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SPECTROGRAPHIC DETERMINATION OF BORON IN  
STEEL<sup>1</sup>

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## ABSTRACT

The spectrographic determination of small amounts of boron in thin steel rods and in massive pieces of steel was investigated by using several types of arc-like discharges. The sensitivity and accuracy in determining boron were found to be affected by variations in the rate of cooling of the electrodes. With  $\frac{1}{32}$ -inch-rod electrodes, the high-voltage alternating-current arc permits determinations down to 0.0006 percent of boron, with an average deviation of  $\pm 4$  percent. With more massive specimens the high-voltage alternating-current arc is insensitive, but an overdamped condenser discharge or similar source provides adequate sensitivity and precision. Determinations as low as 0.0001 percent of boron are made with a direct-current arc to which sodium is added to suppress an interfering Fe II line. In the course of the investigation, a series of six boron steels in two sizes,  $\frac{1}{32}$ - and  $\frac{1}{2}$ -inch rods, were issued as National Bureau of Standards Standard Samples covering the range 0.0006 to 0.019 percent of boron.

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## I. INTRODUCTION

The addition of a few thousandths percent of boron to certain steels results in an increase in the hardenability (depth of hardening). This property suggested the possibility that boron could be substituted for considerably larger portions of strategic alloying elements in steel and, consequently, intensive studies of boron steels were made during the war period. Boron is commonly added to steel in the ladle or ingot molds in the form of ferrobore or by other addition agents containing various beneficial elements. In view of the low concentration of boron required for optimum hardening, usually 0.001 to 0.003 percent, and the effect of variations in concentration, accurate and rapid methods were required for its determination. This problem was brought to the Bureau's attention in connection with an investigation of the properties of boron steels by the Metallurgy

<sup>1</sup> Presented at the meeting of the Optical Society of America on October 19, 1945.

Division of this Bureau, the investigation being sponsored by the War Metallurgy Committee of the National Research Council, National Academy of Sciences. At the request of these groups, and with funds allotted by the Office of Production Research and Development of the War Production Board, methods were developed for the spectrographic determination of boron in solid steel specimens and, in addition, standard samples were made available for the use of other laboratories. A concurrent investigation of the chemical determination of boron by the Chemistry Division of this Bureau permitted intercomparison of methods and provided the chemical determinations essential to the certification of standards. The methods were applied in the analysis of armor plate and of other samples involved in metallurgical investigations.

Methods for the spectrographic determination of boron in various materials have been reported in the literature. For application to steels, Masi [1]<sup>2</sup> described a method employing solutions with spark excitation for determinations in the range 0.03 to 2.0 percent of boron. Schliessmann [2] investigated the sensitivity of detection of a number of components of steels, including boron, using solutions and arc excitation. His results indicated a sensitivity of 0.005 percent for boron. Recently, Gegechkori and Fal'kova [3] described a spark method similar to that of Masi for determinations of boron in the range of 0.1 to 1.0 percent and also an a-c arc method employing filings of the steel sample for the range 0.001 to 0.1 percent of boron. None of these methods employed the steel sample in solid form and all required time-consuming preliminary preparations. Irish [4] has recently described the application of the a-c arc method to the rapid determination of boron in the range 0.002 to 0.05 percent of boron, using rod samples. This method is quite satisfactory for control analysis where samples may be cast in reproducible shape, such as rods  $\frac{1}{2}$  inch in diameter. However, the method must be employed under closely specified conditions, and its sensitivity falls off markedly if the size of the sample is increased. The methods to be described are applicable to rod or massive specimens in control testing and in the testing of fabricated parts. The requirement for the methods, that standard samples be available in suitable form, was met by the certification of six National Bureau of Standards Standard-Sample rods  $\frac{1}{2}$  inch in diameter and a corresponding set of six standards,  $\frac{1}{2}$  inch in diameter, each set covering the range 0.0006 to 0.019 percent of boron.

## II. DETERMINATION OF BORON

### 1. GENERAL ASPECTS OF THE PROBLEM

The concentrations of boron commonly encountered in steels produced in this country are near the limit of spectrographic detection by the usual methods of excitation. As spark excitation is useful only for amounts of boron above 0.005 percent, arc excitation is employed under conditions adjusted to obtain maximum sensitivity. In order to extend the range of determination to 0.001 percent of boron or less, the limiting factors of interference by spectral background and by neighboring iron lines must be minimized. The unfortunate inter-

<sup>2</sup> Figures in brackets indicate the literature references at the end of this paper.

ference of the iron line Fe II 2497.82 Å with the strongest boron line B I 2497.73 Å renders the latter line unsuitable for analytical purposes under many observing conditions, and the next strongest boron line, B I 2496.78 Å, is therefore employed. If the resolving power available in the spectrograph is sufficient to separate the first two lines, an increase in sensitivity up to a factor of two would be realized by use of the strongest boron line. Another possibility is the reduction of intensity of the interfering Fe II line with respect to the strongest B I line. Because of the higher energy required for excitation of the Fe II line as compared to the B I line, the former may be suppressed by employing an excitation source of low energy. Advantage is taken of this phenomenon, when the highest sensitivity is desired, by employing a d-c arc to which sodium is added to maintain low-level excitation.

Another factor of considerable importance in the determination of boron is its volatility (probably as oxide), which is appreciably higher than that of iron; this characteristic is both helpful and troublesome. It permits enrichment of the vapor by boron under certain excitation conditions, thus increasing the sensitivity of detection. A disadvantage lies in the care required to ensure reproducible heating of the electrodes to obtain accurate determinations. Variations in heat conduction from the discharge points on the electrodes may lead to appreciable errors in the analytical results. Because of the effects of heat conduction, it was found necessary for optimum results to apply different excitation procedures in determining boron in thin rods and in more massive pieces.

