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EXPERIMENTAL RESEARCH IN THE USE OF ELECTRETS IN MEASURING EFFLUENTS FROM ROCKET EXHAUST AND A REVIEW OF STANDARD AIR QUALITY MEASURING DEVICES

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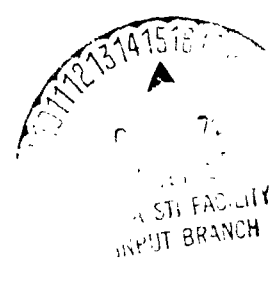
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16. ABSTRACT This report discusses seven standard types of measuring devices used to obtain chemical composition of rocket exhaust effluents. The electrets, a new measuring device developed by MSFC's Space Sciences Laboratory, is investigated and compared with established measuring techniques. The preliminary results obtained show that electrets have multipollutant measuring capabilities, simplicity of deployment, speed of assessment or analysis, and may be an important and valuable tool in measuring pollutants from space vehicle rocket exhaust.					
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OF STANDARD AIR QUALITY
MEASURING DEVICES

I. INTRODUCTION

The primary objective of this report is to demonstrate the feasibility of using electrets, a new measuring device developed by the Space Sciences Laboratory of Marshall Space Flight Center (MSFC), to measure the chemical composition of rocket exhaust from space vehicles. It is noted that there are over 50 measuring techniques presently available to measure atmospheric contamination. The instrumentation used operationally by the Booster Exhaust Study Test Team (BEST), which was composed of personnel from Langley Research Center (LaRC), Kennedy Space Center (KSC), and MSFC, during the sampling of the ground cloud from the Titan III C launches of Viking A and B missions to Mars from Cape Kennedy, Florida, on August 24 and September 9, 1975 is reviewed in this report. The types of instrumentation presented in this report for the measurement of gases are the chemiluminescent hydrogen chloride (HCl) analyzer which is based on light-emitting reaction of the monitored HCl gas with a gaseous reagent; the carbon monoxide (CO) detector, a nondispersive infrared (NDIR) instrument; and bubblers, a dosage type of measuring instrument. To measure particle sizes, a quartz crystal microbalance (mass monitor), an eight-stage Anderson impact filter and a Nuclepore filter were used. A millipore filter, which measures HCl, will also be reported on because of its use during the 6.4 percent model testing of the Space Shuttle booster (solid rocket booster, SRB) at MSFC Test Facility 116. There is a general complication with all these types of instrumentation, i.e., a lack of multipollutant capabilities or a need for the development of a method of high sensitivity, simplicity, and speed of measurement, and for multipollutant capabilities. The electrets can overcome this complication. The experiments with the electrets have shown that they have unique properties that are useful in practical exhaust studies.

II. ELECTRETS

A. Background

Electrets have been known since the latter part of the 19th century. A substantial amount of work has been done on electrets, and a few practical applications, particularly in the area of communications, have been made. In general, however, only qualitative descriptions of the various processes are known.

The particles in tests that have been conducted have either a positive or negative charge and are attracted to the electret's surface. These collected particles or ions on the electrets are then analyzed by taking the mass spectra of the sample.

B. Preparation of Electrets

Electrets have been made in a variety of ways, but the method used most often has been to make "thermoelectrets." A thermoelectret is made by placing a dielectric material between two electrodes, heating the dielectric material to near its softening point, and applying a high direct current field to the material between electrodes. The field is maintained while the dielectric is cooled to room temperature, and the electric field is removed. If the treated dielectric exhibits electric polarization, it is called an electret [1].

It has been found that thermoelectrets of polymers with stable surface charges on either side are suitable devices for attracting charged particles and ions to their surface. Therefore, it was decided to use these electrets for effectively measuring charged gases, vapors, or particles in the atmosphere. The thermoelectret characteristics of polytetrafluorethylene (Teflon) from experimental studies were analyzed by collecting vapors of xylene and acetone under different experimental conditions. Thermoelectrets were prepared by keeping a Teflon foil of 0.0125 cm thickness between two aluminum electrodes. Further details are in Reference 2. Mass spectra of the unexposed electret and the exposed electret were taken by using the Varian M-66 cycloidal mass spectrometer. The experiment results, on the whole, indicated that even small quantities of the gases give a characteristic mass spectra and the peak height of the mass spectra can be considered as a measure of the quantity of the material collected over the surface of the electret (see Section C for the type of material collected).

C. The Use of Electrets to Measure Effluents from Solid Rocket Boosters

The most widely used propellant in solid rocket motors is ammonium perchlorate as the oxidizer with powdered aluminum filler that acts in part as a fuel and partially as a stabilizer to control the burning rate. The exhaust products from this type of fuel contain hydrogen chloride (HCl), aluminum oxide (Al_2O_3), and water (H_2O). To assess the impact of these products in the atmosphere, it is necessary to know not only their quantity but also their distribution in the ground cloud that develops at the launch site after a rocket firing. This report discusses the use of electrets to measure the effluents.

D. Experimental Results from Electrets During Test Firing of 6.4 Percent Scale Model of Space Shuttle Solid Rocket Booster and Liquid Engine at MSFC Test Facility 116

Rocket exhaust effluent transport and chemical kinetic measurements were made using several air quality measuring devices and were compared to electret measurements during the Space Shuttle acoustic model tests at MSFC. A 6.4 percent scale model of the Space Shuttle SRB and the liquid engine of the Space Shuttle main engine (SSME) were test fired.

