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HYDRAZINE IMPURITY SURVEY

G. L. M. Christopher, et al

United Aircraft Research Laboratories

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June 1973

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Hydrazine Impurity Survey

by

G.L.M. Christopher

and

C. T. Brown

Phase I

Special Report

June 1973

United Aircraft
Research Laboratories

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FOREWORD

This work was done at the United Aircraft Research Laboratories, East Hartford, Connecticut, for the Air Force Rocket Propulsion Laboratory [illegible] Phase I of Contract F04611-73-C-0019. The work was carried out during the period January 1, 1973 through March 30, 1973.

Work under Phase I of the Contract F04611-73-C-0019 was performed under the direction of Dr. G. L. M. Christopher, Principal Investigator, with the assistance of Dr. G. Golden, Chief, Analytical Chemistry, Mr. William Lasko, Chief, Materials Analysis, and Dr. C. T. Brown, Chemical Sciences.

This work was conducted under technical management of Captain Ronald J. Meetin, AFRPL (LKDP) with the special cognizance of Captain Charles Mastromonico, AFRPL (LKDP). The contracting officer is G. M. Flock of Edwards Air Force Base, Edwards, California.

Publication of this report does not constitute Air Force approval of the reports' findings or conclusions. It is published only for the exchange and stimulation of ideas.

Ronald J. Meetin, Captain USAF
Project Engineer

AFRPL-TR-73-24

ABSTRACT

A study of the amounts of specific contaminants present in propellant grade (Mil Spec) hydrazine obtained from a number of users has been carried out. A total of twenty-four samples from twelve user groups was analyzed. The contaminants examined included particulate materials, organic impurities, water, carbon dioxide, metals and chloride. The level of particulate contamination, the particle size distribution and approximate identification of chemical types were established by appropriate techniques. Major metal contaminants were determined by atomic absorption and trace metal contaminants were determined by emission spectroscopy. Other contaminants were assayed by gas chromatographic, ultraviolet spectroscopic or colorimetric techniques.

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SECTION I

INTRODUCTION

Problems associated with catalyst contamination effects in catalytic decomposition engines using hydrazine-based propellant systems are encountered throughout the industry. The loss of catalytic activity as a function of bed size, duty cycle and total propellant mass flow throughout the life of the engine can be caused by spallation, sintering, crystallite formation, or true catalytic poisoning. The latter factor has been shown to be responsible for many instances of failure of long life engines. Impurities in hydrazine are the most probable cause for these poisoning effects; however, a systematic study of these effects as a function of specific contaminants with varying concentrations has yet to be performed.

In order to effect such a study, which is the purpose of the present contract, it is necessary to define the nature and concentration levels of contaminants most prevalent in hydrazine. These contaminants can be introduced during the hydrazine manufacturing process, during handling and transfer of hydrazine or by reaction of hydrazine with container materials.

This special report is a summary of a survey of the amount and nature of contaminants found in hydrazine as supplied by a large number of users, both government and industrial.

SECTION II

EXPERIMENTAL PROGRAM

The experimental program for the Air Force Rocket Propulsion Laboratory (AFRPL) Hydrazine Impurity Survey consisted of both chemical and physical analyses of impurities contained in twenty-four samples from twelve user groups. A complete list of the suppliers is included in Appendix A along with the sample designations for each container received.

The analytical scheme (Figure 1) was designed to determine the level of soluble impurities including water, carbon dioxide, aniline, monomethyl hydrazine (MMH), unsymmetrical dimethyl hydrazine (UDMH), isopropyl alcohol (IPA) and chloride. In addition, the amounts of metallic impurities were determined. Particulate matter was also examined after filtration of the hydrazine samples. Particle size distribution, qualitative chemical analysis for the elements present, and particle morphology were determined.

In most cases samples were received in both one quart and one-half pint glass bottles. Each bottle was numbered in the order in which it was received. Where two sample numbers are listed together, both were the same composition from the same lot from a single supplier. They represent different bottles of otherwise identical materials.

The sample handling procedures were as follows:

Samples for Chromatographic Analysis:

Approximately 250 ml (1/2 pt.) of hydrazine sample received in glass bottles with polyseal closure, was transferred, through an air-lock, into a dry-box purged with dry nitrogen. Cleaned and dried 50 ml septum bottles equipped with "Mininert" valves were already contained in the dry box. These valves are all teflon with O-Ring seals and are fitted with septums and a valve which must be opened before a hypodermic syringe can be inserted for chromatographic samples. After 24 hours of conditioning, the sample bottle was opened, part of the contents was transferred to a septum bottle and the closure valve was inserted. The filled septum bottle was removed through the air lock and served to provide samples for chromatographic analysis of H_2O, CO_2, UDMH, MMH and isopropyl alcohol.

Samples for Chemical Analysis of Metallic Materials and Determination of Particulates:

The outside surface of the glass bottle containing approximately 1000 ml (1 qt.) of a hydrazine sample was carefully washed to remove any particulate material. Special attention was given to the region of the bottle neck and the closure. This hydrazine was used for determination of particulates, non-volatile residue, metals, chloride and aniline.

1. ANALYTICAL PROCEDURES FOR NONFILTERABLE IMPURITIES

For convenience the impurities analyzed in this survey have been divided into two groups; filterable and non-filterable impurities. In the latter case there was a further subdivision into metallic and non-metallic impurities. The metallic impurities were analyzed after filtration and evaporation of a large (500 ml) sample of hydrazine (Figure 1). In addition to the above, parameters such as non-volatile residue (NVR) and ash were determined in an attempt to differentiate between materials with high boiling points and inorganic residues. A brief statement of the nature of the type of impurities determined is indicated below:

H_2O, CO_2, UDMH, MMH, Isopropyl Alcohol: These compounds were determined by gas chromatography. Through the use of two chromatographic columns in series, H_2O, CO_2, UDMH, MMH and Isopropyl Alcohol were determined simultaneously. Since well-defined chromatographic peaks are produced by water with each column, two independent measurements of water were obtained with each sample of hydrazine injected into the system. In all cases samples were analyzed in duplicate.

Chloride: Chloride was determined colorimetrically after evaporation of a hydrazine aliquot to dryness. A small amount of NaOH was added before evaporation to ensure retention of chloride.

Aniline: Aniline was determined by ultra-violet spectrophotometry.

Non-Volatile Residue (NVR): Duplicate samples of filtered hydrazine of 250 ml volume were evaporated to small volume in Teflon beakers. Contents of the beakers were transferred to weighed platinum crucibles (using small amounts of pure, filtered isopropyl alcohol for rinsing the beakers) and further evaporated to a syrupy residue. This residue was then heated for 1 hour at 105°C in an oven. The residue weight was used as a measure of NVR.

Ash: The materials in the crucibles from the NVR determination were heated to 650°C for one hour to destroy any organic residues present. The weight of residue after this treatment represents non-volatile inorganic substances.

Trace Metals: A semi-quantitative measurement of trace elements present in the hydrazine was carried out by the use of emission spectrography on one of the ash residues. Elements found to be present in sufficiently large quantities (major metallic impurities) were determined on a quantitative basis.

Major Metallic Impurities: These elements were determined by atomic absorption using a solution prepared from one of the ash residues. A more detailed discussion of the analytical procedures is given in the following sections of the report.

a. Gas Chromatography

The gas chromatographic procedure for the analysis of water, carbon dioxide, monomethyl hydrazine, unsymmetrical dimethyl hydrazine and isopropyl alcohol was based on the use of a dual column chromatograph. The chromatographic equipment is illustrated in Figure 2. The chromatograph may be operated using two detectors simultaneously or in series with two columns to give added versatility in difficult separations. The response of each detector is displayed independently.

Liquids or gases may be introduced into the unit through a heated inlet by means of precision syringes for separation, qualitative identification, or quantitative measurements. Alternatively, gases may be admitted from static or dynamic systems via a solenoid-operated gas sampling valve.

Gas concentrations in the part per million to part per hundred range may be determined. Isothermal or programmed temperature modes of operation up to 400°C yield sufficient vapor pressures to allow samples with boiling points over 500°C to be analyzed. Provisions have been made to allow trapping of separated fractions for infrared identification.

In the course of the chromatographic work, it was found that the columns specified in the military specifications (Refs. 1 & 2) were not sufficient for complete separation of all the filtered impurities noted above.

A gas chromatographic column system was developed consisting of two separate columns of two sections each. In one system a section containing Carbowax 400 (polyethylene glycol 400) liquid phase is followed by a section containing Dowfax 9N9 (nonyl phenyl polyethylene glycol ether). The second system contains a Porapak PS section followed by a section containing Dowfax 9N9. NH_3, CO_2, H_2O and MMH are resolved on the first system and NH_3, CO_2, H_2O, IPA, and UDMH on the second. The Carbowax 400 column described in the military specification (Ref. 1) would not resolve UDMH-IPA and H_2O-MMH; the Dowfax 9N9 column described in the military specifications (Ref. 2) would not effect H_2O-UDMH and MMH-IPA separations.

The three primary gas chromatographic columns used were:

A - one meter x 1/4" length containing 33% Carbowax 400 (polyethylene glycol 400) on 90/100 mesh Anakrom AB (Anakrom B used in the military specification is no longer available);

B - 6 ft x 1/4" length containing 10% Dowfax 9N9 (nonyl phenyl polyethylene glycol ether) on 60/80 mesh T6 (Teflon V used in the military specification is no longer available);

C - 6 ft x 1/4" length containing 50/80 mesh Porapak PS.

All columns were made of 316 or 321 stainless steel. The carrier gas was helium and the flow rate was set to 80 ml/min. These columns were tested and used in a Beckman GC 72-5 with two hot wire thermal conductivity detectors and heated inlets. With this system two independent chromatographic systems were set up to facilitate more rapid total analysis. The procedure used in the military specifications (Refs. 1 & 2) was followed and gave satisfactory results. The procedure is as follows:

1) Condition columns at 150°C for 4 hrs with 50 ml/min He flow

2) Column temperature for analysis - 110°C; He flow rate - 80-120 ml/min

3) Detector and inlet temperature - 150°C

4) Saturate columns by injecting 3 successive 10 microliter samples of propellant. (If more than 30 minutes elapse between elution of the hydrazine and the next injection, re-saturate the column with a 10 microliter injection of hydrazine.

5) Using a clean, dry 10 microliter syringe, carefully fill and set plunger to 3 microliters, wipe tip with tissue without touching the open end of the needle. Quickly inject the sample into the instrument and immediately withdraw the needle.

First attempts to accomplish component separation were made using the military specification methods per se. Figures 3 and 4 show typical chromatograms using these methods. It can be seen that the Carbowax 400 column did not separate UDMH-IPA and H_2O-MMH and the Dowfax 9N9 column did not separate H_2O-UDMH and MMH-IPA. From the separation data obtained on these columns it was concluded that some advantage might be gained by using these two columns in series. This procedure gave better results as can be seen on the chromatogram in Figure 5; however, UDMH and IPA are not separated. Porapak PS (column C) resolves CO_2, H_2O and IPA from the hydrazines. N_2H_4, MMH and UDMH elute from this column after the IPA in that order, but are not well resolved, (Figure 6). This order of elution for the hydrazines is the reverse of that obtained using columns A and B. Since column B gave the same H_2O-IPA elution order as column C and gave clean sharp peaks, the approximate length necessary to place elution time for IPA ahead of the UDMH was calculated from retention data. This calculation indicated a length of 6 ft for column C which when combined with column A, resulted in the chromatogram shown in Figure 7. These column systems have proven valuable in analyzing many propellant samples which were found to contain from <0.01 w/o to over 1 w/o of these various components.

The combination of columns B and C (Figure 7) was used for the analysis of all impurity components with the exception of MMH, which was analyzed using a combination of columns A and B (Figure 5).

Standards were prepared by adding weighed amounts of each impurity specified above to weighed hydrazine samples and extrapolating the results to determine the original amounts of these constituents in the hydrazine by calculating peak height ratios.

b. Ultraviolet Spectroscopy

Aniline in hydrazine was determined by ultraviolet spectroscopy using a Model 14 Cary Recording Spectrophotometer, set at 287 microns. The absorbance of aniline in the samples was measured against doubly distilled water in a 0.5 cm cell. Standards containing known amounts of aniline in doubly distilled water were used for calibration. This calibration resulted in a factor of one (1) absorbance unit per 90 micrograms aniline/ml of a solution which was diluted by a factor of 100. This absorbance X(90) x (100) is equivalent to ppm aniline in the original sample.

c. Colorimetric Analysis for Chloride

Hydrazine samples were analyzed for chloride using a portion of the samples retained (unfiltered) for chromatographic and aniline analysis (Figure 1). Duplicate ten milliliter samples were taken and analyzed according to ASTM method D512-67 (Ref. 3). This procedure is a colorimetric method based on the reaction of chloride ion with a mixture of ferric ammonium sulfate and mercuric thiocyanate to form the colored ferric thiocyanate complex. Absorbance is measured at 460 millimicrons and compared to standards containing known amounts of chloride.

d. Chemical Analysis of Metallic Impurities

Metallic impurities were determined in hydrazine after determination of the non-volatile residue. Samples heated to 650°C were analyzed for both major and minor metallic impurities by atomic absorption and emission spectroscopic procedures. In the former case the heated samples were dissolved in HCl and diluted to 10 ml with deionized water using atomic-absorption procedures ejection of the liquid samples into a flame containing fuel and oxidizer appropriate to the particular metal impurity in question. The major impurities analyzed in this fashion included sodium, iron, chromium, nickel, zinc and cadmium. The atomic absorption apparatus is a Perkin-Elmer Model 303 shown in Figure 8.

Minor metallic impurities were analyzed using ash sample #2 from each filtrate. These filtrates were evaporated in two portions for determination of NVR and subsequently ashed for analysis of major metallic impurities by atomic absorption and minor metallic impurities by emission spectroscopy. A Jarrel Ash-Ebert Mark IV, 3.4 meter spectrograph was used for the latter analysis. This instrument is illustrated in Figure 9.

2. ANALYTICAL PROCEDURES FOR FILTERABLE IMPURITIES

Insoluble impurities were characterized after filtration from hydrazine as follows.

a. Optical microscopy for particle size distribution and particle morphology.

b. Electron probe analysis for the elements present in specific particles.

c. Scanning electron microscopy to determine the topographical features of selected particles.

Particulates from hydrazine samples were collected by filtration through a Teflon filter medium which retained particles larger than 5 microns (μ). Using the procedure in MIL-P-26536 the particulate residues were lifted from a one-quarter section of the filter medium by a stripping technique and optically photographed. The strippings were then coated with a thin layer (several hundred Angstroms) of pure graphite and subjected to Electron microprobe analysis. Strippings from an unused filter were treated in the same manner and served as a blank control specimen.

