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D. W. Golightly and J. L. Weber

Analytical Chemistry Division
Institute for Materials Research
National Bureau of Standards
Washington, D.C. 20234

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STUDIES OF CALIBRATION STANDARDS USED IN THE DEPARTMENT OF DEFENSE EQUIPMENT OIL ANALYSIS PROGRAM

Danold W. Golightly and
Joseph L. Weber, Jr.

Analytical Chemistry Division
Institute for Materials Research
National Bureau of Standards
Washington, D. C. 20234

At the request of the Naval System Air Command, Department of the Navy, studies have been conducted on organo-metallic calibration standards and diluent oil used in the Department of Defense Equipment Oil Analysis Program. Consultation on standards has been provided, and measurements of physical properties of base oil, concentrations of major elements in standards, concentrations of trace contaminants, and stability of solutions have been performed. Results of studies accomplished in fiscal year 1972 are detailed in this report.

Key words: Calibration standards; concentration validity; flash point; lubricating oil; pour point, spectrometric analysis; stability; trace elements; viscosity.

1. INTRODUCTION

The principal objectives of National Bureau of Standards (NBS) studies for the Department of Defense Equipment Oil Analysis Program (DOD EOAP) in fiscal year 1972 were directed toward determinations of physical properties and concentration validities of base oil concentrates. Priority subjects for study were established in a meeting with Navy (DOD EOAP) representatives on February 24, 1972 at Pensacola, Florida. These subjects were:

1. Investigation of the concentration validity of metal concentrates provided by supplier.
2. Characterization of paraffinic hydrocarbon base oil (diluent) in terms of viscosity, flash point, pour point, and trace element content.
3. Investigation of dilution accuracy attainable by Pensacola Navy Air Base Laboratory (PNABL).
4. Characterization of stability of diluted solutions of standards.

The results pertinent to these objectives obtained through the end of fiscal year 1972, and findings on all related studies are detailed and analyzed in this report. Results from the long-term stability study will be supplied at a later date, when the study will have been completed.

2. MEASUREMENTS, RESULTS AND DISCUSSION

A. Concentration Validity of Oil-Base Metal Concentrates

Eleven oil-base concentrates have been analyzed chemically for metal content. The analysis results have been compared with nominal concentrations from the supplier* to ascertain the validity of the metal concentrates.

Liquid oil-base concentrates are received by the Pensacola Navy Air Base Spectrometric Oil Analysis Laboratory (PNABL) as single-element standards that can be blended and diluted to concentrations suited to calibration of optical emission and atomic absorption spectrometers. Each concentrate consists of one metallo-organic compound (an alkyl-aryl sulfonate) in oil**. Representative portions of 12 on-hand concentrates were received by NBS for chemical analysis. Sodium was later deleted from the list of

*Supplier is Continental Oil Company.

**Exact identity of metallo-organics is proprietary information of supplier.

priority elements for DOD EOAP, and replaced by molybdenum. No molybdenum concentrate was received from PNABL in fiscal year 1972.

All analyses, performed by R. Bell of NBS, consisted of classical gravimetric and titrimetric methods. Summaries of these analyses and analytical methods are provided in Tables 1 and 2. (See Appendix A for all tables in this report.)

Results for all eleven elements verify the nominal concentrations and indirectly verify the analytical methodology used by the supplier. Agreement between supplier and NBS values is considered to be attained only in the instances where the mean NBS value falls within the 2 percent relative inaccuracy range set by the supplier on nominal concentrations.

B. Characterization of 245-Type Base Oil

The base oil* has been characterized in terms of physical properties and trace element content. Kinematic viscosity, flash point, and pour point were determined by a cooperating laboratory. Such measurements presently are not being made on a regular basis within the NBS laboratory. Concentrations of 20 key elements were determined by a DC arc-spectrographic technique in the NBS spectrochemistry laboratory.

Results for triplicate determinations of kinematic viscosity, flash point, and pour point are summarized in Table 3. Results from a single DC arc-spectrographic analysis of oxide residue on Ga_2O_3 carrier are presented in Table 4. Further data are forthcoming to supplement the results from spectrographic arc analysis.

*Continental Oil Company, Type 245.

C. Dilution Error and Stability Study*

X-ray fluorescence spectrometry (XRF) affords a means for long-term stability studies and for estimation of possible dilution errors generated in preparation of series of diluted solutions from blended concentrates. The basic response function for calibration, that is, concentration versus relative intensity (counts per second, with a scintillation detector), is linear. Precision for intensity measurements by XRF (for 100 second signal accumulation intervals) was found to be generally better than that demonstrated by spark optical emission spectrometry (for 30 second signal accumulation intervals) for nickel, iron, chromium, and titanium.

Thus, present stability studies (selected samplings from 12 different batches from Pensacola) are being conducted by XRF on Fe, Ni, Cr, and Ti, only. Data will be collected at intervals of 2-3 months to ascertain long-term stability. Sufficient time to complete this study has not elapsed.

Calibration curves for these four mentioned elements are illustrated in Figures 1 through 4. (See Appendix B for all figures in this report.) Solid NBS Standard Reference Material (SRM) alloys have been used to provide reference signals. Thus, for example, the relative intensities of Cr, K-alpha emission from oil-base standards have been ratioed to the relative intensity of Cr, K-alpha emission from NBS SRM 1167, which is a low alloy steel. Because Cr is homogeneously distributed in the SRM and long-term stability of the SRM is certain, this alloy serves as a means to consistently reproduce experimental measurement conditions with minimal uncertainty.

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*Experimental design and preliminary experiments were made possible through the advice and assistance of S. D. Rasberry.

Close observation of the curves in Figures 1 through 4 reveals that the relative intensity ratios do not adhere to strict linearity over the entire concentration range observed. This apparent nonlinearity likely can be attributed to a consistent 10% dilution error made at PNABL for the nominal 300 ppm solution. However, to a first visual approximation, these curves are linear to 50 ppm. This linearity provides a means for estimation of maximum expected errors attributable to dilution. Such a dilution error estimate is possible through first, establishment of a simple linear regression equation for the curve and second, estimation of the deviation of points from the curve along the concentration axis. The equation for the model curve is $C=b_0 + b_1R+E$, where C is concentration in ppm, R is the relative intensity ratio, b_0 is the intercept on the concentration axis, b_1 is the slope of the line, and E is the deviation from the curve. Deviations from five-point (includes zero) curves are illustrated in Table 5. These deviations are presented as relative values in Table 6.

The trend in relative error progresses as one would expect; that is, relative error increases as concentration decreases. Such a trend reflects the uncertainty in measurement of analytical signals as the signal magnitude decreases. The relative deviations in Table 6 should be considered maximal deviations because effects on the slope of the regression curve attributable to normally distributed experimental errors have not been given account in this treatment. However, a conclusion that there are no gross dilution errors is appropriate from these data.

D. Other Observations on Standards

Several series of tests, principally on precision, were performed on the MIL-SPEC spectrometer located at Andrews Air Force Base. Twelve-element standards (designated Batch 5/12/71 by Pensacola Navy Laboratory) at nominal con-

centrations of 0, 3, 10, 30, 50, 100, and 300 ppm, by weight, were used in these studies. Spectrometer standardization was ascertained with 100 ppm 12-element standard (Batch 49, Mar. 31, 72) by Mr. Hare, Sgt. Foust, and Sgt. Speciale of Andrews Air Force Base. Standardization of the instrument was performed immediately before the start of each precision study.

1. MIL-SPEC Requirements on Precision

Analysis of the data collected in the reproducibility study on the MIL-SPEC spectrometer at Andrews Air Force Base (Table 7) indicates a general conformity to latest MIL-SPEC requirements (revised) on maximum deviations of readings. The analysis of data presented in Table 8 represents results from fifteen replications, with no data drops.

2. Analytical Response Functions

Analytical response functions for 12 elements are presented in Figure 5 through 8. In terms of the MIL-SPEC spectrometer, these curves are plots of concentration versus analog readout. Analog readout refers to the ratio of voltages produced on capacitors that accumulate charge during the interval for integration of photocurrent signals.

An ideal curve exhibits linearity over the entire concentration range of interest and, in addition, passes through the 1-1 origin-point on a log-log plot. The curves for several elements (Ni, Pb, Si, Ti, Fe, and likely Sn) approach these linearity and intercept criteria. Change in slope with increasing concentration indicates line broadening and self absorption in the spark source. The net result of decrease in slope is loss of precision with increasing concentration. The slope is loss of precision with increasing concentration. The slopes of these curves correlate well with the data analysis of Table 7.

3. Coefficients of Variation for Organo-Metallic Standard Compared with those for Used-Oil Samples

The percent coefficient of variation provides a base-100 scale for comparison of relative standard deviations. This coefficient is defined as the ratio of standard deviation to the mean, times 100.

Occasionally, the question arises whether the precision of measurement afforded by synthetic standards is significantly different from that incurred with used-oil samples. A limited amount of data (Table 8) addressed to this question was obtained on the MIL-SPEC spectrometer at Andrews Air Force Base. These data, for six elements at similar concentrations, are presented in Table 9.

These limited observations indicate that measurement reproducibility for standards is essentially the same as that for used oil samples from jet and reciprocating engines.

4. Spectrometer Readout for 12-Element Blends versus Readout for 20-Element Blends

Data were collected on the MIL-SPEC spectrometer at Andrews Air Force Base to enable a comparison on spectrometer readouts, at a nominal 100 ppm concentration, for 12-element and 20-element blends. An analysis of these data from five replications is presented in Table 10.

It is of particular interest that the mean values for seven of the 12 elements are not the same for 12-element and 20-element blends (see Table 11). Only Ag, Cr, Cu, Na, and Ni give the same values with a 95% confidence.

Sources for the seven discrepancies rest with one or combinations of three possibilities.

1. Viscosity differences between 12-element and 20-element solutions used in this study,
2. Dilution errors, and
3. Spectral interelement effects.

3. CONCLUSIONS

Nominal concentrations of aluminum, chromium, copper, iron, lead, magnesium, nickel, silicon, silver, tin and titanium in oil-base concentrates were validated by chemical analysis.

Only qualitative descriptions of the effect of variations in viscosity of diluent oil have been reported in the chemical literature. Thus, the quantitative effects on spectrometer response attributable to the observed viscosity differences in this report presently are unknown.

