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Time-resolved spectroscopy in spectrochemical analysis.

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Abstract

Light radiated from transient spark discharges varies as the conditions vary in time in the source. The observation of the variation in time of the radiation emitted give useful information concerning the excitation levels of the variation in intensity of the intensity ratios of the pressure broadening of the wavelength shift of the Stark broadening of the selfabsorption with time of the spectrum lines. These informations can be obtained from time resolved spark spectra taken with the intermediate of an electronically controlled high precision spark source combined with a rotating mirror optical system. From time resolved spark spectra spectrum lines suitable for spectrochemical analysis can be selected on a theoretical basis. In time-resolved spectra the continuous background originating at the beginning of the discharge is separated from the rest of the spectrum. The background free spectrum, in addition to facilitating the construction of straight working curves without background correction is suitable for estimation of minor constituents in metals and alloys. The background separated from the rest of the spectrum can be used as internal standard in spectrochemical analysis. As example by using background as internal standard working curves for the estimation of impurities in aluminium are given.

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Introduction

With the aid of high precision high voltage spark [illegible] time resolved spark spectra can be produced in photographic way. The time resolved spark spectra enables the observation and [illegible] of phenomena impossible in common practice. Moreover a possibility presents itself with the application of time resolved spark [illegible] to improve the spectrochemical analysis resp. to employ new methods. The purpose of present paper is to review the work related to time resolved spark spectra produced with the method worked out in the laboratory of the author.

In spectroscopic practice, where the aim is above all the production of line spectra the instantenous value of the energy used for light excitation is relatively small. Thus the production of line spark spectra is effectuated by means of the discharge of condensers storing relatively low electrical energy through spark gap. To obtain spectra with relatively low energy spark discharges hundreds and thousands of sparks are needed to produce adequate density on the photographic plate. Since for the investigations of time resolved spark spectra a time resolution of an order of magnitude of microseconds or even a smaller one is needed, this signifies that hundreds or thousands of time resolved spectra are to be photographed each on another with such a precision that the resolution desired shall be kept. Therefore to achieve the desired precision however an electronically controlled spark source operating with high precision is required. Taking into account the photographic method the time resolution of spectra is carried out with the aid of a rotating mirror.

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The experimental arrangement.

The experimental arrangement for the production of time resolved spark spectra is shown in figure 1. The lower part of the figure shows the electronically controlled high precision spark source, whilst the upper part illustrates the optical arrangement.

Since the spark source in question has been previously described in detail /1,2,3,4,5/ and because the principle of operation of the optical arrangement has been described too /6/ for the sake of completeness the problem will be dealt with only briefly.

In the spark source part of figure 1, C ist the condenser supplying the excitation energy, which is charged up from the high voltage transformer T through the rectifier diode V1. R1 is a current limiter resistance. The charging voltage of condenser C is distributed evenly by resistances R2 and R3 over the simmetrically adjusted twin controlling spark gap G.

A is the analytical spark gap, which is bridged over by resistance R5, V2 is a thyratron tube in the plate circuit of which the resistance R4 is inserted. The thyratron tube is blocked by meand of the negative bias fed into its grid.

The twin controlling spark gap G is adjusted so that in a charged state of condenser C just no break down shall occur. If in this state a positive voltage signal to the grid of tube V2 is supplied the tube will fire and the charging voltage of condenser C will appear - according to the figure - on the upper half of the twin spark gap G consequently the latter will break down and condenser C will begin to discharge through R5 - upper half of G - R4 - V2. Since practically the discharge of the upper half of G signifies its shortcircuiting therefore the whole char-

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ging voltage of condenser C will appear on the terminals of resistance R4 resp. on the lower half of the twin controlling spark gap G, consequently it will break down too. After this has taken place the path of discharge of condenser C will be R5 - G - G. After that moment however the charging voltage of C will appear on the terminals of R5 viz. on the analytical spark gap A, so A will break down too and after proceeding on the way A - G - G, C discharges and supplies the excitation energy. The discharge circuit is plotted on the figure in thick lines.

