N72-30503

NASA TECHNICAL MEMORANDUM

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QUANTITATIVE X-RAY DIFFRACTION ANALYSIS OF OXIDES FORMED ON SUPERALLOYS

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NATIONAL AERONAUTICS AND SPACE ADMINISTRATION • WASHINGTON, D. C. • SEPTEMBER 1972

1. Report No. NASA TM X-2627	2. Government Accession No.	3. Recipient's Catalog No.
4. Title and Subtitle QUANTITATIVE X-RAY DIFFRACTION ANALYSIS OF OXIDES FORMED ON SUPERALLOYS		5. Report Date September 1972
		6. Performing Organization Code
7. Author(s) Ralph G. Garlick		8. Performing Organization Report No. E-6911
9. Performing Organization Name and Address Lewis Research Center National Aeronautics and Space Administration Cleveland, Ohio 44135		10. Work Unit No. 134-03
		11. Contract or Grant No.
12. Sponsoring Agency Name and Address National Aeronautics and Space Administration Washington, D.C. 20546		13. Type of Report and Period Covered Technical Memorandum
		14. Sponsoring Agency Code
15. Supplementary Notes		

16. Abstract

Methods were developed for quantitative analysis by X-ray diffraction of the oxides Al_2O_3, NiO, Cr_2O_3, CoO, and $CoCr_2O_4$ within a standard deviation of about 10 percent of the weight fraction reported or within 1 percent absolute. These error limits assume that the sample oxides have been well characterized and that the physiochemical structure of the oxides in the samples are identical with those in the synthesized standards. Results are given for the use of one of the techniques in the analysis of spalls from a series of oxidation tests of the cobalt base alloy WI-52.

17. Key Words (Suggested by Author(s)) X-ray diffraction Quantitative analysis Oxides Spalls	18. Distribution Statement Unclassified - unlimited

19. Security Classif. (of this report) Unclassified	20. Security Classif. (of this page) Unclassified	21. No. of Pages 14	22. Price* $3.00

* For sale by the National Technical Information Service, Springfield, Virginia 22151

QUANTITATIVE X-RAY DIFFRACTION ANALYSIS OF OXIDES FORMED ON SUPERALLOYS

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SUMMARY

Techniques were developed for quantitative analysis by X-ray diffraction of the oxides aluminum oxide, nickel oxide, chromium oxide, cobaltous oxide, and cobalt chromate (Al_2O_3, NiO, Cr_2O_3, CoO, and $CoCr_2O_4$) using internal standards. The methods involved were (1) a silicon internal standard used to determine Al_2O_3, NiO, and Cr_2O_3 with diffraction from a loose mixture of the powders, (2) a nickel internal standard used to determine Al_2O_3, NiO, and Cr_2O_3 also with diffraction from a loose mixture of the powders, (3) a nickel oxide internal standard used to determine Cr_2O_3, CoO, $CoCr_2O_4$, and $CoWO_4$ (cobalt tungstate) with diffraction from a paste made by mixing the powders with mineral oil.

Calibration curves for the silicon internal standard loose powder method showed a relative standard deviation (RSD) of about 10 percent for each of the oxides Al_2O_3, NiO, and Cr_2O_3. Calibration curves for the nickel internal standard loose powder method showed a RSD of about 2 percent for these same three oxides. A prototype of these three oxides plus other phases was used to check this method. The results of 10 replications showed the analysis to be 23 percent Al_2O_3 with an RSD of 13 percent compared with 24 percent actual; 30.5 percent NiO with an RSD of 10 percent, compared with 30 percent actual; and 10 percent Cr_2O_3 with an RSD of 10 percent, compared with 10 percent actual.

A similar prototype sample for the NiO, internal standard, paste sample showed this method to be accurate except for $CoWO_2$, the result being: Cr_2O_3 - 3.1 percent measured, 5.5 percent actual; $CoCr_2O_4$ - 39.7 percent measured, 39.5 percent actual; CoO - 39.6 percent measured, 40.0 percent actual; $CoWO_4$ - 9 percent measured, 15.0 percent actual. The reason for the Cr_2O_3 inaccuracy was probably the high error in measuring the low signal from the small amount present. The reason for the $CoWO_4$ inaccuracy was apparently preferred orientation of the phase.

