

Hollow Cathode Discharges

==== Analytical Applications ====

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The low pressure glow discharges considered in this paper are the hollow cathode (Paschen), and the flat cathode (Grimm). Both discharges have similar voltage—current characteristics which are responsible for their radiation stability. The analytical sample is supplied to the discharge through a sputtering mechanism which provides a stable and non-selective source of particles. Some of the fundamental properties of the glow discharge and sputtering phenomena will be discussed, including the relation between the geometry of the discharge, and the nature and pressure of sustaining gas, and current, on the emission characteristics of the discharges. These will be followed by a description of the conventional instrumentation developed for analytical purposes using the hollow cathode and flat discharge. A description of the hollow cathode developed at the National Bureau of Standards (NBS) will follow. The techniques used for the introduction of various conductive and non-conductive materials into the discharge will be discussed. The use of these discharges will be illustrated with examples taken from the literature and from the measurements performed at NBS. The paper will conclude with a discussion of possible future developments of low pressure glow discharges. A collection of references to works on low pressure glow discharges, containing 690 entries, concludes this work.

Key words: Grimm discharge; hollow cathode discharge; low pressure glow discharges; Paschen discharge; planar cathode discharge; pulsed discharge.

1. Introduction

The energy necessary to excite the radiations from various free particles—atoms, ions, molecules, and free radicals—originating from the analytical sample, and required in analytical emission spectroscopy, can be supplied in a variety of forms according to the excitation source used. Table 1 enumerates these sources which are divided in a somewhat arbitrary manner into two general categories: electrical discharges and thermal sources.

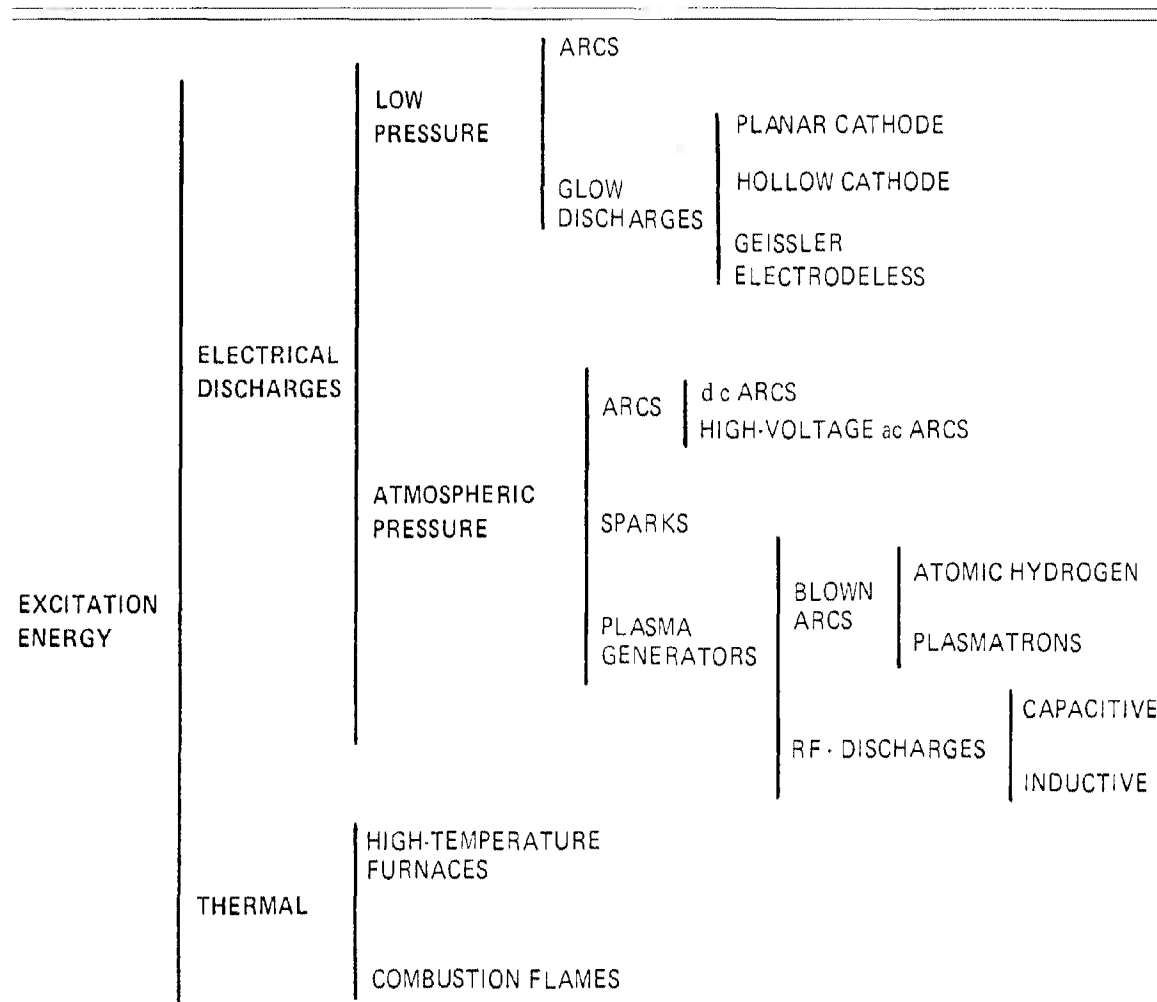
The production of free particles and their excitation through the use of high temperature furnaces or com-

