

# Methods of Collection and Analysis of Toxic Gases from Large-Scale Fire Testing of Aircraft Interior Materials: Equipment/Systems Description

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## EXECUTIVE SUMMARY

The most recent methodology for the collection, identification, and quantification of pyrolysis gases from the combustion of aircraft interior material is outlined in this report.

## INTRODUCTION

### PURPOSE.

This report presents the methodology and techniques employed by the chemistry laboratory to collect, identify, and measure the toxic gases produced in the fire scenarios employed by the Federal Aviation Administration (FAA) Technical Center's Fire Safety Branch to study the potential contribution of aircraft interior materials to fire hazards. The chemistry group performs functions in support of groups within the Fire Safety Branch. Among these functions are the sampling and analysis of fire atmospheres from the C-133 test article. This report presents the methods presently employed.

### BACKGROUND.

A large number of investigators have been and are working in the field of toxic fire gas collection and analysis. There are areas of agreement and areas of controversy between investigators and published reports.

In the case of the controversy, one must appreciate the problems involved in the collection of samples, and in the handling and analytical methods employed to identify and quantify toxic-gas species. In addition, one must appreciate the chemical and physical nature of the mixture of gas species in the fire atmospheres. Fire atmosphere samples are taken when "hot," and in this state different species may condense, react with other species to form new compounds, or neutralize. Discrete samples must be handled in a way that insures their nature until they are analyzed. Analytical methods must be employed that minimize interferences. Discrete sampling in timed sequences result in a vastly different picture than that given by total sampling, when one reviews analytical results of a full-scale fire.

When it is available, reliable, proven, and within budget constrictions the Fire Safety Branch employs automatic analytical equipment. Oxygen (O<sub>2</sub>), carbon dioxide (CO<sub>2</sub>) and carbon monoxide (CO) are analyzed continuously by automatic equipment and a computer data station.

The remaining fire-gas atmosphere is sampled every 30 seconds in two categories, acid gases and organic gases. Different collection and different analytical techniques and methodologies are employed for these two gas categories (references 1, 2, 3, 4, and 5).

In some test programs, animals (rats) have been used to supply insurance that some severely toxic gas has not escaped identification outside of the targeted gas species expected from the composition of the aircraft interior materials evaluated.

## DISCUSSION

### ACID GASES COLLECTION.

In all instances, considerable care is undertaken to check and standardize the various equipment to insure that the data recorded represents the real effect

produced. Absorbtion tubes are used to collect acid gases in the fire tests. This sampling procedure was developed in 1976 (reference 2) at the Technical Center, formerly National Aviation Facilities Experimental Center (NAFEC), and is used currently. A modified version of the original tube has been developed (figure 1). This tube is 16 1/2 centimeters (cm) long with a 4-millimeter (mm) inside diameter (i.d.). It is made of stainless steel and is lined with glass. The tube is packed with 3-mm diameter glass beads to a depth of 14 cm, held in place with a rigid piece of TEFLON™. The beads are rinse coated with a one-molar sodium carbonate solution just prior to use. Excess solution is drawn from the tube by a syringe. A short (7.6 cm) length of 2 mm i.d. capillary tubing is joined to the inlet end of the tube. The tubes are then sealed with a plastic cap.

The tubes are mounted horizontally in an ice-water bath perpendicular to the expected smoke velocity vector (figure 2). The container is an aluminum box insulated with KAOWOOL™. The tubes are held in place with water-tight bulkhead fittings. The outside ends extend just beyond the insulation, and the caps are removed. The interior ends are attached to separate calibrated vacuum lines which pass through the bottom of the box and lead to the solenoid valve assembly (figure 3). These vacuum lines are insulated in a KAOWOOL sleeve. The box has a drain line for the removal of ice water after a test. The solenoid valve assembly is an array of 10 solenoid valves, remotely controlled by a 10-pole relay timer. The main vacuum line is connected to two solenoid valves each connected to a 3 1/2-liter-vacuum bottle. As one bottle is evacuated, the other draws a calibrated-measured (wet gas meter) fire atmosphere sample into and through a sequentially determined sampling tube. A thermocouple is mounted inside one of the two bottles to monitor bottle temperature during a test. The timer with ten positions sequentially opens solenoid valves joined to each sampling tube. The odd number tubes are connected to one vacuum bottle and the even number tubes are connected to the second vacuum bottle. This permits alternative evacuation of vacuum bottles and alternative sampling of tubes. The vacuum bottles are evacuated for 30 seconds and a sample is taken every 30 seconds. The vacuum drawn through the tubes is accomplished within 30 seconds. This means that the samples taken represent a discrete picture of the fire atmosphere at specific time intervals, every 30 seconds for a 5-minute test period.

These analyses are presented in time/concentration graphs, and although not continuous like those for O<sub>2</sub>, CO and CO<sub>2</sub>, can be viewed as a dynamic picture of the changing fire atmosphere. Four of these sampling assemblies are used in the C-133 test article. These sampling stations can be relocated for hazard pattern studies. Time/gas concentration profiles are produced for each station.

The acid gases form anion salts in the absorbtion tubes. These anion salt samples from the fire atmospheres are recovered for analysis by rinsing the tube with a 10-milliliter aliquot of 0.05 molar sodium carbonate solution dispensed by syringe. The syringe is adapted to the tube with a female SWAGELOK™ to luer connector. The rinse washings are collected in autosampler cuvettes for subsequent analysis by ion chromatography (reference 1).

