METHODS FOR IDENTIFICATION OF KNOWN AND UNKNOWN FOOD CONTACT MATERIALS BY MEANS OF HPLC-HIGH RESOLUTION-ACCURATE MASS SPECTROMETRY

Laszlo Hollosi and Michal Godula

Food Safety Response Center - Thermo Fisher Scientific, Dreieich, Germany

INTRODUCTION

Packaging materials (PM) and non-intentionally added substances (NIAS) are currently one of the hottest research topics in food safety relevant applications. Current research especially focuses on identification of non-expected or up-to-now unknown compounds.

Recent poster aims to present such approaches supported by High-Resolution MS (HRMS) based LC-MS methods for identification of known authorized or unauthorized substances. We demonstrate the power of HRMS and TraceFinder 3.1 software with known and unknown compounds.

ANALYTICAL METHODOLOGY

1) Establishment of database (DB) with High-Resolution m/z data

2) Performing analytical measurement
- direct analysis by FIA-HRMS
- LC-HRMS (after extraction)

3) Data mining, evaluation

INSTRUMENTATION

Thermo Fisher Scientific Exactive ™ Orbitrap™ high-resolution benchtop mass spectrometer coupled to Transcend TLX-1 system.
Column: Hypersil Gold C8 HPLC, 100 x 2.1 mm, 3 µm. Mobile phase: A: MeOH, B: water.
Gradient run: 0-1 min: 50%A, 1-6 min ramp to 100%A and hold for 4 min. Flow: 0.4 ml/min.

HR-MS settings:
R=100,000; Ionisation mode: ESI and APCI both in positive/negative; Mass range: 60 -600 m/z; Maximum injection time: 500ms; Fragmentation method: HCD, Sheath gas: 40; AUX gas: 5; Vaporiser temperature: 350°C; Capillary temperature: 300°C.

RESULTS

Targeted screening for known and unknown compounds

2,5-bis(5-tert-buthyl-2-benzoxaolyl)thiophene and 2-hydroxy-benzophenone were used as known model compound for testing and four individual unknown compounds were received from co-operation partner for demonstration of identification process. Thermo Fisher Scientific TraceFinder 3.1 software was used for evaluation.
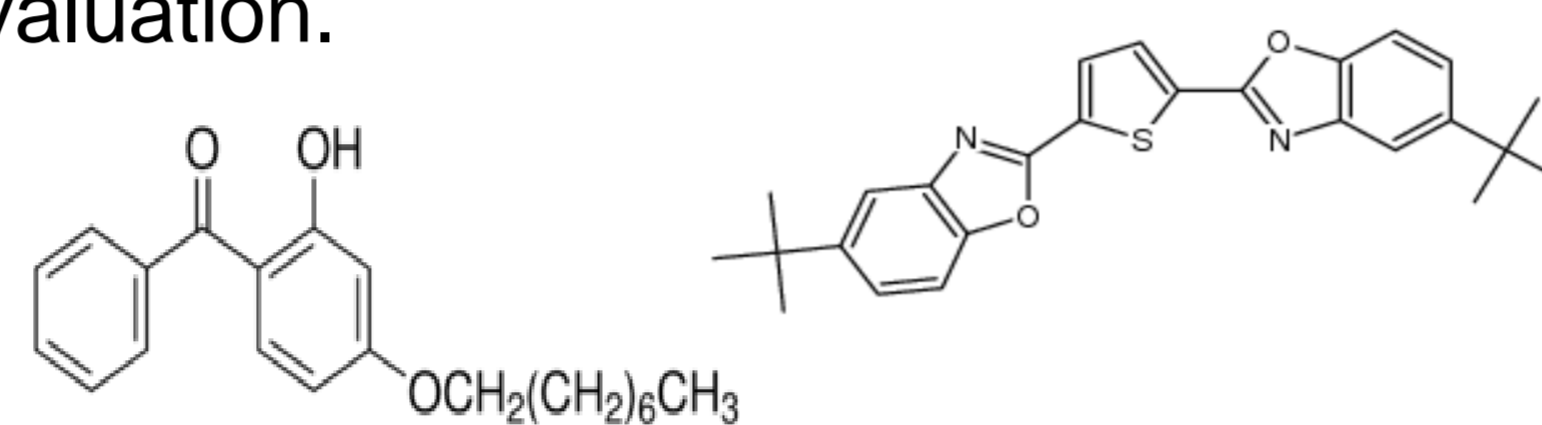
Database establishment

