

PESTICIDES RELEASED FROM BURNING TREATED WOOD¹

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ABSTRACT.--Demands for firewood are high and rising, and pesticide-treated trees are often an obvious source. Wood treated with five herbicides (2,4-D, picloram, hexazinone, dicamba, and dichloroprop) and two insecticides (lindane and chlorpyrifos) were burned under controlled combustion conditions in a horizontal tube furnace to simulate the wide range of thermal environments to be found in domestic wood stoves and fireplaces. Over 95 percent pesticide decomposition occurred when the wood was burned under conditions of rapid (flaming) combustion. Under slow burning (smoldering) conditions, the amounts released were found to depend on the properties of the pesticides. The implications of these findings in terms of the suitability of using treated wood as firewood are discussed.

INTRODUCTION

From earliest times, wood has been man's primary heating fuel. It fell from favor in the United States when oil and coal were cheap and plentiful, but its popularity here is returning. Many homeowners have invested in wood burning stoves, furnaces, and fireplaces, and many public buildings are now heated fully or in part by wood burning. Demands for firewood are high and rising, and pesticide-treated trees are often an obvious source. For example, hardwoods killed by herbicide injection and beetle-killed trees sprayed with insecticide may be selected for firewood.

What happens if this wood is burned in domestic wood stoves and fireplaces? Do the pesticides decompose or are they released into the home or the neighborhood? Does the amount released pose an unreasonable risk to humans? Although there have been comprehensive summaries on the environmental fate and impacts of forest pesticides (e.g. Ghassemi et al. 1981) most of the reports deal with the fate of pesticides in water, soil, and nontarget organisms near the application site. Very little information is available on the fate of pesticides when the treated wood is burned in the open or in a home.

The purpose of the study described here was to begin to establish a database on amounts of pesticides carried over with combustion products when wood treated with organic pesticides is burned under controlled combustion conditions.

The research effort is part of a larger cooperative study with Forest Service Research Unit FS-SE-1153 at the University of Florida; the Pesticide Residue Laboratory, Cooperative Extension Service, University of Georgia; and Forest Pest Management, Region 8 in Atlanta, Georgia. The title of the parent study is "Fate of Herbicides and Insecticides in Trees Used for Firewood." It is sponsored and funded by the National Agricultural Pesticide Impact Assessment Program (NAPIAP).

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In the laboratory study reported here, the amount of pesticide released from burning wood treated with five herbicides and two insecticides in a horizontal tube furnace was measured. Pesticide concentrations in wood ranged from those commensurate with forest management practice to somewhat higher worst-case rates. In most cases, the carry-over amounts of pesticide were found to depend on the properties of the pesticide and the heating rate of the fuel.

MATERIALS AND METHODS

Combustion Procedures

A key factor in conducting this study was to select a combustion procedure that was reproducible and that would simulate the wide range of conditions under which pesticide-treated wood could be burned. It is well known, for example, that wood burns differently in fireplaces than in wood stoves, or under conditions of flaming and smoldering combustion. A horizontal tube furnace procedure was selected for this study. The tube furnace allows small fuel samples to be burned with controlled combustion and flow rates. It also permits quantitative sampling of combustion gases and particles generated during each run. As shown in the photographs (figures 1 and 2) and the line drawing (figure 3), the furnace is an open tube 3 inches in diameter and 2 feet long and can accommodate a 4-foot-long quartz tube. One-half of the quartz tube sits in the electrically heated zone, while the remainder lies outside in a cool (unheated) zone. A 1.5-inch-diameter, 30-inch-long ceramic combustion tube containing a small ceramic sample boat is placed inside the quartz chamber. The furnace can be heated to 1,000°C which is far hotter than necessary for the complete combustion of wood. It can be set to heat the sample boat at a slow rate (0 to 20°C/min) to represent slow burning conditions (e.g. logs on the edge of a fire in a slow burning wood stove). To attain a rapid heating rate (e.g. kindling wood burning in a well developed fireplace fire), the fuel sample can be rapidly inserted from the cool zone into a preheated furnace. Thus, the furnace allows a range of heating rates and temperatures to be applied to the fuel with a high degree of precision in setting and reproducing the test conditions.

