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# OIL FILTER ANALYSIS TECHNIQUE RESEARCH PROGRAM

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Richard H. Hollinger The Franklin Institute Research Laboratories 20th and Parkway Philadelphia, Pa. 19103

July 1978

# Final Report for Period 25 June 1976 - 31 December 1977

Approved for public release; distribution unlimited. DDC

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APPLIED TECHNOLOGY LABORATORY U. S. ARMY RESEARCH AND TECHNOLOGY LABORATORIES (AVRADCOM) Fort Eustis, Va. 23604

### APPLIED TECHNOLOGY LABORATORY POSITION STATEMENT

This report presents the results of an effort to select a technique for determining the quantity of iron captured in a 3-micron aircraft type oil filter. Included in the overall effort was the design and fabrication of two field-usable prototype units that would use the selected technique.

Two prototype X-ray fluorescence analyzers were constructed and tested under laboratory conditions. It was demonstrated that X-ray fluorescence appears to offer one approach for obtaining information on the iron quantity in a superfine filter. However, information on particle size cannot be obtained. In addition, it was noted that the sensitivity of the analyzer decreases at higher iron loadings and that several scans of a filter are necessary in order to increase analyzer accuracy. On the other hand, when the rate of generation of the number of iron particles increases appreciably, it is believed that the filter can be rinsed and the larger particles on the surface can then be inspected with a microscope. This technique, with additional and required modifications, may prove to be a useful method for interrogating debris found in aircraft oil filters.

Because it was found that an on-board, real-time oil debris discrimination capability is required for condition monitoring of oil-wetted components on helicopters, further work on the X-ray fluorescence analyzer is not planned.

Mr. Dominick P. Lubrano of the Military Operations Technology Division served as project engineer for this effort.

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## PREFACE

This program was carried out for the Applied Technology Laboratory, U.S. Army Research & Technology Laboratories (AVRADCOM), Fort Eustis, Virginia, under Contract DAAJ02-76-C-0038. The program was conducted in the Physical and Life Sciences Division of The Franklin Institute Research Laboratories. Mr. Dominick Lubrano of AVRADCOM served as the Technical Monitor for the program, and supplied much useful information and encouragement.

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#### INTRODUCTION

The proposed use of filters having an absolute rating of three micrometers in the recirculating lubrication oil systems of some U.S. Army aircraft is expected to present a problem for those systems in which spectrometric oil analysis is used to give a warning of incipient failure of system components. With the highly efficient filters, little or no suspended metal would remain in the oil, and the result of the spectrometric analysis would be meaningless. The metal particles which normally would be suspended in the oil would, however, be retained on the oil filter which could then be used as a continuing record of the metallic debris generated by the system components, providing a suitable quantitative, nondestructive test technique could be evolved for the analysis of the metal on and in the filter matrix. The object of the work described in this report was to determine which of the many available analytical techniques was the most promising and to reduce the technique to a prototype, flight line usable instrument. Two such prototypes were to be prepared.

#### SCREENING OF TEST METHODS

#### LITERATURE SURVEY AND ANALYSIS

A literature review was conducted by the Science Information Service Department of the Franklin Institute Research Laboratories in order to identify possible test methods that appeared capable of determining the quantity of metal on a filter, the type of metal, and the size distribution of the metal particles. Seventeen possible methods were identified and screened for applicability.

Atom Probe - This method utilizes a field ion microscope and a time-offlight mass spectrometer. While useful for metallurgical analysis, the cost and degree of complication of the apparatus make the method unsatisfactory for the filter analysis program.

Atomic Absorption Spectroscopy - This method is a standard analytical tool and might possibly be adapted for analysis of oil in the field. Since the method is not adaptable to direct analysis of metal retained on a filter, it is not satisfactory for use. However, if the filter can be effectively back-flushed to remove metallic particles without damage to the filter, the atomic absorption spectrometer technique could be used as a backup or check technique for another more direct method.

<u>Auger Electron Spectroscopy</u> - This method is not applicable because of the degree of complication of the apparatus and the fact that it is mainly useful in the analysis of surface layers or very thin films.

Atomic Ionoluminescence - This method involves the bombardment of a surface with positive ions and recording the resultant light emission of the surface atoms. The method applies largely to surfaces and is still in the developmental stage. It is not recommended.

<u>Ion Backscattering</u> - The test surface is bombarded by a low-energy ion beam and the energy of the backscattered ions is measured. The method is effective for thin film analysis only and is not recommended.

Elastic Backscattering - The method uses bombardment of a test surface by heavy, charged particles to determine the composition. The method applies to thin surface layers and is not recommended.

Electron Probe Microscope - Bulk, cost, and degree of complication make this instrument undesirable for field use.

<u>Photoelectron Spectroscopy (ESCA)</u> - The method is relatively new and is used chiefly for the anlysis of surface layers approximately ten angstroms thick. The method is not recommended.

Laser Backscattering - The method uses monochromatic laser radiation of a test surface, and the difference in wavelength of the incident and backscattered radiation may be calibrated with reference standards to permit identification of the sample components. Since only the surface is involved, the method is not recommended

<u>Moessbauer Effect Analysis</u> - This method measures the energy of characteristic conversion electrons released from a surface. The method is largely applicable to surface layers and is not recommended.