bustion flames is a purely thermal phenomenon. When electrical discharges are used for the same purpose, the production of free particles and their excitation often result from a combined effect of the energy developed in the electric field and thermal energy. An extreme case in this regard is illustrated by the dc arc where the thermal energy is the determining parameter. The other extreme, where the production of free particles and their excitation results from the energy generated in electric fields, is illustrated by the low pressure glow discharges, namely the planar and hollow cathode discharge. Although in this case a certain amount of heat is generated as a result of ion bombardment at the cathode, this thermal energy is only incidental to the process and is not necessary for the production and maintenance of the discharge, for the generation of free particles, and for the subsequent excitation processes which occur in these sources.

The basic conditions required from an excitation source are: capability to be supplied in a controlled

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Table 1. Sources of energy used in analytical emission spectroscopy to excite the radiations from atoms, ions, molecules, and free radicals.



manner with the analytical sample in solid, liquid, or gaseous state for both electrically conductive and non-conductive materials. The excitation source should excite all the chemical species of interest with high sensitivity and stability. Furthermore, interferences due to matrix effects, interelement actions, chemical reactions, and selective energy transfers should be as small as possible. These interferences are associated to a large extent with those excitation sources in which the production and excitation of particles result from thermal energy, where the thermal characteristics of each chemical species such as melting, boiling, and vaporization temperatures, vapor pressure, dissociation, are specific for every chemical species and play a determining role. Self-absorption is also a phenomenon often associated with thermal excitation and is responsible for loss of sensitivity and non-linearity between the emission intensity and actual sample concentration. The processes occurring in the excitation source become even more

complex when the energy generated by an electrical field is associated with the thermal energy. From the various sources of excitation mentioned in table 1, the electrical glow discharges produced under low pressure, and in particular the planar cathode and hollow cathode discharges mentioned previously, are less subjected to the processes discussed in the foregoing, and are also practically free of self-absorption.

The sputtering phenomenon, responsible in this case for the production of free particles from the analytical sample, is less subjected to selectivity, and the absence of oxygen from the gas supporting the discharge eliminates the matrix and chemical reactions interferences resulting from the action of oxygen on the sample.

Based on these considerations we have initiated a study of the low pressure glow discharges and some of the results obtained in our preliminary experiments will be discussed in this work together with factual data from the available scientific literature.

2. Production and General Characteristics of Low Pressure Glow Discharges¹

Low pressure glow discharges of the type pertinent to our interests here can be produced using a simple cylindrical glass tube provided with two electrodes of adequate shape, an inlet for the gas sustaining the discharge, and a vacuum connection, as illustrated after Francis [105] in figure 1. The tube is filled with a rare gas, say

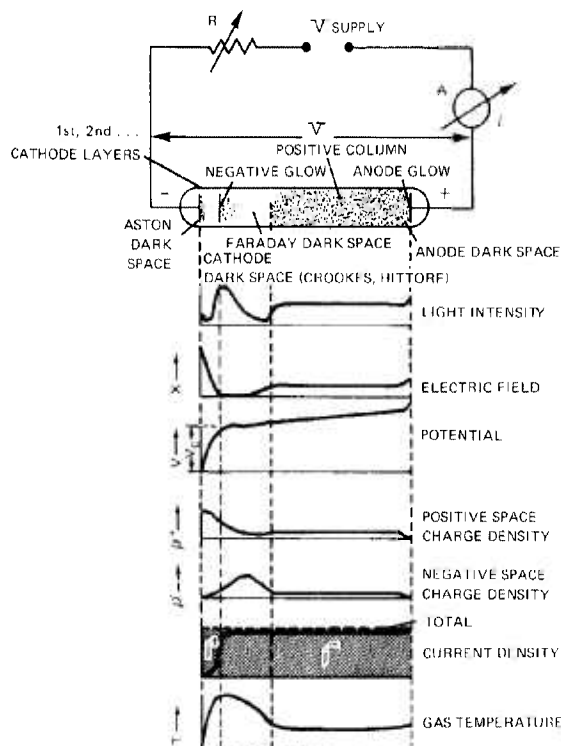


Figure 1—Characteristics of a low pressure glow discharge. After Francis [105].

helium or argon, at a pressure between 1 to 20 Torr, and a dc potential V is applied across the electrodes, through a current limiting resistor R . If the dc potential V , is increased by changing the value of the variable resistance R , a small current i is detected by the sensitive meter A . An intermittent discharge, produced in ran-

¹Most of the information presented in this section is based on the fundamental works of G. Francis [105] and F. M. Penning [257], the bracketed figures indicating references appearing at the end of this paper. Information was also obtained from other sources [94, 213, 252, 257, 365, 386 and 10].

