2009 TTB Expo Presentation

Laboratory Techniques for Small Wineries

Presented by

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Overview

- Basic Assumptions
  - Target audience
  - Basic terminology
- Analytical Tests
  - Wine alcohol determination
  - Sulfur dioxide determination
  - Acidities
  - Fill of wine containers
- Other Practical Information

Basic Assumptions

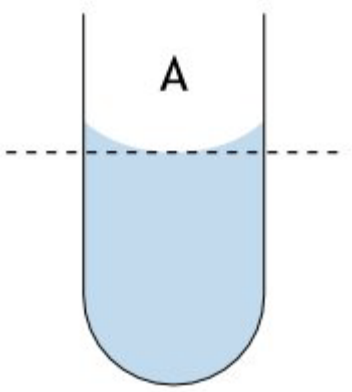
- Audience
  - Small winery
  - Limited laboratory experience
  - Limited laboratory equipment
- Overview of Basic Techniques
  - Basic terminology
  - Basic equipment

Basic Terminology

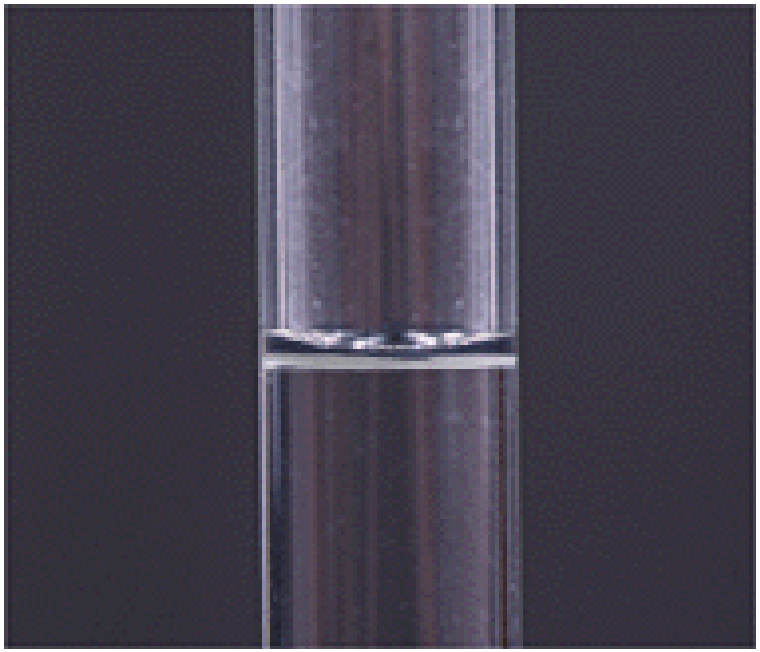
Volumetric glassware:
Calibrated "To Deliver"

Pipettes
Volumetric flasks
Graduated Cylinders
Burettes

Reagents: Substances consumed during a chemical reaction; Chemicals.