When defining and describing the conditions under which woody fuels are normally burned, it is convenient to use the terms "flaming" and "smoldering" combustion. Flaming combustion is an efficient rapid burning process in which about 1 percent of the fuel is converted to a black, sooty visible smoke. Whereas smoldering combustion is an inefficient, slow-burning process in which about 10 percent of the fuel is converted to condensed tars appearing as grey or white smoke. In most wood fires both processes take place, but one is usually dominant, depending on the conditions of the fuel and the surrounding environment. Through preliminary experiments, we found we could simulate these conditions in the

tube furnace. For rapid burning (flaming), we would rapidly insert our fuel sample into the tube furnace preheated to 500°C. For slow burning (smoldering), we heated our fuel samples slowly (20°C/min) from ambient to 500°C.

Exothermic reactions in wood usually begin at temperatures around 320°C. Thus, in the microenvironment surrounding the fuel particles in the furnace, temperatures higher than the furnace setting can occur. Nevertheless, the furnace operating characteristics permit a much higher degree of process control and experimental reproducibility than normally obtained in open combustion experiments.

FUEL.--Although many types of wood are burned in wood stoves and fireplaces, it is likely that wood type has little influence on the problem under study. We picked one wood species, swamp chestnut oak (*Quercus michauxii*), which would be a likely selection for firewood and is readily available near the laboratory. The use of only one species helps reduce experimental variance. Branches were collected and oven-dried to constant weight at 95°C. The dried wood was then ground in a Wiley mill to pass through a 20-mesh screen. The resulting powdered wood was stored in a desiccator at room temperature until used in experimental tests.

PESTICIDE.--The sample unit for a test burn was a small quantity of fuel (0.5 g) treated with a designated level of pesticide. The amount of fuel required in a test burn depended mainly on the acceptable analytical resolution of 1 percent recovery of parent compound applied.

The seven organic pesticides used in this study are identified in Table 1. They are insecticides and herbicides commonly used in forestry applications. A concentrated stock solution of each pesticide was prepared using pesticide grade methanol and stored under refrigeration. An aliquot of each was further diluted with methanol to prepare a working solution for an accurate and precise application with a micropipette. Working solutions of the pesticides were also kept refrigerated.

Actual concentrations of the stock and working solutions of pesticides were determined by chemical analysis. Treatment concentrations in wood ranged from 240 ppm for picloram to 2500 ppm for 2,4-D, levels that would usually qualify as high level concentrations.

Sampling Procedures

For idealized stoichiometric conditions, 1 g of wood requires approximately 6 g of air for complete combustion. Under actual burning conditions, both pyrolytic and oxidative decomposition occurs with the resulting release of smoke gases and particles. For this study, an air flow of 2.8 l/min, supplied from compressed air

cylinders, was maintained through the furnace. This provided both combustion air and a means to sweep the combustion products out of the furnace and into the samplers.

We recognize that these flow conditions could result in an underventilated, fuel-rich condition during rapid exothermic reactions. However, we believe this simulates many of the conditions in which wood is normally burned as discussed earlier.

The hot combustion gases exiting from the furnace were diluted with 26 l/min of air circulated through a Neslab cooling apparatus. The condensing particulate matter and gases were then passed through the sampling system. The main function of the sampling system was to recover parent pesticides exhausted with combustion products. Physical properties of the pesticides indicated that at temperatures less than 100°C they could be condensed to a solid or liquid phase. Cooling the exhaust gases, therefore, allowed recovery of the pesticides and other products by filtration.

The system included a glass transfer tube and a Type A glass fiber filter followed by two polyurethane foam (PUF) plugs (figure 3). Yamasaki et al. (1982), Lewis and MacLeod (1982), and Lewis and Jackson (1982), comprehensively evaluated the use of polyurethane foam plugs for trapping condensable organic chemicals in air. They found that PUF plugs trap pesticides with a very high efficiency while allowing a high flow rate for the air being sampled. These findings were confirmed in our study as shown by consistent non-detectable levels in the second PUF. Prior to use, the PUF samples were Soxhlet-extracted with residue grade methanol distilled in glass. Total sampling of the exhaust gases was possible because of the high efficiency and high flow rates available with the sampling system. Total sampling avoids errors and uncertainties associated with partial or aliquot sampling techniques. Air flow through the filter and PUF's was maintained above 30 l/min by a vacuum pump located downstream from the filter. A thermocouple connected to a recorder was placed a few millimeters in front of the glass fiber filter to monitor the sampling temperature during a test run.

