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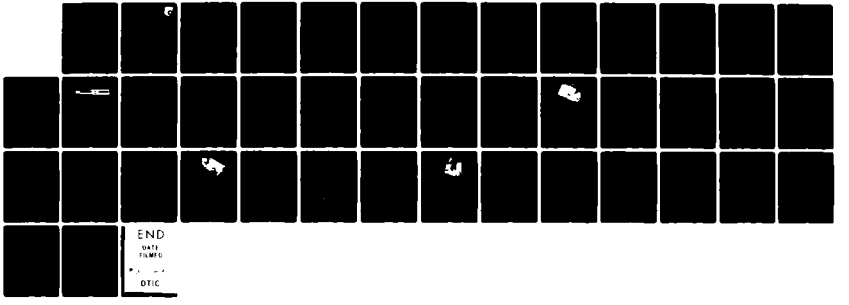

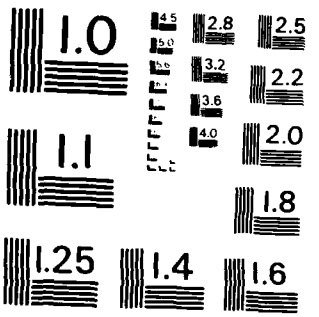

AFWAL-TR-83-2029

X-RAY WEAR METAL MONITOR

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United Technologies Corporation
Pratt and Whitney Aircraft Group
Government Products Division
P. O. Box 2691
West Palm Beach, Florida 33402

MAY 1983

FINAL REPORT FOR PERIOD DECEMBER 1981 - DECEMBER 1982

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This technical report has been reviewed and is approved for publication.

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The objective of this program was a laboratory investigation to study and define the operating parameters and basic equipment requirements of an x-ray fluorescence wear metal monitor system. Specifically, the work has defined the controlling factors of the wear metal detection sensitivity for iron, copper, silver and titanium. The safety and regulatory requirements involved in the use of radioactive materials were also examined.

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The primary findings of the program are:

Using advanced state of the art gas filled proportional counters, the 1-σ wear metal detection sensitivity of ± 1 ppm iron, ± 0.6 ppm copper, 1.8 ppm titanium and ± 3.2 ppm silver was determined under static sampling conditions. Flowing analysis of "used" lubricant demonstrated ± 1 ppm iron and ± 1 ppm copper detection sensitivity.

Laboratory filtered "used" gas turbine lubricant samples analyzed by both flowing loop and individual static sampling showed essentially compatible results.

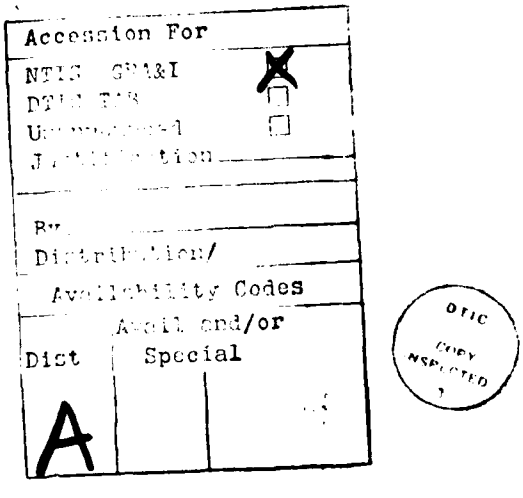
Specially fabricated gas filled gas proportional counter detectors with 13 to 14 percent resolution (FWHM) coupled to low noise (1.3 keV equivalent silicon) hybrid preamplifiers were the controlling instrumentation in achieving the wear metal detection levels.

A commercially designed Curium 244 x-ray excitation source used in the existing wear metal monitor system was ideally suitable for iron and copper detection. Source design modification can be implemented that would reduce the data acquisition time from 1000 sec to about 300 sec.

Titanium and silver measurements were performed using a less than optimal excitation source. Utilization of an annular designed x-ray excitation source is estimated to increase the detector sensitivity to about ± 2 ppm titanium and ± 2 ppm silver in a 300 second data acquisition time.

The specially fabricated hybrid preamplifier was optimized for highest x-ray energy resolution. The temperature stability and maximum operating temperature will be the critical design factor for an in flight wear metal monitor.

TABLE OF CONTENTS



LIST OF ILLUSTRATIONS

LIST OF TABLES

SUMMARY

During this program a laboratory study was conducted to study and define the requirements of an X-ray fluorescent wear metal monitoring system capable of continuously measuring the concentrations of iron, copper, silver, and titanium in the lubricant. The system developed can be made compatible with aircraft propulsion and instrument systems and with flight operations.

The X ray wear metal monitor developed is capable of measuring iron at 1 ppm and copper at 0.6 ppm, or titanium at 1.8 ppm and silver at 3.2 ppm in an engine lubricant. These sensitivites were obtained at a detector operating temperature of 110° F. It was found that temperature control of the preamplifier is critical for satisfactory operation.

The system was tested using lubricants containing known amounts of organometallic standards and by using lubricant samples containing known levels of wear debris as determined by other analysis methods.

During these tests, variations in pressure (2.5 psig to 15.1 psig), and flow (0.4 liters/min to 2.2 liters/min) had no detrimental effect on the results.

The system can be introduced to the Air Force inventory under existing radiological safety regulations and procedures.

SECTION I

INTRODUCTION

Current Air Force operational procedures employ the analysis of engine oil to detect abnormal wear in gas turbine engines. The program employed to conduct this analysis is the Spectrometric Oil Analysis Program (SOAP). This program requires lubricant samples to be periodically taken from the engines and analyzed in a laboratory for various wear metal concentrations. The concentration of the wear metals is usually determined by emission spectrographic analysis. This type of program requires the close and immediate support of an analysis laboratory to produce timely wear metal data.

A continuous in-line oil monitoring system installed on aircraft engines would resolve many of the drawbacks of the current SOAP program. The use of in-line monitoring would eliminate the problem of obtaining SOAP analyses during aircraft deployment. In-line monitoring would also provide continuous "real time" monitoring which would improve the probability of detecting abnormal wear conditions prior to engine failure.

Previous research sponsored by AFWAL/POSL and conducted under Contract F33615-74-C-2024 by Pratt & Whitney Aircraft Corporation demonstrated the capability of X-ray fluorescence to monitor the concentration of iron in lubricating fluids. This capability was shown by rig testing and by monitoring the lubricant in a J57-P-29 engine during test stand operation. Correlation analysis of sensor derived data with atomic absorption analysis data yielded an iron sensor net count sensitivity equivalent to part-per-million iron. Technical Report AFAPL-TR-75-6 dated January 1975 describes this effort (Reference 1).

The objective of this laboratory investigation program was to study and define the operating parameters and basic equipment requirements of an X-ray fluorescence wear metal monitor system. Specifically, the work has defined the controlling factors of the wear metal detection sensitivity for iron, copper, silver and titanium. The safety and regulatory requirements involved in the use of radioactive materials were also examined.

The primary findings of the program are:

- Lowest Level of Wear Metal Analysis
- "Used" Lubricant Analysis Variations
- Monitoring System Controlling Factors.

LOWEST LEVEL OF WEAR METAL ANALYSIS

Using advanced state-of-the-art gas-filled proportional counters, a one-standard deviation wear metal detection sensitivity of 1 ppm iron, 0.6 ppm copper, 1.8 ppm titanium and 3.2 ppm silver was obtained under static sampling conditions. Flowing analysis of "used" lubricant demonstrated 1 ppm iron and 0.6 ppm copper detection sensitivity.

"USED" LUBRICANT ANALYSIS VARIATIONS

Three 2.5 liter "used" gas turbine lubricant samples analyzed by both flowing loop and individual static sampling showed compatible results. There were some differences between the XRF analysis and USAF SOAP measurements.

MONITORING SYSTEM CONTROLLING FACTORS

X-ray Detectors

Specially fabricated gas-filled proportional counter detectors with 13 to 14% resolution (5.9 keV) coupled to low noise (1.3 keV equivalent silicon) hybrid preamplifiers were the critical components in achieving the wear metal detection levels.

X-ray Excitation Sources

Curium-244

A specially designed Curium 244 X-ray excitation source used in the existing wear metal monitor system was ideal for iron and copper detection. Source design modification can be performed that should reduce the data acquisition time from 1000 sec to about 300 sec.

Iron-55 and Americium-241

Titanium and silver measurements were performed using a less than optimal excitation source. Utilization of an annular designed X-ray excitation source is estimated to increase the detector sensitivity to about 2 ppm titanium and 2 ppm silver in a 300 second data acquisition time.

Hybrid Preamplifier

The specially fabricated hybrid preamplifier was optimized for highest X-ray energy resolution. The temperature stability and maximum operating temperature will be the critical design factors for an in-flight wear metal monitor.

Flow Lubricant Sampling Chamber

The basic design of the beryllium window sampling chamber is suitable for consideration in an in-flight system. Size reduction and engine design compatibility will be required.

Signal Processing and Data Reduction

Preliminary discussions with portable X-ray analysis instrumentation vendors indicate that systems currently being designed for portable field applications could be modified for in-flight usage.

SAFETY REQUIREMENTS

Existing U.S. Nuclear Regulatory Commission and U.S. Air Force procedures permit the utilization of radioactive X-ray excitation sources. Approval by the Air Force Radioisotope Committee is required for fleet application. This approval will most likely be contingent on the operational payoff. This payoff must offset the additional procedural, training, and logistic complications resulting from the introduction of a device containing radioactive material into the U.S. Air Force inventory.

SECTION II

WEAR METAL MONITOR SENSOR COMPONENTS

An X-ray fluorescent analysis wear metal monitor having a sensitivity of a part-per-million, and being engine compatible is fundamentally dependent on the following sensor component and elemental analysis characteristics:

- Energy resolution, efficiency and stability of the gas-filled proportional counter detector
- Electronic noise and pulse processing capabilities of the hybrid preamplifier
- Elemental photoelectric cross-section and emission intensity of the radioactive excitation source

The following paragraphs are directed at defining these sensor component characteristics.

