A Simplifed Method
for the Detection of

Impurities in Food Grade CO$_2$

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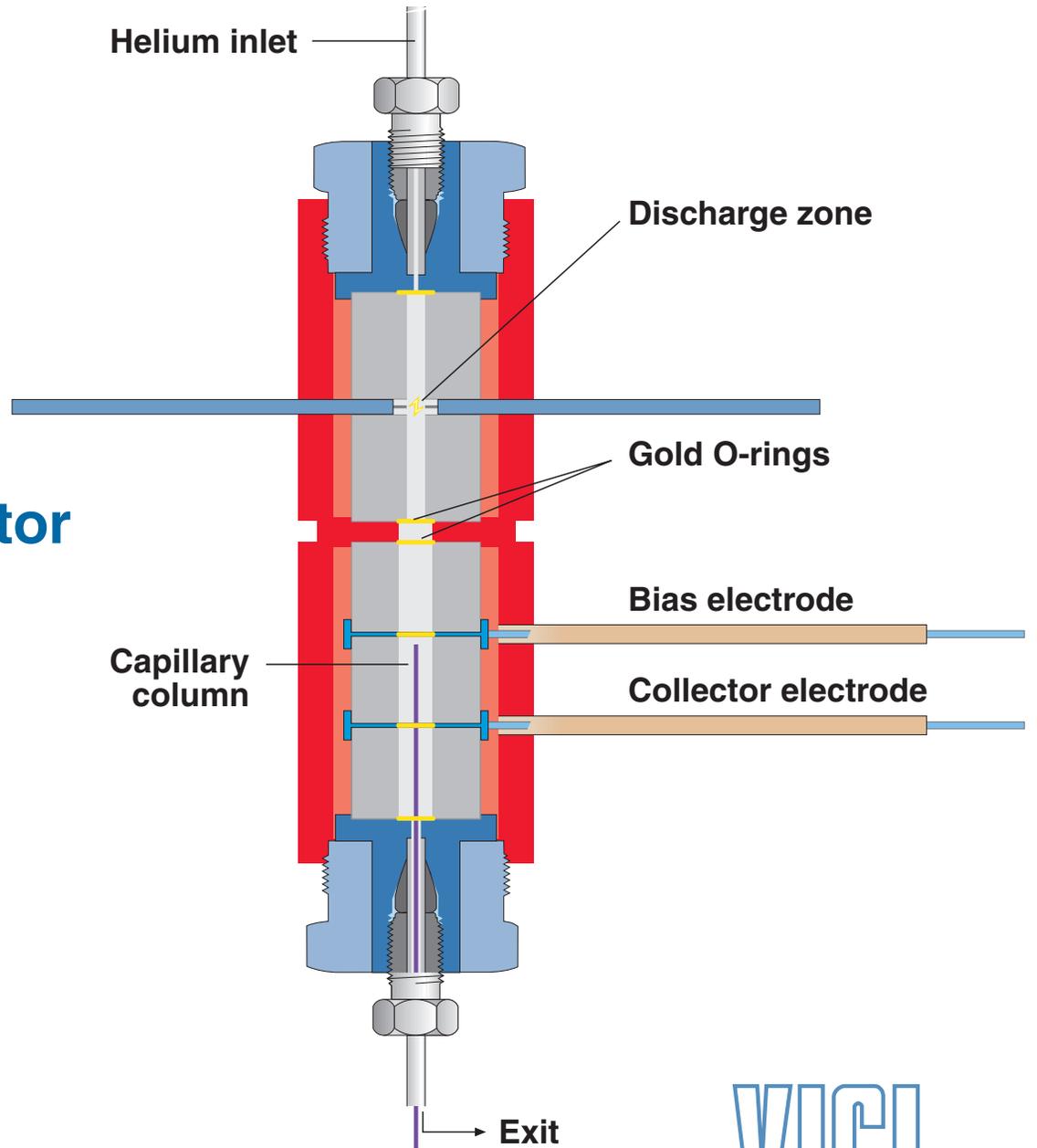Valco Instruments Co. Inc., P. O. Box 55603, Houston TX 77255

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▶ Introduction

Trace level impurities in food grade CO_2 can impart off-flavor to the product or can pose a potential health hazard. The detection and quantification of impurities like permanent gases, inorganics, and aromatics in CO_2 by gas chromatography typically require multiple detectors. The VICI Pulsed Discharge Ionization Detector is very versatile and can detect all of the above impurities. The detector can be configured to operate in either the universal helium ionization mode or the selective photoionization mode. Selective photoionization is achieved by doping the discharge gas with 1-2% argon, krypton, or xenon dopant. In this study, the universal mode was employed for detecting permanent gases and the argon photoionization mode was utilized for detecting inorganics and aromatics in CO_2.

