

Laboratory analytical support in control of use and marketing of plant protection products

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Overview



- Introduction
- Laboratory analysis of PPPs in Austria
- Methods for the analysis of PPPs
- Examples of analysis
- Conclusions

Introduction



Analysis of plant protection products can focus on:

- a) characterisation of a PPP
(and comparison with registration data)
- b) comparison of two or more PPPs
(check for identity, especially parallel import)
- c) finding an undesired contamination
(e.g. presence of additional active substance)

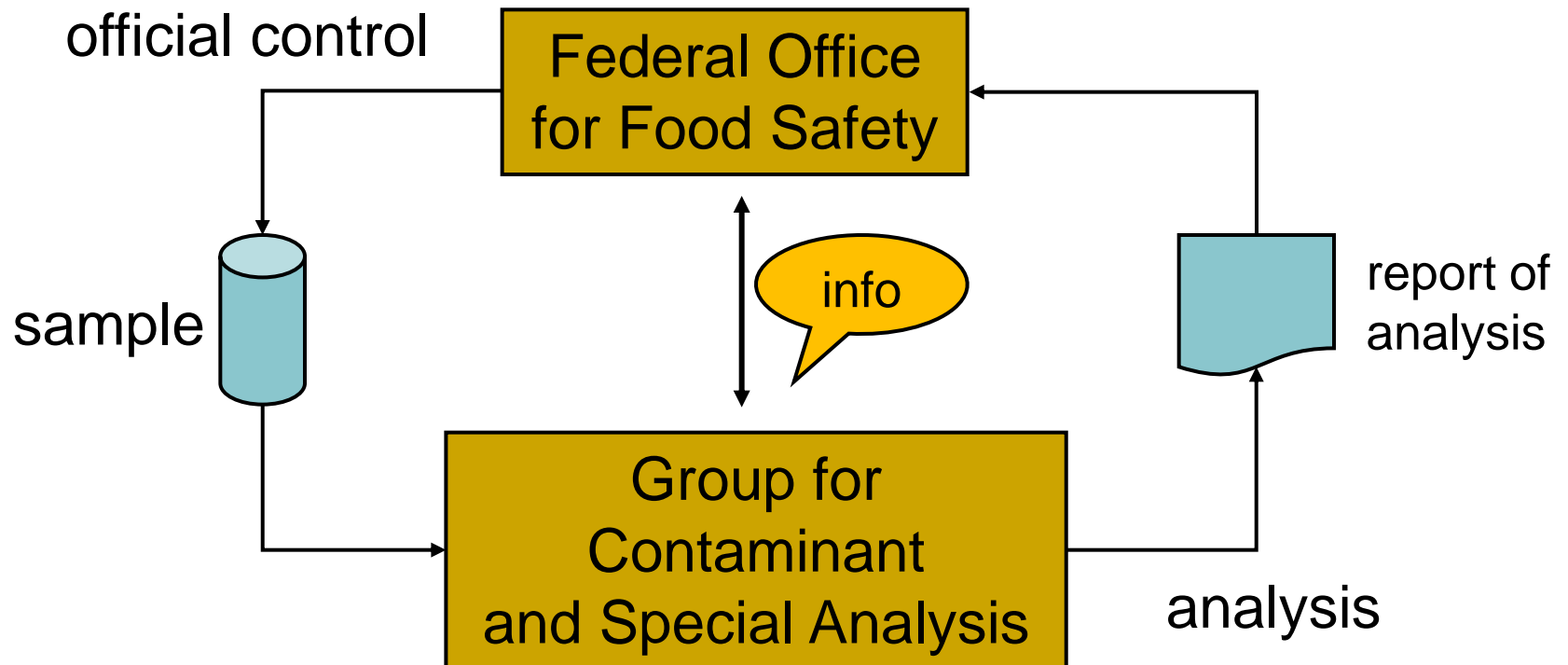
What does checking for identity mean?

- Identity (analytically):
agreement of physical and chemical parameters of two PPPs
(or PPP with reference values)
- (Analytical) non-identity:
as soon as one parameter is not congruent
(considering analytical uncertainty)
- Identity can usually not be proven as this would
involve testing complete composition of PPP
⇒no indication of non-identity

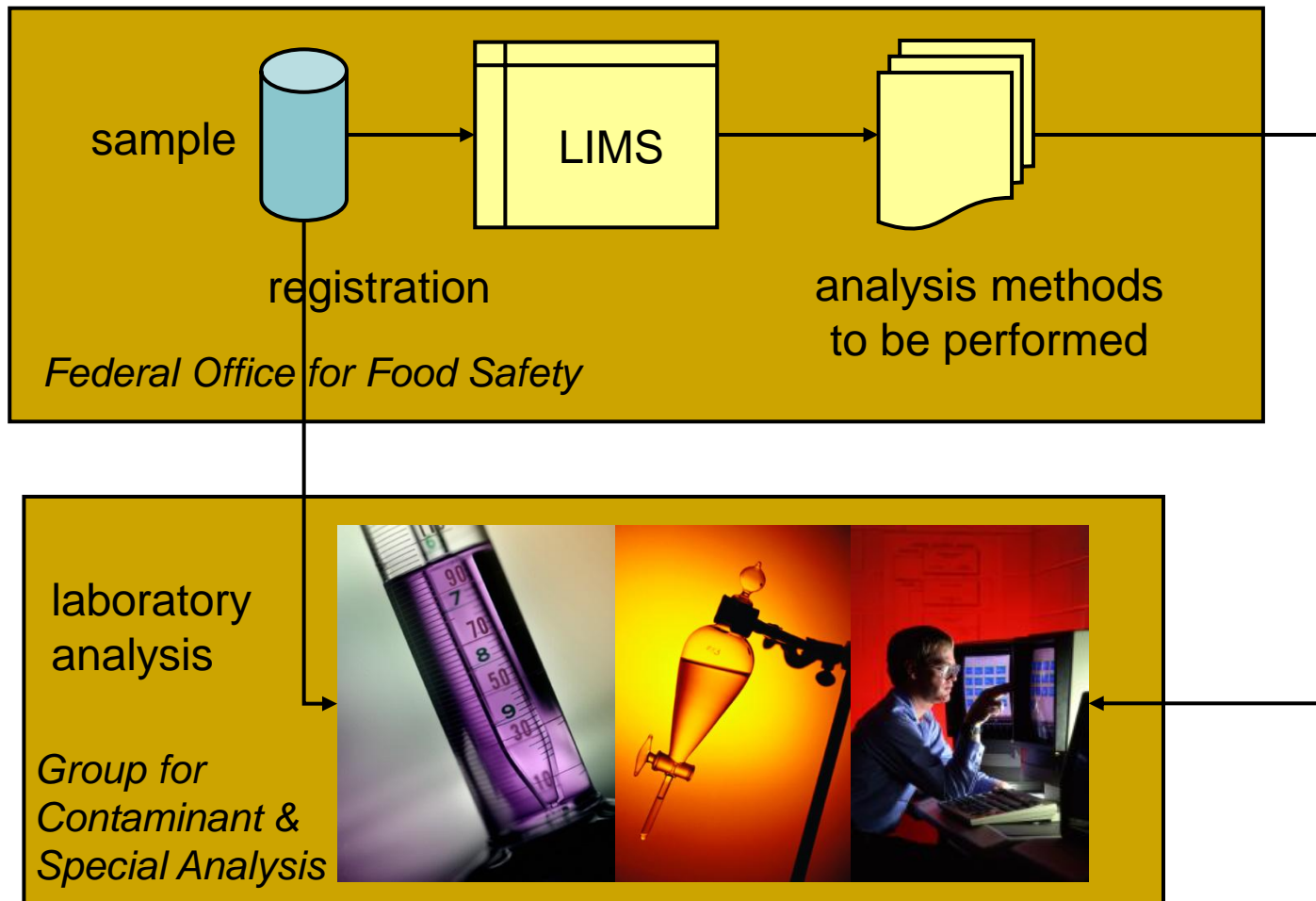
How to go about for finding an undesired contamination?

