TP607

Ultra-Fast profiling of C20-C60 Alkanes in wax samples using LDTD-MS/MS

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OVERVIEW

Purpose

• Rapid evaluation profile of C20-C60 and quantification of C25 in different wax products.

Method

• Wax compounds were dissolved in hexane and dispensed on LazWell plate for fast LDTD-MS/MS analysis

Results

• C20-C60 profile obtained in a few seconds.
• Quantification: Excellent linearity over the calibration range ($R^2 > 0.99$)

Samples are analyzed with a runtime of 10 seconds using LDTD- MS/MS system

INTRODUCTION

C20-C60 Alkanes are the main components of waxes. It's important to measure the composition (amount and type) of wax in crude oil to avoid precipitation during production that may cause serious pipeline blockage problems and/or oil gelling. Usually, C20-C60 wax compositions are analyzed using a GC system (Typical run time: 30 minutes). Laser Diode Thermal Desorption (LDTD) combined to a mass spectrometer is used to generate faster quantification and profiling of C20-C60 alkanes. The LDTD-MS/MS is a ultra rapid analysis (10 seconds) approach in which samples are thermally desorbed. Neutral molecules are channeled, using air as a carrier gas, to a corona discharge region for ionization prior to detection via a mass spectrometer.

LDTD™ Ionization Source:

The LDTD uses a Laser Diode to produce and control heat on the sample support **(Figure 1)** which is a 96 wells plate**.** The energy is then transferred through the sample holder to the dry sample which vaporizes prior to being carried by a gas in a corona discharge region. High efficiency protonation and strong resistance to ionic suppression characterize this type of ionization, and is the result of the absence of solvent and mobile phase. This allows for very high throughput capabilities of 10 seconds sample-to-sample analysis time, without carry over.

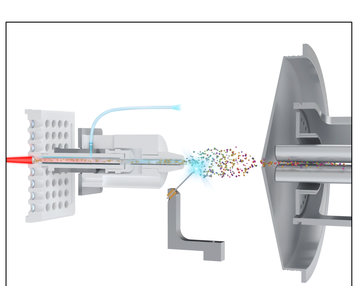

Figure 1 Schematic of the LDTD ionization source.

METHOD

Sample preparation

Stock solution of Pentacosane (C25) was dissolved in hexane and the following standards were prepared:

- Standard curve of Pentacosane
  - 0.5 µg/mL to 50 µg/mL
- Wax samples
  - 100 µg/mL in hexane (10 mg wax sample in 100 ml hexane)
- 4 µL of sample was added in LazWell plate and evaporated to dryness
  - 2 to 200 ng of C25 on LazWell plate

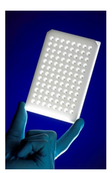

Figure 2 LazWell™ sample plate

Instrumentation

• LDTD model S-960, Phytronix Technologies
• QTRAP® 5500, AB Sciex

MS Parameters (Scan mode)
•APCI (+)
•Scan time : 0.250 s
•NC : 3µA

MS Parameters (Quantification Method)
•APCI (+)
•Scan time : 0.100 s
•CE : 22 eV
•NC : 3µA
•MRM:
➢ Pentacosane: 351 → 71

LDTD Parameters
• Laser power pattern :
- Increase laser power to 45 % in 3.0 s
- Maintain at 45% for 2.0 s
- Decrease laser power to 0 %
• Carrier gas flow : 3 L/min (Air)

In table 1, a list of masses used for positive Q1 scan experiment is shown. As previously presented by Belisle et Al[1], masses are obtained by the double bond formation followed by a positive protonation: M (-2H) (+H).

