

An Improved ASTM D3612 TOGAS System

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Introduction

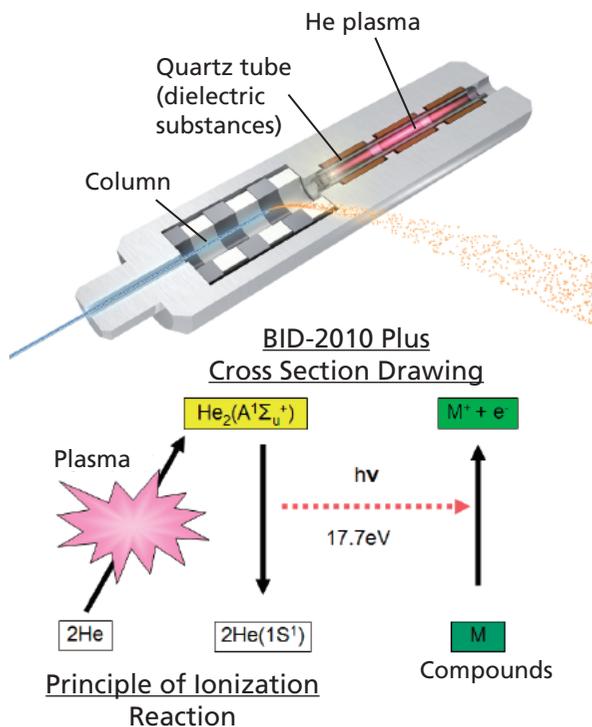
ASTM D-3612 is a GC method that describes the analysis of hydrogen, oxygen, nitrogen, methane, carbon monoxide, C2-C3 in hydrocarbon-based transformer oil. In this study, two changes have been made to the method. The first change was substituting a universal Barrier Ionization Discharge (BID) detector in place of the TCD. Hydrogen detection levels were lowered from 10ppm to 100ppb. The second change was the addition of propane and butane which were incorporated into the analysis.

Two PLOT columns were placed in series separated by a 4-port stream selection valve that directs the gas components to either the BID or FID. H2, O2, N2, CH4, CO are separated by a molecular sieve column then detected by BID while CO2, C2H2, C2H4, C2H6 and C3H8 are separated by the Carboxen-1006 column then detected by FID. CO2 went through a methanizer and was reduced to CH4 then analyzed by FID.

BID-2010 Plus Principals for Detection

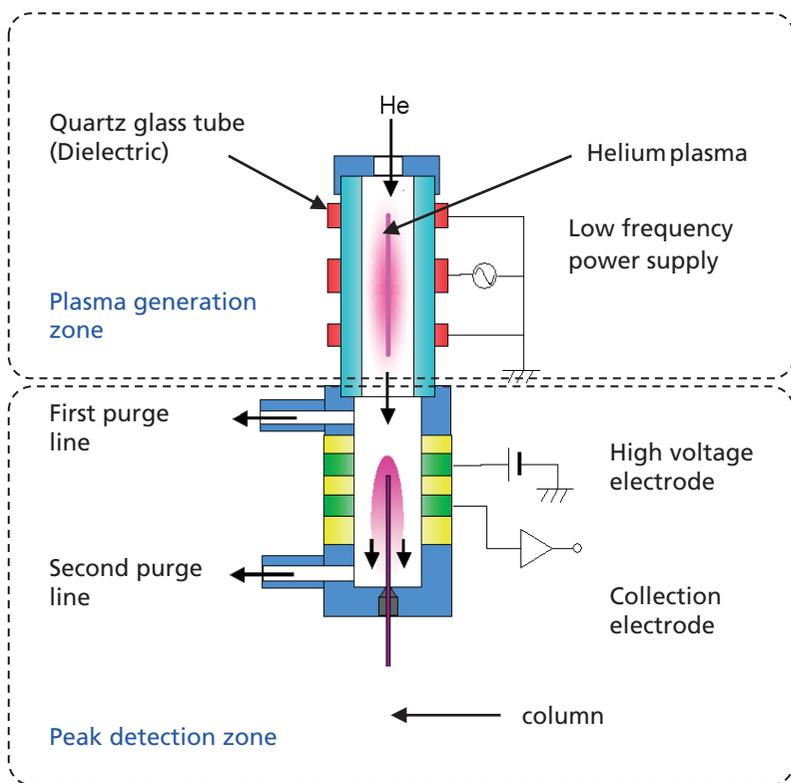
A plasma is generated by applying a high voltage to a quartz dielectric chamber in the presence of helium. Compounds that elute from the GC column are ionized by this He plasma, then, captured with collection electrodes and described as peaks. The photon energy of