Test Procedures

SLOW HEATING.--The cooling apparatus was brought to operational temperature (-60°C), and air flow established. A powdered wood sample (20 mesh, approximately 500 mg) was accurately weighed in the ceramic boat. The pesticide working solution was then applied to the wood sample with a micropipette (typically 150 µl). Without further delay, the ceramic boat was placed into the center of a ceramic combustion tube inside the furnace. The sampling system and furnace controller were turned on. Furnace temperature and filter surface temperature were monitored with thermocouples. When the furnace reached a planned

temperature, 500°C, the heat controller and sampling system were turned off. Filter and PUF samples were removed and prepared for chemical analysis.

RAPID HEATING.--Procedures described above were followed, but the ceramic boat and tube were placed in the quartz tube section (cool zone) that extends outside the furnace. When furnace temperature reached 500°C, the ceramic boat was inserted into the furnace without disrupting the air flow by means of a copper push rod. At this time, the furnace controllers were turned off, but sampling continued for 3 minutes, which proved sufficient for combustion of the fuel sample.

After each test run, the sample train components were prepared for chemical analysis.

Analytical Procedures

The glass transfer tube, glass fiber filter, and the two PUF's from each test were stored at 0°C in individual containers until they could be transported to the lab for residue analysis (Cooperative Extension Service, University of Georgia). Residue extraction was usually initiated upon arrival at the lab. If overnight storage was required, samples were stored at 0°C.

EXTRACTION OF IONIC CHLORINATED HERBICIDES.--The pesticide collector (glass entrance tube, glass fiber filter or PUF's) was transferred to a 125 ml Erlenmeyer flask, and 80 ml of 1.0N NaOH in 10 percent NaCl was added. The flask was placed in an 85°C water bath for 30 minutes and then sonicated for 15 minutes. The extract was filtered through a glass fiber filter and transferred to a 250 ml separatory funnel. The extract was acidified with 6N H₂SO₄ to pH = 2. The acidic aqueous solution was partitioned three times against 100 ml diethyl ether. The ether extracts were combined over dried acidified Na₂SO₄, transferred to a boiling flask, and reduced to dryness using a rotary evaporator.

The residue was methylated with diazomethane and made up to a total volume of 5.0 ml with ethyl acetate for gas chromatographic (GC) analysis.

EXTRACTION OF NONIONIC INSECTICIDES AND HERBICIDES.--The collector was Soxhlet extracted with ethyl acetate. The ethyl acetate extract was concentrated on a rotary evaporator at 45°C to a small volume. Keeper solution (0.1 ml of 1 percent paraffin oil in hexane) was added and the sample reduced to dryness. The residue was made up to 5 ml with ethyl acetate and stored in a refrigerator for GC analysis.

GAS CHROMATOGRAPHY FOR IONIC CHLORINATED HERBICIDES.--GC analysis of the acidic herbicide methyl esters was performed on a Tracor Model 565

gas chromatograph equipped with a Hall 700A detector (halogen mode) and a 2 M x 2 mm I.D. glass column packed with 3 percent OV-101 on Chromosorb WHP. GC parameters were: detector, 325°C; injector, 250°C. The detector reactor temperature was set at 900°C with a hydrogen flow of 30 ml/min, a solvent flow setting of 4, and a solvent vent time of 0.75 min. The helium carrier gas flow was 35 ml/min. The GC column oven was temperature programmed from 150 to 235°C at a rate of 15°C/min.

GAS CHROMATOGRAPHY FOR NONIONIC INSECTICIDES AND HERBICIDES.--GC analysis of lindane was performed on a Tracor Model 222 gas chromatograph equipped with a NI⁶³ electron capture detector and a 2 M x 4 mm I.D. glass column packed with 3 percent OV-1 on Chromosorb WHP. The detector, inlet, and column temperatures were 350, 250, and 200°C, respectively. The carrier and purge gases were 5 percent methane/95 percent argon, and their flow rates were 45 ml/min and 10 ml/min, respectively.