The operation of the optical system is the following. Light coming from spark gap A reaches rotating mirror M driven by a synchronous motor D. Before the spark gap A the slit S2 parallel to the plane of the figure is placed. The symmetry axis of the spark gap A is normal to the plane of the figure. This way it can be achieved that light arising only from a certain part of the spark gap will reach the rotating mirror M. Spark gap A resp. the part of the spark gap screened out by slit S2 is imaged by rotating mirror M onto the slit S1 of the spectrograph and will draw it in time along of the same. The time resolved image of the spark after the resolution according the wavelength taking place at the prism D appears on the photographic plate P.

The connection between the spark source and the optical system is established by photocell I. This occurs in the following way: The light coming from light source H falls onto rotating mirror M with the aid of which it is projected onto photocell I. The photocurrent supplied by photocell I and amplified by amplifier K is used to control the grid of thyratron tube V2. Phase relations are adjusted in such a manner that condenser C is loaded up during

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the half cycle of the a.c. mains and discharges in the follo[illegible] half cycle. Further details are to be find in /6/.

The essential feature of the arrangement of figure 1, consists in that, that related to the signal given onto the gr[illegible] of tube V2 of the spark source the break down of the analytical gap takes place with a time scatter /jitter/ of 0.1 microsecond. This means that the time resolved images of the individual spark appearing on the slit of spectrograph S1 will be superponed o[illegible] each other with a similar accuracy.

Observations in time resolved spark spectra.

Time resolved spark spectra produced with the arrangement according to figure 1 are illustrated in figures 2,3,4,5 and 6. In the upper part of each figure in the middle of the threefold set of spectra the spectrum of the iron is to be seen. The upper and the lower spectrum of the threefold set of spectra shows the spectrum of the element in the middle of the spark gap and near to the cathode in case of a spark gap of 8 mm photographed through a slit of a width of 0.45 mm placed before the spark gap. In the lower part of each figure the time resolved spark spectrum of the element is to be seen. The experimental conditions at the taking of the time resolved spark spectra are summarized in table I.

It can be yet stated at the first sight that the time resolved spark spectra are very complicated, therefore at this place we will treat only their main features. The problem will be dealt with in detail in other papers.

The first what is striking in figures 2,3,4,5 and 6 is that a continuum appears at the beginning of each time resolved

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spark spectrum. The continuum is the stronger and its durati[illegible] the longer the higher the excitation level. As it can be stated above all from figure 4 and 5 in some cases a continuum will appear also in later phases of the discharge, and it is to be find in the neighbourhood of the peak value of the current flowing in the discharge circuit.

A further striking phenomenon consists in that that the intensity of some spectrum lines shows periodicity in near proxi[illegible] to the cathode /figures 2 and 5/. These properties belong first of all to the spark lines of high excitation levels, which cease to radiate as soon as the excitation energy diminishes. The variations of the current flowing in the discharge circuit are follo[illegible] by the periodic intensity variations of these lines and they [illegible] at the zero value of the current or even earlier. All these lines arise at the beginning of the discharge and after a longer or shorter time of radiation they cease to exist. This periodic in[illegible]tensity variation of the spectrum lines can be well employed as a measure of time at time resolved spark spectra. Hence the peri[illegible] of intensity variations in figures 2,4 and 5 is 2.14 microseco[illegible] which is equal to the period of the frequency of the discharge circuit of the spark source.

One part of the lines does not exhibit any intensity variation in the neighbourhood of the cathode, or if it does, [illegible] to a much smaller extent as those belonging to the previous gro[illegible] The lines CdI 2239, CdI 2267, CdI 2306, CdI 2329 of figure 5 and the line CdI 3261 of figure 4 etc. serve as examples. These lines are of lower excitation levels, usually arc lines the excitation energy of which can be covered from the plasma of the spark even

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in the case when the excitation current passes through zero value. Therefore one part of these lines will radiate for a time considerably long enough even after the spark discharge has ceased to exist. Similar lines will appear to the largest extent in the middle of the spark gap, because until the vapours jets from the cathode will arrive as far as here one part of their energy will be lost.

