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# 411400

# U. S. ARMY

TRANSPORTATION RESEARCH COMMAND FORT EUSTIS, VIRGINIA



TCREC TECHNICAL REPORT 62-91

INVESTIGATION OF THE FEASIBILITY
OF AN AIRCRAFT-OIL ANALYSIS SYSTEM
FOR OPERATIONAL USE IN THE FIELD

PHASE I

Task 1D141812D18423 (Formerly Task 9R89-02-015-23)

Contract DA 44-177-TC-789

May 1963

#### prepared by:

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#### **HEADQUARTERS** U S ARMY TRANSPORTATION RESEARCH COMMAND Fort Eustis, Virginia

This report presents the results of a feasibility study of an aircraftoil analysis system for use at the first and second echelons of maintenance.

This Command concurs with the contractor's conclusions and recommendations, and the continuation of the program will be scheduled on this basis.

FOR THE COMMANDER:

Captain

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Ass't Project Engineer

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# INVESTIGATION OF THE FEASIBILITY OF AN AIRCRAFT-OIL ANALYSIS SYSTEM FOR OPERATIONAL USE IN THE FIELD

PHASE I

Report Number KN-676-1(PR)

Prepared by

Kaman Nuclear Garden of the Gods Road Colorado Springs, Colorado

for
U. S. ARMY TRANSPORTATION RESEARCH COMMAND
Fort Eustis, Virginia

#### FOREWORD

This report was written by Dr. Charles W. Whittle and Phillip L. Jessen of Kaman Nuclear. The work was performed during the period starting January 26, 1962, and ending May 30, 1962. This work could not have been performed without the help and cooperation of the Overhaul and Repair Base, U. S. Navy, at Pensacola, Florida, and Trans-Canada Airlines at Montreal, Quebec, Canada. The help of Mr. Leonard Bartone of USATRECOM was invaluable in gaining access to the U. S. Government data.

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#### SUMMARY

Data were gathered from those organizations on the North American Continent which have performed sufficient oil analyses to provide statistically significant information.

Metal content of the lubricating oil for Curtiss-Wright Type R-1820-86 piston engines is compared to engine failures. The purpose of the data analysis is to determine whether or not a correlation exists between various metal contents in lubricating oil and failure of the engine. It was found that correlation exists.

A survey of methods for the determination of metal content is given. Those methods that appear practical for field use are emphasized. It is concluded that good probability of failure prediction is obtainable if the amount of particular metals is measured for any given engine type.

Several potentially practical methods for field measurement appear to exist. The principal problem appears to be that of sample preparation. Vapor phase chromatography of metal chelate groups and mass spectrometry appear to be promising.

An "in-flight" measurement of metal content to provide a warning of imminent failure would be very useful. The feasibility of a device in which the oil is passed through an inductor is investigated. Metal in the oil will cause the losses as measured at the inductor terminals to increase due to the eddy current effect.

An approximate analysis of the effect of small metal particles in the magnetic field of an inductor indicates that metal concentrations of the order of 100 parts per million will be detectable. Results of the data analysis make it apparent that this metal content may be useful in the prediction of imminent failure; the device would not be sufficiently sensitive for use as maintenance test equipment.

#### CONCLUSIONS

Insufficient data exist for most military mechanisms except for piston engines. More data concerning transmissions, gear reducers and turbine engines will need to be gathered before it will be possible to perform rigorous mathematical analyses of the relationship between failure and metal content.

The rather plentiful data for piston engines would indicate that a good probability of predicting failure exists if suitable threshold levels for particular elements are established for each engine type. This would, in turn, mean that any analysis device to be used for this purpose would need to be capable of selectively measuring quantities of different metals.

The relatively low sensitivity indicated in the analysis of the in-flight warning device would mean that such a device would certainly not be capable of providing a warning of failure nearly as far in advance as a ground maintenance analyzer. (This statement is based on the assumption that the ground analyzer has essentially the same sensitivity as a mass spectrograph.) An in-flight device of this type may be quite useful even with its low sensitivity if further experimentation should disclose that the metal content of the lubricating oil rises to quite high values a few minutes before actual loss of function occurs.

The rather qualitative data for the Rolls Royce Conway turbine engine indicate that while lower levels of metal content will need to be detected, the overall detection problem may be simpler because the principal metal that would likely be present in the oil-wetted system would be iron from the antifriction bearings.

#### RECOMMENDATIONS

The benefits to be gained from the successful prosecution of this program are great, and do not require recital here. Since the data for piston engines look quite promising, and the data for turbine engines at least do not look discouraging, it is believed that the development of a prototype of a feasible device should be instituted as a necessary step toward an intermediate goal. This goal is to secure a sufficient amount of data on turbine engine powered aircraft together with additional data on piston powered aircraft of interest. These data can then, in turn, be used to determine just how universally oil analysis may sensibly be applied in the maintenance of aircraft. As experience with the technique is gained, it may very well be both feasible and economical to apply it to all of the various machinery used by the armed services including land and sea-going vehicles. This prototype development should be carried forward to provide a definitive answer to a very important question. Namely, is oil analysis at first echelon maintenance levels a truly practical and feasible process? It is believed that a competently designed analysis system will show that the process is indeed feasible. If this is the case, it will then be possible to construct a number of pieces of "fieldable" analysis equipment which will facilitate the gathering of the additional data that are needed to prove conclusively the feasibility of the technique.

It is recommended that laboratory experiments be conducted to establish the practical sensitivity limits of the in-flight monitoring system. The sensitivity analysis made in this report is approximate and should be verified by experiment. Prototypes of the in-flight device should then be constructed. These prototypes should then be tested by installing them on test-stand engines which are caused to fail in known ways. This will provide a proof test of the in-flight device and establish confidence in its practicality.

Additional analysis of the data is needed to determine correlations of failure as functions of different thresholds of individual metals and various thresholds for several combinations of metals. The failures of oil-wetted parts should be further categorized into two groups, e.g., those failures that would be expected to increase metal content and those which would not be expected to raise metal content. Correlations should then be calculated for each of these categories for each metal and combinations of metals.

#### INTRODUCTION TO THE PROBLEMS

The primary objective of the program is an investigation of the feasibility of designing an operational oil-analysis system capable of operating under field conditions with units at the aviation maintenance company, or lower, level. tron activation analysis, R F spectrometry, ion time-of-flight spectrometry and other analytical techniques will be theoretically and experimentally investigated, utilizing laboratory devices presently in existence at Kaman Nuclear. The feasibility of designing an in-flight warning device, operating on the oil-analysis principle, will be experimentally deter-The program will provide technical information conmined. cerning the feasibility of successfully developing a field oil analysis system, including the in-flight warning system; preliminary design information concerning the optimum field oil-analysis system; and cost information estimating the funds required to construct a prototype field oil-analysis system, including in-flight warning system, for subsequent delivery for evaluation by the United States Army.

Four different problems, all of which are related to the evaluation of failure prediction through oil analysis, have been analyzed. First, it was necessary to determine whether or not sufficient data exist to support a meaningful analysis. A survey of the known workers on the North American Continent was made. 1, 3, 8 (See section entitled "Government and Industrial Organizations Engaged in Metals-in-Oil Analysis") It was found that only a few mechanisms have been analyzed in enough detail to provide a high degree of confidence in correlations. Second, it was necessary to determine from the data whether or not a correlation does, in fact, exist between the amount of various metals in the oil and failure of the device. A considerable amount of data was found to exist for the Curtiss-Wright R-1820-86 engine. It was, therefore, decided to confine rigorous statistical analysis to this engine type. The following analyses were The oil analyses were sorted into a group for performed: which the total metal content was greater than 50 parts per million and a group having total metal content less than 50 parts per million. These two groups were then divided into two subgroups, one in which failure of an oil-wetted part occurred and one in which no failure of oil-wetted parts occurred. This process was also repeated for a total metal content of greater than 100 parts per million. Standard Chisquared\* correlations were then calculated to determine whether or not there was a good probability of predicting engine failure on the basis of total metal content.

<sup>\*</sup> See Appendix

The individual oil analyses were then made into lists on the basis of particular metals exceeding threshold values. These threshold values were, for most of the metals, established by the U. S. Navy Overhaul and Repair Base. In those cases for which threshold values have not been assigned, values for similar engines were used. Correlation tables were then prepared showing the number of instances where metal content was high and failure occurred, where metal content was high and failure did not occur, where metal content was low and failure occurred, and where metal content was low and failure did not occur. Chi-squared probabilities were then calculated to determine whether or not any particular metal or combination of metals was a good indicator of engine failure.

The average amount of metal for all engines having less than 250 hours running time since overhaul was calculated. The same sort of average was calculated for all engines having greater than 750 hours since overhaul. A comparison of these averages showed whether or not there was any significant difference in average metal content as a function of running time since overhaul for normal engines. The reasoning behind this comparison is as follows: It would be expected that follows would become more numerous as the hours since overhaul increased. If the average metal content always increases as the number of hours since overhaul increases, one would expect to find a correlation between metal content and failure even if metal content was not a particularly good indicator of which particular engine was approaching failure.

Graphs showing metal content versus running time since overhaul were made. These provide a qualitative indication of the change in metal content for normal and for failing engines. Although insufficient data exist for a meaningful analysis of the Rolls-Royce Conway engine, it is included here. Turbine engines will undoubtedly show different metal contamination characteristics than piston engines. Turbine engines will be most important in the future as piston engine use diminishes.

The third major phase of the investigation is a study of those methods which would most likely lend themselves to field application of oil analysis. The desirable attributes of a field system would naturally be the following: reasonable size, a high degree of ruggedness, simplicity of operation, simplicity of sample preparation, rapid data read-out, and permanent data recording. The logistics associated with the system should also be simple. That is, the system should not consume any material that is not already readily available at the operation site. This study has consisted largely of

a review of both new and old techniques that are presently within the art. It did not appear desirable to consider those methods which would obviously require an advance in the state of the art to make their use feasible under any conditions.

The fourth major phase is concerned with the development of an in-flight monitor of metal content in the lubricant. One of the more promising methods of performing this measurement involves simply flowing the lubricant through an inductor whose loss is being continuously measured by electronic circuitry. This provides for very simple sample preparation. That is, essentially none is needed. The analysis defines the interaction between metal particles in the oil and the magnetic field in the inductor. This interaction is ultimately defined as a fractional change in the voltage applied across the inductor under certain circuit conditions.

Field application of an oil analysis system would depend to a great extent on a regular formalized method of obtaining samples from operational aircraft. It is assumed that oil samples would be removed from the aircraft at a convenient time, e.g., at the time of an oil change for those engines which require it and at oil replenishment periods for those aircraft which do not require oil changes. It must be pointed out, however, that sampling the oil at shorter intervals than an oil change period may very well be desired. Further work in incipient failure detection is needed to establish proper oil sampling intervals.

#### RESULTS OF COMPUTER DATA REDUCTION

The computer program for testing correlation of failure\* with individual metal resulted in data given in Table 1.

TABLE 1
STATISTICAL DATA USED IN COMPUTER PROGRAM

Engine con- dition at overhaul		inte was	er c erval abov list	intervæls for which no metal content exceed-							
	Cu	Ag	Ni	Fe	Cr	Si	Sn	Al	ed threshold value		
No Discrepancy	15	2	144	59	4	0	8	39	152		
Oil-wetted Part Failure	28	17	124	62	14	0	11	48	121		

Tables for a Chi-squared ( $\chi^2$ ) test \*\*\* is shown in the following diagram. The diagram is also a key for Table 2.

		Number of engine over- haul intervals for which this metal exceeded threshold value (H)	Number of engine overhaul intervals for which this metal did not exceed threshold value (L)
Oil-wetted part failure	(F)		α =
No Discrepancy	<b>(</b> G)		7/

<sup>\*</sup> In order to clarify word usage, the following definitions are given: Discrepancy: A condition of a part which is variant with the normal operation state of that part. Generally in this report, a discrepant part is one which will eventually be harmful to the system. Failure: The end result of a discrepancy. A failure is a complete breakdown of a discrepant part which in turn may result in a shutdown or catastrophic non-operation of an engine.

\*\* Values set by U. S. Navy Overhaul and Repair Base

\*\*\* See Appendix

The fractional probability that engine failure is not dependent on the metal content is  $\alpha$ . If  $\alpha$  is small, high metal content is indicative of abnormal wear in the engine. If  $\alpha$  is large, high metal content is not indicative of engine wear being high or low.

The results are given in Table 2.

	7	TABLE 2	
RESULTS	OF	COMPUTER	PROGRAM

RESULTS OF COMPUTER PROGRAM	
Copper (Cu)  H   L  F   28   267 α = 0.015  G   15   326	
Silver (Ag)  H L  F 17 278 α = <0.001  G 2 339	
Nickel (Ni)  H L  F 124 171 $\alpha = .975$ G 144 197	
<u>Iron (Fe)</u>   H   L   F   62   233   α = 0.28   G   59   282	
Chromium (Cr)	
$ \begin{array}{c ccccccccccccccccccccccccccccccccccc$	
<u>G   4   337                              </u>	
Silicon (Si) No test because no high silicon levels occurred.	
Aluminum (Al)	
HL	
F   48   247	
G 39 402	

#### TABLE 2 (Cont'd)

#### RESULTS OF COMPUTER PROGRAM

Any metal above threshold:

	H	L		
F	174	121	α =	0.41
G	189	152		

Sum of metals for threshold of 50 parts per million

	H	<u> </u>	
F	122	71	$\alpha = 0.40$
G	89	64	

Sum of metals for threshold of 100 parts per million

	H	L				
F	11	71	C	χ	=	0.50
G	6	64	.,*			

The relatively low values for silver, chromium, aluminum, and copper are indicative that high contents of these metals will reliably show abnormal engine wear. Other metals and combinations of metals do not appear to be reliable indicators.

It should be noted that while high values of these three metals are good indicators that abnormal wear is present, low values of these metals are not good indicators that the engine is in good condition. This statement is supported by the fact that, in every metal table, a large number of failures occurred for which no high metal content was observed. The existence of this phenomena does not necessarily infer that the technique is ineffective as an incipient wear detection method. It may, in fact, infer little or nothing relative to the effectiveness of the method. There are many kinds of failures of oil-wetted parts which would not necessarily increase the metal content of the oil. Examples are:

- (a) Connecting rod fracture. (The engine operation would cease before an oil analysis would be made.)
- (b) Valve sticking.

(c) Fracture of bearing races, crankshafts, crankshaft assembly bolts, push rods, etc.

Discrepancies may be noted which are indicative of abnormal wear in the past but which do not necessarily indicate that abnormal wear is presently occurring. A narrow deep groove, or "score", can occur in a sleeve bearing as a result of ingestion of large particles via the lubricating oil. If this groove does not interfere with the major portion of the bearing surface, no abnormal wear would occur but the "scoring" would be noted as a discrepancy at overhaul.

If a gear tooth is partially lost by fracturing and if that loose portion lodges in a "safe" place, a discrepancy would be noted at overhaul but no increase in metal content would be expected.

In the data accumulated on analyses of oil used in the R1820 engine were a number of cases wherein a non-oil-wetted part failed or was discrepant. The incipient failures of these cases could not be expected to be detected by particulate metal count. The number of the non-oil-wetted discrepancies was 76 out of a total number of over 600 overhaul periods investigated.

The number of overhaul periods for which there were discrepancies with below-threshold contamination counts and in which the discrepancies were of the type which would not increase the oil count was approximately 50. The total number of overhaul periods with discrepancies but with no high contamination was 121. There are fairly high uncertainties to be considered before one can draw conclusions from these numbers. Isolating failures or discrepancies into categories which are distinctly separate with respect to expected contamination characteristics is made difficult because of the poorly detailed data on record cards. Without the benefit of data from a controlled experiment, conclusions are likely to be erroneous.

It was necessary to determine whether or not an increase in metal always occurred in an engine as the number of hours since overhaul increased. If such an increase did occur, then one would expect to see some correlation between high metal content and failure regardless of whether or not high metal content in a particular engine is a good indicator of failure in that engine. This would occur because the failure rate for engines would be expected to increase as the number of hours since overhaul increases. If the metal content also increased, independent of failure rate, a false correlation would occur due to both of the parameters' increasing independently, but simultaneously, with hours since overhaul. Table 3 shows the results of this computer run. It will be noted that the average values for each metal are all well below

TABLE 3

COMPARISON OF OIL CONTAMINATION
AT 250 AND 750 HOURS AFTER OVERHAUL

Metal	Average Parts Per Million of Metal For All Engines at <250 Hours	Average Parts Per Million of Metal For All Engines at > 750 Hours	Threshold Values
Cu	5 <b>.</b> 4	4.3	14
Ag	0.23	0.22	3
Ni	1.9	1.4	8
Fe	15	11	35
Cr	2.3	1.7	10
Sn	3.2	<b>3.</b> 5	20
Al	2.0	1.5	8
Average o all Metal		3.3	

the threshold values and that the difference in the average values for the engines having less than 250 hours since overhaul and the engines having greater than 750 hours since overhaul is small compared to the typical variations obtained in Figures 1 through 72. This is also true for the averages for all metals. The average values for all of the individual metals except tin are, as a matter of fact, lower for the engines having the higher time since overhaul. These data would certainly indicate that a fallacy in the correlations will not occur because of this phenomenon.

#### GOVERNMENT AND INDUSTRIAL ORGANIZATIONS

#### ENGAGED IN METALS-IN-OIL ANALYSIS

There are several Government and private organizations which, over a period of several years, have been engaged in analyzing the internal condition of power plants through an examination of the metals found in their lubricating oil. In addition, there have been several organizations which have just seently become active in this field. A number of these groups are interested in power units other than aircraft engine and transmissions. The data these organizations have gathered are not pertinent, except in a very general way, to the particular task related to aircraft. With one exception, as attempt was made to survey these organizations for data.

A list of or inizations located on the North American Continent known to be engaged in metals-in-oil analysis work is given conow. Although the list is believed to be complete as far as Caganizations interested in aircraft are concerned, there may be some organizations omitted that are engaged in analysis of other types of lubricated power plants.

#### 1. Atlantic Refining Company, Philadelphia, Pennsylvania:

Their program is mainly concerned with wear studies on automobile engines in relationship to particular motor oils. No survey was made in connection with this contract.

#### 2. Analysis, Incorporated, Oakland, California:

This company offers oil analysis as a part of their services to industry. They have had some experience with aircraft engines and transmissions although much of their oil analysis work has been with diesel engines. It was determined by telephone that insufficient data on aircraft engines were available to warrant a data-gathering trip.

#### 3. Analysis of Puerto Rico, San Juan, Puerto Rico:

This newly formed company has announced that as a part of their analytical services they are prepared to analyze oil from aircraft engines for metal content. Inasmuch as they have been in business only a short while, it was felt that any data which they may have gathered would be of too fragmentary a nature to be of use in the present study. Therefore, no contact was made.

#### 4. Denver and Rio Grande Western Railroad, Denver, Colorado:

This railroad has pioneered metals-in-oil analysis for locomotives utilizing diesel engines. They credit the use of spectrographic analysis of oil in conjunction with improved maintenance schedules for a large savings of diesel equipment and overhaul and repair time over competing railroads. Discussions with the personnel indicate they are able to categorize the wear patterns of individual diesel power plants with a rather high degree of precision. It has also been noted that large diesel engines deposit a great deal more metal in the lubricating oil during the course of normal wear than do aircraft engines. In conjunction with the present studies, contact was made with this organization in order to obtain additional background information concerning the activities of various groups analyzing lubricating oils for their metal content. Although the discussions were quite helpful in a general sense, the great differences in power plant type between the engines they are interested in and aircraft engines and transmissions prohibit the gathering of data of specific interest to this program.

#### 5. D-X Sunray Oil Company, Tulsa, Oklahoma:

Spectrographic analysis of this oil company is concerned with the improvement of lubricating oils in conjunction with engine wear. No contact was made.

#### 6. Ford Motor Company, Dearborn, Michigan:

The primary interest of this company in conjunction with oil analysis is to determine wear rates on automobile engines. No contact was made.

#### 7. General Motors, Cadillac Division, Cadillac, Michigan:

General Motors has utilized oil analysis in the same manner as Ford Motor Company. No survey was made in connection with this contract.

#### 8. Louisville and Nashville Railroad, Nashville, Tennessee:

The L and N Railroad utilizes spectrographic analysis for the same type of engines as the Rio Grande line. No contact was made.

#### 9. Milwaukee-Chicago Railroad, Chicago, Illinois:

Their spectrographic analysis program is concerned only with diesel locomotive engines. No survey was made.

#### 10. New York Central, Cleveland, Ohio:

New York Central has been one of the pioneers in spectrographic oil analysis for diesel locomotives. Very recently, they have contracted with the U.S. Air Force to monitor some of their aircraft engines and transmissions. At the time the contract was made in connection with this contract, it was determined that their program with the Air Force was only in the formative stages.

#### 11. Pennsylvania Railroad, Philadelphia, Pennsylvania:

The Pennsylvania Railroad's oil analysis program was concerned only with large power plants.

# 12. <u>Pennsylvania State Highway Department, Harrisburg</u>, Pennsylvania:

Although they were reported to be making a metals-inoil analysis, the exact nautre of this work could not be determined. No direct contact was made.

#### 13. Petroleos Mexicanos, Cuidad de Mexico, D.F., Mexico:

Although the exact nature of the spectrographic work is unknown, it is presumed that their program is similar to that of American oil companies.

#### 14. Standard Oil of California, El Segundo, California:

Their oil analysis program is primarily concerned with study of lubricating properties of various oils. No survey was made.

#### 15. Standard Oil of Ohio, Finley, Ohio:

Again, their program is concerned with the improvements of lubricating properties of oil. No contact was made.

#### 16. Trans-Canada Airways, Montreal, Quebec, Canada:

TCA has had a co-operative agreement with Imperial Oil Company, Ltd., at Sarnia, Ontario, to monitor the engine wear of the Rolls Royce Conway and Rolls Royce Dart Jet engines. Both of these engines have lubricating systems which allow facile study of these engines with regard to wear characteristics as determined by matter deposited in oil. Their work on the Conway engine is essentially completed, and the information derived from these studies was obtained in detail through personal contact in connection with this contract. Their work

with the Rolls Royce Dart engine was only in the initial stage at the time of contact and was therefore too fragmentary to lend itself to usefulness in connection with this project at this time. The detailed analysis of the Conway engine will be found elsewhere in this report.