The existence of boron in complexes insoluble in acids was observed in the determination of boron by chemical analysis. As much as one-half of the boron may remain in the minute acid-insoluble residue when the total concentration of boron lies in the range of a few thousandths percent or less. The procedure for chemical analysis, therefore, provides for alkaline fusion of the insoluble material to incorporate it in the solution. This time-consuming operation would necessarily be involved in a solution method for spectrographic analysis and consequently renders this procedure impracticable in comparison with a method of direct excitation of the steel sample. The existence of boron in acid-soluble and acid-insoluble forms presents the possibility that these forms may differ in volatility in spectral excitation and may affect the spectrographic determinations. However, a comparison of chemical and spectrographic results for samples having different fractions of insoluble boron has not disclosed an appreciable effect on the spectrographic results.

## 2. DETERMINATION OF BORON IN THIN-ROD SAMPLES

The determination of boron in thin-rod samples, for example, rods  $\frac{1}{32}$  inch (5.6 mm) in diameter, may be accomplished with satisfactory sensitivity and accuracy by means of the usual arrangement of a 2,500-volt 10-kw a-c arc.<sup>3</sup> Under the conditions employed here for the line B I 2496.78 Å, the method exhibits a sensitivity of 0.0006 percent of boron, with an average deviation of  $\pm 4$  percent of the

<sup>3</sup> A 4-ampere d-c arc was found sensitive to 0.001 percent of boron with light from the entire arc stream or to 0.0006 percent of boron with light taken from a portion of the arc stream nearest the positive electrode. Determinations by this procedure, however, are not as precise as those obtained with the a-c arc.

amount present. The conditions employed in the a-c arc method are as follows:

*Electrodes.*—The tips of two rods  $\frac{1}{32}$  inch (5.6 mm) in diameter are ground to a 150-degree cone (the cone angle should be standardized throughout the procedure, but the value selected is not critical).

*Mounting of electrodes.*—The two rods are mounted in clip holders, with each electrode protruding a fixed distance of 12 mm or more from the holders to minimize heat conduction. The analytical gap is set at 1.8 mm.

*Excitation.*—A 2,500-volt a-c arc with current of 4.5 amperes is used.

*Spectrography.*—A large Littrow quartz spectrograph with a slit width of 20 microns is employed with an Eastman Spectrum Analysis No. 1 plate.

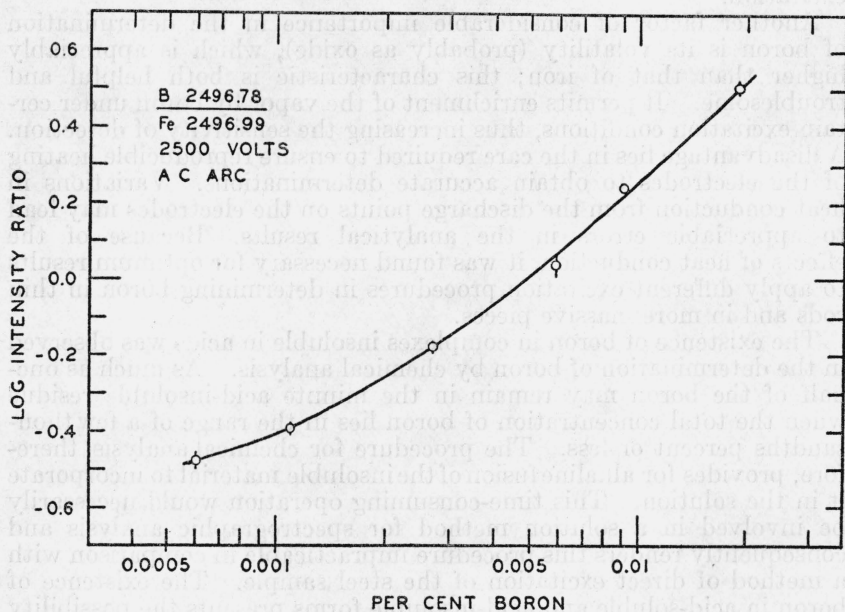


FIGURE 1.—Analytical curve obtained with  $\frac{1}{32}$ -inch-diameter National Bureau of Standards spectrographic boron steel Standard Sample rods and the alternating-current arc.

*Exposure.*—The exposure period is 90 seconds.

*Photometry.*—Photometric measurements are made with an effective slit width of 16 microns. A group of iron lines at 2800 Å, having relative intensities previously determined by a step-sector method, is measured for plate calibration. From photometric measurements and the calibration curve, the intensity ratio of  $\text{Bi}2496.78 \text{ Å}$  and  $\text{Fe}12496.99 \text{ Å}$  is obtained.

The analytical curve shown in figure 1 was constructed from the mean values of four determinations on each of the six National Bureau of Standards boron-steel Standard Samples Nos. 425 to 430, inclusive. The spread of the runs for each standard is indicated by the length of the vertical line at each point; the mean of a set of values is given by the small circle. In order to accommodate the full range of concen-

trations to the latitude of the plate, it was found necessary to reduce the intensity of the boron line above 0.006 percent by means of a step sector or filter. Determinations usually fall below this concentration in routine analysis. The precision of the method is demonstrated in table 1 by the results of repeated runs on the same sample.