A series of spalls produced during the oxidation of WI-52 were found to consist only of CoO, $CoCr_4$, Cr_2O_3, and $CoWO_4$. These were analyzed using the NiO internal standard-paste method. Because of the inaccuracy of the method in $CoWO_4$ determination, this phase was determined by difference.

INTRODUCTION

During high-temperature oxidation of superalloys, more than one oxide is often formed (ref. 1). The relative amounts of the oxides present may change as oxidation proceeds. An understanding of this oxidation may depend on a knowledge of the amounts of the oxides present. A method for quantitative analysis of the oxides is then needed.

Although the elemental composition of a material can be determined by many techniques, these techniques give no indication of the phases present nor of the amounts of these phases. X-ray diffraction is the method for identification of phases and can, in some cases, be used to determine the relative amounts of these phases. This report describes some X-ray techniques used for the quantitative analysis of certain oxides important in the oxidation of superalloys.

The intensities of the lines of an X-ray diffraction pattern for a phase in a powder mixture depend not only on the amount of the phase present but also on the absorbtion of X-rays by the mixture. For example, if a sample contains both a weak and a strong absorbing phase, lines of the weak absorber will be weaker and of the strong absorber stronger than would be expected from the intensities of the pure phases. The absorbtion effects can be corrected for by the use of an internal standard according to the method described as follows:

Alexander and Klug (ref. 2) have shown that the intensity-absorption relationship for X-ray diffraction from a flat cake of powder is

$$I_1 = \frac{K_1 X_1}{\rho_1 \mu_t^*}$$

where I_1 is the intensity of a particular line of phase 1, X_1 is the weight fraction of phase 1, ρ_1 is the density of phase 1, μ_t^* is the mass absorbtion coefficient of the total sample, and K_1 is a constant that depends on the apparatus, the angle of diffraction, and the structure of phase 1. Similarly, for a line from component 2:

$$I_2 = \frac{K_2 X_2}{\rho_2 \mu_t^*}$$

The ratio of the intensity of a particular line for component 1 to a particular line for component 2 then is

$$\frac{I_1}{I_2} = \frac{K_1}{K_2} \frac{\rho_2}{\rho_1} \frac{X_1}{X_2} = (\text{constant}) \frac{X_1}{X_2}$$

that is, the ratio of the intensities of any two lines of the mixture is independent of the mass absorbtion of the mixture. Furthermore, since

$$X_1 = (\text{constant})(X_2)\,\frac{I_1}{I_2}$$

if the weight fraction of phase 2 (X_2) is held constant, then

$$X_1 = C\,\frac{I_1}{I_2}$$

the weight fraction of component 1 is linearly proportional to the intensity ratio. Weight fraction X_2 can be kept constant if phase 2 is an internal standard added to each sample in a constant proportion. For an actual system C is determined by plotting I_1/I_2 for synthetic mixtures of various weight fractions of phase 1. This constant can then be used to determine the weight fraction of phase 1 in any matrix. Quantitative analysis using these principles has been effectively used by other investigators. For example, Copeland and Bragg determined calcium hydroxide in a matrix of hydrated calcium silicates using magnesium hydroxide as an internal standard (ref. 3).

The peak intensity of a reflection from a phase is effected by many factors other than the weight fraction of the phase and absorbtion. Using an internal standard compensates only for absorbtion differences. To make the analysis accurate, the other factors must be either minimized or controlled. The factors, both instrumental and sample, are described in reference 4. To minimize sample factors, for example, sample particles should be small enough to make extinction and microbsorbtion effects negligible, and crystallites should be randomly oriented. Also, stressed particles should not be used. In this study the nature of the materials was such that stress and particle size were not problems. Preferred orientation is usually found when samples contain crystallites whose shape is acicular or tabular and the sample is compacted in the specimen holder. In this study the powders were not compacted, and in most cases the crystals were such that preferred orientation was not a problem.

