

LOW COST VACUUM FREEZE-DRYING

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The major cost of most vacuum freeze-drying systems is the vacuum pump. By replacing this expensive pump with a water aspirator, the cost of vacuum freeze-drying can be reduced by 90% or more. This will provide a system at a price the average lepidopterist can afford.

An excellent treatment of vacuum freeze-drying was done by Dominick (1972). He includes many theoretical and practical ideas which help in understanding this process. The following paper will deal with my modifications of the vacuum freeze-drying process, and complications which arise from these modifications.

Theory

Although a fine treatment of the theoretical aspects of vacuum freeze-drying was given by Dominick (1972), a review of the theory as it applies to this modified system is presented here.

At a fixed temperature and in a closed container, the vapor pressure caused by the evaporation of water (or ice) will be constant, regardless of the amount of vacuum initially applied (Dalton's Law of Partial Pressures). When a desiccating agent is placed in this container (but not in direct contact with the water or ice), the desiccant absorbs some of the water vapor. This causes more water (or ice) to evaporate in an attempt to maintain the partial pressure which is characteristic for that temperature. This means that the major driving force of dehydration is independent of the initial vacuum in the container. However, even though the driving force is independent of the initial vacuum, a container which is initially evacuated will evaporate a piece of ice faster than one with no vacuum. As Dominick explains it, "the more molecules of other gases present (air), the more difficult it is for the molecules evaporating from the water (or ice) to dissipate; then a state of quasi-saturation will be reached near the water (or ice) surface and further evaporation will cease." Actually, the evaporation will only slow down rather than cease. This means that vacuum freeze-drying can take place at any level of vacuum; however, the lower the pressure the faster the process. Table 1 compares the times necessary to dehydrate an average butterfly larva at different vacuum levels.

A water aspirator will attain a vacuum of around 15 mm Hg. My basic idea was to use this as a source of vacuum, in spite of the longer drying

TABLE 1. Comparative times for dehydrating average butterfly larva.

.001 mm Hg	15 mm Hg	760 mm Hg ¹
2 days	14 days	100 days

¹ Data at 760 mm Hg was obtained by Flaschka & Floyd (1969).

times, in order to reduce the cost of a vacuum source from over \$200 to less than \$15.

Equipment

At this point a physical description of an aspirator vacuum freeze-drying system is in order. The basic components can be seen in Fig. 1. In order to aid in the selection of suitable equipment, each of the four components of the system (aspirator, trap, valve, and desiccator jar) will be discussed.

The aspirator can be one of a number of different models. One of the main differences in aspirators is the vacuum they will produce with a given water pressure. Unfortunately, this data is hard to get from many suppliers. For my work I examined four different models, and chose an aspirator made of glass, by Kimax. It has a free air flow of 6.5 liters/min. with 40 lbs. of water pressure and is rated to pull down a 16 oz. jar to 16 mm Hg. in a maximum time of 1½ minutes. This glass aspirator requires a hose clamp, a hose, and an adapter for a kitchen faucet. These can be purchased from most hardware stores. Other aspirators are easier to connect since they come with threads on the aspirator and can be attached directly to the water faucet.

The next component is the trap. This insures that if something goes wrong (such as a sudden change in the water pressure), water will not back up into the desiccator jar. A vacuum trap is excellent, but a home-made trap is almost as good and is very inexpensive. A good home-made trap consists of a thick-walled jar with a two-hole rubber stopper inserted into it. Two pieces of glass or copper tubing are tightly inserted into the holes. One of the pieces of tubing should almost touch the bottom of the jar, and should be connected to the hose going to the aspirator. The other piece of tubing should only be inserted far enough to barely clear the rubber stopper, and should be connected to the hose going to the desiccator jar. In this way, water backing up will have to nearly fill the trap before it can back up into the desiccator, allowing time to shut off the valve to the desiccator jar. Do not reverse the order of the hose connections to the trap, or its purpose will be defeated.

