



Shell Research and Technology Centre, Amsterdam

Analytical Services Amsterdam

To: O'Brien Bvba
Mallekotstraat, 65
2500 Lier
Belgium

Technology and Innovation Support
P.O. Box 38000
1030 BN AMSTERDAM
The Netherlands

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Scope

Testing electro polished and electropolished silica steel coated tubing on adsorption of methylmercaptane.

Samples

- 1- SAMPLE 1: standard RVS weldless 316L SS 1/8"x.020 316L SS smls, received: 18-12-2003
- 2- SAMPLE 2: 1/8"x.020 TrueTube™ EP, received: 18-12-2003
- 3- SAMPLE 3: 1/8"x.020 TrueTube™ EPS, received: 18-12-2003

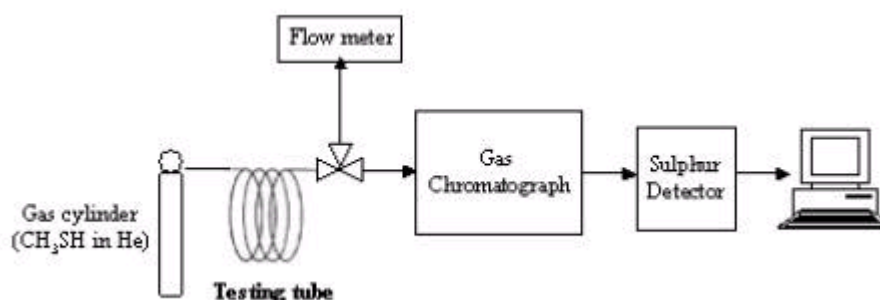
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Experimental procedure

The presence of sulphur compounds in the petroleum industry and other chemical processes causes serious problems in the refinery. One of the problems faced in these industries is for example the corrosion of the equipment due to adsorption of sulphur. Therefore the identification and removal of sulphur is very critical. In few of the foregoing, it is useful to provide a tool to identify and quantify of sulphur compounds. There are numerous sulphur-selective detection techniques commercially available of which the Sulphur Chemiluminescence Detector (SCD) is most favorable in its whole performance. Thus, an analytical technique for selective detection and measurement of sulphur has been set-up. This technique could be useful in determining whether there is a difference in adsorption of a certain sulphur gas compounds i.e. methylmercaptane on different tubings. These are made of materials which have been subjected to different treatments. To reveal the adsorption on different materials, the following three tubings i.e. standard Stainless steel (RVS) weldless 316L SS 1/8"x.020 316L SS 1/8"x.020 TrueTube Electro Polished (EP) and 1/8"x.020 TrueTube Electro Polished Silica lining (EPS) have been subjected to 0.5 ppmv S methylmercaptane in Helium. A schematic outline of the experimental set-up is given in the figure below.



The proposed set-up involves the coupling of the testing tubes (i.d. of 1/8") directly between the gas cylinder with the reference sulphur compound methylmercaptane and an automatic injection valve. The methylmercaptane flowing through the testing tube is injected with a frequency of e.g. every 0.4 minutes, onto a gas chromatograph. The response signal from the gas chromatograph is detected with a sulphur specific detector (SCD) and monitored on a desktop. Dependent on the signal this test might run for e.g. 12 hours (which will yield 1800 data points) and provides information on the adsorption characteristics of the steel surface during first exposure and under steady state conditions. After this period, the cylinder containing the methylmercaptan is replaced by a pure helium supply and the same measurement sequence is repeated. This test provides information on the desorption behaviour of the steel surface.

This test sequence has been carried out for the three different grades of steel tubing supplied.

Equipment:

Gas chromatography equipment consists of:

- A sample introduction system, sample loop (250 μ L) and Valco valve
- A capillary system, Fused SilicaPlot (5m) column and a carrier gas He.
- A detection system, SCD
- H_2 , O_2 en He utilities with a constant flow.
- A data acquisition system, EZ-chrom Elite.