Methods were examined for using appropriate internal standards to determine weight fractions of aluminum oxide, nickel oxide, chromium oxide, cobalt oxide, cobalt chromite, and cobalt tungstate (Al_2O_3, NiO, Cr_2O_3, CoO, $CoCr_2O_4$, and $CoWO_4$). These are some of the most common phases found as products of oxidation on superalloys.

Materials and Procedure

Materials used as internal standards were chosen that had strong reflections well resolved from those of the oxides to be analyzed. The weight fractions of the internal standards used were chosen so intensity ratios expected for actual oxide samples could be most accurately measured. One reflection was chosen for each material that did not overlap any reflections from other materials to be combined with it.

Three different internal standard materials were used for the determination of the oxides: silicon (Si) for Al_2O_3, Cr_2O_3 and NiO, nickel (Ni) also for Al_2O_3, Cr_2O_3, and NiO; and NiO for CoO, Cr_2O_3, $CoCr_2O_4$, and $CoWO_4$. Materials were all acquired in reagent-grade powder form. X-ray diffraction showed all to be single phase except $CoWO_4$ and $CoCr_2O_4$. The $CoWO_4$ contained a small amount of calcium tungstate ($CaWO_4$). The $CoCr_2O_4$ was found to be chiefly Cr_2O_3 and CoO. This powder was heated at 2000° F (1366 K) for 91 hours to induce more complete formation of $CoCr_2O_4$. After treatment the powder was $CoCr_2O_4$ plus a small amount of Cr_2O_3.

Table I(a) shows the reflections used to determine the calibration curves for Al_2O_3, NiO, and Cr_2O_3 using a 0.25 weight fraction Si standard. Table II shows the mixtures used in these determinations. These combinations were mixed for 1 hour in a jar mill. The powder was also mixed immediately before diffraction by vibration in a vial for 4 minutes. Some of the loose powder was then dumped into a rectangular cavity 0.3-millimeter (0.020-in.) deep in a flat specimen holder. Excess powder was then removed with a spatula. After diffraction, the sample was returned to the vial and the process repeated. In this way the intensities were determined 10 times for each mixture. For many of the mixtures both peak height intensity and integrated intensity were determined. A planimeter was used to determine integrated intensity. Integrated intensity gave ratios similar to those from peak heights. Peak height was therefore used in further measurements.

Calibration curves were similarly determined for the same three oxides (Al_2O_3, Cr_2O_3 and NiO) using 0.1 weight fraction nickel as the internal standard. The reflections used are shown in table I(b). The mixtures of oxides used in this case were 100 percent of each of the oxides and the possible 50-50 percent mixtures. A prototype sample containing known amounts of the three oxides was mixed to estimate the application of calibration curves made from a mixture of one or two of the oxides to a mixture of multiple phases, including phases not present in the calibration samples. The mixture was 30.0 percent NiO, 24.0 percent Al_2O_3, 10.0 percent Cr_2O_3, 16.0 percent SiO_2, 17.6 percent WC, and 2.4 percent Co. Relatively large amounts of the unknown must be used for the loose powder methods described above. For example, about 0.5 gram of NiO are needed to fill the sample holder cavity.

For some oxidation tests, very small amounts of spall are collected, and it may be useful to know the proportions of the oxides in these. Therefore, another method was investigated that allows for a smaller sample. This method of sample preparation is similar to that described by Munch and Pierron (ref. 5). In this method the sample (0.2 g for the calibration samples) is put into a small mortar with enough mineral oil so that mulling yields a smooth paste. This paste is then scraped from the mortar onto a section of a glass slide and spread evenly. (Specimens as small as 0.05 g have been used.) Drying the sample for 1 hour was enough so that it retained its shape and the individual particles apparently did not re-orient themselves with further time. This method was used for a 0.1 weight fraction NiO internal standard for the following oxides: CoO, $CoCr_2O_4$, Cr_2O_3, and $CoWO_4$. The reflections used for this analysis are given in table I(c). For these calibrations no mixtures of oxides were used, only 100 percent of each plus 10 percent NiO. Intensities were measured at least five times for each oxide. A prototype sample of these oxides was then made, the mixture being 5.5 percent Cr_2O_3, 39.5 percent $CoCr_2O_4$, 40 percent CoO, and 15 percent $CoWO_4$. This mixture was then used to estimate the accuracy of the method.

