

ACETONITRILE ANALYSIS IN HYDROCARBON (CRUDE C4) BY GAS CHROMATOGRAPHY

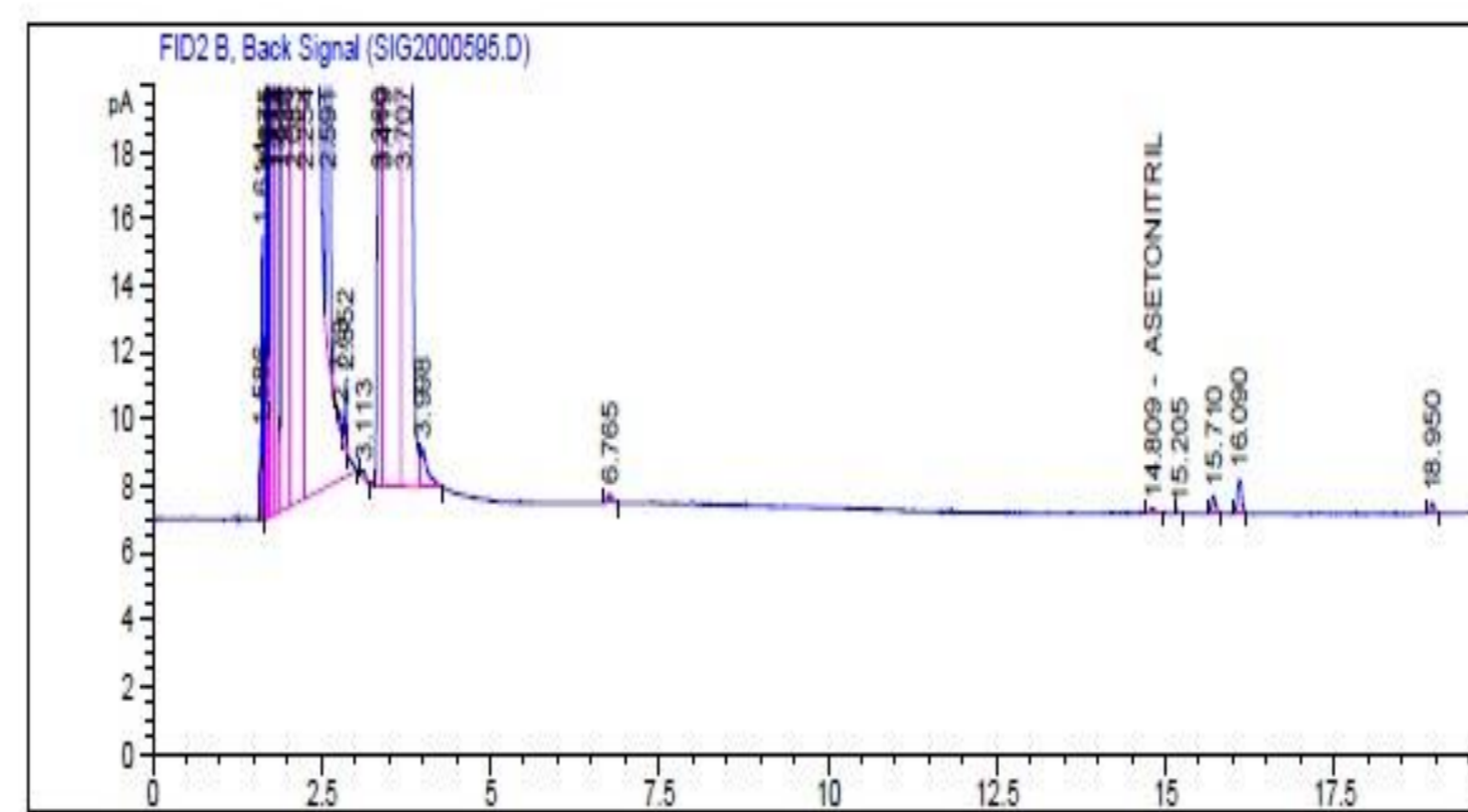


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By this study a new chromatographic method has been developed to determine acetonitrile quantity in crude C4 mix which produced by steam naphtha cracking process. Method validation studies made by a gas chromatograph with a suitable column and valid result had obtained.

Introduction

In the by-product extracted by steam naphtha cracking process Crude C4, consist of approximately 40-50 different components. Acetonitrile, one of these products, damages the process that C4 used as raw material¹. Butadiene Conjugated π system is affected by polar solvents like acetonitrile more than C4 compounds. Therefore, solvents like acetonitrile are not wanted in the system. For this reason, C4 compounds are distilled while rendered with butadiene solvent in the extractive distillation process Acetonitrile that is used as solvent causes catalyst poisoning. In this study, a new method had developed to use controlling to prevent acetonitrile leakage in the product.



