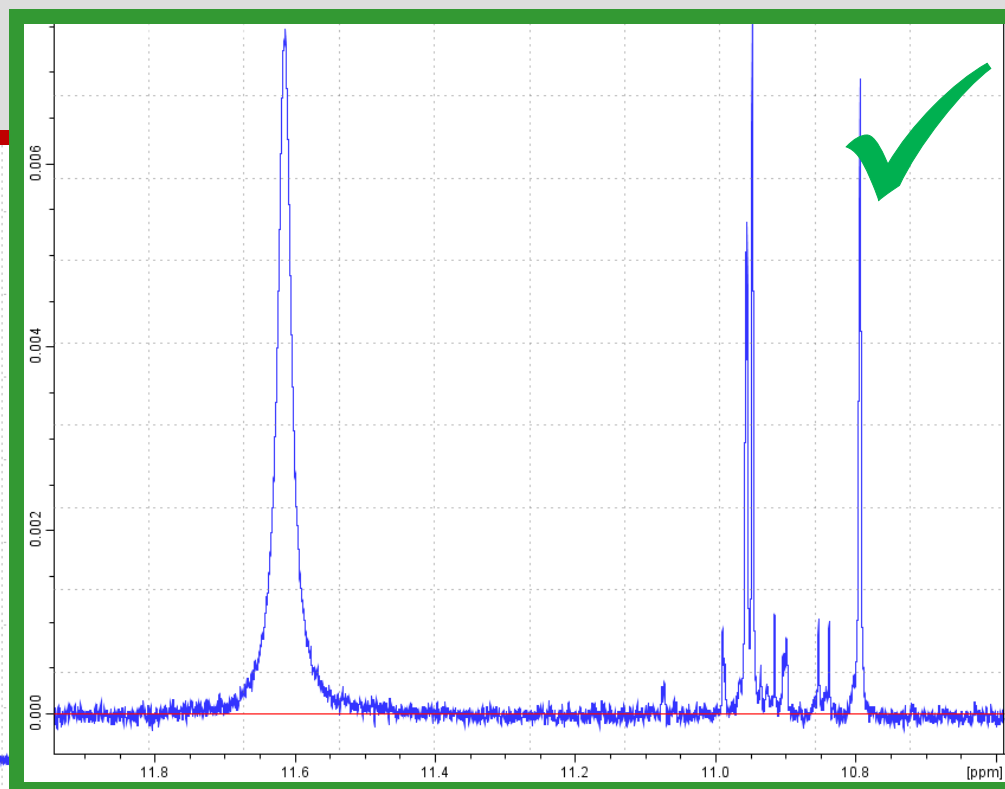
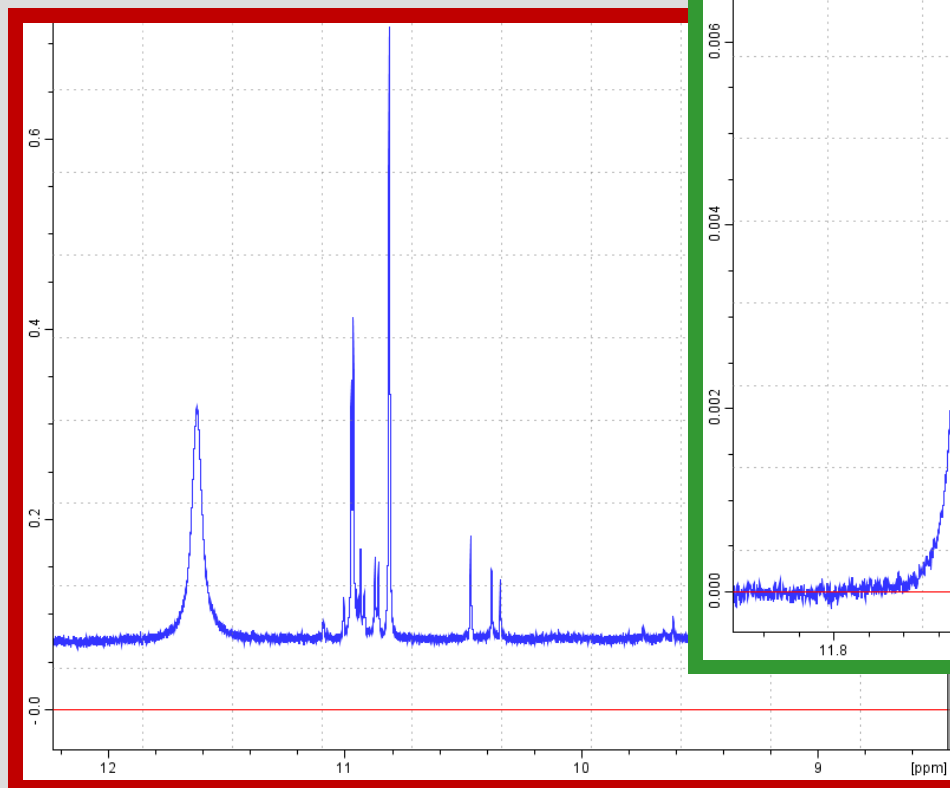


