



Standard Test Method for Analysis of Low-Alloy Steels by X-Ray Fluorescence Spectrometry¹

This standard is issued under the fixed designation E1085; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the wavelength dispersive X-ray fluorescence analysis of low-alloy steels for the following elements:

Element	Concentration Range, %
Calcium	0.001 to 0.007
Chromium	0.04 to 2.5
Cobalt	0.03 to 0.2
Copper	0.03 to 0.6
Manganese	0.04 to 2.5
Molybdenum	0.005 to 1.5
Nickel	0.04 to 3.0
Niobium	0.002 to 0.1
Phosphorus	0.010 to 0.08
Silicon	0.06 to 1.5
Sulfur	0.009 to 0.1
Vanadium	0.012 to 0.6

1.2 The values stated in inch-pound units are to be regarded as standard. The values given in parentheses are mathematical conversions to SI units that are provided for information only and are not considered standard.

1.3 *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 precautionary statements are given in Section 10.

2. Referenced Documents

2.1 ASTM Standards:²

E135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials

¹ This test method is under the jurisdiction of ASTM Committee 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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² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

E305 Practice for Establishing and Controlling Atomic Emission Spectrochemical Analytical Curves

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

E1361 Guide for Correction of Interelement Effects in X-Ray Spectrometric Analysis

E1621 Guide for X-Ray Emission Spectrometric Analysis

3. Terminology

3.1 For definitions of terms used in this test method, refer to Terminology **E135**.

4. Summary of Test Method

4.1 The test specimen is finished to a clean uniform surface and then irradiated with an X-ray beam of high energy. The secondary X-rays produced are dispersed by means of crystals, and the intensities are measured by suitable detectors at selected wavelengths. 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. Concentrations of the elements are determined by relating the measured radiation of unknown specimens to analytical curves prepared with suitable reference materials. Either a fixed-channel, polychromator system or a sequential, monochromator system may be used to provide simultaneous or sequential determinations of elements.

5. Significance and Use

5.1 This procedure is suitable for manufacturing control and for verifying that the product meets specifications. This test method provides rapid, multielement determinations with sufficient accuracy to ensure product quality and minimize production delays. The analytical performance data 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 X-ray spectrometer has changed.

5.2 Calcium is sometimes added to steel to effect inclusion shape control in order to enhance certain mechanical properties of steel. This test method is useful for determining the residual calcium in the steel after such treatment.

5.2.1 Because calcium occurs primarily in inclusions, the precision of this test method is a function of the distribution of the calcium-bearing inclusions in the steel. The variation of determinations on freshly prepared surfaces will give some indication of the distribution of these inclusions.

6. Interferences

6.1 Interelement or matrix effects may exist for some of the elements in 1.1. Mathematical corrections may be used to compensate for these effects. Various mathematical correction procedures are commonly utilized. See Guide E1361 and Practice E1621. Any of these procedures is acceptable that will achieve analytical accuracy equivalent to that provided by this test method.

6.2 Because trace amounts of calcium are being determined with this test method, exercise care not to contaminate the specimen. The presence of calcium in the grinding medium will contaminate the specimen to the extent that erratic and incorrect results will be obtained. Therefore, the grinding medium shall be analyzed for calcium and only those materials that are free of calcium shall be used.

7. Apparatus

7.1 Specimen Preparation Equipment:

7.1.1 *Surface Grinder or Sander With Abrasive Belts, Disks, or Lathe*, capable of providing a flat, uniform surface on the reference materials and test specimens.

7.1.1.1 When calcium is to be determined, 240-grit, calcium-free silicon carbide belts or disks shall be used.

7.2 Excitation Source:

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

7.2.2 *X-Ray Tubes*, with targets of various high-purity elements, that are capable of continuous operation at required potentials and currents, and will excite the elements to be determined.

7.2.2.1 For the determination of calcium only chromium target tubes were tested. Other targets may be used provided they produce data that meets the precision and bias in Section 16.

7.3 *Spectrometer*, designed for X-ray fluorescence analysis, and equipped with specimen holders and a specimen chamber. The chamber may 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.

7.3.1 *Analyzing Crystals*, flat or curved crystals with optimized capability for the diffraction of the wavelengths of interest.

7.3.2 *Collimator*, for limiting the characteristic X rays to a parallel bundle when flat crystals are used. Curved crystals do not require a collimator.

7.3.3 *Detectors*, sealed or gas-flow proportional-type, scintillation counters, or equivalent.