Qualitative results from the tests have been obtained by sampling the rocket exhausts using electrets and comparing these to results obtained using the chemiluminescent HCl analyzer, bubbler, and a millipore filter. Scanning electron microscopic (SEM) and X-ray microprobe analyses have been used to identify the various effluents, the most important being HCl.

Table 1 lists results obtained from the experiments and from various HCl measuring instruments. Concentrations obtained using the chemiluminescent HCl analyzer from the USAF School of Medicine, Brooks AFB, Texas, and bubblers and millipore filters from Arnold Engineering Development Center, Tullahoma, Tennessee, were compared to concentrations obtained simultaneously from the electret studies.

TABLE 1. COMPARISON OF HCl CONCENTRATION MEASUREMENTS USING VARIOUS INSTRUMENTS TO ELECTRET MEASUREMENTS DURING A 6.4 PERCENT SCALE MODEL TEST AT MSFC TEST FACILITY 116

Position (P)	Angle (degrees)/ Distance (m) (Measurement from True North)	Instrumentation	HCl Measurement (ppm)	Measurement Obtained Directly from Electret Counts (Calibrated Values) ^b	Estimated Values (ppm)
1	85/75	Millipore Filter and Electret	21.1 ^a	9 682 ^a	22.65
2	30/75	Electret	30.32 (See Estimated Values)	12 961	30.32
3	75/75	Bubbler	31.3	13 375 ^b	31.30
4	75/70	Electret	37.73 (See Estimated Values)	16 125	37.73
5	50/75	Millipore Filter	45.8	19 572 ^b	45.79
6	45/75	Millipore Filter	55.0	23 503 ^b	54.99
7	65/75	Chemiluminescent	65.4	27 948 ^b	65.39
8	60/75	Bubbler and Electret	66.2 ^a	26 204 ^a	61.31

a. Direct comparison made between millipore filter or bubbler and electret.

b. Calibrated Value (427.35 counts = 1 ppm).

As shown in Table 1, direct comparisons were made at positions one and eight of HCl measurements made with a millipore filter and a bubbler. The electrets were kept beside the above instruments to calibrate the pollution collected on the electret surface. For example, at position one, approximately 75 m from the flame trench and 85° from true north, the millipore filter measured 21.1 ppm. At the same angle and distance, the electret measurement from the SEM and X-ray microprobe analysis gave 9682 counts. Also, at position eight, the bubbler measured 66.2 ppm, and 26 204 counts were obtained from the SEM and X-ray microscope analysis. From the above measurements, a calibration of the counts from the electrets against the instrumentation (millipore filter and chemiluminescent analyzer) was obtained. This was found to be 427.35 counts equal 1 ppm. It may be noted that to obtain consistent results, the SEM analyses of the electrets were done under identical conditions. Also, the exhaust cloud passage time over the test instruments was approximately 10 s, which was determined from photographs taken during the test.

Figure 1 illustrates a summary of the test data and a calibration curve of HCl concentrations (ppm) from various instruments such as millipore filter, bubbler, and chemiluminescent HCl analyzer with the electrets.

In addition to the results in Figure 1, Figure 2 shows the distribution of concentrations of the pollution from the test site at various angles for a constant distance. It appears that when a dense flow of air from the rocket exhaust passes an electret that is adjacent to a millipore filter, bubbler, etc., the reading is high; and when a less dense flow of air from the rocket exhaust passes, a lower measurement of concentration is obtained. Figure 3 is a plot similar to that of Figure 1 but shows the dosage (ppm-s) versus electret electron counts.

E. Experimental Results Obtained During Chamber Testing of the Electrets at Arnold Engineering Development Center

The chamber used for testing at Arnold Engineering Development Center is a 3 ft³ chamber made of plexiglass. The fuel is ignited by triggering an electrically heated nichrome wire. The nichrome wire ignites the solid fuel which then burns for approximately 1 s. The holder of the millipore filter and electrets is placed into the chamber after the firing sequence. Exposure times