The size distribution of the particulates removed from the hydrazine samples were obtained by photographing at 100X, five randomly chosen fields of the remaining 3/4 section of the filters containing the particles from 500 ml. of hydrazine sample. The particles in the size ranges 6-10μ, 11-25μ, 26-50μ, 51-100μ, and >100μ were counted in each field and the data reduced and reported as particles per 100 ml. hydrazine. The area of each field was 1.015 cm^2 and the total filter area was 9.62 cm^2.

The Electron Probe Microanalyzer (Figure 10) was used to qualitatively determine the elements present in the various particulate types found in each sample. This instrument combines techniques from electron microscopy and X-ray spectroscopy in one instrument to achieve exacting analysis in the micro range. With this instrument it is possible to make direct chemical analysis of a sample approximately one $micron^2$ in area and one micron in depth, representing approximately one micro-microgram of material. The test is nondestructive and the sample may be retained for further experimentation.

The Electron Probe Microanalyzer employs an electron optical system using two magnetic lenses which produce an electron focus of approximately one micron in diameter. This focused electron beam is used to excite characteristic X-rays from the specimen. The X-rays are then sorted by means of curved crystal spectrographs, and presented on recorders or scalers as in conventional X-ray spectroscopy. This instrument provides both qualitative and quantitative information of the elemental composition on any specimen area from 1 to 200 microns, from atomic number 5 (boron) up through the remainder of the periodic chart.

Selected particles from some of the hydrazine samples were also examined using a Scanning Electron Microscope (SEM) (Figure 11) in order to determine their topographical features. The AMR-900 Model SEM has a magnification range from 5X to 100,000X, and a resolution of 250Å.

SECTION III

EXPERIMENTAL RESULTS

The experimental results of the hydrazine survey are presented below. The chemical analysis and particulate analysis are presented in separate sections; however total particulates are included in the chemical analysis section.

1. CHEMICAL ANALYSIS OF NON-FILTERABLE IMPURITIES

The results of chromatographic analysis of twenty-four hydrazine samples are summarized in Table I. Water content varied considerably from 0.3 to 1.0 weight percent, while carbon dioxide levels were found to be in the range 0.1 to 0.6 percent. No detectable amounts of UDMH (<0.01 percent) were found in any of the samples. Nine (9) samples were found to contain from 0.01 to 0.08 percent MMH, and four (4) of these nine (9) also contained 0.02 to 0.04 percent IPA. The MMH and IPA are probably present as result of successive use of containers and plumbing for MMH and hydrazine followed by inadequate cleaning with IPA, which in turn may not be thoroughly eliminated before use.

The results of analysis for aniline (the third major impurity in hydrazine) are summarized in Table II. Aniline levels are remarkably constant and range from 0.17 to 0.36 percent. Aniline is used in the hydrazine purification step during manufacture of the latter and the data reflects the fact that a fairly consistant fraction of aniline remains in the product.

The non-volatile residue (NVR) and ash levels found in the hydrazine samples are summarized in Table III. The non-volatile residue reflects the presence of high boiling compounds. Aniline has a relatively low vapor pressure at the evaporation temperature of 105°C. It is also probable that products resulting from the interaction of CO_2 with hydrazine also have relatively low vapor pressure at this temperature. The NVR is believed to contain such materials in addition to any hydrazine hydrochloride normally present in the hydrazine. Attempts to utilize infrared spectroscopy for demonstrating the presence in NVR of compounds derived from hydrazine, CO_2 and HCl were unsuccessful.

The ash samples were analyzed for both major metallic impurities and for metals present in trace quantities. The results of these analyses are summarized in Table IV and V. The major metallic impurities include sodium, iron, chromium, nickel, zinc and cadmium. The quantities of sodium and iron were in general in the range .04 to 0.5 ppm which is about in order of magnitude greater than the other "major" impurities. Iron, chromium and nickel are probably from the stainless steels used in containers, transfer tubing and valves, and may be present either in soluble form or as components of particulates.

Sodium may be introduced as a constituent of soluble salts that are introduced by exposure of hydrazine to the atmosphere, however the majority of the sodium content is probably present due to the use of sodium hydroxide in the manufacturing process for neutralization of hydrochloric acid and chlorine. The hydrochloric acid is a product and the chlorine a reactant in the manufacturing process.

The chloride levels found in the samples (Table IV) are quite large compared to the metals and range from 0.3 to 4.6 ppm. These levels are such that they are much greater than the sum of all the metal concentrations. These results indicate that chloride is associated with either hydrazine, ammonia or possibly aniline in addition to its association with sodium or other metallic ions. The major portion of chloride is probably introduced during the manufacturing process as hydrochloric acid as noted above.

Salt formation of nitrogenous bases with hydrochloric acid also may account for the high levels of NVR since these salts would not be volatile at the temperature used in the test. These materials would volatilize at the high temperature (650°C) used for determining the ash content and the large differences observed between NVR and ash are thus explained. In a similar manner, the interaction between hydrazine and CO_2 could lead to a salt-like product which also could have a relatively low vapor pressure and therefore produce a high NVR.

2. CHEMICAL AND PHYSICAL ANALYSIS OF FILTERABLE IMPURITIES

The particulate level in each sample is summarized in Table III along with the results for non-volatile residue and ash. The particulate levels ranged from a trace (<0.1 ppm) to as high as 2.2 ppm in sample 23-24. In most cases the observed particulate level was below 0.4 ppm. The particulates were examined to determine particle size distribution, chemical elements present and particle morphology.

a. Particle Size Distribution

Particle size distribution data for twelve (12) samples is summarized in Table VI. The size distribution is such that in cases where a large number of particles was found, the majority were less than 25 microns in size. With the exception of samples 23-24, very few particles greater than 50 microns were present. The particle size distribution was determined from random samples as previously described. The samples could only be analyzed as received so the size distribution and the total particulate count (Table III) will not necessarily reflect actual particle levels in a given drum unless the contents were thoroughly stirred and samples were taken before any settling could occur.

b. Chemical Analysis and Morphology

The spectral analysis of the particulates in all twenty-four samples analyzed are summarized in Tables VII through XI. The analyses are segregated according to the general type of particulate found. These types include:

(1) Agglomerates: Dark in appearance and apparently composed of multiple components (size: 5-100μ); two basic sub-types:

A. Low-melting variety (sintered when exposed to electron beam)
B. Stable variety (unchanged when exposed to electron beam)

(2) Glassy Plates: Large, flat and similar in appearance to thin sheets of mica (size: 20-100μ, generally 100-300μ).

(3) Metallics: Small metallic chips (size: 20-100μ).

Representation photomicrographs of the particulates are illustrated in Figures 12 through 15. The three general types of particles are labeled in Figure 12. Also included is identification of Teflon filaments which were transferred from the filters used to trap the particulates.

No specific analysis was performed for oxygen, nitrogen or carbon. Experience with the analytical characteristics of small particulates, however, does indicate that the agglomerates and glassy plates would be oxygen-inch. The major metal elements observed suggest the presence of metal oxides, sand and clays. Electron beam scan photographs of representative particles of each type of contaminant are illustrated in Figures 16, 17 and 18 for a glassy plate phase, a stable agglomerate particle and a metallic particle respectively. It is apparent from these photos that the "glassy" plates contain primarily potassium and silicon, while the agglomerates are silicon and iron and the metals are probably particles of pure iron.

Particulates were also examined using the Scanning Electron Microscope (SEM) to determine their topographical features. The particles were generally plate-like or rough textured, irregularly shaped.

Figure 19 shows particles from Sample 9 (Bell Aerospace, S/N 929). A few flat particles, (9A) and a few granular particles, (9B) were observed. The majority of the particulates from this sample were of rough, irregular shape similar to those shown in C & D.

Figure 20 shows particles from Sample 12 (China Lake, 2037). The particulates are about equally divided between the irregularly shaped particles shown in A and B and those with a more block-like shape as in C & D.

In Figure 21 are particles from Sample 14 (JPL, H-3155). Two rather unusual types of particles are shown in A & B. This type was not observed in the other samples reported here. Sharply cleaved and irregularly shaped plate-like particles were also observed and are shown in C & D, respectively.

Particles from Sample 18 (Sundstrand, H-110) are shown in Figure 22. They consist of the typical plate (A & B) and rough textured, irregularly shaped (C & D) particles.

SECTION IV

SUMMARY AND CONCLUSIONS

From the data presented in the preceding section of this report several general observations may be made regarding the nature of impurities found in the twenty-four samples analyzed.

The major soluble, non-metallic impurities in hydrazine are water, carbon dioxide, aniline and chloride. The amount of aniline was remarkably constant in all the samples analyzed. It is evident that this contaminant is present in the manufactured hydrazine and is not introduced by handling or exposure. Water and carbon dioxide are introduced by exposure to air. The variations in the levels of carbon dioxide and water are such that they constitute a good measure of the exposure of the specific samples to the atmosphere prior to receipt for analysis at UARL. The range of concentrations of carbon dioxide and water, which are 0.1 to 0.6 percent and 0.3 to 1.0 percent respectively, are not large when one considers the large affinity of hydrazine for these constituents.

Chloride concentrations, which ranged from 0.3 to 4.6 ppm, are much larger than the sum of the metallic ion concentrations. Of the major metallic ions - sodium, chromium, iron and nickel - sodium and iron are the most concentrated. Iron, nickel and chromium are probably introduced by reaction of hydrazine with the stainless steels used for containers and associated plumbing. The sodium is primarily introduced during the manufacturing process when sodium hydroxide is used to neutralize the hydrochloric acid produced as a product along with hydrazine. The chloride which is introduced from the hydrochloric acid in this process must be present, not only as metallic chloride salts, but also in combination with hydrazine, ammonia and possibly aniline.

The non-volatile residue (NVR) is a result of high boiling constituents such as aniline and reaction products of hydrazine with carbon dioxide, and chloride. All of these salt-forming compounds have boiling points greater than the evaporation temperature of 105°C, but less than the 650°C used to produce ash samples.

The ash samples are primarily metallic residues; principally iron and sodium. These metallics may be present in solution as hydrazine complexes, or in the form of small particulates less than 5 microns in size.

The remaining non-metallic soluble impurities are monomethyl hydrazine (MMH), unsymmetrical dimethyl hydrazine (UDMH) and isopropyl alcohol (IPA). UDMH was not present in any significant amounts in any of the samples analyzed. MMH and IPA were found in nine and four of the samples analyzed respectively, and are probably the result of successive use of containers and plumbing for hydrazine and MMH and subsequent cleaning of these containers with IPA, accompanied with insufficient flushing of the IPA. Proper cleaning procedures should minimize these problems.

An additional and major problem in propellant contamination is the presence of insoluble particulate materials. Although the vast majority of these particles are less than 25 microns in size, they are still large enough to cause plugging in the small fuel lines used for propellant delivery to small thrust engines. In many cases the maximum particle size was less than 10 microns, but these particles would still cause clogging in the catalyst bed itself unless line filters of less than 10 microns capability are used in the fuel lines. The types of particles analyzed were found to be very similar to metal oxides, sand, clay and individual metallic particles (basically iron). The sand and clay are probably introduced from air suspended dirt during exposure of the hydrazine to the environment. Some of the sodium may also be from this source.

Metal oxides and metallic particles are probably introduced as stainless steel corrosion products (due to exposure to oxygen when containers are empty) and as metal chips from galled threads on container fittings.

In general it appears that many of the contaminant problems including particulate sand and clay, MMH and IPA, may be minimized by more stringent cleaning and handling procedures. The presence of iron, chromium and nickel indicate that better procedures for passivation of stainless steels must be developed to ensure minimal dissolution of these materials.

REFERENCES

1. MIL-P-27402B, May 27, 1969.
2. MIL-P-27404A, May 20, 1969.
3. Colorimetric Determination of Chloride Ion., ASTM Method D512-67 Annual Book of ASTM Standards, Part 23, pp. 21-26, 1972.

APPENDIX A

Source	Supplier Identification	Number of Samples Received	Sample Number
Aerojet General Corporation* Sacramento, California	Drum H-4248	6	none assigned
Bell Aerospace Company Buffalo, New York	Drum S/N 929	3	9, [illegible], 11
Department of the Army Rocky Mountain Arsenal Denver, Colorado	R.R. Car DODX-17074 R.R. Car DODX-17067	2 2	48, 49 50, 51
Hamilton Standard Division of United Aircraft Corporation Windsor Locks, Connecticut	"A" BBL-4318 "B" BBL-2556	3 3	40, 42, 43 41, 44, 45
Jet Propulsion Laboratory California Institute of Technology Pasadena, California	Drum H-3155	2	14, 15
Lockheed Missiles and Space Company Incorporated Sunnyvale, California	A-3937	1	4
Rocketdyne Division of North American Rockwell Corporation Canoga Park, California	PRA Tank #5	2	29, 30

*Samples received too late to be analyzed.