7.3.4 *Vacuum System*, providing for the determination of elements whose radiation is absorbed by air, atomic number 20

(calcium) or lower. The system shall consist of a vacuum pump, gage, and electrical controls to provide automatic pumpdown of the optical path and maintain a controlled pressure, usually 13 Pa (100 μ m Hg) or less, controlled to ± 3 Pa (± 20 μ m Hg).

7.4 *Measuring System*, consisting of electronic circuits capable of amplifying and integrating pulses received from the detectors. For some measurements, pulse height analyzers may be required to provide more accurate measurements. The system shall be equipped with an appropriate recording device.

8. Reagents and Materials

8.1 *Detector Gas (P-10)*, consisting of a 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 and Technology³ and other international certification agencies.

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 have been chemically analyzed in accordance with ASTM standard test methods. These reference materials shall be homogeneous, and free of voids or 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.

10. Hazards

10.1 Occupational Health and Safety standards for ionizing radiation (1)⁴ shall be observed at all X-ray fluorescence spectrometer installations. It is also recommended that operating and maintenance personnel follow the guidelines of safe operating procedures given in current handbooks and publications from the National Council on Radiation Protection and Measurements (2-4) and the U.S. Government Printing Office (5), or similar handbooks on radiation safety.

10.2 X-ray equipment shall be used only under the guidance and supervision of a responsible, qualified person.

10.3 *Monitoring Devices*, either film badges or dosimeters may be worn by all operating and maintenance personnel. Safety regulations shall conform to applicable local, state, and federal regulations. Follow all local, state, and federal radiation standards and regulations related to (1) periodic radiation surveys of the equipment for leaks or excessive scattered radiation, or both; (2) personal film badge survey records; (3) radiation survey records; and (4) equipment maintenance records.

10.4 Special precautions for operators and maintenance personnel shall be posted at the equipment site.

³ Office of Standard Reference Materials, National Institute of Standards and Technology (NIST), 100 Bureau Dr., Stop 1070, Gaithersburg, MD 20899-1070, <http://www.nist.gov>.

⁴ The boldface numbers in parentheses refer to a list of references at the end of this standard.

10.5 Radiation caution signs shall be posted near the X-ray equipment and at all entrances to the radiation area, consistent with state and federal regulations.

10.6 Fail-safe “X-Ray On” warning lights shall be used on the equipment.

11. Preparation of Reference Materials and Test Specimens

11.1 Prepare the reference materials and test specimens to provide a clean, flat uniform surface to be exposed to the X-ray beam. For abrasive sanding, select a grit size and use that grit and size exclusively for all reference materials and test specimens. Aluminum oxide and zirconium oxide belts and discs with a grit size of between 60 and 180 have been found suitable.

11.2 Refinish the surface of the reference materials and test specimens as needed to eliminate surface contamination.

12. Preparation of Apparatus

12.1 Prepare and operate the spectrometer in accordance with the manufacturer’s instructions.

NOTE 1—It is not within the scope of this test method to prescribe minute details relative to the preparation of the apparatus. For a description and specific details concerning the 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 instrument stabilization prior to taking measurements.

12.2 *Tube Power Supply*—Adjust the voltage of the power supply to produce optimum conditions.

12.2.1 The voltage and current established as optimum for the X-ray fluorescence power supply in an individual laboratory shall be reproduced for subsequent measurements.

12.3 *Proportional Counter Gas Flow*—When a gas-flow proportional counter is used, adjust flow of the P-10 gas in accordance with the equipment manufacturer’s instructions. When changing P-10 tanks, the detectors should be adequately flushed with detector gas and adjusted before the instrument is used.

12.4 *Measurement Conditions*—The K- α lines for each element are used. The peak location, when using a scanning spectrometer, should be optimized for each instrument.

12.4.1 *Crystals and Detectors*—The following crystals and detectors are used for the elements indicated:

Element	Crystal ^A	Detector ^A
Nickel	L1, L2	SP, Sc, FP
Chromium	L1, L2	SP, Sc, FP
Manganese	L1, L2	SP, Sc, FP
Silicon	PET, InSb	FP
Molybdenum	L1, L2	Sc
Copper	L1, L2	SP, Sc, FP
Vanadium	L1, L2	SP, Sc, FP
Cobalt	L1, L2	SP, Sc, FP
Sulfur	Ge	FP
Niobium	L1, L2	Sc
Phosphorus	Ge	FP
Calcium	L1	FP

^A L1 = LiF(200), L2 = LiF(220), SP = Sealed Proportional, Sc = Scintillation, and FP = Flow Proportional.