No significant dilution error was observed for 3, 10, 30 and 50 ppm solutions of 12-element standards investigated by x-ray fluorescence.

A study of long-term stability of Fe, Ni, Cr and Ti in oil-base solutions has commenced, but sufficient time has not elapsed for completion of the study.

Limited data indicate that the variance in spectrometer signals from metallo-organic standards is not significantly different from the variance in signals produced from used-oil samples.

4. FUTURE WORK

Studies on the long-term stability of diluted calibration standards, described in section 2C, will continue in fiscal year 1973. However, new investigations will include solutions containing molybdenum in place of sodium.

The significance of kinematic viscosity and flash point of oil-base solutions on accuracy and precision of spectrometric measurements with a rotating disk electrode will be investigated.

Also, error sources in production of calibration standards and analytical methodology that provides quality assurance for dilute oil-base solutions will be subjects for study in fiscal year 1973.

APPENDIX A

TABLES - CONCENTRATIONS, PHYSICAL PROPERTIES, TRACE ELEMENTS, PRECISION STUDIES

Table 1. Concentrations of metals in oil-base concentrates.

Metal Concentrate	Concentrate Lot No.	Nominal Metal Concentration (wt %)[a]	Metal Concentration Determined by NBS (wt %)[b]
Aluminum	11	2.26 ± 0.05	2.29 ± 0.01
Chromium	12	1.91 ± 0.04	1.89 ± 0.01
Copper	11	4.55 ± 0.09	4.61 ± 0.07
Iron	12	2.56 ± 0.05	2.55 ± 0.01
Lead	11	8.18 ± 0.16	8.18 ± 0.03
Magnesium	11	2.07 ± 0.04	2.10 ± 0.01
Nickel	11	3.33 ± 0.07	3.34 ± 0.01
Silicon	11	13.15 ± 0.26	13.11 ± 0.14
Silver	11	5.02 ± 0.10	5.08 ± 0.01
Tin	11	5.89 ± 0.12	5.91 ± 0.02
Titanium	11	6.08 ± 0.12	6.13 ± 0.01

[a] Supplier assures concentrations of metals in concentrates to ±2% relative. Uncertainties shown reflect only this relative 2%.

[b] Values reported are arithmetic means from four replications. Uncertainties are expressed as 95% confidence intervals, and are equal to the product of the t-distribution factor (i.e. the positive number exceeded by 100 $(\frac{\alpha}{2})$ % of the t-distribution with n-1 degrees of freedom) and the estimated standard deviation, divided by the square root of n. The estimated standard deviation is defined as the square root of the sum of the squares of deviations from the mean divided by n-1 degrees of freedom.

Table 2. Analytical methods for NBS chemical analysis of oil-base concentrates.

Element	Method	Details
Aluminum	Gravimetric	8-hydroxyquinoline
Chromium	Potentiometric Titration	Ammonium persulfate-ammonium sulfate
Copper	Gravimetric	Electrodeposition
Iron	Titrimetric	$SnCl_2$ reduction followed by $K_2Cr_2O_7$ titration
Lead	Gravimetric	$PbSO_4$; electrodeposition of soluble lead
Magnesium	Gravimetric	Double-precipitation $Mg_2P_2O_7$
Nickel	Gravimetric	Dimethylglyoxime precipitation
Silicon	Gravimetric	Double dehydration with H_2SO_4
Silver	Gravimetric	AgCl precipitation
Tin	Titrimetric	Test Lead-KIO_3
Titanium	Gravimetric	Cupferron-TiO_2

Table 3. Physical properties of type 245 base oil.

Property Measured	NBS[a]	Supplier[b]
Viscosity at 311 K (100°F)	225 ± 1.8[c]	247
(Centistokes) at 372 K (210°F)	17.5 ± 0.2[c]	19.3
Flash Point (COC)	534 ± 11 K	522 K
Pour Point	258 K[d]	258 K

[a]Measurements made by cooperating laboratory, Penniman and Browne, Inc., Baltimore, Maryland.

[b]Provided by Supplier as typical values.

[c]95% confidence interval, defined in footnote b of Table 1. Triplicate measurements were made.

[d]Arithmetic mean of 3 identical values.

Table 4. Trace metal content[a] in 245-type base oil and comparative data for ASTM #1 oil.

Element	NBS[b]	Supplier	ASTM #1 Oil
Ag	0.01	<0.02	<0.02
Al	1	0.05	<0.02
B	0.05	<0.04	<0.08
Ba	1	<0.08	0.36
Be	0.01	<0.02	<0.02
Cd	0.2	<0.08	<0.08
Cr	0.05	<0.02	<0.02
Cu	0.05	<0.02	<0.09
Fe	1	0.10	0.12
Mg	1	<0.02	0.06
Mn	0.05	<0.08	0.25
Mo	0.01	<0.05	<0.02
Na	0.1	<0.08	<0.08
Ni	0.1	<0.08	0.03
Pb	0.1	0.10	<0.02
Si	1	-	-
Sn	0.05	<0.05	<0.02
Ti	1	<0.02	0.03
V	0.05	<0.08	<0.08
Zn	5	<0.08	<0.02

[a]Micrograms of element per gram of oil.

[b]Actual concentrations are less than or equal to these values.

Table 5. Deviation from linear least-squares curve (in ppm).

Nominal Concentration (ppm)	Element			
	Cr	Fe	Ni	Ti
3	-0.14	+0.09	-0.10	+0.12
10	-0.49	-0.08	+0.10	-0.25
30	+0.33	+0.48	-0.40	+0.62
50	+0.10	+0.30	+0.22	+0.44

Table 6. Relative deviations from linear least-squares curve (%).

Nominal Concentration (ppm)	Element			
	Cr	Fe	Ni	Ti
3	-4.7	+3.0	-3.3	+3.9
10	-4.9	-0.8	+1.0	-2.4
30	+1.1	+1.6	-1.4	+2.1
50	+0.2	+0.6	+0.4	+0.9

Table 7. Analysis of data from reproducibility study (computer printout).

RUN
REPRODUCIBILITY STUDY

MIL SPEC SPECTROMETER, ANDREWS AFB

AG IN D-12 SYNTHETIC STANDARD

STAT ANAL FOR 6 COLUMNS OF 15 NUMBERS

COL:	1	2	3	4
AVERAGE	3.35	10.88	31.32	52.70
MAXIMUM	4.10	11.60	32.40	55.00
MINIMUM	3.20	10.20	29.40	49.60
RANGE	.90	1.40	3.00	5.40
ANALYSIS OF VARIANCE				
STD. DEV.	.23	.38	.98	1.55
95 CONF U LIM	.35	.59	1.51	2.38
95 CONF L LIM	.16	.28	.70	1.11
PCT COEF VAR	6.76	3.53	3.14	2.94
T TEST...95 PCT CONF INTERVAL FOR THE LOT MEAN.				
UPPER LIMIT	3.48	11.10	31.88	53.59
LOWER LIMIT	3.22	10.66	30.76	51.81

COL:	5	6	7	8
AVERAGE	102.80	269.73		
MAXIMUM	113.00	317.00		
MINIMUM	93.00	235.00		
RANGE	20.00	82.00		
ANALYSIS OF VARIANCE				
STD. DEV.	4.95	22.94		
95 CONF U LIM	7.59	35.19		
95 CONF L LIM	3.54	16.44		
PCT COEF VAR	4.81	8.51		
T TEST...95 PCT CONF INTERVAL FOR THE LOT MEAN.				
UPPER LIMIT	105.64	282.89		
LOWER LIMIT	99.96	256.58		

RUN
REPRODUCIBILITY STUDY

MIL SPEC SPECTROMETER, ANDREWS AFB

AL IN D-12 SYNTHETIC STANDARD

STAT ANAL FOR 7 COLUMNS OF 15 NUMBERS

COL:	1	2	3	4
AVERAGE	.09	2.10	9.37	28.2[illegible]
MAXIMUM	1.00	3.90	10.60	30.80
MINIMUM	.00	1.20	8.20	26.30
RANGE	1.00	2.70	2.40	4.50
ANALYSIS OF VARIANCE				
STD. DEV.	.26	.67	.59	1.54
95 CONF U LIM	.40	1.02	.91	2.36
95 CONF L LIM	.19	.48	.43	1.10
PCT COEF VAR	281.93	31.69	6.34	5.45
T TEST...95 PCT CONF INTERVAL FOR THE LOT MEAN.				
UPPER LIMIT	.24	2.48	9.71	29.10
LOWER LIMIT	-.06	1.72	9.03	27.34

COL:	5	6	7	8
AVERAGE	49.68	99.60	313.53	
MAXIMUM	53.40	105.00	322.00	
MINIMUM	45.70	92.00	299.00	
RANGE	7.70	13.00	23.00	
ANALYSIS OF VARIANCE				
STD. DEV.	2.51	4.26	7.14	
95 CONF U LIM	3.86	6.53	10.95	
95 CONF L LIM	1.80	3.05	5.12	
PCT COEF VAR	5.06	4.27	2.28	
T TEST...95 PCT CONF INTERVAL FOR THE LOT MEAN.				
UPPER LIMIT	51.12	102.04	317.63	
LOWER LIMIT	48.24	97.16	309.44	

RUN
REPRODUCIBILITY STUDY

MIL SPEC SPECTROMETER, ANDREWS AFB

CR IN D-12 SYNTHETIC STANDARD

STAT ANAL FOR 6 COLUMNS OF 15 NUMBERS

COL:	1	2	3	4
AVERAGE	2.07	8.89	29.39	50.93
MAXIMUM	3.10	9.40	31.10	54.20
MINIMUM	1.70	8.20	27.50	48.50
RANGE	1.40	1.20	3.60	5.70
ANALYSIS OF VARIANCE				
STD. DEV.	.37	.36	1.07	1.65
95 CONF U LIM	.57	.55	1.64	2.52
95 CONF L LIM	.26	.26	.77	1.18
PCT COEF VAR	17.81	4.01	3.64	3.23
T TEST...95 PCT CONF INTERVAL FOR THE LOT MEAN.				
UPPER LIMIT	2.29	9.09	30.01	51.88
LOWER LIMIT	1.86	8.68	28.78	49.99

