

Determining sulfur impurities in beverage-grade carbon dioxide using the Rt-XLSulfur™ micropacked column

The taste and aroma of a carbonated beverage can be affected by trace impurities from the carbonation process. Therefore, gas producers go to great lengths to purify carbon dioxide (CO₂). Carbon dioxide, a by-product of oil refining, fermentation, and power generating facilities, must be extremely pure to be suitable for a beverage additive. The beverage industry has spent much research time and dollars monitoring the impurities in CO₂.

The most common impurities in CO₂ are hydrocarbons, alcohols, permanent gases, and sulfur compounds. Sulfur impurities are the predominant problem, adding unwanted taste and odor to beverages. The most common volatile sulfur compound (VSC) impurities, listed in Table I, are targeted for monitoring by the International Society of Beverage Technologists (ISBT). Of this group, hydrogen sulfide, carbonyl sulfide, sulfur dioxide, dimethyl sulfide, and methyl mercaptan are the ones most commonly found in beverage-grade CO₂. ISBT guidelines specify Total Sulfur Content* (TSC) as 0.1ppm (v/v) maximum, excluding sulfur dioxide; the maximum level of sulfur dioxide must not exceed 1ppm (v/v) maximum.

The ability to measure reactive sulfur compounds at these levels requires a highly inert chromatographic system. The Restek Rt-XLSulfur™ micropacked column is a powerful analytical tool that can detect sulfurs in CO₂ at levels of 20ppbv, far below the ISBT guideline for TSC. This column also achieves the critical separation of hydrogen sulfide, carbonyl sulfide, and sulfur dioxide as defined in ISBT Procedure 14.0. The Rt-XLSulfur™ micropacked column contains a modified divinyl benzene polymer packed into Sulfinert™ tubing, which is a metal tubing specially deactivated for monitoring ppb levels of active sulfur compounds. Other features of the Rt-XLSulfur™ column include low bleed and thermal stability up to 300°C.

Sample introduction into the column is another critical aspect of obtaining accurate analytical results for sulfur compounds. The sample is introduced onto the column using a Valco® six-port sampling valve, fitted with a 1mL sampling loop (cat. #22845). When the valve, sample loop, and all other surfaces in the sample pathway are deactivated using the Sulfinert™ process, the analyst will see improved response compared to systems using conventional deactivations (Figure 1). The specialized inertness of the Sulfinert™ process is critical for the system to achieve detection limits of 50ppbv for sulfur dioxide and the other target sulfur impurities.

We evaluated the effectiveness of the Rt-XLSulfur™ column and Sulfinert™ sampling system by analyzing bulk CO₂ and CO₂ spiked with a sulfur standard (Figure 2). Notice how even low ppbv levels of sulfur compounds can be detected. We also sam-

*TSC is without SO₂.

pled and measured the TSC* of two top brands of cola and a domestic beer (Figures 3 and 4). The colas show no sulfur content, ensuring that the CO₂ used for carbonation was clean. The beer shows sulfurs that naturally occur during the fermentation process.

This system is sensitive enough to monitor the levels of sulfur in CO₂ during the carbonation process, or in the headspace of the beverage after carbonation. The TSC* generated from headspace sampling of these products demonstrates the ability of the Rt-XLSulfur™ column and the Sulfinert™-deactivated GC system to easily detect sulfur compounds at the 20ppbv level. The combination of the Rt-XLSulfur™ micropacked column and a Sulfinert™-deactivated sample introduction system provides a state-of-the-art, robust sampling and analysis technique for ppbv levels of VSCs in beverage-grade CO₂.

Table I

International Society of Beverage Technologists (ISBT) targets the most common VSCs.

hydrogen sulfide	isopropyl mercaptan
carbonyl sulfide	methyl ethyl sulfide
methyl mercaptan	<i>n</i> -propyl mercaptan
ethyl mercaptan	<i>tert</i> -butyl mercaptan
sulfur dioxide	<i>sec</i> -butyl mercaptan
dimethyl sulfide	diethyl sulfide
dimethyl disulfide	isobutyl mercaptan
carbon disulfide	<i>n</i> -butyl mercaptan
<i>tert</i> -amyl mercaptan	

Figure 1

A Sulfinert™-treated sampling system designed to provide sensitive analysis of trace-level sulfur compounds.

