Application Note : 1303

High-Throughput analysis method for straight chain alkanes Using LDTD–MS/MS

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Introduction

Straight Chain Alkanes are usually analyzed by GC-MS system using Electron Ionization (EI) mode as LC-MS does not properly ionize this type of molecule. To increase the sample throughput, LDTD-MS/MS is used for **10 seconds per sample analysis**.

The Laser Diode Thermal Desorption (LDTD) combined to a mass spectrometer is used to ionize and quantify alkane molecules. The LDTD is a rapid analysis approach in which samples are thermally desorbed. Molecules are channeled, using a carrier gas, to a corona discharge region for ionization prior to detection via a mass spectrometer. Pentacosane and Hexacosane were used as straight chain alkanes to evaluate the ionization process and the development of a quantitative method.

LDTD-MS/MS System

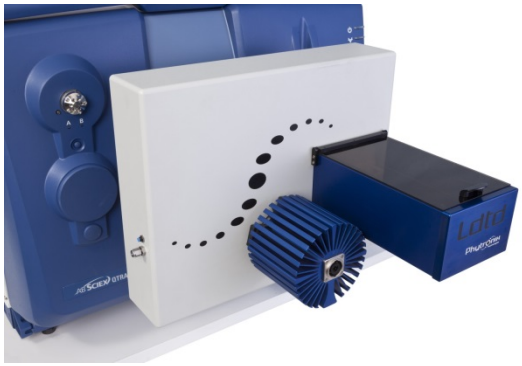

Figure 1: LDTD system on AB SCIEX 5500 Qtrap Mass Spectrometer

Method Development

Sample preparation

Stock solution of Pentacosane and Hexacosane were dissolved in hexane and the following solutions were prepared:

1) Optimization solution of 10 µg/mL in Hexane for both compounds
2) Standard curve of Pentacosane using Hexacosane as internal standard.
   - 50 ng/mL to 5000 ng/mL
   - 4 µL of Sample was added in LazWell plate and evaporated to dryness.

Hydrocarbon optimization

Q1 scan (positive):

Primary mass obtained for Pentacosane (MW: 352.68) was 351 m/z. This mass can be explained by the double bond formation followed by a positive ionization charge: M (-2H) (+H)

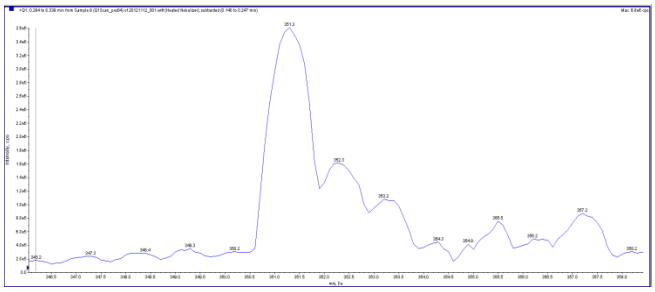

Figure 2: Q1 scan mass spectra of Pentacosane in positive mode

Product ion scan (pos):

Product ion was then generated with the primary mass, 351 m/z. With a low collision energy (CE:15), typical mass spectra was obtained with loss of 14.

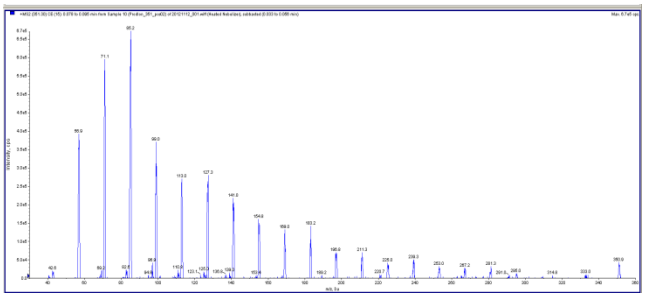

Figure 3: Product ion scan mass spectra of Pentacosane in positive mode

Dehydrogenation mechanism

A double bond formation during LDTD analysis of Hydrocarbon can be explained by the catalytic mechanism for alkane dehydrogenation reported by Weckhuysenet al[1]. Sample was dried in LazWell plate containing stainless steel sheet. Sample was then vaporized and transferred to the ionization region of the mass spectrometer.

1) Reference : B.M. Weckhuysen, R.A. Shoonheydt, Catalysis Today, 51 (1999) 223-232

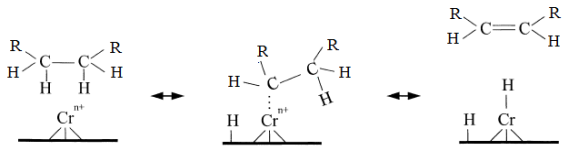

Figure 4: Catalytic mechanism reported by Weckhuysen et al.

LDTD-MS/MS Parameters

Laser power pattern:

- Increase laser power to 45 % in 3.0 s
- Stay at 45% for 2.0 s
- Decrease laser power to 0 %

Carrier gas flow: 3 L/min (Air)

MS Parameters

APCI (+)
Scan time: 0.050 s
CE : 22 eV
NC : 3µA
MRM:

- Pentacosane: 351 -> 71
- Hexacosane: 365 -> 71

Results and Discussion

Linearity Results

As shown in **Figure 5,** excellent linearity (r^2> 0.99) with no signs of carryover effect is achieved within the quantification range (50 to 5,000ng/mL) for Pentacosane.

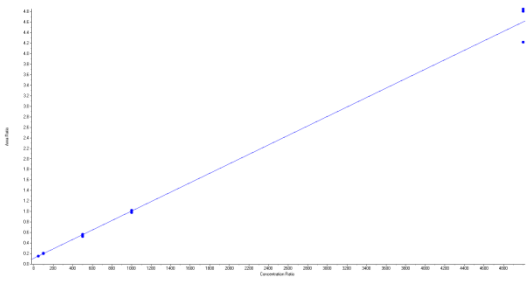

Figure 5: Typical standard curve

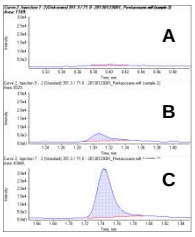

Figure 6:
Typical desorption peak:
A) Blank
B) STD 50
C) STD500

Accuracy and Precision

As shown in **Table 1,** the intra-run accuracy and precision are between 96.8 to 103.8% and 1.3 to 7.8% for Pentacosane.

Drug	S50	S100	S500	S1000	S5000
Nominal conc (ng/mL)	50	100	500	1000	5000
N	3	3	3	3	3
Mean (ng/mL)	49.9	103.8	484.0	990.4	5021.8
%RSD	1.3	4.7	4.7	2.6	7.8
%Nom	99.9	103.8	96.8	99.0	100.4

Table 1: Accuracy and Precision result

Conclusions

Using the LDTD technology, alkanes can be ionized and quantified according to catalytic mechanism reported by Weckhuysen et al.

A fast analysis can be achieved using LDTD-MS/MS system. This system allows a total sample-to-sample analysis time of **8 seconds**.