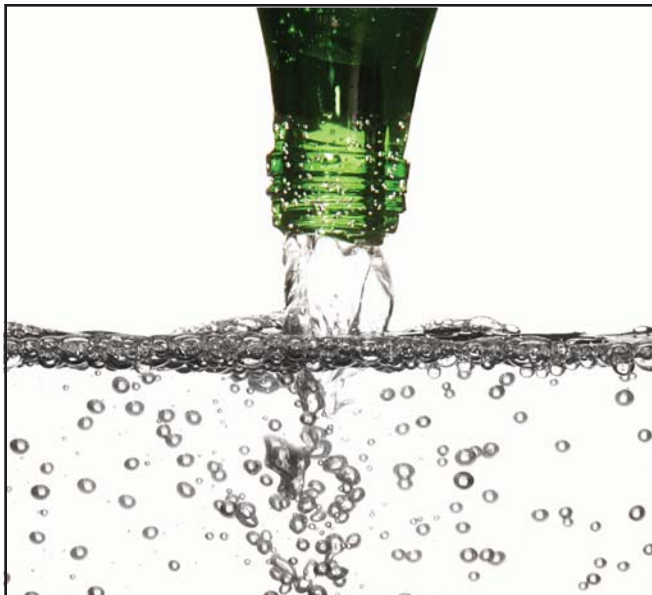
Beverage Grade Carbon Dioxide

Analysis by Gas Chromatography



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INSTRUMENTATION

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The Challenge

Carbon dioxide, used in the production of carbonated soft drinks and other beverages, can be produced from a variety of sources including ammonia plants, fermentation plants, chemical plants, and oil refineries. This variety of production sources results in an array of possible chemical contaminants which can be found in carbon dioxide. In addition, companies are now limited by industry stipulations on maximum levels of these impurities.

The impurities that must be assessed and controlled include hydrogen, oxygen, nitrogen, oxides of nitrogen, oxygenated hydrocarbons, sulfur compounds, phosphine, cyanide, chlorinated compounds, ammonia, light paraffins, olefins, non-volatile hydrocarbons, benzene, and other aromatic hydrocarbons. Because of the wide variety of compounds that must be analyzed, a gas chromatograph (GC) is an excellent tool for the analysis, with its ability to separate and quantify a large range of compounds.

Chemical Analysis (FID/FID/MSD)

Wasson-ECE Instrumentation has a customized solution for the analysis of beverage grade carbon dioxide using two GCs. The first system is configured with dual flame ionization detectors and a mass selective detector (FID/FID/MSD). The power of the MSD is two-fold. First, in the Selected Ion Monitoring (SIM) mode, this detector can measure in the parts-per-billion (ppb) concentration range. Second, because the MSD is programmed to respond only to a known molecular fragmentation pattern, or spectrum, at any given time, it can confirm the presence of each analyte in question rather than relying only on chromatogram retention time. This is very important as different trace impurities have different product impact thresholds. Thus, without the confirmation power of the MSD, it is easy to accidentally confuse the presence of a low impact analyte with that of a higher priority analyte, which would generate a false-positive report.

GC 1: FID/FID Analysis

ANALYTE	CONCENTRATION
Methane	1 ppm
Non-methane hydrocarbons	1 ppm
C_1-C_6 paraffins and olefins	1 ppm
Methanol	0.25 ppm mol
Oxygenates	0.25 ppm mol
BTEX	0.25 ppm mol

GC 1: MSD Analysis

ANALYTE	CONCENTRATION
Acetaldehyde	0.2 ppm
Phosphine	0.3 ppm
Benzene	0.02 ppm
Toluene	0.02 ppm
Ethylbenzene	0.02 ppm
m, p, o-xylenes	0.02 ppm

