

# SELECTIVE ANALYSIS OF TRACE LEVEL CARBONYL SULFIDE IN PROPYLENE BY GAS CHROMATOGRAPHY AND CHEMILUMINESCENCE AS ALTERNATIVE DETECTION FOR ASTM D5303

## INTRODUCTION

In processes producing propylene, trace level carbonyl sulfide (COS) acts as a poison to commercial polymerization catalysts, resulting in deactivation and costly process downtime. The PAC SeNse Chemiluminescence Detector is the most advanced GC Detector available for the selective determination of sulfur containing compounds in hydrocarbon matrices.

Standard methods like ASTM D5303 also address this analysis but is limited towards the analysis of COS within the range of 0.5 to 4.0 ppm mass.



AC SeNse: Sulfur Chemiluminescence Detector

## APPLICATION DEVELOPMENT

The current application is developed to obtain lower detection limits / broader application range compared to ASTM D5303. The analytical set-up is based on the SENSE Chemiluminescence Detector to allow optimum selectivity and lowest detection limits well below 30 ppbM COS in propylene. To broaden the analytical scope the application is developed on a boiling point column to allow compliance with ASTM D5504 type of applications.

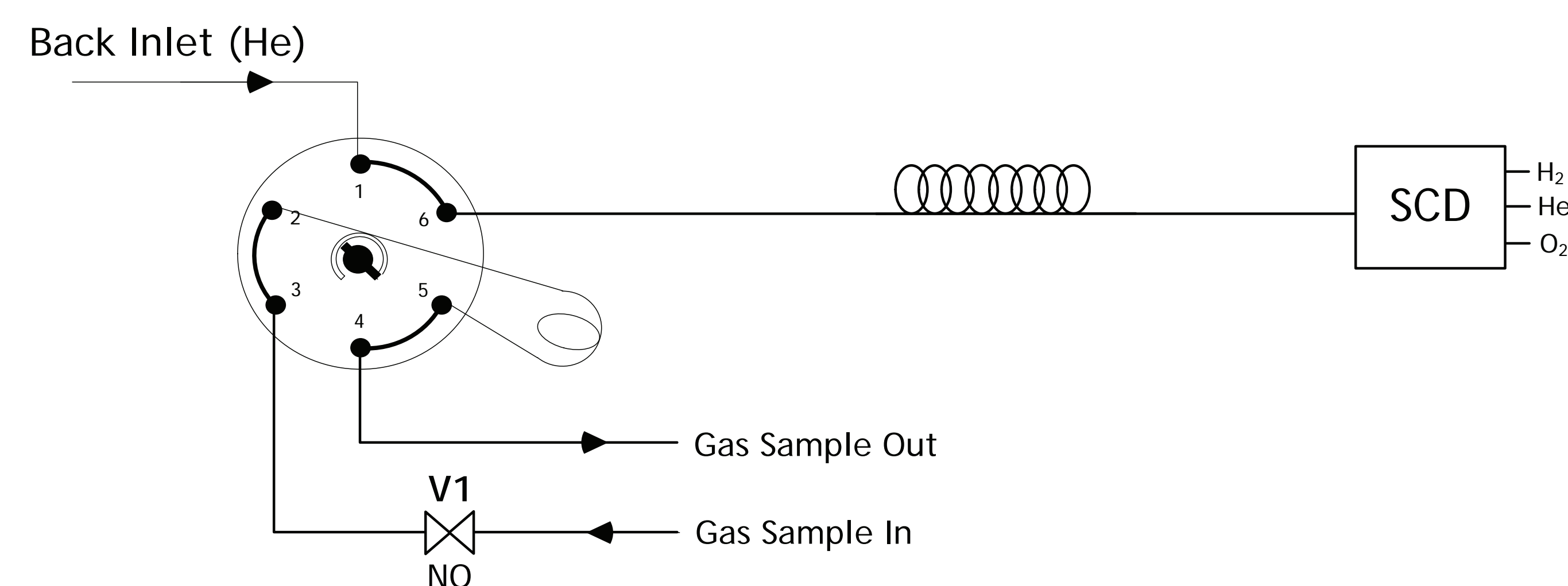


Figure 1: COS in Propylene - Flow diagram

## ANALYTICAL CHALLENGE

The determination of COS in propylene is the most challenging because COS is not separated from the propylene matrix. On many detectors COS response is heavily affected by the propylene matrix which results in erroneous results; particularly when going to the often desired lower levels of 30 ppb Mole

