

The Analysis of Trace Level Sulfurs in Beverage Grade CO₂

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Project Objective

- Develop a robust micro-packed column for the analysis of trace level sulfurs in CO₂ with the following critical characteristics:
 - Thermal stability (310°C)
 - Inert (sub ppb levels)
 - Low bleed (< 20pa @ 310°C)
 - High sample capacity (15,000 ng)
 - Rapid analysis
 - Column longevity

Porous Polymer Column Optimization

- Porous polymer surface modification for the maximum degree of inertness
- Particle classification resulting in a tighter sample bandwidth
- Must resolve SO₂ from all other target sulfur compounds (TSC)

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Column tubing considerations

- Low ID RMS value resulting in increased theoretical plates/efficiency
- Sulfinert deactivation resulting in state-of-the-art inertness
- Flexibility, easily installs in any GC
- Unreactive to sulfurs @ 20 ppbv levels

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Advantages of the Packed Sulfinert™ column

- No cryogenic oven cooling required
- Highly breakable glass column eliminated
- Critical resolution of SO₂, H₂S & COS
- Column has the flexibility to be installed in any model GC
- Cost effective

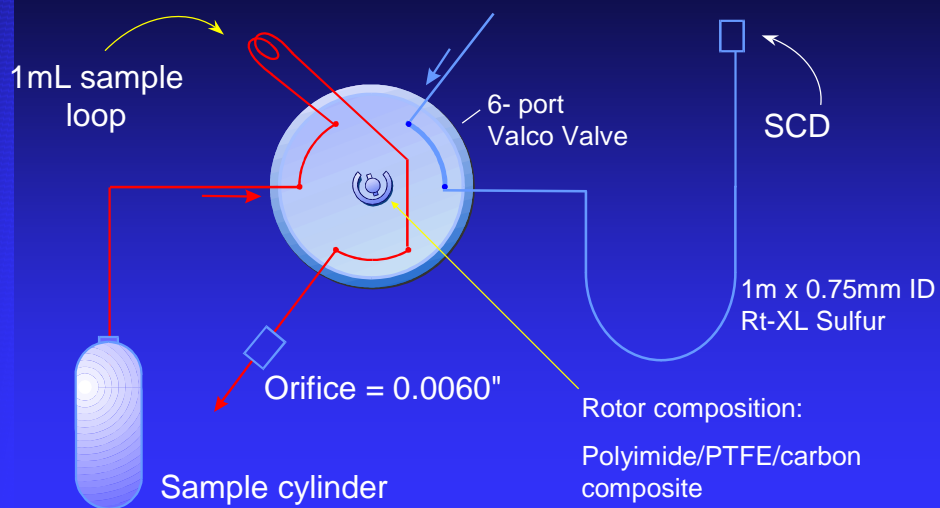
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GC system requirements

- Detection limit of < 0.02 ppm for SO₂ plus the other TSC
- Method of detection: SCD, HECD, FPD or PFPD detector
- All wetted sample pathways Sulfinert™ passivated for maximum inertness

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Sulfinert™-Treated Inlet System



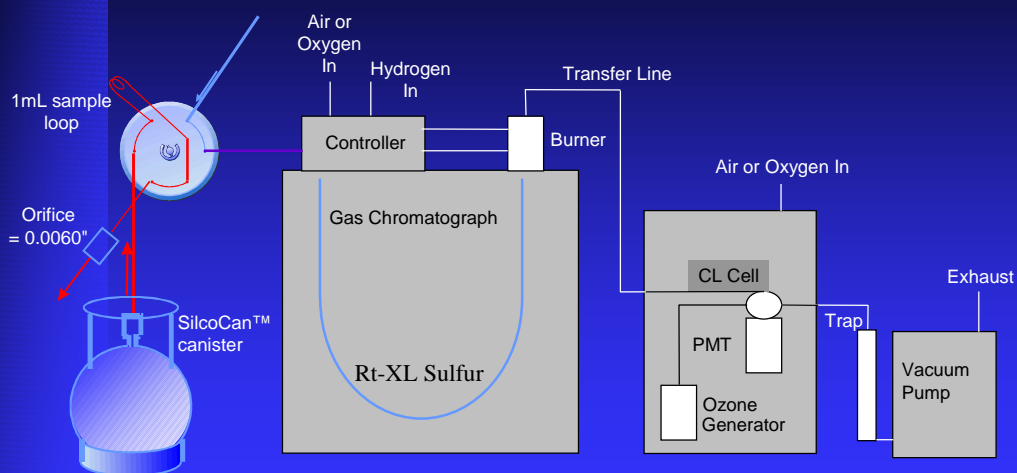
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Chromatographic Parameters for the Rt-XL Sulfur Column

