

THE SPECTROSCOPIC WORKING CURVES FOR MAGNESIUM AND OTHER ELEMENTS IN ALUMINUM ALLOYS

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THE SPECTROSCOPIC WORKING CURVES FOR

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INTRODUCTION

This paper is concerned with the spectrographic determination of 14S and 17S aluminum alloys for which the log relative exposure ratios of the spectral lines of several alloying elements were studied. The alloying elements considered were magnesium, iron, silicon, and manganese.

The wavelength of the various lines as well as the range of concentration for which they were used are included. The working curves for the different alloying elements obtained from the study of the log relative exposure ratios are presented.

A group of six spectroscopic standards obtained from the Aluminum Company of America were used in the development of this work. Eighteen samples of 14S and 17S aluminum alloys were analyzed in terms of these spectroscopic standards, and were designated as secondary standards to be used for subsequent determinations.

A detailed procedure using the internal standard method of line comparison for the spectrographic determination of 14S and 17S aluminum alloys with a condensed spark circuit is given.

A method for the determination of the proper exposure time is outlined. This method can be applied to any spectrographic determination of aluminum alloys.

Two methods of aligning the spark source are presented.

The plates were made on a Bausch and Lomb Littrow type spectrograph and were read on a Bausch and Lomb projection densitometer.

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APPARATUS AND INSTRUMENTS

A condensed spark circuit shown in Fig. I was used as the excitation unit in this work. It consists of a type Q variac R $(1172-41-7)^*$, an external ammeter A (274-13-23) and voltmeter V (806-14-28), a 1 KVA transformer T (519-38-1), a .02 micro-farad maximum capacitance condenser C, and a variable induction coil I. The variac is connected so that it will not have an output greater than 110 volts, which is the maximum voltage for the primary of the transformer. The maximum voltage of the secondary of the transformer is 25,000 volts. The peak voltage of the condenser is 20,000 volts. The induction coil is of a suitable size to eliminate air lines from the spectrum.

In Fig. II, the optical arrangement of spark source and spectrograph is shown. The optical bench supporting the spark electrode stand is directly in line with the slit S of the spectrograph. The lower electrode E is a carbon rod 1/4 in. in diameter. The upper electrode A is a cast aluminum block. The spark source is focused by means of a quartz plano-convex lens 1. The plane surface of the lens is 90 mm. from the center of the carbon electrode and the convex surface of the lens is 814 mm. from the slit housing of the spectrograph. The rotary sector disc R is placed 510 mm. from the slit housing of the spectrograph. The logarithmic sector H is placed as close as possible to the slit. (1)

Light passes from the slit S through the prism P to the lens L to the dispersing prism O. The rear surface of this prism is coated with aluminum which reflects ultraviolet and visible radiation without

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^{*}Physical Chemistry Department Numbers



Fig. I (a) Condensed Spark Circuit (b) Variac Panel



Fig. II Optical system







Fig. IV Optimum position of the electrode images in the logarithmic sector housing absorption bands. (2) (3) The light is reflected and dispersed by this prism and, after passage again through L, forms the spectrum in the curved plane shown.

The method used in aligning the spark source is as follows: the source used in the alignment was the same type as was used in subsequent analyses, i.e. a carbon rod as the lower electrode and a cast aluminum block as the upper electrode. The optical bench was set level and the electrode stand was set at an angle of 90° to the optical bench. The plano-convex lens 1 was removed from the optical bench, the Hartmann diaphragm was removed from the spectrograph, the plate holder was removed from the spectrograph, the prism was set to a visual spectrum, and the alit of the spectrograph was opened to a width of 1 mm." The spectrum of the spark source was viewed from the position of the plate holder while the electrodes were adjusted until the lines of the spectrum were sharp and cut off evenly both at the top and the bottom of the spectrum. An electrode gap of 3 mm. was maintained for all adjustments. Any latteral adjustment of the electrodes was made by carefully shifting the remote end of the optical bench, the knob for the lateral adjustment of the electrodes was maintained at its center position. The plano-convex lens was placed on the optical bench near the electrodes. This produced a circular image on a white card placed in front of the spectrograph lens L. This image should be sharp and coincident with the spectrograph lens. For the lateral adjustment of the image, the remote end of the optical bench was shifted to the right to move the image to the right and vice versa. The plano-convex lens was moved forward and backward on the optical bench until the circular image was sharp and intense.

