

Total Sulfur and Nitrogen Calibration for Gaseous Hydrocarbons and LPG using the Multiloop feature of the Accura sampling device

- Rapid and accurate determination of volatile sulfur and nitrogen
- Fully automated sample introduction and combustion system
- Excellent sensitivity, repeatability & linearity

Keywords: ASTM D6667, UOP 936, ElemeNtS, Accura, Sulfur, Nitrogen, UVF, LPG

INTRODUCTION

In this technical note we will discuss the use of the Multiloop feature of the Accura sampling device. Using this feature, a calibration for gases on the ElemeNtS can be done with the use of just one calibration standard.

Sulfur and Nitrogen can be present in LPG or gases, used for fuel purposes, through the production process or naturally as well as a component of an odorant. In both cases there is a need for measuring the total Sulfur and Nitrogen content for regulatory compliance determination.

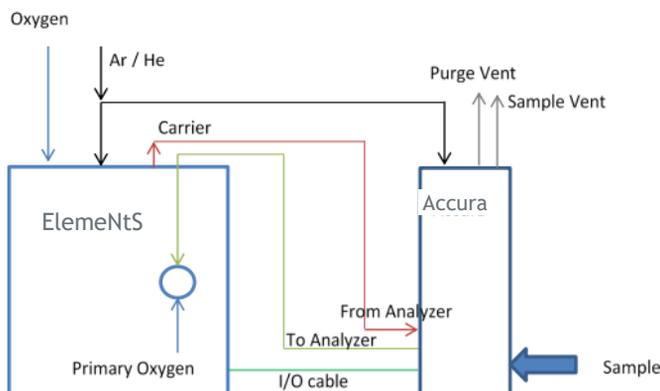
LPG or gases used as a feedstock for various processes should also be monitored for the Sulfur and Nitrogen content, as some process catalysts used in petroleum and chemical refining can be poisoned by Sulfur or Nitrogen bearing materials in these feedstocks.



SAMPLE INTRODUCTION

A gaseous hydrocarbon or LPG sample is introduced into the ElemeNtS by the automated Accura sampling device. The Accura has a vaporizer to convert the liquified gas (LPG) into a gas. After passing the vaporizer the gas or vaporized LPG enters a heated gas sampling valve. When the injection trigger is given from the ElemeNtS analyzer, the valve is switched automatically, and the content of the sample loop transferred to the ElemeNtS with the carrier gas. An additional flow controlled by a built-in Mass Flow Controller is added to ensure there is sufficient flow to empty the sample loop. Additionally the Multiloop feature enables the Accura to inject multiple loops of sample within one analysis.

For calibration purposes, gases with a known concentration of Sulfur and/or Nitrogen should be used. The calibration gases can be introduced into the ElemeNtS in the same way as samples are introduced, by direct connection to the Accura.



MULTILOOP PRINCIPLE

The Multiloop feature of the Accura enables injection of the sample multiple times within the same analysis. Using this principle, a complete calibration can be created using just one calibration gas standard. The use of more than one calibration gas is analytically recommended, but very costly. The cost-effective Multiloop option provides significant savings on calibration gases.

After the first injection the Multiloop injection feature will automatically switch back the sample loop to the load position to fill and inject the sample loop again. Depending on the setting (1-99), this will be repeated. The analysis and integration times need to be optimized for the amount of injection cycles. The total area of all peaks will be combined in the software to generate a calibration curve.

The Multiloop option is only used for the calibration of the ElemeNtS. Regular samples are injected once.

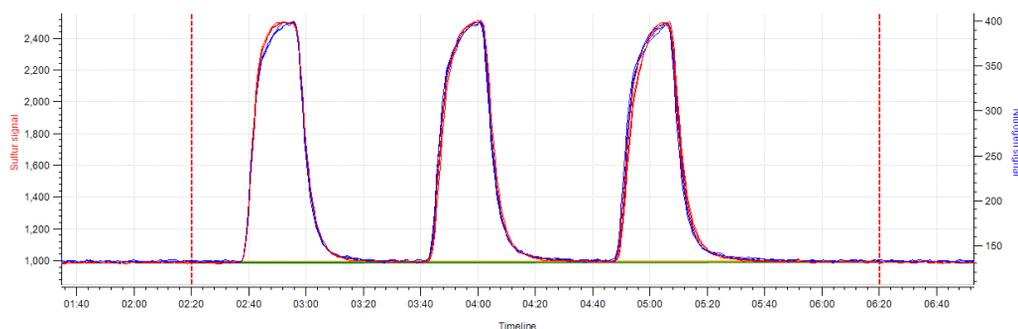


Figure 1: Example of Multiloop injection.