12.4.2 *Counting Time*—Collect a sufficient number of counts so that the precision of the analysis will not be affected

by the variation in the counting statistics. A minimum of 10 000 counts is required for 1 % precision of the counting statistics and 40 000 for 0.5 %.

13. Calibration and Standardization

13.1 *Calibration (Preparation of Analytical Curves)*—Using the conditions given in Section 12, measure a series of reference materials that cover the required concentration ranges. Use at least three reference materials for each element. Measure the reference materials at least two different times on each of two separate days. Prepare an analytical curve for each element being determined (refer to Practice E305).

13.2 *Standardization (Drift Correction)*—Using a control reference material, check the calibration of the X-ray spectrometer at a frequency consistent with statistical process control practice, or when the detector gas or major components have been changed. If the calibration check indicates that drift has occurred, make appropriate adjustments in accordance with the instructions in the manufacturer’s manual. Refer to Practice E305 for frequency of verification of standardization.

14. Procedure

14.1 *Specimen Loading*—Orient the reference materials and test specimens in the specimen chamber so that the relationship between the X-ray beam and the grinding striations is the same for all measurements. This is an essential requirement if the spectrometer is not equipped with a specimen spinner, but is not necessary when a spinner is used.

14.2 *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 elemental combinations (for example, molybdenum and sulfur; molybdenum and phosphorus; iron and cobalt). Therefore, exercise care in the interpretation of intensities when both elements are present. Use mathematical calculations to correct for such interferences. See Practice E1621.

14.5 *Replicate Measurements*—Make a single measurement on each test 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 the accuracy of analysis will improve by making multiple measurements on freshly prepared surfaces of the same specimen or by analyzing more than one 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 are required to correct 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 E1361.

16. Precision and Bias

16.1 *Precision*—An acceptable number of laboratories tested this test method in accordance with Practice E691. Precision data, including repeatability (precision within laboratories), r , and reproducibility (precision between laboratories), R , are provided in Table 1.

NOTE 2—Because calcium exists in inclusions and these may not be uniformly distributed, the r and R values can be expected to be high. To demonstrate the precision of this test method without reflecting the variation in inclusion distribution, three samples were analyzed without

resurfacing between analyses. At concentration levels of (7, 27, and 53) $\mu\text{g/g}$, the r values were 0.4, 0.5, and 0.8, respectively. The R values were 3.3, 3.6, and 2.4, respectively. While greater precision is obtained using this technique, it is not recommended as a general practice, as it may not reflect the general distribution of the calcium in the steel.

16.2 *Bias (Accuracy)*—There is no bias indicated in this test method.

