2017 ASBC Annual Meeting

Rapid Testing methods for Beer Analysis using Infrared Spectrometry and Quality Trait Analysis

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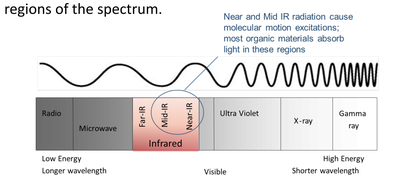
2017 ASBC Annual Meeting
June 4–7, 2017
Sanibel Harbour Marriott
Fort Myers, FL, U.S.A.

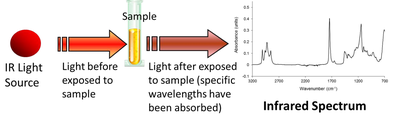
PURPOSE

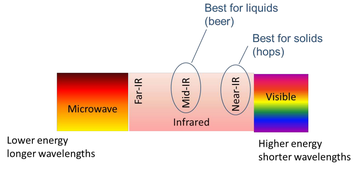
With higher demands in quality and consistency in the craft brewing industry, effective quality control analysis is required. However the investment in a full lab for the instrumentation and personnel can eliminate the profitability. In response to the industry needs for rapid testing and minimal investment, Eurofins Quality Trait Analysis (QTA) has developed a beer testing protocol in which scanning and results are completed in ~2 minutes with no sample preparation. In addition this system can also analyze hop quality in pellets and cones.

Infrared (IR) Analysis

Infrared light is between the visible and microwave regions of the spectrum.







IR Analysis

Advantages
- Rapid Analysis (2 minutes)
- No (or minimal) sample preparation
- Reagentless
- Minimal instrument maintenance
- Multicomponent Analysis

Disadvantages/Challenges
- Expertise in *chemometrics* needed for creation and maintenance of robust, reliable IR calibrations.
- Most labs do not have a chemometrician on staff

MATERIALS AND METHODS

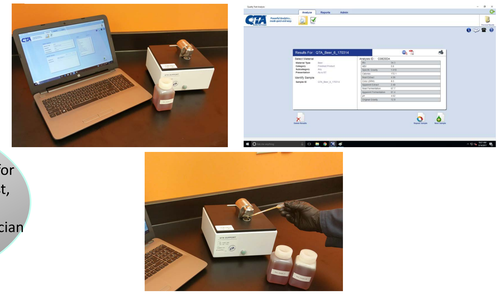
Near IR (NIR) Analysis for Dried Hop pellets or cones: For hop samples, spectra were collected on a Fourier Transform Infrared (FTIR) spectrometer. Spectral range 4000 – 12,000 cm^{-1}, resolution 8 cm^{-1}. Spectra were collected in duplicate for 98 samples encompassing cascade , Ctz, Chinook, el dorado, Amrillo and crystal strains of hops in pellet and cone forms.

Mid IR (MIR) Analysis of finished product beer or wort: For finished product beer and in process wort samples, spectra were collected on a FTIR with adjustable pathlength tumbler for transmission measurement. Spectral range 650 – 4000 cm^{-1} with a resolution of 8 cm^{-1}. Spectra were collected in duplicate for 126 beer samples representing several craft breweries in the Cincinnati area and surrounding cities as well as common varieties found in the grocery store. Spectra from 192 wort samples from brewers in the Ohio region were collected also in duplicate. The samples collected for both beer and wort included a variety of IPA, lager, stout, and porter varieties.

Calibration: The goal of infrared spectral analysis is to produce a result in rapid time that matches as closely as possible to the result obtained from a method traditionally used for testing. Methods traditionally used for testing usually involve sample preparation, reagents, high cost instrumentation, time, and resources. To use infrared instrumentation and obtain a result for a property, a calibration must first be created. The calibration is built by creating a regression equation which relates the spectra to a result for a given property (ie, Alcohol by Volume, ABV). These regression equations can be very complicated and use multivariate mathematical tools to decompose the spectral information. This realm of chemistry is called *Chemometrics*. In order to build a calibration for ABV, a set of samples must be analyzed for ABV by traditional lab methods, and the spectra of those samples must be collected from the IR instrument to be used. Regression algorithms called Partial Least Squares (PLS) are are used for all calibrations. PLS is one of the most commonly used tools for IR calibrations which require multivariate regressions.

