2009 TTB Expo Presentation

Laboratory Analysis for Calories, Carbohydrates, and Protein

Presented by
Norma R. Hill, Chief, Compliance Laboratory

Overview

- **Background:**
  - Scientific Services Division
  - Compliance Sampling and Routine Analysis
  - Terminology
  - Current Requirements
- **Laboratory Analysis for Compliance:**
  - Order of Importance
  - Methods of Analysis
  - Calculations
  - Shortcuts

Scientific Services Division

Scientific Services Division

National Laboratory Center:

- Beverage Alcohol Laboratory
- Nonbeverage Products Laboratory
- Tobacco Laboratory

6000 Ammendale Road
Beltsville, MD 20705
240-264-1580

Compliance Laboratory

490 North Wiget Lane
Walnut Creek, CA 94598
925-280-3625

Routine Compliance Analysis

Compliance Sampling

- TTB-initiated sampling:
  - Product integrity examinations
  - Taxpayer audits
  - Alcohol Beverage Sampling Program

Routine Compliance Analysis

General Types:

1. Alcohol Content and Fill — Protect the Revenue
2. Standard of Identity — Volatile Acidity (wine), Citric Acid (vodka)
3. Limited Ingredients — Sulfites, Methanol, Preservatives, Toxins
4. Label Issues — Government Warning, Serving Facts (nutrition)

Terminology

- Analysis for Compliance:
  - Compliance is assessed against —
    - Title 27 Code of Federal Regulations
    - TTB Rulings, Procedures, Industry Circulars
    - Applicable FDA regulations
      - 21 CFR and Guidance Documents

Terminology

- Analytical Methodology:
  - Official Methods of Analysis
  - AOAC International

    www.aoac.org

  - TTB Official Methods

    laboratory@ttb.gov

Word of Caution

- Be careful choosing testing laboratory to do serving facts label analyses
- Choose laboratories that are familiar with analysis of alcohol beverage products; make sure they are able to produce data equal to data generated by Official Methods of Analysis

Current Requirements

- TTB Ruling 2004-1
- TTB Procedure 2004-1
- NPRM:
  - Ingredient Labeling
  - Allergen Labeling

Serving Facts

Label Information – order of analytical importance

1. Alcohol Content
2. Calories
3. Carbohydrates
4. Protein

Alcohol Content

- Distilled Spirits: AOAC OMA 982.10
  - All glass/ground glass ball and socket type joints
  - 100 ml sample size
  - Chiller for condenser set at 5^0 C
  - Receiving flask set in ice/water
  - Constant temperature water bath set at 20^0 C
  - Density meter is calibrated/certified annually to NIST

Alcohol Content (Continued)

- Wines: AOAC OMA 983.13
  - Gas Chromatography with Flame Ionization Detector
  - Internal Standard – n-butanol
  - Diluter with precision equal to or better than Class A
  - When sample is deemed out of compliance, use AOAC OMA 982.10 to confirm

Alcohol Content (Continued)

- Beers: AOAC OMA 984.14
  - Gas Chromatography with Flame Ionization Detector
  - Internal Standard – n-butanol
  - Diluter with precision equal to or better than Class A
  - When sample is deemed out of compliance, use AOAC OMA 982.10 to confirm

Fat Content

- There are no Official Methods of Analysis
- This is not a problem for most wines, beers, and distilled spirits
- Major problem for cream liqueurs
- Fat interferes with analysis for carbohydrate in cream liqueurs

Protein

Protein Content: Kjeldahl Method

Beer: AOAC 920.53

$$\text{Protein g/serving} = \frac{[(Va \times Na - (Vb \times Nb)] \times 14.007 \times 6.25 \times Vc}{1000 \times Vs}$$

- Dumas method (combustion) is acceptable as long as it is equal to the Kjeldahl protein
- Negligible except for beer, egg and cream liqueurs and some few wines

Carbohydrates

Carbohydrate Content: By Calculation

- "Total carbohydrate content shall be calculated by subtraction of sum of crude protein, total fat, moisture, and ash from total weight of food; this calculation method is described in A. L. Merrill and B. K. Watt, 'Energy Value of Foods — Basis and Derivation,' USDA Handbook 74"

21 CFR 101.9

Calculating Carbohydrates All Foods

Carbohydrate = Total weight of food - Moisture - Protein - Fat - Ash

- Most important calculated value for nutrition is calories
- Most important number to know is calories from fat
- Moisture is primarily water, which has a Sp. Gr. of 1.000
- Protein, fat and carbohydrate are other proximates; for nutritional purposes, they are grouped by their contribution to total calorie count

