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Standard Test Method for X-Ray Emission Spectrometric Analysis Analysis of Stainless and Alloy Steels by X-ray Fluorescence Spectrometry¹

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^{ε1} ~~Note—Additional research report was added editorially in June 2001.~~

¹ This test method is under the jurisdiction of ASTM Committee E-1 E01 on Analytical Chemistry for Metals, Ores and Related Materials and is the direct responsibility of Subcommittee E01.01 on Iron, Steel, and Ferrous Alloys.

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1. Scope

1.1 This test method² ~~provides for~~ covers the X-ray spectrochemical analysis of stainless and alloy steels by wavelength dispersive X-ray Fluorescence Spectrometry for the determination of the following elements in the ranges indicated:

Element	Concentration Range, %	Range %
Chromium	44.0	2.0 to 49.0
Nickel	0.20 25.0	
Cobalt	0.03 to 13.0	
Cobalt	0.03 to 0.40	
Copper	0.05 to 3.50	
Molybdenum	0.05 to 3.00	
Manganese	0.40	0.3 to 2.00
Columbium (Niobium)	0.30 5.0	
Molybdenum	0.15 to 0.70	
Cobalt	0.05 3.5	
Nickel	0.20 to 0	
Nickel	0.20 to 35.0	
Niobium	0.5 to 1.3	
Niobium	0.05 to 1.3	
Phosphorus	0.01 to 0.03	
Silicon	0.05 to 0.20	
Sulfur	0.02 to 0.30	
Titanium	0.002 to 0.04	
Vanadium	0.03 to 0.25	

NOTE 1—~~Unless exceptions are noted,~~ concentration ranges can be extended by the use of suitable reference materials. The detection limit for the elements is lower than the listed value. The ranges represent the nominal levels at which this method was tested.

1.2 This test method is applicable to the control analysis of either chill-cast or wrought samples having a diameter of approximately 25 mm.

NOTE 2—Samples of greater or lesser size than those designated may be used with specially designed sample holders.

1.3 Matrix effects exist between the elements listed. To compensate for these effects, a series of calibration curves are required to cover the designated concentration ranges. The composition of the sample must approximate closely one or more of the reference materials used in the calibration curve which is applied. Mathematical corrections may also be used to solve for matrix effects. (Refer to Practice E 1361.) A variety of such systems is commonly used. Any of these are acceptable that will achieve analytical accuracy equivalent to that reported for the method.

1.4 materials.

1.2 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. Specific hazard precautionary statements are given in Section 7, 10.

² Supporting data for this test method as determined by cooperative testing have been filed at ASTM International Headquarters as RR:E02-1012 and E01-1032. RR: E-1-1032.

2. Referenced Documents

2.1 *ASTM Standards:*

E 135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials³

E 305 Practice for Establishing and Controlling Spectrochemical Analytical Curves³

~~E 353 Test Methods 691 Practice for Chemical Analysis of Stainless, Heat-Resisting, Maraging, and Other Similar Chromium-Nickel-Iron Alloys³~~

~~E 876 Practice for Use of Statistics in Conducting an Interlaboratory Study to Determine the Evaluation Precision of Spectrometric Data a Test Method⁴~~

E 1361 Guide for Correction of Interelement Effects in X-ray Spectrometric Analysis

2.2 *Other Documents:*

~~MNL 7 Manual on Presentation³~~

~~E 1601 Practice for Conducting an Interlaboratory Study to Evaluate the Performance of an Analytical Method³~~

~~E 1622 Practice for Correction of Spectral Line Overlap in Wavelength-Dispersive X-ray Spectrometry³~~

~~E 1806 Practice for Sampling Steel and Control Chart Analysis Iron for Determination of Chemical Composition⁵~~

3. Terminology

3.1 For definitions of terms used in this test method, refer to Terminology E 135.

4. Summary of Test Method

4.1 ~~The sample test specimen is finished to a clean, uniform surface and then irradiated by with an X-ray beam of high energy (short wavelength): energy. The secondary X-rays produced are dispersed by means of crystals and the intensities are measured by suitable detectors at selected wavelengths. Data The outputs of the detectors in voltage pulses are collected counted. Radiation measurements are made based on the time required to reach a fixed number of counts, or on the total counts obtained for a fixed time, or on the integration of voltage for a fixed time. time (generally expressed in counts per unit time). Concentrations of the elements are determined by relating the measured radiation of unknown samples specimens to analytical curves or charts prepared from standard with suitable reference materials of known compositions: materials. A fixed-channel or, polychromator system or a sequential system may sequential, monochromator can be used to provide simultaneous or sequential determinations for measurement of the elements.~~

5. Significance and Use

5.1 This procedure is suitable for manufacturing control and for verifying that the product meets specifications. It provides rapid, multi-element determinations with sufficient accuracy to assure product quality. The analytical performance data included may be used as a benchmark to determine if similar X-ray spectrometers provide equivalent precision and accuracy, or if the performance of a particular spectrometer has changed.

6. Interferences

6.1 Interelement effects or matrix effects exist for some of the elements listed. Mathematical correction may be used to solve for these elements. Various mathematical correction procedures are commonly utilized. See Guides E 1361 and E 1622. Any of these procedures that achieves analytical accuracy equivalent to that provided by this test method is acceptable.

