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Bureau of Mines Report of Investigations/1984

Rapid Identification of Stainless Steel and Superalloy Scrap

By R. D. Brown, Jr., William D. Riley, and C. A. Zieba

UNITED STATES DEPARTMENT OF THE INTERIOR

Report of Investigations 8858

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UNITED STATES DEPARTMENT OF THE INTERIOR
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UNIT OF MEASURE ABBREVIATIONS USED IN THIS REPORT

°C	degree Celsius	min	minute
g	gram	mL	milliliter
h	hour	mm	millimeter
in	inch	nm	nanometer
L	liter	pct	percent
µg	microgram	ppm	part per million
µg/ml	microgram per milliliter	wt pct	weight percent

RAPID IDENTIFICATION OF STAINLESS STEEL AND SUPERALLOY SCRAP

By R. D. Brown, Jr.,[1] William D. Riley,[2] and C. A. Zieba[3]

ABSTRACT

This Bureau of Mines report describes a new methodology for identifying and sorting scrap metals using two modern portable instruments: a thermoelectric sorter and a hand-held emission spectroscope. The method, tested on a 27-sample stainless steel and superalloy array, makes it possible to group and/or identify a typical mixture of high-value metal scrap in two or three steps, whereas as many as seven or eight steps are needed to perform the same separations using chemical spot tests.

A portable fluorescent X-ray analyzer identified 19 of the 27 alloys in the sample array by name or type; these alloys are included in the microprocessor memory. The remaining alloys were identified by using the analyzer in its elemental analysis mode and comparing the results with alloy specification chemistry.

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INTRODUCTION

Conservation of strategic metals such as nickel, cobalt, and chromium, and the minimization of dependence on imports through improved methods of recycling scrap materials are among the objectives of the Bureau of Mines Materials and Recycling Technology program. One aspect of this research has been to improve the efficiency of recycling by introducing and evaluating new technologies to identify and sort scrap metals (9, 11, 13).[4] Because the United States is almost totally dependent on imports of the critical metals nickel, cobalt, and chromium, emphasis has been given to the reduction of downgrading and discarding complex scrap materials containing these elements. The traditional scrap sorting techniques such as object and color recognition, apparent density, magnetic properties, spark testing, and chemical spot tests (8-9, 14) are usually inadequate for sorting the complex materials containing these elements into discrete alloys. Contacts with superalloy recyclers indicate that separation into discrete alloys is ultimately the only acceptable way of recycling these alloys without recovering individual elements through pyrometallurgical or hydrometallurgical processes (1).

Reliable identification techniques are necessary because a penalty is exacted for contamination of a scrap product. Table 1 lists National Association of Recycling Industries-Institute of Scrap Iron and Steel (NARI-ISIS) specifications for some stainless steel and nickel-base alloys. The classification "Sabot," for example, calls for clean 18-8 grade stainless steel clips and solids with minimums for nickel (7.0 pct) and chromium (16 pct); and maximums for molybdenum (0.5 pct), copper (0.5 pct), phosphorus (0.045 pct), and sulfur

[4]Underlined numbers in parentheses refer to items in the list of references preceding the appendix.

TABLE 1. - Some typical NARI-ISIS classifications

Classification	Definition	Specification
Rusten.....	11 to 14 pct straight chrome stainless.	11 to 14 pct Cr, 0.03 pct P (max), 0.03 pct S (max), 0.50 pct Ni (max). Otherwise free of harmful contaminants. Material to be prepared to individual consumer's specifications.
Rusthirty.....	14 to 18 pct straight chrome stainless.	14 to 18 pct Cr, 0.03 pct P (max), 0.03 pct S (max), 0.50 pct Ni (max). Otherwise free of harmful contaminants. Material to be prepared to individual consumer's specifications.
Obole[1].....	Ferronickel-chrome.....	12 pct Cr (min), 12 pct Ni (min), 0.50 pct Cu (max). Free of other foreign elements. Material shall be sold on basis of description and analysis. This category excludes all stainless steel grades that are covered elsewhere.
Sabot.....	18-8 stainless steel (clean scrap).	7.0 pct Ni (min), 16.0 pct Cr (min), 0.50 pct Mo (max), 0.50 pct Cu (max), 0.045 pct P (max), 0.03 pct S (max). Otherwise free of harmful contaminants. Material to be prepared to individual consumer's specifications.

[1]ISIS specification only.

(0.03 pct); and otherwise free of harmful contaminants. Within the 300 series, type 316 stainless steel contains 2 to 3 pct Mo. Other less-common grades such as type 317 also have significant amounts of molybdenum. To meet the Sabot specification, these grades must be removed. In this case, there is an additional incentive for separation because of the high value of molybdenum.

There are several ways to identify scrap metals: by simple comparison to known samples, by group identification, by alloy identification, by qualitative analysis, and by quantitative analysis. Each way, respectively, requires greater accuracy in chemical analysis for successful identification. Since alloy compositions consist of ranges rather than exact compositions, alloys can be identified from elemental analysis of lower precision and accuracy than is ordinarily expected from an analytical laboratory. The sorter can often identify the alloy by determining the amount of only a few of the elements present in a given sample. One alloy can sometimes be picked out of a group on the basis of only one element. The group identification, for example, Sabot, requires still less inherent accuracy in the analysis because the group identification can be made with greater latitude in the actual chemical analysis. Comparison analysis gives the least information--that is, are the standard and sample the same?--but, in some cases, this is enough. The stainless steels again provide a good example. Assume that it has been established that the unknown is 18-8, one of the 300 series. Semiquantitative chemical analysis and comparison with alloy specifications will determine the alloy of the unknown. The presence or absence of 2 to 3 pct Mo will establish whether the sample is one of the high-molybdenum grades (for example, 316 or 317) or not. Table 2 lists the methods studied and how well they supply the required information.

Approximately 25,000 tons of superalloy scrap and 140,000 tons of stainless steels are downgraded yearly during recycling (1, 4). This downgrading represents a loss in inherent value of approximatly $100 million for the superalloys and $225 million for the stainless steels. Scrap identification and segregation are necessary first steps to reduce downgrading.

Techniques such as thermoelectric response, emission spectroscopy, and fluorescent X-ray spectrography offer potential to improve the accuracy and ease with which identification can be accomplished. This report describes how these techniques can be used to identify and sort mixed alloys into alloy groups and into specific alloys. Each of these techniques can be used singly with some success, particularly X-ray spectrography. A combination method that enhances the individual sorting efficiency of the thermoelectric and emission spectroscopy methods has been devised. A flowsheet for this combination method, as applied to stainless steels and superalloys, is presented. A rapid dissolution technique and subsequent atomic absorption analysis procedure for checking the results of more rapid instrumental methods is presented in the appendix.

TABLE 2. - Information grid for methods tested

	Thermoelectric sorter	Portable emission spectroscope	X-ray spectrograph
Elemental analysis.....	Usually no....	Sometimes.....	Yes
Alloy identification...	Sometimes.....	Often.....	Yes
Group identification...	Yes.....	Yes.....	Yes
Element presence.....	No.....	Yes.....	Yes
Comparison.....	Yes.....	Yes.....	Yes

EXPERIMENTAL PROCEDURE

SPECIMEN PREPARATION

Stainless steel and nickel-, iron-, or cobalt-base alloy samples, typical of those alloys prevalent in current scrap operations, were used in this study and are listed in table 3. In practice, alloys are specified by ranges of composition, maximum allowed, minimum required, or "balance" for each element present. The "nominal" values in the table represent the middle of the allowed range or values most often used.

