

## Naphthenic Acids – Inter-laboratory Study 2016

### STUDY INSTRUCTIONS

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#### DESCRIPTION and PURPOSE

The purpose of this inter-laboratory study is to assess the current status of methods for naphthenic acid (NA) analysis of aqueous samples. Specifically, the aim is to assess the various methods in use in their ability to deliver usable data in support of the National Pollutant Release Inventory (NPRI) and Toxicological studies. These samples are intended for laboratories running FTIR, GC or LC/MS systems including HRMS systems.

We are inviting laboratories to participate in this study from the government, academic, petrochemical industry and commercial laboratory communities. Prices for participation are identified in the Application Form.

#### STUDY SCHEDULE

Samples are shipped:       **2016-05-16**  
Data results are due:       **2016-06-30**

#### SAMPLE DESCRIPTION

Nine (9) aqueous samples are provided in the range of 0.0- 30 mg/L total NA as O<sub>2</sub> species. Samples will be provided in triplicate for a total of twenty-seven (27) samples.

All samples will be 'blind'.

Sample composition will consist of six (6) sets of triplicates spiked using a Merichem Commercial NA mixture and three (3) Challenge samples also provided in triplicate.

All samples are provided as 250 mL volumes.

The aqueous matrix will be one of the following;

1. A natural water sample with characteristics similar to a water found in the Fort McMurray area; or
2. A synthetic moderately hard toxicological water with characteristics similar to a water found in the Fort McMurray area.

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#### DATA REPORTING

A Merichem Commercial NA mixture is provided as the quantification reference. It is expected that laboratories will weigh out an appropriate amount to create usable standards for calibration. Please see the definitions below before reporting.

- An Excel file (***Data Report Form NA-ILS 2016.xls***) will be provided for data reporting
  - Report all Total NA results as measured, including those values which are less than Method Detection Limit (MDL) or Reporting Limit (RL).

#### DEFINITIONS:

- **Naphthenic acids** are defined traditionally as mono-carboxylic acids which include chain compounds and compounds with one or more alicyclic ring structures. Total NAs for the purpose of this study are the total  $C_nH_{2n+z}O_2$  species in the acid extractable fraction of the respective samples.
- The **Acid Extractable Fraction** is the concentration of ALL components observed in the acid extract at pH 2. The Acid Extractable fraction includes the total  $C_nH_{2n+z}O_2$  species; along with other naphthenic acids fraction compounds as defined by Headley et al. 2013.
- J. V. Headley, K. M. Peru, M. H. Mohamed, R. A. Frank, J. W. Martin, R. R.O. Hazewinkel, D. Humphries, N. P. Gurprasad, L. M. Hewitt, D. C.G. Muir, D. Lindeman, R. Strub, R. F. Young, D. M. Grewer, R. M. Whittal, P. M. Fedorak, D. A. Birkholz, R. Hindle, R. Reisdorph, X. Wang, K. L. Kasperski, C. Hamilton, M. Woudneh, G. Wang, B. Loescher, A. Farwell, D. G. Dixon, M. Ross, A. Dos Santos Pereira, E. King, M. P. Barrow, B. Fahlman, J. Bailey, D. W. Mcmartin, C. H. Borchers, C. H. Ryan, N. S. Toor, H. M. Gillis, L. Zuin, G. Bickerton, M. McMaster, E. Sverko, D. Shang, L. D. Wilson & F. J. Wrona. Chemical fingerprinting of naphthenic acids and oil sands process waters—A review of analytical methods for environmental samples. J. Environmental Science and Health A 48(10):1145-63. 2013.