#### 17. Union Pacific Railroad, Ogden, Utah:

In common with other railroads doing spectrographic analysis, the UP has concerned itself only with heavy locomotive engines. No contact was made.

#### 18. U. S. Army, Transportation Corps, Ft. Rucker, Ala.

The oil analysis activities at Ft. Rucker have been chiefly engaged in helicopter engines and transmissions. At the present time they have about 800 units under study or on which study has been completed. About half of these have been accumulated recently so that very little data exist. As of March 1962, the following aircraft engines were being monitored:

TYPE AIRCRAFT	NUMBER OF AIRCRAFT	TYPE ENGINE
H-13 H-19 H-21 H-23 H-34 H-37 HU-1 L-19 L-20 L-23 L-26 AC-1 AC-1 G-91T R-4D T-28 U-1A	44 38 7 33 34 31 266 93 97 31 23	0-335-5 R-1300-30 R-1820-103 0-435 R-1820-84 R-2800 T-53 0-470 R-935 0-480 0-435 R-2000 T-53 80302 R-1830-90D R-1830-86 R-1340

The above constitutes a list of units before the recent addition. The 100 or so units not included in the above list are in the inactive files. All of the available information from Ft. Rucker was obtained with the exception of the newly added aircraft. No statistical analyses were made of these engines because of the insufficient quantity of data on any one type of engine.

#### 19. U. S. Navy, Naval Air Station, Pensacola, Florida:

The Overhaul and Repair Base has been one of the leaders in metals-in-oil analysis for aircraft power plants. During the last 5 years, they have had a program which has involved mainly reciprocating engines but which recently has been expanded to include pure jet engines, turbojet engines and transmission units.\* An extensive amount of usable data has been gathered at this facility. The entire inactive, completed files, which comprise about 3,800 aircraft engines and transmissions, have been obtained in accordance with the specification of the contract. These files included about 240,000 individual oil analyses obtained for the following nomenclature types:

#### ENGINE MODEL NUMBER

0-335-5B 0435-6A 0435-23B (23500-7) 0-470-4 R975 R985-14B R-1300-3C R-1300-3D R1340 R1820-80 R1820-82 R1820-84 R1820-86 & 86A R1820-103 R1830-90D R1830-92 R2000 R2800 All R3350 \$14-35-4300-16 J34-46 & 48

<sup>\*</sup>Since the beginning of 1962, they have initiated a program whereby helicopter manufacturers submit oil samples taken from test stand engines and transmissions for analysis. However, this information is still rather fragmentary.

#### VAPOR PHASE CHROMATOGRAPHY

Over the past decade, a new analytical chemical technique has been evolved as a powerful, versatile and useful tool for a variety of identification and estimation tasks. This tool, vapor (or gas) phase chromatography, allows rapid separation of complex mixtures that heretofore were very difficult to handle. This analytical method has gained such wide acceptance that in addition to being an investigational tool, vapor phase chromatography is routinely used as an in-stream monitor for a variety of chemical processes. Through the use of suitable equipment and techniques, qualitative estimates of small amounts of metal can be made to a high degree of accuracy.

Vapor phase chromatography consists of the passage of gas, or vapor, dissolved in solvent gas (called the moving phase), along a column containing a stationary phase. The stationary phase can be either a liquid or a solid. However, most general application techniques call for the use of a solid. The stationary phase is packed in a column, very often in a form of a helix, for compactness. This column, the heart of the system, is generally equipped with a thermostated heating control, a detector which can be one of a number of types, and other necessary control and sample introduction equipment.

Components of the mixture sample are separated on the column based on differences of partition coefficients between the stationary phase and the moving phase. The situation is similar to that encountered with the extraction of a solutesolvent system when one extracts the solute with an immiscible solvent. In the case of vapor phase chromatography, the mixture or solute is partitioned between the weakly attracting stationary phase and the strongly attracting moving phase; thus, as the gas sweeps down the column, the mixture is slightly attracted to the stationary phase with different partition coefficients. The partition coefficients of the component are a function of (a) temperature of the column, (b) vapor pressure of the component, (c) the activity coefficient of the component of the specified stationary phase, and (d) the molecular weight of the stationary phase. Under conditions of operations of the column, (a) and (b) are fixed so that the relative partition coefficients or the components of a sample are related to (b) and (c). In a mixture of two components, A and B, one of the components, A for example, will be retained more tenaciously due to factors (b) and (c) above than will component B. As A and B are swept down the column, they will be separated and, under proper operating conditions of the column, will emerge at the bottom of the column at separate times. A detection device placed at the bottom of the column will sense the emergence of A and B and when coupled with a proper recorder, will plot this emergence as a function of time and amount of A and B.

The time required to elute a component from the column is a function of (a) the linear velocity of carrier gas through the column, (b) the partition coefficient of the component, and (c) the column temperature. In those cases where the partition coefficients of the various components in the system are quite similar, factors (a) and (b) above are adjusted so that the eluting time is sufficiently long to provide adequate separation. Under some conditions, this separation time can be as long as 30 minutes. However, for many separation problems, the elution can be accomplished quite rapidly, i.e., less than 5 minutes.

Recently, vapor phase chromatography has been extended to separate a number of volatile metal chelates\* of acethylacetone and its fluoro- and chloro-derivatives<sup>6</sup>. Although this study is only in the preliminary stages, the separations have been achieved with good results. It is known that the heavy metal ion chelate of acetylacetone and its derivatives are volatile and, furthermore, are stable in the temperature range at which the chelate column must be operated.

Because of the great utility and inherent simplicity, which results in ruggedness, compactness, and ease of operation, vapor phase chromatography shows great promise of providing an in-the-field-type analytical system for metals in oil. While it has been fairly well established that metal chelates can be separated and estimated by vapor phase chromatography, work remains to be done to show that the metals found in lubricating oils can be put in the proper chemical form so as to allow chromatographic separation to be performed on them.

Two workable approaches seem feasible. The first involves a technique of ashing such as has been used previously in arc spectrograph of oil. In this method, the oil sample is rapidly reduced to an ash, eliminating the hydrocarbon portion of the sample. This ash then can be quickly dissolved and chelated with a suitable organic compound. The second method involves the direct extraction of the metals

<sup>\*</sup> Chelates are complexes consisting of an organic portion called a dentate and a metal ion called a legand. In this complex the positive metal ion is strongly associated with two or more negatively charged groups in the organic portion to form a rather stable compound.

in the oil into an aqueous layer through some sort of device such as an ultrasonic mixer. The aqueous solution will then be allowed to react with a suitable chelating agent, so as to prepare a sample suitable for vapor phase chromatographic separation.

Detectors are presently commercially available which are insensitive to hydrocarbon but which have a high sensitivity for compounds such as heavy metal chelates. Through the use of such a device, it will be necessary to eliminate only the bulk of the oil because traces will not interfere with the analysis.

It, therefore, seems possible that an analytical device based on a vapor phase chromatography including ease of operation, ruggedness, high sensitivity and reliability can be constructed which will meet the various criteria necessary for a field unit. The germane problem is one of sample preparation. The feasibility of using either one of the applications outlined above can be rather easily determined by a very simple experiment. It is proposed that the necessary steps to prove these sample preparation concepts be investigated in Phase II of this project.

#### FLAME SPECTROPHOTOMETRY

For a number of years, emission flame spectrophotometry, 5,9 has been an accepted analytical method for a variety of analyses, especially those involving the alkaline metals and alkaline earth metals. The basis for this method of analysis involves the use of a flame to excite atoms from their ground state to some higher energy state. Because these atoms are unstable in these energy states, they quickly revert to the original ground state with the emission of their excess energy. This emission of energy often appears in the form of invisible light photons. Laws concerning the excitation and decay of atoms to and from the ground state require that such transformations be made in discrete energy jumps and that the return from the excited state also be made in discrete energy steps. However, these two processes need not necessarily follow the same path. The results are that definite wave lengths of light are produced from particular atoms. An example is the familiar intense yellow light produced by the burning of a sodium compound in a flame. This is attributed to the emission of light at 5.890 angstroms as the excited sodium atom decays to the ground state. In this case, excited sodium always emits this wave length (among others) so as to provide the basis of a dependable analytical system. It can be seen that an analytical instrument based upon this phenomenon can be devised using a reproducible flame as an excitation source, a prism and slit arrangement to provide monochromatic light, and suitable light intensity sensing equipment. With such an instrument the energy (wave lengths) of the spectro line indicates, oftimes unambiguously, the atom from which it originated; hence this is a qualitative method of analysis. While the intensity of the light, i.e., the number of photons, indicates how many atoms have been excited, it is therefore a quantitative method of analysis. Unfortunately, this type of analysis is not particularly sensitive to iron or other metals found in lubricating oils.

In 1955, a new flame spectrophotometry technique was developed. The essential difference between this new method, known as absorption flame spectrophotometry, and the well-known emission spectrophotometry is that, whereas emission spectrophotometry depends on the light released in the decay form in excited state, absorption spectrophotometry is based on observing the light energy absorbed in the excitation process. For a number of theoretical and practical reasons, the absorption process is quite often a more accurate method of analysis than those methods depending upon the emission analysis.

The following table indicates the accuracies which are obtainable through the use of absorption flame spectrophotometry for metals found in lubricating oils:

TABLE 4
ATOMIC ABSORPTION SPECTROPHOTOMETRY
DETECTION SENSITIVITIES

ELEMENT	SENSITIVITY - PPM		
	Air-Acetylene	Oxyhydrogen	
Iron	0.1 (Note 1)	0.03 (Note 1) 0.1 (Note 2)	
Silver	0.1	0.01 (Note 2)	
Aluminum (Note 3) Chromium	0.5	0.15	
Nickel Silicon (Note 4)	O.Ol NOT DETECT	0.1 ED	
Carbon (Note 4)	NOT DETECTE	ED	
Magnesium (Note 2)	0.002	0.1	

- Note 1: The use of isopropanol enhances the accuracy as high as 10-fold.
- Note 2: Use of isopropanol increased the sensitivity 2-fold.
- Note 3: Aluminum is undetectable by direct means in absorption spectrophotometry. However, the presence of aluminum has been shown to suppress the accuracy of the detection of some elements. Therefore, through the addition of a standard amount of selected elements, the amount of aluminum may be detected by noting the suppression.
- Note 4: Neither silicon nor carbon is detectable by absorption spectrophotometry. Therefore, the presence of dirty oil contaminants does not interfere with the success of the analysis.

Instruments of this type are mechanically and electrically fairly uncomplicated. A complete unit is sufficiently small so as to allow operation on a desk top. It appears, therefore, that the several criteria for a field unit can be met if this analytical method is adopted.

Although this looks like a promising method of analysis, there has been no work done with analyzing metals in a medium consisting of oil. This problem together with the problem of accurately analyzing aluminum will require further investigation. It is suggested these be made items of concern for study in the early portion of Phase II.

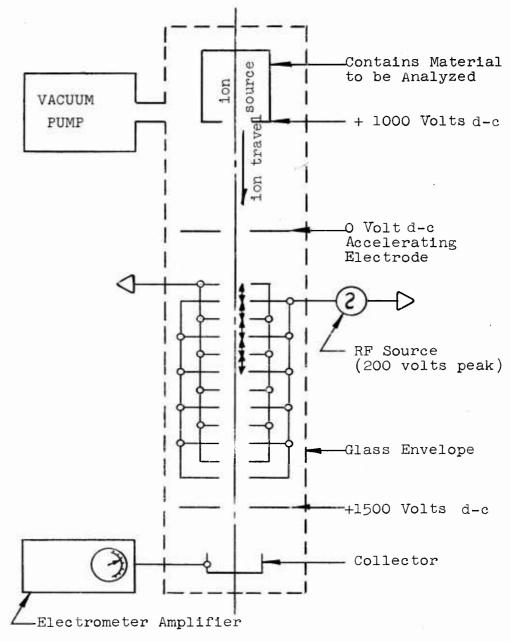
#### RADIO FREQUENCY MASS SPECTROMETER

With its inherent sensitivity and relatively small size, the radio-frequency mass spectrometer (not to be confused with commercial type mass spectrometers) appears to offer an approach to the analysis of metals in oil.

The operation of the R.F. analyzer is based on the fact that ions of the same energy but having different masses are traveling at different velocities. Figure 1 is a sketch of an R.F. analyzer of the Redhead type, which is capable of resolution on the order of 50 and extremely high sensitivity. Referring to Figure 1, the operation of the R.F. analyzer is as follows:

- 1. The material to be analyzed is placed in the ion source, where it is ionized, probably by means of an arc discharge.
- 2. The ions thus created are then accelerated in an axial direction by the +1 kilovolt d-c electrode until they enter the electrode space excited by the R.F. source.
- 3. Assuming the R.F. source is set at a particular frequency, those ions entering space "a" during the right phase of the R.F. cycle will be accelerated, and those entering during the opposite phase will be lost to the electrodes.
- 4. The transit time of the ions accelerated in space "a" will be a function of their mass; therefore, those ions which arrive at space "b" during the proper phase of the R.F. cycle will be accelerated, while the other ions will be lost to the electrodes. A similar selection process will be accomplished in space "c", "d" and "e", until the end of the R.F. electrode structure is reached. In the manner just described, ions of the proper mass will be selectively accelerated by a particular value of the frequency used in the R.F. source, and all others will be rejected.
- 5. The ions leaving the R.F. electrode structure impinge on the collector electrode, where they create a current in the collector circuit with a resulting indication on the electrometer amplifier.

While the above description is brief, it can easily be seen that the R.F. analyzer is capable of fast, sensitive analysis of many materials. For example, if the R.F. source is swept in frequency and if the output of the electrometer amplifier is applied to the vertical deflection plates of an oscillograph whose horizontal sweep is synchronized with



Note: Electrodes, ion source, collector, etc., have circular symmetry.

Figure 1. Radio-Frequency Mass Spectrometer

the frequency sweep, then a visual display of the analysis is presented. If any particular element is present, it will cause a vertical deflection of the oscillograph at a particular point on the sweep and the amplitude of the vertical deflection will be proportional to the number of ions of the material present.

Because of the physical characteristics of ion beam formation, it will be necessary to operate the ion source and glass envelope at a pressure of approximately 10 millimeters of mercury. This pressure is easily obtained by a commercially available ion pump.

Three problems are attendant with the use of mass spectrometers operating on the R.F. principle. The first is that the sensitivity encountered in defining an element in the midst of a background of elements of a similar nature is limited by the natural resolving power. To minimize this interference, it will be necessary to eliminate as much of the oil as possible from the sample so that no ambiguousness exists due to the oil masses near the mass of metallic particles for which an analysis is desired.<sup>2</sup>

Possible methods for eliminating the oil are: vacuum evaporation, combustion in air, or possible force combustion in a stream of pure oxygen. The second problem is the extreme sensitivity inherent in this type of analytical device. This sensitivity dictates (a) that the oil-eliminating problem outlined above be done with extreme thoroughness and (b) that the apparatus be thoroughly purged between analyses to insure sole determining of the single sample under study. third problem relates to the fact that the device must be operated at a fairly high vacuum. Although this pressure is easy to obtain by commercial pumps, a problem is involved through the introduction of a sample into the apparatus. A possible approach to this problem is the use of a ball valve arrangement so that the sample can be introduced with only a slight reduction in the vacuum. These three problems will be examined in detail during the completion of Phase II.

### TIME -OF-FLIGHT MASS SPECTROMETER

The time-of-flight mass spectrometer is similar to the R.F. analyzer in that the material to be analyzed is ionized and the resulting ions are accelerated in an electric field. However, only differences in time of flight of the ions to a collector for ions of various masses are used to obtain separation, and no R.F. selector is utilized.

Figure 2 is a sketch of a time-of-flight analyzer. The material to be ionized is placed in the ionization chamber where it is ionized, probably by means of an arc discharge, since a pulsed arc can be used directly as the source of a burst of ions. The ions resulting from the arc will have a fairly narrow energy spread.

Referring to Figure 2, the burst of ions is created in the ionization chamber in the presence of an accelerating field from the high-voltage d-c.

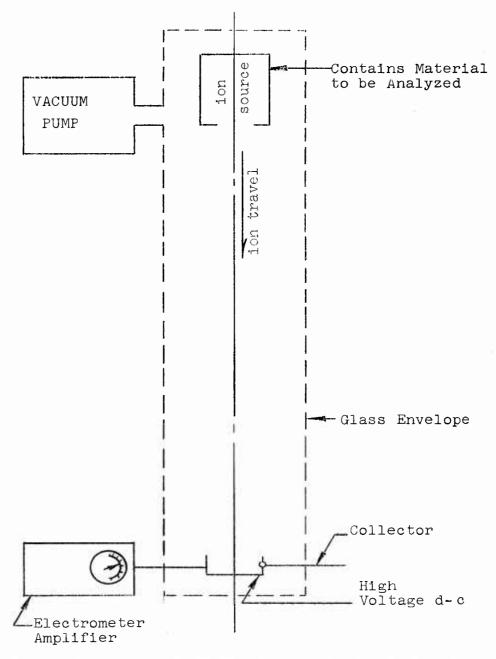
Now, as the ions are accelerated toward the collector, those ions having less mass will travel faster (because of the small energy spread, and assuming equal charges on the ions) and thus will reach the collector sooner than the heavier ones. Actually, as the ions travel toward the collector, ions of equal mass will tend to separate from the others in an axial direction along the tube.

It is easily seen that bunches of ions (grouped according to mass) will arrive at the collector at different times, the bunches of lighter mass arriving first.

The pulsed current created in the collector circuit by the impinging ion bunches can be detected by means of an electrometer amplifier, and the various pulses can be identified as to mass by noting the time of flight from the ionizing pulse to the time of arrival for a particular bunch. The amplitude of any particular collector current pulse is, of course, proportional to the number of ions in the bunch which created the pulse.

The time-of-flight analyzer is somewhat simpler than the R.F. analyzer. However, due to the fact that the ionizing pulse which creates the ions must be much shorter than the ionizing pulse in the R.F. analyzer, less ions can be created with a resulting loss in sensitivity. In addition, since no mass selection is utilized, the resolution will also be less than that of the R.F. analyzer.

It will be necessary to perform laboratory experiments to determine if the sensitivity and resolution of this type of



Note: Tube and electrodes have circular symmetry.

Figure 2. Time -of-Flight Mass Spectrometer

unit are suitable to meet the requirement of a field system for quantitative analysis of oil samples.

The problem attached with the use of a time-of-flight mass spectrometer includes the fact that, like the R.F. analysis, the analysis must be accomplished in vacuo. Therefore, the problem that the sample introduces must be overcome. It is observed that the problems of resolution and background interference that occur with the use of R.F. mass spectrometer will also occur and therefore must be solved in consideration of a time-of-flight mass spectrometer.

### NEUTRON ACTIVATION ANALYSIS:

The quantitative analysis of elemental composition of materials by neutron activation analysis is possible with existing equipments and knowledge. General characteristics of neutron activation analysis may be listed as follows:

- 1. Nondestructive testing can be effected.
- 2. The practicability of this technique differs markedly for the various elements. Some elements are ideally suited for neutron activation analysis; others can scarcely be detected by current methods using existing measurement hardware.
- 3. For elements well suited for neutron activation, extreme accuracies and no preparation of samples can be expected.
- 4. For elements well suited for neutron activation, relatively small and inexpensive nucleonic devices can be used.
- 5. For elements which are not well suited for neutron activation, the converse of statements (3) and (4) is true.
- 6. While techniques using activation of elements are comparatively well along in their development, the processes of inelastic scattering are not well documented at present. For this reason, initial considerations will be limited to activation techniques.

As indicated by the preceding statements, the question of whether neutron activation analysis is a useful tool for oil analysis hinges around the properties of the elements which must be detected.

In neutron activation analysis, the material to be analyzed is first irradiated by a flux of neutrons. Neutrons interact with the elements according to experimentally determined rules. The probability of interaction is usually expressed in terms of the effective cross section of the elemental nucleii. This cross section has units of barns (one barn =  $10^{-24}$  cm²) or millibarns (1 millibarn =  $10^{-3}$  barn). For the larger cross sections, fewer neutrons are required for a given level of activation, thus leading to a smaller number of required source neutrons. The cross sections are generally rather complicated functions of neutron energy but are independent of temperature, density, level of ionization, etc. An approximate acceptable

level for activation analysis is a cross section of 50 millibarns; a more desirable cross section would be of the order of 500 millibarns; a cross section greater than one barn usually simplifies the neutron source requirements, provided that reasonable half-lives exist.

Once the element under study is activated by the neutron flux, it exists in an excited state which decays according to an experimentally determined decay scheme and half-life. The so-called product radiations, which are emitted by the activated elements, may be particles - electrons, positrons, protons, alpha particles - or gamma photons (or some combination of particles and photons). In general, high energy neutrons tend to produce particle radiation (usually accompanied by a gamma ray) while thermal neutrons usually produce gamma radiation only. Both the energy and half-lives of these resultant nuclear radiations give indications of the presence of the product elements.

The nuclear radiation which is emitted by the activated product elements may be measured in a variety of ways. Basically, the detection equipment varies depending on whether ionizing radiation or gamma radiation is being measured. If one requires in addition the measurement of the incident neutron flux, other complications are introduced into the neutron activation system. Detection equipment design depends also on the intensity of the radiation from the product elements - which in turn depends on the intensity of the neutron source, the cross section for activation, the geometry of the scheme of activation and measurement, and the half-life of the particular product elements.

For the particular elements of major concern in oil analysis, the technique of neutron activation analysis looks reasonably applicable. Some of the possible reactions which could conceivably be used are listed in Table 5, along with the product radiation and their half-lives. This table is valid for source neutrons with energies of 14 to 15 Mev. In some instances, care would have to be taken to decrease the thermal neutron background in order to eliminate interference from the thermal reactions. This procedure is not expected to be a difficult one in practice.

