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Fill/Headspace by Weight

Scope and Application

Wine:

Standards of Fill	Fill Tolerance
27 CFR 4.72(a) and (b)	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:

Distilled Opinits.		
Standards of Fill	Standards of Fill	Fill Tolerance
For Containers Other Than Cans	For Cans	
27 CFR 5.203(a)(1)	27 CFR 5.203(a)(2)	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%

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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

This method is preferable for opaque containers and cans. It is also suitable for translucent containers.

Supplemental Documents

Form:SSD:516:001 Calculation for Fill/Headspace by Volume (Formerly BAL:Form:516)

Form:SSD:517:001 Calculation for Fill/Headspace by Weight (Formerly BAL:Form:517)

WG:SSD:1040:003 Sub-sampling (Formerly SSD:WG:112)

WG:Balance:001 Balance and Weights Verification Procedure (Formerly SSD:WG:304a)

SSD:TM:102 Ethanol Determination by Specific Gravity

SSD:TM:516 Fill / Headspace by Volume

Equipment

Top loading balance with 0.01 g accuracy (or better)

Density Meter with 0.0001 g/cm³ accuracy (or better), set at 20 °C

Waterproof Marker

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Reagent and Sample Preparation and Handling

No sample prep is necessary. Homogenization is accomplished using **WG:SSD:1040:003**.

Procedures

Note: All procedures for fill in carbonated samples begin with an unopened bottle or can, and in most cases for non-carbonated samples. Sometimes the sample closure is subject to damage by the process of opening the container. This is particularly evident with wine corks where the closure may break apart. In those instances, the analyst may determine that it is better to open the non-carbonated sample prior to weighing to avoid errors due to closure removal or destruction. The sample is immediately stoppered to prevent loss or damage to the contents.

- 1. For samples in a translucent container, mark the container with a waterproof marker at the Fill Level, and the bottom edge of the closure.
- 2. Weigh full bottle, with closure, (W_f) after stripping all extraneous attachments from the bottle on the top loading balance.
- 3. Distilled Spirits and Wine:

Determine the true specific gravity ($S_{20^{\circ}C}$) of the liquid contents at 20 °C using the densitometer.

For Malt Beverages:

Determine the true specific gravity (S_{20°C}) of the decarbonated beverage at 20°C.

- 4. After analysis of sample is completed, drain the bottle or can by inverting for at least 1 minute after the stream of liquid breaks and drops form.
- Rinse empty bottle with water and dry, inverted at room temperature, for at least 1 hour.
- 6. Weigh the empty can or bottle with closure (W_e).
- 7. For headspace, fill bottle to the brim with water (or to the mark made at the bottom edge of the closure), replace closure and re-weigh (W_{bc}).
- 8. Note the label volume (V_o).

Note: For translucent containers, violative results are confirmed using fill by volume test method (**SSD:TM:516**) or by filling the container with room temperature water to the mark made in Step 1, reweighing, and using 1.0000 for the sample density. This value is then used to confirm the one determined.

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- 1. Prior to weighing: Confirm top loader operating condition by recording two standard weights, for example a 100 g weight and a 1000 g weight. Weight tolerances should be as defined by laboratory protocol, WG:Balance:001.
- Top loading balances should be recalibrated annually and the calibration results should be filed with the balance log book. All weight measurements should be recorded in the log book.
- 3. Quality control of density meter should be performed according to SSD:TM:102

Sources of Uncertainty

- 1. The main source of uncertainty may originate from improper use of a top loading balance. Therefore, particular care should be taken in assuring the proper and timely maintenance and calibration of the balances.
- 2. Accuracy and precision of the density meter may also be a factor of uncertainty. The density meter should be calibrated and results of laboratory control sample run in duplicate should be within established tolerance range.

