

SCREENING OF DIETARY SUPPLEMENTS by ASAP[®] Compact Mass Spectrometry

Jack Henion and Changtong Hao, Advion, Inc.

Advion

INTRODUCTION

Americans spend nearly \$4 billion a year on diet and weight loss supplements. While distributors are largely regulated by the Food and Drug Administration, there are still products on the market that have versions of banned substances, or products that are still being sold months after recall.

In this application note, we demonstrate the use of the Advion *expression*¹ CMS with an atmospheric solids analysis probe (ASAP) ion source to analyze two supplements (figure 1) that contain DMBA, a synthetic stimulant that has not been tested on humans. The substance is the chemical cousin to drug DBAA, which is currently banned by the US FDA. The efficacy and safety of DMBA is entirely unknown, yet it is present in many supplements.

This technique demonstrates the ability for the CMS to rapidly test for and detect DMBA, as well as other ingredients including caffeine and niacin, in less than 30 seconds per sample.

RESULTS AND DISCUSSION

The unknown chemical could readily be detected with the atmospheric solids analysis probe (ASAP/APCI) from the released 820 mg capsule (figure 2). In addition to DMBA (figure 3), the ASAP analysis method can rapidly detect and process multiple samples, and via full-scan acquisition or selected ion monitoring (SIM), detect amounts of other key ingredients including caffeine.

With ASAP/APCI analysis, the ASAP probe with the sample is inserted into the source (figure 4,

5), quickly producing a spectrum for the ion of interest via a full-scan acquisition sample assay (figure 6). DMBA is easily detected in each product powder by this method of analysis. The detector also shows that the Decimate product also contains niacin, while AMPilean does not (figure 7).

The mass spectral analysis shows AMPilean has approximately 2x the DMBA as Decimate (figure 8), and that both samples contain large quantities of caffeine.



FIGURE 1

1. Two dietary supplements for analysis DMBA is 1, 3-dimethyl butylamine, a 'chemical cousin' to DMAA and believed to have similar effects, but not studied in humans. It is now very common in supplements and is promoted for weight loss, memory enhancement and as a sports performance supplement.

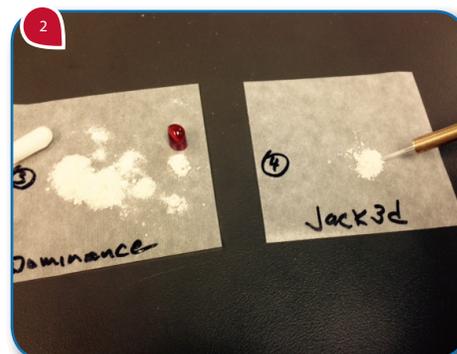
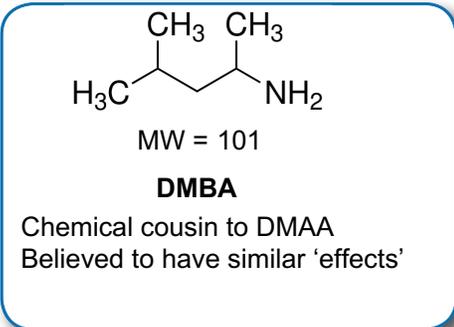
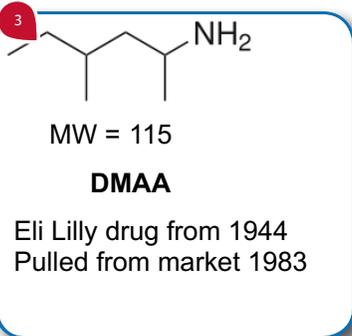


FIGURE 2

2. ASAP/APCI sample method of two capsules Over the counter samples of Decimate Amplified and AMPilean are released and sampled via the glass capillary of the ASAP probe.