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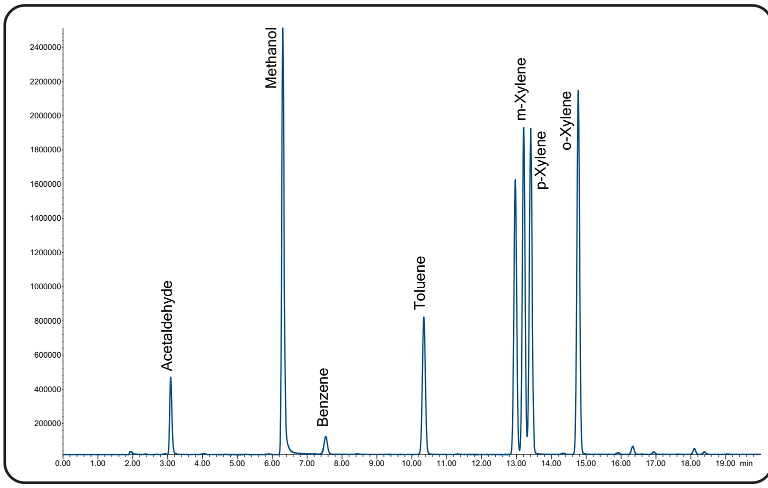
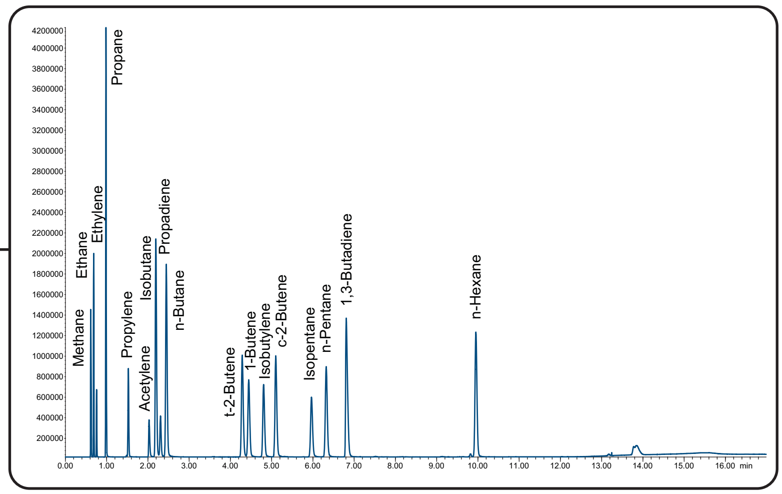
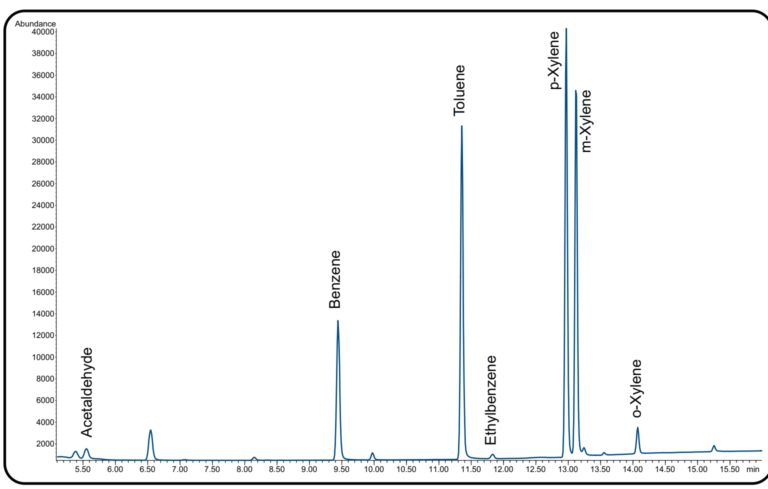
Chromatograms for the Analysis of Trace Impurities in Beverage Grade Carbon Dioxide by Dual FIDs and MSD



0.25 mL gas sample injection of oxygenates and BTEX in carbon dioxide (% levels) by FID1.



0.2 mL gas sample valve injection of ppm level hydrocarbons in carbon dioxide by FID2.