The reactions listed in the table indicate that all the major contaminants in oil should be measurable during reasonable times of measurement (the reactions with half-lives less than 10 seconds and greater than 10 hours have not been included in the table). Conversely, the elements in the oil should not provide appreciable interference: hydrogen, oxygen, carbon, and boron either have reactions resulting in stable isotopes or produce elements with half-lives extremely short or very long.

TABLE 5
NEUTRON REACTIONS FOR OIL CONTAMINANTS

Element	Isotope	Natural Abundance	Reaction	Cross Section*	Product Half-Life	Radiation	Energy of Radiation(Mev)
Copper	Gn <sub>63</sub>	%69	cu <sup>63</sup> (n,2n)cu <sup>62</sup>	550 mb	9.7 min	+8	2.91
Aluminum	A1 <sup>27</sup>	100%	$A1^{27}(n,p)Mg^{27}$	90 mp	9.45 min	188 Y	1.75, 1.59 0.84, 1.02
Silver	Ag 107	51%	Ag <sup>107</sup> (n,2n)Ag <sup>106</sup>	550 mb	24 min	+ 2 2 ×	1.95, 1.45 0.36 0.512
	Ag 109	%67	Ag <sup>109</sup> (n,2n)Ag <sup>108</sup>	800 mb	2.3 min	σ. ×	1.77 0.62, 0.44
Chromium	Cr52	848	cr <sup>52</sup> (n,p)v <sup>52</sup>	80 mb	3.75 min	γ <i>β</i>	2.6 1.44
Iron	Fe 54 Fe 56	00 00 00	Fe54(n,2n)Fg53 Fe56(n,p)Mn56	15 mb 110 mb	8.9 min 2.6 hr	4 2 2 ×	2.5 2.86, 1.05, 0.75 0.84, 1.81, 2.13
Tin	Sn 112	1%	Sn <sup>112</sup> (n,2n)Sn <sup>111</sup>	1.4 b	35 min	+87	1.51
Nickel	N158 N160	000 000 000	N158(n,p)co58 N160(n,p)co60	310 mb 240 mb	9 hr 10.5 min	44	0.025 0.056

 $<sup>^{*}</sup>$  The cross sections listed refer to the approximate values for 14 to 15 MeV neutrons.

In order to determine the neutron flux required to detect the various elements which constitute the contaminants, it is first useful to define a "figure of merit" or an ease of neutron activation analysis. This figure of merit is described approximately as:

F of M = 
$$\frac{(PPM Threshold) (\sigma) (\% Abundance)}{(Produce Half-Life)}$$

It should be realized that this equation is approximate only, ignoring the exponential nature of the decay and assuming that all product radiations can be detected with equal efficiencies. It will serve the purpose of this report, however, if it gives an indication of the neutron flux required for analysis. The resulting figures of merit for the various elements and isotopes are given below:

<u>Element</u>	<u>Isotope</u>	Figure of Merit
Copper Aluminum Silver	Cu <sup>6</sup> 3 Al <sup>2</sup> 7 Ag <sup>10</sup> 7 Ag <sup>10</sup> 9	540 68 35 510
Chromium Iron	Gr52 Fe54 Fe56	179 3
Tin Nickel	Sn112 Ni58 Ni60	23 33 48

The figure of merit values, merely serving as relative indicators, show that copper should be the easiest to measure and that tin should be the most difficult.

As a result of the previous development, one may next compute the neutron flux to produce 100 counts in a hypothetical counter after irradiation of an oil sample containing 14 ppm (wt) copper. The following assumptions are made:

- 1. The oil sample weighs 100 grams.
- 2. The oil can be placed around the source of neutrons such that 25% of neutrons emitted pass through the oil sample.
- 3. The product radiations can be detected with 50% efficiency.
- 4. It takes 30 seconds to transfer the oil from the neutron source to detection equipment.

5. Radiations from the sample are measured for 10 minutes.

The neutron flux (in neutrons/cm<sup>2</sup>) required to produce the 100 counts in 10 minutes is given by:

$$\varphi \text{ (neutrons/cm}^2) = \frac{\text{C M}}{\text{fofafifs Nome}^{-\lambda t_1} (1-e^{-\lambda t_2})}$$

where:  $C = number of counts recorded during time <math>t_2 = 100$ .

M = atomic weight of Cu<sup>63</sup> = 63.54.

 $f_0$  = factor which allows for decay of  $Cu^{62}$  atoms during irradiation. This factor is 0.983 for a 30 second irradiation time.

 $f_a$  = self absorption factor which must be determined. Assumed to be unity in this calculation.

 $f_i$  = isotopic abundance of  $Cu^{63} = 0.691$ .

 $f_s$  = geometry factor of detection, assumed to be 0.5.

 $N = Avogadros number = 6.025 \times 10^{23}$ .

 $\sigma$  = cross section for 14 Mev neutrons = 0.55 barns.

 $m = mass of copper in sample = 1.4 x <math>10^{-3}$  gm.

 $\lambda$  = the e-fold factor for a 9.7-minute half-life= 1.19 x 10<sup>-3</sup> sec<sup>-1</sup>.

 $t_1 = transfer time = 30 sec.$ 

 $t_2$  = measuring time for detection = 600 sec.

Proper substitution of these quantities yields

 $\varphi$  (neutrons/cm<sup>2</sup>) = 8.25 x 10<sup>7</sup> neutrons/cm<sup>2</sup>.

If one assumes that the sample being irradiated has a cross sectional area of 20 cm<sup>2</sup> and that one-fourth of the source neutrons are effective in irradiating the sample, then the total number of neutrons out of the source is:

 $N = 6.6 \times 10^9$  neutrons.

For a nominal 30-second radiation time, the output of the neutron generator per second would amount to

$$\frac{\alpha}{N}$$
 = 2.2 x 10<sup>8</sup> neutrons/second

This level of neutron output is easily obtained with existing neutron generators. It thus appears that the detection of copper in threshold amounts should not present any unsolvable problems.

The most difficult detection problem appears to be that of detecting tin. This should be possible, under ideal conditions, using a neutron source with an output of approximately  $10^{10}$  neutrons/second. Such a generator is well within the state of the art and does not require a large-scale development effort; however, with  $10^{10}$  neutrons/second, the additional shielding necessary to reduce personnel health hazard will probably prohibit the use of such yields in a field device.

The estimates given above must be considered as tentative: very little work has been done on neutron activation analysis on oil. There are many aspects which must be studied in more detail, and some of these aspects may prove to dispell the present comfortable estimates. Present plans call for additional study to determine the influence of some of the unknowns. In addition, some preliminary experimental work is required to determine the degree of interference and the geometries and fluxes required.

## INTERACTIONS OF CONDUCTIVE PARTICLES WITH INDUCTIVE COILS:

Assume that a coil having a diameter of 1 cm and a length of 2 cm and wound with 20 turns is used. The inductance,  $\mathbf{L}_{\text{O}}$ , of this coil may be approximated by: 7

$$L_0 = (0.01)(20^2)(\frac{1}{2.54})$$
 microhenries = 1.57  $\mu$ hy.

If this coil is operated at 1 megacycle and has a Q (quality factor) of 30, the apparent resistance, Rapp, of an antiresonant "tank" circuit is given by

Rapp = 
$$QwL_0$$
 = 30(6.28 x 10<sup>6</sup>)(1.57 x 10<sup>-6</sup>) = 297 $\Omega$ 

If this circuit is excited with 1 watt, the voltage applied to the coil will be equal to E =  $\sqrt{PR}$ 

where: E is voltage applied to the coil.

P = the applied power,

R = Rapp, the circuit resistance.

$$E = \sqrt{(1)(297)} = 17.2 \text{ volts rms.}$$

The flux in the coil is

$$\emptyset_{\text{rms}} = \int_{0}^{\frac{\pi}{2}} \frac{E(t)}{n} \times 10^{8} dt$$

where:  $\emptyset$  = the rms flux in lines:

n = the number of turns, ...

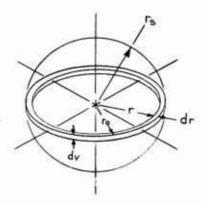
 $E(t) = 17.2 \sin 2\pi \times 10^6 t$  (for one megacycle).

$$\emptyset_{\text{rms}} = \int \frac{\frac{\pi}{2}}{17.2 \times 10^8 \sin 2\pi \times 10^6 \text{ dt}}$$

 $\phi_{rms} = 13.7$  lines, and  $\phi_{rms}(t) = 13.7$  sin wt

where:  $w = 2\pi \times 10^6$ , angular frequency.

Assume that a spherical particle having a diameter r and a conductivity of  $\rho$  is placed in this field. This particle may be approximated by a series of incremental rings as below.



The incremental ring will enclose a portion of the total field determined by the ratio of the ring area to the coil area (if the flux density across the cross section of the coil is assumed to be relatively uniform). The flux through the ring is then given by

$$\emptyset_{\text{ring}} = \emptyset_{\text{rms}} \frac{\pi r^2}{\pi Ro^2} = \underline{\emptyset_{\text{rms}} \gamma^2}_{Ro^2}$$

where: Ro = the coil radius.

The voltage in this ring is given by

$$E_{r} = \frac{d\emptyset_{ring}(t)}{dt} \times 10^{-8} = \frac{r^{2}\emptyset_{rms}\omega \times 10^{-8}}{Ro^{2}}$$

The resistance of the ring is given by

$$R_{ring} = \frac{2\pi r \rho}{dr dv}$$

where: r = ring radius

 $\rho$  = resistivity of material

dr = ring width

dv = ring thickness.

The power in the ring,  $\frac{E^2}{r}$ , is given by

$$dP = \left(\frac{r^2}{Ro^2} w^2 \not \text{grms x } 10^{-8}\right)^2 dr dv$$

$$= \frac{\pi r \rho}{\sqrt{2\pi r \rho}}$$

$$= \frac{y^3 \ (\text{Ørms})^2 \ \text{w}^2 \ \text{x} \ 10^{-16} \ \text{dr dv}}{\text{Ro}^4} \ .$$

The power in a disc of thickness dv and a radius  ${\tt r}_{\tt o}$  is given by

$$P \ \text{disc} = \frac{(\text{//rms})^2 \ \text{w}^2 \ \text{x} \ 10^{-6} \ \text{dv}}{\text{Ro}^4 \ 2\pi \rho} \int_0^{r_0} r^3 \ \text{dr} = \frac{(\text{//rms})^2 \ \text{w}^2 \ \text{x} \ 10^{-16} \ r_b^4 \ \text{dv}}{8\pi R_o^4 \ \rho} \ .$$

 $r_0$  is a function of v as shown below:



The power dissipated in the sphere, Ps, is given by

$$Ps = \frac{(\emptyset rms)^2 w^2 \times 10^{-16}}{8\pi R_0^4 \rho} \left( r_s^2 - v^2 \right)^2 dv = \frac{(\emptyset rms)^2 w^2 \times 10^{-16} r_s^5}{15\pi R_0^4 \rho}.$$

Note: Inductance effects in the ring are ignored because the ring is actually a portion of a solid sphere whose diameter is much smaller than the skin depth.

For a sphere radius of 100 microns,  $10^{-4}$  meters and a typical steel resistivity of  $10^{-5}$   $\Omega$  -cm ( $10^{-7}\Omega$  meters), the power loss per particle, Ps,

$$= \frac{(13.7)^2 (6.28 \times 10^6)^2 \times 10^{-16} \times 10^{-20}}{15\pi(\frac{1}{2} \times 10^{-2})^4 (10^{-7})}$$

= 
$$25.2 \times 10^{-7}$$
 watt per particle.

The voltage change across the  $297\Omega$  coil is given by

$$\frac{(E+\Delta E)^2}{R}$$
 = p +  $\Delta$ 'p if  $\Delta$  <<1,  $(1+\Delta)^2 \approx 1+2\Delta$ 

$$\frac{E^{2}(1+2\Delta)}{R} = p (1+\Delta')$$
, and  $\frac{E^{2}}{R} = p$ 

$$1+2\Delta = 1+\Delta'$$

$$\Delta = \frac{\Delta'}{2}$$

$$\Delta = \frac{25.2 \times 10^{-7} \text{ watts}}{2 \times 1 \text{ watt}}$$
$$= 12.5 \times 10^{-7}$$

$$\Delta E_{rms} = 17.2 \times 0.78 \times 10^{-7} = 21.6 \times 10^{-6}$$
 volts rms.

This voltage can be detected by certain common circuits. Typical of these are superregenerative receivers and superheterodyne receivers. It should be noted that this level is obtained for a single particle. It would appear, then, that a good possibility of detecting particles in this size range does exist.

## GRAPHICAL PRESENTATION OF METAL CONTENT DATA

Figures 3 through 72 are graphs showing the amount of a particular metal in parts per million versus running time since overhaul for various engines. These graphs have been prepared to aid in visualizing the manner in which metal content builds up.

Data points on the graphs were obtained from records maintained at spectrographic analysis laboratories mentioned previously in the report (pp. 12-16). Each point represents a sample analysis and points are plotted consecutively in the time order in which samples were taken. No points have been omitted for the intervals plotted in the graphs. In order to assist in the evaluation of the data, accentuated marks were placed on the abscissa scale at those times after overhaul at which an oil change in the engine was accomplished. It is to be noted that in many cases sampling and oil changes are not coincidental in time and that generally the number of samples exceeds the number of oil changes during the plotted intervals.

The abscissa of Figures 53 through 72 differ in that the scale is stated in Hours Since Oil Change (OC) rather than Hours Since Overhaul (OH) as used in Figures 3 through 52.

Table 6 presents the parts-per-million metal versus time graphs of Figures 10 through 37 in a different manner. It was desired to arrive at an estimate of the wear rate of engines using the contamination versus hours data. One method to calculate wear rate is that of using the equation

$$H = \frac{(T - P_2) / h}{P_2 - P_1}$$

where T = contamination threshold of a particular
 metal - a constant

 $P_1$  and  $P_2$  = contamination levels in parts per million of consecutive samples as measured in an oil analysis program

 $\Delta h$  = hours of engine operating time between samples P<sub>2</sub> and P<sub>1</sub>.

The parameter H, then, is the number of hours of engine operating time remaining before the metal contamination reaches the threshold level, T. If H is small, it is evident that the engine has a high wear rate at a fairly high contamination level. In Table 6, some values of H = 0 are to be noted. This is an indication

that  $P_2$  is equal to or greater than T. In the use of the wear rate formula, care must be exercised that  $\Delta h$  is taken over a sufficiently large interval of time so that measurement errors which contribute to the variation of the contamination levels will not cause an unduly large number of low values of H.

Application of a parameter of this type should be based on a large number of engines with known histories of contamination. The reliability of this type of calculation would be proven only by a detailed analysis of a large number of histories. It is planned to accomplish such calculations for the R1820-86 engine during the second phase of this contract work. For these reasons, no conclusions or statements will be made here about the significance of the data shown in Table 6. They are presented primarily to illustrate another way to use oil analysis data in hopes of eventually arriving at significantly higher utilization of engines or economically directing overhaul periods such that catastrophic failures are eliminated.

TABLE 6
WEAR RATE DATA FOR TYPICAL R1820-86 ENGINES

The data in this table were calculated from the contamination levels versus hours after overhaul curves in Figures 10 through 37. The formula on page 38 of this report was used to make the calculations.

# 1. Engine Number 520-229

Hours after overhaul	•	before Silver (Ag)		T, Will Chromium (Cr)	be reached) Aluminum (Al)
37.8 66.3 99.3 108.2 127.4 158.1 168.6 192.8 211.5	+16.5 +25.6 *** +48.6	+45  +33   & & 48.4	 0 +13.4 +40.8  +130 +56.1	 +82.5  +215  +194 +131	 +33  +19.2 ~ +32

TABLE 6 (Cont'd)
WEAR RATE DATA FOR TYPICAL R1820-86 ENGINES

2. Engine	Number 520	-230			
Hours after overhaul	H (Hours Copper (Cu)	before Silver (Ag)		T, will Chromium (Cr)	be reached) Aluminum (Al)
34.4 72.6 121.5 185.2 185.3 185.4 19.3 19.4 19.4 19.4 19.4 19.4 19.4 19.4 19.4	+73.2 +75.4 +143 +101  +91  +73 * +120 +220 +231  +32 +28.1  +78 +256	+122 +56  +114 *  +115 *  +320 +124  * * * * * * * * * * * * * * * * * *	+83 +90  +35 +54  +174 +1100  +124  +41  +43 +232  +137 +131 0  +145 +148	+220 8  +67  +67  +440  +248 +400  +279 +248  +279 +248  +279 +248  +279 +248  +279 +248  +254 +254	+122 +151  +114  +402  +99 % +187  % % +210 % +72 +141 +27  +72 +65 %
3. Engine	Number 520	-726			
38.8 60 93.8 207.1 236.8 271.3 280.3	+21.2 135  0  +4.5	8 8 +68  8	0	  +29.7	+149 +205  +178 &

TABLE 6 (Cont'd)
WEAR RATE DATA FOR TYPICAL R1820-86 ENGINES

4. Engine Number 520-764						
Hours after overhaul	Copper		Iron C	T, will be hromium A (Cr)	reached) luminum (Al)	
785.1 816.3 853.3 882.5 912.8 940.5 971.7 1001.2 1031.2 1059.4 1090.4 1117.1 1146.0 1169.3	+35 222 +52 +18.9 +67 +43	+114  8 8 8 8 8 8 60  +62  8	425 +16  97 +28  0	230  234  280 8 +35  134 	+29.2  +217 \omega +4 	

Figures 3 through 9 are graphs showing metal content in Engine Model R1820-86, Engine No. BL520159.

The comments taken from the oil analysis records are as follows: Master connecting rod bearing burned and scored. Crankshaft machining and balancing assembly scored and burned on the loaded side of the master rod bearing journal. Crankshaft front section cracked at the hydro oil supply line connector pipe threads. (Sump lite on - full of metal).

It will be noted that all of the metal contents rose very rapidly during the last few hours of operation. All of the metal values except chromium exceeded the threshold values as set by the Navy Overhaul and Repair Base. This engine, then, would appear to be a classical case of failure in which oil analysis using the threshold values would have predicted the failure of the engine.

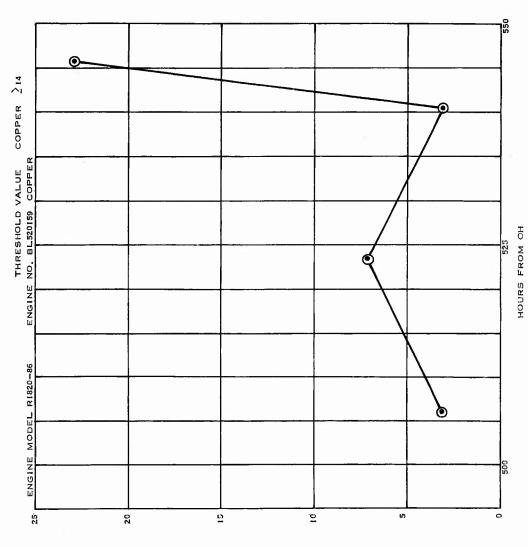


FIGURE 3. CONTAMINATION LEVEL VS HOURS FROM OVERHAUL

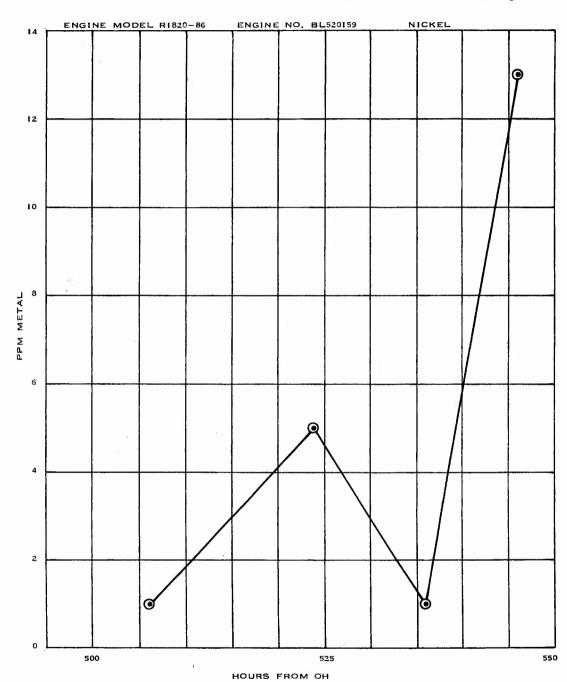


FIGURE 4. CONTAMINATION LEVEL V'S HOURS FROM OVERHAUL

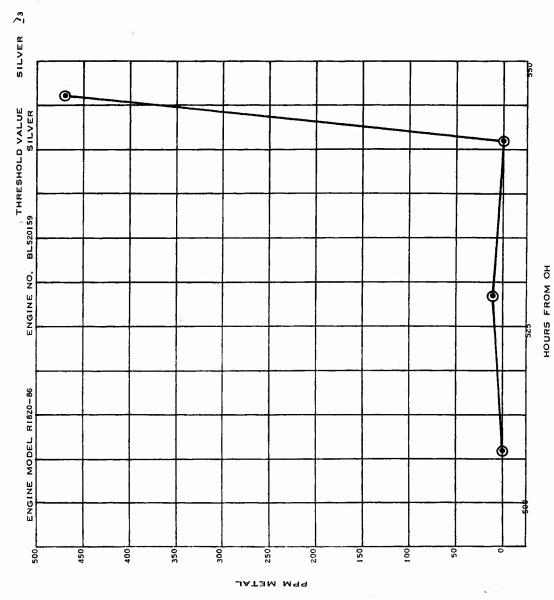
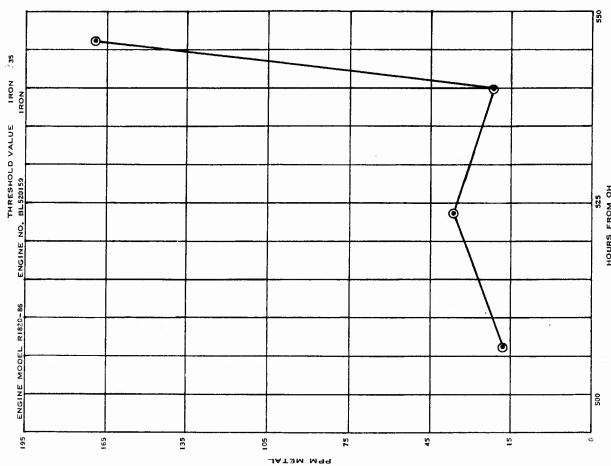