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^{*}One complete revolution of the drum.

The slit of the spectrograph was set to a drum reading of 7* and the focus and tilt were set for position V.** The plate holder was loaded and four exposures were made to determine the vertical position of the plano-convex lens for even illumination of the slit which is essential in using a logarithmic sector. (1) The optimum, position of the images of the electrodes on the logarithmic sector housing obtained by vertical adjustment of the plano-convex lens is shown in Fig. III.

An optional method to be used in aligning the spark source can also be used. The optical bench and electrode stand should be adjusted as in the preceding method. The Hartmann diaphragm was removed from the spectrograph, the plate holder was removed from the spectrograph. the prism was set to a visual spectrum and the slit of the spectrograph was opened to a width of 2 mm. A light bulb was set at the red end of the spectrum at the position of the plate holder. The image from the slit was viewed on a white card and traced to the position of the electrodes. The optical bench was adjusted laterally until the image was in the center of the plano-convex lens. The rotary sector disc, the plano-convex lens, and the electrodes were adjusted vertically with respect to the image. The plano-convex lens was adjusted backward and forward until the image from the slit was focused on the tip of the carbon electrode. Then by starting the spark the image of the electrodes will be in focus on the collimating lens of the spectrograph. The correct vertical position of the plano-convex lens was adjusted as in the preceding method.

The upper electrode with complete dimensions used in this work is shown in Fig. IV. It was made from brass stock and was especially

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^{*}Approximately 40 microns.

^{**}Focus-111, Tilt-245; 2549-3641 A

designed to support the cast aluminum samples.

The spectrograph used was Bausch and Lomb large Littrow mounting with a quartz optical system.

A Bausch and Lomb projection densitometer was used for all line blackness comparisons.

PROCEDURE

A primary prerequisite for the quantitative analysis of aluminum alloys is the preparation of the electrodes. The aluminum blocks should have a clean, smooth surface which was made by surfacing it in a lathe. The smooth surface permitted the sample to rest squarely on the electrode holder. The carbon electrodes were shaped by means of a hand pencil sharpener and flattened off to a point .093 in. in diameter by inserting the carbon through a gauge .093 in. in diameter and rubbing the excess point off on a fine emery paper followed by rubbing it over a clean paper. Thus each carbon electrode has the same size sparking surface. An electrode surface of .093 in. permits good sampling compared to a fine pointed carbon electrode. (4) The carbons for each sample were placed in separate envelopes and never interchanged to avoid contamination. The upper electrode holder was polished with a fine emery paper to ensure a good contact between the holder and the sample.

A step density tablet pattern was exposed on each plate in the darkroom before it was placed in the plate holder. After the plate holder was loaded it was set to the position of the first exposure. Before making any exposures the slit width was set at a drum reading of 7 and the focus and tilt were set for position V. The rotating sector disc should be started if it is to be used. The speed of rotation must be

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fast enough to eliminate the intermittency effect of the photographic emulsion, and yet not so fast that the vibration of the wheel impairs the sharpness of the lines.

The carbon electrode, held by means of a nickel crucible tongs, was inserted into the lower electrode holder, which was spread open by means of a screw driver. A 3 mm. gauge was placed on the top electrode holder and the carbon electrode was raised until it contacted the gauge. Then the gauge was replaced by the aluminum sample. The spark was started by turning the handle of the variac in a clockwise direction until the voltmeter registered 80 volts. The ammeter registered 10.4 amp. The slit of the spectrograph was closed for the first 30 seconds after the spark was started.

Table I shows the change in the value of the Log E_x/E_y ratio for increasing period of prespark. The ratio remained constant for the

TABLE I

Variation of Log E_x/E_y with increasing prespark period.

Time Prespark	of (sec.)	Log E _x /E _y
30		0.41
75		0.41
120		0.50
165		0.63
210		0.61

first 75 seconds of prespark after which it increased. Since time is an important factor in spectrographic analysis, it was decided to use 30 seconds instead of 75 seconds. This 30 second period also makes

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allowances for variation in the size of the samples, since smaller samples may reach their equilibrium sooner than the larger samples. During this presparking period the source was properly aligned by means of the lateral adjustment of the electrode stand. The source is properly aligned when its image covers the quartz window of the logarithmic sector housing. The shutter of the spectrograph was opened for the previously determined time of exposure. After completion of the exposure, the handle of the variac was turned counterclockwise to its 0 position. The electrodes were replaced by the next sample to be exposed and the plate holder was set for the next position of exposure. The same procedure was repeated for each sample to be exposed.