<u>Neutron Activation Analysis</u> - The sample is bombarded with neutrons from a neutron source, and the characteristic energy of gamma radiation for each element is detected. The technique is somewhat similar to X-ray fluorescence and would be an alternative to the X-ray fluorescence technique.

Beta Particle Backscattering - This method uses beta particle bombardment of a sample. It is sensitive to oxide and organic layers on the sample and is useful chiefly for film thickness investigations. The method is not recommended for development.

<u>Electrical Capacitance</u> - A method using electrical capacitance changes was devised by Franklin Institute Research Laboratories (FIRL) to measure the rate of generation of metallic debris in lube oil. It was felt that with the metallic screens on the filter as plates, the metal particles might be detected using a similar system. Calculations showed that for the anticipated changes in metal particle concentration between tests, the capacitance changes would be undetectable. This method is therefore not recommended.

Magnetic Field Effects - Magnetic fields are distorted or lose strength in the presence of finely divided metal particles. The method shows some promise and should be considered.

Radio Frequency Attenuation - Very high frequency electromagnetic radiation such as radar may be reflected or dissipated by metal particles in its path. This is the principle of most passive radar countermeasure methods. The attenuation of such radiation by metal particles on a filter offers a possible method for detecting the quantity of metal particles on a filter and should be considered.

Thermal Effects - Since metal particles will heat and radiate heat at different rates from the oil on the filter, a technique using the differences in thermal properties offers promise and should be considered.

X-Ray Fluorescence - This technique uses the radiated X-rays from a metal when the metal is exposed to high-energy X-ray or gamma radiation. The radiated X-rays have a frequency (energy) characteristic of the particular metal, and the intensity of the raidation is proportional to the quantity of the particular metal present. The method appears to be applicable and should be considered.

#### LABORATORY SCREENING

Three possible physical techniques which might be used as methods to determine quantity of metal on a filter were examined in the laboratory to determine their degree of applicability.

<u>Thermal Effects</u> - The possible use of thermal energy to detect quantity of metal is based on the differences in heat capacity and thermal conductivity (or emissivity) of oil and metals. Induction heating is used to provide direct heating of metals, but it has little effect on organic materials such as lube oil. Most metal objects subjected to induction heating are relatively large. However, the possibility of heating metal particles in the range of tens of microns cannot be eliminated. Several small-scale feasibility tests were conducted in the laboratory with the objective of providing localized heating of iron particles of 44 to 77 micron size range on an oil impregnated filter paper. The particulate concentration corresponded to a concentration of 1 gram distributed over one of the three micron filters with which this program is concerned.

The apparatus shown in Figure 1 was constructed for the tests. Two transmitters were used to supply power of approximately 40 watts to an induction coil of one of three configurations: 1) one turn, flat; 2) flat spiral; and 3) solenoidal.

Spot trials were made at several frequencies in the range from 1 megahertz to 200 megahertz for each of the coil configurations. The first trial showed evidence of heating, but it was later found that the effect was from direct interaction between the magnetic field and the thermistor. These effects were minimized by inserting the thermistor after the sample was heated. There was essentially no detectable heating effect in the air space above the filters having a metal content in the area of immediate interest to the project.

In view of the probable time requirements to work out apparatus which would give measurable heating effects, plus the fact that the type of metal would not be defined in the method, this approach is not recommended for development.

<u>Electromagnetic Effects</u> - Two electromagnetic effects appeared to be promising in view of previous experience. The detection of fine metal particles embedded in a paper of fiberglass substrate by conventional electromagnetic techniques (dc through k band) might capitalize on changes in power dissipation (Q change) in a very high Q circuit at relatively low frequencies, or on the change in reflection coefficient produced by the particles in front of suitable transmission line or waveguide fed antenna at high frequencies.



It was evident that a technique that would be useful in the detection of extremely small amounts of the particulate would show extremely large responses to large amounts of the particulates. No fine particulate metal samples on paper were available at the start of the evaluation, so a gross evaluation of the two prospective methods was conducted, using small pieces of copper wire stuck to a sheet of paper with a blank piece of paper as a control. Later, oil impregnated paper samples, both with and without particulate metals, became available and the evaluations were repeated.

A 3-inch-diameter, cardboard-based, 130-turn coil of 22 gage copper wire was measured on a Boonton-Type 160 - Q meter. Q's were about 190 over the frequency range of 0.8 to 1.2 MHz. A 6-inch x 8.5-inch sheet of paper rolled into a cylinder was inserted inside the coil and Q was remeasured with no detectable change. A similar piece of paper with 20 pieces (about 1/4 inch by 1 inch) of 22 gage copper wire taped randomly to its surface was then measured, and a small change (1 to 3) in Q was observed.

A smaller coil on approximately a l-inch-diameter base was used to repeat the measurements with the paper samples outside the coil. Q's of 150 at 2.86 MHz changed to about 148 with the wire-loaded paper.

