

# NATIONAL BUREAU OF STANDARDS REPORT

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## PREPARATION, SAMPLING AND ANALYSIS OF COMBUSTION PRODUCTS OF AIRCRAFT FINISH MATERIALS

By

J.J. Loftus, E. E. Hughes, D. Gross, A.F. Robertson



U. S. DEPARTMENT OF COMMERCE  
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ABSTRACT

This report describes three techniques which were used in burning samples of aircraft finish materials with the objective of analysis of the resulting decomposition products. In spite of the fact that the methods differed significantly, varying from pyrolysis in a closed vessel to burning in the open, and the fact that the techniques of sampling were varied with the objective of offsetting adsorption problems, the results of mass spectrometric analysis of the products were not greatly different. No halogen compounds were detected in the vinyls known to contain them. It was concluded that the methods used were inadequate to provide detailed information on the composition of the gases released during burning of the materials tested.

1. INTRODUCTION

In connection with a study of methods for more adequately limiting the flammability of aircraft interior finish and construction materials an attempt was made to identify and measure the concentration of decomposition products. Schriesheim (1) had previously made somewhat similar studies by pyrolyzing specimens at temperatures between 250 and 550°C in a closed vessel and analyzing most of the combustion products by means of a mass spectrometer. In his experiments the oxygen concentration in the combustion products was frequently reduced below the five percent level. Since active burning of materials in a self supporting manner appears unlikely under such conditions it was theorized that the decomposition products resulting from pyrolysis experiments of this type might not be most representative of those likely to result from actual fire situations. Because of this three different methods of producing the decomposition products were used. The plan was to observe the influence of varying degrees of confinement of specimen during burning on the pyrolysis products.



The work was undertaken as an analytical or test study rather than as a research investigation. This report describes the sampling techniques used and reports on the results of the analysis.

## 2. EXPERIMENTAL EQUIPMENT AND METHOD

Three degrees of confinement of burning situations were explored:

1. Pyrolysis in a closed though not gas tight heated tube furnace as described in ASTM E 136-59T (2) at 550°C (s)
2. Combustion of the specimen by direct flame impingement in a closed combustion chamber. (B)
3. Collection of a gas sample during burning of the specimen in the radiant panel flame spread test Int. Fed. Std. 00136b (RP)

The equipment and experimental procedure used with each of these will be described in turn.

### 2.1 Heated Tube Furnace

Samples of the combustion gases were collected during the burning of the test materials in an available, electrically-heated tube, furnace (2). Some modifications were made to the furnace to adapt it for this purpose. The principal change was the removal of the inner concentric refractory tube to allow for a tube diameter opening of 4 inches, large enough to accomodate a sampling test assembly. This assembly was made up of a removable furnace lid 5 1/2 inches in diameter which served to support a wire mesh specimen basket at a depth of 6 inches into the tube furnace, and a ST 12/30 taper joint fused to a 1/2 inch I.D. pyrex tube. This tube was positioned to extend a depth of 2 1/2 inches into the furnace and a distance of 3 1/4 inches above the wire basket. For observation purposes the lid was provided with a 1 inch diameter opening, fitted with a mica window.

Figure (3) shows the complete gas sampling assembly used with the furnace. A 100 mm gas drying tube with ST 12/30 pyrex taper joints attached at both stop cock side arms was used to collect samples for transfer to the mass spectrometer for analysis. One arm was fitted into the ST 12/30 pyrex taper joint in the furnace lid and the other connected by rubber tubing to a 250 ml Erlenmeyer filtering flask. A three way stopcock, a mercury manometer and a vacuum pump completed the sampling apparatus.

The furnace was preheated and set to maintain a temperature of 550°C as indicated by a thermocouple located near the center of the furnace. This temperature was sufficiently high to ignite all specimens within 5 minutes and was selected to duplicate a temperature condition used by Schriesheim (1). No air flow was supplied to the furnace.

With the lower stopcock open and the upper closed on the sampling tube the entire sampling system was evacuated through the 250 ml Erlenmeyer flask under a vacuum measured by a displacement of about 13.5 inches of mercury of the manometer. A 0.5 gram specimen of the material to be tested was placed in the wire mesh basket and the furnace lid positioned snugly over the furnace opening. Upon the ignition of the test material (usually within 2 minutes) as evidenced by a burst of flame and the evolution of smoke, the gas tube stopcocks were opened and closed in a single rapid motion during which time a sufficient gas sample was collected in the sampling tube under vacuum.