WEAR METAL X-RAY DETECTOR

Detector Requirements

The ultimate sensor performance is determined by the X-ray detector performance. The X-ray detector converts the X-ray photons into ion pairs that are collected as a series of short current pulses. The fundamental electrical ion pair conversion, the statistics of ion pair production, the intrinsic electronic noise and a myriad of other competing interactions determine the particular X-ray detector quality. Stability and good resolution in the 3 to 9 keV X-ray energy range wear metal elemental analyses. Energy resolution are required for sufficient to identify the specific X-ray photon emitted by the silver, titanium, iron and copper wear metal debris within the excitation backscattering X-ray background and detector effectiveness (efficiency and size) are the essential requirements for the X-ray detector.

Solid state X-ray detectors such as germanium and silicon require cryogenic cooling. A mercuric iodide detector was considered, but the small size, fabrication uncertainties, and temperature limitations were the reason for not pursuing the higher X-ray resolution capabilities of this type detector. The gas filled proportional counter detector provided internal gas gain, minimal temperature dependency, resistance to vibration, mechanical reliability, and operated successfully during gas turbine testing (Reference 2).

Proportional Counter Theory

In the gas porportional counter, the ionizing X-ray photons photoelectrically interact with the proportional counter gas molecules and result in the production of electrons and positive ions. The electron cloud drifts toward the positive center collecting wire and the positive ions drift toward the outer wall (cathode). A potential difference on the order of 700 volts provides sufficient energy in the volume surrounding the wire to ionize additional gas molecules which in turn produce more electrons which repeat the process. (The gas excitation mechanism and charge collection are complex and not fully understood.) Finally, all electrons resulting from the initial X-ray photon are collected on the central wire. These electron avalanches cause a short current pulse to flow to the preamplifier. The current is proportional to the energy of the incident X-ray photon. This process called "gas multiplication" is the principle on which the proportional counter operates. Signal processing of the voltage pulse enables the X-ray spectrum to be resolved and utilized for analytical purposes.

State-of-the-Art Proportional Counters

Direct contact with the three principal U.S. gas proportional counter vendors indicated that no special R&D effort was being directed at improving X-ray proportional counter performance. Discussions about utilizing other than the standard gas mixture or changing the standard mechanical design suggested a lack of commercial interest in getting involved in a one-of-a-kind effort.

Outokumpu Institute of Physics

A literature search showed that only the Outokumpu Institute of Physics in Finland is active in improving the performance of gas proportional counters. Since 1969, Dr. Heikki Sipila and associates at the Outokumpu Institute of Physics, Helsinki, Finland, have been investigating the technology involved in improving the X-ray detection capabilities of the gas proportional counter (Reference 3 through 12). Outokumpu's interest has been in developing specialized X-ray analysis equipment for internal usage and for ore processing and mineral exploration. The Director of the Institute of Physics, Professor Pekka Rautala has generously loaned four advanced gas proportional counter detectors for use in the wear metal monitor program. Specifications for these detectors are given in Table 1 and are shown in Figure 1. Figure 2, obtained from Reference 11, illustrates the excellent separation of the K-Shell X-ray lines of iron (Kα 6.4, keV Kβ 7.0 keV and copper (Kα 8.0 keV, Kβ 8.9 keV) provided by the 10.7%* resolution neon-argon (0.5%) proportional counter. Reference 12 also states that the intrinsic resolution of the counter approaches 8.5% when the gas gain approaches 10. Unfortunately, at present it is possible to achieve this result only by using a cooled preamplifier.

Details regarding specific Outokumpu gas counter construction features obtained from References 3 through 12 are:

1. Removal of electronegative impurities such as O_2, H_2O, SO_2 and halogens to less than 0.1 part per million is essential.
2. Use of high quality production technology
    a. Braze and metal fabrication techniques that eliminate surface contamination
    b. Ion pumping and heating prior to gas fill
    c. Extreme cleanliness
3. Use of anode wire diameter of 13 μm
4. Use of gas gain in the range of 100
5. Use of a noble gas or mixtures of noble gases
6. Use of gas getters within the counter.

* The 5.9 keV X-ray line emitted by radioactive iron-55 is used as an industry standard for comparison of the energy resolution capabilities of X-ray detectors. Percent resolution is the relationship of the Full Width Half Maximum (FWHM; peak width at half intensity) (energy spread) with the 5.9 keV line intensity. Lower resolution percentage indicates superior X-ray detector performance.

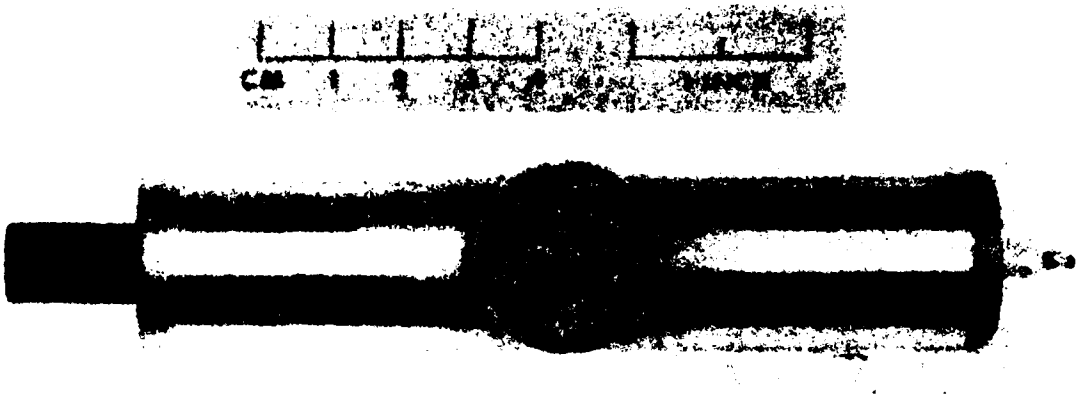
TABLE 1. OUTOKUMPU GAS PROPORTIONAL COUNTER DETECTOR SPECIFICATIONS

Counter Gas	Pressure (Atmosphere)	Percent Resolution Iron-55 OK[1]	Percent Resolution Iron-55 UTRC[2]	Gas Gain (Approximate)	Window Diameter (cm)	Cathode Diameter (cm)	Counter Length (cm)	Weight (g)	Voltage
Argon-Isobutane (2%)	3.7	13.5	14.3	200	1.9	2.5	14.6	116	800
Neon-Argon (0.5%)	7	12	13.0	75	1.9	2.5	14.6	119	620
Neon-Argon (0.5%)	7	11.8	13.1	75	2.2	3.0	14.6	130	700
Argon-Xenon (5%)	2	13	13.7	75	2.9	2.5	14.6	120	720

(1) Outokumpu measurements were performed at 5000 cps — full window illumination using a discrete component preamplifier and a Canberra 2020 amplifier (3 μsec)

(2) United Technologies Research Center measurements performed at 8000 cps — full window illumination using a modified EG&G ORTEC PA242H hybrid preamplifier and a Canberra 2020 amplifier (2 μsec)

(3) All detectors contain a 0.5 mm beryllium window



3.7 ATM Argon-Isobutane

FD 259979

Figure 1. Outokumpu Gas Proportional Detector

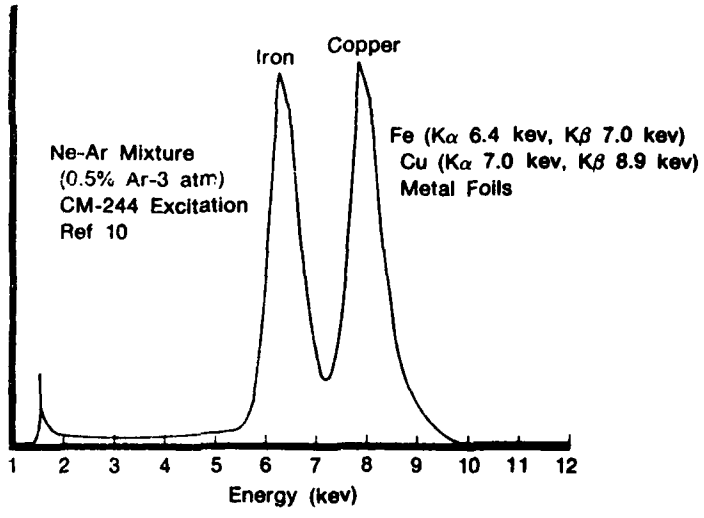


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Figure 2. Institute of Physics X-Ray Spectra from a 10.7% Resolution Proportional Counter

These construction features result in both an increase in the counter lifetime from the nominal 10^{12} counts to an estimated 10^{14} counts or better and a decrease in gas gain shift with changes in count rate.

The use of higher than the standard 1 atmosphere gas filling pressure provides both an increased X-ray detector absorption efficiency and a reduction in the low energy background (wall effect) caused by incomplete charge collection.

Proportional Counter Laboratory Testing

Energy Resolution and Background Reduction

The principal X-ray Kα fluorescent energies of silver, titanium, iron, copper, the Lα energy of silver, and their energy differences are shown below:

	Ag		*Ti*		*Fe*		*Cu*		*Ag*
keV	3.		4.5		6.4		8.1		22.1
ΔkeV		1.5		1.9		1.7		14.1	

The lowest level of wear metal detection is directly related to the intrinsic energy resolving capabilities of the X-ray detector. Sufficient energy resolution is required to permit the specific elemental identification, but the major effect of improved energy resolution for part-per-million analysis is the decrease in the background intensities at the specific elemental X-ray energy. This decrease in background is a square root function with regard to detection sensitivity, i.e., the background must decrease by a factor of four in order to double the detection sensitivity for a particular element.

The measured energy resolution for the four Outokumpu furnished proportional counters, Table I, ranged from 13% to 14.3% for the iron-55 manganese Kα (5.9 keV). The 1% higher resolution obtained by the Institute of Physics, Table I, was obtained with a high quality discrete component preamplifier. The same type Canberra 2020 amplifier was used at both the Institute of Physics and UTRC. Prior P&WA wear metal monitor engine testing (Reference 2), demonstrated the need for a hybridized preamplifier to reduce the acoustic vibration sensitivity; therefore, a hybrid preamplifier was used during the laboratory testing.

To provide a point of reference between the original argon-carbon dioxide (3%) filled gas proportional counter used in the existing wear metal monitor* and a state-of-the-art Outokumpu argon-isobutane (2%) filled counter, the curium-244 lubrication backscattering spectra were compared, Figure 3. The identical LeCroy TRA 510 preamplifier was used for both spectra. The difference between the 22% (iron-55) resolution of the original experimental counter and the 15.5% resolution Outokumpu counter is evident from both the improved definition of the emitted curium X-ray energies and the decrease in the low energy background by the Outokumpu counter.