- Non-selective broad screening approach required
- Combination of gas chromatography and liquid chromatography to cover full polarity range
- Rapid and robust means for identifying peaks required
 - ⇒ coupling with mass spectrometry
 - standard procedure in GC-MS due to availability of large libraries
 - much more tedious and less promising in LC-MS due to lack of libraries

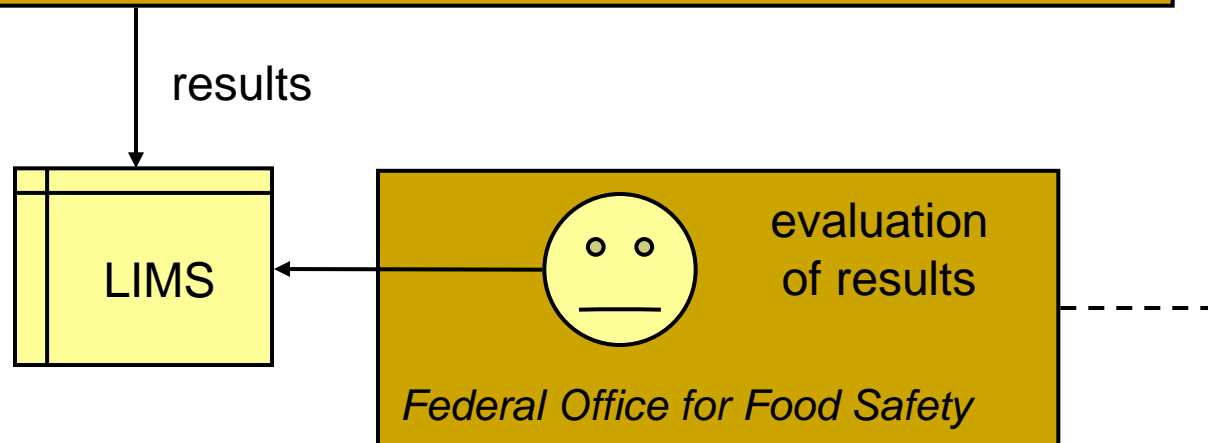
Laboratory analysis in Austria



Laboratory analysis in Austria



Laboratory analysis in Austria



Methods of analysis



Two general types of analysis:

- **Targeted analysis:**
investigation of a specific parameter/compound
- **Screening analysis:**
broad/overall investigation of many/all compounds,
“fingerprinting” or “unknown screening”

Methods of analysis



Targeted analysis:

- physical parameters: e.g. density, viscosity
- chemical parameters: identity and concentration of active ingredient or additive, pH-value

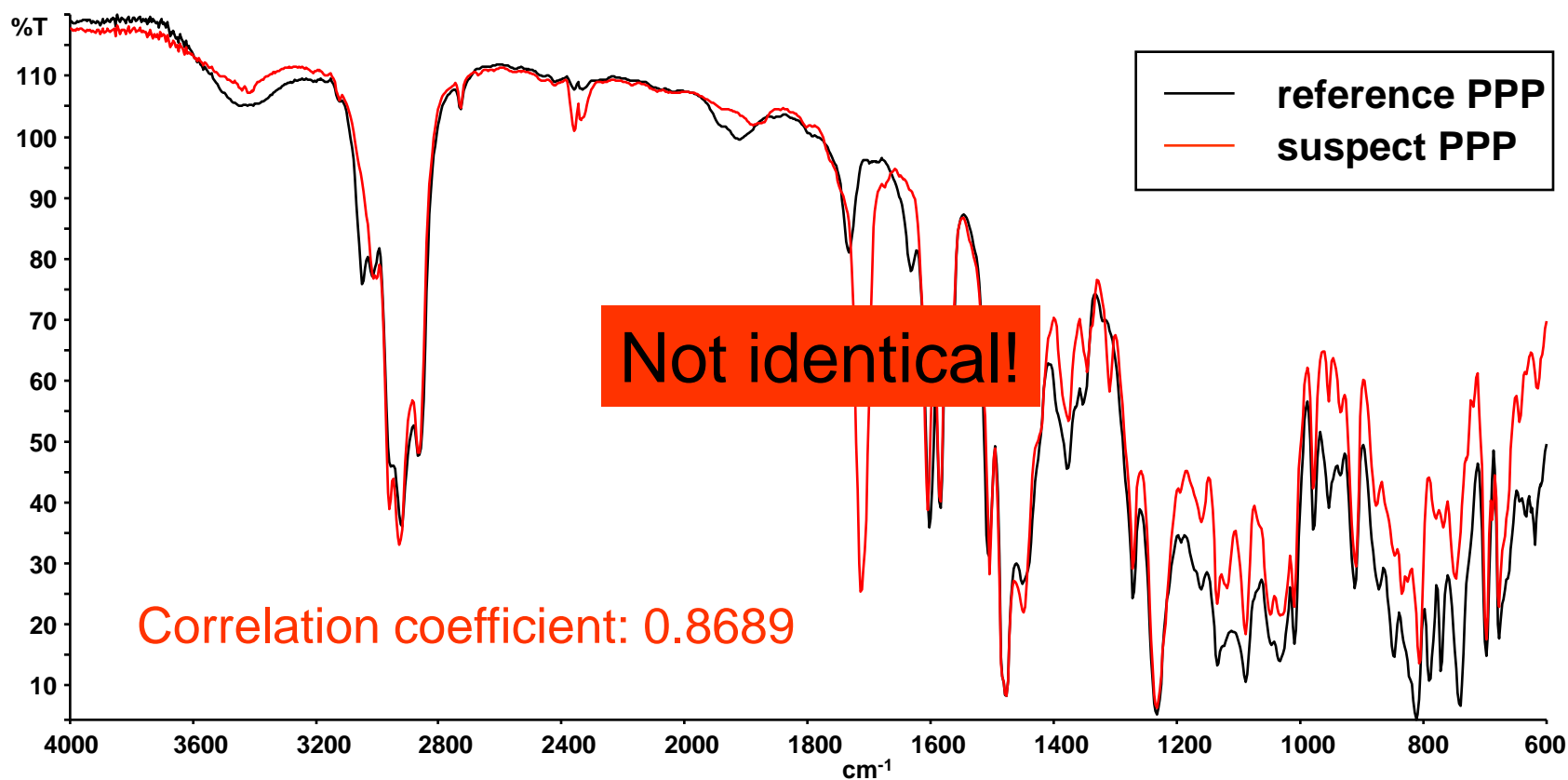
Screening analysis:

