AC SIMDIS^{xlnc}: determine wax content compliant with ASTM D5442

- Carbon distribution / Wax content
- Dedicated SIMDIS^{xlnc} software application
- Intelligent integration protocol
- Meets ASTM D5442 requirements

Keywords: D5442, Wax content, Intelligent Integration, SIMDIS^{xlnc}

Introduction

Petroleum waxes are used for various type of products on the market and, depending on the composition or mixture, can be applied to adhesives, packaging, plastics, and rubber.

Petroleum waxes are essential in the rubber and tire industry, as the greatest natural threats to tires and all other synthetic and natural rubbers are ozone and ultraviolet (UV) light. So-called Antiozonant waxes are applied to these rubber products as protection against these natural elements.

The carbon distribution as well as the ratio of normal paraffins and iso-paraffins in the mixture of a wax determine the protective behavior of the wax on the applied product.

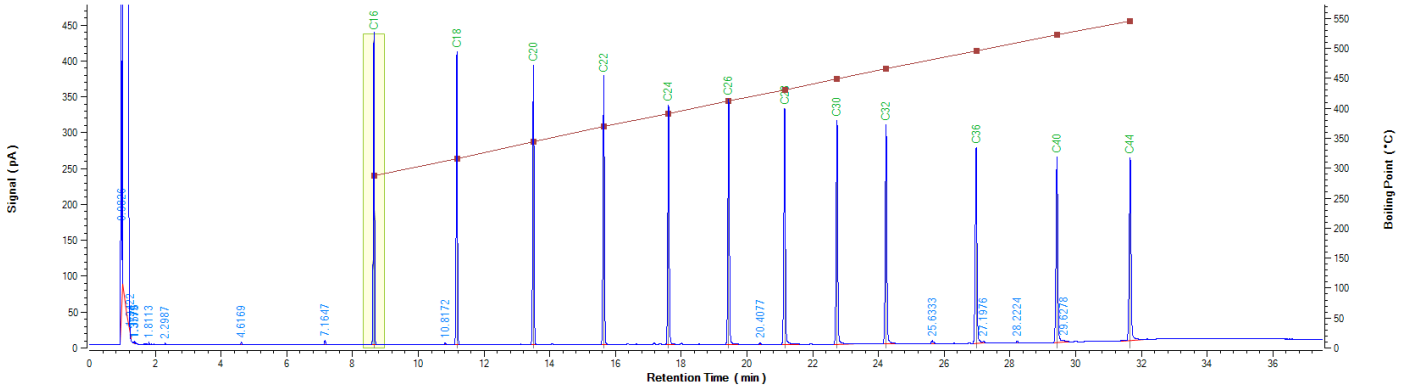
Standardization

ASTM D5442 is the gas chromatography-based test method for the quantitative determination of the carbon number distribution of petroleum waxes (wax content) in the range from n-C17 up to n-C44. Quantification is based on the use of internal standard. In addition to the carbon number distribution, the non-normal hydrocarbons content can be determined, as well as the C45+ content for samples heavier than n-C44.

ASTM D5442 is a Simdis-type analysis. Compared to a regular Simdis method, such as ASTM D2887, it requires several steps to calibrate, identify and quantify the desired properties of a petroleum wax. The new AC ASTM D5442 application in the IRIS based AC SIMDIS^{xlnc} software is specifically designed to perform these steps in a very user-friendly and accurate way and meets all the requirements as specified by ASTM D5442.

SIMDIS^{xlnc} D5442 calibration

To determine the carbon distribution content of a petroleum wax, the first step is to calibrate the system by analyzing a mixture containing known concentrations of normal paraffins in the range of n-C16 to n-C44.



Based on the identification and the specified concentrations, the AC SIMDIS^{xlnc} automatically determines the relative response factors of the normal paraffins to hexadecane (n-C16). The RRF found should not deviate from n-C16 by more than 10% according to the ASTM D5442 specification. If the RRF specification is not met, the software will indicate the outlier in red (see below picture). The response factor found is then applied to the quantification of the carbon number distribution in petroleum wax samples.