RESULTS AND DISCUSSION

Silicon Internal Standard Loose Powder Samples

The calibration curves for NiO, Al_2O_3 and Cr_2O_3 using a Si internal standard are shown in figures 1 to 3. The spread of values shown represents all data points (10 points at 1.0 weight fraction, 20 points at other weight fractions). The curves, which were forced through the origin, can be represented by the equations:

$$\frac{I_{NiO(111)}}{I_{Si(111)}} = (2.16 \pm 0.22)\, X_{NiO}$$

$$\frac{I_{Al_2O_3(104)}}{I_{Si(111)}} = (0.726 \pm 0.068)\, X_{Al_2O_3}$$

$$\frac{I_{Cr_2O_3(104)}}{I_{Si(111)}} = (1.61 \pm 0.14)\, X_{Cr_2O_3}$$

The errors reported represent one standard deviation calculated using the computer techniques described in reference 6. The pooled relative standard deviations were about 10.2 percent of the weight fraction for NiO, 9.4 percent of the weight fraction for Al_2O_3, and 8.7 percent of the weight fraction for Cr_2O_3. Since these errors are relatively high and similar for the three oxides, it was thought that they may be due to variations in the intensity of the Si standard. To check this, a sample of Si powder alone was placed in the holder, and the peak height measured at least 10 times for each of the following scanning speeds: $0.2^\circ 2\theta$/min, $0.4^\circ 2\theta$/min and $2^\circ 2\theta$/min. Considerable variation in the peak height was found, the lowest relative standard deviation (RSD) being 4.5 percent. The cause of this variation is not obvious, since from counting statistics we do not expect this error, and since the variation was random, not either increasing or decreasing with time as would be expected due to an orientation change during settling of the powder.

Nickel Internal Standard-Loose Powder Samples

The calibration curves for NiO, Al_2O_3, and Cr_2O_3 using a Ni internal standard are shown in figures 4 to 6. The curves, which were also forced through the origin, can be represented by the equations:

$$\frac{I_{NiO(111)}}{I_{Ni(111)}} = (3.63 \pm 0.05)\, X_{NiO}$$

$$\frac{I_{Al_2O_3(104)}}{I_{Ni(111)}} = (1.26 \pm 0.03)\, X_{Al_2O_3}$$

$$\frac{I_{Cr_2O_3(104)}}{I_{Ni(111)}} = (2.75 \pm 0.06)\, X_{Cr_2O_3}$$

These standard deviations are much lower than for the Si internal standard, the highest being 2.4 percent for the Cr_2O_3.

The averaged concentrations of 10 replicate determinations of the prototype sample showed good agreement with the actual amount, as shown in table III.

Nickel Oxide Internal Standard-Paste Samples

The results of the standard specimens for the paste samples are shown in table IV. The $CoWO_4$ showed a very high variation of individual peak intensities. In addition, it was observed that the ratio of two peaks of the $CoWO_4$ was highly variable, indicating extremely variable preferred orientation of the monoclinic $CoWO_4$ phase. However, measurement of the sum of two peak intensities did not improve the variability of the measurement of this oxide.

The results for the prototype paste sample as shown in table IV showed good agreement between the actual weight fractions and those determined by intensity ratios except for $CoWO_4$: Cr_2O_3 was 3.1 percent compared with 5.5 percent actual, $CoCr_2O_4$ was 39.7 percent compared with 39.5 percent actual, CoO was 39.6 percent compared with 40.0 percent actual, and $CoWO_4$ was 9.0 percent compared with 15.0 percent actual. The high error for Cr_2O_3 is believed due to the inaccuracy in measuring the low signal from the small amount of Cr_2O_3 present.

