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The Mini Gas Splitter: SCION's Special Tool to Improve Analytical Accuracy

Application Note AN0028

INTRODUCTION

The importance of sulphur removal from various energy sources can be beneficial for many reasons, such as toxicity, corrosion and environmental pollution. Before crude oils are distilled or coal is converted for the use of fuels, sulphur is first removed from the raw feed streams. H_2S is formed during these conversions and treatment steps. When natural gas is extracted from their sources, the gas is treated in gas treatment plants before being used as an energy source. The H_2S, COS and other organic sulphur in the gas are removed from the raw materials.

The analysis of sulphur containing components in different industrial gases is a widely performed measurement in an industrial environment. The methods for the measurement of the sulphur containing compounds can be very broad. The analytical method used is dependant on the range and composition of the gas.

Gas chromatography with pulsed flame photometric detector (PFPD) is usually the method of choice especially where low and ultra low (ppb) levels are to be determined.

Handling gaseous streams contaminated with low levels of sulphur special requires special attention, mainly because of the reactive nature of the components; they tend to be adsorbed onto most materials. The analytical effect is more drastic at lower concentration levels. Attention needs to be given to the transport of the gas; sampling into specially treated containers, transfer into and transport through the analytical device.

This application note discusses the SCION mini gas splitter used to improve analytical accuracy.

DISCUSSION

Once inside the GC, the sample may experience a number of locations where potentially irreversible adsorption may take place before it reaches the detector. The detector is where the concentration is determined; losses in the sample pathway prior to the detector will not be detected, leading to errors in the final reported results.

At SCION Instruments we take great care in designing such analytical solutions. By making use of inert material, deactivated steel transfer tubes and fused silica separation column and avoiding large volumes in the sample path we reduce all adsorption sites in the sample flow path to deliver the highest level of sample transfer to the detector. Figure 1 shows the mini gas splitter used to improve analytical accuracy.

Fig 1. SCION mini gas splitter

The detector used by SCION is the sulphur specific PFPD, operated in sulphur mode (S-mode). This detector shows high sulphur specific response with almost infinite sulphur/carbon selectivity.

The SCION mini gas splitter is used to handle the sample on route to a capillary column, once it has passed via a large sample loop and valve. The reason for the loop to be large is to allow for sufficient sample sensitivity for trace analysis. If the loop and valve were stainless steel they would absorb sulphur, but because the injection valve and loop are produced out of inert Hastelloy-C, no sample adsorption takes place during the injection process. The mini gas splitter serves as the alternative for a standard injector. The advantage being its small size and use of deactivated material. The use of EFC 25 guarantees optimal splitting characteristics as you would expect from a standard injector.

The separation column is a 60 meter, 5µm SCION 1 column; a thick film column avoids adsorption onto the column material. Figure 2 shows an overlay of 2 chromatograms of a sample containing 11ppm each of H_2S, COS and CH_3SH in nitrogen; one sample was analysed using the mini splitter and one without.

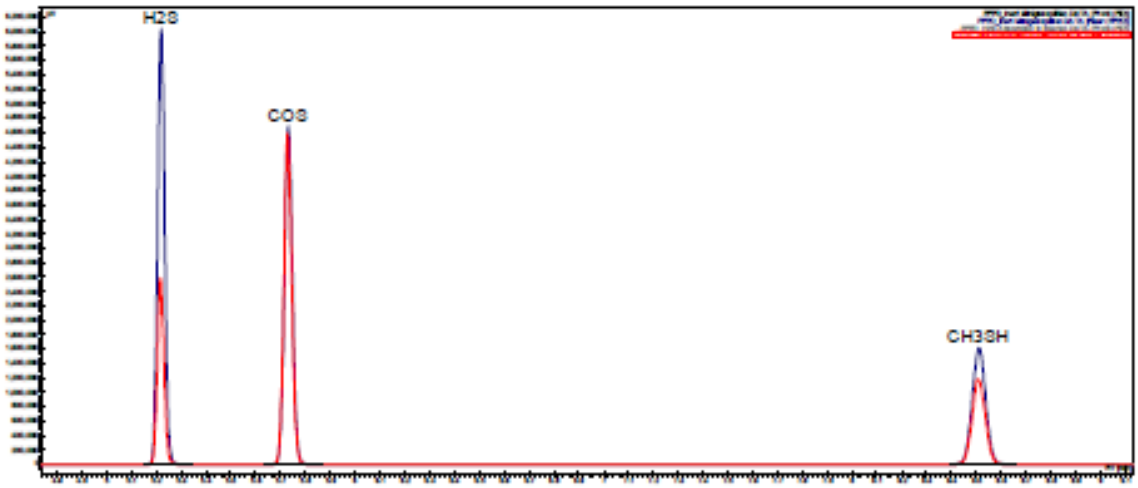

Fig 2. Chromatogram overlay of the same sample; with and without the mini splitter

The blue chromatogram shows analyte detection when the mini gas splitter was used with the red chromatogram showing detection without the mini gas splitter. Repeatability of results using the mini gas splitter was evaluated over three injections. Table 1 details the repeatability data from the three injections.

In combination with the observed equimolar response towards sulphur proved that no adsorption occurs when using the mini gas splitter.

Table 1. Repeatability data; area (mV/sec)

	H_2S	COS	CH_3SH
Run 1	2090.2	2109.8	2056.6
Run 2	2067.6	2101.1	2049.7
Run 3	2047.4	2060.9	2050.1
Average	2068.4	2090.6	2052.1
RSD%	1.04	1.25	0.19

CONCLUSION

From this short study in which the analysis of sulphur components was examined in more detail the following may be concluded:

- With the use of the mini gas splitter adsorption has been eliminated
- Reporting analysis results with an error is avoided
- Use of the PFPD provides equimolar response to sulphur and provides good repeatability

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