Designation: E 1257 – 93 (Reapproved 1998)ε1

Standard Guide for Evaluating Grinding Materials Used for Surface Preparation in Spectrochemical Analysis[1]

This standard is issued under the fixed designation E 1257; 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.

ε[1] NOTE—Footnote 1 was corrected in October 1998.

1. Scope

1.1 This guide gives recommendations for the evaluation of various grinding materials used to prepare the surfaces of specimens to be analyzed by optical emission or X-ray emission spectroscopy.

1.2 *This standard does not purport to address all of the safety problems, 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.*

2. Referenced Documents

2.1 *ASTM Standards:*

E 135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials[2]

3. Terminology

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

4. Significance and Use

4.1 The grinding materials used for the preparation of the surfaces of specimens prior to analysis by optical emission or X-ray emission spectroscopy can contaminate the surface and thus produce erroneous results. This guide provides examples of the effects of these contaminations and recommendations for evaluating grinding materials to eliminate or reduce these effects in spectrochemical analysis.

4.2 The examples given in this guide are not the only contaminations which can occur. Especially in X-ray spectrometry, all phases of the surface preparation should be examined for potential contamination effects.

4.3 Analytical significance of the contaminations observed depends on the needs of the analyst for the particular application at a given concentration level.

5. Evaluation of Grinding Materials by Direct Analysis

5.1 Table 1 shows an example of semiquantitative spectrographic analysis of various grinding belts from different producers. An examination of these analyses identifies the elements most likely to contaminate the surface of the specimen. The more critical the element and the lower its concentration in the specimen, the more important are low-level concentrations in the belts.

5.1.1 For example, using the 80-grit zircon belt in the determination of 0.5 % chromium, the trace level of chromium in the belt should cause no problem, but in the determination of 0.02 % aluminum, that belt probably will cause a problem. In the determination of calcium at ppm levels in steel, even low levels of calcium in the belts cause problems.

5.2 Figs. 1-6 show energy dispersive X-ray analyses of various belts and the same logic applied in 5.1 can be used with these analyses. Major components in the belts will cause greater problems in the determination of these elements.

5.2.1 Direct analysis of the grinding material is particularly useful in such analyses as the determination of calcium in steel, where the analyte is generally too inhomogeneous to use the methods described in Section 6. This analysis requires a virtually calcium-free belt as in Fig. 2.

6. Evaluation of Grinding Materials by Specimen Examination

6.1 The effect of grinding materials depends on the analytical method. In optical emission analysis, the preburn will, in general, volatilize the grinding material left on or driven into the surface (see 6.3). For X-ray emission analysis, the material left on the surface will be analyzed as being specimen material.

6.2 Table 2 shows X-ray emission analyses of a steel specimen after surfacing with various grinding materials. By tabulating the results in this manner, it becomes obvious what problems are occurring from the various grinding materials. Where there is no change from material to material, beyond the precision of the method of analysis and the homogeneity of the material, no contamination has occurred. But where the concentration of a given element appears higher, there has been contamination. Such is the case with the determination of silicon using the silicon carbide belt and the bonded diamond

[1] This guide is under the jurisdiction of ASTM Committee E-1 on Analytical Chemistry for Metals, Ores and Related Materials and is the direct responsibility of Subcommittee E01.20 on Fundamental Practices and Measurement Traceability.

Current edition approved Jan. 15, 1993. Published March 1993. Originally published as E 1257 – 88. Last previous edition E 1257 – 88.

[2] *Annual Book of ASTM Standards*, Vol 03.05.