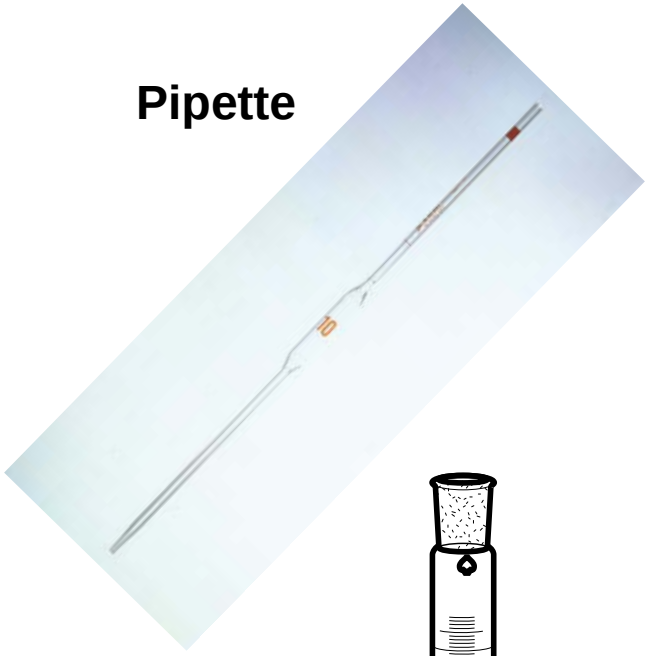
How to read a water meniscus

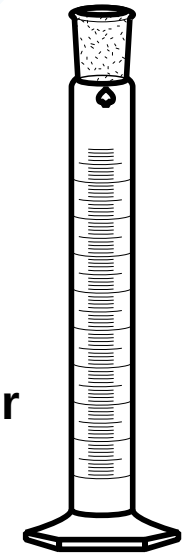


Volumetric Glass

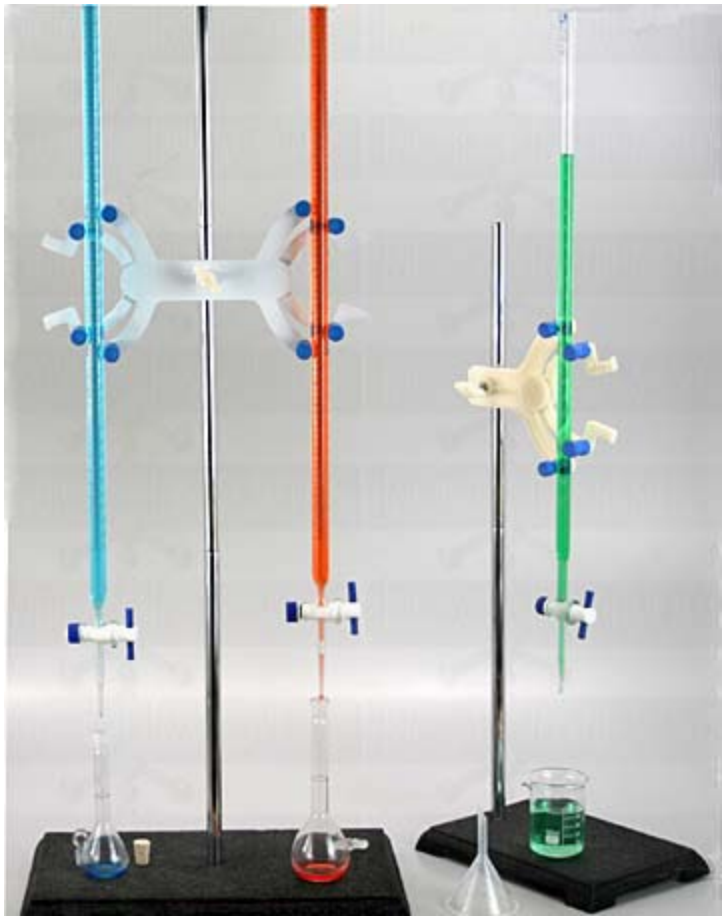
Volumetric flask

Pipette



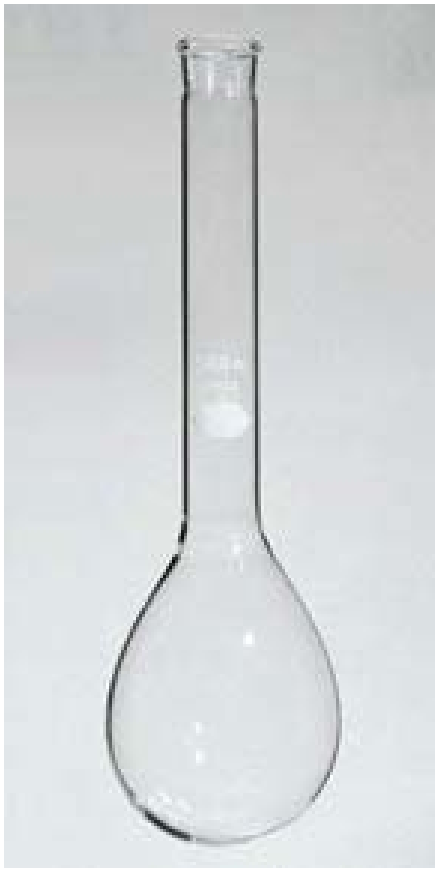


Graduated cylinder

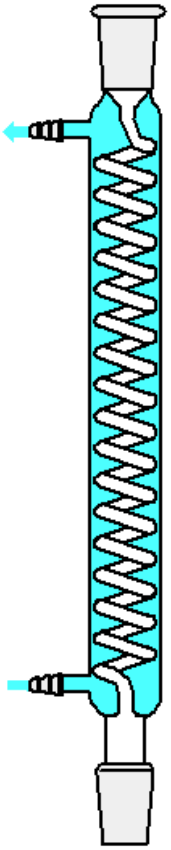


Burettes

Common glassware



Flasks



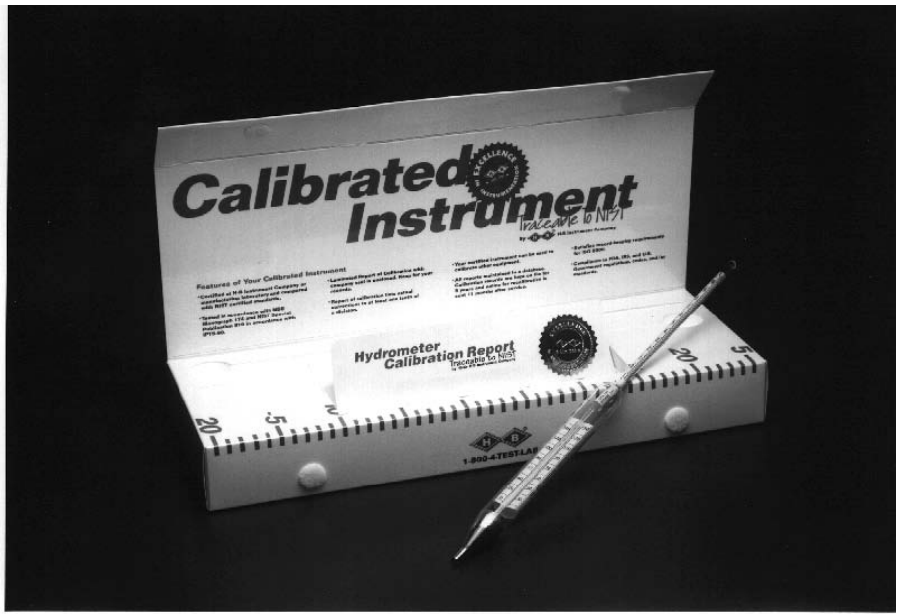