The paste method was used in the analysis of a series of spalls containing only these oxides. These spalls were produced during the cyclic oxidation of a cobalt base alloy (WI-52) at 2000° F. Because of the poor accuracy for the $CoWO_4$ standard specimen, X-rays were not used for its determination in the WI-52 spall samples. Since the spalls consisted of only Cr_2O_3, $CoCr_2O_4$, CoO, and $CoWO_4$, the first three phases were determined from intensity ratios, and the $CoWO_4$ by difference. These results are also shown in table V. The analysis was valuable in this case in showing how the amount of $CoCr_2O_4$ and CoO in the spall depended on the surface preparation of the samples.

CONCLUDING REMARKS

The methods described in this report were adequate for quantitative analysis of the phases studied except for $CoWO_4$. An accuracy within about 10 percent of the amount reported was obtained on oxide blends for weight fractions above about 0.1 for each of the phases. For lower weight fractions counting errors in determining the height of the peaks will cause more error, but even these lower values were good to about 1 percent absolute. The applicability of such methods to other systems depends chiefly on the following:

1. The phases present in the sample should be known and their X-ray patterns well characterized. This is necessary to insure that there is no superposition of other lines on those used in the analysis.

2. The individual phases used in making a standard should be the same as the phases to be determined.

3. A homogeneous mixture of the unknown with the correct weight fraction of the internal standard should be achieved.

4. There is little lattice strain in the phase to be analyzed.

5. The crystallites should be large enough so that line broadening due to small crystallites is not a problem.

6. There is little preferred orientation in the phase to be analyzed.

The analysis of the spalls from the oxidation of Co base alloy WI-52 suggests the application of such techniques to other superalloy spalls if the oxides formed are well characterized. For such oxides it should be possible to achieve an accuracy of ±10 percent of the weight fraction reported. At present there is no other method for quantitative analysis of such phases.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, June 7, 1972,
134-03.

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TABLE I. - REFLECTIONS USED IN QUANTITATIVE ANALYSIS

(a) Silicon internal standard - loose powder method

Phase	Miller indices, hkl	Interplanar spacing, d, m	Intensity ratio, $[I/I_1]^a$
Si	(100)	3.14×10^{-10}	100
NiO	(111)	2.41	91
Al_2O_3	(104)	2.55	90
Cr_2O_3	(104)	2.67	100

(b) Nickel internal standard - loose powder method

Phase	Miller indices, hkl	Interplanar spacing, d, m	Intensity ratio, $[I/I_1]^a$
Ni	(111)	2.03×10^{-10}	100
NiO	(111)	2.41	91
Al_2O_3	(104)	2.55	90
Cr_2O_3	(104)	2.67	100

(c) Nickel oxide internal standard - paste method

Phase	Miller indices, hkl	Interplanar spacing, d, m	Intensity ratio, $[I/I_1]^a$
NiO	(200)	2.09×10^{-10}	100
CoO	(200)	2.13	100
Cr_2O_3	(104)	2.67	100
$CoCr_2O_4$	(311)	2.51	100
$CoWO_4$	(110)	3.73	30
$CoWO_4$	(031)	1.76	14

[a] Ref. 8.

TABLE II. - MIXTURES USED FOR CALIBRATION CURVES FOR DETERMINATION OF NiO, Al_2O_3, AND Cr_2O_3 USING SILICON INTERNAL STANDARD

[Standards are these mixtures plus 0.25 weight fraction silicon.]