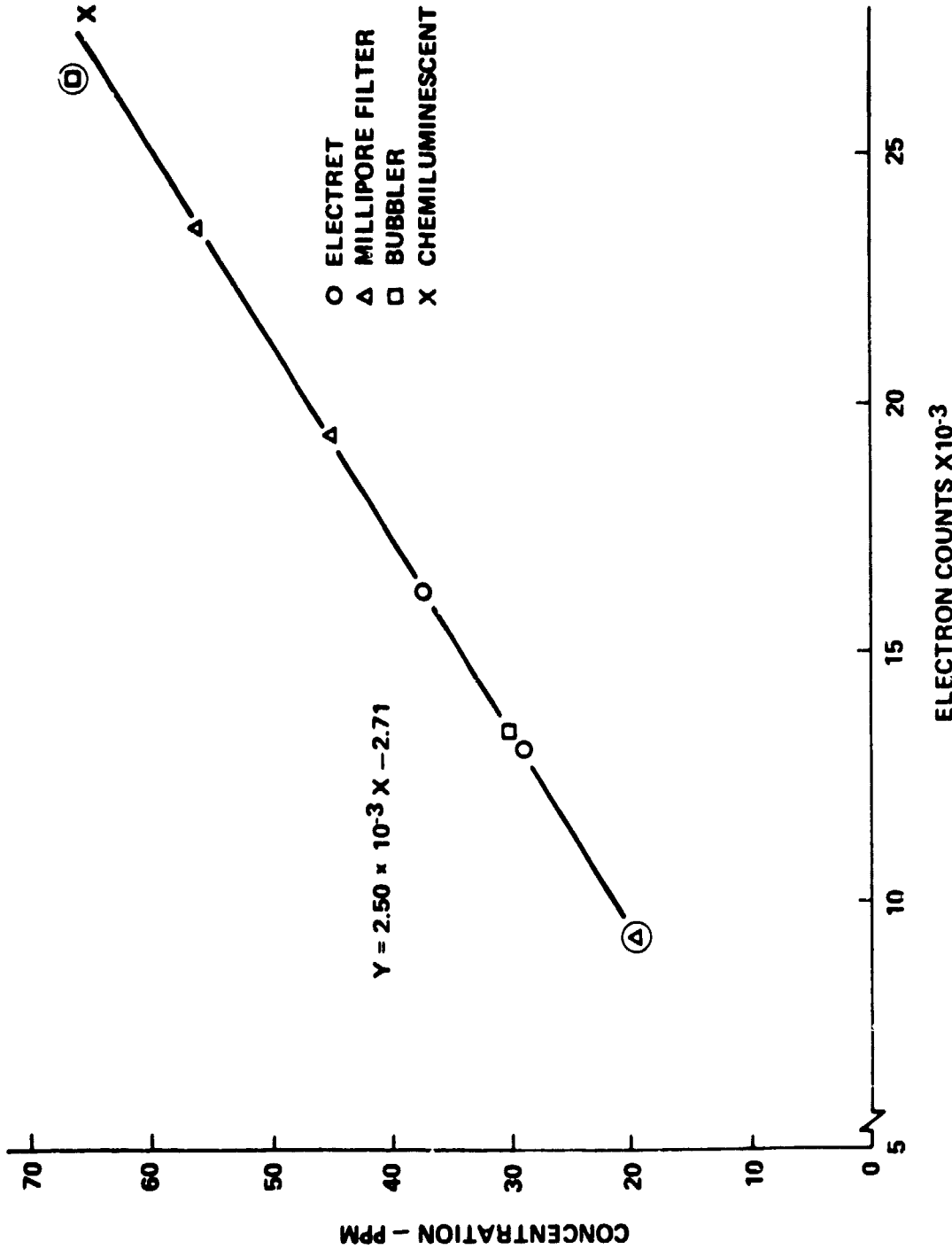


Figure 1. Summary of test data and a calibration curve of HCl concentrations (ppm) from various instruments to electret electron counts during 6.4 percent SRB tests at MSFC.