It is without any more detailed investigations a stimulating phenomenon, that in time resolved spark spectra the width of some lines due to various excitation conditions prevailing in the spark gap will vary in its width strongly with the time of discharge. As examples serve the lines ZnII 2502 and ZnII 2557 in figure 2 and CdI 3403, CdI 3466, CdI 3467, CdI 3610, CdI 3612, CdI 3614 in figure 4. The appearence of the Stark effect due to the charged particles present in the spark gap is responsible for these broadenings of large extent as well as for the variations in the broadenings.

The periodicity of the intensity of spectrum lines originating from the neighbourhood of the cathode are due to the vapour jets breaking out from the cathode. These vapour jets will appear during each cycle of the current circulating in the discharge circuit. Until the metal vapours will reach the middle of the spark gap they will be spread evenly in the space partly owing to the diffusion and partly in consequence of eddying. This even distribution of the vapours is promoted to a large extent by the fact that at the middle of the spark gap the vapours arrive from both electrodes. From the anode the vapours will reach the middle of the spark gap with a phase delay related to the cathode of 180 electric degrees. Spectra taken at the middle of the spark gap are

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illustrated in figures 5 and 6. Figure 7 illustrates the variation in time of the profile of line ZnII 2502 in a time resolved spark spectrum taken at the middle of the spark gap.

More detailed observations show that a certain part of the lines appearing at the beginning of the spark discharge will strongly broaden at the initial phase of the discharge and their wavelength will be shifted towards the red. This is due to the high pressure prevailing at the beginning of the spark discharge. This effect ceases to exist on the basis of previous observations within 2-3 microseconds and the line returns into the position of its corresponding wavelengths.

An intensive examination of the lines broadened owing to the Stark effect shows that some of those lines are shifted in wavelengths. This shifting however has a duration much longer than the shift arising owing to the pressure and described in the previous paragraph. This shift of the wavelength of a longer duration is similarly due to the Stark effect. Figure 8 illustrates the variation of the profile of the asimmetrically broadened and wavelength shifted line of ZnI 3072 as function of time in a time resolved spark spectrum.

Further observations demonstrate that with the aid of time resolved spark spectra the dependence of the self absorption of spectrum lines from the excitation conditions can be studied. In figures 5 and 6 spectrum lines with self absorption are illustrated. In figure 5 the course of self absorption of the resonance line CdI 2288 is well observable. In the neighbourhood of the cathode this line has at the beginning no self-absorption. With the progression of time resp. with the diminishing of the

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excitation energy the self-absorption grows. The resonance line MgI 2852 in figure 6 shows a similar course. The latter spectrum illustrates the radiation appearing at the middle of the spark. Figure 9 illustrates the variation in time of the resonance line CdI 2288 in the middle of the spark gap.

In figures 5 and 6 further selfabsorbed lines can be observed by which however the selfabsorbed width is smaller than in case of the previous resonance lines. These are the resonance lines CdII 2265, MgII 2795 and MgII 2802 belonging to the second spectra.

Time resolved spectrochemical analysis.

Selection of spectrum lines.

A time resolved spark spectrum may be a serious aid for the spectrum analyser concerning the selection of his analytical lines. Present practice demonstrates that in spectrochemical analysis ~~preferring~~ above all arc lines are employed. That has been achieved in an empirical way. A time resolved spectrum shows that arc lines are much less sensitive to the excitation conditions than spark lines are. The reason for is the following. The average value of the excitation energy ~~will~~ during an individual discharge will diminish in the course of time from a highest initial value until a zero value. That is to say that the energy conditions of the spark will run through each value of the excitation function of a spectrum line assuming that the highest energy of the spark is high enough for this /8/. Taking in consideration excitation energies occurring in spectrochemical practice, the energy of the spark is usually higher as the highest energy level of the excitation

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conditions of arc lines applied for analyses. Consequently starting from zero intensity a similar spectrum line will reach in the course of time a highest intensity value and then with the progression of time diminishes again proceeding towards zero intensity. Should the excitation energy now vary to a slighter extent that will signify that the intensity conditions of the line in question are shifted in principle only in time, but the value of the intensity integrated in time does not vary. Whilst in the case of a line where the higher energy levels of its excitation function are not included in the excitation range employed and the variations of the intensity between the two zero values of the spectrum line described before are only partly included into the energy range subject of investigation, than the variation of the excitation energy will change the integrated intensity to a large extent.