All of the identification techniques used in this study can be interfered with by mill scale and other surface impurities. Therefore, unless it was clean,

TABLE 3. - Major element concentration of alloys used in this study

Alloy class	Major alloying elements, wt pct (nominal)							Others
	Fe	Ni	Cr	Mo	Co	Cu	Ti	
Stainless steel:								
201.....	70	5	18	n.m.	n.m.	n.m.	n.m.	Mn
301.....	73	7	17	n.m.	n.m.	n.m.	n.m.	Mn
303.....	72	9	18	n.m.	n.m.	n.m.	n.m.	Mn
304.....	69	9	19	n.m.	n.m.	n.m.	n.m.	Mn
316.....	72	10	16	2.5	n.m.	n.m.	n.m.	Mn
321.....	72	9	18	n.m.	n.m.	n.m.	5	Mn
347.....	70	9	17	n.m.	n.m.	n.m.	n.m.	Mn, Ta + Cb
Nickel-base alloys:								
Monel 400.....	1.0	67	n.m.	n.m.	n.m.	31	n.m.	Mn
Monel K500.....	1.0	67	n.m.	n.m.	n.m.	29	.6	Al, Mn
Inconel 600.....	8.0	75	16	n.m.	n.m.	n.m.	n.m.	Cu, Mn
Inconel 625.....	2.5	61	22	9.0	n.m.	n.m.	.2	Cb, Al, Mn
Inconel X-750.....	7.0	70	15	n.m.	n.m.	n.m.	2.5	Cu, Cb, Al, Mn
Inconel 718.....	19	53	19	3.0	1.0	n.m.	.9	Cu, Cb, Al, Mn
Hastelloy B-2.....	2.0	65	1.0	28	1.0	n.m.	n.m.	Mn
Hastelloy S.....	1.0	66	16	15	n.m.	n.m.	n.m.	Mn
Hastelloy C-276...	6.0	55	16	16	2.5	n.m.	n.m.	Mn, W
Hastelloy G.....	19	42	22	6.5	2.5	n.m.	n.m.	Mn, Ta, Cb
Nickel 200.....	n.m.	99.0	n.m.	n.m.	n.m.	n.m.	n.m.	
Nickel 201.....	n.m.	99.0	n.m.	n.m.	n.m.	n.m.	n.m.	
Nimonic 75.....	3.0	75	19	n.m.	n.m.	n.m.	.4	Mn
Nimonic 80A.....	n.m.	75	19	n.m.	n.m.	n.m.	2.4	Al, Mn
Nimonic 90.....	n.m.	59	19	n.m.	16.5	n.m.	2.4	Al, Mn
Incoloy 800.....	46	32	21	n.m.	n.m.	n.m.	n.m.	Mn
Incoloy 825.....	30	42	21	3.0	n.m.	2.2	n.m.	Mn
Iron-base alloy:								
Haynes 20 MOD.....	46	26	22	4.0	n.m.	n.m.	.4	
Cobalt-base alloys:								
Haynes 188.....	3	22	22	n.m.	36	n.m.	n.m.	W, Mn
Haynes 25.....	2	10	20	n.m.	51	n.m.	n.m.	W, Mn

n.m. Not major.

each sample was prepared by wet grinding using 240-grit silicon carbide abrasive belts, followed by degreasing with acetone and air drying. Wet grinding was chosen to prevent any heat-related structural changes that might affect the thermoelectric measurements. Since many critical applications of cobalt- and nickel-base alloys require wear- or oxidation-resistant coatings, it is good procedure to remove the surface layer before identification is attempted whenever the presence of a coating is suspected.

THERMOELECTRIC INSTRUMENTS

Thermoelectric instruments for identification of metals and alloys are based on the Seebeck effect (the principle of thermocouples). The magnitude of the potential difference depends on the temperatures of the two junctions and on their composition. Thus, if an unknown metal forms a junction with a known metal at a known temperature difference, the unknown metal may be identified. However, this potential is also sensitive to changes in microstructure and surface conditions due to heat treatment, cold working, etc. The magnitude of this change in potential is small for the alloys used in this study, usually less than 1 pct. An extreme example is 304 stainless steel; the instrument reading increases 6 pct when the cold work increases from 0 to 75 pct. In this case, the austenite structure is being transformed into a structure like martensite, owing to the cold work. This kind of effect is fairly small when compared with the changes in instrument readings that occur with changes in composition from alloy to alloy. For example, nickel ranges from 32 to 75 pct for the superalloy samples used in these tests. The corresponding change in instrument readings (from 611 to 708) is more than 15 pct. (See tables 3-4.)

A Technicorp model 850/950 WT Alloy-Separator[5] was used to measure the

TABLE 4. - Thermoelectric measurements for stainless steels, Monels, and superalloys

Alloy class	Average thermoelectric response (copper-base probe tip), arbitrary units, SS 302 standard
Stainless steel:	
201	585
301	597
303	603
304	602
316	587
321	591
347	586
Nickel-base alloys:	
Monel 400	343
Monel K500	352
Inconel 600	698
Inconel 625	644
Inconel X-750	677
Inconel 718	640
Hastelloy B-2	726
Hastelloy S	660
Hastelloy C-276	650
Hastelloy G	626
Nickel 200	466
Nickel 201	460
Nimonic 75	691
Nimonic 80A	708
Nimonic 90	693
Incoloy 800	611
Incoloy 825	625
Iron-base alloy:	
Haynes 20 MOD.	610
Cobalt-base alloys:	
Haynes 188	632
Haynes 25	637

[5]Reference to specific equipment does not imply endorsement by the BuMines.

thermoelectric response of the alloys listed in table 3. Newell (9) has described other thermoelectric instruments that could be used for these measurements. The Alloy-Separator is supplied with changeable probe tips of various proprietary compositions. With a wide variety of probe tips, more effective separations can be performed. The probe tip is the "known" alloy in the couple and is maintained at a constant temperature, so the voltage signal generated is a function of the composition and, to a lesser degree, the microstructure of the sample to be identified. The unit operates in both digital and analog modes. The digital mode was used for these tests.

The unit, shown in figure 1, is operated by installing one of the selectable probe tips and allowing the probe about 10 min to heat to equilibrium temperature. Then the circuit is closed by clipping the unknown specimen to the lead wire and touching the hot probe tip to the specimen. The instrument is calibrated to an arbitrary value using a standard of type 302 stainless steel.

The calibration, requiring several seconds, should be checked periodically. The measurements were repeated many times in order to establish the precision obtainable with this type of device. Samples of each alloy listed in table 3 were

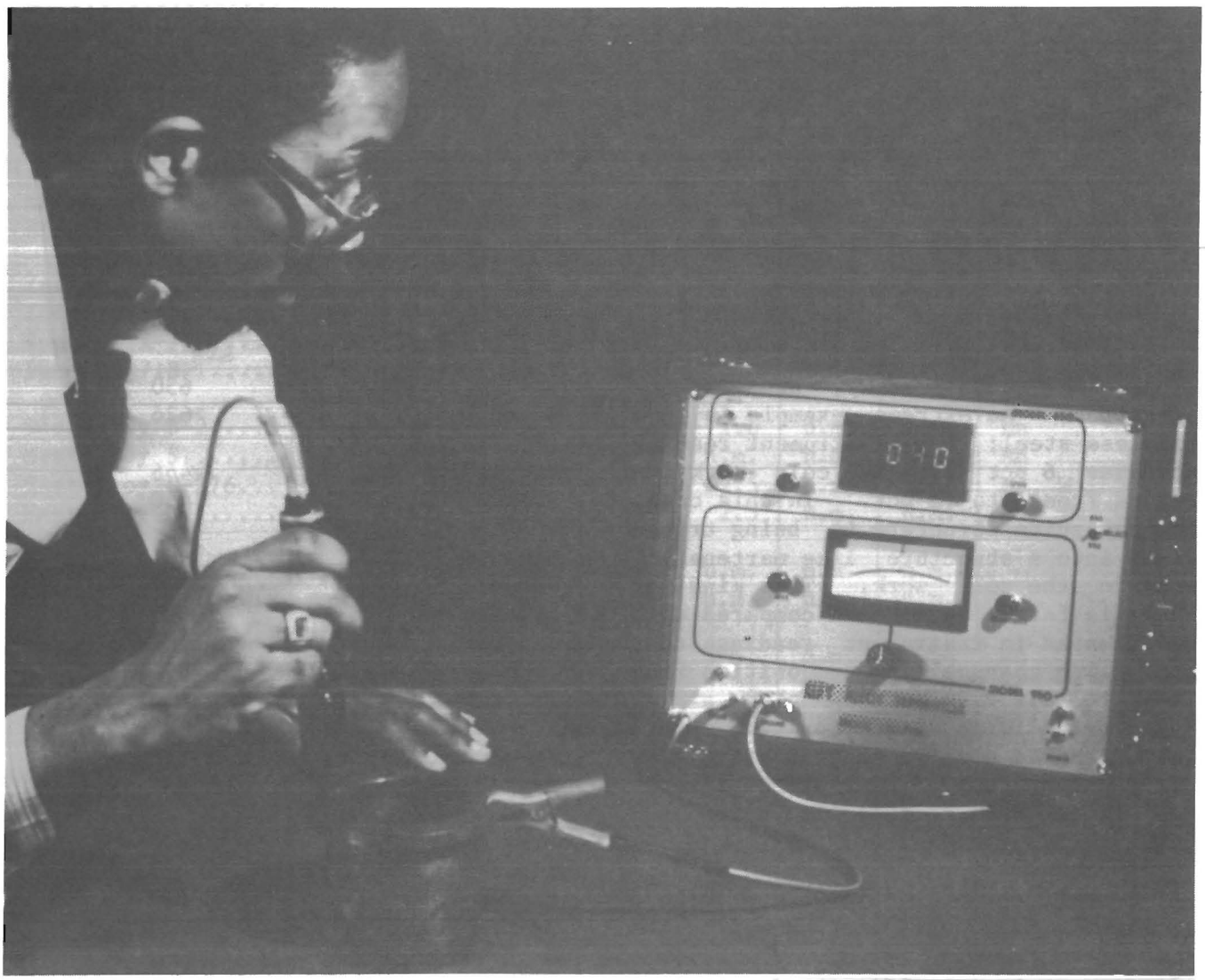

FIGURE 1. - Bureau researcher measuring the thermoelectric response of stainless steel sample.

measured at least five times, with periodic restandardization of the thermoelectric sorter. The unit was stable during runs of several hours, over several days. Some of the samples were measured several times, over a period of weeks. The system was quite stable, showing the same small amount of deviation, <2 pct, in the long-term testing as in the day-to-day tests.