Condenser

Adapters

Analytical Tests

Determination of Alcohol in Wine

Basic Equipment
Distillation – Hydrometry

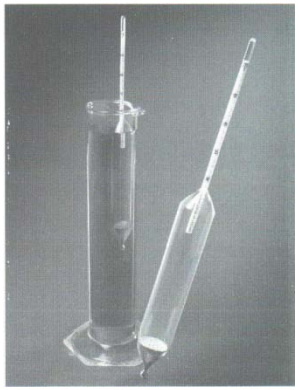


Hydrometer
Either
Specific Gravity divisions 0.001
Proof divisions 0.2° proof
Percent Alcohol divisions 0.1%

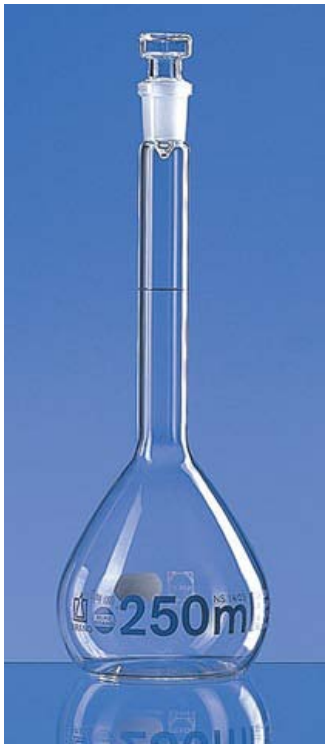
Basic Equipment
Distillation – Hydrometry