#### ACID GAS ANALYSIS.

The acid gases, hydrogen fluoride (HF), hydrogen chloride (HCl), and hydrogen bromide (HBr) are identified and quantified by ion chromatography. Anion concentrations are measured with a Dionex Model 10 Ion Chromatograph. A silver/silver chloride potentiometric detector is used for chloride and bromide and a solid-state

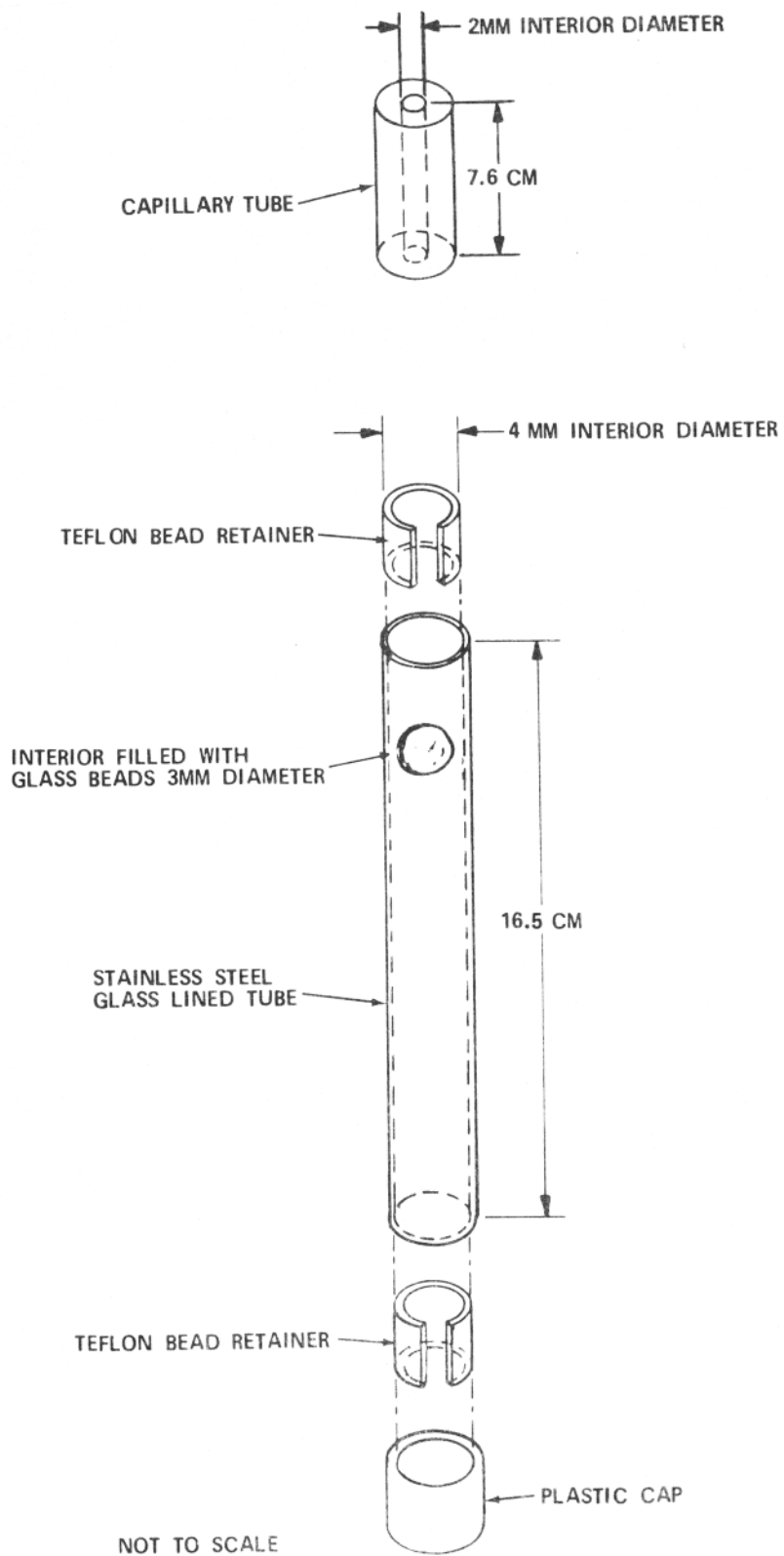


FIGURE 1. ACID GAS COLLECTION TUBE



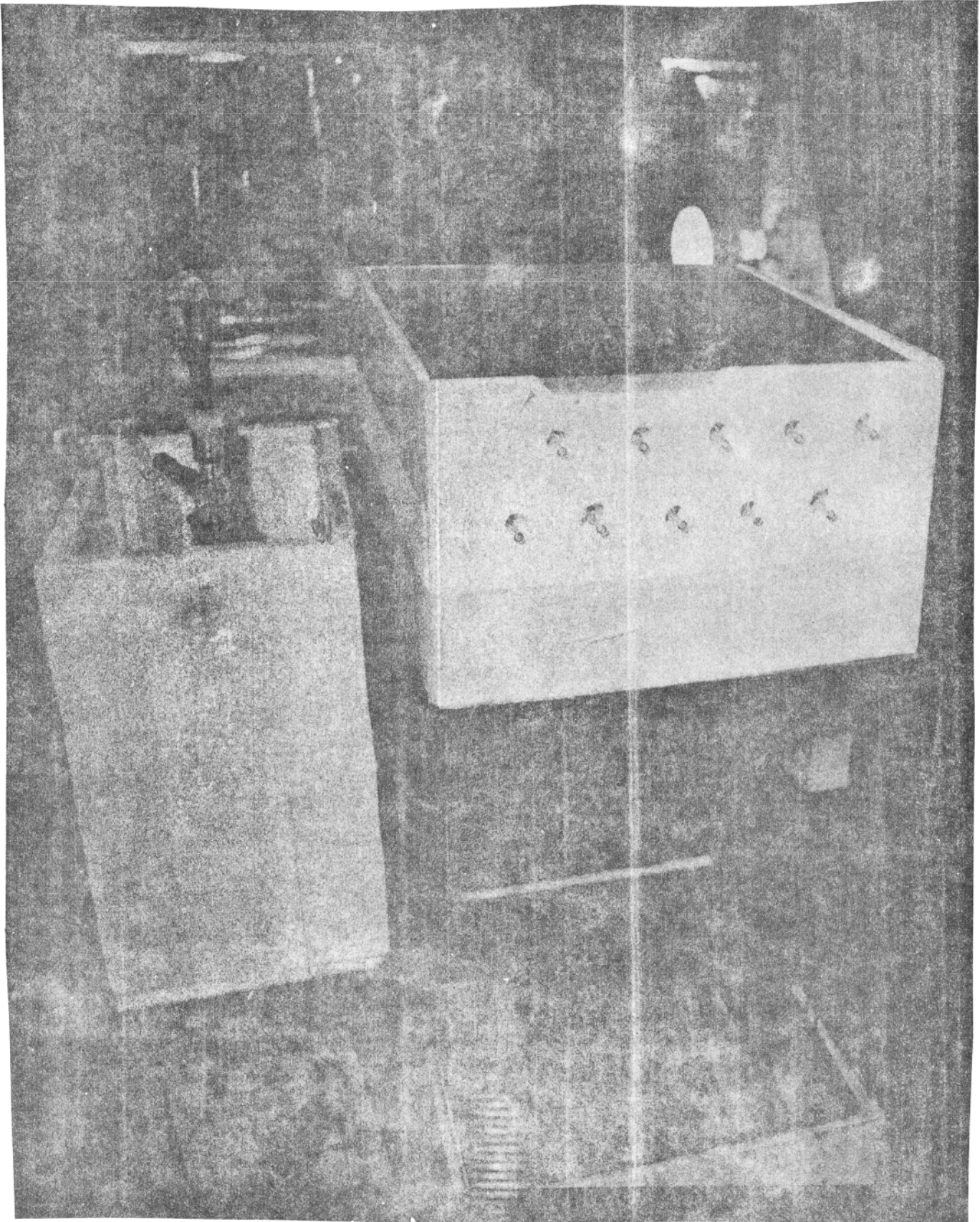


FIGURE 2. ACID GAS COLLECTION STATION

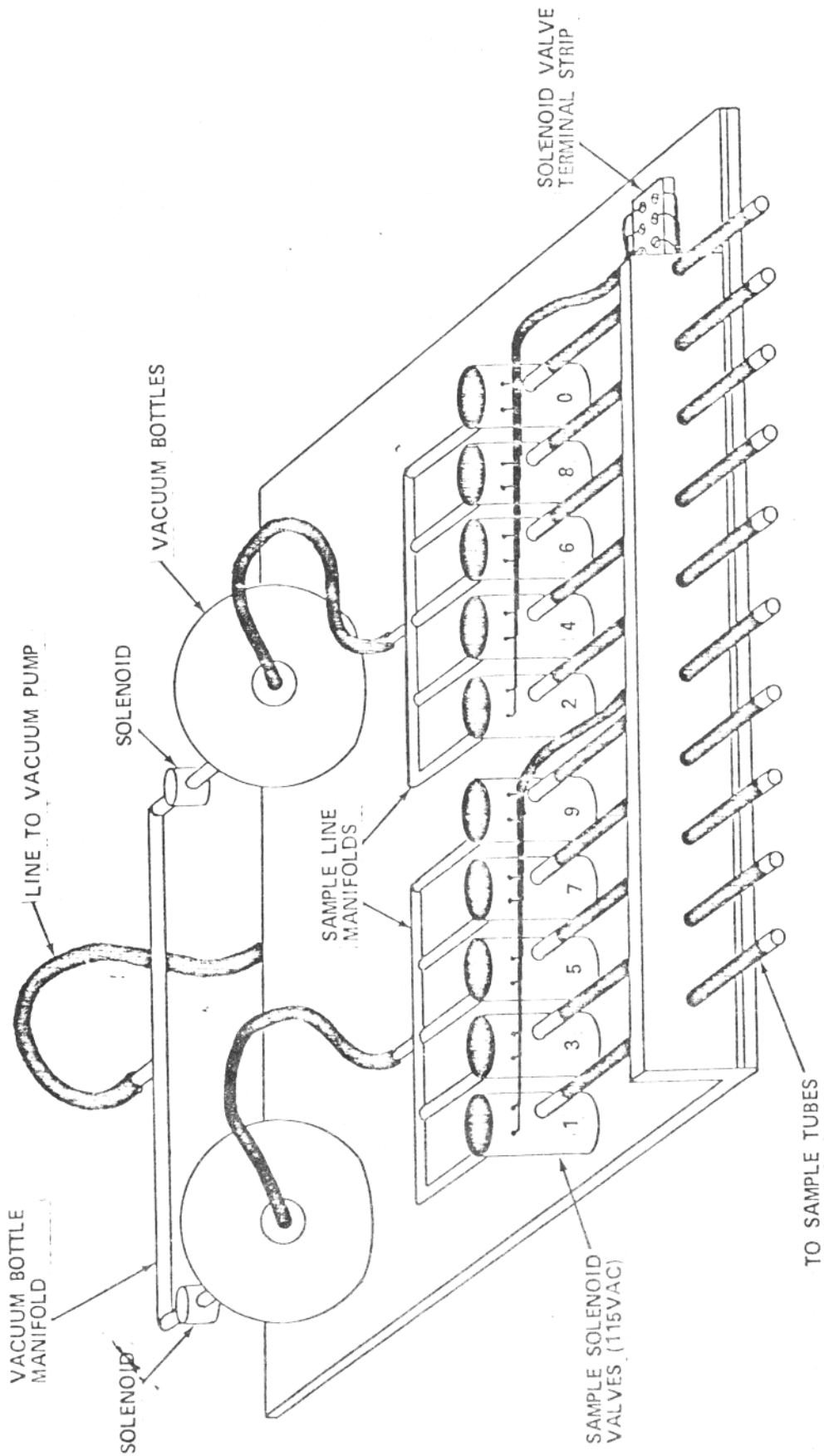


FIGURE 3. SOLENOID VALVE ASSEMBLY

fluoride ion selective electrode is used for fluoride. The millivolt signals from the detectors are amplified to fall into a 0- to 5-volt range. This is compatible with the analog-to-digital converters to the computer. A PDP 11/03 computer is used for data collection, calculation, and reduction. The reduced data is stored on a RL-01 disc. The PDP 11/03 is linked through a RS-232 interface to a Tektronix 4051 graphics terminal with a hard copy unit and a 4662 flat bed plotter. Software has been written to present and document the data in a variety of ways. Time/gas concentration graphic profiles are produced for each station and placed in the computer permanent file (reference 1).

