



## SETTING UP A WINERY FOR BASIC MUST AND WINE ANALYSIS

**T. STEINER and P. PIERQUET**  
**Dept. of Horticulture and Crop Sciences**  
**The Ohio State University/OARDC, Wooster, OH.**



When setting up a wine laboratory, one must not underestimate the importance of a proper equipped laboratory. The wine laboratory is one of the most important places in the winery. To produce an “award winning wine” it is essential that you utilize the wine laboratory to its fullest potential. By not paying special attention to your basic wine analysis is relative to buying a BMW sports car and never changing your oil. Sooner or later this will come back to hurt you. If you place a lot of time, effort and money in your winery why would you not be properly equipped with a well thought out wine analysis lab? There are several important factors to consider when setting up a properly equipped laboratory.

### 1. **ROUTINE ANALYSIS**

Before we make any decisions about the physical size and location of the wine laboratory, we need to determine what basic must and wine analysis procedures are necessary to produce a quality wine. This will help us in later decisions as to the overall size, shelf space, storage and special needs based on specific laboratory procedures.

#### **A. Soluble Solids:**

Knowledge of the sugar content is important to the winemaker in determining the maturity of the grapes, the amount of amelioration needed, the approximate alcohol content and the completeness of fermentation.

Soluble Solids content are measured using a Brix (Balling) hydrometer that measures the density of an aqueous solution. The Brix (Balling) hydrometer is calibrated in degrees corresponding to percent of sucrose in water at 20°C (68 °F) or grams of sucrose per 100 grams of water at 20°C. As fermentation proceeds there is an increase in Ethanol. Since ethanol is less dense than water, most dry wines will have a negative Brix (Balling) reading. To determine accurate residual sugar readings after fermentation you would have to utilize another procedure such as the <sup>®</sup>Clinitest or Gold Coast Method.

#### **B. pH and Titratable Acidity:**

Acid levels significantly influence wine pH that typically falls between 3.2 to 3.7 on a pH scale. Monitoring pH is important to help determine ripeness of the grape; color stability of must and wine along with chemical and microbial stability. The major acids present in wine are

tartaric and malic acid. Acids are responsible for the fresh crisp taste of wine. Wines with a low acid content appear to be “flat” and insipid. Wines with high acidity appear to be tart and puckery.

Orange tinted rose’s and brownish purple red wines may indicate wines with a high pH value. As wine pH lowers towards 3.0 the color of our rose’s and red wines will become a brighter pink and a deep ruby red respectively. Wine stability will also benefit from a lower pH. As the pH increases towards 4.0 we have a serious threat for microbial growth, physical and chemical instability.

Therefore, we can see it is very important to monitor our pH and acid levels properly. Detailed analysis will give us the basis for determining any chemical additions needed to the must or wine to affect our pH and acid levels in achieving proper quality control in addition to a more balanced wine. Wine pH can be measured using a pH meter accurate to .01 pH units. I would recommend a benchtop meter with a minimum two point calibration curve and able to read in both pH and mV modes. Total Titratable Acidity is determined by a direct titration procedure. The wine acids are titrated with a 0.1N standardized sodium hydroxide solution to a phenolphthalein endpoint of 8.2 on the pH scale.

### **C. Sulfur Dioxide:**

One of the most important quality control procedures that a winery should perform in premium quality wine production is through the proper use and monitoring sulfur dioxide (SO<sub>2</sub>). Sulfur dioxide serves three major functions in winemaking: 1) control of undesirable microorganisms 2) denaturation of browning enzymes 3) an antioxidant.

It is important to regularly monitor your SO<sub>2</sub> concentrations immediately after initial addition following primary fermentation. Sulfur dioxide in wine occurs in two forms, bound and free with their sum equaling the total SO<sub>2</sub> concentration. Generally, we must be able to maintain between 20 to 40 parts per million “free” SO<sub>2</sub> depending on wine pH after primary fermentation to produce quality sound wines. Since free SO<sub>2</sub> binds wine substances such as aldehydes, anthocyanins, proteins and aldo-sugars, we need to monitor our free SO<sub>2</sub> concentration regularly through the life of the wine. We also need to be aware that the maximum amount of total SO<sub>2</sub> permissible in wines made in the United States is 350 ppm.

