APPLICATION NOTE

C-K-001-2015/A1

N Determination in Engine Oil

according to the Kjeldahl method

Reference: **ASTM D 3228 – 03 Total Nitrogen in Lubricating Oils and Fuel Oils by Modified Kjeldahl Method**

Tested with **VELP Scientifica DKL 20 Automatic Kjeldahl Digestion Unit** (Code S30100210) and **UDK 169 Automatic Kjeldahl Analyzer with AutoKjel Autosampler** (Code S30200160)

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NITROGEN DETERMINATION IN ENGINE OIL
KJELDAHL METHOD

Introduction

Oil additives are used to improve the base oil into a high performing lubricant. By utilizing the same base stock many different oils can be manufactured, each with its own unique properties, by adding different additive packages.
Combined nitrogen additives in petrochemical fuels give origin by combustion to nitrogen oxides whose emission in the atmosphere is subjected to limitations. Combustions occurring at relatively low temperatures (1400-1700 °F, 750-950 °C) produce nitrogen oxides mostly from fuel-bound nitrogen and not from atmospheric nitrogen.
The use of lubricating oil triggers the production of insoluble oxidation products.
To prevent deposits and reduce the risk of damage, the nitrogen content in lubricating oils must be kept low.
For this reason Nitrogen is one of the quality control parameters of crude oil, lubricants and fuel oils.

Nitrogen Determination in Engine Oil according to the Kjeldahl Method

Thanks to the high level of precision and reproducibility and to its simple application, Kjeldahl is nowadays the most used method for determining nitrogen and protein contents in the food and feed industry. It also has several other applications in environmental control (phenols and nitrogen in water, sludge, soil and lubricants) and in the chemical and pharmaceutical industry according to official AOAC, EPA, DIN e ISO procedures.
The modern Kjeldahl method consists in a procedure of catalytically supported mineralization of organic material in a boiling mixture of sulfuric acid and sulfate salt at digestion temperature higher than 400 °C. During the process the organically bonded nitrogen is converted into ammonium sulfate. Alkalizing the digested solution liberates ammonia which is quantitatively steam distilled and determined by titration.

Sample

Engine oil — Accepted nitrogen content*: 0.015 to 2.0 % N

*The ASTM D 3228 – 05 method covers the determination of nitrogen in fuel oils when present in the concentration from 0.015 to 2.0 % N.

Sample Digestion

Stir the engine oil into a beaker using a VELP magnetic stirrer at 700 rpm.
Using a pipette put 1.500 g of sample into a nitrogen-free weighing boat (code CM0486000) and place it into a 250 ml test tube.
For each sample, add in the test tube:

- 2 catalyst tablet ST (code CT0006609; 3.5 g K_2SO_4, 0.0035 g Se)
- 18 ml concentrate sulphuric acid (96-98%)
- 5 ml of hydrogen peroxide (~ 30%)

Prepare some blanks with all chemicals and without the sample.
Connect the Digestion Unit to a proper Aspiration Pump (JP code F30620198) and a Fume Neutralization System (SMS Scrubber code F307C0199) to neutralize the acid fumes created during digestion phase.
Digest the samples for 80 minutes at 150 °C, plus 80 minutes at 250 °C, plus 80 minutes at 350 °C and 30 minutes at 420 °C, according to the method “crude oil, lubricants and fuel oils” (n° 23 on DKL 20).

Distillation and Titration

Let the test tubes cool down to 50-60 °C.
Condition the **UDK 169 with Autokjel Autosampler** unit by performing the Automatic Check-up and Wash-down in the Menu-System.

Distill the samples according to the following parameters (pre-defined method n° 23):

- H_2O (dilution water): 70 ml
- NaOH (32 %): 70 ml
- H_3BO_3 (4 % with indicators): 30 ml
- H_2SO_4 (0.1 N) as titrant solution
- Protein factor: none

Distillation & Titration analysis time: from 4 minutes for one test.

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Typical Results on Engine Oil

The results are automatically calculated by UDK 169 as percentage of nitrogen.

Sample quantity (g)	Nitrogen %
1.513	0.055
1.499	0.059
1.518	0.059
1.501	0.063
1.503	0.060
1.508	0.061
1.518	0.060
1.504	0.060
1.501	0.059
1.512	0.060
Average ± SD%	**0.060 ± 0.002**

The complete procedure was verified by using 5 ml of glycine standard solution (3%) containing 28 mg of nitrogen, as reference substance. The obtained recovery falls into the expected range: between 98 % and 102 %.

Conclusions

The obtained results are reliable and reproducible in accordance with the expected values: all data fall in the indicated range.

Benefits of Kjeldahl method by using DKL 20 and UDK 169 with AutoKjel Autosampler are:

- High level of precision and reproducibility
- Maximum productivity and full automation
- Worldwide official method
- Reliable and easy method
- Time saving
- Moderate running costs

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