- spectroscopy (FTIR)
- separation technique: GC-MS, LC-UV, LC-MS

Examples of analysis

Screening by FTIR spectroscopy

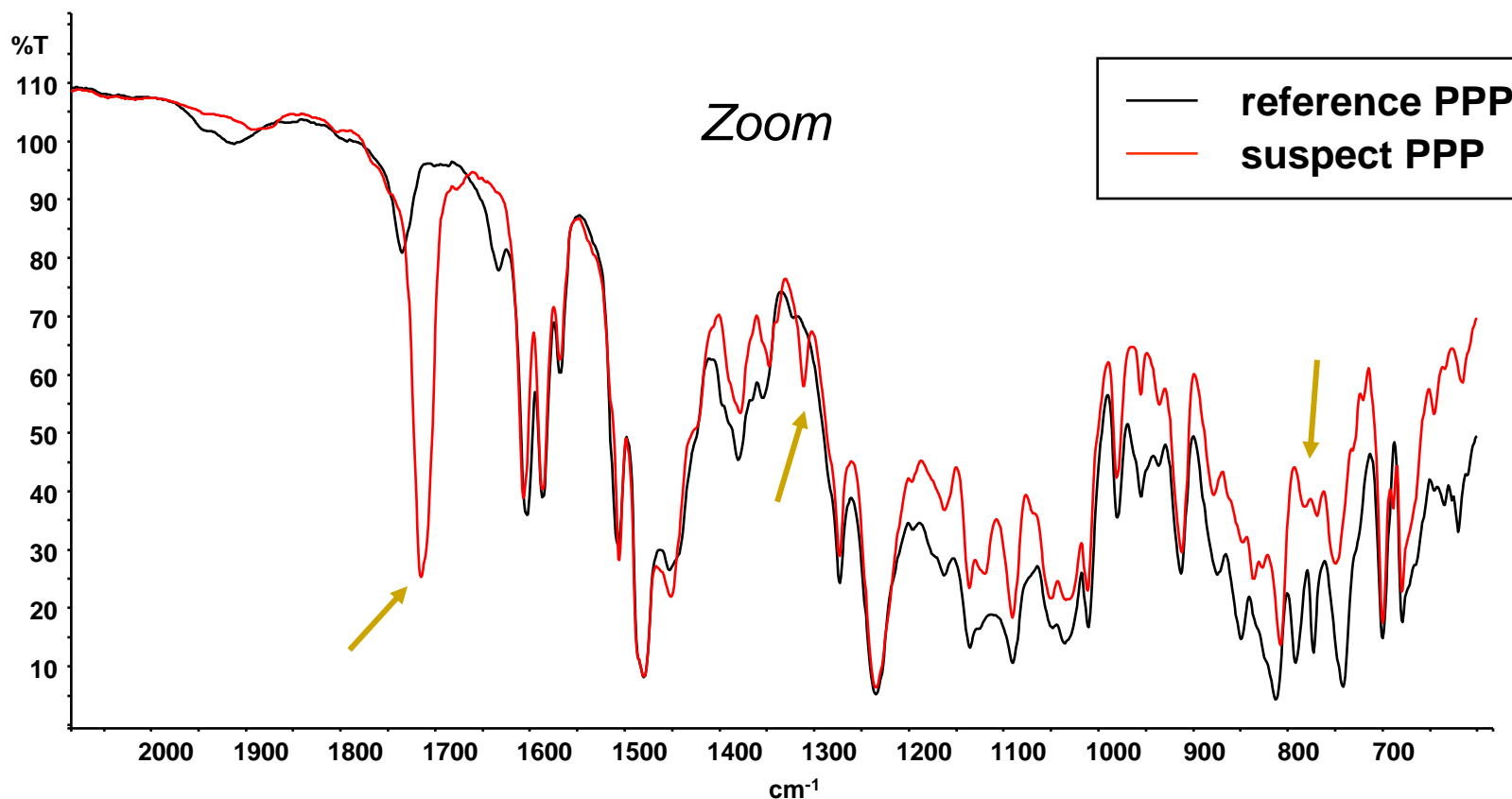
Case 1



Examples of analysis

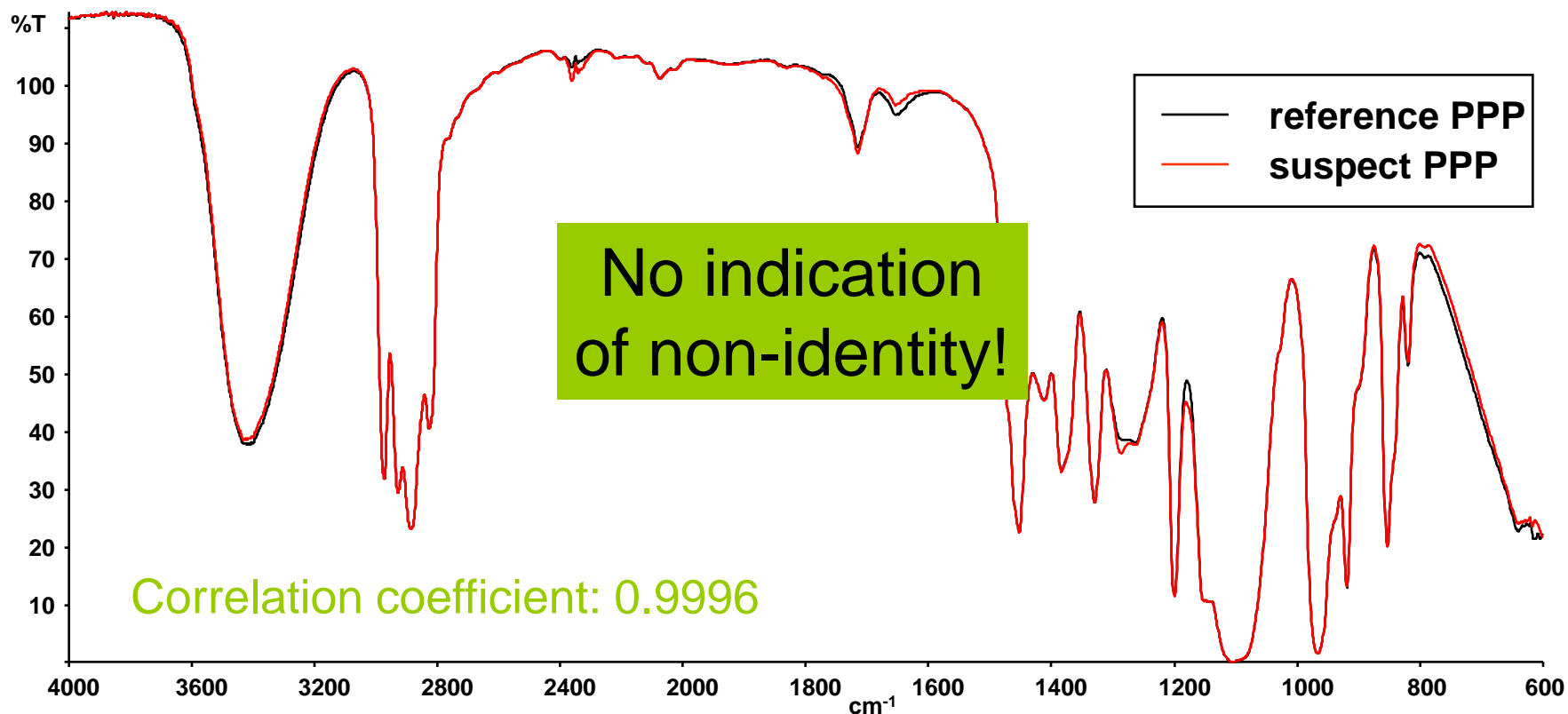
Screening by FTIR spectroscopy

Case 1



Examples of analysis

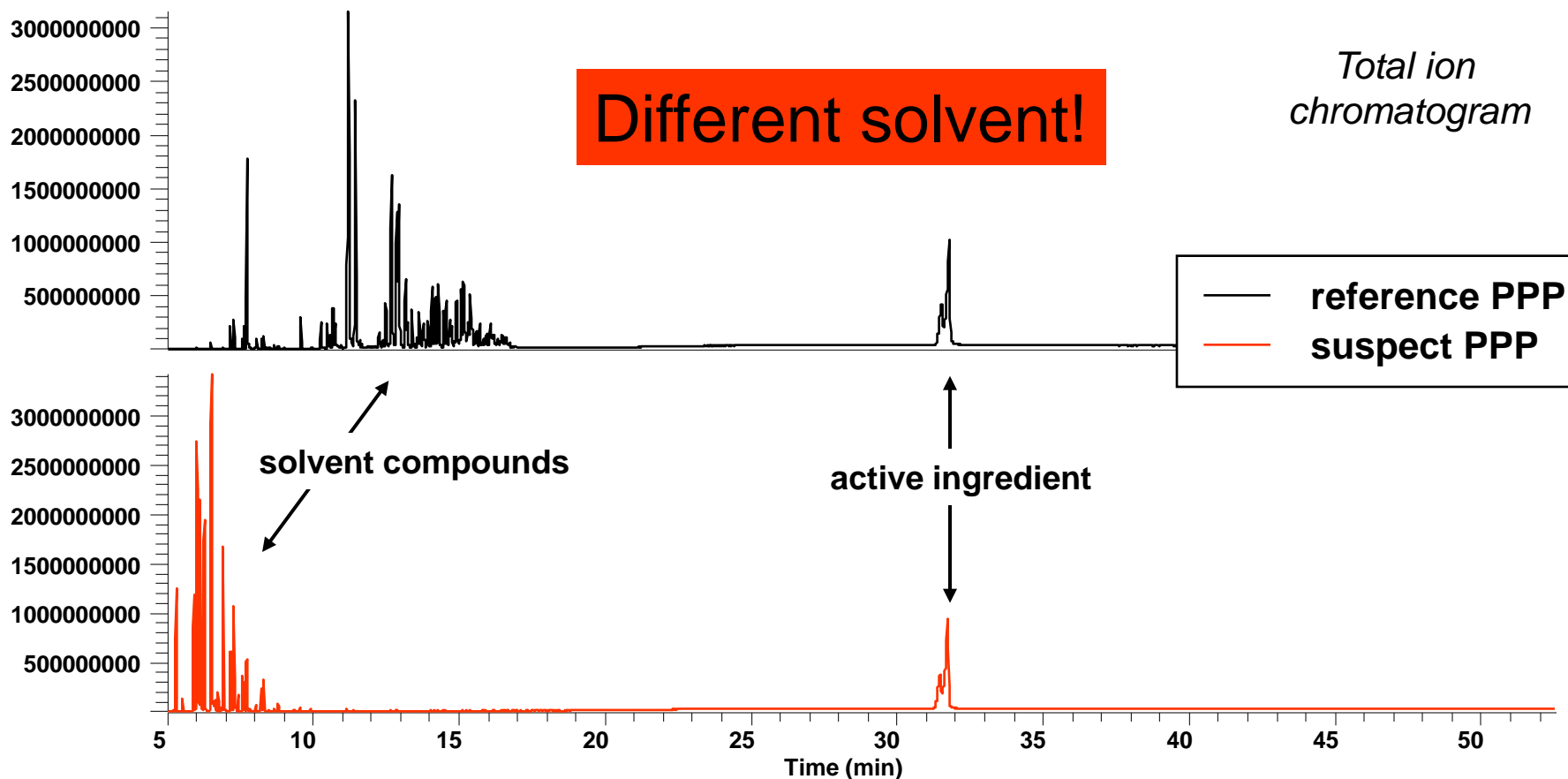
Screening by FTIR spectroscopy