The determination of spark and arc lines with the aid of time resolved spark spectra is according to the aforesaid a very simple task.

With the aid of time spark spectra lines exhibiting self-absorption can be easily discovered. Many lines will not be selfabsorbed during the beginning of spark discharge, but only towards the end of it. Examples are in figure 5 the line CdI 2288 and in figure 6 the line MgI 2582. These lines often does not show any self/absorption in the spectra taken in the usual way. As a matter of course the analyst will employ a selfabsorbing line for analytical purposes only in very exceptional cases.

Moreover in the qualification of lines it is a well known fact that strongly diffuse, asymmetric or lines the wavelength of which

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vary in time are similarly slightly adequate for being employed to spectrochemical analyses. But however just time resolved spectrochemical analysis renders the application of otherwise not very suitable lines possible in such a manner that only the suitable part of the time resolved line will be utilized. A characteristic example is the compulsory utilization of the first part of self-absorbing lines only at the beginning of the discharge.

The time resolved spark spectrum may be a valuable aid in the determination of the correct wavelengths of spectrum lines. Strongly diffuse lines become sharp with the diminishing of the excitation energy thus an accurate determination of the wavelenghts of lines is rendered possible. Particularly suitable examples are to be seen in figure 6 where the lines MgII 2790, MgII 2798, MgII 2928 and MgII 2936 have similar behaviour.

The separation of the background, the air lines and of other spark lines from the spark spectrum.

As far as we succed in separating the background arising at the beginning of the discharge the air lines and further lines of high excitation levels from the spectrum thus practically only the arc spectrum will remain. This spectrum without background and from the point of view of spectrochemical analysis without superfluous lines is exceedingly suitable for carrying out spectrochemical analyses. The photoelectric solution of this problem is due to Steinhaus, Crosswhite and Dieke . /12,13,14/. For photographic quantitative analyses however such spectra are to be produced in which the intensity of the spectrum lines is equal in their total length. Spectra of that type can be produced with the optical arrangement illustrated in figure 10. This arrangement

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differs from that of figure 1 as far as between the rotating mirror M and the slit of spectrograph S1 the slit S2 and the imaging lens L are inserted. The aperture of the slit S2 is in parallel with the plane of the figure.

For sake of separating the undesired part of the spectrum the light source A, resp. its part screened by slit S3 is imaged in time resolved onto the screen S2. With the aid of the screen S2 the desired part is screened out from the time resolved spark image appearing on the screen. After that the spark image appearing on the screen S2 is imaged onto the collimator lens of the spectrograph with the aid of the lens L. In such a manner a spectrum with a degree of purity to be seen in the lower spectrum of figure 12 can be produced.

To demonstrate what can be achieved with the aid of the above described spectrochemical analytical method in table II some reproducibility results for the determination of impurities in aluminium are presented. Because these results were obtained at an initial state of the method it is to be seen what might be expected from the method in question. Though for time resolved quantitative spectrum analyses in general the optical apparatus illustrated in figure 10 is employed, in carrying out the above-mentioned reproducibility test an optical arrangement illustrated in figure 1 has been used. The working conditions were the followings.

Flat ended aluminium electrodes of 5,4 mm in diameter and pointed carbon counter electrodes of 5,5 mm in diameter were used. Aluminium was the anode. The spark gap was 3 mm, and light arising from the central portion of 1 mm has been utilized for

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carrying out analyses. The width of the slit of the spectrograph was 30 microns. Presparking time 120 seconds. Further working conditions are the same as at the taking of the other spectra.

In spectra taken according to the above described method the intensity so of aluminium as well as of impurity lines will have a highest value in time. The ratio of these highest intensity values has been formed. A high speed microphotometer of Zeiss type has been employed. Slit width of the photometer was 80 microns, slit height 4 mms.

The background as internal standard.

By using the optical arrangement according to figure 11 and an electronically controlled high precision spark source the possibility to produce a spectrum from the initial and a spectrum from the later phase of a time resolved spark spectrum separated each from another but simultaneously is given.