Thermometer
Calibrated divisions 0.2°F

Hydrometer cylinder
Clear glass 2.5” diameter



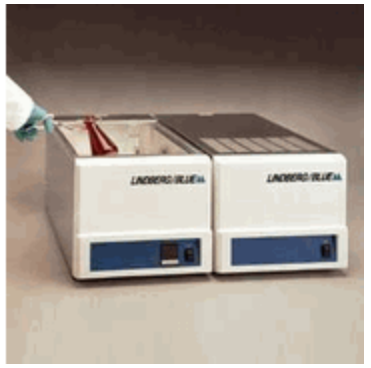
250 mL volumetric flask



Basic Equipment
Distillation – Hydrometry

Distillation Apparatus
Electric or gas heater
Condenser

Constant Temperature Waterbath



Conversion table as appropriate:
Table 1 of the Gauging Manual (www.ttb.gov)
Table 913.02 of the Official Methods of Analysis (18th Ed.)
Alcohol Correction Table Bureau of Internal revenue Bulletin 7 (1937)

Alcohol Correction Table

% Read	At 57° F	At 58° F	At 59° F	At 61° F	At 62° F	At 63° F	At 64° F	At 65° F	At 66° F	At 67° F	At 68° F	At 69° F	At 70° F	At 72° F	At 74° F	At 76° F	At 78° F	At 80° F
1	0.14	0.10	0.05	-0.05	-0.10	-0.16	-0.22	-0.28	-0.34	-0.41	-0.48	-0.55	-0.62	-0.77	-0.93	-----	-----	-----
2	0.14	0.10	0.05	-0.05	-0.11	-0.17	-0.23	-0.29	-0.35	-0.42	-0.48	-0.56	-0.63	-0.78	-0.94	-1.10	-1.28	-1.46
3	0.14	0.10	0.05	-0.06	-0.12	-0.18	-0.24	-0.30	-0.36	-0.43	-0.50	-0.57	-0.64	-0.80	-0.96	-1.13	-1.31	-1.50
4	0.14	0.10	0.05	-0.06	-0.12	-0.19	-0.25	-0.32	-0.38	-0.45	-0.52	-0.59	-0.67	-0.83	-1.00	-1.17	-1.35	-1.54
5	0.15	0.10	0.05	-0.07	-0.13	-0.20	-0.26	-0.33	-0.40	-0.47	-0.54	-0.62	-0.70	-0.86	-1.03	-1.21	-1.40	-1.60
6	0.17	0.11	0.06	-0.07	-0.14	-0.20	-0.27	-0.34	-0.42	-0.50	-0.57	-0.66	-0.74	-0.90	-1.09	-1.27	-1.46	-1.66
7	0.18	0.12	0.06	-0.07	-0.14	-0.21	-0.29	-0.36	-0.44	-0.52	-0.60	-0.68	-0.77	-0.94	-1.13	-1.32	-1.52	-1.73
8	0.19	0.13	0.06	-0.08	-0.16	-0.23	-0.31	-0.39	-0.47	-0.55	-0.64	-0.73	-0.81	-0.99	-1.18	-1.38	-1.59	-1.80
9	0.21	0.14	0.07	-0.08	-0.16	-0.24	-0.32	-0.41	-0.50	-0.58	-0.67	-0.76	-0.86	-1.04	-1.25	-1.46	-1.67	-1.89
10	0.23	0.16	0.08	-0.08	-0.17	-0.25	-0.34	-0.43	-0.52	-0.61	-0.71	-0.80	-0.90	-1.10	-1.32	-1.54	-1.76	-1.99
11	0.25	0.16	0.08	-0.09	-0.18	-0.27	-0.37	-0.46	-0.56	-0.65	-0.75	-0.85	-0.96	-1.16	-1.39	-1.61	-1.84	-2.09
12	0.27	0.18	0.09	-0.10	-0.20	-0.29	-0.39	-0.49	-0.59	-0.70	-0.80	-0.91	-1.02	-1.23	-1.46	-1.70	-1.94	-2.20
13	0.29	0.19	0.10	-0.10	-0.21	-0.31	-0.42	-0.52	-0.63	-0.74	-0.85	-0.97	-1.08	-1.31	-1.55	-1.80	-2.05	-2.31
14	0.32	0.21	0.11	-0.11	-0.22	-0.32	-0.44	-0.55	-0.66	-0.78	-0.91	-1.02	-1.14	-1.39	-1.65	-1.91	-2.17	-2.44
15	0.35	0.23	0.12	-0.12	-0.24	-0.35	-0.48	-0.60	-0.71	-0.84	-0.97	-1.10	-1.23	-1.50	-1.76	-2.03	-2.30	-2.58
16	0.37	0.24	0.12	-0.13	-0.26	-0.38	-0.52	-0.65	-0.77	-0.90	-1.03	-1.17	-1.31	-1.60	-1.88	-2.16	-2.44	-2.72
17	0.40	0.26	0.13	-0.14	-0.27	-0.41	-0.54	-0.68	-0.82	-0.96	-1.10	-1.25	-1.40	-1.70	-1.99	-2.28	-2.58	-2.87
18	0.44	0.29	0.14	-0.14	-0.29	-0.44	-0.58	-0.73	-0.88	-1.03	-1.18	-1.33	-1.49	-1.80	-2.10	-2.41	-2.72	-3.02
19	0.47	0.32	0.16	-0.15	-0.30	-0.46	-0.62	-0.78	-0.94	-1.10	-1.26	-1.42	-1.58	-1.90	-2.22	-2.54	-2.86	-3.17
20	0.51	0.34	0.17	-0.16	-0.32	-0.49	-0.66	-0.82	-0.98	-1.15	-1.33	-1.48	-1.65	-2.00	-2.32	-2.65	-2.98	-3.33
21	0.53	0.35	0.18	-0.17	-0.34	-0.51	-0.68	-0.85	-1.02	-1.20	-1.38	-1.54	-1.72	-2.06	-2.41	-2.76	-3.10	-3.45
22	0.56	0.38	0.19	-0.17	-0.36	-0.53	-0.71	-0.90	-1.07	-1.25	-1.44	-1.61	-1.78	-2.13	-2.48	-2.84	-3.20	-3.56
23	0.58	0.40	0.20	-0.18	-0.37	-0.55	-0.74	-0.92	-1.11	-1.30	-1.49	-1.66	-1.84	-2.20	-2.56	-2.93	-3.30	-3.67
24	0.60	0.40	0.20	-0.18	-0.38	-0.56	-0.77	-0.96	-1.16	-1.35	-1.54	-1.72	-1.91	-2.27	-2.65	-3.03	-3.40	-3.78