Troubleshooting

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Baseline correction

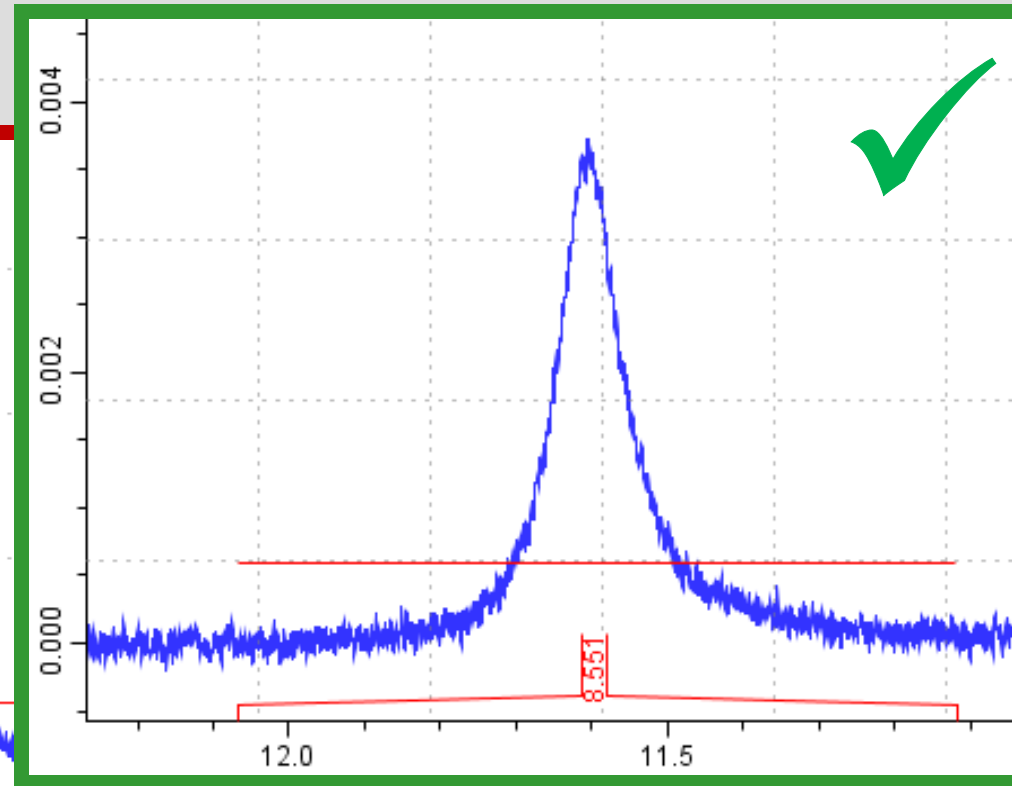
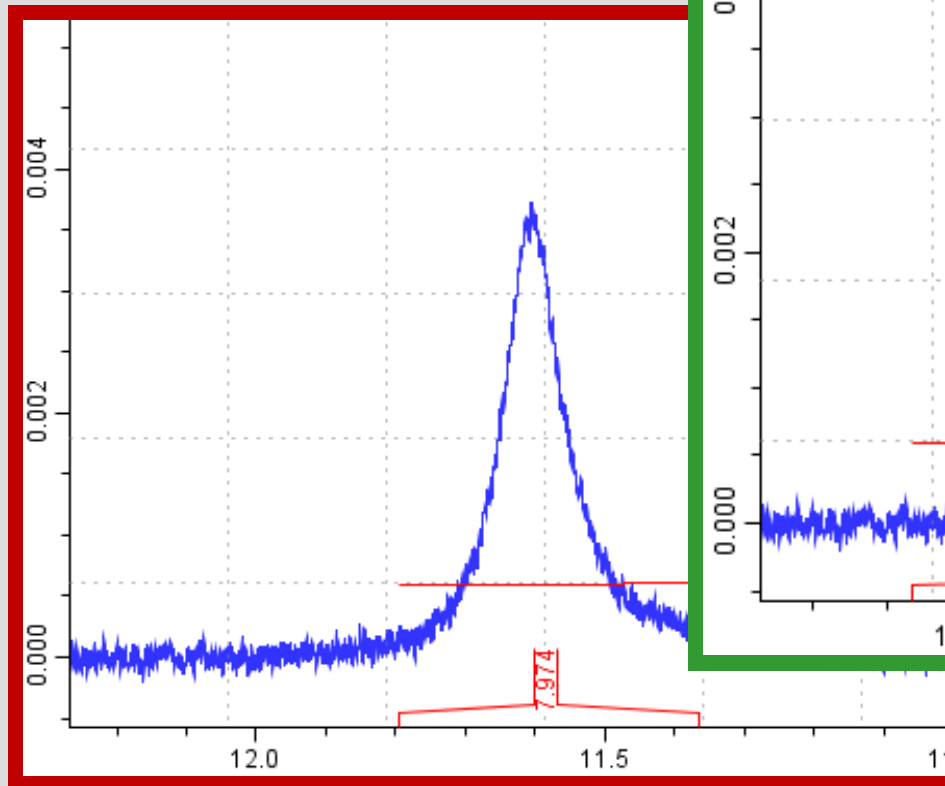