There are several methods available for testing free, bound and total SO<sub>2</sub> in wines. Measurement of free SO<sub>2</sub> with a “@Titrets” kit would be recommend for only a very rough estimate of SO<sub>2</sub> levels. These may be utilized only for quick SO<sub>2</sub> estimations of tanks and barrels. The Ripper method is arguably the most common procedure used in many wineries

today for SO<sub>2</sub> determination. The Ripper method is a redox titration that is more accurate than the “Titrets” kit. There are some limitations however in accuracy with this method. 1) Certain compounds mostly in red wine can reduce iodine causing some erroneous results. 2) Detection of end points in red wine can be difficult. 3) Volatilization of SO<sub>2</sub> can occur during titration. 4) This procedure cannot be performed on wines with sorbic acid addition. With these limitations in mind the aeration-oxidation procedure is highly recommended for precise results.

#### **D. Ethanol Content:**

Most table wines have an alcohol content of 10-14% by volume. A wine with low alcohol content may be perceived as being slightly thin in body and will be more susceptible to microbial spoilage than wines with greater alcohol content. The other end of the spectrum includes wines being too high in alcohol concentration that provide a delicate wine with a “hot” sensory evaluation on the palate. The accurate concentration of alcohol must be known also to abide by federal (TTB) and state (ODLC) regulations for alcohol concentrations and label laws.

The ebulliometric method for alcohol determination is the most recommended method for the wine industry. This method is based on the boiling point of a mixture of ethanol and water. Ethanol will lower the boiling point of water. As the ethanol concentration increases, the boiling point of the aqueous solution will lower. This method also has some limitations. Certain compounds such as sugars can influence the boiling point of a wine. Wines with higher sugar concentrations need to be diluted with water below 2% as to yield a boiling point of 96°C to 100°C. Therefore the result must be multiplied by the dilution factor to give the correct result. This dilution factor is questioned as to its integrity since you are multiplying the relative error back into the equation.

It should be noted that wines analyzed for alcohol content with higher than 2% residual sugar concentration by the ebulliometer approximates that of the actual alcohol content. The better choice for alcohol measurement for wines of high sugar content would be by the distillation method.

#### **E. Protein Stability:**

Precipitation of protein may cause deposits to form in white wines. Protein precipitation does not appear in red wines due to the natural high tannin concentration removing protein during the vinification process. There are several factors that cause precipitation of proteins such as heat, shaking, heavy metals and ultraviolet light.

The most common method for obtaining protein stable wines is to fine with bentonite. It is recommended to perform bentonite fining trials in the laboratory to determine the correct amount to add in the cellar.

A simple method for the determination of protein stability is to expose a measured amount of wine to elevated temperatures for a specified amount of time and observe for haze formation which indicates protein instability and the need for bentonite fining trials.

#### **F. Tartrate Stability:**

Tartaric acid and its salts, potassium bitartrate and calcium tartrate are naturally occurring in grape juice and wine. Unless potassium bitartrate is removed during the wine making process, the formation of a crystalline deposit will form in the bottled wines when subjected to lower temperatures. Although these deposits are not considered as spoilage or a human health concern, many consumers consider them a major defect.

The three most common methods of tartrate removal are chilling, contact seeding in conjunction with chilling and ion exchange.

One of the most common procedures for estimating tartrate stability is by the cold and or freeze tests. This procedure subjects a known amount of sample to a reduced temperature for a specified period of time. The absence of a crystal formation would indicate the wine being tartrate stable.

#### **G. Malolactic Fermentation:**

The bacterial conversion of malic acid to lactic acid and carbon dioxide in wine is termed malolactic fermentation. This bacterial fermentation is caused by a certain lactic acid bacteria and usually occurs at the end or just after alcoholic fermentation.