FIGURE 5. CONTAMINATION LEVEL VS HOURS FROM OVERHAUL





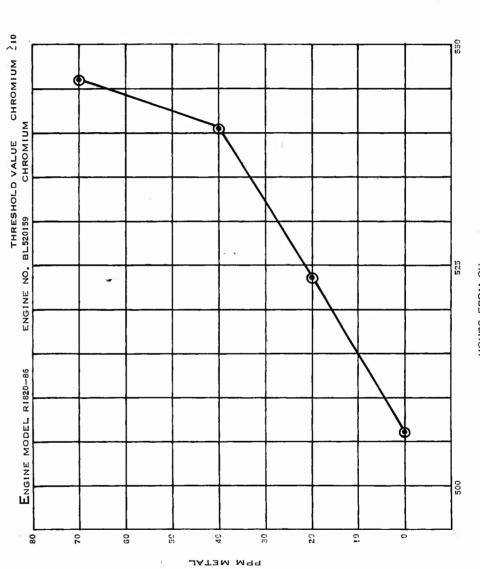


FIGURE 7. CONTAMINATION LEVEL VS HOURS FROM OVERHAUL

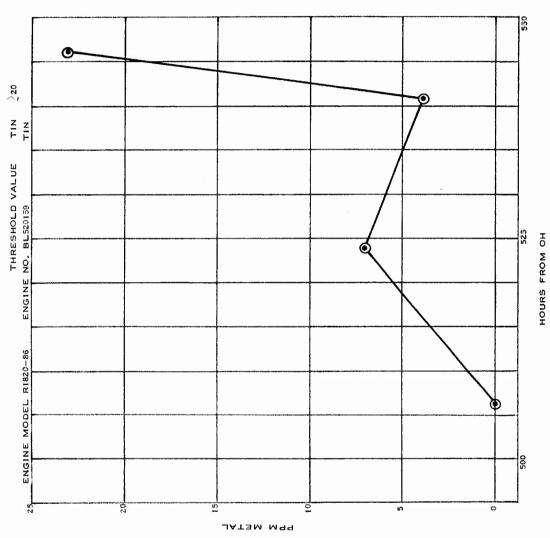


FIGURE 8. CONTAMINATION LEVEL VS HOURS FROM OVERHAUL

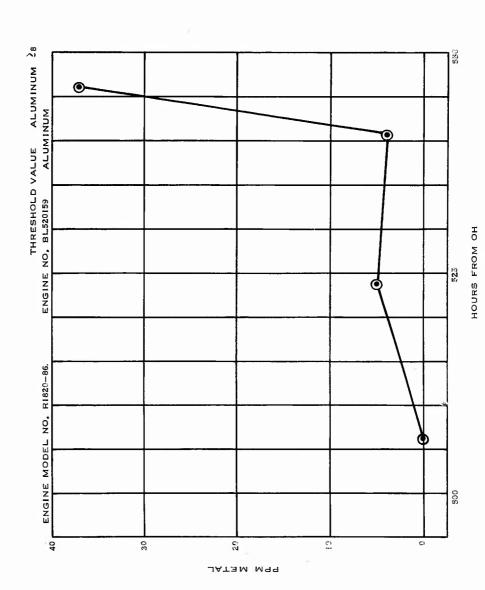
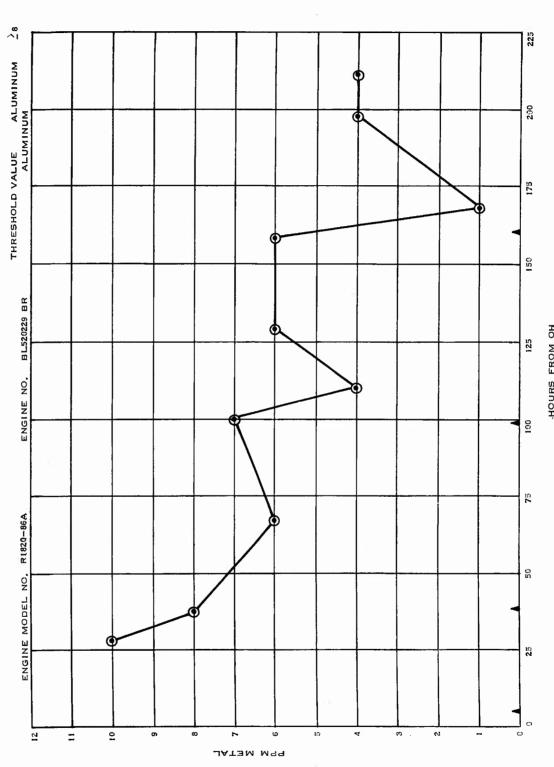


FIGURE 9, CONTAMINATION LEVEL VS HOURS FROM OVERHAUL

Figures 10 through 16 are graphs showing metal content in Engine Model No. R1820-86A, Engine No. BL520229 BR.

The comments taken from the oil analysis records are as follows: Crankshaft machining and balancing assembly bent. Supercharger front housing cracked through one mounting lug boss. (Overboost, aircraft crashed April 14, 1961.)

The failures described are ones that would not necessarily raise the metal content in the lubricant. It will be noted that aluminum, iron and nickel slightly exceed the threshold values and that the remaining elements did not exceed the threshold values. Oil analysis would not have been successful, in all probability, in detecting that a defect existed. As mentioned, however, there is little reason to expect that it would, since the particular defects are not such that they would be expected necessarily to increase the metal content.



HIGURE 10, CONTAMINATION LEVEL VS HOURS FROM OVERHAUL

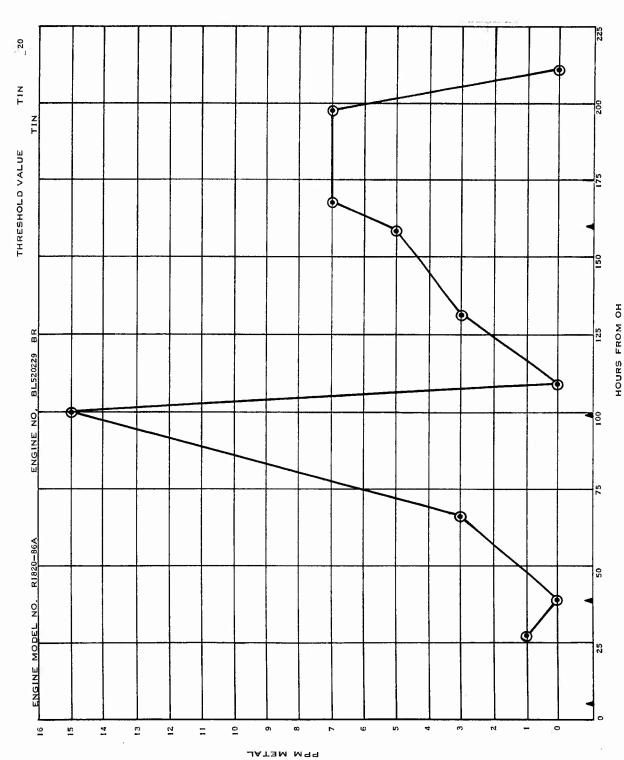
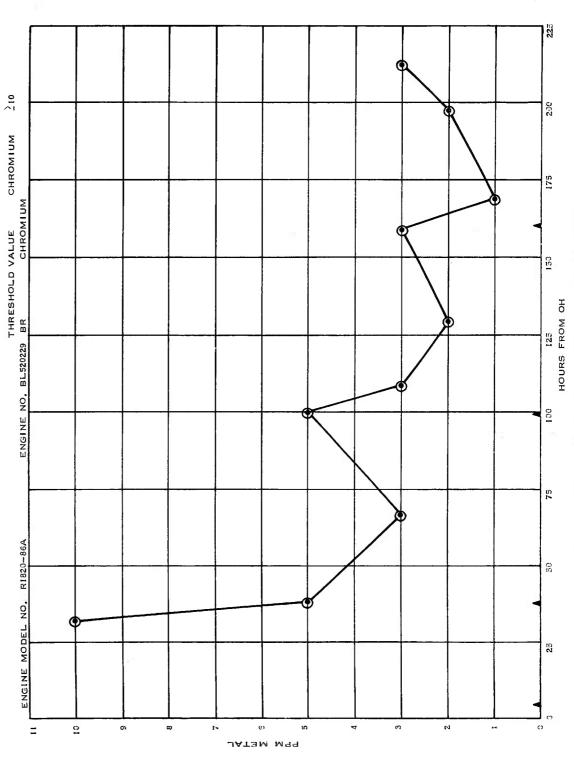


FIGURE II. CONTAMINATION LEVEL VS HOURS FROM OVERHAUL

# FIGURE 12, CONTAMINATION LEVEL VS HOURS FROM OVERHAUL



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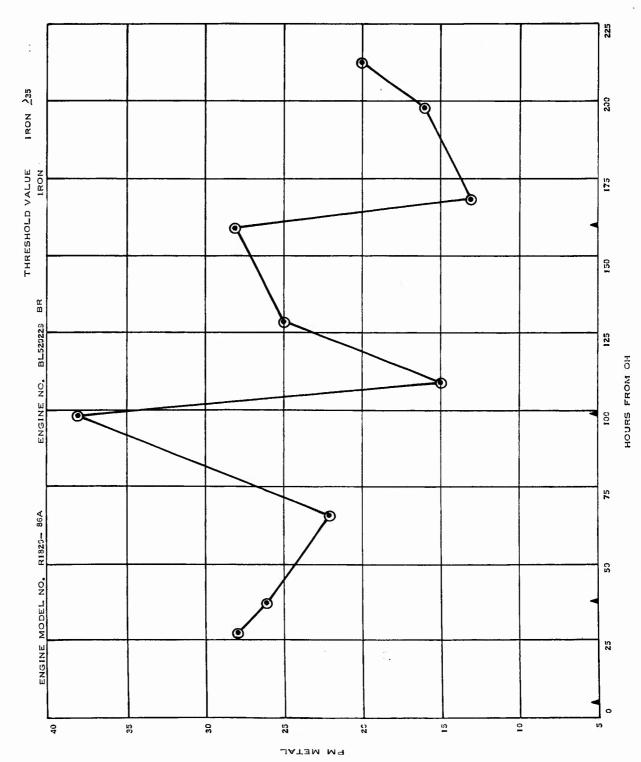
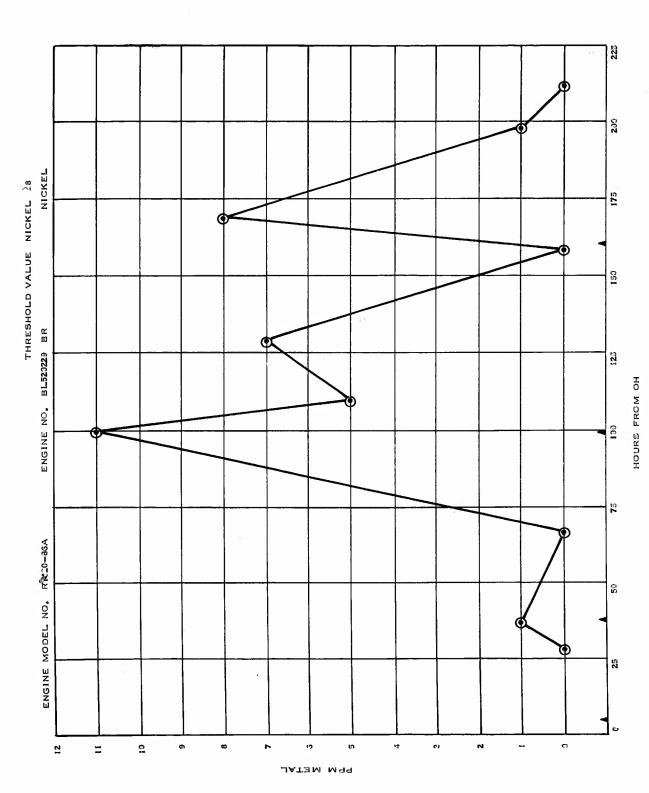


FIGURE 13, CONTAMINATION LEVEL VS HOURS FROM OVERHAUL



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FIGURE 14, CONTAMINATION LEVEL VS HOURS FROM OVERHAUL

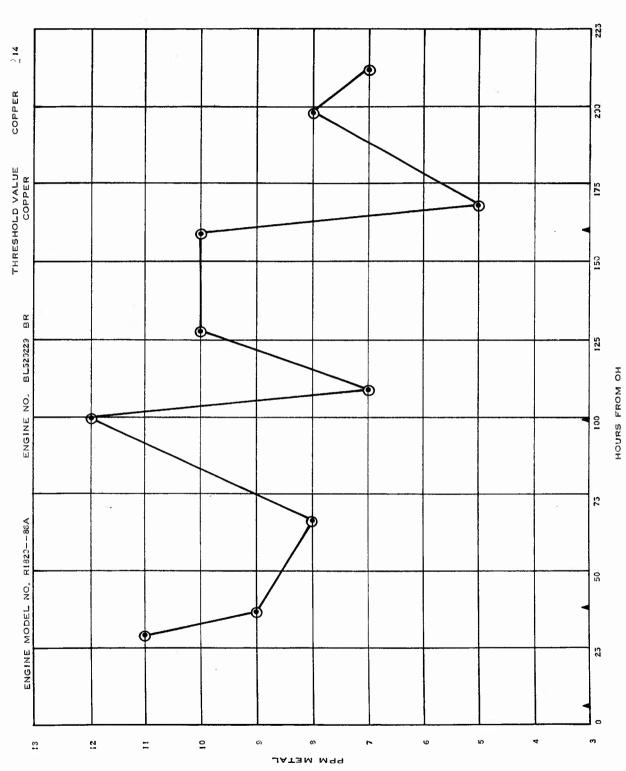


FIGURE 15. CONTAMINATION LEVEL VS HOURS FROM OVERHAUL

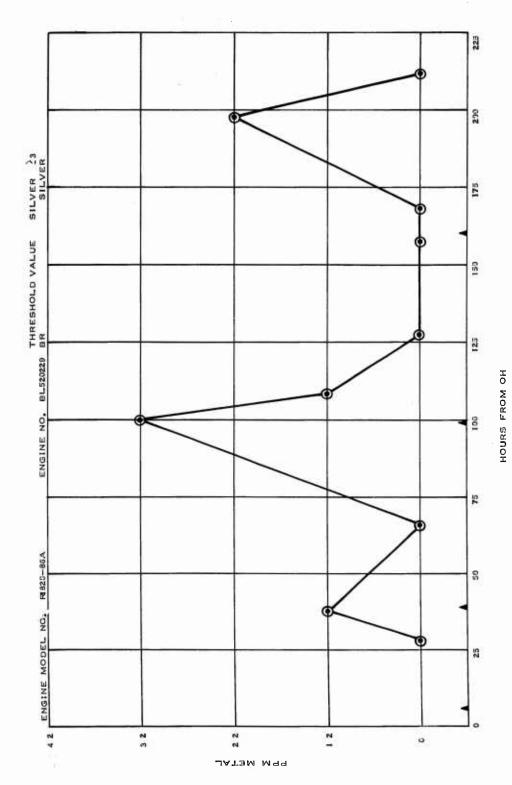


FIGURE 16, CONTAMINATION LEVEL VS HOURS FROM OVERHAUL

Figures 17 through 23 are graphs showing metal content in Engine Model R1820-86A, Engine No. BL520230.

The comment taken from the oil analysis records is as follows: No discrepancies.

These figures have been included to illustrate the variation in measurements in metal content on an engine that appears to be normal. It will be noted that on one occasion nickel exceeded the threshold value for one measurement by indicating a value of 13 parts per million as compared to the threshold value of 8 parts per million. It should also be noted that the rates indicated on these graphs are in many instances quite high. For example, on Figure 17, the value of copper increased from 0 parts per million to 6 parts per million over approximately 30 operating hours. And nickel in one instance increased from 0 parts per million to 5 parts per million over approximately 30 operating hours. These rapid rates of change in an apparently normal engine would indicate that rate of metal build-up may not be a very good indicator of discrepancies.

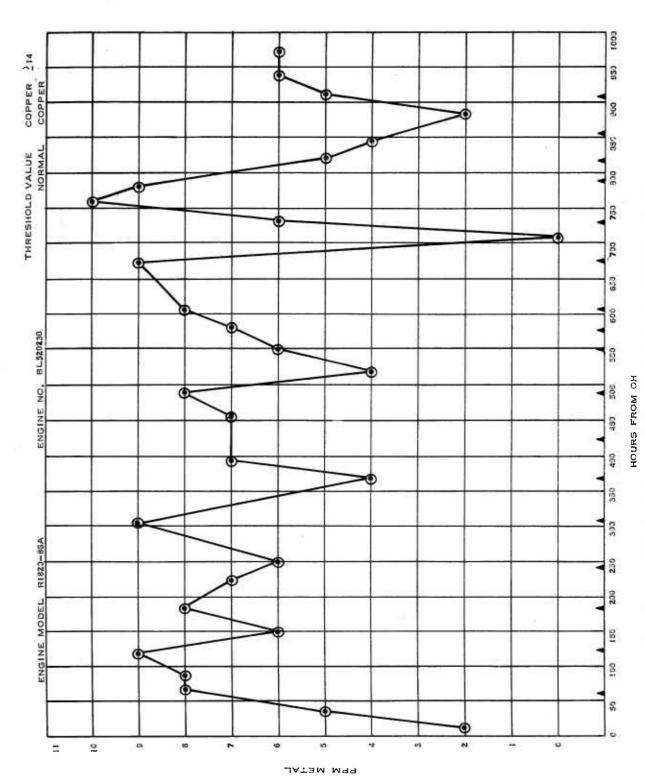
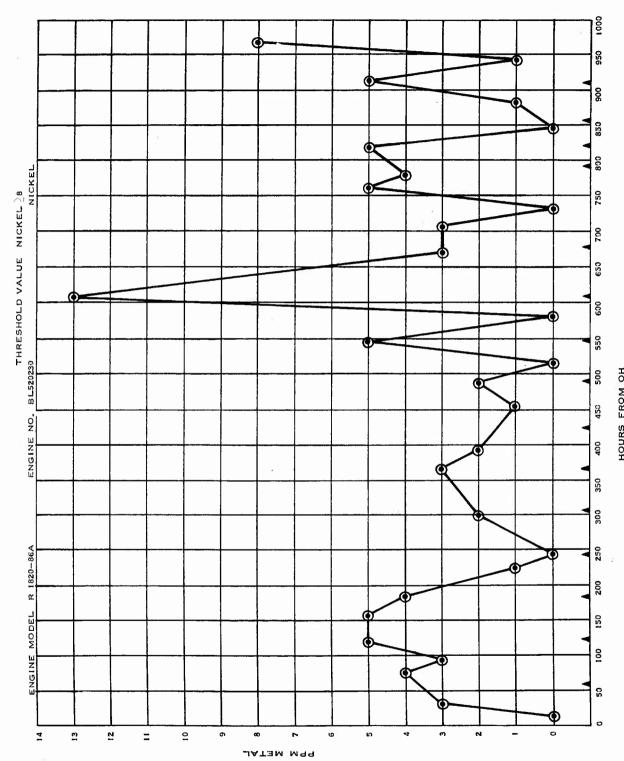
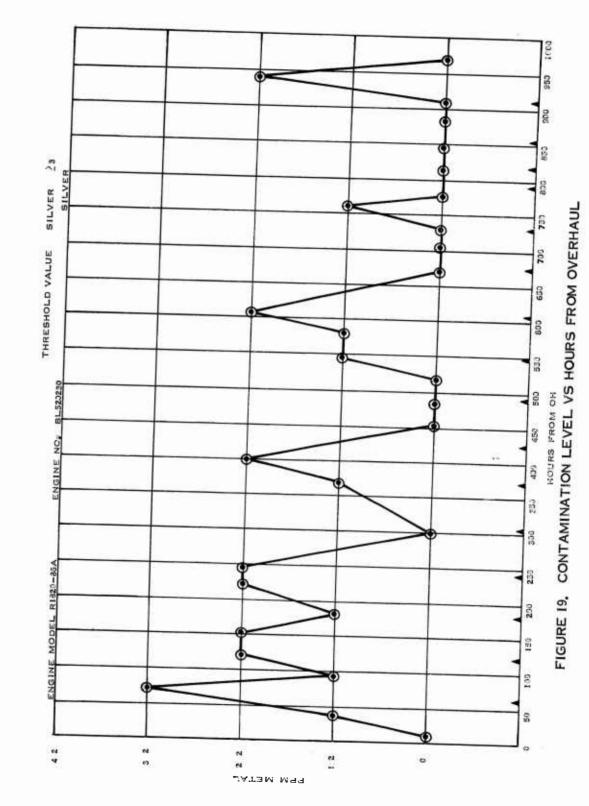


