

Option B – Diaphragm Pump

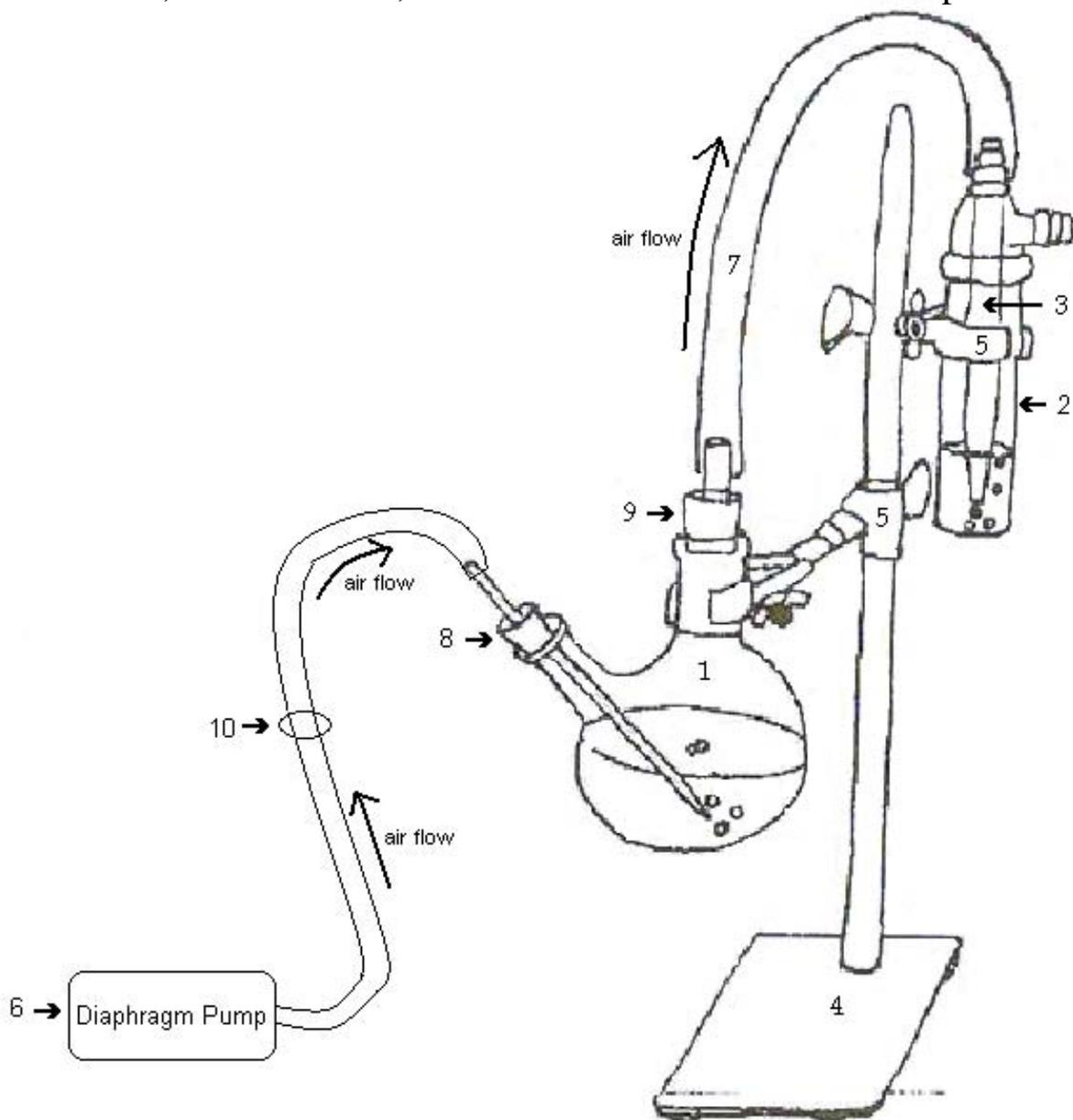


Apparatus for Aeration Oxidation Assembly for Free SO₂

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|------------------------|------------------------------|
| 1. Sidearm Flask | 6. Diaphragm Pump |
| 2. Bubbler Tube | 7. Vacuum Tubing |
| 3. Bubbler Insert | 8. #1 Stopper with Pipet |
| 4. Support Stand & Rod | 9. #4 Stopper with Glass Rod |
| 5. Clamps | 10. Tubing Clamp |

Not shown: 20ml Volumetric Pipet

Chemicals (not shown): 16oz NaOH 0.01N, 32oz H₃PO₄ 25%, 1 oz H₂O₂ 30%, 2oz HCl 0.01N, ½oz indicator, 1oz each dilute acid & base in dispensers



APPARATUS

1. Refer to the diagram. Set up the support stand and rod, positioning one clamp about $\frac{1}{3}$ of the way up the rod so it is pointing forward and slightly to one side. The other clamp should be about $\frac{2}{3}$ of the way up the rod and at a 90° angle to the other clamp.
2. Fasten the ground-glass neck of the round-bottomed flask in the lower clamp. The stopper with the pipet in it goes into the sidearm, with the tip of the pipet reaching almost to the bottom of the flask.
3. Attach the diaphragm pump to the 1ml pipet via the tubing supplied as shown. Attach one tubing clamp onto the tubing as shown.
4. Separate the impinger (bubbler) into 2 pieces, one a male part, the "insert", and a female part, the "tube". Clamp the tube in the upper clamp a little below the ground-glass junction.
5. Connect the free end of the small piece of tubing (with the stopper attached) to the TOP of the impinger insert and fit the insert into the impinger tube. If the tubing is too tight to make the connections, soften the ends in hot water; if it is too loose, trim a small piece off the tubing until it fits.
6. Complete the connections by securing the rubber stopper into the top arm of the flask.