The third component is the valve. This item is hard to find at a low

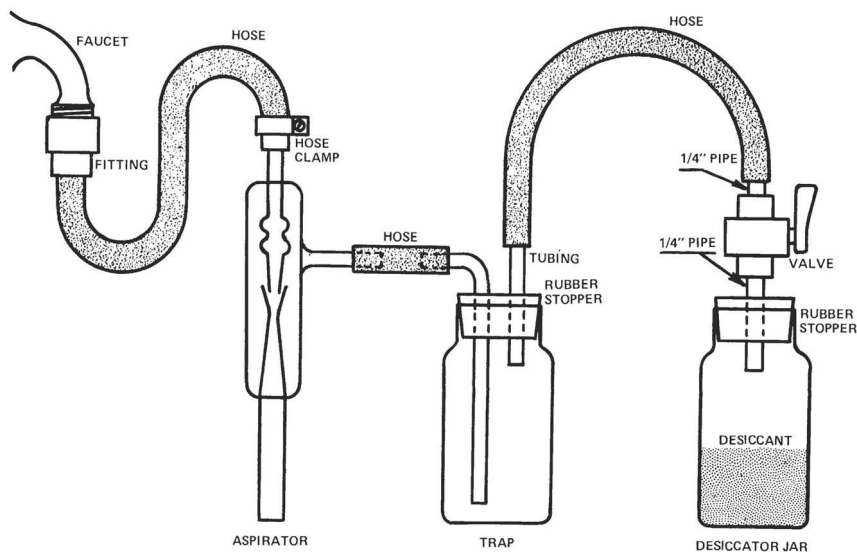


Fig. 1. Diagram of an aspirator vacuum freeze-drying system.

cost. The simplest choice is to buy a stopcock, but current costs for stopcocks are around \$8–\$15 (U.S.) each. Polyethylene one-way valves are inexpensive and in theory should work, but I have had numerous experiments ruined because they failed to operate properly. Several other types were tried with some success, but my best results have been obtained with equipment that can be bought at many hardware stores. Most good hardware stores carry various valves to fit $\frac{1}{4}$ " pipe. Obtain one with two female connections. Also purchase a three inch piece of $\frac{1}{4}$ " pipe. Cut this pipe in half, and attach half to each end of the valve. Bore a $\frac{1}{4}$ " hole through the rubber stopper you are going to use as a desiccator lid. As seen in Fig. 1, the pipe (which is attached to the valve) is inserted through the hole in the stopper. The valve, pipe, and stopper cost about \$2.50.

The last component is the desiccating jar. This jar should be thick-walled and fitted with a one-hole rubber stopper. I use the same type of jar as in the trap. These are thick-walled square 8 oz. bottles with a wide mouth, have a cap size of 43 mm, and take a #7 rubber stopper. One word of caution: before using any jar with a vacuum, test it by wrapping some towels around it and connecting it to the vacuum source. Even jars of the same brand should be checked individually to make sure

TABLE 2. Efficiency of desiccants.

Material	Residual Water per Liter of Air, 30.5°C (in mg)
$\text{CaCl}_2 \cdot \text{H}_2\text{O}$	1.5
$\text{Ba}(\text{ClO}_4)_2$, anhydrous	0.82
NaOH , sticks	0.80
CaCl_2 , anhydrous	0.36
$\text{Mg}(\text{ClO}_4)_2 \cdot 3\text{H}_2\text{O}$	0.03
Silica gel	0.03
KOH , sticks	0.014
Al_2O_3	0.005
CaSO_4 , anhydrous	0.005
CaO	0.003
$\text{Mg}(\text{ClO}_4)_2$, anhydrous	0.002
BaO	0.0007
P_2O_5	lower

they don't implode. Tape can be wrapped around the jar as a further safety measure.

A vacuum hose or a large diameter thick walled hose should be used in connecting the trap to both the aspirator and the desiccator jar. A washing machine hose is excellent for connecting the aspirator to the faucet.

One additional part of the system needs to be discussed. This is the desiccant. I use CaSO_4 which is impregnated with an indicator. The desiccant is blue when it is active, and turns red when it can no longer absorb water. This desiccant can be reactivated by placing it in a shallow dish in an oven set for 450°F. One brand name for this desiccant is Drierite (with indicator). Other desiccants can be used, but CaSO_4 , as seen from Table 2 (Bower, 1934), is one of the most efficient. The last point in this discussion of desiccants is to be sure not to overload the system. The 8 oz. jar should be no more than one third full of desiccant. This should be enough for any large butterfly larva or a couple of small ones. With very large larva such as those of Sphingidae or Saturniidae, be sure to start with freshly activated desiccant. A larger jar might be required on some of the largest larva. Remember that at best the CaSO_4 can absorb only about $\frac{1}{4}$ of its weight in water.

One piece of equipment not needed in this vacuum freeze-drying system is a large freezer. Several 8 oz. jars can be stored in a home refrigerator freezer and still leave plenty of room for frozen foods.

