

2009 TTB Expo Presentation

Laboratory Techniques for Small Distilleries

Presented by

Norma R. Hill

Scientific Services Division



Overview

- Basic Assumptions:
 - Target audience
 - Basic terminology
- Analytical Tests:
 - Proofing Rules
 - Distilled spirits alcohol determination
 - Solids determination and obscuration
 - Fill and Headspace of containers

Basic Assumptions

- Target Audience:
 - Small distillery
 - Limited laboratory experience
 - Limited laboratory equipment
- Overview of Basic Techniques:
 - Basic terminology
 - Basic equipment

Basic Terminology

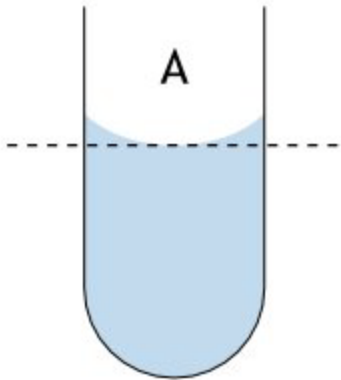
- **Proof Gallon:** A gallon of liquid at 60⁰F. which contains 50 percent by volume of ethyl alcohol having a specific gravity of 0.7939 at 60⁰F. referred to water at 60⁰F. as unity, or the alcoholic equivalent thereof.
- **Apparent Proof:** Proof measured on the sample neat. It is not the same as true proof. It is influenced by dissolved solids and other solvents.
- **True Proof:** A defined measure of the alcohol content of the sample.
- **Obscuration:** A convention used to correct for dissolved solids.
- **Fill:** The net contents of the container.
- **Headspace:** The volume left at the top of an almost filled container before sealing.

Basic Terminology

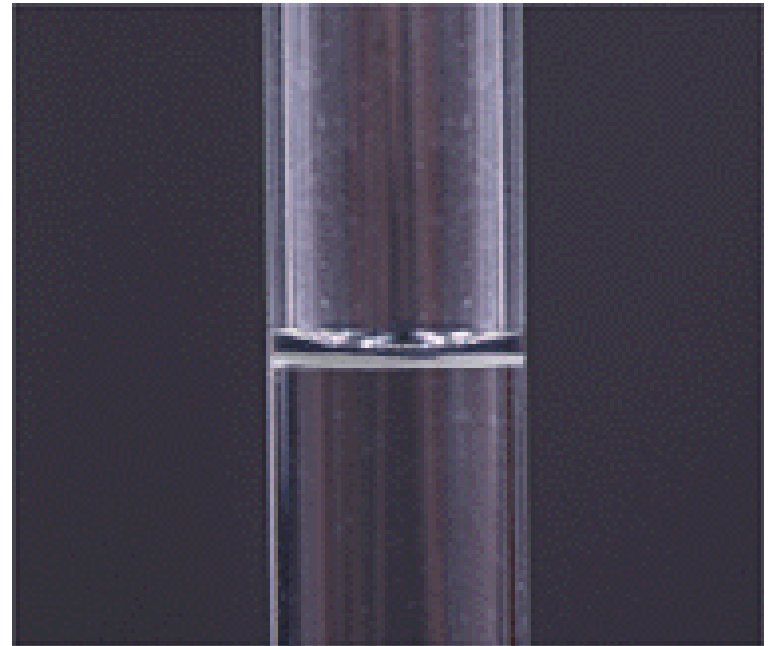
**Volumetric glassware:
Calibrated “To Deliver”**