FIGURE 17. CONTAMINATION LEVEL VS HOURS FROM OVERHAUL



HOURE 18, CONTAMINATION LEVEL VS HOURS FROM OVERHAUL



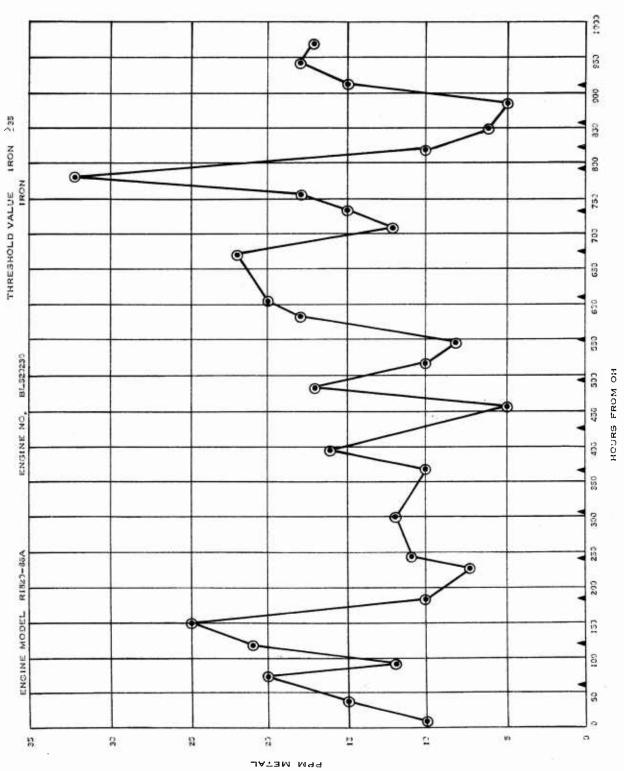


FIGURE 20. CONTAMINATION LEVEL VS HOURS FROM OVERHAUL

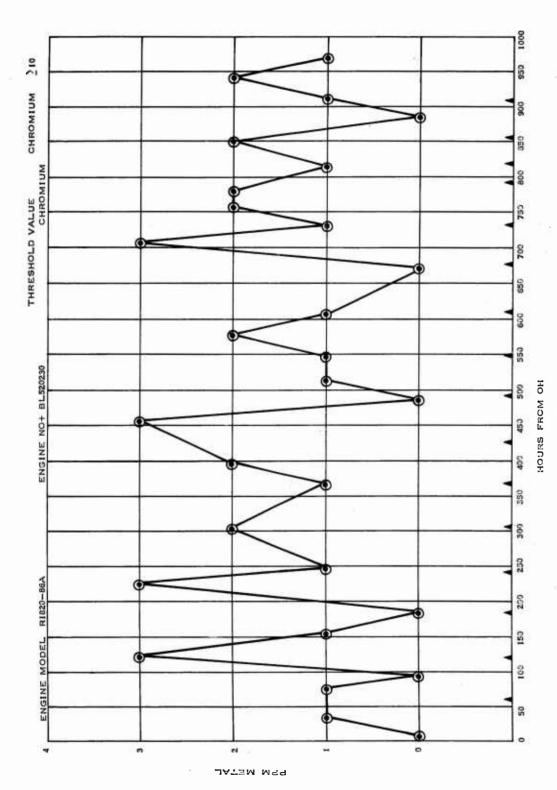


FIGURE 21, CONTAMINATION LEVEL VS HOURS FROM OVERHAUL

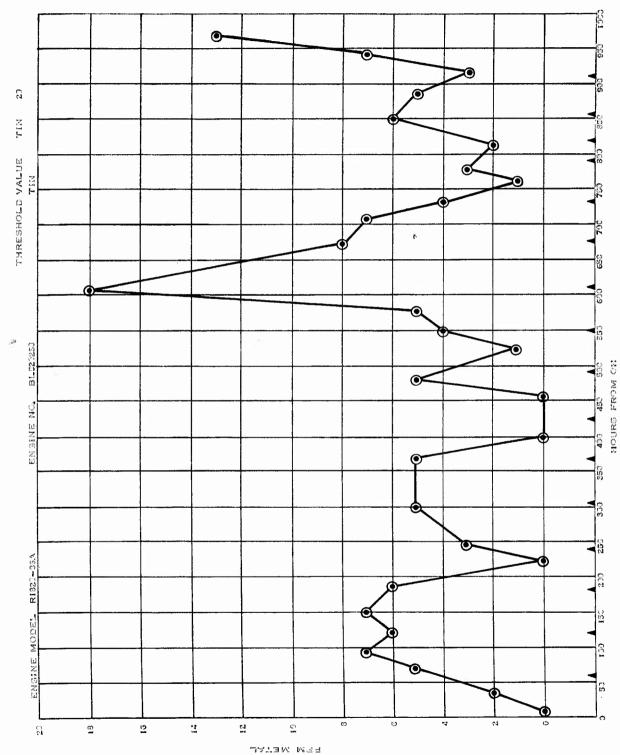
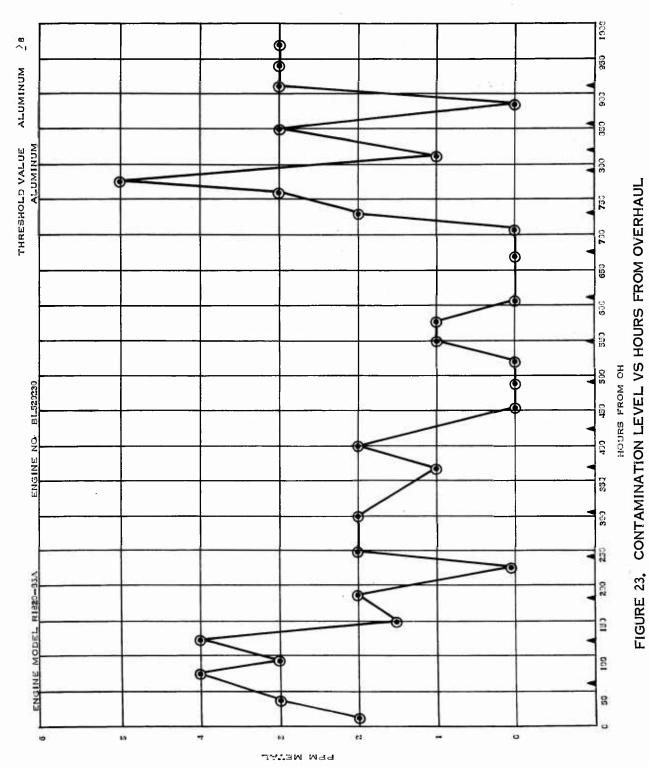


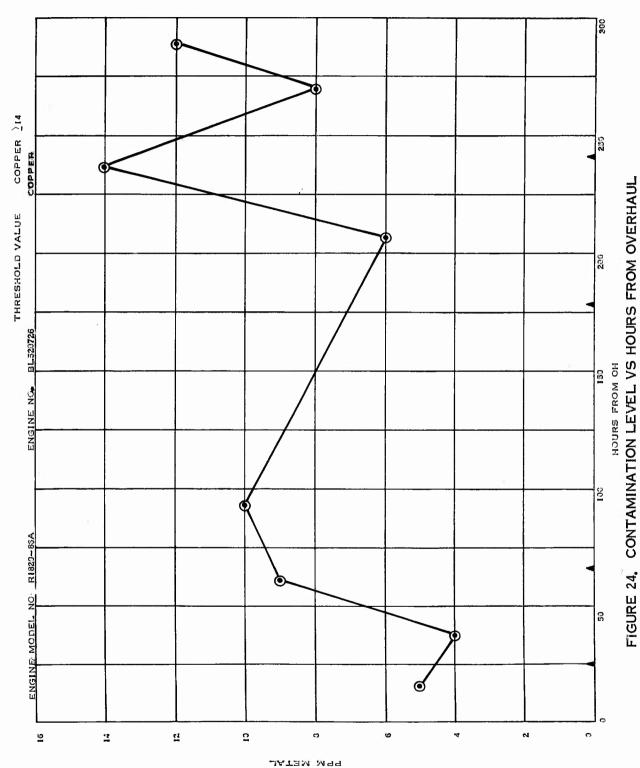
FIGURE 22, CONTAMINATION LEVEL VS HOURS FROM OVERHAUL

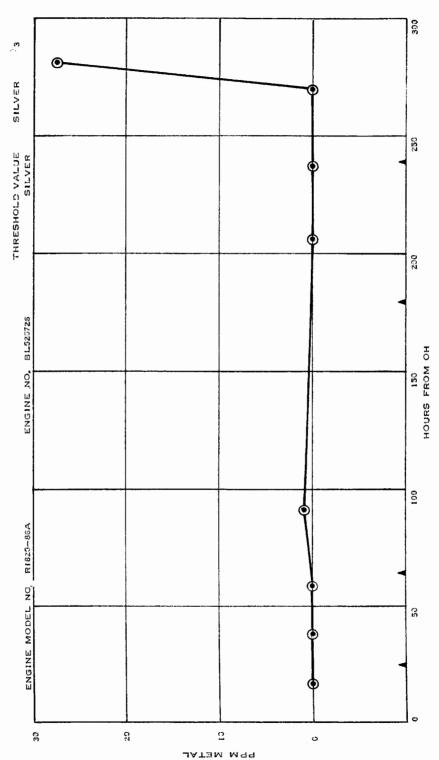


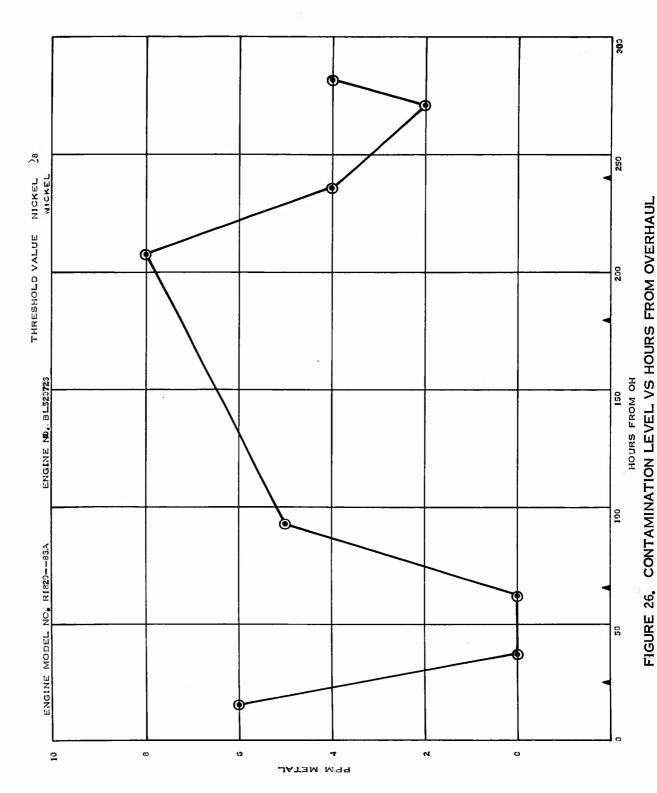
GS

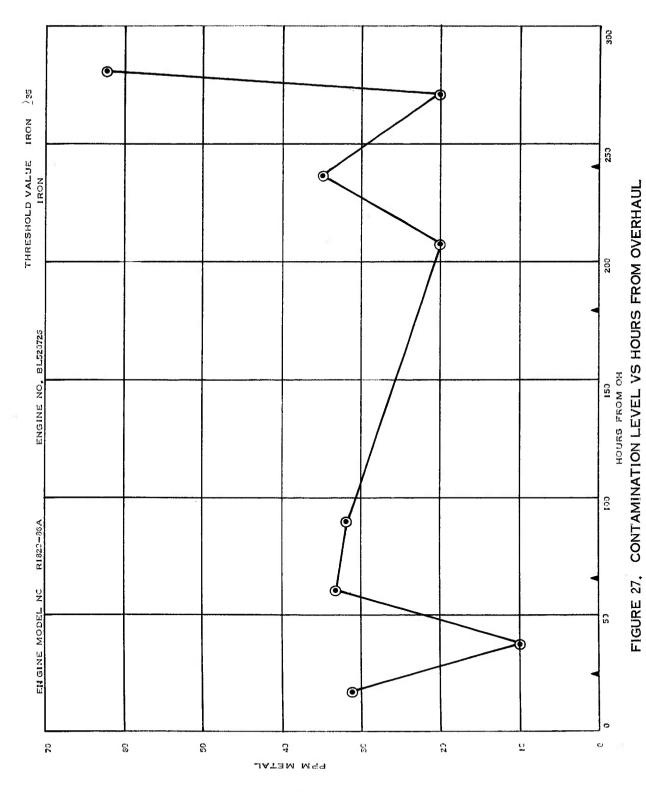
Figures 24 through 30 are graphs showing metal content in Engine Model No. R1820-86A, Engine No. BL520726.

The comments taken from the oil analysis records are as follows: Contamination - master connecting-rod bearing burned and scored. Oil passages to bearing were open. Crankshaft machinery and balancing assembly burned and lightly scored on the loaded side of the crank pin journal.

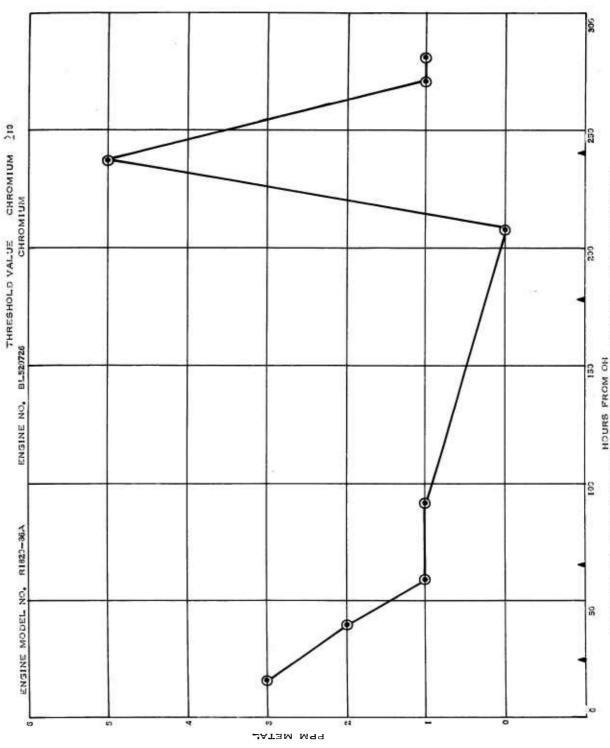


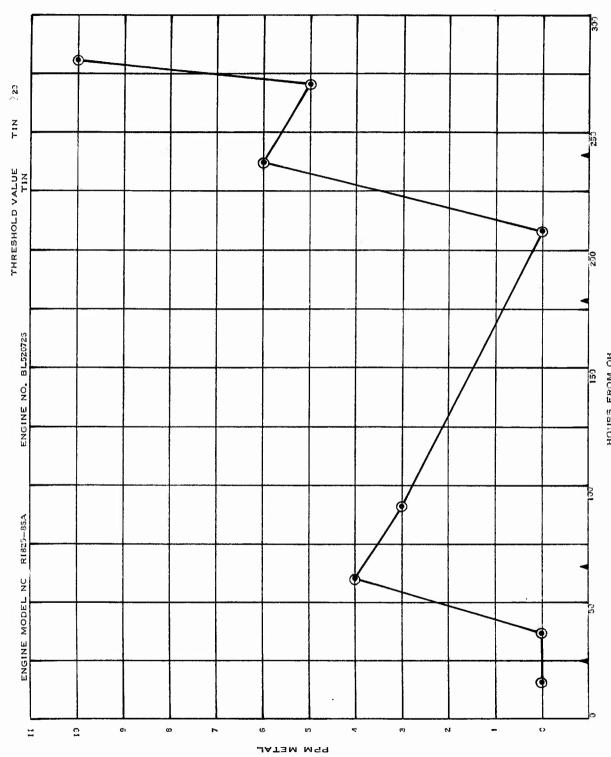




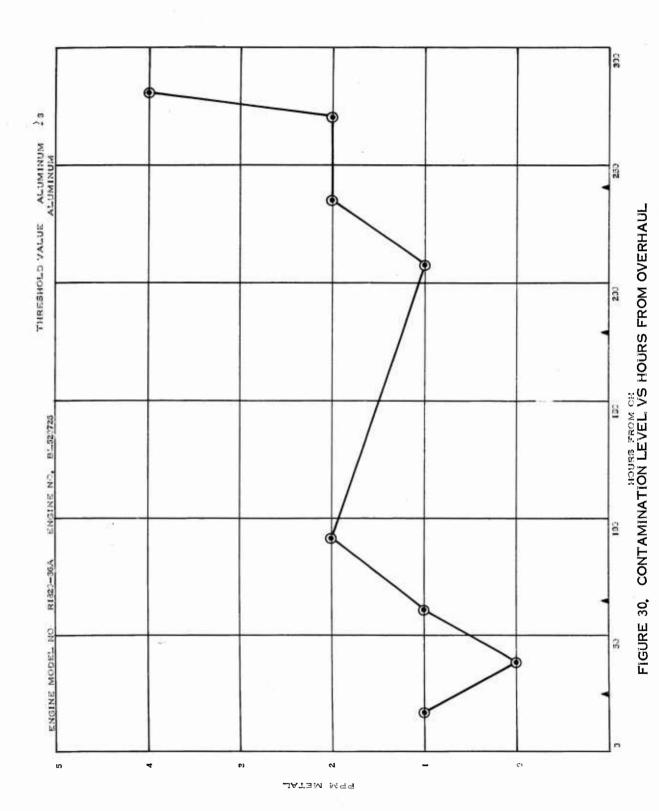


## FIGURE 28, CONTAMINATION LEVEL VS HOURS FROM OVERHAUL





ноика гком он FIGURE 29, CONTAMINATION LEVEL VS HOURS FROM OVERHAUL



Figures 31 through 37 are graphs showing metal content in Engine Model R1820-86, Engine No. BL520764.

The comments taken from the oil analysis records are as follows: Contamination - master connecting-rod bearing fractured through 13 locking splines.

These figures were included because of an interesting effect that is apparent. The discrepancy noted should certainly have caused an increase in metal content. It will be noted that the last oil analysis on the engine indicated that all of the metal contents were proper. It is also quite apparent that, at the oil analysis performed at 1,060 hours from overhaul, all of the metals except silver and tin showed a significant increase over their previous values and that iron did exceed the threshold value by a considerable amount. This would leave one to suspect that the failure actually occurred at this point and was not detected until inspection. The fact that the various metal contents decreased after this time would indicate that it is possible to miss a period of high metal contamination if oil analyses are not performed sufficiently often.