TABLE 1.—Precision of 2,500-volt a-c arc method  
[Seventeen runs of NBS Standard Sample 427 on one plate.]

Boron content	Deviation	Boron content	Deviation
<i>Percent</i>	<i>Percent</i>	<i>Percent</i>	<i>Percent</i>
0.00282	0.00012	0.00262	0.00008
.00256	.00014	.00288	.00018
.00271	.00001	.00286	.00016
.00261	.00009	.00269	.00001
.00270	.00000	.00266	.00004
.00281	.00011	.00253	.00017
.00277	.00007	.00258	.00012
.00278	.00008	.00262	.00008
.00275	.00005		
		<sup>1</sup> 0.00270	<sup>1</sup> 0.00009
Average deviation from the mean, $\pm 4\%$			

<sup>1</sup> Average.

The accuracy of the results is difficult to estimate. The agreement of the plotted points with the smooth curve in figure 1 indicates an accuracy approaching the precision of the spectrographic results in these cases where carefully determined chemical values are available for comparison. In routine analyses, a comparison of spectrographic determinations (average of three runs) and single runs by chemical analysis showed an average deviation of  $\pm 0.0003_8$  percent, or  $\pm 19$  percent of the amount present.

The sensitivity of detection of boron in steel with the a-c arc source depends on the temperature of the molten pool from which the steel is being vaporized into the arc stream. As the temperature is raised, the ratio of boron to iron vapor in the arc steam is increased. The temperature in turn depends on the rate of cooling of the rods and is markedly affected by the method of supporting the electrodes and by their diameter. This can be demonstrated by supporting two  $\frac{7}{32}$ -inch rods in closely fitting water-cooled copper clamps. The temperature of their tips will depend on the length of rod projecting from the clamps. In figure 2 the intensity ratio of boron to iron is plotted as a function of the distance to which the tips of the rods projected from the clamps. The marked effect of the variation of electrode temperature is apparent in the figure. At projections greater than 14 mm the temperature of the tips was unaffected by variations in the distance of projection as most of the cooling was by radiation and conduction into the air rather than by conduction through the rods into the clamps. This figure illustrates the importance of a proper method of supporting the electrodes and its possible effect on the sensitivity and the accuracy of the determinations.

Figure 3 shows another aspect of the same effect. Here the temperature of the tips of the rods decreased as the rod diameter was increased. As in figure 1, the intensity ratio of boron to iron is great-

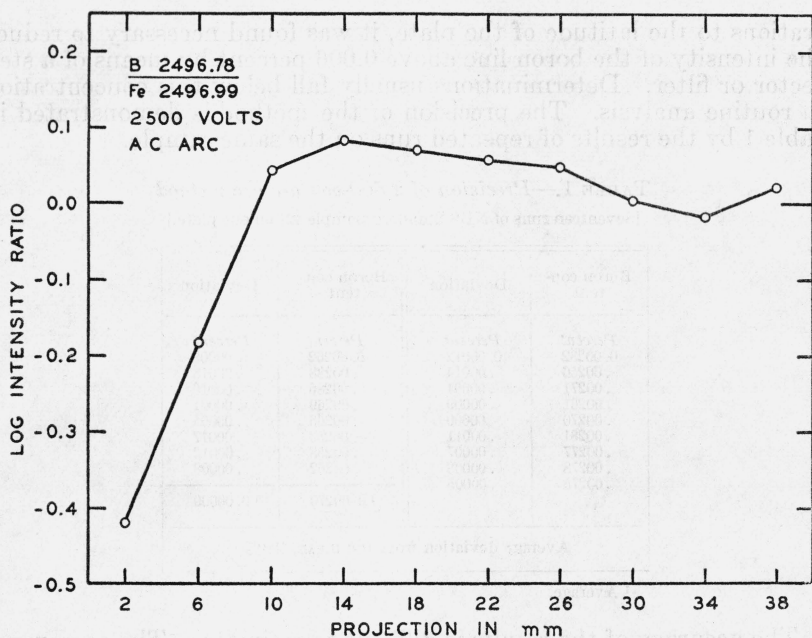


FIGURE 2.—Effect of variation in projection of  $\frac{1}{32}$ -inch-diameter rods from water-cooled copper clamps on the intensity ratio of boron to iron lines in the alternating-current arc.

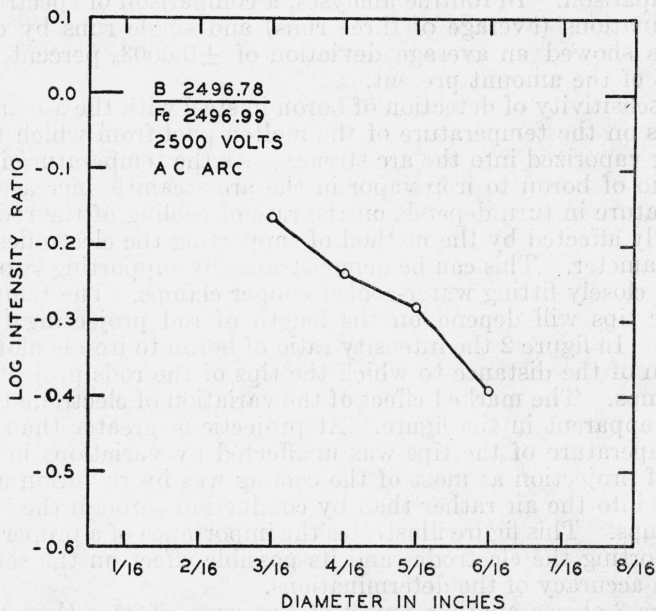


FIGURE 3.—Effect of variation of rod diameter on the intensity ratio of boron to iron lines in the alternating-current arc.

est where the temperature is highest. For this reason the ordinary 10-kw a-c arc provides inadequate sensitivity for determinations of boron on large samples, such as rods  $\frac{1}{2}$  inch in diameter or heavy fabricated pieces.