**Pipettes
Volumetric flasks
Graduated Cylinders
Burets**

**Reagents: Substances consumed during
a chemical reaction; Chemicals.**



How to read a water meniscus

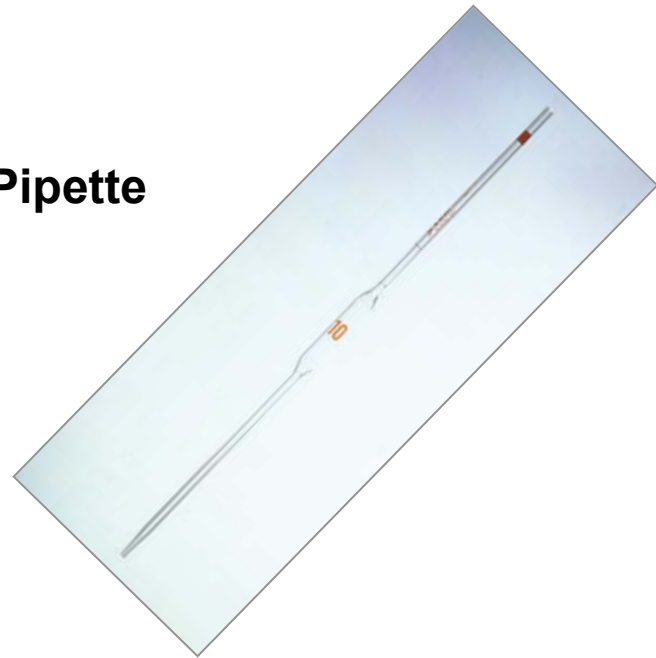


Volumetric Glass



Volumetric flask

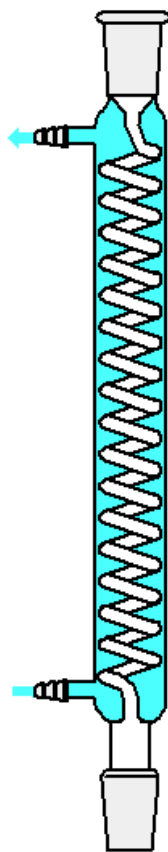
Pipette



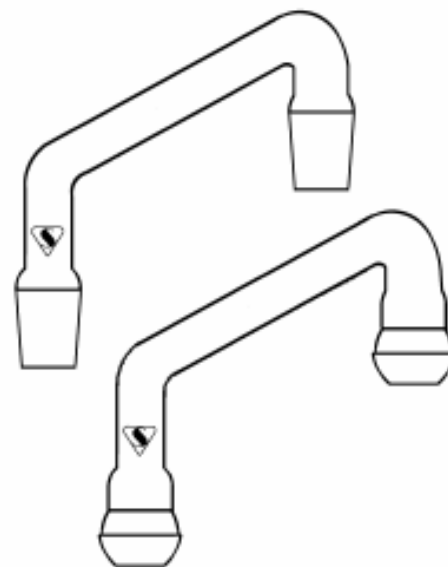
Common Glassware



Flask



Condenser



Connecting adapter

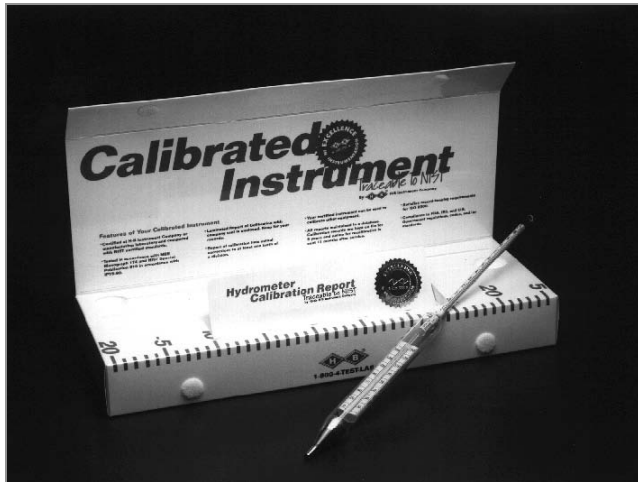
Analytical Tests

Determination of Alcohol in Spirits

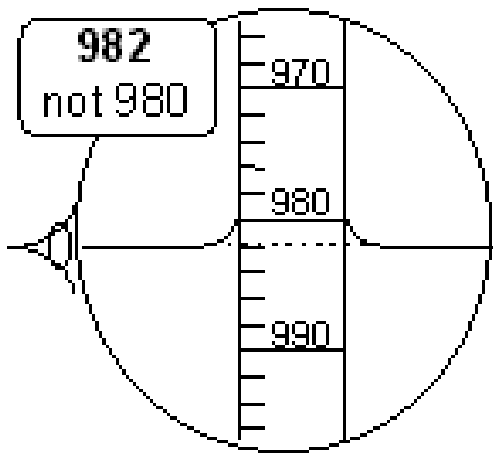
Distillation with Hydrometry or Densitometry

Basic Equipment

Distillation – Hydrometry



Hydrometers:
Specific Gravity divisions 0.0005
Proof divisions 0.2° proof



How to read the hydrometer



Hydrometers 27 CFR 30.22

Precision	Range	Subdivision
F	0 to 20	0.2°
G	20 to 40	0.2°
H	40 to 60	0.2°
I	60 to 80	0.2°
K	75 to 95	0.2°
L	90 to 110	0.2°
M	105 to 125	0.2°
N	125 to 145	0.2°
P	145 to 165	0.2°
Q	165 to 185	0.2°
R	185 to 206	0.2°

Thermometers 27 CFR 30.22

Type	Range	Subdivision
Pencil type	10° to 100°	1°
V-back	10° to 100°	1°
Glass shell (earlier model)	40° to 100°	1/2°
Glass shell (later model)	40° to 100°	1/4°

