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

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Keywords: High-throughput, alkanes, LDTD

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

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)

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.

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**Dehydrogenation mechanism**

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


**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.

**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.

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

**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.