COL:	5	6	7	8
AVERAGE	100.00	304.40		
MAXIMUM	103.00	321.00		
MINIMUM	96.00	293.00		
RANGE	7.00	28.00		
ANALYSIS OF VARIANCE				
STD. DEV.	2.75	7.12		
95 CONF U LIM	4.22	10.92		
95 CONF L LIM	1.97	5.10		
PCT COEF VAR	2.75	2.34		
T TEST...95 PCT CONF INTERVAL FOR THE LOT MEAN.				
UPPER LIMIT	101.58	308.48		
LOWER LIMIT	98.42	300.32		

RUN
REPRODUCIBILITY RUN MIL SPEC SPECTROMETER, ANDREWS AFB

CU IN D-12 SYNTHETIC STANDARD

STAT ANAL FOR 7 COLUMNS OF 15 NUMBERS

COL:	1	2	3	4
AVERAGE	.04	3.21	11.26	32.85
MAXIMUM	.10	3.60	11.80	34.50
MINIMUM	.00	3.00	10.40	31.20
RANGE	.10	.60	1.40	3.30
ANALYSIS OF VARIANCE				
STD. DEV.	.05	.16	.39	1.06
95 CONF U LIM	.08	.24	.60	1.63
95 CONF L LIM	.04	.11	.28	.76
PCT COEF VAR	126.77	4.83	3.50	3.24
T TEST...95 PCT CONF INTERVAL FOR THE LOT MEAN.				
UPPER LIMIT	.07	3.30	11.49	33.46
LOWER LIMIT	.01	3.12	11.03	32.24

COL:	5	6	7	8
AVERAGE	53.49	103.53	279.27	
MAXIMUM	56.60	107.00	300.00	
MINIMUM	50.90	99.00	250.00	
RANGE	5.70	8.00	50.00	
ANALYSIS OF VARIANCE				
STD. DEV.	1.48	2.77	15.52	
95 CONF U LIM	2.28	4.26	23.81	
95 CONF L LIM	1.06	1.99	11.12	
PCT COEF VAR	2.78	2.68	5.56	
T TEST...95 PCT CONF INTERVAL FOR THE LOT MEAN.				
UPPER LIMIT	54.34	105.12	288.16	
LOWER LIMIT	52.64	101.94	270.37	

RUN
REPRODUCIBILITY STUDY

MIL SPEC SPECTROMETER, ANDREWS AFB

FE IN D-12 SYNTHETIC STANDARD

STAT ANAL FOR 7 COLUMNS OF 15 NUMBERS

COL:	1	2	3	4
AVERAGE	.27	2.93	9.69	29.81
MAXIMUM	.60	3.40	10.50	32.90
MINIMUM	.00	2.50	8.90	27.90
RANGE	.60	.90	1.60	5.00
ANALYSIS OF VARIANCE				
STD. DEV.	.19	.23	.55	1.46
95 CONF U LIM	.30	.35	.85	2.24
95 CONF L LIM	.14	.16	.40	1.05
PCT COEF VAR	71.14	7.80	5.70	4.90
T TEST...95 PCT CONF INTERVAL FOR THE LOT MEAN.				
UPPER LIMIT	.38	3.06	10.01	30.64
LOWER LIMIT	.16	2.80	9.38	28.97

COL:	5	6	7	8
AVERAGE	51.41	104.87	331.73	
MAXIMUM	56.40	110.00	360.00	
MINIMUM	46.90	96.00	312.00	
RANGE	9.50	14.00	48.00	
ANALYSIS OF VARIANCE				
STD. DEV.	2.45	3.87	12.23	
95 CONF U LIM	3.76	5.94	18.76	
95 CONF L LIM	1.76	2.77	8.76	
PCT COEF VAR	4.77	3.69	3.69	
T TEST...95 PCT CONF INTERVAL FOR THE LOT MEAN.				
UPPER LIMIT	52.82	107.09	338.74	
LOWER LIMIT	50.01	102.65	324.72	

RUN
REPRODUCIBILITY STUDY

MIL SPEC SPECTROMETER, ANDREWS AFB

MG IN D-12 SYNTHETIC STANDARD

STAT ANAL FOR 6 COLUMNS OF 15 NUMBERS

COL:	1	2	3	4
AVERAGE	2.66	10.25	30.80	50.72
MAXIMUM	3.00	11.30	32.90	55.80
MINIMUM	2.40	9.40	28.90	46.50
RANGE	.60	1.90	4.00	9.30
ANALYSIS OF VARIANCE				
STD. DEV.	.15	.59	1.37	3.12
95 CONF U LIM	.23	.91	2.10	4.79
95 CONF L LIM	.11	.43	.98	2.24
PCT COEF VAR	5.65	5.79	4.43	6.16
T TEST...95 PCT CONF INTERVAL FOR THE LOT MEAN.				
UPPER LIMIT	2.75	10.59	31.58	52.51
LOWER LIMIT	2.57	9.91	30.02	48.93

COL:	5	6	7	8
AVERAGE	100.00	295.67		
MAXIMUM	108.00	319.00		
MINIMUM	92.00	265.00		
RANGE	16.00	54.00		
ANALYSIS OF VARIANCE				
STD. DEV.	4.87	15.38		
95 CONF U LIM	7.47	23.60		
95 CONF L LIM	3.49	11.02		
PCT COEF VAR	4.87	5.20		
T TEST...95 PCT CONF INTERVAL FOR THE LOT MEAN.				
UPPER LIMIT	102.79	304.49		
LOWER LIMIT	97.21	286.85		

RUN
REPRODUCIBILITY STUDY

MIL SPEC SPECTROMETER, ANDREWS AFB

NA IN D-12 SYNTHETIC STANDARD

STAT ANAL FOR 7 COLUMNS OF 15 NUMBERS

COL:	1	2	3	4
AVERAGE	.01	4.11	11.60	32.59
MAXIMUM	.10	6.60	12.20	33.60
MINIMUM	.00	3.50	10.90	31.40
RANGE	.10	3.10	1.30	2.20
ANALYSIS OF VARIANCE				
STD. DEV.	.03	.75	.37	.59
95 CONF U LIM	.04	1.14	.57	.90
95 CONF L LIM	.02	.53	.27	.42
PCT COEF VAR	387.30	18.14	3.19	1.80
T TEST...95 PCT CONF INTERVAL FOR THE LOT MEAN.				
UPPER LIMIT	.02	4.54	11.81	32.92
LOWER LIMIT	-.01	3.69	11.39	32.25

COL:	5	6	7	8
AVERAGE	54.69	103.33	296.87	
MAXIMUM	58.50	114.00	329.00	
MINIMUM	51.60	97.00	269.00	
RANGE	6.90	17.00	60.00	
ANALYSIS OF VARIANCE				
STD. DEV.	1.55	5.33	16.78	
95 CONF U LIM	2.38	8.17	25.73	
95 CONF L LIM	1.11	3.82	12.02	
PCT COEF VAR	2.84	5.16	5.65	
T TEST...95 PCT CONF INTERVAL FOR THE LOT MEAN.				
UPPER LIMIT	55.58	106.39	306.48	
LOWER LIMIT	53.80	100.28	287.25	

RUN
REPRODUCIBILITY STUDY

MIL SPEC SPECTROMETER, ANDREWS AFB

NI IN D-12 SYNTHETIC STANDARD

STAT ANAL FOR 6 COLUMNS OF 15 NUMBERS

COL:	1	2	3	4
AVERAGE	2.06	9.15	30.05	52.58
MAXIMUM	2.50	9.70	32.60	55.60
MINIMUM	1.40	8.40	28.10	49.20
RANGE	1.10	1.30	4.50	6.40
ANALYSIS OF VARIANCE				
STD. DEV.	.28	.41	1.34	1.88
95 CONF U LIM	.43	.62	2.05	2.88
95 CONF L LIM	.20	.29	.96	1.34
PCT COEF VAR	13.46	4.43	4.45	3.57
T TEST...95 PCT CONF INTERVAL FOR THE LOT MEAN.				
UPPER LIMIT	2.22	9.39	30.81	53.65
LOWER LIMIT	1.90	8.92	29.28	51.51

COL:	5	6	7	8
AVERAGE	106.27	336.73		
MAXIMUM	111.00	355.00		
MINIMUM	99.00	313.00		
RANGE	12.00	42.00		
ANALYSIS OF VARIANCE				
STD. DEV.	3.51	11.48		
95 CONF U LIM	5.39	17.61		
95 CONF L LIM	2.52	8.23		
PCT COEF VAR	3.31	3.41		
T TEST...95 PCT CONF INTERVAL FOR THE LOT MEAN.				
UPPER LIMIT	108.28	343.31		
LOWER LIMIT	104.25	330.15		

RUN
REPRODUCIBILITY STUDY

MIL SPEC SPECTROMETER, ANDREWS AFB

PB IN D-12 SYNTHETIC STANDARD

STAT ANAL FOR 7 COLUMNS OF 15 NUMBERS

COL:	1	2	3	4
AVERAGE	.28	2.12	9.11	30.79
MAXIMUM	1.00	4.40	10.30	32.50
MINIMUM	.00	.70	6.80	27.80
RANGE	1.00	3.70	3.50	4.70
ANALYSIS OF VARIANCE				
STD. DEV.	.38	.99	.93	1.22
95 CONF U LIM	.58	1.51	1.42	1.87
95 CONF L LIM	.27	.71	.67	.87
PCT COEF VAR	135.79	46.53	10.18	3.96
T TEST...95 PCT CONF INTERVAL FOR THE LOT MEAN.				
UPPER LIMIT	.50	2.69	9.65	31.49
LOWER LIMIT	.06	1.55	8.58	30.09

COL:	5	6	7	8
AVERAGE	53.23	107.87	292.53	
MAXIMUM	56.80	114.00	311.00	
MINIMUM	49.70	103.00	279.00	
RANGE	7.10	11.00	32.00	
ANALYSIS OF VARIANCE				
STD. DEV.	2.21	3.25	9.46	
95 CONF U LIM	3.40	4.98	14.52	
95 CONF L LIM	1.59	2.33	6.78	
PCT COEF VAR	4.16	3.01	3.23	
T TEST...95 PCT CONF INTERVAL FOR THE LOT MEAN.				
UPPER LIMIT	54.50	109.73	297.96	
LOWER LIMIT	51.96	106.00	287.11	

