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The Pyrolysis Toxic Gas Analysis of Aircraft Interior Materials

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April 1982

Final Report

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Dominic Timoteo and Stanley J. Sternik

16. Abstract

Selected aircraft interior materials previously reported are tested by a new methodology. Gas and ion chromatographs linked to computers are utilized to identify and quantify gases evolved from a specific thermal exposure. Results are compared to those reported by other methods and instruments. Time concentration profiles are utilized to "Fingerprint" and identify the material by this test evaluation.

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METRIC CONVERSION FACTORS

Approximate Conversions to Metric Measures

Symbol	When You Know	Multiply by	To Find	Symbol
		LENGTH		
in	inches	*2.5	centimeters	cm
ft	feet	30	centimeters	cm
yd	yards	0.9	meters	m
mi	miles	1.6	kilometers	km
		AREA		
in^2	square inches	6.5	square centimeters	cm^2
ft^2	square feet	0.09	square meters	m^2
yd^2	square yards	0.8	square meters	m^2
mi^2	square miles	2.6	square kilometers	km^2
	acres	0.4	hectares	ha
		MASS (weight)		
oz	ounces	28	grams	g
lb	pounds	0.45	kilograms	kg
	short tons (2000 lb)	0.9	tonnes	t
		VOLUME		
tsp	teaspoons	5	milliliters	ml
Tbsp	tablespoons	15	milliliters	ml
fl oz	fluid ounces	30	milliliters	ml
c	cups	0.24	liters	l
pt	pints	0.47	liters	l
qt	quarts	0.95	liters	l
gal	gallons	3.8	liters	l
ft^3	cubic feet	0.03	cubic meters	m^3
yd^3	cubic yards	0.76	cubic meters	m^3
		TEMPERATURE (exact)		
°F	Fahrenheit temperature	5/9 (after subtracting 32)	Celsius temperature	°C

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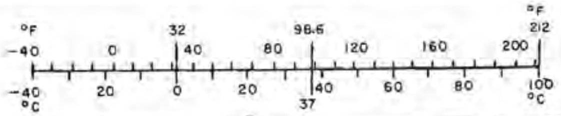
*1 in ≡ 2.54 (exactly). For other exact conversions and more detailed tables, see NBS Misc. Publ. 286, Units of Weights and Measures, Price $2.25, SD Catalog No. C13.10:286.

Approximate Conversions from Metric Measures

Symbol	When You Know	Multiply by	To Find	Symbol
		LENGTH		
mm	millimeters	0.04	inches	in
cm	centimeters	0.4	inches	in
m	meters	3.3	feet	ft
m	meters	1.1	yards	yd
km	kilometers	0.6	miles	mi
		AREA		
cm^2	square centimeters	0.16	square inches	in^2
m^2	square meters	1.2	square yards	yd^2
km^2	square kilometers	0.4	square miles	mi^2
ha	hectares (10,000 m^2)	2.5	acres	
		MASS (weight)		
g	grams	0.035	ounces	oz
kg	kilograms	2.2	pounds	lb
t	tonnes (1000 kg)	1.1	short tons	
		VOLUME		
ml	milliliters	0.03	fluid ounces	fl oz
l	liters	2.1	pints	pt
l	liters	1.06	quarts	qt
l	liters	0.26	gallons	gal
m^3	cubic meters	35	cubic feet	ft^3
m^3	cubic meters	1.3	cubic yards	yd^3
		TEMPERATURE (exact)		
°C	Celsius temperature	9/5 (then add 32)	Fahrenheit temperature	°F



PREFACE

The Federal Aviation Administration (FAA) Technical Center's Aircraft Fire Safety Program targets the development of a small-scale fire test for cabin interior materials including flammability, smoke, and toxic gas emissions. The emission of toxic gases by a burning material is an important element of the overall hazard. In addressing this aspect of the program, the Technical Center has developed data on toxic gas production from pyrolysis of aircraft interior materials. Reliable, reproducible test data are required to conduct and communicate the program. The latest analytical equipment, automated and supported by computer hardware and software is employed to identify, quantify, and document gas yields.

This report presents a specific methodology for fire gas analysis and documentation. The information is reduced to selected tabulations and graphic plots. This graphic presentation data can represent an identification, "Fingerprint" of the materials test event. Yields of selected toxic gases measured by methods used in the past are compared with results obtained by the current methodology.

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INTRODUCTION

PURPOSE.

The purpose of this report is to present the most recent methodology for aircraft material toxic pyrolysis gas generation, collection, analysis, and documentation, and to compare results with those obtained using other techniques for confirmation of data.

BACKGROUND.

Past studies of the toxic gases from pyrolysis of aircraft interior materials have been conducted over a 5-minute exposure at 600° centigrade (C) in an airflow of 2 liters per minute (references 1 and 2). The total yields of eight selected toxic gases were measured from samples collected in liquid-filled impingers by differential pulse polarography (DPP), ion selective electrode (ISE), and ultraviolet and visible spectroscopy. Carbon monoxide was collected in a plastic bag and the yield was measured using a nondispersive infrared analyzer.

This report expands on the collection technique, automatic chemical analyzers, and computer application for the identification, quantitation, and documentation of effluent gases from the above exposure in three distinct measurement groupings. Yields for 13 materials are used for comparison. The first measurement group is oxygen (O2), carbon monoxide (CO), and carbon dioxide (CO2), using automatic commercial gas analyzers. A computer collects data points and plots the progressive changing concentrations of these three gases in graphic form. These plots are referred to as text "Fingerprints," and are utilized to monitor subsequent test burns conducted to analyze for other fire results. The "Fingerprint" plots depict the decomposition history of the material under test. This technique also depicts autoignition very graphically. Subsequent analyses are for total yields of the other gas groupings. The second group is hydrogen cyanide (HCN) and other organic gases measured by gas chromatography (GC). The third group is hydrogen fluoride (HF) and hydrogen chloride (HCl), and other acid gases measured by ion chromatography (IC). Collection technique, chemical analysis procedure, and computer application are distinct for each gas group. This report includes examples of computer prints, plots, and computer augmentation.

EXPERIMENTAL

GENERAL APPROACH.

The test evaluation of an aircraft interior material for toxic gas emission under thermal degradation is divided into three parts. Each part encompasses a specific family of gases. The final test and analysis methodology is based on a number of considerations including state-of-the-art automation, chemical analyzers, the accumulated experience of past investigators, and guidelines suggested by the Committee on Fire Gas Toxicology of the National Research Council (reference 3), the American Society of Test Methods (ASTM), Committee E 5 (reference 4), and others (references 5, 6, 7, and 8). Triplicate tests are run, and in most cases, triplicate chemical analyses are run. Results are statistically treated and reported. The aircraft interior materials selected for this study (table A-1 of the appendix) were the same as those used in previous work (references 1 and 2). The selection was made on the basis of previous gas yields and the scope of the field to be studied.

THE GAS FAMILY GROUPS

The gas family groups consist of the following:

Part A	Oxygen, Carbon Monoxide, Carbon Dioxide.	
Part B	Major Gases:	Hydrogen Cyanide
	Minor Gases:	Acrolein, Acetonitrile, Acrylonitrile, Benzene, Acetone, Propane.
Part C	Major Gases:	Hydrogen Fluoride, Hydrogen Chloride.
	Minor Gases:	Hydrogen Bromide, Sulphur Oxides, Nitrogen Oxides

PART A - FIRE GASES.

A procedure for the continuous automatic monitoring of oxygen, carbon monoxide, and carbon dioxide concentrations from a combustion tube furnace test is used to "Fingerprint" the rate of thermal decomposition of a material at a constant temperature exposure for a fixed interval.

A 250 plus or minus 5-miligram environmentally (ASTM) conditioned sample is placed in a tare weighed ceramic boat. The boat is inserted inside a 25-millimeter Vycor combustion tube inside a Lindberg single-zone tube furnace (CTF) stabilized at 600° C (figure 1). The temperature is measured with a Leeds Northrup potentiometer. A dry airflow of 2 liters per minute is applied. The Vycor tube is positioned so that 10 centimeters is extended beyond the furnace to reduce buildup of combustion products in a hot zone that could support a flash-fire in the tube, yet remain warm enough to minimize condensation. The tube is attached to a 10-milimeter glass "T" with a 4-millimeter bore. The "T" is a stopcock with a silicone rubber stopper. One leg of the "T" is connected to a 12-liter Saran™ bag, and the other leg to a continuous gas analyzer module (figure 2). This module was designed and constructed at the Federal Aviation Administration (FAA) Technical Center, and is described in the next section.

At the initiation of the test, all legs of the stopcock "T" are opened and the Saran bag is emptied. The analyzer module draws 1/2 liter per minute of dry air through the combustion tube. The sample is placed in the furnace and a calibrated 2 liters per minute of dry air enters the system. The system allows for a 1 and 1/2-liter-per-minute flow to enter the Saran bag and the remaining 1/2 liter per minute enters the module.

After 5 minutes, the stopcock is switched on connecting the Saran bag to the module only. The average concentration of gases represented in the bag is the yield for the test run. The combustion tube and furnace are then removed. After a cooling period, the ceramic boat and ashes are weighed, and the weight loss is recorded. The dry airflow for the test is calibrated with a wet gas meter corrected for temperature and pressure (Boyles Law) and monitored through a Matheson #603 flowmeter.

The automatic gas analyzer module flow diagram is presented in figure 2. The automatic gas analyzer module was assembled to continuously measure the oxygen, carbon monoxide, and carbon dioxide effluent from a combustion tube furnace burn test. The carbon monoxide and carbon dioxide are analyzed with a Beckman Model 864 nondispersive infrared analyzer. The oxygen is analyzed with a Beckman OM-11 polarograph analyzer. All systems plumbing is with 1/4-inch stainless-steel swagelok fittings. The module has been equipped with all necessary calibration gas bottles and rotometers for testing blended gas samples. The automatic analyzers are hooked up in series. The pump to the oxygen analyzer draws 1/2 liter per minute through the entire system. Downstream of the furnace and stopcock is a 10-millimeter by 65-millimeter section of glass tubing packed with 25-millimeters of Alcoa type F-1 activated alumina 30/60 mesh held in place with glass wool. This is attached to a Gelman 2220 stainless steel inline filter holder equipped with a 47-millimeter polypropylene filter disc. All fittings prior to the filter are Teflon™ swagelok and after the filter stainless steel swagelok. This configuration adequately protects the analyzer sensors from tar effluent buildup, yet results in no more than a 20-millimeter pressure differential due to clogging from start to finish of a test. Both filters are replaced after each test run. The polypropylene discs are retained for future comparisons. During the test duration, a uniform 1/2 liter per minute flow is maintained. Calibration of the test module with certified full-scale standard gases is performed before every test. Two ranges are used for the carbon monoxide and carbon dioxide analyzers. The high range is calibrated 0-10 percent CO and 0-20 percent CO2. The low range is 0-2 percent CO and 0-5 percent CO2. Generally, low range is used for materials with fiber glass as a reinforcement, or decompose slowly. The high range is used for materials which decompose quickly and are prone to autoignition.

Computer programs have been developed inhouse to document the results from the automatic gas analyzer module (reference 9). The analyzer instrument output is set at 0 to 5 volts direct current and is compatible with the analog-to-digital converters on the DEC PDP 11/03 computer system. Each combustion tube furnace burn has three files established, one for each gas analyzer, and data are collected directly on an RL-01 5-megabite disc in real-time.

The PDP 11/03 is linked through an RS-232 interface to a Tektronix 4051 graphics terminal with a hard copy unit and a Tektronix 4662 flat bed plotter.

After a test burn is completed, file output can be documented and displayed in a variety of options. Computerization allows the complete data bank to be instantly accessible and has the advantage that any material test result can be compared to any other directly and visually. As many overlays, displays, and plots can be compared as desired. Data remain in the file and can easily be recalled and/or augmented.

The time concentration plots of material decomposition provide insight not available with total yield information alone. Within the series of three burns of the same material, direct comparison of each burn event can explain variations in yield of carbon monoxide which is toxic, and carbon dioxide which is less of a hazard but indicative of a more complete oxidation. Autoignition, flashover, events of a material are clearly shown by a sudden decrease in oxygen concentration with a concomitant increase in carbon oxides. During the test, this flashover is also visual and audible.

In a comparison of materials, sample 14 (figure 3) shows burns 1, 3, and 4; burn 1 was somewhat more gradual with the exception of an early large carbon monoxide peak that is reflected in the final carbon monoxide yield. Burn 3 was the most oxidative burn having the largest carbon dioxide peak. Burn 3 also produced the lowest hydrogen cyanide yield, pointing to the theory that an oxidative burn can carry hydrogen cyanide over to nitrous oxides in the effluent gas mix. This reaction yield is also reflected in material 15 (figure 4), burn 4. The potential relative flammability hazard of a material is graphically displayed for study. As an example, material sample 6A (figure 5) depicts very gradual decomposition, in addition to low gas yields. Material sample 32 (figure 6) likewise undergoes relatively slow oxidation 2 minutes into the burn event, and demonstrates very good reproducibility between tests. In comparison, material sample 104 (figure 7) decomposes quickly and autoignites. The burn event is almost completely over in 1 1/2 minutes. An analysis of the results from material sample 100 (figure 8) is interesting. It resulted in the highest standard deviation in hydrogen cyanide yield. One possible explanation can be seen in burn 3 which showed double the hydrogen cyanide total yield and did not undergo the autoignition events seen in burns 1 and 2. Consequently, organic gas concentrations are also considerably higher because oxidative combustion did not occur.

Additional "Fingerprints" are depicted in figures 9 through 13.

It has often been reported that no two fires are exactly alike, despite very carefully controlled conditions, uniform temperatures and airflows, sample size and conditioning, etc (reference 11).

PART B ORGANIC GASES.

In part A, the description of gas collection is presented for the automatic analyzer module and the Saran bag sample. The bag sample is used for the gas chromatographic analyses of the organic gas family group including hydrogen cyanide, referred to as part B gases (figure 14). At the end of a test run burn, the Saran bag is mixed and kneaded for uniformity. Gas samples are extracted by volumetric syringe, or adsorbed on to 30/60 mesh GC porous thermally desorbed polymer Tenax in a 12 centimeter by 1/4-inch glass lined stainless steel tube. The tubes are sealed with Parafilm and stored in a freezer for later analysis. Samples remain quantitative for 1 week at minus 17° C. The adsorbed sample is connected in a backflush mode to a Perkin Elmer 3920 GC equipped with flame ionization and nitrogen phosphorous detectors. Data is acquired on a Perkin Elmer Sigma 10B computer data station. A program for the collection of raw data, calculation of quantities of identified gases, and documentation is started. Examples of various documentation output are presented in figures 15 through 20.

The GC is calibrated daily from a cylinder of standard hydrogen cyanide gas with nominal 300-parts-per-million concentration. The cylinder is checked every month by titration with silver nitrate and has been found to be stable at this concentration for this period. Ten millimeters of the hydrogen cyanide calibration gas is drawn from a flowing stream into a Tenax tube with a 10-millimeter gas tight syringe adapted to accept the 1/4-inch outer diameter tube. This has been found to give identical results to direct syringe injection with the exception of slight retention time variations. The alkane series is calibrated with direct syringe injection of Scott standard mixtures. Those compounds, which are liquid at room temperature, are calibrated by injection of 10 microliters of headspace from the pure compound in a septum vial. Parts per million concentrations are calculated via the Clausis-Claperon equation for vapor pressure to determine the amount present. The reproducibility using this technique is plus or minus 15 percent. This technique has obvious advantages of speed and convenience. Since there is no solvent peak, time consuming preparations are bypassed and one need not worry about solvent evaporation changing the concentration of the calibration sample.

All information on the Sigma 10B tapes is then printed out for reference and tabulation. The total information is then transferred via RS-232 interface to magnetic discs on the larger PDP 11/03 computer for filing and random rather than sequential access. The raw data can be augmented and recalled for plotting and comparison study. The results for as many gases as detected are placed in the permanent file for both GC detectors. As other peaks are identified and calibrated, the data bank can be updated to include the information.

The HCN yields for this report are listed in table A-2 of the appendix, along with statistical comparison of this and the older method.

HCN yields for the Nitrogrn Phosporous detector (NPD) are presented in table A-2 of the appendix. While HCN is seen with the flame ionization detector (FID), the NPD, because of its high selectivity, is more accurate due to incomplete peak resolution from propane. That is, propane has approximately ten-fold the response of HCN at identical concentration on FID, while HCN has one-hundred times the response of propane on NPD so peak resolution is not necessary. For each material burn, three Tenax samples are drawn. The three analyses are averaged and the burn yield and the percent RSD for the analytical technique reported. The results for the three different burns of the same material are reported as the yield for that material and the percent RSD associated with the yield may be thought of as the repeatability of the combustion process, as oppose to the analytical technique.

Part C - Acid Gases.