Table 1 Scan mode mass

	M/Z	width	Time (sec)
C20	281.15	0.3	0.002
C21	295.15	0.3	0.002
C22	309.15	0.3	0.002
C23	323.15	0.3	0.002
C24	337.25	0.3	0.002
C25	351.25	0.3	0.002
C26	365.25	0.3	0.002
C27	379.25	0.3	0.002
C28	393.25	0.3	0.002
C29	407.25	0.3	0.002
C30	421.35	0.3	0.002
C31	435.35	0.3	0.002
C32	449.35	0.3	0.002
C33	463.35	0.3	0.002
C34	477.35	0.3	0.002
C35	491.35	0.3	0.002
C36	505.35	0.3	0.002
C37	519.45	0.3	0.002
C38	533.45	0.3	0.002
C39	547.45	0.3	0.002
C40	561.45	0.3	0.002
C41	575.45	0.3	0.002
C42	589.45	0.3	0.002
C43	603.55	0.3	0.002
C44	617.55	0.3	0.002
C45	631.55	0.3	0.002
C46	645.55	0.3	0.002
C47	659.55	0.3	0.002
C48	673.55	0.3	0.002
C49	687.55	0.3	0.002
C50	701.65	0.3	0.002
C51	715.65	0.3	0.002
C52	729.65	0.3	0.002
C53	743.65	0.3	0.002
C54	757.65	0.3	0.002
C55	771.65	0.3	0.002
C56	785.65	0.3	0.002
C57	799.75	0.3	0.002
C58	813.75	0.3	0.002
C59	827.75	0.3	0.002
C60	841.75	0.3	0.002

1) Belisle et al., Poster WP-05-62, ASMS 2013

RESULTS

Q1 scan of different wax products:

C20-C60 profiles of different products are obtained in **10 seconds** using Q1 scan positive mode.

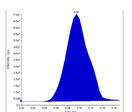

Figure 3 Typical desorption peak.

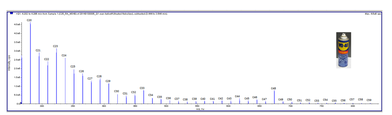

Figure 4 C20-C60 profile of WD-40 product.

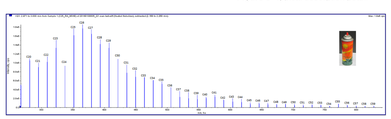

Figure 5 C20-C60 profile of Releasall product.

Figure 6 C20-C60 profile of Teflon grease product.

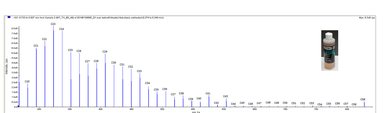

Figure 7 C20-C60 profile of Mascon-Tap product.

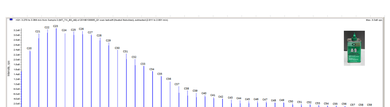

Figure 8 C20-C60 profile of B9 oil product.

Figure 9 C20-C60 profile of B3 oil product.

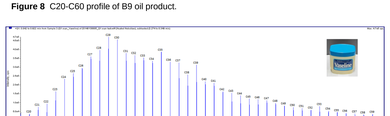

Figure 10 C20-C60 profile of Vaseline product.

C25 quantification results:

Quick quantification using a calibration range of 2 to 200 ng of alkane (C25 for this application) on LazWell plate is obtained in **10 seconds** using MRM positive mode.

Table 2 C25 concentration evaluation in product

Product	Conc. (mg/g Wax sample)
Releasall	11.3
Mascou-Tap	7.8
B3 oil	25.5
WD-40	2.0
Teflon grease	4.8
A9 oil	171.6
Vaseline	11.6

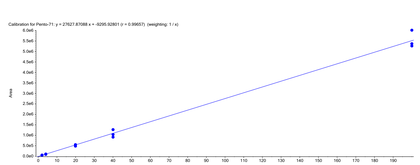

Figure 11 Typical standard curve of C25

CONCLUSIONS

- **LDTD Ion Source coupled to a mass spectrometer enables specific and fast C20-C60 profile of different wax compounds**
- **LDTD-MS/MS gives accurate and precise quantification of C25 over a range of 2 to 200 ng on plate**
- **LDTD provides High-Throughput analysis of Alkanes in 10 seconds sample-to-sample**