## SELECTIVITY / DETECTION LIMIT

Selectivity and detection limit are tested on 15 ppbM COS in propylene by diluting a sulfur standard with pure propylene. A matrix analysis of pure propylene is compared with the 15 ppbM COS analysis (Fig. 2). No baseline disturbance is observed. Quantitative results are obtained by use of external standard calibration.

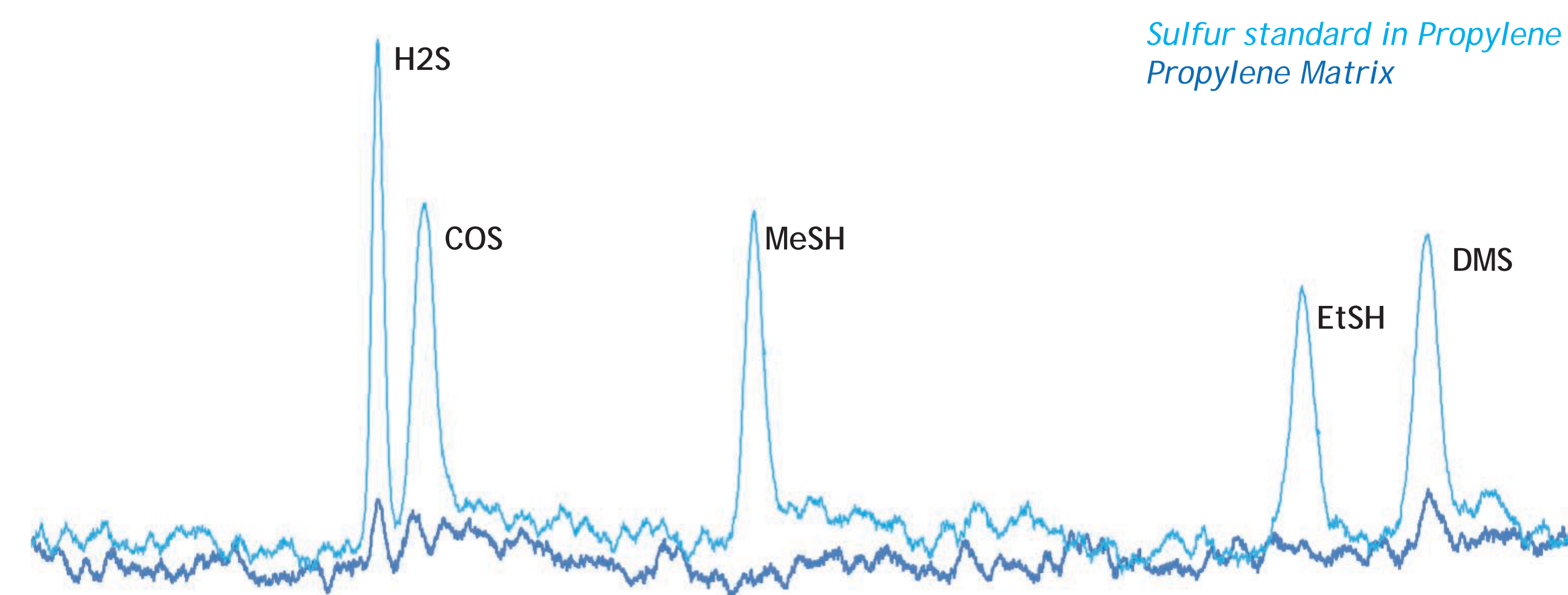


Figure 2: Chromatogram of 15 ppbM COS in Propylene matrix

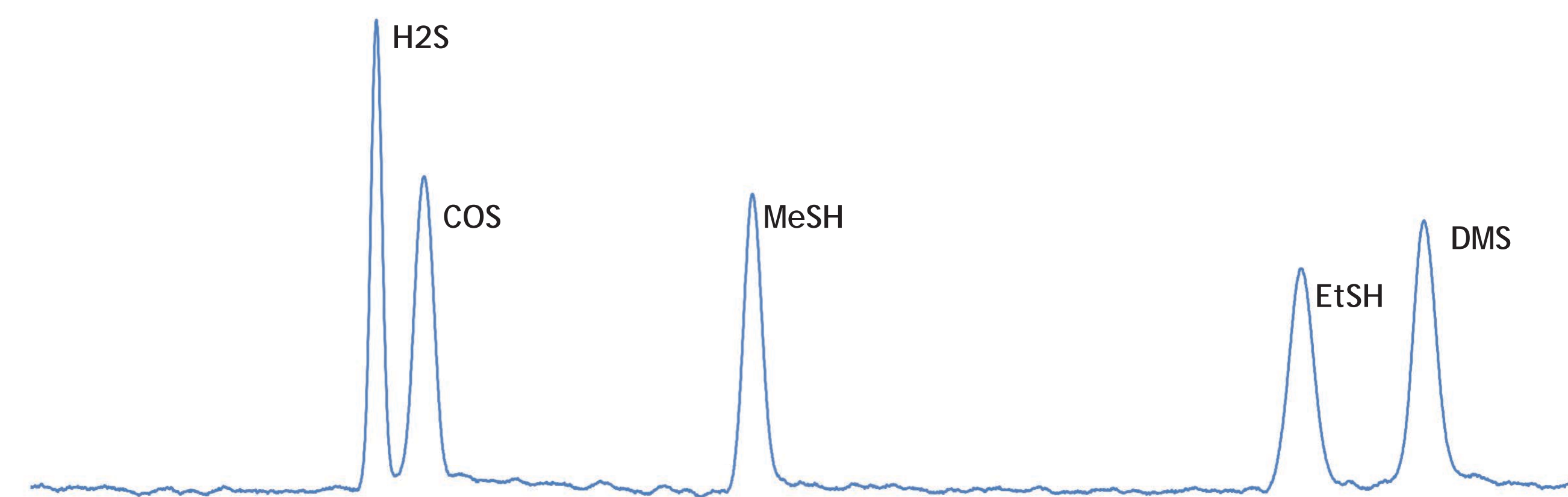


Figure 3: Chromatogram of 60 ppb M COS in Propylene matrix

## RECOVERY CARBONYL SULFIDE IN PROPYLENE

To determine the recovery / influence of propylene matrix at low levels a 1.5 ppmM sulfur calibration mixture (N2 balance) is diluted with propylene by dynamic dilution using 2 mass flow controllers to 60 ppbM. (Fig. 3)

The found recovery is compared to the target value based on the response factor obtained from a 1.5 ppmM Sulfur calibration standard (Nitrogen balance) (Table 1)

Component	Target Value (ppb M)	Found Value (ppb M)	Recovery
H2S	62.3	60.8	98%
COS	63.1	64.2	102%
MeSH	61.0	61.1	100%
EtSH	61.9	61.4	99%
DMS	61.4	67.1	109%

Table 1: Recovery COS in Propylene calibrated with a Sulfur calibration standard (Nitrogen balance)

All sulfur compounds including the coelution of carbonyl sulfide with propylene recovered with 10% of the target value.

## TRACE LEVEL CARBONYL SULFIDE IN PROPYLENE

- LDL COS in Propylene < 30ppbM
- Selectivity S/C > 5 10e7
- Also fits ASTM D5504 application

SeNse Sulfur Chemiluminescence Detector:  
Best in market for COS propylene

