

High throughput identification of anabolic steroid esters by compact atmospheric solid analysis probe mass spectrometry system

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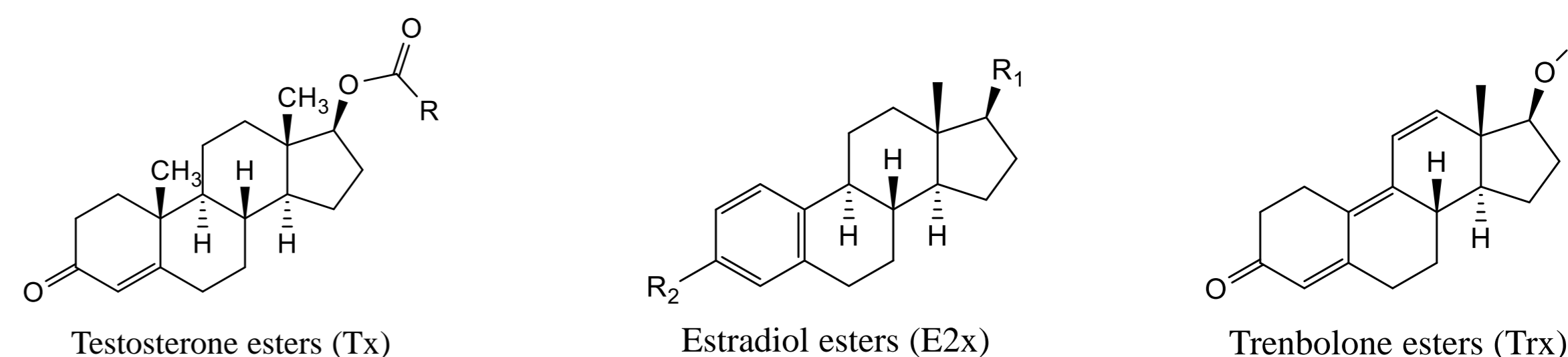


Introduction

Rapid and easy-to-perform (on-site) analysis methods have a bright future in forensic and food analysis. Implementing on-site pre-screening of target compounds in samples would reduce the number of suspicious samples to be transported to the control laboratories for their analysis. Therefore, it would improve the efficiency of control and monitoring programs. Ambient ionization mass spectrometry (AIMS) techniques offer simplified sample preparation and sample introduction protocols [1].

This work, evaluated the feasibility of a compact atmospheric solids analysis probe (ASAP) - single quadrupole mass analyzer system for the screening and rapid identification of anabolic steroid esters. The applicability was demonstrated to rapidly identify of steroid esters in oily injection solutions, providing test results in less than 2 min.

Anabolic steroid esters



Model compounds: T acetate, T propionate, T isocaproate, T enanthate, T decanoate, T benzoate, T phenylpropionate, T cypionate, Nortestosterone phenylpropionate, boldenone undecylenate, E2 dipropionate, E2 valerate, E2 benzoate, Trenbolone, Tr acetate, Tr enanthate, drostanolone enanthate.

Instrumentation

- Atmospheric solid analysis probe (ASAP) ionization source
- Single quadrupole mass analyzer
- Minimal sample preparation
- Real time results