Accurate mass database was established for 600 potential target compounds. HRMS precursor and fragment masses were identified for known compounds in LC-HRMS experiments.
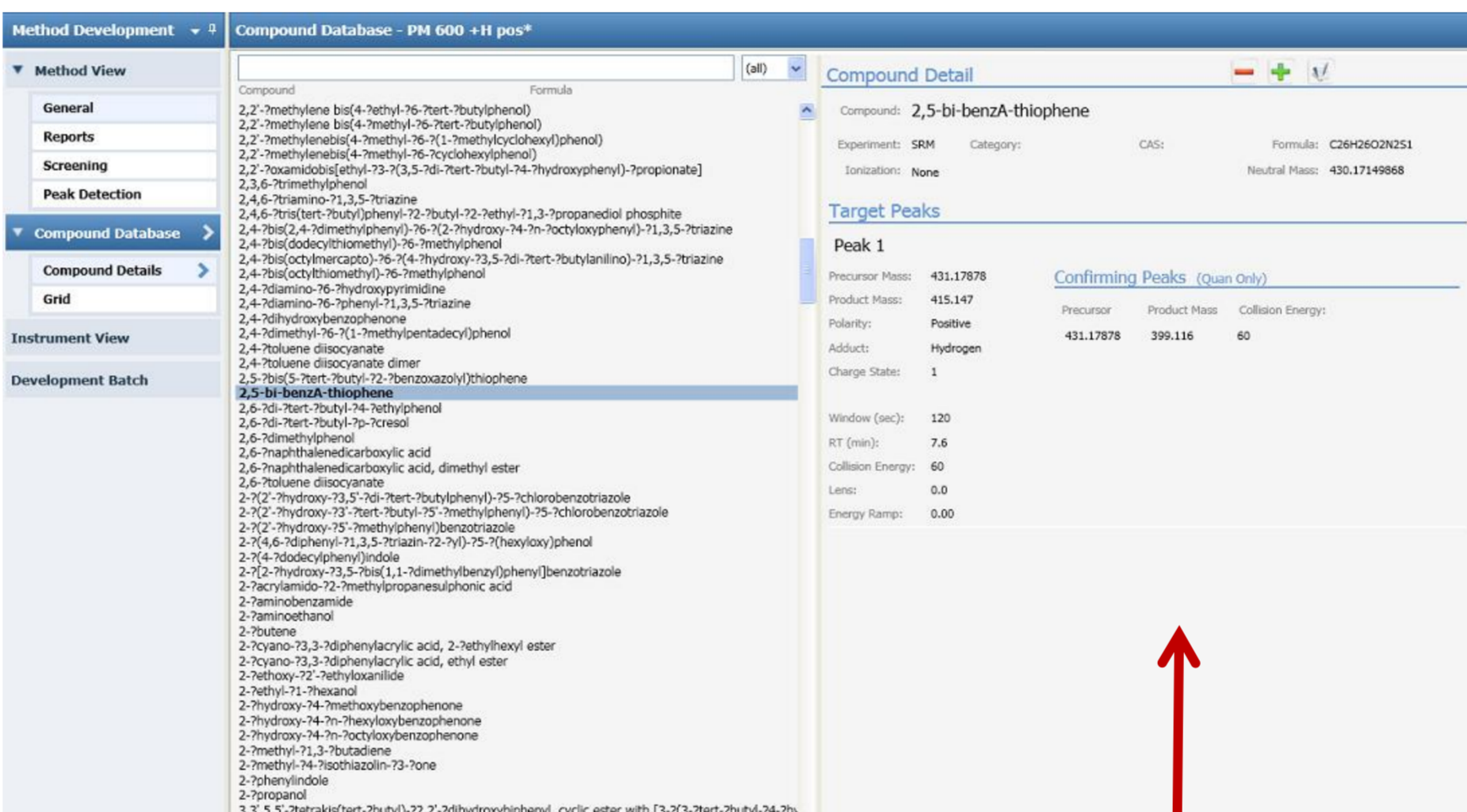

