

Application News

Gas Chromatography

Using the Shimadzu GC System in the Fuel-Grade Ethanol Production Laboratory

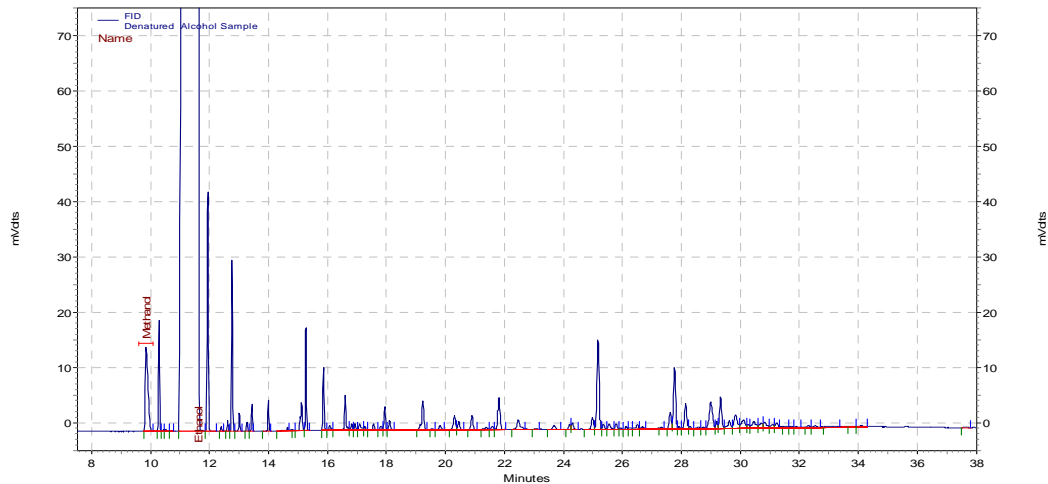
Gas Chromatography (GC), sometimes referred to as gas-liquid chromatography, is a mature chromatographic procedure in which the moving phase is a gas. A stationary phase, a liquid coating on a solid support or tubing is also necessary. Over time, highly efficient GC columns have been developed, based on the use of narrow fused silica capillary tubing and very thin polymeric coatings on the inside walls of these capillaries. This has led to analyses that show great levels of chromatographic separation, an important consideration when the sample can have many peaks.

With GC the stationary phase and the mobile phase (a carrier gas, such as helium) are fixed throughout the analysis. The only variables that can be used to change the separation conditions are the carrier gas flow rate and the temperature of the oven. The separation of the molecules in the sample is strongly dependent on the affinities of the analyte molecules for the stationary and mobile phases as the analytes move toward the detector. If the linear velocity of the carrier gas is held constant, then the kinetics of the interactions between the sample components and the column liquid phase is largely determined by the temperature of the column. Increasing the column temperature in a reproducible manner, during the analysis, can often improve the separation of many compounds and greatly shorten the analysis time.

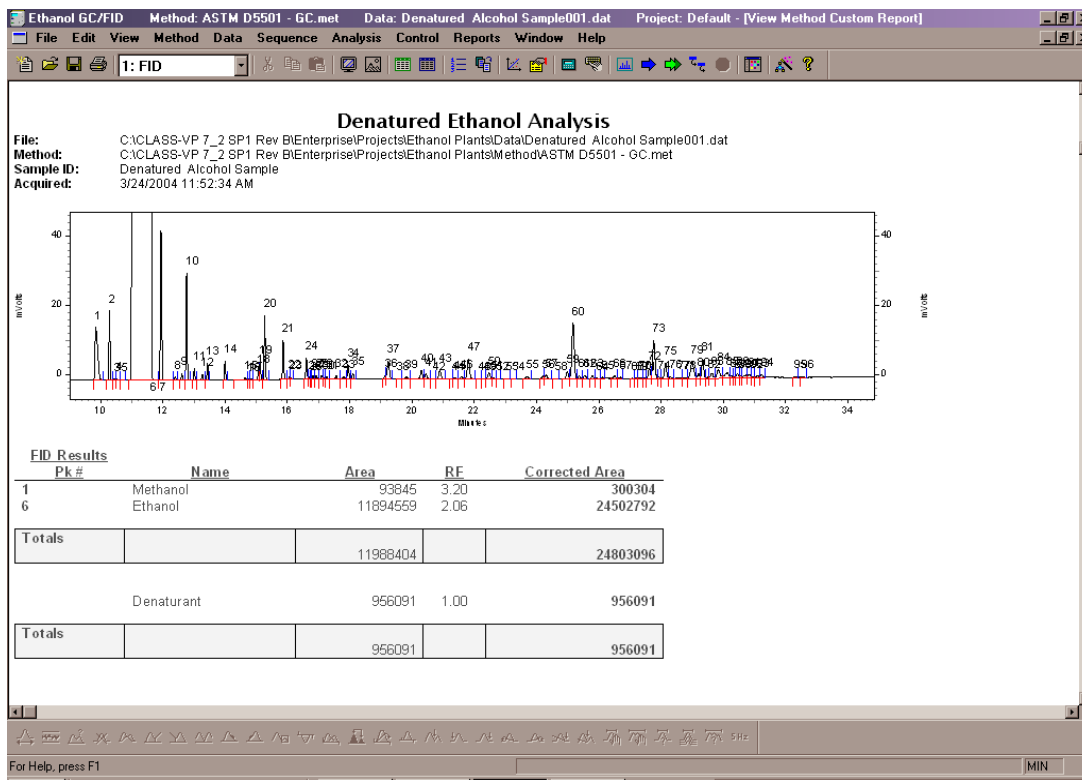
In the case of the ethanol production plant, GC analysis is used to determine the total amount of denaturant added to the final ethanol product prior to shipment. This analysis is required in certain ethanol markets. The denaturant, being a petroleum distillate product, can contain hundreds of different compounds. The GC analysis for **ASTM D5501** certification measures the amount of methanol, ethanol, and total amount of denaturant as area percents. These area percents are corrected, per the ASTM method, to give corrected volume percentages for these materials.

The GC equipment that is used to perform the certification analysis is comprised of several components that make the analysis easy and reliable. The necessary components are 1) the mobile phase, helium, 2) a temperature-programmable gas chromatograph, 3) an autosampler for sample introduction, 4) the analytical capillary column, 5) a flame ionization detector, and 6) the data acquisition system. The quality of the gas chromatograph is determined by the consistency of its temperature programming and its ability to accurately control the carrier gas flow.

Injection of the denatured ethanol finished product for GC analysis results in a representative chromatogram.



The data system method is configured to determine three critical values: the methanol peak area, the ethanol peak area, and the sum of all other peak areas, which is referred to as “denaturant”. An example of a typical GC analysis report is shown below.



From these areas, the ethanol lab is able to calculate the mass response corrected area percentage of these components. Applying the appropriate specific gravity and Karl-Fisher water analysis values into the calculation, the ethanol lab can provide a certificate of analysis that accurately details the percentage of methanol and denaturant in the fuel-grade ethanol product.

Typical Instrumentation: Gas Chromatograph: GC-17AAF, GC-2014 or GC-2010; Automatic Injection System: AOC-20i; Analog-to-Digital Converter: SS420x Interface (GC-17AAF only); Shimadzu CLASS-VP Software; Capillary Column: Restek Rtx-1 PONA, 100 m x 0.25 mm i.d., 0.5 µm film; Temperature Program: 35°C (Hold 12 minutes), 30°C/min temperature ramp to 250°C (Hold 19 minutes).