

Testing program for determination of distribution of sulfur compounds in shale gasoline

1. General

Shale gasoline contains a wide spectrum of sulfur compounds. Usually treatment methods are not removing all sulfur compounds. Different treatment methods are affecting different sulfur compounds. In order to understand selectivity of different methods we need to understand distribution of sulfur compounds in raw shale gasoline and gasoline after the treatment with different methods.

2. Objectives

- Determination of distribution of sulfur compounds in shale gasoline produced by Enefit140 and Enefit280 plants;
- Determination of changes in distribution of sulfur compounds in shale gasoline after treatment with caustic solution and scavengers;

3. Preparatory activities

3.1. Determination of the best method for determination of sulfur compounds in shale gasoline

- Literature review (GC-SCD, GC-MS; 2D-GC, etc.);
- Contact Adapter.ee;
- Select the best equipped laboratory in Estonia for determination of distribution of sulfur compounds.

3.2. Sample collection and preparation

- Order canisters for gasoline sampling (volume depends on the sample size requested by the laboratory);
- Take xL sample from Enefit140 and xL sample from Enefit280;

3.3. Perform gasoline treatment tests in the Enefit Energiatootmine laboratory

- Treatment with caustic solution;
- Treatment with scavengers;
- Collect treated shale gasoline samples.

3.4. Procurement and preparation of agreement

- Procurement process for selection of the third party laboratory for determination of distribution of sulfur compounds in shale gasoline samples;

- Preparation and signing of the agreement.

3.5. Team

- Testing coordinator;
- Third party laboratory;
- Enefit Energiatootmine laboratory;
- Analyst for interpretation of results.

4. Main testing method

Based on the literature review and feedback from the universities the main testing method was selected to be GC-MS

Description of the testing method is given below [Wikipedia]:

Gas chromatography–mass spectrometry (GC-MS) is an analytical method that combines the features of gas-chromatography and mass spectrometry to identify different substances within a test sample. Applications of GC-MS include drug detection, fire investigation, environmental analysis, explosives investigation, and identification of unknown samples. Additionally, it can identify trace elements in materials that were previously thought to have disintegrated beyond identification. GC-MS allows analysis and detection even of tiny amounts of a substance.

The GC-MS is composed of two major building blocks: the gas chromatograph and the mass spectrometer. The gas chromatograph utilizes a capillary column which depends on the column's dimensions (length, diameter, film thickness) as well as the phase properties. The difference in the chemical properties between different molecules in a mixture and their relative affinity for the stationary phase of the column will promote separation of the molecules as the sample travels the length of the column. The molecules are retained by the column and then elute (come off) from the column at different times (called the retention time), and this allows the mass spectrometer downstream to capture, ionize, accelerate, deflect, and detect the ionized molecules separately. The mass spectrometer does this by breaking each molecule into ionized fragments and detecting these fragments using their mass-to-charge ratio.

5. General description of tests

A short description of tests to be performed is given below:

1. Collection of raw gasoline samples from the Enefit140 and Enefit280 plants;
2. Performance of caustic treatment of mixed raw gasoline samples from the Enefit140 and Enefit280 plants according to the best procedure determined in earlier tests;
3. Performance of treatment with scavenger of mixed raw gasoline samples from the Enefit140 and Enefit280 plants using the best scavenger determined in earlier tests;
4. Shipment of prepared samples (raw gasoline from Enefit140 plant, raw gasoline from Enefit280 plant, shale gasoline treated with caustic solution and shale gasoline treated with scavenger) to the third party laboratory
5. Measure distribution of sulphur compounds in untreated shale gasoline samples taken from Enefit140 and Enefit280 plants using the GC-MS method;
6. Measure distribution of sulphur compounds in shale gasoline samples after the caustic treatment according to the most promising recipe using the GC-MS method;

7. Measure distribution of sulphur compounds in shale gasoline samples after the treatment with the most promising scavenger using the GC-MS method.

6. Guidelines for shipment and storage of samples

- Shale gasoline samples should be preferable stored and transported in closed dark glass bottles;
- Shale gasoline bottles should be nitrogen blanketed to avoid oxidation of sulfur compounds;
- Shale gasoline samples should be preferable stored in a refrigerator at around 0°C;
- Shale gasoline samples should be analyzed as quickly as possible due to possible changes in the composition.

7. Presentation of results

Results should be presented in the following table:

Sulphur concentration in different shale gasoline samples.

Sulphur Compounds	Enefit140 shale gasoline	Enefit280 shale gasoline	Enefit140 shale gasoline after caustic treatment	Enefit280 shale gasoline after caustic treatment	Enefit140 shale gasoline after scavenger treatment	Enefit280 shale gasoline after scavenger treatment
Units	ppm	ppm	ppm	ppm	ppm	ppm
Methanethiol						
Ethanethiol						
...						
...						

8. Report

Results should be presented in a report.