In figure 11 A is the spark gap with the slit S1 before it. The spark gap is imaged in time resolved with the aid of rotating mirror M onto the plane of the sides of reflecting rhombuses R1 and R2 facing the rotating mirror. The phase position of rotating mirror is adjusted in such a manner that light arising at the initial stage of the spark discharge shall fall onto rhombus R1 that one arising in the later stage of it onto rhombus R2. This way light coming from the light source is divided in two beams. The image of the spark gap A formed onto the sides of the rhombuses R1 and R2 is imaged by lenses L1 and L2 onto the collimator lens of the spectrograph. The task of rhombuses R3 and R4 is to bring near the two light beams travelling far each from another. S2 is the slit of the spectrograph.

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In figure 12 a spectrum taken with the aid of optical arrangement illustrated in figure 11 is shown. In the upper spectrum the background, the spark lines and the air lines are included, and in the lower one above all the arc lines are contained.

The basis of the modern spectrochemical analysis forms the internal standard line method. The method in question is comfortable and agreable if in the spectrum to be analysed in proximity to the lines of the alloying element or of the impurities an adequate comparison line is at disposal. If the standard line is as far in wavelength, that the sensitivity of the plate varies at this distance so the forming of the ratio of intensities will encounter difficulties. If an adequate internal standard line is not at our disposal such an element has to be added to the material to be analysed which has the suitable comparison lines. This is similarly a cumbersome procedure.

Taking into consideration the spectrum of figure 12 it can be regarded as quite an evident idea to employ as internal standard the background of the spark spectrum. If other difficulties would not be encountered a very agreable method could be employed, since the background of the spark spectra is everywhere present in near proximity of the lines of the alloying or impurity elements. This way in addition of increasing the sensitivity of the spectrochemical analytical method by the separation of the background, the background can be utilized as a comparison basis /standard basis/. This is all the more conceivable for in spectroscopic practice the utilization of the background as internal standard has yet been met with elsewhere /10,1[illegible]16/.

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The applicability of the method to spectrochemical analyses can be decided on the basis of reproducibility measurements. Measurements of this type has been carried out and in Table III those results are represented, which are obtained at the occasion of the comparison of the impurity lines of aluminium with the background. These data though being fairly preliminary ones, the results of the up to date experiments show that the method can be regarded as a well enough founded one for practical application. In figure 13 a detail of the plate, where the data of Table III were obtained is illustrated. The plate gives the impression of a good reproducibility of the background. In figure 14 and 15 working curves obtained in this way are illustrated for the determination of the impurities Cu and Si in aluminium.

Taking the spectra of figure 13 and so at the determination of the data included in Table III the experimental conditions were the following. In figure 11 from the time resolved spark spectra a first phase corresponding to 20 microseconds was falling onto rhombus R1. The focal length of mirror M is 140 mm, the distance A-M 224 mm, the distance M-R1 330 mm. From the viewpoint of the light yield the adjustement in question cannot be regarded as the most convenient. A Hilger Spekker photometer has been used to divide the light of the spark into two beams. The built in slits of the original photometer has been removed and the rhombuses R1 and R2 were approximated as much one to another, that their edges came nearly into contact. The width of the slit of the spectrograph is 60 microns. At the photometering the slit width was 100 microns, slit heigth 10 mm. Taking the working curves of the figure 14 and 15 exposure was 30 seconds. Further conditions are similar to the previous ones.

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Concerning the photometering in the spectrum we note: Hence the width of the slit before the analytical gap is 1 mm, the resolution in time of the time resolved spark spectrum for the sake of the separation of the background diminishes. Therefore radiation corresponding to a duration of about 20 micro - seconds counted from the beginning of the spark discharge has to be directed onto the rhombus R1 of figure 11.Owing to the small time resolution in question, in the part of the spectrum including the background analysis lines will appear also. Therefore the comparison with the background takes place slightly displaced from the analytical line in the direction of the dispersion.

Some practical remarks.