### 3. DETERMINATION OF BORON IN MASSIVE SAMPLES

If the steel samples are in the form of irregular massive pieces, it is advantageous to have a method for the determination of boron that does not require machining the steel to thin rods having a closely controlled diameter. The high-voltage a-c arc provides a sensitivity of only 0.005 percent with the boron line 2496.78 Å when applied to massive pieces of steel, and therefore other modes of excitation were investigated. Three sources were found useful, namely, an overdamped condenser discharge, a high-current a-c arc, and a high-current d-c arc of low excitation energy. The procedures to be described for these excitation sources are applied with the point-to-plane electrode technic, in which one electrode is the steel sample having a machined flat surface at least  $\frac{1}{2}$  inch in diameter and the other electrode is a graphite rod.

#### (a) OVERDAMPED CONDENSER DISCHARGE

A commercial source of excitation, the Multisource unit, providing a wide selection of discharge conditions has been described by Hasler [5]. One mode of excitation involving the discharge of a condenser bank through relatively high values of inductance and resistance results in an overdamped discharge with favorable characteristics for the determination of boron. The method exhibits a sensitivity of 0.0006 percent of boron, with an average deviation of  $\pm 5$  percent of the amount present and thus, with irregularly shaped steel specimens, matches the results obtained by the high-voltage a-c arc as applied to thin-rod samples. The conditions employed are as follows:

*Electrodes.*—A flat surface having a diameter of  $\frac{1}{2}$  inch (13 mm) or more is machined on the steel specimen. A counter electrode is prepared from  $\frac{1}{4}$ -inch (6.3 mm) boron-free graphite by cutting a 120-degree cone on the end.

*Mounting of electrodes.*—The steel electrode (positive) is mounted with its flat surface in a horizontal plane; the counter electrode (negative) is mounted vertically with an electrode gap of 6 mm. The half of the electrode gap above the steel electrode is centered on the optical path, and the image of this portion of the discharge is projected to the collimator lens of the spectrograph by means of a lens ( $f=34$  cm) at the slit of the spectrograph.

*Excitation.*—An overdamped condenser discharge of the Multisource unit is employed with capacitance of 60  $\mu$ f, inductance 480  $\mu$ h, resistance 50 ohms, charging voltage 940 volts, and full initiator power.

*Spectrography.*—A large Littrow quartz spectrograph with a slit width of 20 microns is employed with an Eastman Spectrum Analysis No. 1 plate.

*Exposure.*—The exposure period is 90 seconds.

*Photometry.*—Photometric measurements are made as described for the a-c arc method, except that Fe I 2487.37 Å is employed.

The analytical curve shown in figure 4 was constructed from the mean values of three determinations on each of the six National Bureau of Standards Standard Samples 825 to 830, inclusive, having a diameter of  $\frac{1}{2}$  inch. The agreement of the points with the smooth curve indicates an accuracy approaching the precision of the spectrographic determinations. The precision of the method is demonstrated in table 2 by the results of repeated runs on Standard Sample 826.

Comparison of routine spectrographic determinations (average of three runs) and chemical determinations, by single runs, for boron in 24 steel samples showed an average deviation of 0.0003<sub>6</sub> percent, or  $\pm 17$  percent of the amount present.

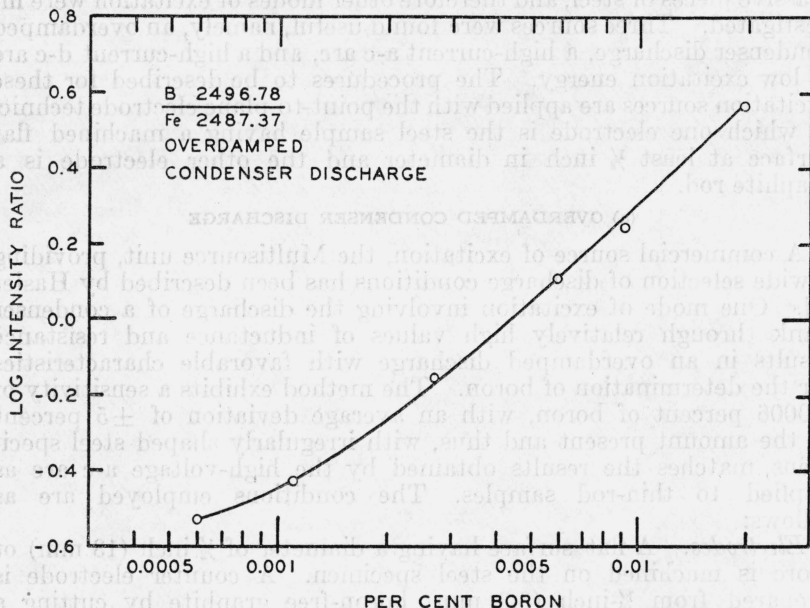


FIGURE 4.—Analytical curve obtained with  $\frac{1}{2}$ -inch-diameter National Bureau of Standards spectrographic boron steel Standard Samples and the overdamped condenser discharge.

