The Analysis of Trace Level Sulfurs in Beverage Grade CO_2

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Project Objective

- Develop a robust micro-packed column for the analysis of trace level sulfurs in CO_2 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 SO_2 from all other target sulfur compounds (TSC)

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

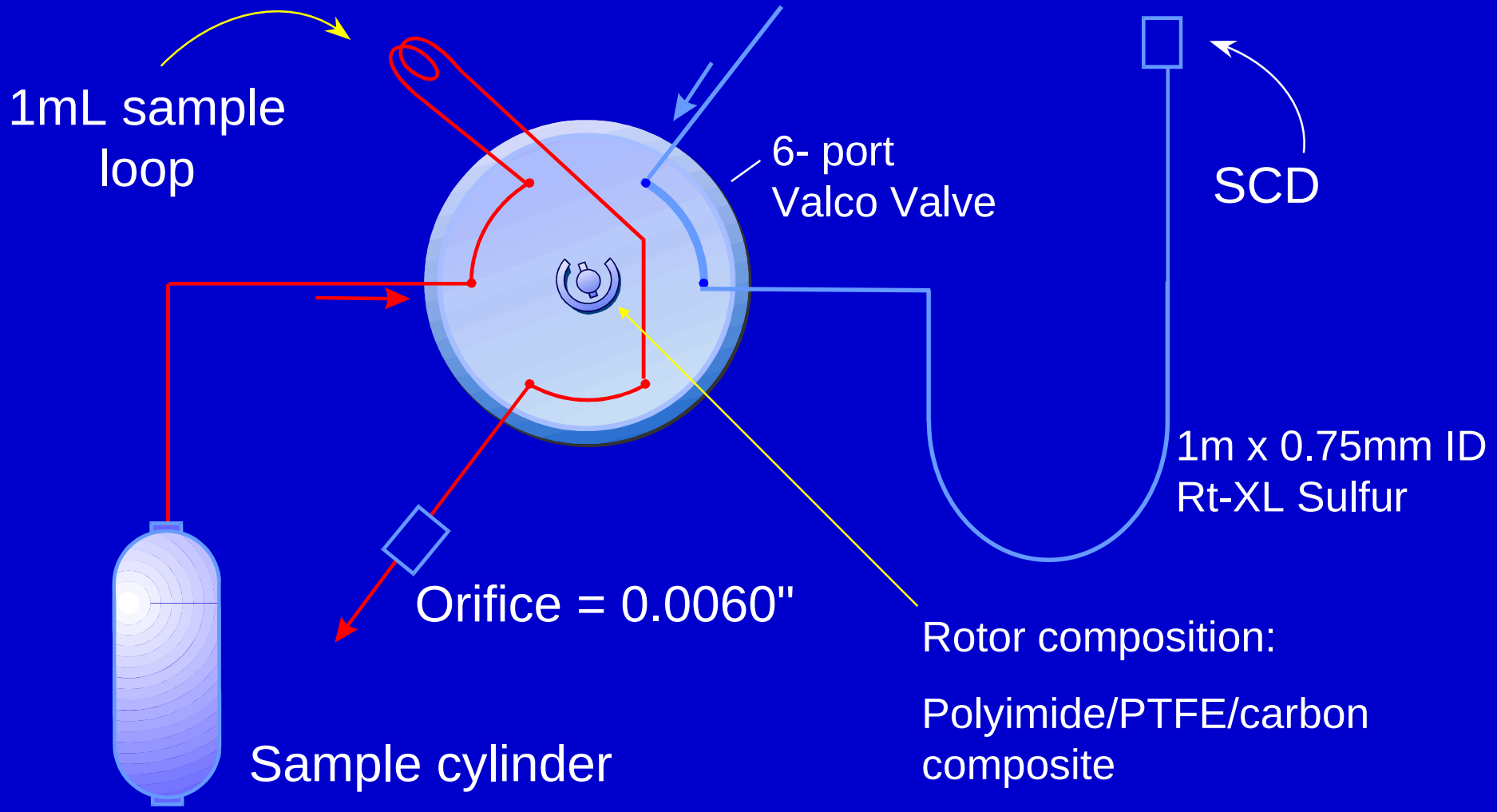
Advantages of the Packed Sulfinert™ column

- No cryogenic oven cooling required
- Highly breakable glass column eliminated
- Critical resolution of SO_2, H_2S & COS
- Column has the flexibility to be installed in any model GC
- Cost effective