TABLE 1 Semiquantitative (Spectrographic) Analysis of Grinding Belt Abrasives

Concentration,%	80-Grit Silicon Carbide			80-Grit Alumina			80-Grit Zircon
	No. 1	No. 2	No. 3	No. 1	No. 2	No. 3	
10+	Si	Si	Si	Al	Al, Ca	Al	Al, Ca, Zr
1–10	Ca	Ca			Ti		Si, Na, Fe
0.1–1	Ba, Mg	Fe, Al, Na		Mg, Si, Ca, Ti	Fe, Si, Na	Ca	Ti, Zn
0.05–0.5	B		Fe, B		Mg		
0.01–0.1	Mn, Na	B, Mg	Al	Ba, B	Zr	Na	Mg
0.005–0.05	V, Cu, Ti, Ni	Mn, Ti	V, Ca, Na, Ni	Mn, Zr, Cu, Na	B	B, Fe, Si	B, Mn, Sr
Trace–0.01	Mo, Zr, Sr	Ba, V, Zr, Cu, Ni, Sr	Ba, Mn, Mg, Pb, Cr, Zr, Cu, Ti, Sr	Ni	Ba, Mn, Cr, V, Cu, Ni, Sr	Mn, Mo, Cu, Sr, Mg	Ba, Pb, Cr, V, Mo, Cu