Mixture	Weight fraction		
	NiO	Al_2O_3	Cr_2O_3
1	1.00	0	0
2	.75	.25	0
3	.75	0	.25
4	.50	.50	0
5	.50	0	.50
6	.25	.75	0
7	.25	0	.75
8	0	1.00	0
9	↓	.75	.25
10	↓	.50	.50
11	↓	.25	.75
12	↓	0	1.00

TABLE III. - SUMMARY OF ANALYSIS OF PROTOTYPE OXIDE MIXTURES

Method of analysis	Oxides present	Weight percentages		Relative standard deviation, percent[c]
		Added	Determined[b]	
Nickel internal standard	Al_2O_3	24	24	13
	NiO	30	30.5	10
	Cr_2O_3	10	10	10
	WC[a]	17.6	----	--
	Co[a]	2.4	----	--
	SiO_2[a]	16	----	--
Nickel oxide internal standard	$CoWO_4$	15	9	--
	$CoCr_2O_4$	39.5	39.7	--
	Cr_2O_3	5.5	3.1	--
	CoO	40	39.6	--

[a] Extraneous phases not present in standards.

[b] Average of 10 replications.

[c] $$RSD = \frac{\sqrt{\frac{\sum d^2}{\eta - 1}}}{\text{average percent}} \times 100.$$

TABLE IV. - INTENSITY RATIOS FOR STANDARD SPECIMENS

[NiO internal standard-paste samples; each standard is phase listed +0.1 NiO.]

Phase	Ratio measured	Average ratio for 1.0 weight fraction	Range of apparent weight fractions (5 values)
Cr_2O_3	$\frac{I_{Cr_2O_3(104)}}{I_{NiO(200)}}$	3.75	0.98 - 1.04
CoO	$\frac{I_{CoO(200)}}{I_{NiO(200)}}$	2.99	0.98 - 1.02
$CoCr_2O_4$	$\frac{I_{CoCr_2O_4(311)}}{I_{NiO(200)}}$	1.81	0.94 - 1.04
$CoWO_4$	$\frac{I_{CoWO_4(100) + (031)}}{I_{NiO(200)}}$	1.72	0.70 - 1.47

TABLE V. - QUANTITATIVE X-RAY ANALYSIS OF SPALLED SCALES FROM GROUND-LAPPED AND GROUND-POLISHED WI-52 FOR 20 HOUR CYCLES AT 2000° F (1266° C)[a]

[Analysis used NiO internal standard - paste method.]

Cycle	Phase content of spall, wt. %			
	Cr_2O_3	$CoCr_2O_4$	CoO	$CoWO_4$ (by diff.)
Ground-lapped				
1	3	50	34	13
2	2	50	37	11
3	4	50	35	11
4	3	45	45	7
5	2	45	36	17
6	3	47	39	11
Ground-polished				
1	11	22	54	13
2	3	29	59	9
3	3	21	56	20
4	4	23	51	22

[a] From ref. 6.

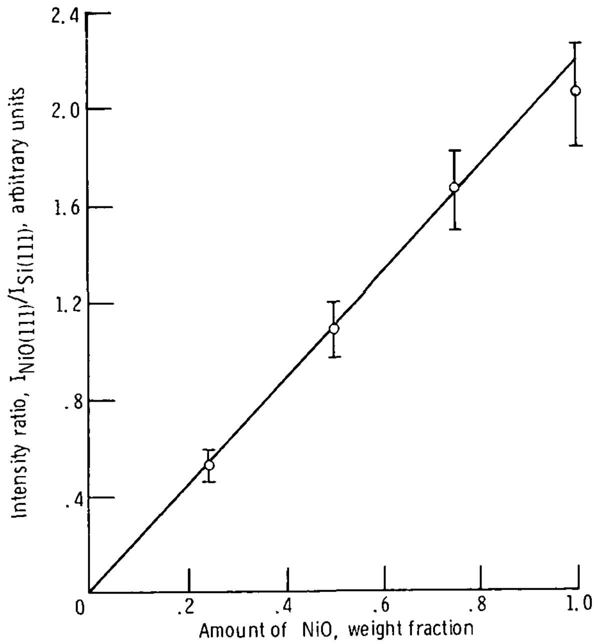

Figure 1. - Calibration curve for quantitative determination of nickel oxide (NiO) using 0.25-weight-fraction silicon internal standard. Slope, 2.16; number of data points, 20, except 10 at 1.0 weight fraction; pooled standard deviation; 10.2 percent. Ranges on curve are one standard deviation.