Some research has also shown a benefit of either co-inoculation or inoculating near half completion of primary fermentation. One would have to take a closer look at compatible yeast and bacterial strains for this purpose in avoiding any competitive factors which can slow or stop either the primary or secondary fermentation process.

Also, both pH and temperature play a vital role in the success of malolactic fermentations. A pH below 3.2 and temperatures below 70° F make it a less conducive environment for successful malolactic fermentation to complete.

Other factors that influence successful malolactic fermentation are bacterial strain, aeration, alcohol, sulfur dioxide and amount of inoculum. One of the main effects of malolactic fermentation is the increase in pH

and the decrease in titratable acidity. Therefore wines having a relatively low pH and high acidity may benefit from a properly induced malolactic fermentation.

Depending on chemical conditions and winemaking style, it is important to be able to monitor the process of malolactic fermentation. The simplest and most common procedure for monitoring malolactic fermentation is by paper chromatography. Malolactic fermentation can be confirmed with the absence of a malic acid spot along with the formation of a lactic acid spot on the finished chromatogram. Another more accurate and technical way to analyze for malolactic completeness is through the use of an enzymatic assay utilizing a spectrophotometer.

#### **H. Volatile Acidity:**

Measuring the volatile acidity will provide a good indication of wine spoilage. A wine high in volatile acidity is affected by spoilage organisms typically in the production of both acetic acid and ethyl acetate. It is the production of acetic acid and ethyl acetate that gives us a sharp pungent “vinegar” aroma.

Formation of volatile acidity can occur from many factors such as pH, available nitrogen, fermentation temperature and length, oxidative conditions and surface area. The federal law limits the maximum amount of volatile acidity in both red and white wines. The legal limit for white wines is 0.12% along with 0.14% for red wines.

Therefore it is essential to monitor the volatile acidity. Since acetic acid is a “fixed” acid, it is essential to analyze the concentration of acetic acid with a distillation method. This is determined by passing a current of steam through the wine sample then collecting the distillate. The distillate is titrated with a standardized sodium hydroxide solution to a phenolphthalein endpoint.

#### **I. Sensory Evaluation:**

The process of producing a quality wine must not rely on chemical evaluation alone. Sensory evaluation plays a critical role in wine processing. Through sensory evaluation a winemaker can tell if a wine has an “off” color, odor, flavor profile or pleasing to the nose and palate. It is important for the winemaker to perform numerous sensory evaluations on a wine throughout the vinification process in drawing a conclusive decision based on the wines attributes as a varietal or blend.

Therefore, it is essential to have a separate sensory evaluation area free from any distractions and odors. The sensory lab is best separated from the wine lab to reduce chemical smells that will interfere with the sensory evaluation. The counters and walls of the sensory evaluation room

should be white in color with plenty of light from incandescent or natural lighting. Fluorescent lighting will tend to create brown hues in red wines. It also makes it convenient to have a sink located in the room for sensory evaluation of wines.

## **2. SIZE**

- A. Make sure that you have enough space for your laboratory routine analysis.
- B. Include proper amount of storage space on shelves, in cabinets and drawers for equipment, glassware and chemicals.
- C. The laboratory should be large enough to accommodate several people at once. (Size recommendation being no smaller than 11' wide x 16' long.)
- D. Include room for future expansion

## **3. LOCATION**

- A. Laboratory should be centrally located between crushing/pressing pad, fermentation, cold storage and bottling areas if possible.
- B. The Laboratory should be enclosed with a separate intake and exhaust vent.
- C. The sensory lab should be located separately from the wine lab in a location free of disturbances and odors.
- D. Include plans for laboratory to be located in relation to utilities: water, natural gas, heating and cooling.

## **4. LABORATORY EQUIPMENT**

- A. See attachment listing the essential equipment

## **5. LABORATORY CHEMICALS**

- A. Store chemicals according to the specific manufacturer directions that are typically on the label.
- B. Keep all acids, bases, solvents and oxidizers in their own designated area separate from each other and in enclosed cabinets approved for that use.
- C. Label all incoming chemicals with receiving date and initials.
- D. See attached list of laboratory analytical chemicals.