OPTICAL EMISSION

A Clandon Metascope, a small hand-held spectroscope that employs an Amici straight-line prism with a wavelength range of 420 to 650 nm, was used for this study (9, 13). An 80-nm wavelength range can be viewed in the spectroscope at one time. Samples are excited by a vibrating tungsten or molybdenum electrode driven by an ac solenoid.

The Metascope, shown in figure 2, consists of two units: (1) a variable transformer, which provides power to a variable-voltage vibrating-electrode ac arc source used to vaporize and excite the sample, and (2) a hand-held unit containing the prism and optics necessary to observe the spectral lines in the desired range. The spectra can be viewed through an eyepiece or recorded using a camera.

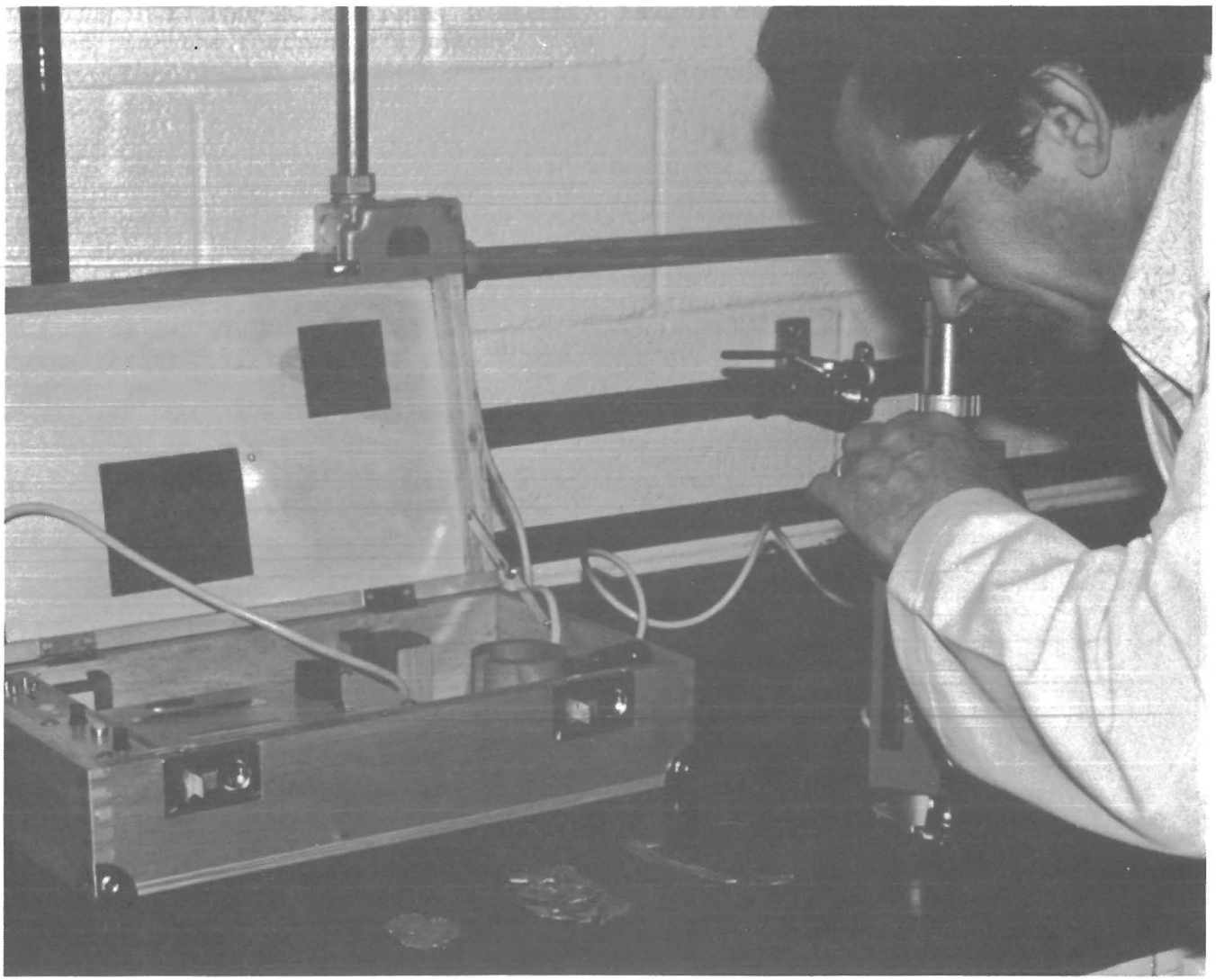

FIGURE 2. - Bureau researcher examining type 316 stainless steel sample for molybdenum content with the Clandon Metascope.

The instrument is calibrated by adjusting the vernier drum so that the eyepiece pointer is located using lines of known wavelength. Other lines can then be observed by adjusting the vernier to bring the desired range of wavelengths into view. The wavelengths corresponding to the elements of interest are found on calibration tables supplied with the instrument.

The Metascope can be used in different modes. The simplest is to determine the absence or presence of a given element in the sample by looking for a specific line or lines corresponding to that element. Knowledge of alloy specifications is then used to decide if the sample is a given alloy. Further, this device can be used to semiquantitatively determine the amount of a given element present in the sample by comparing the intensity of the line in question with its intensity for a standard. The relative amounts of an element present in two samples can be determined in this way without reference to an absolute standard. Finally the alloy can be grouped or specifically identified if the operator has the experience to recognize the spectral patterns corresponding to the various alloys.

X-RAY FLUORESCENT SPECTROGRAPH

A Texas Nuclear model 9266 Alloy Analyzer, shown in figure 3, was used for this study. This energy dispersive instrument generates X-rays using iron-55

FIGURE 3. - Texas Nuclear 9266 Alloy Analyzer.

and cadmium-109 radioactive sources and a scintillation counter for detection. It offers a combination of both alloy identification and elemental analysis. The instrument consists of a hand-held probe containing the sources, filters, the scintillation detector, and associated electronics, and a microprocessor unit which controls the source and filter selections. Alloys with spectral intensities and compositions stored in the instrument memory can be identified by name. Other alloys can be identified by comparing the elemental analysis with alloy chemistry specifications. The elements that can be used for alloy identification are Cb, Co, Cr, Cu, Fe, Mn, Mo, Ni, Ti or V, and W. Intensity-composition data for 66 alloys are presently stored in the instrument's memory. To operate the unit, a clean, flat surface is presented to the analyzer, and either the identification or the analysis mode is selected.

COMBINATION METHOD

A combination method has been developed using the thermoelectric sorter and the optical spectroscope. The new procedure requires only two or three steps to identify each alloy. The thermoelectric sorter is used first to identify some samples by alloy and to place others into groups of alloys. Then the optical spectroscope reduces groups into specific alloys. This procedure is much more efficient than previous spot testing schemes, which required seven or eight steps for making the same identifications.

RESULTS AND DISCUSSION

THERMOELECTRIC MEASUREMENTS

Table 4 lists the results of the thermoelectric measurements for the 27 alloys tested, using a copper-base alloy probe and type 302 stainless steel as a standard. Table 5 lists results for only the nickel-base alloys, using a nickel-base probe and both type 302 stainless steel and nickel 201 as standards. The selection of the best probe-standard combination for a given array of unknowns is an empirical process. Incoloy 825 and Hastelloy G are poorly resolved using the copper alloy probe or the nickel alloy probe with the stainless 302 standard. The nickel alloy probe with the Ni 201 standard significantly increases the ease of separation. The resolution between values for the Inconel 625-Hastelloy C-276 pair is poor for the nickel probe-stainless steel standard combination, better for the copper-stainless steel combination, and excellent for the nickel-nickel combination. However, this combination is not always the best. For Inconel 600 and Nimonic 75, either probe with the 302 stainless standard gives a wider difference in readings than the nickel-nickel combination.