Bureau of Internal Revenue Bulletin 7 (1937)

Reagents

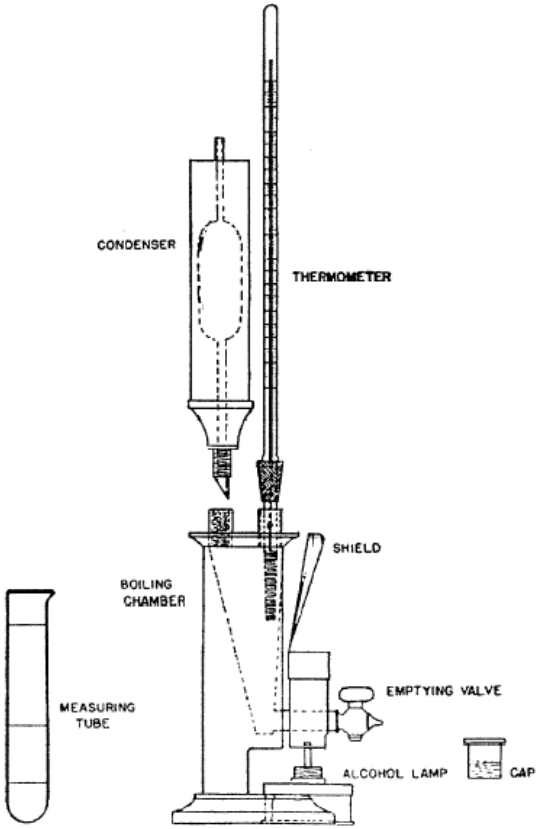
- Reagents:
  - Antifoam
  - Distilled water
  - 2 N aqueous Sodium Hydroxide (NaOH) solution
- Also:
  - Ice
  - Boiling Beads/Chips