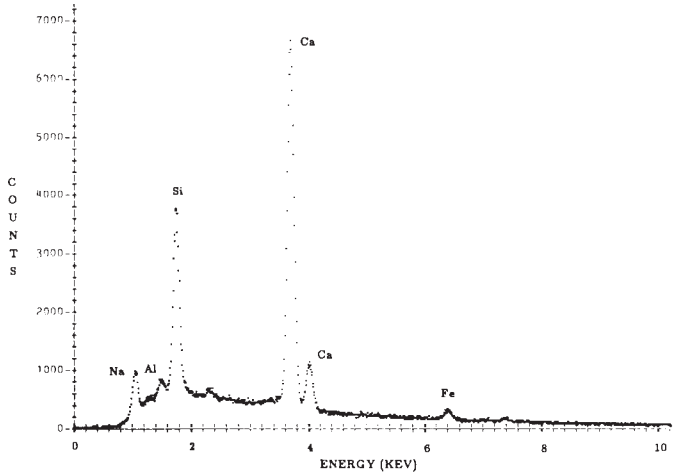

FIG. 1 EDX Analysis of Silicon Carbide Grinding Belt, 60-Grit

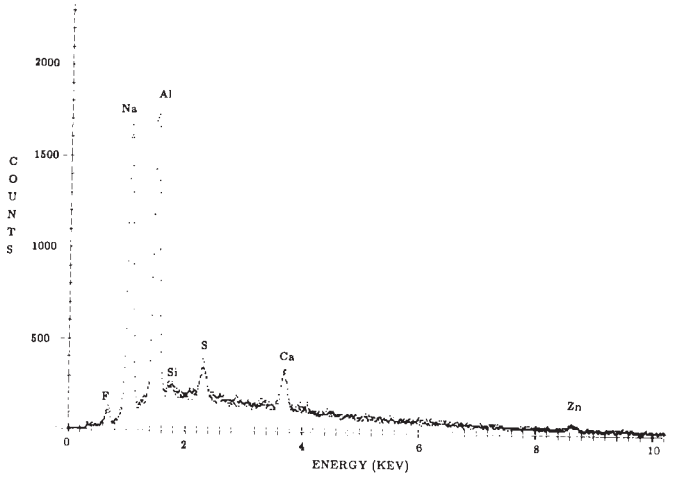

FIG. 3 EDX Analysis of Alumina Grinding Belt, 60-Grit

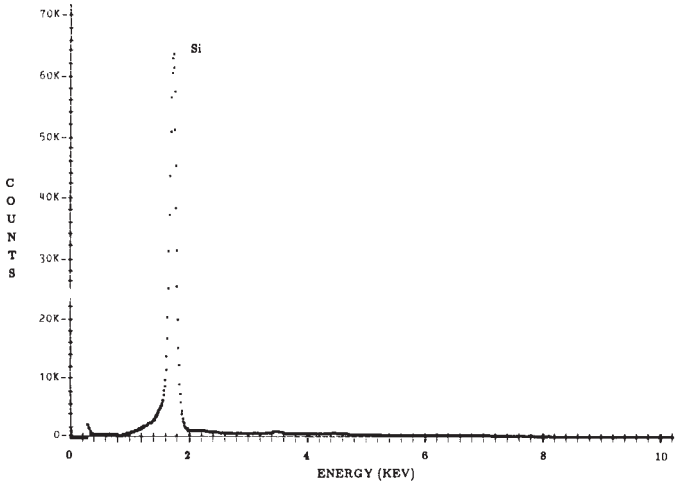

FIG. 2 EDX Analysis of Silicon Carbide Grinding Belt, 240-Grit

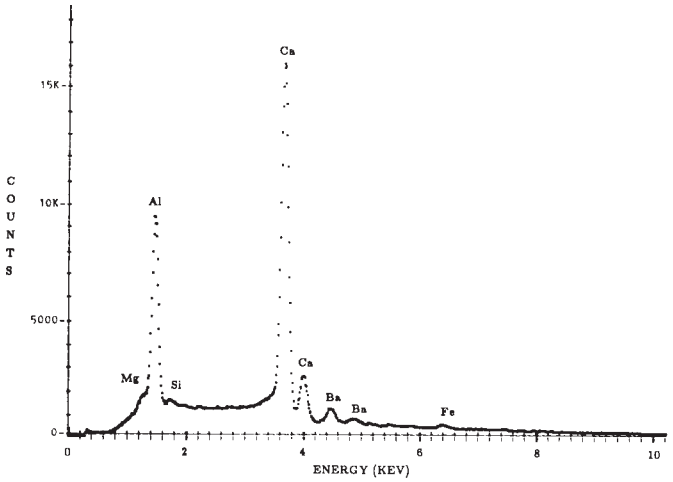

FIG. 4 EDX Analysis of Alumina Grinding Belt, 120-Grit

wheels; with the determination of zirconium using zircon belts; with the determination of aluminum using the alumina and zircon belts, the bonded diamond wheels, and the surface grinder; and with the determination of nickel using the metal bonded diamond wheel.

6.2.1 This method requires the use of homogeneous materials to attain the required precision to detect low levels of contamination. Materials should be examined by replicate determinations using the same grinding material beforehand to assure that they are homogeneous. If inhomogeneity seems to be excessive for one element, that may come from the grinding material, for example, silicon from silicon carbide, repeat the examination using a different grinding material.

6.2.2 Generally this method is convenient because it determines the contamination which actually occurs in the type of material being analyzed and does not require analysis of the grinding material itself. An exception is the calcium determination mentioned in 5.2.1.

6.3 In optical emission analysis, a finite time is required to clean the specimen surface (by volatilization). Intensity-time studies show that preburn periods as long as 20 s can be required to reach stable intensity ratios for elements comprising the grinding matrix. Fig. 7 shows time studies for carbon in a specimen surfaced with silicon carbide, alumina, zircon, and