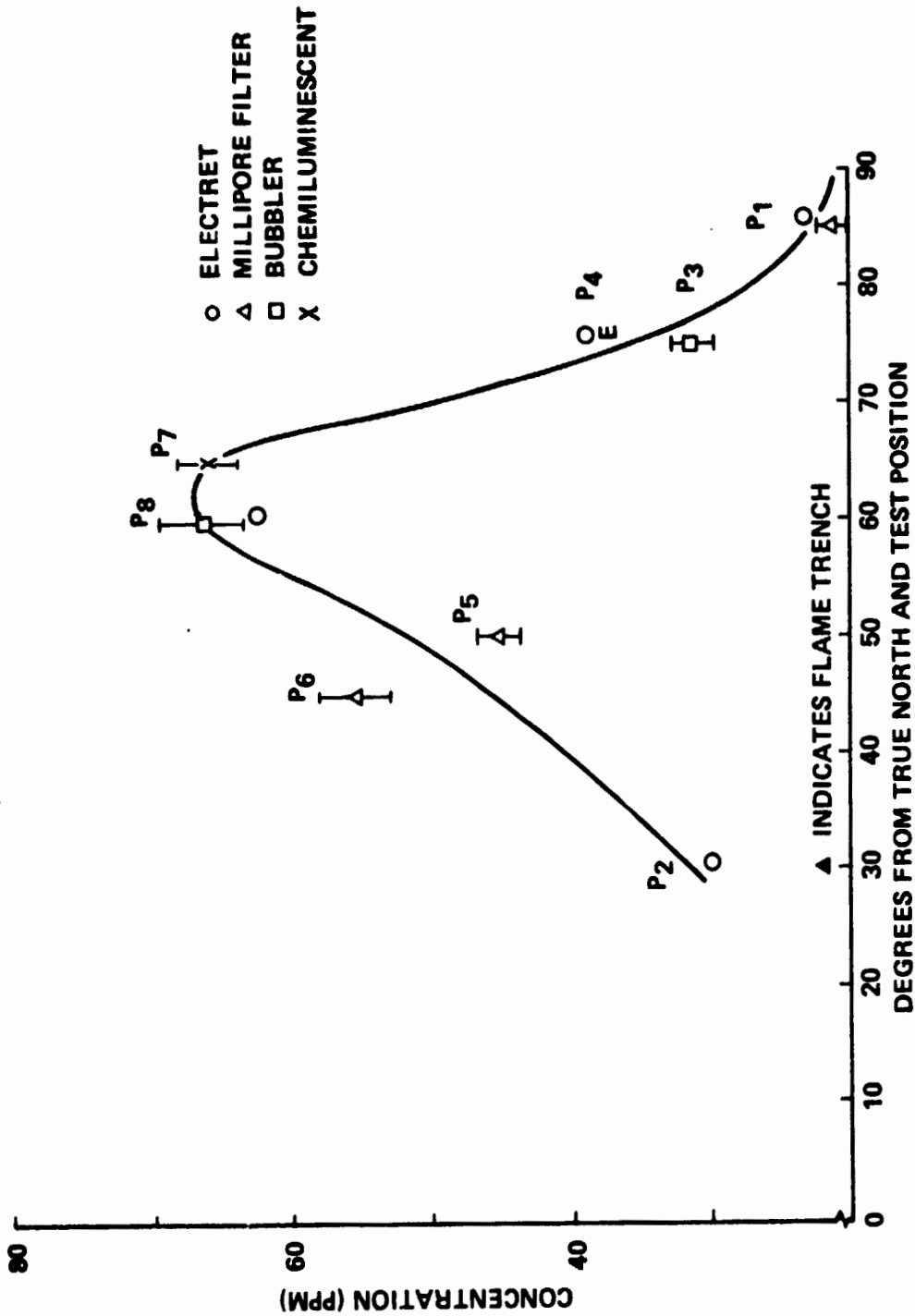


Figure 2. Measured distribution of rocket effluents (ppm) versus degrees from true north during 6.4 percent scale model testing at MSFC Test Facility 116.

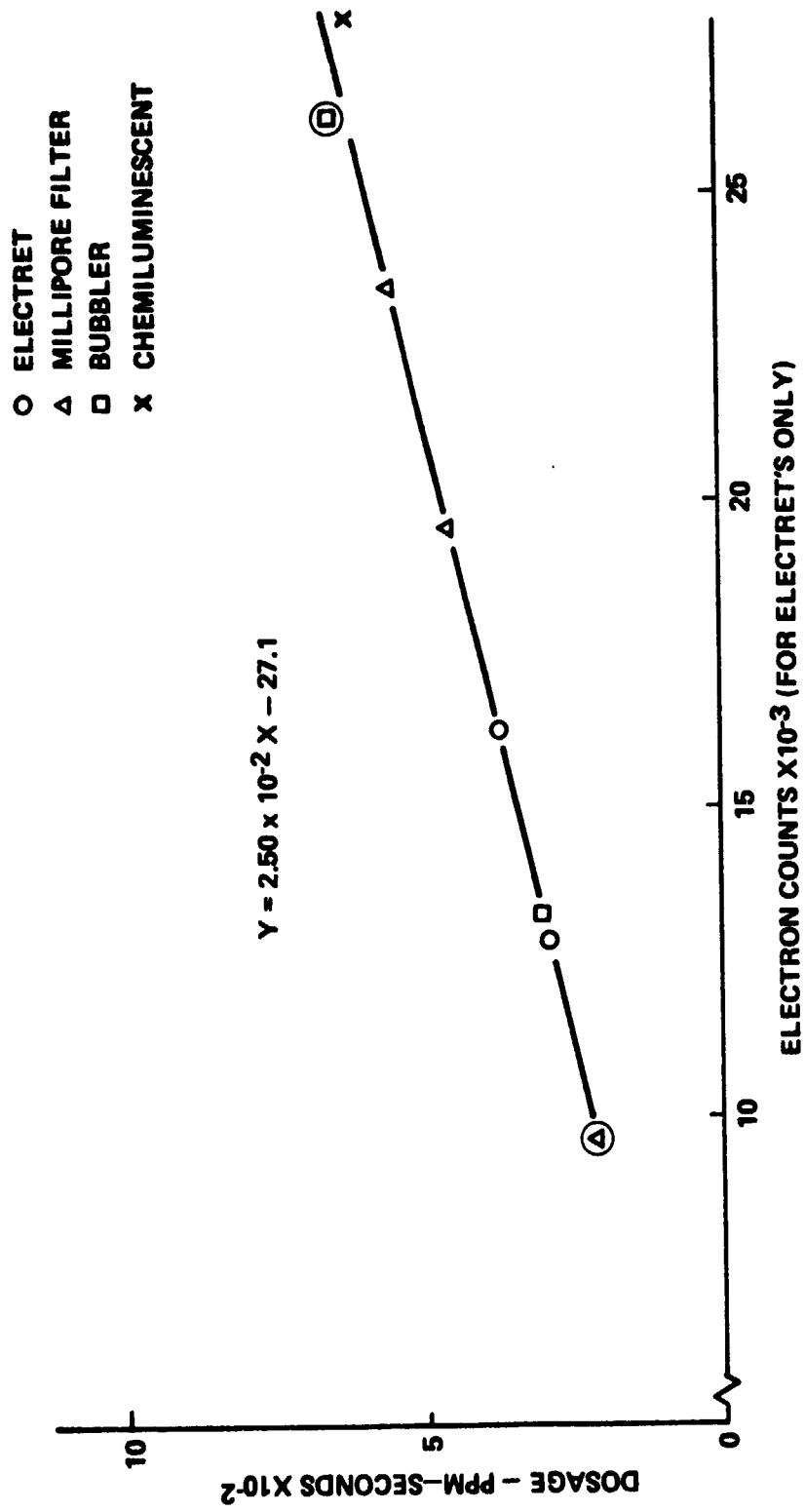


Figure 3. Comparison of HCl dosages (ppm-s) from various instruments to electret electron counts during 6.4 percent SRB tests at MSFC.

