Fill/Headspace by Volume

Scope and Application

This method is used to assess whether alcoholic beverage containers are filled in accordance with 27 CFR. This method is applicable to all alcoholic beverages, but is preferred only for products that are not amenable to analysis by SSD:TM:517 Fill/Headspace by Weight, such as gelatins where specific gravity cannot be determined by densitometer, due to the higher associated measurement uncertainties when determined by volume as opposed to by weight. This method may be used for all types of container types (e.g., bottles, cans, ceramic containers).

Wine:

Standards of Fill 27 CFR 4.72(a) and (b)	Fill Tolerance 27 CFR 24.255(b)
≥ 15 L in 1 L increments	± 1.0%
4 L – 14 L in 1 L increments	± 1.5%
3 L, 1.5 L, 1 L	± 1.5%
750 mL	± 2.0%
500 mL	
375 mL	± 3.0%
355 mL, 250 mL, 200 mL	
187 mL, 100 mL	± 4.5%
50 mL	± 9.0%

A liter of wine is defined at 20°C (68°F) in 27 CFR 24.10.

Wine container sizes without a fixed tolerance are to be filled according to good commercial practice (27 CFR 4.37(d)(1)). TTB laboratories consider good commercial practice to be ±2.5% for 500 mL, ±3.0% for 355 mL, ±4.0% for 250 mL and 200 mL. Samples beyond these limits are flagged.

The fill tolerances and standards of fill in 27 CFR Part 4 apply to wine that contains not less than 7 percent alcohol by volume. They do not apply to cider, perry, or mead that contain less than 7 percent alcohol by volume nor sake. Where there is no TTB regulation, containers beyond the limits listed above are flagged.

Headspace is defined as the volume of the container occupied by air after closure.
27 CFR 4.71(a)(3):
(i) *187 mL or more.* If the net contents stated on the label are 187 milliliters or more, the headspace must not exceed 6 percent of the container's total capacity after closure.
(ii) *Less than 187 mL.* If the net contents stated on the label are less than 187 milliliters, except as described in (a)(3)(iii) of this section, the headspace must not exceed 10 percent of the container's total capacity after closure.
(iii) *Exception.* Wine bottled in clear containers with the contents clearly visible, with a net content stated on the label of 100 milliliters or less, may have a headspace that does not exceed 30 percent of the container's total capacity after closure.

Distilled Spirits:

Standards of Fill For Containers Other Than Cans 27 CFR 5.203(a)(1)	Standards of Fill For Cans 27 CFR 5.203(a)(2)	Fill Tolerance 27 CFR 19.356(b)
1.8 L, 1.75 L, 1.0 L		± 1.5%
900 mL, 750 mL, 720 mL, 700 mL		± 2.0%
375 mL, 200 mL	355 mL, 200 mL	± 3.0%
100 mL, 50 mL	100 mL, 50 mL	± 4.5%

Proof spirits is defined at 60°F in 26 U.S.C. 5002(a)(10), so fill is accordingly determined at 60°F.

Headspace is defined as the volume of the container occupied by air after closure. Headspace for containers 200 mL or greater must be no more than 8% of the total bottle capacity. (27 CFR 5.202(b))

Malt Beverages:
Filling shall be conducted in accordance with “good commercial practice” (27 CFR 25.142(d)). Historically, the TTB laboratories have considered good commercial practice as ± 2% of the labelled fill for malt beverages, beyond which samples are flagged.

A gallon is defined for malt beverages at 4°C (39.1°F) in 27 CFR 7.1.

Mandatory label information requires the net contents (except when blown, branded, or burned, in the container) for malt beverages must be displayed on the brand label (27 CFR 7.63(a)(5)).

Levels and Limitations

Not applicable

Supplemental Documents

BAL:Form:516 Fill-Headspace by Volume

Equipment

Class A calibrated fill flasks of various sizes

Traceable Thermometer

Gauging Manual

Class A Graduated Cylinder

Reagent and Sample Preparation and Handling

Not applicable

Procedures

Note: Begin with an unopened beverage container which has been equilibrated to room temperature for wine and distilled spirits and equilibrated to refrigeration temperature for malt beverages.

Fill for opaque or ceramic containers*:*

Note: most tests on the sample cannot be performed after this procedure as the results may be affected. See **SSD:TM:517** Fill/Headspace by Weight for alternative method.

1. Drain the contents of a newly opened bottle into a clean, dry calibrated fill flask by inverting for approximately 1 minute after the stream of liquid breaks and drops form.
2. Read the fill flask using the divisions above or below the indicated volume mark. This is the “Actual Volume”.
3. Determine the temperature of the sample by measuring the room temperature to the nearest tenth of a degree, using a traceable thermometer.

