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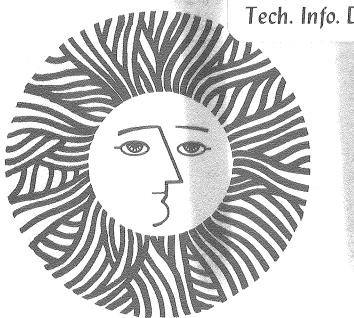
INSTRUCTIONS FOR OPERATING LBL FORMALDEHYDE SAMPLER

L.Z. Fanning, J.R. Allen, and R.R. Miksch

September 1981

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INSTRUCTIONS FOR OPERATING LBL FORMALDEHYDE SAMPLER

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CONTENTS OF THIS PACKAGE

- 1) One LBL Formaldehyde Sampler consisting of one small refrigerator and one pump box.
- Two sampling lines.A) One connecting line (to connect pump box to the refrigerator).
- 3) Three shipping containers.
- 4) "Blue Ice" to keep samples cold during shipping.

Keywords: aldehydes, formaldehyde, indoor air pollution, measurement, sampler

The LBL formaldehyde sampler consists of two parts: 1) a pump box and 2) a small refrigerator housing sampling bubblers. The pump box contains two pumps, a timer, a flow controller, an electrical cord, and a ten-foot piece of tubing to connect the refrigerator to the pump box. The small refrigerator contains four columns of bubbler sampling trains attached to a metal plate. Two sampling trains each are plumbed in parallel to two sampling ports on the back of the refrigerator. The two sampling lines supplied are to be attached to these ports to allow two locations to be sampled at once (usually one indoor and one outdoor). The refrigerator also contains a rack for holding bubbler tubes. (See parts list.)

In the sampling process, air is drawn through a sampling line attached to the fitting at the back of the refrigerator and into a primary bubbler containing a trapping solution. This trapping solution can be distilled water or an aqueous solution of some compound that reacts with formaldehyde. From this bubbler the air goes through a second bubbler containing the same trapping solution as the first bubbler. (To maintain sample integrity, all parts that the air sample contacts are made of Teflon, polypropylene, and stainless steel.) The air then goes into the third bubbler, which contains no liquid. This bubbler contains a hypodermic needle that serves as a flow-control orifice. The hypodermic needle, in conjunction with the flow controller in the pump box, ensures a constant air-flow rate. The refrigerator contains four columns of these sets of three bubblers.

After samples have been collected, the bubbler bottoms are detached and the contents of the first and second bubblers in each column are

poured together, capped, and labeled. The use of a refrigerated primary and secondary bubbler whose contents are combined at the end of a sampling period ensures 95% collection efficiency. After the bubbler tubes are capped and labeled, they are stored either in the rack supplied in the refrigerator or in one of the styrofoam shipping boxes with some frozen blue ice. LBL has found that formaldehyde samples collected in water degrade significantly in a matter of hours if they are not kept cool, whereas refrigerated samples remain stable for as long as a month.

SET-UP AND OPERATING PROCEDURE

UNPACKING

- each of the four lower corners of the crate and then lifting the crate off the sampler.
- 2. Note that the sampler has two parts: 1) The sampler itself, which is a refrigerator and 2) a pump box containing two pumps, a flow controller, a vacuum gauge, and a timer. The pump box also serves as the shipping container for an electrical cord and a ten-foot piece of polyethylene tubing (see Figures 1 and 2).
- 3. Plug the electrical cord at the back of the refrigerator into the female socket on the pump box marked "SAMPLE OUTLET" or "REFRIGERATOR OUTLET". Remove the electrical cord from the pump box. Plug the female end of this cord into the male socket on the pump box marked "POWER INLET" or "INLET POWER". Plug the male end of this cord into a grounded wall socket. (Note: One or both pumps may come on if the power switch on the timer happens to be in the "ON" position.)
- 4. Above the switch on the pump box marked "1 PUMP, 2 PUMPS", there is a Swagelok tube fitting. With the aid of a 9/16-inch open-end wrench or a small crescent wrench, attach the 1/4-inch polyethlene tube with fittings on both ends here and to the fitting marked "PUMP" on the refrigerator. The fittings should be lightly "wrench-tight" at both attachments, not "finger-tight" and not too

tight.

- 5. When the refrigerator and pump box are plugged in, open the refrigerator and place your hand on the bottom of the freezer unit to check that it is getting colder.
- 6. Allow the refrigerator to cool at least 15 minutes before sampling.