Chlorpyrifos analysis was conducted using a Tracor Model 550 gas chromatograph equipped with a flame photometric detector and a glass column as above. The hydrogen and air flow were optimized for maximum response and the detector temperature was at 220°C. The carrier gas was nitrogen at a flow of 40 ml/min.

The hexazinone analysis was conducted on a Tracor Model 550 gas chromatograph equipped with a model 702 nitrogen-phosphorous detector. Operating conditions were: column, 3 percent OV-101 on Ultrabond 20 M (100/120 mesh); column temperature, programmed from 130-235°C at 12°C/min; carrier flow rate, 35 cc/min; detector temperature, 275°C.

QUALITY CONTROL.--Precision and accuracy of analytical methods were examined in laboratory quality control tests. Extractions and analytical procedures were applied to a set of fresh PUF's spiked with pesticides and to a set of glass fiber filters containing smoke condensate also spiked with pesticides. Recoveries and standard deviation for 50 µg/sample spiking levels are shown in Table 2. Large variations in recovery rates indicate difficulties that were probably caused by pesticide/sample matrix interaction either during the extraction or the chromatographic procedure. It should be noted that high blanks were not found for untreated PUF's or smoke laden filters. These analytical uncertainties were not a serious problem in achieving the objectives of this study, but would have to be resolved if more accurate recovery rates were needed to meet the needs of a more comprehensive parametric study.

RESULTS AND DISCUSSION

Parent pesticides recovered from the exhaust gases of burning treated wood in the tube furnace are given in Tables 3, 4, and 5. These values have not been adjusted for recovery rates shown in Table 2. Even if a correction factor had been applied, however, it would not have altered the overall findings of this study. The recovered amount of pesticide is reported as percent of that applied to the wood sample. The amount of pesticide applied is given as parts per million on a dry weight basis. Also reported in Tables 3, 4, and 5 are particulate emissions as a percent of dry fuel. These values are good indicators of combustion efficiency. Under slow combustion conditions (Table 3), particulate emissions of approximately 10 percent correspond to values reported for wood burning in wood stoves with closed dampers (Knight 1983). The particulate values under 3 percent found in rapid combustion simulations (Table 4), correspond to values of particulate production from wood stoves with open dampers (Knight 1983) and fireplaces (Dasch 1982). They also correspond to values found in the flaming (rapid combustion) and smoldering (slow combustion) of forest fuels such as burning pine needles (McMahon and Tsoukalas 1978). Thus, these particulate results are strong evidence that the combustion tube furnace is simulating the wide range of desired oxidative thermal environments likely to be found in domestic wood stoves and fireplaces.

Table 3 gives results of test runs in which samples were subjected to slow heating. The recovery of pesticides from these runs ranged from 0 for picloram to an average of 176 percent for dichloroprop. Analytical interference caused by chemicals in wood smoke and PUF's may explain the high values for dichloroprop. Recoveries of 2,4-D and dicamba were about 92 percent. Average percents of recovery for the other three pesticides were 43 for lindane, 28 for chlorpyrifos, and 11 for hexazinone. Although there was a wide range of recoveries among the pesticides with slow heating treatment, recoveries among replicate runs for each pesticide were consistent and in line with what we know about the physical properties of these chemicals. As expected, pesticide carryover was highly dependent on the physical properties of the pesticide when heating rate was slow. Low picloram recovery was expected because it is known to decompose at 215°C.

Table 4 gives results of test runs in which samples were inserted into the tube furnace preheated to 500°C. Pesticide recovery for these runs ranged from 0 for picloram and hexazinone to nearly 55 percent for one run of dicamba. Average recovery of 2,4-D was only 1.6 percent, which is markedly less than the recovery of 2,4-D when slow heating was used. Similarly, recoveries of chlorpyrifos, dichloroprop, and hexazinone were all reduced by rapid heating

compared to slow heating. Lindane, on the other hand, appeared unaffected by heating rate, and the dicamba runs gave mixed results. A closer look at the particulate emissions gives a clue to what happened. In several of the rapid heating runs, the high particulate yields (~10 percent) indicate that the wood sample actually burned slowly. Apparently, the addition of the pesticide to the wood suppressed the rapid combustion reaction at 500°C for some of the runs. In the other runs, particulate emission yields (~1 percent) indicate rapid combustion was achieved. Six additional test runs were made with wood treated with lindane and chlorpyrifos to help explain this phenomenon. For three runs, the samples were inserted into the furnace preheated to 500°C as before and for the other three, the furnace was set at 600°C. Results are given in Table 5. Apparently, 500°C is a borderline condition for rapid combustion, while 600°C consistently yielded low particulate emissions (~0.5 percent).