RUN
REPRODUCIBILITY STUDY

MIL SPEC SPECTROMETER, ANDREWS AFB

SI IN D-12 SYNTHETIC STANDARD

STAT ANAL FOR 7 COLUMNS OF 15 NUMBERS

COL:	1	2	3	4
AVERAGE	.03	2.54	9.77	30.29
MAXIMUM	.10	3.40	10.50	33.40
MINIMUM	.00	2.00	8.30	28.20
RANGE	.10	1.40	2.20	5.20
ANALYSIS OF VARIANCE				
STD. DEV.	.05	.37	.65	1.59
95 CONF U LIM	.07	.56	.99	2.44
95 CONF L LIM	.03	.26	.46	1.14
PCT COEF VAR	146.39	14.49	6.64	5.24
T TEST...95 PCT CONF INTERVAL FOR THE LOT MEAN.				
UPPER LIMIT	.06	2.75	10.15	31.20
LOWER LIMIT	.01	2.33	9.40	29.38

COL:	5	6	7	8
AVERAGE	52.05	103.80	304.80	
MAXIMUM	56.40	110.00	324.00	
MINIMUM	48.30	96.00	291.00	
RANGE	8.10	14.00	33.00	
ANALYSIS OF VARIANCE				
STD. DEV.	2.17	3.78	9.47	
95 CONF U LIM	3.34	5.80	14.52	
95 CONF L LIM	1.56	2.71	6.78	
PCT COEF VAR	4.18	3.64	3.11	
T TEST...95 PCT CONF INTERVAL FOR THE LOT MEAN.				
UPPER LIMIT	53.29	105.97	310.23	
LOWER LIMIT	50.80	101.63	299.37	

RUN
REPRODUCIBILITY STUDY

MIL SPEC SPECTROMETER, ANDREWS AFB

SN IN D-12 SYNTHETIC STANDARD

STAT ANAL FOR 6 COLUMNS OF 15 NUMBERS

COL:	1	2	3	4
AVERAGE	.10	5.93	26.24	48.17
MAXIMUM	.80	7.40	28.00	52.10
MINIMUM	.00	4.10	23.70	45.30
RANGE	.80	3.30	4.30	6.80
ANALYSIS OF VARIANCE				
STD. DEV.	.25	1.00	1.37	2.35
95 CONF U LIM	.38	1.53	2.11	3.61
95 CONF L LIM	.18	.71	.98	1.68
PCT COEF VAR	247.85	16.81	5.23	4.88
T TEST...95 PCT CONF INTERVAL FOR THE LOT MEAN.				
UPPER LIMIT	.24	6.50	27.03	49.51
LOWER LIMIT	-.04	5.36	25.45	46.82

COL:	5	6	7	8
AVERAGE	100.33	308.40		
MAXIMUM	105.00	315.00		
MINIMUM	92.00	300.00		
RANGE	13.00	15.00		
ANALYSIS OF VARIANCE				
STD. DEV.	3.94	5.07		
95 CONF U LIM	6.04	7.77		
95 CONF L LIM	2.82	3.63		
PCT COEF VAR	3.93	1.64		
T TEST...95 PCT CONF INTERVAL FOR THE LOT MEAN.				
UPPER LIMIT	102.59	311.31		
LOWER LIMIT	98.07	305.49		

RUN
REPRODUCIBILITY STUDY

MIL SPEC SPECTROMETER, ANDREWS AFB

TI IN D-12 SYNTHETIC STANDARD

STAT ANAL FOR 6 COLUMNS OF 15 NUMBERS

COL:	1	2	3	4
AVERAGE	2.28	9.45	29.49	49.65
MAXIMUM	2.60	10.10	31.70	53.70
MINIMUM	1.60	8.70	27.30	45.20
RANGE	1.00	1.40	4.40	8.50
ANALYSIS OF VARIANCE				
STD. DEV.	.23	.46	1.40	2.53
95 CONF U LIM	.36	.71	2.15	3.88
95 CONF L LIM	.17	.33	1.00	1.81
PCT COEF VAR	10.25	4.88	4.75	5.10
T TEST...95 PCT CONF INTERVAL FOR THE LOT MEAN.				
UPPER LIMIT	2.41	9.71	30.29	51.10
LOWER LIMIT	2.15	9.18	28.68	48.20

COL:	5	6	7	8
AVERAGE	102.33	293.40		
MAXIMUM	107.00	306.00		
MINIMUM	95.00	273.00		
RANGE	12.00	33.00		
ANALYSIS OF VARIANCE				
STD. DEV.	3.96	9.75		
95 CONF U LIM	6.07	14.95		
95 CONF L LIM	2.84	6.98		
PCT COEF VAR	3.87	3.32		
T TEST...95 PCT CONF INTERVAL FOR THE LOT MEAN.				
UPPER LIMIT	104.60	298.99		
LOWER LIMIT	100.06	287.81		

Table 8. Analysis of data from used-oil samples (computer printout).

```
>RUN
REPRODUCIBILITY STUDY  MIL SPEC SPECTROMETER, ANDREWS AFB

USED LUB OIL SAMPLE MIL-L-22851 FROM RECIP ENGINE

COLUMN CODE: 1-FE 2-AG 3-AL 4-CR 5-CU 6-MG 7-NA 8-NI


STAT ANAL FOR   8          COLUMNS OF     15          NUMBERS

COL:            1           2             3           4
----          ----        ----          ----        ----

AVERAGE        32.62         .13          7.76        1.83
MAXIMUM        36.00         .80          9.80        2.40
MINIMUM        30.60         .00          6.10        1.30
RANGE           5.40         .80          3.70        1.10
ANALYSIS OF VARIANCE

STD. DEV.       1.48         .19           .78         .31
95 CONF U LIM   2.27         .29          1.19         .48
95 CONF L LIM   1.06         .13           .56         .22
PCT COEF VAR    4.53      140.79         10.03       17.12
T TEST...95 PCT CONF INTERVAL FOR THE LOT MEAN.

UPPER LIMIT    33.47         .24          8.21        2.01
LOWER LIMIT    31.77         .03          7.31        1.65


COL:            5           6             7           8
----          ----        ----          ----        ----

AVERAGE        13.44         .81          1.91        9.30
MAXIMUM        15.30        1.40          2.50       10.30
MINIMUM        12.70         .70          1.70        8.00
RANGE           2.60         .70           .80        2.30
ANALYSIS OF VARIANCE

STD. DEV.        .59         .18           .21         .55
95 CONF U LIM    .91         .28           .32         .85
95 CONF L LIM    .42         .13           .15         .40
PCT COEF VAR    4.41       22.22         10.98        5.96
T TEST...95 PCT CONF INTERVAL FOR THE LOT MEAN.

UPPER LIMIT    13.78         .92          2.03        9.62
LOWER LIMIT    13.10         .71          1.79        8.98
```

RUN
REPRODUCIBILITY STUDY MIL SPEC SPECTROMETER, ANDREWS AFB

USED LUB OIL SAMPLE MIL-L-22851 FROM RECIP ENGINE

COLUMN CODE: 1-PB 2-SI

STAT ANAL FOR 2 COLUMNS OF 15 NUMBERS

COL:	1	2	3	4
AVERAGE	343.73	3.11		
MAXIMUM	362.00	3.60		
MINIMUM	323.00	2.70		
RANGE	39.00	.90		

ANALYSIS OF VARIANCE

STD. DEV.	11.65	.29		
95 CONF U LIM	17.87	.45		
95 CONF L LIM	8.35	.21		
PCT COEF VAR	3.39	9.47		

T TEST...95 PCT CONF INTERVAL FOR THE LOT MEAN.

UPPER LIMIT	350.41	3.28		
LOWER LIMIT	337.05	2.94		

RUN
REPRODUCIBILITY STUDY MIL SPEC SPECTROMETER, ANDREWS AFB

USED LUB OIL SAMPLE MIL-L-23699 FROM JET ENGINE

COLUMN CODE : 1-FE,2-CU,3-SI,4-SN,5-TI

STAT ANAL FOR 5 COLUMNS OF 15 NUMBERS

COL:	1	2	3	4
AVERAGE	.72	.11	3.25	5.35
MAXIMUM	1.10	.30	4.00	7.00
MINIMUM	.40	.00	2.40	3.60
RANGE	.70	.30	1.60	3.40
ANALYSIS OF VARIANCE				
STD. DEV.	.22	.09	.47	1.03
95 CONF U LIM	.34	.14	.73	1.57
95 CONF L LIM	.16	.06	.34	.73
PCT COEF VAR	31.15	82.85	14.60	19.18
T TEST...95 PCT CONF INTERVAL FOR THE LOT MEAN.				
UPPER LIMIT	.85	.16	3.53	5.93
LOWER LIMIT	.59	.06	2.98	4.76

COL:	5	6	7	8
AVERAGE	.03			
MAXIMUM	.10			
MINIMUM	.00			
RANGE	.10			
ANALYSIS OF VARIANCE				
STD. DEV.	.05			
95 CONF U LIM	.07			
95 CONF L LIM	.03			
PCT COEF VAR	146.39			
T TEST...95 PCT CONF INTERVAL FOR THE LOT MEAN.				
UPPER LIMIT	.06			
LOWER LIMIT	.01			

Table 9. Comparison of coefficients of variation obtained with organo-metallic standards and with used-oil samples.

Element	Concentration (ppm)			Percent Coefficient of Variation	
		Measured			
	Nominal for Standard	Standard	Used Oil	Standard	Used Oil
Cr	3	2.1	1.8	18	17 [a]
Fe	30	29.8	32.6	4.9	4.5[a]
Ni	10	9.2	9.3	4.4	6.0[a]
Pb	300	293	344	3.2	3.4[a]
Si	3	2.5	3.3	15	15 [b]
Sn	10	5.9	5.3	17	19 [b]

[a]Used oil from reciprocating engine test stand, sample MIL-L-22851, Pensacola.

[b]Used oil from jet engine test stand, sample MIL-L-23699, Pensacola.