SETTING-UP SAMPLING TRAINS

- 1. There are four columns of three bubbler caps each, attached to the metal plate inside the refrigerator. They are labeled from left to right as columns 1, 2, 3, and 4. Generally, columns 1 and 2 are for sampling an indoor location (i.e., kitchen, living room, bedroom) and columns 3 and 4 are for sampling outdoor ambient air; however, check the labeling on the metal rack inside the refrigerator as this arrangement can vary. The first cap in each column is for the primary bubbler, the second cap is for the secondary bubbler, and the third cap is for the needle used as a flow-control orifice.
- Check the back cap in each column to be certain there is a needle in place. The needles supplied are precalibrated.
- 3. Place an empty bubbler tube over each needle orifice and push it firmly up onto the cap fitting. At this point there should be four bubblers across the back with no fluid in them. Make certain they are firmly attached.
- 4. If you are sampling for formaldehyde only, place 10 milliliters of sampling solution in each of eight bubbler tubes. If you are going to use LBL's pararosaniline method of analysis, the solution will be

distilled water.

- 5. If you are sampling with two solutions, (for example, water for formaldehyde and MBTH for total aldehydes) take two tubes of one solution and attach them to the first and second caps in column 1. Then take two tubes of the other solution and attach them to the first and second caps in column 2. Repeat with rows 3 and 4 (i.e., place the same solution in column 3 as in column 1; the same solution in column 4 as in column 2). Make sure you have the same solution in the primary and secondary bubblers in each column.
- 6. Attach indoor and outdoor sampling lines to the ports marked "IN" or "INDOORS" and "OUT" or "OUTDOORS" at the rear of the refrigerator in one of the upper corners. Fifty feet (25 feet each) of 3/8-inch tubing has been provided for these lines. The indoor and outdoor sampling lines have been labeled inside the refrigerator as well. The sampler is plumbed such that replicate samples are taken from each of two sampling lines.

SAMPLING

- 1. On the front of the pump box is a switch labeled "l PUMP" and "2 PUMPS". Set the switch to "2 PUMPS" and leave it there. This sampler is supplied with various needles which give different flow rates. This sampler is supplied with # ______ needles providing flow rates of ______ standard liters per minute (slpm).
- 2. Set the timer in the pump box for the desired sampling time.
 Operating instructions for the timer are included at the end of this instruction manual. The timer is capable of a fifteen-hour sampling

time. (Sampling for 2 hours at 1 slpm gives a minimum detection limit of 50 ppb when samples are analyzed with pararosaniline.) Be certain that nothing is blocking the path of the timer hands as the timer will stop if the hands are stopped.

- Turn on the timer (upper right-hand corner of dial) to start sampling.
- 4. Be sure that the vacuum gauge in the pump box registers at least 6 inches (goes to the point on the dial that corresponds to 6). If it does not, there is a serious leak somewhere: Be certain that all bubblers are securely in place and that the "PUMP" line is tightly attached at both ends.
- 5. Verify that all the bubblers (the first two tubes in each column) are bubbling. If not, reseat the tubes and try again.

AFTER SAMPLING

1. The sampler will run for the amount of time set on the timer and then stop. It is not necessary to remove the tubes from the bubbler caps until you are ready to change them for a new sample or move the sampler. The tubes will be kept cold by the refrigerator and are quite stable in this position. When you are ready to move the sampler or change the tubes to sample again, take the first two bubblers in column 1 and combine them. Label the tube with all pertinent information (location of sample, date, time beginning and time ending) and cap it securely. The laboratory doing the analysis will also need to have a sampling tube filled with the solution you used. Place the water or other sampling solution into a tube as if

you were going to sample with it; then cap it, label it as a "BLANK", and send it along with each shipment. (See the section on "DATA SHEETS" for labeling instructions.)

2. Follow the same procedures for bubblers in columns 2, 3, and 4.

SHIPPING OF SAMPLES

- 1. Samples must be kept cool at all times. Either store the capped samples in the rack in the refrigerator door or place them in the styrofoam shipping boxes with the frozen blue ice provided.
- 2. Samples should be sent back to LBL or to the laboratory where they are to be analyzed within two weeks after they are collected. It is best to ship them in weekly or semi-weekly lots of up to 50 samples per lot. In addition to packing samples with frozen blue ice, ship by the fastest route available. (We have had good luck with Federal Express.) Data sheets should be included with the shipment.

SUMMARY GUIDELINES

SAMPLES

- 1. Keep the samples cool at all times. When they are not inside the refrigerator, they should be in a shipping container with frozen blue ice. (Ordinary ice will melt in a matter of hours in the insulated shipping containers damaging the labels used on the sample tubes.)
- 2. There are generally two sizes of blue ice available (6 1/2 x 3 1/2 x 1 1/4 inches and 7 x 6 3/4 x 1 1/4 inches). When shipping or storing the samples, at least four pieces of the smallest blue ice should be included in the styrofoam shipping container. If the larger size blue ice is used, three pieces should be included. Because the cold transfer from the blue ice will be from top to bottom, the shipping container should be packed with the blue ice on top of the samples. (Make certain that the blue ice is well frozen before using it for shipping.)
- 3. When labeling the samples, use waterproof ink only. Felt tip or fountain pens are inappropriate. Ball point pens are acceptable.
- 4. Make sure that the plastic caps on the bubbler tubes are secure to prevent leakage of sample contents.