In spectrochemical analyses it is a well known fact, that as a rule the more sparklike the discharge the less influence is exercised by the metallurgical history of the sample and by the alloying variations onto the analytical results /11/. A sparklike discharge however can involve a background so significant that it can signify a serious obstacle in carrying out the analysis. This is very characteristic for aluminium and its alloys which has been experienced so by the author as well as by others /9/. If for the reduction of the background selfinduction is applied in the discharge circuit this will consequently result in a more arclike discharge and irregular evaporation and cratering, which signifies a drawback concerning the reproducibility /9/. The abovedescribed method in principle is a perfect solution of the problem.

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T a b l e I.

Experimental conditions at taking the time resolved spectra of figures 2,3,4,5 and 6.

Capacity of condenser C /figure 1/:	10,000 pF
Charging voltage of condenser C :	20,000 V
Sparking frequency :	50/sec.
Self induction of the discharge circuit:	residual selfinductions of the leads
Frequency of the discharge circuit:	about 0.5 Megaherz
Duration of one discharge :	about 45 microseconds
Spark gap :	8 mm
Form of the electrodes :	rooflike, with edges parallel to the optical axis
Screening slit width of the spark gap /figure 1,S2/ :	0,45 mm
Spektrograph :	ISZP 22, medium type
Width of the slit of the spectrograph :	5 microns
Photographic emulsion :	Agfa blau rapid
Exposure time :	2 minutes, at figure 6, 1,5 minutes
Revolution per minute of the rotating mirror M :	3000
Focal length of rotating mirror M :	18 cm
Slit S2 is imaged by rotating mirror M /figure 1/ in proportion	1 : 3 onto slit S1.
Number of sparks needed for one exposure :	6000 and 4500 respectively
Developer :	high speed
Developing time :	90 seconds
Developing temperature :	20° C

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Reproducibility results for the determination of [illegible] in aluminium with the aid of time resolved spectr[illegible]. The numerical values given are the average deviation of 10 measurements.

Element	Average deviation %	Lines used	Percentage of the impurity in the sample
Cu	1,2	Cu 3247/Al 2659	0,01
Mn	2,1	Mn 2949/Al 2669	0,115
Si	2,2	Si 2881/Al 2669	0,57
Mg	2,7	Mg 2852/Al 3064	0,11
Fe	1,3	Fe 2395/Al 2669	0,11
Mean value	1,9		

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Table III.

Reproducibility results for the determination of impurities in aluminium with the aid of time resolved spark spectra. As internal standard the background separated in time of spark spectra was used. The numerical values given are the average deviations of 10 measurements.

Element	Average deviation %	Line used	Percentage of the impurity the sample
Cu	2,4	Cu 3247/Bg	0,01
Mn	1,6	Mn 2949/Bg	0,115
Si	1,5	Si 2881/Bg	0,57
Mg	1,8	Mg 2852/Bg	0,11
Mean value	1,8		

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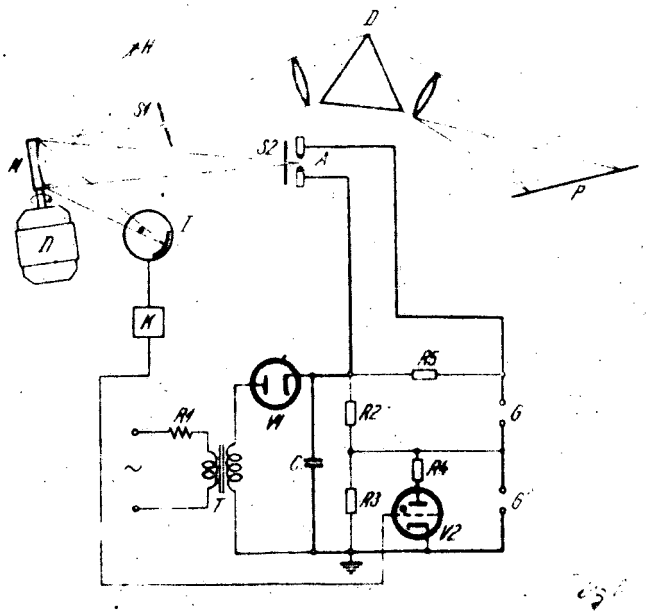

Figure 1.

[illegible] arrangement for the production [illegible] resolved [illegible] lower part of the figure illustrate[illegible] [illegible]lly controlled high precision spark source [illegible] [illegible]ical system.