17. Keywords

17.1 fluorescence; low-alloy steel; spectrometric; X-ray

TABLE 1 Repeatability and Reproducibility

Sample	Certified Value, %	X	S_r	S_R	r	R
Nickel						
1	0.031 $\pm$ 0.001	0.031	0.0013	0.0061	0.0037	0.0171
2	0.250 $\pm$ 0.004	0.250	0.0024	0.0104	0.0067	0.0292
3	1.30 $\pm$ 0.01	1.317	0.0051	0.0421	0.0141	0.1180
4	0.20 $\pm$ 0.01	0.203	0.0031	0.0093	0.0087	0.0260
5	0.182 $\pm$ 0.005	0.183	0.0026	0.0085	0.0072	0.0239
6	0.202 $\pm$ 0.004	0.203	0.0025	0.0089	0.0071	0.0251
7	2.97 $\pm$ 0.02	3.006	0.0189	0.1133	0.0530	0.3174
8	0.025	0.032	0.0011	0.0045	0.0032	0.0126
9	0.16	0.157	0.0031	0.0064	0.0087	0.0180
Chromium						
1	1.26 $\pm$ 0.01	1.27	0.0044	0.0096	0.0123	0.0270
2	0.25 $\pm$ 0.01	0.258	0.0023	0.0108	0.0064	0.0304
3	0.062 $\pm$ 0.002	0.067	0.0017	0.0092	0.0048	0.0257
4	2.37 $\pm$ 0.03	2.36	0.0159	0.0288	0.0445	0.0806
5	0.157 $\pm$ 0.006	0.156	0.0064	0.0113	0.0179	0.0316
6	0.201 $\pm$ 0.006	0.201	0.0024	0.0099	0.0066	0.0276
7	0.56 $\pm$ 0.008	0.575	0.0038	0.0128	0.0106	0.0360
8	0.015	0.014	0.0010	0.0029	0.0029	0.0081
9	1.96	1.95	0.0319	0.0483	0.0893	0.1353
Manganese						
1	2.11 $\pm$ < 0.01	2.113	0.0065	0.0116	0.0183	0.0326
2	0.91 $\pm$ 0.03	0.913	0.0053	0.0123	0.0148	0.0346
3	0.322 $\pm$ 0.007	0.314	0.0016	0.0025	0.0044	0.0071
4	0.55 $\pm$ 0.01	0.564	0.0036	0.0064	0.0102	0.0179
5	0.97 $\pm$ 0.006	0.997	0.0041	0.0126	0.0114	0.0353
6	0.75 $\pm$ 0.006	0.756	0.0029	0.0102	0.0081	0.0686
7	0.97 $\pm$ 0.02	0.908	0.0054	0.0245	0.0151	0.0686
8	0.025	0.023	0.0006	0.0079	0.0016	0.0221
9	0.325	0.319	0.0034	0.0080	0.0095	0.0223
Silicon						
1	0.019 $\pm$ 0.003	0.026	0.0036	0.0173	0.0102	0.0484
2	0.235 $\pm$ 0.009	0.258	0.0071	0.0544	0.0199	0.1524
3	1.06 $\pm$ 0.01	1.14	0.0247	0.1919	0.0690	0.5373
4	0.18 $\pm$ 0.01	0.195	0.0059	0.0452	0.0166	0.1265
5	0.175 $\pm$ 0.005	0.195	0.0060	0.0439	0.0168	0.1231
6	0.456 $\pm$ 0.005	0.503	0.0100	0.0924	0.0281	0.2588
7	0.22 $\pm$ 0.01	0.234	0.0063	0.0476	0.0176	0.1334
8	0.14	0.166	0.0076	0.0390	0.0214	0.1091
9	0.75	0.821	0.0203	0.1394	0.0567	0.3902
Molybdenum						
1	0.79 $\pm$ 0.01	0.792	0.0021	0.0081	0.0058	0.0225
2	0.090 $\pm$ 0.003	0.089	0.0005	0.0006	0.0013	0.0016
3	0.028 $\pm$ 0.002	0.026	0.0003	0.0009	0.0009	0.0025
4	0.93 $\pm$ 0.01	0.942	0.0025	0.0084	0.0071	0.0235
5	0.087 $\pm$ 0.005	0.089	0.0004	0.0008	0.0011	0.0024
6	0.153 $\pm$ 0.003	0.149	0.0007	0.0013	0.0019	0.0035
7	0.98 $\pm$ 0.01	1.003	0.0023	0.0159	0.0064	0.0444
8	0.005	0.002	0.0002	0.0011	0.0007	0.0032
9	1.35	1.38	0.0091	0.0159	0.0254	0.0444
Copper						
1	0.032 $\pm$ 0.002	0.033	0.0008	0.0022	0.0021	0.0062
2	0.109 $\pm$ 0.002	0.110	0.0010	0.0021	0.0029	0.0059
3	0.425 $\pm$ 0.004	0.428	0.0017	0.0133	0.0048	0.0371
4	0.134 $\pm$ 0.005	0.138	0.0013	0.0028	0.0035	0.0078
5	0.060 $\pm$ 0.003	0.058	0.0009	0.0023	0.0027	0.0064
6	0.153 $\pm$ 0.005	0.152	0.0012	0.0026	0.0033	0.0074
7	0.50 $\pm$ 0.01	0.513	0.0025	0.0209	0.0069	0.0585
8	0.035	0.039	0.0010	0.0027	0.0027	0.0074