GC system requirements

- Detection limit of < 0.02 ppm for SO_2 plus the other TSC
- Method of detection: SCD, HECD, FPD or PFPD detector
- All wetted sample pathways Sulfinert™ passivated for maximum inertness

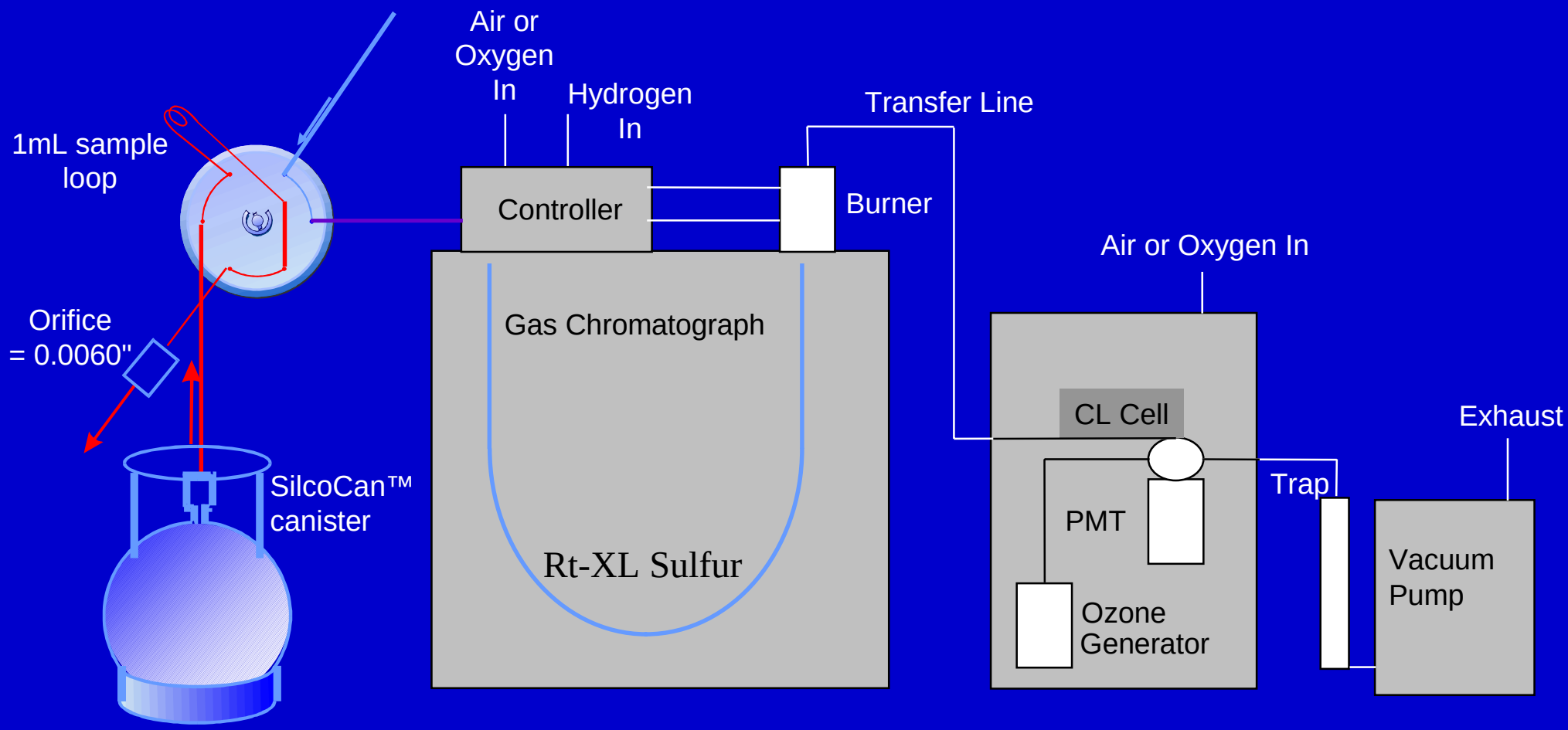
Sulfinert™-Treated Inlet System



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

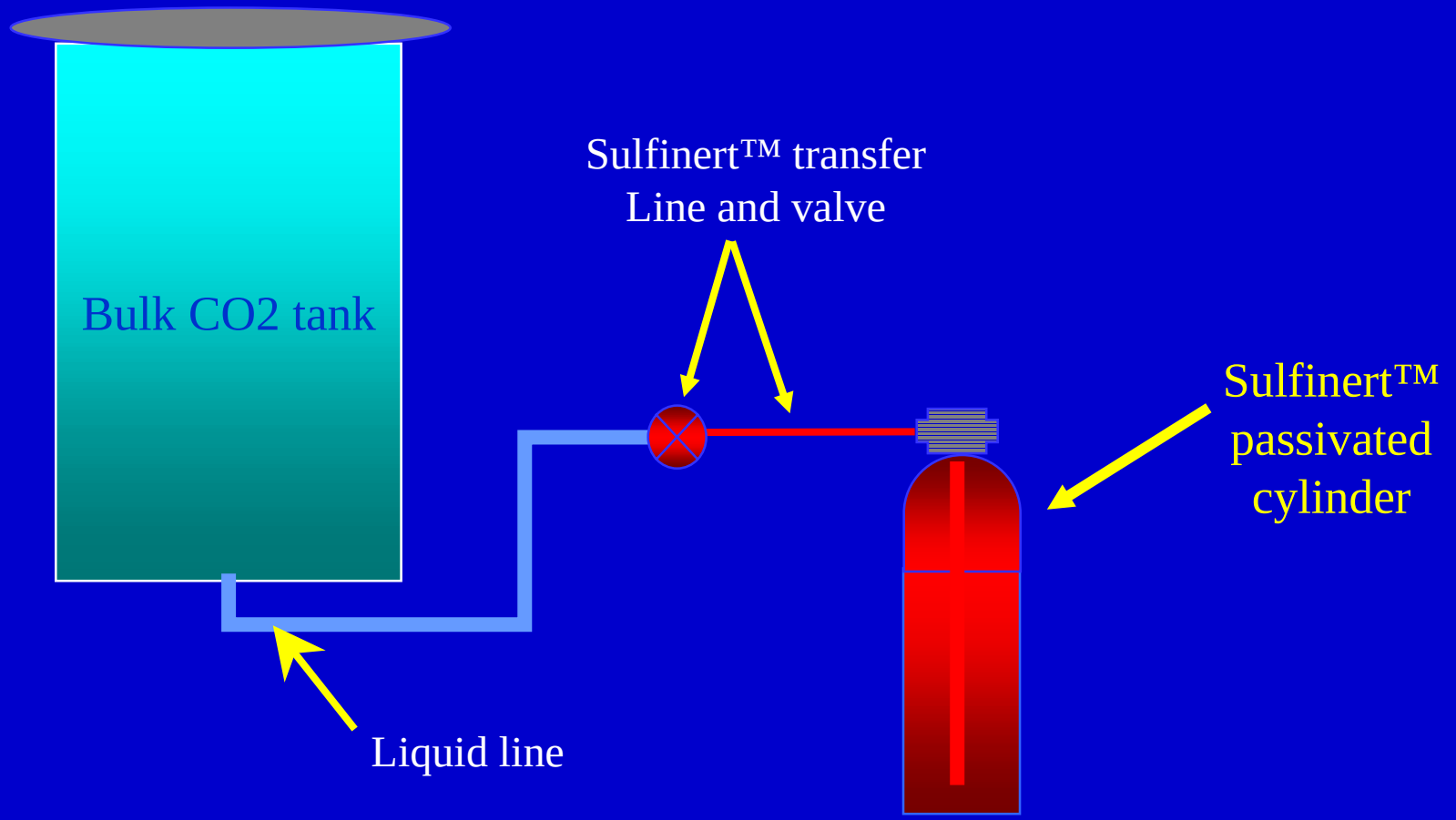
Block diagram of Analytical System



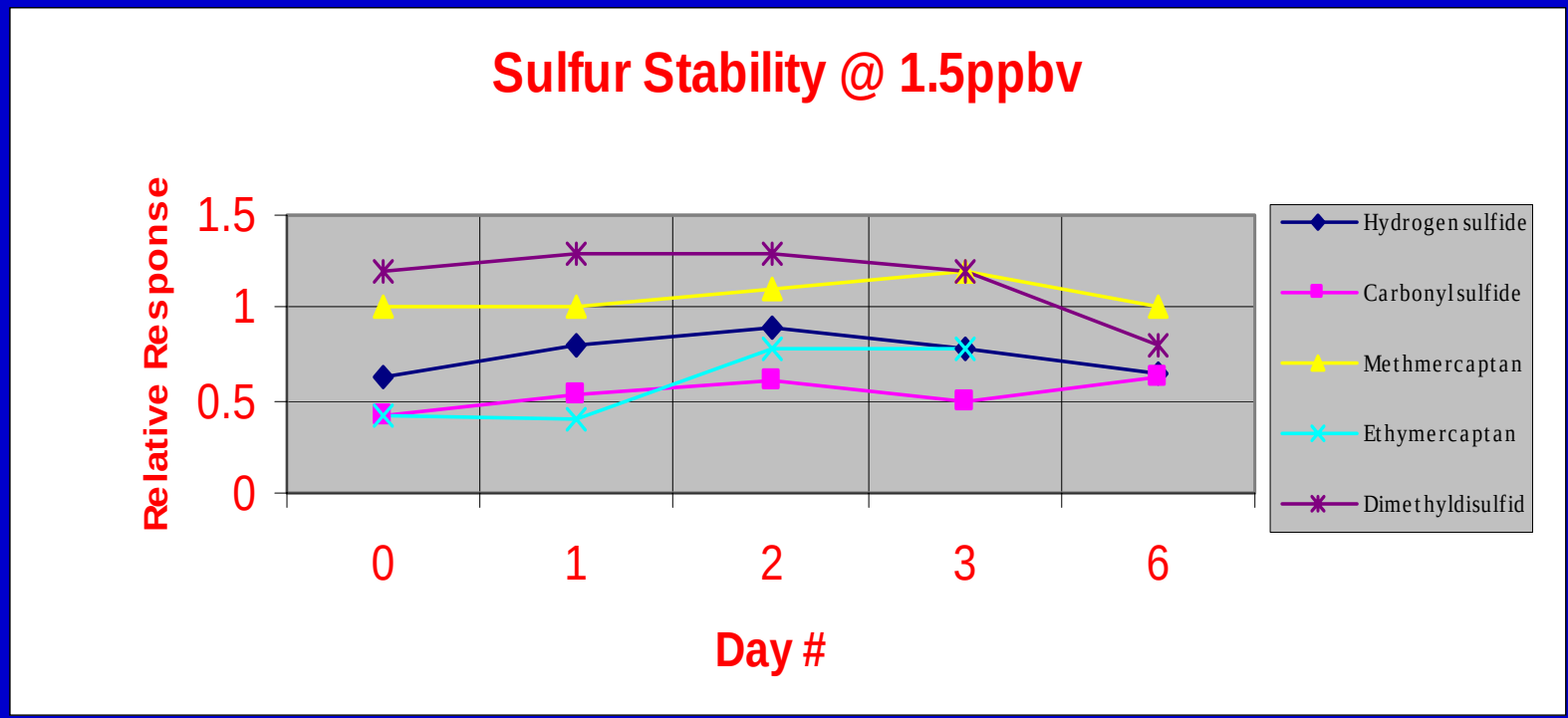