PROCEDURE

1. Prepare 100ml of 1% hydrogen peroxide by diluting 3ml of 30% H_2O_2 to 100ml with distilled or deionized water (make more or less dilute H_2O_2 as needed). Do not use the 3% H_2O_2 in the drugstore, the color change is poor and the results will be too low! The 30% H_2O_2 has a shelf life of about 6 months in the refrigerator; the diluted solution may be refrigerated overnight but should not be kept longer.
2. Fill the bubbler tube to the etched mark with the diluted peroxide. Add 4 to 6 drops of indicator solution. The peroxide-indicator mixture will be a purplish-gray to green color. If it is a bright green, your water may be too

alkaline (even distilled/deionized is not neutral). Use a different source of water or you will get falsely lower results. Adjust the mixture with the dilute base until the solution turns an olive green color that persists for 30 seconds.

3. Clamp the bubbler tube in place and fit the bubbler insert into it.
 4. Pipet 20ml of the wine to be tested into the sidearm flask. Add 20ml of 25% H₃PO₄ (phosphoric acid) and stopper the flask according to the diagram. The tip of the pipet in the small stopper must be below the level of the liquid in the flask. Check all connections to be sure they are tight
 5. Turn on the diaphragm pump to create air pressure in the system. Air is pushed through the pipet in the flask and out through the bubbler into the peroxide, bringing the free SO₂ with it. Use tubing clamp to adjust air flow. Aspirate 10 minutes. Both wine and peroxide should bubble vigorously throughout aspiration. Bubbling longer than 10-15 minutes may cause a small percentage of bound SO₂ to dissociate, resulting in a slightly higher reading.
- (Note: If the peroxide mixture in the bubbler tube does not turn purple after 1 minute of aspiration, the test wine contains essentially no free SO₂, so you need not continue the aspiration.)
6. Turn the diaphragm pump off and disconnect the stopper from the flask, allowing the remaining liquid to run out of the bubbler insert into the tube (shake gently if necessary).
 7. Titrate the peroxide in the bubbler tube with 0.01 NaOH to the olive green color obtained prior to aspiration.

8. Calculate the free SO₂ in parts per million:

Free SO₂ (ppm) = Normality of NaOH used x 1600 x ml of NaOH used

If the NaOH is exactly 0.01N:

Free SO₂ (ppm) = 16 x ml of NaOH used

Blank: For vinegar or wines with illegally high volatile acidities, run a second sample of the wine, but this time put a few drops of diluted peroxide

in the flask with the wine and the phosphoric acid before aspiration. Proceed in the same way as usual. When you titrate the blank, subtract the results you get for the blank from the result you got from the original sample. The difference is the free SO₂ corrected for acetic acid.

Checking Indicator: The indicator is light sensitive and should be stored in the dark. Even so, it will deteriorate after 3-6 months, so it should be checked frequently to be sure it retains its potency. To check it, set up the peroxide/indicator mixture as described above. Add dilute acid until it turns deep purple, then dilute base until it turns bright green. *When the indicator will no longer achieve bright royal purple and bright emerald green*, but instead turns only bluish-purple and turquoise blue no matter how much dilute acid or base is added, the indicator is dead and must be replaced, and the result will be falsely low.

Standardizing NaOH: The NaOH solution will lose strength very quickly after it is opened because it reacts with air. The more dilute the solution, the faster it deteriorates, so this strength changes more quickly than 0.1N NaOH. Its strength should be tested at least once every week, oftener if using it regularly.

To standardize the NaOH, pour about 100ml of boiling distilled water into a beaker or flask. Add a squirt of phenolphthalein indicator, neutralize with dilute base to a very faint pink color. Add 5ml of HCl 0.01N titrate with sodium hydroxide to the same faint pink. Calculate the new normality (divide 0.05 by ml of NaOH used).

TROUBLESHOOTING

Sources of error (effects in parentheses):

peroxide too old – indicator turns bright purple with added to peroxide mixture. (results variable)

indicator too old – will not turn bright purple and green when dilute acid, then dilute base are added. (results low)

peroxide turns green as soon as indicator is added – bubbler tube has residual ions from previous titration; wash thoroughly and try again. Or, distilled/deionized water is slightly alkaline; change brands. (results low)

wine not bubbling vigorously – stopper connections not tight or air pressure insufficient. (results low)

peroxide not bubbling vigorously – same as above.

peroxide turns green when bubbling commences – no free SO₂ in wine.

endpoint not distinct – usually from using drugstore H₂O₂. (results low)

peroxide goes bright green with first drop of NaOH – very low free SO₂, or using too strong a normality of NaOH. (result OK or not titratable)

NaOH is no longer 0.01N – if NaOH has decayed, more NaOH will be needed for titration. (results too high)

high result in sparkling wine or wine with high V.A. – sparkling wine needs degassing, or a blank should be run. (results too high)