Eastman spectrum analysis #1 plates were used for all analyses. The plates were exposed from the bottom to the top. Sixteen separate exposures were made through the #3 or center opening of the Hartmann diaphragm. For the exposure of the logarithmic sector pattern of one of the aluminum alloys, the Hartmann diaphragm was removed to utilize the full height of the spectrograph slit. (5) The slit width was set at a drum reading of 7** for all exposures including the logarithmic sector pattern.

After the exposures were made on the plate, the plate was processed according to the standardized procedure necessary for duplication of results. The plate was developed for 4 minutes in E_astman D-11 developer maintained at a constant temperature of 18° C. by means of a water thermostat. The plate was agitated 5 seconds of every minute of development. After development, the plate was washed in water for 15 seconds and placed in the fixer for 5 minutes. The plate was agitated

^{*}See page

^{**}Approximate 40 microns.

the first and last ten seconds of the fixing period. The developer and fixer were contained in upright glass vats 2x4x12 in. The plate, having been fixed, was washed for 15 minutes in running water, rinsed in distilled water, sponged off, and dried under a ventilation hood.

When the plate was dried, the various lines* to be measured on the densitometer were spotted and labeled on the plate. The densitometer must be turned on one half hour before it is to be used. (6) The scale of the densitometer has 38 divisions and the galvanometer was connected so that it registered a deflection between 36 and 38 for no illumination and deflected towards the zero end of the scale for full illumination passing through the densitometer. Thus the readings of the various lines are in terms of blackness. The position of the deflection for no illumination is known as the galvanometer zero and should remain constant throughout the reading of a plate. The galvanometer zero should be set to the same position for each plate because it has a definite effect upon the deflections obtained when reading the blackness of the lines. The plate is mounted in a mechanical stage between the illuminating chamber and the projection lenses of the densitometer with the glass side of the plate toward the operator.

The lines of the spectra were properly focused and the galvanometer deflection with the light passing through a clear portion of the plate was set to zero by means of the iris diaphragm. This setting is known as the clear plate reading and should be checked at least after reading the lines of each spectrum. The same spot on the plate should be used for the clear plate reading every time it is set. Before measuring the blackness of a line, the slit was made parallel to the line

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^{*}See Table III.

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by means of the adjustment provided. The galvanometer deflections for each line were read by traversing the line very slowly across the slit until the maximum deflection is obtained. The line of the element being analyzed and the reference line were read for each spectrum before proceeding to the next spectrum as compared to reading the reference line in every spectra and then reading the line of the element under analysis in every spectra. With this method any variations due to the apparatus which would be present in the reading of one line of the spectrum would be apt to be present in the reading of the other line or lines of that spectrum.

TREATMENT OF DATA

Table II shows a typical set of data for the spectrographic determination of magnesium in 14S and 17S aluminum alloys. The characteristic curve of the emulsion of the plate shown in Fig. V was ovtained by plotting the galvanometer deflections as ordinates against the step number of the step density tablet as abscissa. The step numbers are proportional to Log E, where E is relative exposure. The working curve shown in Fig. VI was obtained by plotting Log $\mathbf{E}_{M\sigma}/\mathbf{E}_{A1}$ against Log % Mg. Log \mathbf{E}_{Mg} was obtained by referring the galvanometer deflection of the magnesium line to the characteristic curve from which its corresponding Log E value on the horizontal axis was read. This procedure was repeated for the reference line. Then by subtracting $\log E_{A1}$ from Log E_{Mg} , Log E_{Mg}/E_{A1} was obtained. The working curve of Fig. VI was constructed from the data of the three standards SA268, SS17, and SA376. Then by referring the Log E_{Me}/E_{A1} ratio for each sample to the working curve, the corresponding log % magnesium was obtained and converted to % magnesium.

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Fig. V - Characteristic curve from the step density tablet of plate $\neq 100$.



Fig. VI - Working curve for the quantitative analysis of magnesium in 148 and 178 aluminum alloys for plate #100.