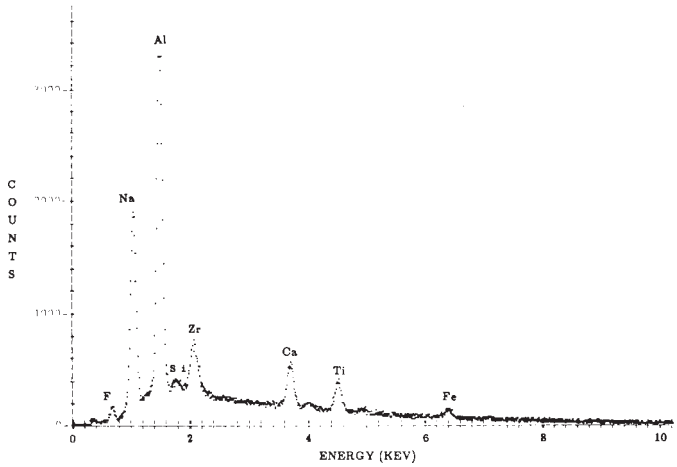

FIG. 5 EDX Analysis of Zircon Grinding Belt, 60-Grit

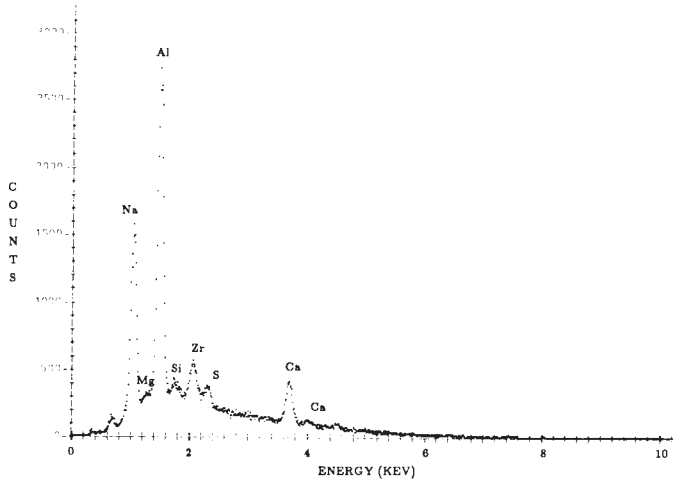

FIG. 6 EDX Analysis of Zircon Grinding Belt, 120-Grit

a lathe. Fig. 8 shows the time studies for aluminum. Under the conditions used for these tests, a total of 4800 discharges occurred in the 20-s period. Sources running at lower rates may require longer preburn periods. Fig. 8 shows that, compared to a specimen surfaced with lathe, the aluminum value found at the end of the preburn period can be higher on the alumina and zircon ground specimens. Analytical significance will depend on the concentration level. Similar results can be shown for other elements, especially silicon and zirconium under the conditions used. Different source conditions may show different results.

6.3.1 Although the preburn generally removes most of the grinding material, in analyses where the highest accuracy is desired, grinding materials containing the analyte element should be avoided.

7. Keywords

7.1 grinding material; specimen preparation; spectrochemical analysis

TABLE 2 X-Ray Fluorescence Analysis of a Steel Specimen Using Various Grinding Media

	Apparent Concentration, %, Using						
Element	Si C Belt	Alumina Belt	Zircon Belt	Resin Bonded Diamond	Metal Bonded Diamond	Diamond Paste	Surface Grinder
Molybdenum	0.057	0.056	0.058	0.057	0.057	0.057	0.058
Niobium	0.034	0.032	0.032	0.033	0.032	0.032	0.033
Copper	0.315	0.310	0.320	0.316	0.317	0.316	0.317
Nickel	0.297	0.296	0.292	0.295	0.322[A]	0.296	0.295
Cobalt	0.014	0.011	0.010	0.011	0.010	0.011	0.012
Manganese	1.39	1.40	1.39	1.40	1.39	1.40	1.40
Chromium	0.197	0.193	0.197	0.196	0.195	0.196	0.195
Vanadium	0.061	0.059	0.060	0.059	0.060	0.059	0.059
Titanium	0.024	0.025	0.024	0.024	0.025	0.024	0.024
Phosphorus	0.012	0.011	0.012	0.011	0.012	0.012	0.011
Silicon	0.444[A]	0.234	0.234	0.245[A]	0.293[A]	0.235	0.236
Antimony	0.006	0.005	0.005	0.005	0.006	0.005	0.006
Tin	0.023	0.022	0.023	0.023	0.022	0.023	0.022
Aluminum	0.016	0.070[A]	0.085[A]	0.026[A]	0.090[A]	0.015	0.030[A]
Zirconium	0.050	0.051	0.066[A]	0.051	0.050	0.051	0.050

[A]Elements that exhibit contamination from grinding media.