APPENDIX A (continued)

Source	Supplier Identification	Number of Samples Received	Sample Number
Rocket Propulsion Laboratory Edwards Air Force Base Edwards, California	Drum 3103 Batch 43-72	2	31, 32
	Drum H-1993 Batch 43-72	2	33, 34
	Drums-2763, 2266, 3027 Batch 38-72	2	35, 36
	Drum 1810 Batch 38-72	2	46, 47
Rocket Research Redmond, Washington	Drum H-1799	3	1, 2, 3
Shell Development Company Chemical Research Laboratory - KATY Houston, Texas	H-4670	2	5, 6
	H-4903	2	16, **
TRW Systems Group Redondo Beach, California	no designation	3	37, 38, 39
Sunstrand Aviation Division of Sunstrand Corporation Rockford, Illinois	Drum H-110	1	17
		1	18

**Larger sample received broken and not given sample number.

APPENDIX A (conclusion)

Source	Supplier Identification	Number of Samples Received	Sample Number
United States Naval Weapons Center China Lake, California	Lot H-2002	2	7, 8
	Lot-2037	2	12, 13
	Lot-681	2	19, 20
	Lot-3139	2	21, 22
	Lot-3456	2	23, 24
	Lot H-794	2	25, 26
	Lot H-2604	2	27, 28

Table I

GAS CHROMATOGRAPHIC ANALYSES OF HYDRAZINE CONTAMINANTS

UARL I.D.#	USER	SAMPLE I.D.	H_2O %(Wt.)	CO_2 %	MMH %	UDMH %	IPA %
1,3	ROCKET RESEARCH	H-1799	0.5	0.2	<0.01	<0.01	<0.01
5,6	SHELL	H-4670	0.4	0.2	0.08	<0.01	<0.01
7,8	CHINA LAKE	H-2002	0.6	0.5	<0.01	<0.01	<0.01
10,11	BELL	S/N 929	0.5	0.3	<0.01	<0.01	<0.01
12,13	CHINA LAKE	2037	1.0	0.3	<0.01	<0.01	<0.01
14,15	JPL	H-3155	0.3	0.2	<0.01	<0.01	<0.01
17,18	SUNDSTRAND	H-110	0.3	0.3	0.03	<0.01	<0.01
19,20	CHINA LAKE	Lot 681	1.0	0.4	<0.01	<0.01	<0.01
21,22	CHINA LAKE	H-3139	0.6	0.5	<0.01	<0.01	<0.01
23,24	CHINA LAKE	H-3456	0.9	0.4	<0.01	<0.01	<0.01
25,26	CHINA LAKE	H-794	0.6	0.6	<0.01	<0.01	<0.01
27,28	CHINA LAKE	H-2604	0.7	0.6	<0.01	<0.01	<0.01
4	LOCKHEED	A 3937	0.3	0.4	<0.01	<0.01	<0.01
16	SHELL	H-4903	0.4	0.5	<0.01	<0.01	<0.01
29,30	ROCKETDYNE	PRA Tank #5	0.5	0.1	0.04	<0.01	0.04
31,32	EDWARDS	Drum 3103	0.4	0.2	0.04	<0.01	0.02
33,34	EDWARDS	H-1993	0.4	0.2	0.02	<0.01	0.02
35,36	EDWARDS	Drum 2763	0.3	0.2	0.01	<0.01	0.02
37,38	TRW	None	0.4	0.2	0.02	<0.01	<0.01
40	HAM. STAND.	4318	0.7	0.5	<0.01	<0.01	<0.01
41	HAM. STAND.	2556	0.4	0.6	<0.01	<0.01	<0.01
46,47	EDWARDS	Drum 1810	0.3	0.5	0.06	<0.01	<0.01
48,49	ROCKY MTN. ARS.	DODX 17074	0.4	0.2	<0.01	<0.01	<0.01
50,51	ROCKY MTN. ARS.	DODX 17067	0.3	0.5	0.03	<0.01	<0.01

Table II

ANILINE CONTENT OF HYDRAZINE SAMPLES

UARL I.D.#	USER	SAMPLE I.D.	ANILINE %
1,3	ROCKET RESEARCH	H-1799	0.27±.02
5,6	SHELL	H-4670	0.28
7,8	CHINA LAKE	H-2002	0.33
10,11	BELL	S/N 929	0.26
12,13	CHINA LAKE	2037	0.36
14,15	JPL	H-3155	0.17
17,18	SUNDSTRAND	H-110	0.26
19,20	CHINA LAKE	Lot 681	0.34
21,22	CHINA LAKE	H-3139	0.34
23,24	CHINA LAKE	H-3456	0.36
25,26	CHINA LAKE	H-794	0.34
27,28	CHINA LAKE	H-2604	0.33
4	LOCKHEED	A 3937	0.30
16	SHELL	H-4903	0.23
29,30	ROCKETDYNE	PRA Tank #5	0.25
31,32	EDWARDS	Drum 3103	0.30
33,34	EDWARDS	H-1993	0.29
35,36	EDWARDS	Drum 2763	0.24
37,38	TRW	None	0.32
40	HAM. STAND.	4318	0.32
41	HAM. STAND.	2556	0.27
46,47	EDWARDS	Drum 1810	0.27
48,49	ROCKY MTN. ARS.	DODX 17074	0.31
50,51	ROCKY MTN. ARS.	DODX 17067	0.24

Table III

PARTICULATE, NVR AND ASH IN HYDRAZINE SAMPLES

UARL I.D.#	USER	SAMPLE I.D.	PARTICULATES ppm	NVR (105°C) ppm	ASH (650°C) ppm
1,3	ROCKET RESEARCH	H-1799	0.2	8.8	1.8
5,6	SHELL	H-4670	0.3	3.0	2.0
7,8	CHINA LAKE	H-2002	0.1	12.2	1.9
10,11	BELL	S/N 929	0.3	21.0	<1.0
12,13	CHINA LAKE	2037	0.3	9.5	3.6
14,15	JPL	H-3155	<0.1	13.2	<1.0
17,18	SUNDSTRAND	H-110	0.2	19.4	1.7
19,20	CHINA LAKE	Lot 681	0.3	12.4	1.0
21,22	CHINA LAKE	H-3139	<0.1	12.5	<1.0
23,24	CHINA LAKE	H-3456	2.2	24.5	1.0
25,26	CHINA LAKE	H-794	0.2	9.0	<1.0
27,28	CHINA LAKE	H-2604	<0.1	7.8	<1.0
4	LOCKHEED	A 3937	0.13	15.9	<1
29,30	ROCKETDYNE	PRA Tank #5	0.35	46.9	1.0
31,32	EDWARDS	Drum 3103	0.25	17.9	<1
33,34	EDWARDS	H-1993	<0.1	20.9	<1
35,36	EDWARDS	Drum 2763	<0.1	17.9	<1
37,38	TRW	None	<0.1	28.5	<1
40	HAM. STAND.	4318	0.11	11.3	<1
41	HAM. STAND.	2556	0.37	18.7	<1
46,47	EDWARDS	Drum 1810	<0.1	10.5	<1
48,49	ROCKY MTN. ARS.	EODX 17074	<0.1	11.6	<1
50,51	ROCKY MTN. ARS.	DODX 17067	0.11	13.0	<1

TABLE IV

MAJOR METALS AND CHLORIDE
IN HYDRAZINE SAMPLES

UARL I.D. #	USER	SAMPLE I.D.	Na ppm	Fe ppm	Cr ppm	Ni ppm	Zn ppm	Cd ppm	Cl ppm
1,3	ROCKET RESEARCH	H-1799	0.04	0.14	0.006	0.004	0.008	0.01	2.2
5,6	SHELL	H-4670	0.35	0.29	0.016	0.01	0.006	0.04	2.1
7,8	CHINA LAKE	H-2002	0.23	0.10	0.001	0.002	0.004	0.02	3.1
10,11	BELL	S/N 929	0.26	0.26	0.012	0.01	0.04	0.04	3.5
12,13	CHINA LAKE	2037	0.30	0.23	0.006	0.02	0.008	0.03	2.0
14,15	JPL	H-3155	0.10	0.14	0.024	0.01	0.01	0.02	3.5
17,18	SUNDSTRAND	H-110	0.27	0.59	0.006	0.08	0.01	0.18	1.6
19,20	CHINA LAKE	LOT 681	0.10	0.21	0.006	0.03	0.02	0.07	2.4
21,22	CHINA LAKE	H-3139	0.22	0.04	0.002	0.004	0.02	0.07	4.2
23,24	CHINA LAKE	H-3456	0.23	1.9	0.073	0.20	0.02	0.004	4.6
25,26	CHINA LAKE	H-794	0.19	0.30	<0.001	0.002	0.066	0.04	3.5
27,28	CHINA LAKE	H-2604	0.17	0.30	0.007	0.002	0.006	0.04	3.0
4	LOCKHEED	A 3937	0.04	0.04	<0.004	0.005	0.019	0.005	3.1
29,30	ROCKETDYNE	PRA TANK #5	0.07	0.50	0.02	0.05	0.02	<0.001	4.3
31,32	EDWARDS	DRUM 3103	0.03	0.04	<0.004	0.007	0.03	0.02	1.5
33,34	EDWARDS	N-1993	0.04	0.05	0.007	0.02	0.04	0.02	2.1
35,36	EDWARDS	DRUM 2763	0.05	0.04	<0.004	0.005	0.03	<0.001	0.7
37,38	TRW	NONE	0.06	0.15	0.03	0.02	0.02	0.05	0.3
40	HAM. STAND.	4318	0.04	0.08	<0.004	0.002	0.04	0.03	0.8
41	HAM. STAND.	2556	0.05	0.10	<0.004	0.005	0.06	0.04	1.2
46,47	EDWARDS	DRUM 1810	0.04	0.06	<0.004	0.003	0.008	0.03	1.9
48,49	ROCKY MTN. ARS.	DODX 17074	0.09	0.04	<0.004	0.002	0.01	0.02	3.5
50,51	ROCKY MTN. ARS.	DODX 17067	0.08	0.05	<0.004	0.002	0.02	0.02	1.4

TABLE V

TRACE METALS IN HYDRAZINE SAMPLES SUBMITTED FOR IMPURITY SURVEY
SEMIQUANTITATIVE (EMISSION SPECTROSCOPY)

UARL I.D. #	USER	SAMPLE I.D.	Ba ppm	Ca ppm	Al ppm	Mo ppm	Cu ppm	Si ppm	Mn ppm	Mg ppm	Pb ppm	Cs ppm	Ag ppm
1,3	ROCKET RESEARCH	H-1799	<0.01	0.02	0.01	0.001	0.005	0.02	<0.001	0.002	<0.001	0.006	<0.001
5,6	SHELL	H-4670	<0.01	0.03	0.008	0.009	0.008	0.02	0.002	0.006	<0.001	0.02	<0.001
7,8	CHINA LAKE	H-2002	0.02	0.02	0.005	0.001	0.002	0.02	0.002	<0.001	<0.001	0.001	<0.001
10,11	BELL	S/N 929	<0.01	0.10	0.007	0.01	0.008	0.02	0.005	0.03	0.02	0.01	0.005
12,13	CHINA LAKE	2237	<0.01	0.001	0.006	0.002	0.005	0.05	0.002	0.01	<0.001	0.006	<0.001
14,15	JPL	H-3155	<0.01	0.03	0.008	0.002	0.01	0.02	0.002	0.01	<0.001	0.02	<0.001
17,18	SUNDSTRAND	H-110	<0.01	0.04	0.003	0.002	0.01	0.02	0.002	0.02	<0.001	0.006	<0.001
19,20	CHINA LAKE	Lot 681	<0.01	0.04	0.02	0.004	<0.002	0.008	0.001	<0.001	<0.001	0.006	<0.001
21,22	CHINA LAKE	H-3139	<0.01	0.02	0.01	0.002	0.007	0.02	<0.001	0.006	<0.001	0.002	<0.001
23,24	CHINA LAKE	H-3456	<0.01	0.04	0.01	0.004	0.008	0.03	0.03	0.008	<0.001	0.07	<0.001
25,26	CHINA LAKE	H-794	<0.01	0.02	0.001	0.001	<0.002	0.008	<0.001	0.002	<0.001	<0.001	<0.001
27,28	CHINA LAKE	H-2604	<0.01	0.02	0.002	0.001	0.008	0.008	<0.001	0.002	<0.001	0.007	<0.001
4	LOCKHEED	A 3937	<0.01	0.008	0.01	0.01	0.006	0.04	<0.001	0.02	0.02	<0.004	<0.004
29,30	ROCKETDYNE	PRA TANK #5	<0.01	0.006	0.03	0.01	0.01	0.07	0.03	0.04	0.006	0.02	<0.004
31,32	EDWARDS	DRUM 3103	<0.01	0.008	0.01	0.008	0.001	0.05	<0.001	0.04	0.004	<0.004	<0.004
33,34	EDWARDS	H-1993	<0.01	0.008	0.01	0.002	0.02	0.06	0.002	0.06	0.005	0.007	<0.004
35,36	EDWARDS	DRUM 2963	<0.01	0.008	0.02	0.01	0.006	0.08	<0.001	0.07	0.02	<0.004	<0.004
37,38	TRW	NONE	<0.01	0.008	0.008	0.002	0.006	0.02	0.003	0.004	0.003	0.03	<0.004
40	HAM. STAND.	4318	<0.01	0.01	0.006	0.004	0.004	0.02	<0.001	0.007	0.02	<0.004	<0.004
41	HAM. STAND.	2556	<0.01	0.01	0.02	<0.001	0.006	0.01	<0.001	0.007	<0.002	<0.004	<0.004
46,47	EDWARDS	DRUM 1810	<0.01	0.008	0.01	<0.001	<0.002	0.04	<0.001	0.01	<0.002	<0.004	<0.004
48,49	ROCKY MTN. ARS.	DODX 17074	<0.01	<0.004	0.02	<0.001	0.005	0.14	<0.001	0.01	<0.002	<0.004	<0.004
50,51	ROCKY MTN. ARS.	DODX 17067	<0.01	<0.004	0.03	<0.001	0.004	0.03	<0.001	0.01	<0.002	<0.004	0.02

TABLE VI

Particle Size Distribution (Particles/100 ml)

UARL I.D.#	User	Sample I.D.	Size Range (in microns)				
			6-10	11-25	26-50	51-100	>100
1,3	Rocket Research	H-1799	23	7	2	0	0
5,6	Shell	H-4670	32	13	10	1	0
7,8	China Lake	H-2002	3	4	2	1	0
10,11	Bell	S/N 929	31	14	5	0	1
12,13	China Lake	2037	11	3	2	1	1
14,15	JPL	H-3155	10	3	2	0	0
17,18	Sundstrand	H-110	21	10	7	1	0
19,20	China Lake	Lot 681	3	5	1	0	0
21,22	China Lake	H-3139	3	2	0	0	0
23,24	China Lake	H-3456	50	40	32	17	10
25,26	China Lake	H-794	10	6	1	0	0
27,28	China Lake	H-2804	13	6	2	1	0

Table VII

Results of Spectral Scan Analyses

(Fig. 12)

Sample	Particle Type	Number Analyzed	Elements Observed	
			Major	Minor
No. 1	Glassy Plates	3	Fe,K,Ti,Si	Al,Mn
	Metallic	1	Fe	Mn
	Agglomerates: Stable	2	Fe,Si,Ca,P,K	Cr,S,Ni,Al
No. 5	Glassy Plates	2	K,Si,Fe,Al	Ti,Mg
	Agglomerate: Unstable	1	Fe,Cl	Si,Na
	Agglomerate: Stable	1	K,Si,Fe,Ti	Al,Ca,Cl,Mg
	Small-Orange Fluorescent	1	Si,Cl	Ca,Ti
No. 7	Glassy Plates	2	Si,Fe,K,Ti	Al
	Metallic	1	Fe	Mn,Cr
	Agglomerates: Stable	2	Fe,Ca,Si	Al,K,Mg
	Agglomerates: Unstable	2	Cl,Fe,Si	Na,Ca
No. 23	Agglomerates: Unstable	2	Si,Fe,Cl	Ca,Al,K,Cr
	Metallic	1	Fe	Ti,Al,Cl,Si,Ca
	Glassy Plates	2	K,Fe,Si,Ti	Al,Ca,Cr
	Agglomerates: Stable	2	Fe,K,Si,Ca	Al,Ti
Control	Oxide Particle	1	Ti	Al
	Particles: Unstable	3	Si,Ca,Cl,Ti	K,S