The pyrolysis acid gases are identified and measured by ion chromatography. These gases are generated in the combustion tube furnace (figure 1) described in Parts A and B. The collection method for Part C is different. An airflow of 2 liters per minute is drawn through the CTF using a vacum pump. The combustion gases exiting the tube are divided equally into four streams monitored by rotometers, and collected in liquid filed bubblers (figure 1). Four bubblers are used for balance and multiple testing and to control foaming. A series backup bubbler is used to evaluate for collection efficiency. The yields reported in table A-3 of the appendix, are obtained by combining the yields from the first and its backup bubbler. The bublers contain 25 milliliters of 5/100 molar sodium carbonate solution. Sodium carbonate solution produces less interference than sodium hydroxide. The backup bubbler can collect up to 25 percent of the effluent gas. Yields in table A-3 of appendix A are combined yields for two bubblers.

Anion concentrations are measured with a Dionex Model 10 Ion Chromatograph, modified for use with a Dionex autoion system 12 Autosampler. An injection valve actuated by solenoid valves was added in parallel with the existing air toggle controlled injection valve. The air toggle controlled valve is used for manual syringe injection, and the electrically controlled valve is for automatic injection. Another addition to the system is an injector valve which diverts the eluent through the appropriate valve. Three potentiometric detectors are used downstream of the conductivity detector. Two detectors are used for measuring chloride and one for fluoride. A bioanalytical TL 6 thin layer silver, silver chloride cell followed by a mercury, mercury chloride thin layer cell with a double junction silver, silver chloride reference electrode are used to measure chloride. The silver, silver chloride thin layer cell is illustrated in figure 21.

A thin layer flow cell housing an Orion solid-state fluoride electrode and double junction silver, silver chloride reference electrode has been installed further downstream. This flow cell is composed of an electrode holder and a 30-mil polyethylene spacer sandwiched between plexiglas blocks. The electrode holder screws into the block. An O-ring around the fluoride electrode presses against the block to form a leakproof seal. A salt bridge also rests in this block and is held in place by a recessed O-ring. A silver, silver chloride reference electrode rests in the salt bridge containing a 10-percent potassium nitrate solution (figure 22).

These cells are housed in a grounded aluminum box (faraday cage) to protect them from any surrounding electromagnetic fields. This box is lined with Kaowool™ board insulation. A coil of 1/4-inch outer diameter (O.D.) copper tubing lies on the bottom of the box. A water line from a constant temperature circulating bath passes through the system to maintain a constant box temperature. Room temperature fluctuations of plus or minus 5° C result in a box temperature change of 1° C. A 3- x 300-millimeter low capacity anion exchange column is used as a separatory column. A 6- x 250-millimeter cation exchange suppressor column is used. Glass water jackets and a constant temperature circulating water bath control the temperature at the suppressor column and the detectors. The bath temperature is maintained at 25° plus or minus 2/100 of a degree centigrade. A 45/100 micron disposable syringe filter is used to protect the analytical column from contamination from samples.

The potentiometric detectors are sensitive to changes in the eluent flowrate. A 5-foot coil of 1/4-inch O.D. diameter Teflon lined stainless steel tubing, added at the pressure gauge, results in a more than usual constant pressure of 400 pounds per square inch with a pressure differential of 5 pounds per square inch per pump cycle. This coil depulses the eluent flow, resulting in a stable baseline.

The injection valve is equipped with a 100-microliter sample loop. The eluent is a 2.4 millimolar sodium bicarbonate, 2.6 millimolar sodium carbonate buffer. The eluent flow is 3.1 liters per minute (reference 10). A calibration curve is constructed for chloride and fluoride by using standard solutions containing both ions. Five standard solution concentrations are entered into the PDP 11/03. The program to control the autosampler is run. The standard solutions are injected under program control every 6 1/2 minutes. The resulting calibration curves appear on the cathode-ray tube (CRT) (figures 23 and 24). The chromatographer reviews the calibration curves and proceeds to run samples if the curves are satisfactory. Calibration curves for each test are stored on disc.

The millivolt signal from each detector is amplified so that it falls into a 0- to 5-volt range, which is compatible with the analog-to-digital converters to the computer. The PDP 11/03 computer is used for data acquisition and reduction. A file is set up in memory for each detector, and data is collected in real-time. Reduced data is stored on an RL-01 5-megabite disc. The PDP 11/03 is linked through an RS-232 interface to a Textronix 4051 Graphics Terminal with a Hard Copy Unit, and a Textronix 4662 Flat Bed Plotter. Examples of computer output are presented in figures 25 through 28. Programs are written to present reduced test data as plots of time versus concentration, or as a tabulated report.

STATISTICAL CHARTS.

Figures 29 through 32 present the stastistical comparison of the yields of the toxic gases tested in this program and those yields reported by other chemical analysis.

COMPARISON OF METHODS.

The yields obtained using the old and new methods for fluoride and chloride analysis compare well. This can be seen in figures 30 and 31. Perfect agreement between methods is indicated by a solid line. Bromide is known to be an interference for the polarographic analysis of chloride. These anions interact catalytically at the mercury electrode. Hydrogen chloride yields obtained by DPP in which both anions are present are in error by as much as 50 percent (reference 2). Materials 14, 15, 32, and 42 have both HCl and HBr as thermal decomposition products. Chloride yields obtained using DPP are significantly lower for these materials than yields obtained using IC with a Ag/AgCl detector. Bromide is not an interference for chloride in the IC method, as these species are separated prior to the detector.

Figure 32 illustrates the different yields obtained using IC with two different metal/chloride salt of metal IC detectors. Yields can be seen to be generally higher with the Hg/Hg_2Cl_2 detector. This is probably due to the incomplete return to baseline from a tailing bromide peak from a previous sample. Brommide peaks obtained using the Ag/AgCl detector are symmetrical.

CONCLUSIONS

The conclusions reached from this project and the comparison of results achieved from the new methods of combustion gas analysis (computer aided gas and ion chromatography) versus the old methods (differential pulse polarography) are:

1. New methods of analysis give comparable results to old methods.

2. New methods via computerization and automation allow for large number of samples to be analyzed.

3. New methods lend themselves to expansion to analyses of other gas species.

4. New methods employ commercial equipment that can be adopted and utilized by most laboratories.

5. Fingerprints can be viewed as an identification of a material and/or a monitoring of a burn event that is used to study other fire hazards.

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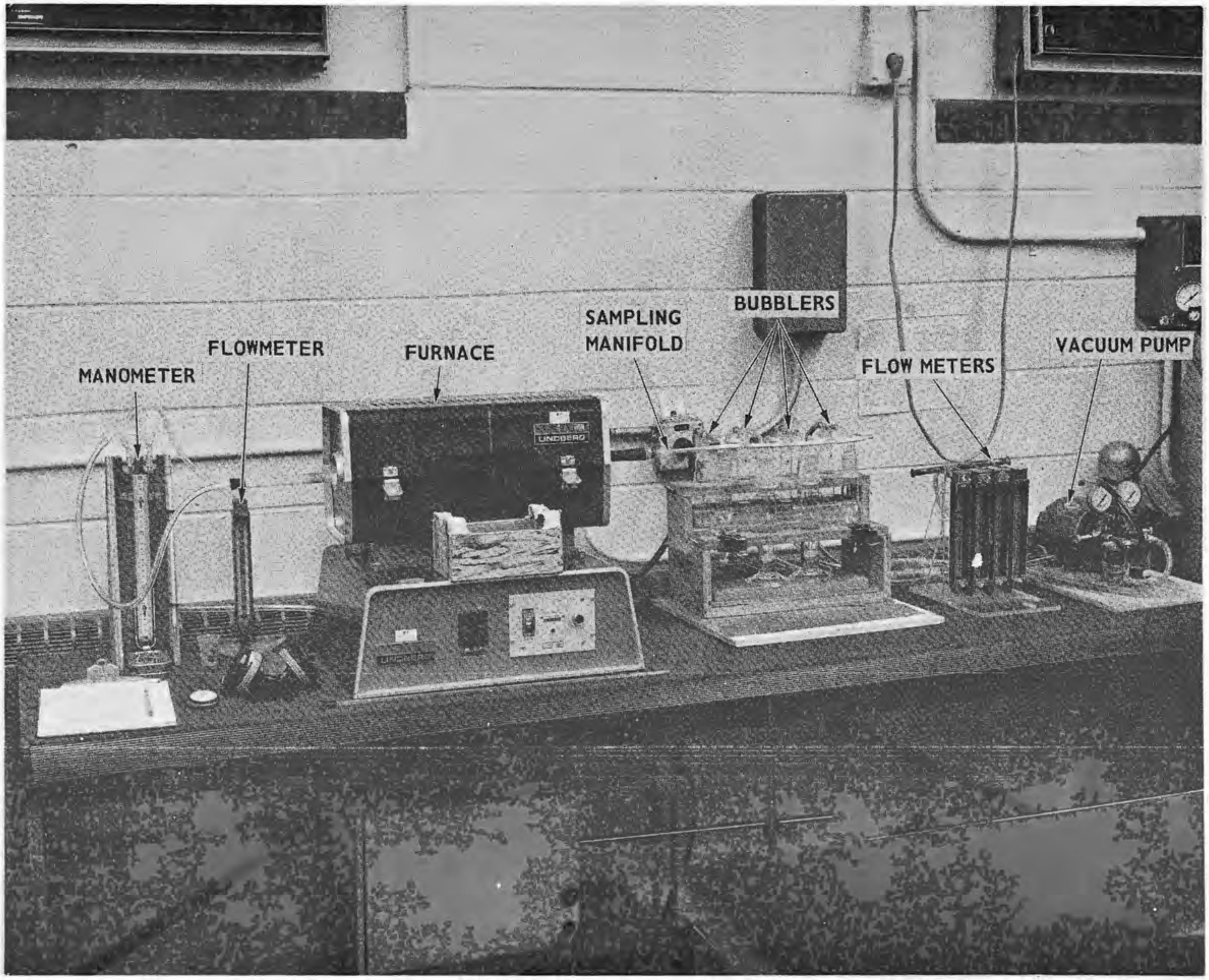