* Model X-1-51, 10 mil berryllium 1.75 in. diameter side window, 3 in. wide × 1 in. depth × 6 in. length, Ar-CO_2 Ar-CO_2 (3%), 1 atm (1978).

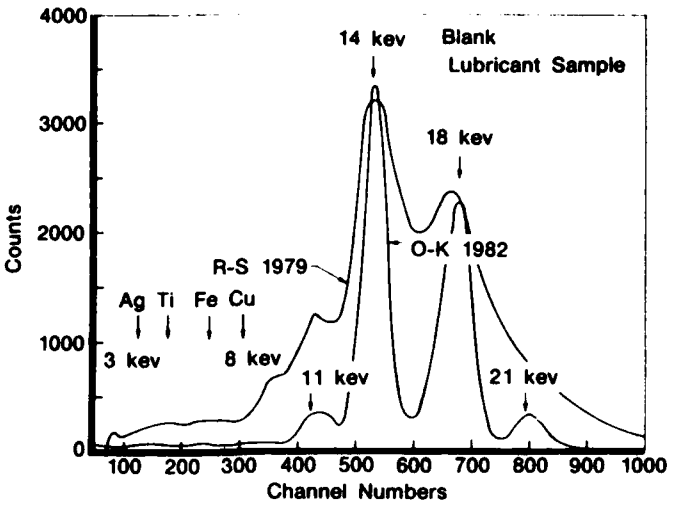

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Figure 3. Improved Curium-244 Backscatter Energy Resolution

Silver, Titanium, Iron and Copper Metal Foil X-Ray Fluorescence

Curium-244 Excitation

The excellent X-ray energy peak resolving capabilities of the 7-atmosphere neon-argon (0.5%) proportional counters for silver, titanium, iron and copper X-rays excited from metal foils by curium-244, Figure 4, is typical for the four Outokumpu proportional counters. The peak resolving capabilities of the original wear metal monitor 1 atmosphere argon-carbon dioxide (3%) proportional counter for the same element exhibits poorer energy resolution, Figure 5.

Americium-241 Excitation

Silver detection is more probable when K-shell electrons are excited by the 59 keV gamma radiation from Americium-241. The approximately 200 times greater lubricant depth range of the 22 keV K-shell silver X-ray vs the 3 keV L-shell X-ray increases the probability for silver X-ray interactions. The energy resolving capabilities for titanium, iron, copper and silver K-shell X-rays by the 3.7 atmosphere argon-isobutane (2%) proportional counter, for metal foils excited by Americium-241 is clearly shown in Figure 6.

Iron-55 Excitation

Titanium detection is more probable when it is excited by the 5.9 keV X-ray from iron-55. As can be noted in the excitation of titanium foil, Figure 7, the iron-55 backscatter is in close proximity to the 4.5 keV titanium X-ray.

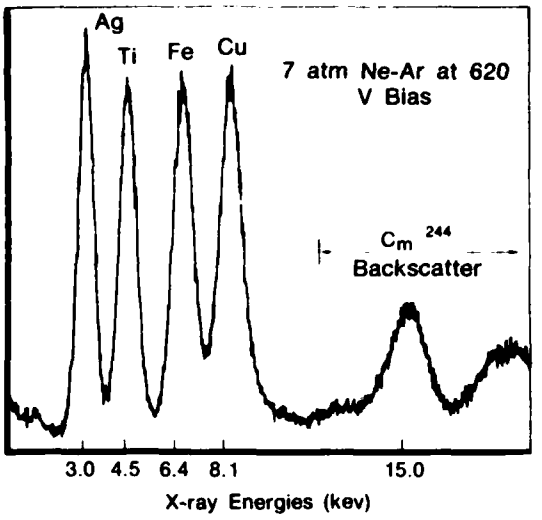

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Figure 4. Metal Foil Curium-244 Excitation, Ortec PA 242H

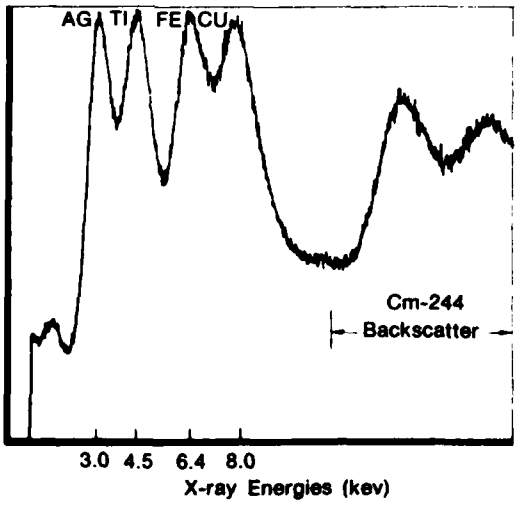

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Figure 5. Metal Foil Curium-244 Excitation, Reuter-Stokes

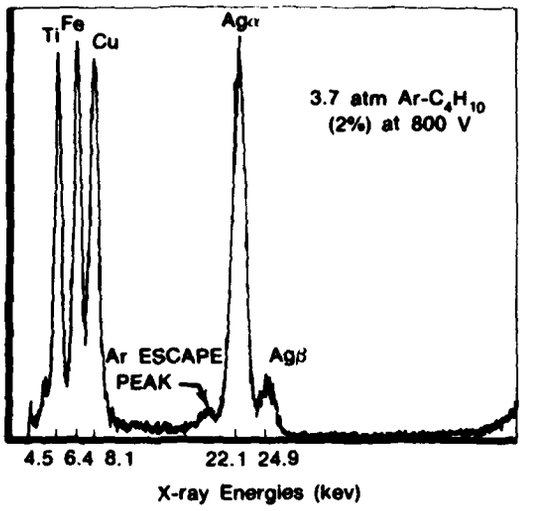

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Figure 6. Metal Foil Americium-241 Excitation

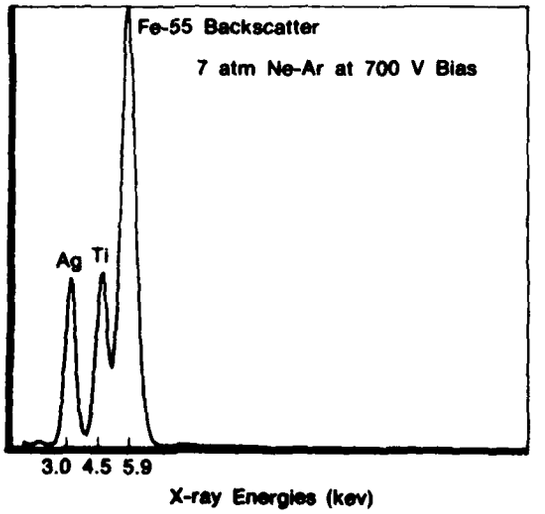

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Figure 7. Metal Foil Iron-55 Excitation

Other Proportional Counter Characteristics

Count Rate vs Gain Shift

A consistent X-ray peak location is required for energy dispersive X-ray analysis. Counting rate dependent gain decrease is an undesirable feature of gas filled proportional counters. For a count rate shift of 15,000 counts per second to 20,000 counts per second the position of the 5.9 keV iron-55 X-ray peak channel shifted 0.5% (1 channel out of 226 channels) using an Outokumpu neon-argon counter, Table 2.

TABLE 2. GAIN SHIFT VS COUNT RATE

Count Rate (cps)	*Peak Channel*	*% Shift*
2297	231	0
4208	230	0.4
9486	229	0.9
12091	227	1.7
15253	226	2.2
18153	226	2.2
21151	225	2.6

7 atmosphere neon-argon proportional counter (3 cm diameter)
Iron-55 5.9 keV X-ray
−700 volts bias
ORTEC PA242H

Gas Gain vs Bias Voltage

Proportional counter gas amplification is a function of the magnitude of potential difference between the anode and cathode. The internal gas gain for the 3.7 atmosphere argon-isobutane counter was measured by Outokumpu, Table 3. The method used is based on current measurements in which the counter is used as an ionization chamber.

TABLE 3. INTERNAL GAS GAIN CHARACTERISTICS OF THE OUTOKUMPU 3.7 ATMOSPHERE ARGON-ISOBUTANE PROPORTIONAL COUNTER

High Voltage	*Gas Gain*
600	25
650	40
700	70
750	120
800	200
850	350
900	600
950	1200
1000	2300

The change in the 5.9 keV X-ray peak location on a multichannel analyzer display as a function of bias voltage, Figure 8, for a neon-argon counter, shows the need for a stable reproducible high voltage supply. A one-volt change in detector bias voltage yields a 3.6% change in X-ray peak location (Figure 8).

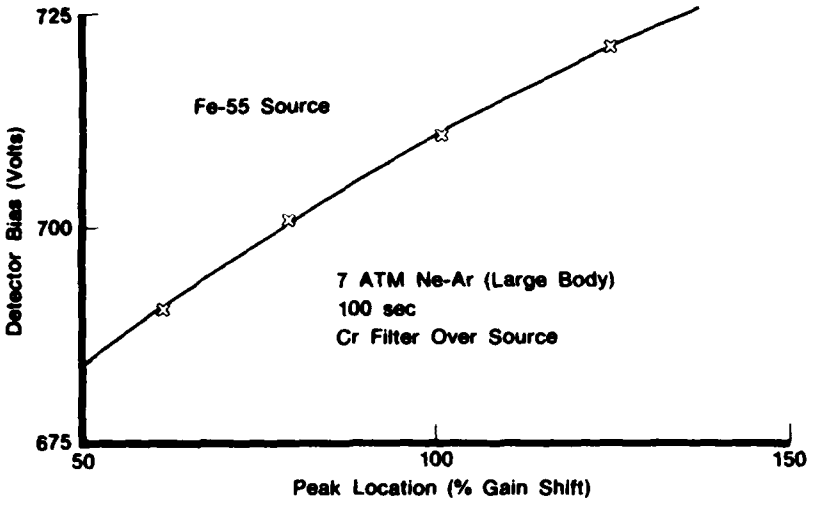

Figure 8. Voltage vs Gain Shift

Preamplifier

The charge sensitive preamplifier is mounted as close as practical to the gas proportional counter detector. This close coupling is required to reduce input capacitance caused by cabling and to decrease microphonic noise, ground loops and radio frequency pick-up, all of which are sources of noise in the charge-sensitive preamplifier.