Fill for glass containers:

1. Etch/mark the bottle at the liquid level and the bottom edge of the closure (if applicable) making note of the sample temperature by measuring the room temperature to the nearest tenth of degree, using a traceable thermometer.
2. After analyses are complete, empty the remaining sample, rinse the bottle with water, and fill bottle with water to the mark made at the liquid level.
3. Transfer the water into a calibrated fill flask by inverting for approximately 1 minute after the stream of liquid breaks and drops form.
4. Read the fill flask at the bottom of the meniscus using the divisions above or below the indicated volume mark. This is the “Actual Volume”.
5. For **headspace** measurements drain the sample bottle and fill to brim with water. For closures that are inserted into the bottle (e.g., corks), mark for the bottom edge of the closure to aid in repositioning. The bottle closure is replaced so that any liquid that would normally be lost by the capping process is displaced.
6. Using the graduate cylinder, determine the volume of the water by inverting for approximately 1 minute after the stream of water breaks and drops form. This volume is the “Bottle Capacity”.
7. Return the remaining sample to the bottle.

Quality Control

Only Class A calibrated fill flasks and a traceable thermometer are used for determination of fill.

Sources of Uncertainty

1. The main source of uncertainty in this method comes from using graduated cylinders, which have a higher level of associated uncertainty than fill flasks. Both graduated cylinders and fill flasks have a higher level of associated uncertainty than the analytical balance used in SSD:TM:517 Fill/Headspace by Weight. SSD:TM:517 Fill/Headspace by Weight is preferred over SSD:TM:516 Fill/Headspace where applicable for the product type.
2. Uncertainty may originate from using uncalibrated fill flasks, graduated cylinders that are not class A, and/or a thermometer that is not traceable. Therefore particular attention should be paid while using this equipment.
3. Uncertainty may arise due the incorrect volume readings from the bottom of the meniscus. Demonstration of correct volume reading skills is a part of the chemists' competency certification for this method.
4. Wines and malt beverages analyzed by this method will have a higher level of uncertainty than distilled spirits due temperature variability and the lack of temperature correction factors.

Calculations

Fill

% Fill = (Adjusted Actual Volume / Label volume) X 100

For distilled spirits, the Adjusted Actual Volume is defined as the Actual Volume multiplied by a correction factor to adjust for temperature, obtained from the Gauging Manual 27 CFR 30.67 Table 7. This table converts volumes of distilled spirits at various room temperatures to that at 60°F. A liter of wine is defined at 20°C (68°F) in 27 CFR 24.10, and a gallon of malt beverage is defined at 4°C (39.1°F) in 27 CFR 7.1. Fill for wine is determined at room temperature, and fill for beer is determined at refrigerated temperature, which are typically close to the defined temperatures, so no temperature correction factors are used for these commodities.

Example: Sample is a 750 mL wine bottle at 20% alcohol.
Temperature is 76°F.
Number of divisions above mark is 2.
Fill flask: 750 mL with each division mark equal to 1.6 mL.

1. Determine the Actual Volume:
   a. 2 X 1.6 mL = 3.2 mL
   b. 750 mL + 3.2 mL = 753.2 mL
2. Convert alcohol % by volume to proof (2 X ABV)
   a. 20% ABV x 2 = 40 proof
3. Using the temperature of the room and the proof of the sample, determine the correction factor using proof in the Gauging Manual, Table 7
   a. Correction for 76°F, at 40 proof is 0.997
   b. If the temperature of the room falls between two temperatures listed in Table 7, take the average of the correction factors

4. Apply the correction factor to the actual volume:
    a. 753.2 mL X 0.997 = 751 mL = Adjusted Actual Volume
5. Calculate the % Fill as defined above:
    a. 751 mL / 750 mL X 100 = 100.1%

Headspace

% Headspace = ((Bottle Capacity – Actual Volume) / Bottle Capacity) X 100

Example: Bottle Capacity is 790 mL and the Actual Volume is 753.2 mL.

Calculation: ((790 mL – 753.2 mL) / 790 mL) X 100 = 4.7%

Reporting Results

Fill is reported as % of label contents with one decimal, i.e. xxx.X% and as mL with no decimal place, i.e. XXX mL. Headspace is reported as % with one decimal place, i.e. xxx.X%.

Safety Notes

Normal laboratory safety protocol should be followed.

References

	Standards of Fill	Fill Tolerances	Fill Temperature	Headspace
Wine	27 CFR 4.72 (a) & (b)	27 CFR 24.255(b)	27 CFR 24.10	27 CFR 4.71(a)(3) (i), (ii) & (iii)
Distilled Spirits	27 CFR 5.203(a) (1) & (2)	27 CFR 19.356(b)	26 U.S.C. 5002 (a)(10)	27 CFR 5.202(b)
Malt Beverages	none	27 CFR 25.142(d)	27 CFR 7.1	none

27 CFR 30.67 for correction of volume of spirituous liquors to 60°F.

Location of Validation Package

Quality System Files

Required Training, Certification and Re-certification

1. In-house training by a certified chemist.
2. Demonstrate competency by taking written test and by performing the technique.
3. Recertification every 5 years.

Revision History

Revision 4 – Minor edits to Scope and Application section; Added requirement to equilibrate samples to room temperature prior to analysis; added use of traceable thermometer; added statement to read fill flask at the bottom of the meniscus; updated example in calculations section; and updated reference in References section.

Revision 5 – Limited Scope and Application due to measurement uncertainty; Updated CFR references; Addressed temperatures for wine and beer to make this method applicable to these products in line with the fill temperatures as defined in the CFR.