#### ORGANIC GAS COLLECTION.

Absorbtion tubes are used to collect organic gas samples in the fire tests. This procedure was developed at the Technical Center and has been used subsequently (reference 1). The sample tube is glass-lined stainless steel. It is 12 centimeters long and is packed with a 30/60 mesh gas chromatograph grade porous polymer, TENAX™ absorbent, held in place with a wad of glass wool. The tubes are thermally desorbed in the gas chromatograph prior to use and are then sealed with PARAFILM and stored in a freezer. The sample tubes are frozen inside a water filled glass jacket, resembling a popsicle and then arranged in an automatically timed sampling station (figures 4 and 5). A vacuum timer system draws calibrated (Bubblemeter) samples of the fire atmosphere into and through the tubes, sequentially. CO<sub>2</sub> is discharged, posttest, in the station to cool the tubes and prevent HCN desorbtion. The tubes and sampling station are recovered immediately after the fire test. The tubes are removed from the station, sealed, and placed in a freezer for subsequent analysis. Three of those stations are used in the C133 test article (figures 6, 7, 8, 9 and 10). The timed samples represent eight discrete 30-second batch samples over a 4-minute fire test.

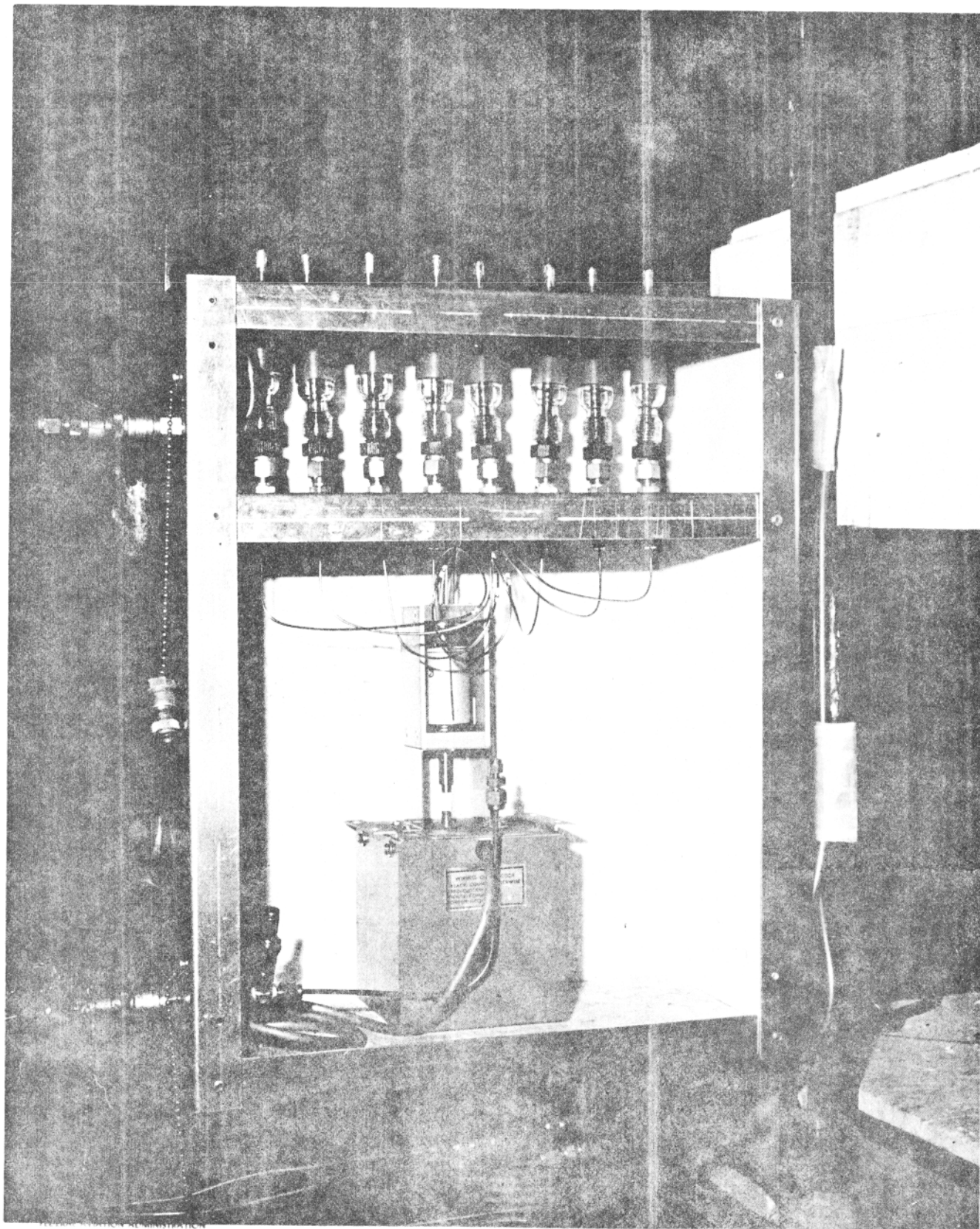
#### ORGANIC GAS ANALYSIS.

The organic gases specifically targeted for analysis are Hydrogen Cyanide (HCN) and Benzene. The absorbed organic gas sample tube is connected in a backflush mode to a PERKIN ELMER 3720 gas chromatograph equipped with a flame ionization and a nitrogen phosphorous detector. The gas chromatograph is calibrated weekly from samples whose concentration is determined monthly by redundant test methodology (reference 1). Data is acquired on a PERKIN ELMER SIGMA 10B minicomputer data station. A software program for the collection of raw data, calculation of quantities of identified gases and documentation is started. This information is then transferred to the PDP 11/03 computer for Time/gas concentration plot preparation and permanent filing (reference 1).