These same measurements were repeated when the filter paper particulate samples became available. Plain filter paper, oil impregnated filter paper, and oil and metallic particulate impregnated filter paper were used in the measurements. The oil and particulate impregnated paper corresponded to a metal particle concentration of 1 gram on the surface of a 3-micron filter. The samples were approximately 1 inch in diameter. With either coil, and at any of the frequencies, there was no detection of change in Q with the filter paper samples either inside, on top of, or outside the coils.

The equipment used in the evaluation of radio frequency transmission techniques consisted of a pulsed magnetron source (1 microsecond pulse width, 1000 Hz PRF), and an isolator feeding a waveguide horn antenna with another antenna a short distance away. The receiving antenna was terminated in a matched waveguide load through a directional coupler and received power was monitored using the output of the directional coupler to feed a power meter. All measurements were performed with the isolator output at approximately 8 watts of average power at a frequency of 33.2 GHz.

Preliminary measurements with the 1/8-inch pieces of 0.01-inchdiameter wire taped to a piece of paper demonstrated that fairly large changes in transmitted power could be obtained by inserting the metalloaded paper between the horns when they were about 1 inch apart.

The first set of measurements used horns with openings  $5/8 \ge 7/8$  inch. Transmission measurements were made with the horns approximately

1/8 inch and 1 inch apart. The samples were plain filter paper, oil impregnated filter paper, and oil impregnated filter paper that was loaded with approximately 5 mg of fine metal particles, simulating a one-gram filter loading. At 1/8-inch separation, measured received power varied from 8 watts empty to about 7.2 watts, but the amount of transmission seemed more sensitive to placement of the sample between the horns than to the nature of the sample. The same overall result was observed at 1inch separation with received power varying from 2.85 watts to 2.6 watts. This measurement sequence was repeated using open waveguide ends instead of horns. Results were the same, i.e., placement played a larger role than sample type. Powers varied between 3.45 and 3.0 watts for 1/4-inch separation of the waveguides and between 680 and 640 mW for 1-inch separation.

A similar set of measurements was performed with 3/8-inch x 3/8-inch horns using 1/4-inch separation and the same samples as above. Results were the same: there was no difference between the samples.

A final set of measurements using the 3/8-inch x 3/8-inch horns separated by 5.5 inches were performed. During this sequence, in addition to the 5 mg particulate loaded sample, a specially prepared filter paper impregnated with oil and 15 mg of metal particles was used, simulating a three-gram loading. Measurements were made for the samples at different locations between the horns. Results are shown in Table 1. The data were repeatable to a few milliwatts. The table shows that the 15-mg sample could easily be identified from the other samples, but the method was relatively insensitive to the 5-mg sample.

If the 5-mg loaded sample adequately represents the maximum density of metal that must be detected and measured, neither of the methods described is adequate for the task. It should be noted that the 5-mg sample, corresponding to one gram on the filter, would correspond to 83 ppm in a 12-liter lube oil system, or proportionately higher in lower volume systems. The system could thus be in the failure region before detection would be made. For this reason the methods are not recommended.

X-Ray Fluorescence - The feasibility of developing an X-ray fluorescence technique for monitoring particulate matter trapped on a three-micron filter was evaluated by a series of tests of samples cut from unused and 100-hour-service-used filters. The service-used filters were analyzed in three conditions:

- a) Oil wetted as removed from service.
- b) After flushing in the filtering direction with a clean solvent to remove the oil which may act as a filter for the X-rays.
- c) After immersion in an ultrasonically agitated solvent.

A G.E. diffractometer with a copper tube, solid-state detector, and multichannel analyzer (MCA) was employed in these preliminary

TABLE 1	RECEIVED	POWER	FOR	SAMPLES	

	Received Power in Milliwatts						
Samp1a	Sample	Location	in Inch	es From	the Tran	smitting	Horn
Туре	0,5	1.0	1,5	2.0	3,0	4.5	5.0
Filter Paper	42	46	49	44	50	45	39
Filter Paper With Oil	39	42	53	56	44	45	44
Filter Paper With Oil and 5 mg of Metal	40	45	50	51	51	43	44
Filter Paper With Oil and 15 mg of Metal	38	62	63	64	71	49	38

evaluations. Small segments cut at random from the filters were flattened and held in the usual diffractometer specimen position. The area covered by the X-ray beam was centered on one fold of the flattened filter. A cylindrical graphite collimator tube directed the beam onto the specimen, and the scattered radiation entered the detector through a 0.2 degree receiving slit.

Since the fiberglass matrix of the filter emits its own characteristic radiation along with that of material embedded on it, it was first analyzed in a Scanning Electron Microscope (SEM) using Energy Dispersive Analysis to determine which elements were present and, in particular, to determine whether or not iron was present on an unused filter. It was found that the filter contained a series of elements but was free of iron. However, iron was detected in the scattered radiation in the diffractometer setup. This iron fluorescence undoubtedly originated on the various slits and holders in the system and can be eliminated by appropriate choice of slit materials and specimen shielding. Since the objective was to determine if small amounts of iron on the filter can be quantitatively measured, in this initial phase no extended effort was made to eliminate the iron radiation background. Rather, increases in iron fluorescence above the background reading were considered to be of primary interest.