- 1 meter x 0.75mm ID micro-packed column with Sulfinert™ deactivation
- 10mL/min. helium
- 60°C > 260°C @ 15°C/min. > hold 10 min.
- GC/SCD (Sievers) Detection

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Block diagram of Analytical System



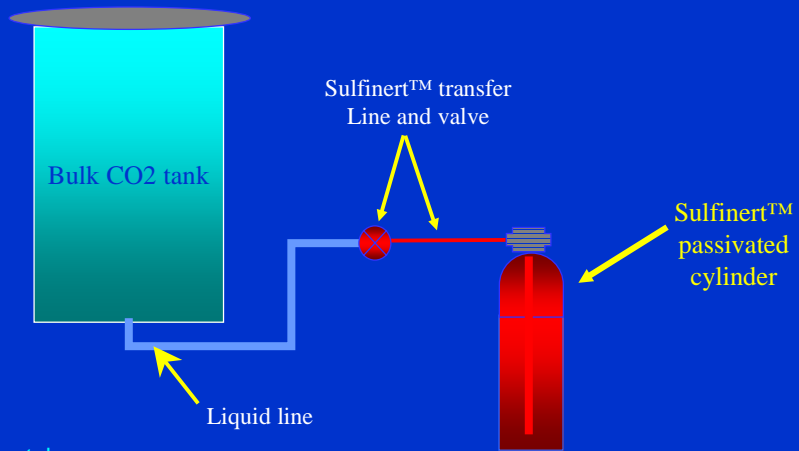
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Sampling system integrity for CO₂ liquid sample acquisition

- Passivated vessel to collect the liquid CO₂, conforming to the International Society of Beverage Technologists (ISBT) procedure 1.0
- Connections between the bulk CO₂ and the sample vessel must be passivated to prevent adsorption of the sulfur compounds

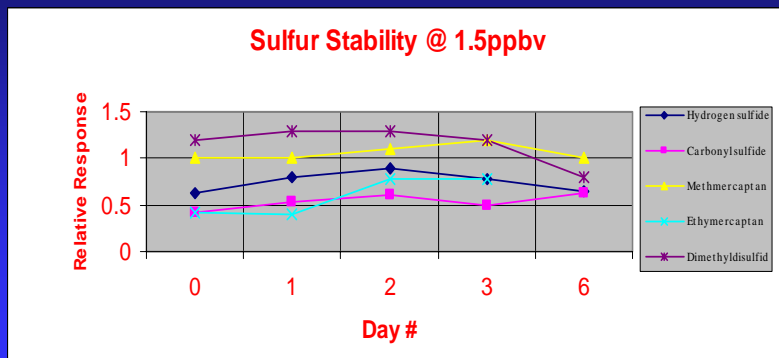
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Sample Acquisition System



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Sulfinert™ Vessel Inertness



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Response Factors @ 1.5ppbv

	Day 0	Day 1	Day 2	Day 3	Day 6		
	RRF/DMS	RRF/DMS	RRF/DMS	RRF/DMS	RRF/DMS		% RSD
Hydrogen Sulfide	0.62	0.9	0.85	0.77	0.7		14.6
Carbonyl Sulfide	0.42	0.53	0.6	0.5	0.62		15.07
Methyl Mercaptan	1	1	1.1	1.2	1		8.43
Ethyl Mercaptan	0.42	0.39	0.77	0.77	0.35		39.15
Dimethyldisulfide	1.2	1.3	1.3	1.2	0.8		17.87

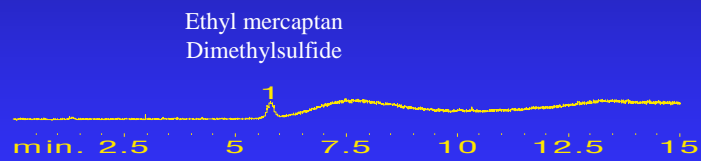
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Beverage Sampling Data