Table VIII

Results of Spectral Scan Analyses

(Fig. 13)

Sample	Particulate Type		Number Analyzed	Elements Observed	
				Major	Minor
No. 4	Agglomerates:	Stable	2	Ca	P,Si
	Glassy Plate		1	Si	--
	Metallic		2	Fe,Cl	Cr,Cl
No. 16	Agglomerates:	Stable	1	Ca	Si,Ca
	Agglomerates:	Unstable	2	K,Fe,Si,Ca	Mg,Al,Ti
	Glassy Plate		2	K,Fe,Si	Ti,Al,Mg,Ca
No. 19	Agglomerates:	Unstable	4	Ca,Al,K,Cl	Si,S,Mo,Ca
	Metallic		1	Fe,Cr,Ni	--
No. 25	Agglomerates:	Stable	4	Ca,Si,Fe	Fe,Cd,K,Cl,Al
	Agglomerates:	Unstable	1	Ca	Fe,Si

Table IX

Results of Spectral Scan Analyses

Sample	Particle Type	Number Analyzed	Elements Observed	
			Major	Minor
No. 9	Agglomerates: Stable	3	Ca,Fe,Si	Cr,Fe,Ca,Al
	Agglomerates: Unstable	3	Si,Ca,Fe,Cr	Fe,Al,Mg
No. 12	Glassy Plate	2	Fe,Cr,K,Si, Al,Ca	Ti
	Agglomerates: Stable	1	Ca,Fe	Si
	Agglomerates: Unstable	2	Ca,P,Cl	Fe,Cr,Ca
No. 14	Agglomerates: Stable	1	Cl,Fe,Ca	Cr,Si
	Agglomerates: Unstable	3	Ti,Ba,Cl,Ca, Fe,Si,K,P	Fe,Si,S, Cl,Al,Cr
	Metallic	1	Fe	--
No. 18	Glassy Plate	1	Si	--
	Agglomerates: Stable	2	Ca,Fe	Si,Cr
	Agglomerates: Unstable	3	Ti,Ca,Cl	Si,Fe

Table X

Results of Spectral Scan Analyses

(Fig. 14)

Sample	Particulate Type	Number Analyzed	Elements Observed	
			Major	Minor
No. 21	Agglomerates: Stable	2	Ti,Ca,Si	Ca,Fe
	Agglomerates: Unstable	3	Ca,K,Cl	Si,Fe,Ti,K
No. 27	Agglomerates: Stable	1	Si	---
	Agglomerates: Unstable	3	Ca,Fe,Al,Cl, Si,K	Si,K,Cd,S, Cl,Fe
No. 29	Agglomerates: Stable	1	Si,Ca,Fe	Al
	Agglomerates: Unstable	2	Ca,Cl	Fe,S,K,Cl
	Metallic	2	Fe,Si	Si,Ca
No. 31	Agglomerates: Stable	2	Fe,K,Si,Ti	Ca,Mg
	Agglomerates: Unstable	2	K,Fe,Ti,Si	Mg,Ca
	Metallic	1	Fe,Si	K,Ca,Ti
No. 40	Agglomerates: Stable	1	Mg,Ca	Fe
	Agglomerates: Unstable	3	Ca,Cl,K	K,S
No. 41	Agglomerates: Stable	2	Fe,Ca,K,Si	K,Ti
	Agglomerates: Unstable	1	Ca,Fe,Si	K
	Metallic	2	Fe,Cr	Ti,Ca

Table XI

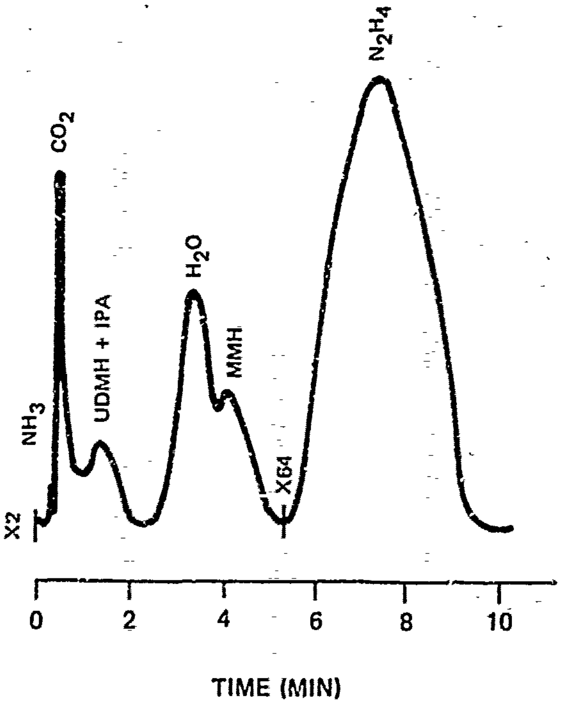
Results of Spectral Scan Analyses

(Fig. 15)

		Number	Elements Observed	
Sample	Particle Type	Analyzed	Major	Minor
No. 33	Agglomerates: Stable	3	Fe,Si,K,Ti, Al	Al,Ti,K
	Metallic	2	Fe,Si,K	Ti,Al
No. 35	Agglomerates: Stable	3	Fe,Cr,Al,S, Si,K	Cr,Ca,Ni, Al,Ti
	Metallic	2	Fe,Si,K	Al,Ti,Si
No. 37	Agglomerates: Stable	3	Ca,Fe	--
	Agglomerates: Unstable	2	Ca,Cl,Fe,Si	--
No. 46	Agglomerates: Stable	1	Cr	Si,Fe,Cl
	Agglomerates: Unstable	1	Si	Al,Cl,Ca
	Metallic	3	Si,Fe,K	Fe,K,Ca,Mg, Ti,Al
No. 48	Agglomerates: Unstable	2	Al,Si	--
	Metallic	3	Fe,Al,K,Si	Ca,Fe
No. 50	Agglomerates: Stable	1	Ca	S
	Agglomerates: Unstable	3	S,Cl,Ca,Si	Cl,Al
	Metallic	1	Al	--

CHROMATOGRAPHIC SEPARATION OF HYDRAZINE IMPURITIES

(COLUMN A – 1m x 1/4 IN. OD 33% CARBOWAX 400 ON 90/100 MESH ANAKROM AB)



GAS CHROMATOGRAPH

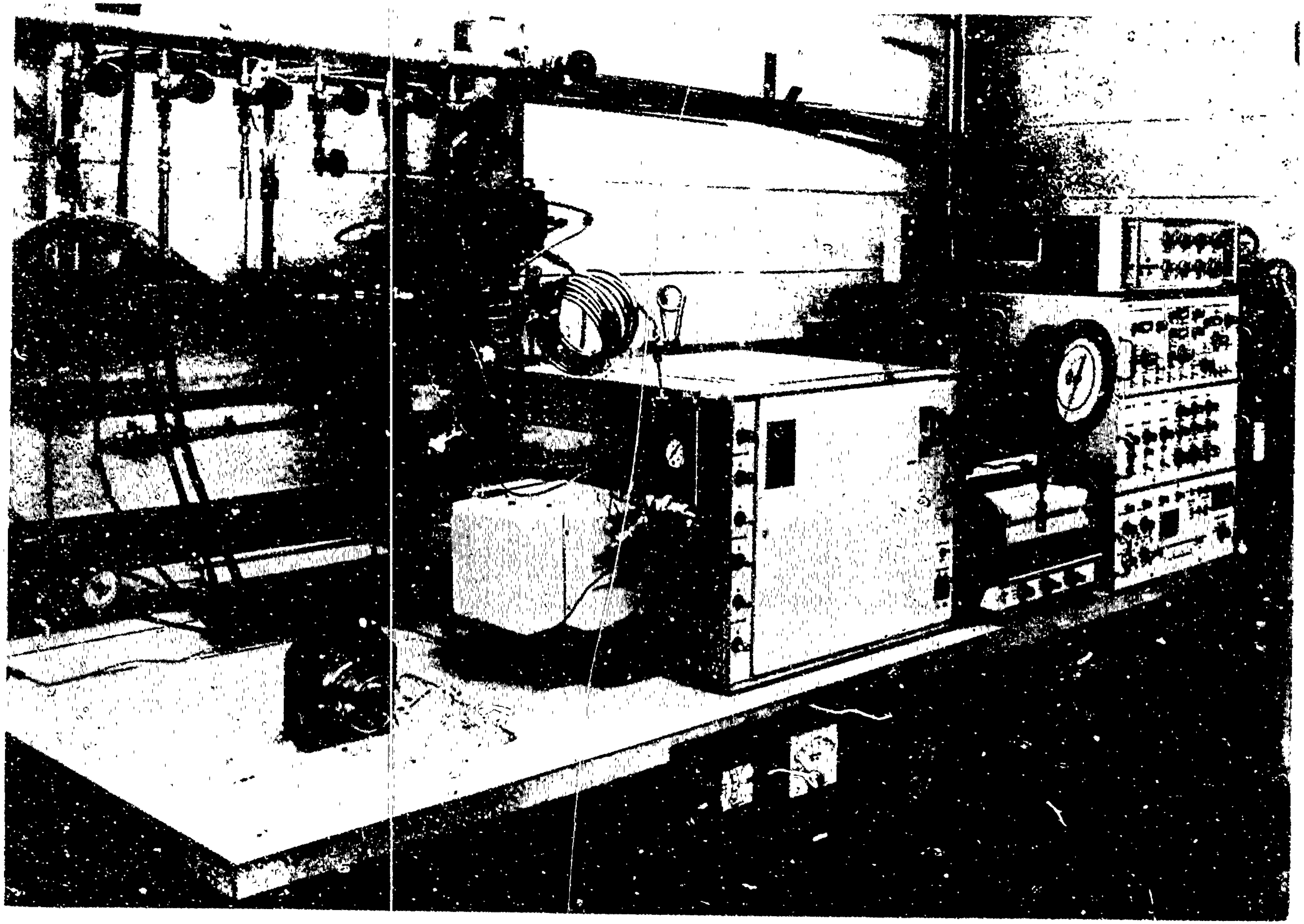


FIG. 2

FIG. 3

CHROMATOGRAPHIC SEPARATION OF HYDRAZINE IMPURITIES

(COLUMN A – 1m x 1/4 IN. OD 33% CARBOWAX 400 ON 90/100 MESH ANAKROM AB)

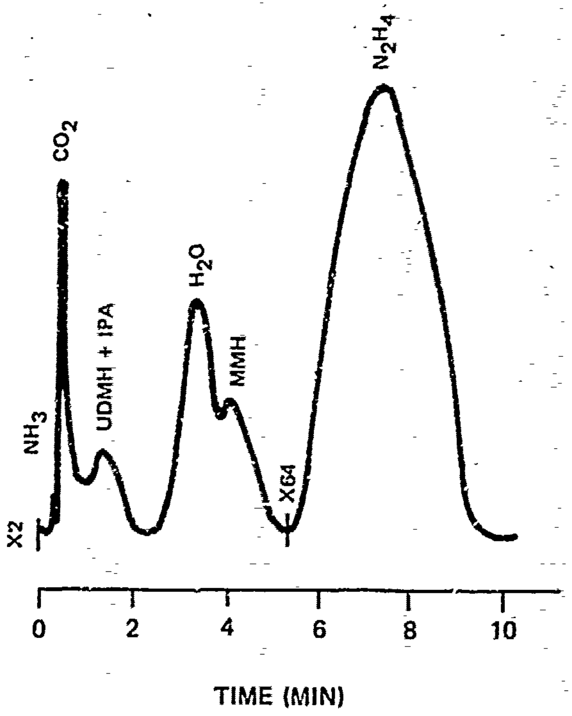


FIG. 4

CHROMATOGRAPHIC SEPARATION OF HYDRAZINE IMPURITIES

(COLUMN B – 6 FT x 1/4 IN. OD 10% DOWFAX 9N9 ON 60/80 MESH T6)

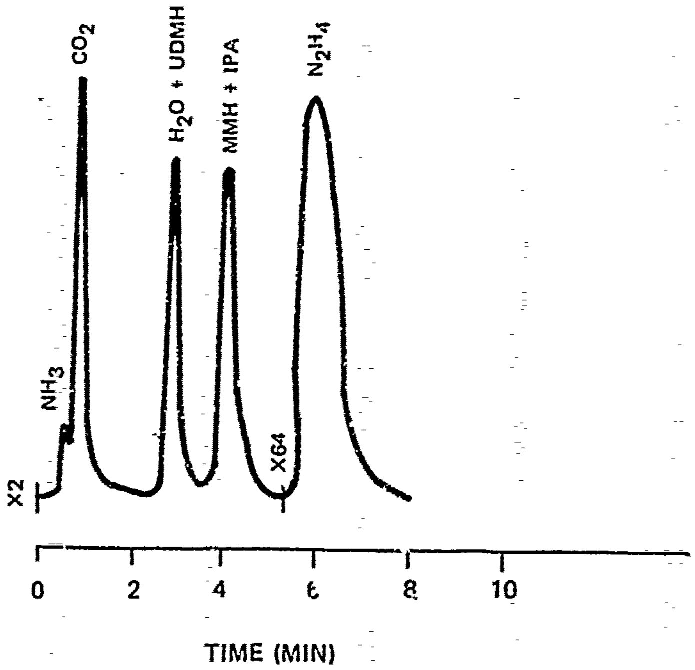


FIG. 5

CHROMATOGRAPHIC SEPARATION OF HYDRAZINE IMPURITIES

(COLUMN A + B)

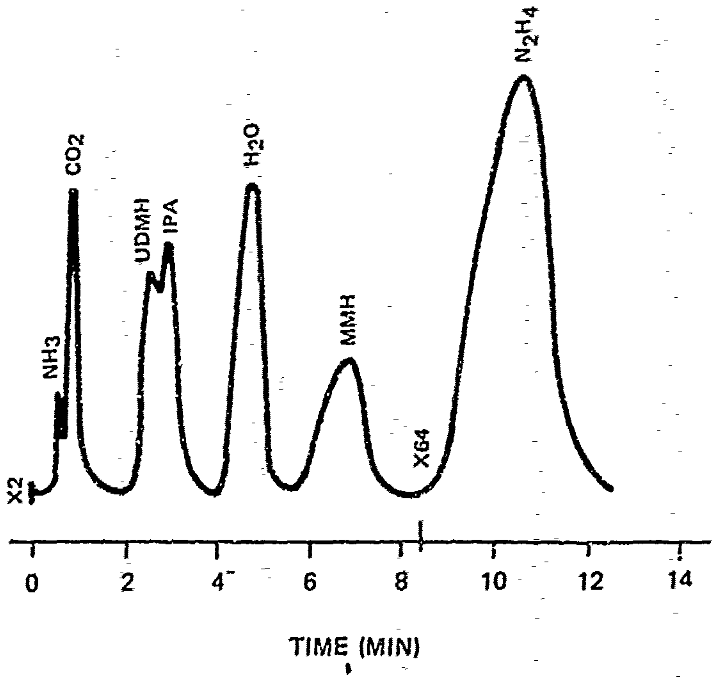


FIG. 6

CHROMATOGRAPHIC SEPARATION OF HYDRAZINE IMPURITIES

(COLUMN C – 6 FT x 1/4 IN. OD 50/80 MESH PORAPAK PS)

