BUCK Scientific

Application Note #AA3009

Analytical Methodology for the Characterization of Steels and Iron Alloys by Atomic Absorption Analysis

The analysis of irons and steels has always been a very important job in the industrial and commercial environment. The grading of steels and iron alloys is critical to characterizing their respective strengths to corrosion and stress. There are also many protocols for the several hundred types of steels available. Fe is always the primary component, with elements such as Ni and Cr as major consstituents, and Mn, Zn, B, P, Cu, V, Ti, Co, Pb, Mo, Si, and Sn as essential minor elements. Much of the standard methods of analyses have been documented and issued by agencies like the ASTM (American Society of Testing and Materials), and the ISO (International Standards Organization). Some of the procedures listed here are taken from the ASTM Standards, volume 03.05 (Chemical Analysis of Metals); methods E350 (Low Alloy Steels), E351 (Cast Irons), E352 (Tool and High Alloy Steels, and E353 (Stainless Steels). All these standards employ Flame Atomic Absorption Spectrophotometry (FAAS).

Preparation of the samples and calibration of the instrumentation is actually quite easy using the Buck Scientific Model 210VGP AA system. The unique components allow interference-free measurements at high levels of sensitivity and precision. Aspiration of the sample by the high efficiency nebulizer provides tremendous sample throughput, and the proprietary "in-line" Deuterium (D_2) Lamp Background Corrector gives the highest energy of any commercially available system. These features combined with the single-mirror, high-resolution monochromator and low noise electronics create an integrated analytical tool of remarkable power.

An example of a "universal" sample preparation technique is listed below. This procedure gives complete recovery of all components with no losses or chemical interferences, and can be used for Certified Standards from ASTM, NIST / NBS, BCS, IUPAC, or other regulatory agencies. Two basic dilutions cover the range of concentrations from 0.001% up to 25.0%.

Preparation of Irons and Steels for Flame AAS Analyses: Aqua Nasty ™

[1] Weigh 1.0gm sample chips into a tared 4oz HDPE Nalgene bottle.

[2] Add 10ml HCl, 2ml HNO_3, 1ml HF, and 1gm $K_2S_2O_8$. Heat in water bath or in microwave oven for 5 – 10 minutes.

[3] Dilute to 100grams final weight, or cool and dilute to 100ml volume. This is the 1% solution (1:100 dilution factor) for minor elements.

[4] Weigh or pipet 1gm or 1ml of this to 100ml to make 100 ppm solution (1:10,000 dilution factor) for Major components.

The Buck 210VGP AA can accurately determine these major and minor elements, depending on the dilution used, with typical precision levels of 1% or better if using bracketted standards. The entire procedure can determine over 40 elements per hour for high turnover and high quality results.

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Analysis of Irons and Steels by Atomic Absorption Analysis

Samples: Standard Reference Materials from NIST / NBS, ASTM, and BCS
Preparation: "Aqua Nasty" method previously listed
Calibration: Buck Certified Atomic Absorption Standards
Instrument: The Buck 210VGP Atomic Absorption Spectrophotometer
Conditions: Air / Acetylene flame, Integrate mode, Normal parameters
N_2O flame for Sn, Si, Ti, V, and Mo

NOTE – All values listed below are in Weight Percent (%wt) in the original Steel sample; [K] = Known assay, [M] = measured data.

Element	Cast and Pig Irons		Stainless #316S		Tool Steel No. 3	
	[K]	[M]	[K]	[M]	[K]	[M]
Cr	2.45	2.48	18.1	18.0	0.69	0.70
Ni	1.17	1.19	12.9	13.0	0.21	0.20
Mn	0.79	0.80	1.77	1.75	0.18	0.19
Co	0.54	0.56	0.27	0.25	4.82	4.80
Al	0.35	0.33	0.88	0.89	0.22	0.24
Cu	0.67	0.67	0.24	0.23	.079	.080
Ag	.011	.010	.009	.008	-	-
Mo	0.48	0.50	2.11	2.10	0.48	0.47
Ti	0.17	0.15	0.44	0.45	-	-
Si	2.86	2.83	0.66	0.68	0.22	0.21
V	0.15	0.16	0.21	0.22	0.13	0.12
Pb	0.17	0.16	.028	.029	.059	.060
Zn	.088	.090	-	-	.028	.029
Sn	.013	.011	.035	.036	.029	.030
Mg	.097	.098	-	-	.040	.039
Bi	.046	.045	-	-	-	-
As	0.12	0.11	-	-	-	-

The above data shows the powerful flexibility and stability of the Buck 210 system for the wide-ranging requirements of the Iron and Steel industry. The excellent correlation between the Known and Measured values ranges from 0.6% to 3.0% (average RSD = 1.6%), exemplifying the high precision of the instrument. The fact that a single preparation gives superb reproducibility on three (3) widely varying metals, with a range of difficult elements, reveals the accuracy of the chemistry. The above components provide for an unmatched system in economy and performance.

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