Examples of analysis

Screening by GC-MS

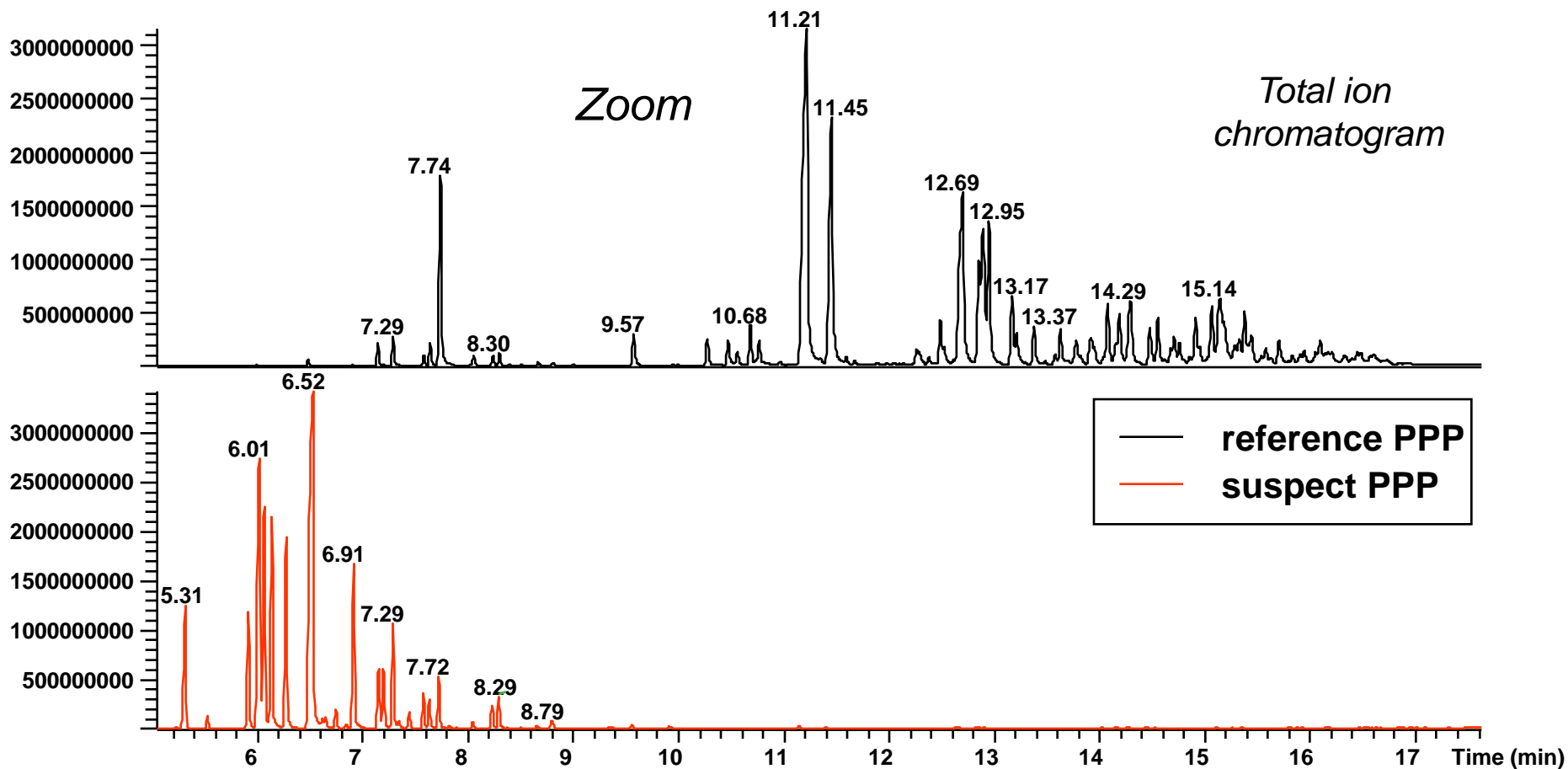
Case 1



Examples of analysis

Screening by GC-MS

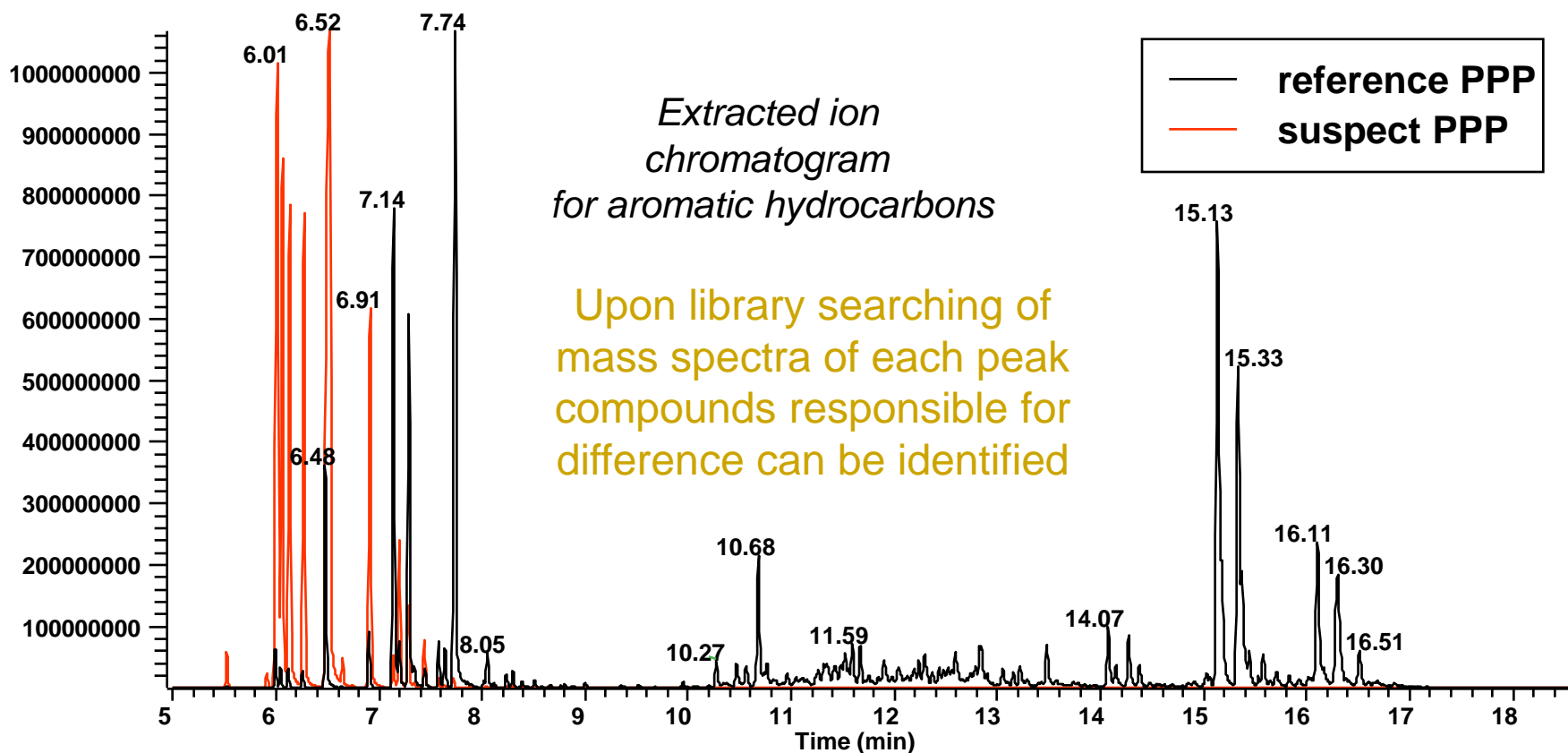
Case 1



Examples of analysis

Screening by GC-MS

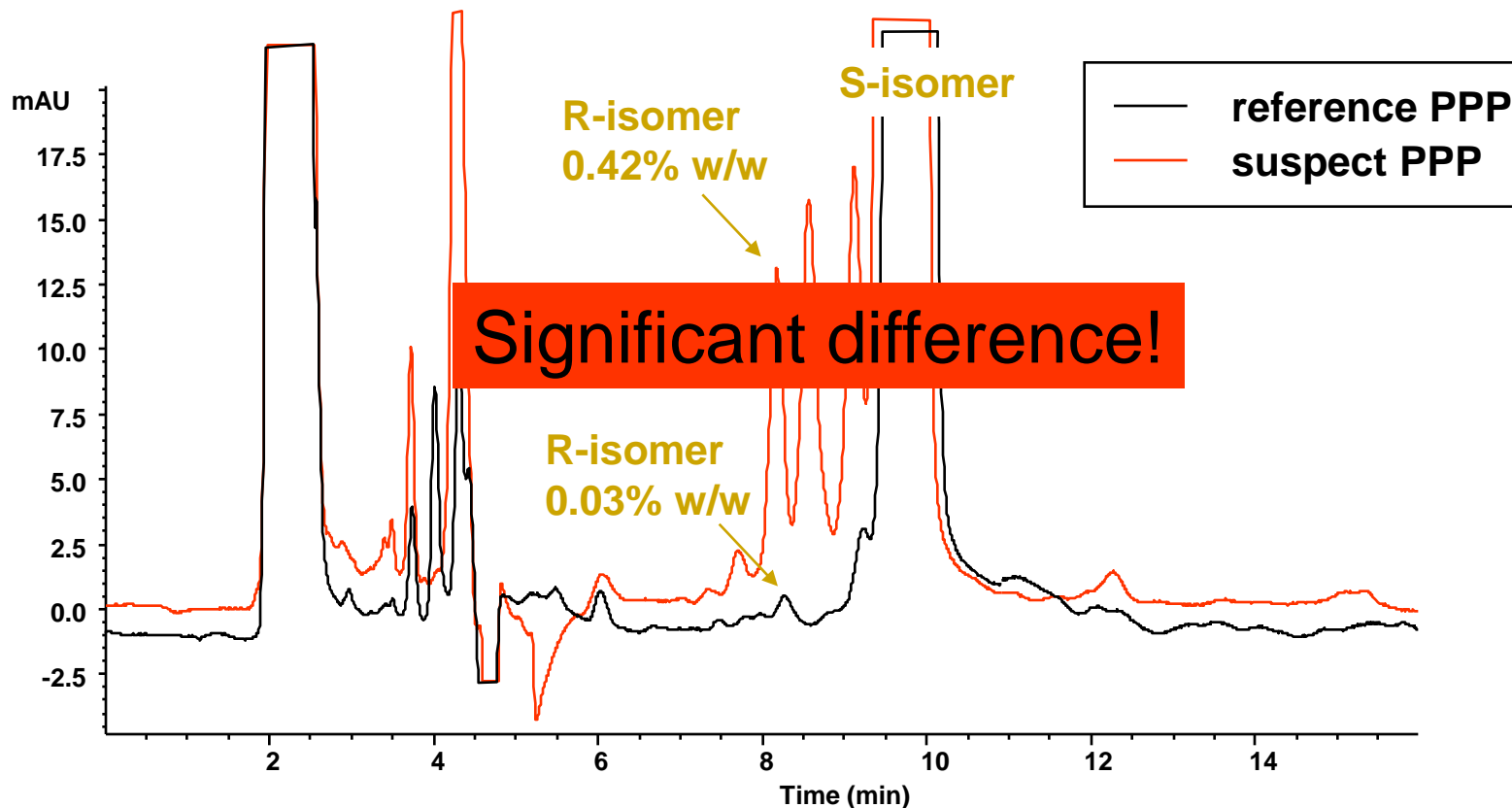
Case 1



Examples of analysis

Analysis of active ingredient

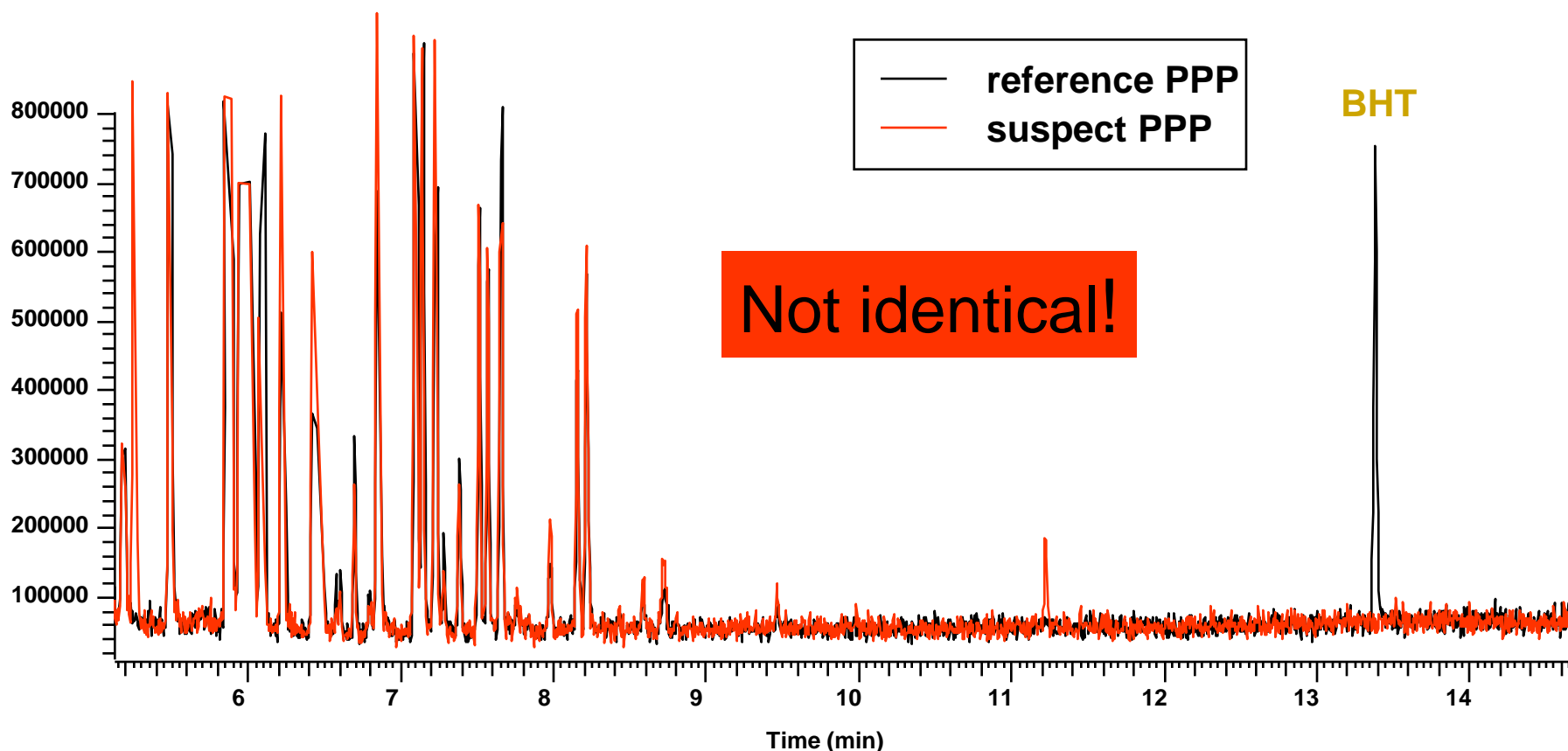
