

Application
Data Sheet

No. 18

GC

Gas Chromatography

Water Determination in Liquefied Petroleum Gas using GC BID and Ionic Liquid Column "Watercol"

Water in petrochemical feedstocks can cause problems for processors. Freezing of pipe lines and valves and poisoning of expensive catalysts are just a few examples. Monitoring water in petroleum from an upstream source to the downstream processing plant is critical to insure uninterrupted operation. Unlike the Karl Fischer analysis, the GC water analyzer does not suffer from the adverse effects of the petroleum matrix which can skew the KF results. GC method can bypass the undesirable chemical interference that would otherwise be present like "Sulfur interaction". Shimadzu's proprietary BID and Supelco's water analysis column "Watercol" are combined to separate and measure the water in a formulation of feedstock and provide a sensitive and accurate result. Measurements can be made down to sub-ppm level of water detection.

Instruments Used and Analysis Conditions

Instruments Used

Software	Labsolutions GC
Gas chromatograph	Tracera (GC-2010 Plus A + BID-2010 Plus)
Sample injection	Valco Internal Liquid Sample Injector with Splitter Injection Unit
Gas purifier	Supelco High Capacity Gas Purifier (P/N:29541-U)

Analysis Conditions

Column	Supelco Watercol 1910 60 m x 0.25 mmID, 0.20 µm
Column temperature	35 °C(2.0 min) – 5 °C/min – 150 °C(15 min) Total. 40 min
Carrier gas controller	Constant Linier Velocity
Gas type	Helium(Research grade 99.9999+ %)
Injection volume	2 µL
Split ratio	1:5
Liner velocity	45 cm/sec (Column flowrate 3.78 mL/min)
Transfer line temperature	175 °C (After Internal Liquid Sample Injector to GC column Oven)
Detector temperature	200 °C
Discharge gas volume	50 mL/min(He)

Results

Analysis of water in Liquid Petroleum Products

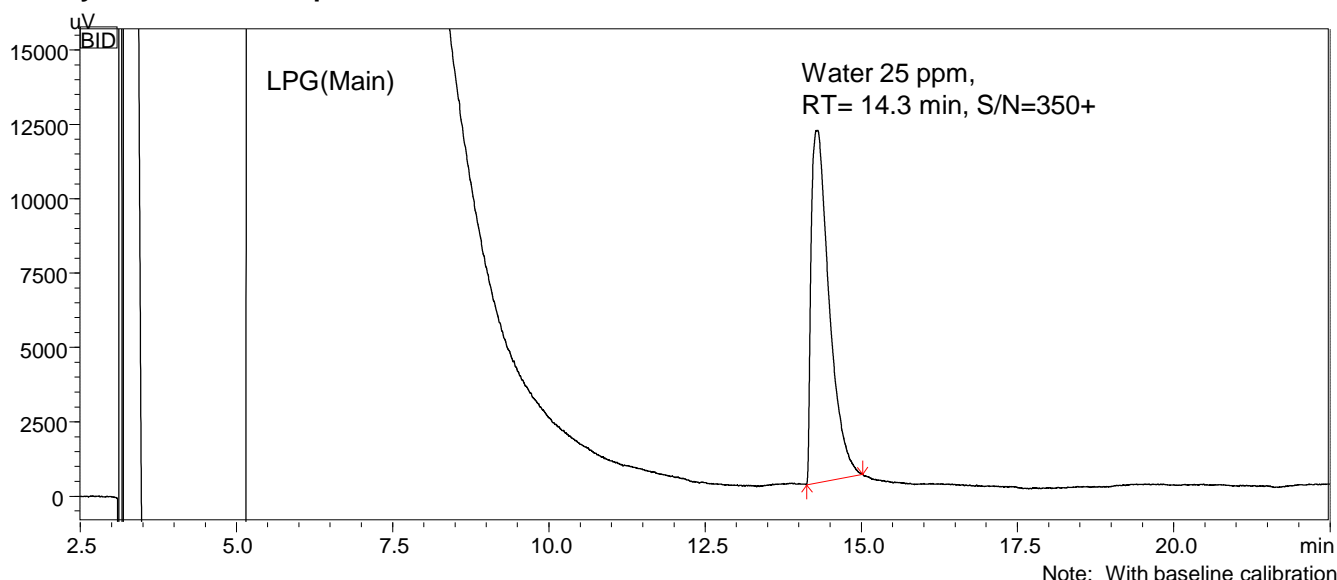


Fig. 1: Chromatogram for water determination (25 ppm) in LPG. Quantification of Limit(S/N=10) and Detection of Limit(S/N=3.3) can be down to 0.66 ppm and 0.22 ppm respectively.