Figure 1. TraceFinder 3.1 database compound definition page with precursor and fragment masses.
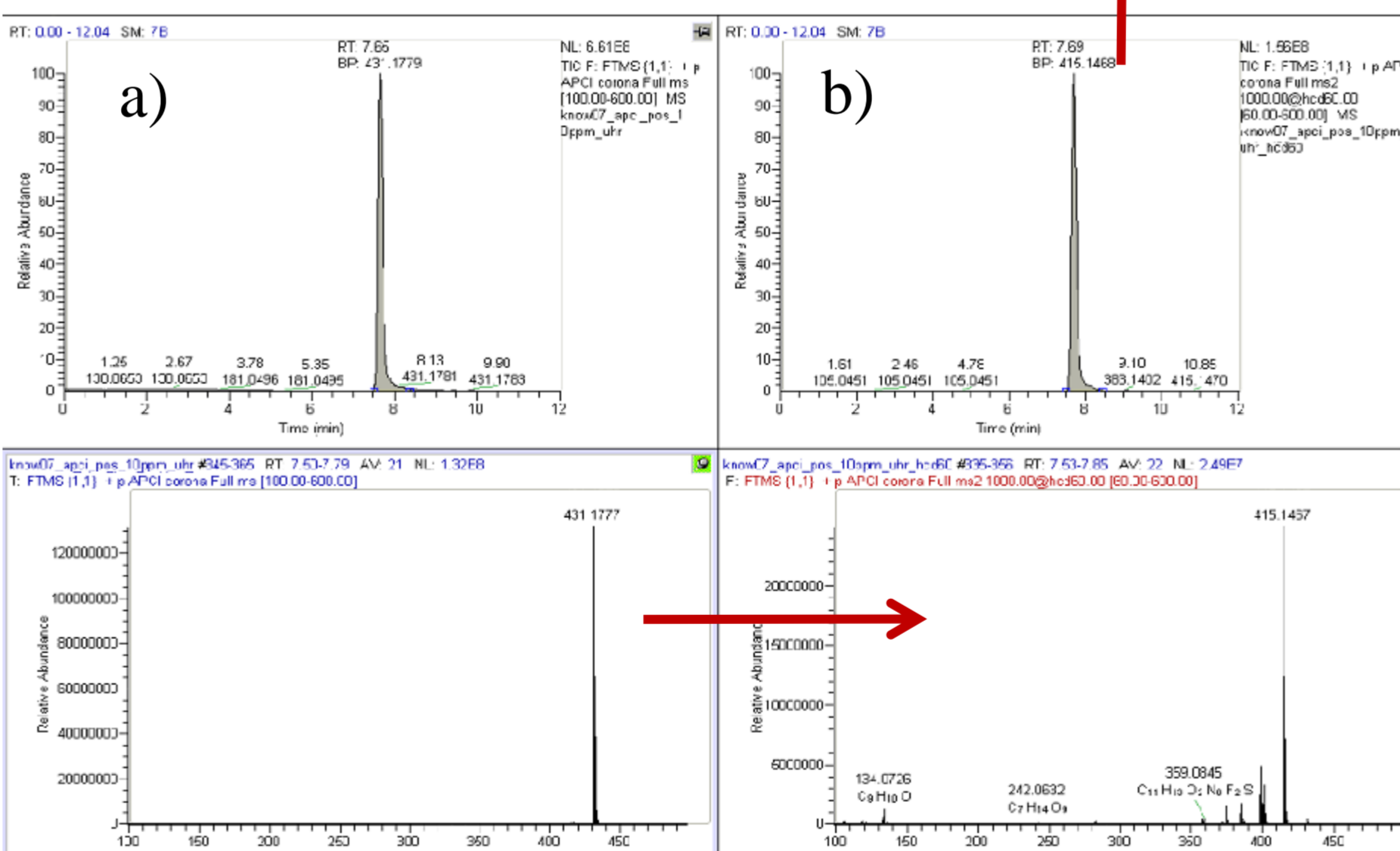

Figure 2. Fragmentation profile for 2,5-bis(5-tert-buthyl-2-benzoxaolyl)thiophene with a, Ecoll=0 kV and b) Ecoll=60 kV.