FIGURE 1. COMBUSTION TUBE FURNACE AND GAS SAMPLING SYSTEM

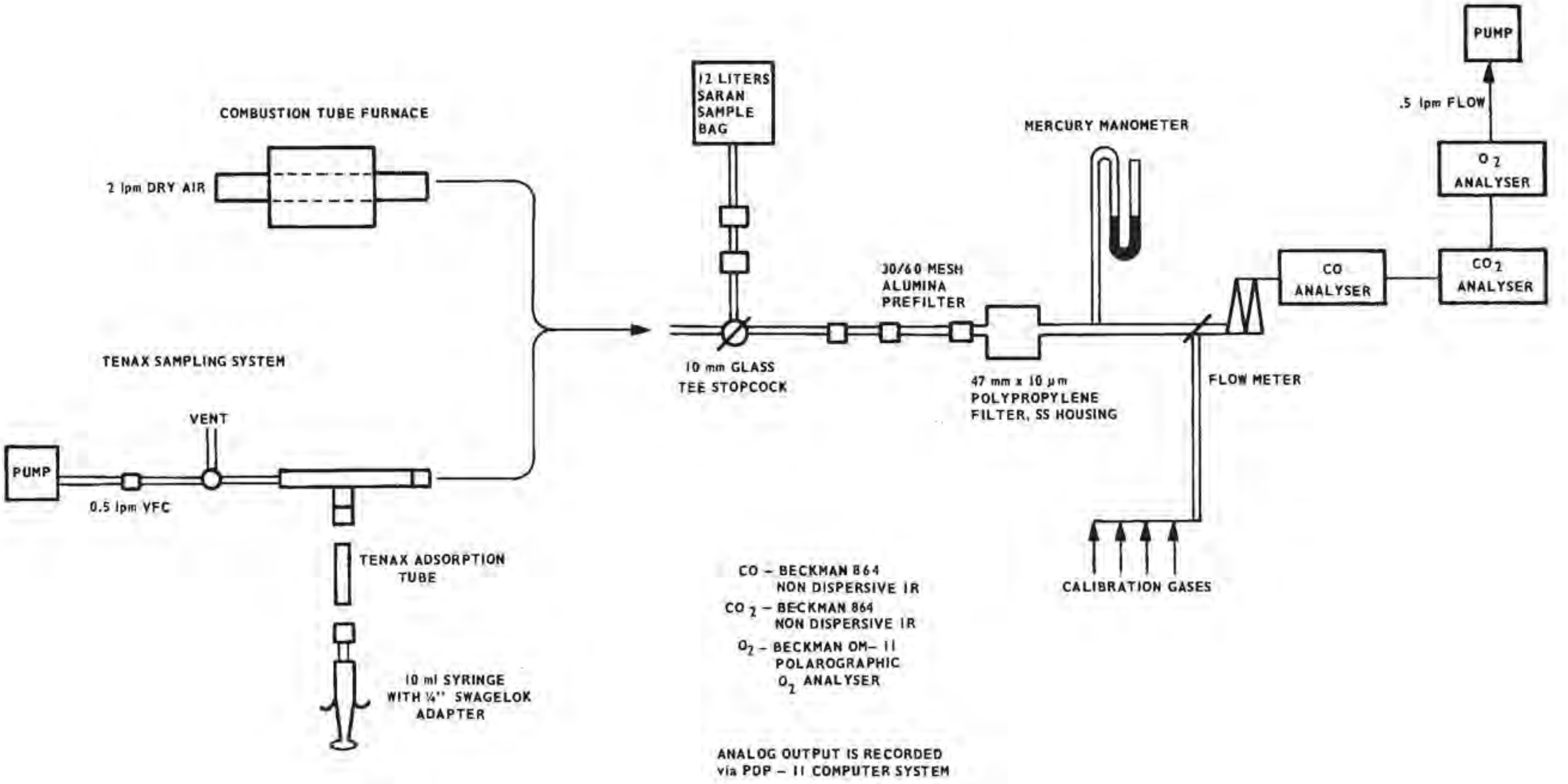


FIGURE 2. AUTOMATIC GAS ANALYSER

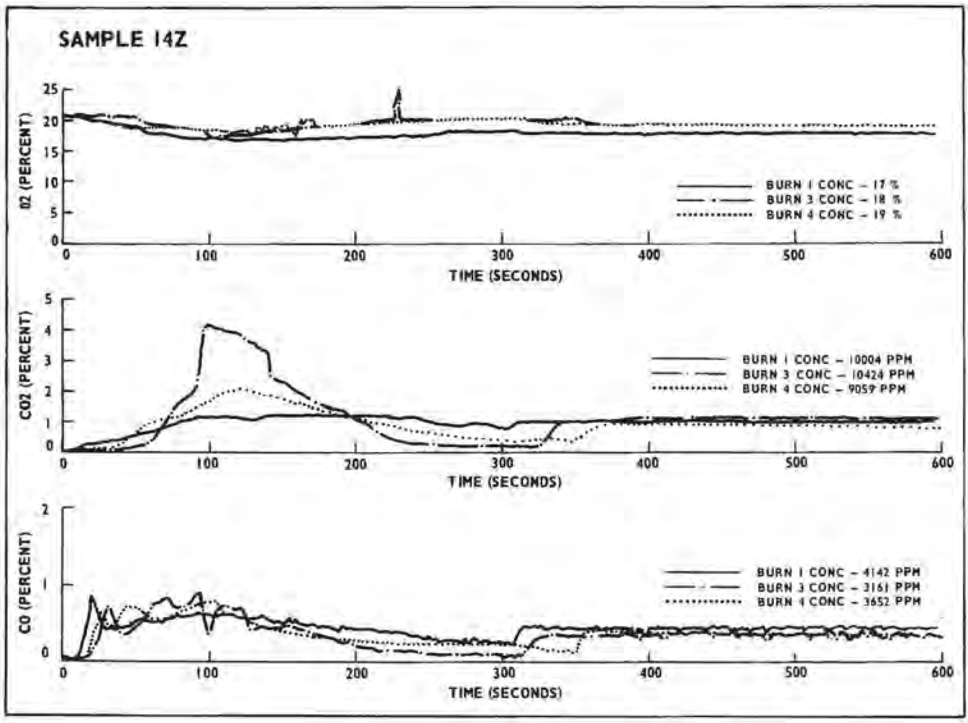


FIGURE 3. FIRE GAS FINGERPRINTS, 14Z

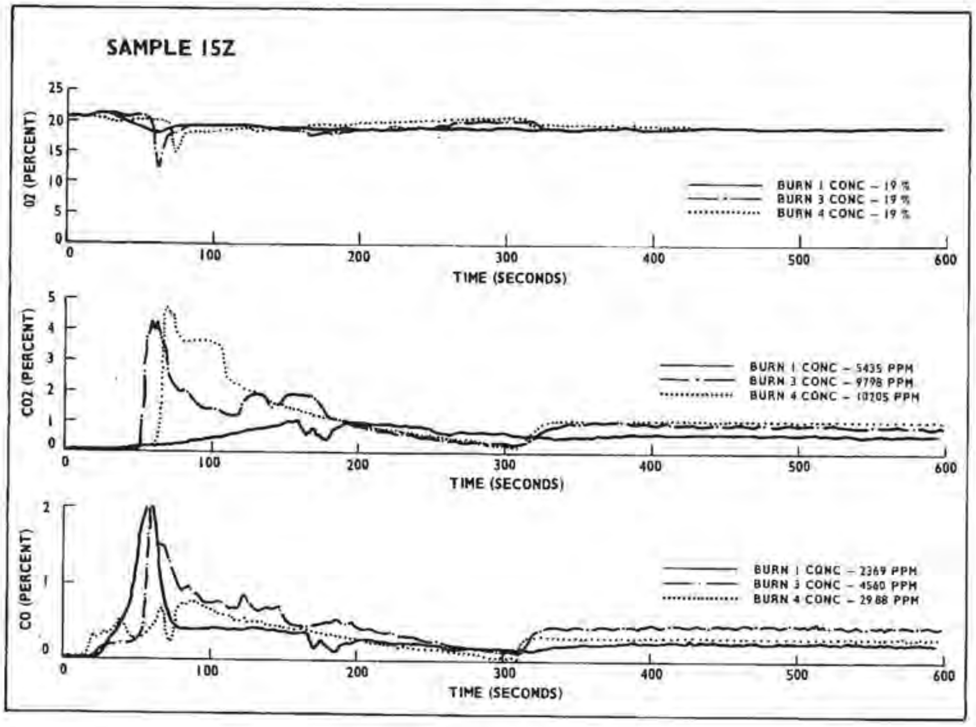


FIGURE 4. FIRE GAS FINGERPRINTS, 15Z

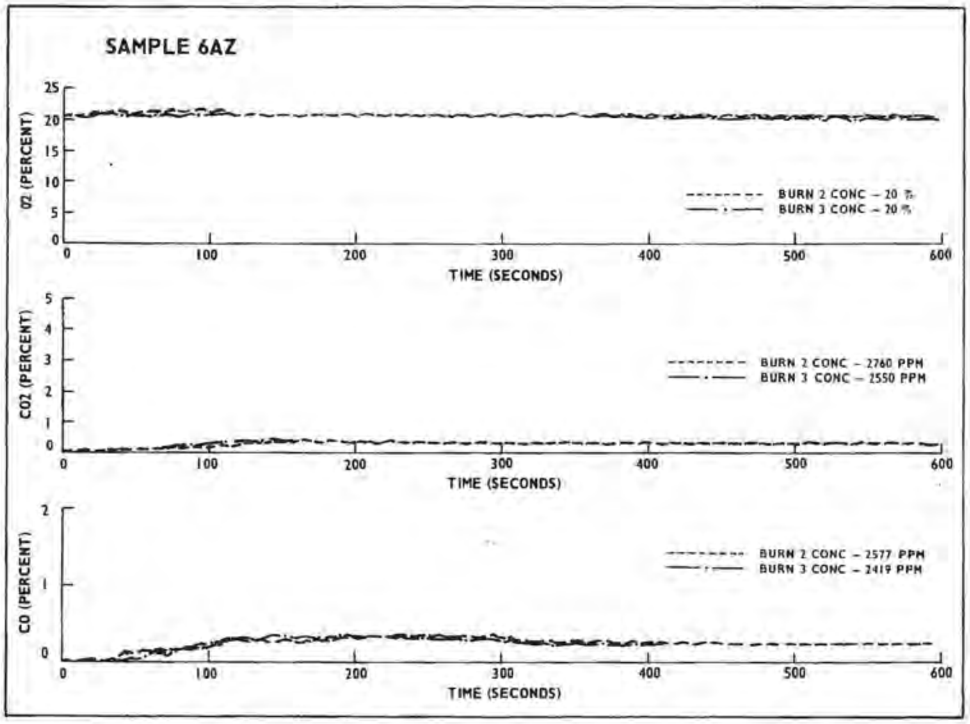


FIGURE 5. FIRE GAS FINGERPRINTS, 6AZ

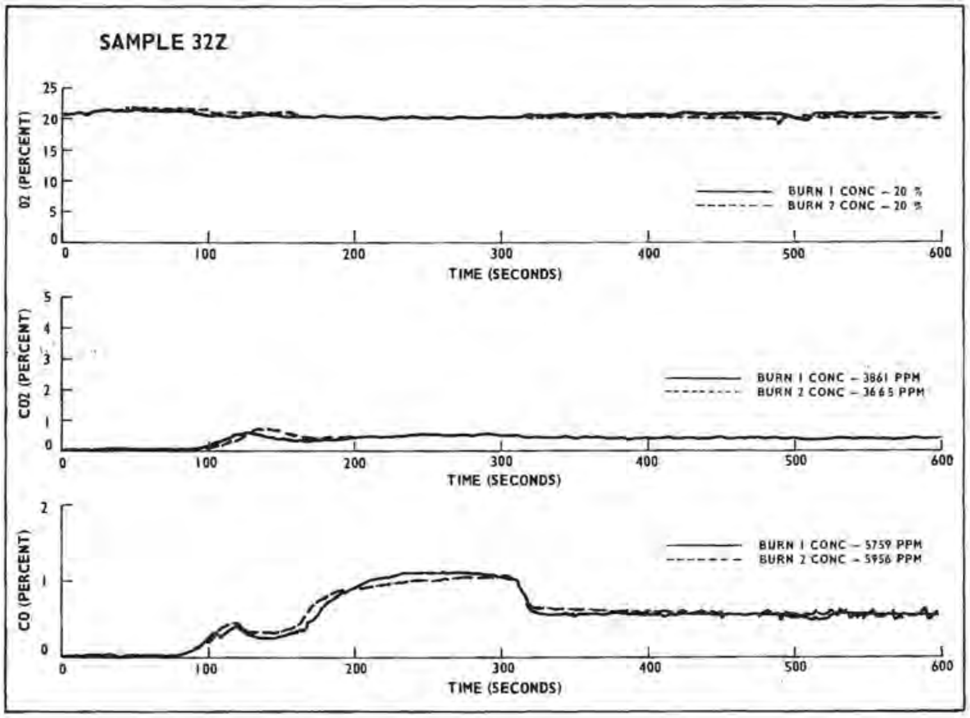


FIGURE 6. FIRE GAS FINGERPRINTS, 32Z

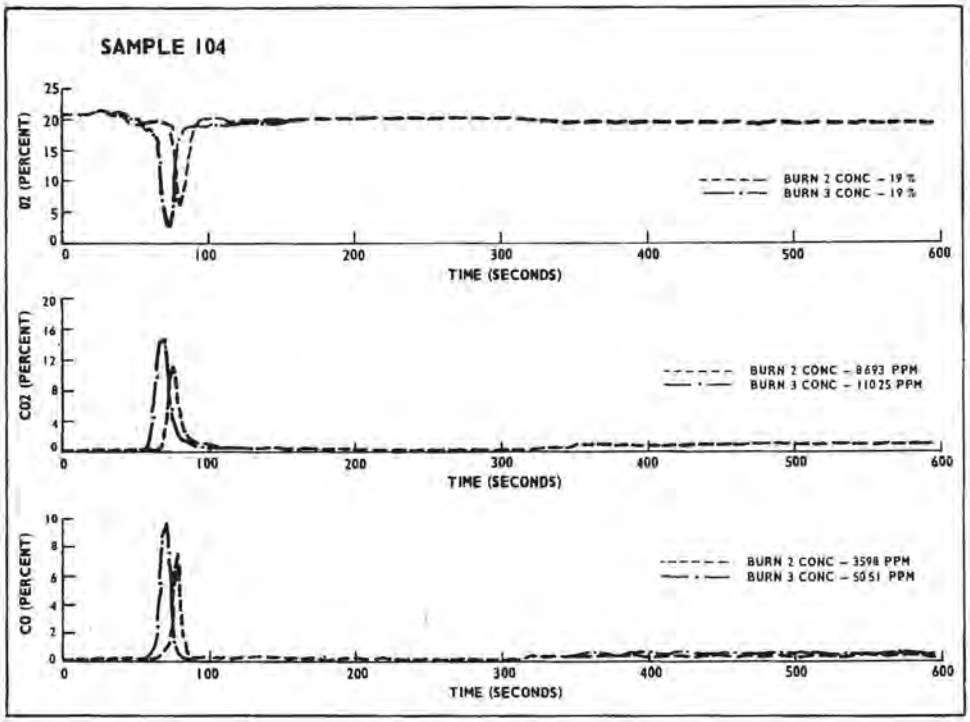


FIGURE 7. FIRE GAS FINGERPRINTS, 104

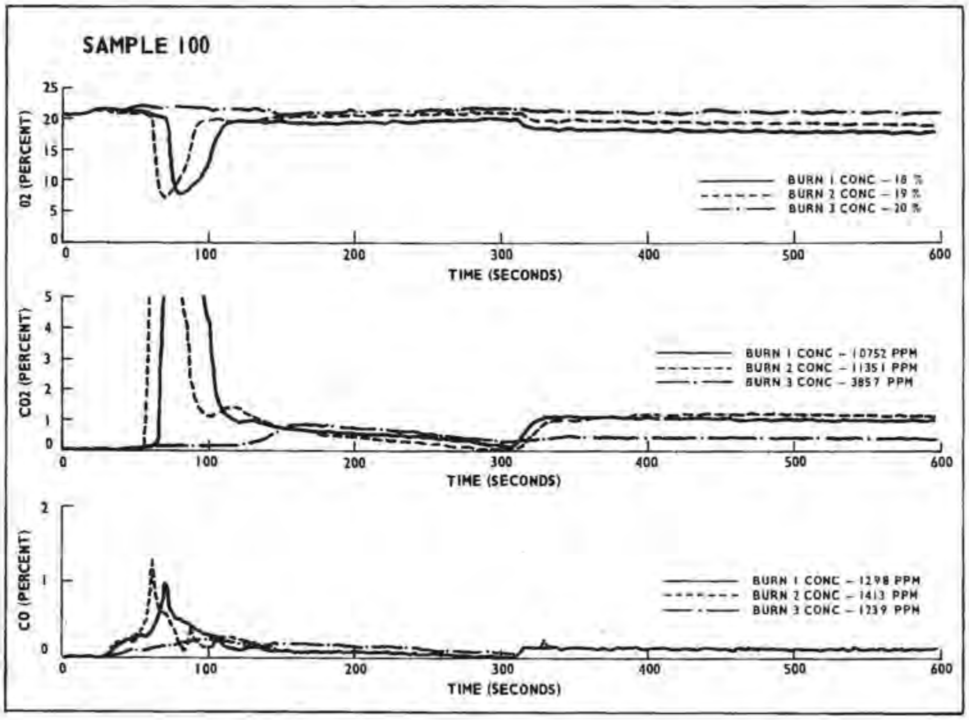


FIGURE 8. FIRE GAS FINGERPRINTS, 100

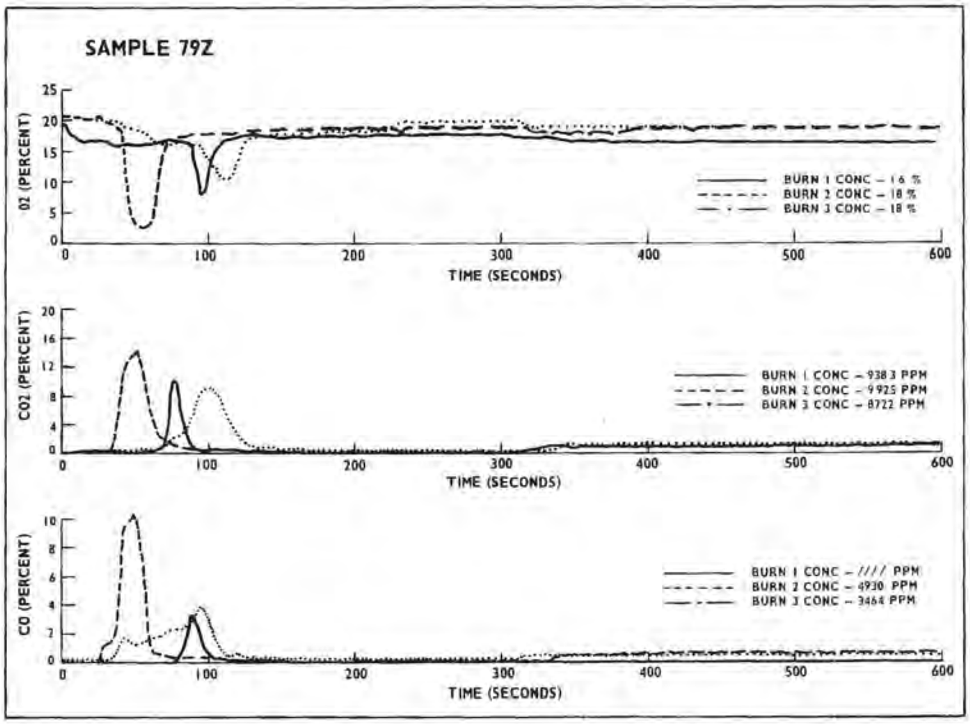


FIGURE 9. FIRE GAS FINGERPRINTS, 79Z

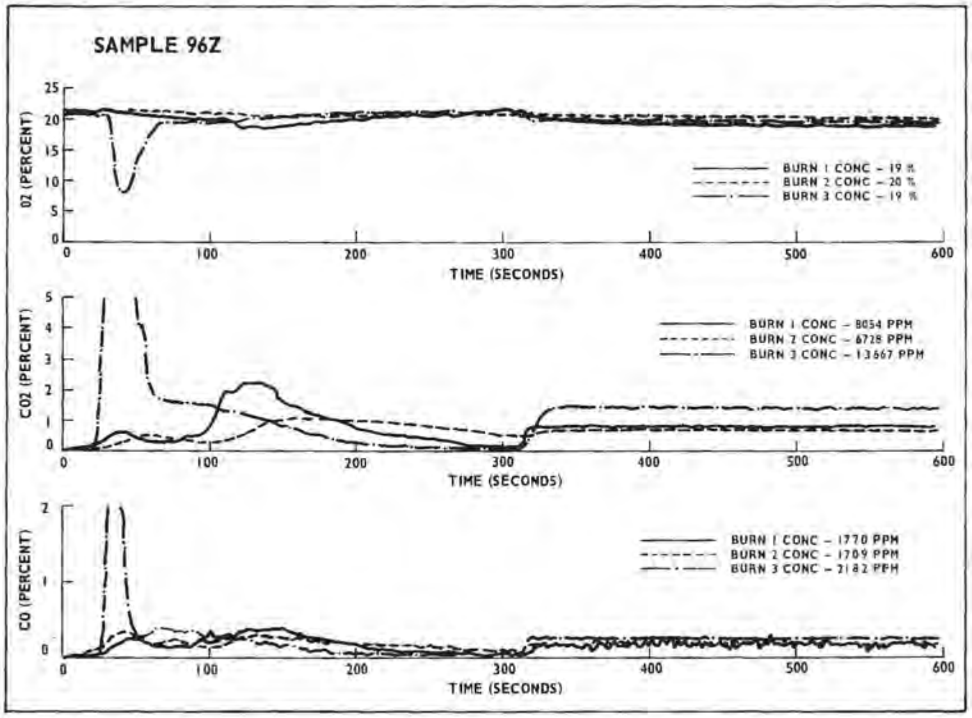