FIGURES

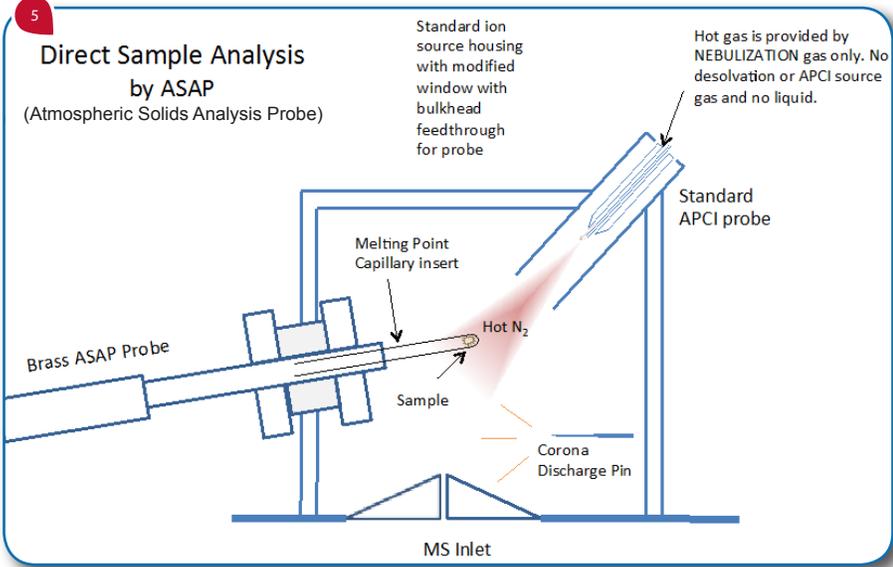
- 3. Composition of DMAA and DMBA
DMAA was pulled from the market in 1983, while it's chemical cousin DMAA is still available, believed to have similar effects to DMAA.
- 4. ASAP Probe Inserted into **expression^L**
The brass probe with glass capillary containing the sample are inserted into the ASAP/APCI source for analysis.
- 5. ASAP Direct Sample Analysis Schematic
Inside the APCI source, sample is volatilized by the hot nitrogen gas and undergoes atmospheric pressure chemical ionization via the corona discharge to produce positive or negative ions of the chemical entities on the surface of the glass capillary.

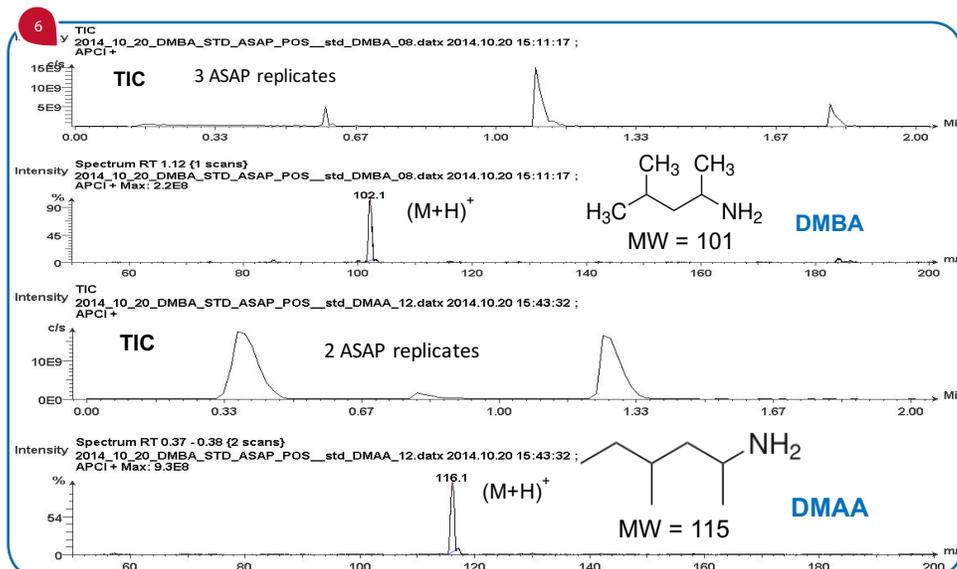
CONCLUSIONS

We used the **expression^L** CMS with an ASAP ion source to analyze the chemical constituents of two dietary supplements without any additional sample preparation or derivatization using a simple solid analysis mass spectrometry setup.

Additionally, we used full-scan mass spectral acquisition to show the mass spectra of each of these constituents, identifying that Decimate and AMPilean each contain large quantities of DMBA and caffeine, but with AMPilean containing twice as much as the Decimate supplement (figure 8). We also determined that Decimate contains niacin, while AMPilean does not.

