Screening of selective androgen receptor modulators by hand-held laser diode thermal desorption-transportable mass spectrometry

Ane Arrizabalaga-Larrañaga, Marco Blokland, Michel Nielen

WAGENINGEN UNIVERSITY & RESEARCH

European Union Reference Laboratory

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.. However, today, only a few studies reported ambient ionization of food contaminants and drug analysis with a (trans)portable MS system.

This work developed a handheld laser diode thermal desorption electrospray ionization (LDTD-ESI) mass spectrometry method to rapidly screen illegal substances in solid samples. The applicability was demonstrated to rapidly identify selective androgen receptor modulators (SARMs) in powders and pills [1].

LDTD-ESI-MS Set up

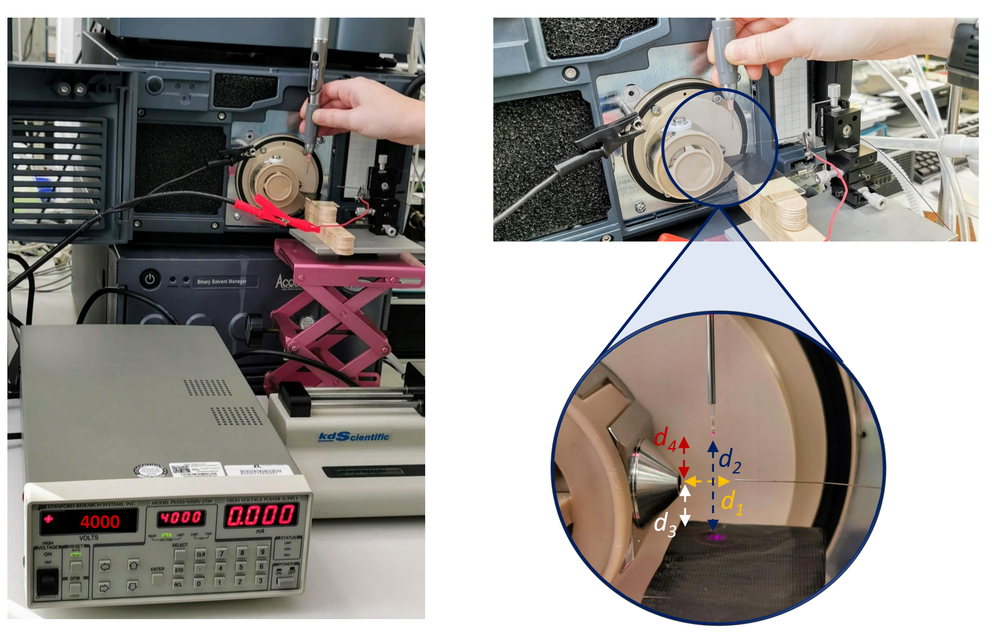

Figure 1: Instrumental setup of the laser ablation electrospray (LDTD-ESI) source with a battery-powered handheld 940 nm diode laser coupled to the transportable single quadrupole MS

Results

Method development

- The solid surface characteristics, including its chemical composition, texture and color severely affects the energy transfer processes and, therefore, the desorption efficiency.

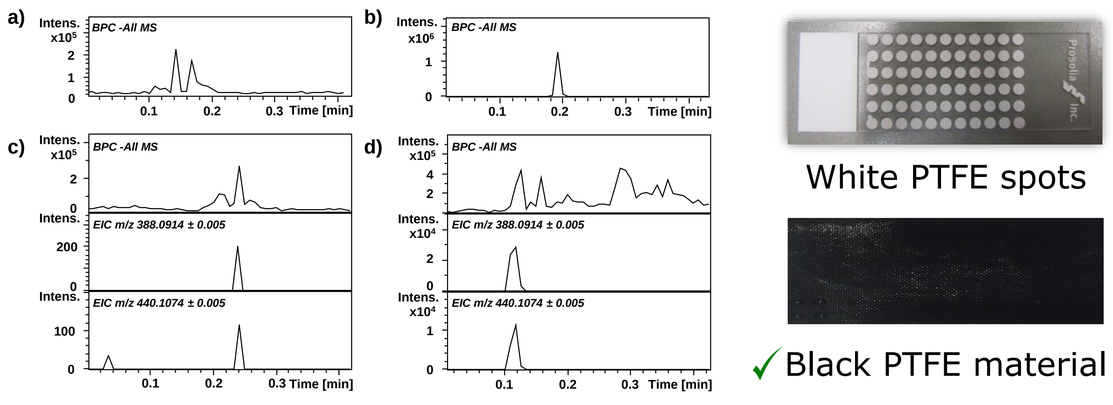

Figure 2. Chronograms obtained by LDTD-ESI−HRMS: base peak chronogram of a blank with (a) white PTFE substrate and (b) black PTFE substrate and extracted ion chronogram of SARMs ostarine and andarine spotted onto (c) white PTFE substrate and (d) black PTFE substrate.