## 2.2 Closed Combustion Chamber

This device shown in Figure (1) comprised a completely closed system within which the specimen could be burned. Its main features included the combustion chamber, ignition burner, adjustable specimen holder, air and gas metering equipment and the gas sampling tube. In use, air and gas flows were adjusted to a level found to produce stable combustion conditions (0.003-0.004 cu ft/min gas and 0.2 cu ft/min air). The burner was lit and a one half gram sample of the specimen was placed in a silica dish on the sample support and the equipment assembled with the sample tube cocks open.

The specimen was then raised into a position so that the burner flame, which was about 1 1/4 inches in length, impinged on it serving to ignite it. The burner was fastened in this position and gases were allowed to continually escape through the sampling tube. After operation in this manner for about 10 minutes and before flaming from the specimen had stopped, the outer cock and then the inner cock of the sampling tube were closed.

This method of burning the specimen and collected gas samples was used with the hope that the interior surfaces of the equipment and sampling tube might become saturated and thus some of the gas products otherwise likely to be adsorbed on the walls might still remain in the gas or vapor phase.



### 2.3 Radiant Panel Gas Samples

The radiant panel flame spread test method (3) was modified by insertion of the gas sampling equipment shown in Figure (2) in the air suction line used for collecting smoke samples. With the objective of reducing adsorption difficulties expected with organic type hose, the line between the smoke sample holder and the sampling tube was replaced with stainless steel tubing.

The cocks were adjusted to permit bypassing the sampling tube and the test was run in this way until the flames reached the nine inch position or maximum smoke evolution rate had been achieved. The cocks were then adjusted to pass the gas through the sampling tube for the time period during which specimen flaming continued. This ranged from 0.6 min to over 2 minutes and the cocks were then again adjusted to permit bypassing the tube.

This technique was intended to provide a gas sample representative of those likely to be produced in open fires where considerable dilution with fresh air was likely.

### 3. Results

Two series of experiments were performed. In the first series, three materials, selected to represent a wide range of combustion products, and a blank were tested by each of the combustion methods previously described. The materials tested were a cotton fabric, a vinyl floor covering incorporating a foamed rubber pad, and a drapery material fabricated with cotton, mohair, and viscose fibers together with a metal foil. These were designated as specimens 1, 25, and 41 respectively.

These samples were first analyzed with the mass spectrograph directly without pretreatment (i.e. concentration). The analysis, Table 1, showed that only the components of air with increased carbon dioxide, and some carbon monoxide and in one case hydrogen were present. There was evidence of other compounds but their concentration was so low they could not be identified. An attempt was then made to concentrate the unidentified portion of several samples. The samples were cooled with liquid nitrogen, the air and other noncondensibles were pumped away and a spectrum was obtained of the residue after volatilization. Since water and carbon dioxide constituted the greater part of the condensible portion it was impossible to determine the presence of anything else without first removing them. The volatilized residue was then treated with a drying agent to remove water and with Ascarite to remove the carbon dioxide. The remaining sample after this treatment was too minute to allow identification of any compounds. Furthermore, the treatment prevented any estimate of the quantity of the unidentified compounds.



In general, the results for  $O_2$ , CO, and  $CO_2$  seem to indicate that the excess air increased from S to B to RP. This is to be expected, although the absence of this trend for Specimen No. 41 could not be explained unless an error occurred in either labeling the specimens or reporting the data. The rather unusual analysis reported under BLANK (RP) probably can be attributed to the fact that the combustion products of the radiant panel diluted with fresh air were being analyzed. The complete absence of any indication of halogens in specimen No. 25 for any of the sampling methods suggests that either the concentration was below the sensitivity of the spectrometer used or the sampling and transfer techniques were inadequate.

Because of the large quantities of air present in the samples it was decided to use the Heated Tube furnace for the next group of tests, with the objective of increasing concentration of decomposition products. In addition, no extraneous combustion products would be introduced in an electrically-heated furnace. This was done and the 14 specimens shown in Table 2 were pyrolyzed by this method. The one marked BLANK was a sample obtained from the heated furnace but without introducing any specimen. The samples were analyzed without treatment and the mass spectra obtained, Table 2, represented the entire gaseous phase in the sample tubes.

The major portion of each sample consisted of air, carbon dioxide, carbon monoxide, nitrogen in excess of that in air and argon in excess of that in air. The latter components are the result of the depletion of oxygen in part of the air due to the combustion process. In every case the above named components constituted 98% or more of the sample, with the remainder consisting of a complex mixture of organic compounds. The value in the column headed unidentified fraction is the percentage of the total ion currents at all mass numbers, which would not be ascribed to the identified compounds. It is not the actual concentration of the unidentified fraction but is probably within one order of magnitude of the actual concentration.