#### SUMMARY

The gas collection-sampling, identification, and quantification methods described here have been used for 2 1/2 years in a large number of full- and small-scale aircraft fire and fire extinguisher tests. It is believed that these analyses are representative and descriptive of the real-fire event.

Reports have been published and distributed to the Fire Community describing this methodology in the FAA Fire Safety Programs (references 1, 2, 3, 4, and 5).



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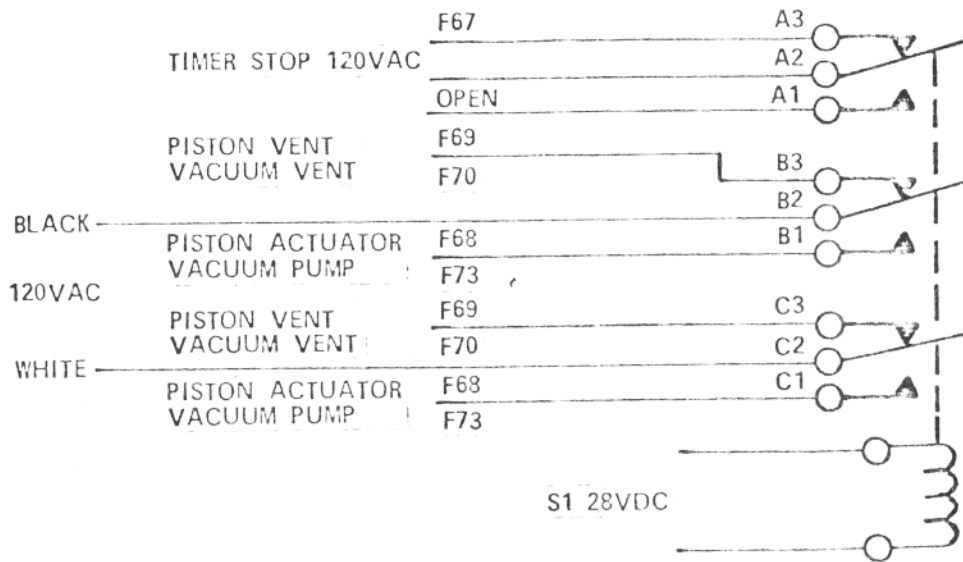
FIGURE 4. ORGANIC GAS COLLECTION STATION

RELAY 1 - ON PRETEST RELAY 1 - ON PRETEST - OFF END TEST

SOLENOID FUNCTION

INPUT VOLTAGES

WIRE NO.