Identification of known compounds

Identification was conducted both by TraceFinder 3.1 quantitative and screening methods. Fragment ions were used for confirmation.
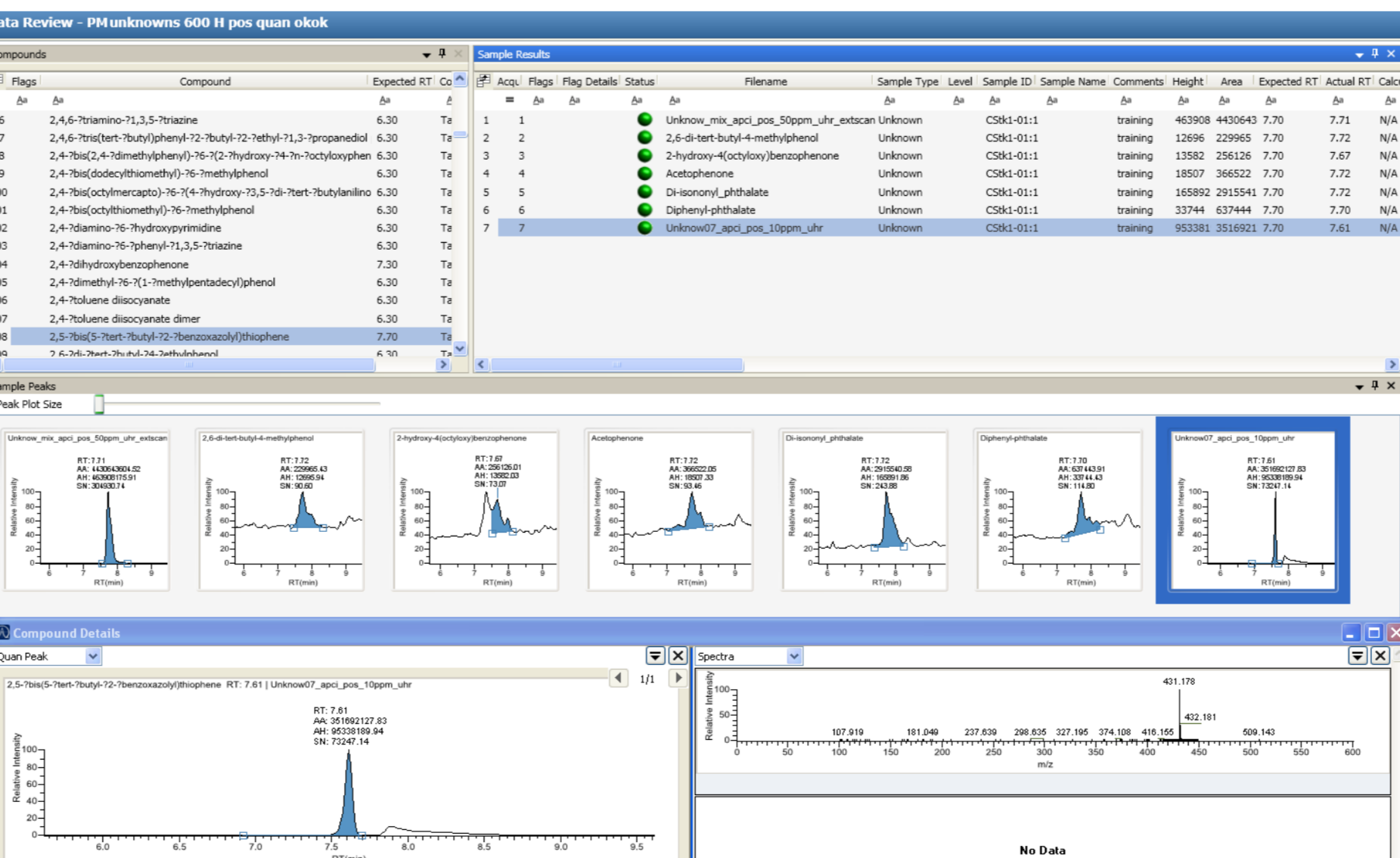

Figure 3. Identification of 2,5-bis(5-tert-buthyl-2-benzoxaolyl)thiophene in TraceFinder result page for quantitative methods.