- Beverage grade CO₂ blank
- 20ppb sulfur standard in beverage grade CO₂
- 20 ppb SO₂ in beverage grade CO₂ standard
- Headspace analysis of beer
- Analysis of a hard lemon alcoholic beverage
- Analysis of a top brand of cola

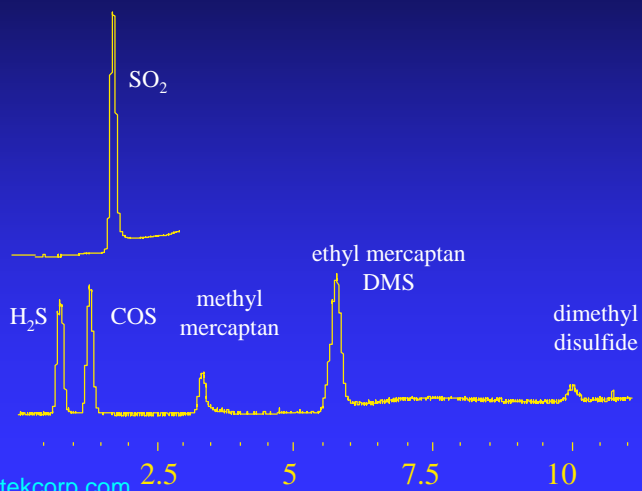
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Beverage Grade CO₂ blank



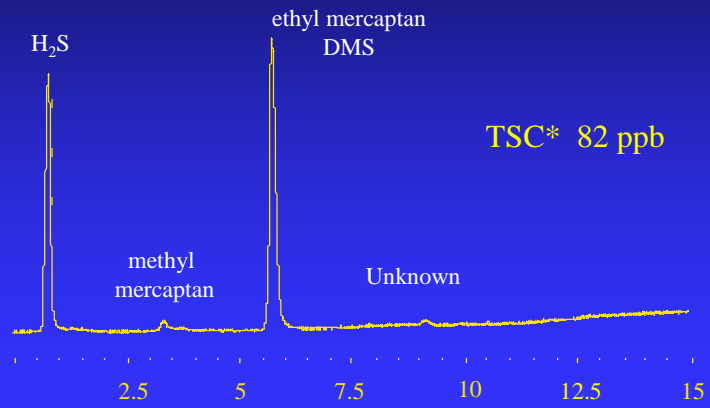
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20 ppb Sulfur Standard in Beverage Grade CO₂



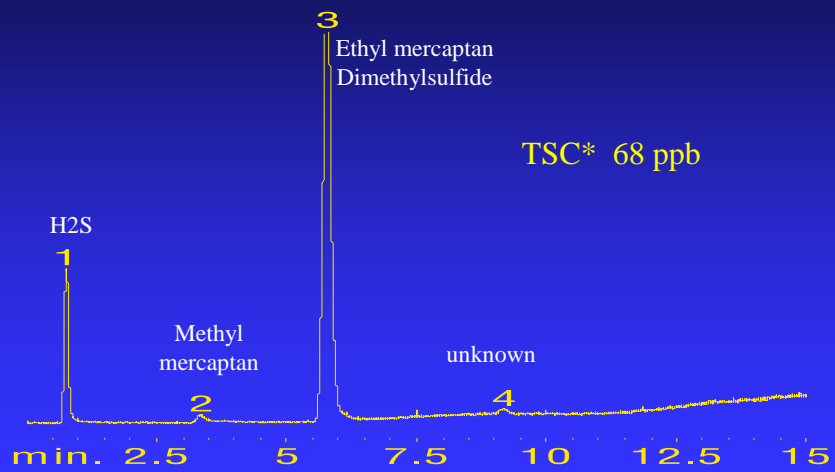
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Brand "B" Beer Headspace Sample