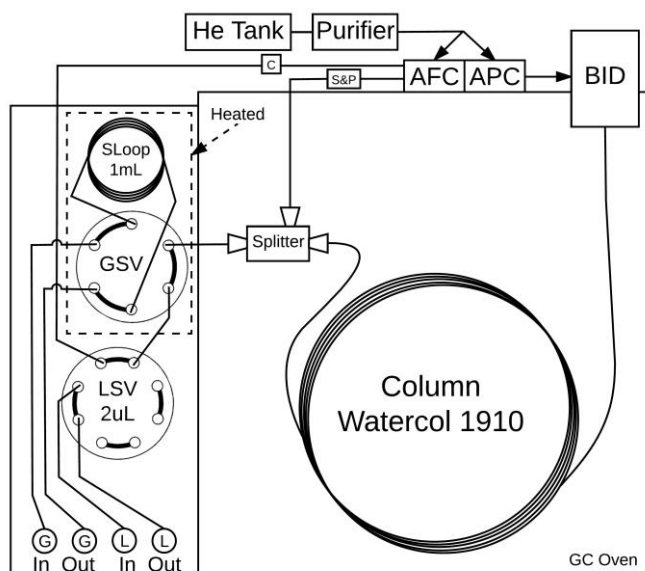


Fig. 2:Flow diagram of GC configuration.



Fig. 3:Tracera(GC-2010 Plus with BID-2010 Plus) equipped with valve oven box.

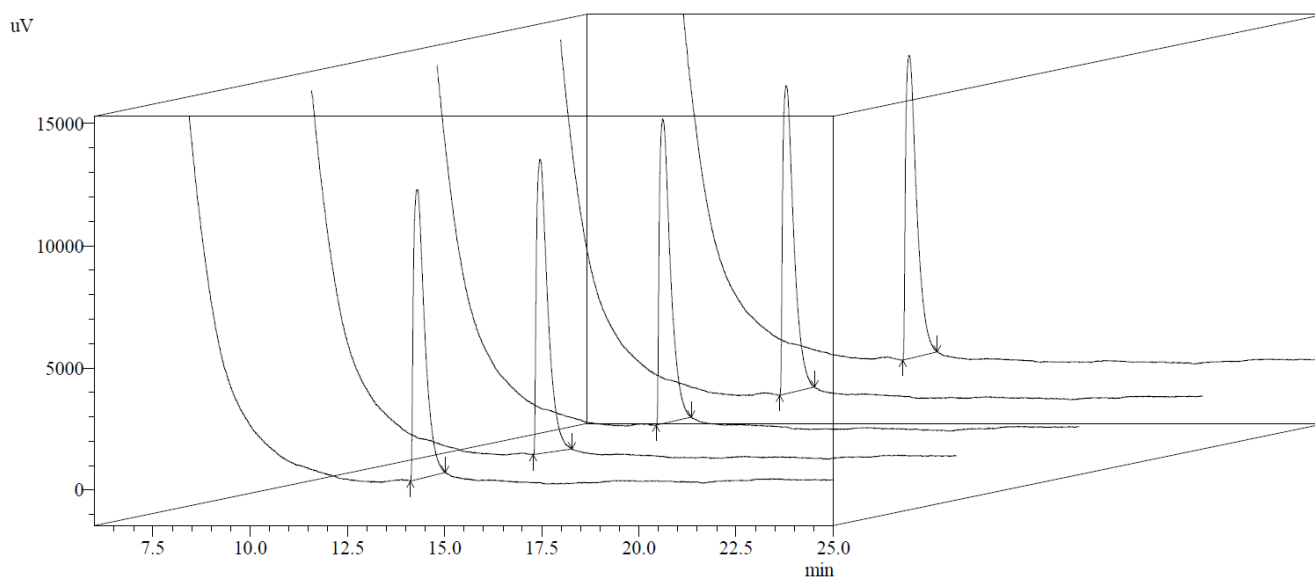


Fig. 2: Overlaid chromatograms of Water in LPG analysis(n=5).

Table 1: Repeatability of Water analysis(n=5).

Water	No.1	No.2	No.3	No.4	No.5	Average	Standard Deviation	%RSD
RT(min)	14.285	14.288	14.286	14.296	14.304	14.292	0.008	0.06
Area(µV·s)	244,037	249,854	246,884	242,950	238,428	244,430	4,296	1.76
Height(µV)	12,418	12,600	12,468	12,045	11,851	12,276	315	2.57
Conc.(ppm)	24.96	25.56	25.25	24.85	24.39	25.00	0.44	1.76

Typical repeatability of water determination (25 ppm) was confirmed as less than 2 %RSD shown in Table1. Besides even if sample contains sulfur component like ethyl mercaptan, Supelco's Watercol can separate water from LPG and sulfur components and Shimadzu's BID can offer trace water detection with GC-level reliability.