## **6. ORGANIZATION**

### **A. Laboratory**

- 1. Design specific areas for each analytical procedure (see fig. 2.50-2.53). It is essential to have laboratory equipment ready to use at all times.

2. Procedures requiring gas, water, vacuum or a sink should be located close to the source.
3. The wine lab should be equipped with a double stainless steel sink, refrigerator, and oven or incubator.
4. Label all drawers, cabinets, shelves, etc. with the contents they contain for quicker access.
5. Have laboratory analytical procedures written up and accessible in the lab in the event the laboratory technician is not available to run samples.

## **B. Paperwork**

1. It is extremely important to keep precise and organized records of wine analysis. This will enable you to trace your analytical data for any given sample back to the specific date when the procedure was performed. There should be separate data books for must analysis, fermentation analysis and post fermentation analysis.
2. Keep a logbook with the standardization of titration chemicals. This will include their concentration and date of standardization.
3. Keep all laboratory equipment and chemical purchases listed in a file for future reference.

## **C. Personnel**

1. Appoint a laboratory supervisor that is in charge of all laboratory analysis, procedures and purchases.

## RECOMENDED WINE LABORATORY EQUIPMENT

<u>EQUIPMENT</u>	<u>UNITS</u>	<u>EST. UNIT COST</u>	<u>QUANTITY</u>	<u>Total COST</u>	<u>SUPPLIER</u>	<u>CAT. NO.</u>
Pyrex Beakers	12/cs-50 mls.	\$61.00	1	\$61.00	Fisher	02-540G
Pyrex Beakers	12/cs-150 mls.	\$60.00	1	\$60.00	Fisher	02-540J
Pyrex Beakers	12/cs-250 mls.	\$62.00	1	\$62.00	Fisher	02-540K
Pyrex Beakers	6/cs-1000mls.	\$87.00	1	\$87.00	Fisher	02-540P
Pyrex Beakers	4/cs-2000 mls	\$113.00	1	\$113.00	Fisher	02-540R
Graduated Cylinders	10 mls.	\$20.00	2	\$40.00	Fisher	08-570B
	25 mls.	\$28.00	2	\$56.00	Fisher	08-570C
	100 mls.	\$38.00	2	\$76.00	Fisher	08-570E
Volumetric Flasks	25 mls.	\$72.00	2	\$144.00	Fisher	10-212BB
	100 mls.	\$86.00	2	\$172.00	Fisher	10-212B
	500 mls.	\$116.00	1	\$116.00	Fisher	10-212E
	1000 mls.	\$141.00	1	\$141.00	Fisher	10-212F
Burets	25 mls.	\$95.00	3	\$285.00	Fisher	03-700B
	50 mls.	\$153.00	1	\$153.00	Fisher	03-700C
Membrane Filters	.45um X 47mm	\$153.00	100/pk	\$153.00	Fisher	09-905-17
Funnels	****	****	****			
Separatory Funnels	500 mls.	\$191.00	1	\$191.00	Fisher	10-435-5D
Repipettors	10 mls.	\$178.00	1	\$178.00	Fisher	13-687-62B
Acid Dilutor	10mls	\$390.00	1	\$390.00	Fisher	13-687-35
Repeater Pipet(glass)	100 mls.	\$267.00	1	\$267.00	Fisher	K759300-0100
Pasteur Pipets	250/pk.	\$41.00	1	\$41.00	Fisher	13-678-8B
Rubber Bulbs	24/pk.	\$41.00	1	\$41.00	Fisher	03-448-26
Pipet Pump	10 mls.	\$39.00	1	\$39.00	Fisher	13-683-1C
	25 mls.	\$47.00	1	\$47.00	Fisher	13-683-1D
Serological Pipets	5 mls. 12/cs.	\$145.00	1	\$145.00	Fisher	13-664F
	10 mls. 12/cs.	\$168.00	1	\$168.00	Fisher	13-664G
	25 mls. 12/cs.	\$223.00	1	\$223.00	Fisher	13-664H
Digital Scale	****	****	1	****		
Lighted Stirplate	****	\$676.00	1	\$676.00	Fisher	11-474-526
Stirring Magnets	6-pack	\$33.00	1	\$33.00	Fisher	14-513-58SIX
pH Meter Kit	****	\$984.00	1	\$984.00	Fisher	13-636-AB15
Aeration/Oxidation	Free and Total	\$290.00	1	\$290.00	R&D Glass	
Stand & Clamps		\$125.00	1	\$125.00	R&D Glass	
Ebulliometer	DuJardin-Salleron	\$1,050.00	1	\$1,050.00	Vinquiry	*****
Fermentation Tubes		\$24.95	2	\$49.90	ScienceLab.com	10-601-5
Chromatography Kit		\$55.60	1	\$55.60	PIWC	PCKV
#80 Vol. Acid Still	****	\$580.00	1	\$580.00	R&D Glass	