Table 10. Analysis of data for comparison of spectrometer response for 12-element and 20-element blends (computer printout).

```
>RUN
REPEATABILITY RUN

D-20 SYNTHETIC STANDARD

COLUMN CODE: 1-MN 2-MO 3-V 4-ZN
```

STAT ANAL FOR 4 COLUMNS OF 5 NUMBERS

COL:	1	2	3	4
AVERAGE	80.80	61.00	77.80	23.20
MAXIMUM	82.00	62.00	79.00	25.00
MINIMUM	80.00	60.00	75.00	21.00
RANGE	2.00	2.00	4.00	4.00
ANALYSIS OF VARIANCE				
STD. DEV.	.84	1.00	1.79	1.48
95 CONF U LIM	2.15	2.57	4.59	3.81
95 CONF L LIM	.47	.56	1.00	.83
PCT COEF VAR	1.04	1.64	2.30	6.39
T TEST...95 PCT CONF INTERVAL FOR THE LOT MEAN.				
UPPER LIMIT	81.96	62.39	80.28	25.26
LOWER LIMIT	79.64	59.61	75.32	21.14

```
>RUN
REPEATABILITY RUN

D-20 SYNTHETIC STANDARD

COLUMN CODE: 1-NI 2-PB 3-SI 4-SN 5-TI 6-B 7-BA 8-CD


STAT ANAL FOR   8          COLUMNS OF      5              NUMBERS

COL:            1           2              3              4
----          ----        ----           ----           ----

AVERAGE       101.00      111.00         106.00         107.60
MAXIMUM       102.00      114.00         108.00         110.00
MINIMUM       100.00      109.00         104.00         106.00
RANGE           2.00        5.00           4.00           4.00
ANALYSIS OF VARIANCE

STD. DEV.        .71        1.87           1.58           1.52
95 CONF U LIM   1.82        4.80           4.06           3.89
95 CONF L LIM    .40        1.05            .88            .85
PCT COEF VAR     .70        1.69           1.49           1.41
T TEST...95 PCT CONF INTERVAL FOR THE LOT MEAN.

UPPER LIMIT   101.98      113.60         108.19         109.71
LOWER LIMIT   100.02      108.40         103.81         105.49


COL:            5           6              7              8
----          ----        ----           ----           ----

AVERAGE        91.00       64.80          81.00          50.00
MAXIMUM        93.00       66.00          85.00          51.00
MINIMUM        89.00       62.00          78.00          49.00
RANGE           4.00        4.00           7.00           2.00
ANALYSIS OF VARIANCE

STD. DEV.       1.58        1.64           3.24            .71
95 CONF U LIM   4.06        4.22           8.32           1.82
95 CONF L LIM    .88         .92           1.81            .40
PCT COEF VAR    1.74        2.54           4.00           1.41
T TEST...95 PCT CONF INTERVAL FOR THE LOT MEAN.

UPPER LIMIT    93.19       67.08          85.50          50.98
LOWER LIMIT    88.81       62.52          76.50          49.02
```

```
>RUN
REPEATABILITY RUN

D-20 SYNTHETIC STANDARD

COLUMN CODE: 1-FE 2-AG 3-AL 4-BE 5-CR 6-CU 7-MG 8-NA
```

STAT ANAL FOR 8 COLUMNS OF 5 NUMBERS

COL:	1	2	3	4
AVERAGE	102.80	100.00	102.00	62.60
MAXIMUM	104.00	103.00	103.00	63.00
MINIMUM	101.00	97.00	100.00	61.00
RANGE	3.00	6.00	3.00	2.00
ANALYSIS OF VARIANCE				
STD. DEV.	1.30	2.55	1.22	.89
95 CONF U LIM	3.35	6.54	3.14	2.30
95 CONF L LIM	.73	1.43	.68	.50
PCT COEF VAR	1.27	2.55	1.20	1.43
T TEST...95 PCT CONF INTERVAL FOR THE LOT MEAN.				
UPPER LIMIT	104.61	103.54	103.70	63.84
LOWER LIMIT	100.99	96.46	100.30	61.36

COL:	5	6	7	8
AVERAGE	100.80	98.80	100.40	104.20
MAXIMUM	102.00	100.00	103.00	115.00
MINIMUM	99.00	96.00	97.00	100.00
RANGE	3.00	4.00	6.00	15.00
ANALYSIS OF VARIANCE				
STD. DEV.	1.30	1.79	2.30	6.57
95 CONF U LIM	3.35	4.59	5.91	16.87
95 CONF L LIM	.73	1.00	1.29	3.67
PCT COEF VAR	1.29	1.81	2.29	6.31
T TEST...95 PCT CONF INTERVAL FOR THE LOT MEAN.				
UPPER LIMIT	102.61	101.28	103.60	113.32
LOWER LIMIT	98.99	96.32	97.20	95.08

```
>RUN
REPEATABILITY RUN

D-12 SYNTHETIC STANDARD

COLUMN CODE: 1-FE 2-AG 3-AL 4-CR 5-CU 6-MG 7-NA 8-NI


STAT ANAL FOR   8          COLUMNS OF     5          NUMBERS

COL:            1          2              3          4
----          ----       ----           ----       ----

AVERAGE       100.60      99.00          95.80      99.80
MAXIMUM       105.00     100.00         102.00     104.00
MINIMUM        98.00      95.00          92.00      98.00
RANGE           7.00       5.00          10.00       6.00
ANALYSIS OF VARIANCE

STD. DEV.       3.21       2.24           3.77       2.68
95 CONF U LIM   8.24       5.74           9.67       6.89
95 CONF L LIM   1.79       1.25           2.11       1.50
PCT COEF VAR    3.19       2.26           3.93       2.69
T TEST...95 PCT CONF INTERVAL FOR THE.LOT MEAN.

UPPER LIMIT   105.05      102.10         101.03     103.52
LOWER LIMIT    96.15       95.90          90.57      96.08


COL:            5          6              7          8
----          ----       ----           ----       ----

AVERAGE       100.40      95.80         105.20     100.20
MAXIMUM       104.00     101.00         115.00     105.00
MINIMUM        99.00      93.00         100.00      98.00
RANGE           5.00       8.00          15.00       7.00
ANALYSIS OF VARIANCE

STD. DEV.       2.07       3.11           6.14       3.19
95 CONF U LIM   5.32       7.99          15.76       8.20
95 CONF L LIM   1.16       1.74           3.43       1.79
PCT COEF VAR    2.07       3.25           5.84       3.19
T TEST...95 PCT CONF INTERVAL FOR THE LOT MEAN.

UPPER LIMIT   103.28      100.12         113.72     104.63
LOWER LIMIT    97.52       91.48          96.68      95.77
```

```
>RUN
REPEATABILITY RUN

D-12 SYNTHETIC STANDARD

COLUMN CODE: 1-PB 2-SI 3-SN 4-TI
```

STAT ANAL FOR 4 COLUMNS OF 5 NUMBERS

COL:	1	2	3	4
AVERAGE	107.20	99.20	99.80	96.80
MAXIMUM	110.00	104.00	106.00	102.00
MINIMUM	105.00	96.00	97.00	93.00
RANGE	5.00	8.00	9.00	9.00
ANALYSIS OF VARIANCE				
STD. DEV.	2.28	3.96	3.63	3.63
95 CONF U LIM	5.85	10.17	9.33	9.33
95 CONF L LIM	1.27	2.21	2.03	2.03
PCT COEF VAR	2.13	3.99	3.64	3.75
T TEST...95 PCT CONF INTERVAL FOR THE LOT MEAN.				
UPPER LIMIT	110.37	104.70	104.84	101.84
LOWER LIMIT	104.03	93.70	94.76	91.76

Table 11. Comparison of spectrometer readout for 12 elements in 12-element and 20-element blends with nominal 100 ppm concentrations for all elements.

Element	12-Element Blend		20-Element Blend	
	Mean Concentration ppm	95% Confidence Interval ppm	Mean Concentration ppm	95% Confidence Interval ppm
Ag	99.0	96.6-101.4	100.0	97.3-102.7
Al	95.8	91.8- 99.8	102.0	100.7-103.3
Cr	99.8	96.9-102.7	100.8	99.4-102.2
Cu	100.4	98.2-102.6	98.8	96.9-100.7
Fe	100.6	97.2-104.0	102.8	101.4-104.2
Mg	95.8	92.5- 99.1	100.4	97.9-102.9
Na	105.2	98.7-111.7	104.2	97.2-111.2
Ni	100.2	96.8-103.6	101.0	100.2-101.8
Pb	107.2	104.8-109.6	111.0	109.0-113.0
Si	99.2	95.0-103.4	106.0	104.3-107.7
Sn	99.8	95.9-107.6	106.6	106.0-109.2
Ti	96.8	92.9-100.7	91.0	89.3- 92.7

APPENDIX B

FIGURES - ANALYTICAL RESPONSE FUNCTIONS BY X-RAY FLUORESCENCE (XRF) AND OPTICAL EMISSION SPECTROMETRY (OES)