Identification of unknown compounds

Identification was conducted both by TraceFinder 3.1 screening and quantitative methods based on the established database search. Extracted ion chromatograms (XIC) were identified and compared to FIA observed or online available spectral fingerprints. Spectra were recorded in both ESI and APCI ionisation mode.
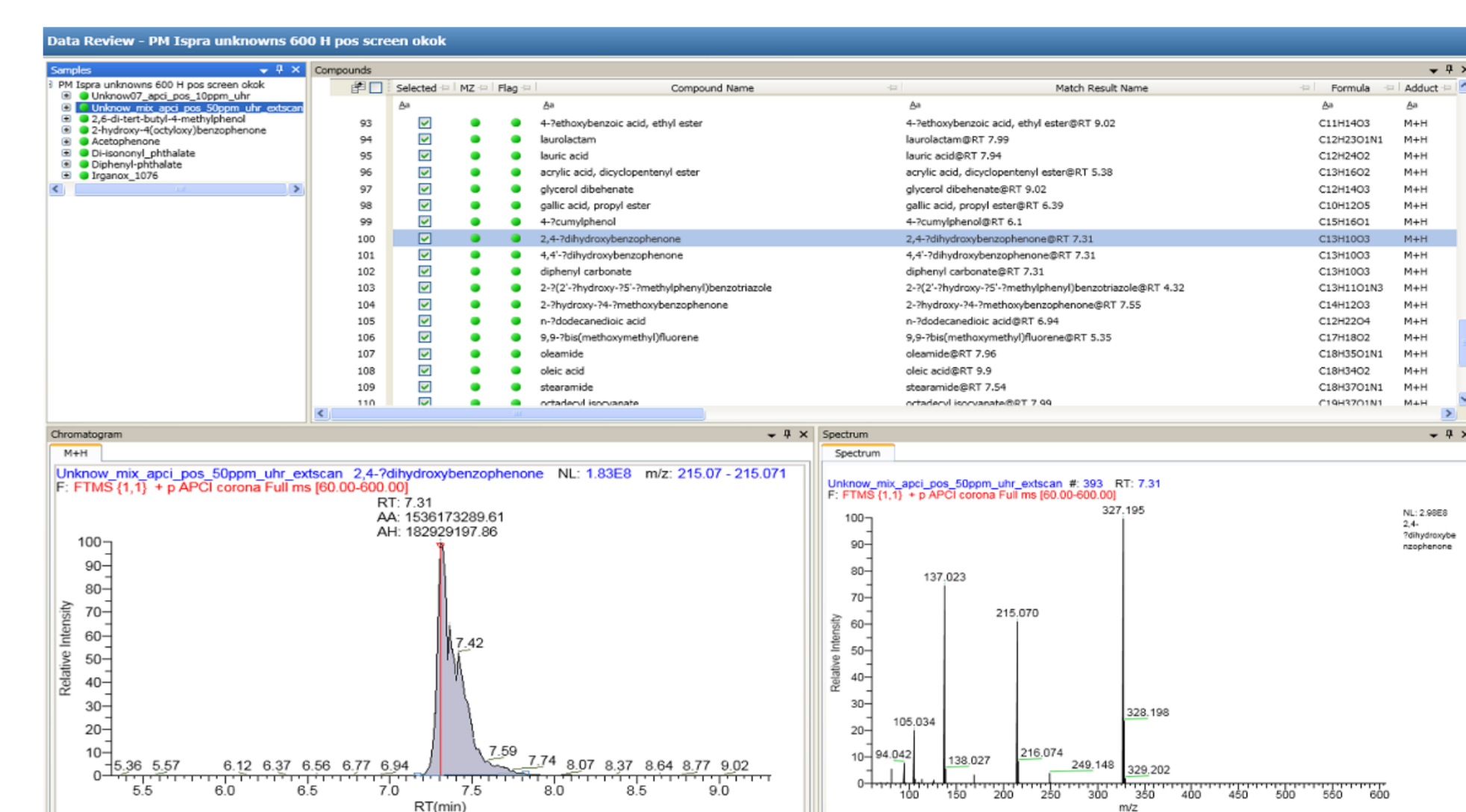