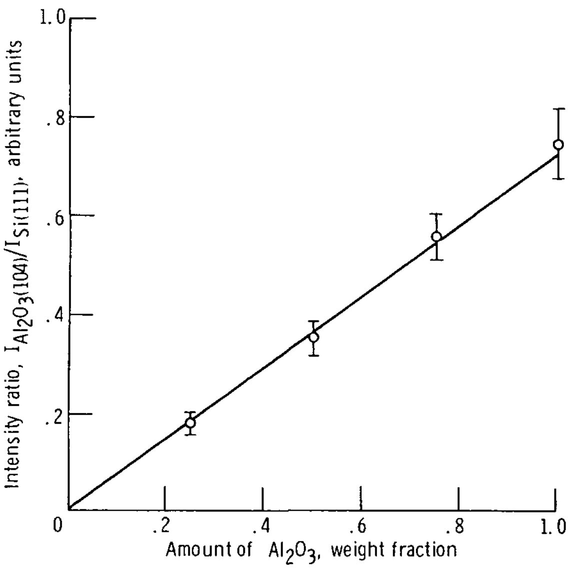

Figure 2. - Calibration curve for quantitative determination of aluminum oxide (Al_2O_3) using 0.25 weight fraction silicon internal standard. Slope, 0.726; number of data points, 20, except 10 at 1.0 weight fraction; pooled standard deviation, 9.4 percent. Ranges on curve are one standard deviation.

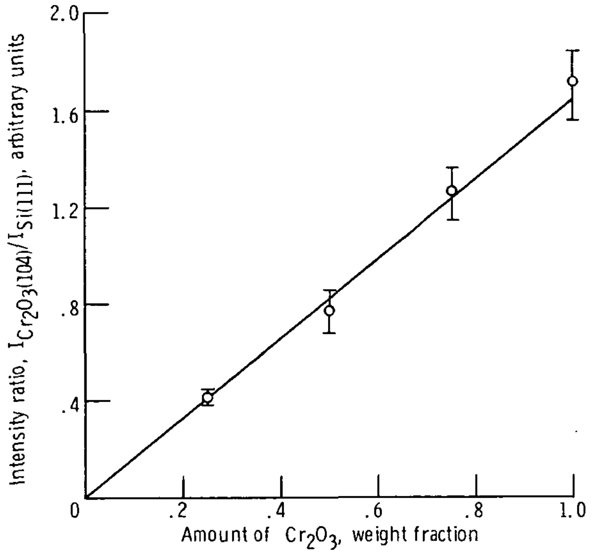

Figure 3. - Calibration curve for quantitative determination of chromium oxide (Cr_2O_3) using 0.25 weight fraction silicon internal standard. Slope, 1.61; number of data points, 20, except 10 at 1.0 weight fraction; pooled standard deviation, 8.7 percent. Ranges on curve are one standard deviation.

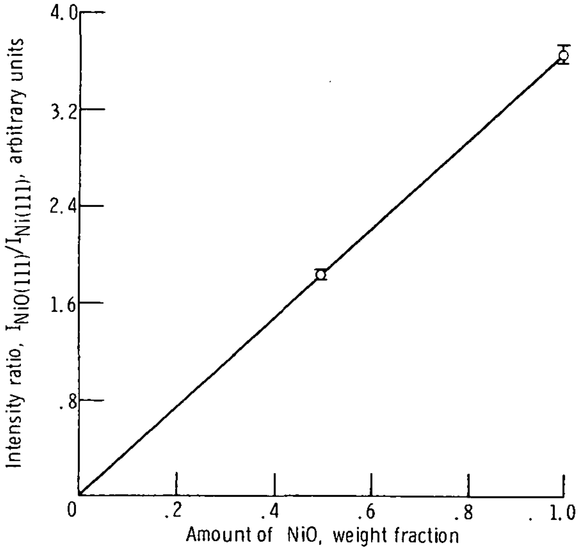

Figure 4. - Calibration curve for quantitative determination of nickel oxide (NiO) using 0.1 weight fraction nickel internal standard. Slope, 3.63; number of data points, 20 at 0.5 weight fraction, 10 at 1.0 weight fraction; pooled standard deviation, 1.4 percent. Ranges on curves are one standard deviation.