SETTING UP THE ACCURA AND ELEMENTS FOR MULTILOOP INJECTION

To set up the amount of Multiloop injections on the Accura, go to the “Method” screen on the user interface of the Accura. Settings such as the vaporizer temperature, loop temperature and MFC flow can be set, as well as the amount of Multiloop injections. To set the amount of Multiloop injections, click on the number and change it to the desired quantity.

In the IRIS ElemeNtS plugin open the “Method editor” and go to the “Gas” tab to create a new or edit an existing gas method. Instrument settings such as Flows, Temperatures and High Voltage settings can all stay default. The cycle time and integration settings should be extended to include all injected peaks. A method with ten Multiloop injections will take about 15 minutes. Note that this settings will also apply to the regular sample injection, taking 15 minutes for one sample injection.



Figure 2: Accura method screen.

CALIBRATION

The ElemeNtS was calibrated using the Multiloop option. A calibration gas containing 15 ppm Sulfur and Nitrogen was injected 1, 2, 3 and 6 times respectively. This corresponds to total concentrations of 15, 30, 45 and 90 ppm used to create the calibration curve.

Table 1: Calibration data Multiloop

Calibration Multiloop 1-6		
Concentration (ppm)	S counts	N counts
15,0	29411	5126
30,0	58031	9834
45,0	86493	14544
90,0	171950	28718
Intercept		
	972	398
Slope		
	1900	315
R2		
	1,0000	1,0000

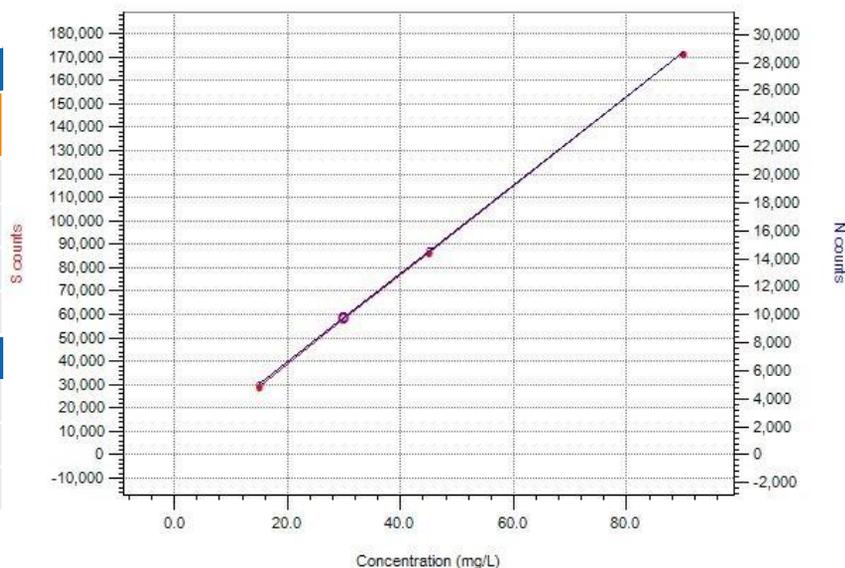


Figure 2: Calibration curve Multiloop.

CONCLUSION

The Multiloop feature of the Accura enables injection of the sample multiple times within the same analysis. It is a cost-effective solution to calibrate the ElemeNtS analyzer using just one calibration gas. A calibration performed with the Multiloop option offers excellent linearity and is possible because the ElemeNtS instrument has a very broad linear dynamic detector range.

The Multiloop option is no direct replacement for a normal multi-level calibration, as it does not correct for sample specific interference induced by increasing sample concentration. It does offer a proven cheaper alternative for a conventional calibration using multiple gases.

Antek's lab instruments provide reliable, precise elemental analysis for total nitrogen and sulfur, speciated nitrogen and sulfur, fluoride, chloride, and bromide. Antek products are recognized by global regulating bodies, leading scientific research institutions, and process laboratories as the instrument of choice for selective multi-element detection.