External Standard Report

Sorted By : Signal
Calib. Data Modified : 1/26/2018 2:23:20 PM
Multiplier : 1.0000
Dilution : 1.0000
Sample Amount : 1.00000 [mg/kg] (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs

Signal 1: FID2 B, Back Signal

RetTime [min]	Type	Area [pA*s]	Ant/Area	Amount [mg/kg]	Grp Name
14.889	VB	6.85384e-1	24.28564	16.64499	ASETONITRIL

Figure 1. Analysis chromatogram

Experimental Methods

For the study, it is decided to use HP-5 and LOWOX Column, FID Detector and Backflush System which is found convenient by the results of trials. Because of the Liquid-Gas mix nature, it is impossible to give sample homogeneously to Gas Chromatography. So that, sample had taken into steel cylinder and pressurized with inert gas. Consistent repeatability is seen by providing sample with Liquid Sampling valve (LSV). To ensure the response time of Acetonitrile parameter in gas chromatography, pure acetonitrile fed to the GC and then 10-2000 ppm standard studies were done. By the results, it is seen that the study is successful.

RESULTS

Details

EXPERIMENTAL STEPS;

- Because of the light and the heavy components, it so important how to feed sample to GC. In the case of liquid sample feed, it is discovered that the heavy components passes to LOWOX Column and repeatability cannot ensured in this case.
- For this reason, sample had taken into steel cylinder and pressurised with inert gas and fed column by LSV.
- It is provided the sample fed to HP-5 Column for disposing of the heavy fractions and it is provided to feed light fractions to LOWOX Column.
- Also because of the heavy fractions, considering the sample compositions it is decided to use backflush system and time shift was done.
- It is seen that when the sample fed to the LOWOX Column just nitrile and oxygenated light components were fed by the help of Backflush system.
- To confirm retention time (RT) and response factor (RF) of acetonitrile, analysis were done in different solvents.
- Because of the different RT and RF in every different solvents, standards had prepared as the same as real sample.

Details

EXPERIMENTAL SET-UP

- Agilent Technologies 7890B
 - GSV : Gas sampling valve
 - LSV : Liquid sampling valve
 - ALS : Automatic Liquid Injection
- Backflush