The implications of these experimental results in terms of domestic wood burning devices are:

1. Under conditions of rapid (flaming) combustion, most organic pesticides decompose readily, with higher temperatures causing more complete decomposition. Indeed, thermal decomposition is a well-known disposal technique for many waste organic compounds. With a well-ventilated, fully developed fire in a wood stove or fireplace (where the temperature can reach 800-1000°C), one can expect over 95 percent decomposition of the pesticides evaluated in this study. Thus, a good hot fire is recommended for the burning of all pesticide-treated forest fuels.

2. Wood treated with organic pesticides will release high percentages of parent pesticide when the sample is heated very slowly. This may occur in stoves and fireplace fires that are not fully developed. The percent of pesticide released will depend on the physical and thermal properties of the compound. Relatively stable compounds such as lindane and dicamba as well as compounds with significant vapor pressures can be expected to be released if the wood is heated very slowly.

3. Thermally unstable compounds such as picloram decompose readily under both slow and rapid combustion. However, decomposition of the parent compound does not necessarily eliminate problems; some pesticide decomposition products may be toxic.

4. Because of the uncertainty of ventilation and temperature in many domestic wood-burning devices, a cautious approach to the use of woods treated with organic pesticides is well advised. Thus, the indoor burning of this wood should be avoided unless it is known to have very low residue levels. For example, in some hardwoods killed by stem injection of herbicide, there is growing evidence that there are not significant

pesticide residues more than 1-2 feet from the point of injection (Bush et al. 1985³; Woolson 1985⁴). Thus, some of the wood available from stem-treated trees should be usable as firewood. As such findings are published, risk can be more completely assessed and guidelines published based on pesticide properties, method of application, amount applied, and planned use of the treated wood.

5. As a final note of caution, we point out that in this study we have been discussing the burning of unprocessed forest fuels treated with "organic" pesticides. The burning of finished or processed wood treated with a wood preservative is another matter. For example, wood treated with "inorganic" chromated copper arsenate (CCA) salts should not be burned because of the high concentrations of arsenic in the smoke and ashes (McMahon, Clements, and Bush 1985).

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TABLE 1.--Pesticides used in this study.

Common name	Chemical name	Forestry application
2,4-D ^a	2,4-dichlorophenoxyacetic acid as the triisopropanolamine salt	Herbicide
Picloram ^a	4-amino-3,5,6-trichloropicolinic acid as the triisopropanolamine salt	Herbicide
Lindane	1,2,3,4,5,6 hexachlorocyclohexane	Insecticide
Chlorpyrifos	0,0-diethyl-0-(3,5,6-trichloro-2-pyridyl) phosphorothioate	Insecticide
Hexazinone	3-cyclohexyl-6-(dimethylamino)-1-methyl-1,3,5-triazine-2,4(1H,3H)-dione	Herbicide
Dicamba	3,6-dichloro-o-anisic acid or 2-methoxy-3,6-dichlorobenzoic acid	Herbicide
Dichloroprop	2-(2,4-dichlorophenoxy) propionic acid	Herbicide

^afrom a Tordon 101R mixture

TABLE 2.--Pesticide recovery from spiked polyurethane foam (PUF) and filter samples containing smoke condensate.^a

Pesticide	PUF	Filter
	-----% Recovery-----	
2,4-D	92 (5) ^b	134 (11)
Picloram	111 (10)	102 (5)
Lindane	89 (9)	96 (5)
Chlorpyrifos	101 (3)	108 (8)
Hexazinone	117 (3)	127 (1)
Dicamba	111 (2)	97 (5)
Dichloroprop	126 (13)	138 (8)

^aspiking level 50 µg/sample
^bmean (+ S.D.)