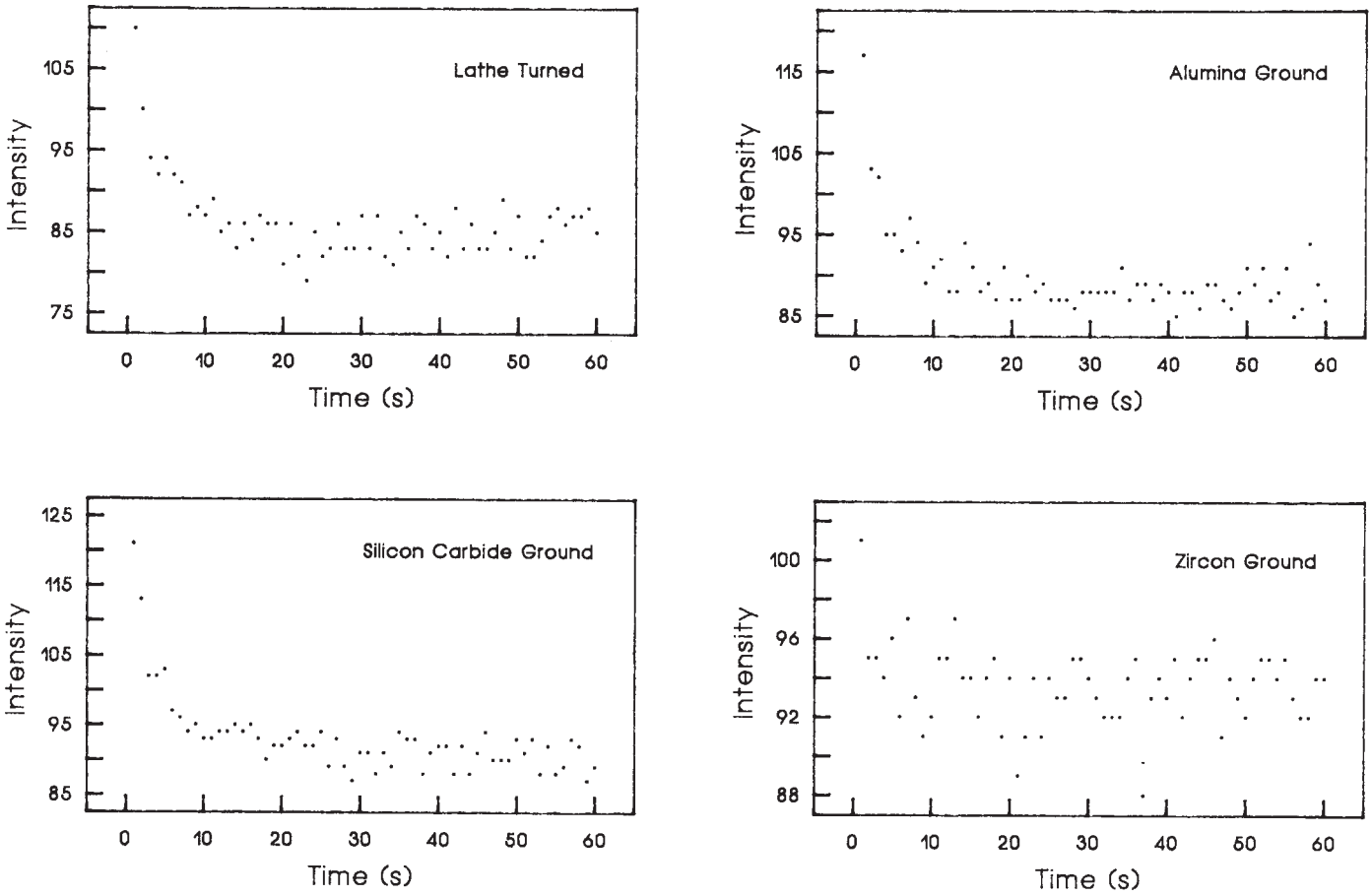

FIG. 7 Time Studies: Carbon—Optical Emission Analysis of High-Purity Iron