NOTE: Bracketed numerals in roman type identify the 400 citations assembled in the main body of Section 5. Collection of References to Works on Low Pressure Glow Discharges. Bracketed numerals in italic type identify papers in a 290-citation addendum to this collection, also part of Section 5.

dom bursts, is thus observed at very low values of i of the order of 10^{-18} A. As the potential V is further increased, the current i increases rapidly and rises to values exceeding those determined by the resistance R . Under these conditions a dark self-sustained discharge is produced and the voltage at which this phenomenon is observed is called the breakdown voltage V_b (of the order of 1000 V). Such discharges are called dark discharges or Townsend discharges, and are characterized by currents of the order of 10^{-6} A under practically constant voltage. This is illustrated in figure 2 by the region AB [257]. As the current is further increased, the voltage decreases through a transitional region CD (subnormal glow discharge), and reaches a constant value V_n at the point D. A visible glow discharge is now produced at the normal cathode fall potential V_n (of the order of 200 to 300 V). This potential remains practically constant for large variations of the current from about 10^{-4} A to about 0.1 A.

With a further increase of the current the limited cathode area becomes current saturated, whereupon the voltage rises and the discharge enters in the abnormal mode of operation described by portion EF of the curve on figure 2. If the current is increased again, the voltage goes first through a sharp increase followed at F by a transition region and a sudden drop, reaching values of the order of several tens of volts for currents of the order of 10 A; this is the arc discharge. A significant characteristic of this type of discharge is heating of the sustaining gas.

The glow discharge defined by the points D-E-F on figure 2 is of interest to the analytical spectroscopist and

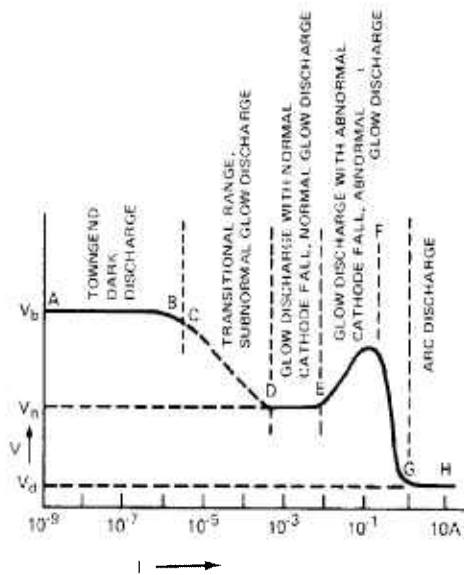


Figure 2—Voltage-current characteristics of a self-sustained low pressure glow discharge. After Penning [257].

can be produced directly by raising the potential V to the value V_n . The appearance of this discharge varies, for the same gas, with the geometry of the electrodes and that of the discharge tube, the distance between the electrodes, the pressure of the sustaining gas, the current, and the nature of the electrodes. These parameters were selected here to produce a discharge which is closely related in its properties to the discharge used in analytical applications.

2.1 Characteristics of the Low Pressure Glow Discharge

The glow discharge described schematically in figure 1 consists of a number of alternating dark and luminous zones. Their existence, disposition, and size depend on the experimental conditions; however, the example chosen here describes the general case of a discharge obtained in a glass tube 30 cm long and 5 cm wide, provided with flat copper electrodes having a diameter of 2.5 cm placed inside the tube 15 cm apart. The gas is helium at a pressure of 10 Torr and the current is 50 to 100 mA. Under these conditions the following zones are observed: at the cathode there is a very thin dark layer called the Aston primary or dark space. This is followed by a weakly luminous cathode layer, and a second dark zone called the Crookes or Hittorf dark space. Following this dark space, and sharply defined toward the cathode, is the strongly luminous negative or cathode glow. The luminous intensity of the cathode glow decreases toward the anode as it merges into another dark space called the Faraday dark space. Between this dark space and the anode there is another luminous zone, called the positive column, separated from the anode by another thin dark space and an anode glow at the surface of the electrode. The variation of the light intensity, electric field, potential, positive space charge density, negative charge density, current density, and gas temperature are described qualitatively in figure 1 [105].

An increase of pressure results in a compression of the cathode dark space, negative glow and Faraday dark space which contract toward the cathode. A decrease of pressure produces a reverse effect, and, if the voltage is not increased, the discharge goes out. A decrease of the distance between the cathode and anode produces a shortening of the anode glow, which disappears altogether where the anode is in the proximity of the cathode. When the anode is near the cathode edge of the negative glow, the voltage necessary to sustain the discharge rises rapidly and the discharge is said to be obstructed. This proves that the positive column is not essential for maintaining the low pressure glow discharge, while the part of the discharge at the cathode, including the Aston and Crookes dark spaces, are indis-

pensable. An increase in voltage results in an increase in the radiation intensity of the discharge, in particular at the cathode, and the definition of the various zones becomes sharper [105].

Unlike the conductivity of electricity in solids, the elementary processes occurring when an electrical current passes through a gas are numerous and complex. They have been summarized by Penning in the diagram from figure 3 [257]. From these, the excitation and ionization processes are the most significant in relation to the subject discussed here.