The short current pulses from the detector are integrated by the charge-sensitive preamplifier to produce a voltage step at its output. The output rise time is equal to the collection time of the detector (approximately 10 nanoseconds), its amplitude is equal to the input charge divided by the capacitance of the feedback capacitor (approximately 1 pf), and its decay time constant is determined by the preamplifier feedback components. The preamplifier output signals are amplified and shaped by the main amplifier.

The electrical and mechanical design of the preamplifier and the physical connection to the detector are critical to the resolution, gain stability, linearity and count rate capability of the X-ray spectrometer. Of particular concern in obtaining the best energy resolution is the fact that electrical noise modulates the statistically varying detector signal causing apparent energy spread. Low noise is also essential because operating the proportional counter under decreased gas gain conditions requires that the noise contribution be at a spectral energy position of about 1 keV.

Prior engine and vibrational testing were performed using a LeCroy TRA-510 hybrid preamplifier (Reference 1). Hybrid preamplifiers provide a smaller package and decreased probability for acoustical noise pick-up during engine operation compared to discrete component preamplifiers. The three hybrid preamplifier vendors were contacted and their preamplifiers were tested using the 3.7 atmosphere argon-isobutane proportional counter. The 5.9 keV X-ray iron-55 peak resolution at 10,000 counts per second was as follows:

Preamplifier	*Percent*
LeCroy TRA-510	15.5
AMPTEC A203	17
ORTEC H242A	14.5
ORTEC H242 Modified	14.3

For count rate shifts of 15,000 counts per second to 20,000 counts per second, the portion of the 5.9 keV X-ray peak channel shifted about 0.8% for the A203, 0.5% for the H242A and 2.4% for the TRA-510.

The split metal case packaging, microdot input connectors and extra features of the standard ORTEC H242A were not suitable for flowing lubrication testing. Modifications were performed by ORTEC. The timing channel was removed. The input field effect transistor was repositioned and the circuit tuned for maximum energy resolution. The schematic diagram of the PA242 (modified), Figure 9, shows that for other than the input field effect transistor, feedback resistor and voltage conditioning (resistors, capacitors, inductors), the preamplifier circuit is contained within the HPA 1006 hybrid can. The round board, overly large resistors in the high voltage filtering and the oversized enclosure box represent a workable breadboard version of the preamplifier, Figure 10. This breadboard was designed for lowest noise. The large filtering resistors were on-hand low noise resistors. The resistor size can be reduced by a factor of about 10. The preamplifier noise was measured at zero capacitance to be 1.3 keV silicon equivalent and the gain is 438 mV/MeV silicon equivalent. The high gain resulted from the use of only stray capacitance (approximately 0.1 picofarad) in the feedback loop. This was done to reduce preamplifier noise to the absolute minimum. This high gain has resulted in sensitivity to temperature changes. Tests show that a 0.06% /°C change results from the high preamplifier gain configuration. Addition of 0.5 picofarad capacitance to the feedback circuit would reduce the temperature coefficient by about a factor of 5.

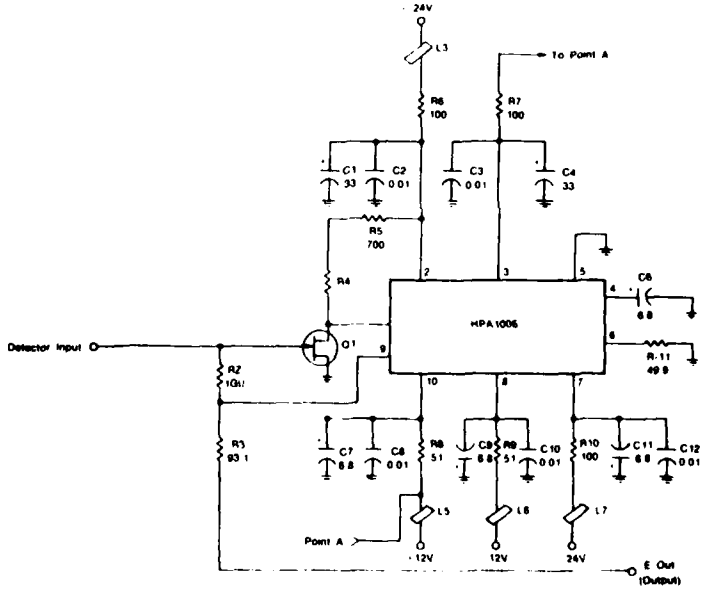

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Figure 9. Ortec Model PA 242 (Modified) Schematic

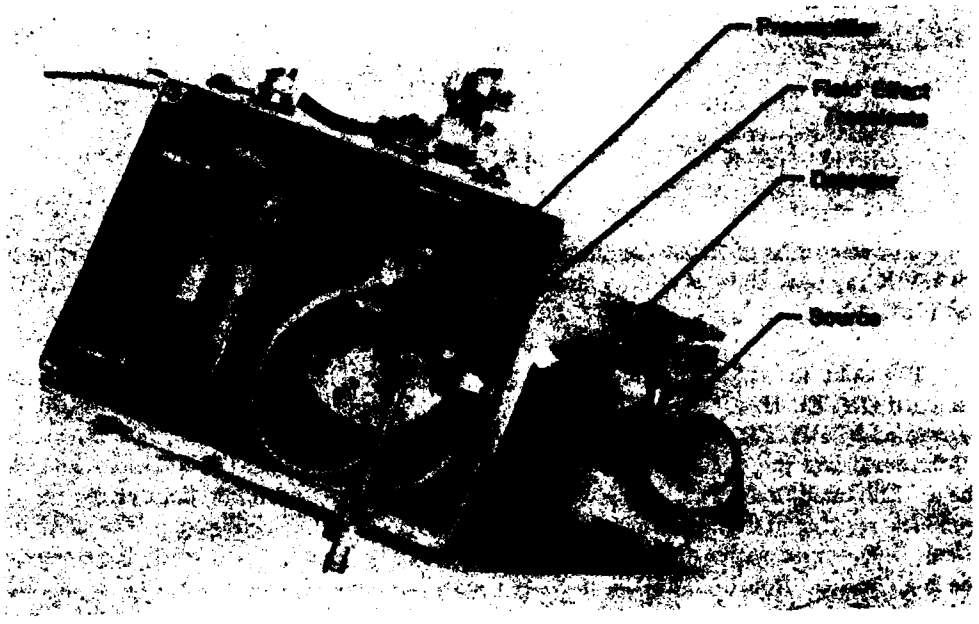

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Figure 10. Source Detector Assembly

RADIOACTIVE X-RAY EXCITATION SOURCES

There are practical limits in the selection of the most applicable radioactive material to provide the optimum X-ray excitation source. The excitation source must emit sufficient X or gamma radiation above the photodetector absorption energies for the elements being analyzed but not emit interfering radiation in the energy range of the element being analyzed. The elemental fluorescent yield, excitation energy path length, and elemental photoelectric cross section also affect the trace analysis detection limits. Other factors such as a sufficiently long half-life to minimize source replacement and Nuclear Regulatory Commission procedures affect excitation source selection. The radioactive material source characteristics and X-ray detection factors are summarized in Tables 4 and 5 for curium-244, iron-55, americium-241 and cadmium-109.

TABLE 4. RADIOACTIVE MATERIAL EXCITATION SOURCE CHARACTERISTICS

	Principal X-ray Emission (keV)	*Principal Emission (keV)*	*Half-life (Year)*	*Specific Activity (millicure/ milligram)*	*Theoretical Yield per Disintegration (%)*
Curium-244	14-21	none	18	81	10[1]
Americium-241	12-22	59.6	433	3.4	36[2]
Iron-55	5.9-6.5		2.7	40	28[3]
Cadmium-109	22.1-24.9	88.7 (4%)	1.24	—	100

1. L X-ray emissions from alpha decay of Cm-244
2. Gamma ray emissions from alpha decay of Am-241
3. Manganese K X-rays from electron capture decay of Fe-55

TABLE 5. X-RAY FLUORESCENCE ANALYSIS FACTORS

	Minimum Excitation Energy (keV)	*Principal X-Ray Emission Energy (keV)*	*Fluorescent Yield*	*Excitation Photoelectric Cross Section*				*Relative[1] Detection Efficiency*		*Oil Depth for 80% Absorption (mm)*
				14 keV (cm^2/g)	*6 keV*	*60 keV*	*22 keV*	*7 atm Neon (%)*	*3.7 atm Argon (%)*	
Iron	7.1	6.4	3.5×10^{-1}	79.7	82.5	0.95	21.6	37	78	2
Copper	8.9	8.0	4.5×10^{-1}	103	114	1.32	28.6	21	74	4
Titanium	4.9	4.5	2.2×10^{-1}	50.3	437	0.55	13.1	46	58	0.6
Silver "L"	3.3	2.9	5.6×10^{-2}	54.4	468	—	13.7	14	13	0.2
Silver "K"	25.5	22.1	8.3×10^{-1}	—	—	5.43	—	2	10	45

1. **Assume 2 cm pathlength and a 0.5 mm beryllium detector window**
2. **Assumed carbon absorption coefficient with a density of 0.987 (g/cm^3)**

Curium-244

The X-ray excitation of iron in lubricant at the ppm level was previously evaluated (Reference 2), using a 60 millicure curium-244 source. Curium is a relatively clean source of L X-rays in the 12 to 22 keV energy range. As can be seen from Tables IV and V, and Figure 4, the emitted X-rays are of sufficient energy to fluoresce copper, iron, titanium and silver X-rays.

The curium-244 source, Figure 11, used in this program was fabricated by Isotope Products Laboratory to UTRC specifications. A solution containing about 800 micrograms of curium-244 was absorbed into two Vycor porous "Thirsty" glass* disks 4 mm diameter by 0.5 mm thick. The glass pores were sealed by firing at 1000°C. The disks are positioned in a gold holder within a welded stainless steel capsule. The stainless steel capsule contains a 0.9 cm diameter beryllium window about 0.5 mm thick. The berrylium window was brazed to the steel capsule using 71.8% gold-28.2% copper braze. An annular configuration is estimated to provide about a three times increase in source activity over the disk source used.