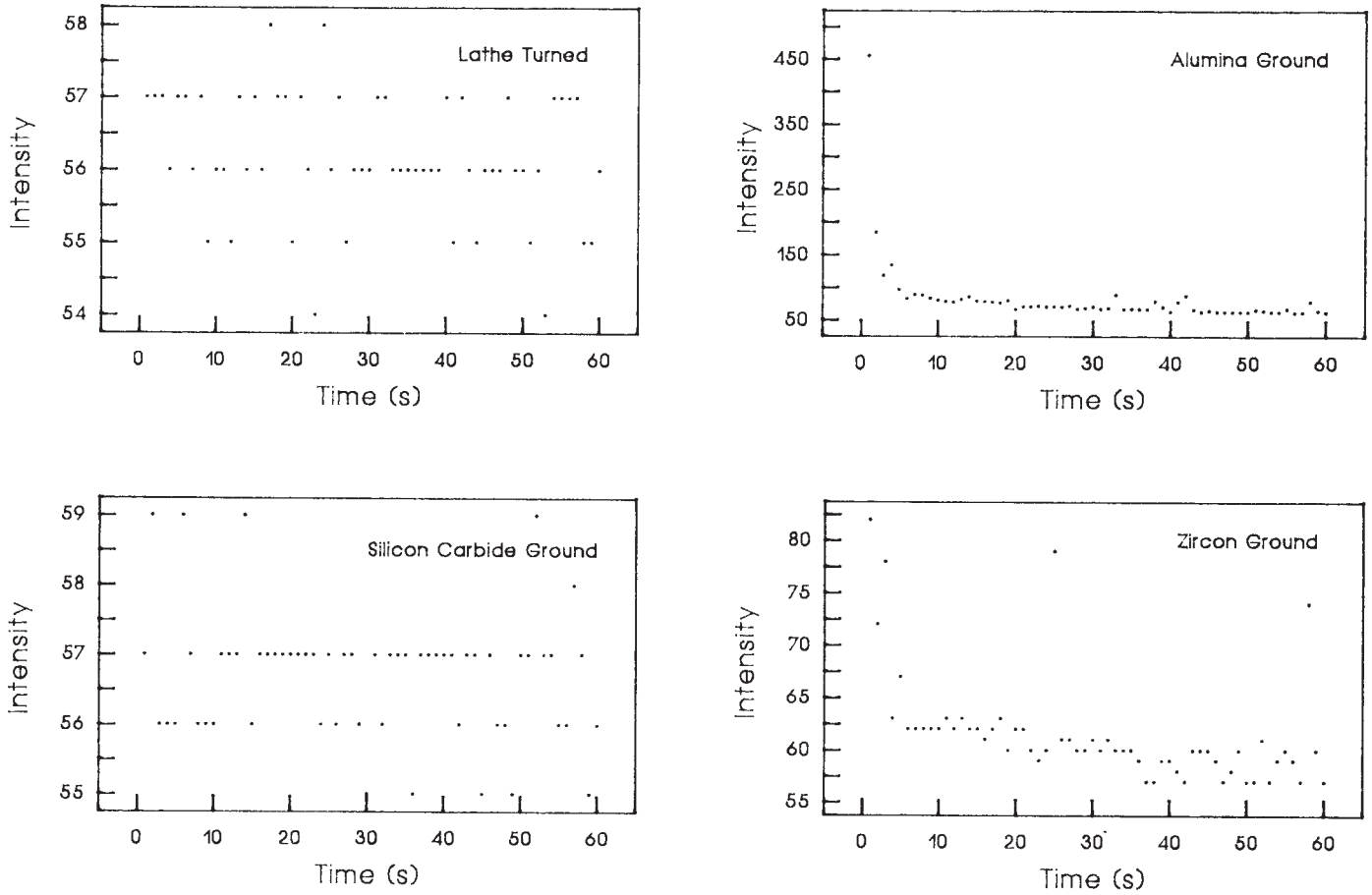

FIG. 8 Time Studies: Aluminum—Optical Emission Analysis of High-Purity Iron