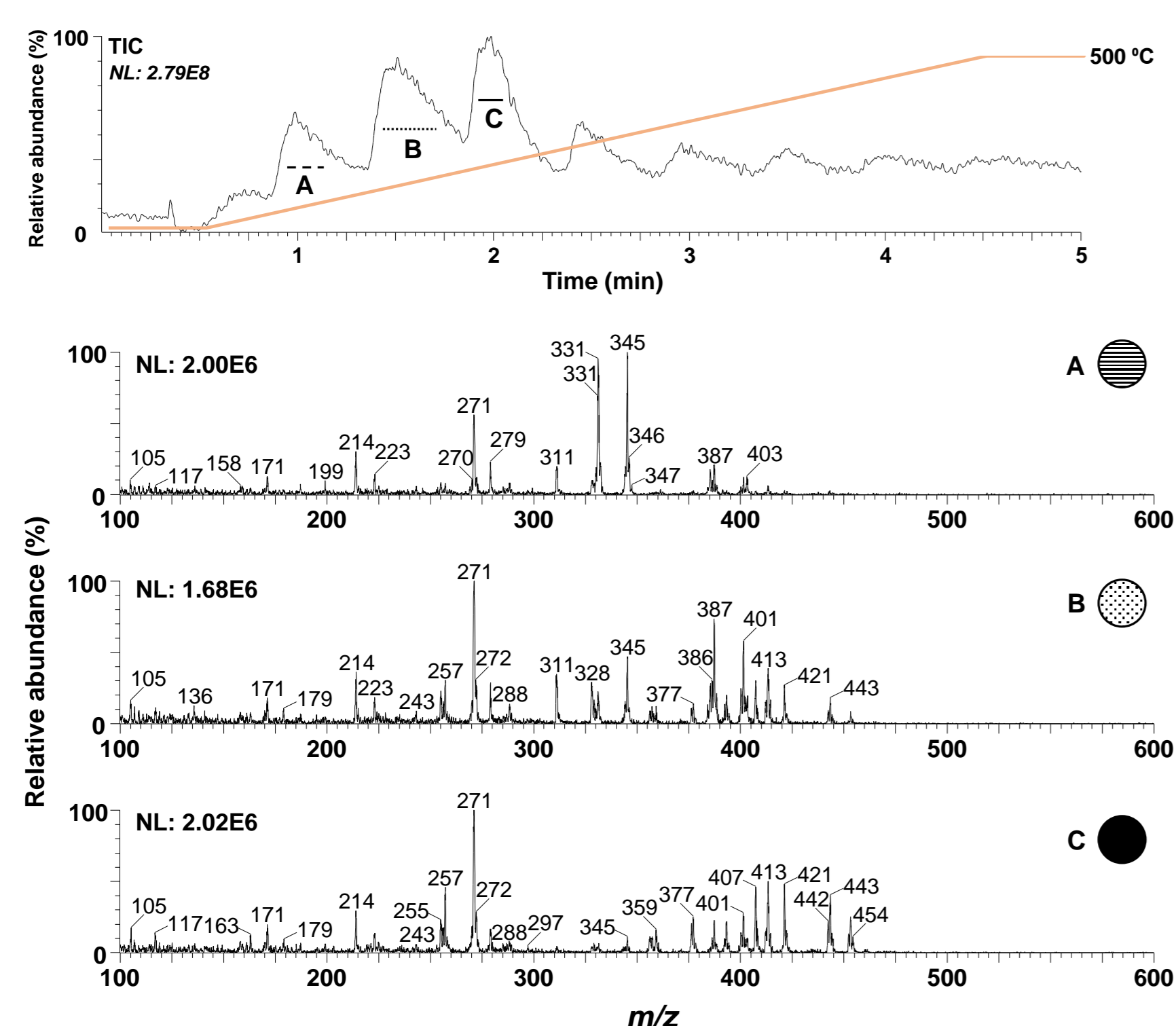


Figure 1: RADIUM ASAP Direct Mass Detector

Results

Method development

- Optimized parameters: sample introduction, sample solvent and source temperature.



- **Objective:** complete vaporization with no thermal degradation
- The side chain of target compounds influence on the desorption temperature
- **Result:** to ensure the complete desorption of all target compounds, the source temperature was set at **450 °C**

Figure 2. Upper trace, desorption temperature profile of the studied steroid esters; lower traces mass spectra of regions A (0.85–1.15 min), B (1.4–1.7 min), and C (1.9–2.1 min).