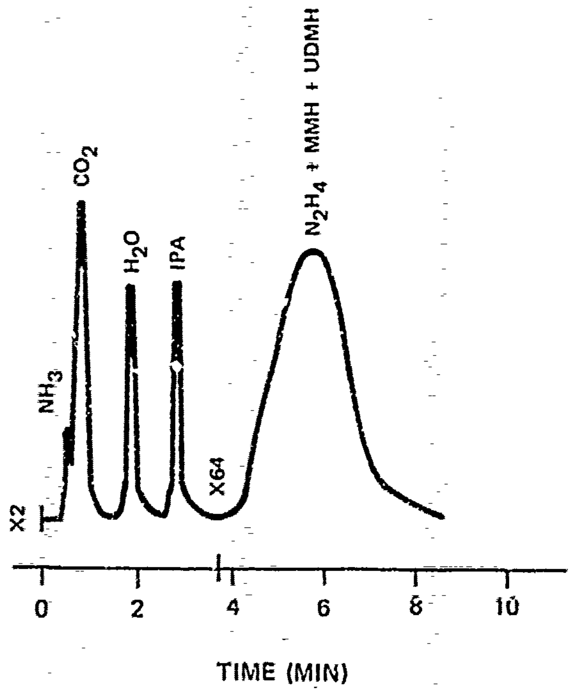


FIG. 7

CHROMATOGRAPHIC SEPARATION OF HYDRAZINE IMPURITIES

(COLUMN B + C)

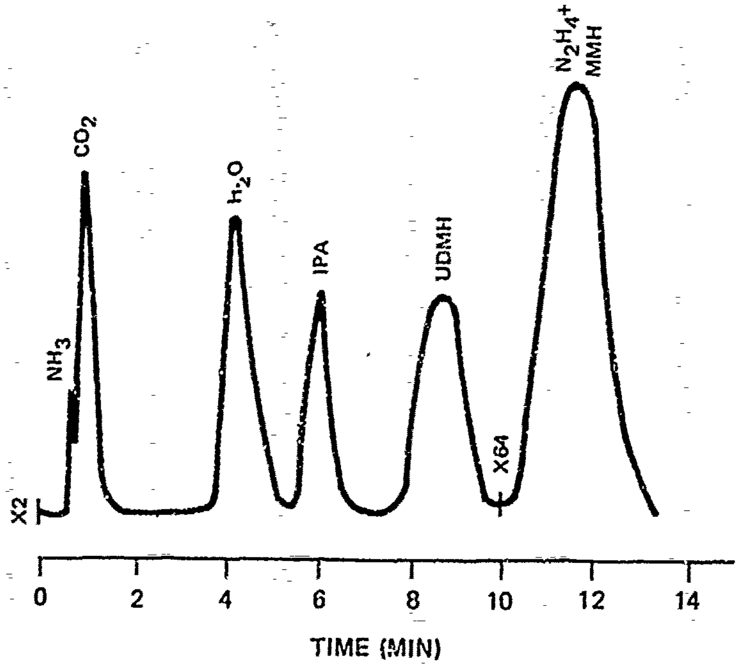


FIG. 8

ATOMIC ABSORPTION SPECTROMETER

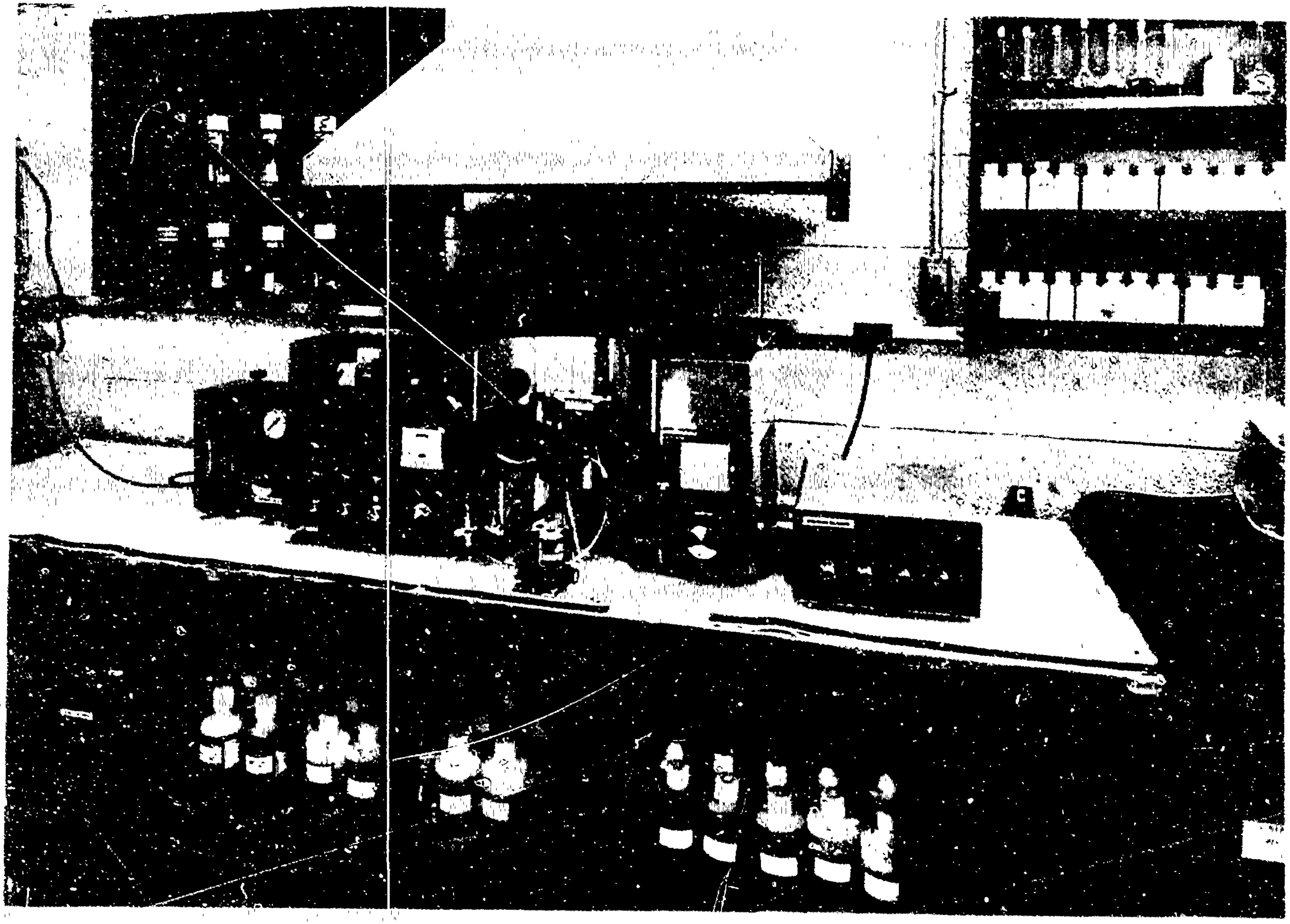
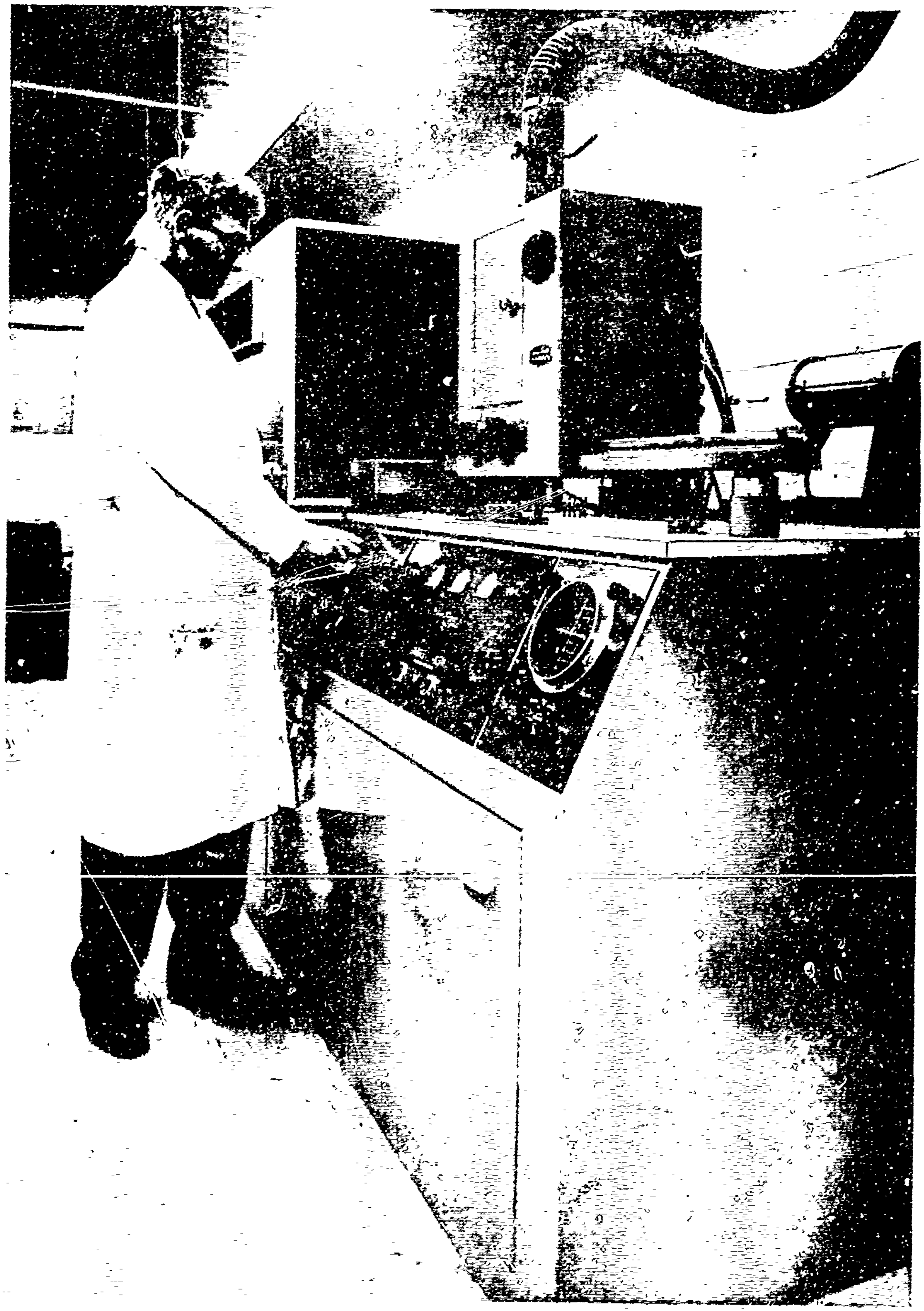
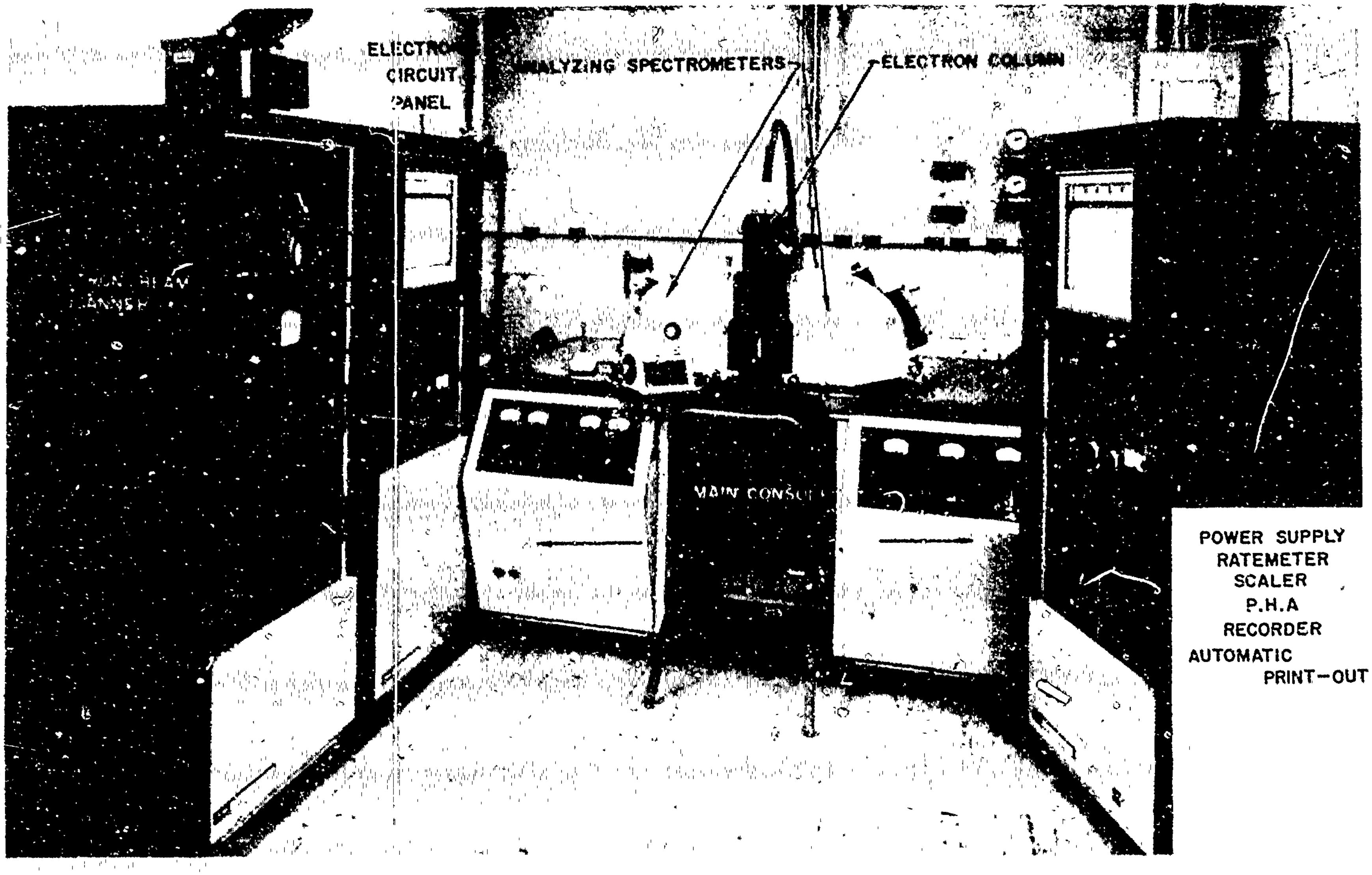