FIGURE 10. FIRE GAS FINGERPRINTS, 96Z

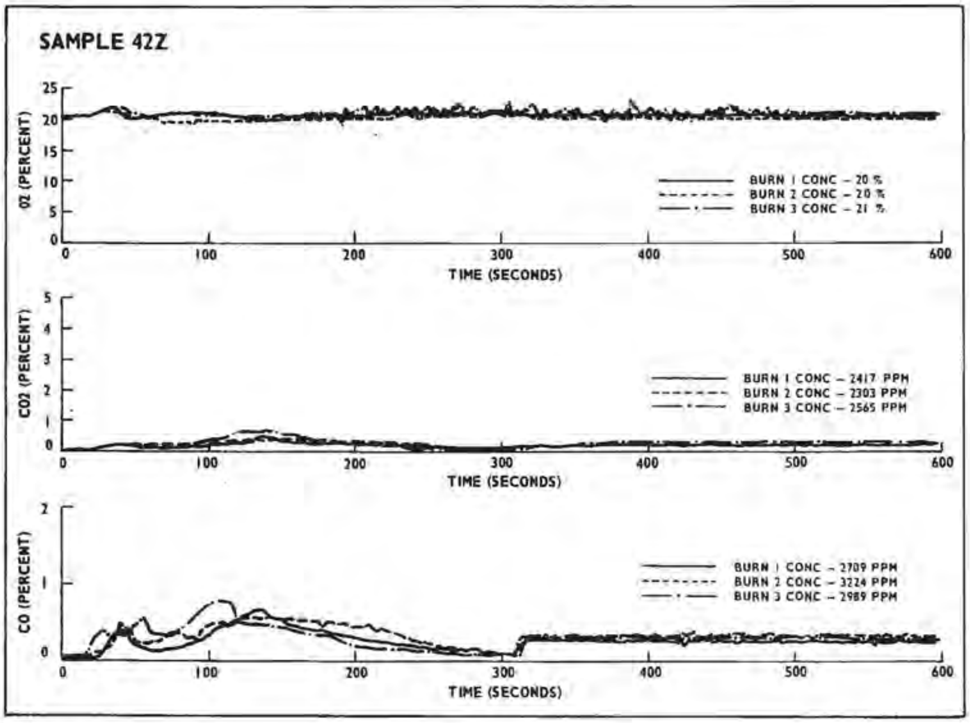


FIGURE 11. FIRE GAS FINGERPRINTS, 42Z

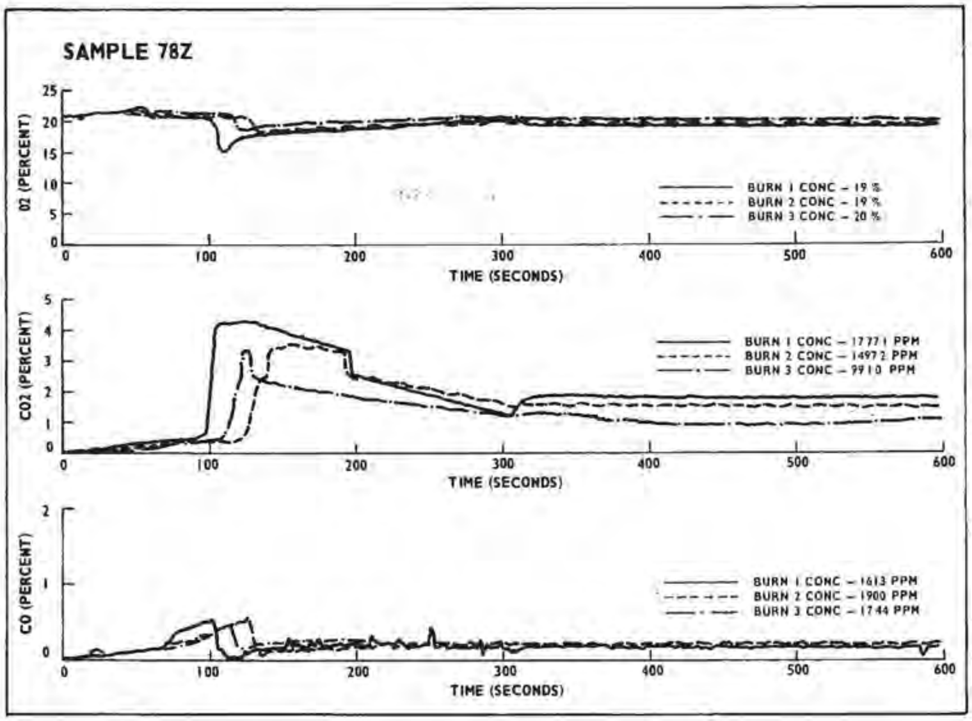


FIGURE 12. FIRE GAS FINGERPRINTS, 78Z

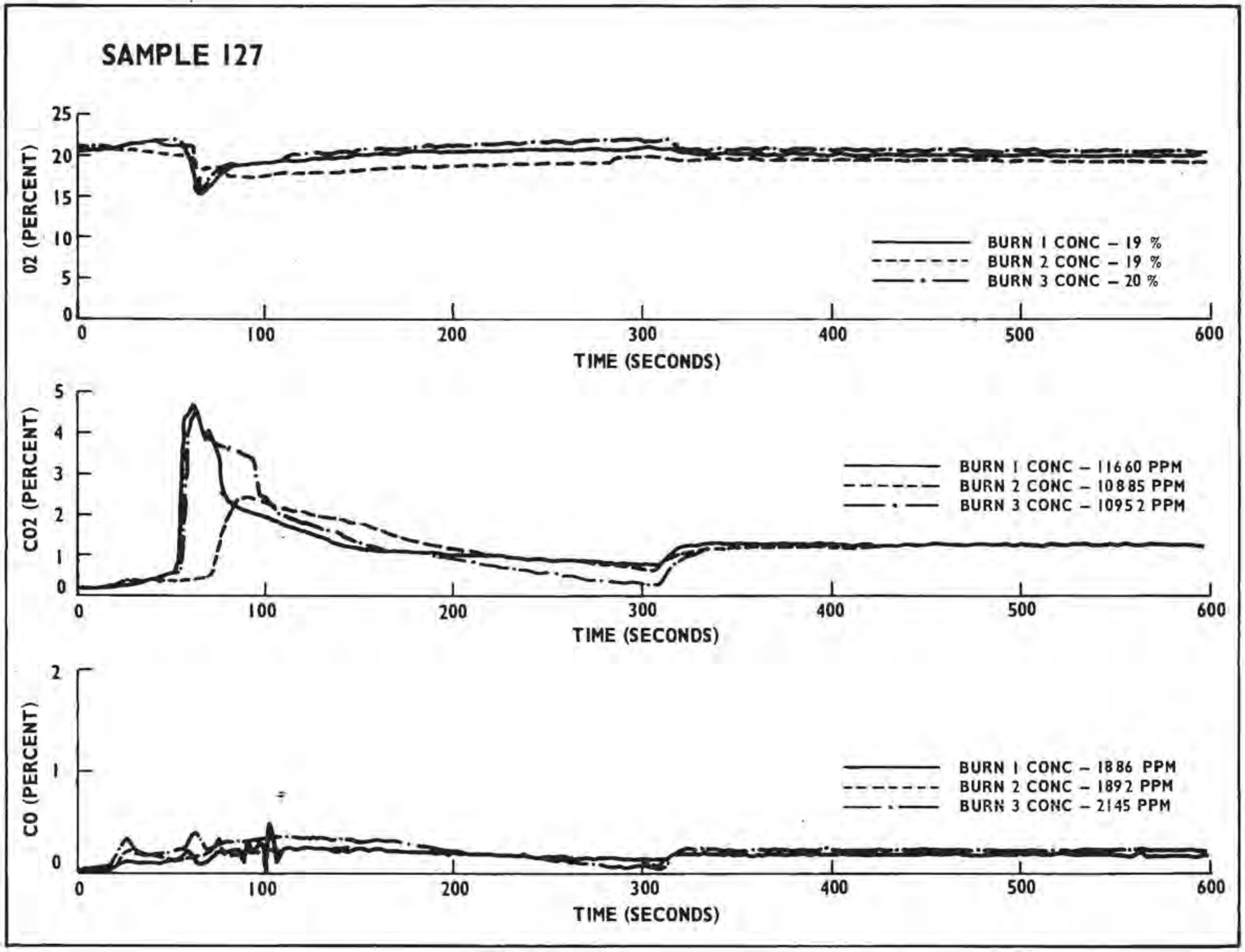
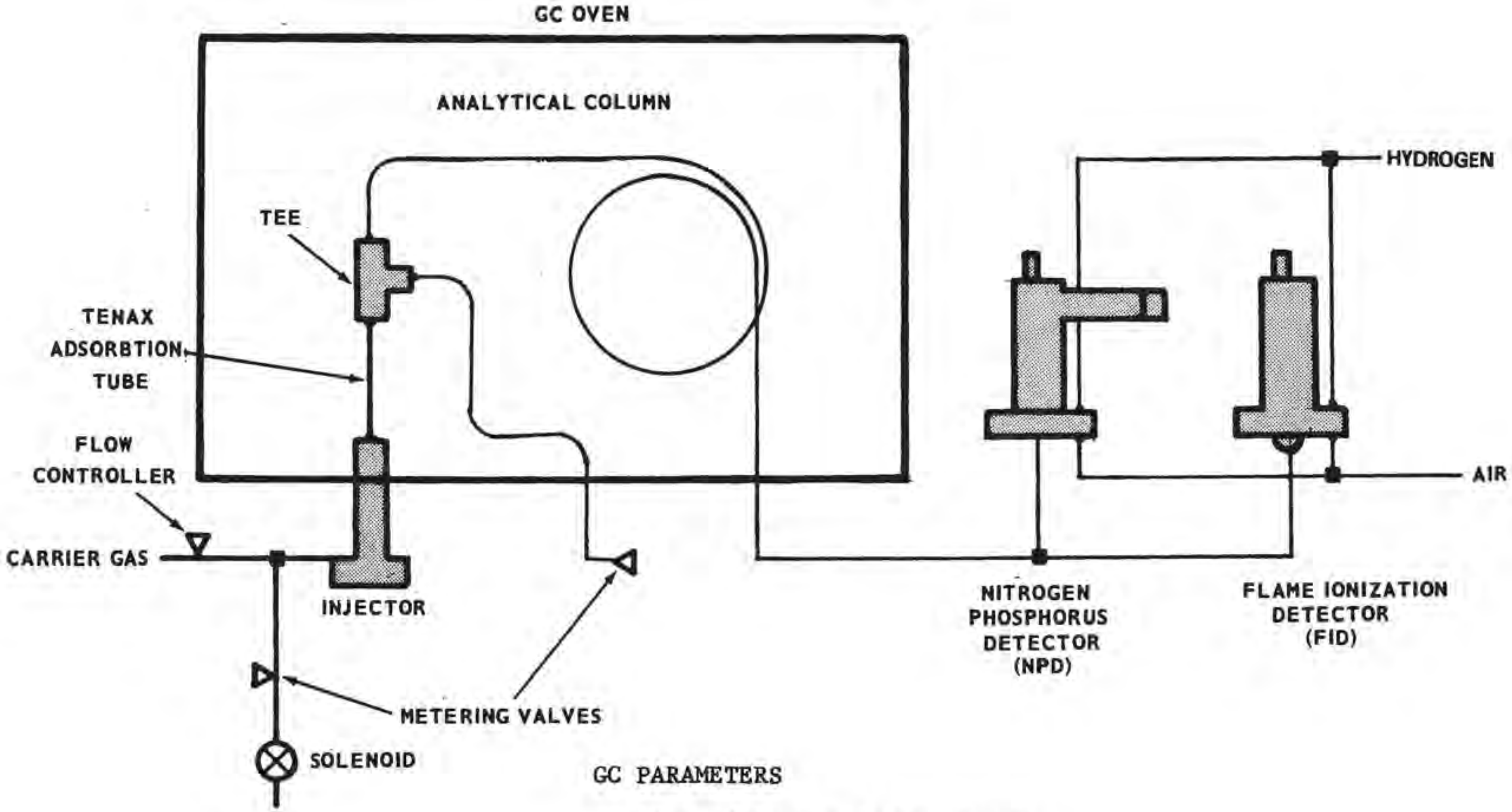


FIGURE 13. FIRE GAS FINGERPRINTS, 127



GC PARAMETERS

4' x 1/8" Porapak QS 50/80
4' x 1/8" Porapak S 80/100

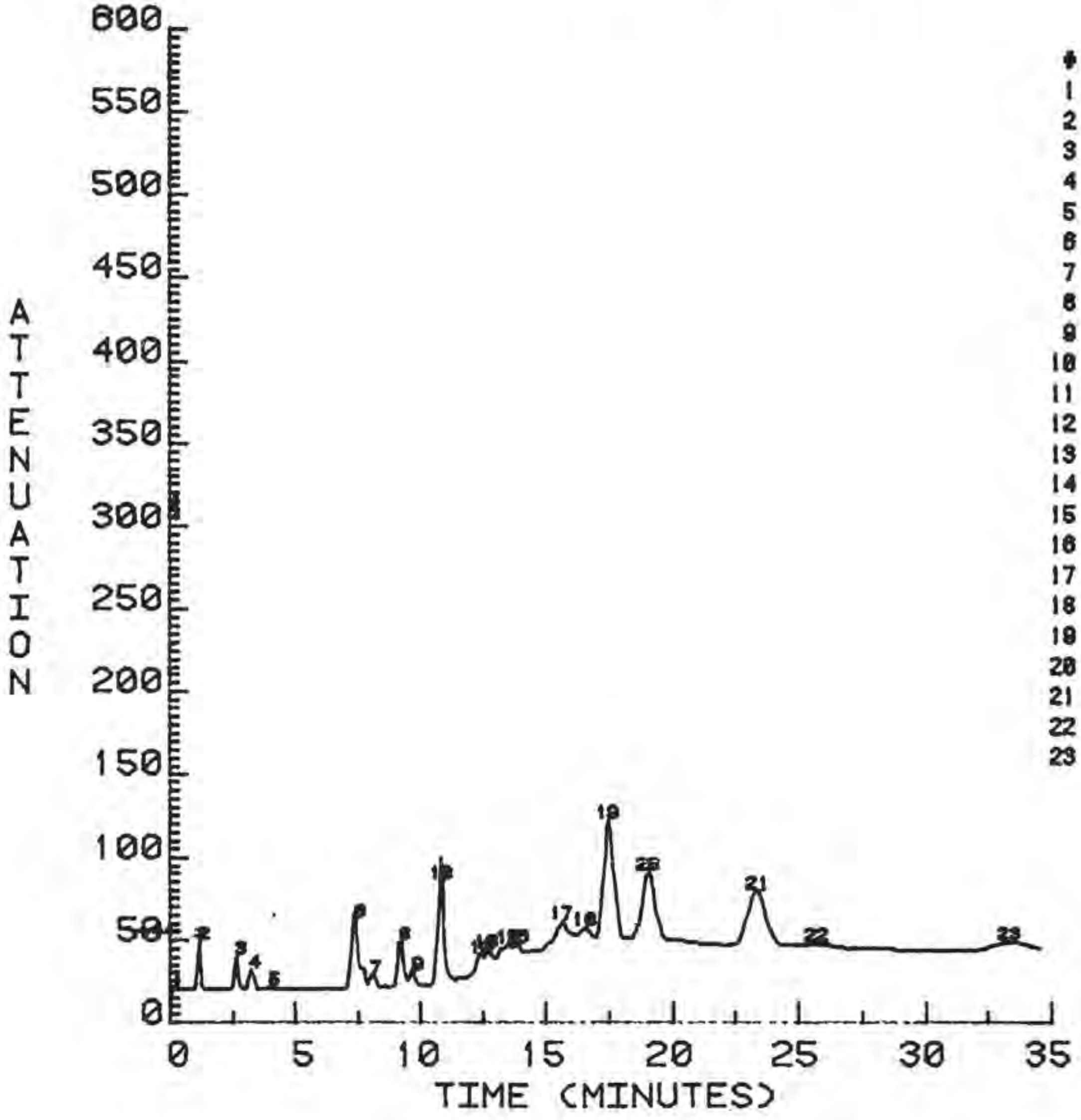
40 ml/min Tenax purge
30 ml/min analytical column flow

Injector 110° C
Interface 150° C

Temperature Program
80 - 220° C, 16°/min
4 min lag 32 min hold

FIGURE 14. GAS CHROMATOGRAPH CONFIGURATION

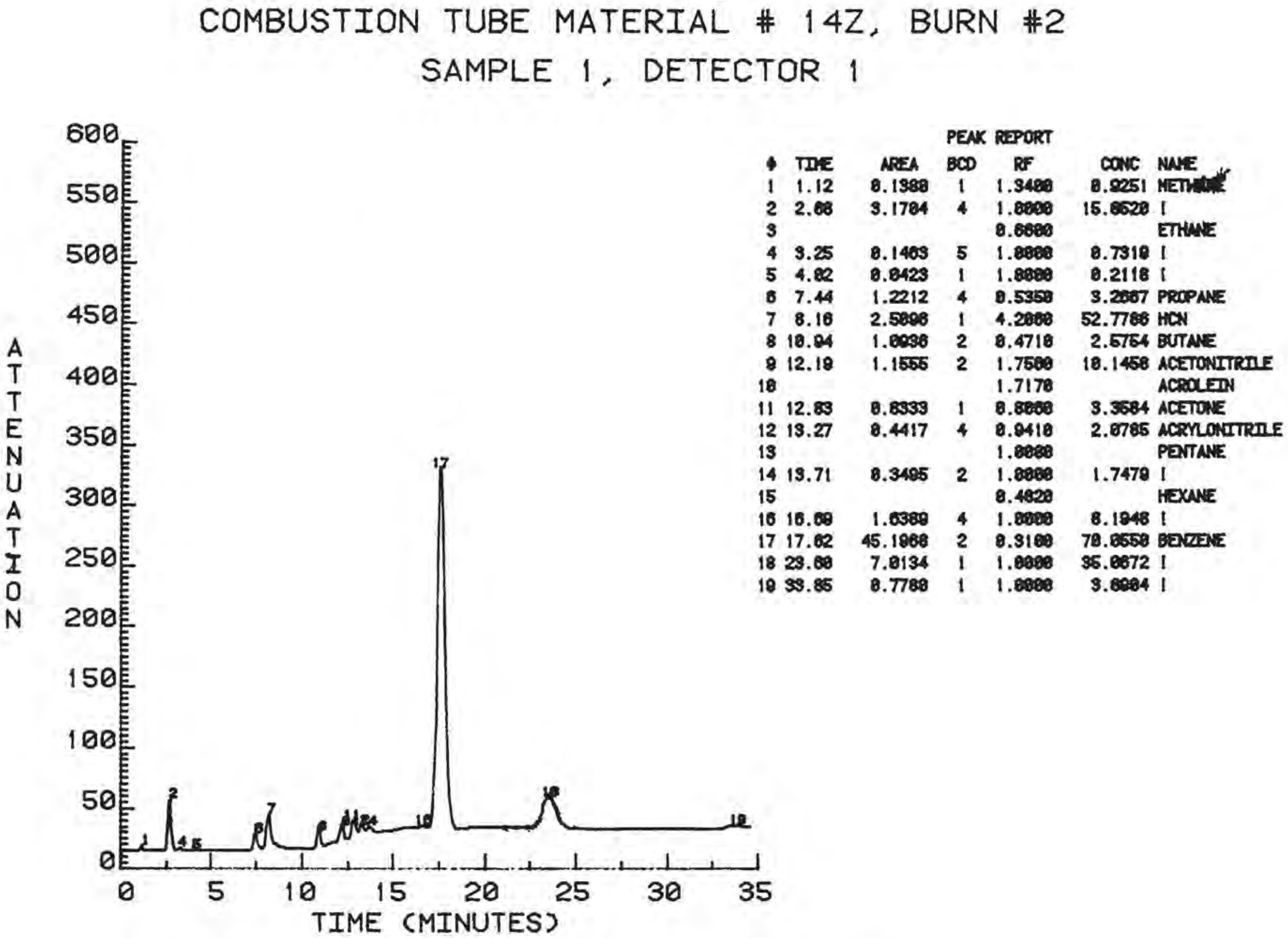
COMBUSTION TUBE MATERIAL # 6AZ, BURN #2
SAMPLE 1, DETECTOR 1