TABLE 5. - Thermoelectric response of nickel-base alloys (nickel-base probe tip)

Alloy	SS 302 standard	Ni 201 standard
Inconel 600.....	703	986
Inconel 625.....	656	917
Inconel 718.....	650	920
Inconel X-750.....	679	952
Incoloy 800.....	615	865
Incoloy 825.....	633	883
Hastelloy C-276.....	655	929
Hastelloy B-2.....	728	1,018
Hastelloy G.....	635	895
Hastelloy S.....	668	945
Nimonic 75.....	690	989
Nimonic 80A.....	713	1,020
Nimonic 90.....	693	962

The operator must be aware that values for different alloys are sometimes unresolved and that overlap can exist. A value of 615 could represent a superalloy or a work-hardened 304 stainless sample. But a value of 590 is clearly a stainless steel, while one of 660 is clearly a superalloy.

The tables reveal two key items. There is a good first separation between the stainless steels, Monels, nickels, and superalloys by groups. Further separations within the superalloy group are possible as described in the section "Combination Method."

EMISSION SPECTROSCOPE

The optical spectroscope was used for both elemental and alloy identification. From the first series of measurements, specific identifying elements were determined, such as molybdenum to separate 316 from other stainless steels and cobalt to separate Nimonic 90 from other Nimonics. In the second series of measurements, discrete alloy identification was attempted.

In the first case, the Clandon Metascope was used to separate 316 stainless steel (2 to 3 pct Mo) from the rest of the 300 series (<0.5 pct Mo) stainless steels. Figure 4A shows a spectrum for 302 stainless steel, which contains <0.5 pct Mo, with the marker on the 547.69-nm nickel line in the center of the field of view. Figure 4B, with the marker on the 553.30-nm Mo line and with the 547.69-nm Ni line just to the right, shows that the molybdenum emission is easily discernible for type 316 stainless steels. The spectra can be compared easily if viewed "on end" with the line of sight nearly parallel to the page.

This instrument also allows the sorter to segregate mixed scrap on the basis of a particular element. Figure 5A shows the spectrum for a Nimonic alloy. In this case, the instrument was adjusted to the 547.69-nm nickel line. The nickel line is very intense in the Nimonic alloys (59 to 75 pct Ni); thus, high-percentage-nickel scrap can be separated from low-nickel scrap on the basis of the observed intensity of the nickel line.

Use of the spectroscope mainly for single-element determinations of presence or relative amount is not difficult and should be learned quickly by most operators. Comparing photographs of the spectra, while more tedious, is much easier and requires considerably less skill than mental comparison of visual observations. While it does require much more operator skill, spectral pattern recognition can be used to identify alloys or groups of alloys.

Figure 4A and 4B show that 302 and 316 stainless steels have similar spectra except for the molybdenum lines. In figure 5, the spectrum of the Nimonic alloy shown is substantially different from that for the stainless steel. For example, in the stainless steel, directly to the right of the 547.69-nm Ni line is a series of iron lines; they are much less intense in Nimonics than in the stainless steel. Examination of figure 6 shows distinct differences between the spectra of a Hastelloy and a stainless steel. The Hastelloy spectrum has two strong lines to the left of the nickel line, which are the 553.30- and the 550.65-nm lines for molybdenum. These lines are very intense, indicating that this alloy has a much higher concentration of molybdenum than does stainless steel.

Two problems are the "memory effect," resulting from the carryover of some of the preceding sample as residue on the electrode, and reproducible image intensity. The first problem was solved by cleaning the electrodes with emery boards between each use. The second problem required that the operator take special care to properly hold the instrument on the sample to obtain a stable, reproducible arc.

X-RAY ANALYZER

The Texas Nuclear fluorescent X-ray spectrograph is oriented toward the identification of superalloys. Seventeen of the 27 alloys used for this work are included in the memory of the instrument. The device identified 16 of the 17 by name. The elemental analysis for each superalloy in the array was sufficient so that the correct identification could be made by comparison of X-ray-determined values and alloy specifications. While this mode is relatively tedious, it

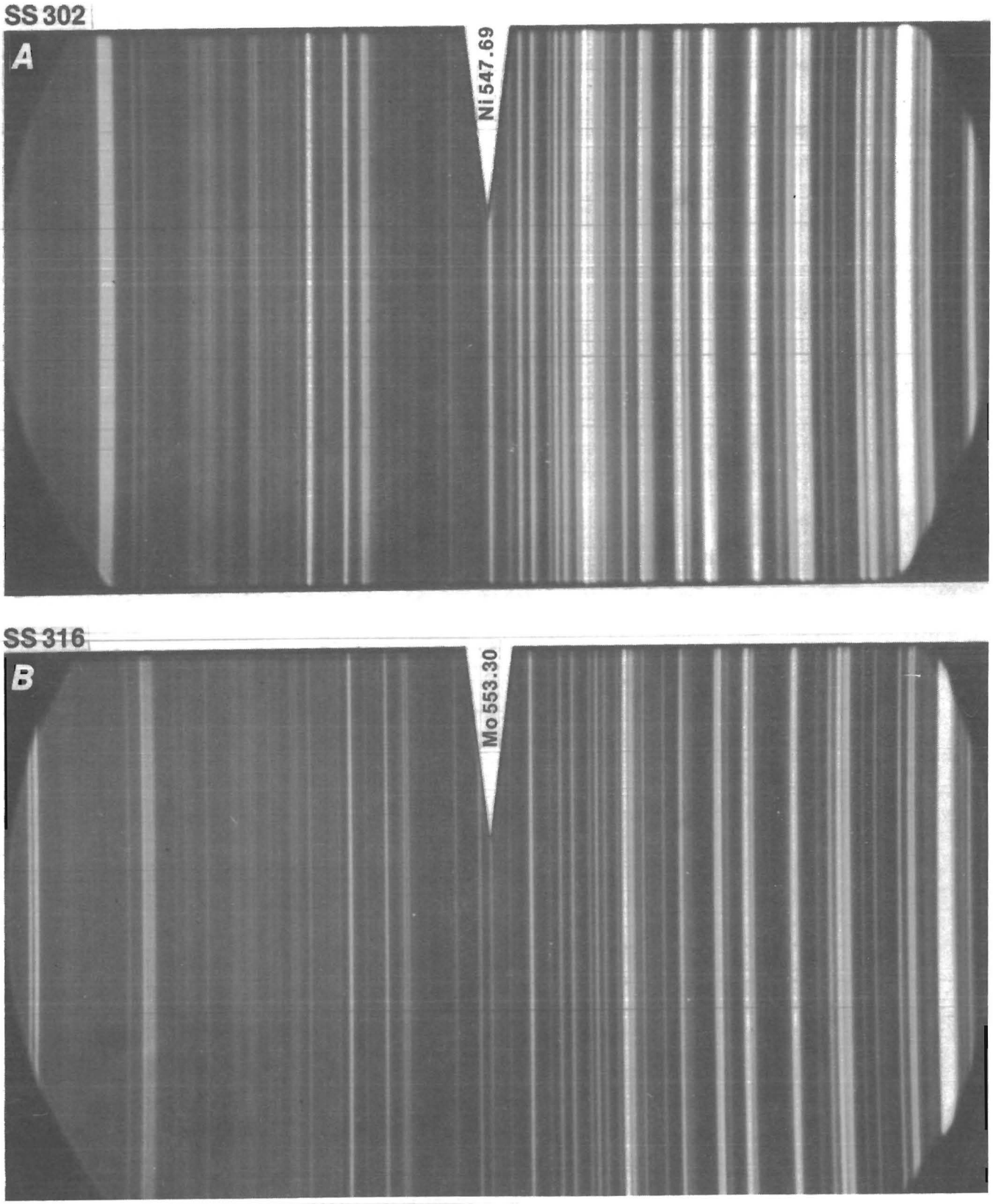

FIGURE 4. - Comparison of spectra for 302 stainless steel and 316 stainless steel. *A*, 302 stainless steel (pointer set on the 547.69-nm nickel line); *B*, 316 stainless steel (pointer set on the 553.30-nm molybdenum line).