TABLE 2.—Precision of overdamped condenser discharge

[Sixteen runs of Standard Sample 826 on one plate]

Boron content	Deviation	Boron content	Deviation
<i>Percent</i>	<i>Percent</i>	<i>Percent</i>	<i>Percent</i>
0.00105	0.00005	.00103	.00007
.00116	.00006	.00125	.00015
.00117	.00007	.00106	.00004
.00099	.00011	.00108	.00002
.00115	.00005	.00113	.00003
.00109	.00001		
.00109	.00001	1.00110	1.00006
.00115	.00005		
.00098	.00012		
.00114	.00004		
.00108	.00002		
		Average deviation from the mean $\pm 5$ percent.	

<sup>1</sup> Average.



The operating conditions for the excitation source were selected to provide optimum sensitivity and reproducibility. The maximum value of capacity is necessary to provide sufficient intensity of the boron line. The effect of resistance in the circuit, as plotted in figure 5, shows that the intensity ratio of boron to iron reaches a maximum at 15 ohms but that the ratio of boron to background reaches a maximum at 20 ohms and remains nearly constant above that value. In view of the gain in the intensity of the boron line at higher resistance values,

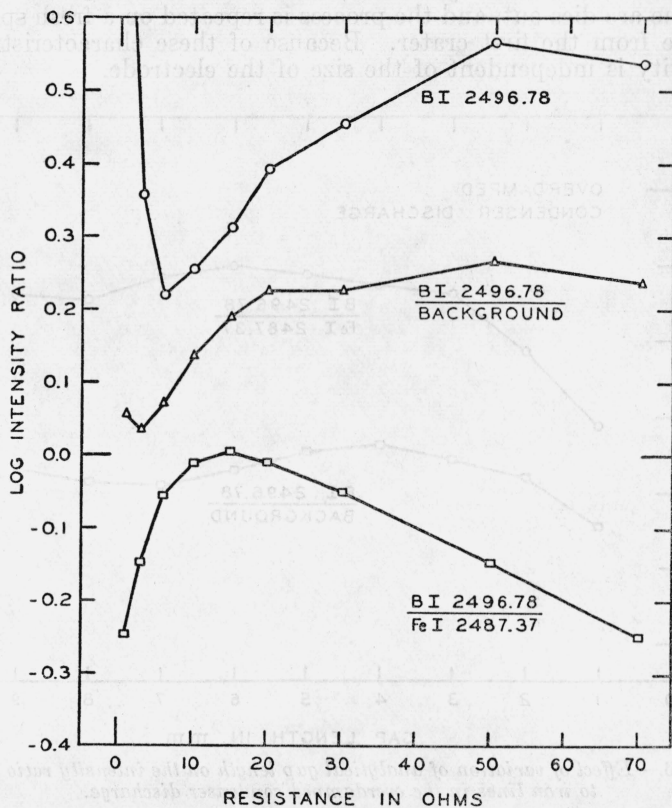


FIGURE 5.—Effect of variation of resistance on the intensity ratio of boron to iron lines in the overdamped condenser discharge.

a resistance of 50 ohms was adopted. Under these conditions the available variation in inductance has little effect on the discharge, but as larger inductance increased the intensity level slightly, the maximum value of  $480 \mu\text{h}$  was employed. The intensity level also depends on the distance between the electrodes, as shown in figure 6. At larger gap distances the intensity ratio of boron to iron is little affected by change in the gap; the optimum gap distance apparently lies between 4 and 6 mm. The electrode polarity in which the steel electrode is positive provides maximum sensitivity; reversal of polarity reduces the intensity ratio of boron to iron by a factor of 4.5.

The high sensitivity of this discharge apparently results from a combination of a violent attack on the surface of the steel and a subsequent overdamped condenser discharge with a duration sufficient to volatilize and excite the metal under arc-like conditions. As each pulse of current passes between the electrodes, a molten pool of steel about  $\frac{1}{2}$  mm in diameter is formed and is explosively volatilized, ejecting droplets and leaving a minute crater on the steel surface. The boron volatilizes from the surface of the molten metal before heat conduction into the body of the steel electrode is well established. Then the arc dies out, and the process is repeated on a fresh spot at a distance from the first crater. Because of these characteristics the sensitivity is independent of the size of the electrode.

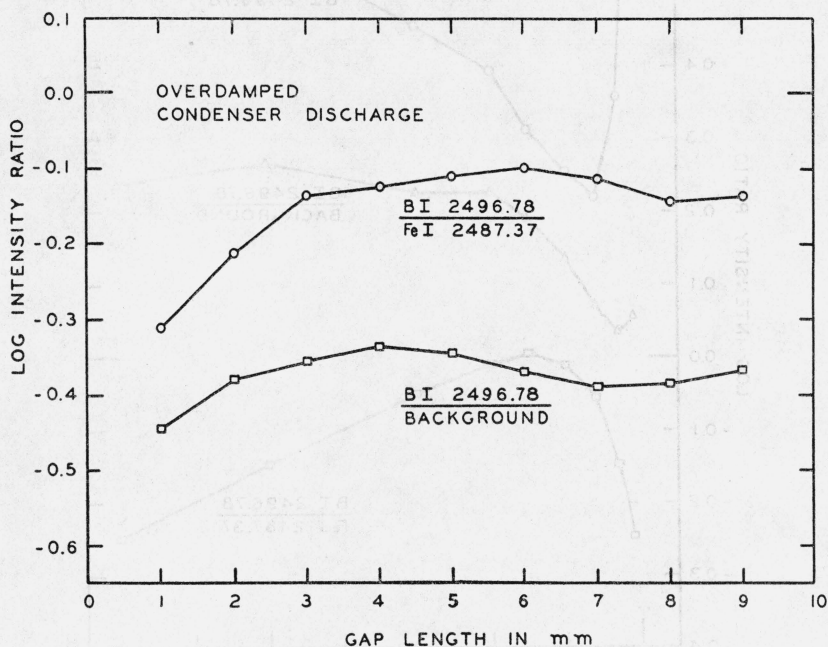


FIGURE 6.—Effect of variation of analytical gap length on the intensity ratio of boron to iron lines in the overdamped condenser discharge.