Troubleshooting

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Integration range

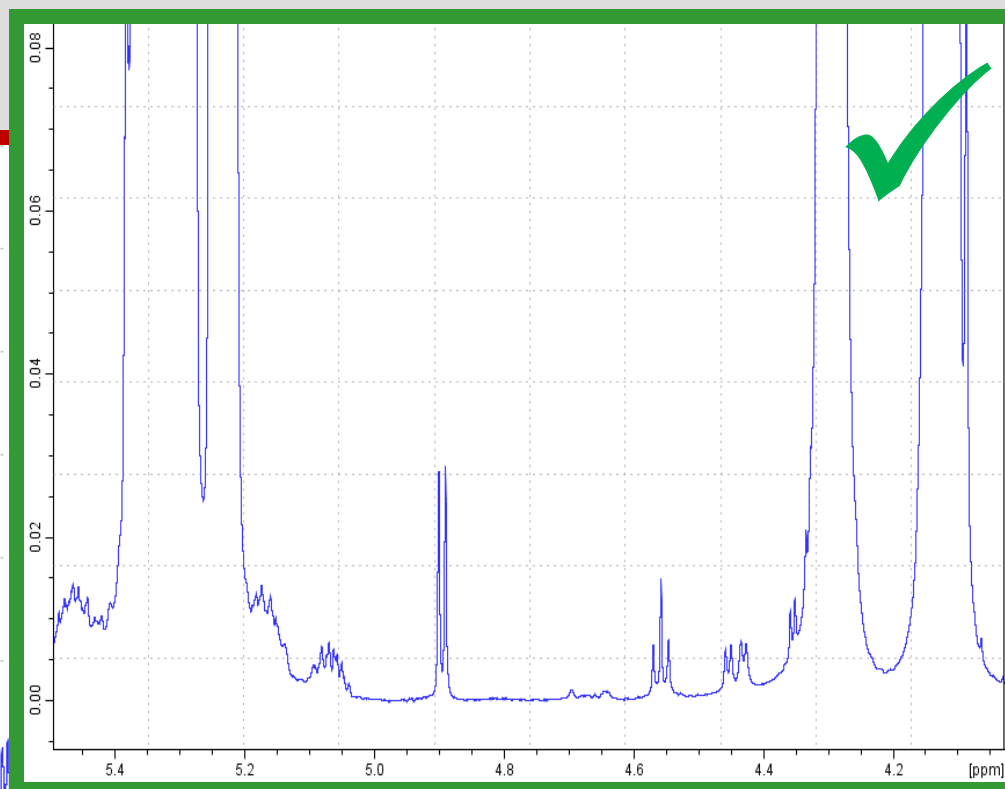
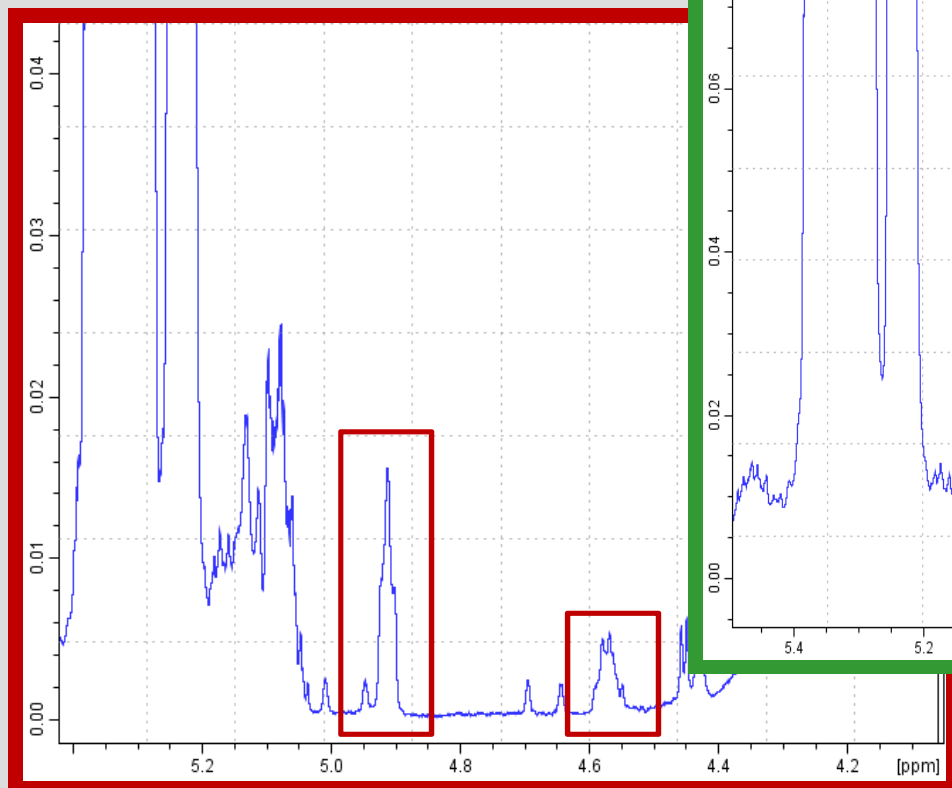