Sampling system integrity for CO_2 liquid sample acquisition

- Passivated vessel to collect the liquid CO_2, conforming to the International Society of Beverage Technologists (ISBT) procedure 1.0
- Connections between the bulk CO_2 and the sample vessel must be passivated to prevent adsorption of the sulfur compounds
- What are the options, and how are the ISBT criteria adhered to?

Sample Acquisition System



Sulfinert™ Vessel Inertness



Response Factors @ 1.5ppbv

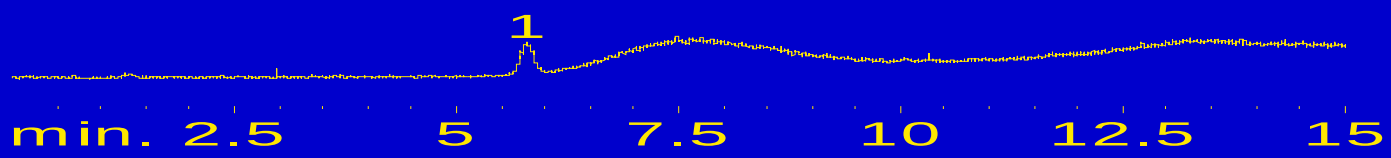
		Day 0	Day 1	Day 2	Day 3	Day 6		
		RRF/DMS	RRF/DMS	RRF/DMS	RRF/DMS	RRF/DMS		% RSD
Hydrogen Sulfide		0.62	0.9	0.85	0.77	0.7		14.6
Carbonyl Sulfide		0.42	0.53	0.6	0.5	0.62		15.07
Methyl Mercaptan		1	1	1.1	1.2	1		8.43
Ethyl Mercaptan		0.42	0.39	0.77	0.77	0.35		39.15
Dimethyldisulfide		1.2	1.3	1.3	1.2	0.8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