A series of determinations of boron made on steel specimens of different sizes and shapes but from the same heat of metal showed close agreement. If the sample provides a flat surface  $\frac{1}{2}$  inch in diameter or larger and is sufficiently massive to prevent excessive heating, an accurate determination can be made.

The possibility that the National Bureau of Standards boron-steel standards could be employed for the determination of boron in cast iron was investigated by use of this excitation method. A comparison of spectrographic and chemical results on three cast-iron samples showed the former to be low by 30 percent, indicating that cast-iron standards must be used.

If the Multisource unit is not available, a low-voltage a-c arc may be employed with comparable accuracy but with sensitivity limited to

0.001 percent of boron. This source offers the advantage, over the usual high-voltage a-c arc, in that larger currents may be passed through the arc, and consequently the selective volatilization of boron from massive specimens may be increased with a gain in sensitivity. The 220-volt a-c arc is not self-sustaining, but an auxiliary circuit similar to that described by Brockman and Hochgesang [6] provides a weak spark for steady ignition of the arc. The arc is run at a current of 7.5 amperes from a 220-volt a-c line with an electrode gap of 3 mm and an exposure of 30 seconds.

#### (b) DIRECT-CURRENT ARC OF LOW EXCITATION ENERGY

In the course of investigating the effect of boron in armor plate, determinations as low as 0.0001 percent of boron were required. The resolving power of the large Littrow quartz spectrograph was not sufficient to permit the use of the strongest boron line 2497.73 Å, and the second strongest line 2496.78 Å did not allow detection below 0.0006 percent of boron with the excitation procedures just described. As was stated in the discussion of the general aspects of the problem, an excitation means was found that permitted the use of B<sub>I</sub>2497.73 Å by minimizing the interference of the iron spark line Fe<sub>II</sub>2497.82 Å.

The energy needed to excite the boron line is 4.9 electron volts as compared to 16.1 volts for the spark line. If the energy of the electrons in the arc is reduced by introducing an element of low ionization potential, the intensity of the iron spark line will fall with respect to the boron arc line. This idea was tested in a d-c arc with the point-to-plane electrode combination in which the graphite counter electrode was drilled and then filled with a salt of an alkali metal. Of the elements tried, only sodium and potassium were effective. Sodium changed the ratio of B<sub>I</sub>2497.73 Å to Fe<sub>II</sub>2497.82 Å from unity at 0.003 percent to unity at 0.0003 percent of boron. Potassium was even more effective but reduced the general intensity level excessively. Not all sodium salts were satisfactory; anhydrous sodium sulfite provided superior sensitivity and precision of results.

For maximum accuracy with this method, the temperature of the steel electrode should be closely controlled. Large specimens of identical size and shape are least subject to variations. The physical form and concentration range of the National Bureau of Standards spectrographic boron steel Standard Samples are not suitable for application to this method.

The following conditions are employed:

*Electrodes.*—The anode is a steel block cut 1½ inches (38 mm) square by ½ inch (13 mm) thick and machined flat on both sides. The cathode consists of a ⅛-inch (3.2 mm) pure-graphite rod 15 mm long having a cup 2 by 2 mm filled with dry, powdered sodium sulfite.

*Mounting of electrodes.*—The steel block anode is mounted horizontally as the upper electrode. The cathode is inserted into a hole 5 mm deep in a ¼-inch graphite rod that serves as a mounting of low heat conductivity. The electrode gap is set at 6 mm, and light from a section of the gap 4 mm long at the steel electrode is focused on the collimator of the spectrograph.

*Excitation.*—A d-c arc is employed with current of 10 amperes drawn from a 220-volt line.

*Spectrography.*—The spectrum is photographed on a large Littrow quartz spectrograph with a slit width of 20 microns and using an Eastman No. 33 plate.

*Exposure.*—The exposure period is 90 seconds.

*Photometry.*—The plate is calibrated as described for the a-c arc method, and the intensity ratio of B I 2497.73 Å to Fe I 2496.99 Å is determined.

The analytical curve shown in figure 7 was constructed by this method from the mean values of three determinations on a set of five reference samples in which boron had been determined chemically. The agreement of repeated runs, as indicated by the vertical lines through the plotted points, is poor in comparison with the