RELAY 2 - ON - TEST START - OFF END TEST OPEN

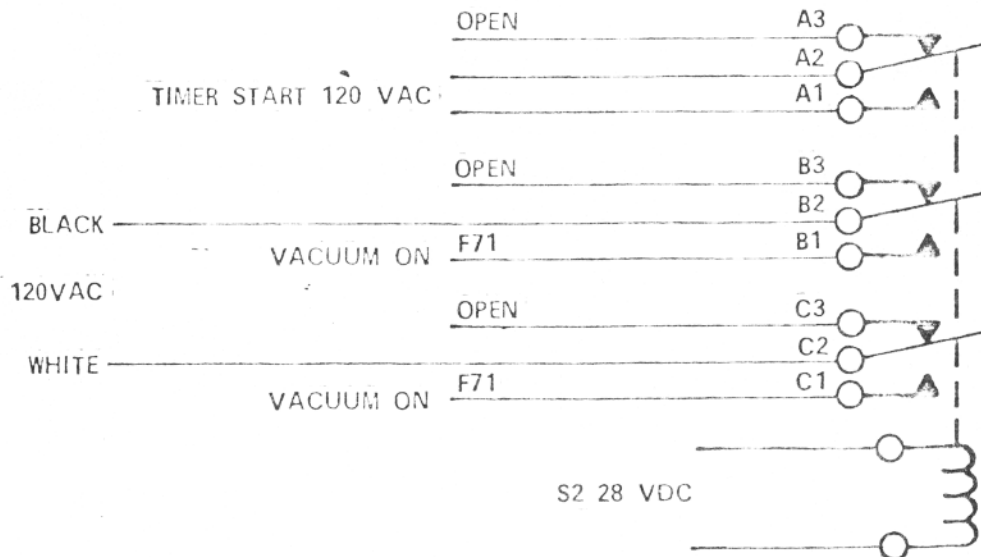


FIGURE 5. ORGANIC GAS TIMER ELECTRIC SEQUENCER

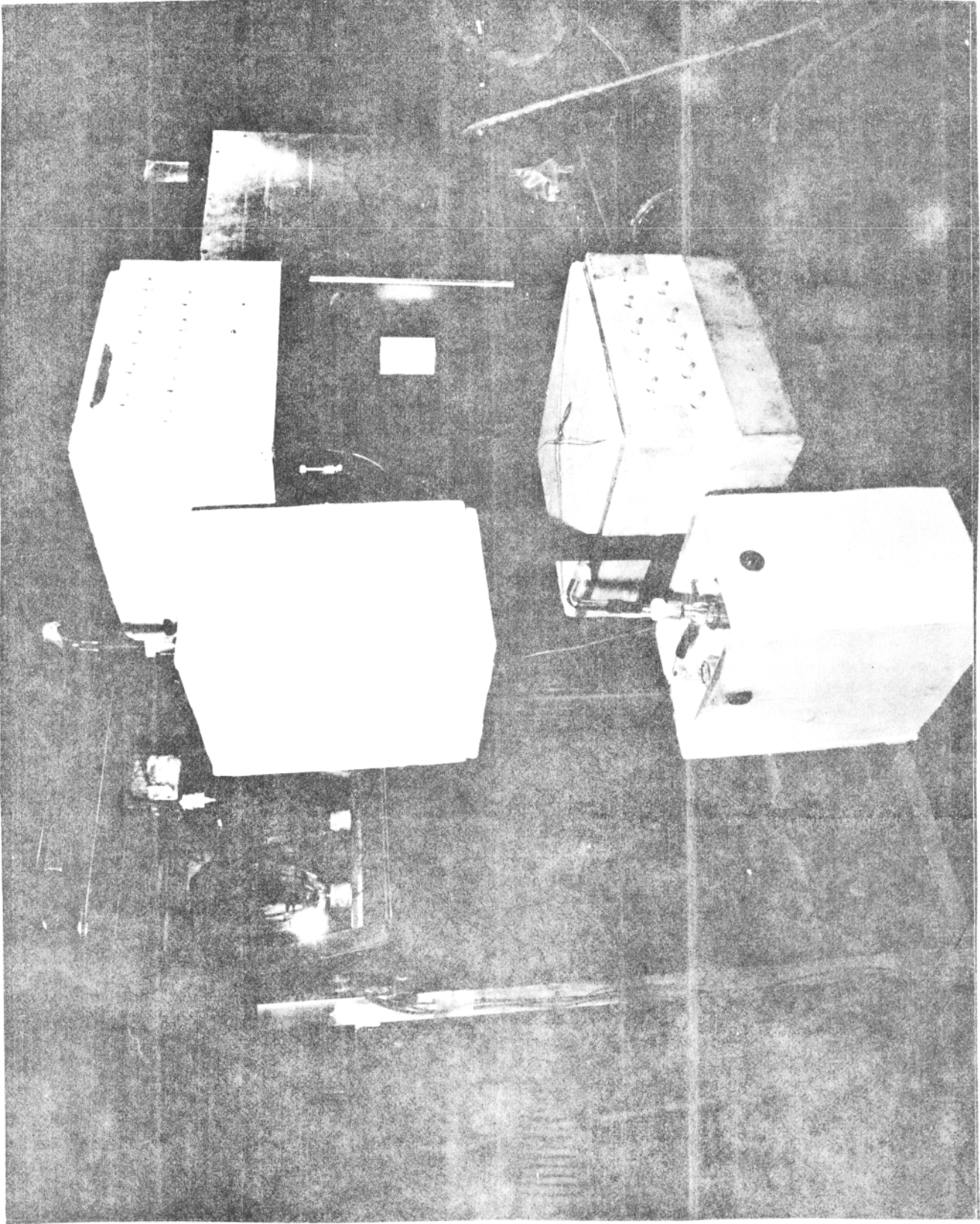


FIGURE 6. TOXIC GAS COLLECTION STATIONS (FRONT VIEW)