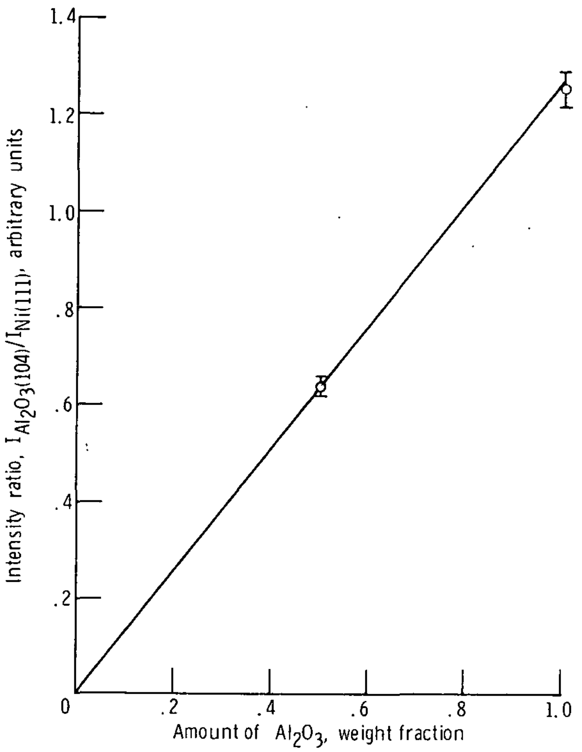

Figure 5. - Calibration curve for quantitative determination of aluminum oxide (Al_2O_3) using 0.1 weight fraction nickel internal standard. Slope, 1.26; number of data points, 20 at 0.5 weight fraction, 10 at 1.0 weight fraction; pooled standard deviation; 2.4 percent. Ranges on curves are one standard deviation.

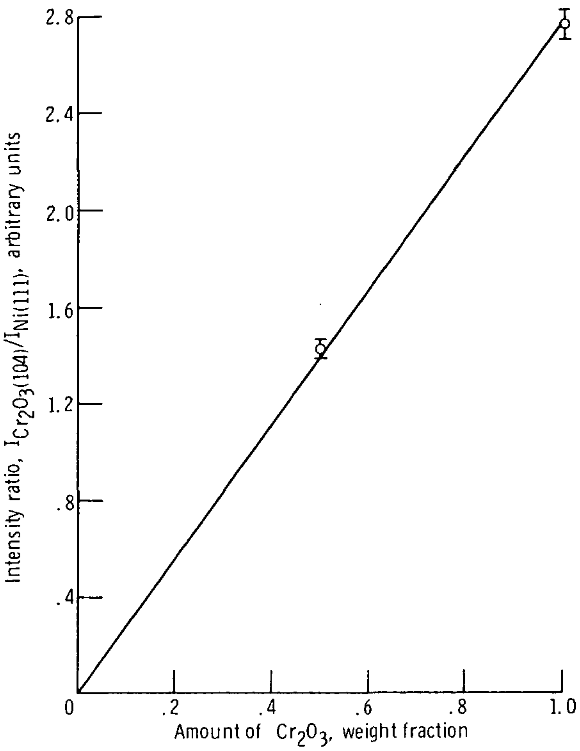

Figure 6. - Calibration curve for quantitative determination of chromium oxide (Cr_2O_3) using 0.1 weight fraction nickel internal standard. Slope, 2.75; number of data points, 20 at 0.5 weight fraction, 10 at 1.0 weight fraction; pooled standard deviation; 2.2 percent. Ranges on curves are one standard deviation.

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