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

This standard is issued under the fixed designation E1257; 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 guide covers recommendations for the evaluation of various grinding materials used to prepare the surfaces of specimens to be analyzed by ~~optical~~ spark atomic emission or X-ray ~~emission spectroscopy.~~fluorescence spectrometry.

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:*[2]

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

3. Terminology

3.1 For definitions of terms used in this guide, refer to Terminology E135.

4. Significance and Use

4.1 The grinding materials used for the preparation of the surfaces of specimens prior to analysis by ~~optical~~ spark atomic emission or X-ray ~~emission spectroscopy~~fluorescence spectrometry 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~~that 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.~~composition.

5. Evaluation of Grinding Materials by Direct Analysis

5.1 Table 1 shows an example of semiquantitative ~~spectrographic~~spectrometric 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~~ mass fraction in the specimen, the more important are low-level ~~concentrations~~contaminants 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~~μg/g 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.

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

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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.

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~~

TABLE 1 Semiquantitative (Spectrographic) Analysis of Grinding Belt Abrasives

Composition, %	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

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

Element	~~Apparent Concentration,~~ %, Using						
	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~~

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

Element	Apparent Composition, %, Using						
	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.

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.

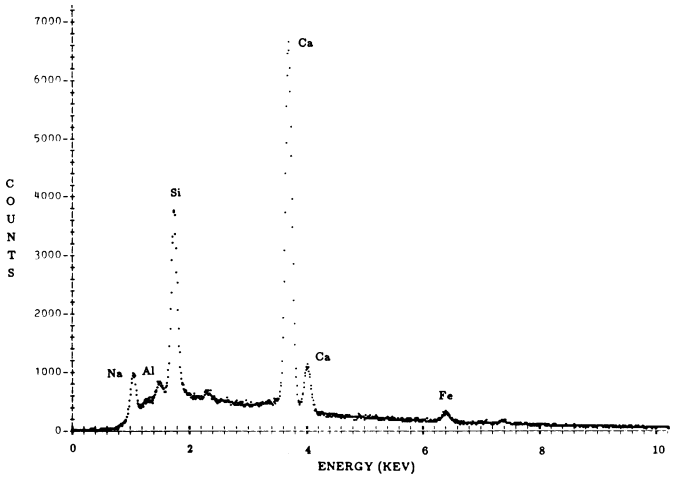

FIG. 1 EDX Analysis of Silicon Carbide Grinding Belt, 60-Grit

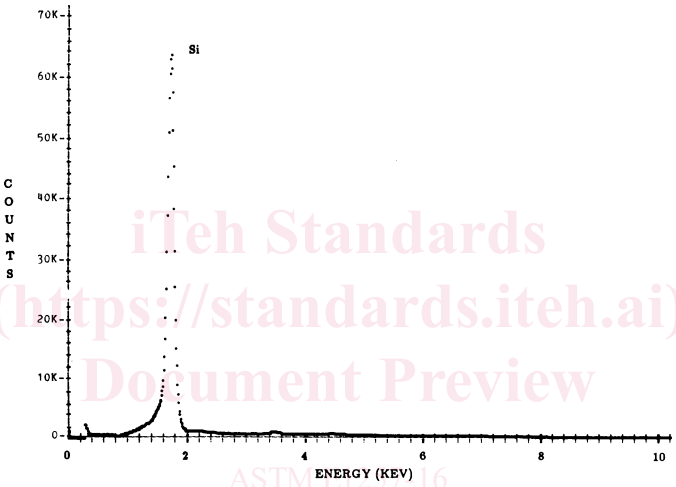

FIG. 2 EDX Analysis of Silicon Carbide Grinding Belt, 240-Grit

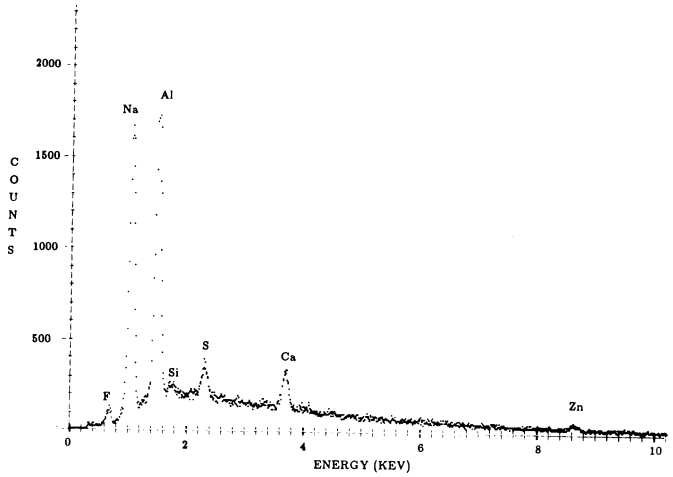

FIG. 3 EDX Analysis of Alumina Grinding Belt, 60-Grit

6. Evaluation of Grinding Materials by Specimen Examination

6.1 The effect of grinding materials depends on the analytical method. In ~~optical~~ spark atomic 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~~fluorescence analysis, the material left on the surface will be analyzed as being specimen material.