Details | Components | Used by | Audit trail

Short name	Boiling Point (°C)	Window - Relative (%)	Window - Absolute (min)	Reference component	Retention time (min)	Area (%)	RRF
C16	287.00	5.00	0.20	☐	8.6535	0.01	1.00
C18	316.00	5.00	0.20	☐	11.1764	0.01	1.00
C20	344.00	5.00	0.20	☐	13.5043	0.01	1.00
C22	369.00	5.00	0.20	☐	15.6435	0.01	1.00
C24	391.00	5.00	0.20	☐	17.6156	0.01	1.08
C26	412.00	5.00	0.20	☐	19.4446	0.01	1.01
C28	431.00	5.00	0.20	☐	21.1478	0.01	1.01
C30	449.00	5.00	0.20	☐	22.7407	0.01	1.02
C32	466.00	5.00	0.20	☐	24.2338	0.01	1.02
C36	496.00	5.00	0.20	☐	26.9739	0.01	1.09
C40	522.00	5.00	0.20	☐	29.4308	0.01	**1.12**
C44	545.00	5.00	0.20	☐	31.6610	0.01	1.08

Since this Simdis application requires regular calibration, usually based on the same calibration mixture, SIMDIS^{xlnc} will automatically apply the last known identification and concentrations to the new calibration analysis, minimizing operator involvement.

SIMDIS^{xlnc} D5442 sample analysis

Sample analysis by ASTM D5442 requires a very different software approach than a regular Simdis application, as it uses:

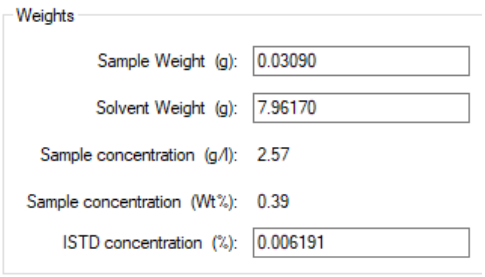
- an internal standard.
- a unique manner of integration

to correctly quantify the carbon distribution content of a Petroleum Wax. This requires a different software approach, where the AC SIMDIS^{xlnc} is optimized to perform the identification, integrate, and quantification in a single automated workflow.

INTERNAL STANDARD

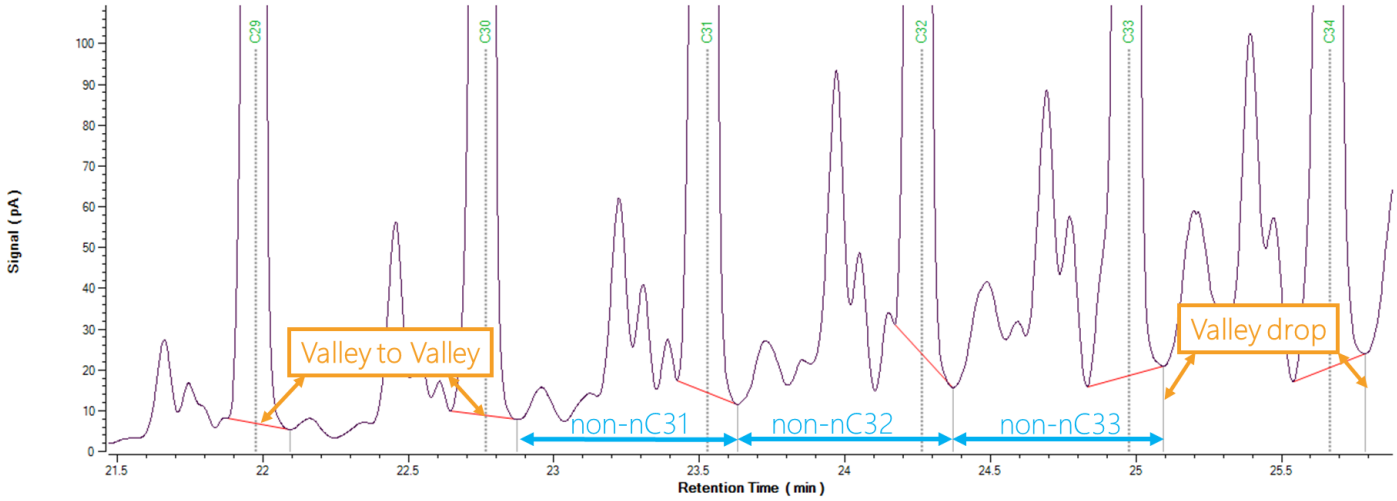
The operator's only action is to dissolve a small portion of the sample with a pre-prepared internal standard solution of cyclohexane containing a small concentration of the internal standard n-hexadecane (n-C16) and start the analysis.