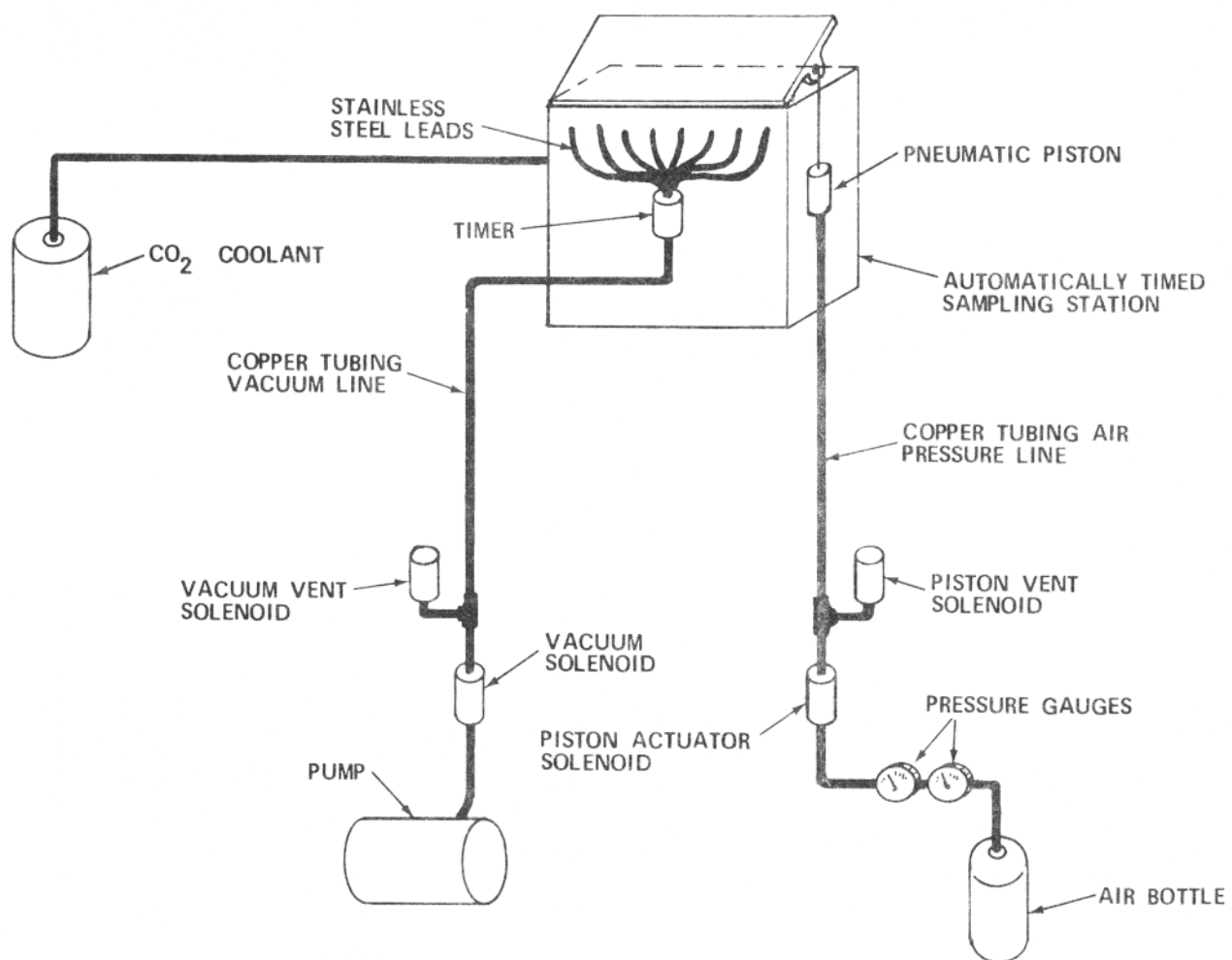


FIGURE 7. ORGANIC GAS COLLECTION DIAGRAM

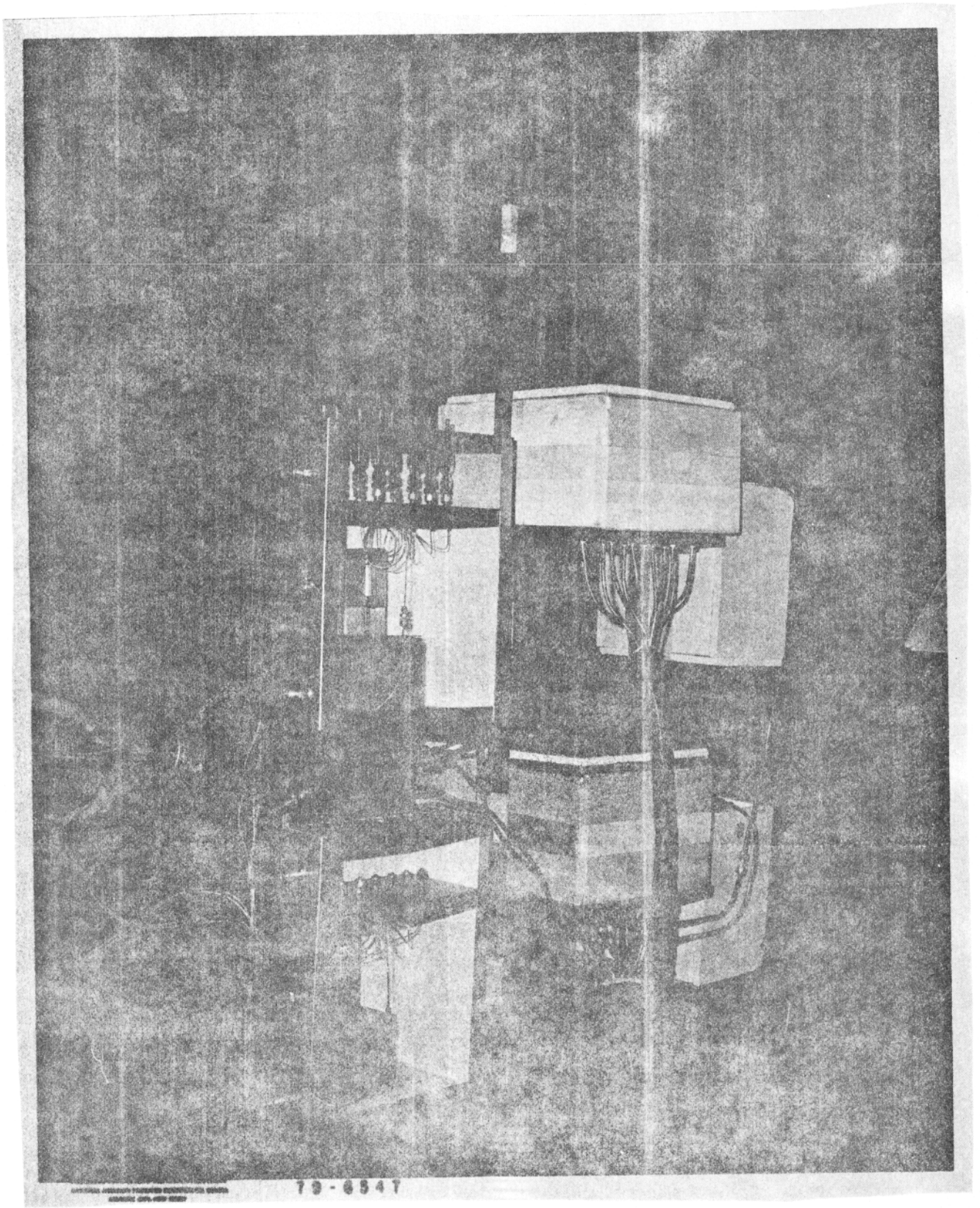


FIGURE 8. TOXIC GAS COLLECTION STATIONS (REAR VIEW)