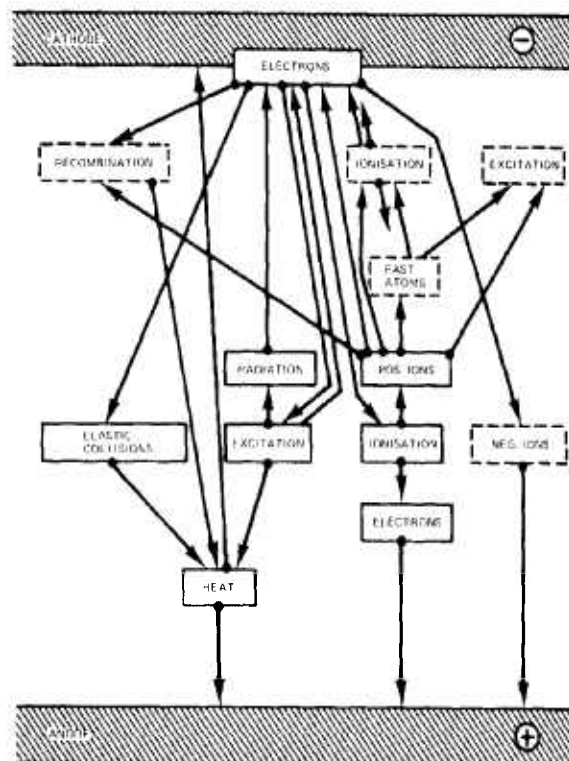


Figure 3 Diagrammatic summary of several elementary processes occurring in a low pressure glow discharge. Each process originates at the dot and ends at the arrowhead. After Penning [257]. [Courtesy of Philips Technical Library and Servire BU; Katwijk aan Zee, Netherlands, Publisher.]

The Aston dark space is characterized by the presence of electrons of low energy originating from the cathode. In the first thin layer of the negative glow these electrons are accelerated sufficiently to excite the particles found in this zone. They lose this energy in the Crookes or Hittorf dark zone. The excitation occurring in the strongly luminous negative glow is produced by the numerous electrons resulting from the ionization process occurring in the cathode dark space as well as by the few faster ones originating from the cathode. Positive ions are also generated in this zone and are attracted toward the cathode. As a result of their impact

on the cathode, atoms from this cathode are ejected and reach the glow discharge zone producing a sputtering of the material from which the cathode is made. Under these conditions a mixture of atoms from the supporting gas and from the material from the cathode are always present in the negative glow zone where they are strongly excited by collision with the ions and electrons present there [105].

The Faraday dark space is characterized by the presence of ground-state particles and low energy electrons which have lost this energy in the negative glow zone. After these electrons gain once more sufficient energy, from the electric field which accelerates them toward the anode, they produce the luminous positive zone. A spectroscopic examination of the radiations excited in this zone reveals the presence of the atomic spectra from helium which is the sustaining gas used in this example.

The same examination of the negative glow discharge reveals that, in addition to the radiations from the sustaining gas, strong emissions from the sputtered particles from the cathode are excited and emit radiations from neutral atoms, ions, and molecules. It is this basic property, i.e., the generation of free particles through sputtering and their subsequent excitation by non-thermal processes in the negative glow region, that makes the low pressure glow discharge a valuable source in analytical spectroscopy.

Further information concerning various characteristics of low pressure glow discharges will be found in references 10, 177, 180, 226, and 252.

3. The Low Pressure Glow Discharge as an Excitation Source in Emission Spectroscopy

3.1 The Planar Discharge

Described diagrammatically in figure 1, the actual aspect of this simplest form of low pressure glow discharge is illustrated in figure 4 which was obtained under the conditions described previously. The copper cathode is at left, the copper anode at right, and the principal zones seen are the strongly luminous negative glow at the cathode, followed by the Faraday dark space, the positive column, and the anode glow. The thin Aston and Crookes dark spaces, and the faint cathode layer and the anode dark space cannot be distinguished on this photograph.

As discussed previously, the zone of interest is the negative glow since the particles sputtered from the cathode, which in this case is the analytical sample, are strongly excited in this zone and produce the spectra of the neutral and ionized atoms together with pertinent

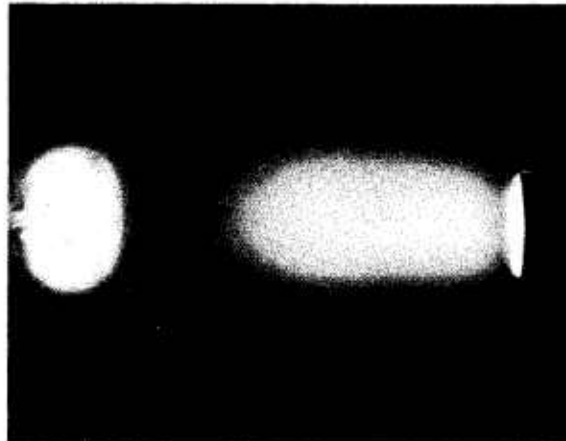


Figure 4—A low pressure glow discharge produced between two copper disc electrodes in helium at 10 Torr and 100 mA. The cathode is at left, the anode at right.

molecular spectra. The positive column and the anode glow, which are not needed to produce or maintain the discharge, can be eliminated altogether by bringing the anode electrode near the cathode, and by placing this anode outside the field of examination of the cathode glow. It is from this basic and simplest form of the flat or planar glow discharge that Grimm has developed a discharge tube that is now used routinely as an excitation source in analytical emission spectroscopy [128, 129]; a diagrammatic description of this discharge tube is given in figure 5. It consists of a cylindrical anode and cathode unit, made usually of a copper alloy, and separated by an insulator. The distance between the cathode and the anode is about 0.2 mm. The metallic analytical sample, in the shape of a flat disc, is placed against the cathode provided with a vacuum tight "O" ring, and is in electrical contact with the cathode. The anode is closed with a vacuum tight window. An adequate gas, usually