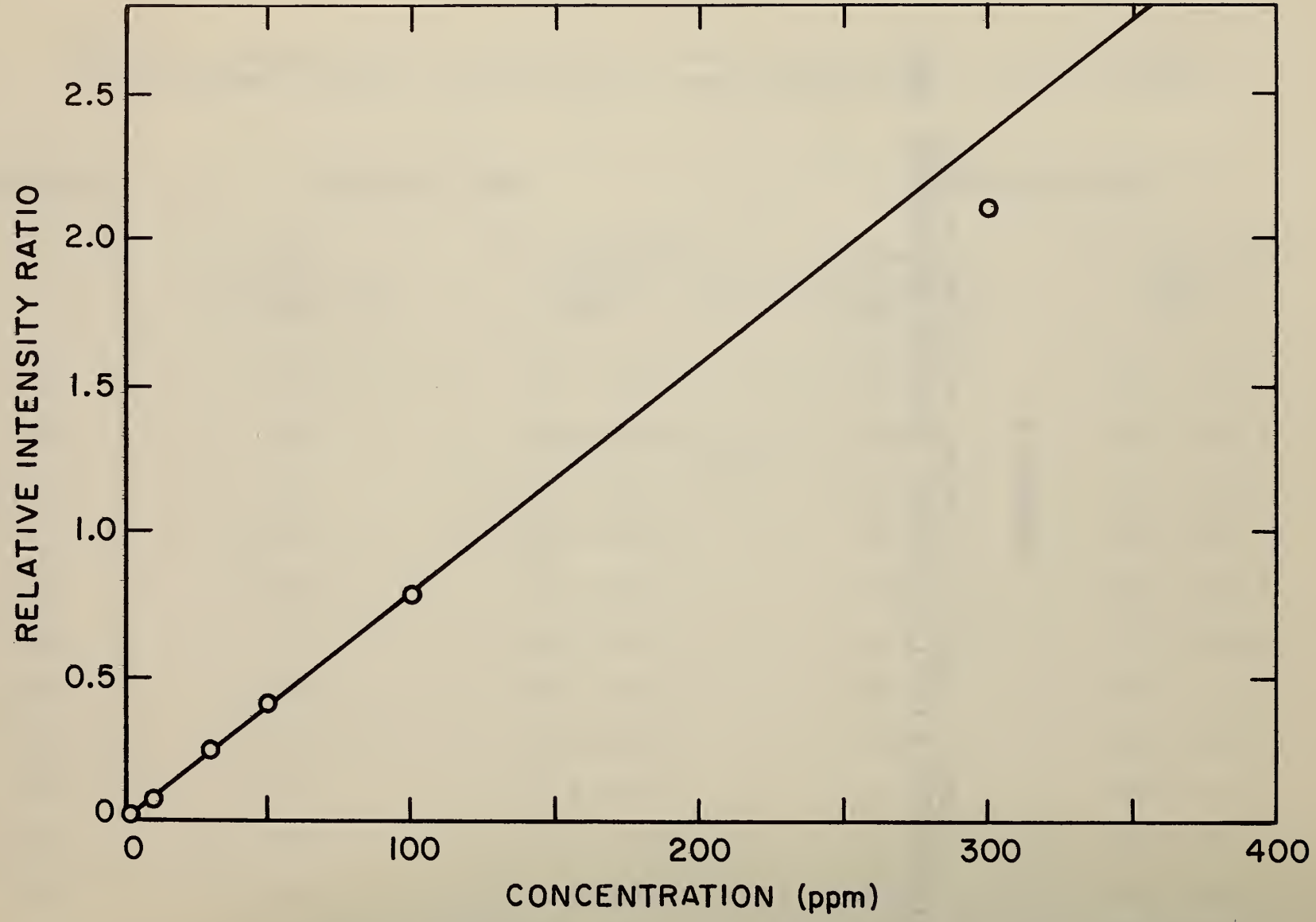

Figure 1. Chromium - x-ray fluorescence analytical response function.

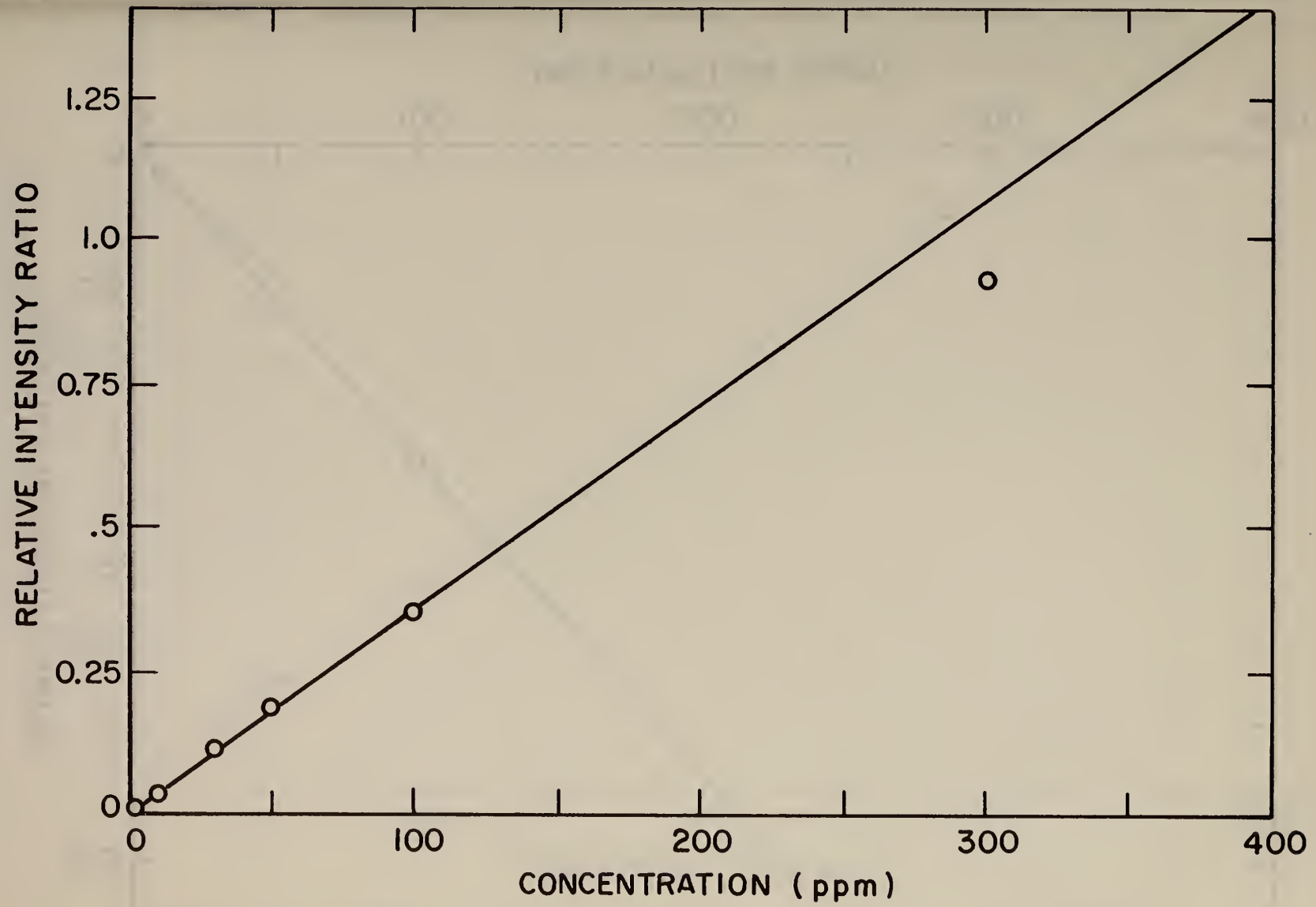

Figure 2. Iron - x-ray fluorescence analytical response function.

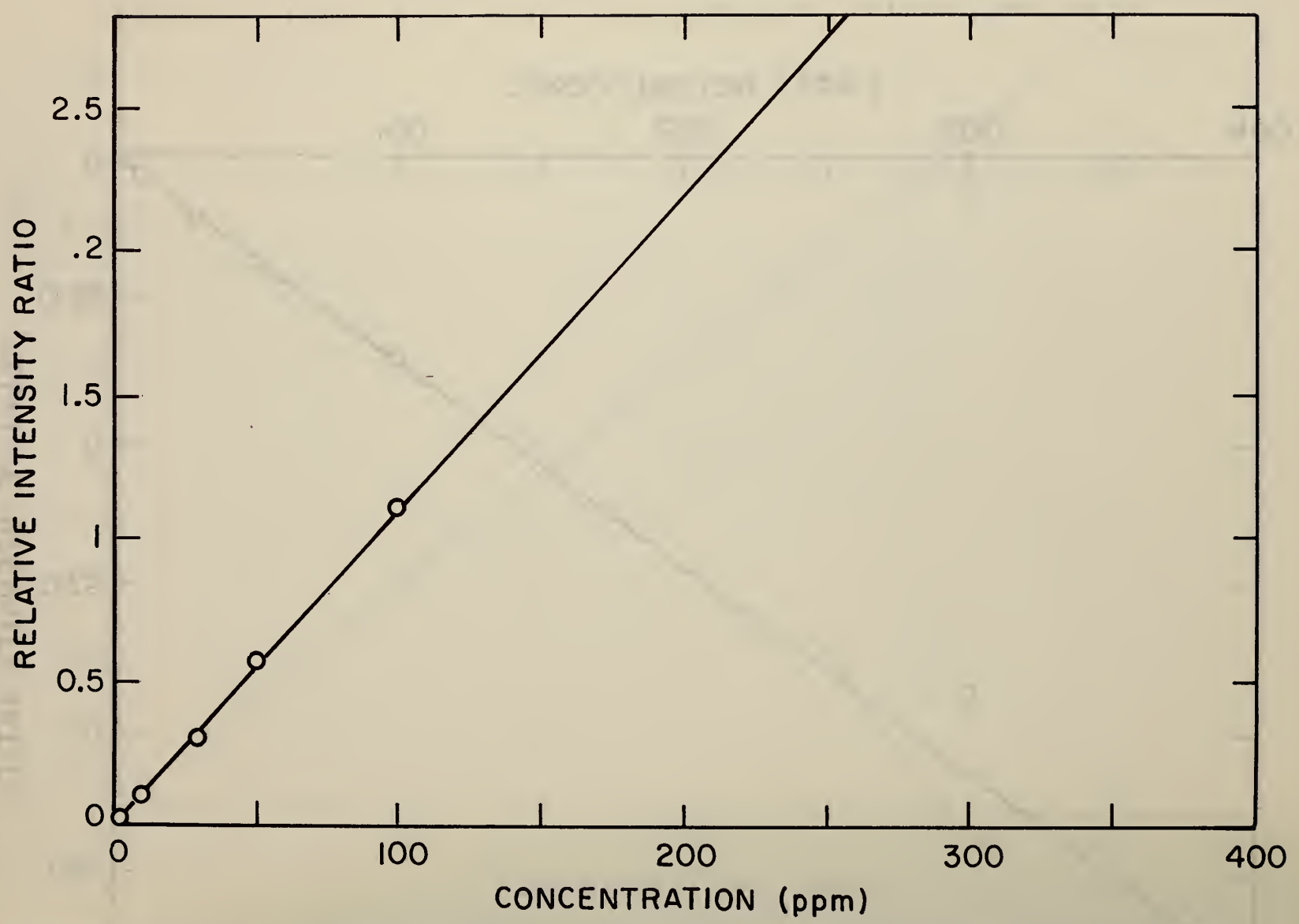

Figure 3. Nickel - x-ray fluorescence analytical response function.