TABLE II

Data from the spectrographic determination of magnesium in 14S and 17S aluminum alloys for plate #100.

Sample	Al 3	060	Mg 2780	Log	EAI	Log E	Log]		Log %	Me %	Mg
15	14.	65	6.55	5.	.84	4.82	-1.	.02	-,58	4 •	26
SA 268	13.	55	6.75	5.	.74	4.88	-0.	.86	-,53	8 •	29
16	13.	95	10,25	5.	.79	5.39	-0,	.40	40	7.	39
13	16.	00	12.25	5.	.97	5.61	-0.	.36	39	5.	40
8	14.	40	11.50	5.	.81	5.53	-0,	.28	37	4•	42
14	16.	70	13.95	6.	.00	5.79	-0.	21	35	2.	44
12	14.	55	13.45	5.	.83	5.73	-0.	10	32	2 •4	48
7	14.4	45	13.35	5.	82	5.72	-0.	10	32	2 •4	48
3	16.0	00	18.35	5.	97	6.10	+ 0.	13	25	6 •!	56
SS17	15.0	65	18.85	5.	92	6.13	+ 0.	21	23	7.	58
2	15.	50	19.35	5.	90	6.17	+ 0.	27	21	8.6	60
1	14.	75	19.45	5.	86	6.18	+0.	32	20	4.6	63
5	16.0	05	19.25	5.	98	6.16	+0.	18	24	2 •{	57
11	14.'	75	20.65	5.	86	6.25	+ 0.	39	18	2.6	66
9	15.7	70	24.35	5.	92	6.52	+ 0.	60	12	3.7	75
SA376	15.7	70	25 .75	5.	92	6.65	+0.	73	08	5 . 8	32
Step # Galvanom	eter	1	2	3	4	5	6	7	8 75 65	9	10
Deltect	lon	0.00	0.00	T*M	ພ. 30	7.DU	TO . 00	23.00	00.00	01.CO	31.40

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In the determination of magnesium in the 14S and 17S aluminum alloys the best results were obtained by using the characteristic curve plotted with the data for the step density tablet as compared to the characteristic curve obtained from the logarithmic sector pattern. A typical characteristic curve obtained from the logarithmic sector pattern is shown in Fig. VII.

The proper exposure time is an important factor in any spectrographic analysis. In any case, the exposure should be such that the blackness of the reference line and the alloy line is about equal when the percentage of the alloying element is average. Thus, no error can result when determining the Log $E_x E_y$ ratio in spite of extreme error in the slope of the characteristic curve. On the other hand, if there is a large difference between their blackness even slight error in the slope of the characteristic curve can cause appreciable error in the Log $\mathbf{E}_{\mathbf{x}} / \mathbf{E}_{\mathbf{y}}$ ratio. Another factor to be considered in determining the exposure time is that the best results are obtained when the straight portion of the characteristic curve is used. This straight portion of the characteristic curve covered a range of 5-25 in terms of galvanometer deflections. In keeping with the foregoing, the percentage range to be covered in the analysis will be governed by the fact that the blackness of the alloy line should be kept within the deflection range of 5-25. It follows that the reference line should have a blackness which will give a deflection of 13-16 inasmuch as this would be the deflection of the alloy line at its average percentage.

The first step in determining the exposure time to make a survey plate of all or at least a representative number of the samples to be analyzed using the same exposure time for each sample. From this

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plate the two samples having the smallest and largest amount of the alloying element to be determined were identified. A second plate was made on which the exposure time for the two trial samples was varied so that the proper exposure time could be determined by measuring the alloy lines of each spectrum of the two samples for the various exposure times. The exposure time which gave the proper galvanometer deflections for the alloy line of the two samples was used for all the samples to be analyzed within the percentage range represented by the two samples.

The exposure time for any percentage range should be checked from time to time due to any changes which may have taken place. It has been found that changes in the atmosphere cause variations in operating conditions. (6) After every sample within a given percentage range has been analyzed the approximate galvanometer deflection will be known for the alloy line of each sample. Thus any subsequent checks on the exposure time for this particular percentage range can be made by varying the exposure time for any one of the samples. The exposure time that gives the proper galvanometer deflection for the alloy line of this sample can be used for all the samples as above.