REPRODUCIBILITY OF TEST
 DATA IS POOR

of 30, 60, 90, and 120 s were determined using a digital timer. The mixing of the exhaust products results from the heat of the fuel and the resultant turbulent mixing. This lasts for several minutes, and observations indicated that it is a slow mixing process.

The density or distribution of the exhaust products for the 1 s firing in a chamber is not as uniform as a typical exhaust cloud observed [3] in the atmosphere where there is a mass flow of air over the instruments (wind speeds of 2 to 15 m/s depending on the test day). By observation [3] the distribution sensed by the millipore filters and electrets in the chamber is a very slow mixing process in comparison to the mass flow of exhaust clouds observed in rocket testing in the atmosphere. It appears, however, that the localized reading for both instruments in the chamber is the best reading. The flow at this point is more dense and uniform. After a period of time, the distribution becomes spotty in parts of the chamber and decay accrues for the exhaust gases. This can be seen in Figure 4 and Table 2. In Figure 4, the concentration for the millipore filter, after 60 s, two readings of 90 s, and 120 s drops off dramatically. In Figure 5, however, the electron counts generally increase with greater exposure time.

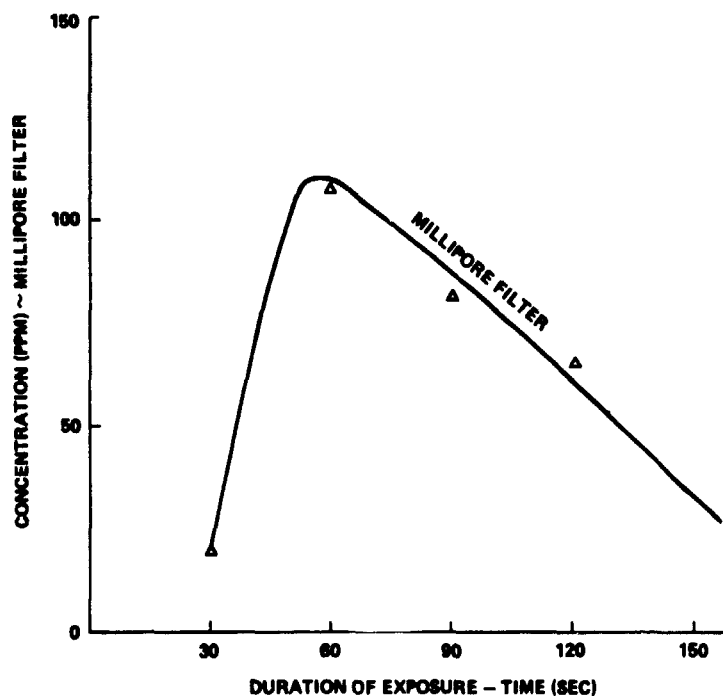


Figure 4. Duration of exposure (s) versus concentration measurements of HCl from millipore filter during chamber tests at Arnold Engineering Development Center.

TABLE 2. CHAMBER TEST DATA; HCl CONCENTRATION MEASUREMENTS FROM MILLIPORE FILTER VERSUS ELECTRET ELECTRON COUNTS FOR TIMES OF 30, 60, 90, AND 120 s AT ARNOLD ENGINEERING DEVELOPMENT CENTER

Instrumentation	Time of Exposure (s)			
	30	60	90	120
Electret Electron Counts	7366	10 845	10 263	15 648
Millipore Filter (ppm)	20	107	81	67

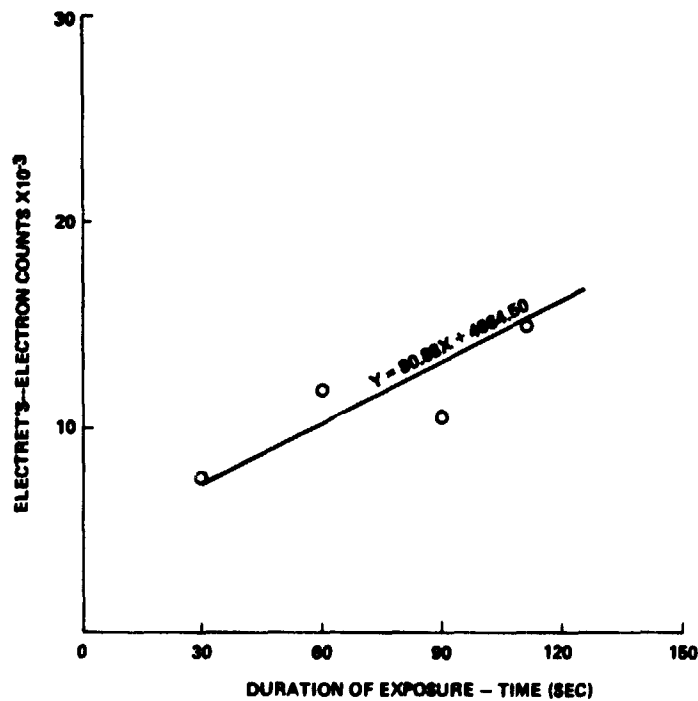


Figure 5. Duration of exposure (s) versus electret electron counts during chamber tests at Arnold Engineering Development Center.