Materials:

- Carrier gas He of quality at least 5, 99999% purity
- Sulfinert material has been used for the whole system
- A gasbombe with methylmercaptane with the following conditions has been used:
 - a 10 liter w.i. 150 bar cylinder with methylmercaptane in Helium
 - concentration: 0.5 ppmv S
 - standard deviation: +/- 20%

Results and Discussion

Different tubings have been subjected to methylmercaptane to reveal their adsorption behaviour. For the results see Figure 1 below. The figure shows on the x-axis the retention time (min) e.g. the run time each tubing has been subjected to sulphur and on the y-axis the methylmercaptane signal e.g. the amount sulphur detected with the SCD.

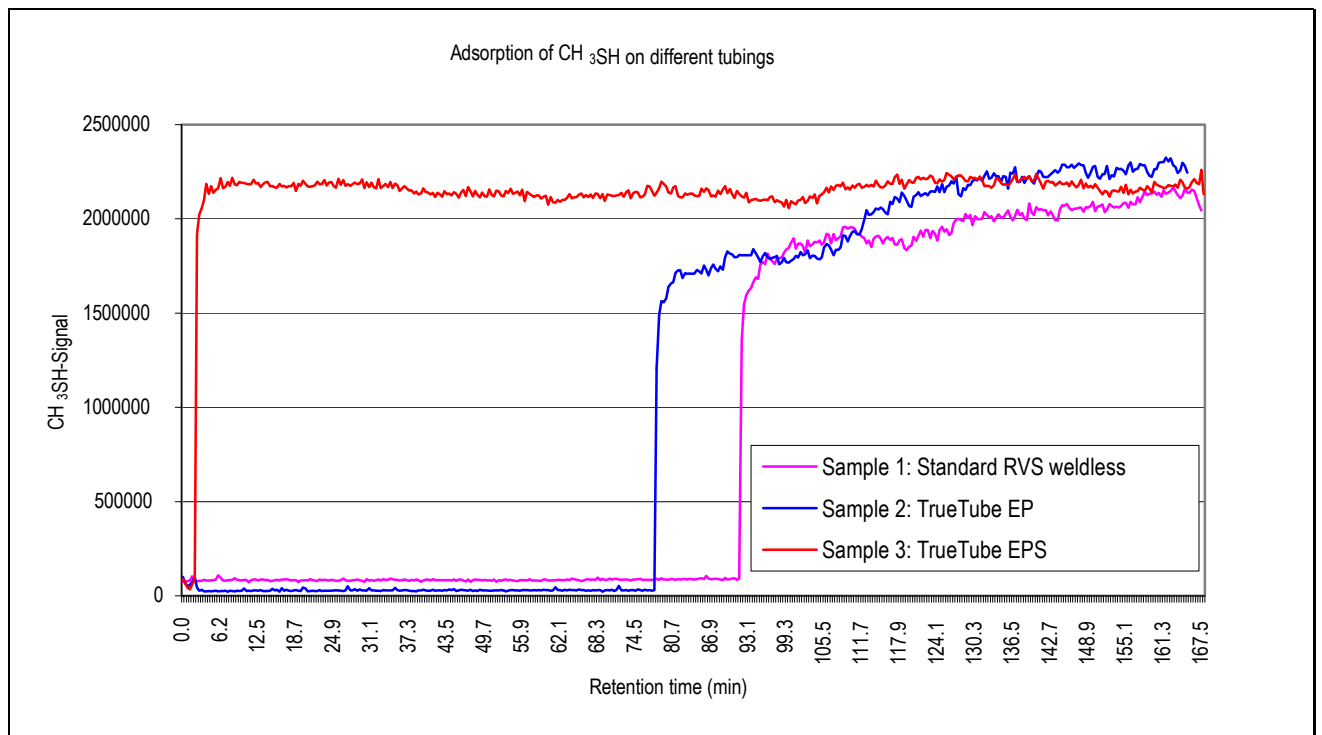


Figure 1: Adsorption behaviour of methylmercaptane on different tubings.