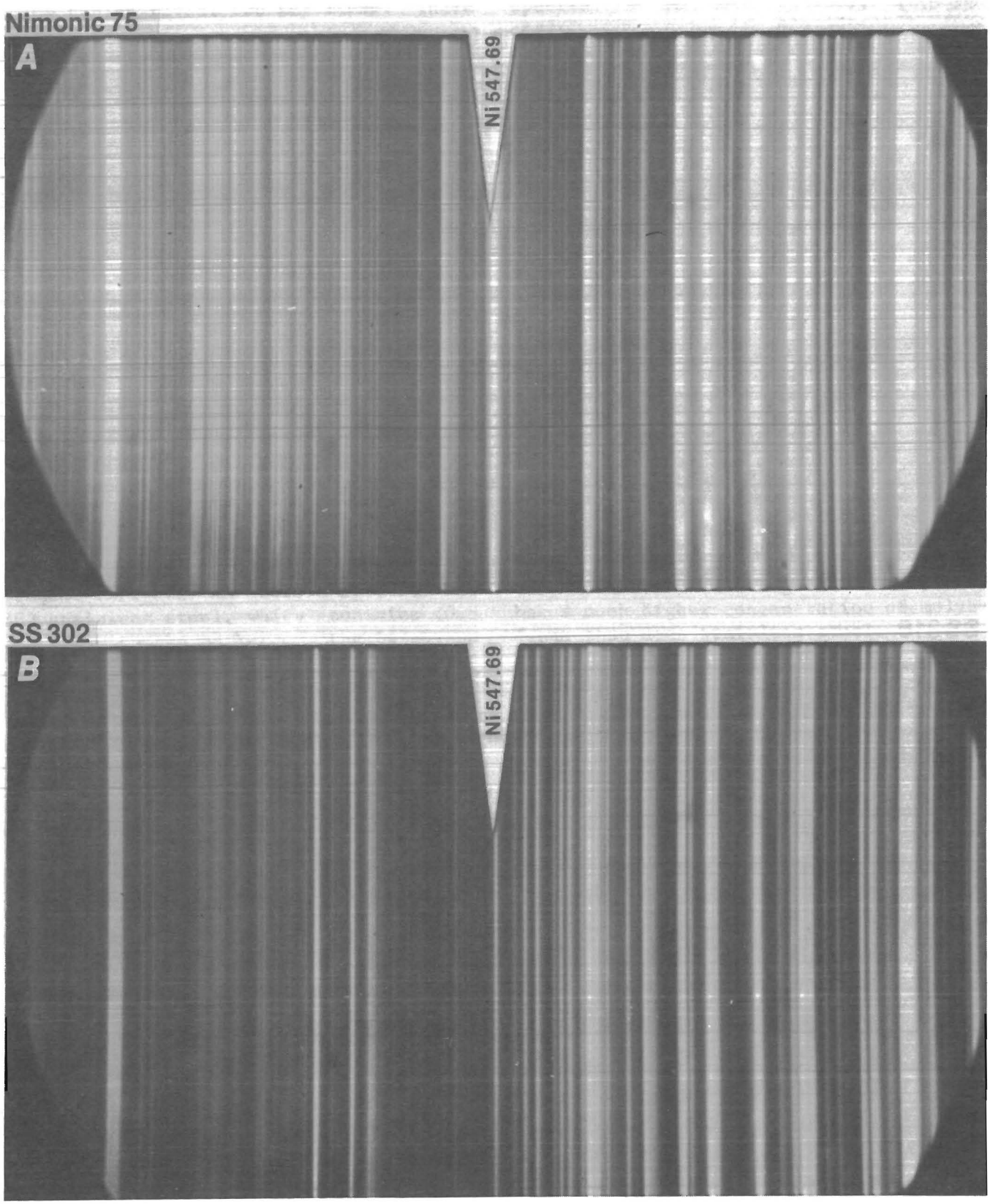

FIGURE 5. - Comparison of spectra for a Nimonic and a stainless steel. *A*, Nimonic 75 (pointer set on the 547.69-nm nickel line); *B*, 302 stainless steel (pointer set on the 547.69-nm nickel line).

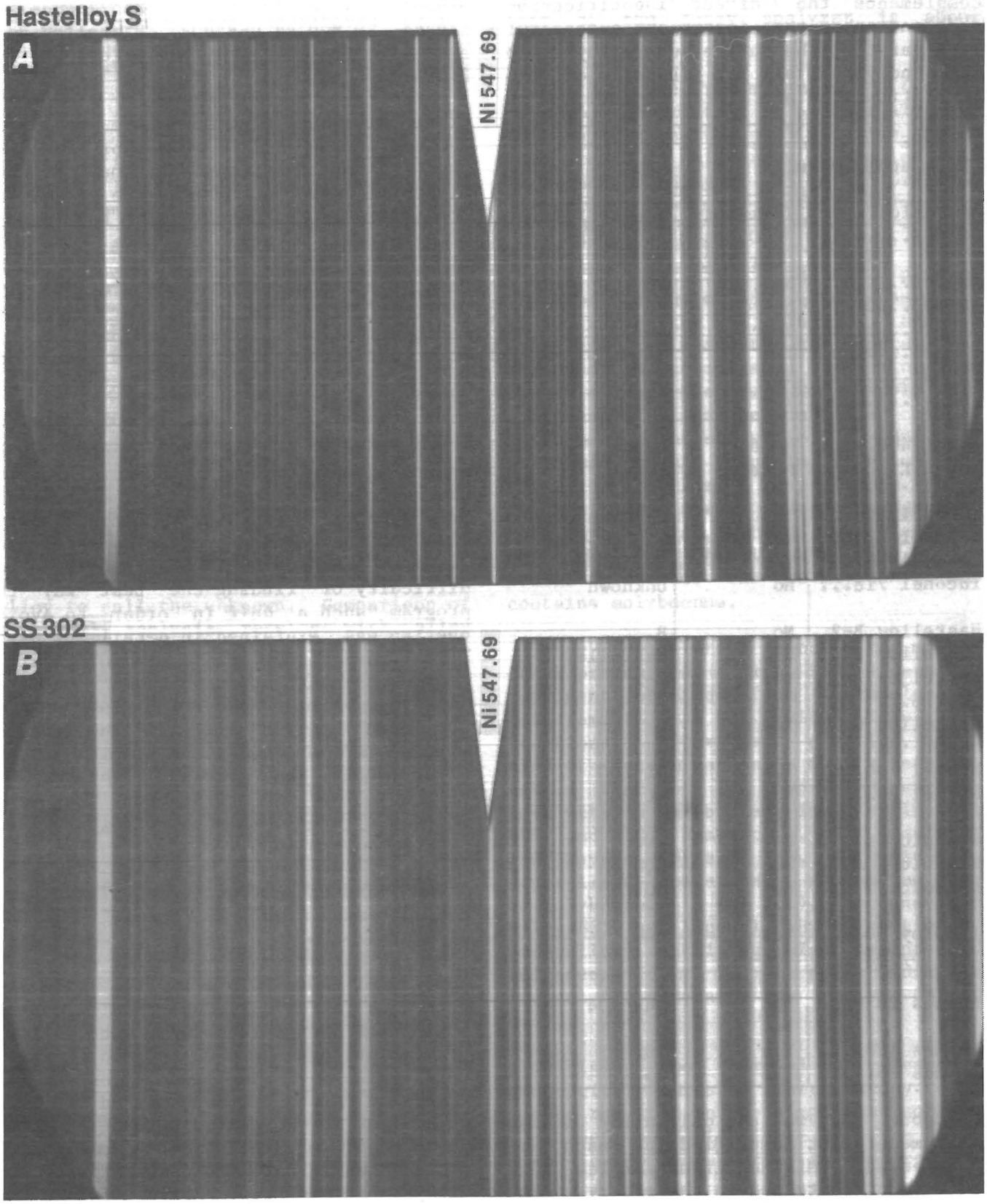

FIGURE 6. - Comparison of spectra for a Hastelloy and a stainless steel. *A*, Hastelloy S (pointer set on the 547.69-nm nickel line); *B*, 302 stainless steel (pointer set on the 547.69-nm nickel line).

complements the direct identification mode and makes the instrument successful in separating the entire superalloy group into individual alloys. Details of the identifications are listed in table 6.

TABLE 6. - Fluorescent X-ray spectrographic results

Alloy	In memory?	Identification
SS 201.....	"200," set for 202.	200 or SS
SS 301.....	No	SS
SS 303.....	No	SS
SS 304.....	Yes	304
SS 316.....	Yes	316
SS 321.....	Yes	321
SS 347.....	Yes	347
Monel 400.....	Monel	Monel
Monel K500....	Monel	Monel
Inconel 600...	Yes	Unknown or 600
Inconel 625...	Yes	625
Inconel X-750.	Yes	X-750
Inconel 718...	No	Unknown
Hastelloy B-2.	No	B
Hastelloy S...	No	Hastelloy C-4
Hastelloy C-276.	Yes	C-276
Hastelloy G...	Yes	G
Nickel 200....	Yes	Ni 200
Nickel 201....	No	Ni 200
Nimonic 75....	No	600
Nimonic 80A...	No	600
Nimonic 90....	No	601
Incoloy 800...	Yes	800
Incoloy 825...	Yes	825
Haynes 20 MOD.	No	UHB 904
Haynes 188....	Yes	188
Haynes 25.....	Yes	25

Inconel 600 was the only alloy in the unit memory not identified for each sample tested. Misidentified samples were alloys not contained in the memory. The Nimonics were identified as Inconels, Haynes 20 MOD as UHB 904, Hastelloy B-2 as Hastelloy B, and Hastelloy S as Hastelloy C-4.