From Table 2, the localized instantaneous value for the electret count of 7366 and the millipore measurement of 20 ppm at 30 s compare favorably when converted using 427.35 counts equal 1 ppm from the atmospheric tests. In this case, 17.25 ppm would be obtained from the electret measurement versus 20 ppm for the millipore filter.

F. Typical Output Curves from X-Ray Microprobe Analysis

A typical computer output of the X-ray microprobe analysis [4] of the rocket exhaust effluents is shown in Table 3 and Figure 6. Because this method has the added advantage of obtaining the entire spectrum of pollutants from a rocket exhaust, it is a powerful tool in studying contaminants.

TABLE 3. DATA OUTPUT FROM ANALYZER, TEST 45

Kilo-Electron-Volts (keV)	Counts (2000 s)	Symbol
0.688	12 979	Γ
1.494	125 890	Al
1.747	331 732	Si
2.128	4 268	Au
2.312	16 765	S
2.643	10 432	Cl
3.396	2 489	K
3.708	1 373	Ca
5.449	5 523	Cr α
5.951	620	Cr β
6.421	14 336	Fe α
7.101	1 458	Fe β
7.527	2 450	Ni

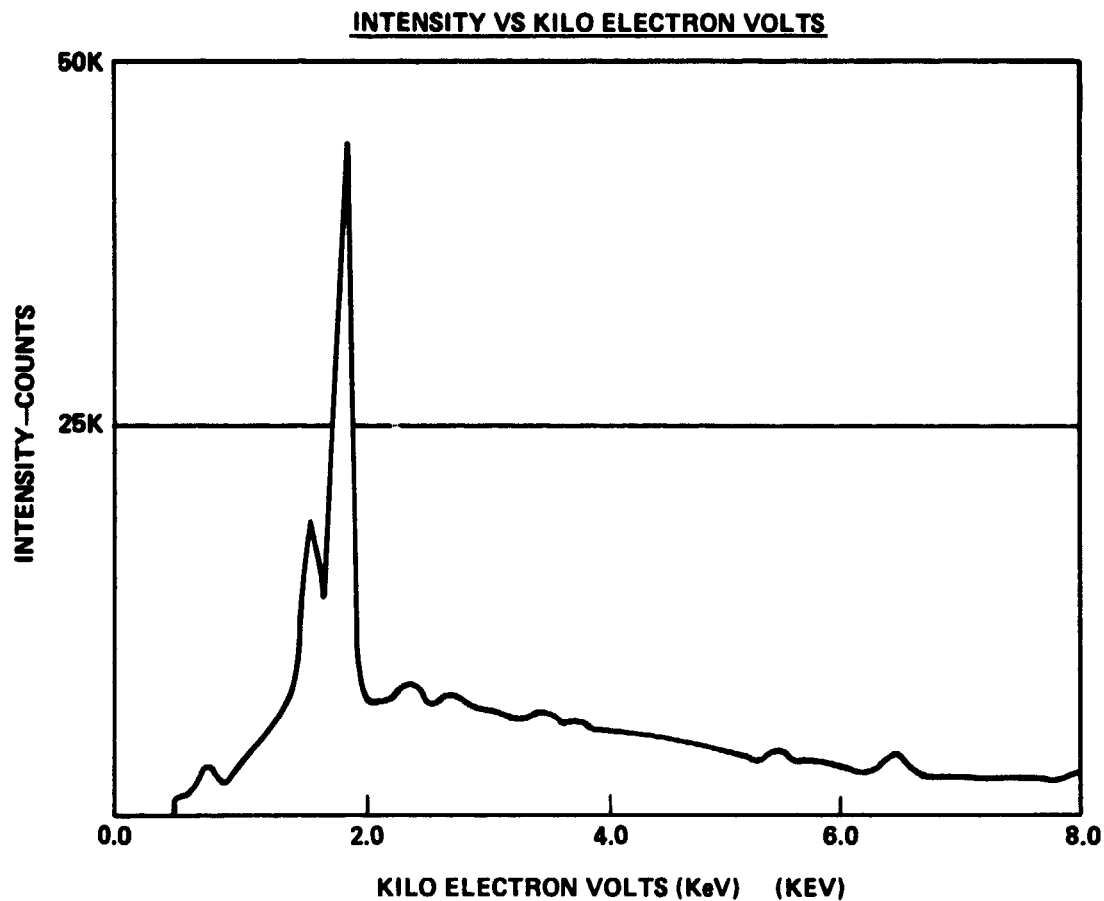


Figure 6. Electron volt spectra output, Test 45.

G. Conclusions and Recommendations

Electrets of polymers were used successfully for the first time in collecting rocket exhaust effluent measurements. The exposed electrets were analyzed by scanning electron microscope and X-ray microprobe analyses. It was possible by these studies to identify the various effluents coming from the rockets at the time of firing. Direct comparisons of values with electrets were made with the results obtained from the chemiluminescent, bubbler, and millipore filters for samples kept under identical conditions. This aided in calibrating the intensity of the X-ray spectra with respect to the concentration of the products

collected on electret surfaces. The results of these comparisons show a high degree of correlation. The entire spectra of the pollutants coming from the rocket may be obtained from the electrets. Other methods showed only the concentration of HCl. However, more extensive investigations must be performed to obtain quantitative results with the electrets.