TABLE 3.--Products from heating pesticide treated wood samples from ambient to 500°C (20°C/Min) in a horizontal tube furnace (slow heating).

Pesticide	Application rate ppm	Pesticide recovered %	Particulate emissions % ^a
2,4-D	2579 (79) ^b	92 (15)	11.2 (0.5)
Picloram	243 (8)	0 ^c	11.2 (0.5)
Lindane	381 (6)	43 (8.2)	10.8 (0.5)
Chlorpyrifos	504 (7)	28 (4.9)	10.8 (0.5)
Hexazinone	415 (5)	11 (2.3)	9.2 (2.0)
Dicamba	720 (26)	92 (5.1) ^d	10.8 (0.2)
Dichloroprop	400 (14)	176 (8.0) ^d	10.8 (0.2)

^apercent of dry fuel

^bmean (+ S.D.), n=3

^c98 ± 4 percent was recovered as picloram decomposition product 2,3,5-trichloro-4-aminopyridine (4A-TCP)

^dThis high recovery reflects an enhanced gas chromatographic response to dichloroprop in the presence of smoke condensate.

TABLE 4.--Products from burning pesticide treated wood samples inserted into a horizontal tube furnace preheated to 500°C (rapid heating).

Pesticide	Application rate ppm	Pesticide recovered %	Particulate emissions % ^a
2,4-D	2,586 (17) ^b	1.6 (1.3)	1.3 (0.4)
Picloram	243 (6.3)	0 ^c	1.3 (0.4)
Lindane	383 (4.1)	41 (4.0) ^d	10.4 (1.0) ^d
Chlorpyrifos	508 (5.7)	4.2 (3.4)	10.4 (1.0) ^d
Hexazinone	416 (11)	0	0.7 (0.4)
Dicamba	723 (5)	32 (30) ^d	3.4 (4.8) ^d
Dichloroprop	406 (10)	6.5 (1.6)	3.4 (4.8) ^d

^a percent of dry fuel

^b mean (+ S.D.), n=3

^c 64 ± 23 percent was recovered as picloram decomposition product 2,3,5-trichloro-4-aminopyridine (4A-TCP)

^d These high values are explained in the text and do not occur when the furnace is heated to 600°C (See Table 5)

TABLE 5.--A comparison of pesticide recoveries from burning treated wood samples inserted into a horizontal tube furnace at 500°C and 600°C.

Pesticide and furnace temperature	Application rate ppm	Pesticide recovered %	Particulate emissions % ^a
<u>Lindane</u>			
500°C	280 (9.2) ^b	76 (29)	7.1 (4.0)
600°C	273 (9.6)	0	0.46 (0.10)
<u>Chlorpyrifos</u>			
500°C	514 (17)	2.5 (4.4)	7.1 (4.0)
600°C	501 (18)	0	0.46 (0.10)

^a percent of dry fuel

^b mean (+ S.D.), n = 3

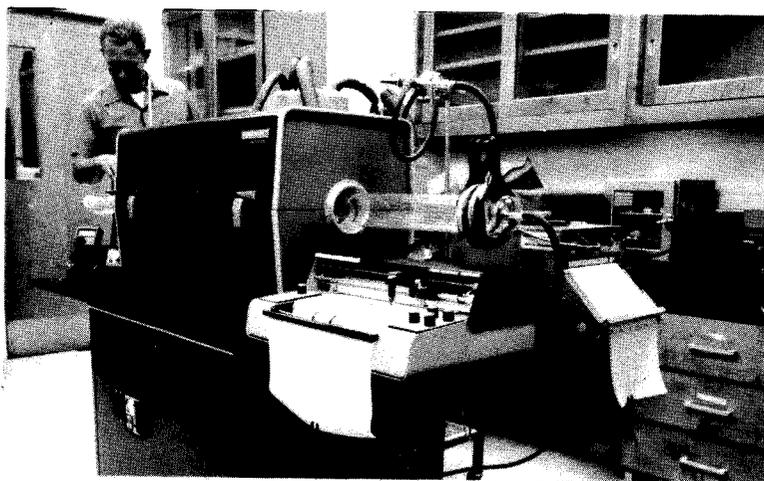


FIGURE 1.--Overall view of horizontal combustion tube furnace and associated equipment.

