CASE STUDY

Food Analysis

Customer Story:
Tsingtao Utilizes Headspace Sampling to Ensure World-Class Beer Quality Standards

Tsingtao Brewery Co. is ranked as the largest brewery in China and the sixth largest in the world. Maintaining the stability and consistency of product quality among the company's many plants is very important for the branding of Tsingtao Beer. The technical center of Tsingtao recently established standard operating procedures designed to identify problems or changes occurring in the brewing or fermentation process that affect the taste and quality of the final product.

Tsingtao worked with PerkinElmer technical staff in China to select the best instruments and methodologies to maintain the stability and uniform quality of Tsingtao's products. The instrumentation solutions are based on PerkinElmer's TurboMatrix headspace trap and Clarus gas chromatography (GC) solutions for comprehensive analysis of flavor compounds and defects in beer. These instruments have been installed in more than 30 plants and are playing an important role in helping Tsingtao Brewery improve its market positions in the highly competitive global beer market.

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Tsingtao Brewery was founded in 1903 by German settlers in Qingdao, China. Since its introduction, Tsingtao Beer has grown to become the number one beer in China. Introduced to the United States in 1972, Tsingtao soon became the top-selling Chinese beer in the U.S. market and has maintained this leadership position ever since. The Tsingtao Brewery itself has grown from four plants in 1996 to 48 today. Tsingtao Beer, a well-hopped standard pilsner with 4.7% alcohol content, accounts for most of the company's production. The Tsingtao brand is sold in more than 50 countries worldwide and accounts for more than 50 percent of China's total beer exports. In fact, Tsingtao is the number-one branded consumer product exported from China.

Challenge of maintaining consistent beer quality

Beer is a highly complex mixture of many compounds including sugars, proteins, alcohols, esters, acids, ketones and terpenes. The chemical content of beer is responsible for its flavor. Aroma is an important part of beer flavor and the aroma is largely determined by the volatile organic compounds (VOCs) in beer. Breweries around the world are focusing more attention on characterizing the chemical content of beer products as part of process control, quality assurance and product development. Traditionally, Tsingtao relied upon human taste testers to ensure the quality of its product but more recently it has been supplementing human testers with scientific and molecular analysis that provides objective and measurable quality standards.

Tsingtao's technical center is responsible for developing standard testing methods and rolling them out to the company's breweries. The Tsingtao technical center identified two key areas to begin monitoring the company's product. 2,3-butanedione and 2-3-pentanedione are vicinal diketones (VDK) that produce a butter-like flavor and can cause an adverse odor at high levels. VDK concentrations typically range from 1-50 ppb in lighter beers but they can reach several hundred ppb in darker beers. Acetaldehyde is reduced to ethanol by yeast during secondary fermentation but oxidation of the finished beer may reverse this process and convert ethanol back to acetaldehyde. Acetaldehyde has the taste and aroma of fresh-cut green apples and has also been compared to grass. Typical levels of acetaldehyde are in the range of 1 to 20 ppb.

Headspace sampling is the state-of-the-art method for sampling the aroma of beer and other food products. The beer sample is placed into a vial and sealed. The vial is heated to release the vapor into the headspace or empty area of the vial. The vapor is then extracted and analyzed using gas chromatography. At equilibrium the concentration in the headspace phase is proportional to the original concentration in the sample. Determining the concentration of the headspace phase enables the composition of the sample to be established. Polar compounds in beer are more soluble in water than in air so only less than 0.5% of the compound in the sample may pass into the headspace. The headspace trap technique can enhance detection limits by a factor of 100 by injecting the entire headspace volume into the trap, pausing to allow the headspace to refill with vapor and repeating the injection process several times.

Selecting equipment and developing method

Tsingtao selected PerkinElmer as its primary instrumentation partner because of PerkinElmer's technological leadership in headspace sampling and its strong technical team in China. Six PerkinElmer gas chromatography application specialists based in both Northern and southern China worked together with the Tsingtao technical center to develop analysis methods for VDKs and acetaldehyde. PerkinElmer also leveraged its global resources by providing technical expertise based in the U.S. to assist with the application. These analyses are performed using a PerkinElmer TurboMatrix automated headspace sampler and a Clarus 500 GC. The Clarus 500 GC was configured with an electronic capture detector (ECD) for VDKs and with a flame ionization detector (FID) for acetaldehyde.

