APPLICATION NOTE

High precision nitrogen and protein analysis in beer brewing

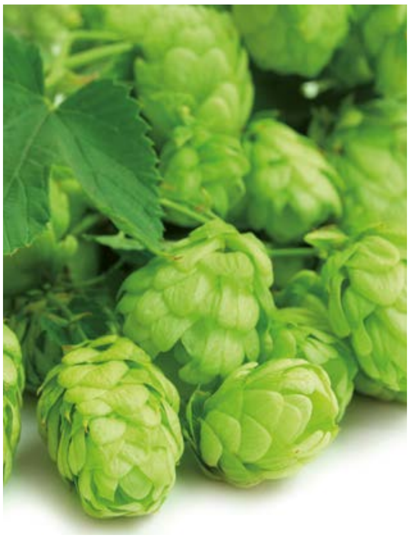
Introduction

The brewing process is affected at multiple stages by the protein content of the associated liquids and solids, ultimately influencing total protein content of the beer. Protein content of beer is related to many of its key properties, most notably the flavor, as well as foam building and retention capabilities. The ability to measure protein concentrations in grains of the grain bill, as well as in wort, clarifying agents, spent grain, and beer itself, increases the quality and profitability of any brewing operation. Thus, protein determination methods need to be accurate for a wide variety of sample types.

BEER AND BREWERIES

rapid MAX N exceed

The protein content of food and agricultural products is often determined by measuring the total nitrogen content of a sample and multiplying by an appropriate factor, as outlined in many international norms, including those from the American Society of Brewing Chemists (ASBC), the Association of Official Agricultural Chemists (AOAC), and the European Brewery Convention (EBC). The Dumas combustion method provides a reliable and matrix-independent option for determining total nitrogen in a wide variety of samples.



This cost-effective method involves high-temperature combustion of sample followed by separation and detection of the resulting gases, without needing toxic or corrosive solutions. Measurements can typically be completed in a few minutes with little-to-no sample preparation. This Dumas combustion approach facilitates frequent protein monitoring for all stages during the brewing process and provides timely results for any needed corrections to reliably maintain the expected product taste and texture.

Instrumentation

With over 110 years of experience producing elemental analyzers and more than 50 years of experience producing dedicated N/Protein analyzers, in 2015 Elementar released the rapid MAX N exceed analyzer, which combines high-throughput and ease of operation with reliable determination of nitrogen, even at low concentrations and in difficult samples. The 90-position autosampler utilizes stainless steel crucibles that can hold up to 5 mL of liquid or 5 g of solid. Enabled by easy-to-use software and a flexible autosampler, time-critical samples can easily be added to any tray position and run immediately without rearranging previously loaded samples.

The crucibles are introduced to the combustion furnace by a smart-design gripper arm which includes the oxygen inlet. By dosing the oxygen directly on top of the sample, less oxygen is necessary to get complete combustion, which is a key aspect of our unrivaled low price-per-sample. Further savings are made by our proprietary EAS REGAINER® and EAS REDUCTOR® technology. This system utilizes a non-toxic, metal-free method for binding excess oxygen and regenerating the metals that reduce the nitrogen oxides from combustion to nitrogen gas for reliable detection. Additional savings can also be made by using argon, instead of the typical helium, as a carrier gas.

Because the rapid MAX N exceed can measure up to 1 g of organic material, samples can be quite heterogeneous, such as milled malt or spent grain, and still yield accurate, reproducible results. With a robust three-stage gas drying system, routinely measuring several grams of aqueous solutions, like wort, syrups, or beer, present no challenge for the instrument. In addition, Elementar's unique, upright crucibles can be used for liquids or solids without the need for any chemicals or additional materials, such as sample liners or absorbers.

International Standards

Many international standards have been written which outline the precision to which protein measurements within the brewing process must be made. "Nitrogen in Beer, Wort, and Brewing Grains, Protein (Total) by Calculation, Combustion Method", **AOAC 997.09-2008**, also includes sample results for malt and other brewing products. The stated precision for beer is less than 0.75 %RSD or 0.002 %N (20 mg/l) SD and the sample data for malt has a RSD of 2.2%. "Crude Protein in Grains and Oilseeds, Generic Combustion Method", **AOAC 992.23**, requires a RSD less than 2% for ten consecutive measurements. The EBC has separate standards for barley (3.3), malt (4.3), wort (8.9), and beer (9.9). Each method has a Kjeldahl and Dumas version. For example, "Total Nitrogen in Beer: Dumas Combustion Method", **EBC 9.9.2**, specifies a precision of 2% RSD. Lastly, "Food products -- Determination of the total nitrogen content by combustion according to the Dumas principle and calculation of the crude protein content -- Part 2: Cereals, pulses and milled cereal products", **DIN EN ISO 16634-2**, requires that the largest difference between two sample measurements to be less than 0.1 %N. These are a few examples of the many international standards for determining protein content in food and beverages by means of the Dumas method.