\* Indicates non-discount prices



## RECOMENDED WINE LABORATORY EQUIPMENT

Stand & Clamps		\$125.00	1	\$125.00	R&D Glass	
Brix Hydrometers	-5 °to 5°	\$20.00	1	\$20.00	Vinquiry	*****
	0 to 10	\$20.00	1	\$20.00	Vinquiry	*****
	10° to 20°	\$20.00	1	\$20.00	Vinquiry	*****
Hydrometer Cylinder		\$60.00	1	\$60.00	Vinquiry	20-064-0250
Wash Bottles	6/pack	\$40.00	1	\$40.00	Fisher	02-897-10
Thermometer	-20°C to 150° C	\$15.00	1	\$15.00	Fisher	14-985C
Hot Plate	****	\$490.00	1	\$490.00	Fisher	11-102-49SH
Safety Glasses	****	\$8.00	2	\$16.00	Fisher	17-230-A
Lab Gloves - Nitrile	100/pk.	\$39.00	1	\$39.00	Fisher	19-050-550A
Spectronic 20D+	Digital	\$2,142.00	1	****	Fisher	14-385-130
Lab-Jack	6" X 6"	\$239.00	1	\$239.00	Fisher	14-673-51
<b>EST. TOTAL COST</b>				\$8,376.50		
<b>Vendor Contact Info</b>						
Fisher	(800)766-7000					
Vinquiry	(707)838-6312					
Gusmer	(715)258-5525					
PIWC	(814)725-1314					
R&D Glass	(510)547-6464					
ScienceLab.com	(800)901-7247					

\* Indicates non-discount prices

# RECOMENDED LABORATORY CHEMICALS

<u>CHEMICALS</u>	<u>CONC.</u>	<u>UNITS</u>	<u>EST. COST</u>	<u>QUANTITY</u>	<u>SUPPLIER</u>	<u>CAT. NO.</u>	
Hydrochloric Acid	.1N	1L	\$51.00	1	Fisher	SA54-1	
Sulfuric Acid	1.0N	1L	\$49.00	1	Fisher	SA212-1	
Phosphoric Acid	85%	1L	\$163.00	1	Fisher	A242-1	
Sodium Hydroxide	.01N	1L	\$68.00	1	Fisher	SS284-1	
	.1N	4L	\$83.00	1	Fisher	SS276-4	
	1N	4L	\$89.00	1	Fisher	SS266-4	
Ethyl Alcohol	95%	4L	\$195.00	1	Fisher	A995-4	
Sodium Thiosulfate	.025N	1L	\$66.00	1	Fisher	SS370-1	
Methyl Red	****	10.0 g.	\$47.00	1	Fisher	M296-10	
Phenolphthalein	****	100.0 g.	\$92.00	1	Fisher	P79-100	
Starch, Soluble	****	100.0 g.	\$60.00	1	Fisher	S516-100	
S02 Indicator	****	60 ml	\$9.00	1	Vinquiry	*****	
pH Buffer	pH 4.00	500.0 ml	\$35.00	1	Fisher	SB101-500	
	pH 7.00	500.0 ml	\$34.00	1	Fisher	SB107-500	
Storage Solution	****	1.0 Liter	\$57.00	1	Fisher	SE40-1	
Potassium Iodide	Granular	100 g.	\$223.00	1	Fisher	P410-100	
Iodine	Flakes	100 g.	\$395.00	1	Fisher	I37-100	
Iodine	.1N	1L	\$68.00	1	Fisher	S186-1	
Sodium Bicarbonate	****	500.0 g.	\$52.00	1	Fisher	S233-500	
Hydrogen Peroxide	30%	100 ml	\$68.00	1	Fisher	H325-100	
Gold Coast Solution	#1	250 ml	\$10.00	1	Vinquiry	*****	
	#2	250 ml	\$32.00	1	Vinquiry	*****	
	#3	250 ml	\$32.00	1	Vinquiry	*****	
	(Sulfuric acid solution)	#4			1	Fisher	See above
		#5	250 ml	\$10.00	1	Vinquiry	*****
		#6	250 ml	\$10.00	1	Vinquiry	*****
Potassium Metabisulfite		500.0 g.	\$198.00	1	Fisher	P197-500	
EST. TOTAL			\$2,196.00				