He is extremely high (17.7 Electron Volt). Therefore, allowing highly sensitive detection of every compound except Ne (Neon) and Helium which is the plasma gas. BID is a near-universal plasma detector.



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Structure of BID



Three electrodes are placed on the quartz tube. High voltage is applied to electrodes, and plasma is generated.

Compounds are ionized by plasma, and signal is collected at collection electrode.

Features of BID-2010 Plus

BID-2010 Plus Tracera is a novel universal detector based on dielectric barrier plasma ionization. Tracera makes it possible to conduct many kinds of applications and achieve simple, highly sensitive analysis.

1. High Sensitivity

Detection Sensitivity over 100x Higher Than TCD, 2x Higher than FID

2. Novel Universal Detector

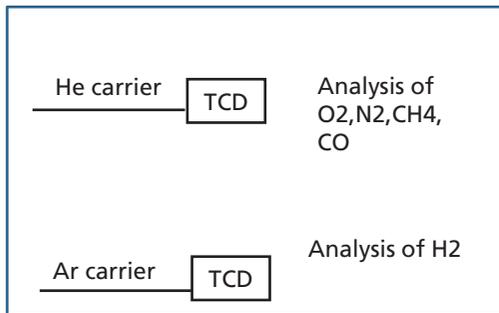
Single Detector Approach for Your Complex Analyses

3. Long-Term Stability

Long-Term Stability with New Discharge Design

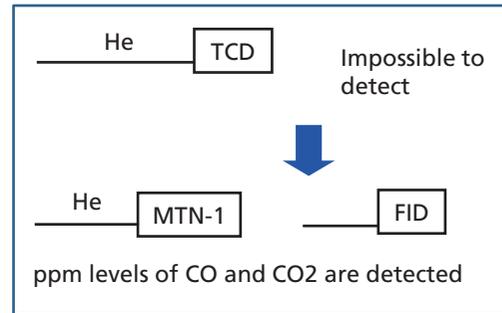
The BID Can Replace Multiple Detection Schemes