Measurements

To demonstrate the suitability of the rapid MAX N exceed for the measurement of beer and brewing products, four different data sets are presented here. Starting with the source grain, the flour of four different grains, representing the diversity of a potential grain bill, were analyzed in ten replicates, with 500 mg per replicate. The average difference between two successive analyses (diff. N) and the relative standard deviation (RSD) was calculated to compare to international standards AOAC 992.23 (RSD < 2%) and ISO 16634-2 (diff. N < 0.1%). The results are summarized in Table 1 below. Next, the analysis of three types of malt were performed and compared to sample data from AOAC 997.09-2008. Each sample was measured five times, with 500 mg for each measurement. The results are summarized in Table 2 below. Finally, six different wort samples and six different beer samples were analyzed on the rapid MAX N exceed. In order to avoid foaming and to remove CO_2, the beer was filtered twice before analysis. The samples were measured into the standard stainless-steel crucibles. The upright crucible design is ideal for liquid samples. For all samples, 2.5 ml were used for each measurement. The wort samples were measured four times and the beer samples were analyzed ten times each. The average nitrogen content and the resulting absolute standard deviation (SD) are presented below and compared with the maximum accepted standard deviation according to the international EBC standards 8.9.2 and 9.9.2. The results are summarized in Table 3 below and Table 4 on the following page.

Table 1. Results for four different flour types. Diff. N is the average difference of two successive measurements.

SAMPLE	N [%]	PROTEIN [%]	RSD [%] (AOAC 992.23: < 2%)	DIFF. N [%] (ISO 16634-2: < 0.1%)
wheat flour	1.78	10.1	0.46	0.012
rye flour	1.25	7.13	0.61	0.008
buckwheat flour	1.49	9.30	1.24	0.019
grape seed flour	1.91	11.9	0.93	0.019

Table 2. Results for three different malt types. The AOAC 997.09 sample data has an RSD of 2.2%.

SAMPLE	N [%]	PROTEIN [%]	RSD [%]	Factor better than AOAC
wheat malt	1.62	10.1	0.54	4.1
summer barley malt	1.77	11.1	0.80	2.8
Munich malt	1.61	10.0	0.23	9.6

Table 3. Results for two different wort types and six different beer types. The AOAC 997.09 requirement is 20 mg/l N SD.

SAMPLE	N [mg/l]	SD [mg/l]	EBC SD [mg/l]	Factor better than AOAC
wort from summer barley	677	4	14	5
wort from winter barley	655	10	13	2
wort from winter barley	709	6	14	3
wort from winter barley	649	6	13	3
wort from winter barley	660	10	13	2
Kara (dark) wort	751	2	15	10

Table 4. Results for six different beer types. The AOAC 997.09 requirement is 20 mg/l N SD.

SAMPLE	N [mg/l]	SD [mg/l]	EBC SD [mg/l]	Factor better than AOAC
German pilsner	640	3	13	7
German wheat beer	750	5	15	4
German double bock beer	1250	5	25	4
Guiness	580	3	12	7
German export beer	800	6	16	3
German non-alcoholic beer	390	2	8	10

Summary

In the brewery process, a variety of liquid and solid materials, and their protein content, play an important role in the quality and properties of the final product. The various steps and processes are covered by a collection of international standards.

The rapid MAX N exceed from Elementar has been shown to be capable of measuring the various components of the brewery process significantly better than specified by the applicable standards. Beginning with the raw materials through malt and wort on to the final product, the measurements made by the rapid MAX N exceed are consistently at least twice as precise as required, and often as much as ten times more precise.

The crucible sample-handling technology utilized in the rapid MAX N exceed makes switching between liquid and solid samples trivial, requiring no additional chemicals or supplies. The tall upright crucibles easily handle liquids or light powders without concerns of sample loss. The possibility for higher sample weights means that even inhomogenous samples can be analyzed with little-to-no prior treatment.

The innovative EAS REGAINER® and EAS REDUCTOR® technology contribute to long instrument uptime and low cost-per-sample. With a 90-position autosampler, the rapid MAX N exceed can reliably be run unattended, even overnight, increasing laboratory throughput and productivity. The three-furnace combustion system ensures complete combustion, making measurements consistent across all sample types without the need for sample-specific calibrations.

The rapid MAX N exceed is a robust, precise, easy-to-use instrument for all of the N/Protein analysis applications of the brewery and beer industries.

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