PEAK REPORT

#	TIME	AREA	BCD	RF	CONC	NAME
1	0.10	0.0140	1	1.0000	0.0701	I
2	1.13	0.8339	1	1.3400	5.5875	ME[illegible]
3	2.62	1.0385	2	1.0000	5.1926	I
4	3.23	0.6590	1	0.6600	2.1750	ETHANE
5	3.94	0.0467	1	1.0000	0.2335	I
6	7.35	5.0512	4	0.5350	13.5120	PROPANE
7	8.06	0.3740	6	4.5030	8.4359	HCN
8	9.10	2.3251	4	1.0000	11.6256	I
9	9.66	0.8002	1	1.0000	4.0014	I
10	10.60	6.1946	2	0.4710	14.5880	BUTANE
11				1.7560		ACETONITRILE
12	12.45	0.1758	4	1.7170	1.5094	ACROLEIN
13	12.75	0.5284	1	0.8060	2.1295	ACETONE
14				0.9410		ACRYLONITRILE
15	13.53	0.9791	4	0.4160	2.0367	PENTANE
16	13.67	0.6981	4	1.0000	3.4906	I
17	15.66	3.5632	4	1.0000	17.8160	I
18	16.56	1.8016	4	0.4820	4.3420	HEXANE
19	17.50	10.3977	1	0.3100	16.1165	BENZENE
20	19.07	7.6662	1	1.0000	38.3312	I
21	23.41	8.7046	4	1.0000	43.5232	I
22	25.67	0.5462	5	1.0000	2.7312	I
23	33.40	2.0273	1	1.0000	10.1365	I

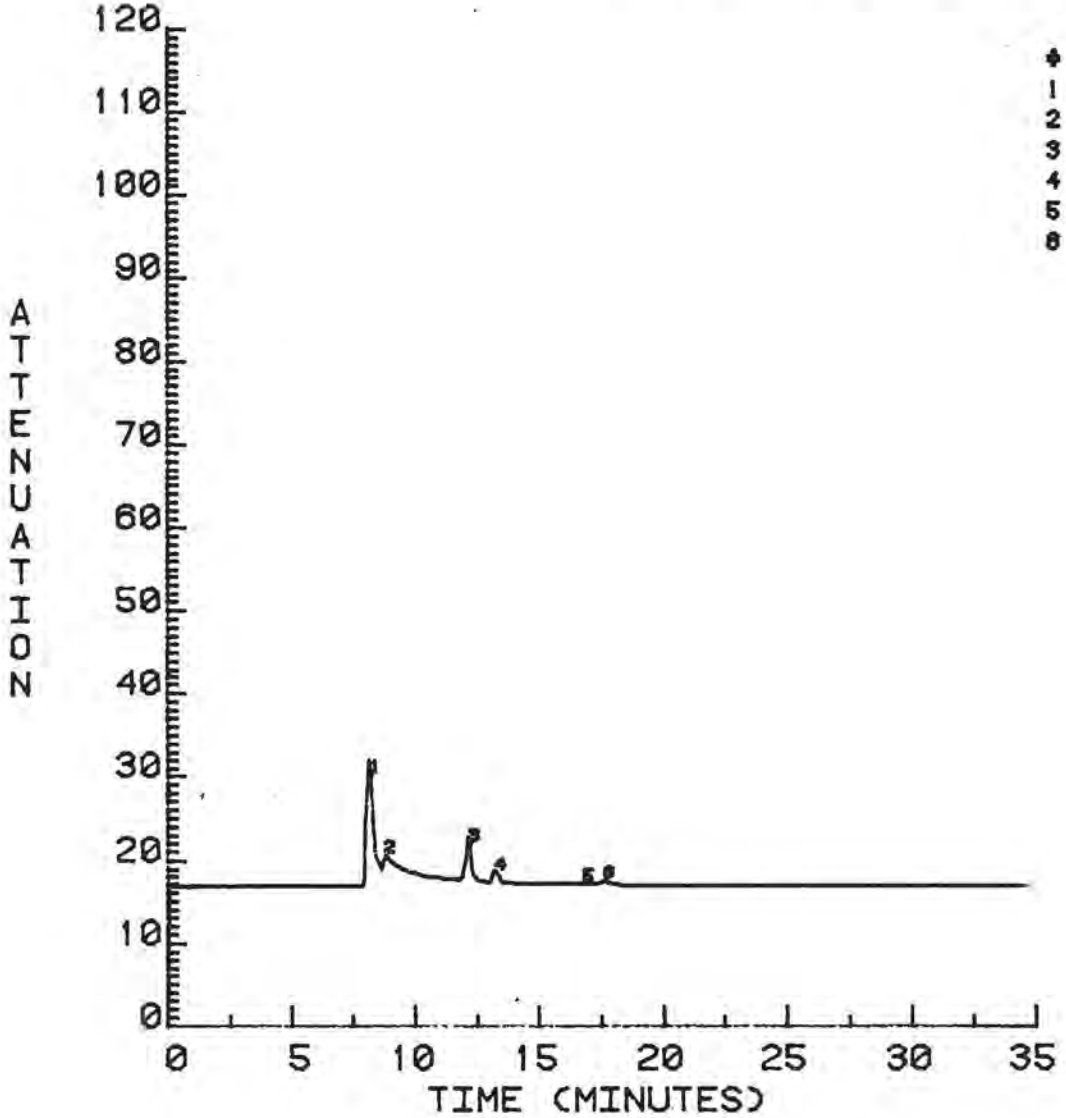
FIGURE 15. ORGANIC GAS FINGERPRINTS 6AZ, DETECTOR 1


COMBUSTION TUBE MATERIAL # 14Z, BURN #2
SAMPLE 1, DETECTOR 1

PEAK REPORT

#	TIME	AREA	BCD	RF	CONC	NAME
1	1.12	0.1380	1	1.3400	0.9251	METHANE
2	2.60	3.1704	4	1.0000	15.8520	!
3				0.6000		ETHANE
4	3.25	0.1463	5	1.0000	0.7310	!
5	4.02	0.0423	1	1.0000	0.2110	!
6	7.44	1.2212	4	0.5350	3.2667	PROPANE
7	8.16	2.5098	1	4.2000	52.7706	HCN
8	10.94	1.0936	2	0.4710	2.5754	BUTANE
9	12.10	1.1555	2	1.7500	10.1450	ACETONITRILE
10				1.7170		ACROLEIN
11	12.83	0.8333	1	0.8000	3.3584	ACETONE
12	13.27	0.4417	4	0.9410	2.0785	ACRYLONITRILE
13				1.0000		PENTANE
14	13.71	0.3495	2	1.0000	1.7479	!
15				0.4020		HEXANE
16	16.60	1.6389	4	1.0000	8.1948	!
17	17.62	45.1960	2	0.3100	70.0550	BENZENE
18	23.60	7.0134	1	1.0000	35.0672	!
19	33.85	0.7780	1	1.0000	3.8904	!

FIGURE 16. ORGANIC GAS FINGERPRINTS 14Z, DETECTOR 1

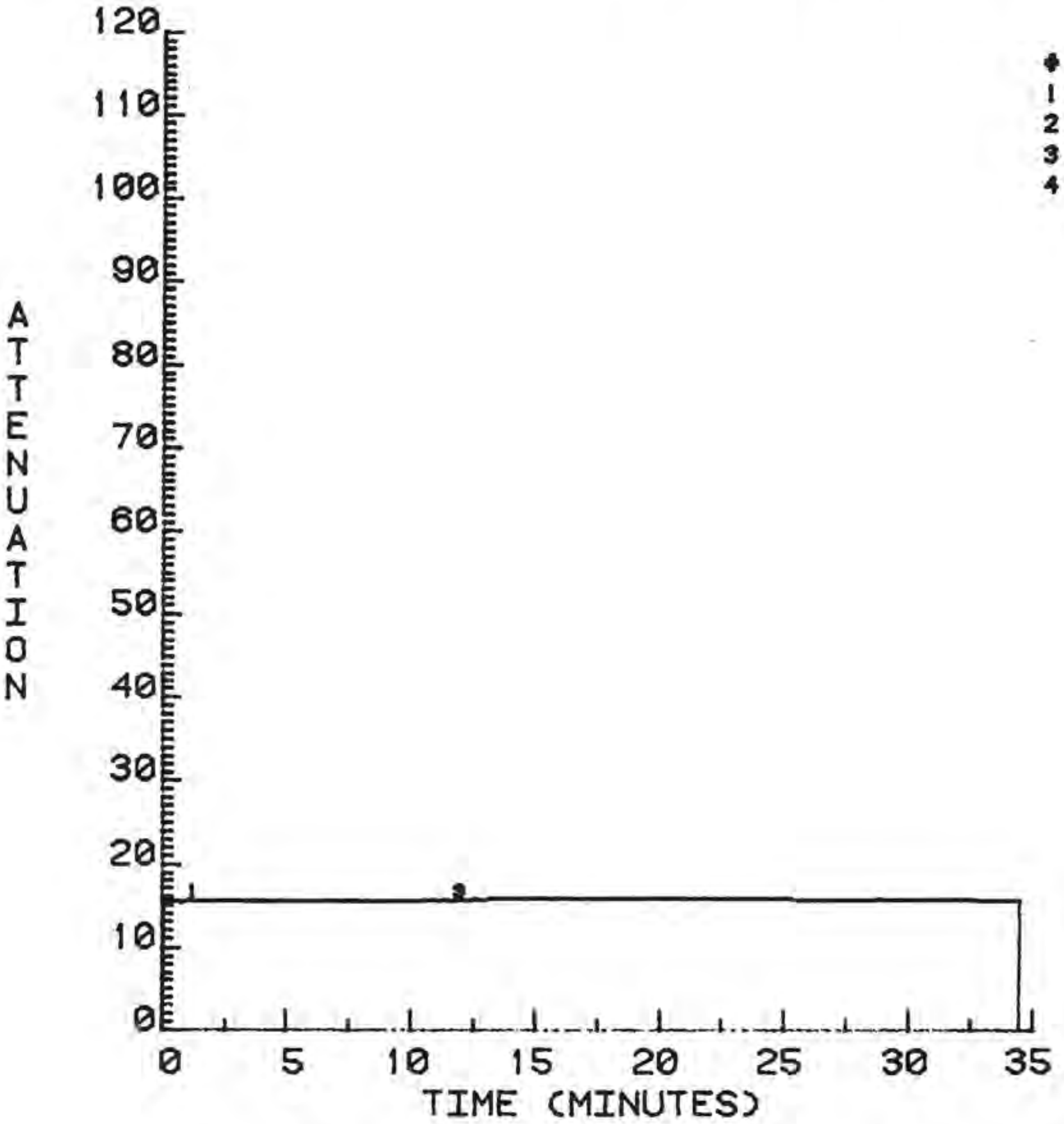


COMBUSTION TUBE MATERIAL # 14Z, BURN #2
SAMPLE 1, DETECTOR 2

```
                   PEAK REPORT
#  TIME     AREA   BCD    RF        CONC  NAME
1  8.17   1.3271    4   5.4410   36.1049  HCN
2  8.80   0.3212    1   1.0000    1.6062  I
3 12.20   0.4202    2   2.9350    6.1667  ACETONITRILE
4 13.28   0.1186    1   3.4650    2.0715  ACRYLONITRILE
5 16.70   0.0191    4   1.0000    0.0959  I
6 17.61   0.0703    1   1.0000    0.3517  I
```

FIGURE 17. ORGANIC GAS FINGERPRINTS 14Z, DETECTOR 2

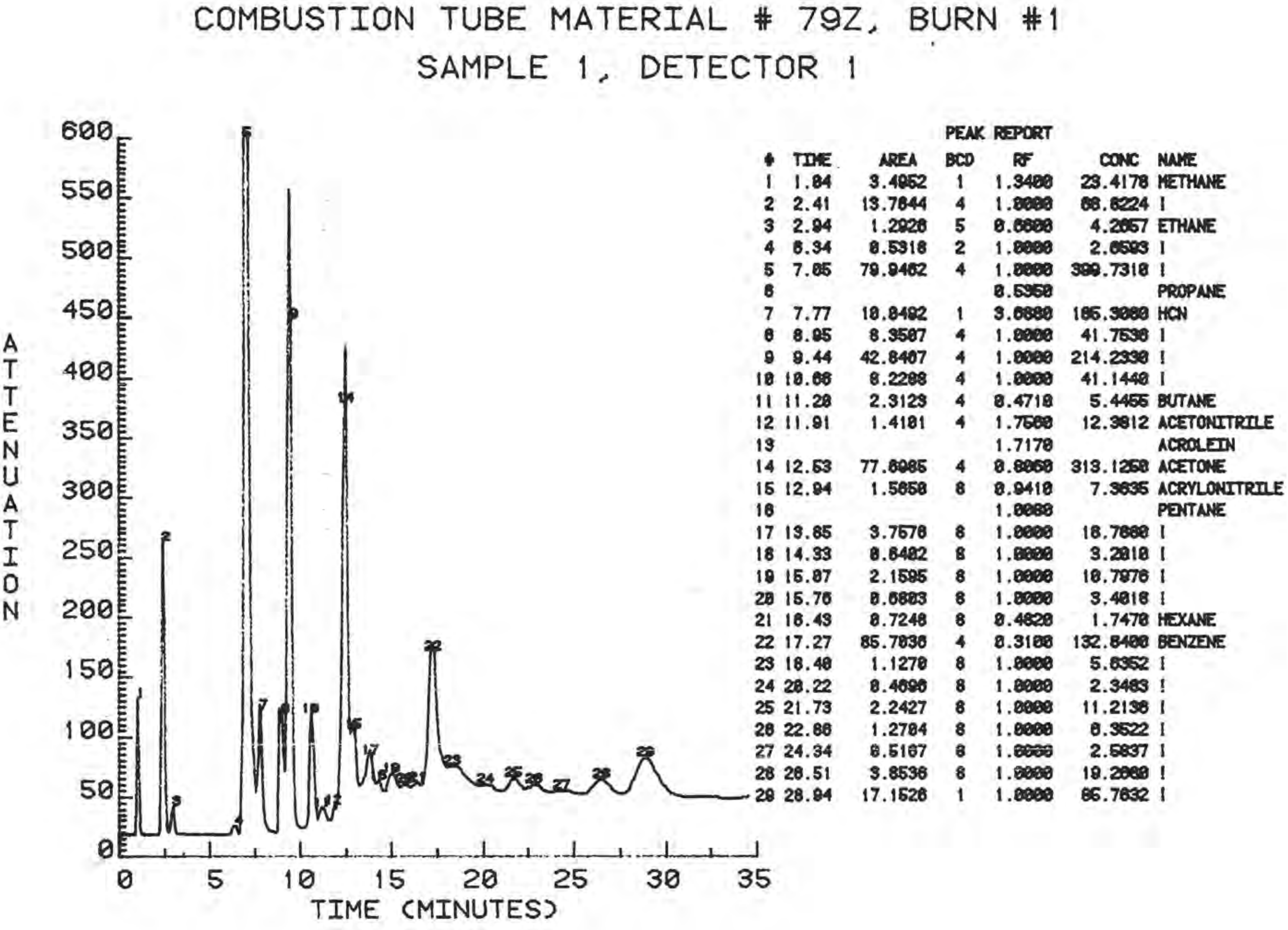
COMBUSTION TUBE MATERIAL # 32Z, BURN #1
SAMPLE 1, DETECTOR 2