Basic Equipment
Distillation – Densitometry

Densitometer

100 ml volumetric flask

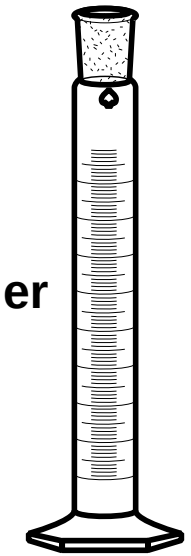
Basic Equipment
Ebulliometry



Ebulliometer

50 ml pipette

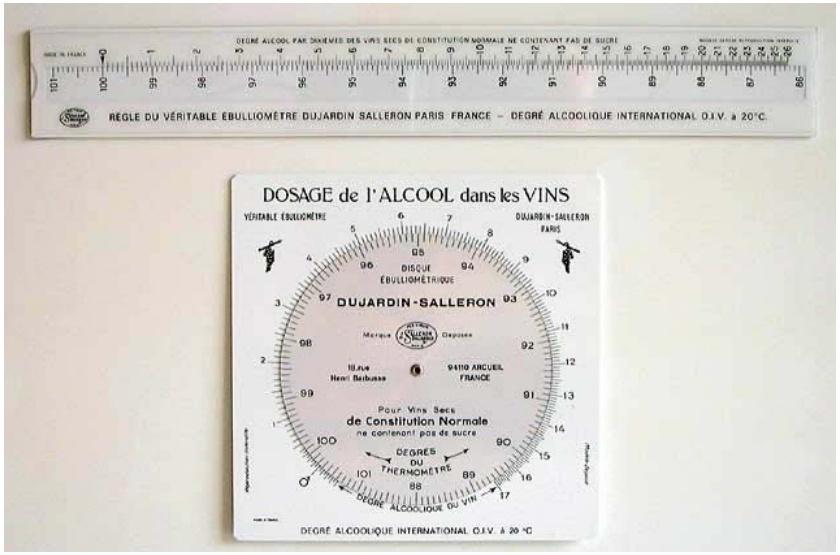
100 mL graduated cylinder



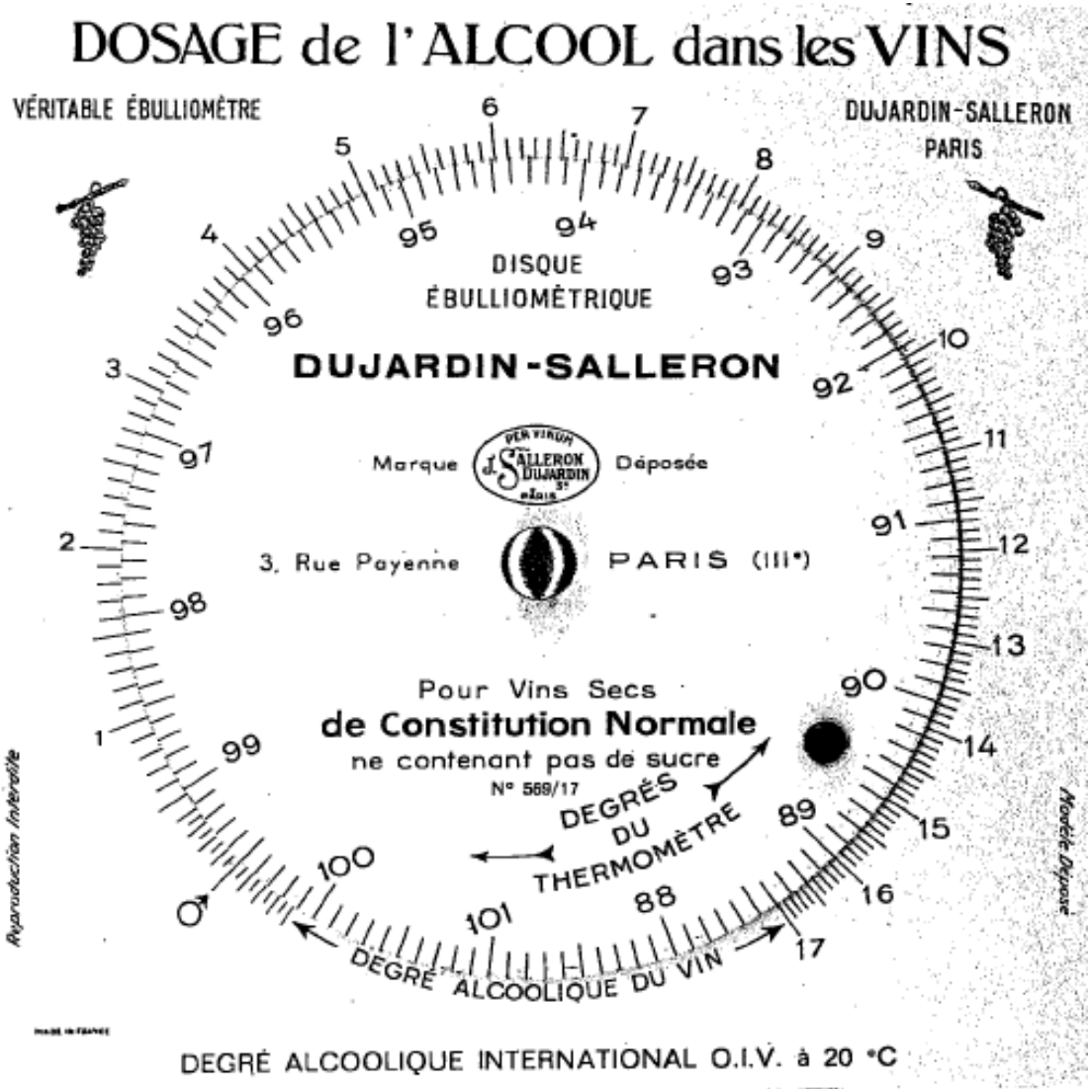
Basic Equipment & Reagents
Ebulliometry

- Alcohol/micro Bunsen burner
- 200 mL volumetric flask
- Narrow range ebulliometer thermometer
- Slide rule/conversion table

Reagents:
Antifoam
Distilled water



Interpreting the Slide Rule



Boiling point of the water?