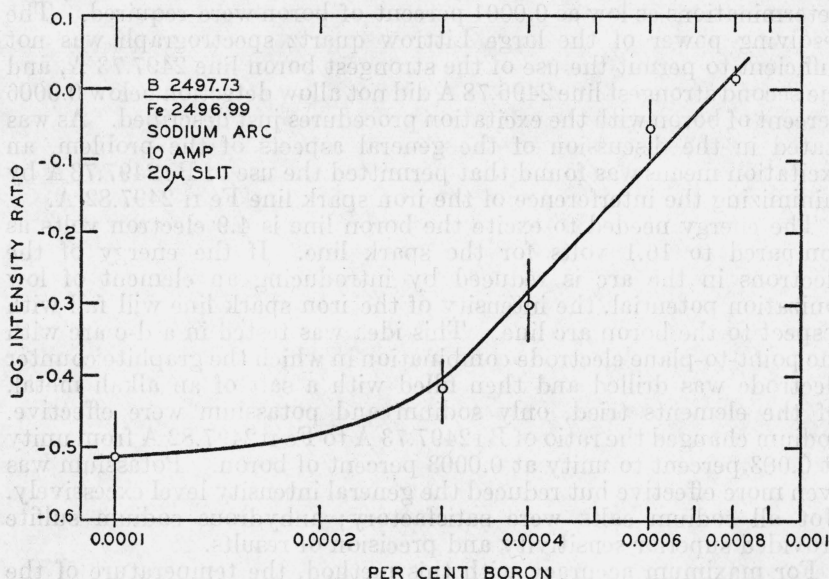


FIGURE 7.—Analytical curve obtained for the determination of boron in steel with the sodium arc.

methods just described. However, the mean of two determinations for an analytical sample provided results good to 0.0001 percent in most cases. The method is mainly useful for its high sensitivity and is not recommended for determinations above 0.001 percent of boron.

### III. SPECTROGRAPHIC BORON-STEEL STANDARDS

The application of spectrographic methods involving direct excitation of the metallic sample requires standard samples, in proper forms, with uniform distribution of the element to be determined, and with accurate evaluation of its concentration. As a part of the investigation of methods for determining boron in steel, a series of boron steels was made available as National Bureau of Standards Standard Samples. The steels were obtained through the generous cooperation of the Bethlehem Steel Co., Bethlehem, Pa. For each standard, the steel of selected composition was hot-rolled to  $\frac{1}{16}$ -and

$\frac{1}{32}$ -inch rods, the rods were annealed, straightened, and finally finished to  $\frac{1}{8}$  and  $\frac{1}{16}$  inches, respectively, by centerless grinding. The finished rods, with the exception of Standard Sample 828 and part of 826, are in the annealed state.

The rods were received in 10-foot lengths at this Bureau, where a test sample was taken from both ends of each length. The samples were examined spectrographically, and the boron was found to be uniform within the precision of the spectrographic methods. The uniformity of compositions was gratifying, particularly as the boron was added in the furnace only in two cases, the remaining additions being made in the ladle or ingot molds. The boron contents of the standards were determined in the Chemistry Division of this Bureau by K. D. Fleischer. Boron in the 12 standards was determined by a distillation-tumeric colorimetric method and also by a distillation-titration method in the case of standards 427, 429, 430, 827, 829, and 830. The certified Standard Samples<sup>4</sup> and the modes of preparation are described in table 3.

TABLE 3.—Spectrographic boron-steel Standard Samples

Standard Sample number <sup>1</sup>	Type of steel	Addition agent	Added in—	Total boron
425, 825	Mn-Ni-Cr B. O. H. (NE9450).....	Ferroboron (11% boron).....	Ingot.....	<i>Percent</i> 0.0006 .0011 .0027 .0059 .0091 .019
426, 826	Cr-Mo B. O. H. (SAE 4150).....	Grainal No. 79 (0.5% boron).....	Ladle.....	
427, 827	do.....	Ferroboron (12% boron).....	do.....	
428, 828	Mn-Cr B. O. H.....	Ferroboron (11% boron).....	do.....	
429, 829	Ni-Cr-B (basic electric).....	do.....	Furnace.....	
430, 830	do.....	do.....	do.....	

<sup>1</sup> Standard Samples in the 400 series are rods  $\frac{7}{32}$  inch in diameter, 4 inches long, and those in the 800 series are rods  $\frac{1}{4}$  inch in diameter, 2 inches long.

The  $\frac{1}{32}$ -inch Standard Samples are intended for use as self-electrodes in the analysis of rods prepared in similar shape, for example, by the method described in section II-2. The 4-inch length may be cut at the center, yielding two rods, each 2 inches long. The larger standard rod provides a cross-sectional surface  $\frac{1}{2}$  inch in diameter for application in the analysis of steel specimens of similar or larger size. If the Standard Samples are to be used with a Petry stand or a similar flat-top stand, it is convenient to mount them in a steel-disk adaptor fitted with six holes and setscrews for clamping the  $\frac{1}{2}$ -inch rods. Cutting the rods lengthwise to bridge the opening in the stand is not recommended. The  $\frac{1}{2}$ -inch standard rods also serve for the preparation of specimens of unusual shapes, such as square rods, and as a source of millings or turnings if these are desired.

The validity of applying the  $\frac{1}{2}$ -inch standards to the analysis of more massive specimens has been established by comparing the results on specimens having a variety of sizes but from the same heat of steel. If the specimen provides a flat surface  $\frac{1}{2}$  inch in diameter or larger and has sufficient mass to avoid excessive heating, the Multisource unit and low-voltage a-c arc methods will apply accurately. Application of the usual d-c arc or of the sodium-arc procedure require samples and standards of identical size and shape.

<sup>4</sup> Full information on current National Bureau of Standards Standard Samples, including the procedure to be followed in their purchase, is given in the Supplement to NBS Circular C398, which can be obtained free upon application to the National Bureau of Standards, Washington 25, D. C.

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WASHINGTON, January 7, 1946.