The spectrum lines that can be used in the spectrographic analysis of aluminum alloys have been worked out to completion. The percentage range for which a line can be used will vary from one unit of laboratory equipment to another, however, the approximate percentage range for which each line can be used has been determined. The spectrum lines used and the percentage range they covered in this work are given in Table III. The only analysis for which the optimum exposure conditions were realized was that for 0.2-0.9% magnesium. The alloy line had a galvanometer deflection of 5-25 and the aluminum reference line had a

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TABLE III

Spectrum lines used in the quantitative analysis of 14S and 17S aluminum alloys with their approximate percentage range of analysis and galvanometer deflections.

Alloying Element	Percentage Range	Alloy Wavelengt	Line h Gal. Def.	Aluminum Wavelength	Ref.Line Gal.Def.	Corresponding Working Curve
Mg	0.2 - 0.9	2780	5-25	3060	13-16	Fig. VI
Te	0.2 - 0.9	2743	10-21	3050	22.25	Fig. VIII
Si	0.2 - 1.3	2882	6-25	3050	22.25	Fig. IX
Mn	0.4 - 1.0	2888	6-23	3050	22.25	Fig. I
Mg	0 - 0.6	2780	5-25	3060	22.25	Fig. XI
Fe	0.4 - 2.0	2743	2-22	30 57	20-23	Fig. XII

galvanometer deflection of 13-16. In the analysis of 0-0.6% magnesium the galvanometer deflections were in order for the magnesium line, but those for the reference line were high. This was due to the fact that the 0-0.6% range of magnesium required a longer exposure time than the 0.2-0.9% range of magnesium.

The 3060 A aluminum line is used as the reference line whenever possible in the analysis of aluminum alloys. However, there are instances when it is necessary to use an aluminum line other than the 3060A. In the determination of iron, silicon, and manganese, it was necessary to use the 3050A aluminum line as the reference line. The same exposure time was used for the analysis of iron, silicon, and manganese, however, this exposure time was less than that used for the analysis of magnesium. Thus the 3060 A line gave a deflection of less than 5 and it was necessary to select another aluminum reference line which had a deflection within the range of 5-25. In the analysis of iron, 0.4-2.0%, both the 3060A

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TABLE IV

Data for Fig. VII to XII.

Fig. VII

Step #	l	2	3	4	5	6	7
Deflection	3.80	9.60	17.40	24.55	28.30	30.80	32 .35

Fig. VIII

Fig. IX

Sample	Log E _{Fe} /EAL	Log 🖇 Fe	% Fe	Sample	Log E _{Si} /E _{Al}	Log 🖇 Si	% S1
SA 275	-1.89	538	.29	SA 275	-2.16	 569	.27
SS17	-0.35	252	•56	S S17	-1.60	-,444	•36
SA376	+0.34	119	.76	SA376	+1.04	+ .029	1.07

Fig. I

Fig. XI

Sample	Log E _{Mn} /E	Log % Mn	5Mn	Sample	log E _{Mg} /E _{Al}	Log % Mg %Mg	
SS17	-1.51	276	•53	15	-0.75	585	.26
SA275	-0.70	114	•77	7	-0.01	 357	.44
SA 376	-0.50	086	.82	3	+ 0.25	276	.53

Fig. XII

Sample	Log E _{Fe} /E _{Al}	Log 🖇 Fe	% Fe
14	-2.93	347	•45
7	-2.51	252	•56
SA376	-1.96	119	•76



Fig. VII - Characteristic curve from the logarithmic step sector pattern of the aluminum 3057 line for plate #147.



Fig. VIII - Working curve for the quantitative analysis of iron in 14S and 17S aluminum alloys for plate #109.



Fig. IX - Working curve for the quantitative analysis of silicon in 145 and 175 aluminum alloys for plate #112.



Fig. I - Working curve for the quantitative analysis of manganese in 148 and 178 aluminum alloys for plate #110.



Fig. XI - Working ourve for the quantitative analysis of 0 - .05 magnesium in aluminum alloys for plate #133.



Fig. XII - Working curve for the quantitative analysis of .4 - 2.00% iron in aluminum alloys for plate #144.

and the 3050A lines were too light and it was necessary to use the 3057A aluminum line. Table IV includes the data for the curves in Figs. VII to XII. The characteristic curve from the logarthmic sector pattern of the 3057A aluminum was used in the analysis of iron, silicon, and manganese.