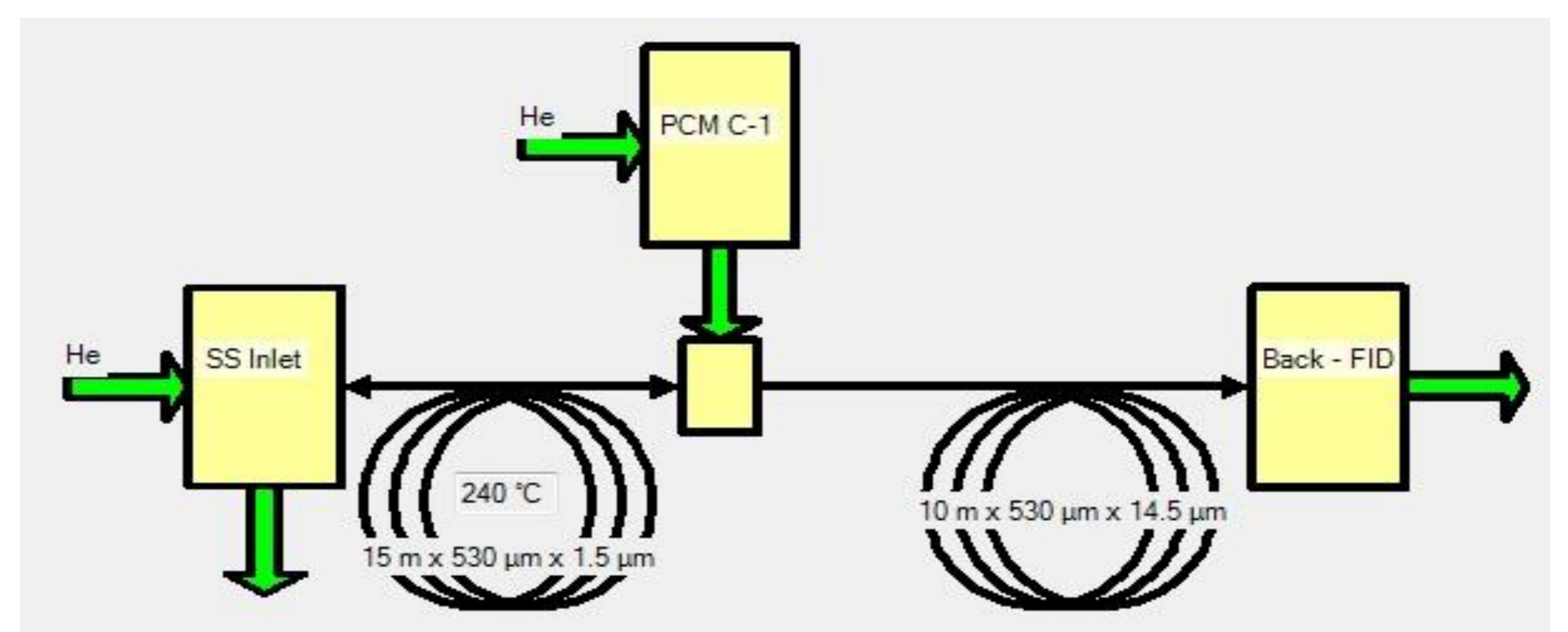


Figure 2. Backflush system

- FID Detector
- HP-5 & LOWOX Column

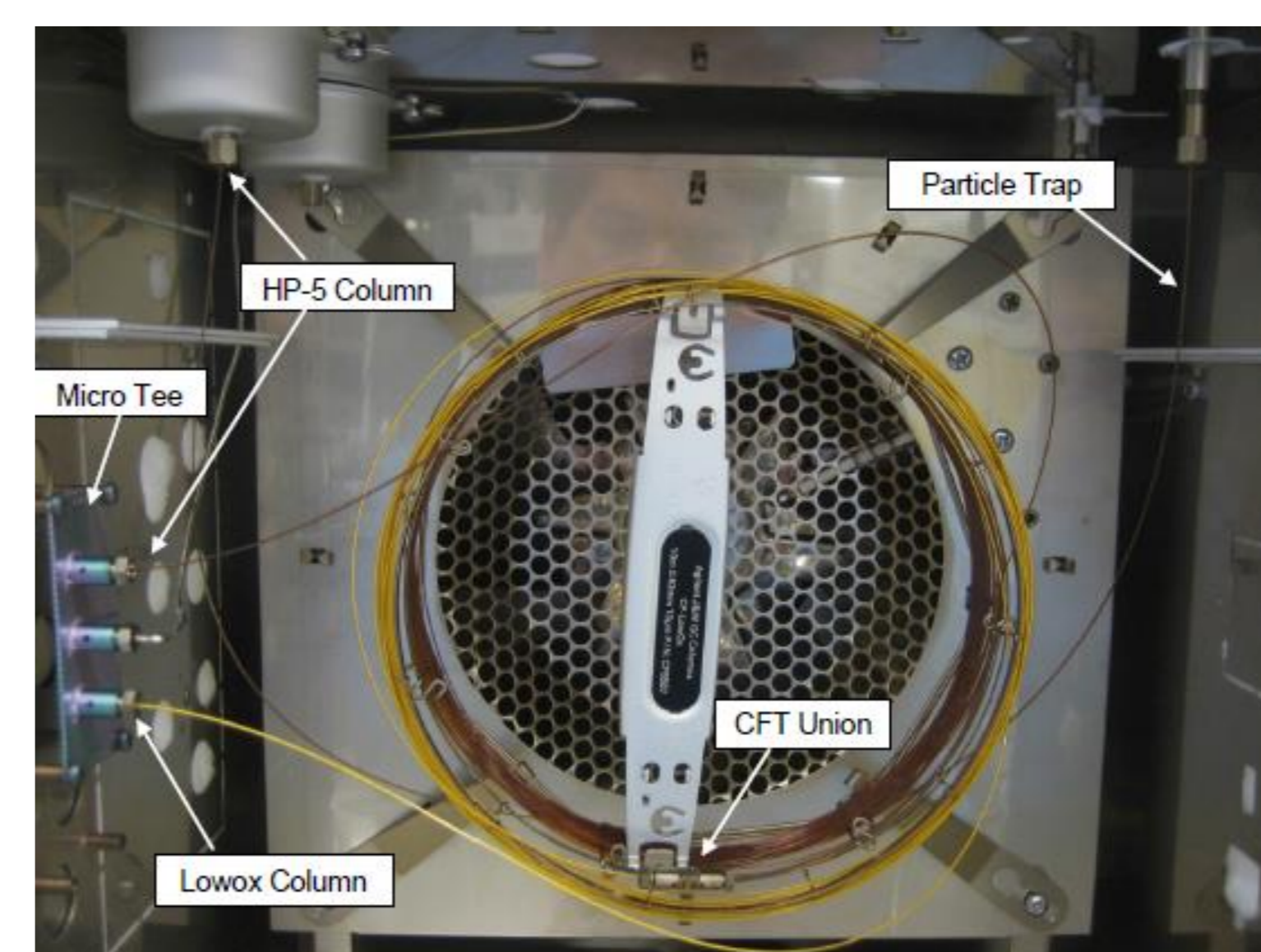


Figure 3. Instrument configuration

Conclusion;

Acetonitrile as up to 3 ppm has dedected in crude C4 with appropriate system configuration and right system parameters.

Acknowledgements

1- Survey of industrial Chemistry – Philip j. cehnier University of Wisconsin-Eau Claire