As can be seen from the results in Figure 1, there is a difference in methylmercaptane adsorption on the different tubings. As can be seen in Figure 1, TrueTube EPS an immediate signal has been detected after just two minutes. On the contrary, the TrueTube EP and the Standard RVS weldless a signal after subsequently 78 and 92 min. This means that the TrueTube EPS releases sulphur immediately and has the least tendency to adsorb sulphur than the other two tubings. However, the TrueTube EP on its turn adsorbs less sulphur than the Standard RVS weldless tubing. No major differences have been monitored after the release of sulphur the signal of methylmercaptane for the three tubings remains constant.

The effect of desorption is demonstrated in Figure 2(a+b) below. The axes indicate the same as for the adsorption figure. As can be seen from Figure 2, the TrueTube EPS shows the least sulphur release upon flushing with Helium because less sulphur has been adsorbed on this tubing. On the contrary, the TrueTube EP and the Standard RVS weldless tubings show the highest sulphur release upon flushing with Helium because a large amount of sulphur has been adsorbed on these two tubings. However, there is a slight difference in sulphur desorption of the later two tubings meaning that the TrueTube EP release on its turn sulphur easier than the Standard RVS weldless tubing.

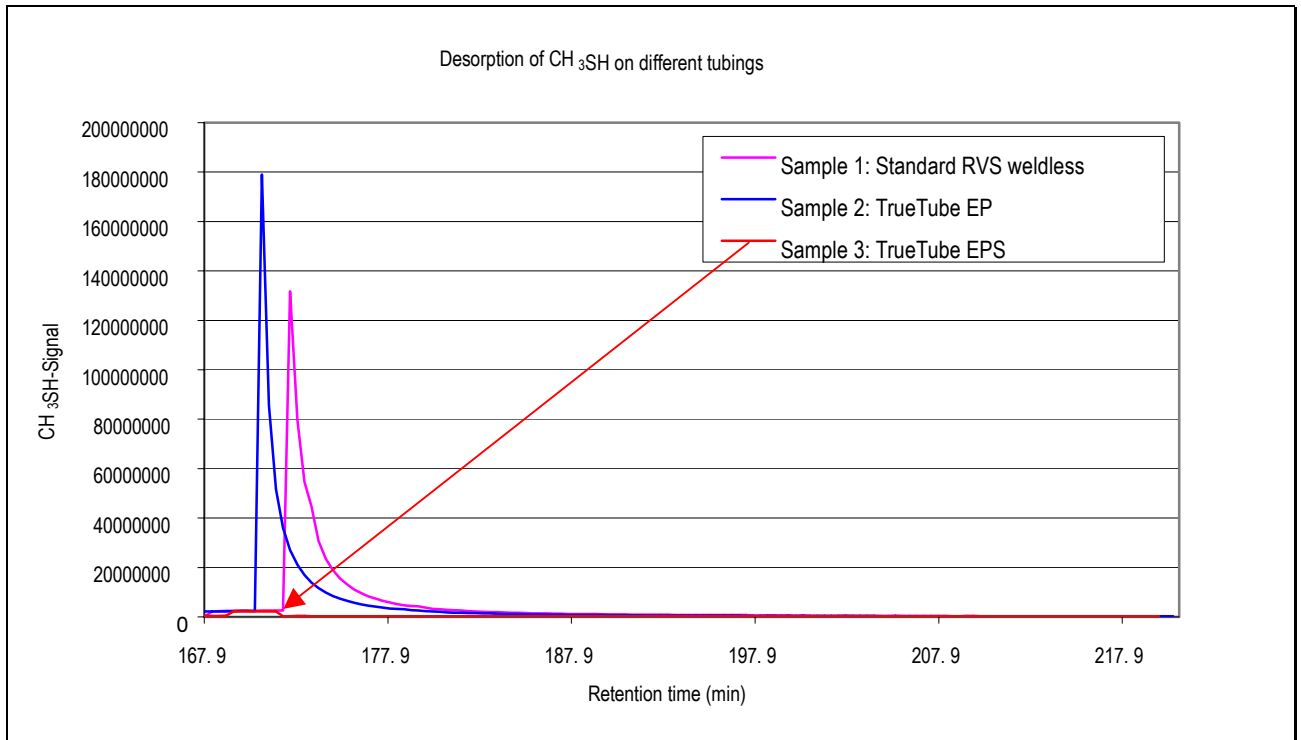


Figure 2a: Desorption behaviour of methylmercaptane on different tubings.