Basic Equipment

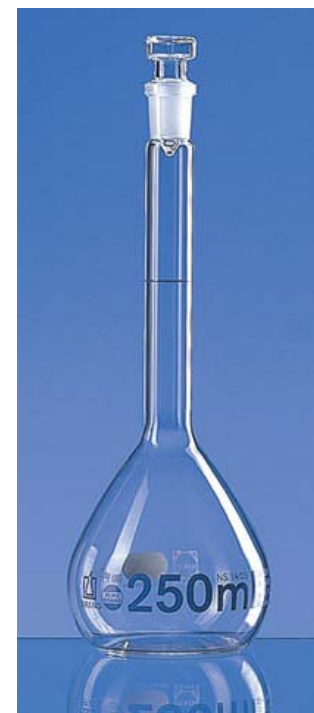
Distillation – Hydrometry



Thermometer
Calibrated divisions 0.2°F



Hydrometer & cylinder
Clear glass 2.5" diameter



250 mL volumetric flask

Basic Equipment

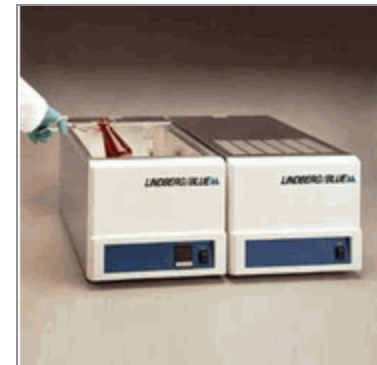
Distillation – Hydrometry



Distillation Apparatus
Electric mantle heater
Condenser



Constant Temperature Waterbath



Conversion table as appropriate:

The Gauging Manual and Tables 27 CFR 30 (www.ttb.gov)

Table 913.02 of the Official Methods of Analysis (18th Ed.)

Reagents

- Reagents:
 - Antifoam
 - Distilled water
- Also:
 - Ice
 - Boiling Beads/Chips

Basic Equipment

Distillation – Densitometry



Densitometer



100 ml volumetric flask

Analytical Tests

Determination of Solids in Spirits

Solids up to 600 mg/L
Obscuration

Determination of Solids Basic Equipment



25 mL pipette

**Analytical
Balance**



Desiccator



Aluminum weighing dish



Drying oven

Calculations

- Obscuration is an empirical factor. It has been determined experimentally that 100 mg of solids in 100 mL of sample will obscure (lower) the apparent proof by 0.4° proof.
- Obscuration =
$$\frac{[(\text{Wt. dish} + \text{residue}) - \text{tare}] \times 400}{\text{Sample size}}$$
- True Proof = Apparent Proof + Obscuration

Proofing Rules

Proofing Rules

80 – 100° Proof

- Solids Range 0 – 400 g/100L:
 - Sample may be compliant on either true proof or apparent proof.
 - Obscuration may be determined using the evaporation or distillation method.

Proofing Rules

80 – 100⁰ Proof (Continued)

- Solids Range 400 – 600 g/100L:
 - Sample must be compliant on True Proof.
 - True proof is determined from the Apparent Proof plus the Obscuration.
 - Obscuration may be determined using the evaporation or distillation method.

Proofing Rules

80 – 100⁰ Proof (Continued)

- Solids range over 600 g/100L:
 - Sample must be compliant on True Proof.
 - True Proof is determined by distillation.

Proofing Rules

Proof < 80 or >100⁰ Proof

- Solids range 0 – 400 g/100L:
 - Sample may be compliant on either true proof or apparent proof.
- Solids Range 400 – 600 g/100L:
 - Sample must be compliant on True Proof.
 - True proof is determined from the Apparent Proof + the Obscuration.
- Solids range over 600 g/100L:
 - Sample must be compliant on True Proof.
 - True Proof is determined by distillation.
- Obscuration must be determined by the distillation method.

Analytical Tests

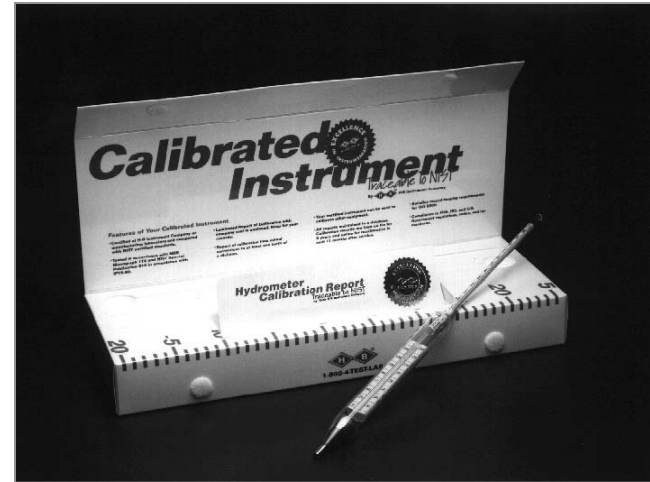
Fill/Headspace

Basic Equipment

Fill/Headspace



Top loader balance



Calculations

Absolute Fill at 60⁰F =

$$(\text{Weight}_{\text{full}} - \text{Weight}_{\text{empty}}) / (\text{Density @ } 20^{\circ}\text{C} \times 1.0008)$$

Headspace as % of Volume =

$$\frac{[(\text{Weight}_{\text{filled to brim}} - \text{Weight}_{\text{empty}}) - \text{Label Volume}] \times 100}{(\text{Label Volume})}$$

Contact Information

Norma R. Hill

Phone: 925-280-3642

Fax: 202-435-7275

Compliance.Laboratory@ttb.gov