PEAK REPORT

#	TIME	AREA	BCD	RF	CONC	NAME
1	1.03	0.0050	1	1.0000	0.0253	I
2	.			3.9440		HCN
3	11.78	0.0000	2	2.9360	0.1400	ACETONITRILE
4	.			3.4650		ACRYLONITRILE

FIGURE 18. ORGANIC GAS FINGERPRINTS 32Z, DETECTOR 2



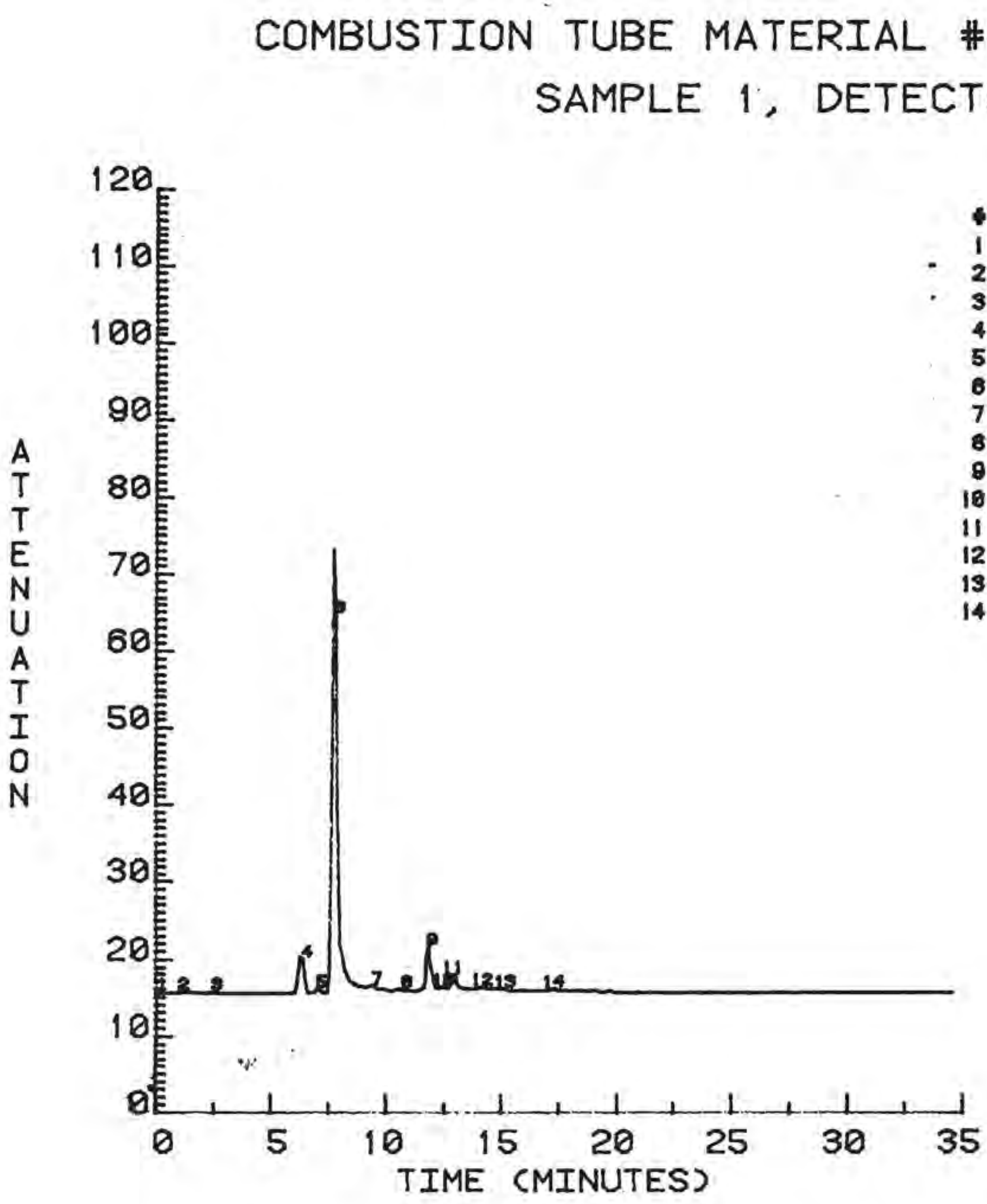
COMBUSTION TUBE MATERIAL # 79Z, BURN #1

SAMPLE 1, DETECTOR 1

PEAK REPORT

#	TIME	AREA	BCD	RF	CONC	NAME
1	1.04	3.4952	1	1.3400	23.4178	METHANE
2	2.41	13.7644	4	1.0000	68.8224	!
3	2.94	1.2926	5	0.6600	4.2657	ETHANE
4	6.34	0.5318	2	1.0000	2.6593	!
5	7.05	79.9462	4	1.0000	399.7310	!
6				0.5950		PROPANE
7	7.77	10.8492	1	3.6000	165.3000	HCN
8	8.95	8.3507	4	1.0000	41.7536	!
9	9.44	42.8467	4	1.0000	214.2330	!
10	10.66	8.2288	4	1.0000	41.1440	!
11	11.20	2.3123	4	0.4710	5.4455	BUTANE
12	11.91	1.4101	4	1.7560	12.3812	ACETONITRILE
13				1.7170		ACROLEIN
14	12.53	77.6985	4	0.8060	313.1250	ACETONE
15	12.94	1.5650	8	0.9410	7.3635	ACRYLONITRILE
16				1.0000		PENTANE
17	13.65	3.7576	8	1.0000	18.7880	!
18	14.33	0.6402	8	1.0000	3.2010	!
19	15.07	2.1595	8	1.0000	10.7976	!
20	15.76	0.6803	8	1.0000	3.4016	!
21	16.43	0.7248	8	0.4820	1.7470	HEXANE
22	17.27	85.7036	4	0.3100	132.8400	BENZENE
23	18.40	1.1270	8	1.0000	5.6352	!
24	20.22	0.4696	8	1.0000	2.3483	!
25	21.73	2.2427	8	1.0000	11.2136	!
26	22.86	1.2704	8	1.0000	6.3522	!
27	24.34	0.5167	8	1.0000	2.5837	!
28	26.51	3.8536	8	1.0000	19.2680	!
29	28.94	17.1526	1	1.0000	85.7632	!

FIGURE 19. ORGANIC GAS FINGERPRINTS 79Z, DETECTOR 1



PEAK REPORT

#	TIME	AREA	BCD	RF	CONC	NAME
1	0.11	0.0038	1	1.0000	0.0190	I
2	1.05	0.0043	1	1.0000	0.0216	I
3	2.42	0.0097	1	1.0000	0.0486	I
4	6.35	0.4094	2	1.0000	2.0473	I
5	7.07	0.0426	2	1.0000	0.2132	I
6	7.70	4.9427	4	3.3940	83.8780	HCN
7	9.42	0.0261	5	1.0000	0.1308	I
8	10.67	0.0155	2	1.0000	0.0777	I
9	11.91	0.4582	4	2.9358	6.7247	ACETONITRILE
10	12.54	0.0220	8	1.0000	0.1102	I
11	13.00	0.1048	1	3.4650	2.0710	ACRYLONITRILE
12	14.20	0.0232	2	1.0000	0.1164	I
13	15.17	0.0100	1	1.0000	0.0500	I
14	17.33	0.0284	1	1.0000	0.1424	I