The mass numbers which contributed the major portion of the ion currents at the unidentified masses are those which represent ion fragments of unsaturated hydrocarbons containing from one to three carbon atoms. Some of the large remainders arise from pattern variation and other errors involved in the calculation of the identified portion. There is also evidence of the presence of aromatic compounds, principally benzene, at a concentration of about 2-5% of the unidentified fraction.

Because of the apparent inability of the mass spectrometer to detect halogens from materials including those containing poly (vinyl chloride) under the conditions of testing and sampling used, the very sensitive qualitative Beilstein test for halogens was performed on small samples of most of the specimens listed in Table 2. Table 3 shows the results of application of this test method. In addition, moist litmus paper was held in the smoke produced during performance of the Beilstein test, and it was found that with the single exception of specimen No. 4 all specimens produced an acid indication. Specimen 4 produced a basic indication while specimen 75 showed initial acid and then basic indication.

#### 4. DISCUSSION

There are problems associated with the collection and transfer of combustion gases for analysis by the mass spectrometer, which probably accounts for the lack of indication of certain toxic components which are presumably present. Although the products analyzed in producing Table 2 were collected using a closed system, maintained at high temperature, with no air supplied, equally severe conditions might occur during fires in aircraft compartments.

It was considered possible that certain compounds ( $\text{Cl}_2$ ,  $\text{HCl}$ , and oxides of nitrogen) which were not found in the gaseous phase may have been adsorbed on solid carbon particles or in tar aerosols. However, no analyses were made of the liquid (including tar) portions of the samples (some of which were colored) since it seemed that the information obtained would not justify the effort. It may be noted that during analysis of the vacuum pyrolysis of cellulose (4) the tar fraction was analyzed by prior heating in a furnace opening directly into the mass spectrometer ionization chamber. Other research studies employing gas chromatography (5,6) have also found it desirable or necessary to carry the combustion products directly into the analyzer without collection and handling.

Since such techniques are apparently necessary, it may not be profitable to consider the bottle collection of combustion gases from full scale field studies. Where direct analysis instruments (e.g. of the infrared absorption type for  $\text{CO}$  and  $\text{CO}_2$  and the magnetic susceptibility type for  $\text{O}_2$ ) are available for field use, their use is recommended. Care should be taken to keep the length of the sampling lines as short as possible. The use of colorimetric gas detecting tubes (7,8) for analysis of known or suspected toxic gases should be considered. Such tubes should also be placed as close as possible to the area being monitored to reduce the amount of adsorption along the surfaces of sampling tubes.



[9]

It has been suggested<sup>^</sup> that for the majority of fires and almost irrespective of the type of lining materials used, the principal toxic product would be carbon monoxide and that even if more lethal gases were produced, the volume of CO would be sufficiently great for it to constitute the major hazard. However, the lack of adequate information on the synergistic toxic effects of CO and very small quantities of other toxic products makes a complete analysis of combustion products highly desirable.



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Table 1 Three Methods of Decomposition Sampling  
Concentration of compound in mole percent

	Cotton #1*				Floor Covering #25				Drapery #41				Blank			
	S	B	RP	S	S	B	RP	S	S	B	RP	S	B	RP	S	RP
N <sub>2</sub>	72.5	77.6	74.4	81.1	80.8	74.4	77.6	77.8	80.4	78.3	76.2	75.4				
O <sub>2</sub>	9.3	10.0	16.3	14.0	14.0	16.3	10.8	20.1	14.5	19.8	20.5	18.4				
H <sub>2</sub> O	6.5	4.1	5.1	-	-	5.1	0.4	0.1	-	0.0	0.4	3.9				
CO <sub>2</sub>	9.7	7.4	2.7	3.0	4.0	2.7	7.7	0.6	4.2	0.9	0.3	1.5				
Ar	0.8	0.9	0.8	0.9	0.9	0.9	0.9	0.9	0.9	0.9	0.9	0.9				
CO	1.2	-	0.6	0.7	0.2	0.6	2.6	0.6	-	-	1.7	-				
H <sub>2</sub>	-	-	-	0.3	-	-	-	-	-	-	-	-				

S Heated Tube furnace pyrolysis; rapid sampling in evacuated tube.