REFRIGERATOR

 The bubblers should be checked to see that they are bubbling. If not, connections must be checked. All Swageloks (metal fittings in the sampling lines) should be just wrench-tight, not finger-tight and not over-tight.

- 2. Fluid may collect in the third bubbler containing the flow-control orifice. If this happens, empty it and place the tube back on the line again. Under some conditions (e.g., very cold, very dry weather and a very long sampling time) water will evaporate. In this case, use a larger sampling volume, perhaps 15 or 20 milliliters instead of the 10 suggested. (We have not encountered such a problem yet, but the possibility does exist.)
- 3. If you are sampling for short times and going from site to site, you should keep blue ice in the sampler to keep the samples cool.
- 4. All sampling line used to bring the sample to the refrigerator must be either Teflon or polyethylene with an outside diameter of 3/8-inches.
- 5. If there appears to be no bubbling from the fourth column of bubblers while the first three are bubbling properly, check to see whether the line passing into the second bubbler on that column is frozen. (This line is closest to the freezing unit of the refrigerator.)

DATA SHEETS

We have found that using a "data sheet" is the most convenient way of labeling the sample tubes and providing the necessary information for each set of samples.

The bubbler bodies, or tubes themselves, should be labeled in simple, easy-to-read terms that can be reproduced on the data sheet. An example of such a label is shown below.

Sample Label

ALDEHYDE: A FORMALDEHYDE: F	DATE ON/ OFF	INDOOR: I OUTDOOR:O	(HOUR & MIN.) TIME ON/OFF (A/PM)
A	3/26/81 3/27/81	I	4:45 P.M. 11:35 A.M.
		:	

Below is a sample data sheet which should correspond to the label.

ALDEHYDE DATA SHEET

TUBE	LOCATION	DATE ON/ OFF	TIME ON/OFF	FLOW RATE (slpm)	REMARKS:
A 3 In	House #6 (Grayson) (near kitchen, Door)	11/6/80 11/7/80	8:00 A.M. 6:00 P.M.	1.3	Electricity off for 3 hours (about) so time of sample suspicious, Refrig. Was still cold.
F 16 Out	House #1 (Smith)	12/5/80 12/6/80	10:00 A.M. 9:00 P.M.	1.0	

PARTS LIST FOR CONSTRUCTION OF FORMALDEHYDE SAMPLER

PART	CATALOG #	SUPPLIER		
SAMPLER				
2.5 ft ³ refrigerator	34H 7606N	Sears' Roebuck Contact Sales		
100 ml. polypropylene round centrifuge tubes (bubbler bodies)	VWR# 60817-540 (Nalge PP-3111- 0032)	Van Waters & Rogers P.O. Box 3200 San Francisco, CA 94119		
Closures for centrifuge tubes (caps)	Bel Art Cat #478 (F19920)	American Hospital Corp. Scientific Products 255 Caspian Way Sunnyvale, CA 94086		
Teflon Tubing .	Continuous	Port Plastics 1047 N. Fair Oaks Avenue Sunnyvale, CA 94086		
PUMP BOX				
Air Cadet Vacuum, Pressure Pump	7530-40	Cole Parmer Instrument Co. 7425 North Oak Park Avenue Chicago, IL. 60649		
Flow Controller	Model #63BU	Moore Products 1122 B Street Hayward, CA		
15-hour Interval Timer	8635	Curtis Matheson 470 Valley Drive Brisbane,CA 94005		
SHIPPING CONTAINERS				
Fiberboard Insulated Mailers	Model #314	Polyfoam Packers Co. 6415 North California Ave. Chicago, IL. 60645		
Blue Ice	1034	Divajex 1557 Red Hill Ave. Tustin, CA 92680		
MISCELLANEOUS				
Cooling Fan	All items listed under "Miscellaneous" were available as stores items at Lawrence Berkeley Laboratory but can be obtained at any scientific supply house			
Pressure Gauge				

PROBLEMS:

If you experience any problems or if any questions arise, please call one of the following persons at the Lawrence Berkeley Laboratory:

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Richard Allen	(415)	486-4167
Kristin Geisling	(415)	486-6072
Buck Koonce	(415)	486-4061

(If using FTS-federal telecommunications system, please replace the 486-prefix with 451-)

Address: Lawrence Berkeley Laboratory

Building 70, Room 201 #1 Cyclotron Road Berkeley, CA. 94720

OPERATING INSTRUCTIONS FOR TIMER

TIMER FACE AND HANDS

On the face of the timer is an outer ring of black numerals (0 through 14) enumerated in a clockwise direction, and an inner ring of red numerals enumerated in a counter-clockwise direction. The black numerals are used to set the timer to run for a specific time (interval timing). The red numerals are used to indicate elapsed time. The three black marks between each numeral serve to divide the hour into fifteen-minute intervals. One hand has a "hooked" or "right-angle-shaped" end as opposed to the other hand which is straight, and has a red tip. When the timer is in operation, the hand with the hooked end-the slower-moving of the two-will make one complete revolution of the dial face in fifteen hours. The other hand is a "sweep" hand and will make one complete revolution of the dial face in fifteen minutes. For operating the pumps through the timer, the toggle switch in the upper right-hand corner of the timer face must be in the "ON" position.

