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Fill/Headspace by Volume

Scope and Application

This method is used to assess most types of alcohol beverages to ensure that they are filled in accordance with 27 CFR. This method may be used for all types of container types (e.g., bottles, cans, ceramic containers).

27 CFR 5.47 (b)(1) states "The following tolerances shall be allowed: (1) Discrepancies due to errors in measuring which occur in filling conducted in compliance with good commercial practice." Historically, TTB laboratories have considered good commercial practice for 750 mL and 1.75 L containers to be +/- 2% of the label fill for distilled spirits.

According to 27 CFR §24.255, the fill tolerance for wines should not exceed 1.0 % for containers 15.0 liters and above, 1.5 % for 1.0 liter to 14.9 liters, 2.0 % for 750 mL, 3.0 % for 375 mL, 4.5 % for 187 mL and 100 mL, and 9.0 % for 50 mL.

Current standards of fill for wines and distilled spirits are as follows:
Wine—3L, 1.5L, 1L, 750 mL, 500 mL, 375 mL, 187 mL, 100 mL, 50 mL (27CFR 4.72)
Distilled Spirits—1.75L, 1.00L, 750 mL, 375 mL, 200 mL, 100 mL, 50 mL (27CFR 5.47a)

The wine standards of fill do not apply to sake. There are no standards of fill for malt beverages or sake.

Headspace is defined as the volume of the container occupied by air after closure. For wine samples, the headspace may be 6% of the total capacity if the net contents are 187 mL or more. For all other wine containers, the headspace must be less than 10% (27CFR 4.71(a)(3)). For distilled spirits the headspace for containers 200 mL or greater must be no more than 8% of the total bottle capacity.

Levels and Limitations

Not applicable

Equipment

Type A calibrated fill flasks of various sizes

Traceable Thermometer

Gauging Manual

Graduated Cylinder

Reagent and Sample Preparation and Handling

Not applicable

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Procedures

Note: All procedures for fill begin with an unopened bottle.

Fill for opaque or ceramic containers*:*

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. (Note: most of analyses on the sample can not be performed after this procedure. See **SSD:TM:517** Fill/Headspace by Weight for alternative method).

2. Read the fill flask using the divisions above or below the indicated volume mark

3. Determine the temperature of the sample by measuring the room temperature to the nearest tenth of a degree.

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.
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 using the divisions above or below the indicated volume mark.
5. Return the remaining sample to the bottle.
6. For **headspace** measurements drain the sample bottle and fill to brim with water. For closures that are inserted <u>into</u> 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.
7. 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”.

Quality Control

Only Type A or calibrated fill flasks and calibrated thermometers are used for determination of fill.

Sources of Uncertainty

1. The main source of uncertainty may originate from using uncalibrated fill flasks, graduated cylinders, and/or thermometer. Therefore particular attention should be paid while using this equipment. Class A glassware is preferred.

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2. 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.

Calculations

Fill

% Fill = (Contents Volume**/Label volume)X100

**Contents after adjusting for temperature using ATF Gauging Manual Table 7

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. ***Convert alcohol % by volume to proof (2 X ABV)***
2. ***Look up the correction factor using proof in the ATF Gauging Manual, Table 7***
3. Correction for 76°F, at 40 proof is 0.997, so . . .

Calculation: 2 X 1.6 = 3.2 mL
750 + 3.2 = 753.2 mL
753.2mL X 0.997 = 751 mL volume of wine
(751/750) X 100 = 100.1%

Headspace

% Headspace = ((Bottle capacity – Bottle contents)/Bottle capacity) X 100

Example: Bottle capacity is 790 mL.
Volume of contents is 753.2 mL.

Calculation: ((790 – 753.2)/790) 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

27 CFR §24.255; §5.46 and §5.47 (1)

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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.

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