B Enclosed burner; continuous sampling.

RP Radiant Panel; sampling during active flaming.

\* Materials may be identified as follows:

- # 1--Rug (UP) Wool backed by Cotton with Neoprene base
- #25--Laminate (F) Vinyl backed by Fiberglas on Foam pad (1/4")
- #41--Rug (UP) Nylon, Dacron

Table 2

## Products Identified by Mass Spectrometer-Composition in Moie Percent

Material		Composition								
No.	Type	Air	CO <sub>2</sub>	N <sub>2</sub>	CO	Ar	H <sub>2</sub> O	Unidenti- fied Resi- due		
1.	Rug (UP)	39.1	8.2	48.7	2.6	0.6	0.8	1.7		
4.	Fabric (UC)	35.9	13.4	38.4	3.3	0.4	8.4	1.1		
9.	Fabric (UC)	77.6	2.0	16.7	3.6	0.1	---	1.9		
11.	Sheet (R)	58.8	6.4	27.6	6.9	0.3	---	0.8		
16.	Fabric (UC)	46.2	6.4	44.8	1.9	0.3	0.5	2.0		
21.	Fabric (C)	70.0	2.4	24.0	3.2	0.4	---	1.9		
22.	Fabric (C)	70.6	2.5	23.6	2.9	0.3	---	1.7		
24.	Fabric (C)	84.9	1.7	11.5	1.8	0.1	---	0.6		
28.	Fabric (C)	82.8	2.2	13.3	1.6	0.2	---	0.3		
29.	Laminate (F)	58.7	3.8	33.1	3.9	0.4	---	1.6		
31	Sheet (R)	65.6	1.9	31.8	---	0.4	0.4	2.4		
34	Sheet (R)	65.5	2.2	28.4	3.4	0.3	---	2.3		
35	Sheet (SR)	71.7	2.3	20.3	5.4	0.3	---	1.8		
Blank	(no specimen)	94.5	---	5.4	---	0.01	.5	0.09		

Note: UP - Unpadded; C - Coated; UC - Uncoated; F - Flexible; R - Rigid; SR - Semi-rigid.



TABLE 3

List of Materials as Classified by Beilstein Test

Materials showing negative indication (no halogen):

1, 4, 11, 16, 65, 82, 88

Materials showing positive indication (halogen present)