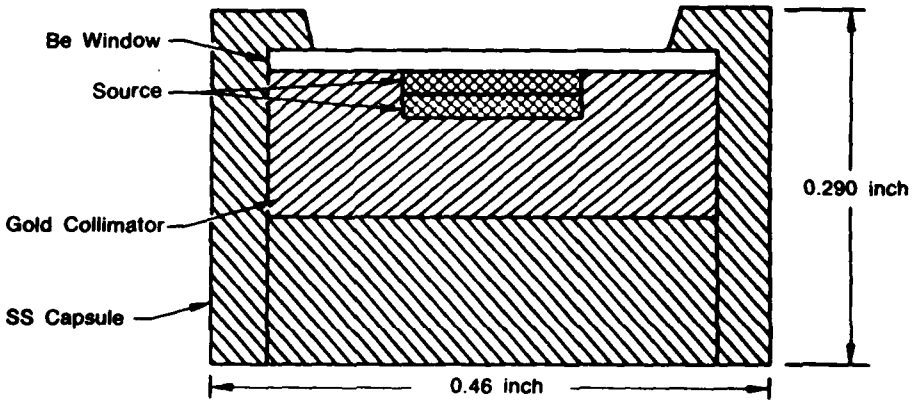

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Figure 11. Curium 244 X-ray Source

Iron-55

The excitation photelectric cross section for titanium and silver is 10 times as great for the 5.9 Kev X-rays from Iron-55 than it is for the 14-22 Kev X-rays from curium-244, see Tables 4 and 5, and Figure 7. An available line configuration source was used to evaluate the applicability of iron-55 excitation.

The 50 millicurie source** was fabricated by electrodeposition of approximately 1.3 milligrams of iron-55 on a nickel substrate in an area 3 mm wide by 25 mm long. The nickel foil was contained in a welded stainless steel capsule with 0.13 mm brazed beryllium window. To limit the X-ray beam to the sample chamber window, a lead collimator was used to permit use of

* 96% silica with the remainder chiefly boric oxide; internal surface area about 250 sq meter/gram.
** Isotope Products Laboratory Model PH55-50.

only 9.5 mm of the source length (approximately 20 millicures). An annular source configuration would be superior and is estimated to be able to provide about six times more source output than the modified line source being used.

Americium-241

The photoelectric cross section times the fluorescence yield is about two times higher for K-shell silver X-ray fluorescence by the 59.6 keV americium-241 gamma than for L-shell silver excitation by curium-244 see Tables 4 and 5, and Figure 6. The longer lubricant pathlength for the 22.1 keV silver K X-ray, Figure 12, increases the probability for silver atom interactions. An available 45 millicurie 3 mm diameter spherical source* was used to evaluate the applicability of americium excitation for silver detectability. The source was encapsulated in a welded stainless steel capsule with a 0.2 mm steel window. To restrict the beam to the sample chamber window, the source capsule was enclosed in a tungsten collimator. An improved annular source configuration is estimated to provide about a threefold increase in source activity over the spherical source used.

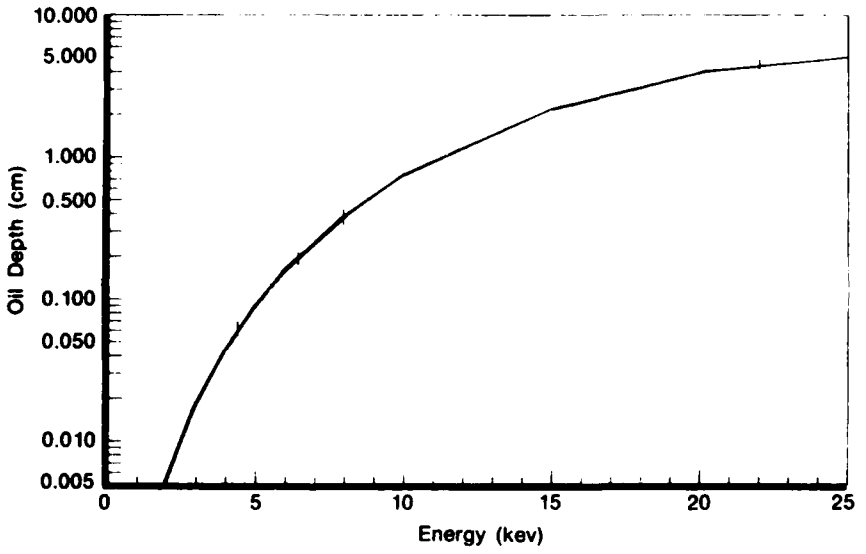

Figure 12. Calculated 80% Absorption in Oil

Cadmium-109

The 22 keV X-ray emitted during the electron capture decay of cadmium-109 can excite copper, iron, titanium and silver X-rays, see Table 4, and Figure 13. The photoelectric cross section is about one third that of curium but cadmium has about 10 times more X-ray output per millicure than curium, Table 5. Cadmium-109 results from cyclotron production and the use is not controlled by the U. S. Nuclear Regulatory Commission. The major disadvantage is the unsuitability of its 1.24 year half-life for practical Air Force field usage.

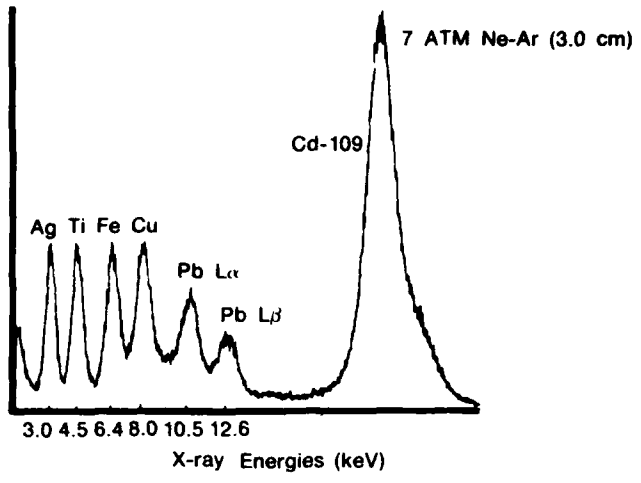

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Figure 13. Cd-109, Pure Metal XRF Superimposed Over Lubricant Backscatter

An available 3 millicure 4 mm diameter disk source* was used. The Cadmium-109 was encapsulated in an ion-exchange resin, sealed in a welded Monel capsule with a brazed 1 mm beryllium window. To restrict the excitation beam to the sample chamber window the source capsule was enclosed in a lead collimator. An annular source configuration is estimated to be able to provide about a 6 times greater source activity than the disk source used.

SECTION III

EXPERIMENTAL MEASUREMENTS

LOWEST LIMITS OF DETECTION

The lowest limit of detection for iron, copper, titanium and silver in MIL-L-7808 lubricant was measured using four different proportional counter detectors and three different X-ray excitation radioactive material sources, see Table 6. The detection levels, counts per ppm, were determined using a series of Conostan organometallic standards in MIL-L-78082 based oil, see Figures 14 through 17. Six milliliter lubricant standard samples (3.4 mm depth) were used for iron, copper and titanium and 25 milliliter lubricant standard samples (15 mm depth) were used for silver measurements. The sampling measurements were performed, see Figure 18, using an aluminum sampling holder containing a 0.13 mm Kapton window.

TABLE 6. ONE-σ FROM BACKGROUND

Gas Proportional Counter	*Iron (ppm)*	*Copper (ppm)*	*Titanium (ppm)*	*Silver (ppm)*	*Count Rate (cps)*
Ne-Ar (0.5%) (3.0 cm diameter)	1.0	0.6	5.0	-	3550
Ne-Ar (0.5%) (2.5 cm diameter)	1.0	0.6	-	-	2000
Ar-Xe (5%)	1.0	0.4	-	-	8150
Ar-Isobutane (2%)	1.3	0.6	-	-	9450
			Iron-55 Excitation		
Ne-Ar (0.5%) (3.0 cm diameter)			1.8		1050
			Americium-241 Excitation		
Ar-Isobutane (2%)	-	-	-	3.2	180

The lowest limit of detection for these measurements was defined as being the (background counts)$^{1/2}$ divided by the counts per ppm. The background counts are the total counts in each spectral region of interest for blank lubricant samples, Figures 19, 20 and 21.

EXCITATION SOURCE AND GAS PROPORTIONAL DETECTOR EVALUATION

Curium-244 Excitation — Iron, Copper and Titanium Measurements

Measurements of the wear metal elements were initially performed using the curium-244 excitation source. The X-ray lubricant backscatter spectrum, as detected by the 7 atmosphere Ne-Ar counter, Figures 19 and 20, show the background contribution from the excitation source, the gold source holder, and the iron contamination in the beryllium detector window. The 18.3 keV and 14.3 principal X-ray excitation energies provide a continuous incomplete energy capture low energy background over the 3 to 8 keV region of interest. The expanded spectrum shows the regions of interest for silver, titanium, iron and copper that are integrated to determine the X-ray fluorescence responses. The silver X-ray background consists of electronic noise and low energy background from the principal curium excitation energies. The copper X-

ray background consists of backscattering from the gold source holder and from the curium backscatter. The iron background consists of both iron contamination in the beryllium detector window and curium low energy backscatter.