\* Indicates non-discount prices

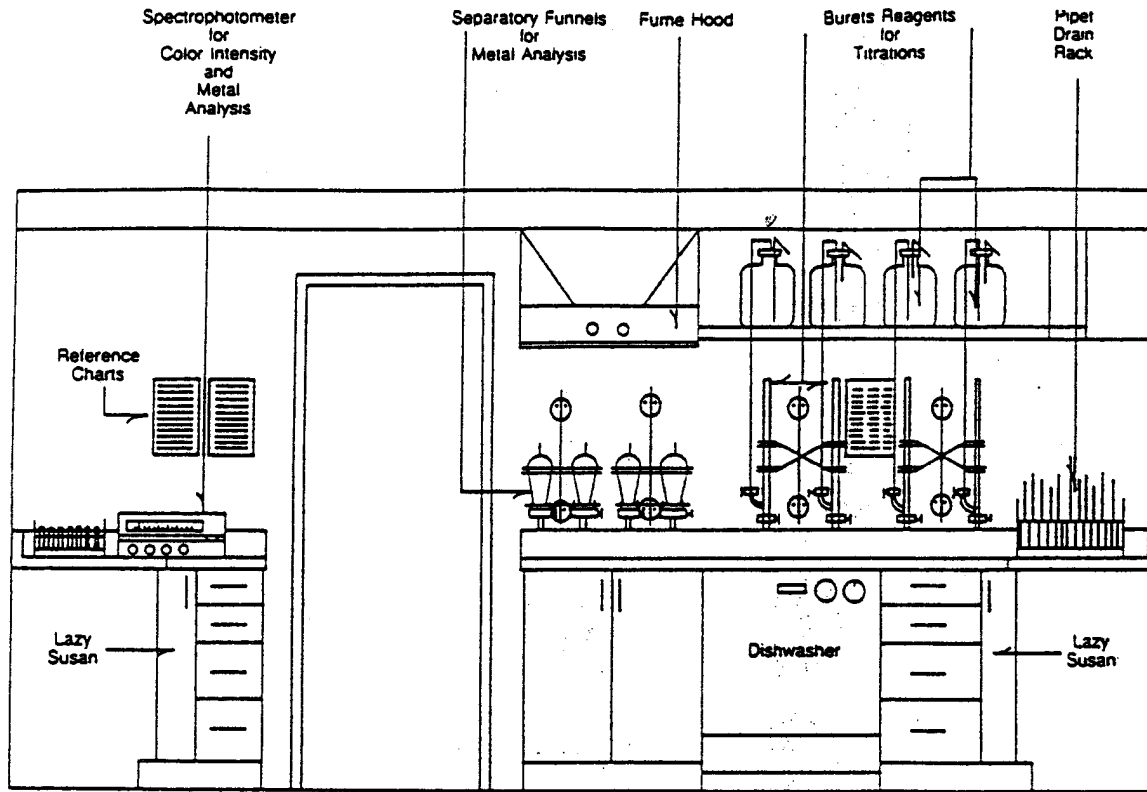


FIG. 2.50. INTERIOR ELEVATION OF ANALYTICAL LABORATORY