Beverage Sampling Data

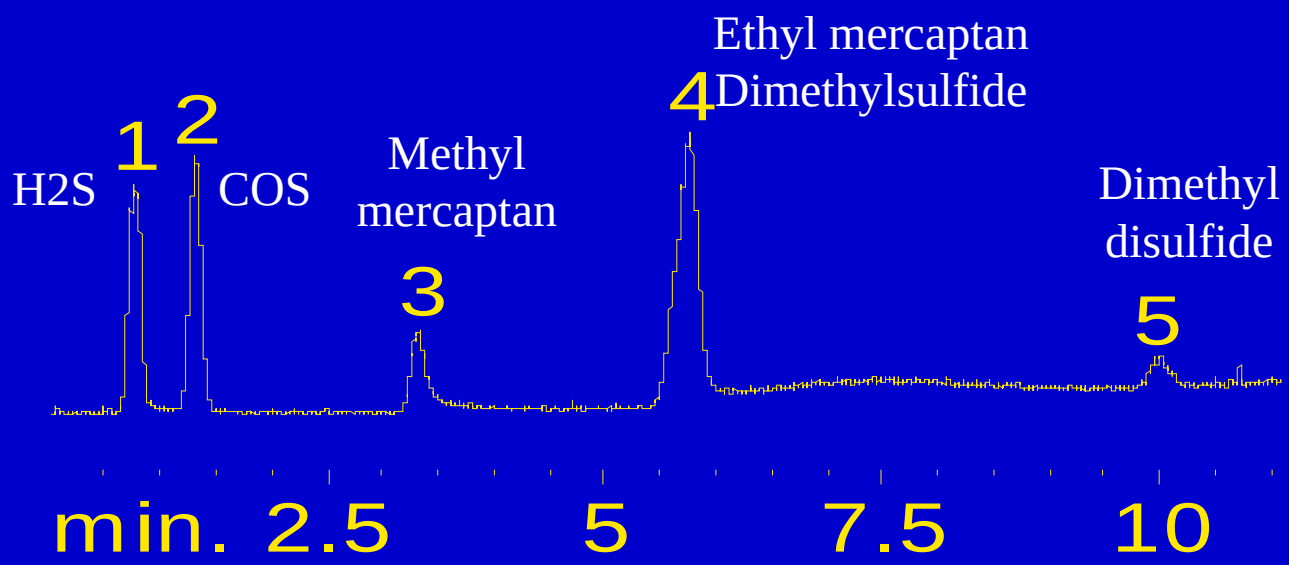
- Beverage grade CO_2 blank
- 20ppb sulfur standard in beverage grade CO_2
- 20 ppb SO_2 in beverage grade CO_2 standard
- Headspace analysis of beer
- Analysis of a hard lemon alcoholic beverage
- Analysis of a top brand of cola