7. Apparatus ~~NOTE 3—It is not within the scope of this test method to prescribe all details of equipment to be used.~~

~~Equipment varies between laboratories.~~

5.1 *Sample*

7.1 *Specimen Preparation Equipment :*

5

~~7.1.1 Surface Grinder, with 60 to 600-grit aluminum oxide belts or Sander With Abrasive Belts or Disks, or Lathe, capable of providing a flat, uniform surface on the reference materials and test specimens. Aluminum oxide and zirconium oxide belts and discs with a uniform flat finish.~~

~~5.2- grit size of between 60 and 180 have been found suitable.~~

7.2 *Excitation Source:*

5.2.1 *X-Ray Generator*

~~7.2.1 X-ray Tube Power Supply, providing a constant potential or rectified power of sufficient energy to produce secondary radiation of the sample specimen for the elements specified. The generator may be equipped with a line voltage regulator and a current stabilizer.~~

³ Annual Book of ASTM Standards, Vol 03.05.

⁴ Annual Book of ASTM Standards, Vol-03-06: 14.02.

⁵ ASTM Manual Series, ASTM, 6th Edition Annual Book of ASTM Standards, 1990: Vol 03.06.

~~57.2.2 X-Ray Tubes, with targets of various high-purity elements; that are capable of continuous operation up to the at required potentials and currents shown in Table 1.~~

~~NOTE 4—X-ray tubes with tungsten, gold, and rhodium targets were used in that will excite the testing of this method.~~

~~5.3— elements to be determined.~~

~~7.3 Spectrometer, designed for X-ray emission analysis, using air or vacuum, analysis and equipped with specimen holders and a specimen chamber. The chamber should shall contain a specimen spinner, and must be equipped for vacuum or helium-flushed operation for the determination of elements of atomic number 20 (calcium) or lower.~~

~~57.3.1 Analyzing Crystals, flat or curved lithium fluoride LiF(200) crystals with optimized capability for the diffraction of the wavelengths of interest. The use of synthetic multilayer structures can be found in state-of-the-art equipment.~~

~~7.3.2 Collimators or LiF(220).~~

~~5.3.2 Collimator Slits, for limiting controlling the divergence of the characteristic X-rays to a parallel bundle when flat crystals are used in X rays. Use per the instrument. For curved crystal optics, no collimator is necessary.~~

~~5.3.3— equipment manufacturer's recommendations.~~

~~7.3.3 Detectors—Sealed, sealed or gas-flow proportional type, scintillation counters or equivalent.~~

~~57.3.4 Vacuum System, providing for the determination of elements whose radiation is absorbed by air (for example, silicon, phosphorus, and sulfur). The system shall consist of a vacuum pump, gage, and electrical controls to provide automatic pump down of the optical path, and to start the analysis at maintain a controlled pressure of, usually 13 Pa (100 μ m Hg) or less, controlled to $\pm 20 \mu$ m.~~

~~5.4 $\sqrt{3}$ Pa ($\sqrt{20}$ m Hg). A helium-flushed system is an alternative to a vacuum system.~~

~~7.4 Measuring System—An, consisting of electronic circuits capable of amplifying and integrating pulses received from the detector tube. The system should be equipped with visual and automatic recording devices.~~

~~6. Reagents~~

~~6.1 Detector Gas (P-10), consisting of 90 % argon and 10 % methane.~~

~~7. Hazards~~

~~7.1 Guidelines on ionizing radiation given detectors. For some measurements, a pulse height selector in *Occupational Health and Safety Standards*⁶ shall be observed at all X-ray emission spectrometer installations. It is also recommended that personnel follow conjunction with the guidelines of safe operating procedures given in the NIST Handbook *X-Ray Protection, HB76*,⁷ the booklet *Radiation Safety Recommendations for X-Ray Diffraction and Spectrographic Equipment*,⁸ #MORP 68-14, by T. M. Moore and D. J. McDonald, 1968, and the U.S. Government Handbook 93, *Safety Standard for Non-Medical X-Ray and Sealed Gamma-Ray Sources*, Part 1, general, or similar handbooks of latest issue.~~

~~7.2 X-ray equipment should detectors may be used only under the guidance and supervision of a responsible, qualified person.~~

~~7.3 Suitable monitoring devices, either film badges or dosimeters, shall be worn by all personnel using the equipment.⁹ To meet local, state, and federal radiation standards, periodic radiation surveys of the equipment for leaks and excessive scattered radiation~~

~~Federal Register, Vol. 36, No. 105, May 29, 1971, Section 1910.96 or of latest issue of Subpart G, available~~

~~⁶ Available from Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20025; or *National Bureau of Standards Handbook 111*, ANSI N43.2-1971-20025.~~

~~⁷ Available from Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20025; Department of Health, Education, and Welfare, Rockville, MD 20850.~~

~~⁸ Available from U.S. Department of Health, Education, and Welfare, Rockville, MD 20850; Seimens Gammasonics, Inc., 2000 Nuclear Drive, Des Plaines, IL 60018.~~

shall be made by a qualified person using an ionization-chamber detector.⁹ required to provide more accurate measurements. The personal film badge survey record, the radiation survey records, and a maintenance record system shall be available upon request.

7.4 Special precautions for the operator shall be posted.

7.5 X-ray caution signs shall be posted near the X-ray equipment and at all entrances to the radiation area.

7.6 Fail-safe “X-ray On” warning lights shall be used at the X-ray tube equipped with an appropriate device.

8. Preparation of Reference Materials Reagents and Samples

8.1 Grind the samples to provide Materials

8.1 *Detector Gas (P-10)*, consisting of a flat, clean area over the entire surface to be exposed to the X-ray beam. Adhere rigorously to the preparation technique established. mixture of 90 % argon and 10 % methane, for use with gas-flow proportional counters only.

9. Reference Materials

9.1 *Certified Reference Materials* are available from the National Institute of Standards national and Technology⁹ and other international sources.