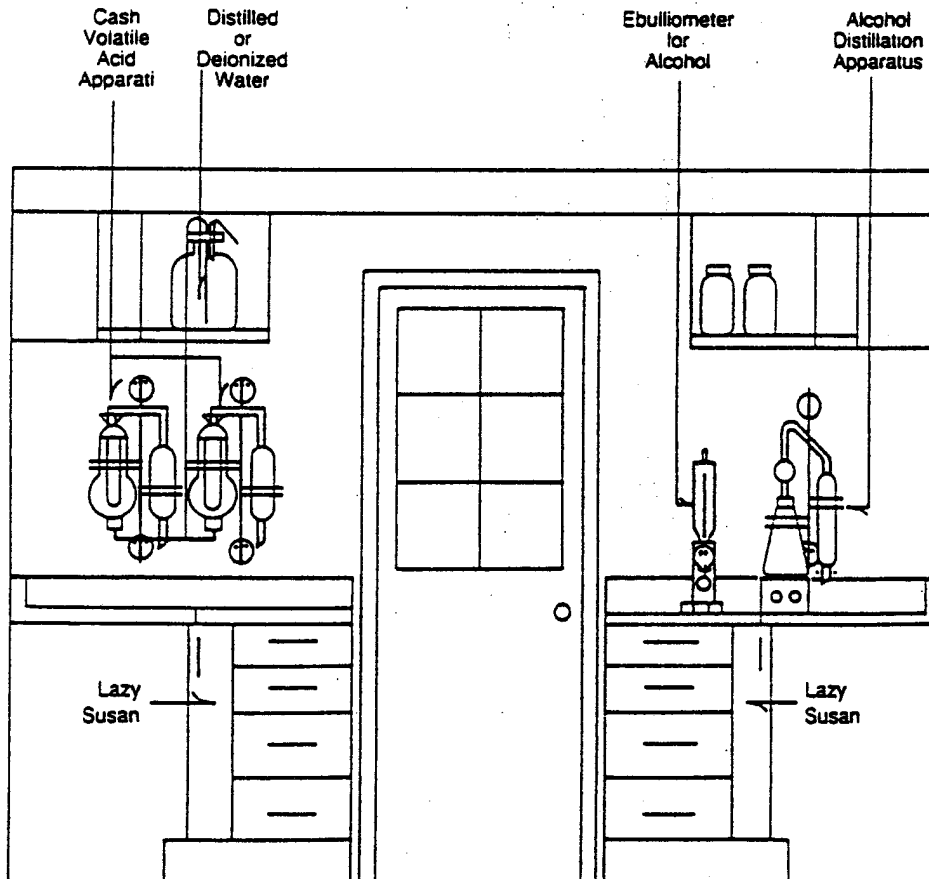


FIG. 2.51. INTERIOR ELEVATION OF ANALYTICAL LABORATORY

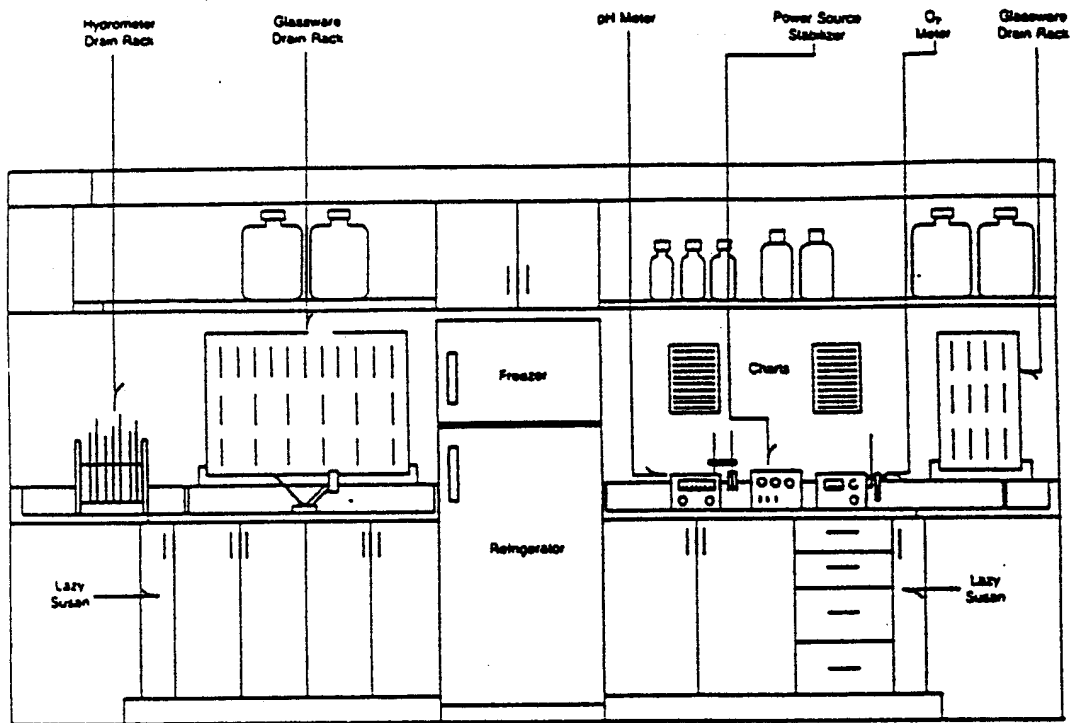