Six separate analyses were made for each alloying element analyzed in each sample. Results typical of those obtained in the various analyses are shown in Table V.

TABLE V

Results of six separate analyses and their mean values for magnesium in 14S and 17S aluminum alloys.

			% Mg				Mean %
Sample	1	2	3	4	5	6	Mg
3	•53	•56	•53	•52	•49	•52	•53
6	•66	.61	•62	•59	.61	.62	.61
11	.67	•66	•6 6	.71	•68	•66	•67
15	.24	.26	•25	•25	• 25	•29	•26
14S	.31	•30	.29	.28	•29	•29	• 30
175	•49	.51	•53	.48	.51	.51	.51

Table VI shows the final mean value for all analyses. It

also includes the chemical analyses furnished with the samples.

TABLE VI

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Spectrograph	ic and	chemical	analyses	of	each	alloying	element
analyzed in each	sample.	•					

	% M	g	% S	i	%I	'e	% Mn		
Sample	Spec.	Chem.	Spec.	Chem.	Spec.	Chem.	Spec.	Chem.	
1	•60	•48	•34	•24	•33		•64	.57	
2	•59	•49	•34	• 33	•38	•41	•63	•59	
3	•53	.78	.37	•34	•42	•46	•6 4	•60	
4	•66	. 67	•44	.42	•44	.41	•64	•57	
5	.62	•55	•41	•40	•37	•35	•60	•61	
6	.61	•67	•39	.41	•40	•68	•66	•59	
7	•44	•43	.41	•42	•56	•52	.63	•60	
8	•42	•34	.61	•49	•49	•47	•75	•59	
9	.81	•92	. 48	.47	•33	•36	•46	•41	
10 .	•42	•42	•34	•39	•4 5	•45	•65	.61	
11	•67	•60	.42	•37	.42	•42	.61	•59	
12	•43	•43	.80	•69	.37	•42	.80	.71	
13	•38	•38	.73	•70	.42	•54	.79	.71	
14	.41	•38	.82	.70	•45	•42	.80	•69	
15	•26	.26	.70	•65	.4 8	•59	•74	•73	
16	.37	•45	.72	.73	.43	•69	•83	.71	
14S	•30		•63		.37		•73		
175	.51		•45		•43		•66		
R115	.15				1.24				
R112	.13				1.22				
R77	•089				1.59				
R84	.097				1.74				
R99	•22				1.53				
R 98	.097				1.74				

DISCUSSION

Spectrographic analysis is readily assuming an important position in a large percentage of the modern industries. This is especially true in the production of aluminum alloys. Therefore, it is anticipated that the detailed procedure given in this paper will enable anyone in this laboratory with a fundamental knowledge of spectroscopy to gain experience which will be of exceeding value to them in any future spectrographic work. The method used in this laboratory is similar to those used by various industries.

The accuracy of the method used in this work is considered to be within 5% of the amount present. (1) (5) The majority of the results obtained in the analyses as shown in Table V are within 5% of the mean value. When the accuracy is not within 5% of the mean value it has been found that the mean value is always in the correct order. Such deviations from the mean value are attributed to segregation which is common to cast structures. (7)

In order to conserve the primary standards, the samples analyzed were designated as secondary standards and were used in determining the working curve for additional analyses as in Figs. XI and XII. The values obtained in the spectrographic analyses shown in Table VI were used to plot these curves. It was impossible to obtain a straight line for the working curve when the values of the chemical analyses were used. Therefore, the spectrographic values were used to plot all working curves. The chemical analyses are given in Table VI for comparison only.

The R-series of samples were beyond the range of the primary and secondary standards, i.e., their concentration of magnesium was less than that of the standards and their concentration of the iron was

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greater than that of the standards. However, in order to analyze them the working curves obtained from the standards were extrapolated, shown in Figs. XI and XII. The results were low, but better results would have been obtained if their ranges of concentration had been within that of the standards.

SUMMARY

- 1. A method of alignment of the spark source has been given.
- A detailed procedure of spectrographic determination of 14S and 17S aluminum alloys has been presented.
- 3. The characteristic curves and the working curves for the determination of magnesium, iron, silicon, and manganese in 14S and 17S aluminum alloys have been shown.

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