Lab Methods for Calibrating the IR spectra: IR results are calibrated to traditional lab methods for beer and hops. Lab methods for each trait are as follows: Alcohol by Volume (ABV) was measured according to the American Society of Brewing Chemists (ASBC) method 4G (using Near Infrared and Original Extract Content), specific gravity according to ASBC method 2B (digital density meter). For free amino nitrogen in wort ASBC method 12 (ninhydrin) was used. For alpha and beta acids as well as Hops Storage Index, ASBC method 6A was used. Each of these methods are detailed on the ASBC website (methods.asbcnet.org). All other traits for finished product beer and wort were measure by an Anton Paar DMA 4500 density meter with attached Beer Alcolyzer.

Analysis of Finished Product Beer using Mid-IR Instrument



Analysis of Hop Flower or Hop Pellet using Near-IR Instrument

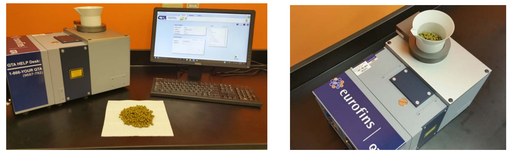
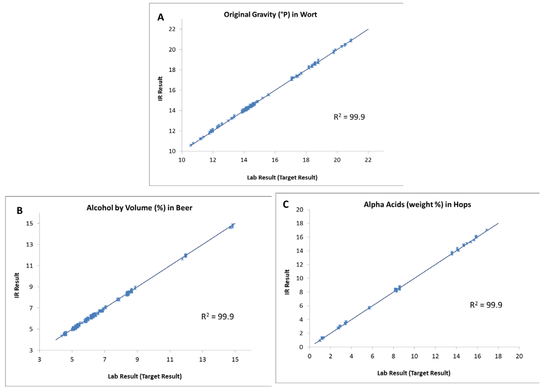


Figure 1. Correlation Plots illustrate how close the IR result is to the testing method traditionally used. The goal is to have the IR result match the traditional method as closely as possible.



IR Method Error – In plots A-C above, the vertical distances of the data point to the line represent the IR errors. The subtractive difference: IR Result – Lab Result is the error. All data points in the calibration contribute to the error computation.

The **Standard Error of Prediction (SEP)** is the IR method error, computed as a standard deviation:

$$SEP = \sqrt{\frac{\left(\sum_1^n (\text{IR result} - \text{Lab result})\right)}{n}}$$

Table 1. List of Current IR methods for Beer, Hops, and Wort including range of levels included in the calibration as the Standard Error of Prediction (SEP) of each Calibration

Material/Product	Trait	Range	SEP
Beer	ABV (%)	4.36 - 14.85	0.25
	IBU	4 - 102	7.0
	FAN (%)	24 - 315	35
	Specific Gravity	0.9981 - 1.028	0.0008
	Real Extract (°P)	1.04 - 9.54	0.15
	Apparent Extract (°P)	1.44 - 7.02	0.25
	Original Gravity (°P)	7.48 - 21.38	0.35
	Real Fermentation (%)	55.16 - 86.56	1.5
	Apparent Fermentation (%)	64.97 - 106.6	1.7
	Calories (per 12 oz)	92.47 - 297.5	4.5
	Color (SRM)	1.59 - 183.6	7.0
	pH	3.43 - 5.02	0.07
Wort	IBU	16 - 99.5	6.0
	Specific Gravity	1.043 - 1.086	0.003
	FAN (%)	190 - 310.1	8.5
	Original Gravity (°P)	10.6 - 20.9	0.35
Hops	Alpha Acids, %	0.7 - 18.3	0.5
	Beta Acids, %	2.9 – 7.4	0.3
	Hops Storage Index	0.229 – 0.356	0.01

Conclusions

- QTA analysis offers rapid analysis for both hops and finished product beer.
- Reagentless, multi-component analysis, no sample preparation.
- Results display in ~ 2 minutes.
- Instrument expertise, knowledge in spectroscopy/chemistry not required.
- Quality Trait Analysis (QTA) has several chemometricians on staff dedicated to development and maintenance of reliable calibrations.
- QTA calibrations can be customer-specific or industry wide, according to customers request.