Beverage Grade CO_2 blank

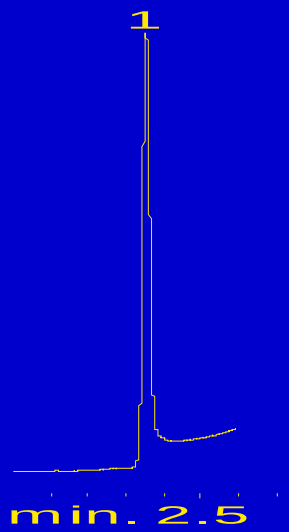
Ethyl mercaptan
Dimethylsulfide



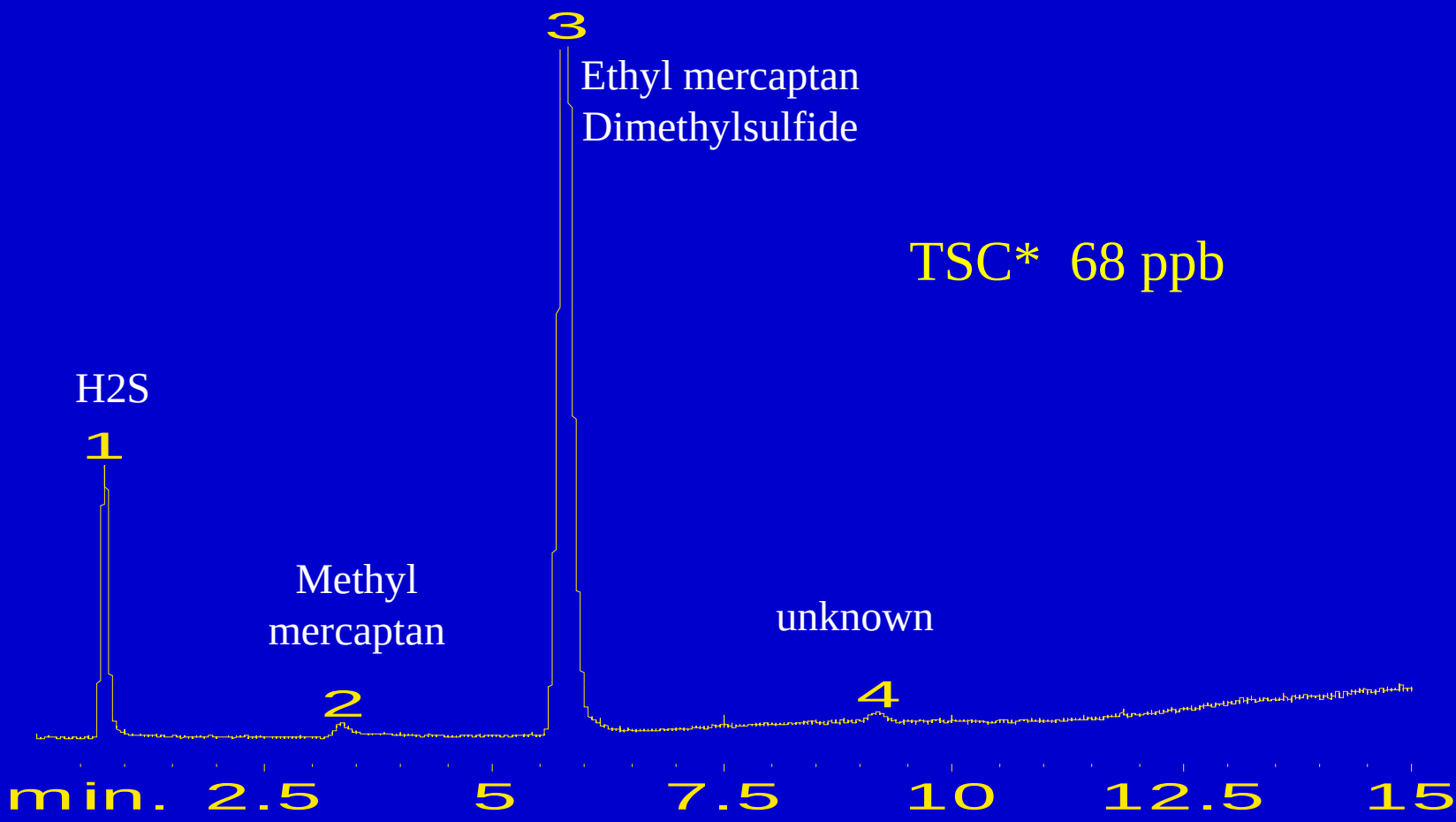
20 ppb Sulfur Standard in Beverage Grade CO_2