The software requires the input of sample weight, ISTD solution weight and the concentration of n-C16 in the ISTD solution.



INTEGRATION

After an analysis is finished, the sample signal is automatically processed by the SIMDIS^{xlnc} software, which starts by subtracting the blank analysis signal. The normal-paraffins, including the ISTD, are identified using the calibration analysis and integrated as per below chromatogram via ~~a~~ valley-to-valley integration. The found normal-paraffin peak area is used for quantification.



The non-normal hydrocarbons are integrated and quantified via a so-called valley drop integration, where the area is taken from the valley drop of the normal-paraffin of interest to the valley drop of the previous normal-paraffin. The earlier integrated normal-paraffin peak is excluded from this area.

In above chromatogram an example is shown of the valley drop lines and the valley drop area of the non-normal hydrocarbon nC31, nC32 and nC33.

SIMDIS^{xlnc} processes all steps fully automatic, from importing the data, integration, Identification and to its final steps quantification and reporting.

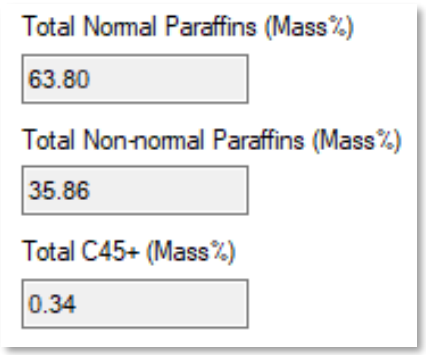
The AC ASTM D5442 application in SIMDIS^{Xlnc} contains two unique reports:

- Totals report where the Total normal paraffins, Total Non-normal Paraffins (hydrocarbons) and Total C45+ are reported.
- Carbon distribution content / Wax content where the found concentrations are expressed per carbon number.

Wax Content Report

Carbon Number	Normal Paraffin (Mass %)	Non-normal Paraffin (Mass %)
17	0.00	0.01
18	0.00	0.01
19	0.01	0.01
20	0.02	0.02
21	0.06	0.02
22	0.18	0.03
23	0.48	0.04
24	0.99	0.07
25	1.52	0.07
26	2.14	0.11
27	3.00	0.20
28	3.77	0.38
29	5.03	0.70
30	5.84	1.18
31	6.68	1.74
32	6.15	2.57
33	6.26	2.78
34	5.12	3.24
35	4.45	3.33
36	3.31	3.38
37	2.28	3.15
38	1.72	3.12
39	1.40	2.68
40	1.08	2.20
41	0.86	1.74
42	0.64	1.33
43	0.48	0.99
44	0.34	0.73

Typical "Carbon distribution content / Wax content" report



Typical "totals report"

Conclusion

The AC ASTM D5442 application on AC SIMDIS^{XLNC} is the go-to software application for "ASTM D5442: Petroleum wax analysis" on the market today, as it is:

- Designed to perform all critical steps as per ASTM D5442 requirements:
  - calibrate for boiling-point retention curve & determine the found RRF against n-C16.
  - integrate and identify normal paraffins and non-normal paraffins.
  - quantify using ISTD and RRF found during calibration.
  - report total wax content and carbon content per carbon number.
- Easy to use, workflow-oriented software
- All-round compatibility, it is compatible with every Chromatography Data Handling Software (that can produce a CDF). SIMDIS^{XLNC} has been tested against ChemStation, EZChrom, OpenLab ChemStation and EZChrom Editions, Atlas, Chromeleon, ChromPerfect and Galaxie/Kompass CDS.

Do you wish to have an online demo of the AC SIMDIS^{XLNC} capabilities, even with your own datafiles? Please contact your sales representative to schedule an appointment with the AC application team.

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Part number: AN-2021-C-009