9.2 *Reference Materials* with matrices similar to that of the test specimen and containing varying amounts of the elements to be determined may be used; provided they are have been analyzed in accordance with ASTM standard methods or similar procedures established by the certifying body. These reference materials shall be homogeneous and free of voids and porosity.

9.3 The reference materials shall cover the concentration ranges of the elements being sought. A minimum of three reference materials shall be used for each element. A greater number of calibrants may be required if the analyst chooses to perform mathematical corrections for interelement effects. See Guide E 135361.

10. Preparation of Apparatus

10.1 *Start-up*—Energize the power supply and electronic circuits Hazards

10.1 OSHA Standards for ionizing radiation⁹ shall be observed at least 1/2 h prior to taking measurements.

10.2 *Power Supply*—Adjust the voltage of the power supply to produce secondary fluorescence according to the expression:

$$V_f = 12.350/\lambda K_{abs} \quad (1)$$

where:

V_f = the minimum voltage required for exciting the element, and

λK_{abs} = wavelength of all X-ray emission spectrometer installations. It is also recommended that operating and maintenance personnel follow the *K*-adsorption edge guidelines of the fluorescent element.

10.2.1 Ideally, the safe operating voltage should approximate or exceed $V = 3V_f$. Adjust the procedures given in current to produce rate handbooks and publications from National Institute of secondary fluorescence within the desired limits of statistical precision Standards and detector linearity. Typical operating voltage Technology⁶ and current used by different laboratories for

⁹ A two-stage mechanical pump meeting the requirements can be purchased

⁹ *Federal Register*, Vol 36, No. 105, May 1971, Section 1910.96 or of latest issue of Subpart G, available from Precision Scientific Co., Chicago, IL 60647; Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20025; or Sargent-Welch Scientific Co., Skokie, IL 60076; *National Bureau of Standards Handbook III*, ANSI N43.2-1971.

TABLE 2 Analytical Information for Copper

Element Material	No. of Line Designations	2θ angle, deg	Min SD (s ^M , E 1601)	SD (s _R , E 1601)	Reproducibility Index (R, E 1601)	R _{rel} %
Chromium	K α	69.35	0.2294	LiF 0.001572	0.043	42.9
1	6	0.104	0.0012	0.001572	0.013	12.9
Nickel	K α	48.66	0.1659	LiF 0.001564	0.024	44.8
2	6	0.160	0.0012	0.001564	0.024	14.8
Copper	K α	45.93	0.1542	LiF 0.001682	0.041	21.0
3	5	0.054	0.0013	0.001682	0.011	21.0
Molybdenum	K α	20.29	0.0740	LiF 0.004222	0.035	6.0
4	6	0.584	0.0034	0.004222	0.035	6.0
Manganese	K α	62.97	0.2103	LiF 0.001634	0.045	65.1
5	5	0.023	0.0013	0.001634	0.015	65.1
Columbium (Niobium)	K α	21.40	0.0748	LiF 0.002206	0.062	44.0
6	6	0.441	0.0017	0.002206	0.062	14.0
Cobalt	K α	52.79	0.1790	LiF 0.002723	0.046	4.3
7	6	0.370	0.0021	0.002723	0.016	4.3
8	6	0.311	0.0026	0.003222	0.016	5.1
9	6	3.249	0.0197	0.023712	0.166	5.1
10	6	0.075	0.0010	0.001252	0.016	5.1
11	6	0.132	0.0011	0.0011	0.023	17.3

^AThe 2θ angles represent the theoretical values for a lithium fluoride crystal. The actual position for the peak intensities of the elements should be experimentally determined for each spectrometer.

testing the method are shown in Table 1. The values in Table 1 should U.S. Government Printing Office¹⁰, or similar handbooks on radiation safety.

10.2 X-ray equipment shall be used as a guide only.

NOTE 5—The voltage and current established as optimum for only under the X-ray emission power supply in an individual laboratory is reproduced for subsequent measurements. guidance and supervision of a responsible, qualified person.

10.3 Detector Gas Flow—Adjust the flow of P-10 gas in accordance with the equipment manufacturer's instructions. Some detectors require a rapid flow rate and others only a bubble per second. Optimum rate should Monitoring Devices, either film badges or dosimeters¹¹ may be determined worn by each laboratory; all operating and the detectors should be adequately flushed with detector gas before the instrument is used.

10.4 Loading Samples—Orient the reference materials maintenance personnel. Safety regulations shall conform to applicable local, state, and specimens in the sample chamber so that the relationship between the X-ray beam and the grinding striations are the same for all measurements. The precaution need not be taken when a sample spinner is utilized. federal regulations.

11. Procedure

11.1 Excitation—Expose the sample to primary X radiation in accordance with the conditions described in Section 9.

11.2 Radiation Measurements—Measure the radiation intensities Preparation of the analytical lines listed in Table 2.

11.2.1 Counting—Obtain Reference Materials and record the counting rate Test Specimens

11.1 The analyst must choose a measurement for each element. Fixed time area or fixed count modes can be used. Obtain at least 16 000 counts for diameter from the lowest concentrations within options built into the specification.

11.2.2 Voltage Integration—Obtain spectrometer. All test specimens and record the voltage integration measurement for each element. Fixed time or fixed voltage modes can be used. A minimum reference materials must have a flat surface of 30-s integration time is necessary for greater diameter than the lowest concentrations.

11.2.3 Interference of Chromium on Manganese—Many spectrometers will not completely resolve Mn K α from Cr K β . Care must chosen viewed area.