1.0 mL gas sample valve inject of a 1.0 ppm standard blended in carbon dioxide by MSD.

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Chemical Analysis (SCD/PDHID)

The second GC is configured with a sulfur chemiluminescence detector (SCD) and cryogenic cooling to quantify trace level sulfur compounds in the low part-per-million (ppm) range. A pulsed discharge helium ionization detector (PDHID) is also included on the second system to detect low level permanent gases.

GC 2: SCD Analysis

ANALYTE	CONCENTRATION
Hydrogen sulfide	0.03 ppm
Carbonyl sulfide	0.03 ppm
Sulfur dioxide	0.1 ppm
Ethyl mercaptan	0.03 ppm
Dimethyl mercaptan	0.03 ppm
Carbon disulfide	0.03 ppm
Isopropyl mercaptan	0.03 ppm
Ethyl methyl mercaptan	0.03 ppm
Butyl mercaptan	0.03 ppm
Isobutyl mercaptan	0.03 ppm
Diethyl mercaptan	0.03 ppm
Dimethyl disulfide	0.03 ppm
2-Methylthiophene	0.03 ppm
3-methylthiophene	0.03 ppm
Diethyl disulfide	0.03 ppm
Phenyl mercaptan	0.03 ppm
Total sulfur	0.06 ppm

GC 2: PDHID Analysis

ANALYTE	CONCENTRATION
Oxygen	0.5 ppm mole
Argon	0.5 ppm mole
Nitrogen	0.5 ppm mole
Carbon monoxide	0.5 ppm mole
Methane	0.5 ppm mole

Sample Phase

The carbon dioxide samples can be introduced as a pressurized liquid or in the gas phase. A selection valve is provided to address the appropriate sample input. In the case of pressurized liquid, an internal vaporizer converts the sample to gas phase with fused silica-lined internal components so that the loss of trace sulfur or oxygenated components is avoided. In the case of heavy hydrocarbons/oils analysis, the sample is syringe-injected through a hot inlet and gas injection valve.

Non-Volatile Organics Concentrator (NVOC) for Liquid Carbon Dioxide

Liquid carbon dioxide may have heavy organic contaminants such as ethylene glycol, or pump oil. The NVOC concentrates these analytes into a vial which contains a measured amount of solvent. The heavy organics dissolve in the solvent for syringe injection. The NVOC extracts the heavies from the captured, known volume of carbon dioxide. The matrix is flashed off with heat in a controlled fashion while the heavies remain in liquid phase.

