

Trace Moisture Analysis System



Analyzing moisture and how much of it is critical to the performance of petroleum products, as well as infrastructure and product integrity. For example, moisture in petrochemical feedstock can cause pipelines and valves to freeze, as well as poison some catalysts. One popular method used to analyze for moisture is Karl Fischer Titration (KFT). KFT has a wide dynamic range but has difficulty in measuring relatively low amounts of moisture. Besides, there are couples of well-known problems like side reaction and interference.

Shimadzu's proprietary BID and MilliporeSigma's* moisture analysis column "Watercol" are combined to separate and measure the moisture in a formulation of feedstock and provide a sensitive and accurate result. Measurements can be made down to ppm level of moisture detection without any interference.

- ▶ Down to ppm levels of moisture analysis using Shimadzu's proprietary Barrier Discharge Ionization Detector (BID)
- ▶ Watercol, MilliporeSigma's* unique Ionic Liquid GC column, enables separation of moisture from typical organic components
- ▶ Leads to more precise moisture analysis than Karl Fischer Titration (KFT)

Moisture Analysis of Liquid Petroleum Gas

A new test method work item (WK59649): A New Standard Test Method for the Determination of Water in LPG using Gas Chromatography (GC) and BID, which offers 100 times higher sensitivity than conventional Thermal Conductivity Detector (TCD) has been submitted by the ASTM D02 H00 committee. This method uses the new GC/BID/liquid/gas sampling valve with the Watercol (MilliporeSigma*) GC column. Be on the lookout for the official method once it is approved by ASTM.

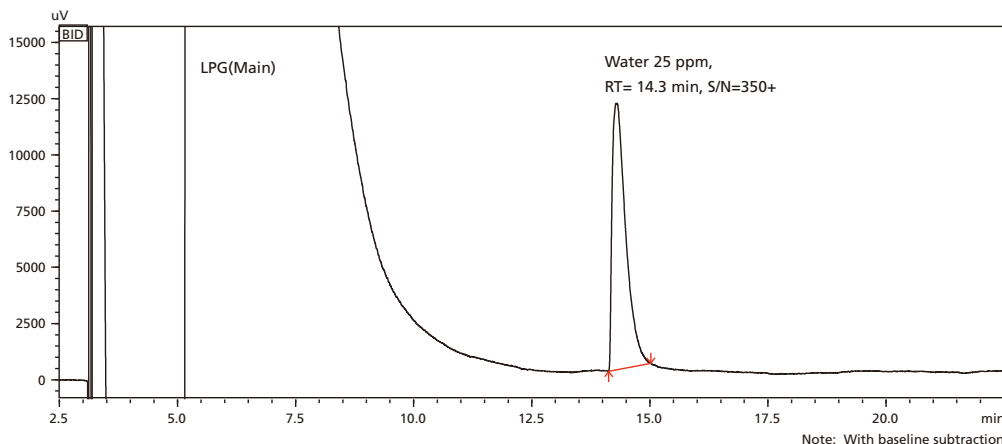


Fig. 1: Chromatogram for moisture determination (25 ppm) in LPG. Quantification of Limit (S/N=10) and Detection of Limit (S/N=3.3) can be down to 0.66 ppm and 0.22 ppm respectively.

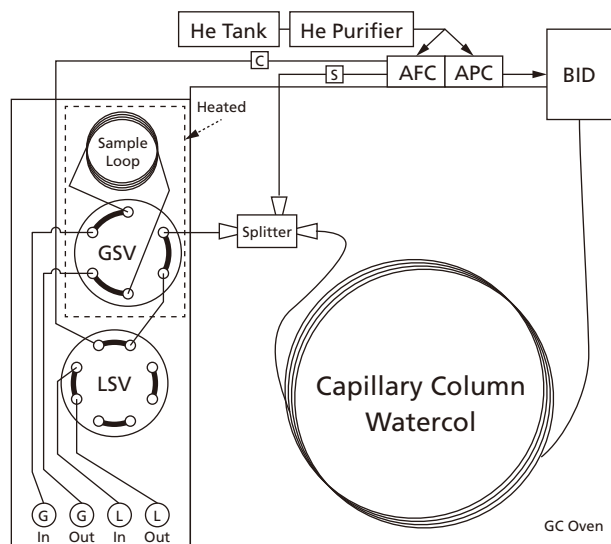
Excellent Repeatability

Typical repeatability of moisture determination (25 ppm) was confirmed as less than 2% RSD shown in Table 1. Besides even if sample contains sulfur component like ethyl mercaptan, MilliporeSigma's Watercol can separate moisture from LPG and sulfur components and Shimadzu's BID can offer trace moisture detection with GC-level reliability.

Table 1: Repeatability of moisture 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($\mu\text{V}\cdot\text{s}$)	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

Flow Diagram of GC system



Trace Moisture Analysis System Specification

Sample Type	Gas, Liquid (there are some restrictions on sample types)
Analytes	Moisture
Target Concentration	Down to 1ppm Moisture
Analysis Time	<15 min
Quantitative Method	External Standard Method (Moisture Standards are offered by specialty chemical vender)
Instruments	GC: Shimadzu Nexis GC-2030 and BID-2030 with Liquid/Gas Sampling Valve Data Integration by LabSolutions LC/GC Software
Capillary Column	MilliporeSigma Watercol 1460/1900/1910
Standard Method	ASTM WK59649 (Ongoing)
Carrier Gas	Helium (Purity >99.9999%) Pressure 500-900 kPa Helium Purifier is needed as well.
System Dimension and Weight	Height : 44 cm (17.3 in) Width : 51.5 cm (20.3 in) Depth : 54 cm (21.3 in) Weight : 43.5 kg (96 lbs) (Valve oven box would be attached to GC body depending on sample types.)

*MilliporeSigma, the life science business of Merck KGaA
Watercol is a trademark of MilliporeSigma.



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