Analysis of H₂, O₂, N₂, CH₄, CO



Using two TCDs

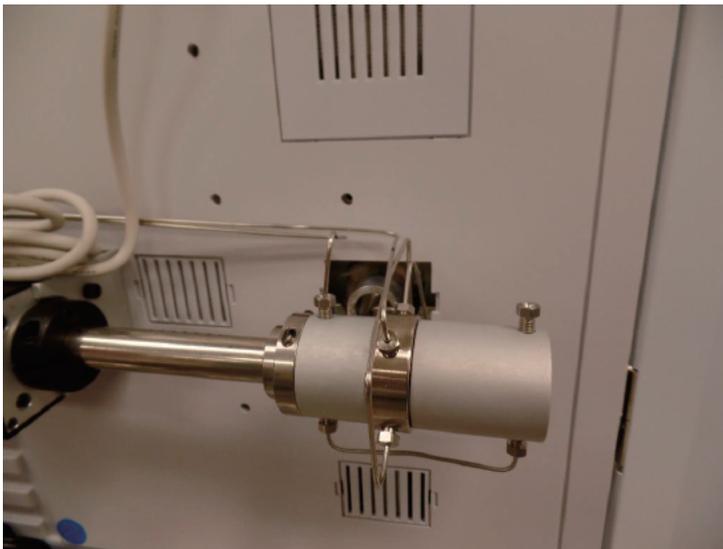
Analysis of ppm level of CO, CO₂



Using a Methanizer and FID

BID will detect all of these analytes at low levels

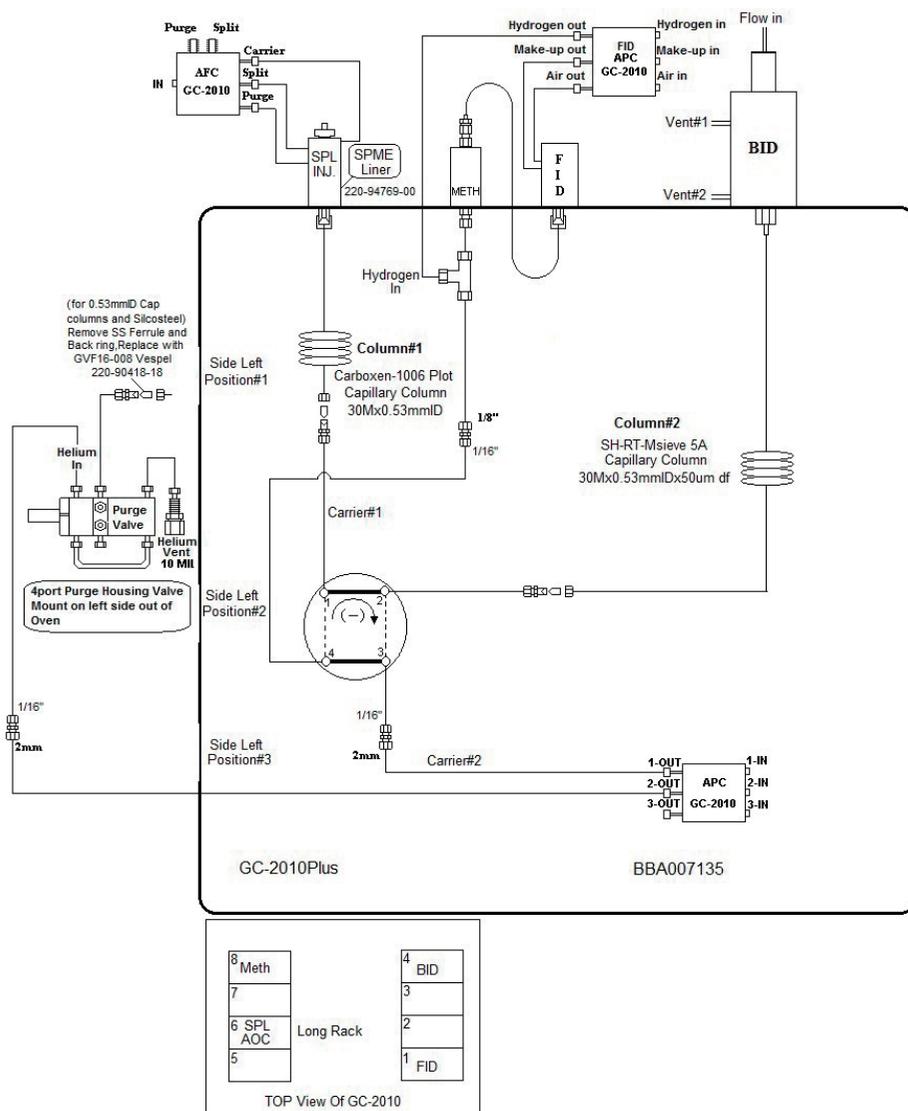
GC-2010 Plus Tracera BID with Valves



Main body of BID-2010 Plus

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ASTM D3612 TOGAS BID Drawing