Data for the filter specimens described above are presented in Table 2.

The data were analyzed statistically to determine if real differences (95 percent confidence level) existed between the mean values. The "t" test (Student's t) was applied. A "t" value for each set of means to be compared was calculated according to the following equation:

$$t = \sqrt{\frac{(n_1 - 1)v_1 + (n_2 - 1)v_2}{n_1 + n_2 - 2}} \sqrt{\frac{1}{n_1} + \frac{1}{n_2}}$$

t = Student's t

where:

 $\overline{x_1}$ ,  $\overline{x_2}$  = Means to be compared  $v_1$ ,  $v_2$  = Variances of  $\overline{x_1}$  and  $\overline{x_2}$  respectively  $n_1$ ,  $n_2$  = Number of data points for  $\overline{x_1}$  and  $\overline{x_2}$  respectively

For three samples in each mean, a "t" value of 2.78 must be exceeded for the compared mean values to show significant differences at a 95 percent confidence level. The "t" values are shown in Table 3.

As indicated by the "t" values there are significant differences between the iron intensities from the used and unused (or clean) filters.

Filter Condition	Integrated Count Mean <sup>a</sup>	Standard Deviation of Mean	Variance of Mean
Unused	652.3	17.6	310
Used (oil removed)	1190	169.3	28675
Used (with oil and particulates)	1100,6	97.9	9592.3
Used (treated ultrasonically)	998.3	87.5	7662.3
Used <sup>b</sup> (treated ultrasonically)	1097.3	43.2	1862.3

## TABLE 2. X-RAY FLUORESCENCE DATA FOR IRON

a Mean of three values at different locations except as noted.

b Ihree values at same location.

# TABLE 3. "t" VALUES FOR COMPARED MEANS OF IRON FLUORESCENCE

Means Compared	t	Result
Unused Filter - Used Filter (no oil)	5.47	significant difference
Unused Filter - Used Filter (with oil and particulates)	7.80	significant difference
Used Filter (no oil) - Used Filter (with oil and particulates)	0.79	no significant difference
Used Filter (no oil) - Used Filter (treated ultrasonically)	1.74	no significant difference
Used Filter (oil and particulates) - Used Filter (treated ultrasonically)	1.35	no significant difference
Unused Filter - Used Filter (treated ultrasonically)	6,72	significant difference

Also, there is no significant difference in intensity between the used filter from which the oil was removed by direct flushing in the direction of the filtration and the unflushed, used filter. This indicates that reduction in intensity of the iron fluorescence owing to a filtering effect of the oil is not a major factor. The comparison of means of the used, used-flushed, and used-ultrasonically cleaned filters shows no significant difference. This indicates that the ultrasonic cleaning, or treating, of the filter segment unsupported in the ultrasonic bath is not an efficient process. Later experiments using a fully supported 3-micron filter in an ultrasonic bath, while reverse flushing with filtered nitrogen, effectively removed most contaminants.

#### RESULTS OF SCREENING OF TEST METHODS

The X-ray fluorescence method for analysis of particulates appears to offer the best approach for devising an instrument for field use.

The overall makeup of the debris on a filter was determined by analyzing material backflushed off one of the 3-micron filters. The filter was suspended in trichloroethylene and ultrasonically agitated while nitrogen gas was flowed through it in the reverse direction. The filter was rotated to several positions during flushing. The material shaken loose was then filtered onto a Millipore filter and analyzed in the SEM. The composition of several regions of agglomerated particulates was determined by energy dispersive X-ray analysis (EDXA). It is apparent that while there are small amounts of many elements, the primary constituent is iron. Based upon this result, it is evident that a measurement of the iron on a filter would be a most reasonable approach in the development of a prototype, nondestructive technique.

The tests described on used and unused filters indicate that the iron content of the particles on a filter can be analyzed and significant differences in X-ray intensity can be obtained.

While the iron quantity can be obtained, the particle size cannot. However, when the rate of generation of iron increases appreciably, the filter can be rinsed, and the larger particle sizes can be inspected with a standard stereo-microscope or inspection microscope. Since the increased rate of generation of iron containing debris signals the point of initial failure of a bearing or gear, the detection of this point is critical, and filter removal and analysis, once it is detected, is felt to be justified.

The method is relatively easy to use and off-the-shelf hardware can be utilized for a field instrument.

If required later, other, separate analyzers for elements other than iron may be added with no, or minor, alteration of the basic device.

### DESIGN OF PROTOTYPE UNITS

### OPERATING PRINCIPLES

The X-ray fluorescence filter analyzer consists of two units, a lead shielded mechanical unit and an electronic unit. The mechanical unit receives the filter to be tested on a spindle where it is held tightly. The spindle, during operation, rotates and raises and lowers the test filter in a helical fashion. During filter rotation the filter is scanned by X-rays from a radiation source and the characteristic X-rays fluoresced by the iron on the filter are detected by a proportional counter.