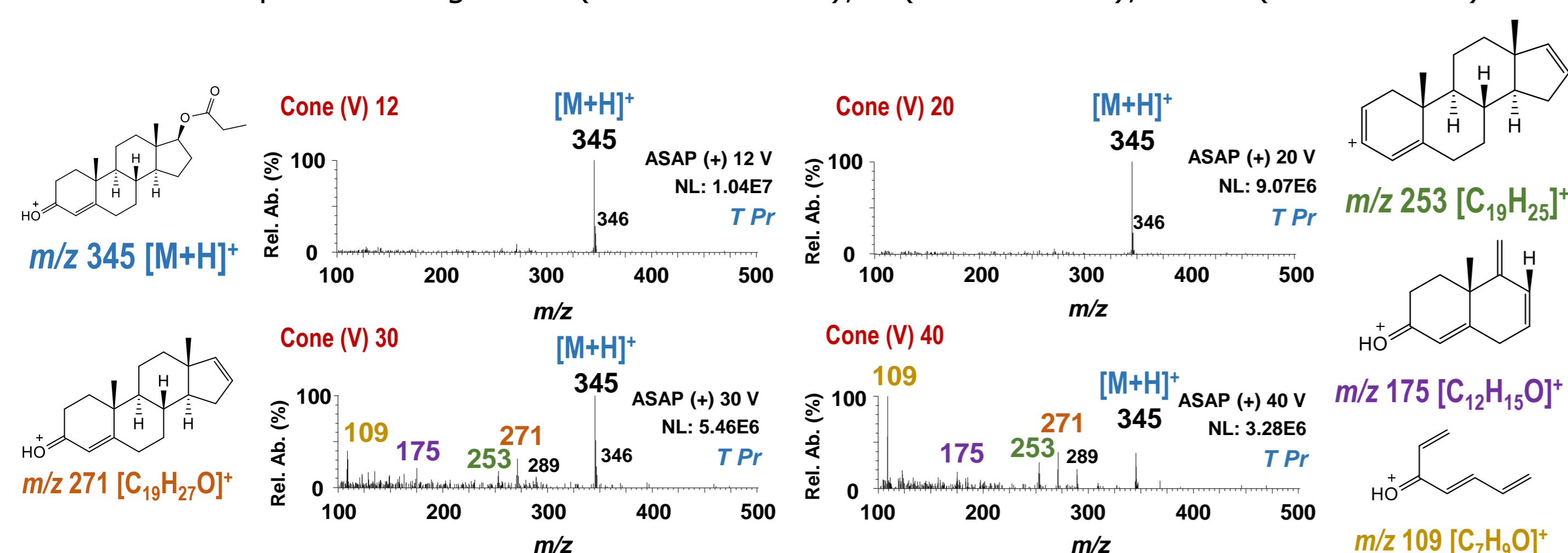


Figure 3. Mass spectra for testosterone propionate by ASAP-MS at the four selected cone voltages.

- Four different cone voltages (**12, 20, 30 and 40 V**) which allow the characterization of analytes are optimized for identification purposes.

Conclusions

- The applicability was demonstrated by identifying anabolic steroid esters such as testosterone, trenbolone, and estradiol in real samples.
- This approach can be beneficial to support the control and enforcement authorities since it can rapidly pinpoint suspicious samples for further confirmatory analysis

Screening of samples

- As a response to the potential illicit application of steroids in doping or in food production systems, the applicability of the developed ASAP-MS method was evaluated by analyzing 7 samples.

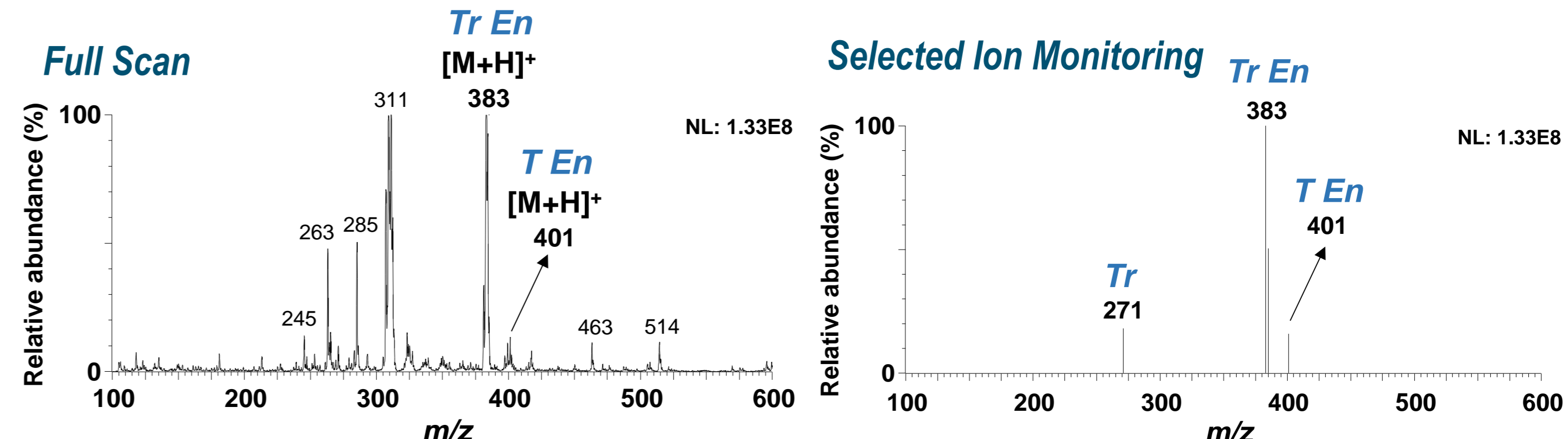


Figure 4. ASAP-MS full scan and selected ion recording spectra of an analyzed sample.

- Use of real time library matching workflow by matching the spectra of four acquisitions in full scan mode at selected cone voltages.