11.2 Prepare the reference materials and test specimens to provide a clean, flat uniform surface to be exercised in exposed to the interpretation X-ray beam. One surface of manganese intensities for samples with varying chromium concentration. Manganese must a reference material may be determined designated by the producer as the certified surface. The same surface preparation medium shall be used for all reference materials and test specimens.

11.3 Refinish the surface of curves established from the reference materials with a chromium content equivalent and test specimens as needed to eliminathe oxidation.

12. Preparation of Apparatus

12.1 Prepare and operate the sample, or mathematical calculations must be used to correct for chromium interference. spectrometer in accordance with the manufacturer's instructions.

NOTE 6—The use of LiF(220) as 2—It is not within the analyzer crystal may resolve Cr K β and Mn K α sufficiently scope of this test method to simplify prescribe minute details relative to the manganese determination.

12. Calibration, Standardization, and Verification

12.1 Calibration—Irradiate preparation of the calibrants and potential standards in apparatus. For a random sequence,

TABLE 3 Precision and Bias for Cobalt

Element	Material	No. of Samples	Number of Measurements	Coefficient of Variation, %	Minimum Value	Standard Deviation, SD	Relative Standard Deviation, RSD, %	Bias, %		Repeatability, %	Reproducibility, %	
								Mean	Standard Error			
Chromium	X-ray	1	25	0.27	18.55	18.50	2	25	0.37	17.54	17.44	
		1	25	7	0.233	0.0027	2	25	0.00335	0.028	12.2	
		3	25	0.30	17.34	17.24						
		3	25	2	17.34	17.24						
		4	25	0.26	17.25	17.20						
		5	25	0.26	15.91	15.93						
		6	25	0.24	17.78	17.70	7	25	0.20	17.09	-17.10	
		6	25	0.047	0.0013	0.0016167	7	25	0.049	109	173.0	
		8	25	0.33	13.34	13.34						
		9	25	0.26	11.72	11.73						
Nickel	Chemical	1	25	0.45	9.19	9.13						
		2	25	0.62	9.91	9.82						
		3	25	0.70	8.24	8.16						
		4	25	0.54	12.35	12.29						
		5	25	0.53	4.63	4.74						
		5	25	0.029	0.0015	0.001874						
		6	25	2.22	0.24	0.24						
		6	25	0.009	324	0.7						
Copper	X-ray	7	25	2.22	0.29	0.29						
		8	25	3.07	0.21	0.22						
		9	25	2.41	0.31	0.31						
		1	25	0.75	0.27	0.245						
		2	25	2.72	0.16	0.153						
		3	25	2.22	0.21	0.20						
		4	25	4.90	0.10	0.105						
		5	25	1.17	3.33	3.38						
		6	25	4.69	0.074	0.060	7	25	6.85	0.0750.068		
Molybdenum	Chemical	6	25	0.292	0.0030	0.0036687	7	25	0.072	24.5		
		8	25	5.42	0.086	0.078						
		9	25	6.16	0.074	0.065						
		1	25	1.04	0.36	0.35						
		2	25	1.30	0.27	0.26						
		3	25	1.83	0.32	0.30						
		4	25	0.36	2.73	2.73						
		5	25	1.68	0.23	0.23						
		6	25	16.21	0.017	0.019						
		6	25	0.024	0.0015	0.0019						
828	X-ray	7	25	0.94	0.48	0.48						
		7	25	0.006	25.48	0.48						
		8	25	3.85	0.098	0.101	9	25	4.76	0.079	0.074	
		8	25	6	6	0.018	9	25	0.0022	0.002598	0.074	
Manganese	Chemical	1	25	0.88	1.421.41							
		1	25	0.88	122.6							
		2	25	0.92	1.63	1.61	3	25	0.90	1.85	1.78	
		2	25	7	7	0.156	3	25	0.0026	0.003287	0.78	
		4	25	0.53	1.60	1.60						
		4	25	0.53	13.60	1.602						
		5	25	1.62	0.52	0.55						
		5	25	8	0.52	0.55						
		6	25	2.36	0.39	0.40						
		7	25	2.40	0.45	0.47						
021	X-ray	7	25	0.222	0.0015	0.47						
		8	25	0.94	1.09	1.00						
		8	25	0.013	6.09	1.00						
		9	25	1.63	0.45	0.46						
		9	25	6	0.402	0.46						
		Columbium	2	25	0.82	0.70	0.66					
		Columbium0020	2	25	0.00254	0.106	26.5					
		(Niobium)	5	25	1.47	0.34	0.36					
		(Niobium)	5	25	10	6	0.036					
		Cobalt	1	20	1.80	0.22	0.204					
Cobalt2	1	20	0.0017	0.002034	0.04							
19	X-ray	2	20	2.88	0.19	0.20						
		2	20	2.88	60.19	0.20						
		3	20	4.91	0.14	0.13						
		3	20	11	0.14	0.13						
		4	25	2.37	0.36	0.37						
		5	15	7.93	0.044	0.050						
		5	15	0.019	0.0012	0.00						
		6	15	9.32	0.029	0.020						
		6	15	0.007	0.029	0.020						
		1469	745	8.50	0.044	0.031						
	3745	8.50	0.044	0.031	7							
	8	15	14.65	0.025	0.018							
	9	20	6.65	0.046	0.040							

4 These relevant standard deviation values are reproducibility data and were obtained by pooling data from five cooperating laboratories. Repeatability data for the

bracketing these radiations with irradiations of any materials intended for use as verifiers. (A verifier may be used as a calibrant even though it is irradiated only as a verifier.) There will be several calibrants for each element description and for each alloy group spanning specific details concerning the required concentration ranges. Repeat with different random sequences at least three times. Using the averages operation of a particular spectrometer, refer to the manufacturer's manual.