The electrical unit consists of the electronic signal processing equipment to perform the actual count of the fluoresced iron radiation detected by the proportional counter. The unit consists of the following items:

- a) A preamplifier to boost the low level signal from the proportional counter
- b) A high voltage power supply for biasing the proportional counter
- c) An amplifier and a single-channel analyzer which screen out all but signals from iron radiation
- d) A digital readout counter to record the iron radiation count
- e) A low voltage power supply for the equipment.

#### SELECTION OF EQUIPMENT

The following items were selected for radiation measurements and radioactive excitation of the iron on the filter:

- a) Radiation source New England Nuclear, cadmium 109 source, aluminum mounted, 50 millicuries.
- b) Proportional counter LND Inc., No. 425 B, quadrilateral, side window, proportional counter.
- c) Preamplifier Ortec Inc. No. 121.
- d) Amplifier and single-channel analyzer Ortec Inc. No. 490 B.
- e) Counter Ortec Inc. No. 775.

f) High voltage bias supply - Ortec Inc. No. 459.

g) Low voltage power supply and rack - Ortec Inc. No. 401 M/402 M.

The mechanical unit was designed and built at The Franklin Institute Research Laboratories.

The overall Filter Analyzer is shown in Figure 2.

The assembly and detail drawings for the mechanical unit are shown in Appendix A.





### TESTS OF PROTOTYPE FILTER ANALYZERS

#### PRELIMINARY TESTS AND ANALYZER MODIFICATIONS

In the preliminary tests, extreme variability in counts on the same test filter was observed. The variability was traced to the following causes:

- 1. Placement of the cadmium-109 radiation source directly above the proportional counter cell in the mechanical unit resulted in some downward scattered radiation reaching the cell.
- In addition, radiation from the source caused fluorescent radiation scatter from the walls and components in the mechanical unit to help flood the counter cell beyond its capacity to count.
- 3. The setting for the window in the single channel analyzer was too wide, allowing relatively intense lead fluorescent radiation to be included in the count.
- 4. The same wide window setting allowed relatively intense silver fluorescent radiation to be included in the count.
- 5. Line voltage variations were thought to cause fluctuations in the bias supply voltage to the proportional counting cell.
- 6. The bias supply voltage was subject to variation because the integral voltmeter was not sufficiently sensitive to allow reproducible bias voltage settings, thus changing the sensitivity of the proportional counter.

In order to correct these conditions, the following actions were taken:

- 1. A lead shield, later replaced by an aluminum shield, was placed between the upper surface of the proportional counter and the radiation source to absorb the downward scattered radiation.
- 2. Reduction in the window width set on the single-channel analyzer partially reduced the effect of both lead and silver fluorescent radiation scatter.

- 3. Interposing four 0.0015-inch-thick nickel discs between the source and the interior of the mechanical unit further reduced the lead and silver interference. The discs were made an integral part of the source holder.
- 4. An external line voltage regulator was used to nullify line voltage fluctuations.
- 5. An external, high voltage, digital voltmeter was used to assist in setting reproducible bias voltages. An integral high voltage, digital voltmeter is highly desirable.

### FILTER LOADING METHOD

Calibration of the Filter Analyzers - The filter analyzers were calibrated by using special filters of identical size to those for aircraft use, but having no metal media support components. These filters were supplied by Aircraft Porous Media, Glen Cove, Long Island, New York and were rated at  $\beta_7 = 100$ . The filters were loaded with powdered iron in increments by sifting the iron uniformly over the surface of the filter. The powdered iron used in the hand loading was in the size range below 44 micrometers. The size range was selected after a preliminary experiment, using cylinders of filter paper of the same height and diameter of the test filters and each hand loaded with one gram of powdered iron, indicated a count difference dependent on the size of the iron. Since, in practice, the filters are protected by a nominal 40 micrometer pre-filter it was decided (after conferring with AVRADCOM, Fort Eustis personnel) that the size range of 44 micrometers should be used for calibration.

<u>Calibration Curves</u> - The calibration curves obtained for the two prototype filter analyzers are shown in Figures 3 through 6. The calibrations are drawn for two filter conditions, with the protective outer paper cylinder and without the protective outer paper cylinder. There are pronounced differences in the curves showing the two test filters with paper covers and without paper covers, but only minor differences when the curves for the two test filters are compared under the same, i.e., cover/no cover, conditions. The calibration curve for Analyzer No. 1 is consistently lower than that for Analyzer No. 2. Minor differences in source intensity, source to filter distance, and proportional detector sensitivity all account for the differences between Analyzers.

Effect of Filter Position - There was some question whether filter position on the internal spindle of the analyzer mechanical unit would have an effect on the count for the same iron loadings. Experiments were conducted in which the filter was scanned, turned 180 degrees and scanned a second time. Three counts were made under each condition. Four such tests were made. The data is summarized in Table 4. Statistical comparisons of the mean values of the counts in each test show



Figure 3. Calibration Curve, Filter No. 1, No Paper 25







Figure 5. Calibration Curve, Filter No. 2, No Paper





no significant differences in the mean count for changed positions. This means that the analyzers scan the entire filter and that no special reference position marking is required on the filter itself.