FIGURE 20. ORGANIC GAS FINGERPRINTS 79Z, DETECTOR 2

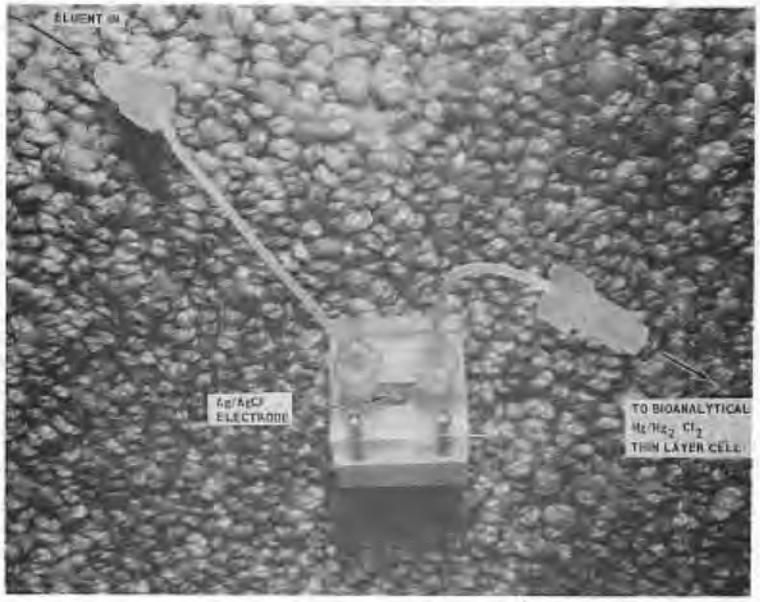


FIGURE 21. BIOANALYTICAL Ag/AgCl THIN LAYER CELL

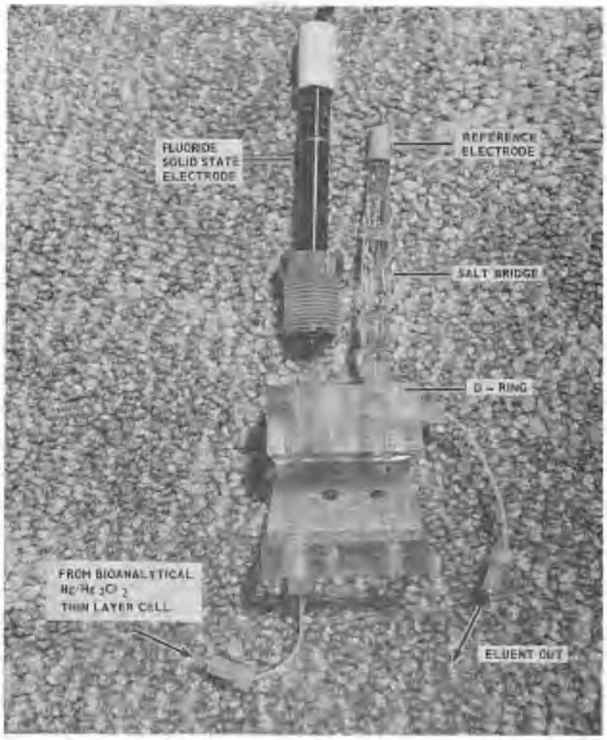


FIGURE 22. FLUORIDE THIN LAYER CELL

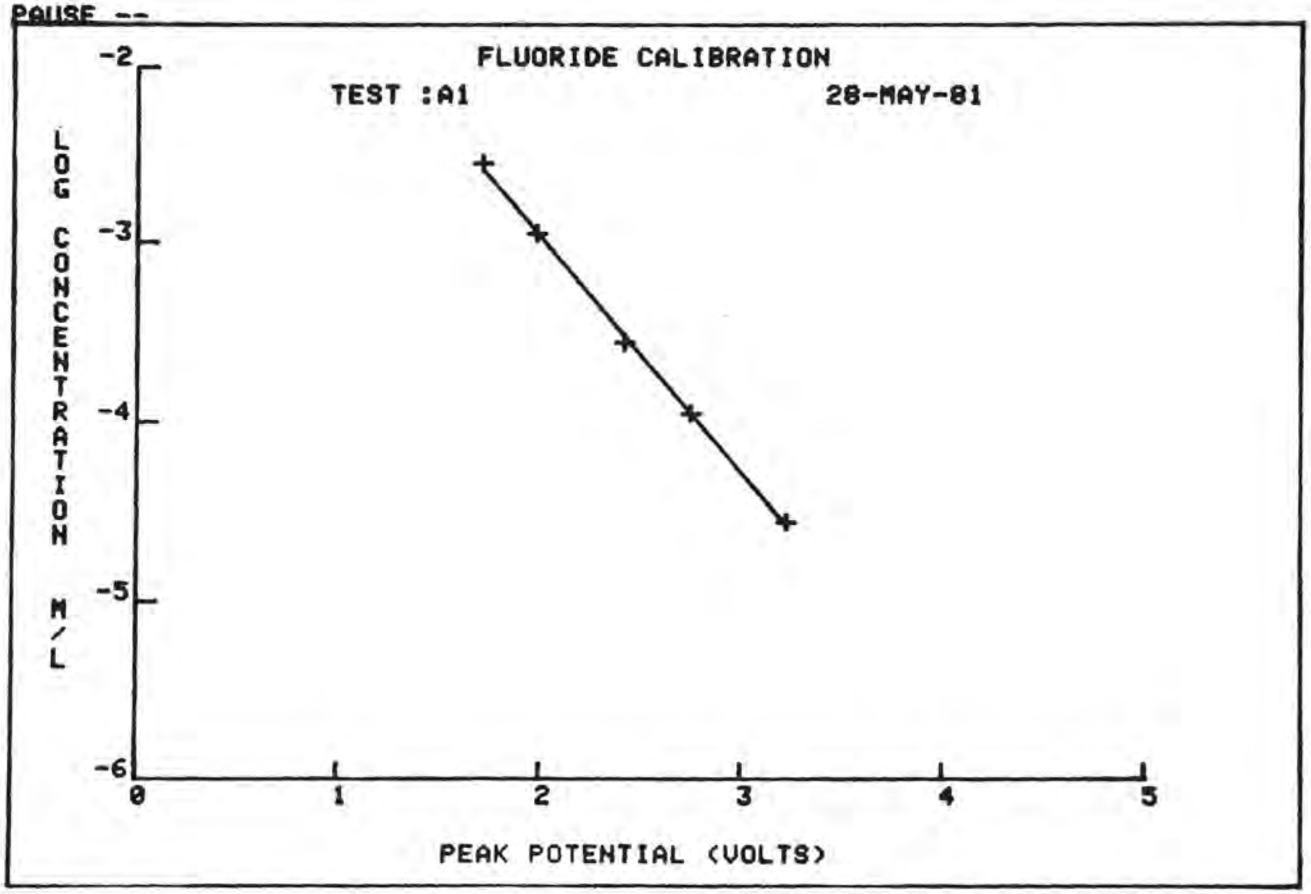


FIGURE 23. FLUORIDE CALIBRATION CURVE OBTAINED USING A FLUORIDE THIN LAYER CELL

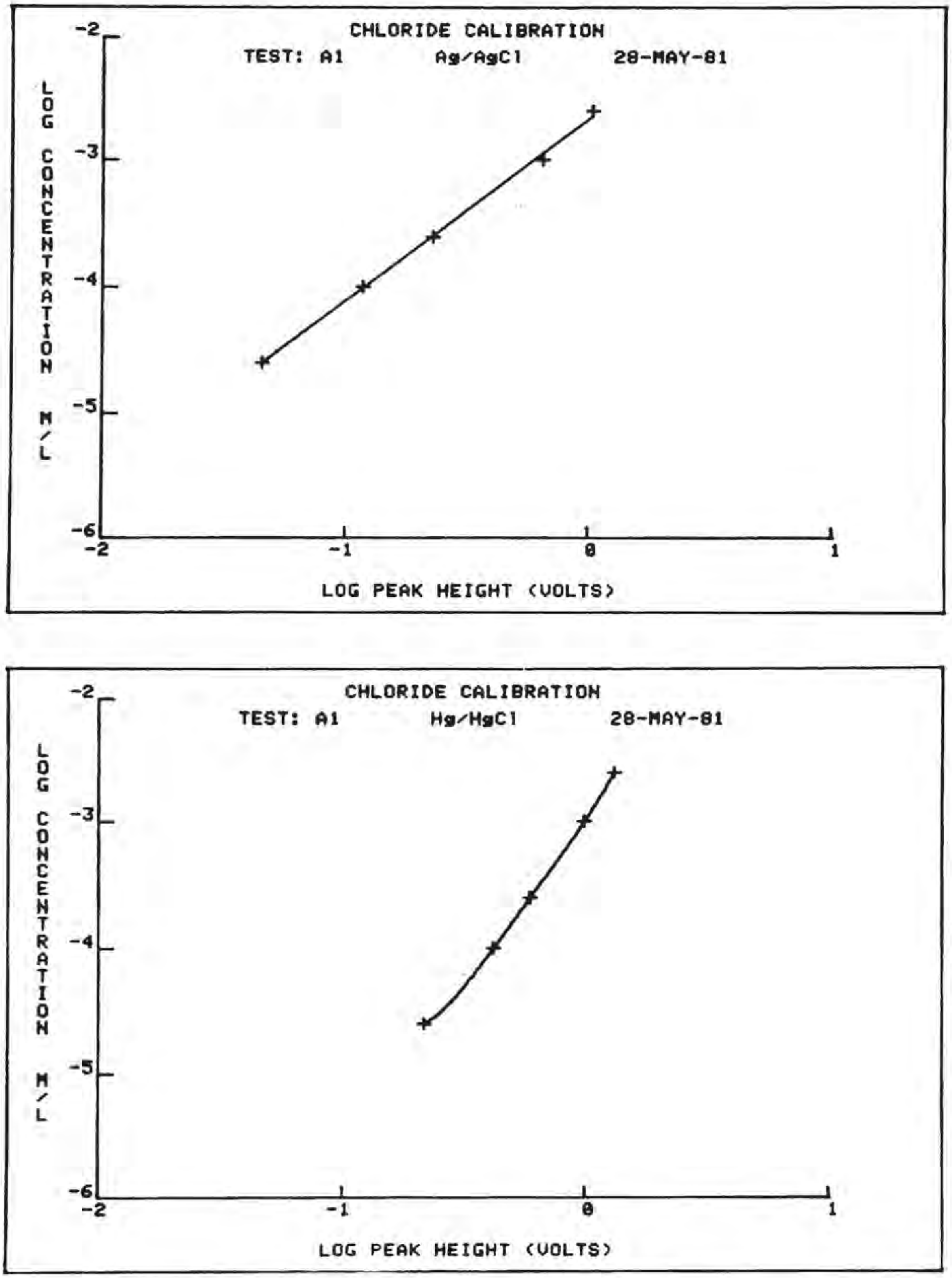


FIGURE 24. CHOLRIDE CALIBRATION

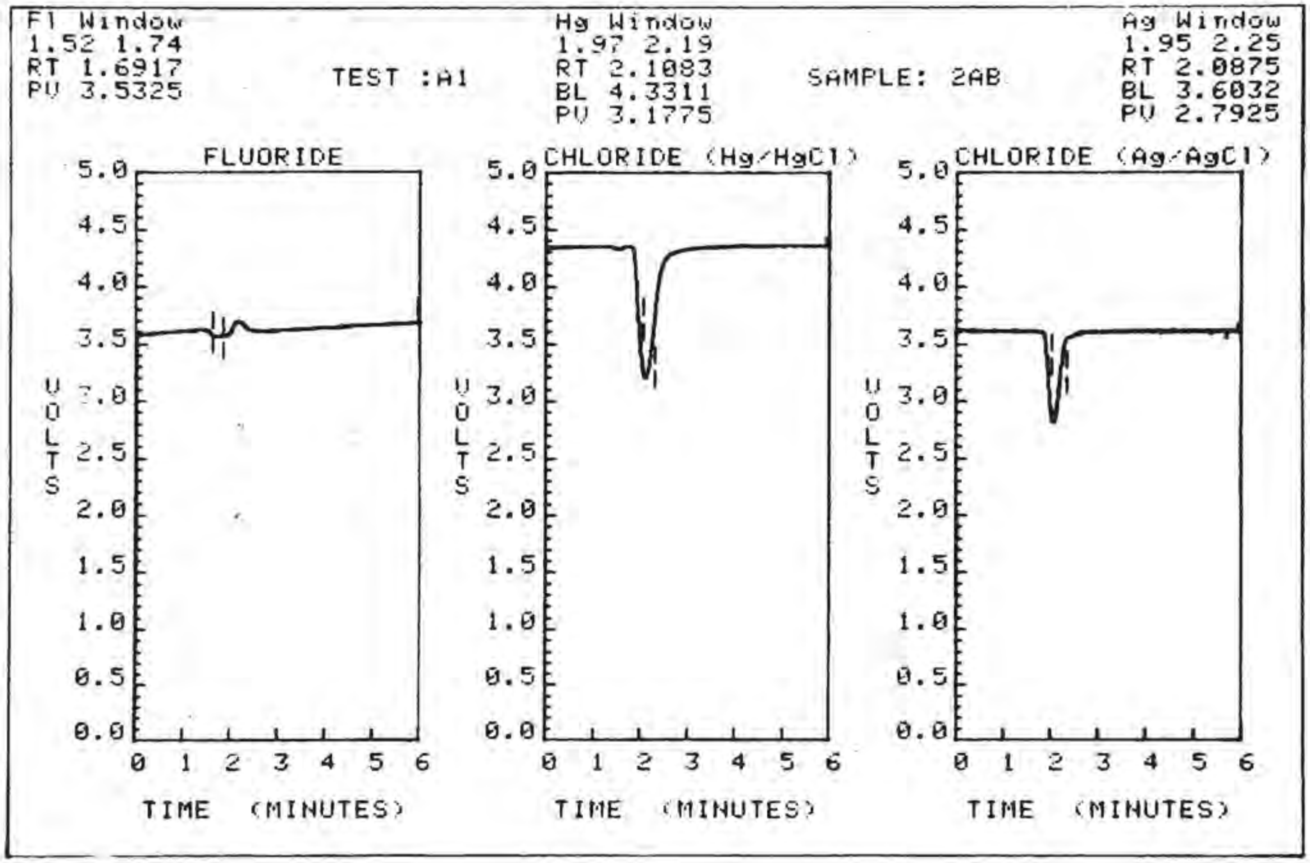


FIGURE 25. CHROMATOGRAMS OF COMBUSTION PRODUCTS OF A PLASTIC SHROUD (MATERIAL 100)

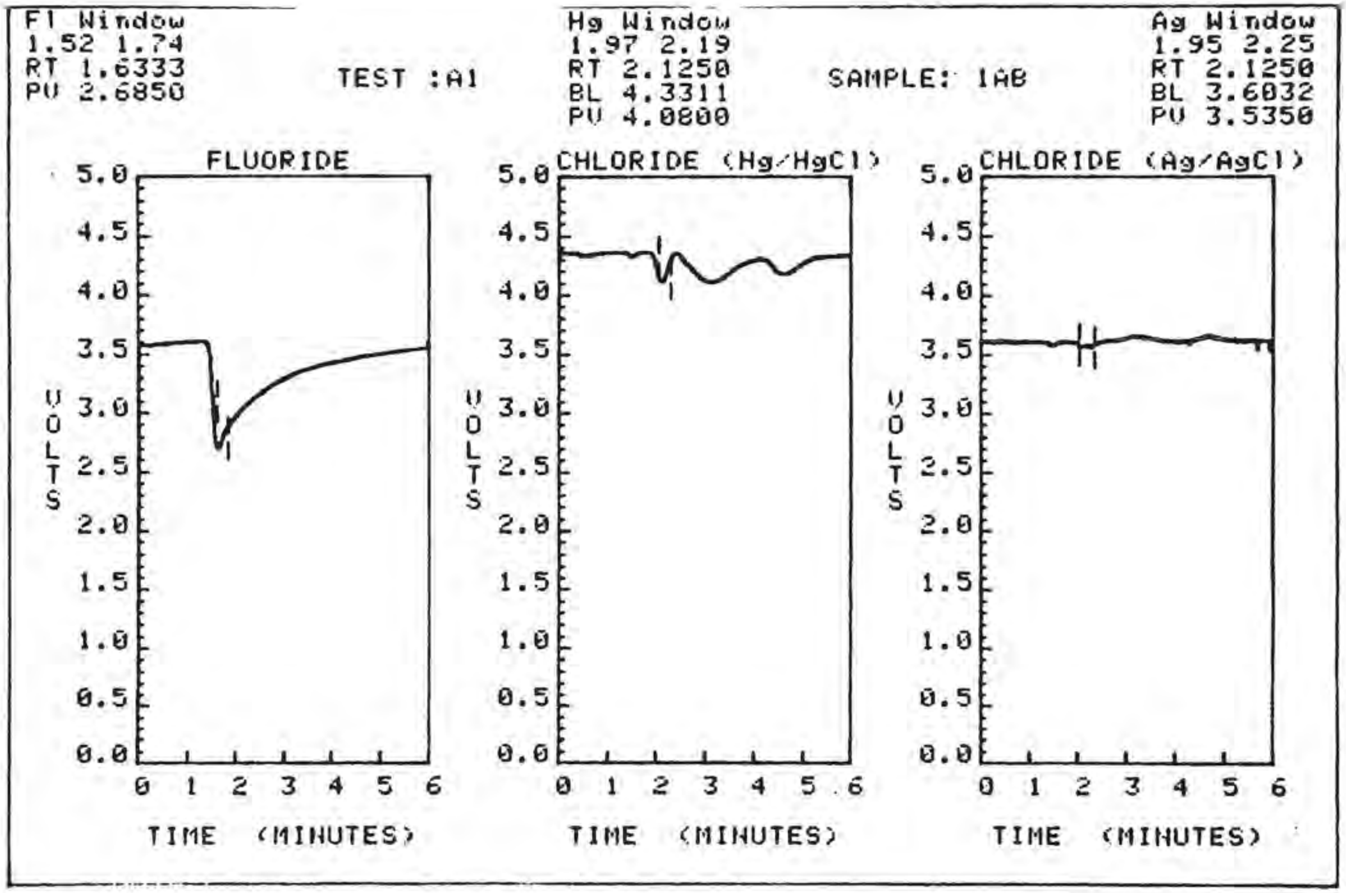


FIGURE 26. CHROMATOGRAMS OF COMBUSTION GASES FROM A PANEL (MATERIAL 14)

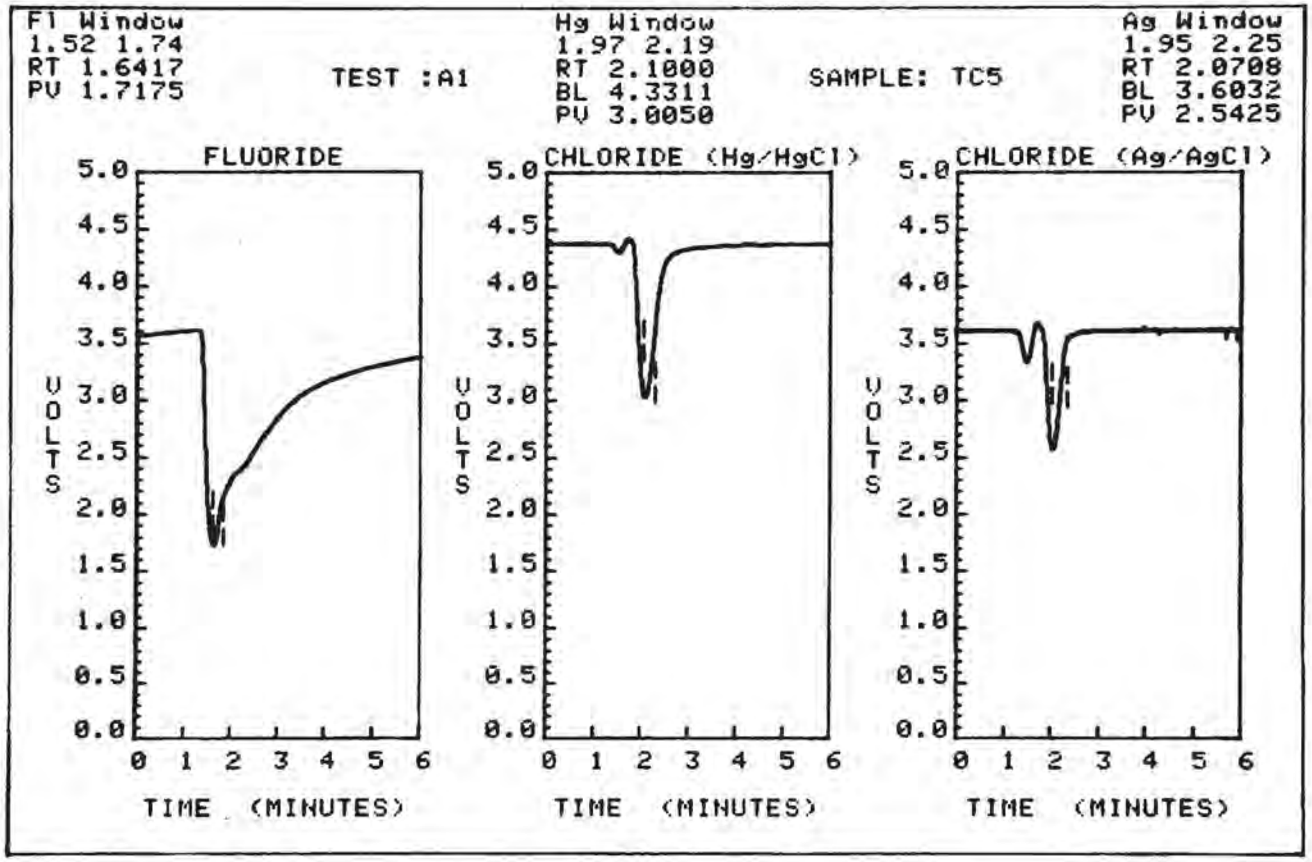


FIGURE 27. CHROMATOGRAMS OF A STANDARD OF FLUORIDE AND CHLORIDE

Combustion Tube Furnace Test #B-5

Burn Date: July 18, 1981
Analysis: July 18, 1981

SAMPLE NUMBER	SAMPLE NAME	WEIGHT MG	HYDROGEN FLUORIDE (F)				HYDROGEN CHLORIDE (AG/AGCL) (CL)				COLLECTING SOLN. VOL. MILLILITERS
			RETENTION TIME-MINS	PEAK POT. VOLTS	CONCENTRATION MOLES/LITER	YIELD MG/G	RETENTION TIME-MINS	PEAK POT. VOLTS	CONCENTRATION MOLES/LITER	YIELD MG/G	
1	CA1	250	1.68	3.242	0.2790E-04	0.9	2.07	3.480	0.2520E-04	1.5	100.0
2	CA2	250	1.66	2.625	0.1116E-03	3.6	2.05	3.447	0.1008E-03	5.9	100.0
3	CA3	250	1.63	2.187	0.2790E-03	8.9	2.05	3.330	0.2520E-03	14.7	100.0
4	CA4	250	1.63	1.668	0.1116E-02	35.7	2.05	2.970	0.1008E-02	58.8	100.0
5	CA5	250	1.65	1.335	0.2790E-02	89.3	2.05	2.597	0.2520E-02	147.0	100.0
6	BNK	250	1.55	3.445	0.1581E-04	0.3	1.95	3.485	0.1329E-04	0.4	50.0
7	078	248	1.72	3.418	0.1689E-04	0.3	2.08	2.900	0.1234E-02	36.3	50.0
8	096	252	1.73	3.273	0.2393E-04	1.9	2.07	2.895	0.1252E-02	181.1	250.0
9	100	254	1.64	3.447	0.1571E-04	1.2	2.07	2.900	0.1234E-02	177.2	250.0
10	127	253	1.63	3.482	0.1444E-04	1.1	2.07	2.800	0.1605E-02	231.3	250.0
11	06A	253	1.65	1.705	0.1036E-02	16.4	2.07	3.055	0.7694E-03	22.2	50.0
12	015	251	1.64	1.337	0.2505E-02	39.9	1.94	3.497	0.8662E-07	0.0	50.0
13	018	246	1.64	1.570	0.1432E-02	233.0	1.94	3.500	0.2933E-08	0.0	500.0
14	028	252	1.64	1.678	0.1106E-02	87.8	2.11	3.472	0.4502E-04	6.5	250.0
15	042	252	1.65	1.360	0.2373E-02	75.4	2.08	2.910	0.1200E-02	69.5	100.0
16	IC3	250	1.65	2.097	0.4031E-03	12.9	2.08	3.357	0.2180E-03	12.7	100.0
17	IC5	250	1.65	1.315	0.2644E-02	84.7	2.08	2.585	0.2583E-02*	150.7	100.0

COMBUSTION TUBE FURNACE TEST #B5

BURN DATE: 16-JUL-81
ANALYSIS DATE: 16-JUL-81

SAMPLE NUMBER	SAMPLE NAME	WEIGHT MG	HYDROGEN CHLORIDE (Hg/HgCl) (L)				HYDROGEN CHLORIDE (Ag/AgCl) (L)				COLLECTING SOLN. VOL. MILLILITERS
			RETENTION TIME-MINS	PEAK POT. VOLTS	CONCENTRATION MOLES/LITER	YIELD MG/G	RETENTION TIME-MINS	PEAK POT. VOLTS	CONCENTRATON MOLES/LITER	YIELD MG/G	
1	CA1	250	2.06	3.645	0.2520E-04	1.5	2.07	3.400	0.2520E-04	1.5	100.0
2	CA2	250	2.05	3.475	0.1008E-03	5.9	2.05	3.447	0.1008E-03	5.9	100.0
3	CA3	250	2.07	3.313	0.2520E-03	14.7	2.05	3.330	0.2520E-03	14.7	100.0
4	CA4	250	2.05	3.013	0.1008E-02	58.8	2.05	2.970	0.1008E-02	58.8	100.0
5	CA5	250	2.06	2.763	0.2520E-02	147.0	2.05	2.597	0.2520E-02	147.0	100.0
6	BNK	250	2.16	3.795	0.0000E+00	0.0	1.95	3.405	0.1329E-04	0.4	50.0
7	078	248	2.09	2.980	0.1148E-02	33.8	2.08	2.900	0.1234E-02	36.3	50.0
8	096	252	2.08	2.960	0.1242E-02	179.7	2.07	2.895	0.1252E-02	181.1	250.0
9	100	254	2.09	2.967	0.1206E-02	173.1	2.07	2.900	0.1234E-02	177.2	250.0
10	127	253	2.08	2.902	0.1545E-02	222.6	2.07	2.800	0.1605E-02	231.3	250.0
11	06A	253	2.09	3.055	0.8455E-03	24.4	2.07	3.055	0.7694E-03	22.2	50.0
12	015	251	2.13	3.637	0.2783E-04	0.8	1.94	3.497	0.8662E-07	0.0	50.0
13	018	246	2.15	3.725	0.1338E-05	0.4	1.94	3.500	0.2933E-08	0.0	500.0
14	028	252	2.10	3.472	0.1024E-03	14.8	2.11	3.472	0.4502E-04	6.5	250.0
15	042	252	2.10	2.950	0.1291E-02	74.7	2.08	2.910	0.1200E-02	69.5	100.0
16	IC3	250	2.10	3.215	0.4110E-03	24.0	2.08	3.357	0.2180E-03	12.7	100.0
17	IC5	250	2.08	2.212	0.2961E-02*	[illegible]	2.08	2.585	0.2583E-02*	150.7	100.0