21, 22, 24, 28, 29, 31, 34, 35,

51, 52, 53, 58, 59, 60, 61, 62,

63, 66, 69, 70, 74, 75, 85, 86, 87



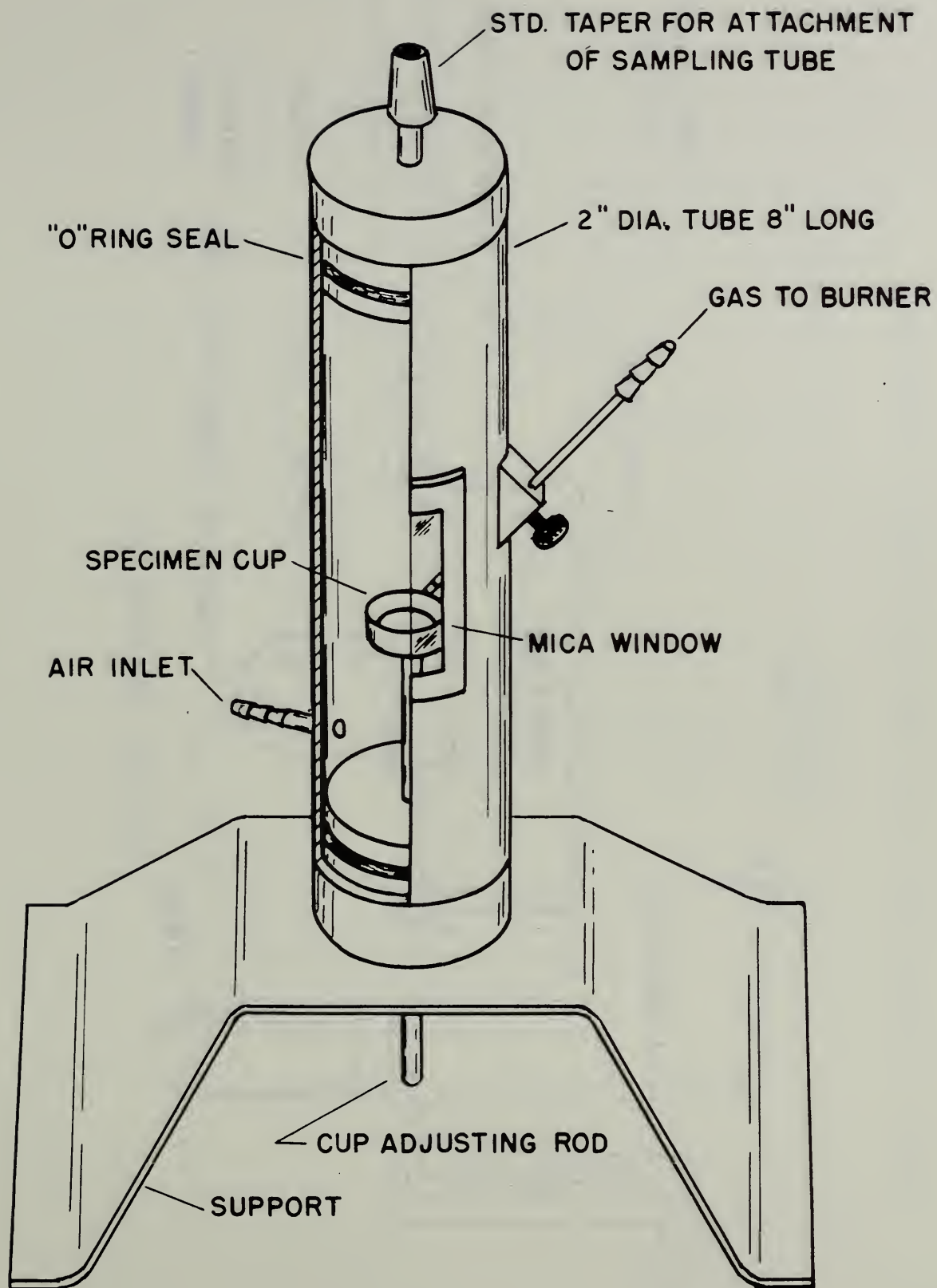


FIG. I- ENCLOSED BURNER



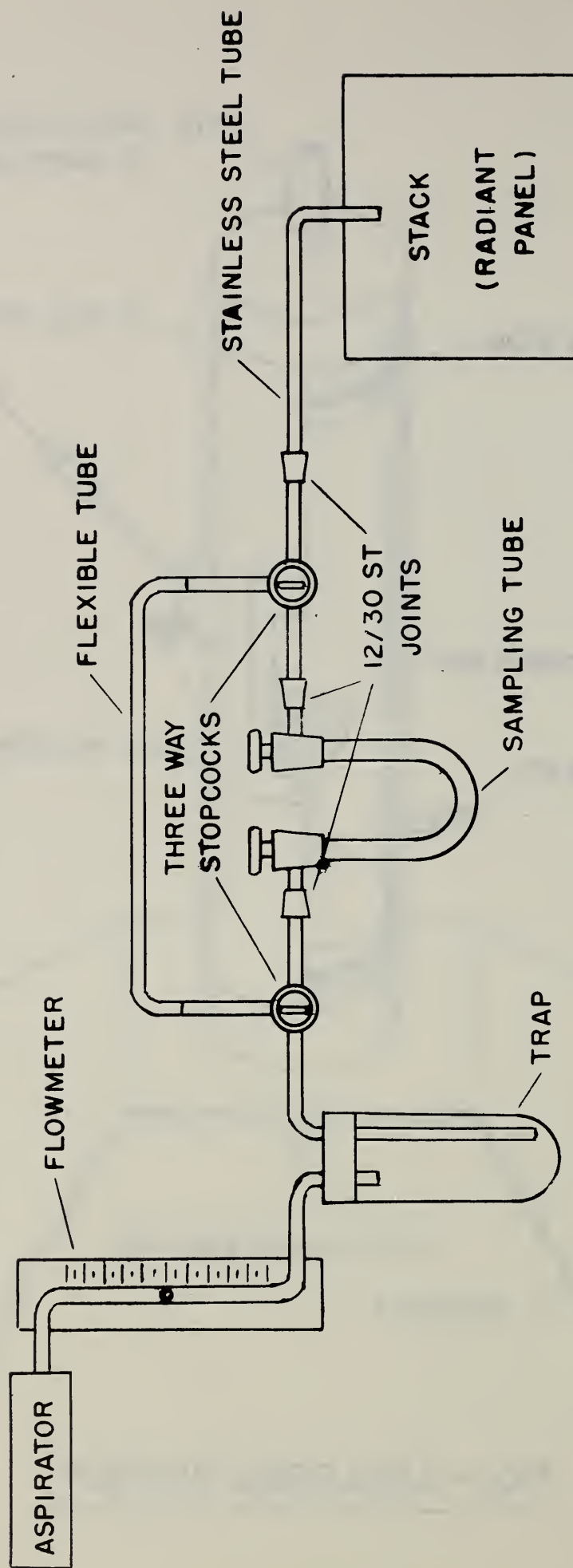