  Protein: grams per serving x 4

  Carbohydrate: grams per serving x 4

  Fat: grams per serving x 9

Calculating Carbohydrates Alcohol Beverage Products

“Moisture” is not clearly defined:

- Is major component
- Small error in measurement leads to very large error in carbohydrate calculation
- *Alcohol contribution leads to significant errors in conversion from weight to volume at temperature*

Calculating Carbohydrates Alcohol Beverage Products

The Official Methods of Analysis for beer and wine:

- Remove “moisture” from equation entirely
- Start with “total solids”
- Carbohydrate = total solids – (fat + protein + ash)
- Account for alcohol contribution to caloric count separately

Calculating Carbohydrates Alcohol Beverage Products

Carbohydrate in wine: AOAC 985.10

Carbohydrates = Extract – ash

- There is no fat in standard wine
- For most wines, protein is negligible

Extract:

- Weigh sample, evaporate half, reconstitute and determine density or use the specific gravity of the reconstituted residue from distillation
- Use AOAC Table 970.90 to get extract
- Or (dry wines) evaporate a small quantity to dryness, weigh residue

Calculating Carbohydrates Alcohol Beverage Products

Wine Extract: AOAC 920.62

OMA 920.62(a) to determine wine extract is equivalent and preferred in wines where the extract >3 g/100 mL and required when extract >6g/100mL.

- From Alcohol % by volume, look up the specific gravity or use the Specific gravity of the distillate
- Determine the Specific Gravity of the wine (neat)
- $SG_{(DealcWine)} = SG_{(Wine)} + 1 - SG_{(Distillate)}$
- Use the calculated Dealcoholized specific gravity to read degrees Brix from the AOAC Table 942.33.
- Correct to g/100 mL by multiplying by the specific gravity of the wine ($SG_{(Wine)}$)

Calculating Carbohydrates

Alcohol Beverage Products

Carbohydrate in beer: AOAC 979.06

Carbohydrates = Extract – (ash + protein)

- There is no fat in standard beer

Extract:

- Weigh sample, evaporate half, reconstitute and determine density or use the specific gravity of the reconstituted residue from distillation
- Use AOAC Table 970.90 to get extract

Ash

Typical ash values:

- Beers: 0.1 grams/100 ml.
- Typical Wines:
  - For white wines, ash is 0.20 +/- 0.05 g/100 mL
  - For red wines, ash is 0.28 +/- 0.06 g/100 mL

Calculating Carbohydrates

Alcohol Beverage Products

Carbohydrate in spirits:

Carbohydrates = Total solids – (ash + protein + fat)

- There is no fat in standard spirits (except cream liqueurs)
- For most spirits, protein is negligible

For low solids:

- Tare, evaporate to dryness and weigh
- If less than 0.5 grams/serving it is negligible

Calculating Carbohydrates

Alcohol Beverage Products

Carbohydrate in spirits:

Cordials and Liqueurs: OMA 940.09

- Residue from distillation
- Reconstitute and measure specific gravity
- Read sugar level from Table 942.33
- Correct to g/volume using specific gravity of sample neat
- Carbohydrates assumed to be sugar

Calculating Calories

Alcohol Beverage Products

Calorie Content: By Calculation

- Beer: OMA 971.10
- Wine: OMA 979.07
- Using standard enthalpy calculations, alcohol yields 6.93 calories/gram, sugars yield 4 calories/gram; other carbohydrates average 2.4 calories/gram; also, protein at 4 calories/gram and fat at 9 calories/gram should be included where detected
- Sugars are defined in 21 CFR 101.9 as the sum of all free mono- and disaccharides

Calculating Calories Alcohol Beverage Products

For all products:

Calories = [(6.9 x A) + (4 x S) + (2.4 x (TC –S)) + (4 x P) + (9 x F)]

- A = alcohol content by weight
- S = sugar content by weight
- TC = total carbohydrate
- P = protein
- F = fat
- To get calories per serving use conversion factors:
  - Spirits: 1.5 ounces uses 0.44
  - Wines: 5 ounces : 1.48
  - Malt Beverages: 12 ounces : 3.55

Contact Information

Norma R. Hill

Phone: 925-280-3642
Fax: 202-435-7275
Compliance.Laboratory@ttb.gov