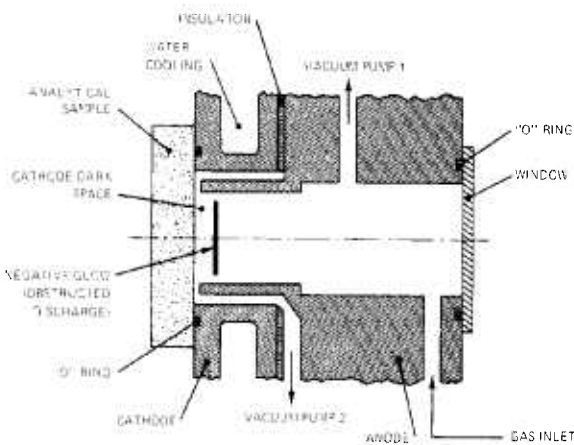


Figure 5—Diagrammatic description of the planar low pressure glow discharge developed by Grimm [128, 129].

argon or helium, is introduced in the lamp at a pressure of 1 to 20 Torr and flows continuously through the unit. The cathode body is water cooled.

Under these conditions, when a dc current of 100 to 600 mA and 700 to 2500 V is supplied to the lamp, a glow discharge is produced in front of the cathode, inside the anode space. This discharge is the negative glow described previously in figures 1 and 4. It has the characteristics of an obstructed discharge, and, is suspended free of any material contacts with the two electrodes, being isolated from the walls of the anode by a circular gap, which in fact is the anode dark space, and separated from the cathode by the cathode dark space. The intensely luminous negative glow has a thickness of the order of the mean free pathlength of the particles, and consists mostly of excited particles sputtered continuously and uniformly from the analytical sample and from the supporting gas. The dc current can be supplied to the discharge in an uninterrupted manner or in a pulsed mode. The pulsed mode is generally used with analytical sample subjected to overheating when undergoing the sputtering process.

The glow discharge source developed by Grimm can be obtained from RSV-Präzisionmessgeräte, GmbH Hauptstrasse 60, D-8031 Seefeld 2, West Germany,² including the source, the vacuum system, and the dc power supply for continuous or pulsed operation. The

²In order to adequately describe materials and experimental procedures, it is occasionally necessary to identify a commercial product by a manufacturer's name or label. In no instance does such identification imply endorsement by the National Bureau of Standards, nor does it imply that the particular product or equipment is necessarily the best available for that purpose.

RSV company is represented in North America by Labserco Ltd. Unit 8 1100 Invicta Drive, Oakville, Ontario L6H 3K9. A similar low pressure glow discharge source using a planar cathode is manufactured in this country under the name "Cathaquant" by the Spectrogram Corp., 385 State Street, North Haven, CT 06473. A detailed study of the functioning characteristics of this discharge source was made by Grimm [128, 129], Boumans [40], and by Dogan, Laqua, and Massmann [85, 86] and El Alfy, Laqua, and Massmann [93] at the Institut für Spektrochemie und Angewandte Spektroskopie in Dortmund, Germany, and all the data used, and the statements made in the following discussion, are taken from these basic contributions. Further contributions were made by Boumans [41].

The current-voltage characteristic of the discharge is illustrated in figure 6(a) for various pressures, using argon as the carrier gas and nickel for the cathode. This parameter depends, for a given current, also on the type of carrier gas and its pressure and on the type of cathode sample material. The relative intensity of the Ni 3610.46 Å line as a function of the current supplied to the lamp and for several argon pressures also is given in figure 6(b). These measurements were made with a 1.5 m direct reading grating spectrometer [129].

The linear relation between the concentration of a chemical element and the corresponding signal intensity obtained with the Grimm glow discharge source is illustrated in figure 7 for Sb in Cu-Pb-Sn(a), Cu in Al(b), Zn in Al(c), and Mn in Al(d) matrices, in comparison with a conventional spark.

The matrix effect is illustrated in (c) and (d) of figure 7 for Zn and Mn in Al and in AlMg and AlMgSi alloys,

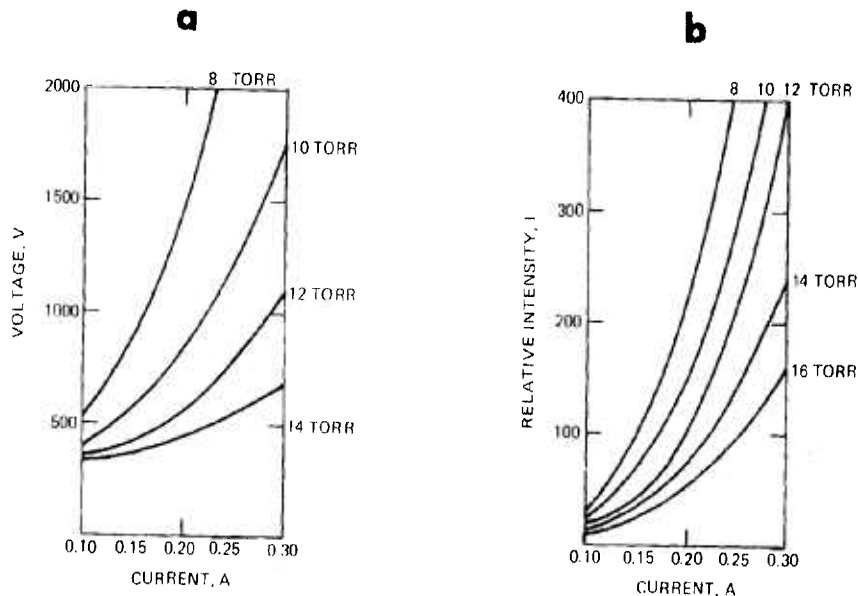


Figure 6— Voltage-current characteristics of the discharge from figure 5, in argon (a), and (b), the relative intensity of the Ni radiation at λ 3610.46Å as a function of the current supplied to the discharge in argon [129].