12.1.1 *Start-up*—Turn on the power supply and electronic circuits and allow sufficient time for each point, determine analytical curves as directed in Practice E 305. instrument warm-up prior to taking measurements.

12.2 *Standardization*—Following Tube Power Supply—The power supply conditions should be set according to the manufacturer's recommendations, standardize on an initial setup or any time that it is known or suspected that readings have shifted. Make manufacturers recommendations.

12.2.1 The voltage and current established as optimum for the necessary corrections either by adjusting the controls on the readout or by applying arithmetic corrections. Standardization with X-ray tube power supply in an individual laboratory shall be done any time verification indicates that readings have gone out of statistical control. reproduced for subsequent measurements.

12.3 *Verification*—Verification shall be done at least at Proportional Counter Gas Flow— When a gas-flow proportional counter is used, adjust the beginning flow of a work shift. Analyze verifiers in replicate to confirm that they read within the expected confidence interval, as defined P-10 gas in 12.4.

12.3.1 Check verification after standardizing. If confirmation is not obtained, run another standardization or investigate why accordance with the instrument is malfunctioning.

12.3.2 Repeat equipment manufacturer's instructions. When changing P-10 tanks, the verification at least every 4 h or if detectors should be adequately flushed with detector gas before the instrument is used. After changing P-10 tanks b, check pulse height selector according to the manufacturer's instructions.

12.4 *Measurement Conditions*—The K-L $\gamma_3(K\alpha)$ lines for more than 1 h. If readings each element are not in conformance, repeat the standardization.

12.4 The confidence interval will used. When using a scanning (sequential) spectrometer, locations shall be established from observations of calibrated according to the repeatability of the verifiers manufacturer's guidelines.

12.4.1 *Crystals and determining Detectors*—The following crystals and detectors are used for the confidence level as prescribed in Practice E 876 or by establishing the upper and lower limit of elements indicated:

Element	Crystal	Detector
Chromium	L1, L2	SP, Sc, FP
Cobalt	L1, L2	SP, Sc, FP
Copper	L1, L2	SP, Sc, FP
Manganese	L1, L2	SP, Sc, FP
Molybdenum	L1, L2	Sc
Nickel	L1, L2	SP, Sc, FP
Niobium	L1, L2	Sc
Phosphorus	Ge	FP, SP
Silicon	PET, InSb	Fb, SP
Sulfur	Ge	FP, SP
Titanium	L1, L2	SP, Sc, FP
Vanadium	L1, L2	SP, Sc, FP
L1 = LiF(200),		
L2 = LiF(220),		
SP = Sealed Proportional,		
Sc = Scintillation, and		
FP = Flow Proportional.		

12.4.2 *Counting Time*—Collect a control chart as prescribed in Manual MNL 7. The latter is the preferable approach since it also monitors the consistency sufficient number of counts so that the statistics precision of the measurements and provides a way analysis will not be affected by the variation in the counting statistics. A minimum of maintaining a record 10 000 counts is required for one percent precision of performance. the counting statistics, 40 000 for one-half percent.

13. Calibration and Standardization

13.1 *Calibration (Preparation of Analytical Curves)* —Using the percent concentration conditions given in Section 12, measure a series of each element from reference materials that cover the appropriate required concentration ranges. Use at least three reference materials for each element. Prepare an analytical curve.

NOTE 7—Radiation measurements can be automatically printed curve for each element being determined (refer to Practice E 305). For information on an electric typewriter. The output signals correction of interelement effects in X-ray Spectrometric Analysis refer to Guide E 1361. Information on correction of spectral line overlap in wavelength dispersive X-ray spectrometry can also be entered directly into found in Practice E 1622.

13.2 *Standardization (Analytical Curve Adjustment)* —Using a computer so that control reference material, check the concentrations can be read directly.

13.1.1 If plotting is done in terms calibration of ratio of radiation measured to the X-ray spectrometer at a selected reference, determine frequency consistent with SPC practice or when the ratio of detector gas or major components have been changed. If the observed radiation measurement to calibration check indicates that of the selected reference and read concentration from the spectrometer has drifted, make appropriate analytical curve.

13.1.2 Concentrations can be read from tables prepared from adjustments according to the analytical curves relating radiation measurements and concentrations. instructions in the manufacturer's manual. Refer to Practice E 305 for frequency of verification of standardization.

14. Precision and Bias ²Procedure

14.1 Precision—The precision of this Specimen Loading—Place the reference materials and test specimens in the appropriate specimen holding container. If the spectrometer is equipped with an automated loading device, repeatability may be improved by submitting nine stainless steel alloys to five different laboratories. Using their own reference samples as well as NBS Certified Reference Materials for calibration, these five laboratories analyzed loading and unloading all specimens from the nine unknowns on five different days. same holder. The container shall have a suitable opening to achieve the required precision data are shown in Table 3. an acceptable amount of time. The holder must be equipped to keep the specimen from moving inside the holder.

14.2 Bias—The agreement between results obtained by chemical methods Excitation—Expose the specimen to primary X radiation in accordance with Section 12.

14.3 Radiation Measurements—Obtain and record the counting rate measurement for each element. Either fixed count or fixed time modes may be used. Obtain at least the predetermined minimum counts for all specimens.

14.4 Spectral Interferences—Some X-ray spectrometers will not completely resolve radiation from several metal combinations (for example, molybdenum and sulfur; molybdenum and phosphorus; and iron and cobalt). Therefore, care must be exercised in the interpretation of intensities when both elements are present. Mathematical calculations must be used to correct for the interferences.