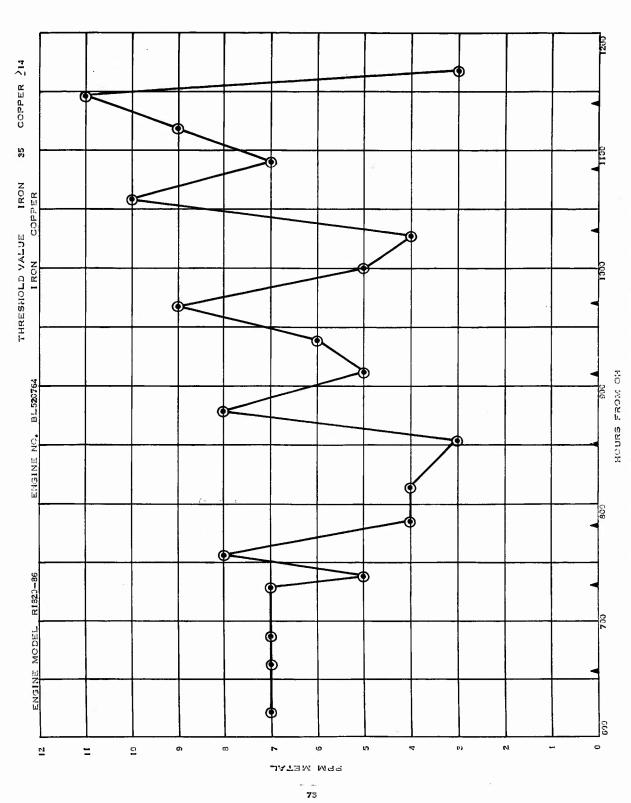


FIGURE 31. CONTAMINATION LEVEL VS HOURS FROM OVERHAUL

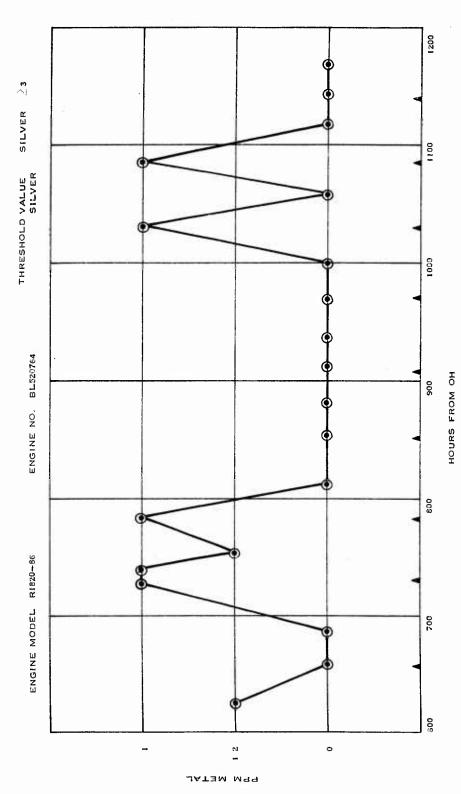
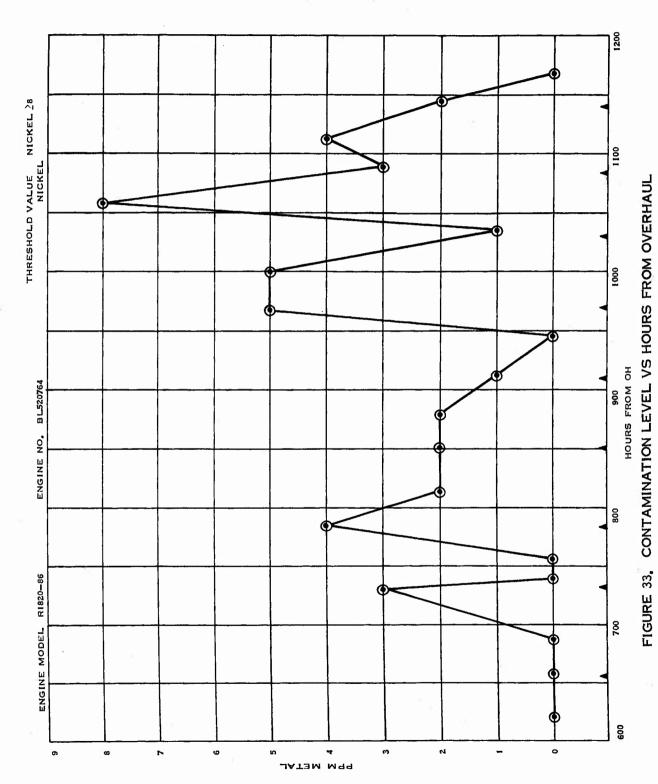
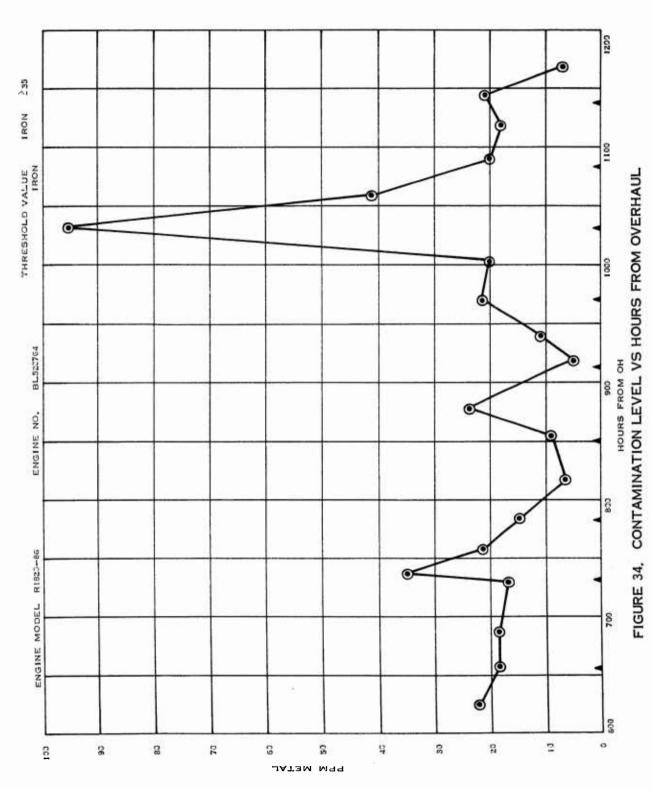
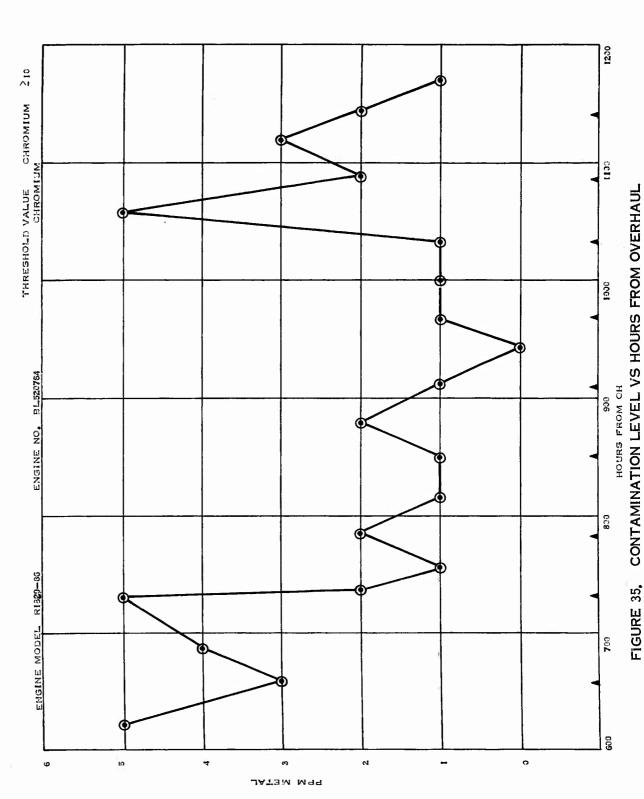


FIGURE 32, CONTAMINATION LEVEL VS HOURS FROM OVERHAUL







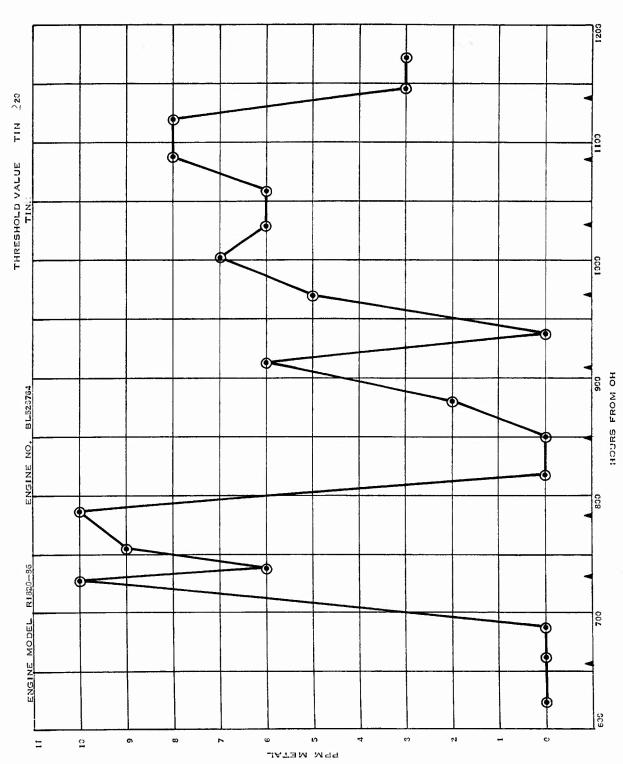
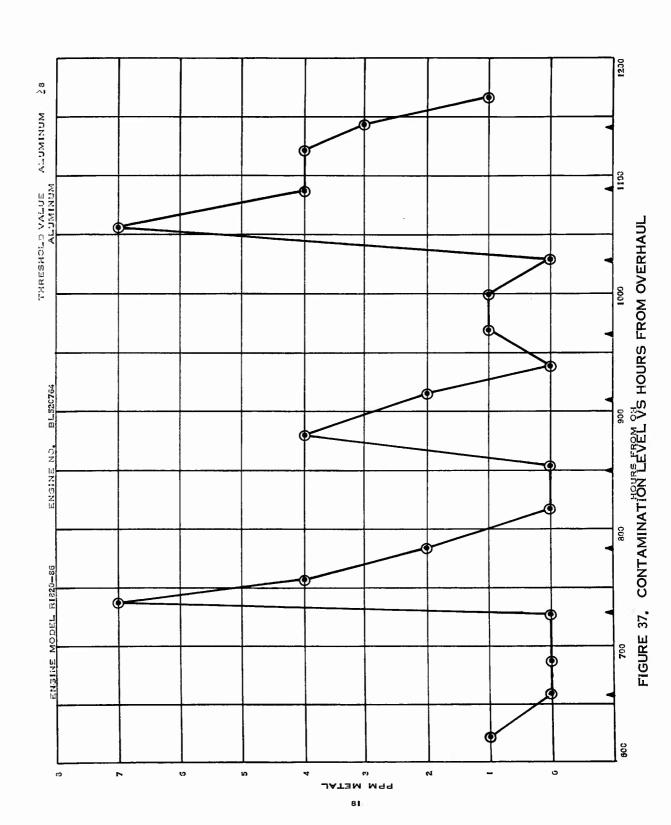


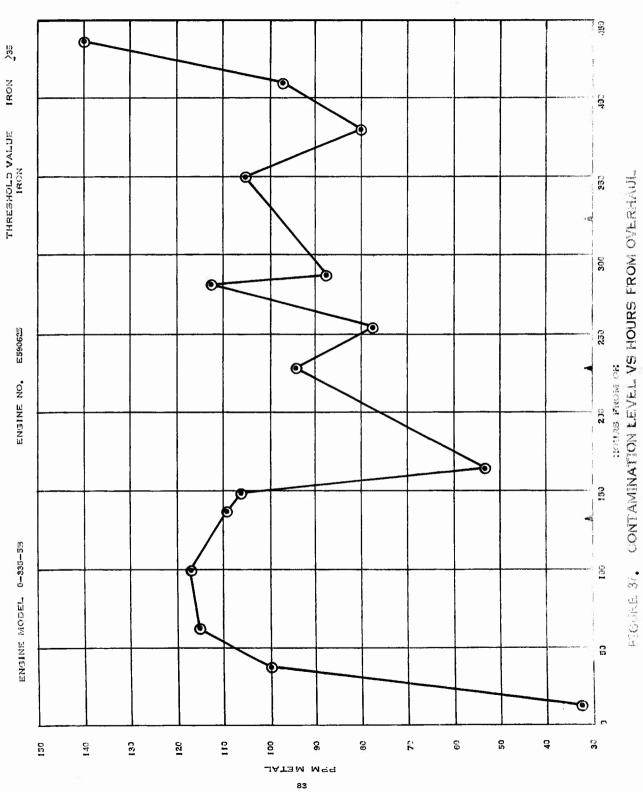
FIGURE 36, CONTAMINATION LEVEL VS HOURS FROM OVERHAUL

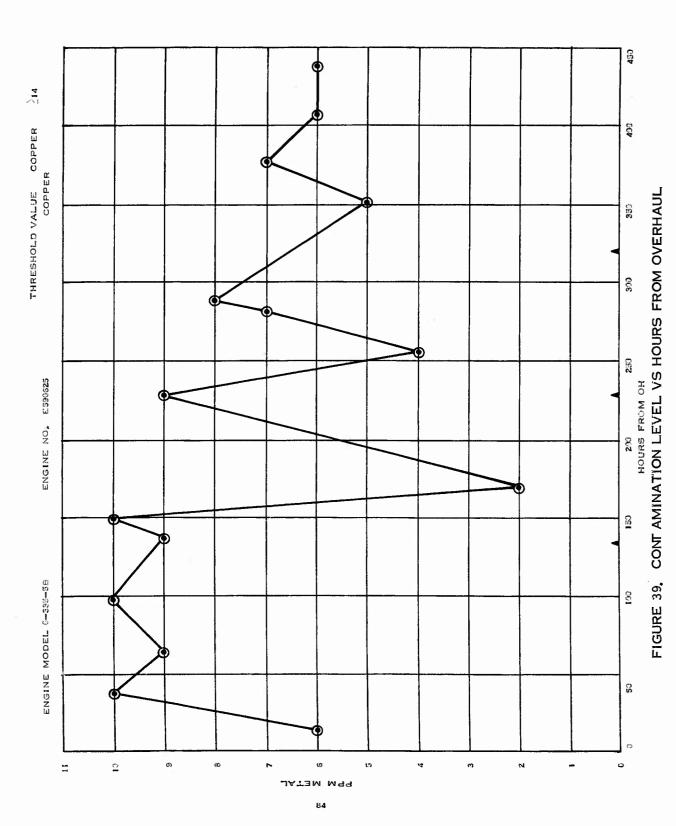


Figures 38 through 44 are graphs showing metal content in Engine Model 0-335-5B, Engine No. E590625.

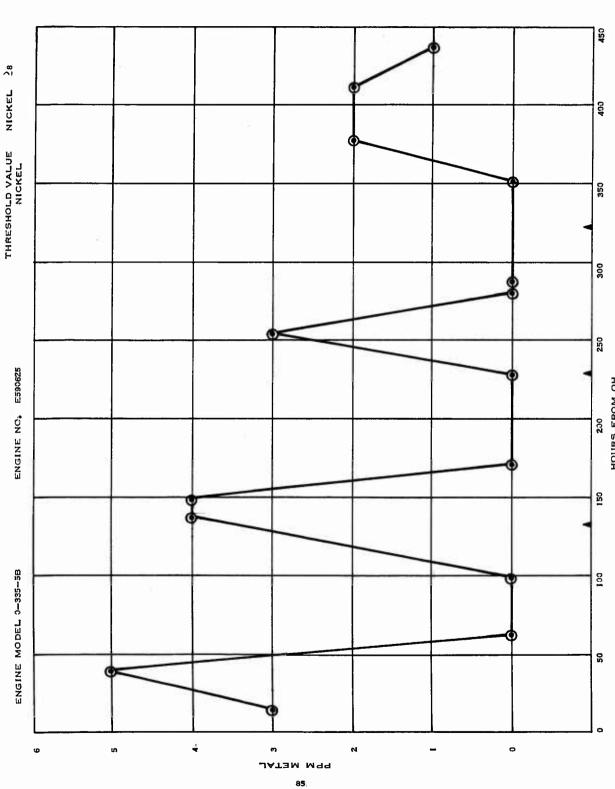
The comments taken from the oil analysis records are as follows: Excess oil leak. DOP November 7, 1960. No discrepancies.

These graphs are included to show that all data do not correlate particularly well. The fact that no discrepancies were discovered does not, of course, mean that no discrepancies existed. It will be noted that iron and aluminum did exceed the threshold values on at least one occasion of oil analysis.





## FIGURE 40, CONT AMINATION LEVEL VS HOURS FROM OVERHAUL



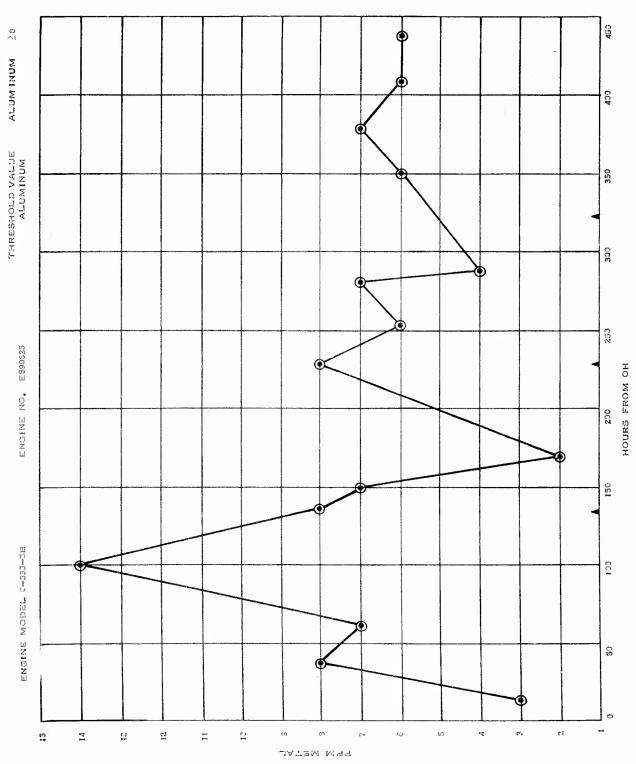
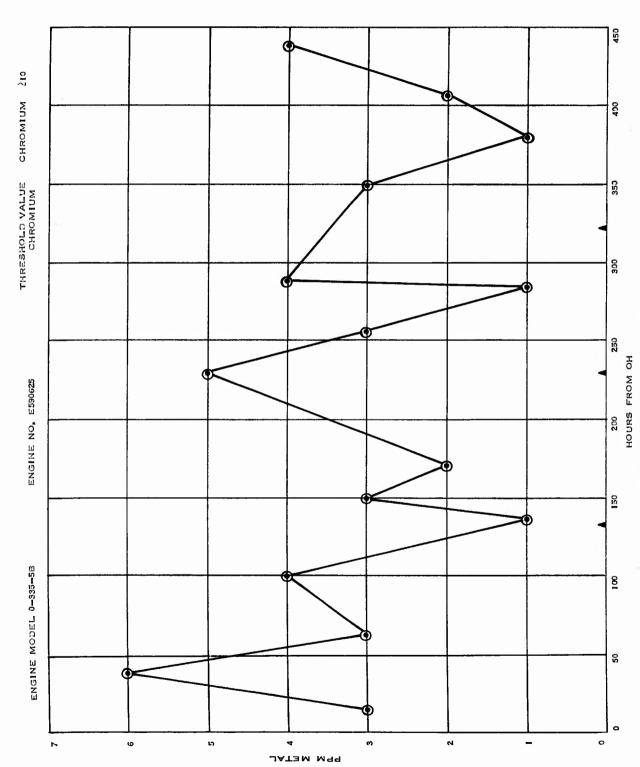


FIGURE 41, CONT AMINATION LEVEL VS HOURS FROM OVERHAUL



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FIGURE 42, CONTAMINATION LEVEL VS HOURS FROM OVERHAUL

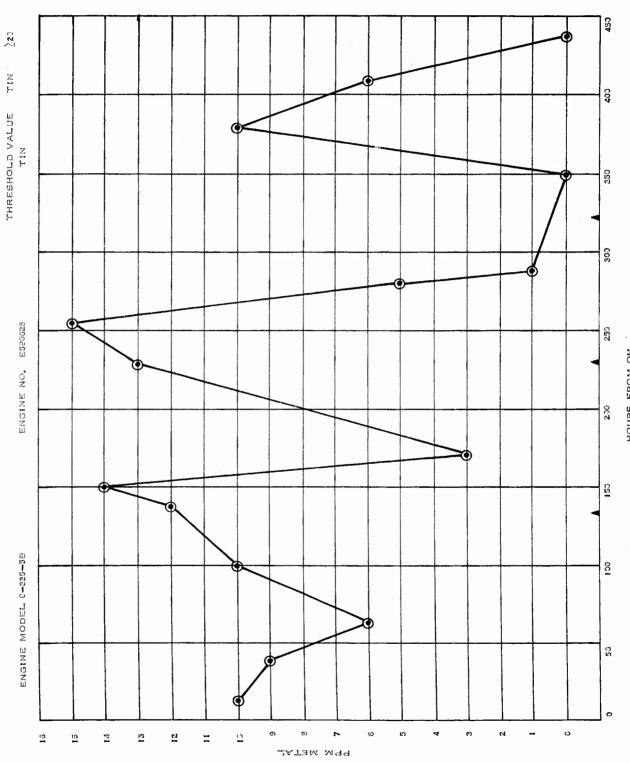


FIGURE 43, CONTAMINATION LEVEL VS HOURS FROM OVERHAUL

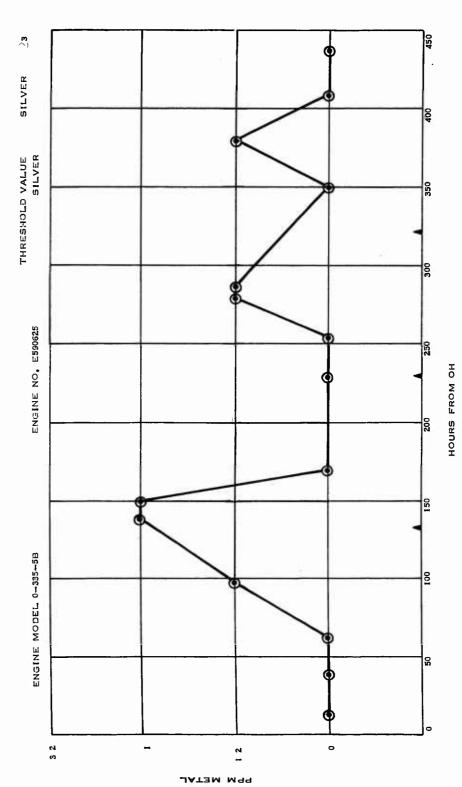
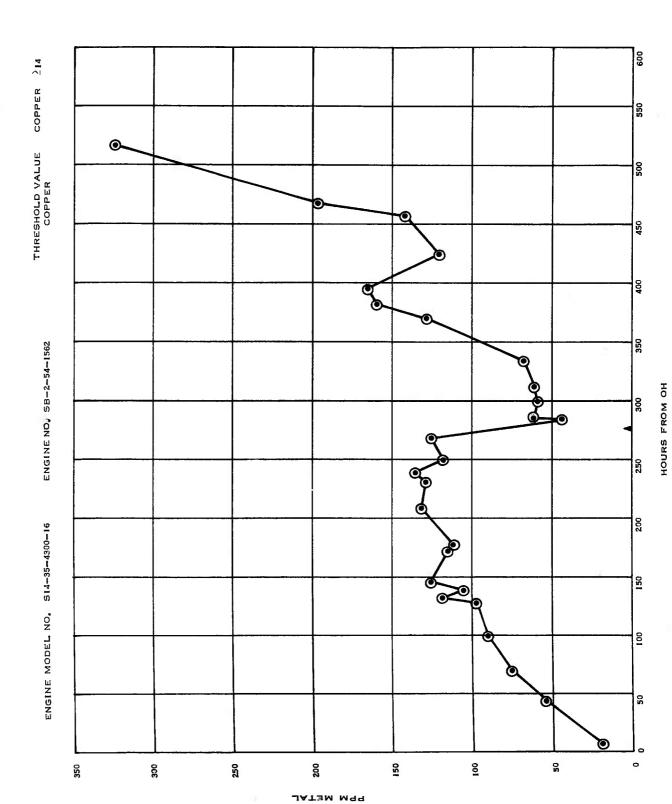


FIGURE 44, CONTAMINATION LEVEL VS HOURS FROM OVERHAUL

Figures 45 through 52 are graphs showing metal content in Engine Model No. S14-35-4300-16, Engine No. SB-2-54-1562.

The comment taken from the oil analysis records is as follows: DOP.

These figures are included merely to show that the pattern of metal content is considerably different for various types of engines. The nickel, chromium, and tin content in this engine are particularly different from the R-1820.