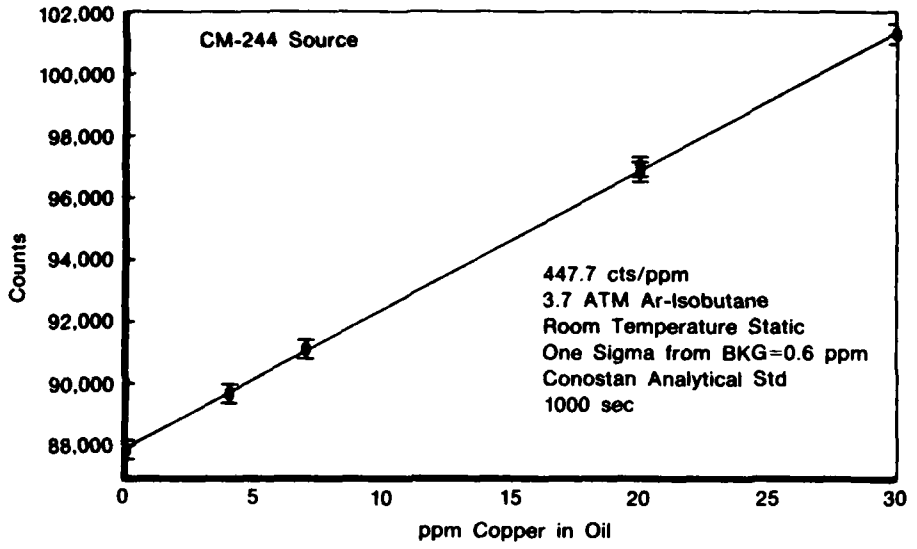

Figure 14. Detection of Copper in Oil

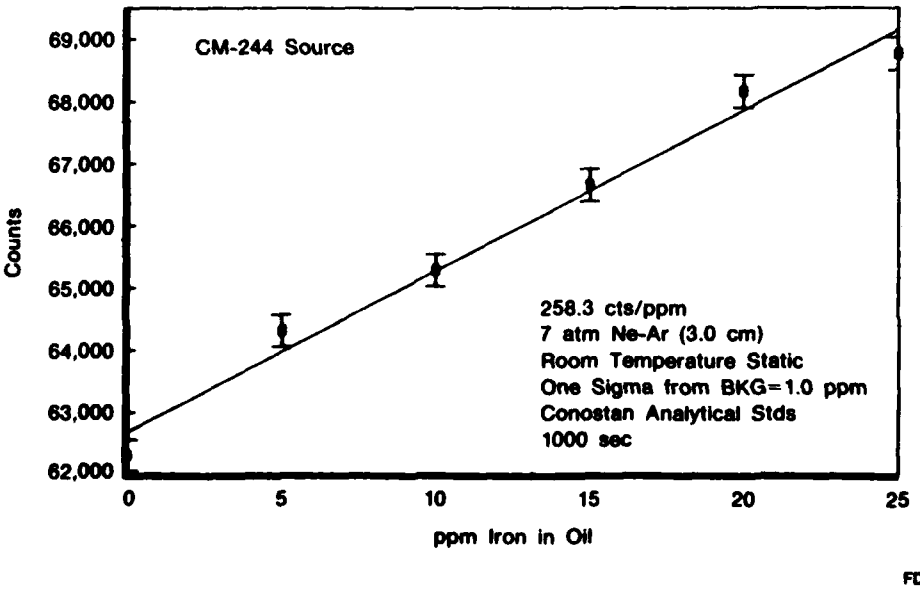

Figure 15. Detection of Iron in Oil

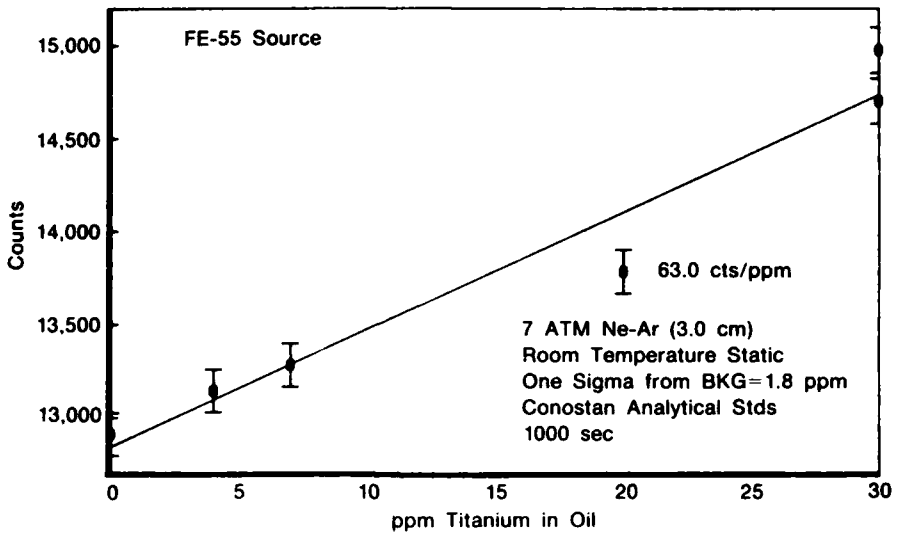

Figure 16. Detection of Titanium in Oil

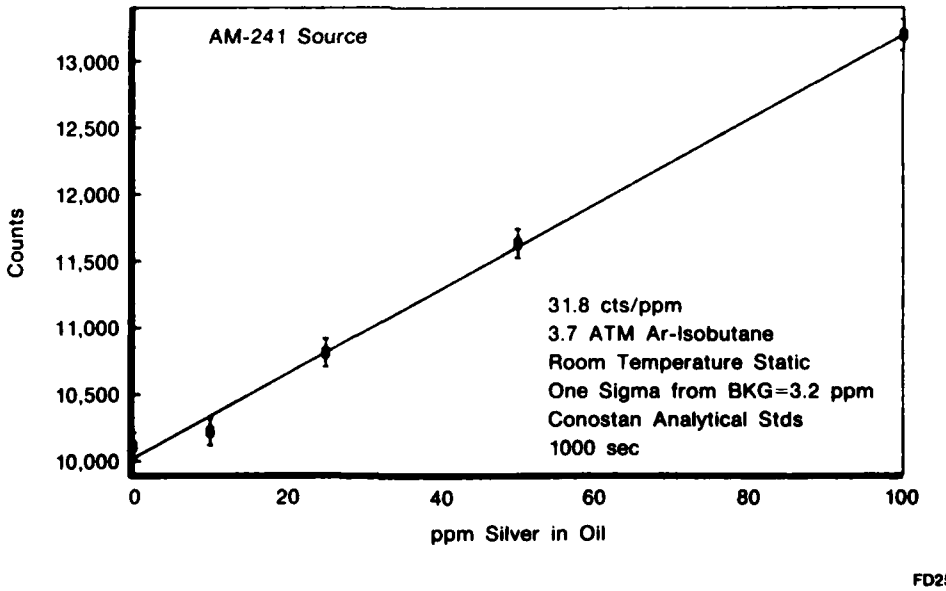

Figure 17. Detection of Silver in Oil

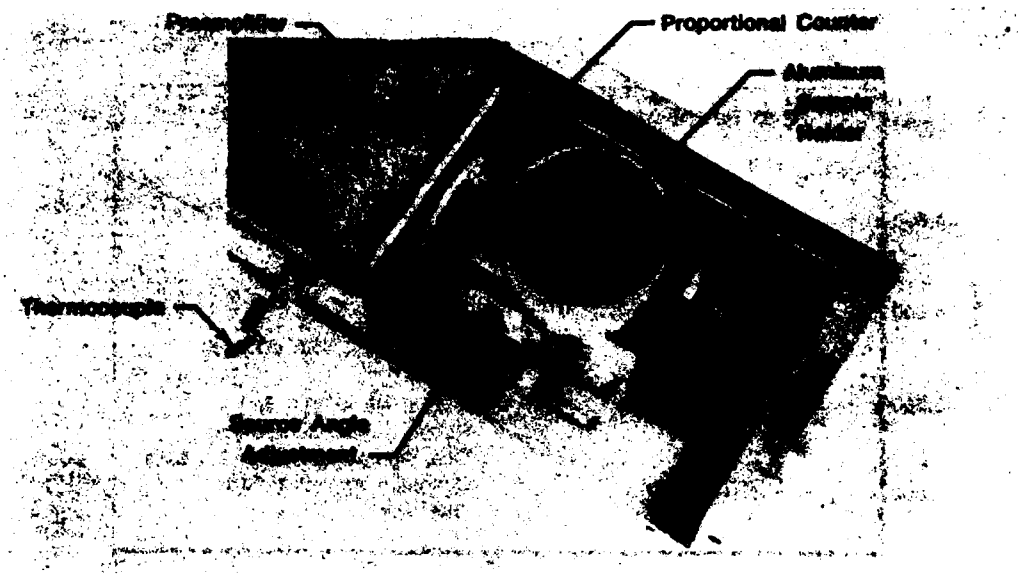

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Figure 18. XRF Lubricant Static Sample Holder

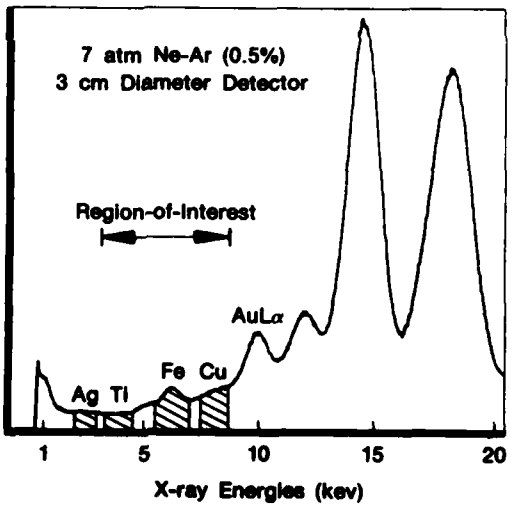

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Figure 19. Curium-244, Lubricant Backscatter Spectrum, Full Scale

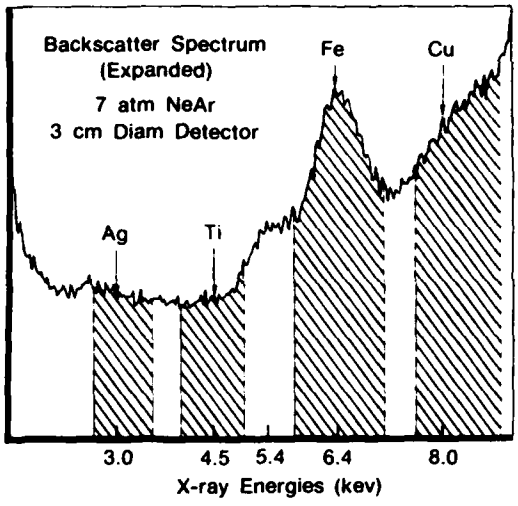

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Figure 20. Curium-244, Lubricant Backscatter Spectrum, Expanded Scale

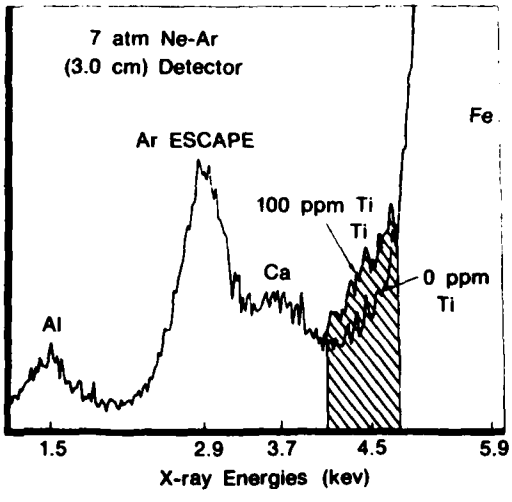

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Figure 21. Titanium Calibration in Oil, Iron-55 Source

For titanium X-rays the curium-244 backscatter is the major background contributor. Use of lower level contaminated beryllium would not significantly increase the iron detection sensitivity in as much as the sensitivity is a function of the square root of background. The complete elimination of iron background contribution from beryllium contamination would increase the iron detection sensitivity by about 18%. The gold X-ray background effect on

copper detectors is compensated in part by the higher copper photoelectric excitation cross section of the gold X-ray energizer.