The misidentifications are possible because of the limited number of elements used for positive identifications of the alloys in memory. The device compares the values of the unknown with the ranges of expected values; positive correlation gives an identification. In each case, the misidentifications are actually "correct" for the particular elements matched by the instrument. But one or more additional matching tests would be needed in each case to resolve the misidentifications.

The program in the instrument can use up to seven elemental tests for a positive identification, but in most cases uses only three or four. Precise knowledge of the sorting program used in the Texas Nuclear device is proprietary. The difficulty of finding the best way to program such a unit in order to avoid overlap was explained in detail by Marr (5). The designer has to aim for separations (identifications) based on the usual compositions occurring in practice for each of the alloys. The ranges allowed in the composition specifications often involve some overlap. This is why strict alloy identification for the stainless steels is more difficult than for the superalloys.

The Texas Nuclear unit identifies both Monels tested simply as "Monel." In the elemental analysis mode, it can distinguish between Monel 400 (no titanium) and Monel K500 (0.6 pct Ti). The expected range for molybdenum and columbium in Inconel 600 appears to be set too low. Consequently the unit failed to identify this alloy. The iron content of Hastelloy B is 6 pct as compared to less than 2 pct for Hastelloy B-2. Therefore measurement of the iron concentration would discriminate between these two alloys. This same logic applies to

the Hastelloy S and C-4 and the Haynes 20 MOD and UHB 904 unresolved pairs, using cobalt and copper concentration respectively. Nickel 201 is simply extra-low-carbon pure nickel. Using the analyses for iron, titanium, and cobalt would resolve the Nimonic-Inconel misidentifications. The elemental analysis for Inconel 718 is easily matched to its composition specification. The unit is set to identify stainless steels with 7.5 to 10 pct Mn as "200," the same range as for type 202. Stainless steel 201 has 5 to 7.5 pct Mn and is sometimes identified as "200" and sometimes as "stainless." But in all cases the high manganese reading in the elemental analysis mode would indicate the 200 series. The instrument successfully identifies each individual 300 series stainless steel in its memory, but problems arise for similar alloys not in memory. Sometimes these alloys are identified as one of the stainless steels in memory. Sometimes the instrument cannot decide which particular stainless alloy to call the unknown. Comparison of elemental analysis results with alloy specification is tedious here, because the specifications do overlap. But the instrument is successful in first indicating stainless steel, and then specifically indicating the special stainless steels: 316 and 317 (Mo), 321 (Ti), 347 (Cb), etc. The "ordinary" stainless steels not in the memory, for example, 301 and 303, fit right into the Sabot scrap classification (table 1), so in real world practice, the problem disappears.

COMBINATION METHOD

The Secondary Resources group at the Bureau's Avondale Research Center has devised a combination scheme that takes advantage of the high efficiencies and low cost of two of the methods studied, thermoelectric and optical emission analysis. The costs of the thermoelectric sorter and optical spectroscope are about $4,200 and $5,600, respectively, whereas that of the X-ray analyzer is about $23,000.

The thermoelectric measurements (tables 4 and 5) show that it is relatively easy to identify the 200 and 300 series stainless steels in this array of samples. An instrument reading of 585 to 615 produces a fraction that may contain 201, 301, 303, 304, 316, 321, and 347 stainless steels, Incoloy 800, and Haynes 20 MOD (table 4). All that is needed to produce the Sabot grade of 18-8 stainless steel is to remove the 201, 316, 321, and 347 stainless steels and the two superalloys. This can be accomplished by using the portable spectroscope. For example, 316 stainless steel can be identified by its molybdenum content, while 321 and 347 stainless steels contain titanium and columbium-tantalum, respectively. The 201 stainless steel contains manganese; both superalloys contain larger amounts of nickel and chromium than any of the stainless steels, and the Haynes 20 MOD contains molybdenum.

Using the relative amounts of molybdenum present as a key, the superalloy component obtained from thermoelectric measurements can be divided into three fractions. After some practice, this kind of determination is fairly easy to make. Fraction 1, comprising superalloys with no molybdenum, includes Inconel 600 and 750; Nimonic 75, 80A, and 90; and Haynes 25 and 188. Fraction 2, superalloys containing medium amounts of molybdenum (3 wt pct), includes Incoloy 825 and Inconel 718. Fraction 3, superalloys containing high amounts of molybdenum (6 to 28 wt pct), includes Inconel 625 and Hastelloy B-2, S, C-276, and G.

Combining the thermoelectric response and optical emission spectroscopy techniques resulted in the simplified sorting flowsheet shown in figure 7. The first step in the flowsheet is to subject the

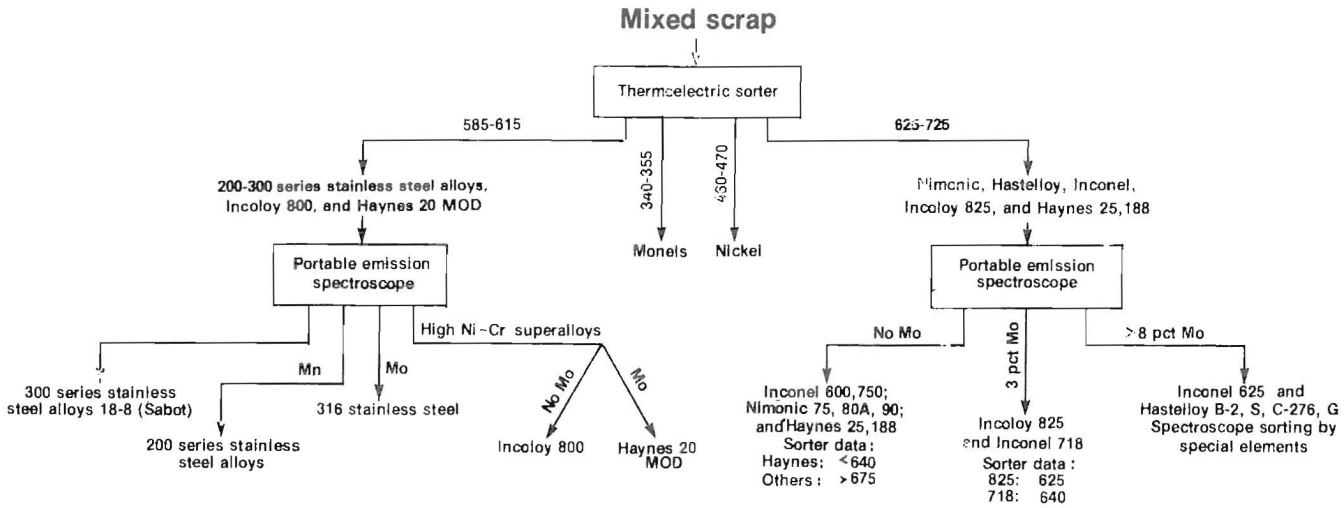

FIGURE 7. - Instrumental identification technique for sorting stainless steels, nickel alloys, and superalloys; readings in arbitrary units.

samples to thermoelectric measurements. This results in--

1. A 200-300 series stainless steel-Incoloy 800-Haynes 20 MOD fraction (585-615).

2. A Monel fraction (340-355).

3. A pure nickel fraction (460-470).

4. A superalloy fraction (625-725).

Using the spectroscope, we can then identify and separate from each other low-molybdenum 18-8 grade stainless steels (Sabot), 316 stainless steel, Haynes 20 MOD, Incoloy 800, and the 200, 321, and 347 stainless steel fractions, respectively, by the presence of nothing special, molybdenum, high nickel and chromium with molybdenum, high nickel and chromium alone, manganese, titanium, or colubium and tantalum (table 3). The same spectroscopic technique can be applied to the superalloy fraction. This results in the three superalloy fractions of high, medium, and low molybdenum content. By reapplying the thermoelectric data, or observing the spectra again, these fractions can be further broken down into alloy classes. For example, the medium molybdenum fraction can be segregated into the individual alloys Inconel 718 and Incoloy 825 on the basis of the thermoelectric readings: 625 for Incoloy 825, and 640 for Inconel 718. The high-molybdenum fraction can be separated into the individual Hastelloy and Inconel components by using thermoelectric readings or further observations of the spectra. The flowsheet in figure 7 for the combined procedure shows graphically how these separations are completed in two or three steps.

The increase in ease and efficiency is shown by comparing this to a flowsheet (fig. 8) for making a similar separation by a traditional chemical spot test routine.