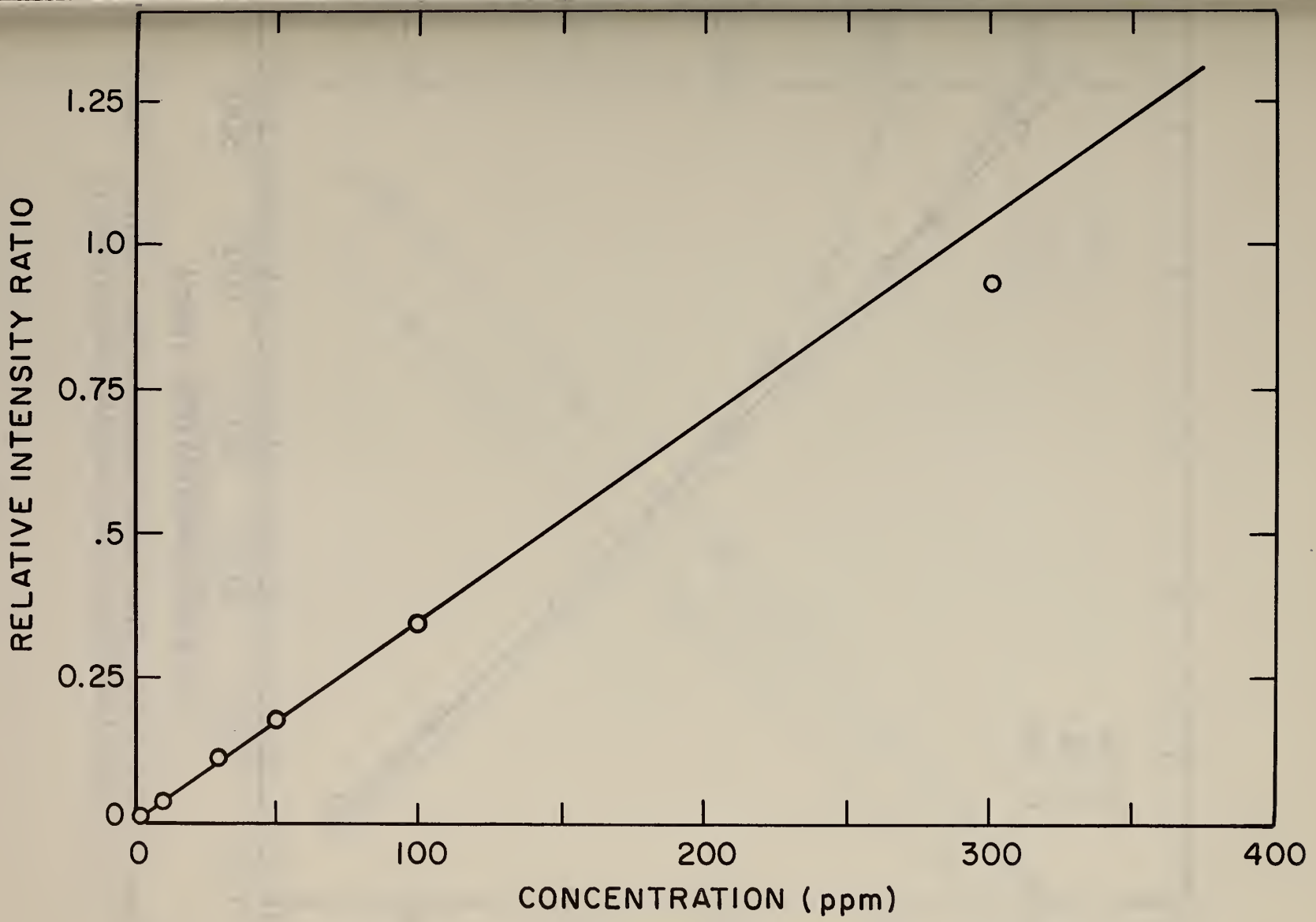

Figure 4. Titanium - x-ray fluorescence analytical response function.

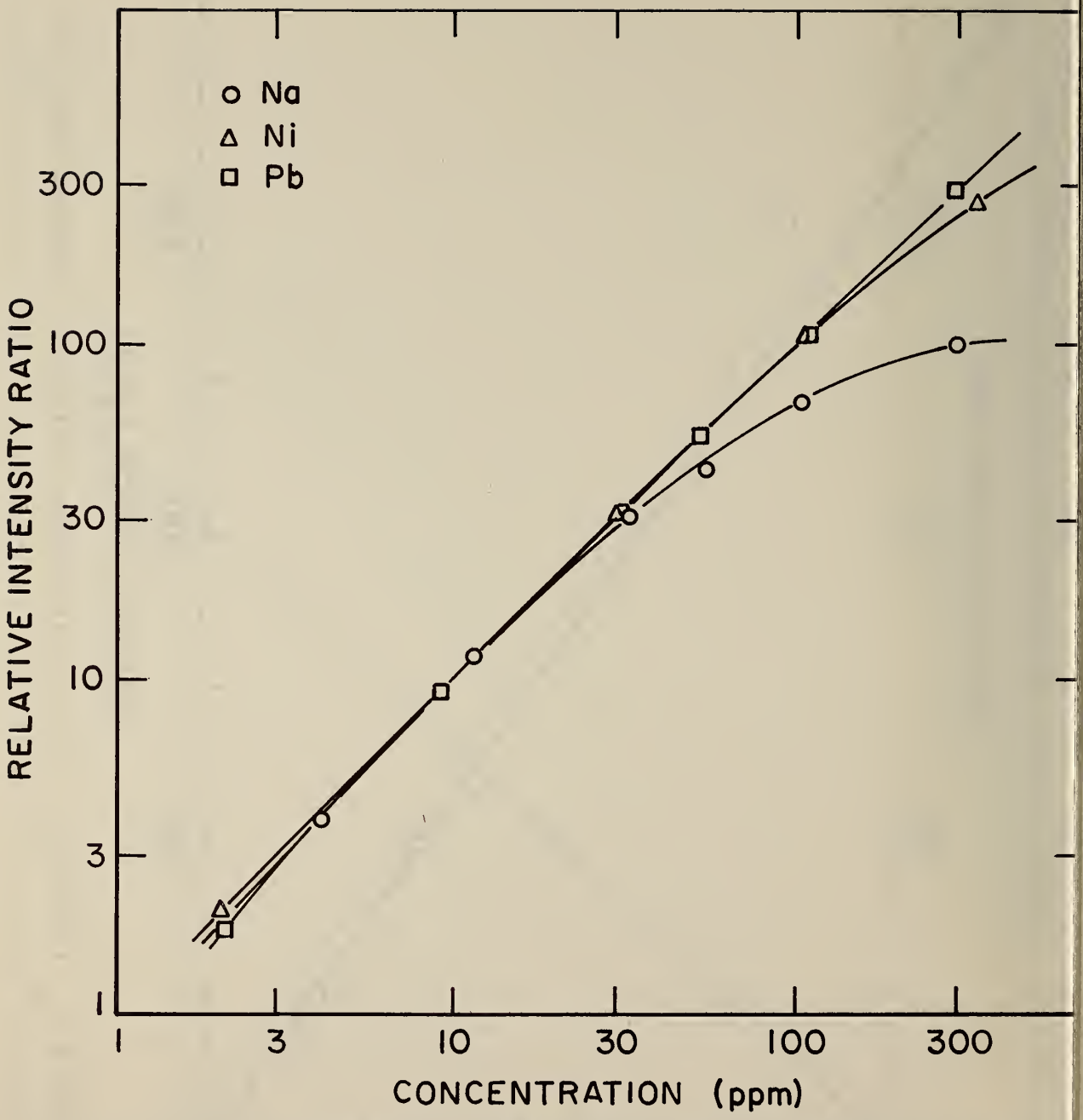

Figure 5. Analytical response functions for sodium, nickel, and lead by optical emission spectrometry.