DMBA is easily detected in each product powder by ASAP/APCI screening of dietary supplement powders requires less than 30 sec per sample.

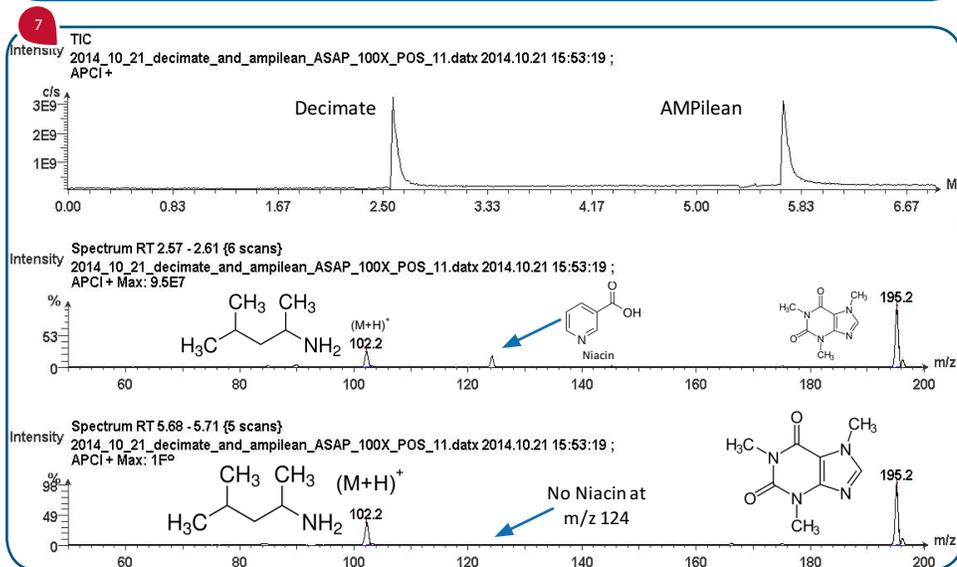




FIGURES

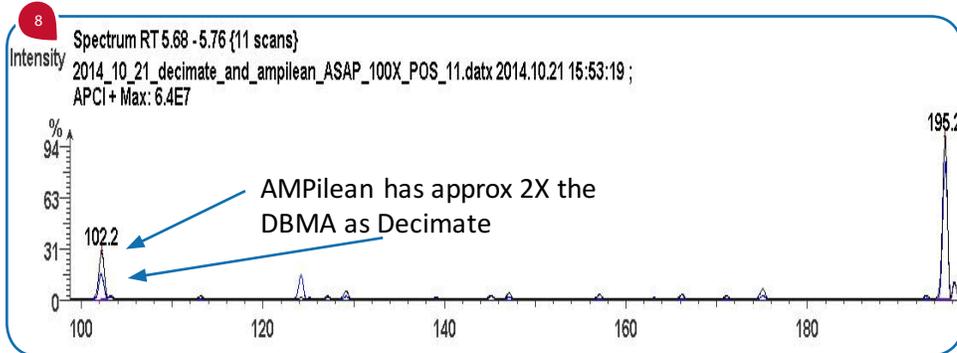
6. ASAP Positive Ions for Standard DMBA and DMAA

Determining the molecular weight of both DMBA and DMAA for a comparative analysis of the two dietary supplement products.



7. ASAP Positive Ion for Decimate and AMPilean

The molecular weight of 101 (DMBA) was detected in both samples, however niacin was only detected in the Decimate sample.



8. Spectral Overlay of m/z 102 for DMBA in Decimate and AMPilean

The intensity of AMPilean showed that it contains approximately two times the amount of DMBA as Decimate, as shown on the mass spectral analysis.

Advion

www.expressioncms.com
info@advion.com

Advion is a leader in mass spectrometry & synthesis solutions. The **expression** CMS is a high performance, compact, affordable single quadrupole mass spectrometer. Its compact size allows it to fit into space-limited labs for direct access and immediate results for chemists requiring mass confirmation, reaction monitoring, quality control and purity analysis.