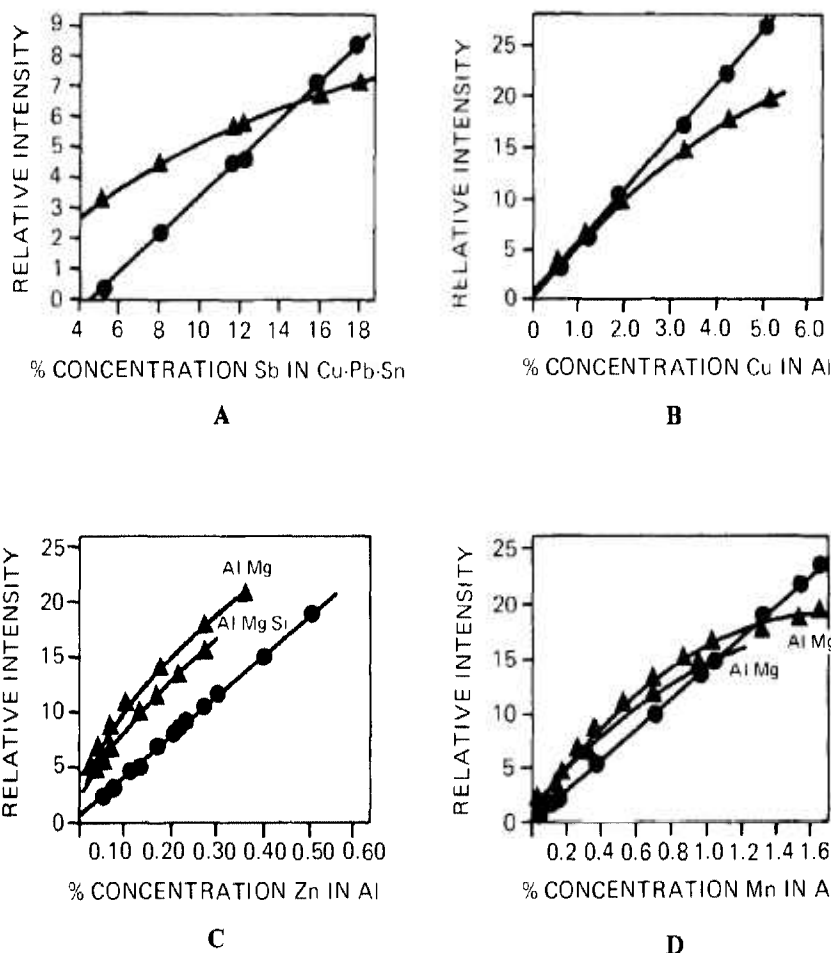


Figure 7—Relation between the concentration of a chemical element and the corresponding signal intensity obtained in a Grimm glow discharge source in comparison with a spark source for: Sb in Cu-Pb-Sn (A); Cu in Al (B); Zn in Al, Al-Mg, and Al-Mg-Si (C); and Mn in Al, Al-Mg, and Al-Mg-Si (D) samples. After Grimm.

● GLOW DISCHARGE
 ▲ SPARK

determined with the glow discharge source and a conventional spark source. In all cases the glow discharge was operated in argon at a pressure of 5 to 7 Torr and a discharge current of 0.15 to 0.25 A. A preburn of 10 to 40 s and an integration time of 3 to 40 s were used for the glow discharge.

The detection sensitivity of the planar glow discharge was determined for several elements by Grimm [129] and is given in table 2.

Table 2. Detection sensitivity for the glow discharge source after Grimm [129]

Element	Line Å	Detection limit (ppm)
Beryllium	2494.73	2
Silicon	2881.58	5
Iron	3020.64	80
Magnesium	2795.53	80
Chromium	4254.35	20
Molybdenum	3864.11	10
Aluminum	3961.52	100
Lead	4057.83	100

This detection sensitivity can be increased by looking at the negative glow sidewise [85], or by using the glow-discharge in the hollow cathode mode; this mode of operation will be discussed later in this work.

Further physical characteristics of the glow discharge source were investigated by Dogan, Laqua, and Massmann [85], using a lamp similar to that developed by Grimm, but with additional water cooling of the analytical sample. This provision permits the use of higher currents without overheating the sample.

When a new sample with a fresh surface is submitted to the discharge, the current exhibits a certain instability. This is due to surface impurities and oxides. After the surface has been cleaned through the sputtering process, the discharge stabilizes and "burns" quietly. Aluminum surfaces require a longer cleaning time (40s) while zinc cleans after a shorter preburn (3s) both in argon. The preburn time depends also on the carrier gas and is shorter in krypton than in argon or neon.