FIG. 9

EMISSION SPECTROGRAPH



ELECTRON PROBE MICROANALYZER



AMR–900 SCANNING ELECTRON MICROSCOPE

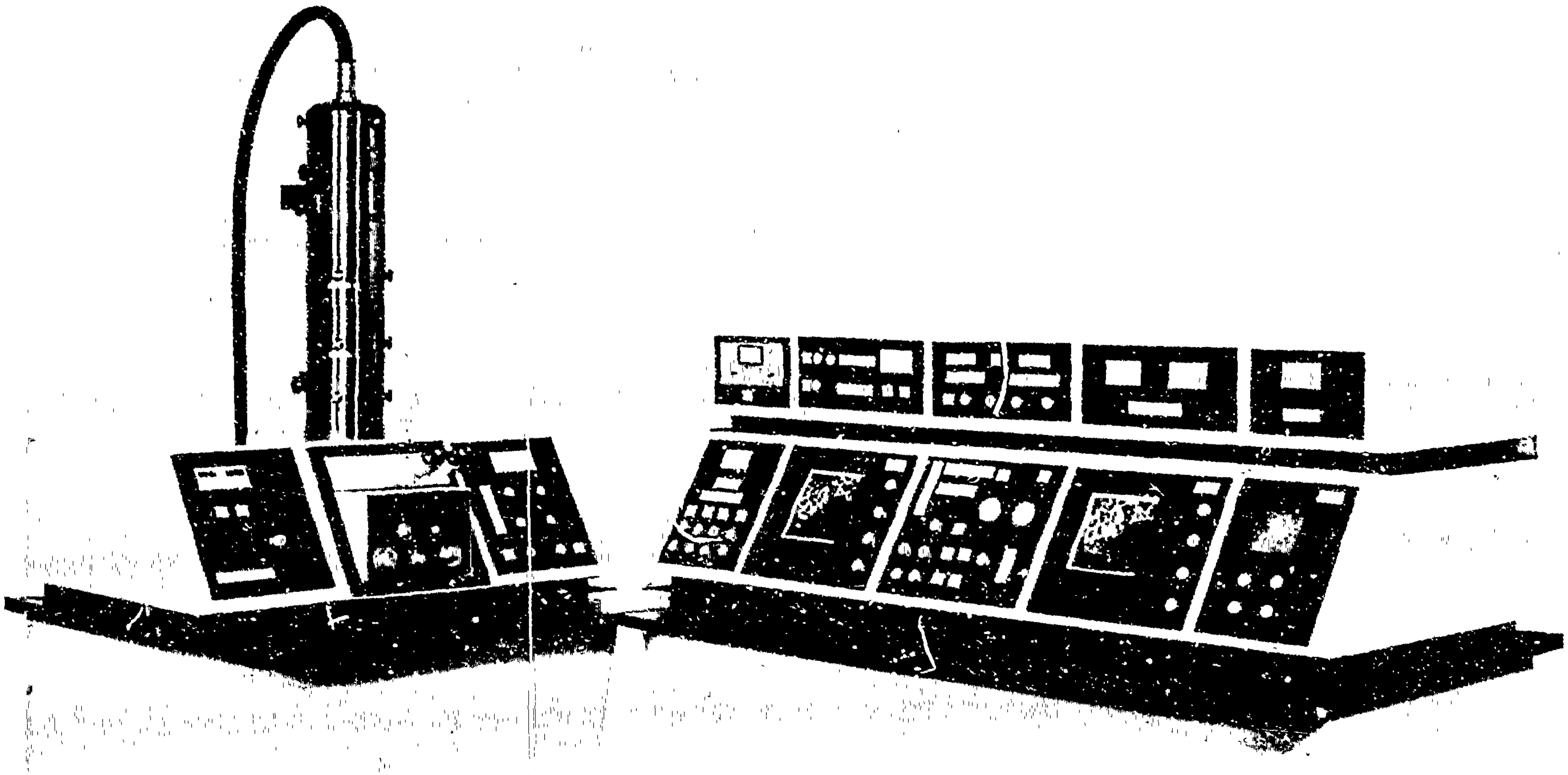
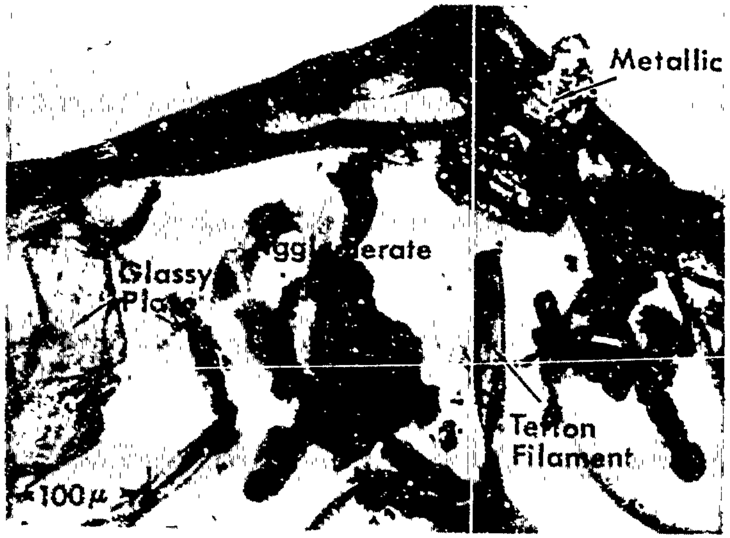


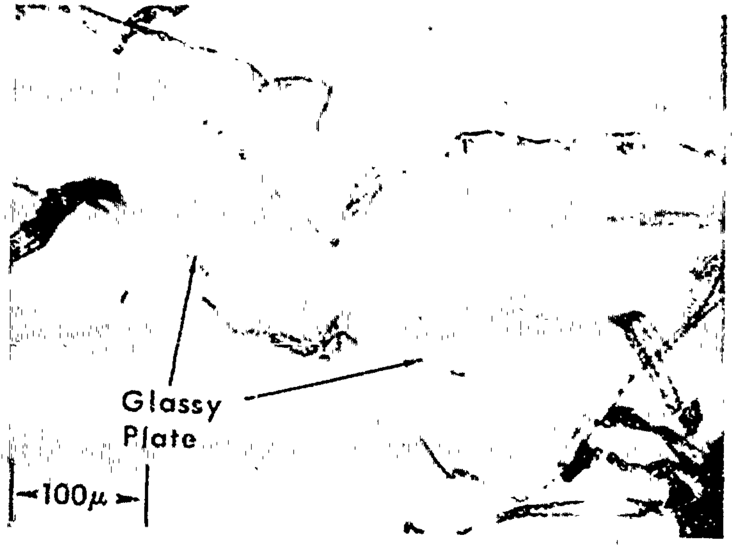
FIG. 11

FIG. 12

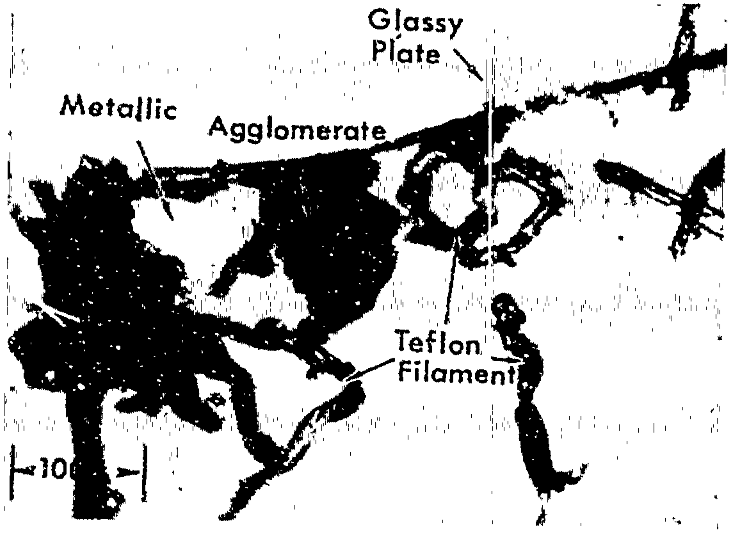
OPTICAL PHOTOMICROGRAPHS OF PARTICULATES FROM HYDRAZINE SAMPLES



SAMPLE 1 (ROCKET RESEARCH – DRUM H-1799)



SAMPLE 5 (SHELL DEVELOPMENT – DRUM H-4670)



SAMPLE 7 (CHINA LAKE – DRUM H-2002)

SAMPLE 23 (CHINA LAKE – DRUM H-3456)

FIG. 13

OPTICAL PHOTOMICROGRAPHS OF PARTICULATES FROM HYDRAZINE SAMPLES

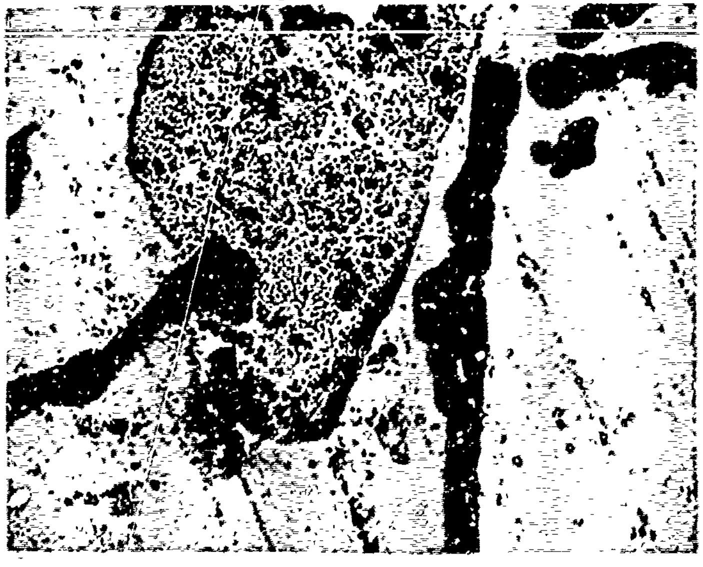
SAMPLE 4 (LOCKHEED - DRUM A - 3937) 50μ

SAMPLE 16 (SHELL - DRUM H - 4903) 50μ

OPTICAL PHOTOMICROGRAPHS OF PARTICULATES FROM HYDRAZINE SAMPLES

SAMPLE 29 (ROCKETDYNE - PRA TANK NO. 5) 50 μ

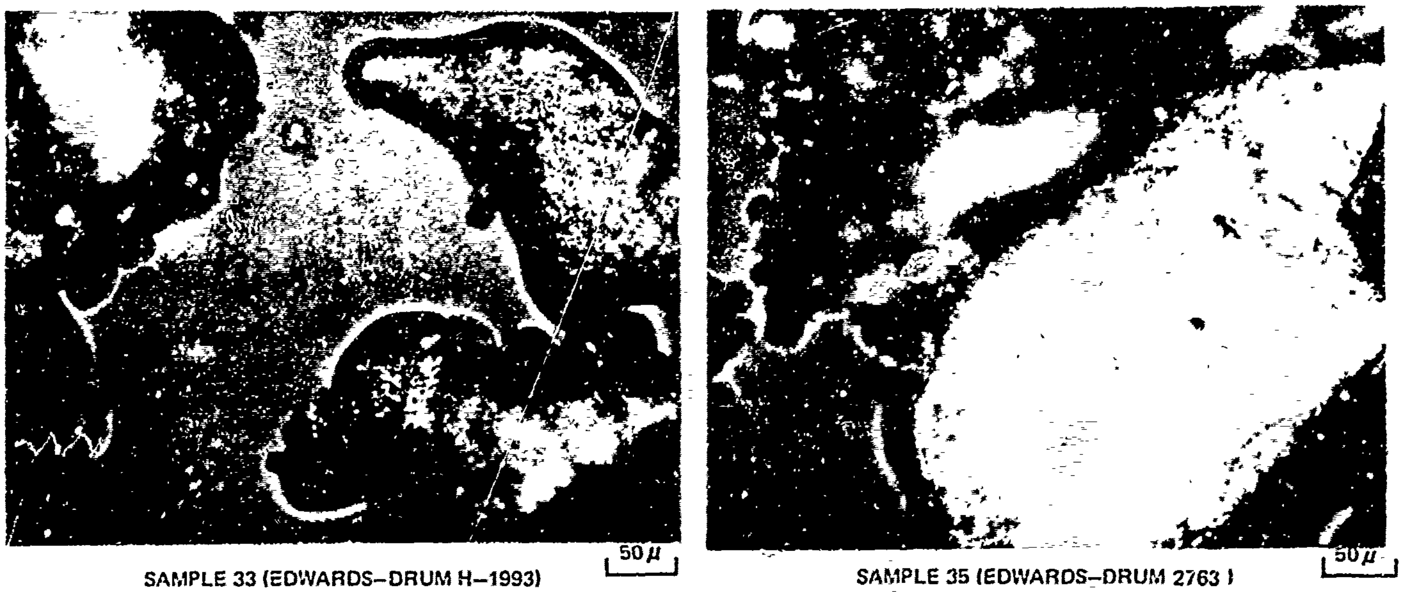
SAMPLE 31 (EDWARDS - DRUM 3103) 50 μ



SAMPLE 41 (HAMILTON STANDARD - BB 2556) 50μ

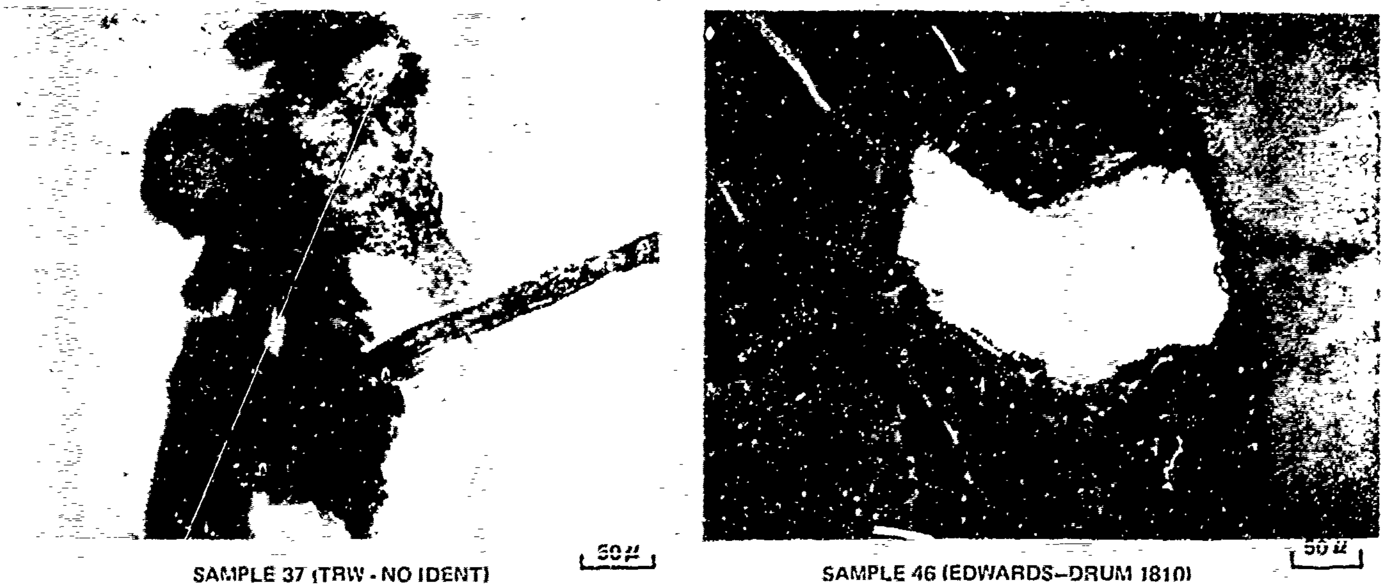
FIG. 15

OPTICAL PHOTOMICROGRAPHS OF PARTICULATES FROM HYDRAZINE SAMPLES



SAMPLE 33 (EDWARDS-DRUM H-1993)