Alcohol content if wine boils at 91.15 degrees?

Basic Equipment
Ebulliometry

Electronic Ebulliometer

Basic Equipment
Near Infrared

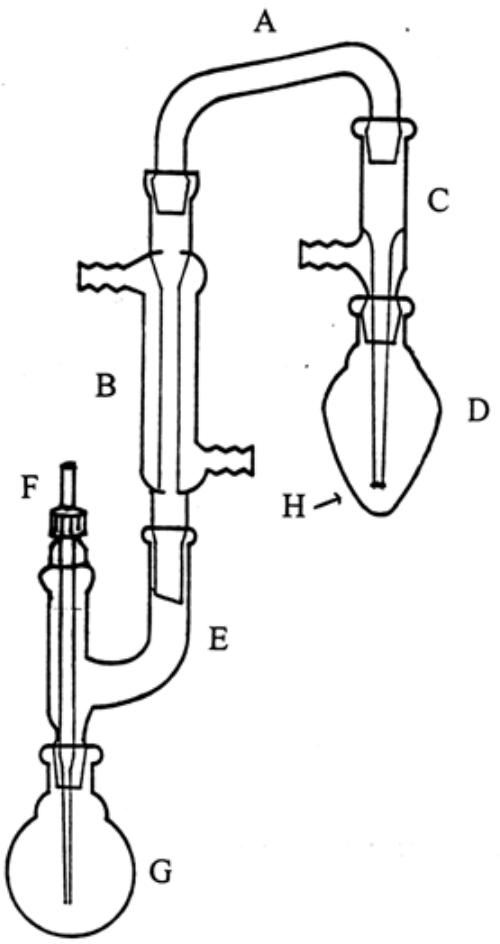
Analytical Tests

Determination of Sulfite in Wine (Sulfur Dioxide)

Aeration-Oxidation
Ripper

Basic Equipment

Sulfur Dioxide – Aeration Oxidation



apparatus for SO_2 determination by aeration-oxidation

A. Connecting adapter 19/22
B. Graham Condenser
C. Vacuum adapter
D. 50 mL pear flask
E. Claissen adapter
F. Pasteur pipette
G. Round bottom flask
H. Sintered end of vacuum adapter