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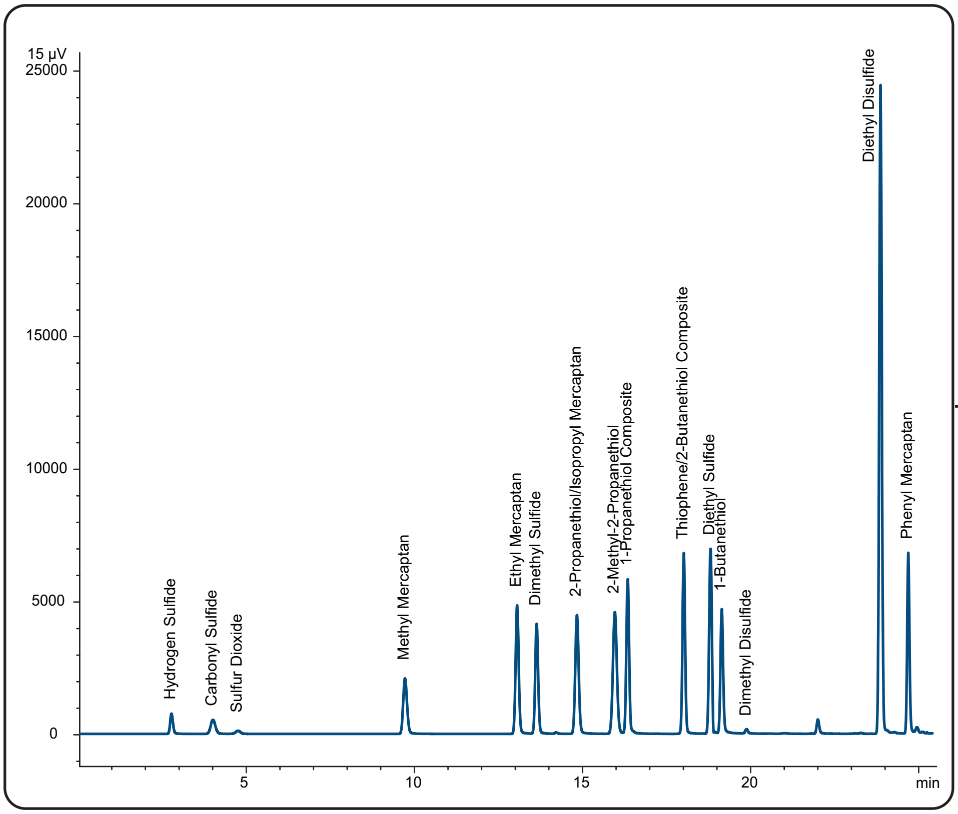
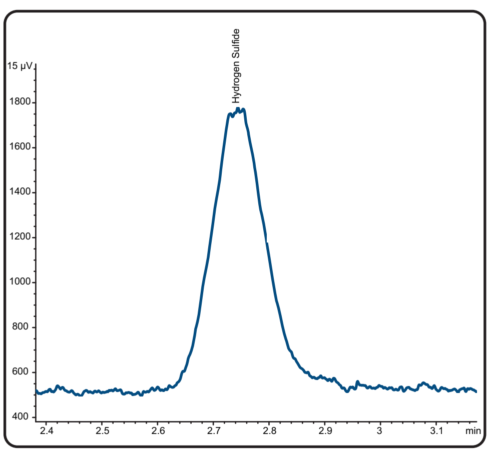
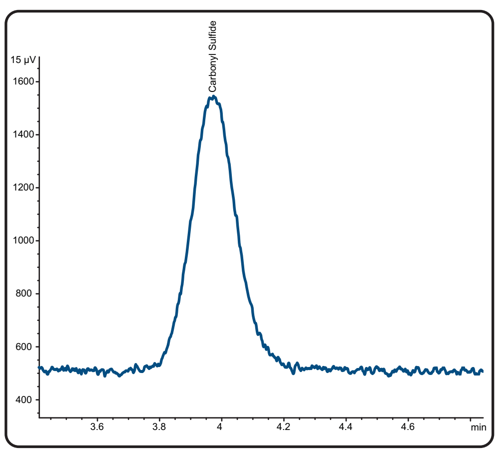
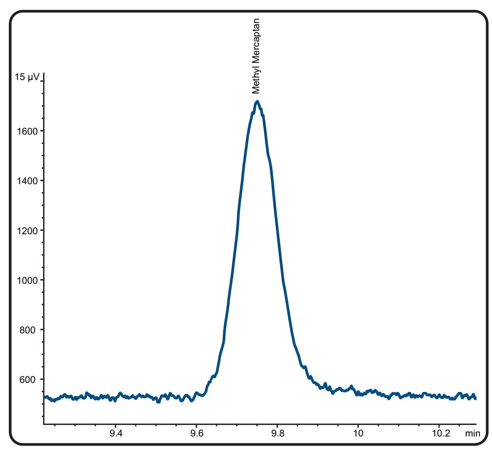
Chromatograms for the Analysis of Trace Sulfurs in Beverage Grade Carbon Dioxide by SCD



3.0 mL gas sampe injection of 20 ppm sulfur standard blend in air, spiked with sulfur dioxide, by SCD.