Case 2



Examples of analysis

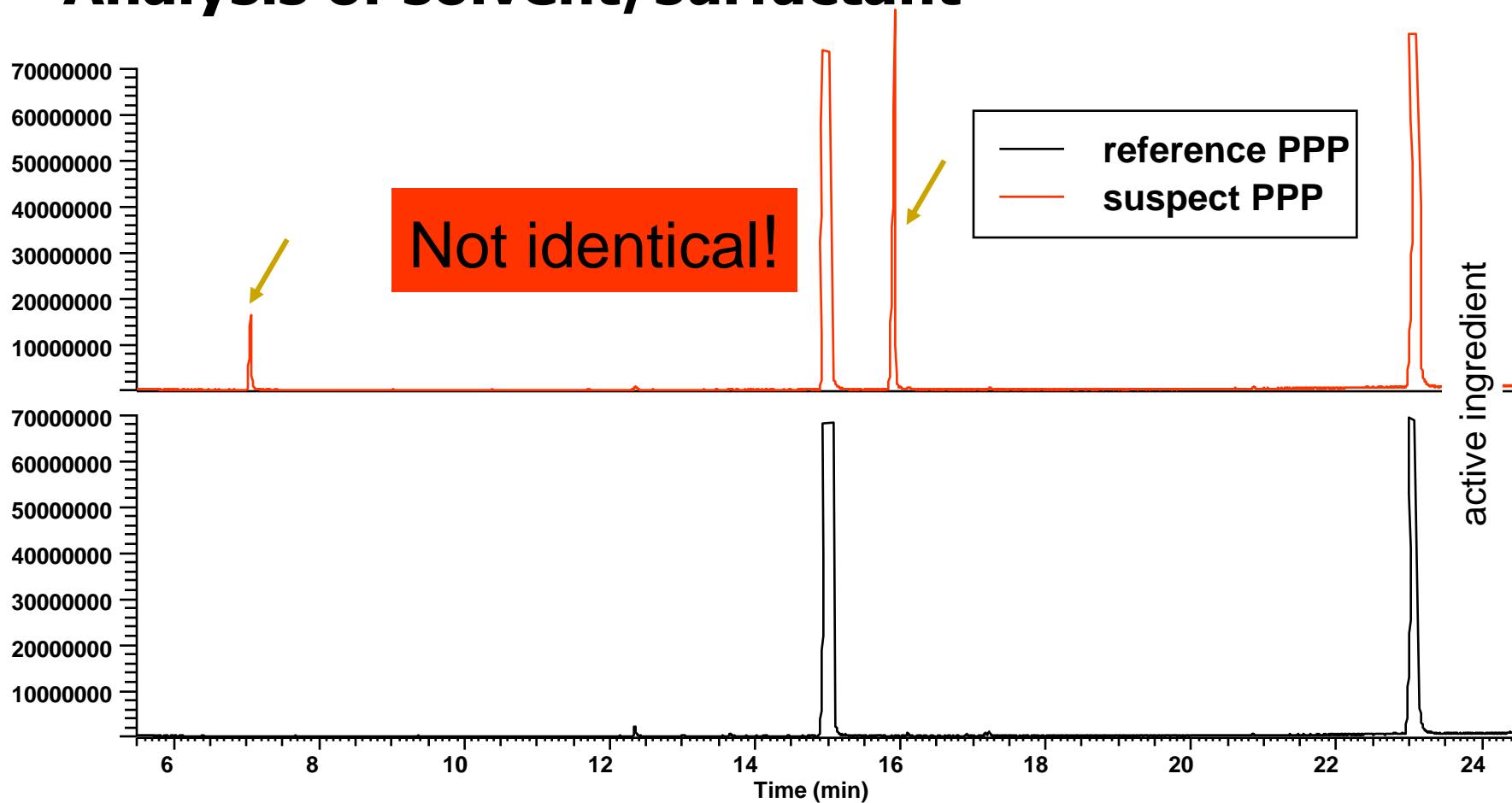
Analysis of preservative

Case 2



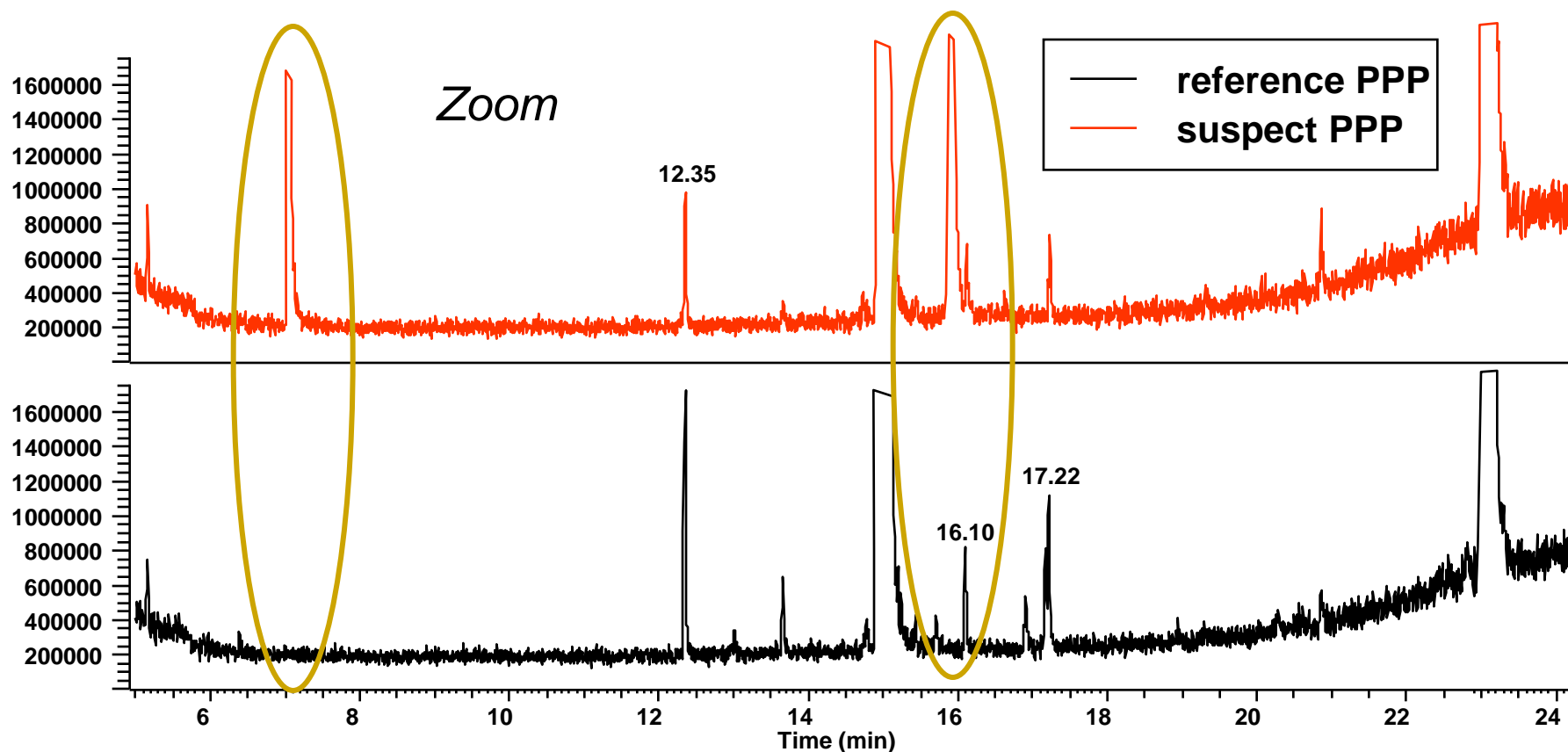
Examples of analysis

Analysis of solvent/surfactant



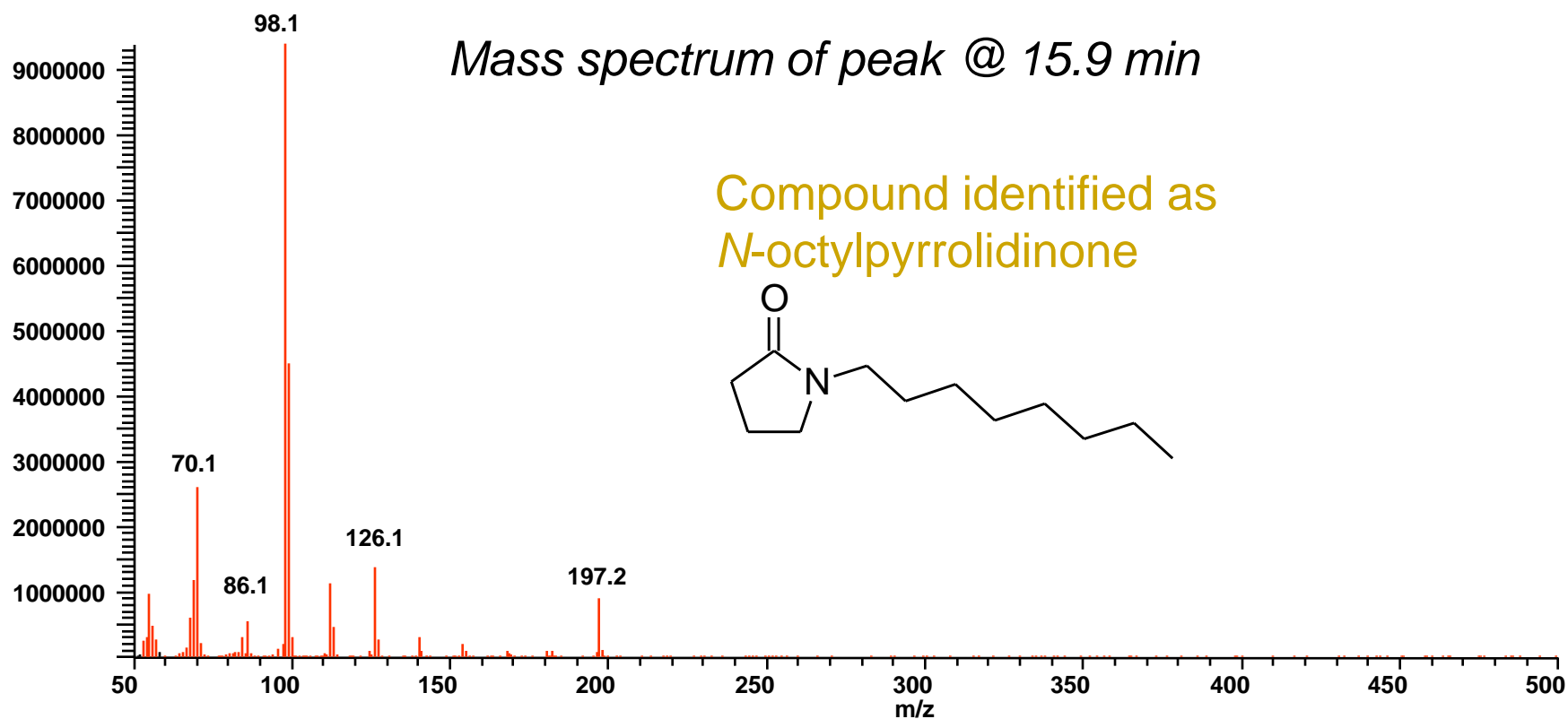
Examples of analysis

Analysis of solvent/surfactant



Examples of analysis

Analysis of solvent/surfactant