Reagents

- Reagents:
  - 0.01 N Sodium Hydroxide (NaOH)
    Buy premixed or dissolve 0.41 grams NaOH to 1 liter
  - 0.3% hydrogen peroxide
    Dilute 10 mL of 3% hydrogen peroxide to 100 mL
  - Indicator Solution
    0.100 g methyl red and 0.05 g methylene blue in
    100 mL of 50% ethanol in water
  - 25% phosphoric acid or 4 M hydrochloric acid
  - Standardized 0.01 N sulfuric acid (for calibrating the 0.01 N NaOH)

Basic Equipment

Sulfur Dioxide – Ripper Method

Reagents

- Reagents:
  - 1 N Sodium Hydroxide (NaOH)
    Buy premixed or carefully dissolve 41 grams NaOH
    to 1 L water
  - 1+3 Sulfuric Acid Solution
    Add 10 mL of concentrated sulfuric acid carefully to 30 mL of water
  - 1% Starch Indicator Solution
    Mix starch in cold water, drop in boiling water, heat until clear
  - 0.020 N Iodine solution standardized against sodium thiosulfate
  - Standardized sodium thiosulfate solution

Analytical Tests

Wine Acidities

Total Acidity

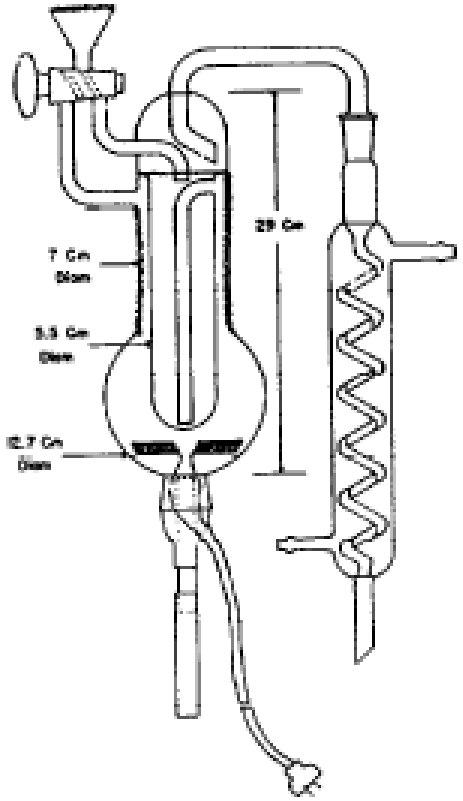
Volatile Acidity

Basic Equipment

Titratable Acidity

Basic Equipment

Volatile Acidity



Cash Still

Basic Equipment

Volatile Acidity

Self Evacuating Cash Still

Reagents:

0.3% peroxide to correct for sulfite
0.1 N sodium hydroxide
Indicator: Phenolphthalein

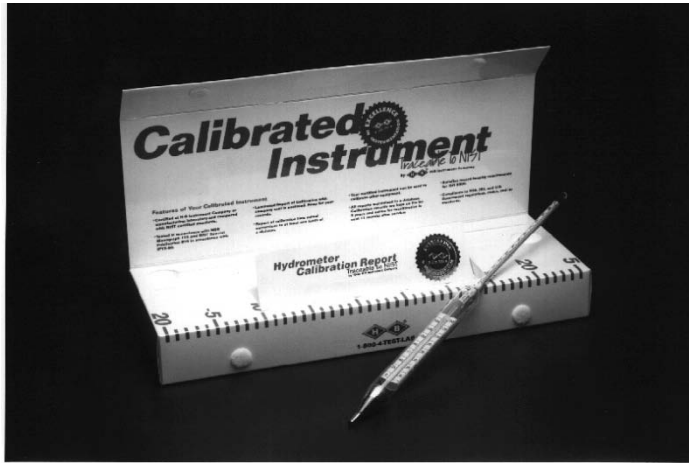
Analytical Tests

Fill

Basic Equipment

Fill

Top loader balance



Contact Information

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