Calculations

Fill

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Absolute fill at temperature T, V_T = (W_f - W_e) / D_T
                                                                                                      equation 1
          where D_T = density of beverage at temperature T
D_T / D_{20^{\circ}C} = D_{H2O T} / D_{H2O 20^{\circ}C}
          where D_{H2OT} = density of water at temperature T
          solving for D_T = D_{20^{\circ}C} * D_{H20 T} / D_{H20 20^{\circ}C}
                                                                                                      equation 2
by definition S_{20^{\circ}C} = D_{20^{\circ}C} / D_{H2O 20^{\circ}C}
          solving for D_{20^{\circ}C} = S_{20^{\circ}C} * D_{H2O 20^{\circ}C}
                                                                                                      equation 3
substituting the expression for D_{20^{\circ}C} from equation 3 into equation 2:
          D_T = S_{20^{\circ}C} * D_{H2O \ 20^{\circ}C} * D_{H2O \ T} / D_{H2O \ 20^{\circ}C}
          D_{H2O\ 20^{\circ}C} cancels out to give: D_T = S_{20^{\circ}C}^* D_{H2O\ T}
                                                                                                      equation 4
substituting the expression for D<sub>T</sub> from equation 4 into equation 1:
          V_T = (W_f - W_e) / (S_{20^{\circ}C}^* D_{H2O T})
                                                                                                      equation 5
Wines are reported at 20°C, so use D_{H2O,20^{\circ}C} = 0.99823 in equation 5 to get:
          V_{20^{\circ}C} = (W_f - W_e) / (S_{20^{\circ}C} * 0.99823)
                                                                                                      equation 6
Distilled spirits are reported at 60^{\circ}F, so use D_{H2O 60^{\circ}F} = 0.99902 in equation 5 to get:
          V_{60^{\circ}F} = (W_f - W_e) / (S_{20^{\circ}C} * 0.99902)
                                                                                                      equation 7
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Malt beverages are reported at 4° C, so use $D_{H2O \ 4^{\circ}C} = 1$ in equation 5 to get: $V_{4^{\circ}C} = (W_f - W_e) / S_{20^{\circ}C}$ equation 8

Fill as % of label contents = $V_T \times 100 / V_0$

equation 9

Headspace

Volume of the bottle, $V_b = (W_{bc} - W_e) / D_{H2O T}$

equation 10

Wines are reported at 20°C, so use $D_{H2O\ 20^{\circ}C}$ = 0.99823 in equation 10 to get: $V_b = (W_{bc} - W_e) / 0.99823$ equation 11

Distilled spirits are reported at 60° F, so use $D_{H2O 60^{\circ}}$ F = 0.99902 in equation 10 to get: $V_b = (W_{bc} - W_e) / 0.99902$ equation 12

Headspace % = $[(V_b - V_T) / V_b] \times 100$

equation 13

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%.

Compliance is assessed on wine and distilled spirits based on the fill tolerances given in the 27 CFR. Compliance is not assessed on malt beverages, rather any container filled beyond $\pm 2\%$ of the labelled volume is noted in the "Summary of Findings" narrative section of the results report and called out in the LIMS Findings test by selecting "Warning" under Fill Compliance along with "Flagged" under Summary Determination (when there are no other compliance issues being reported).

Safety Notes

Normal laboratory safety protocol should be followed.

References

Standards of Fill	Fill Tolerances	Fill Temperature	Headspace
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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.

Official Methods of Analysis (2019) 21st Ed., AOAC INTERNATIONAL, Rockville, MD, **Method 990.17** (*Calculation from Measured Net Weight*). <u>www.eoma.aoac.org</u> [accessed on March 25, 2021]

(<u>Note</u>: Similarities – calculation and weighing/measuring steps; Differences – TM-517 does not include the CO_2 density correction referenced in OMA 940.17, does not follow same method of degassing, and does not dry using an oven).

ASBC Methods of Analysis. 8th Revised Ed., 1992. **Method Fills-1**. *Total Contents of Bottles and Cans by Calculation from Measured Net Weight*. Issued 1992. American Society of Brewing Chemists, St. Paul, MN, USA.

(<u>Note</u>: Similarities – calculation and weighing/measuring steps; Differences – TM-517 is different from Fills-1 in terms of the drying method and correction for residual CO₂ after degassing).

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

Rev. 3 -- Allows for verifying noncompliant results using water; Changes calculation for malt beverages to use SG rather than density (8/21/2009)

Rev. 4 – Edits for consistency and clarity. Addition of compliance statement.

Rev. 5 – Changed section 3 under Procedures from Apparent Specific Gravity to True Specific Gravity. (08/30/2017)

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- Rev. 6 Added regulatory references, changed wine reporting to 20°C, corrected and explained calculations, and clarified compliance reporting.
- Rev. 7 Changes to regulations and references. Change to balance readability. No changes were made to the method procedure.
- Rev. 8 Updated CFR references for 27CFR parts 5 and 7.
- Rev. 9 Changed document IDs to the new ID structure.