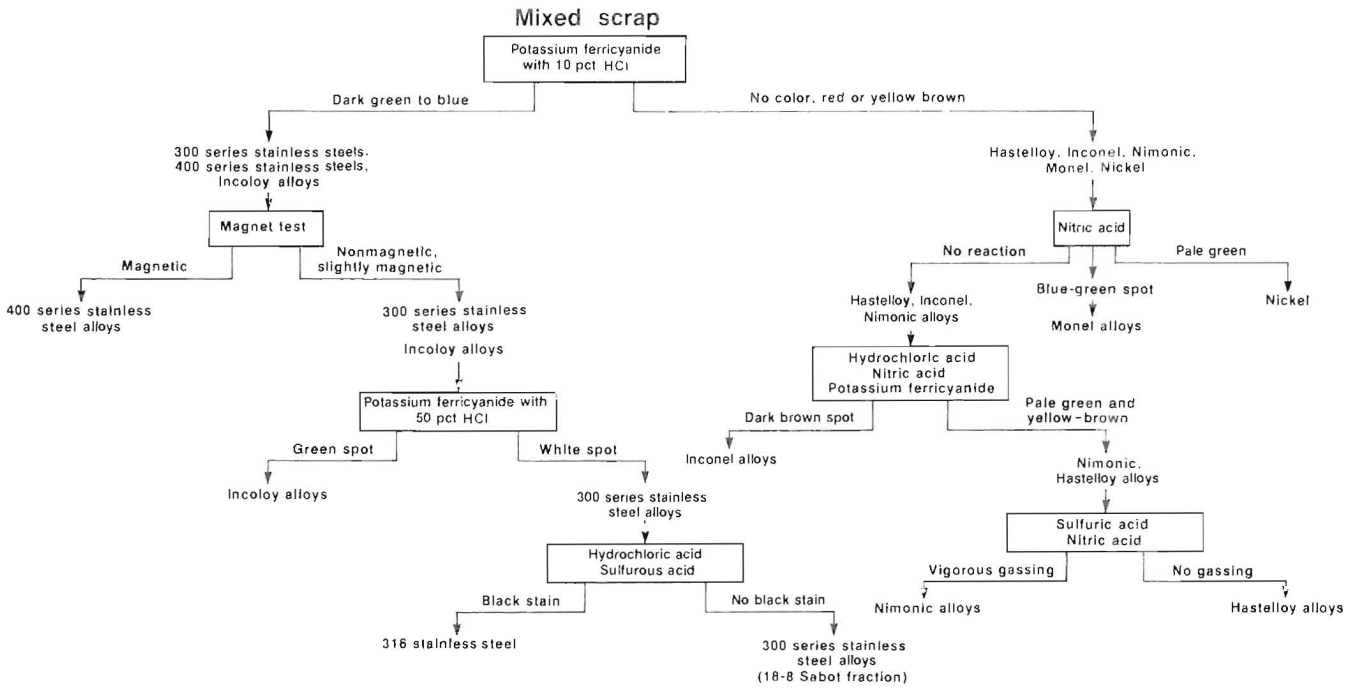

FIGURE 8. - Conventional metals identification techniques for separating nickel-base and stainless alloys.

CONCLUSIONS

Traditionally, the identification and sorting of scrap metals has been carried out by skilled sorters using various physical and chemical tests. New techniques, such as thermoelectric response, emission spectroscopy, and fluorescent X-ray spectrography, improve the accuracy and ease of rapid identification. Twenty-seven stainless steel and superalloy samples were used in laboratory investigations of these techniques. The selection of an identification method is generally determined by the potential value offered by the increased degree of separation. The use of the techniques described above may not be warranted for less valuable materials. In addition, a certain amount of presorting is necessary; it is extremely useful to know what types of alloys may be present so that standards for instrument calibration may be selected.

Thermoelectric response is inherently a comparison method of identification because individual elements cannot be quantitatively determined. The measured response is the resultant of effects from all the elements present; the effect from a single element is difficult to isolate except when the specimen array is very well defined.

Although it is inherently capable of quantitative analysis, emission spectroscopy is generally also used as a comparison method. The response is due to the amounts of individual elements present, but it is difficult to recognize specific alloy patterns or to accurately estimate quantitative amounts without considerable training and skill. It is fairly easy to learn the skills necessary for recognizing the patterns of alloy types and for estimating relative amounts of an element present in different samples. The thermoelectric sorter and optical emission spectroscope have been combined to form an efficient, low-cost sorting system.

The X-ray spectrograph identifies many alloys directly through an integral computer programmed to process the X-ray counts from a number of elements present

into a readout of individual alloys. Sometimes an alloy is misidentified because of the small number of programmed identifying elements, but results are generally excellent when this class of instrument is used within the expected range of alloys. While it is a tedious process compared with direct readout of the alloy name (but still only several minutes per unknown), the correct identification of any alloy within the design range can be made by using the elemental analysis mode and comparing the results with alloy specifications.

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APPENDIX.--ANALYSIS OF STAINLESS STEELS AND SUPERALLOYS BY FLAME ATOMIC ABSORPTION SPECTROSCOPY: RESULTS FOR Co, Cr, Cu, Fe, Mn, Mo, and Ni

INTRODUCTION

The increasing use of new alloys is making rapid scrap identification by the traditional color, spark, and chemical spot tests more complex. Flame atomic absorption spectroscopy (FAAS) is an alternative to these traditional tests and a good method of spot checking or verifying results obtained by more rapid methods. By comparing the results with chemical composition tables for alloys, a positive identification of the sample can be made.

Atomic absorption spectroscopy is highly regarded as an analytical technique owing to its relatively high speed of analysis, selectivity, sensitivity, and simplicity (12).[1] In atomic absorption, a dissolved sample is aspirated into a flame where it is converted into an atomic vapor which absorbs light from a primary light source (hollow cathode lamp). A series of calibration standards is prepared for each element such that all of the samples to be analyzed fall within the linear range of this standard curve. Based on the linear relationship between the absorbance and concentration, the concentration of the elements in the sample can be determined.

A procedure has been reported using a rapid, high-pressure, acid dissolution technique carried out in polycarbonate bottles which allows total analysis of samples from single solutions (2, 7). This method was found suitable for the stainless steels; however, the other alloys required that an acid digestion bomb be used to achieve complete dissolution. Decomposition with HF under pressure in an acid digestion bomb is definitely advantageous in comparison to standard time-consuming decomposition procedures, particularly when considering superalloys. A relatively interference-free matrix is created by the addition of a boric acid solution (2).

[1]Underlined numbers in parentheses refer to items in the list of references preceding the appendix.

Using this method, stainless steels, nickel-base alloys and superalloys, iron-base alloys, and a cobalt-base superalloy have been successfully dissolved and analyzed for Co, Cr, Cu, Fe, Mo, Mn, and Ni.

EXPERIMENTATION

Equipment

1. Drill press.

2. Boiling water bath (4-L beaker with ~2 in H_2O, four bottles fit at one time).

3. Polycarbonate bottles, 250-mL, wide-mouthed with polypropylene screw caps.

4. Acid digestion bomb (Parr #4749, 23-mL).

5. Liquid dispensers, Eppendorf pipets (0.1- to 0.5-mL), pipets (1- to 10-mL), graduated cylinders (10- to 100-mL), plastic and glass.

6. Atomic absorption spectrophotometer (Perkin-Elmer model 373).

7. 50-nm nitrous oxide burner head.

8. Hollow cathode lamps for Co, Cu, Cr, Fe, Mn, Mo, and Ni.

9. Welder's grade acetylene, high-purity nitrous oxide, and dry end oil-free compressed air.

10. Fume hood.

11. Oven.

Reagents and Solutions

1. Standard reference solutions of 1,000 µg/mL for each element of interest.

2. Boric acid solution (1.5-wt-pct)--mix 15 g high-purity boric acid crystals (99.9 pct) with 1 L H_2O.

3. HCl-HF acid mixture (7:3)--use concentrated reagent-grade acids.[2]

4. HNO_3--concentrated reagent-grade acid.

Sample Preparation

1. Drill samples--turnings or drillings are acceptable.

2. If the sample is a stainless steel, place a 0.100- to 0.250-g sample into 250-mL polycarbonate bottles. If the sample is something other than a stainless steel, go to step 9.

3. Pipet (plastic) 5-mL of the 7:3 HCl-HF acid mixture, followed by 2 mL of HNO_3, into each bottle. Screw caps on tightly.

4. Place bottles in the boiling water bath for 15 min.

5. Remove bottles and cool in running water for 2 min.

6. When cool, remove caps from the bottles and add 93 mL of the 1.5-wt-pct boric acid solution to each bottle. Screw caps on tightly.

7. Return the bottles to the boiling water bath for 15 min.

8. Remove bottles and place in cold running water until completely cool. The solutions are now ready for analysis. Refer to the analysis procedure section.