Sample	Certified Value, %	X	s _r	S _R	r	R
9	0.10	0.093	0.0019	0.0028	0.0052	0.0078
Vanadium						
1	0.003 ± 0.0006	0.003	0.0005	0.0023	0.0015	0.0065
2	0.072 ± 0.002	0.074	0.0005	0.0043	0.0015	0.0121
3	0.441 ± 0.001	0.442	0.0128	0.0358	0.0358	0.0608
4	0.013 ± 0.000	0.013	0.0006	0.0017	0.0018	0.0048
5	0.002 ± 0.000	0.003	0.0006	0.0024	0.0017	0.0067
6	0.102 ± 0.004	0.102	0.0007	0.0044	0.0021	0.0123
7	0.50 ± 0.01	0.531	0.0040	0.0143	0.0112	0.0401
8		0.002	0.0004	0.0015	0.0012	0.0043
9	0.045	0.038	0.0008	0.0022	0.0023	0.0063
Cobalt						
1	0.179 ± 0.004	0.172	0.0015	0.0277	0.0041	0.0776
2	0.025 ± 0.001	0.027	0.0010	0.0026	0.0028	0.0074
3	0.012 ± 0.002	0.014	0.0009	0.0026	0.0028	0.0074
4	0.011 ± 0.001	0.010	0.0014	0.0072	0.0039	0.0203
5	0.012 ± 0.003	0.011	0.0007	0.0038	0.0020	0.0107
6	0.042 ± 0.003	0.044	0.0009	0.0037	0.0026	0.0102
7	0.030 ± 0.003	0.027	0.0013	0.0047	0.0035	0.0132
8		0.008	0.0010	0.0057	0.0027	0.0160
9	0.01	0.005	0.0008	0.0060	0.0022	0.0167
Sulfur						
1	0.044 ± 0.002	0.050	0.0012	0.0093	0.0034	0.0259
2	0.022 ± 0.000	0.024	0.0016	0.0068	0.0044	0.0190
3	0.0067 ± 0.0006	0.006	0.0007	0.0016	0.0020	0.0045
4	0.030 ± 0.0015	0.025	0.0009	0.0042	0.0025	0.0118
5	0.088 ± 0.0015	0.085	0.0026	0.0191	0.0073	0.0535
6	0.046 ± 0.0015	0.046	0.0015	0.0117	0.0041	0.0328
7	0.0083 ± 0.0005	0.006	0.0007	0.0016	0.0020	0.0045
8	0.002	0.002	0.0008	0.0016	0.0022	0.0045
9	0.039	0.040	0.0014	0.0073	0.0040	0.0205
Niobium						
1	0.003 ± 0.001	0.002	0.0002	0.0008	0.0005	0.0022
2	0.049 ± 0.002	0.049	0.0004	0.0026	0.0010	0.0072
3	0.102 ± 0.002	0.106	0.0007	0.0072	0.0019	0.0203
4	0.002 ± 0.001	0.001	0.0003	0.0007	0.0008	0.0020
5		0.000	0.0002	0.0002	0.0004	0.0007
6	0.030 ± 0.001	0.028	0.0003	0.0018	0.0008	0.0049
7	0.030 ± 0.001	0.028	0.0003	0.0017	0.0009	0.0048
8	0.000	0.00	0.0001	0.0001	0.0002	0.0003
9		0.021	0.0005	0.0017	0.0015	0.0048
Phosphorus						
1	0.005 ± 0.001	0.008	0.0007	0.0019	0.0019	0.0052
2	0.034 ± 0.001	0.035	0.0007	0.0018	0.0019	0.0049
3	0.074 ± 0.002	0.076	0.0008	0.0033	0.0019	0.0092
4	0.018 ± 0.002	0.019	0.0007	0.0017	0.0021	0.0049
5	0.068 ± 0.004	0.067	0.0008	0.0028	0.0023	0.0082
6	0.023 ± 0.001	0.021	0.0005	0.0015	0.0014	0.0041
7	0.056 ± 0.002	0.060	0.0010	0.0021	0.0028	0.0059
8	0.005	0.004	0.0023	0.0023	0.0065	0.0065
9	0.035	0.033	0.0009	0.0027	0.0025	0.0075
Calcium, µg/g						
1	73	69.5	1.98	2.52	5.54	7.06
2	10	9.6	0.47	1.63	1.31	4.57
3	61	61.8	1.53	1.75	4.29	4.69
4	40	37.9	1.68	1.68	4.69	4.69

REFERENCES

- (1) ANSI/NBS Handbook 114, *General Safety Standard for Installations Using Non-Medical X-Ray and Sealed Gamma-Ray Sources*, available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, <http://www.ansi.org>.
- (2) *National Council on Radiation Protection and Measurements Report 102*, <http://www.ncrp.com>.
- (3) ANSI N43.2-2001, *Radiation Safety for X-ray Diffraction & Fluorescence Analysis Equipment*, available from Health Physics Society (HPS), 1313 Dolly Madison Blvd., Suite 402, McLean, VA, 22101, <http://www.hps.org>.
- (4) NTIS Publication PB282500, *Radiation Safety for X-Ray Diffraction and Fluorescence Analysis Equipment*, available from National Technical Information Service (NTIS), 5285 Port Royal Road, Springfield, VA, 22161, <http://www.ntis.gov>.
- (5) Moore, T. M., and McDonald, D. J., *Radiation Safety Recommendations for X-Ray Diffraction and Spectrographic Equipment*, MORP 68-14, 1968, Document Number PB182558, available from National Technical Information Service (NTIS), 5285 Port Royal Rd., Springfield, VA 22161, <http://www.ntis.gov>.

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