A better visualization of the difference in desorption of the TrueTube EPS compared to the other two tubings is demonstrated in Figure 2b below.

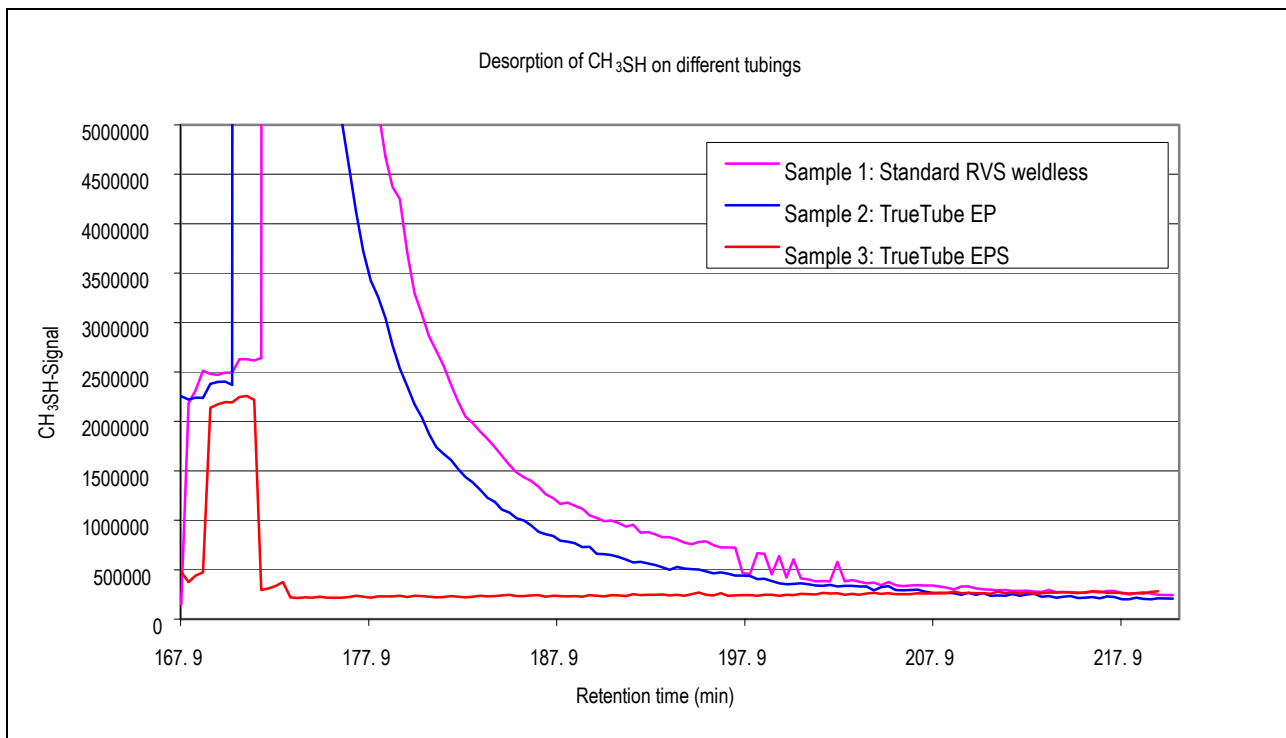


Figure 2b: Desorption behaviour of methylmercaptane on different tubings with other y-axis.

Conclusion

- The adsorption tendency of sulphur increases subsequently from TrueTube Electro Polished Silica lining (EPS), TrueTube Electro Polished (EP) to Standard Stainless Steel (RVS) weldless tubing.
- The TrueTube Electro Polished Silica lining (EPS) shows a barely desorption effect because no sulphur has been adsorbed.
- TrueTube Electro Polished (EP) releases sulphur easier than the Standard Stainless Steel (RVS) weldless tubing.
- It seems that Electro Polished tubing with Fused Silica treatment is the best way of preventing sulphur from adsorption to these tubings.