The adjustable buzzer in the lower right-hand corner of the timer face notifies the operator that the time interval has elapsed; "HI" indicates the loudest setting; softer settings can be obtained by moving the knob toward the "OFF" position.

SETTING TIMER INTERVALS, EXAMPLE:

As noted, the outer circle of large black numbers are used to set the timing interval. To set the timer for 2 hours, 45 minutes, proceed as follows:

- 1. With the power switch off, rotate the slow-hand (hooked end) clock-wise until it rests on the third 15-minute graduation beyond the numeral 2.
- 2. If the fast hand (red-painted tip) is not already set on the numeral 0, use the large plastic knob in the center of the timer face to rotate it to the 0 position.
- 3. Turn power switch to "ON" and timing will begin.

Time intervals are limited to a minimum of 15 minutes and a maximum of 15 hours. When air samples must be taken in excess of 15 hours, both hands should be set at the zero position and the outlet selector switch (discussed below) moved to the right. This setting will allow the pumps to run independently of the timer.

WHITE AND BLACK RECEPTACLES

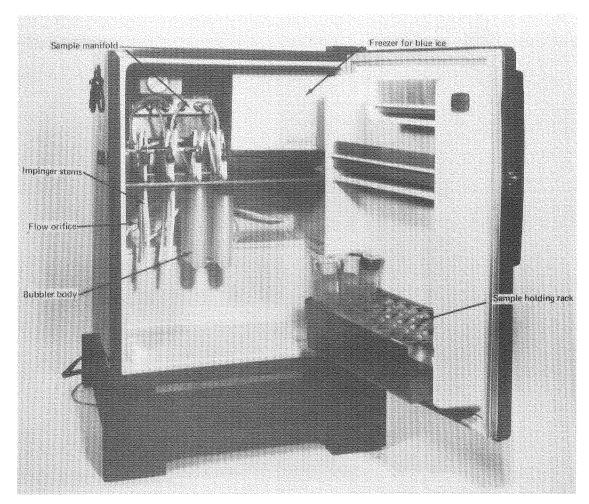
These receptacles are located on the left side of the timer case.

The pumps are usually plugged into the white receptacle since their positioning next to the timer case obstructs access to the black receptacle. (For this reason, the black receptacle will never be used).

The white receptacle is controlled by the outlet selector switch located in the upper left-hand corner of the timer face. The power switch in the upper right-hand corner must be in the "ON" position for the outlet selector switch to work. Pictographs on either side of the outlet selector switch show a black "X" across the white square and a white "X" across the black square, both in the upper (timer at zero) and lower (timer running) portions of the pictograph; the "X" indicates that

no power is available at that particular receptacle. When the hands are at zero, moving the outlet selector switch to the left will result in neither outlet (white or black) having power; if switched to the right, only the white receptacle will have power. When the timer is running, moving the outlet selector switch to the right will result in the black receptacle having power; if switched to the left, both receptacles will have power.

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XBB805-6058A

Figure 1. Formaldehyde/Aldehyde sampler.

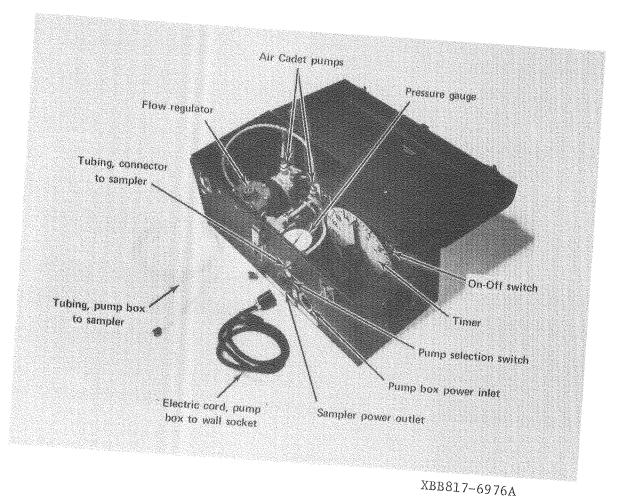


Figure 2. Sampler pump box with connections.

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