▶ Experimental

A VICI model D-4 was used to analyze impurities in CO_2. (A cross-section of the detector is shown in Figure 1.) Standards for benzene, toluene, m-xylene, hydrogen sulfide, formaldehyde, carbonyl sulfide, acetaldehyde, and methanol in CO_2 were generated using a Dynacalibrator Model 190 (VICI Metronics) with respective permeation tubes. A certified blend of H_2, O_2, N_2, CH_4, and CO in helium was used as standard for the permanent gases. The experimental setup is illustrated in Figure 2.
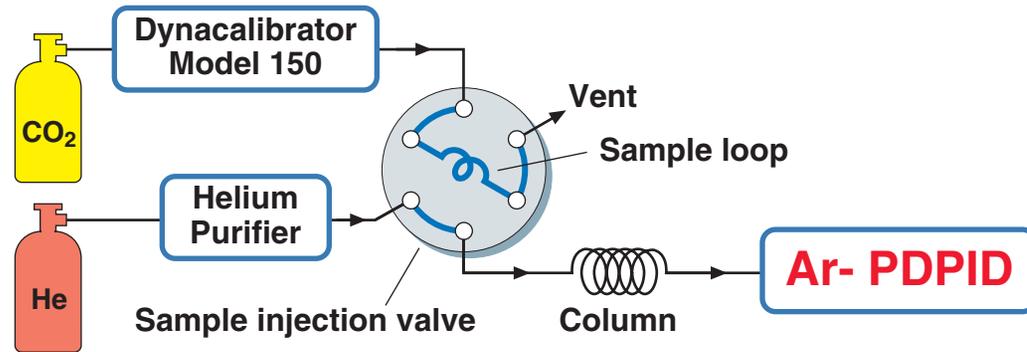
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Figure 1

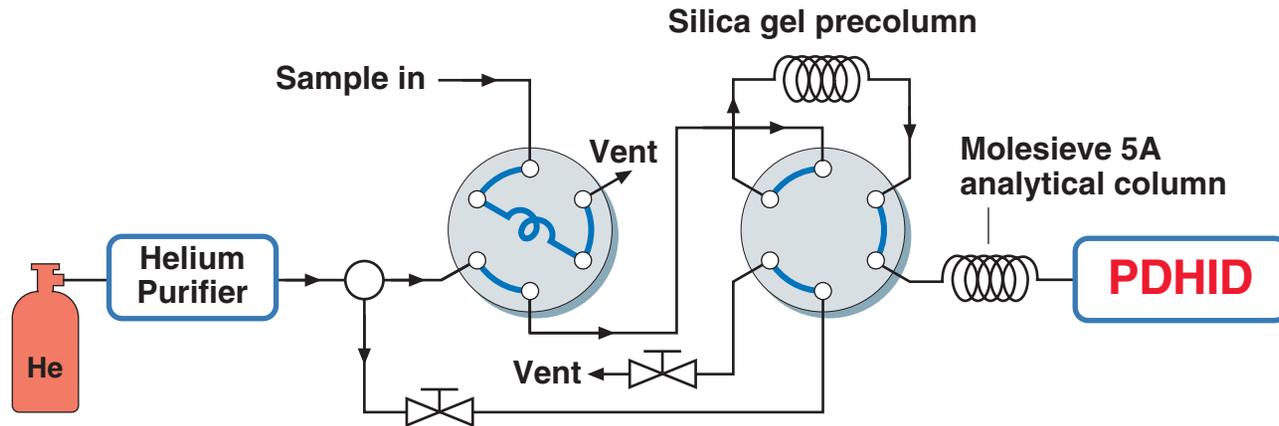## Pulsed discharge helium ionization detector

Helium inlet

Discharge zone

Gold O-rings

Bias electrode

Collector electrode

Capillary column

Exit

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Figure 2

Experimental setup for:

aromatics and inorganics

Dynacalibrator Model 150

CO_2

Helium Purifier

He

Sample injection valve

Vent

Sample loop

Column

Ar- PDPID

permanent gases

Silica gel precolumn

Sample in

Vent

Molesieve 5A analytical column

Helium Purifier

He

Vent

Vent

PDHID

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Minimum Detectable Quantities

	(S/N=3, ppb)
Benzene	**0.65**
Toluene	**0.40**
m-Xylene	**0.36**
Hydrogen Sulfide, H_2S	**10.5**
Formaldehyde, HCHO	**8.2**
Carbonyl Sulfide, COS	**10.6**
Acetaldehyde, CH_3CHO	**25.4**
Methanol, CH_3OH	**46.2**