FIGURE 28. TYPICAL REPORT OF HF AND HCL YIELDS

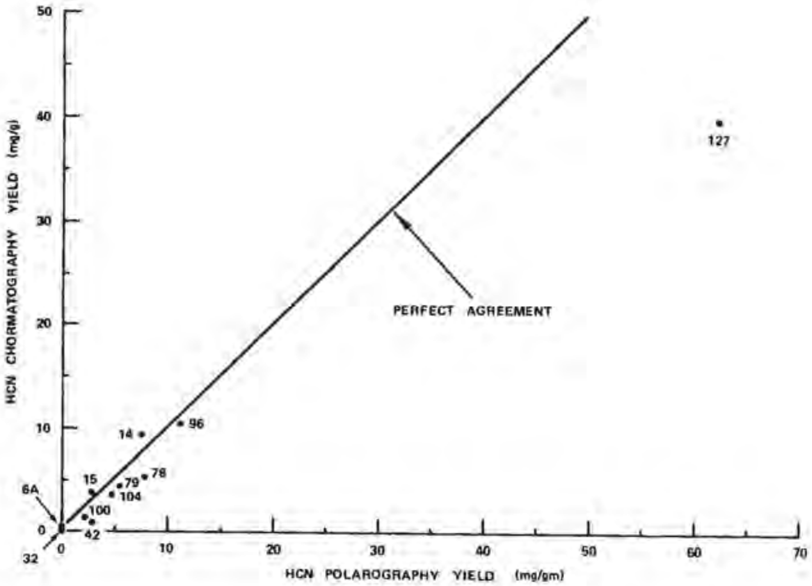


FIGURE 29. GAS CHROMATOGRAPHY/POLAROGRAPHY YIELD CORRELATION

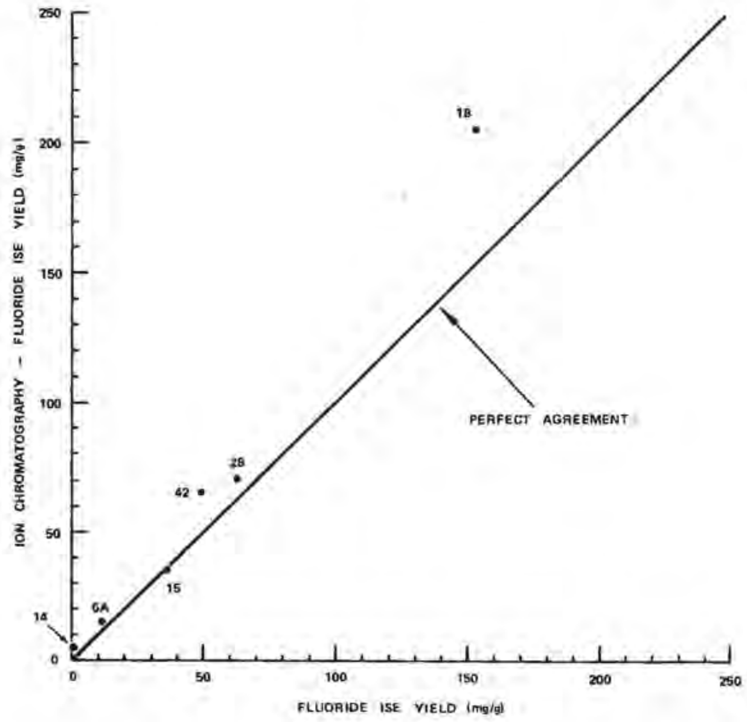


FIGURE 30. COMPARISON OF HF YIELDS OBTAINED USING ION CHROMATOGRAPHY WITH A FLUORIDE ISE WITH YIELDS OBTAINED USING A FLUORIDE ISE

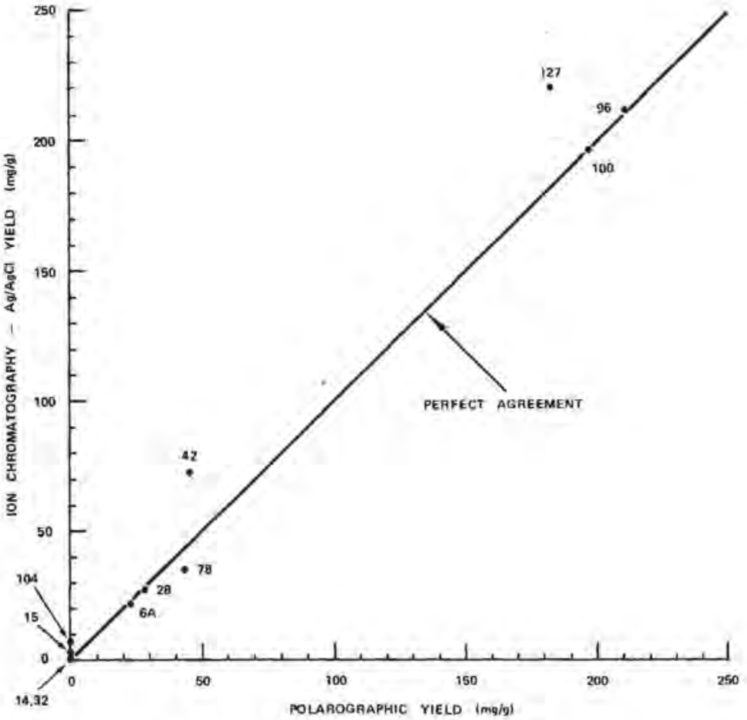


FIGURE 31. COMPARISON OF HCl YIELD OBTAINED USING ION CHROMATOGRAPHY (Ag/AgCl) WITH YIELDS OBTAINED USING POLAROGRAPHIC ANALYSIS

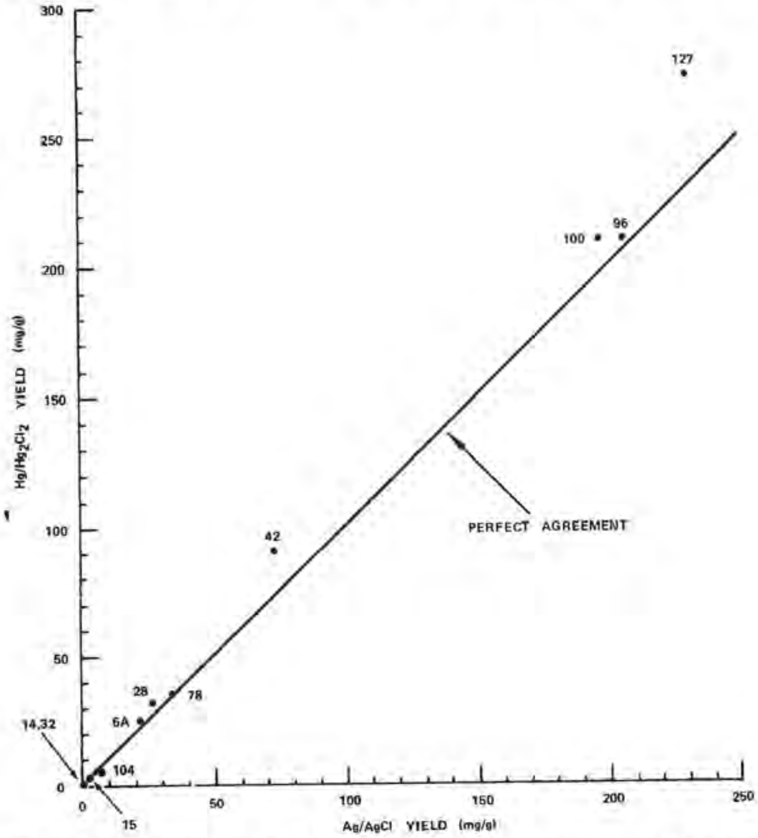


FIGURE 32. COMPARISON OF HCl YIELDS OBTAINED USING ION CHROMATOGRAPHY USING Hg/Hg_2Cl_2 AND Ag/AgCl ELECTRODES

APPENDIX A

TABLES

THIS APPENDIX SHOWS (a) LISTING OF MATERIALS, (b) YIELDS OF ORGANIC GASES, AND (c) COMPARISON OF ACID GASES.

TABLE A-1. MATERIALS DESCRIPTION

Sample Number	Cabin Use	Chemical Description
6A	Composite Panel	Polyvinyl Fluoride Aramid Honeycomb Phenolic Fiberglass
14	Composite Panel	Polyvinyl Fluoride Aramid Honeycomb Epoxy Fiberglass
15	Panel Face	Polyvinyl Fluoride (Face for 14)
18	Panel Component	A Clear Film of Polyvinyl Fluoride
28	Batt Cover	Polyvinyl Fluoride Aluminized Nylon Scrim
32	Molded Part	Polycarbonate
42	Acoustic Skin	Polyvinyl Fluoride
78	Fabric	Aramid
79	Seat Cushion	Fire Retarded Polyether Urethane
96	Fabric	Wool 49%, Polyvinyl Chloride 51% Fire Retarded
100	Plastic Shroud	Fire Retarded Polyvinyl Chloride
104	Seat Cushion	Fire Retarded Polyester Urethane
127	Drapery	Modacrylic

TABLE A-2. HYDROGEN CYANIDE ANALYSIS RESULTS

Gas Chromatograph and Polarograph (2) yields of hydrogen cyanide in milligrams per gram.

The GC yields are calculated from the nitrogen phosphorous detector. Three analyses are run on each of three test burns. The percent is relative standard deviation.

Sample No.	Burn 1 (%)	Burn 2 (%)	Burn 3 (%)	Yield %	Polarograph
6A	0.042 - 4.1	0.055 - 20.2	0.060 - 21.2	0.052 - 17.9	0
14	11.99 - 2.3	7.54 - 16.8	8.58 - 17.4	9.37 - 24.8	7.5
15	3.68 - 9.9	5.24 - 20.7	2.15 - 2.5	3.69 - 41.8	2.9
32	0	0	0	0	0
42	0.57 - 18.4	0.94 - 50.2	0.94 - 14.3	0.82 - 26.1	3.2
78	3.66 - 10.5	6.84 - 19.2	5.43 - 20.6	5.31 - 30	7.0
79	4.35 -	5.16 - 16.4	4.28 - 4.6	4.59 - 10.7	5.8
96	10.1 - 7.7	11.27 - 11.1	10.52 - 10.8	10.63 - 5.6	11.2
100	1.23 - 25.1	1.0 - 19.1	2.34 - 7.3	1.52 - 71.7	2.2
104	3.12 -	2.87 - 17.6	3.66 - 2.9	3.21 - 12.6	5.0
127	39.87 - 10.2	45.99 - 12.5	32.96 - 4.8	39.6 - 16.4	62.4

TABLE A-3. COMPARISON OF ACID GAS YIELDS

Material		Percent Wt. Loss	HF Yields (mg/g)	RSD (%)	HCl Yields (mg/g)	RSD (%)
6A	ISE/DPP	34.8	11.6	13.5	22.0	13.0
	IC1	31.1	15.4	15.0	22.8	14.7
	IC2				25.1	36.8
14	ISE/DPP	75.2	0.3	68	0	--
	IC1	76.6	4.4	57	1.5	68
	IC2				1.2	85
15	ISE/DPP	96.7	36.0	9.7	0	--
	IC1	95.2	35.7	12.7	2.7	56
	IC2				2.5	92
18	ISE/DPP	97.9	152.	12.4	0	--
	IC1	98.2	205	21.0	0	--
	IC2				0	--
28	ISE/DPP	97.1	63.1	5.0	27.7	4.1
	IC1	96.8	70.6	25.6	26.3	14.2
	IC2				31.7	8.0
32	ISE/DPP	97.0	--	--	0	--
	IC1	92.5	--	--	1.5	34.0
	IC2				1.5	14.0
42	ISE/DPP	77.7	48.8	14.5	45.2	4.7
	IC1	78.8	65.5	7.3	73.1	6.2
	IC2				90.5	12.4
78	ISE/DPP	90.7	--	--	43.1	23.2
	IC1	86.6	--	--	34.3	8.6
	IC2				36.5	12.3
79	ISE/DPP	98.9	--	--	0	--
	IC1	97.6	--	--	0	--
	IC2				0	--
96	ISE/DPP	94.8	--	--	205	13.8
	IC1	98.4	--	--	206	3.1
	IC2				211	13.8
100	ISE/DPP	90.4	--	--	197	10.7
	IC1	88.8	--	--	197	10.4
	IC2				210	21.3
104	ISE/DPP	97.3	--	--	0	--
	IC1	96.0	--	--	7.3	111.
	IC2				5.1	62.

127	ISE/DPP	97.6	--	--	182.	36.3
	IC1	98.4	--	--	231.	4.7
	IC2				273.	10.3

ISE/DPP = Solid state ISE for fluoride and DPP for chloride.

IC1 = IC with a Ag/AgCl electrode and a fluoride ISE.

IC2 = IC with an $Hg/Hg_2 Cl_2$ electrode

Reported yields are the average of three tests. One analysis is performed per test.

APPENDIX B

EQUIPMENT

APPENDIX B

EQUIPMENT

Tenney Mite Environmental Chamber Setting (ASTM D618 61 24 Hrs 23° C 50 percent R H)

Lindberg STB Combustion Tube Furnace

Leeds Northrup Potentiometer

Precision Scientific Wet Test Meter

Matheson Flowmeter #603

Beckman Oxygen Analyser OM 11

Beckman Model 861 Infrared Analysers

Tenax Polymer Adsorbent

Perkin Elmer 3920 Gas Chromatograph

Perkin Elmer N Detector

Chemical Data System 190 Probe

General Electric Hydrogen Generator

Perkin Elmer Recorder 56

Dionex System 10 Ion Chromatograph

Dionex Auto Ion™ System 12 Auto Sampler

Bioanalytical TL6 thin layer cell

Orion Solid State Fluoride Electrode

Tektronix 4631 Hard Copy Unit

Tektronix 4662 Interactive Digital Plotter

Tektronix 4051 Cathode Ray Tube (CRT)

Digital PDP 11/03 Computer

Digital Decwriter II

Perkin Elmer Sigma 10B Chromatograph Data System

PDP 11/03 Software Programs were developed in-house

COMPUTER SOFTWARE PROGRAMS FOR CHEM LAB AUTOMATION 3/17/81

1. GASANA

   - automates the collection of data from the burning of a material in a combustion tube furnace.

   - creates and controls data base of collected data.
   - supporting subroutines

     1) HELP
     2) LIST
     3) DIRECT
     4) COLECT
     5) READ
     6) GAPLOT

2. ION

   - automates the collection of data from an Ion Chromatograph separation hydrogen fluoride and hydrogen chloride
   - controls data base of collected data
   - allows access to data base
   - gives reports and plots of results of data analysis
   - supporting subprograms:

     1. IONCCL
     2. IONCCO
     3. ION3CL
     4. ION3CO
     5. IONRPT
     6. IONPLT

3. SIGTRN

   - automates the transfer of data from Sigma 1- microprocessor to DEC PDP-11/03
   - controls and provides access to data base of transferred data
   - generates reports
   - supporting subprograms:

     1. RDHEAD
     2. HDLIST
     3. TSLIST
     4. REPORT
     5. SIGFUL
     6. TSDATA
     7. PKTRNS
     8. FLCHNG
     9. SIGCTF
     10. SIGCOM
     11. ASCRL

    12. TTSUB
    13. HELP

4. GPPDRV
    - program to produce plots on CRT or flatbed plotter of x, y data stored on disks
    - used mainly to plot gas chromatograms
    - allows user to create data files via keyed in data
    - controls keyed in data base
    - supporting subroutines:

        1. HELP
        2. RDFLNM
        3. ADDATA
        4. LIST
        5. MODIFY

DOT/FAA
CT-82/13
The Pyrolysis toxic gas
analysis of aircraft interior
materials /
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