Beer samples are degassed prior to headspace analysis to prevent dissolved carbon dioxide from influencing vial pressure during the headspace heating process and to minimize GC baseline disturbances from CO_2 eluting during chromatography. A sample of beer is placed into a headspace vial. A typical chromatogram showing the presence of VDK is shown in Figure 1. A chromatogram showing the presence of acetaldehyde along with 2-propanol, which is used as an internal standard, is shown in Figure 2. Both analyses can be performed simultaneously on the same HS-GC system by splitting the GC column effluent between the FID and ECD detectors and using the column to separate all of the components. If these screening tests indicate that the targeted components exist at undesirable levels, then more specific analyses can be performed as follow-up.

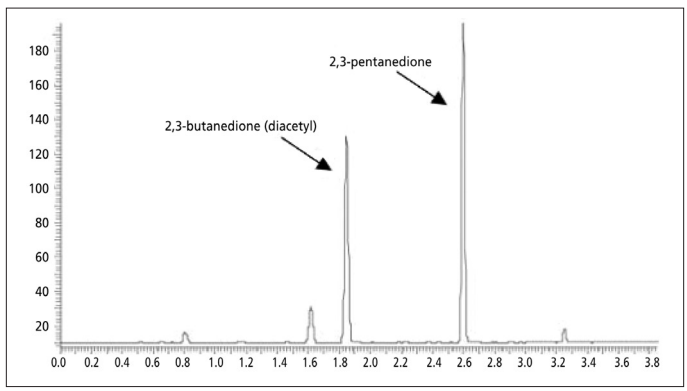

Figure 1. VDK determination at 10 ppb concentration with ECD.

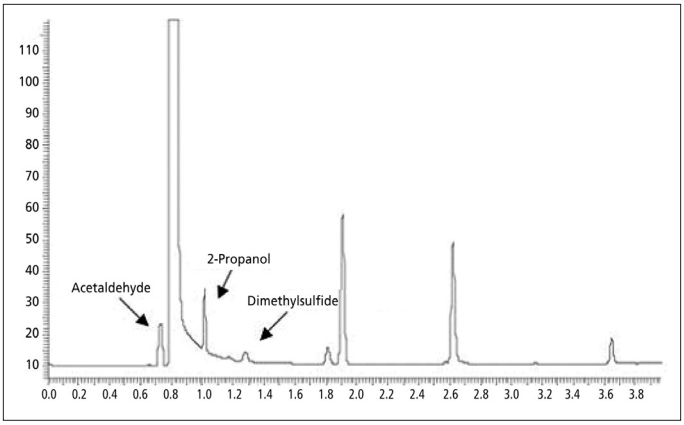

Figure 2. Acetaldehyde determination at 10 ppm concentration with FID.

Impact of new methods on quality and product development

Once the final methods were developed, they were rolled out to breweries across China. In addition to monitoring product quality, the instruments are used to quickly determine the root cause of any quality incident that might occur. For example, a batch of beer products was once found to contain an unusual odor. The whole batch of beer was removed from the product flow and samples were sent to the technology center for analysis with solid phase micro extraction (SPME), another technique for extraction and concentration of VOCs and GC with mass spectroscopy (MS) detectors. Total ion chromatography (TIC) results were obtained only two hours later and compared to the analysis results for the regular product. A small phenol peak was found in the TIC of the controlled products, leading to the identification of the root cause of the problem.

Scientific analysis is also providing valuable input to the product development process. The technology center runs flavor fingerprint analysis with SPME-GCMS to help determine the differences between Tsingtao beers and other branded products in the market. The results are studied to determine why certain products are preferred by specific groups of consumers and guide new product development. New products developed by the research and development department are tested in the same way.

The quality control solution of analyzing the VDK and acetaldehyde of the beer with TurboMatrix headspace and Clarus GC is playing a very important role in maintaining the stability and uniformity of Tsingtao's products. By using scientific analysis to supplement human testing, Tsingtao has ensured even higher levels of consistent quality and improved the product development process.

Using PerkinElmer GC-HS with PFPD detector to analyze ppt level sulfur in beer sample.

Using PerkinElmer GC/MS to analyze the fragrance of hops in beer.

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