Test No.	Counts in Filter Pos. 1	Counts in Filter Pos. 2	Mean Count Filter Pos. 1	Mean Count Filter Pos. 2
1	31532 30375 30096	29696 29574 29341	30668	29537
2	33995 34025 34025	33489 33130 33414	34015	33344
3	32354 32811 32313	32313 32220 32269	32493	32267
4	37918 37917 37899	37627 37529 37514	37911	37557

TABLE 4. EFFECT OF FILTER POSITION

Sensitivity of Iron Determination - In the first calibration tests on Filter No. 1 in both analyzers, the first weight increment of iron added to the filter was 0.025 gram. In previous work on a capacitance type iron detector, it was learned that an estimate for debris generation in a UH-1 main transmission is one part per million, or one milligram per liter, per hour of running time. For approximately a 12-liter system, this corresponds to 12 milligrams per hour, and for a sampling period of 20 hours of operation it corresponds to 240 milligrams or 0.240 gram. The 0.250-gram iron addition corresponds, roughly, to the amount of iron which might be expected to be deposited on the filter in 20 hours. Since some of the iron generated might be of larger particle size it was decided to use smaller increments of iron weight in several subsequent additions in order to determine the sensitivity of the method. Table 5 shows the count data for six scans at each of the first five iron additions for Analyzer No. 1, using Filter No. 1 with no protective paper. Table 6 shows the same data for Analyzer No. 2. Significant count differences are obtained with an increment of iron weight of only 0.05 gram of iron.

Weight of Iron in Grams	Raw Counts	Mean Count
0.00	28710 284 28555 283 28503 284	468 361 28506 441
0.25	532535125220751251477510	250 965 51865 936
0.35	63270 618 61956 613 60867 602	367 382 61603 278
0.45	72009 716 71022 699 68998 694	500 924 70505 177
0.50	73361 735 73263 755 75131 735	551 188 74002 517

TABLE 5. ANALYZER NO. 1 SENSITIVITY DATA

TABLE 6. ANALYZER NO. 2 SENSITIVITY DATA

Weight of Iron in Grams	Raw Counts	Mean Count
0.00	314293223105431130815306	208 121 31213 548
0.25	627836266219361061448613	506 063 61902 317
0.35	73766 738 73972 733 72891 724	817 886 73386 883
0.45	83627 831 84212 836 82783 829	22 544 83387 932
0.50	89861 898 89554 887 88706 891	806 711 89290 102

Table 7 indicates, however, that the sensitivity falls off as higher iron loadings are reached. While a difference in mean count of 3497 counts for 0.05 gram of iron is obtained at 0.45 to 0.50 gram of iron, a count difference of 33995 counts is obtained for 1.0 gram of iron at 3.0 to 4.0 grams of iron. At this level a count of 1699 would be expected for a 0.05 gram increment. Since this value is well within the maximum deviation, it would appear that above 3.0 grams the sensitivity to 0.05 gram is highly questionable. Likewise, the sensitivity to 0.1 gram is questionable above the 4.0-gram level, to 0.25 gram above the 8.0-gram level, and to 0.5 gram above the 11.0-gram level. The high counts at the higher iron concentrations may be causing the detector to miss some counts because of "dead" time, the time needed to recharge after making a count. Also the possibility exists that, at higher concentrations, one iron particle may rest on top of another and "shield" it.

Table 7 also indicates the inadvisability of making only one scan on a filter. The final column shows the high and low counts to be expected in view of the maximum count deviation. The maximum deviation is just slightly higher than two standard deviations and so approximately 95 percent of all counts for a given level may be expected to lie in the indicated range. If only one reading is taken, the chance exists that at one level it may be high and at the next level it may be low, thus leading to an error. At least three scans should be made, and the mean (average) value calculated and used in determining the iron level.

Two used filters that had been cycled with contaminated transmission oil were tested in both analyzers. Used Filter No. 3 had been cycled six times with eight gallons of oil, and Used Filter No. 4 had been cycled six times with two gallons of oil. After tests in the analyzers the filters were cut apart, and the medía were ashed and acid extracted. The extract was analyzed for iron content by atomic absorption and colorimetric methods. The results are shown in Table 8.

The actual iron content on the test filters is below the minimum detectable limit on the analyzers (0.05 gram).

Colorimetric methods detected no iron.

While the time on the test oils was sufficiently long that higher concentrations of iron should have been achieved, the multi-pass filter loading technique does not deposit all the contaminants on the filter surface as would normal scavenge pump circulation of the oil. This is largely a result of lower flow velocities in the multi-pass system which allows contaminants to settle in the filter bowl and/or the sump so that the particulates never actually reach and deposit upon the filter surface. This observation is borne out to a degree by data which shows that a filter in normal use retains 93 percent of its original dirt holding capacity after 100 system running hours. With the normal dirt holding capacity of 25 grams, a total of 1.75 grams was deposited, or 0.35 grams of contaminant, on the average, for a sampling period of 20 hours. The average deposition rate is well within the detection capability of the X-ray fluorescence unit.