Troubleshooting

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Solvent

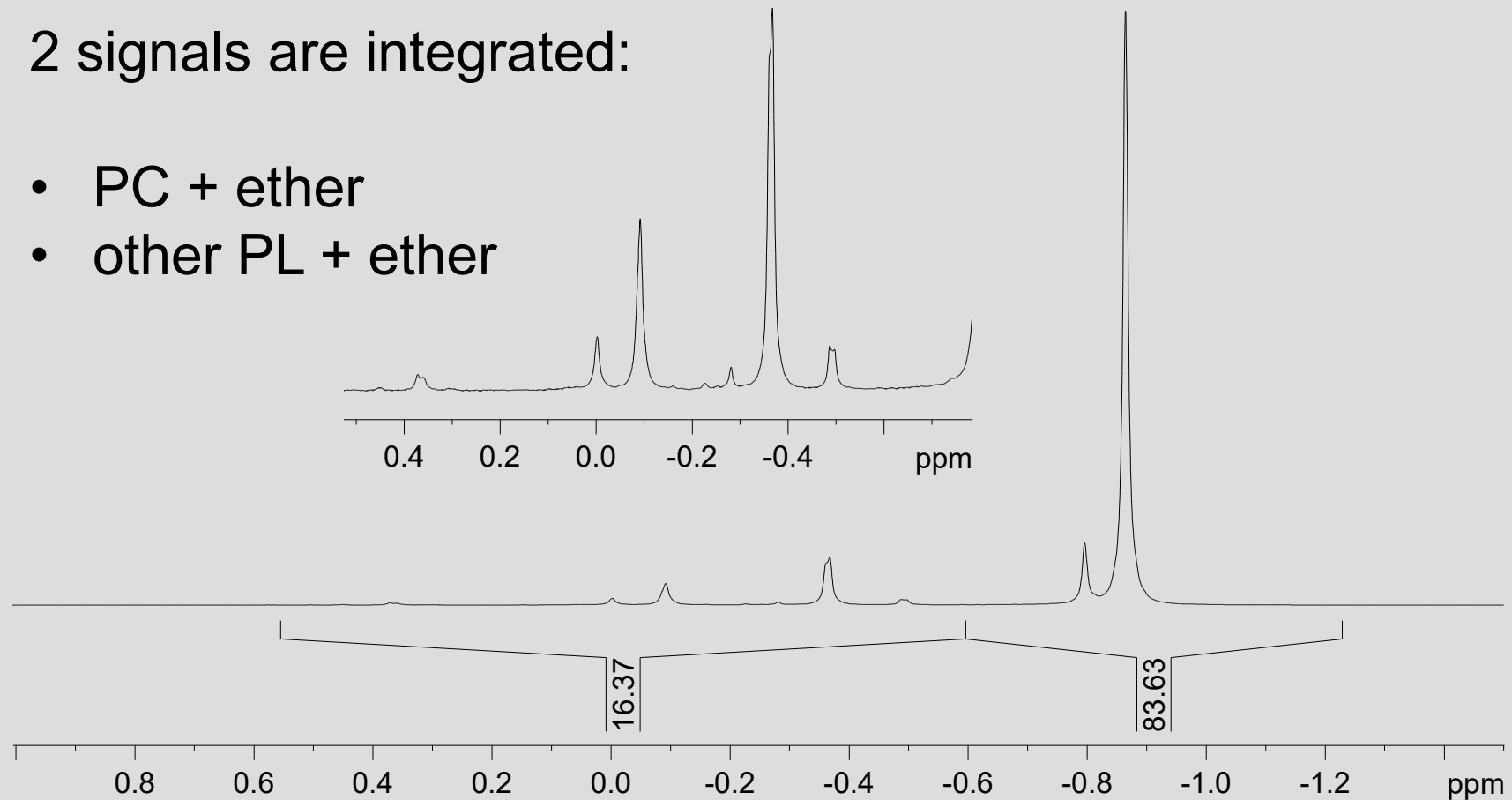




Analysis of Krill Oils

2 signals are integrated:

- PC + ether
- other PL + ether



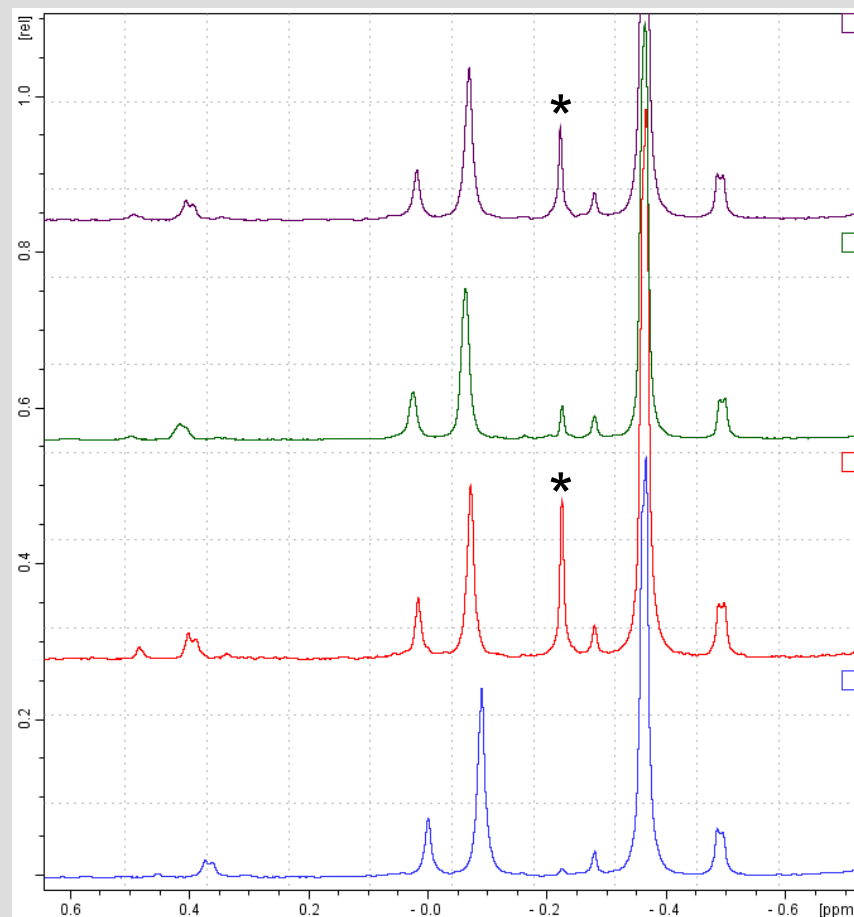
Analysis of Krill Oils

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PC	Mean	SD	SD [%]
Krill 1	94.66	4.27	4.51
Krill 2	116.07	4.23	3.64
Krill 3	92.67	11.47	12.38
Krill 4	52.22	2.78	4.47
Krill 5	99.98	1.65	1.65
Other PL	Mean	SD	SD [%]
Krill 1	241.09	5.44	2.25
Krill 2	280.55	4.98	1.78
Krill 3	378.04	109.45	28.95
Krill 4	194.49	3.86	1.99
Krill 5	146.74	2.28	1.55

Krill 3



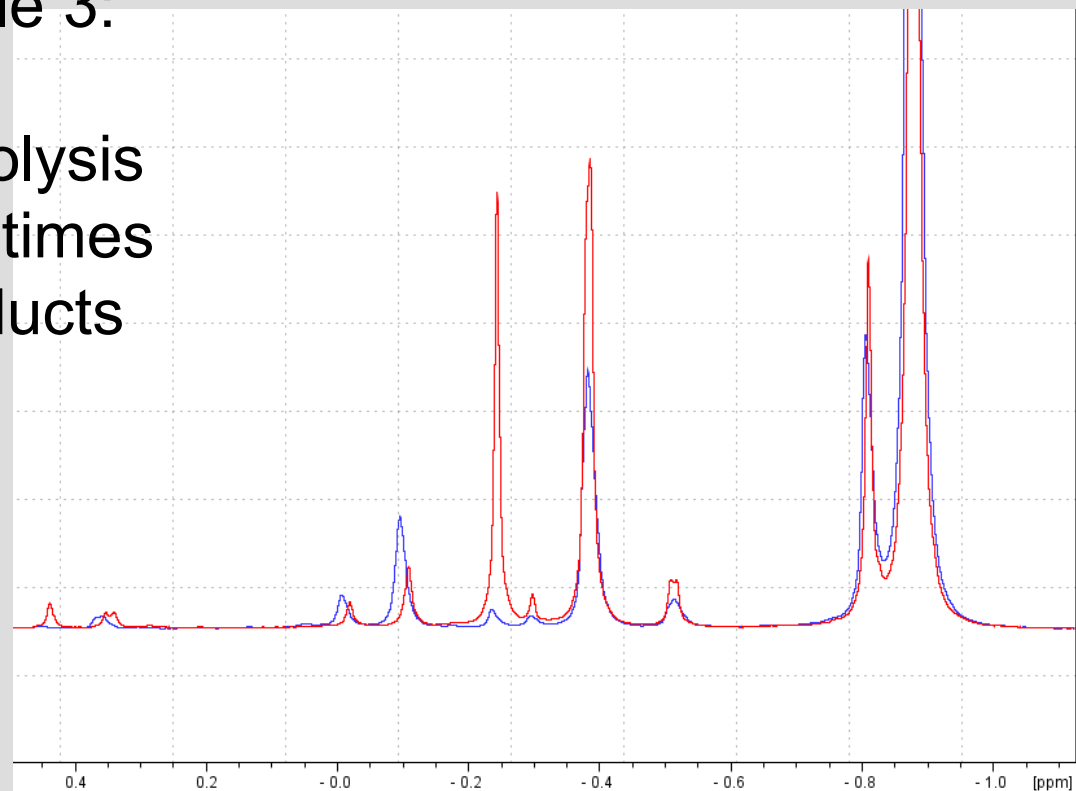
Analysis of Krill Oils

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Problem with sample 3:

- sensitive to hydrolysis
- different storage times
- degradation products



Round Robin Test Findings & Outlook

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Findings:

- Analysis must be performed in a shorter time range
- still great backlog demand for optimal NMR spectrum evaluation

Future evaluations of these round robin tests:

- Comparison with magnetic field strength, measuring date
- Impact by measuring parameters

Future projects:

- Research projects in the area of food and pharmaceuticals
- Organisation of further round robin tests to proof NMR methods
- Increase acceptance of NMR as a powerful tool in quantitative analysis

**Quantitative NMR
Foundation Meeting**

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Thank You for Your Attention!