III. CHEMILUMINESCENT HCl ANALYZERS

A. Description

This detector was developed by NASA under contract and is now available commercially. The HCl detector is designed to continuously monitor HCl air concentrations from 0.1 to 50 ppm. In this concentration range, the response time to 90 percent signal and 10 percent recovery is a few seconds. Since these terms will be used again, an explanation is presented as follows [5]:

"Response time has been defined as that time required for the output of the detector to reach 90 percent of its final (equilibrated) output upon exposure to a fixed pollutant concentration. Time zero for a response time measurement is that time at which the pollutant concentration is introduced into the inlet of the detector. Recovery time is defined as that time required for the output of the detector sampling a fixed concentration to decay to 10 percent of the original value after removing the pollutant gas stream from the inlet of the detector. Time zero for a recovery time measurement is that time at which the pollutant concentration is removed from the inlet of the detector."

B. Discussion

HCl is detected by chemiluminescence accompanying the reaction of luminol. The inlet column for the sampling stream is coated with NaBr and NaBrO₃. HCl reacts with this coating to produce hypochlorite, hypobromite, and possibly bromine, all of which react with luminol to produce chemiluminescence which is proportional to the concentration of HCl in the incoming stream. With most other instruments sufficient HCl, especially at low concentrations, is removed by the adsorption or reaction at the inlet to

cause a lag in response of the instrument. However, in this chemiluminescent instrument, this adsorption or reaction produces species that are essential to the detection process. The response of this instrument is rapid in comparison to other HCl detecting instruments. Laboratory and field evaluations of the chemiluminescent HCl detector have shown the unit to be a reliable HCl detector, as reported in Reference 5. Laboratory results have shown detector limits of 0.05 ppm and an absolute accuracy of at least ± 5 percent. Field results have shown similar detection limits and stable operation at ambient temperatures of 40°C. Future laboratory evaluations will be in the area of interference studies and detection of HCl (gas) and HCl (aerosol) mixtures. Based on the results in Reference 5, the following conclusions apply:

1. The detection limit for the HCl detector is below 0.05 ppm.
2. In the range of 0.05 to 50 ppm, the accuracy of the HCl detector is 5 percent.
3. Response time for the HCl detector ranges from less than 1 s at 50 ppm to approximately 20 s at 0.05 ppm.

IV. CO DETECTORS

A. Description

The CO detector is designed to continuously monitor CO in gas streams and ambient air at concentrations from 0.2 to 200 ppm. The instrument is a nondispersive infrared instrument featuring dual-isotope fluorescence to account for signal attenuation as the result of other pollutants in the sample stream. Response time to 90 percent signal and 10 percent recovery is typically 20 to 40 s.

B. Discussion

Both the nondispersive-infrared, dual-isotope-fluorescent CO detector and the chemiluminescent HCl detector have been used in a number of field monitoring programs. To date no major problems have been encountered in their operation. Minor problems with zero drift and/or small sensitivity

changes (15 percent) have been observed as the result of the field operational environment (35° to 40°C). However, in each case appropriate and rather simplified field procedures can be implemented to correct for these minor problems. Based on the results presented in Reference 5, the following conclusions apply:

1. The detector limit for the CO detector is approximately 0.2 ppm.
2. In the range of 0.2 ppm to 200 ppm, the accuracy of the CO detector is 5 percent or 0.2 ppm, whichever is larger.
3. Response time for the CO detector to 90 percent of reading ranges from 40 s (0 to 20 ppm range) to 20 s (0 to 200 ppm range).

V. BUBBLERS

A. Description

Basically, the bubbler is a dosage type instrument. It sucks air at a rate of 3000 cm³/min into 20 cm³ of distilled water. It uses coulometric detection for the detection of chloride.

B. Discussion

There is a detection limit of 3 ng/20 μl of water. Also, the detection limit is a function of how pure one can get the water. Basically, the detection limit is 80 to 100 ppm-s, which is marginal. As an example, if the concentration is 0.8 ppm for 10 s, no measurement is obtained. However, if a 0.8 ppm measurement for 100 s is made, a measurement may be obtained. The accuracy is 10 percent of the reading above 200 ppm-s; below this the accuracy goes up [6].

VI. MILLIPORE FILTERS

A. Description

A millipore filter consists of a plastic membrane backed with a cellulose pad. The membrane and pad are housed in a disposable plastic container. The accuracy of the determination of an HCl concentration using this device is defined

by the various measurements that must be made, i. e. , time of exposure, flow rate through filter, quantity of water used in the analysis, and the hydrogen ion concentration. Assuring reasonable uncertainties for each of these measurements yields a net uncertainty of ± 15 percent in the HCl concentration.

B. Discussion

The pore size of the millipore filter membrane is $0.4 \mu\text{m}$. The flow rate to sample the exhaust gas is 5 l/min. The membrane is 1.5 in. (3.28 cm) in diameter. The membrane collects the aluminum oxide particles. The HCl is absorbed by the membrane and the cellulose backing pad. The dosage absorbed by a specific filter is determined by removing the membrane and pad and macerating them in 20 cm^3 of distilled water. The resulting pH change in the water is then measured. Thus, the hydrogen ion concentration can be determined, and the quantity of HCl follows from this determination [6, 7].