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FIGURE 45, CONTAMINATION LEVEL VS HOURS FROM OVERHAUL

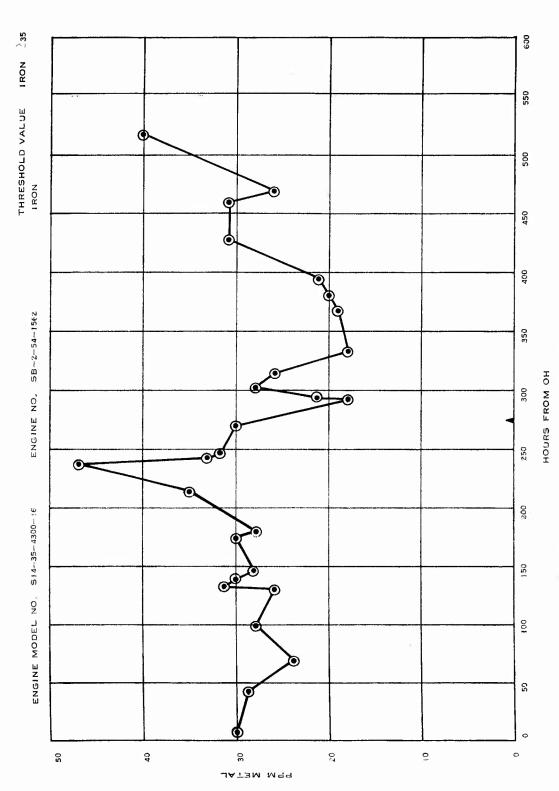


FIGURE 46. CONTAMINATION LEVEL VS HOURS FROM OVERHAUL

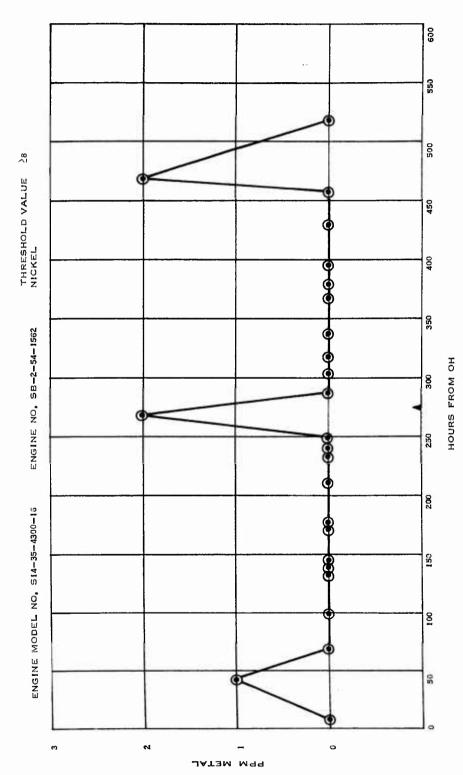


FIGURE 47. CONTAMINATION LEVEL VS HOURS FROM OVERHAUL

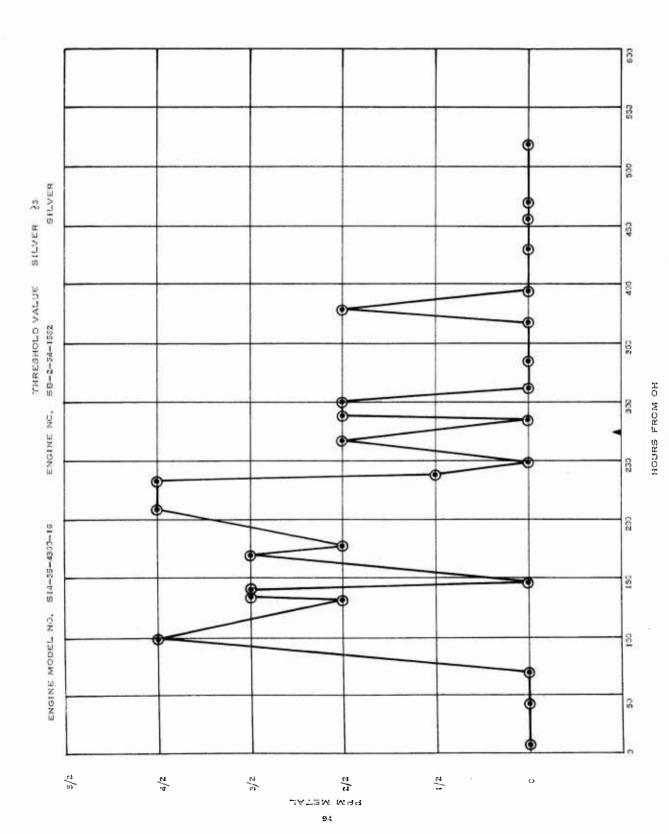
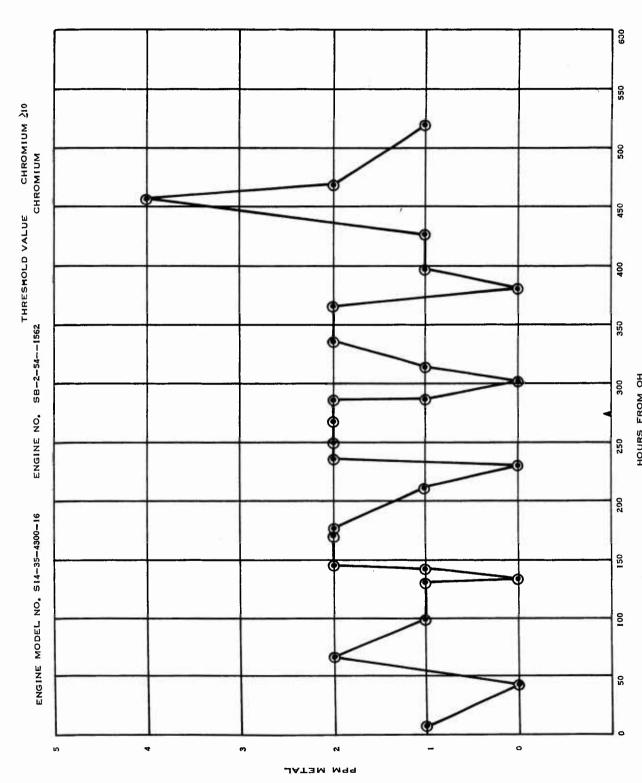


FIGURE 48, CONTAMINATION LEVEL VS HOURS FROM OVERHAUL



HOURS FROM OH FIGURE 49, CONTAMINATION LEVEL VS HOURS FROM OVERHAUL

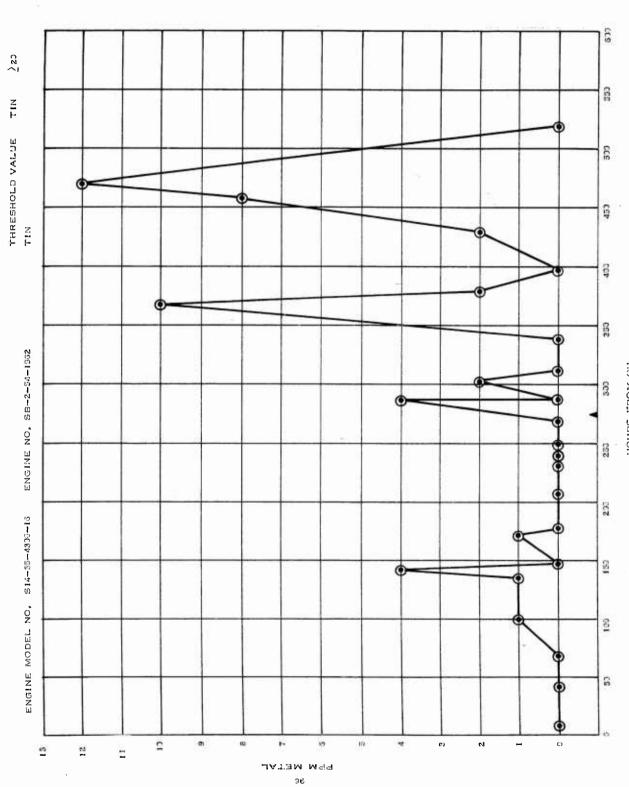
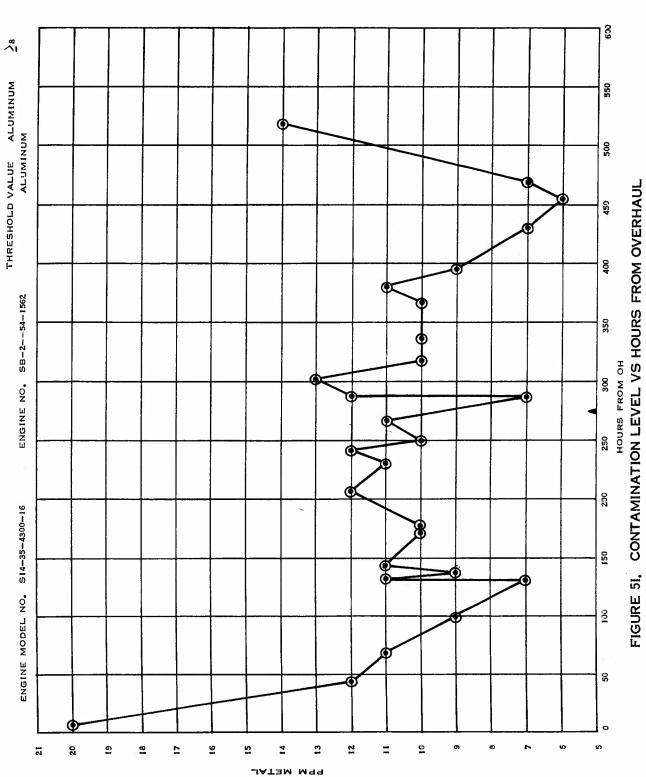
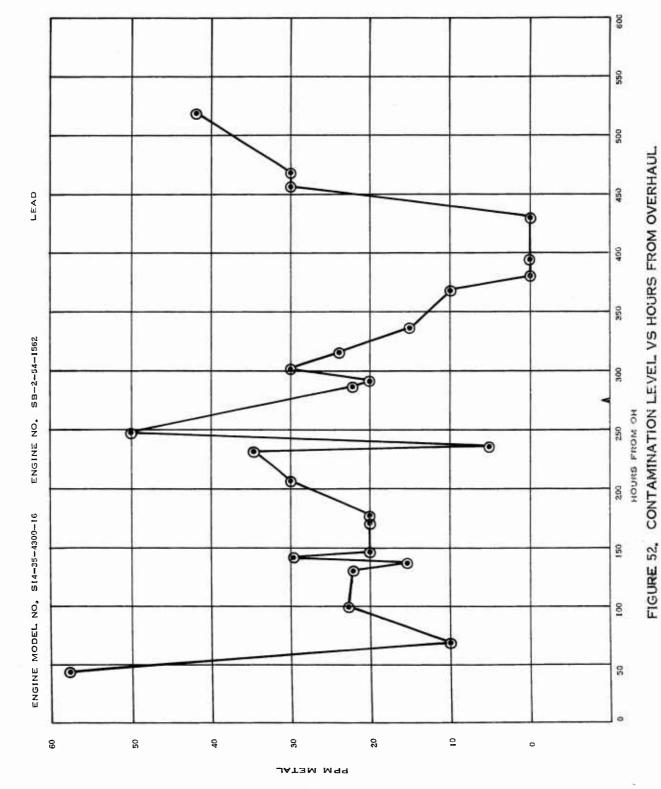


FIGURE 50, CONTAMINATION LEVEL VS HOURS FROM OVERHAUL





Figures 53 through 62 are graphs showing metal content in Conway Engine No. 6014.

The comment taken from the oil analysis records is as follows: Engine removed, due time expired.

Figures 53 through 72 are included to show typical metal content patterns for one type of turbine engine. It will be noted that most of the metal content values are much lower for the jet (turbine) engine than for piston engines and that many of the metals are present in levels that are sufficiently low that it would be suspected that these may be impurities introduced by new oil. Also, a number of these materials are not present except as impurities in the oil-wetted portion of the engine. It would be expected, then, that probably iron would be one of the better indicators of discrepancy.

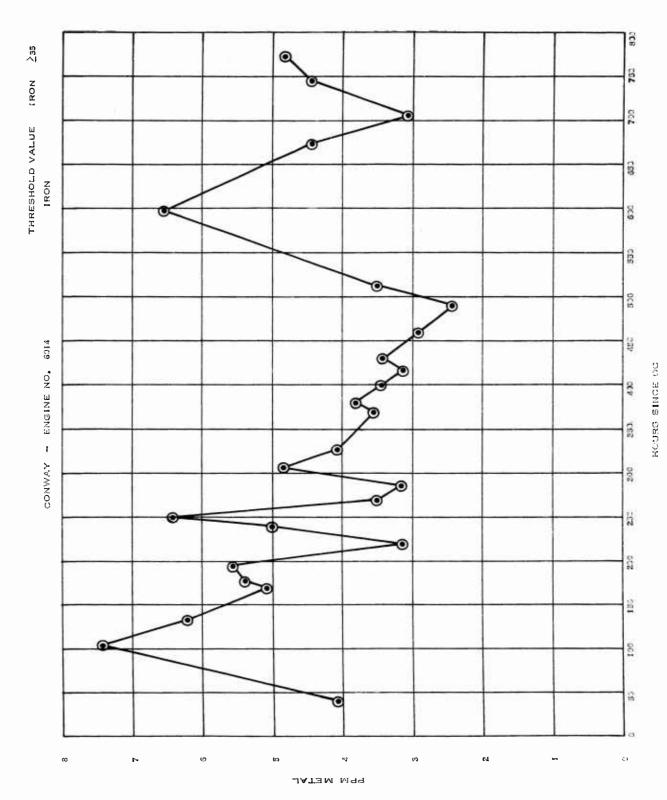


FIGURE 53, CONTAMINATION LEVEL VS HOURS SINCE OIL CHANGE

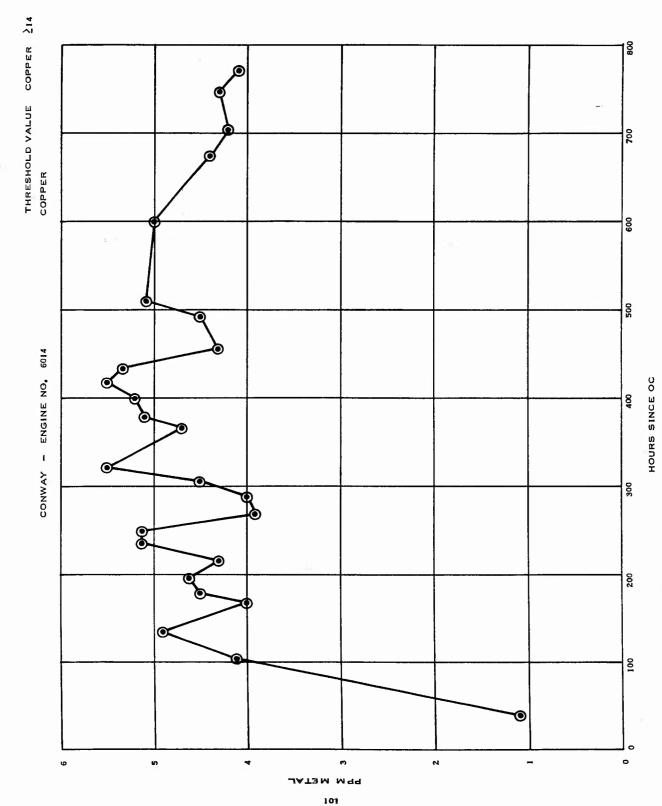


FIGURE 54, CONTAMINATION LEVEL VS HOURS SINCE OIL CHANGE

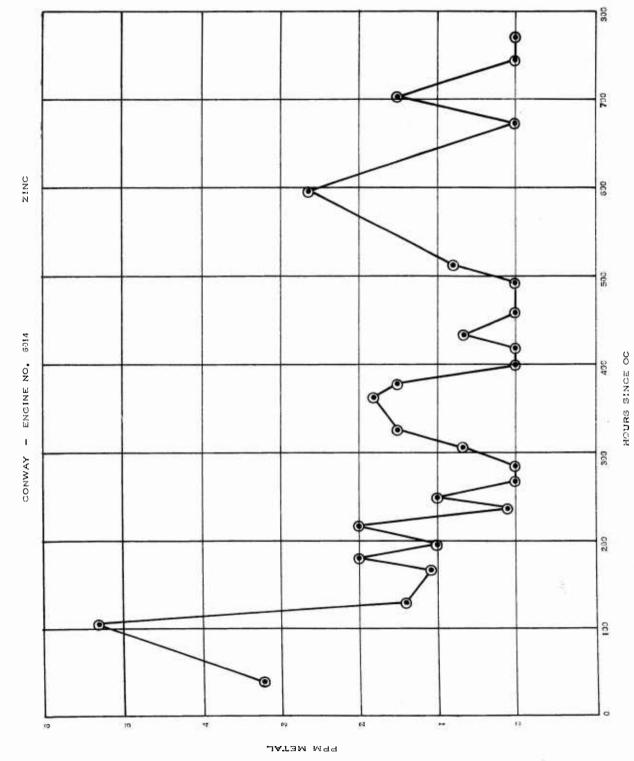
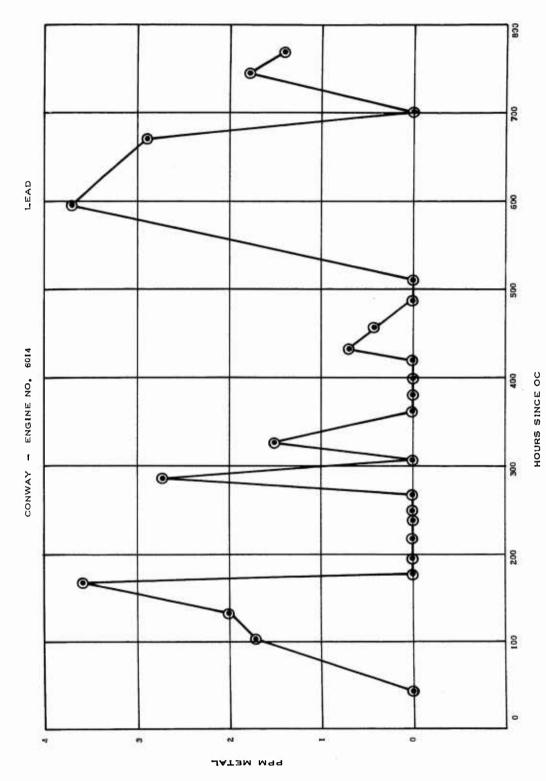
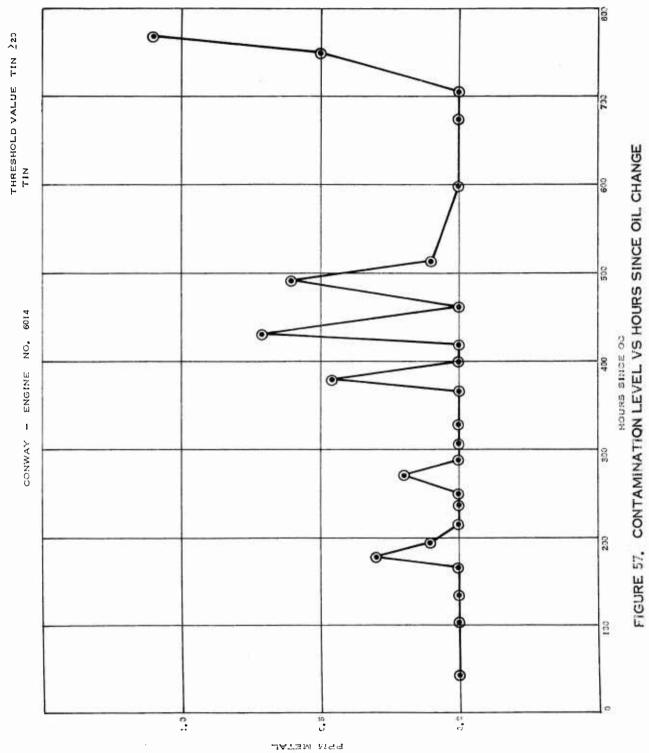


FIGURE 55. CONTAMINATION LEVEL VS HOURS SINCE OIL CHANGE





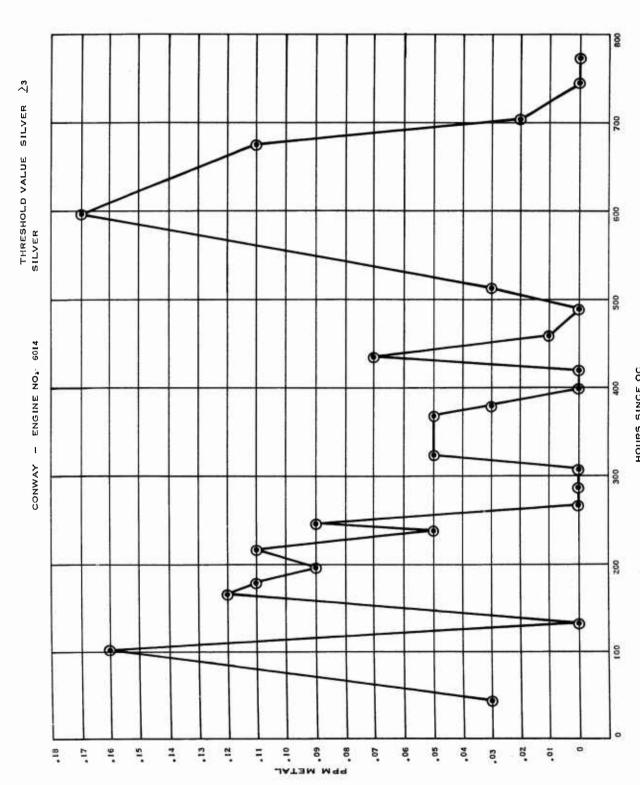


FIGURE 58, CONTAMINATION LEVEL VS HOURS SINCE OIL CHANGE

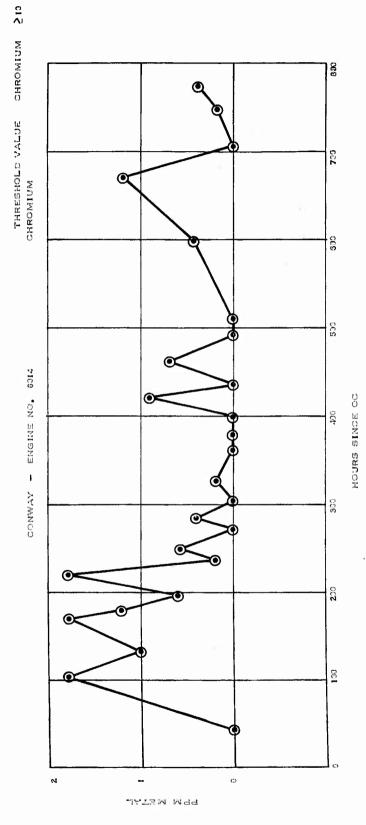
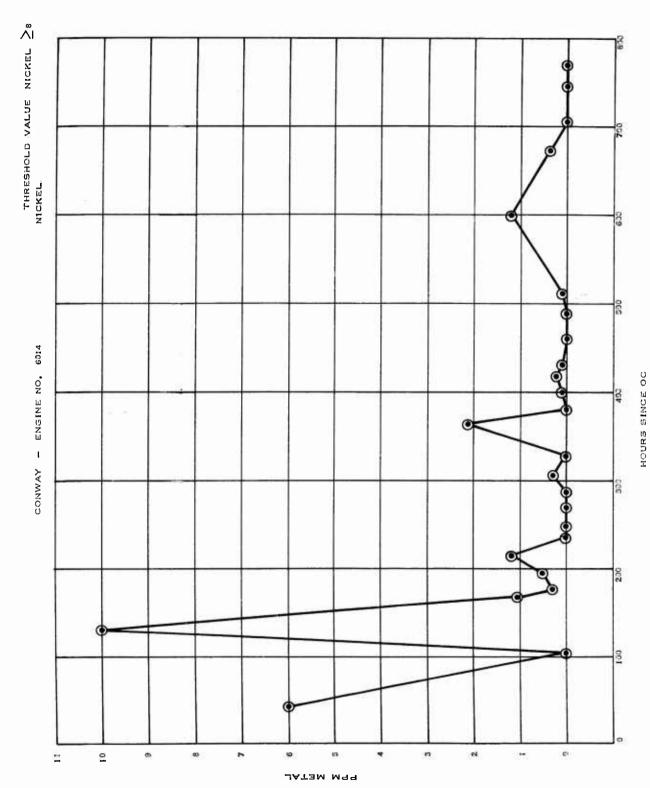