Weight of I (grams)	ron Mean Count	Mean Count Difference	Max. Deviation (counts)	Mean Count - Max. Dev. Mean Count + Max. Dev.
0.00	28506		204	28302 28710
0.25	51865	23359	1388	50477 43253
0.35	61603	9738	1667	59936 63270
0.45	70505	8902	1507	68998 72012
0.50	74002	3497	1186	72816 75118
0.75	88079	14077	1862	86217 89951
1.00	108686	20607	1793	106893 110479
1.50	134241	25562	2060	123181 136301
2.00	157474	23233	1405	156069 158879
3.00	203424	45950	3049	200375 206473
4.00	237419	33995	3473	233946 240892
5.00	250008	22589	2857	257151 262865
8.00	315563	55555	2965	312598 318528
11.00	348549	32986	4902	343647 353451
15.00	369677	21128	4698	364976 374375

## TABLE 7. DEVIATION OF RESULTS

b

Analyzer No.	Filter No.	Count	Iron Content From Curve	Actual Iron Content
1	3	32812	0.0 grams	Color AA - 0.0044 gms
2	3	36517	0.0 grams	Color AA
1	4	30864	0.0 grams	Color AA - 0.0020 gms
2	4	35746	0.0 grams	Color

## TABLE 8. RESULTS WITH USED FILTERS

#### FILTER ANALYSIS OPERATIONS

### EQUIPMENT PREPARATION

<u>General</u> - The mechanical unit is an aluminum container shielded internally with sheet lead. It contains a radioactive source which is covered by a lead shield when the mechanical unit is open. Since the shield covering the source can be raised by depressing the interlock device above the source, the shielded lid of the mechanical unit should be kept closed at all times except when inserting a filter.

Before the application of 110 volts, 60 Hertz power to the equipment, check to be sure that the POWER switch on the Model 402 M Power Supply and the HIGH VOLTAGE switch on the Model 459 High Voltage Bias Supply are in the OFF position.

Wiring Installation - The following sequence should be followed in attaching the electrical lead wiring to the mechanical and electrical units:

- Connect the Model 121 Preamplifier INPUT jack to the OUPUT jack on the Model 425 B Proportional Counter contained in the mechanical unit. A coaxial cable, 6 inches in length with SHV type connectors, is supplied for the purpose.
- Connect the permanently attached, gray power cable from the Model 121 Preamplifier to the mating, multi-prong connector on the back of the Model 490 B Amplifier and Single Channel Analyzer.
- Connect the BIAS jack on the Model 121 Preamplifier to the 0-5 Kv jack on the back of the Model 459 High Voltage Bias Supply. A coaxial cable, 12 feet in length with SHV type connectors, is supplied for the purpose.
- 4. Connect the OUTPUT jack on the Model 121 Preamplifier to the INPUT jack on the front of the Model 490 B Amplifier and Single Channel Analyzer. A coaxial cable, 12 feet in length with BNC type connectors, is supplied for the purpose.
- 5. Connect the SCA OUTPUT jack on the front of the Model 490 B Amplifier and Single Channel Analyzer to the INPUT jack on the front of the Model 775 Counter. A coaxial cable, 6 inches in length with BNC connectors, is supplied for the purpose.

- 6. Connect the permanently attached gray cable from the electrical unit to the MOTOR CONTROL INPUT jack on the mechanical unit.
- Connect the GATE jack on the Model 775 Counter to the GATE OUTPUT jack on the Motor Control at the left of the electrical unit. A coaxial cable, 12 inches in length with BNC connectors, is supplied for the purpose.

<u>Panel Settings</u> - The following settings should be made on the front panels of the equipment in the electrical unit.

- 1. Turn the POWER switches on the Model 402 M Power Supply and Motor Control to the ON position. Allow approximately two minutes for warm-up.
- Make the following settings on the front panel of the Model 490 B Amplifier and Single Channel Analyzer:
  - a) COARSE GAIN to 4
  - b) DIFF-INT switch to DIFF
  - c) FINE GAIN to 3
  - d) WINDOW control to 2.00 and lock control
  - e) LOWER LEVEL control to 0190 and lock control.
- 3. Set the DISCRIMINATOR control on the Model 775 Counter to 4 and the COUNT-STOP switch to COUNT.
- 4. Set the OUTPUT VOLTAGE control on the Model 459 High Voltage Bias Supply to 1.820 and lock the control.
- 5. Turn the HIGH VOLTAGE switch on the Model 459 High Voltage Bias Supply to the ON position and allow approximately 30 minutes for warm-up.
- 6. Push the RESET button on the Model 775 Counter. The counter should reset to zero and remain there. The indicator light beside the COUNT-STOP switch should not be lighted.

Filter-Scanning Operation - The following sequence of operations should be carried out in determining the quantity of iron on a filter:

- 1. Open the shielded lid on the mechanical unit and slide the filter over the vertical aluminum tube. When the filter is properly seated, the tube will project approximately 0.25 inch above the filter.
- 2. Close and lock the shielded lid of the mechanical unit.

- 3. Press the SCAN button on the Motor Control. Hold the SCAN button in the depressed position until the light beside the STOP-COUNT switch on the Model 775 Counter is illuminated.
- 4. The counter will count until a full filter scan is completed. The light will then go out to indicate completion of the scan.
- 5. Record the count indicated on the counter readout display.
- 6. Remove the filter from the mechanical unit.