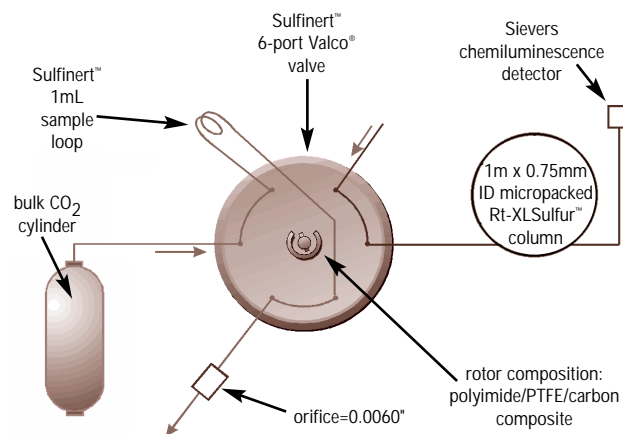


Figure 2

The Rt-XLSulfur™ column and the Sulfinert™-deactivated GC system easily detect sulfur compounds at the 20ppbv level.

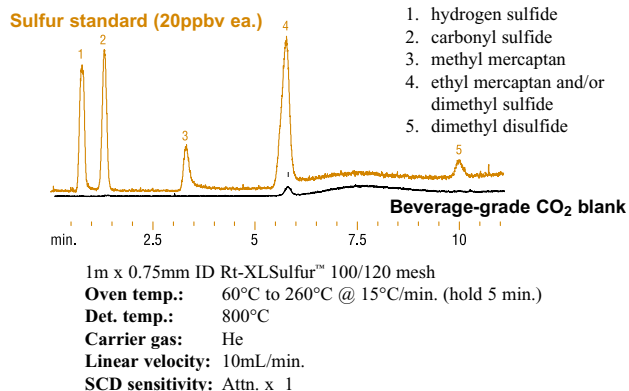


Figure 4

A sample of domestic (US) beer contains ppbv levels of sulfurs from the fermentation process.

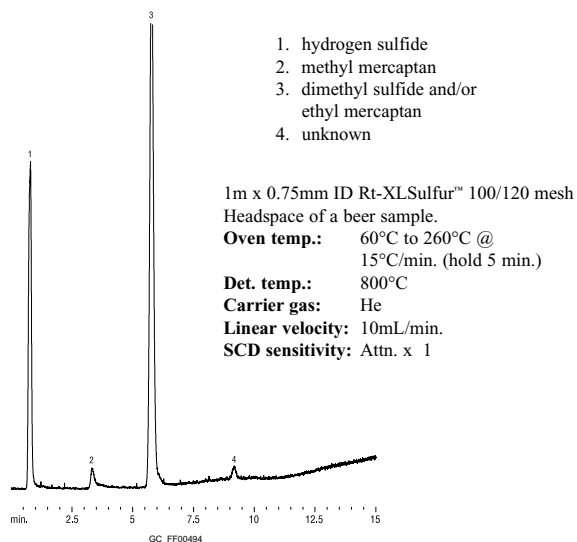
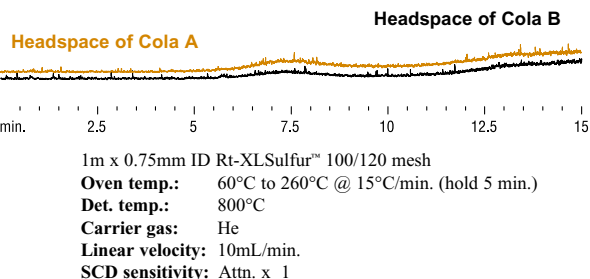


Figure 3

Samples of two popular colas show no sulfur compounds.



Installation Kits

Fits column IDs →	0.75mm	1mm	2mm
For valve applications	21062	21065	21067
For split applications	21063	—	—
For all Agilent GCs	21064	—	—
For direct injections	—	21066	—

Rt-XLSulfur™ Packed and Micropacked Columns

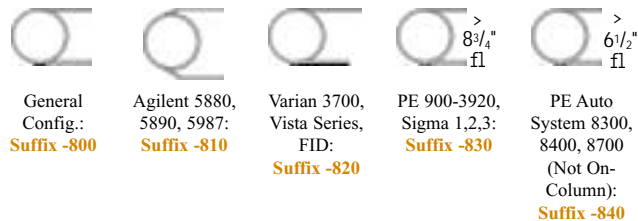
OD	ID	1-meter	2-meter
1/16*	1.00	19804	19805
0.95mm*	0.75	19806	19807
1/8*	2.00	80484**	80485**
3/16	3.20	80482**	80483**

Sulfinert™ Sample Loops

size	cat.#	size	cat.#	size	cat.#
5µL	22840	50µL	22844	1cc	22848
10µL	22841	100µL	22845	2cc	22849
20µL	22842	250µL	22846	5cc	22850
25µL	22843	500µL	22847		

Packed Column Configurations

Custom configurations are available. Please contact Customer Service (ext. 3) or your local Restek representative.



6-Port Valco® Valve

The 6-port Valco® valve was coated with Sulfinert™ treatment on a custom basis. For custom Sulfinert™ quotes, call customer service at ext. 3, or contact your local Restek representative.

* Installation kit must be purchased when using valve applications.
 ** Please include configuration suffix number when ordering.

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