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Figure Table.

Figure 1./ Experimental arrangement for the production of time resolved spark spectra. The lower part of the figure illustrates the electronically controlled high precision spark source, the upper one the optical system.

Figure 2./ Part of the time resolved spark spectrum of Zn taken in near proximity of the cathode of a spark gap of 8 mm through a slit of 0.45 mm wide /lower spectrum./ In the upper part of the figure in the middle of the threefold set of unresolved spectra the spectrum of the iron is to be seen. Above the iron spectrum is the spectrum of Zn photographed through a slit of 0.45 mm wide in the middle of the spark gap, below the spectrum of the iron the same as above it but in near proximity to the cathode.

Figure 3./ Part of the time resolved spark spectrum of Zn taken in the middle of the spark gap of a gap of 8 mm through a slit of 0.45 mm wide/lower spectrum/. In the upper part of the figure in the middle of the threefold set of unresolved spectra the spectrum of the iron is [illegible] Above the iron spectrum is the spectrum of Zn photographed through a slit of 0.45 mm wide [illegible] of the cathode of the spark gap [illegible] spectrum of the iron the same as above [illegible] the middle of the spark gap.

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Figure 4./ Part of the time resolved spark spectrum of Cd taken in the near proximity of the cathode of a spark gap of 8 mm through a slit of 0.45 mm wide /lower spectrum/. In the upper part of the figure in the middle of the threefold set of unresolved spectra, the spectrum of iron is to be seen. Above the iron spectrum is the spectrum of Cd photographed through a slit of 0.45 mm wide in the middle of the spark gap, below the spectrum of the iron the same as above is, but in near proximity to the cathode.

Figure 5./ Part of the time resolved spark spectrum of Cd taken in near proximity of the cathode of a spark gap of 8 mm through a slit of 0.45 mm wide/ lower spectrum/. In the upper part of the figure in the middle of the threefold set of unresolved spectra the spectrum of the iron is to be seen. Above the iron spectrum is the spectrum of Cd photographed through a slit of 0.45 mm wide in the middle of the spark gap, below the spectrum of the iron the same as above but in near proximity to the cathode.

Figure 6./ Part of the time resolved spark spectrum of Mg taken in the middle of a spark gap of 8 mm through a slit of 0.45 mm wide /lower spectrum/. In the upper part of the figure in the middle of the threefold set of unresolved spectra the spectrum of iron is to be seen. Above the iron spectrum is the spectrum of Mg photographed through a slit of 0.45 mm wide in near proximity of the cathode of the spark gap. Below the spec

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of iron the same as above it, but [illegible] the [illegible].

[illegible] Variation of the profile of the line [illegible] in time resolved [illegible] in the middle of the spark gap.

I = relative intensity, A = angstrom, t = [illegible].

Figure 8./ Variation of the profile of the [illegible] and asymmetrically broadened Zn I 2502 line resolved spark spectrum due to the [illegible] the middle of the spark gap.

I = relative intensity, A = angstrom, t = [illegible].

[illegible] The variation in time of the profile of the [illegible] in time resolved spark spectrum [illegible] middle of the spark gap.

I = relative intensity, A = angstrom, t = [illegible].

[illegible] 10./ Optical arrangement for the production of [illegible] resolved spark spectra for spectrochemical [illegible].

Figure 11./ Optical arrangement for the separate but simultaneous production of sparklike spectrum originating from the initial phase of the spark discharge and of arclike one arising from a later phase of [illegible].

Figure 12./ Parts arising from the initial phase / upper spectrum and from the later phase /lower spectrum/ of the [illegible] resolved spark spectrum of aluminium containing

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impurities. The width of the slit of the spectrograph is 15 microns.

Figure 13./ Time resolved spark spectra of aluminium [illegible] impurities for reproducibility tests. The [illegible] spectra including the background are taken from the initial phase of the discharge.

Figure 14./ Working curve for the determination of Cu in aluminium with the aid of time resolved spark spectra. As comparison standard serve the background separated in time of the spark spectra.

Figure 15./ Working curve for the determination of Si in aluminium with the aid of time resolved spark spectra. As comparison standard serves the background separated in time of the spark spectra.

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