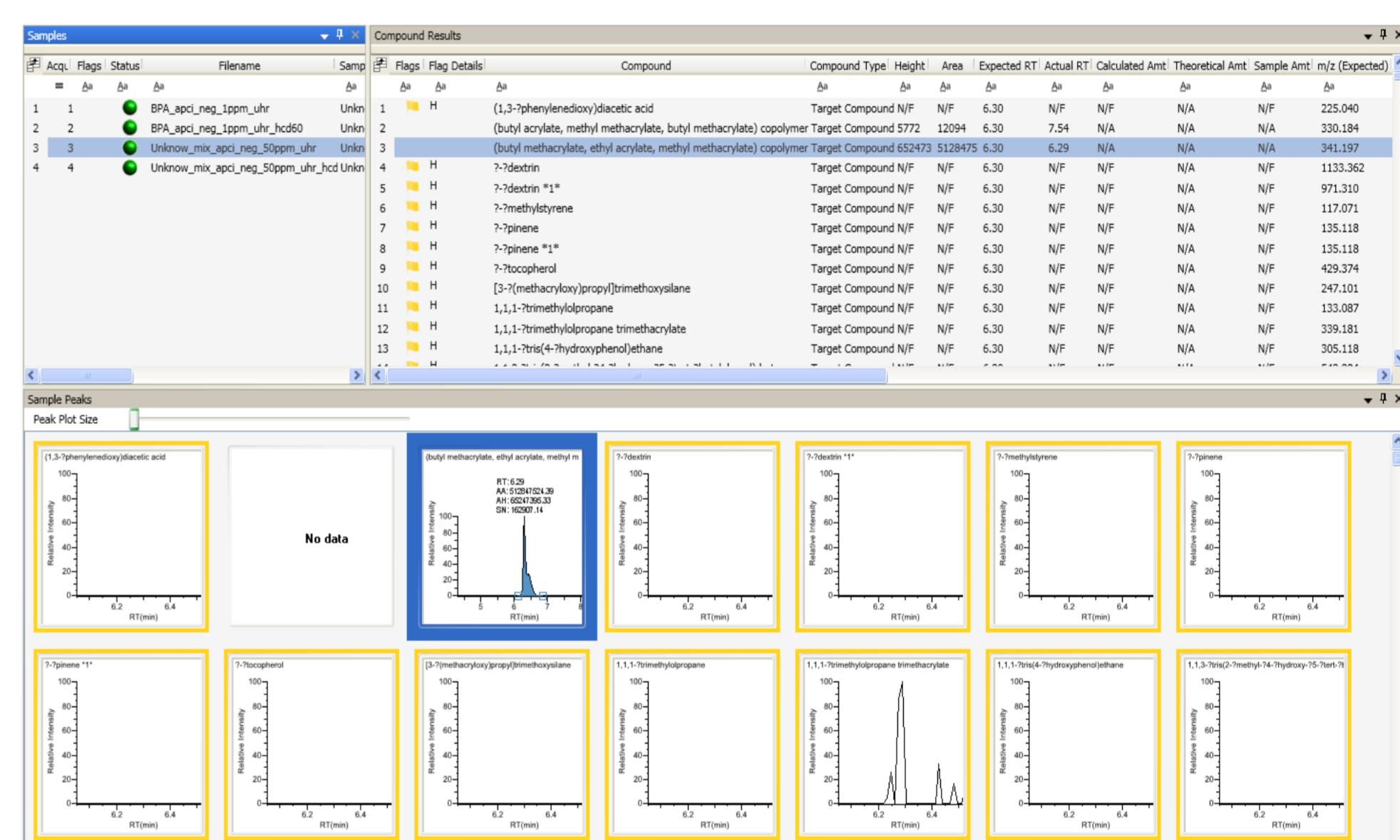**Figure 4.** APCI screening method result with compound prediction for an unknown compound.

Figure 5. Quick overview of possible target compound identity in quantitative method result window (APCI ionisation).