SAMPLE 35 (EDWARDS-DRUM 2763)



SAMPLE 37 (TRW - NO IDENT)

SAMPLE 46 (EDWARDS-DRUM 1810)

SAMPLE 48 (ROCKY MTN,-DODX-17074)

SAMPLE 50 (ROCKY MTN.-DODX 17067)

FIG. 16

ELECTRON BEAM SCAN ANALYSIS OF THE "GLASSY" PARTICULATE SHOWN IN SAMPLE 5

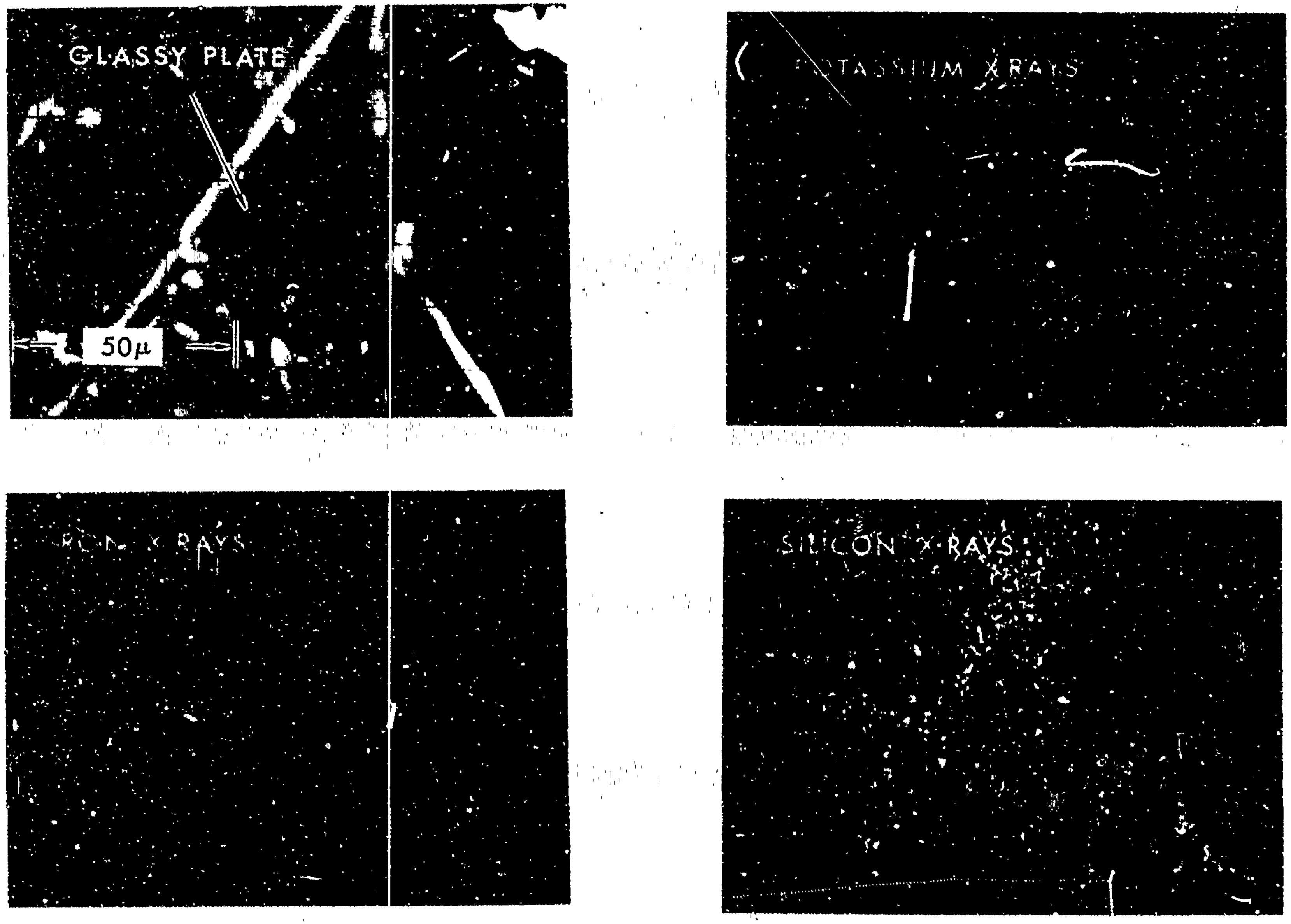
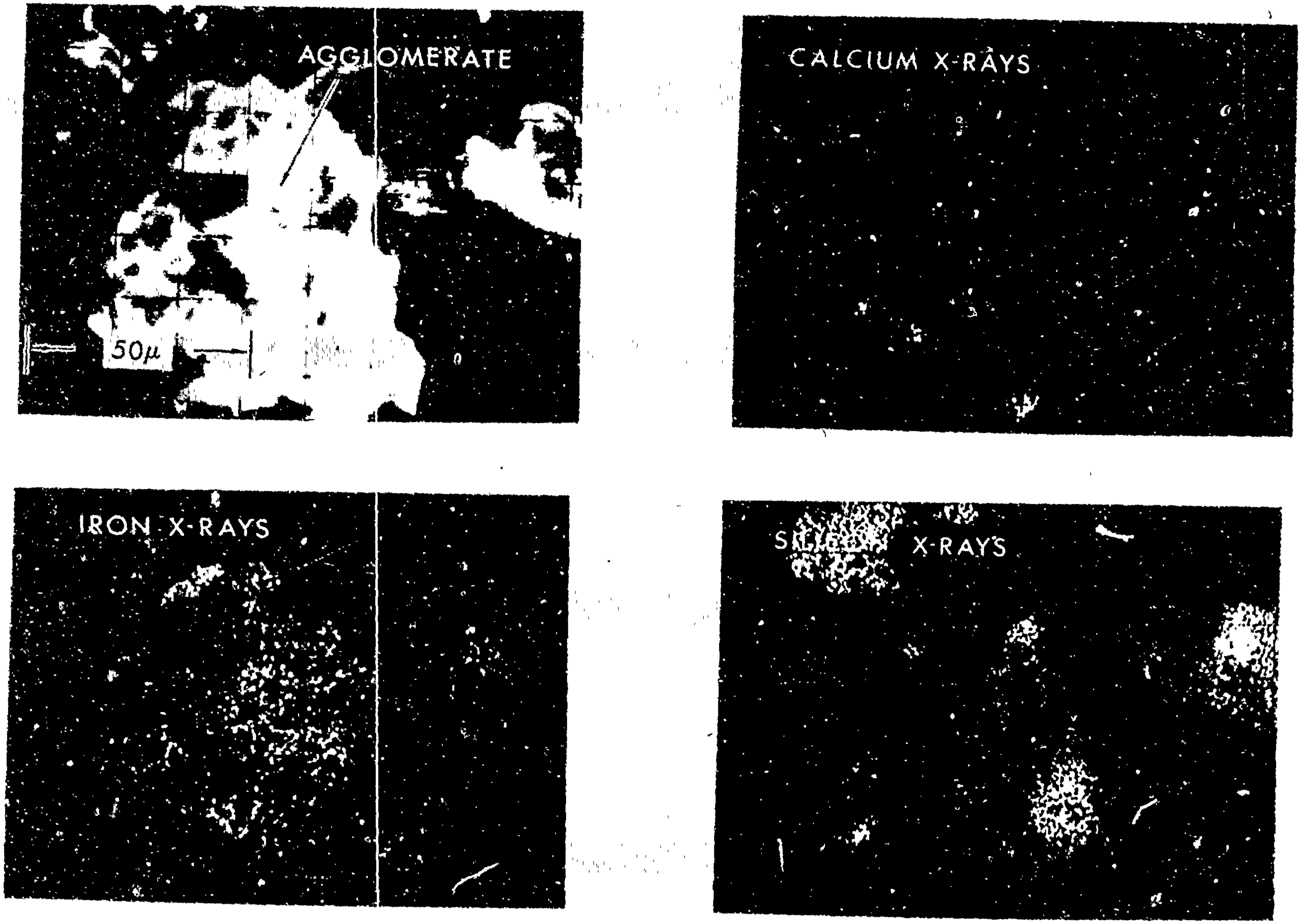


FIG. 17

ELECTRON BEAM SCAN ANALYSIS OF A TYPICAL AGGLOMERATE PARTICULATE



ELECTRON BEAM SCAN ANALYSIS OF A TYPICAL METALLIC PARTICULATE

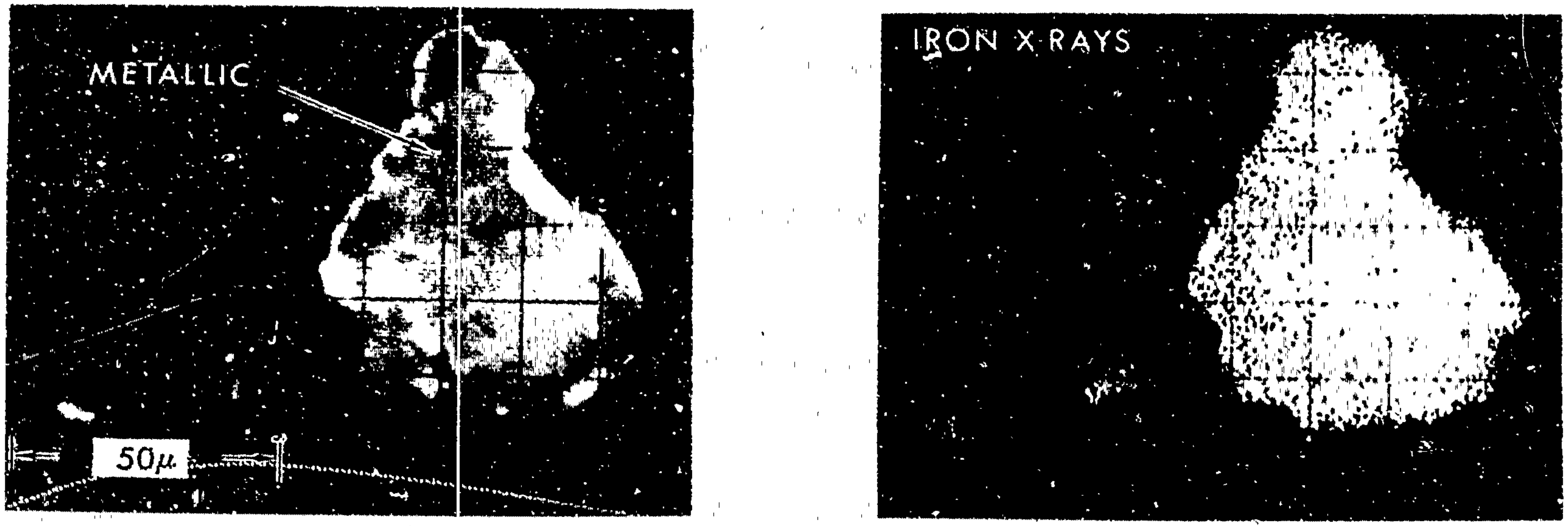
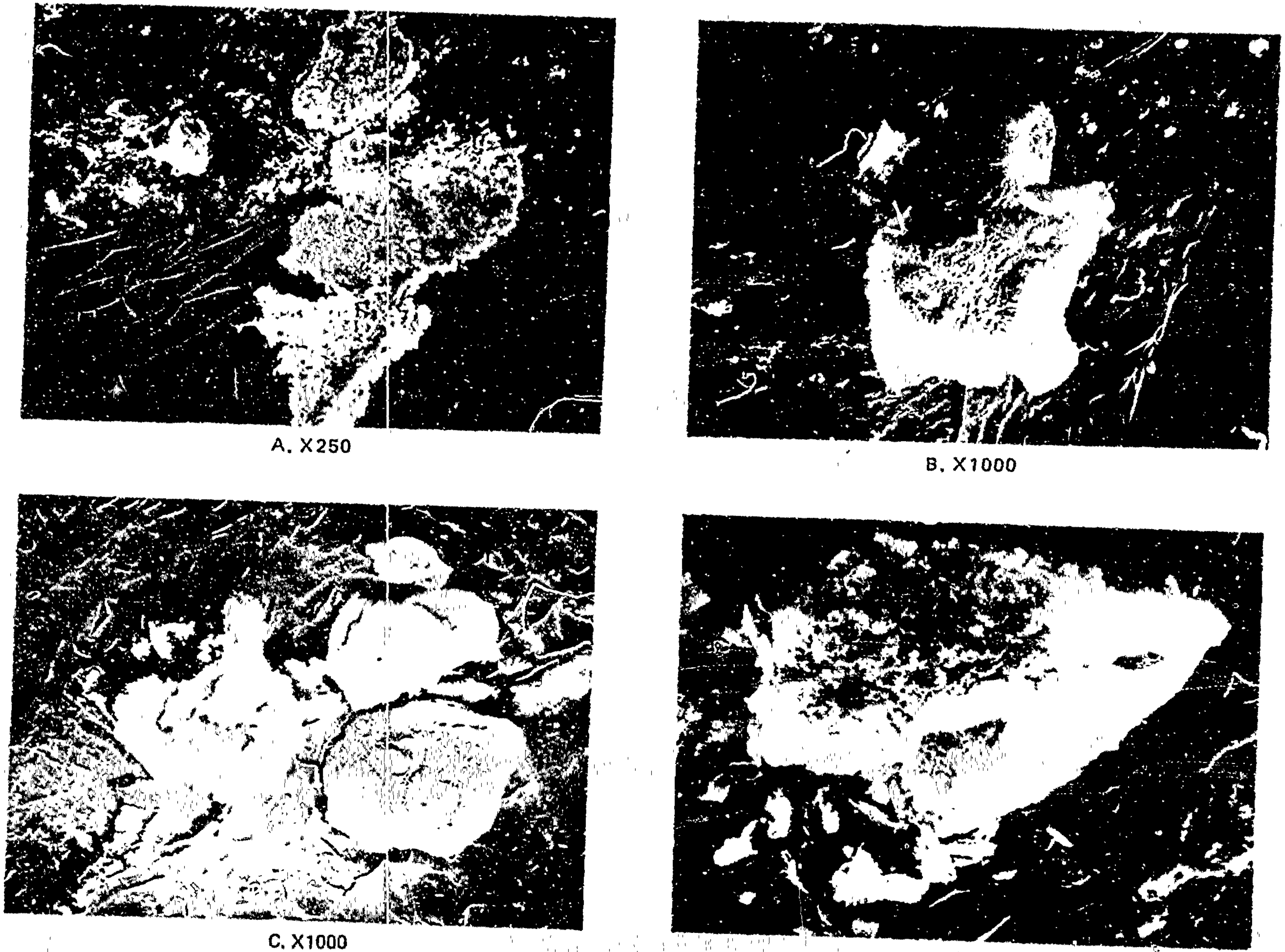