The copper, iron and titanium 1-sigma detection sensitivities for 1,000 second data acquisition times as determined using the 7 atmosphere neon-argon (3 cm diameter) were 0.5, 1.0 and 5 parts per million. Silver was not detectable using 100 ppm silver concentration. The detection sensitivity for the 7 atmosphere neon-argon (2.5 cm), 3.7 atmosphere Ar-C_4H_{10}, and Ar-Xe proportional counters are given in Table VI.

Iron-55 Excitation — Titanium Measurements

The iron-55 lubricant backscattering spectra, Figure 21, of both zero ppm and 100 ppm titanium illustrate the close proximity of the 4.5 keV titanium X-radiation to the base of the 5.9 keV manganese Kα energy emitted by the iron-55 excitation source. Although the 7 atmosphere neon-argon proportional counter contains only 0.5% argon, a 2.9 keV argon escape peak is positioned directly at the silver Lα 3 keV spectrum position. Use of 100 ppm silver standards indicated no silver sensitivity.

The calcium peak was due to the material used for source positioning. The 1.8 ppm titanium sensitivity is a 2.7 times improvement over the detection levels achieved with the curium-244 source. A properly designed annular iron-55 source should be capable of reducing the detection levels to about 1 ppm. The close proximity of the 5.9 keV backscatter to the titanium peak will require careful attention to gain shift stability.

Americium-241 Excitation-Silver Measurements

The americium-241 lubricant backscatter spectrum, Figure 22, for zero ppm of silver illustrates the low interference in the region of interest for the 22.1 keV silver X-ray energy. The background at the region of interest is essentially incomplete energy capture from the approximately 50 keV inelastic Compton scattering of the 59.6 keV gamma energy.

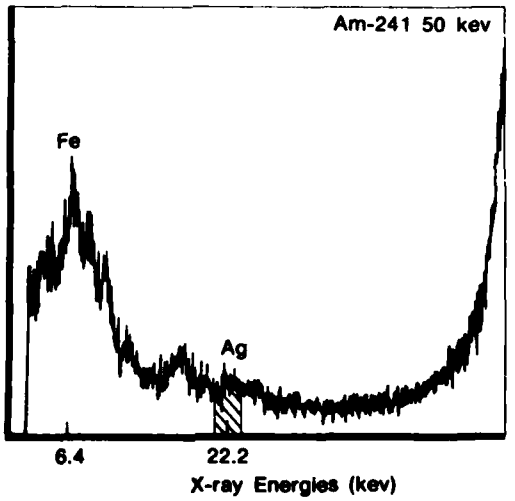

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Figure 22. Americium-241 Lubricant Backscatter Spectrum

The high level of background in the range below 10 keV due to Compton escape prevents the use of americium-241 for low level trace analysis of copper, iron and titanium. The 3.2 ppm lower limit of silver detection could be improved to about 2 ppm with a properly designed annular americium-241 source.

"USED" GAS TURBINE ENGINE LUBRICATION FLOWING MEASUREMENTS FOR IRON AND COPPER

Flowing lubricant sampling measurements were performed using an existing sampling chamber used during early engine testing, Figure 23. The nozzle configuration of the aluminum chamber is designed to disperse the lubricant flow uniformly over the 0.13 mm thick beryllium window. Measurements were performed using the 3 cm diameter 7-atmosphere neon-argon (0.5%) proportional counter and the 60 millicurie curium-244 excitation source. The closed loop sampling system, Figure 24, includes a water bath and circulation pump. A water bath is required to reduce the localized heating effect on the temperature-limited organometallic standards.

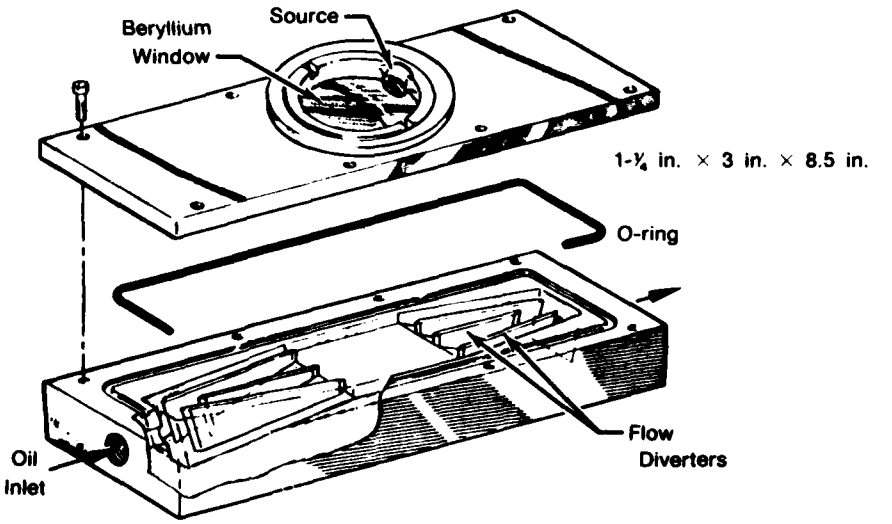

Figure 23. Oil Sampling Chamber

The 4-liter capacity system was calibrated using iron and copper organometallic standards in MIL-L-7808 base oil. Three approximately 2.5 liter "used" gas turbine engine lubricant samples (USAF furnished) were measured for iron and copper concentrations. The engine lubricant samples were heated to 110°F and circulated at a flow rate of 0.5 liter per minute at 5 psig. Pump suction was from the bottom of the beaker and return was above the beaker lubricant level. X-ray analysis data acquisition time was 1000 sec. Because of differences between the flowing measurements and the USAF SOAP data, 6-milliliter samples were obtained immediately after shaking the 1-gallon storage container and after the containers were undisturbed for 24 hours (75°F). Pipetted undisturbed samples were obtained from the bottom, middle and top.

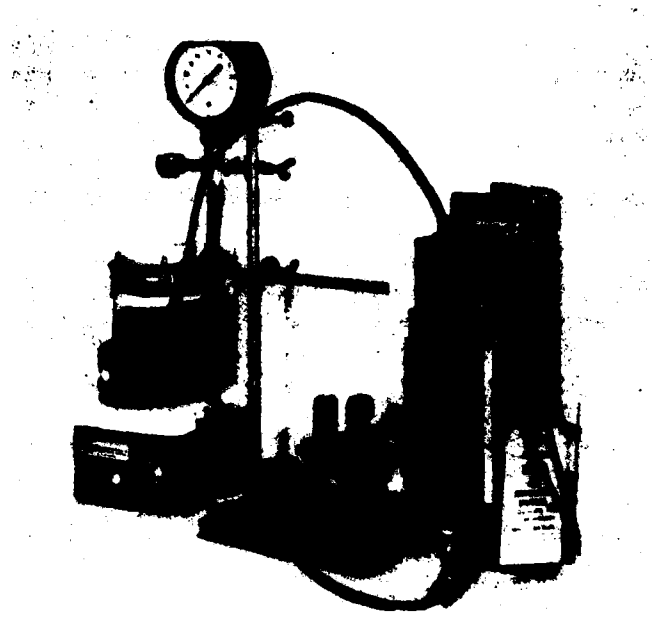

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Figure 24. Flowing Lubricant Sampling Loop

The analytical results are given in Table 7. The XRF metal levels are internally consistent within the 1 ppm iron and 0.6 ppm copper. There are variations between the 15 and 30 hour static sampling values. The several ppm differences would be expected from "used" gas turbine engine lubricant samples due to particle setting, Reference 13. The AF SOAP iron values are about twice the XRF flowing values. For the 0 to 4 ppm copper data the SOAP and XRF flowing data are the same for one sample and differ by 3 ppm on another sample. Considering the fact that two different analytical measurement methods were used and the fact that different amounts of lubricant were sampled by each method, the results are within the range of other acceptable analytical techniques such as emission and atomic absorption spectroscopy, References 14 and 15.

Flow Rate, Pressure and Temperature Testing

Flow Rate and Pressure

The chamber inlet pressure in the closed loop sampling system shown in Figure 24, was varied from 2.5 psig to 15 psig as shown in Table 8 using "used" lubricant sample OP-201-15. Correspondingly, the flow rate ranged from about 0.4 liters per minute to about 2.2 liters per minute. The lubricant temperature was maintained at 110°F. X-ray fluorescence analysis data acquisition time was 1000 seconds. Data was obtained over about a 5-hour period.

The variation analysis showed 1.6 ppm iron and 1.1 ppm copper. These results showed that under these test conditions, variations in pressure and flow rate had no detrimental effect on XRF analysis.