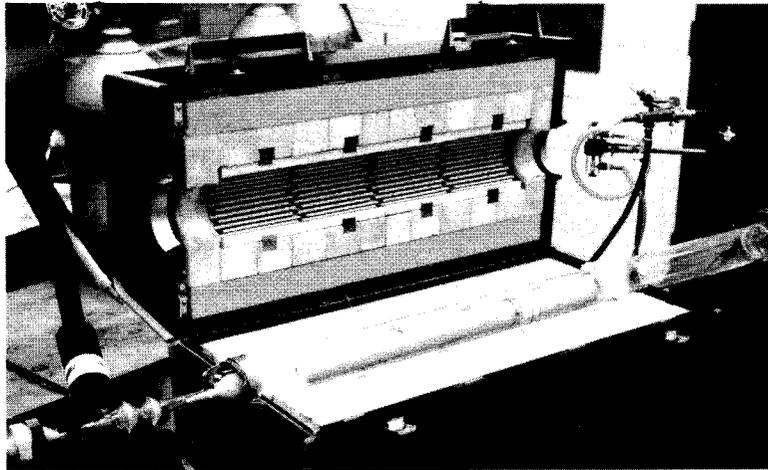


FIGURE 2.--Close-up view of quartz chamber and combustion tube in furnace. Air flow moves from right to left into glass transfer tube and filter sampling systems.

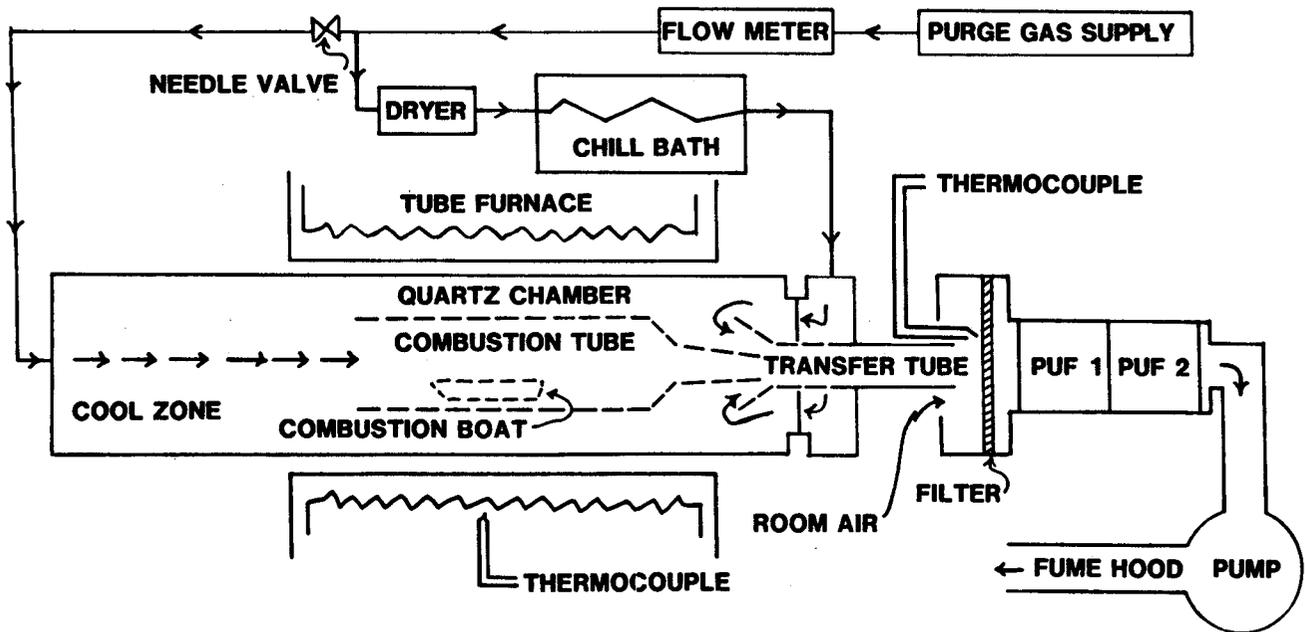


FIGURE 3.--Schematic drawing of horizontal combustion tube furnace and associated sampling systems (not to scale).