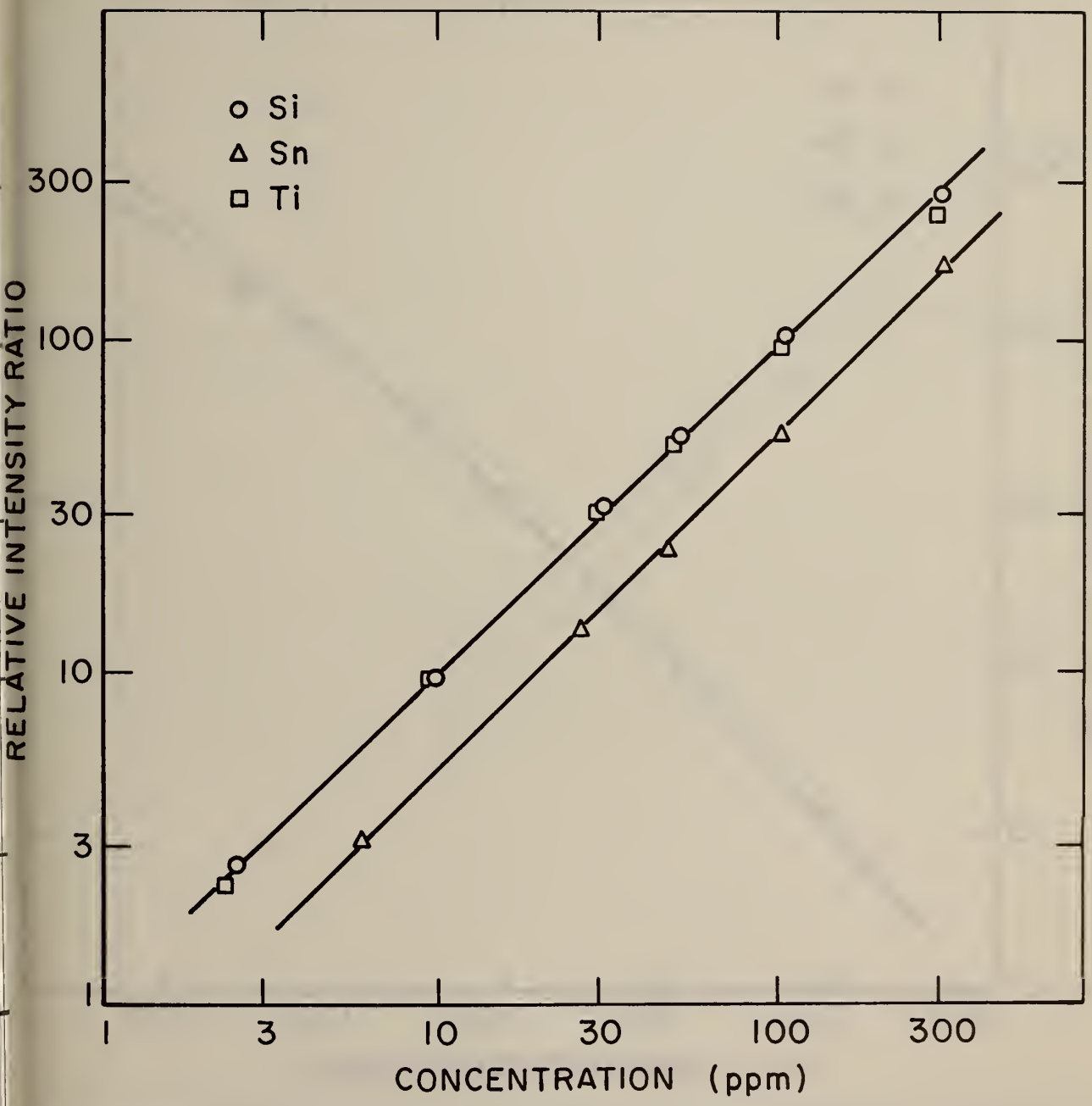

Figure 6. Analytical response functions for silicon, tin, and titanium by optical emission spectrometry.

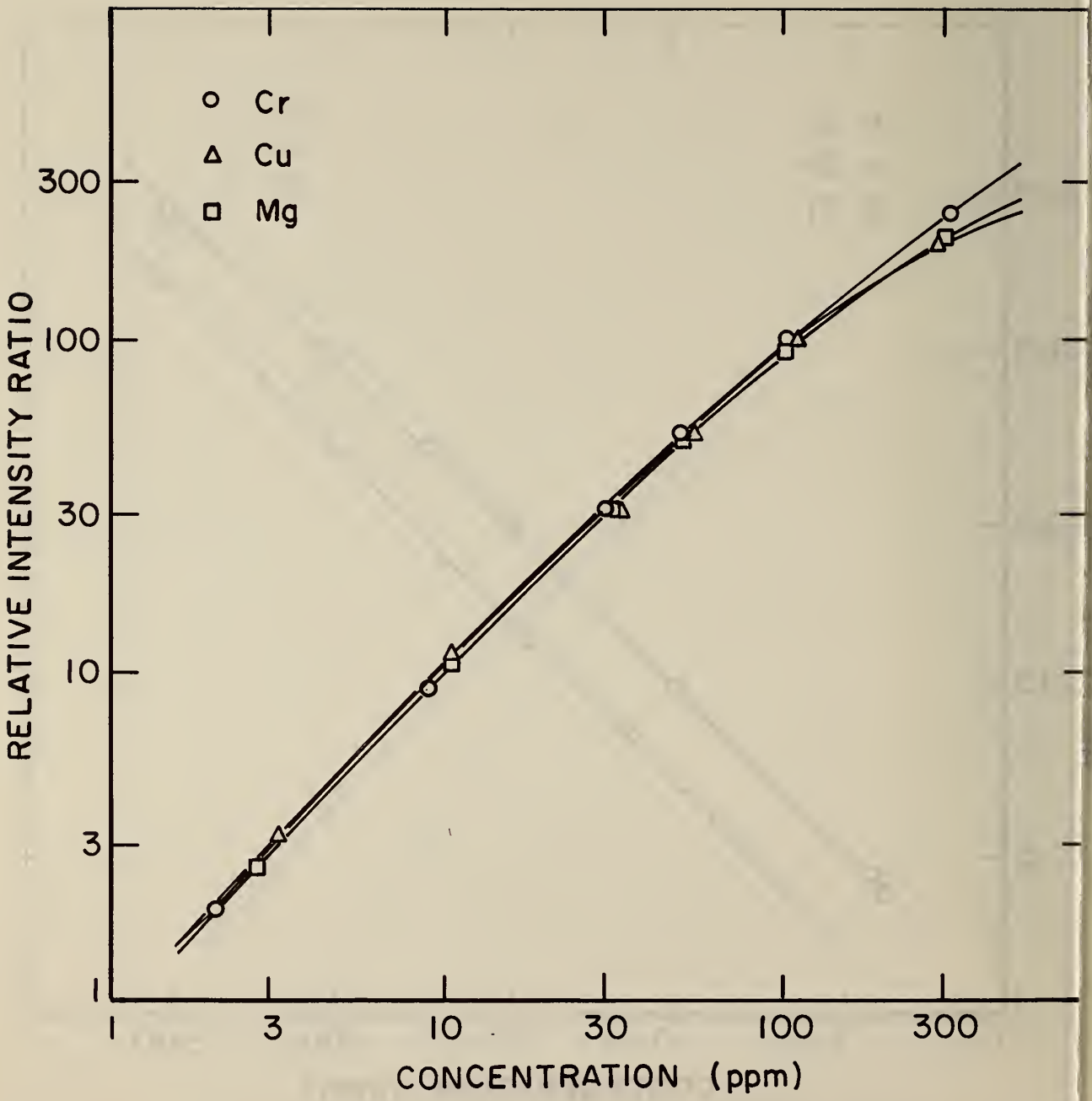

Figure 7. Analytical response functions for chromium, copper, and magnesium by optical emission spectrometry.

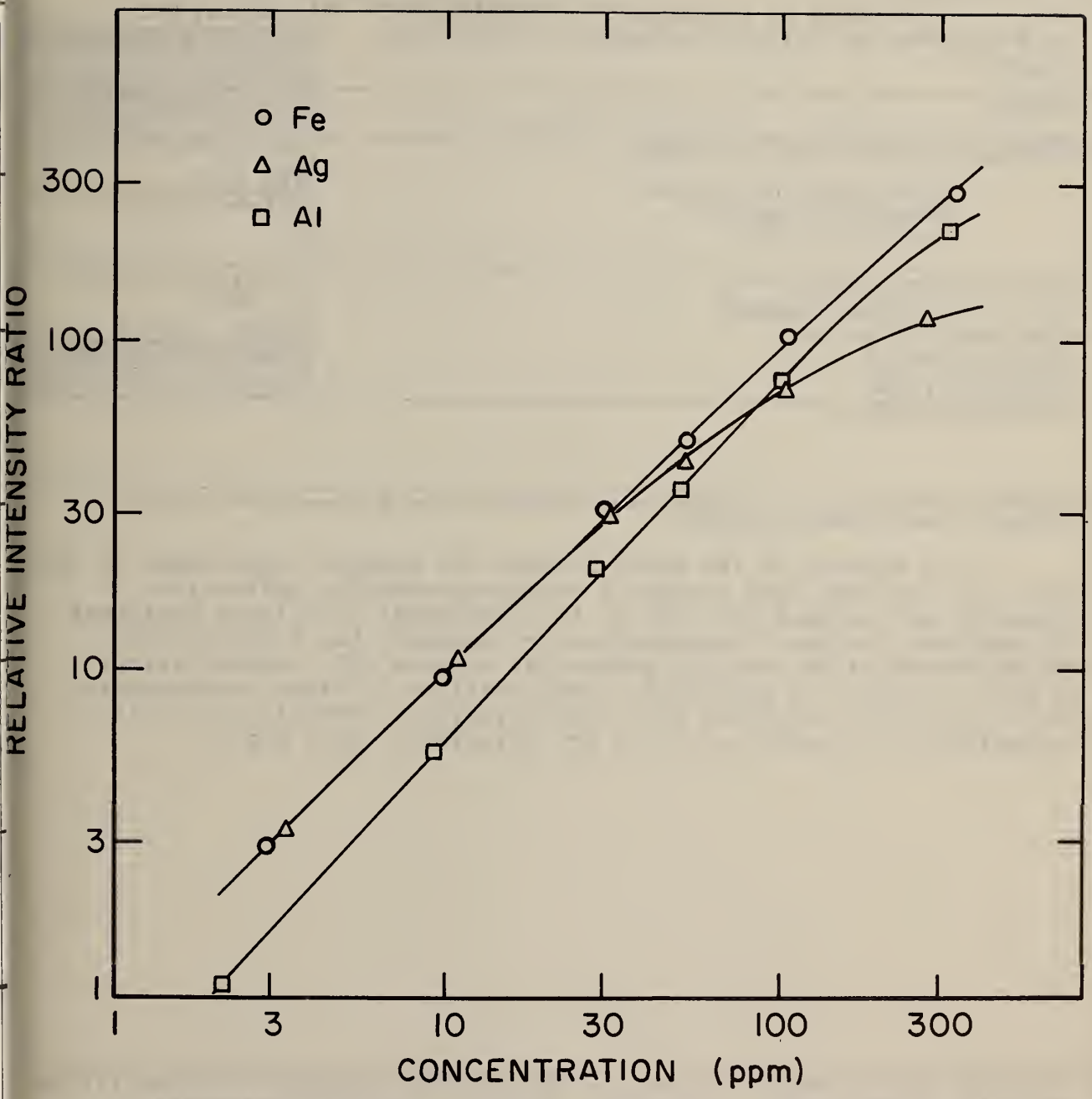

Figure 8. Analytical response functions for iron, silver, and aluminum by optical emission spectrometry.

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At the request of the Naval Systems Air Command, Department of the Navy, studies have been conducted on organo-metallic calibration standards and diluent oil used in the Department of Defense Equipment Oil Analysis Program. Consultation on standards has been provided, and measurements of physical properties of base oil, concentrations of major elements in standards, concentrations of trace contaminants, and stability of solutions have been performed. Results of studies accomplished in fiscal year 1972 are detailed in this report.

17. KEY WORDS (Alphabetical order, separated by semicolons) Calibration standards; concentration validity; flash point; lubricating oil; pour point; spectrometric analysis; stability; trace elements; viscosity.

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