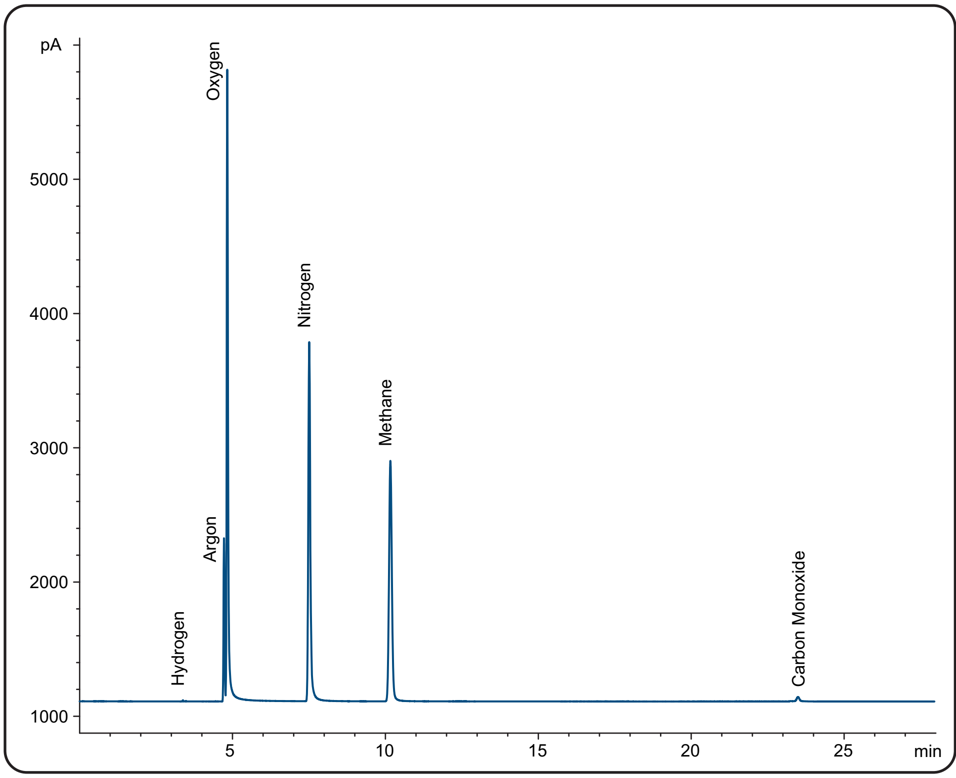






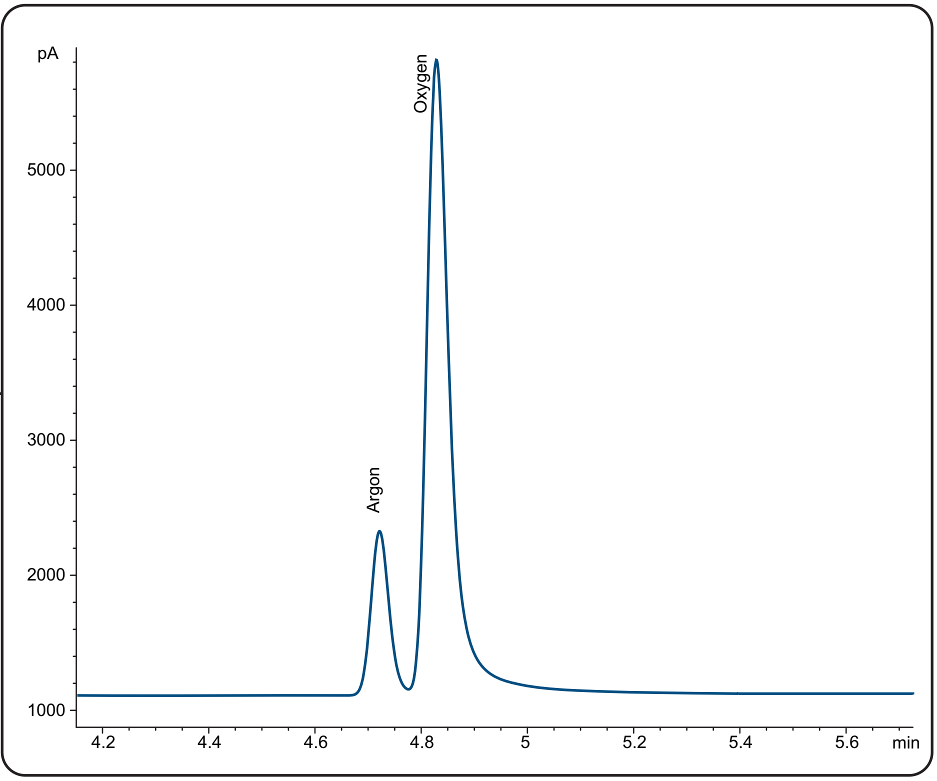
Beverage Grade Carbon Dioxide

Chromatograms for the Analysis of Permanent Gases in Beverage Grade Carbon Dioxide by PDHID



0.1 mL gas sample injection of permanent gases in carbon dioxide by PDHID.

- 5.0 ppm Ar
- 28.0 ppm O_2



Enlargement showing argon and oxygen separation on a PDHID.

- LDL* (Ar peak): 11.6 ppb
- LDL* (O_2 peak): 6.9 ppb

* LDL denotes the concentration at which the signal is 2.5 times greater than the background noise signal.

Wasson-ECE Instrumentation

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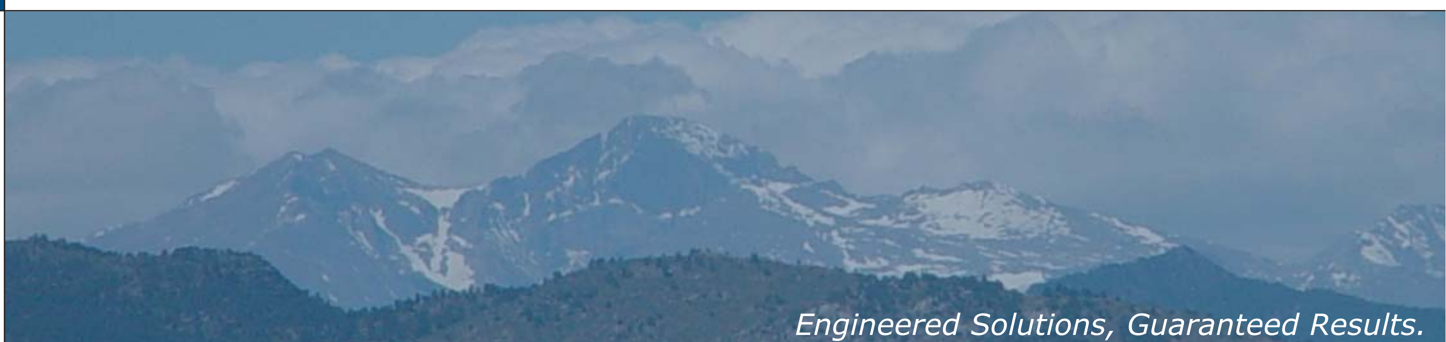
Wason-ECE Instrumentation specializes in configuring and modifying new or existing gas chromatographs exclusively from Agilent Technologies to become guaranteed, turn-key analytical systems. Our customers describe their objectives and their samples: analytes, concentration ranges, phases, temperature, throughput, and any special needs. From this dialog we configure a task specific instrument. We add extra ovens, valves, plumbing, flow control, columns, electronics, software, etc., to yield a complete solution. This saves our clients valuable time and delivers instruments that are state-of-the-art and ready for use upon installation.

The complete analytical method is developed, tested, and documented utilizing our experience working with numerous companies with similar needs and goals. The resulting chromatograms and reports are inspected by our application chemists and you, to ensure system performance and design quality. Our field engineers then install each system and provide training. After installation, and throughout the life of the chromatograph, our support chemists are ready to help. We can assist with application details, questions, training, calibration, maintenance, and on-site service. Wasson-ECE brings experience and efficiency to your project, giving you confidence in the quality of your results.

Please contact us for more information.



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