FIG. 18

FIG. 19



A. X250

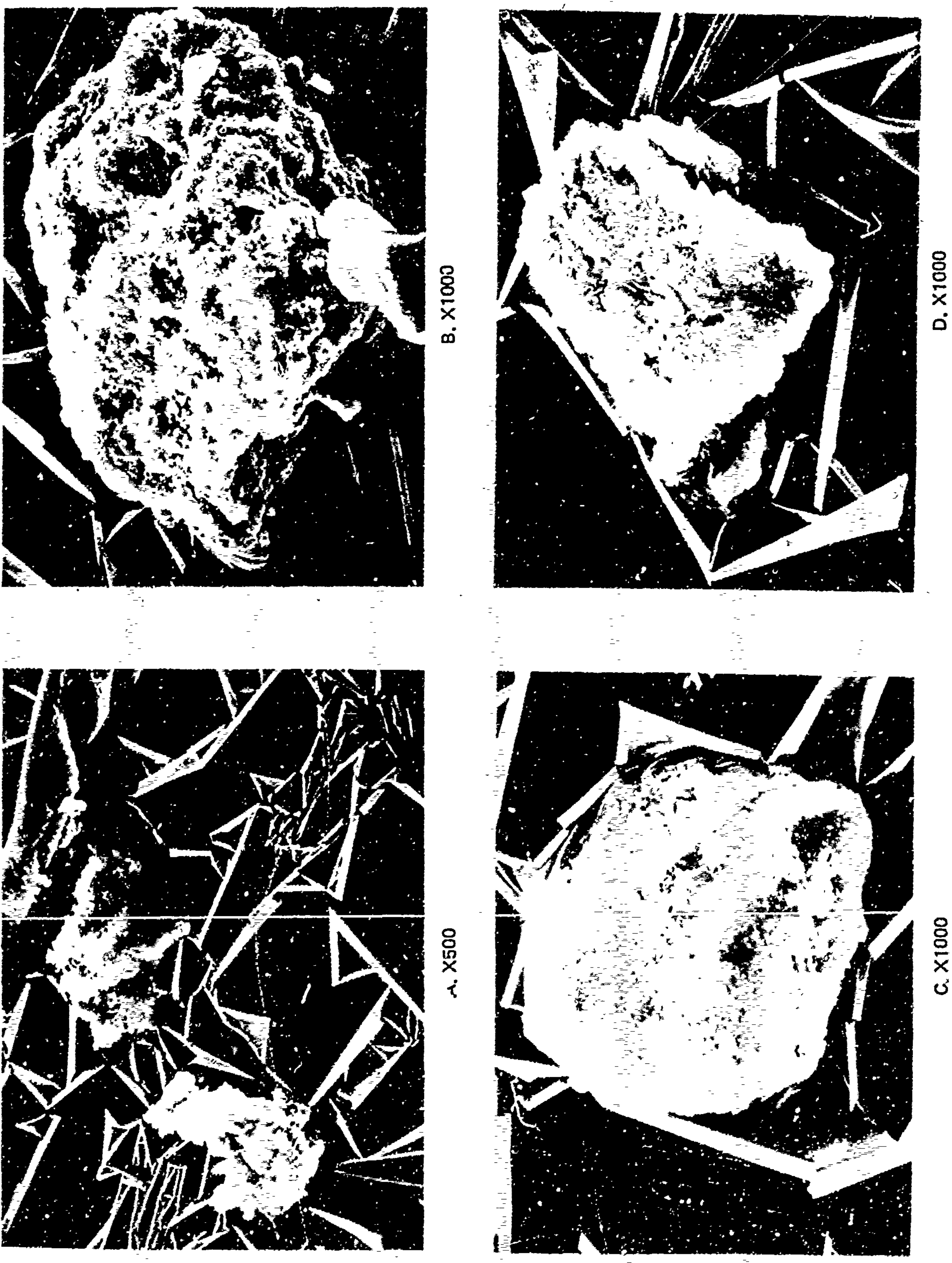
B. X1000

C. X1000

D. X2500

SCANNING ELECTRON MICROGRAPHS – SAMPLE 9
(BELL AEROSPACE, RUN S/N 929)

FIG. 20



A. X500

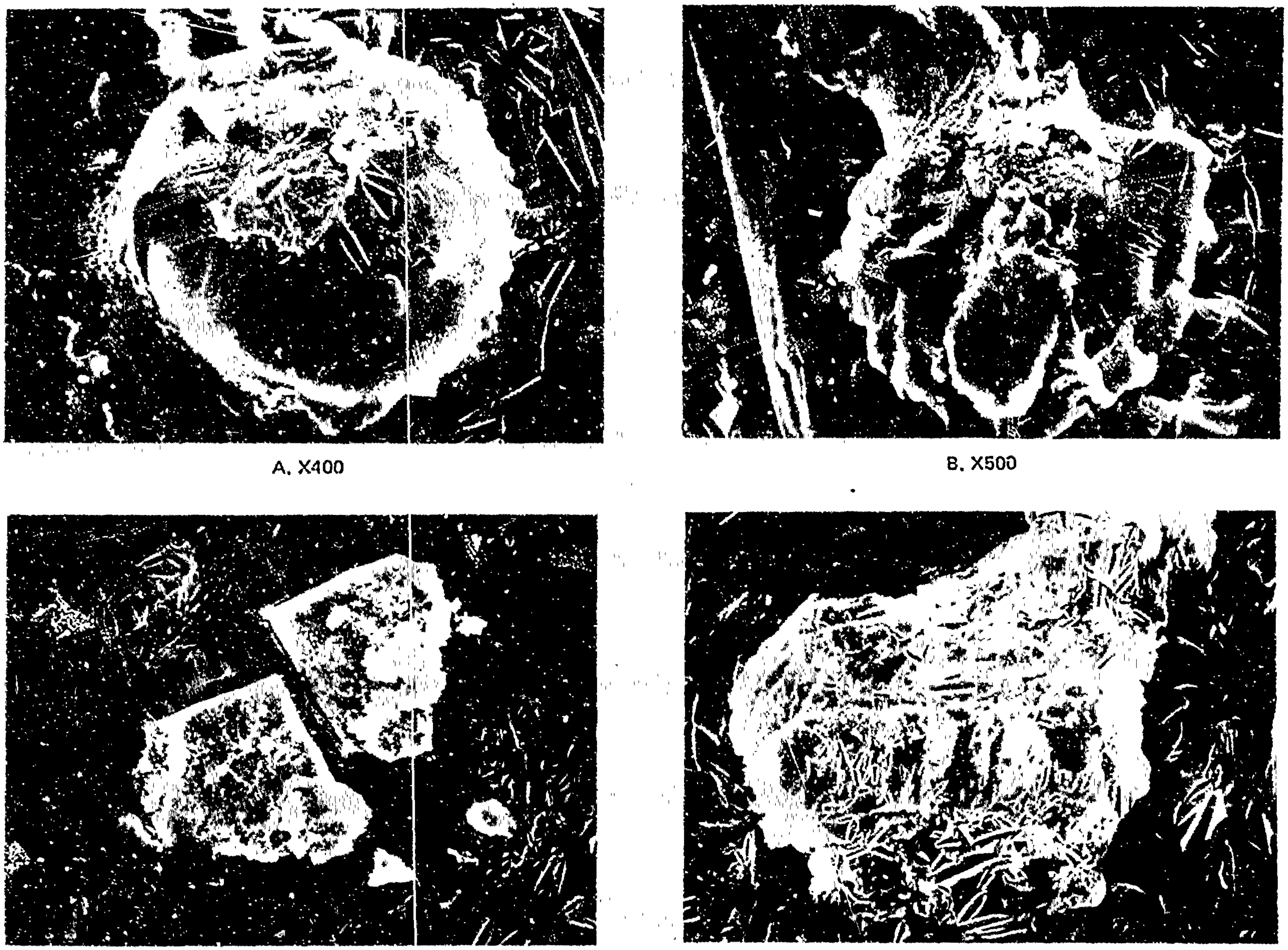
B. X1000

C. X1000

D. X1000

SCANNING ELECTRON MICROGRAPHS – SAMPLE 12
(CHINA LAKE, RUN 2037)

FIG. 21



A. X400

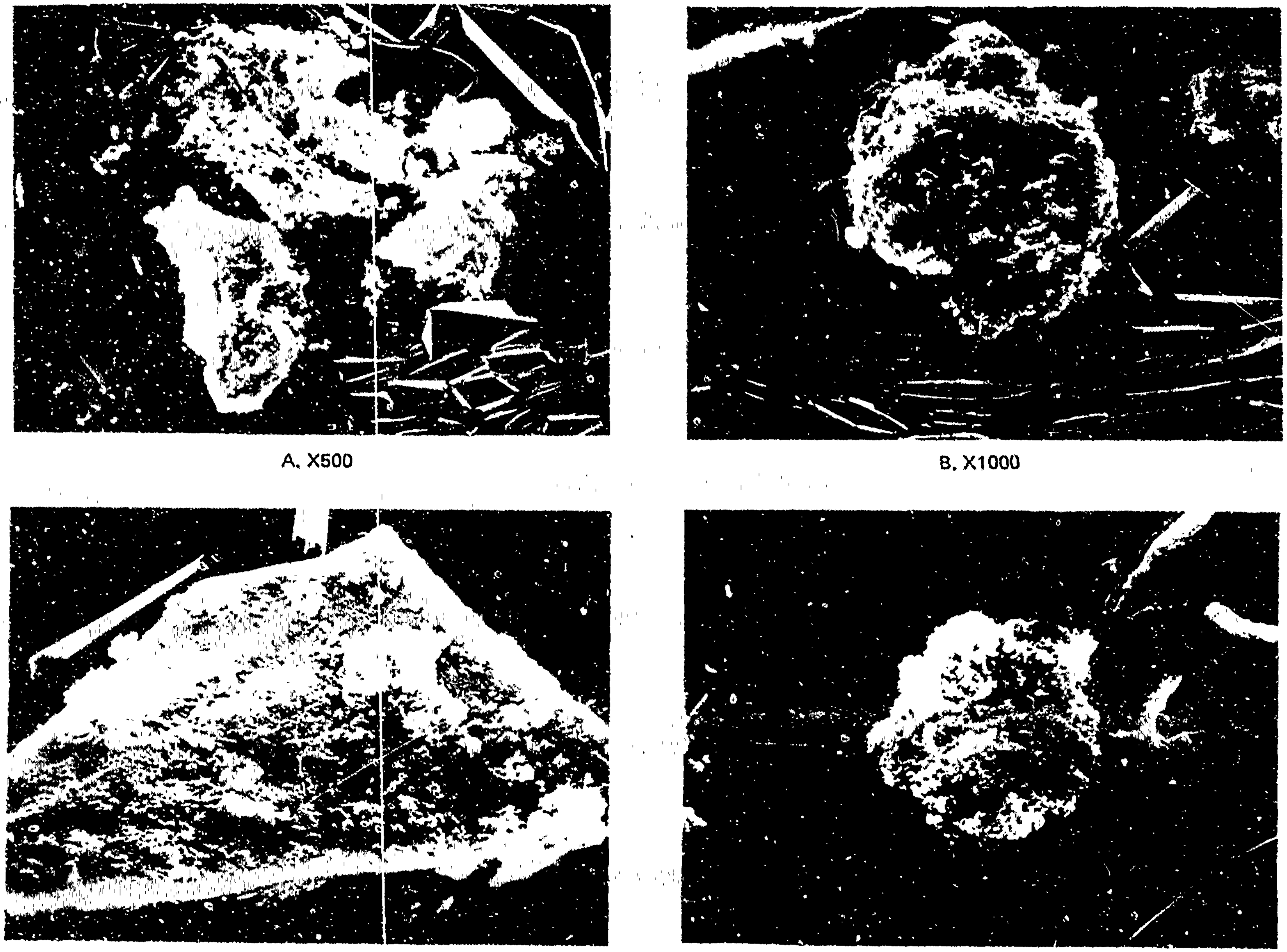
B. X500

C. X1000

D. X1000

SCANNING ELECTRON MICROGRAPHS – SAMPLE 14
(JPL, RUN H–3155)

FIG. 22



A. X500

B. X1000

C. X1000

D. X1000

SCANNING ELECTRON MICROGRAPHS – SAMPLE 18
(SUNDSTRAND, RUN H-110)

END

DATE

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9-4-73

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