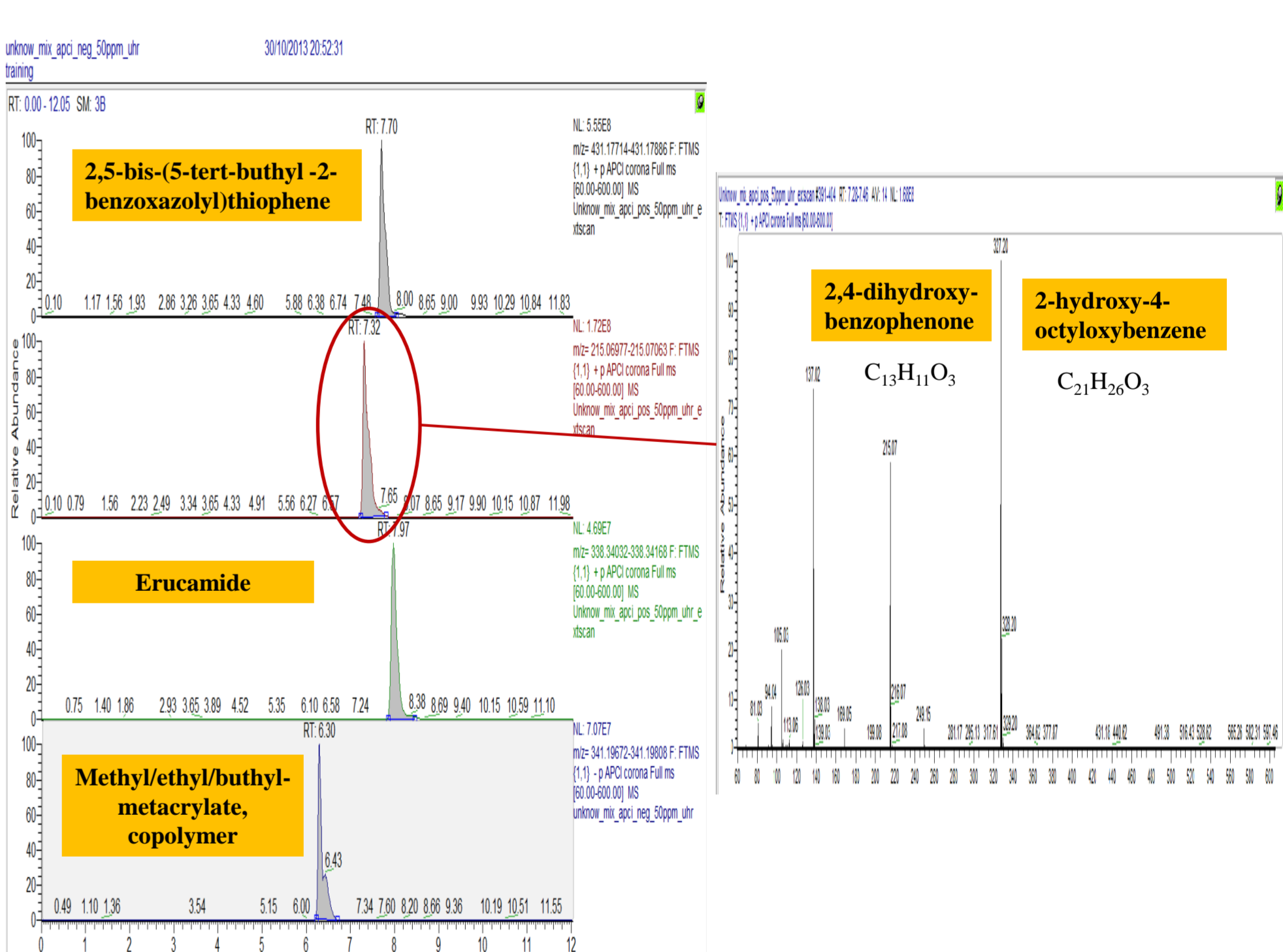### Identified unknown compounds

Figure 6. Overview of the identified four unknown compounds.

SUMMARY

➢ All test compounds could be identified correctly

➢ HRMS is essential for high confidential identification of both known and unknown compounds

➢ adequate software support with correct databases is necessary to apply in order to get correct results.

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