FIG. 2 - SAMPLING SYSTEM FOR RADIANT PANEL

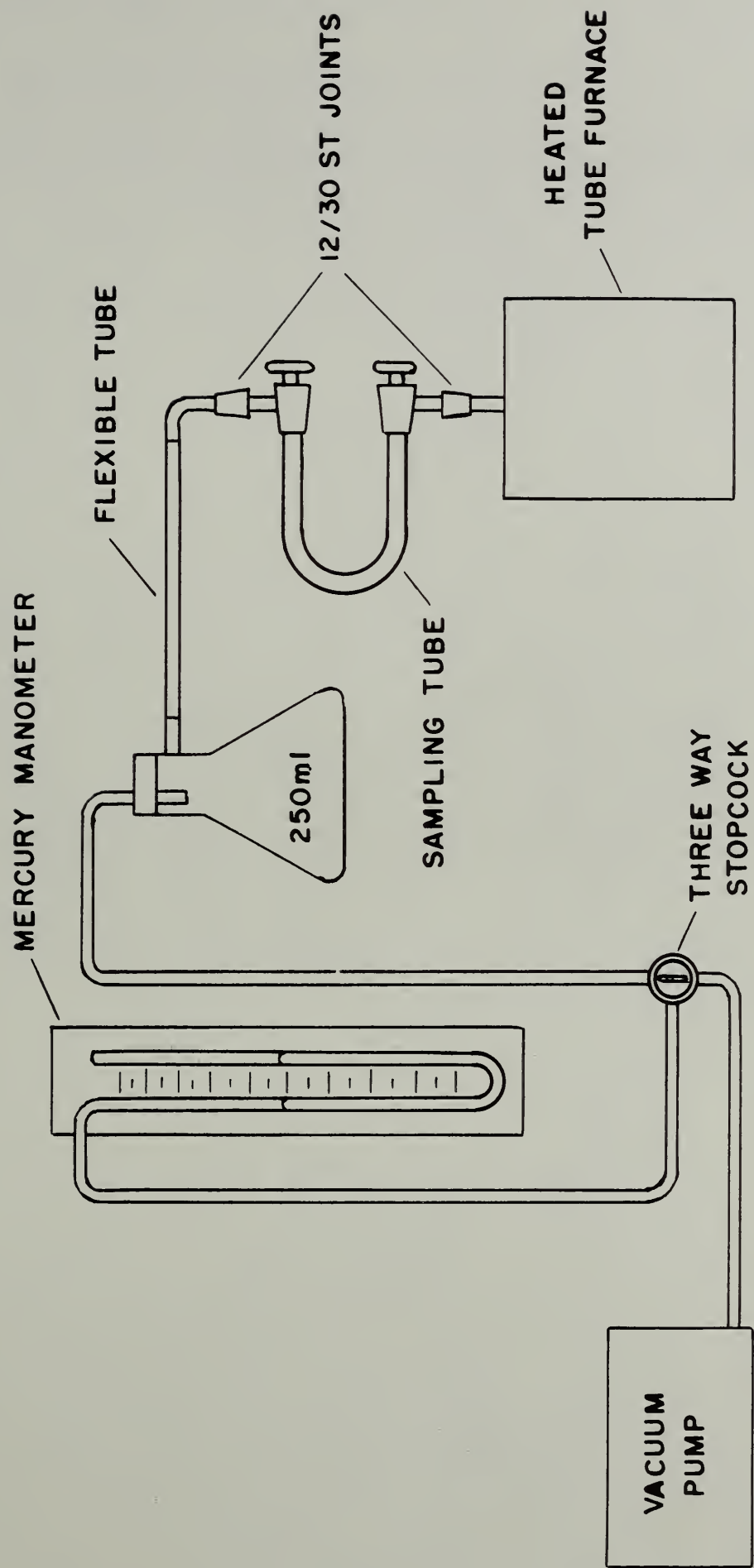


FIG. 3 - SAMPLING SYSTEM FOR TUBE FURNACE







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