Procedure

The basic vacuum freeze-drying process consists of the following steps: (1) freezing the larva in the desiccating jar; (2) pulling a vacuum; (3) keeping this jar frozen until the specimens are dehydrated; (4) thawing and releasing the vacuum. Each of these will be discussed in turn.

The first step, freezing the larva, seems fairly simple, but there are a few cautions. Be sure that the jar is tightly sealed. Also, allow plenty of time for the larva to freeze. I usually wait until the next day before pulling a vacuum.

The second step, pulling the vacuum, involves several complications. The first major problem is to determine the amount of vacuum you are creating. The same aspirator will produce widely differing vacuum with different water pressures. One method of checking the vacuum is to connect the aspirator to a simple manometer. Since this involves an added expense (unless you happen to have one laying around the house), I use a less accurate but simpler method. I connect an empty 8 oz. desiccator jar, equipped with a valve, to the aspirator. The water is turned on for 40 seconds, and the valve is closed. Next, the hose is disconnected, the jar and valve are submerged in a pan of water, and the valve is now opened. Water will then rush into the jar. A good aspirator operating with the correct water pressure should create a vacuum sufficient to fill an 8 oz. jar to within $\frac{1}{4}$ oz. of being completely full. This is about 98% full if one is working with another size jar.

Because of changes in water pressure one should check the vacuum just prior to pulling down the desiccator jar. If the faucet is equipped with an aerator, this should be removed to prevent it from reducing the water pressure. Be sure to note the position of the cold water faucet handle when checking the vacuum, so that it can be returned to the same position when the desiccator jar is connected. It is best to work late at night when the water pressure is the highest. A most important aspect in evacuating the desiccator jar is speed. Have everything set up and tested before removing the jar from the freezer. Only 40 seconds are needed to evacuate the jar, and the total time out of the freezer should not exceed one minute.

The third step, keeping the jar in the freezer until the specimens are dehydrated, deserves a few comments. The length of time required is dependent upon numerous factors. The main variables are the temperature of the freezer, degree of vacuum, size of larve, and type of desiccant. In the system I have described, I have found one week insufficient but two weeks adequate for most types of butterfly larva. One last word of

TABLE 3. Comparative costs of freeze-drying systems (1974 U.S.A. prices).

Item	Conventional System	Aspirator System
Pump	\$200.00	\$12.00
Trap	none	0.50
2 Valves	none	5.00
2 Desiccators	50.00	1.00
Freezer	130.00	none
Hose & Connections	7.00	12.00
Total	\$387.00 (minimum)	\$30.50 (maximum)

caution: be sure the jar remains frozen—do not pack warm foods around it.

The last step is to remove the jar from the freezer after sufficient time has elapsed for dehydration. Before letting it thaw, test to be sure the desiccator jar still has a vacuum. I recommend moistening a finger tip and then with the finger over the valve, open and close the valve. If upon removing the finger, a distinct pop is heard, one can be sure the vacuum is still being held. If for some reason there is no vacuum present, one can return the jar to the freezer and repull the vacuum later. If the vacuum has to be repulled, replace the stopper and valve with dry ones just prior to repulling the vacuum. I have found this procedure to be satisfactory as long as the larvae are not allowed to thaw before they are completely dehydrated. Upon removing the jar from the freezer, do not release the vacuum for 24 hours. Experiments I have done indicate that the larvae should be sufficiently dehydrated at this point to prevent discoloration or spoilage, however the wait of 24 hours allows the dehydration to come to a completion. When releasing the vacuum, proceed slowly to avoid collapsing the specimens.

Additional Comments and Results

Since the main reason for using an aspirator vacuum freeze-drying system rather than a conventional system is cost, a comparison of costs is given in Table 3. The figures given are based on 1974 prices. Another benefit of this system is its portability. It weighs less than one pound and can be used anywhere there is running water and a refrigerator freezer.

I have successfully used this system on a wide variety of butterfly and moth larvae. A few suggestions can be made for avoiding poor results. Larvae that are preparing to moult or that are parasitized often do not turn out well. If the frozen larva becomes covered by desiccant,

such as by tilting the jar during the process of pulling the vacuum, the grains of desiccant can dent it. Leaves of larval foodplants can curl during dehydration, therefore when possible avoid putting leaves in the desiccator. Finally a little fading of some colors (especially greens) occurs during vacuum freeze-drying.

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