New Adapter for On-the-Fly Sampling During Distillation

Contributed Article
The following was generated with the assistance of an outside source using Sigma-Aldrich products. Technical content was generated and provided by:
Thomas J. Bruno¹ and James J. Breier²
1. National Institute of Standards and Technology (NIST), Thermophysical Properties Division, Boulder, Colorado, USA
2. Sigma-Aldrich, Milwaukee, Wisconsin, USA
mike.buchanan@nist.com

Introduction
Distillation is the most widely used bulk separation method used in the laboratory as well as industry. Beyond purification, it is widely used to characterize complex fluids (such as fuels) through measurement of the distillation curve, a plot of the boiling temperature against volume distilled. A common theme in both of these applications is the desire to understand the composition. In purification, the goal is to monitor the distillation progress, and in fluid characterization, one seeks to relate the composition to the temperature data.

Features and Benefits
A distillate sampling adapter (Figure 1) installed following a condenser or distillation column can provide this important capability without the need for cumbersome, expensive, and often unreliable fraction collectors. The flow of the distillate is focused to drop into a 0.05 ml “hammock” that is positioned directly below the flow path. The sampling port, equipped with a vacuum tight valve, allows access to the hammock with a standard chromatographic syringe, through a septum. To sample the distillate, one simply positions the chromatographic syringe, preferably equipped with a blunt tipped needle, in the well of the hammock. It is a simple matter to withdraw samples as the distillation progresses. The sample can then be directly injected into the gas chromatograph or spectrometer, or injected into an autosampler vial for analysis later. Indeed, any analytical technique that is applicable for liquid samples ranging in volume from 1 to 50 microliters can be used to characterize the distillate.

Application Examples
This adapter has been used for many complex fluid analyses, including gasolines (including oxygenates and bio-derived alternatives), diesel fuels (including oxygenates and biodiesel fuels), rocket kerosenes, jet fuels, crude oils (including bio-crudes), transformer fluids, waste oils, and aeronautical accelerants. Some of the analytical techniques applied to distillate fraction analysis have included gas chromatography (with mass selective, flame ionization, and chemiluminescence detection), FTIR spectroscopy, Karl Fischer coulometric titration, and refractometry. The ability to easily and quickly couple quantitative analysis with the distillation opens the door to thermochemical determinations such as the enthalpy of combustion of fuels, as a function of distillate cut. The adapter has also been used to measure corrosivity of crude oil fractions, with a copper coupon test performed at various distillate cuts.

Conclusion
The distillate sampling adapter is offered with several joint options and is designed to work with existing modular distillation glassware. It is simply a matter of replacing the existing vacuum adapter with a distillate sampling adapter. No further modification to the setup is required. The simple concept eliminates the need for cumbersome, expensive, and often unreliable fraction collectors.

References