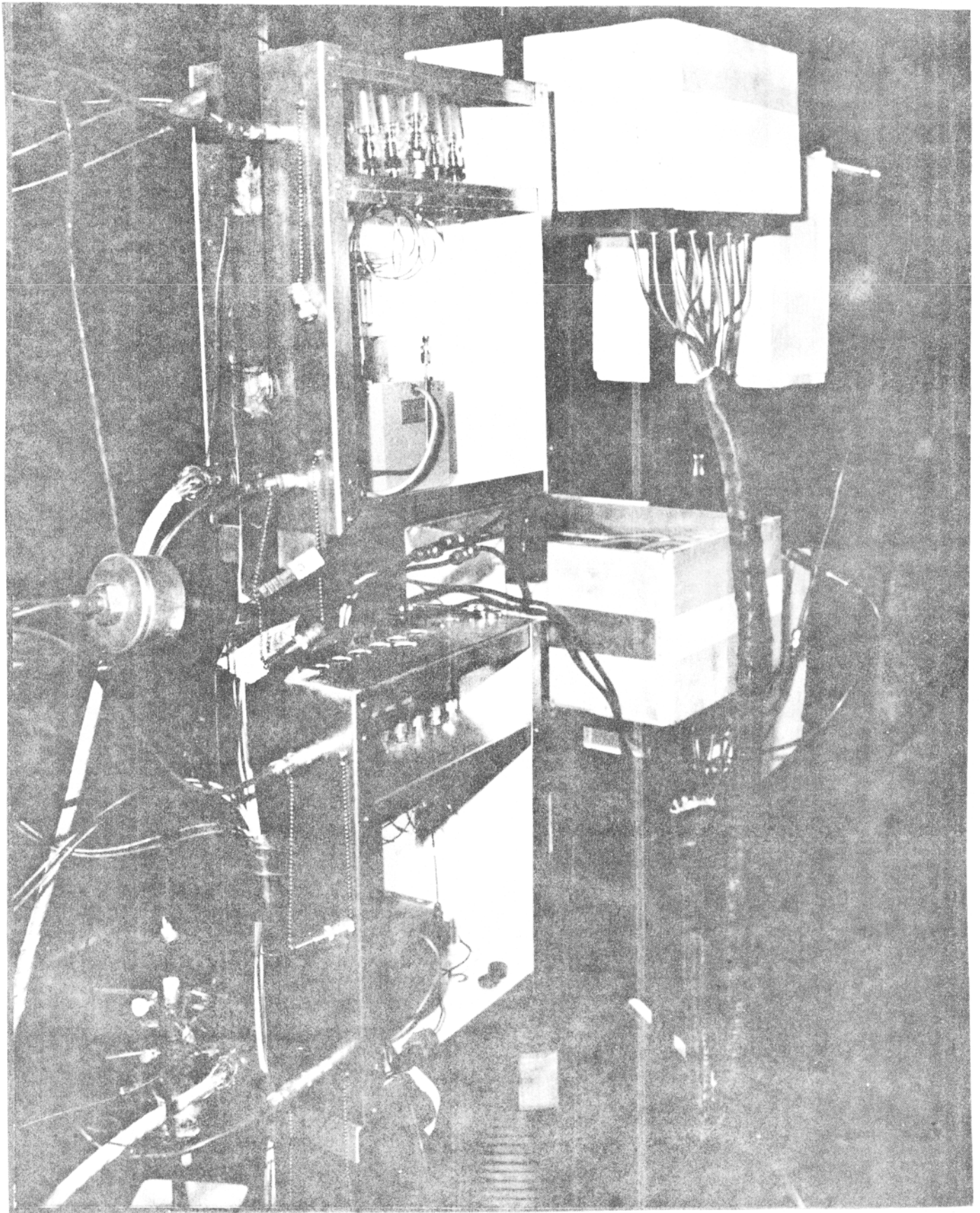
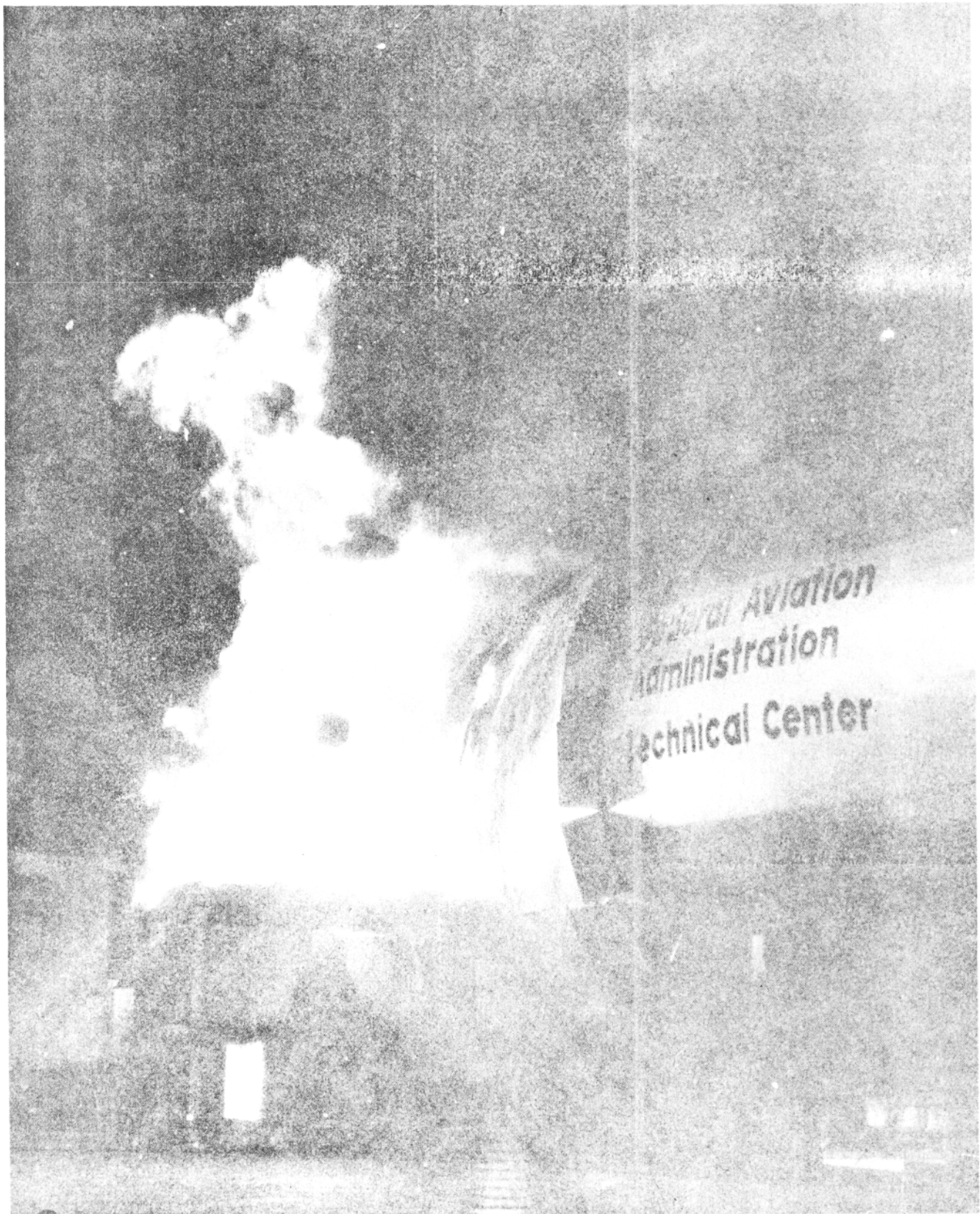


FIGURE 9. TOXIC GAS COLLECTION STATIONS (SIDE VIEW)



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FIGURE 10. CABIN FIRE SAFETY C-133 TEST ARTICLE

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