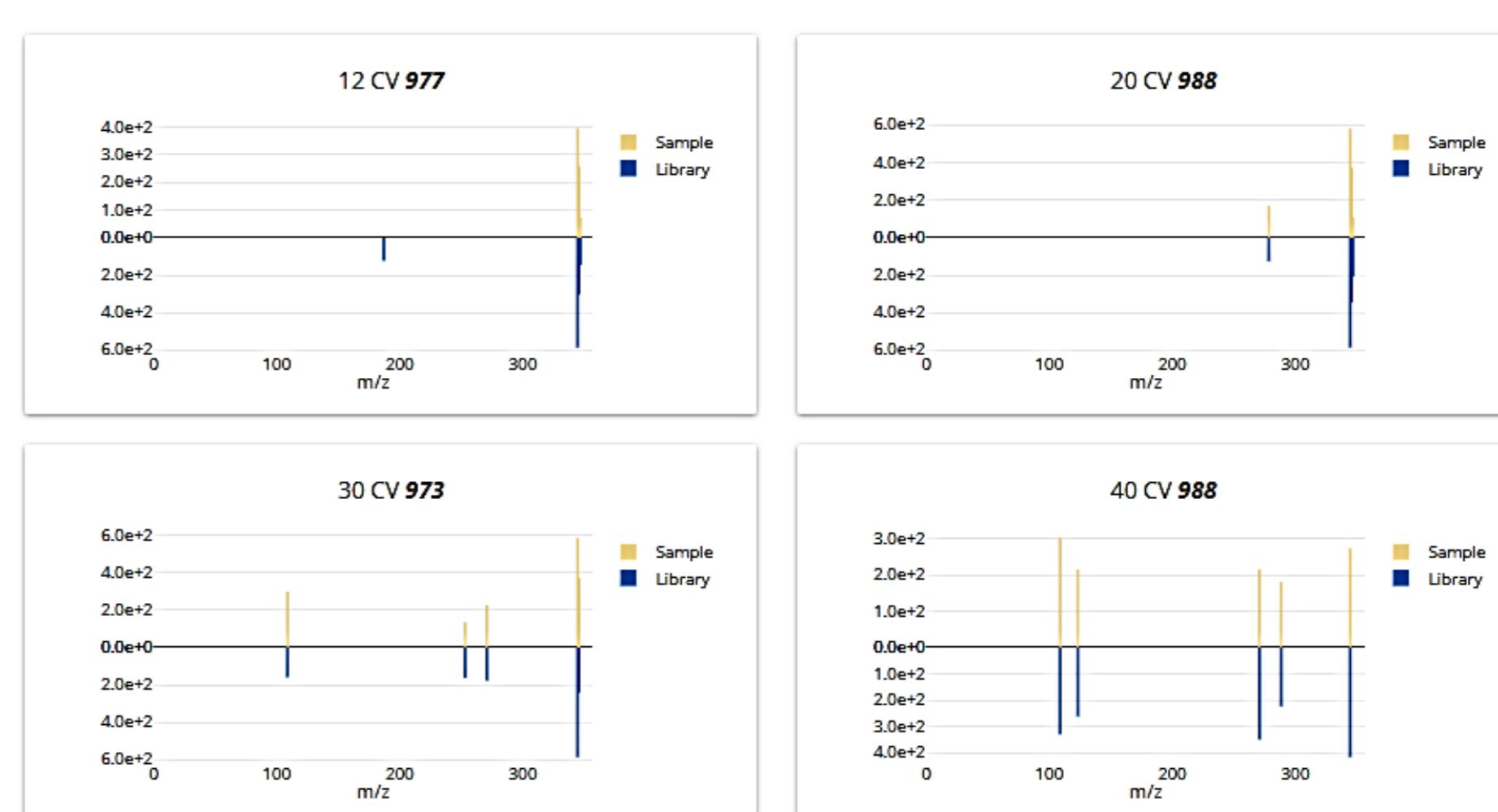


Figure 5. Library matching results from (A) high match score identification of testosterone propionate.

- Establish library matching identification criteria
- Average match score ≥ 800
- Function 1 (12V) ≥ 850
- Function 2,3,4 (20,30,40 V) ≥ 825

- Such a simple analytical workflow enables analysis of a large number of samples in a short analysis time (1 min) using a small sample (2 μ L) in a fully automated data interpretation process.

References

[1] A. Arrizabalaga-Larrañaga et al. Ambient Ionization mass spectrometry in food analysis, Food Toxicology and Forensics, (2021) 271–312. DOI: 10.1016/B978-0-12-822360-4.00006-6.

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