TABLE 7. XRF ANALYSIS OF USED GAS TURBINE ENGINE OIL (ppm)

Sample No.	USAF Value	Flowing	Homogeneous Static	Segregation Tests		
				Top	Middle	Bottom
			Fe*		1000 sec	
OP-201-13	10	4.9	5.6	2.2	2.1	3.2
OP-201-14	14	5.5	4.0	4.5	5.8	4.5
OP-201-15	22	12.4	18.5	19.2	19.1	21.5
			Cu*		1000 sec	
OP-201-13	0	2.4	0	0	1.2	0
OP-201-14	2	1.8	0	0.7	0.6	0
OP-201-15	1	3.7	3.0	2.0	2.5	2.7
			Fe**		500 sec	
OP-201-13	10	—	8.1	3.3	10.6	8.6
OP-201-14	14	—	4.6	4.2	8.3	6.2
OP-201-15	22	—	13.3	10.3	7.8	15.7
			Cu**		500 sec	
OP-201-13	0	—	1.0	2.1	1.6	1.6
OP-201-14	2	—	2.7	2.3	2.8	2.5
OP-201-15	1	—	1.1	2.2	2.2	2.4

*Lubricant standing undisturbed ~ 15 hours
**Lubricant standing undisturbed ~ 30 hours

TABLE 8. FLOW RATE AND PRESSURE TEST DATA

psig	Flow (liters/min.)	Oil Temp (°F)	Pre-amp Temp (°F)	Integrated Counts for 500 sec	
				Fe (223-263)	Cu (280-330)
2.5	0.40	110-111	86-90	32530	32877
2.5	0.39	109-110	88-90	32886	32713
5.0	0.96	109-112	88-91	33005	33122
5.0	0.90	110-112	91	33914	33144
7.5	1.32	110	91	33089	33110
7.5	1.26	110	91	33173	33376
10	1.56	110	91-92	33345	33364
10	1.64	110	92	33384	33188
15	2.08	110-111	92-93	33529	33415
15	2.20	108-111	92-93	32970	33750
15	2.10	109-111	92-93	33536	34452
			Average	33215	33319 Counts
			1 σ	379	467 Counts

• Counts per ppm for 500 sec.
Fe 216
Cu 438
• OP-201-15 flowing results 12.4 ppm Fe 3.7 ppm Cu (Table 7)

Temperature

The temperature in the closed loop system, Figure 24, was ranged from 130°F to 210°F using "used" lubricant sample OP-201-15, see Table 9. The lubricant pressure was maintained at 5 psig. The magnetic coupled gear pump (micropump) used to circulate the lubricant had a maximum operating temperature of 210°F. Air was circulated over the preamplifier housing using a fan. To evaluate preamplifier temperature effects, the preamplifier was not temperature stabilized. The preamplifier temperature ranged from 86°F to 110°F and showed a gain shift of 1.7%.

TABLE 9. TEMPERATURE TEST DATA

Oil Temp. (°F)	*Pre-amp Temp. (°F)*	*Integrated Counts for 500 sec.*		*Difference Between Adjacent Oil Temp Steps (ppm)*		*Iron Peak Shift from 130°F Oil Temp (%)*
		Fe	*Cu*	*Fe*	*Cu*	
130	88	36613	29955			
150	94	37221	30706	2.8	1.7	0.8
170	93-100	37875	31358	3.0	1.5	0.8
190	104	38908	32281	4.8	2.1	1.2
210	109	39984	33360	5.0	2.5	1.2

• Counts per ppm for 500 sec.
Fe 216
Cu 438
• OP-201-15 flowing results 12.4 ppm Fe, 3.7 ppm Cu (table 7).

X-ray fluorescent analysis data acquisition time was 500 seconds and two readings were taken at each of the five 20°F temperature steps. Comparison of the extremes between each pair of the 500-second readings showed about 2 ppm iron and 2 ppm copper for the 130°F, 150°F, 170°F and 210°F steps. The 190°F step showed 5 ppm iron and 2 ppm copper between the extremes of the two readings. Using the XRF data over the temperature steps from 170°F to 210°F would yield a data difference of 14 ppm iron and 8 ppm copper. This unsatisfactory result is attributed to the temperature instability of the preamplifier.

SECTION IV

SAFETY REQUIREMENTS

AIR FORCE POLICY

AF Regulation 161-16, dated 25 April 1978, sets Air Force policy for using radionuclides and specifies procedures for getting licenses and permits to receive, possess, use and transfer radionuclides. It is to be used in conjunction with Title 10, Code of Federal Regulations and Air Force Technical Order 00-110N-3.

Air Force installations under Federal jurisdiction are subject to Nuclear Regulatory Commission authority. Regulations issued and enforced by the NRC are in Title 10 CFR 0-170. Title 10 CFR has licensing criteria and radiation protection standards. Compliance with the provisions of Title 10 CFR is mandatory for the Air Force.

Radioactive materials used Air Force wide are under the administrative control of the Air Force Radioisotope Committee located at AFMSC/SGTZ Brooks AFB, Texas 78235. Radioactive material used only at WPAFB is approved and administered by the Base Radiological Hazard Committee. WPAFB Supplement 1 to AFR 161-16 and WPAFBR 161-1 provide guidelines.

The radioactive materials used in the X-ray wear metal monitor require specific approval under the Base's NRC Byproduct Material License prior to Air Force acquisition. This approval could be provided under existing licensing provisions or may require a license admendment.

The Base Health Physicist is Secretary to the Radiological Hazard Committee and in this capacity is responsible for advising the Base Commander. He has the authority to enforce rules and regulations. This includes general responsibility regarding the use, storage and disposal of all radioactive material. All organizations desiring to utilize radioisotopes must submit a letter to the Secretary, Radiological Hazard Committee detailing the intended use. Written approval or disapproval will be returned to the originating organization following committee appraisal.

RADIOACTIVE XRF EXCITATION SOURCES

The radioactive sources used in this program were obtained from commercial radioisotope vendors. The materials were encapsulated using procedures approved in accordance with NRC authority. The proper handling of the radioactive sources requires an understanding of sections contained in Title 10 CFR Part 20 Standards for protection Against Radiation. Details regarding radiation signs, shipping, storage, handling and disposal are also contained in WPAFBR 161-1 Section A - General Guidance.

The safe usage of XRF radioactive excitation sources is relatively uncomplicated. The dose rate from an assembled X-ray fluorescent wear metal monitor can be reduced by internal localized shielding to meet regulatory requirements. It should be noted that only Amerecium-241 requires any shielding considerations. The normal enclosure for the sampling chamber and detector reduces the contact dose rate to less than 1 milligram per hour for Curium-244, Iron-55, and Cadmium-109. The dose rates for the XRF sources used in this program and the decrease in dose rate as a function of shielding material (Table 10) illustrates the limited shielding required.

TABLE 10. XRF DOSE RATES

Radioisotope	*Unshielded[1] Dose Rate at 15 cm (mr/hr)[2]*	*Dose Rate at 10 cm Above Steel Shielding Material (mr/hr)*		
		0.64 mm	*1.52 mm*	*2.9 mm*
Curium-244 60 mCi	60	0.7	0.35	0.2
Iron-55 20 mCi	110	0.1	—	—
Americium-241 45 mCi	14	14	6.5	2.2
Cadmium-109 3 mCi	21	0.6	0.3	0.2

[1] Unshielded dose rates are from a bare source *not* enclosed in the X-ray fluorescent wear metal monitor.
[2] 0.1 mr/hr is background reading on the Victoreen 440 Ionization Survey Meter.

SECTION V

CONCLUSIONS

This program has demonstrated the following under laboratory conditions.

- One sigma wear metal detection under static sampling conditions of ±1 ppm iron, ±0.6 ppm copper, ±1.8 ppm titanium and ±3.2 ppm silver using the gas-filled proportional counter detector sensor components of an X-ray fluorescent wear metal monitor system.

- Flowing analysis of "used" gas turbine engine lubricant demonstrated ±1 ppm iron and ±0.6 ppm copper sensitivity for the updated X-ray fluorescence wear metal monitor.

- Changes in pressure (2.5 psig to 15 psig) or changes in flow rate (0.4 liters/min to 2.2 liters/min) do not affect the accuracy of the flowing XRF measurements.

- Testing in the lubricant temperature range (130°F to 210°F) with temperature transients of 20°F yielded unsatisfactory iron and copper measurements due to thermal instability in the XRF sensor preamplifier.

- Safety analysis indicates that existing USAF procedures can permit the use of a radioisotope excitation source in an in-flight wear metal monitor. Actual approval will most likely be contingent on the operational payoff of the device.

SECTION VI

RECOMMENDATIONS

The capabilities of the X-ray fluorescence wear metal monitor to measure the concentration of iron and copper debris in flowing oil has been demonstrated under laboratory conditions. Further work is recommended to resolve the following:

- Thermal stabilization of engine mountable electronic package.
- Optimization of silver and titanium analysis X-ray excitation sources.
- Investigate multiple X-ray excitation source configurations.
- Validation of flowing lubrication analysis.

Considerations of these factors are as follows:

Thermal Stabilization of Engine Mounted Electronic Package

Operational conditions predicate a hostile temperature environment for the engine mounted wear metal sensor electronic components. Temperature stabilization is an essential requirement for the front end of the preamplifier.

a. Gain Stability and Maximum Operating Temperature

The gain temperature sensitivity of the hybrid preamplifier must be reduced by a factor of 5 to 10 without producing a significant loss in X-ray energy resolving capabilities. Preamplifier components selected for the highest temperature operation must be assembled and tested to permit long term operation at temperatures of about 100°C.

b. Heating and Cooling System

A combined heating and cooling system capable of stabilizing the engine mounted electronic package within $\pm$ 10°C is required.

Optimization of Silver and Titanium Analysis X-Ray Excitation Sources

The radioactive X-ray excitation sources used to measure silver and titanium lubrication concentration levels were not mechanically compatible with the flowing lubricant sensor assembly. Radioactive sources, designed for the sensor assembly, that can provide maximum X-ray excitation are required to evaluate silver and titanium measurements in flowing lubricant.

Investigate Multiple X-Ray Excitation Source Configurations

In the present approach, three engine-mounted sensors would be necessary to measure iron, copper, silver and titanium. Combining two or three radioactive materials into a single excitation assembly would reduce the number of engine mounted sensors. A segmented source holder incorporating a rotatable shutter assembly could permit individual radioactive material X-ray excitation. The technical feasibility and laboratory demonstration of this concept is required.

Validation of Flowing Lubrication Oil Analysis

The validity of flowing lubrication oil analysis to characterize the wear metal debris concentration levels of actual engine oil samples must be demonstrated on a larger sampling

basis than the three 2.5 liter engine oil samples used in this program. At least 25 engine oil samples that have prior engine SOAP long term histories are required. Wear metal analytical differences between SOAP and X-ray fluorescence flowing oil monitoring systems must be investigated to determine the character of the wear metal debris causing significant differences.

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