Shut Down - When it is desired to shut down the filter analyzer, first turn the HIGH VOLTAGE switch on the Model 459 High Voltage Bias Supply to the OFF position. The POWER switches on the Model 402 M Power Supply and the Motor Control Unit should then be turned to the OFF position.

#### GENERAL INSTRUCTIONS

It is not necessary to perform the wiring installation sequence except when the wiring has been removed for transportation or storage of the filter analyzer. In most instances the wiring will remain in place for continuing use of the filter analyzer. Likewise, an analyzer being used on a frequent basis will require only that the settings of the electrical unit be checked from time to time, except for the HIGH VOLTAGE setting on the Model 459 High Voltage Bias Supply as noted. For a filter analyzer in continuing use during a day, setting the bias voltage at the start of the day is necessary, but the setting can be maintained during the day until it is desired to shut down the equipment.

### CONCLUSIONS

The prototype filter analyzers have demonstrated capability to detect iron on lube oil filters quantitatively. The ability of the analyzers to detect small increases in iron concentration is lessened at higher iron concentrations. The ability to detect increases in iron concentration which might signal impending component failure is sufficient even at the higher levels of iron concentration if a minimum 20-hour sampling period is used. The use of the analyzer requires that at least three scans of each filter be made, and an average value be calculated in order to increase accuracy.

#### RECOMMENDATIONS

The following recommendations are made for further test and improvement of the filter analyzers:

- 1. The two prototype analyzers as tested are not fully automatic with regard to synchronization of starting the scan in the mechanical unit and the count in the electrical unit. This should be done in order to facilitate field use. The operating instructions are drawn to reflect the automatic operation.
- The analyzers currently use a cadmium-109 source with 1.5 years half life. The use of an Americium source having a much longer half life should be considered in order to avoid a shift in calibration because of decreasing source intensity.
- 3. The sensitivity of the current analyzers to small incremental increases in iron concentration decreases at high iron loadings on the filters. Work should be undertaken to improve the high level sensitivity.
- 4. The analyzers should be tested in the field.

























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4       1       1/8 DIA. X. G20 LG.       C25         5       2       3/8 DIA X 3/LCG       NYLON         G       1       1/8 X 1/2 X 2/2       2024 AL         7       1       3/8 X 1/8 X 1/16       2024 AL         8       1       1/32 DIA X 5LG.       PIAMO WIZE         9       2       3       T2UARC E-RING-NO 5155-18       VILE         10       2       4       TRUARC E-RING-NO 5133-12       VILE	3	1		3/16 DIA X GGOLG	CRS
5     2 <sup>3</sup> /8 DIA X 3/10LG     UYLOU       G     1 <sup>1</sup> /8 X 1 <sup>1</sup> /2 X 2 <sup>1</sup> /2     2024 AL       7     1 <sup>3</sup> /8 X 1 <sup>1</sup> /2 X 1 <sup>1</sup> /16     2024 AL       8     1 <sup>1</sup> /32 DIA X 5LG1.     DIAU0       9     2     3     TEUARC E - RINGT- NO 5135-18     DIAU0       0     2     4     TRUARC E-RINGT- NO 5133-12     DIAU0	4	1		1/8 DIA. X. 620 LG.	CRS
G     1     1/0 × 1/2 × 2/2     2024 AL       7     1     3/8 × 1/0 × 1/1 ×     2024 AL       8     1     1/32 DA × 5LG.     PIAMO WIZE       9     2     3     TEUARC E - RINGT- NO 5135-18       10     2     4     TRUARC E - RINGT- NO 5133-12	5	2		3/8 DIA X 3/16 LG	NYLON
7     1     3/8 × 1/8 × 1/1/6     2024 A       8     1     1/32 D/A × 5LG1.     PIANO WIZE       9     2     3     TEUARC E - RINGT- NO 5135-18       10     2     4     TRUARC E-RINGT- NO 5133-12	6	1		1/0 X 11/2 X 21/2	2024 AL
8         1         1/32 D/A X 5LG1.         PIAMO WILE           9         2         3         TEUARC E-RING-NO 5135-18         WILE           10         2         4         TRUARC E-RING-NO 5135-18         WILE	7	1		3/8×11/8×17/16	2024 A
9 2 3 TEVARC E-RING-NO 5133-12	8	1		1/32 D/A X 5LG.	WIRE
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2	1	CONTAINER	C12
2	1	CONTAINER TOP	Ø19
3	1	CONTAINER LID	214
4	11	SOURCE SHUTTER	016
0		SOURCE HOLDER	0.16
4	1	COUNTER SUPPORT	0.7
7	1	SHUTTER DETAILS	018
	1	SUDECE HOLDER MOUNT	0,9
9		DE STA-CO TOCHE CLAMP	20100
10	1	DE TA LO E WASHER	@2712"
1.1	1	DE STA-CO SPONDLE ANSY	5-710-
12	2.	MENASTER CARE DE PULL	
1.5	4	SOUTHEN -77 THREDSTUDS	106.210
4	1	LND, NC. PROP CONTER	420 2
15	11	SPURCE	1
17	1	30 PANEL LOCK	

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