14.5 Replicate Measurements—Make a single measurement on each test method specimen. The performance of an X-ray spectrometer is not improved significantly by making multiple measurements on the same surface of the specimen. Confidence in Table 3. the accuracy of analysis will improve by making multiple measurements on freshly prepared surfaces of the same specimen.

15. Calculation of Results

15.1 Using the radiation measurements for the test specimen and the appropriate analytical curves, determine the concentration of the various elements.

15.1.1 If mathematical calculations must be made to correct the concentrations for interelement effects, any one of a number of correction procedures may be employed. Refer to the equipment manufacturer's manual for the recommended procedure for the instrument being used. See Guide E 1361.

16. Precision and Bias

16.1 Precision—An acceptable number of laboratories tested this method in accordance with Practice E 691. Precision data, including repeatability (precision within laboratories), r , and reproducibility (precision between laboratories), R , are provided in Tables 1-12.

16.2 Bias (Accuracy)—Bias data calculated in accordance with Practice E 1060 are included in Table 13.

17. Keywords

157.1 spectrometric analysis; stainless steels; X-ray emission

TABLE 1 Typical Statistical Operating Informing Voltages and for Currents

Test Material	No. of Labs	% Chromium	Min SD ($S_{M, E 1601}$)	Reproducibility SD ($S_{R, E 1601}$)	Reproducibility Index ($R, E 1601$)	R_{rel} %
Voltage, kV (Current, mA)†	6	17.818	0.05258	0.06684	0.306	1.7
1 Chromium	60(45)	60 (24)	50 (40)	20 (8)	40 (8) 0.237	1.5
2 Nickel	60(45)	60 (32)	50 (40)	60 (32)	40 (16) 0.933	3.9
3 Copper	60(45)	60 (32)	50 (40)	60 (32)	40 (24) 0.933	3.9
4 Molybdenum	60(45)	60 (32)	50 (40)	60 (32)	40 (24) 0.640	2.7
5 Manganese	60(45)	60 (32)	50 (40)	60 (32)	40 (24) 0.333	1.8
6 Columbium(Niobium)	60(45)	60 (32)	50 (40)	60 (32)	40 (24) 0.254	12.3
7 Cobalt	60(45)	60 (32)	50 (40)	60 (32)	40 (24) 0.247	1.4
8	60(45)	60 (32)	50 (40)	60 (32)	435 0.435	2.5
9	60(45)	60 (32)	50 (40)	60 (32)	435 0.769	3.9
10	60(45)	60 (32)	50 (40)	60 (32)	435 0.769	3.9
11	60(45)	60 (32)	50 (40)	60 (32)	435 0.293	1.4
	60(45)	60 (32)	50 (40)	60 (32)	435 0.688	3.6

TABLE 4 Statistical Information for Manganese

Test Material	No. of Labs	% Manganese	Min SD ($S_{M, E 1601}$)	Reproducibility SD ($S_{R, E 1601}$)	Reproducibility Index ($R, E 1601$)	R_{rel} %
1	7	0.9656	0.0080	0.0286	0.0800	8.28
2	7	0.4243	0.0035	0.0331	0.0926	21.82
3	7	0.3143	0.0109	0.0495	0.1387	44.12
4	7	1.6587	0.0078	0.0488	0.1367	8.24
5	7	0.7637	0.0049	0.0263	0.0735	9.63
6	7	1.4665	0.0052	0.0840	0.2352	16.04
7	7	1.7348	0.0110	0.0572	0.1601	9.23
8	7	1.6327	0.0100	0.0392	0.1099	6.73
9	7	0.4430	0.0039	0.0350	0.0980	22.11
10	7	4.9410	0.0174	0.1570	0.4396	8.90
11	7	0.9595	0.0054	0.0338	0.0946	9.86

TABLE 5 Statistical Information for Molybdenum

Test Material	No. of Labs	% Molybdenum	Min SD ($S_{M, E 1601}$)	Reproducibility SD ($S_{R, E 1601}$)	Reproducibility Index ($R, E 1601$)	R_{rel} %
1	7	0.433	0.0016	0.001986	0.008	1.9
2	7	0.166	0.0020	0.002473	0.013	8.1
3	5	0.016	0.0014	0.001468	0.009	54.9
4	7	0.464	0.0078	0.008461	0.026	5.7
5	5	0.005	0.0014	0.001495	0.011	235.6
6	7	3.085	0.0060	0.007455	0.090	2.9
7	7	0.566	0.0029	0.003452	0.028	4.9
8	7	0.333	0.0043	0.005506	0.009	2.8
9	7	2.066	0.0070	0.008213	0.170	8.2
10	7	2.205	0.0070	0.008213	0.096	4.4
11	7	2.637	0.0065	0.008128	0.192	7.3

TABLE 6 Statistical Information for Nickel

Test Material	No. of Labs	% Nickel	Min SD (s_{M_i} , E 1601)	Reproducibility SD (s_{R_i} , E 1601)	Reproducibility Index (R, E 1601)	R_{rel} %
1	7	10.938	0.03123	0.04002	0.210	1.9
2	7	2.110	0.01039	0.01334	0.210	10.0
3	6	0.284	0.00382	0.00508	0.056	19.5
4	7	21.175	0.06064	0.07502	0.453	2.1
5	7	9.231	0.04841	0.05806	0.219	2.4
6	7	18.582	0.07075	0.08675	1.623	8.7
7	7	8.156	0.02684	0.03339	0.172	2.1
8	7	10.053	0.0392	0.04989	0.187	1.9
9	7	34.113	0.11373	0.13766	0.467	1.4
10	7	13.247	0.06444	0.0779	0.863	6.5
11	6	3.31	0.0152	0.0609	0.1704	5.2