FIG. 2.52. INTERIOR ELEVATION OF ANALYTICAL LABORATORY

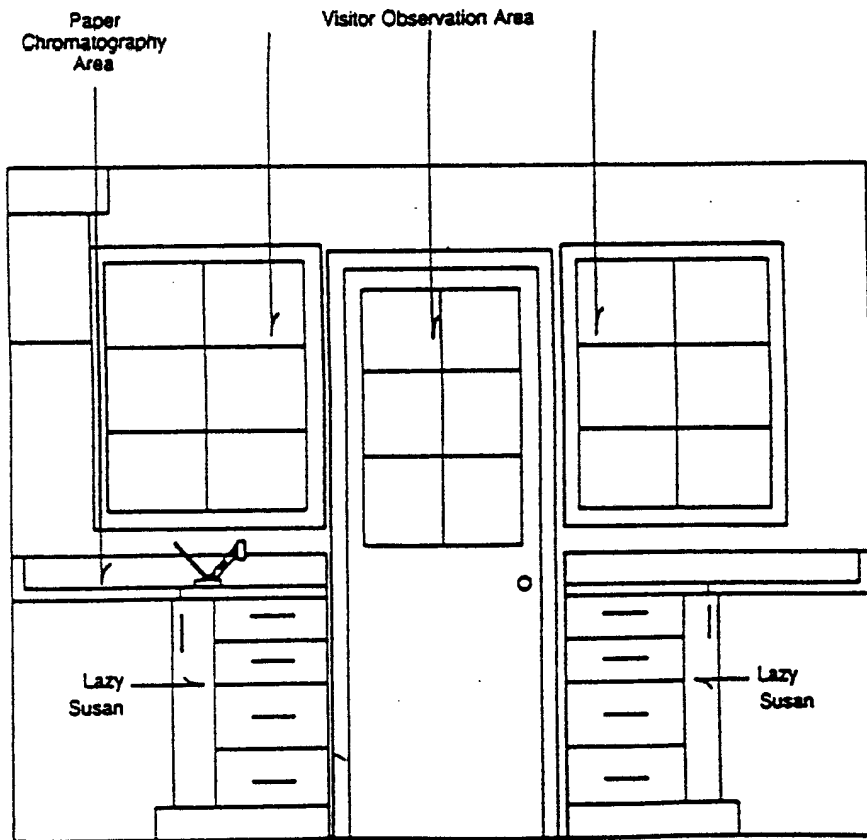


FIG. 2.53. INTERIOR ELEVATION OF ANALYTICAL LABORATORY