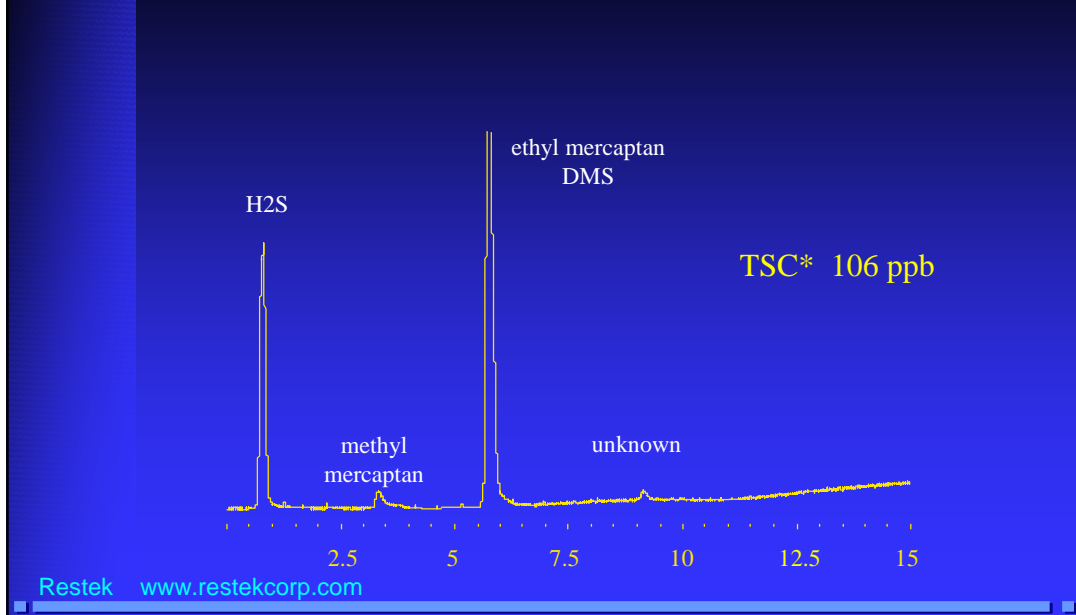
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Brand "F" Beer Headspace Sample

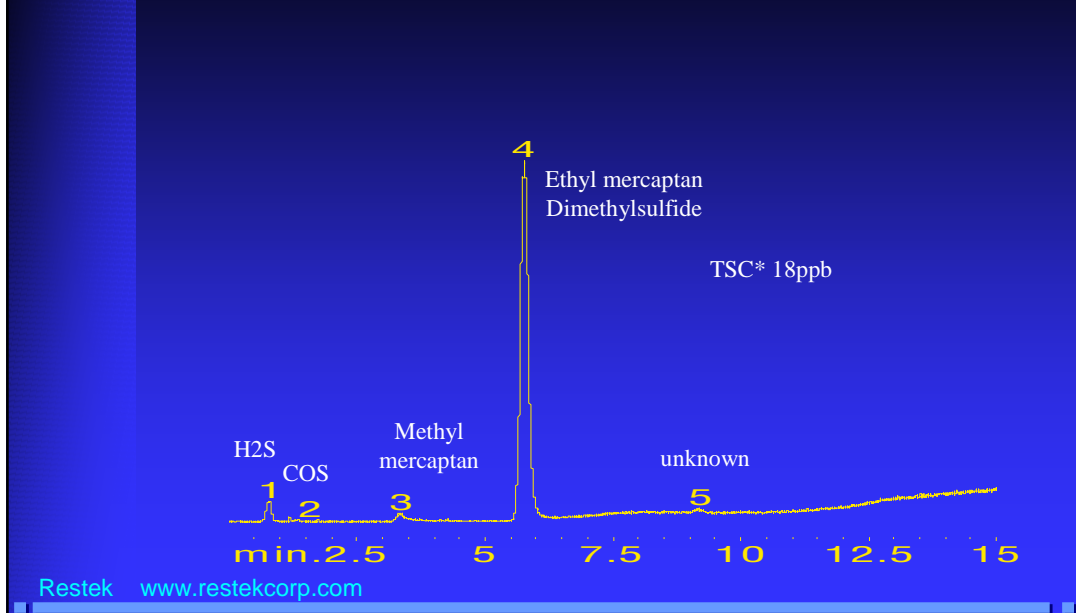


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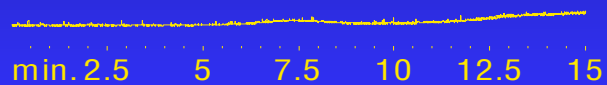
Brand "K" Beer Headspace Sample



Hard Lemon Beverage Headspace Sample



Top Brand Cola Headspace Sample



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Conclusion

- The Rt-XL Sulfur micro-packed column is a robust, low cost, rapid turn-around analytical tool for ppb level analysis of sulfur compounds.
- Sulfinert™ treatment for steel surfaces is unsurpassed for low-level ppb containment and transfer of highly reactive sulfur compounds.

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