TABLE 7 Statistical Information for Niobium

Test Material	No. of Labs	% Niobium	Min SD (s_{M_i} , E 1601)	Reproducibility SD (s_{R_i} , E 1601)	Reproducibility Index (R, E 1601)	R_{rel} %
1	7	0.194	0.0008	0.001022	0.025	12.7
2	6	0.015	0.0003	0.000405	0.016	106.2
3	5	0.007	0.0002	0.000317	0.015	203.5
4	7	0.072	0.0004	0.000536	0.020	27.7
5	7	0.943	0.0048	0.00582	0.094	10.0
6	7	1.151	0.0032	0.004043	0.206	17.9
7	7	0.016	0.0003	0.000357	0.017	104.1
8	6	0.015	0.0004	0.000400	0.015	98.8
9	7	0.483	0.0010	0.0010	0.087	18.1
10	7	0.213	0.0008	0.0008	0.031	14.3
11	7	0.067	0.0003	0.0003	0.016	23.9

TABLE 8 Statistical Information for Phosphorus

Test Material	No. of Labs	% Phosphorus	Min SD (s_{M_i} , E 1601)	Reproducibility SD (s_{R_i} , E 1601)	Reproducibility Index (R, E 1601)	R_{rel} %
1	7	0.020	0.008	0.000967	0.003	14.7
2	7	0.023	0.0012	0.001359	0.004	17.0
3	7	0.016	0.0008	0.000913	0.003	17.5
4	7	0.027	0.0029	0.003078	0.008	30.1
5	7	0.020	0.0011	0.001291	0.004	21.3
6	7	0.011	0.0028	0.003007	0.012	108.3
7	7	0.028	0.0013	0.001537	0.005	16.3
8	7	0.021	0.0010	0.001129	0.004	18.9
9	7	0.017	0.0010	0.001284	0.008	47.4
10	7	0.026	0.0045	0.004624	0.013	48.6
11	6	0.025	0.0017	0.001598	0.005	19.7

TABLE 9 Statistical Information for Silicon

Test Material	No. of Labs	% Silicon	Min SD (s_{M_i} , E 1601)	Reproducibility SD (s_{R_i} , E 1601)	Reproducibility Index (R, E 1601)	R_{rel} %
1	7	0.825	0.0110	0.013934	0.065	7.9
2	7	0.552	0.0076	0.009823	0.063	11.4
3	7	0.598	0.0089	0.010898	0.071	11.9
4	7	1.679	0.0159	0.019746	0.080	4.8
5	7	0.502	0.0085	0.011178	0.058	11.6
6	7	1.788	0.0167	0.020035	0.170	9.5
7	7	0.575	0.0179	0.020622	0.056	9.7
8	7	0.395	0.0070	0.008612	0.057	14.6
9	7	0.256	0.0070	0.008622	0.074	29.0
10	7	0.321	0.0065	0.007876	0.061	18.8
11	7	0.321	0.0092	0.011595	0.061	18.8

TABLE 10 Statistical Information for Sulfur

Test Material	No. of Labs	% Sulfur	Min SD (s_{M_i} , E 1601)	Reproducibility SD (s_{R_i} , E 1601)	Reproducibility Index (R, E 1601)	R_{rel} %
1	5	0.0086	0.0016	0.0020	0.0058	64.22
2	5	0.0039	0.0014	0.0025	0.0071	180.79
3	5	0.0169	0.0031	0.0039	0.0109	64.86
4	5	0.0241	0.0017	0.0020	0.0055	23.00
5	5	0.0198	0.0010	0.0020	0.0057	28.91
6	5	0.0225	0.0017	0.0067	0.0187	82.84
7	5	0.2494	0.0044	0.0208	0.0583	23.39
8	5	0.0041	0.0018	0.0023	0.0065	156.32
9	5	0.0024	0.0009	0.0027	0.0076	316.47
10	5	0.0041	0.0019	0.0023	0.0065	157.16
11	5	0.0185	0.0020	0.0043	0.0121	65.58

TABLE 11 Statistical Information for Titanium

Test Material	No. of Labs	% Titanium	Min SD (s_{M_i} , E 1601)	Reproducibility SD (s_{R_i} , E 1601)	Reproducibility Index (R, E 1601)	R_{rel} %
1	7	0.0091	0.0012	0.0053	0.0150	163.92
2	3	0.0008	0.0004	0.0012	0.0034	413.75
3	3	0.0008	0.0004	0.0013	0.0036	415.93
4	7	0.0489	0.0010	0.0040	0.0112	22.93
5	6	0.0014	0.0010	0.0013	0.0037	270.24
6	6	0.0073	0.0093	0.0101	0.0284	384.64
7	6	0.0007	0.0003	0.0007	0.0019	276.07
8	7	0.4355	0.0025	0.0136	0.0383	8.78
9	7	0.0015	0.0004	0.0011	0.0030	193.63
10	7	0.0021	0.0002	0.0011	0.0030	139.99
11	6	0.956	0.0198	0.0136	0.0662	69.22