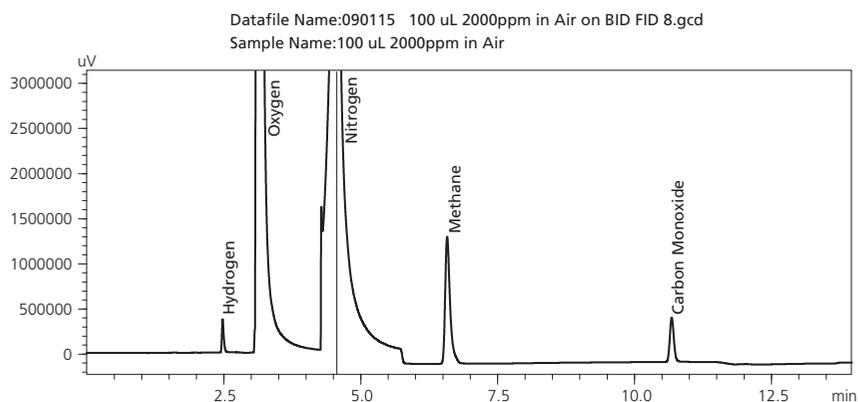


Running Conditions

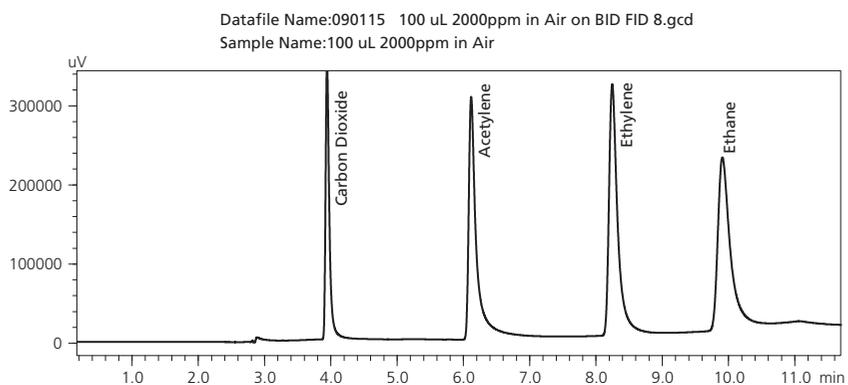
- Columns:
 - Carboxen-1006 PLOT 30mX0.53mm
 - SH-RT-MSieve 5A PLOT 30mX0.53mmX50µm
- Injection mode: Direct; INJ Temp: 100.0 °C; Column flow: 24.0mL/min;
- INJ Pressure: 2.8min at 110.0KPa, 5KPa/min to 150KPa hold 0.20min.
- Oven Temp: 2.8min at 70 °C, 40 °C/min to 95 °C, 7 °C/min to 150 °C hold 0.72min.
- FID: 200.0 °C; H2 65.0mL/min, Air 400.0mL/min; Makeup He 20.0mL/min.
- BID: 200.0 °C; Discharge gas 50.0mL/min.
- 4-port valve timing: 2.8min Event 91; 12.00min Event -91.
- Methanizer Temp: 380.0 °C

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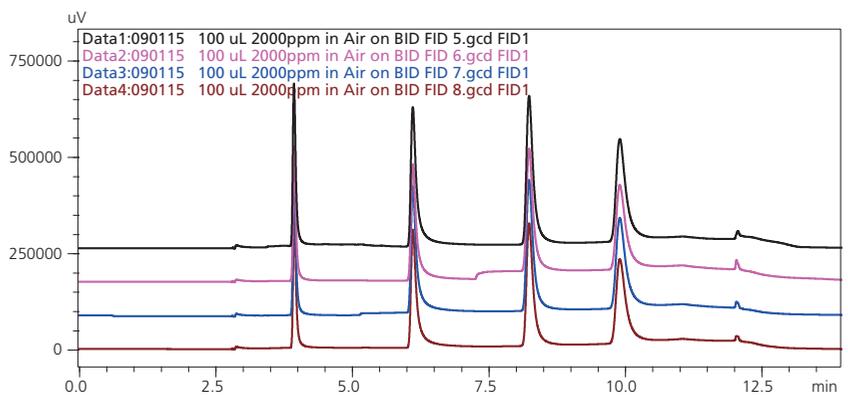
BID Chromatogram