The analytical sample is supplied to the discharge as an electrical conducting flat disc with a smooth surface. It is pressed against the cathode which is provided with

an "O" ring to insure a vacuum tight seal. The surface sample exposed to the discharge is 0.28 cm². It is desirable that this surface be representative of the sample, homogeneous, and with particles having a diameter less than 0.1 mm for electrically conducting samples. Non-conducting samples can be mixed with a conducting powder material such as copper, silver, or graphite and pressed to produce the disc-shaped sample.

The amount of material sputtered during the discharge, for the same discharge conditions, i.e., current, gas, pressure, exposed surface, time, depends on the sample material. For instance, the amount of material sputtered in one minute is 0.34 mg for aluminum, 1.0 mg for copper, 3.1 mg for zinc, and smaller amounts for graphite and carbon. When arranged in increasing order, carbon sputters least and is followed by aluminum, iron, steel, copper, brass, and zinc. The amount of sputtered material per unit of time increases with the increase of current. For a power of 90 w the amount of aluminum sputtered is 0.30 mg/min in neon, 0.34 mg/min in argon, and 0.38 mg/min in krypton.

As already mentioned, the spectra excited in a low pressure glow discharge are characterized by the presence of radiations originating from the atoms, ions, molecules, and free radicals from the analytical sample and the carrier gas. The initial emissions originate from the impurities and oxide layers formed at the surface of the sample. After these layers are eroded through the sputtering process, the spectra observed are emitted by the elements which constitute the analytical sample, together with those from the carrier gas, and sometimes with the molecular emission from OH and N₂ as impurities. The intensities of these emissions depend on the concentration of the corresponding elements in the sample, the discharge parameters, the geometry of the discharge lamp, and on the carrier gas. In general an increase in current supplied to the discharge increases the radiation intensity. For instance, a twofold increase in current from 60 mA to 120 mA produces a fivefold increase in the intensity of magnesium emission at λ 2852.13 Å, at an optimum argon pressure of about 8 Torr.

Another property of the discharge is the continuum emission from the carrier gas. For argon this emission is stronger toward the longer wavelengths, while for krypton it is stronger for the short wavelengths.

All the parameters discussed above should be taken into consideration when the Grimm low pressure glow discharge is used for quantitative analyses since they all affect the sensitivity, precision, and accuracy of the measurements.

The essential characteristics of the low pressure glow discharge can be summarized as follows.

—The excitation conditions prevailing in the discharge produces for *all known chemical species*, radiations originating from atoms, ions, and molecules (free radicals) with *narrow natural spectral line width* and radiations which are, under the experimental conditions described in this work, *practically free of self-absorption*.

—The generation of particles and their supply into the discharge results from a *non-thermal and a practically non-selective cathode sputtering process*.

—The discharge is produced in an *inert gas* at low pressure and may be used directly as an excitation source in the vacuum ultraviolet.

—As a result of its *current-voltage characteristics*, the discharge is *particularly stable*.

—The discharge is silent.

The use of the Grimm discharge for the quantitative analysis of electrically conductive and non-conductive samples was studied in detail by Dogan, Laqua, and Massmann [86], and by El Alfy, Laqua and Massmann [93], and some of their results will be summarized here. The detection limits measured for several trace elements in aluminum are given in table 3 and were obtained using argon as a carrier gas at a current of 100 mA and 900 V and with a preburn of 45 s and an exposure of 6.5 min. These detection limits compare favorably with those obtained with sparks and interrupted ac arcs.

Lower detection limits were observed in krypton for atomic radiations, while neon produced lower detection limits for radiations originating from ions. With argon the detection limits were similar for both atomic and ionic radiations; therefore, this gas presents the best compromise.

Table 3. Detection limits for several elements determined with the Grimm discharge lamp [86].

Element and wavelength, Å*	Detection Limits in Percent for	
	Medium quartz spectrograph	3.m grating spectrograph
Cu I, 3247.54	4.0×10^{-5}	1.5×10^{-6}
Fe I, 3020.64	3.0×10^{-3}	2.1×10^{-4}
Mg I, 2852.13	1.2×10^{-4}	8.5×10^{-6}
Mg II, 2795.53	1.1×10^{-4}	8.5×10^{-6}
Mn I, 2794.82	1.8×10^{-4}	1.4×10^{-5}
Mn II, 2576.10	1.1×10^{-4}	2.2×10^{-5}
Mn II, 2593.73	2.4×10^{-4}	4.6×10^{-5}
Si I, 2881.58	4.0×10^{-4}	2.7×10^{-5}
Si I, 2516.11	1.1×10^{-4}	9.4×10^{-6}
Ti I, 3653.50	1.7×10^{-3}	—
Ti II, 3349.41	3.8×10^{-3}	1.6×10^{-5}
Zn I, 3345.02	1.5×10^{-3}	3.6×10^{-5}

*As given in *Tables of Spectral-line Intensities*, W. F. Meggers, Ch. H. Corliss and B. F. Scribner, NBS Monograph 145, Part I, 1975.

The detection limits measured for the same elements in electrically non-conducting materials produced somewhat higher values. In this case the non-conductive sample was mixed with a conducting powder such as copper (reduced) silver, aluminum or graphite in a ratio of up to 1 to 1 sample to metal by volume, and the mixture was pressed (8 to 10 tons/cm²) into a circular disc 1 to 2 mm thick.