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Aromatics

pA
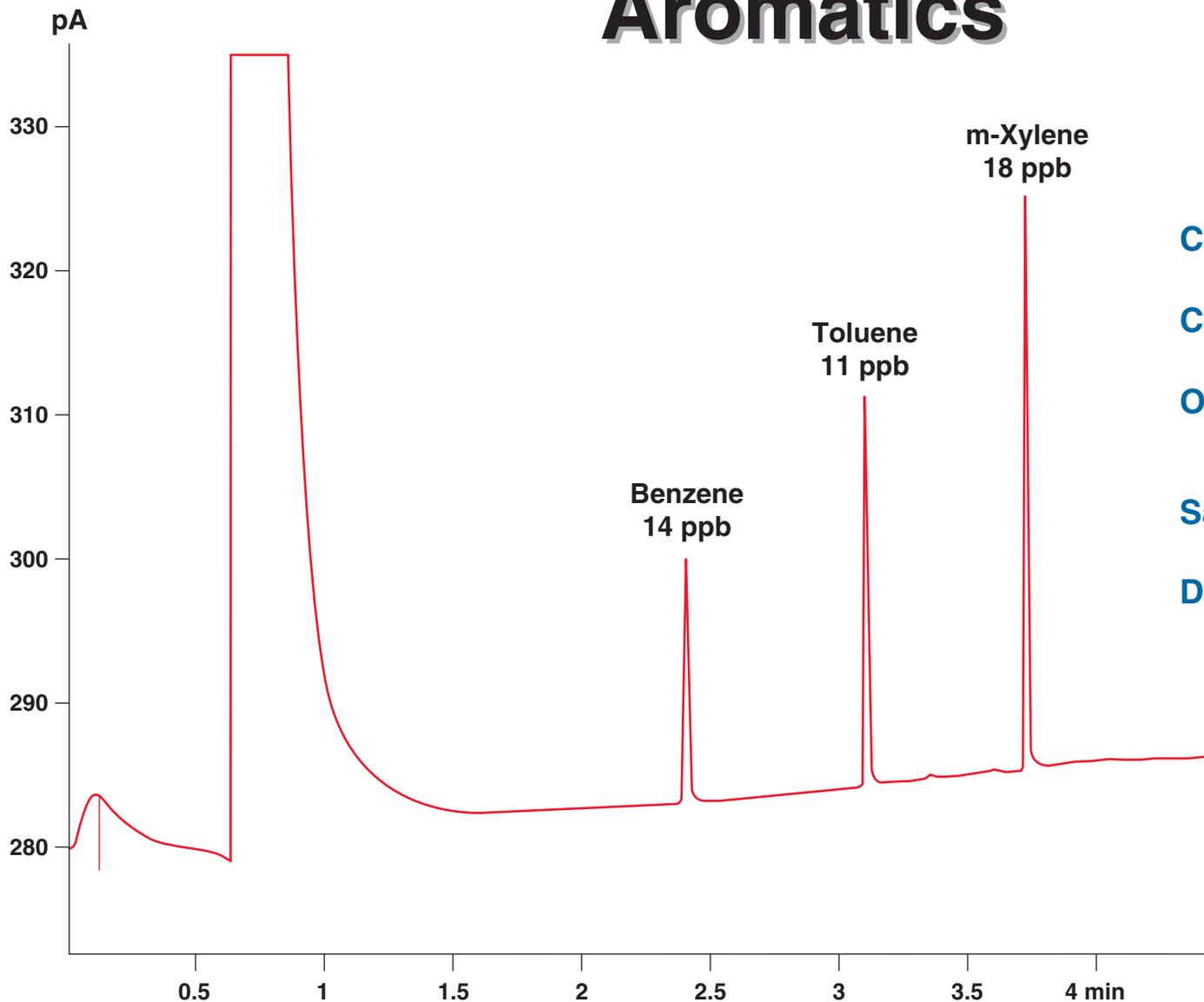
Column
DB5, 30 m x 0.25 mm x 1.0 μm

Carrier flow
4 mL /min

Oven temp
35℃ for 1 min to
145℃ @ 40℃ /min

Sample loop
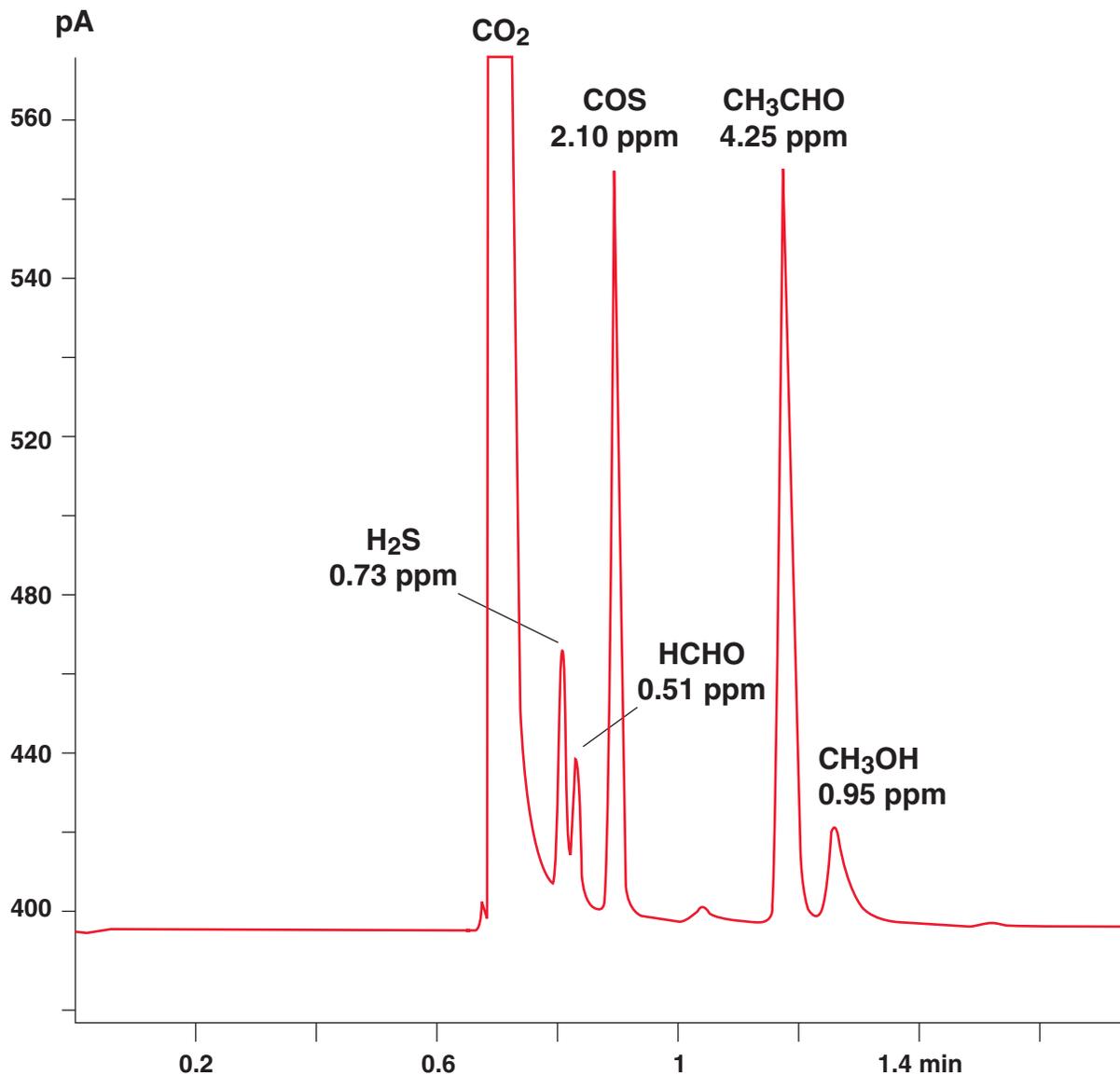250 μL

Detector mode
Ar-PDPID

Chart labels: m-Xylene 18 ppb, Toluene 11 ppb, Benzene 14 ppb

Y-axis: 330, 320, 310, 300, 290, 280

X-axis: 0.5, 1, 1.5, 2, 2.5, 3, 3.5, 4 min

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Inorganics

pA

560

540

520

480

440

400

0.2   0.6   1   1.4 min

CO_2

COS
2.10 ppm

CH_3CHO
4.25 ppm

H_2S
0.73 ppm

HCHO
0.51 ppm

CH_3OH
0.95 ppm

Column
DB1, 30 m x 0.32 mm x 4.0 µm

Carrier flow
5 mL /min

Oven temp
30℃

Sample loop
50 µL

Detector mode
Ar-PDPID

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Permanent gases

volts

methane
7.8 ppm

nitrogen
6.7 ppm

oxygen
4.8 ppm

Precolumn
8' x 1/8" silica gel

Analytical column
10' x 1/8" Molesieve 5A

Carrier flow
30 mL /min

Oven temp
110℃

Sample loop
1 mL

Detector mode
PDHID

VICI

Conclusions

▶ **The Pulsed Discharge Detector is an ideal detector for the analysis of food grade CO_2:**

- **Most impurities in CO_2 can be analyzed up to ppb level without sample preconcentration**

- **The response is linear**

- **The PDD is very easy to operate**

▶ **The use of permeation tubes to generate standards in CO_2 is convenient and economical, and provides the flexibility to customize**

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