FID Chromatogram

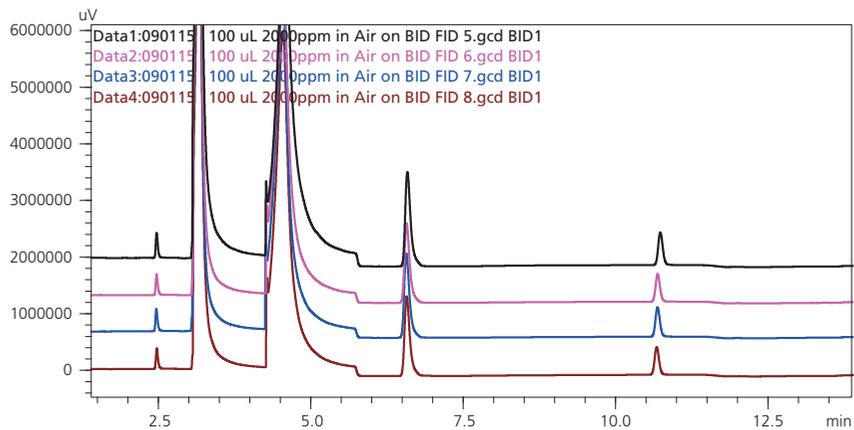


Repeatability FID



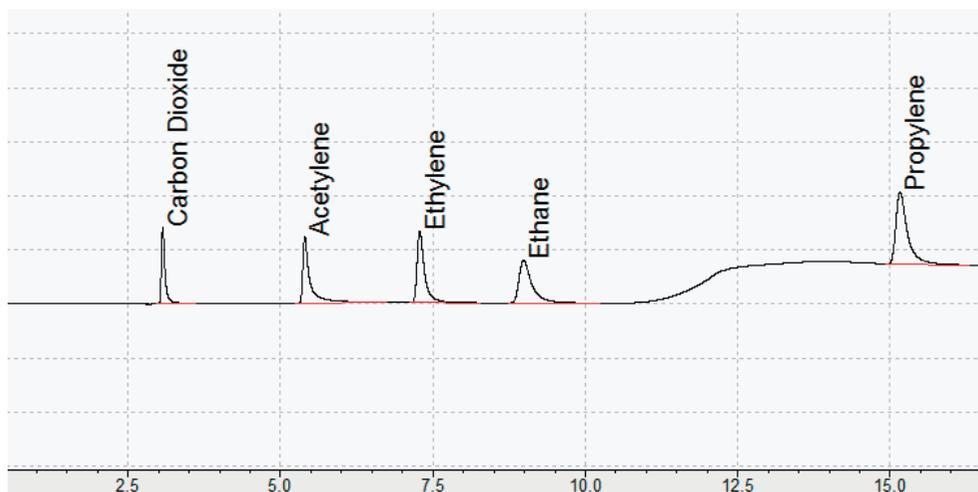
	Carbon Dioxide	Acetylene	Ethylene	Ethane
RSD%(n=4)	9.51	9.51	9.45	9.55

Repeatability BID



	Hydrogen	Oxygen	Methane	Carbon Monoxide
RSD%(n=4)	8.38	4.26	9.12	10.11

Propylene Was Detected at RT 15 Min



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Conclusions

- Hydrogen and Methane were detected at lower concentrations by employing the BID detector.
- Propylene was incorporated into the analysis.
- Butane can be determined, if needed, with a longer run time.
- A headspace autosampler such as an HS-20 or HS-10 can be added for automation as per ASTM3612 Method C.



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