- ESI solvent composition was optimized to 0.1% ammonia and 0.1% formic acid in methanol/water (50:50, *v/v*) in negative and positive ion mode, respectively.

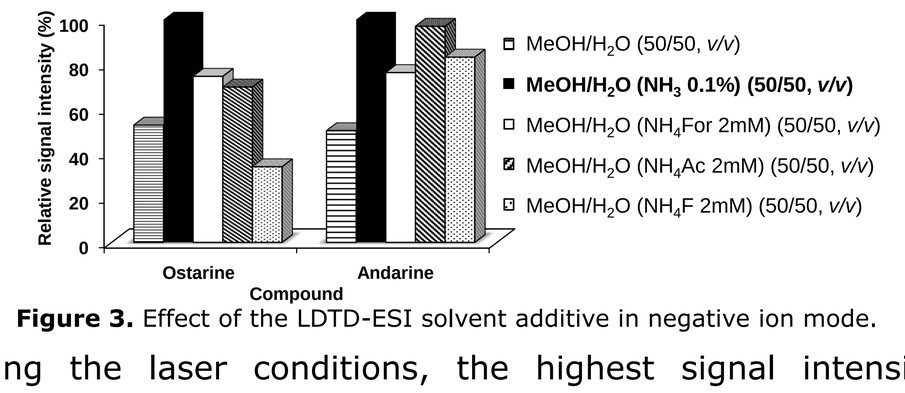

Figure 3. Effect of the LDTD-ESI solvent additive in negative ion mode.

- Regarding the laser conditions, the highest signal intensity was observed when applying 3 W peak power and CP1 pulse mode

Screening of samples

As a response to the potential illicit application of SARMs in sports doping or in food production systems, the applicability of the developed LDTD-ESI−HRMS method was evaluated by analyzing 10 samples.

- No signal when analyzing directly the outside and inside of the pills (Fig.4A)
- ✓ Identification of SARMs when powder was placed onto the sample stage (Fig.4B)

Figure 4. Direct analysis of (A) pills from outside part and (B) powders placed into the substrate by LDTD-ESI-MS.

In the transportable MS system (single Quadrupole analyzer), samples were analyzed in full scan mode applying cone voltage to perform in-source fragmentation for identification purposes.

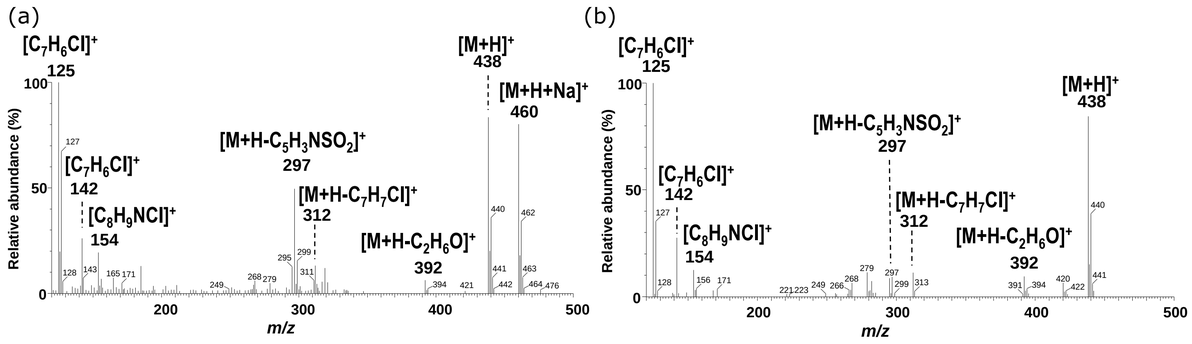

Figure 5. LDTD-ESI−transportable MS full-scan spectrum of (a) sample 1 and (b) stenabolic standard obtained by applying 30 V.

Conclusions

- Applicability of the developed LDTD-ESI-MS set up has been demonstrated by identifying 10 SARMs in real samples
- These initial results demonstrate the applicability of the simplified LDTD-ESI-MS method for future on-site analysis in solid samples

References

[1] A. Arrizabalaga-Larrañaga, M.W.F. Nielen, M.H. Blokland, Hand-Held Diode Laser for On-Site Analysis Using Transportable Mass Spectrometry, Analytical Chemistry, 93 (2021) 8122-8127.

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Wageningen University & Research
P.O. Box 123, 6700 AB Wageningen
Contact: ane.arrizabalarranaga@wur.nl
T + 31 628 04 37 82
www.wfsr.nl