VII. MASS MONITORS

A. Description

The Celesco quartz crystal microbalance (mass monitor) is an impactor device in which particulates are collected on an oscillating quartz crystal and a rotating polycarbonate disk. The sensing crystal provides real time data on mass loading, while the rotating disk collects material for time resolved elemental analysis.

B. Discussion

Located behind the sensing crystal in the mass monitor is a second crystal that serves as a frequency reference and a temperature compensator. The two crystals, in separate resonant circuits, produce a beat frequency that is increased when mass is added to one of the crystals. This beat frequency is converted to an analog voltage that is proportional to the total mass collected. The analog signal is further differentiated to give a signal proportional to instantaneous mass loading. Both signals are scaled appropriately and recorded directly on a two-channel strip chart recorder in terms of total material collected in micrograms and instantaneous mass loading in micrograms per cubic

meter. The size of particles collected on the crystal is in the range of 0.1 μm to 100 μm diameter. Using scanning electron microscopy, pictorial identification can be made of particles collected on the polycarbonate disk. Both the sensing crystal and disk are coated with an adhesive to increase collection efficiency and to prevent reentrainment of the particles. This instrument can sample continuously to ambient levels of 50 $\mu\text{g}/\text{m}^3$ for a period of 3 to 4 h, at which time the crystal must be changed [8].

VIII. EIGHT STAGE ANDERSON IMPACT FILTERS

A. Description

The Anderson nonviable sampler is an eight stage impact filter. The unit will effectively sample in any position (preferably upright). The particles are impacted on the plates, and the jet stages propel each particle to its particular collection plate for analysis. For further details, see Reference 8.

B. Discussion

Larger particles normally impacting on the top stages could plug the smaller holes in the lower stages of this filter. With the collection plates in place and jet stages in the proper sequence, the sampler is placed in the desired areas for a sample collection. The variety of materials that can be collected with this sampler is naturally unlimited. The collected sample may be analyzed in many different ways, such as microscopically, chemically, colormetrically, gravimetrically, radiologically, etc. [8].

IX. NUCLEPORE FILTERS

A. Description

The filters used are made from polycarbonate sheets that are approximately 10 μm thick. The filters are made by boring holes with fission fragments and then enlarging the holes to the desired size by placing the filters in an etch solution. This process yields a thin, uniform pore-sized, nonhygroscopic, transparent collection surface of uniform weight for particulates.

B. Discussion

This clean, smooth filter material is relatively free of metallic impurities and lends itself well to scanning electron microscopy and neutron activation analysis. The filter is clamped in a holder, and a vacuum pump is attached to the suction tube and turned on with flow rates of 6 to 140 l/min depending on pore size, pump capacity, etc. Particles are drawn to the filter surface and collected primarily by diffusion and impaction, with electrostatic forces playing a small part. The particles collected can then be subjected to the above analysis techniques [8].

X. SUMMARY OF THE SENSORS AND BASIC USAGE

A description of electrets and their multipollutant spectra capability was presented. The response time to 90 percent signal and 10 percent recovery was noted to be a few seconds for the HCl analyzer and 20 to 40 s for the CO detector. It was also noted that if a 0.8 ppm measurement of HCl is to be made by the bubbler, 100 s is the time interval before a measurement may be made successfully. The accuracy is 10 percent of the reading above 200 ppm-s. In addition, the millipore uncertainty in measuring HCl is ± 15 percent. The particulates collected by the mass monitor, eight stage Anderson filter, and Nuclepore filter may be analyzed in many different ways, such as microscopically, chemically, colorimetrically, gravimetrically, radiologically, etc.

XI. CONCLUSIONS AND RECOMMENDATIONS

It has been shown that electrets of polymers have been used successfully for the first time in collecting rocket exhaust effluents. It was possible by the use of scanning electron microscope and X-ray microprobe analyses to obtain spectra, giving the electret a multipollutant capability. In addition, simplicity of deployment and speed of assessment and analysis make the electrets a valuable tool in measuring pollutants from a space vehicle rocket exhaust. An overview of the gas measuring instrumentation, such as the chemiluminescent HCl analyzer, CO detector, bubbler, and millipore filter, was presented. Also, particulate measuring instrumentation, such as the mass monitor, nuclepore filter, and eight-stage Anderson filter, were reviewed. Primarily, the electret is a complementary device to other measuring systems. However, more extensive investigations must be performed to obtain quantitative results with the electrets.

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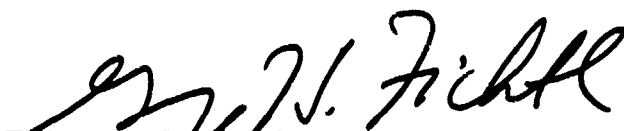
APPROVAL

EXPERIMENTAL RESEARCH IN THE USE OF ELECTRETS IN MEASURING EFFLUENTS FROM ROCKET EXHAUST AND A REVIEW OF STANDARD AIR QUALITY MEASURING DEVICES

By Michael Susko

The information in this report has been reviewed for security classification. Review of any information concerning Department of Defense or Atomic Energy Commission programs has been made by the MSFC Security Classification Officer. This report, in its entirety, has been determined to be unclassified.

This document has also been reviewed and approved for technical accuracy.



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