TABLE 12 Statistical Information for Vanadium

Test Material	No. of Labs	% Vanadium	Min SD (s_{M_i} , E 1601)	Reproducibility SD (s_{R_i} , E 1601)	Reproducibility Index (R, E 1601)	R_{rel} %
1	7	0.028	0.0016	0.001844	0.016	55.7
2	7	0.057	0.0017	0.00085	0.015	26.3
3	7	0.091	0.0013	0.001633	0.016	17.3
4	7	0.091	0.0010	0.001334	0.013	140.0
5	7	0.015	0.0007	0.000828	0.003	19.2
6	7	0.077	0.0010	0.001217	0.019	24.9
7	7	0.071	0.0008	0.001036	0.012	16.7
8	7	0.137	0.0014	0.001828	0.025	18.2
9	7	0.062	0.0011	0.001436	0.013	21.3
10	7	0.231	0.0024	0.003079	0.014	6.1
11	6	0.024	0.0008	0.00983	0.010	43.9

TABLE 13 Bias Information

Element	Assumed True Value, %	Average Spectrometer Value, %	Number of Laboratories	Deviation from Assumed True Value
<u>Chromium</u>	17.81	17.82	6	0.01
	15.64	15.63	6	-0.01
	24.14	23.91	6	-0.23
	23.98	24.01	6	0.03
	18.09	18.15	6	0.06
	2.06	2.06	6	0.00
	17.21	17.24	6	0.03
	17.68	17.66	6	-0.02
	19.81	19.95	6	0.14
	21.17	21.19	6	0.02
	18.90	19.01	6	0.11
<u>Copper</u>	0.104	0.102	6	-0.002
	0.162	0.160	6	-0.002
	0.58	0.58	6	0.00
	0.44	0.44	6	0.00
	0.36	0.37	6	0.01
	0.30	0.31	6	0.01
	3.26	3.25	6	-0.01
	0.075	0.075	6	0.00
	0.14	0.13	6	-0.01
	<u>Cobalt</u>	0.22	0.23	7
0.31		0.29	6	-0.02
0.16		0.16	7	0.00
0.23		0.22	7	-0.01
0.41		0.40	6	-0.01
0.034		0.032	6	-0.002
<u>Manganese</u>	0.96	0.97	7	0.01
	0.42	0.42	7	0.00
	0.315	0.314	7	-0.001
	1.66	1.66	7	0.00
	0.788	0.764	7	-0.024
	1.50	1.47	7	-0.03
	1.68	1.74	7	0.06
	1.63	1.63	7	0.00
	0.46	0.44	7	-0.02
	5.09	4.94	7	-0.15
	0.99	0.96	7	-0.03
<u>Molybdenum</u>	0.164	0.166	7	0.002
	0.46	0.46	7	0.00
	3.15	3.08	7	-0.07
	0.55	0.57	7	0.02
	0.33	0.33	7	0.00
	2.10	2.07	7	-0.03
	2.19	2.20	7	0.01
	2.59	2.64	7	0.05
<u>Nickel</u>	10.88	10.94	7	0.06
	2.16	2.11	7	-0.05
	0.29	0.28	7	-0.01
	21.16	21.18	7	0.02
	9.21	9.21	7	0.00
	18.2	18.6	7	0.4
	8.14	8.16	7	0.02
	10.01	10.05	7	0.04
	34.1	34.1	7	0.0
	13.10	13.25	7	0.15
	3.31	3.30	7	-0.01
<u>Niobium</u>	(.16)	0.194	7	
	0.050	0.072	7	0.022
	0.99	0.94	7	-0.05
	1.22	1.15	7	-0.07
	0.01	0.02	7	0.01
	0.51	0.48	7	-0.03
	0.22	0.21	7	-0.01
	0.07	0.07	7	0.00
<u>Phosphorus</u>	0.021	0.021	7	0.000
	0.026	0.023	7	-0.003

TABLE 13 *Continued*

Element	Assumed True Value, %	Average Spectrometer Value, %	Number of Laboratories	Deviation from Assumed True Value
	0.018	0.016	7	-0.002
	0.029	0.027	7	-0.002
	0.018	0.020	7	0.002
	0.011	0.011	7	0.000
	0.030	0.028	7	-0.002
	0.022	0.021	7	-0.001
	0.018	0.017	7	-0.001
	0.028	0.026	7	-0.002
	0.027	0.025	7	-0.002
<u>Silicon</u>	0.80	0.82	7	0.02
	0.545	0.552	7	0.007
	0.58	0.60	7	0.02
	1.66	1.68	7	0.02
	0.52	0.50	7	-0.02
	1.80	1.79	7	-0.01
	0.58	0.58	7	0.00
	0.40	0.48	7	0.08
	0.24	0.21	7	-0.03
	0.32	0.32	7	0.00
	0.067	0.067	7	0.000
<u>Sulfur</u>	0.0064	0.0086	5	0.0022
	0.001	0.004	5	0.003
	0.015	0.017	5	0.002
	0.024	0.024	5	0.000
	0.019	0.020	5	0.001
	0.025	0.022	5	-0.003
	0.309	0.249	5	-0.060
	0.0018	0.0041	5	0.0023
	0.002	0.002	5	0.000
	0.003	0.004	5	0.001
	0.026	0.018	5	-0.008
<u>Titanium</u>	(.01)	0.0091	7	
	0.050	0.049	7	-0.001
	0.45	0.44	7	-0.01
	0.002	0.002	7	0.000
	0.002	0.002	7	0.000
	0.11	0.10	7	-0.01
<u>Vanadium</u>	0.030	0.028	7	0.002
	0.056	0.057	7	0.001
	0.08	0.09	7	0.01
	0.09	0.09	7	0.00
	0.07	0.07	7	0.00
	0.13	0.14	7	0.01
	0.059	0.062	7	0.003
	0.23	0.23	7	0.00

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