9. For samples other than stainless steels, place a 0.100-g sample into the Teflon cup of an acid digestion bomb.

10. Pipet (plastic) 5 mL of the 7:3 HCl-HF acid mixture, followed by 2 mL of HNO_3, into the cup.

11. Assemble the bomb, making sure that it is closed as tightly as possible.

12. Place the bomb in a preheated oven at 150° C for approximately 1 h.

13. Allow the bomb to completely cool before opening (~3 h).

14. Disassemble the bomb. Pour the solution into a 250-mL polycarbonate bottle and add 93 mL of the 1.5-wt-pct boric acid solution. The solution is now ready for analysis.

Analysis Procedure

Table A-1 summarizes the standard atomic absorption conditions for the elements of interest. All dilutions were made with distilled water in volumetric flasks, using a micropipet. The standard curve for each element was prepared by diluting a standard reference solution (1,000 ppm) down to the linear working range as given in table A-1. Each sample solution was diluted so that the resulting element concentration fell within the linear working range of the element's

[2]Particular care should be taken when working with HF. It is an extremely hazardous liquid and vapor. It causes burns which may not be immediately painful or visible. Refer to the chemical label for specific details.

TABLE A-1. - Standard flame atomic absorption conditions (10)

Element	Wavelength, nm	Slit width, nm	Gas mix	Flame type	Standards, µg/mL
Co	240.7	0.2	N_2O-C_2H_2	Lean	1,3,5
Cr	357.9	.7	N_2O-C_2H_2	Rich	1,3,5
Cu	324.8	.7	Air-C_2H_2	Lean	1,3,5
Fe	248.3	.2	N_2O-C_2H_2	do.	1,3,6
Mn	279.5	.2	N_2O-C_2H_2	do.	1,3,5
Mo	313.3	.7	N_2O-C_2H_2	Rich	10,30,50
Ni	232.0	.2	N_2O-C_2H_2	Lean	1,3,5

standard curve. Therefore, the necessary dilutions of each sample solution vary, depending on the element of interest and the original sample weight.

For the elements where interferences have been experienced when using an air-acetylene (air-C_2H_2) flame, a nitrous oxide-acetylene (N_2O-C_2H_2) flame was used to reduce or eliminate potential chemical and/or matrix interferences. The 50-mm-N_2O burner head was positioned at the optimum height for analysis of chromium and was used at that height for the remainder of the elements. All of the elements requiring an N_2O-C_2H_2 flame were run consecutively. Furthermore, to insure consistent flame conditions, the elements requiring a lean flame were run one after another, followed by those requiring a rich flame. Otherwise, the elements were run in random order.

Distilled water was used to set the zero point. During the analysis procedure, the zero point was checked following each sample, and the standard curve was rerun at the end of each element analysis to check for drift. Standard linear regression analysis was applied to the data, and the elemental concentrations were determined for each sample and converted to weight percent.

RESULTS

Tables A-2, A-3, A-4, and A-5 summarize the analytical data obtained for samples of stainless steels, a cobalt-base superalloy, nickel-base alloys, and iron-base alloys, respectively. Standard deviations are reported, as are the alloy specification for each element (6) and the actual heat analysis where available.

TABLE A-2. - FAAS results for the analysis of stainless steels, weight percent

Element	FAAS	Specification (6)
	SS 304	
Co.....	0.057±0.010	NS
Cr.....	19.1 ±1.6	18-20
Cu.....	.116± .050	NS
Fe.....	71.6 ±1.8	Balance
Mo.....	.174± .060	NS
Mn.....	1.68 ± .070	2 (max)
Ni.....	8.72 ± .83	8-12
	SS 316	
Co.....	0.193±0.303	NS
Cr.....	16.5 ±1.0	16-18
Cu.....	.397± .050	NS
Fe.....	72.0 ±2.3	Balance
Mo.....	3.09 ± .71	2-3
Mn.....	1.85 ± .090	2 (max)
Ni.....	12.5 ± .83	10-14

NS Not specified.

TABLE A-3. - FAAS results for the analysis of a cobalt alloy, (Haynes 25), weight percent

Element	FAAS	Specification (6)	Heat analysis
Co.....	50.5 ±1.9	Balance	50.92
Cr.....	20.1 ± .040	19-21	19.83
Cu.....	.112± .10	NS	NS
Fe.....	2.38 ± .020	3	2.31
Mo.....	.295± .010	NS	.31
Mn.....	1.52 ± .040	1- 2	1.46
Ni.....	10.5 ± .63	9-11	10.04

NS Not specified.

TABLE A-4. - FAAS results for the analysis of nickel-base alloys, weight percent

Element	FAAS	Specification (6)	Heat analysis
	MONEL 400		
Co	0.258±0.060	NA	NA
Cr	.635± .46	NA	NA
Cu	33.4 ±1.8	31.5	NA
Fe	1.56 ± .010	1.25	NA
Mo	.344± .93	NA	NA
Mn	.843± .57	1	NA
Ni	63.6 ±2.5	66.5	NA
	HASTELLOY G		
Co	2.26 ±0.38	2.5	1.8
Cr	20.1 ± .070	21 -23.5	21.62
Cu	2.08 ± .030	1.5- 2.5	1.75
Fe	19.3 ± .75	18 -21	19.34
Mo	7.70 ± .57	5.5- 7.5	6.63
Mn	1.36 ± .020	1 - 2	1.33
Ni	45.5 ±1.9	Balance	44.08
	HASTELLOY C-276		
Co	2.38 ±0.070	2.5	2.18
Cr	15.7 ± .47	14.5-16.5	16.12
Cu	.230± .21	NA	NA
Fe	6.27 ± .080	4 - 7	6.02
Mo	20.7 ±2.2	15 -17	15.29
Mn	.560± .010	1	.43
Ni	50.5 ±1.9	Balance	56.02
	HASTELLOY B-2		
Co	0.193±0.010	1	<0.10
Cr	.106± .010	1	.40
Cu	.047± .010	NA	NA
Fe	1.00 ± .010	2	.86
Mo	34.2 ± .62	26 - 30	27.84
Mn	.117± .010	1	.16
Ni	70.5 ±1.0	Balance	70.60
	INCONEL 625		
Co	0.225±0.040	NA	NA
Cr	21.1 ± .85	21.5	NA
Cu	.365± .020	NA	NA
Fe	4.56 ± .080	2.5	NA
Mo	10.1 ± .060	9	NA
Mn	.140± .010	.2	NA
Ni	57.2 ± .31	61 (Ni-Co)	NA
	INCOLOY 825		
Co	0.04 ±0.010	NA	NA
Cr	22.1 ± .11	21.5	NA
Cu	2.06 ± .070	2.2	NA
Fe	29.9 ±1.4	30	NA
Mo	3.77 ± .040	3	NA
Mn	.270± .010	.5	NA
Ni	41.4 ± .92	42	NA

NA Not available.

TABLE A-5. - FAAS results for the analysis of iron-base alloys, weight percent

Element	FAAS	Specification (6)	Heat analysis
	INCOLOY 800		
Co	0.066±0.010	NA	NA
Cr	18.5 ±2.7	21	NA
Cu	.605± .010	.38	NA
Fe	47.5 ± .23	Balance	NA
Mo	.265± .010	NA	NA
Mn	1.26 ± .46	.75	NA
Ni	33.0 ±1.6	32.5	NA
	HAYNES 20 (MOD)		
Co	0.510±0.030	NS	NA
Cr	21.1 ± .54	21-23	21.87
Cu	.09 ± .010	NS	NA
Fe	45.8 ±1.4	Balance	46.16
Mo	5.93 ± .34	4- 6	4.23
Mn	.897± .030	2.5 (Max)	.84
Ni	24.8 ± .50	25-27	25.89

NA Not available.
NS Not specified.

DISCUSSION AND CONCLUSIONS

Using the standard atomic absorption conditions presented in table A-1, significant interferences were not encountered. The experimental values are in agreement with the published standard values and heat analyses, where available.

During the initial dissolution procedure, a reduction in the acid volume may occur owing to the reaction of the acid with the sample. However, with a sample of less than 0.500 g, the volume loss is less than 1 mL. Therefore, the final volume is reduced by less than 1 pct after the addition of the H_3BO_3 solution (2).

The determination of several elements in a single sample frequently requires that more than one dissolution procedure be performed (3). These conventional methods are time consuming, particularly for the dissolution of superalloys. The reported procedure, dissolution and analysis, requires at most 6 h for the autoclave-dissolved samples and 1-1/2 h for the bottle-dissolved samples. The 6 h includes 1 h for the dissolution and 5 h for the autoclave to return to room temperature. The actual sample analysis requires about 45 min. This time includes running the sample and the standard curve for each of the seven elements. Consequently, up to a point, the more samples run at one time, the more efficient the procedure becomes.

Comparing the experimental results with chemical composition tables for the alloys provides a sufficient method for alloy identification or verification.

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