Standard No.	Type of steel	Element	Concentration
100	SAE 1045	Boron	0.0005
101	SAE 1045	Boron	0.0010
102	SAE 1045	Boron	0.0015
103	SAE 1045	Boron	0.0020
104	SAE 1045	Boron	0.0025
105	SAE 1045	Boron	0.0030
106	SAE 1045	Boron	0.0035
107	SAE 1045	Boron	0.0040
108	SAE 1045	Boron	0.0045
109	SAE 1045	Boron	0.0050
110	SAE 1045	Boron	0.0055
111	SAE 1045	Boron	0.0060
112	SAE 1045	Boron	0.0065
113	SAE 1045	Boron	0.0070
114	SAE 1045	Boron	0.0075
115	SAE 1045	Boron	0.0080
116	SAE 1045	Boron	0.0085
117	SAE 1045	Boron	0.0090
118	SAE 1045	Boron	0.0095
119	SAE 1045	Boron	0.0100
120	SAE 1045	Boron	0.0105
121	SAE 1045	Boron	0.0110
122	SAE 1045	Boron	0.0115
123	SAE 1045	Boron	0.0120
124	SAE 1045	Boron	0.0125
125	SAE 1045	Boron	0.0130
126	SAE 1045	Boron	0.0135
127	SAE 1045	Boron	0.0140
128	SAE 1045	Boron	0.0145
129	SAE 1045	Boron	0.0150
130	SAE 1045	Boron	0.0155
131	SAE 1045	Boron	0.0160
132	SAE 1045	Boron	0.0165
133	SAE 1045	Boron	0.0170
134	SAE 1045	Boron	0.0175
135	SAE 1045	Boron	0.0180
136	SAE 1045	Boron	0.0185
137	SAE 1045	Boron	0.0190
138	SAE 1045	Boron	0.0195
139	SAE 1045	Boron	0.0200
140	SAE 1045	Boron	0.0205
141	SAE 1045	Boron	0.0210
142	SAE 1045	Boron	0.0215
143	SAE 1045	Boron	0.0220
144	SAE 1045	Boron	0.0225
145	SAE 1045	Boron	0.0230
146	SAE 1045	Boron	0.0235
147	SAE 1045	Boron	0.0240
148	SAE 1045	Boron	0.0245
149	SAE 1045	Boron	0.0250
150	SAE 1045	Boron	0.0255
151	SAE 1045	Boron	0.0260
152	SAE 1045	Boron	0.0265
153	SAE 1045	Boron	0.0270
154	SAE 1045	Boron	0.0275
155	SAE 1045	Boron	0.0280
156	SAE 1045	Boron	0.0285
157	SAE 1045	Boron	0.0290
158	SAE 1045	Boron	0.0295
159	SAE 1045	Boron	0.0300
160	SAE 1045	Boron	0.0305
161	SAE 1045	Boron	0.0310
162	SAE 1045	Boron	0.0315
163	SAE 1045	Boron	0.0320
164	SAE 1045	Boron	0.0325
165	SAE 1045	Boron	0.0330
166	SAE 1045	Boron	0.0335
167	SAE 1045	Boron	0.0340
168	SAE 1045	Boron	0.0345
169	SAE 1045	Boron	0.0350
170	SAE 1045	Boron	0.0355
171	SAE 1045	Boron	0.0360
172	SAE 1045	Boron	0.0365
173	SAE 1045	Boron	0.0370
174	SAE 1045	Boron	0.0375
175	SAE 1045	Boron	0.0380
176	SAE 1045	Boron	0.0385
177	SAE 1045	Boron	0.0390
178	SAE 1045	Boron	0.0395
179	SAE 1045	Boron	0.0400
180	SAE 1045	Boron	0.0405
181	SAE 1045	Boron	0.0410
182	SAE 1045	Boron	0.0415
183	SAE 1045	Boron	0.0420
184	SAE 1045	Boron	0.0425
185	SAE 1045	Boron	0.0430
186	SAE 1045	Boron	0.0435
187	SAE 1045	Boron	0.0440
188	SAE 1045	Boron	0.0445
189	SAE 1045	Boron	0.0450
190	SAE 1045	Boron	0.0455
191	SAE 1045	Boron	0.0460
192	SAE 1045	Boron	0.0465
193	SAE 1045	Boron	0.0470
194	SAE 1045	Boron	0.0475
195	SAE 1045	Boron	0.0480
196	SAE 1045	Boron	0.0485
197	SAE 1045	Boron	0.0490
198	SAE 1045	Boron	0.0495
199	SAE 1045	Boron	0.0500
200	SAE 1045	Boron	0.0505

The 200 standard samples are intended for use as self-electrodes in the analysis of rods prepared in similar shape. For example, by the method described in section II-2, 200 1/2-inch long rods may be cut from the center of each rod, each 2 inches long. The 200 standard rods provide a cross-sectional surface 1/2 inch in diameter for analysis in the analysis of steel specimens of similar size. The standard samples are to be used with a 1/2-inch rod of a similar diameter. It is convenient to mount them in a 1/2-inch diameter lathe with six holes and set screws for clamping the 1/2-inch rods. The 1/2-inch standard rods are also for the preparation of specimens of unusual shapes and sizes, and as a source of millings or turnings if these are desired. The validity of applying the 1/2-inch standards to the analysis of more massive specimens has been established by comparing the results on specimens having a variety of sizes and forms. A standard of steel. If the specimen provides a flat surface 1/2 inch in diameter and has sufficient mass to avoid excessive heating, the 1/2-inch standard rods and low-voltage arc method will apply equally. Application of the standard rods to the analysis of steel specimens requires samples and standards of identical size and shape.

1. All information on current National Bureau of Standards research, including the procedures to be followed in their application, is given in the supplement to NBS Circular 500, which may be obtained free upon application to the National Bureau of Standards, Washington, D. C.