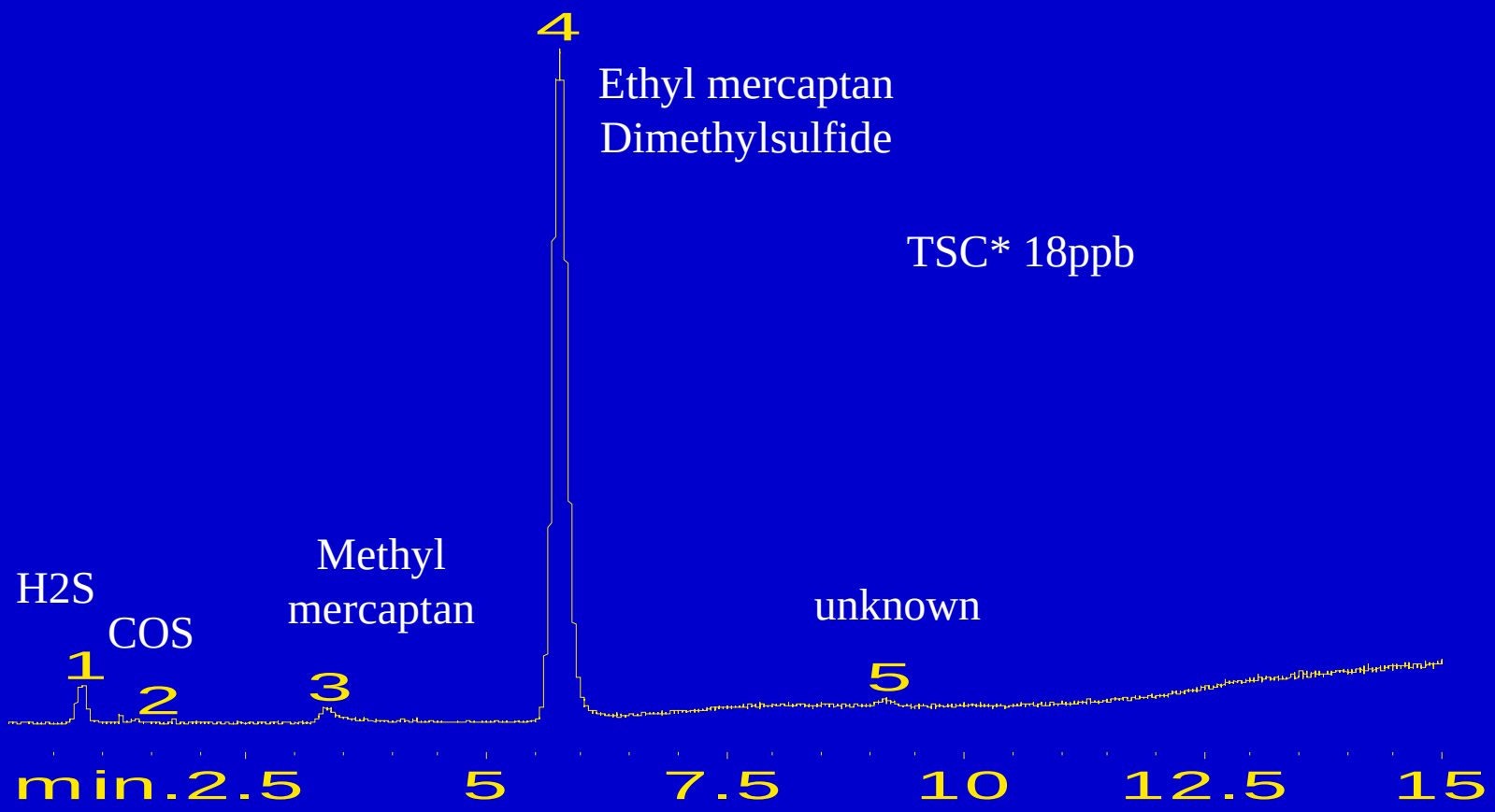
20 ppb SO_2 in CO_2 Standard



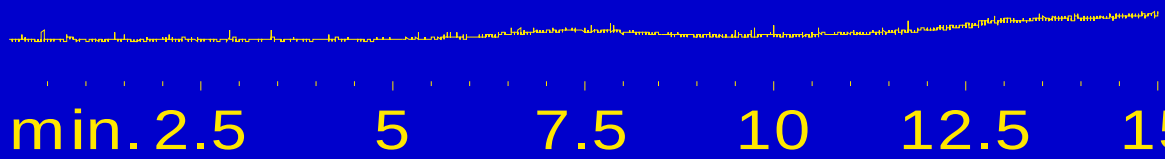
Brand “F” Beer Headspace Sample



Hard Lemon Beverage Headspace Sample



Top Brand Cola Headspace Sample



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.

Acknowledgements

- Seivers Instrument Inc. for their cooperation.

- Lou D’agostaro of DCG Partnership 1 Ltd. of Pearland Texas for supplying us with the sulfur standards.

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