chemical analysis 101

measurement of consituents in food products

Objective

While having conversations with some of our representatives around the world there seems to be, in some cases, a lack of knowledge as to what primary methods the food industry presently uses to determine certain constituents and the TEV (Total Existing Variance) or lab precision of those primary methods. I will also touch on the accreditation of the methods that are discussed which is important.

Food Types and Specific Primary Methods

FAT Analysis

Dairy foods are somewhat unique in the measurement of fat as the fat molecule or globule takes a special process to extract for the determination of the content of fat in a sample. Methods used are the Gerber method along with the more automated method using the basic premises, Mojonnier, are the primary methods approved by IDF (International Dairy Federation) NCIMS (National Conference on Interstate Milk Shipments, a US regulatory group associated with the FDA) and AOAC (Association of Official Analytical Chemists). The method uses ether extraction to separate the fat from the other constituents in the milk which takes several steps and from 15-20 minutes for a complete analysis. Most laboratories will have many tests run at once so the technician is processing new samples as the other samples are running through the hydrolysis. Accuracy or TEV for milk from skim or almost 0 fat to approximately 4% butterfat is 0.01%-0.02%. Cream at 40% fat the TEV would be around 0.2%-0.4%.

Babcock is a more rapid method than the Gerber but less accurate. Usually the TEV is in the high 100's or in the 10th's instead of the low 100's. The speed of the analysis of less than 10 minutes is the reason organizations will use this method to save time over better accuracy.

Fat Analysis for Other Products

Fat analysis is performed using the Soxhlet method which is again an ether extraction method for all products other than dairy. Products include meat, grains and grain products and prepared foods. This test is performed by first weighing each sample, approximately 6 grams, in a Soxhlet tube. The sample is then placed in a drying oven and the moisture is completely removed, 3+ hours. The sample is then taken to ether extraction which takes from 2 hours for automated systems to 12 hours using a traditional method. The sample is then weighed again and the difference is the fat content.

Babcock can also be used but I've found it is not often the chosen method.

Protein Analysis

The AOAC method for dairy is the Kjeldahl method which has three steps. Digestion of a specific weighed sample (usually 4-6 grams) in a high heat acid solution for several hours to an ammonium sulfate solution. Step two s a distillation of the sample to separate the ammonia from the sample. Step 3 is the titration of the sample using a colorimetric method and matched to a established color range specific to the sample type that is analyzed.

Another method that is AOAC approved is the combustion method; Leco is a common brand name that uses high pressure to determine the protein level. The combustion method usually obtains slightly higher results (0.2-0.4) than the Kjeldahl method.

Moisture Analysis

There are several approved methods for determining moisture in most products. Vacuum oven is usually the most accurate followed by convection oven and finally an oven. All include weighing the sample to a specific weight. In most cases you are looking at a minimum of 12 hours of drying and then weighing the sample at the end of the drying process to determine what is left after the moisture content is removed. The moisture content is the converse of the solids that are left in the vial. Usually moisture TEV by the vacuum oven are around 0.1%.

Sugars

HPLC chemical method is the normal primary instrumentation for determination of sugars in products and are usually very accurate with a TEV in the low 10th to low 100ths.

Use of Secondary Methods for Determination of Constituents

There are several technologies that are in use today for rapid analysis of one or all of the constituents that are necessary. Since the primary method had an inherit error of some degree and the secondary method will have an inherit error of some degree, it is NOT an acceptable practice to calibrate one secondary method to another secondary method as you are adding the instrument error to the primary method error, thus, making the new calibration less accurate than if calibrated to a primary method.

Examples of Secondary Methods

MidInfrared (IR) is used for fluid type products such as milk, whey, juices and wine. Most common instruments today use FTIR (Fourier Transform Infrared). They usually can be calibrated to two times the primary methods TEV on dairy fluid products like milk and whey. Top three competitors are Foss, Bentley and Delta Instruments. The instruments are used for fat, moisture (solids), lactose, sugars, protein, acids among other constituents. There are still a few filter base instruments in the marketplace but they tend to have calibration shifts and

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are not robust. The product to be analyzed is pumped into a small cuvette, usually with an aperture of less than 52 microns. This process makes the instrument very maintenance extensive because of the necessary cleaning out of caustic samples so as not to have carryover or build up with the cuvette.

NIR (Near Infrared) instruments are used for more viscous products that have from 45%+ solid content. Company's use Transmission and reflectance technologies for determining the constituent content. Transmission is used when it is possible to have a pathlenght between the probes of less than 10mm. Since most products at this viscosity will be hard to pump through this small pathlength the technology is very product dependent. When used, however, the analysis results can be within the 2X's the TEV. NIR reflectance technology is sending the energy into the product and collecting the reflected energy and then producing a predicted result. This can work well with an homogenous product but for meat, as an example, a surface measurement can be several % off of the true measurement. Top NIR manufactures in food are: Foss, Perten, Unity, Bruker, and ABB.

The benefit of both IR and NIR technologies is that both can measure all the constituents in less than a minute with relatively good accuracy if calibrated properly. Good defined as 2X's the primary TEV or lab precision.

Another secondary method widely used in the CEM Smart Trac system which measures moisture by placing a small amount of sample on specific consumable pad and putting the sample into a special microwave to dry. The pad is then placed into an NMR (Nuclear Magnetic Renounces Instrument). This system is highly susceptible to technician errors and takes 4-6 minutes for full sample preparation and analysis.

On-line or In-line Analysis Instruments

There are several in-line type instruments that are used. FTIR for fluid products are expensive ($150K+) and are high maintenance because of the small orifice the product must be pumped through to obtain an analysis.

NIR is difficult to calibrate needing several 100's of samples in most cases with chemical analysis. NIR-Transmission can be relatively accurate on homogenous product but NIR-Reflectance on products such as meat which is heterogeneous, has poor TEV and accuracy. Typical NIR in-line analysis instruments run from $80K-$150K.

Density Meters are use a lot in the dairy industry but are unreliable and much less accurate than the alternatives. They are, however, much less expensive with the price usually in the $30K-15K price range.

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