FIGURE 59, CONTAMINATION LEVEL VS HOURS FROM OIL CHANGE



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FIGURE 60. CONTAMINATION LEVEL VS HOURS SINCE OIL CHANGE

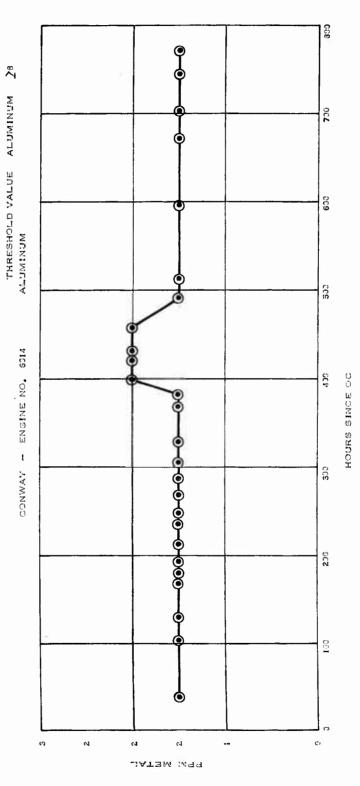
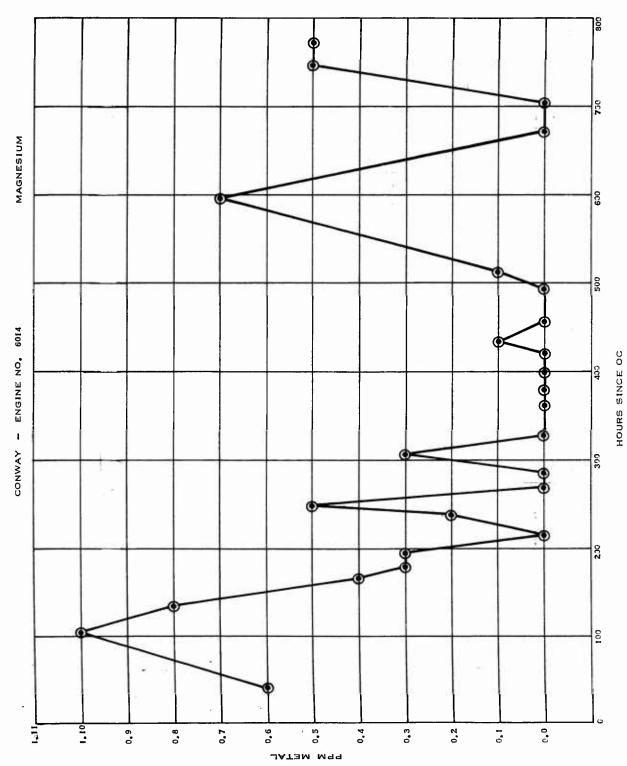


FIGURE 61, CONTAMINATION LEVEL VS HOURS SINCE OIL CHANGE



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FIGURE 62, CONTAMINATION LEVEL VS HOURS SINCE OIL CHANGE

Figures 63 through 72 are graphs showing metal content in Conway Engine No. 6022.

The comment taken from the oil analysis records is as follows: No discrepancies.

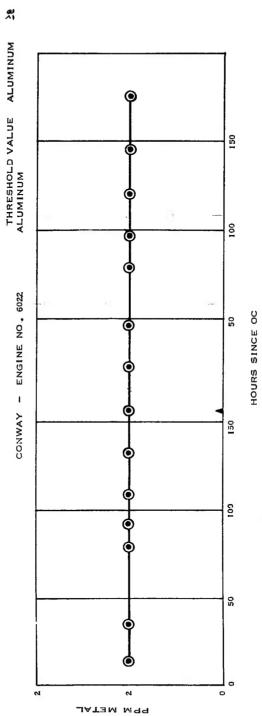


FIGURE 63, CONTAMINATION LEVEL VS HOURS SINCE OIL CHANGE

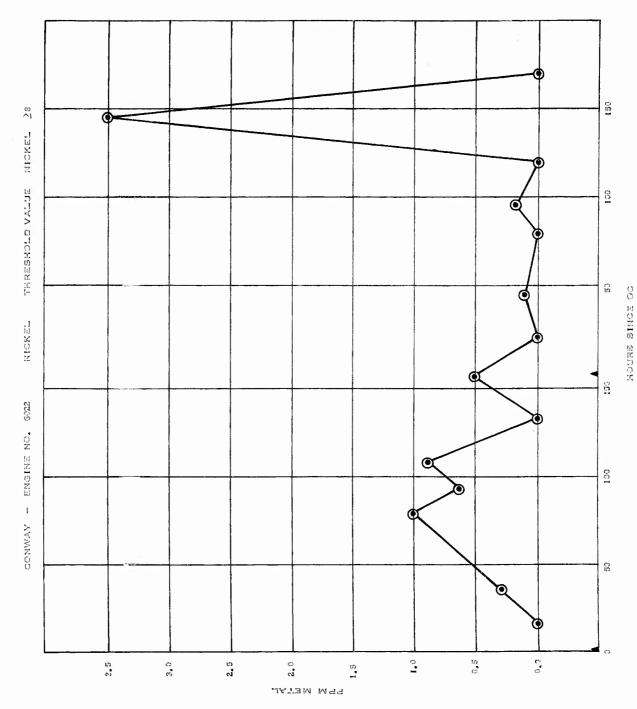


FIGURE 64. CONTAMINATION LEVEL VS HOURS SINCE OIL CHANGE

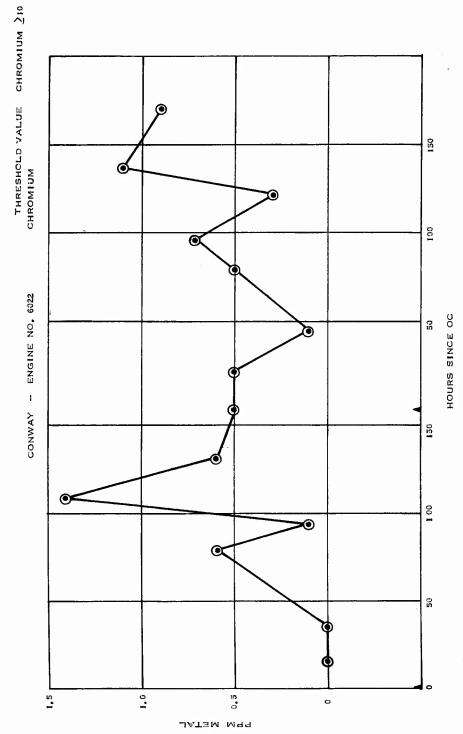


FIGURE 65. CONTAMINATION LEVEL VS HOURS SINCE OIL CHANGE

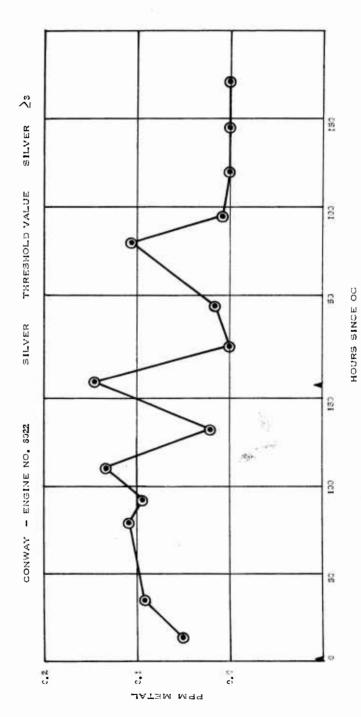


FIGURE 66, CONTAMINATION LEVEL VS HOURS SINCE OIL CHANGE

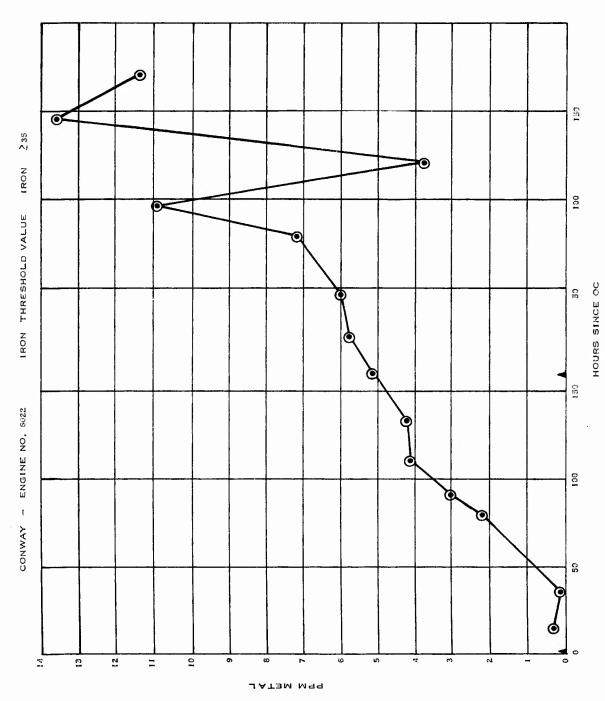


FIGURE 67. CONTAMINATION LEVEL VS HOURS SINCE OIL CHANGE

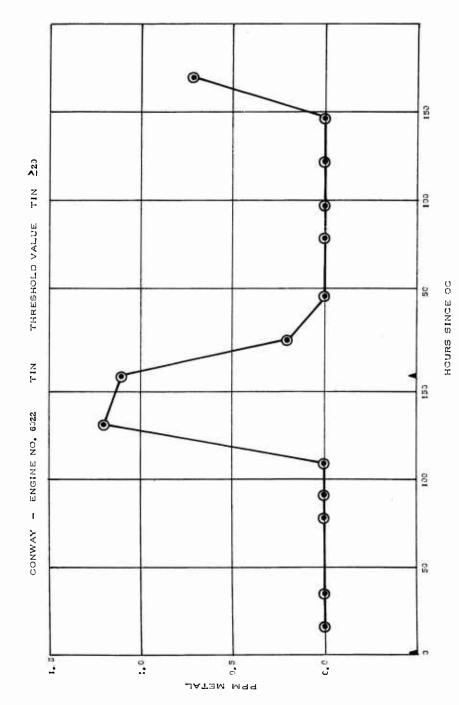


FIGURE 68, CONTAMINATION LEVEL VS HOURS SINCE OIL CHANGE

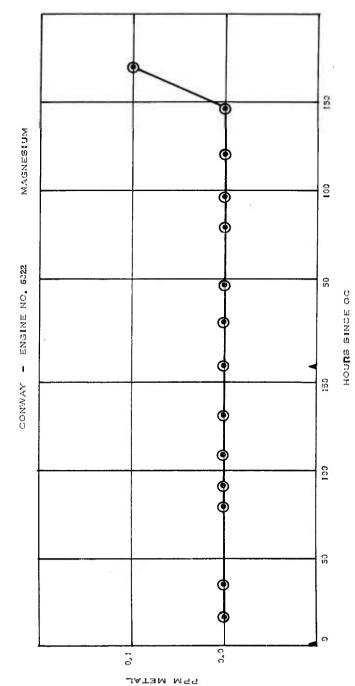
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<u>></u>14

FIGURE 69, CONTAMINATION LEVEL VS HOURS SINCE OIL CHANGE

HOURS SINCE OC

# FIGURE 70, CONTAMINATION LEVEL VS HOURS SINCE OIL CHANGE



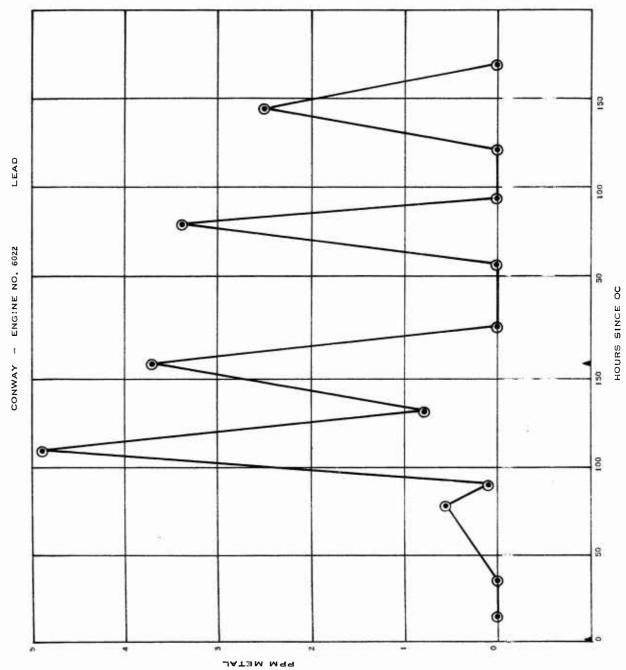


FIGURE 71. CONTAMINATION LEVEL VS HOURS SINCE OIL CHANGE

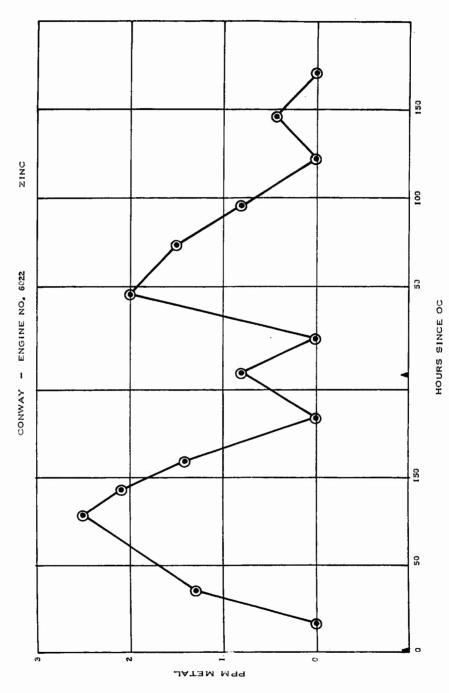


FIGURE 72. CONTAMINATION LEVEL VS HOURS SINCE OIL CHANGE

### BIBLIOGRAPHY

- 1. Cattaneo, A. G., and Starlsman, E. S., <u>Level and Lubrica-tion Factors in Piston Ring and Cylinder Wear</u>, <u>Technical Memorandum</u>, undated and unnumbered, Shell Development Co., Emeryville, California.
- 2. Churchill, H. V., and Churchill, J. R., "Evaluation of Spectrographic Analytical Data," <u>Industrial and Engineering Chemistry</u>, Vol. 17, December 15, 1945, pp. 751-754.
- 3. Dyment, J. T., and Eden, J. J., Trans Canada's Experience With the Rolls-Royce "Conway" Turbine Engine, Society of Automotive Engineers, 1961 SAE Aeronautic Meeting, N. Y., N. Y., paper No. 345C.
- 4. Menzies, A. C., "A Study of Atomic Absorption Spectroscopy," Analytical Chemistry, Volume 32, No. 8, July, 1960, pp. 898-904.
- 5. Sawyer, R., "Absorption Spectrophotometry," <u>Instruments</u> and Control Systems, Volume 34, No. 11, November, 1961, pp. 2049-2053.
- 6. Staff Report, "Gas Chromatography Splits Metal Chelates," Chemical and Engineering News, Volume 40, No. 14, April, 1962, p. 50.
- 7. Terman, F. E., Radio Engineers' Handbook, First Edition, McGraw-Hill Book Co., New York, N. Y., 1943, pp. 53-54.
- 8. Tischler, A. O., <u>Determination of Iron Contamination of Used Lubricating Oil for Use in Measuring Rates of Wear in Aircraft Engines</u>, National Advisory Committee for Aeronautics, Washington, D. C., Report No. RB4C25, March, 1944.
- 9. Willis, J. B., "Determination of Lead and Other Heavy Metals in Urine by Atomic Absorption Spectroscopy,"

  Analytical Chemistry, Volume 34, No. 6, May, 1962, pp. 614-617.
- 10. Wolf, F. L., <u>Elements of Probability and Statistics</u>, McGraw-Hill Book Co., New York, N. Y., 1962, p. 314.

### APPENDIX

To aid the reader in the interpretation of the statistical tests performed on the data, a brief discussion of the rationale behind the tests is given below.

From a particular population we select "N" individuals at random. Each individual may be classified as having a characteristic  $A_1$  or a characteristic  $A_2$  when the characteristic  $A_2$  is simply that of not  $A_1$ . Also each individual may be classified as having characteristic  $B_1$  or  $B_2$ . Our interest is in whether or not there is a relationship between the two classification characteristics. Classifying each member of our sample of size N, we may construct the following table:

	A	As		
B <sub>1</sub>	N <sub>11</sub>	Naı	N .1	
B <sub>2</sub>	N <sub>ls</sub>	Nss	N.2	
	N <sub>1</sub> .	N <sub>s</sub> .	N.	= N

 $N_{11}$  is the number of individuals having both characteristic  $A_1$  and characteristic  $B_1$ ;  $N_{21}$  for characteristics  $A_2$  and  $B_1$  etc., the marginal totals are given by  $N_{.1}$ ,  $N_{.2}$ ,  $N_{1.}$ , and  $N_{2.}$ . (The dot indicates summation over that subscript--that is,  $N_{1.}=N_{11}+N_{12}$ .)

If the two classification schemes are indeed independent of each other, we would expect the proportion  $\frac{N_{11}}{N_{1.}}$  to be about the same proportion as the proportion  $\frac{N_{11}}{N}$  -- that is,

$$N_{11} \simeq \frac{N_{11}N_{11}}{N}$$

and similarly for the other three Nij (i = 1 or 2, j = 1 or 2). Due to sampling variability, it is not reasonable to expect an exact equality for any given sample; but an excessive difference

would lead us to suspect an interrelationship between the two classifications:

Let 
$$Mij = \frac{N.j.Ni.}{N}$$

We call Mij the expected value of Nij. A single number (statistic) which provides a measure of the differences between Nij and Mij in all four cells is

$$X^{2} = \sum_{i,j=1}^{2} \frac{(Nij-Mij)^{2}}{Mij}$$

Other measures are, of course, possible, but we shoose  $X^2$  because, given that the classifications are independent and that the sample is random, we are able to determine mathematically the values of  $X^2$  which are excessive and which are not--that is, we can find the statistical distribution of the random variable  $X^2$ . The exact form of this distribution is difficult to compute, but a good approximation is given by the well-known chi-square distribution (with one degree of freedom in our case of a 2 by 2 classification table). Tables of the chi-square distribution may be found in almost every textbook on applied statistics and in most handbooks of mathematical tables (Reference 10, page 121).

To make the test, we first put the data in a table as shown above and then compute the value of  $X^2$ . To improve the chisquare approximation, it is desirable to alter the formula for  $X^2$  to

$$X^{2} = \sum_{i,j=1}^{2} \frac{\left( \left| \text{Nij-Mij} \right| - \frac{1}{2} \right)^{2}}{\text{Mij}}$$

This alteration helps correct for the fact that the exact distribution of  $X^2$  is discrete, whereas the chi-square distribution is continuous and is referred to as a continuity correction. We now enter a chi-square table and read off the significance level  $\alpha$  of the value of  $X^2$ . (Many tables of chi-square give 1- $\alpha$  rather than  $\alpha$ , but by remembering that  $\alpha$  varies inversely with respect to  $X^2$ , one can determine the proper value of  $\alpha$ .) The interpretation of  $\alpha$  is as follows: Assume we have obtained 2.71 as the value of  $X^2$ . A chi-square table gives us  $\alpha = 0.10$ . This means that if the two classifications are indeed independent, then there is a probability of 0.10 of obtaining a value of  $X^2$  of 2.71 or larger. Therefore, either an event with a probability of 0.10 has occurred or the classifications are not independent. The decision as to which of these alternations to accept rests

with the experimenter. Of course, the smaller  $\alpha$  is, the more inclined we are to accept the latter; but the critical value chosen for  $\alpha$  will depend upon the cost of making a mistake, and this is usually quite difficult to determine. As a guide, 0.05 and 0.01 are commonly used critical values of  $\alpha$  -- that is, if  $\alpha \leq$  0.05 (or 0.01), we reject the hypothesis that the classifications are independent; otherwise not; but it must be stressed that it is up to the experimenter (and not the statistician) to decide whether or not the computed value of  $X^2$  is significantly high.

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An approximate analysis of the effect of small metal particles in the magnetic field of an inductor indicates that metal concentrations of the order of 100 parts per million will be detectable.

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Metal content of the lubricating oil for GurtissMetal content of the lubricating oil for Gurtiss Wright Type R1820-86 piston engines is compared to engine failures. A survey of methods for the to engine failures. A survey of methods for the determination of metal content is given. Several potentially practical methods for field eral potentially practical methods for field measurements appear to exist. An "in-flight" metal particles in the magnetic field of an inductor indicates that metal concentrations of measurement of metal content to provide a warning of imminent failure would be very useful. the order of 100 parts per million will be An approximate analysis of the effect detectable. An approximate analysis of the effect of small metal particles in the magnetic field of an inductor indicates that metal concentrations of measurement of metal content to provide a warn-ing of imminent failure would be very useful. the order of 100 parts per million will be

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