Examples of analysis

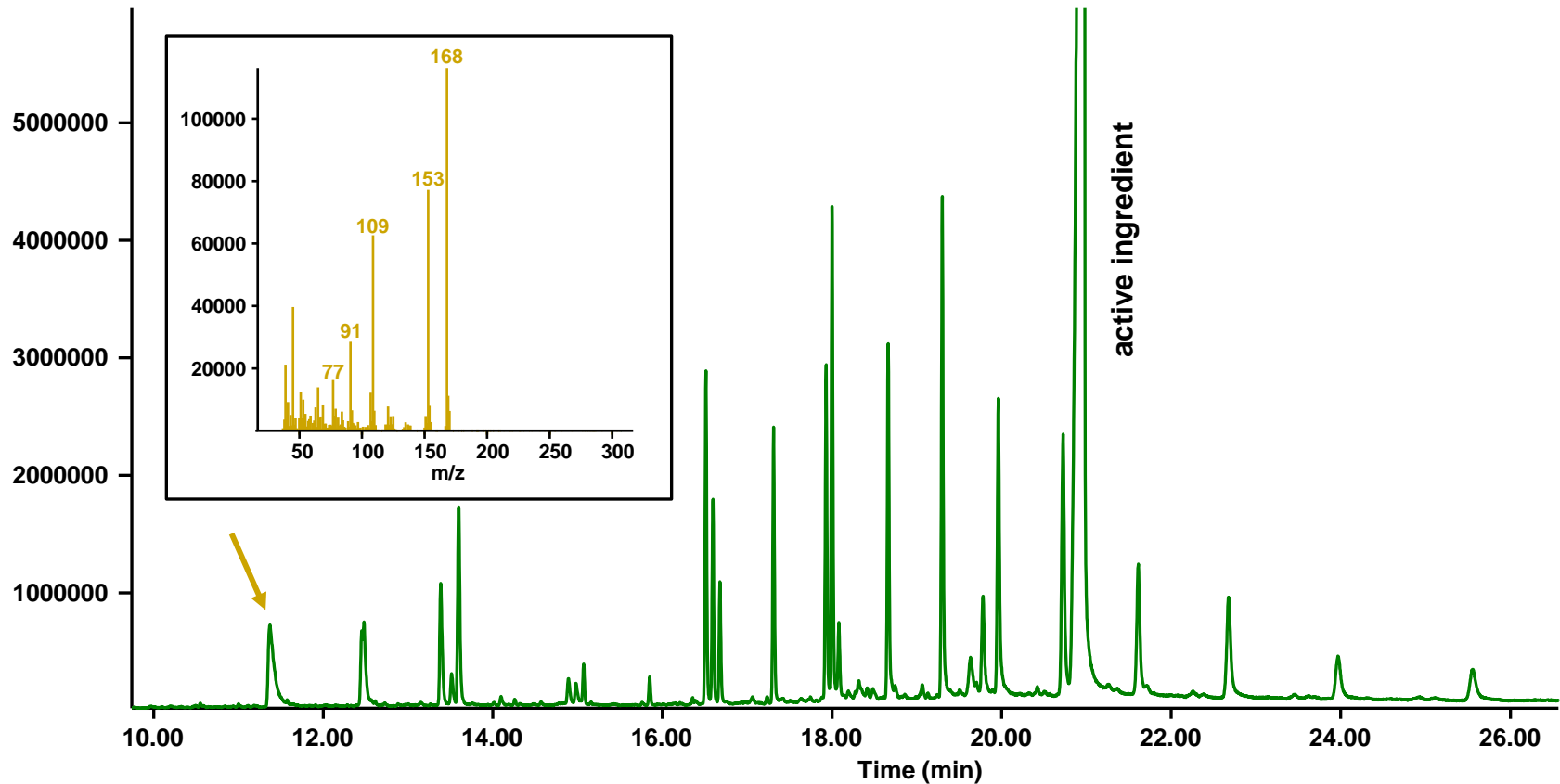


Analysis of solvent/surfactant

- Signal @ 7.1 min identified as N-Methylpyrrolidinone, a solvent
- This compound should not be used due to toxicity issues
- Non-identity already observed by FTIR spectroscopy (correlation coefficient 0.9757)

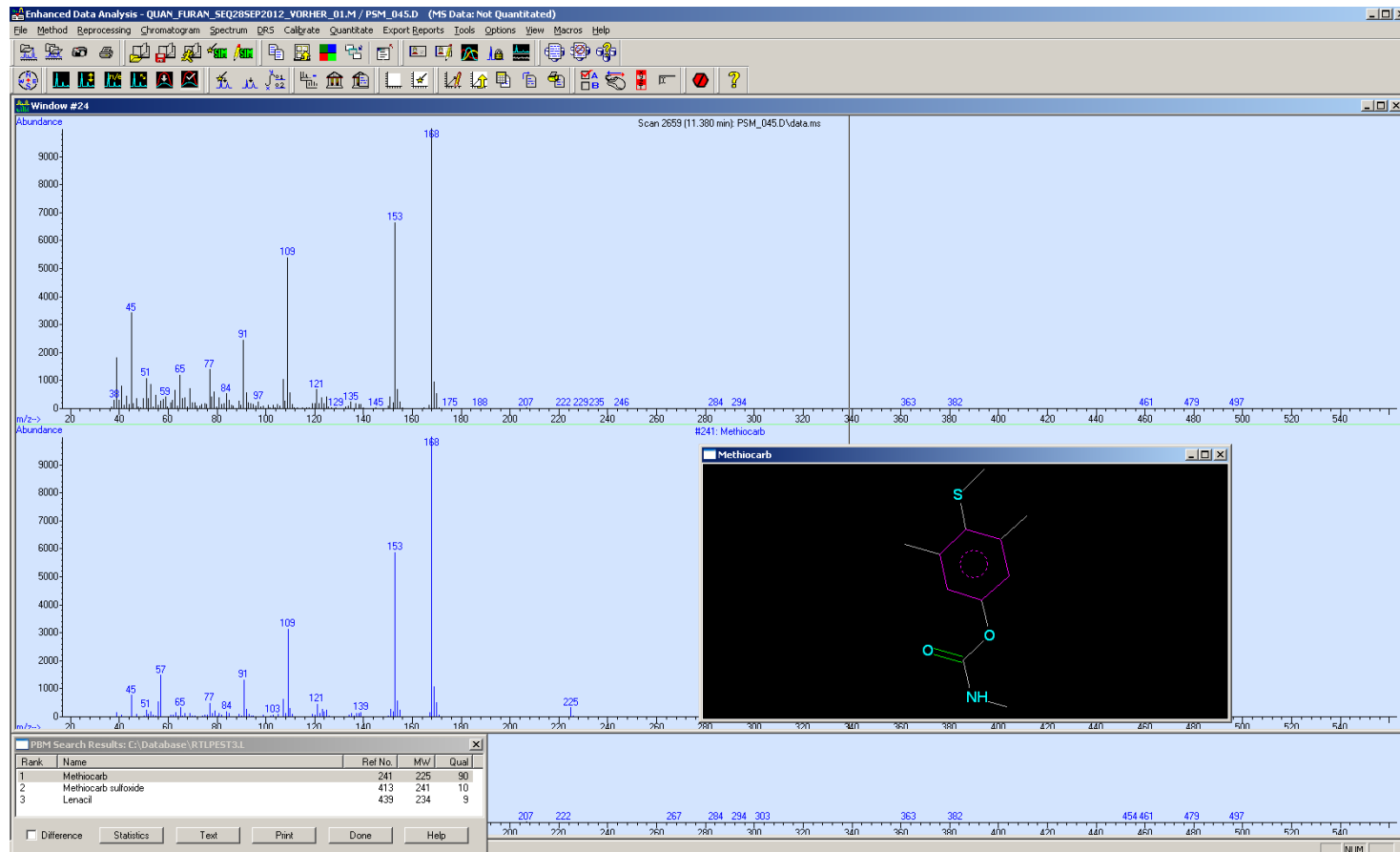
Examples of analysis

Analysis for undesired contaminations



Examples of analysis

Analysis for undesired contaminations



Analysis for undesired contaminations

- Signal @ 11.5 min identified as Methiocarb by GC-MS
- Also confirmed by LC-MS/MS
- Quantification yielded a concentration of 440 mg/l formulation (0.04% w/v)
- This corresponds to 0.4% relative to the active ingredient

Conclusions



- From our experience concentration of active ingredient is mostly not a issue, however impurities or isomeric composition may be one
- Additives often differ from reference
- Mass spectrometry very useful for identification of differences in solvent composition, surfactants, preservatives, etc. as well as contaminations

**Thank you
for your attention!**