Table 4 presents a comparison between analytical data obtained through chemical procedures (Chem.) and by emission spectrometry with the Grimm discharge source (GDS) on a variety of non-conducting sample materials [93]. These data are the average of 10 separate measurements. In this case 50 mg of the pulverized sample was mixed with 950 mg of copper powder (Merck No. 2715) and pressed into a 10 mm diameter disc at a force of at least 8 tons. The standards were obtained by mixing and diluting the oxides of Si, Al, Fe, Ti, Mg, and Mn in a Ca CO₃ matrix to produce the desired range of known concentrations. The discharge was operated in argon at a current of 160 mA, using a preburn of 60 to 180 s and an exposure time of 2 to 10 min. The spectra were recorded on photographic plates and the spectral line densities were measured with a micro-densitometer using copper as an internal standard.

These data demonstrate that the Grimm source is capable of performing spectrometric analyses with excellent precision and accuracy, and that the precision is probably related to the inevitable limitations of the photographic plate used as a receptor. Table 4 also shows that the concentration spread for the 10 elements determined extends from about 0.01 to several tens percent, covering the minor and major constituents range.

A modified Grimm discharge is described in references 4, 37, 43, 44, and 223.

The measuring sensitivity of the low pressure planar glow discharge can be increased by producing the discharge in the hollow cathode mode.

3.2 The Hollow Cathode Discharge

3.2.1 Production of the Discharge

The aspect of a low pressure glow discharge is illustrated in its simplest form in figure 4, which was obtained with a single flat cathode. If a second flat cathode is now placed in the discharge tube and the two cathodes are separated by a gap of approximately 25 mm, the discharge produced takes the form observed in figure 8.

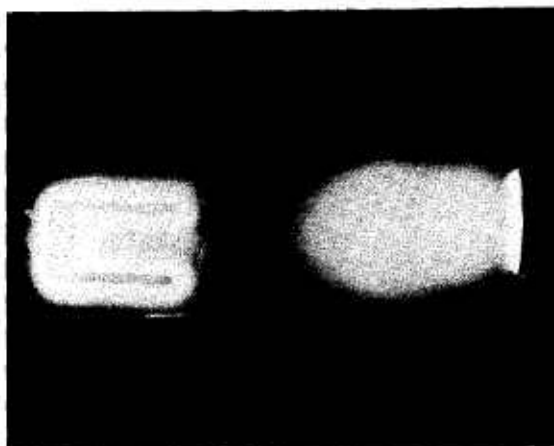


Figure 8-- A low pressure glow discharge produced between two flat copper cathodes separated by a gap of 25 mm, and a flat copper anode, in helium at 10 Torr and 100 mA.

Table 4. Comparison between chemical (Chem.) and spectrometric analyses obtained with the Grimm discharge source (GDS), [93].

Sample	Limestone ^a		Granit ^a		Basalt ^a		Flintclay ^a		Argillaceous limestone ^b		Pb-Ba-Glass ^b		Cement ^c	
	Chem.	GDS	Chem.	GDS	Chem.	GDS	Chem.	GDS	Chem.	GDS	Chem.	GDS	Chem.	GDS
Ca	34.10	34.0	0.83	0.83	4.64	4.30	0.28	-	29.44	29.5	0.15	-	29.50	30.5
Si	3.99	4.0	33.97	34.0	22.90	23.0	27.90	28.0	6.55	6.6	30.30	30.0	6.60	6.5
Al	1.27	1.30	7.27	7.50	8.73	8.5	11.10	11.0	2.21	2.20	0.10	0.12	1.56	1.60
Fe	0.69	0.68	1.48	1.50	6.81	6.9	4.85	4.75	1.10	1.08	0.03	0.27	1.12	1.10
Mg	0.45	0.45	0.26	0.30	4.49	4.50	1.18	1.20	1.30	1.30	0.02	-	0.83	0.80
Mn	0.07	0.072	0.04	0.046	0.12	0.12	0.04	0.042	0.03	0.035	0.07	0.068	-	0.03
Ti	0.08	0.078	0.13	0.15	0.69	0.67	0.56	0.58	0.01	0.012	0.007	-	-	0.06
Na	0.16	0.12	2.88	2.90	3.27	3.25	0.94	0.87	-	-	4.25	4.25	-	-
K	0.32	0.35	3.87	4.10	0.17	0.17	3.18	3.40	-	-	6.96	7.0	-	-
C	10.21	10.30	0.07	0.07	0.36	0.36	0.04	0.033	-	-	-	-	-	-

^aZentral Geologischen Instituts, Berlin.

^bNational Bureau of Standards, Washington.

^cCentre d'Etudes et de Recherches de l'Industrie des Lants Hydrauliques, Paris.

In this case the discharge follows the outline of the two flat cathodes, and is otherwise identical in structure and properties with the discharge obtained when a single flat cathode is used (Fig. 4).

If the distance between the two cathodes is decreased to about 8 mm or less, the two separate cathode layers from the preceding figure 8 are seen to coalesce into a single cathode layer having a high radiation intensity, as illustrated in figure 9. This is the hollow cathode discharge. In practice, instead of using two flat cathode electrodes, a cylindrical cathode is used as illustrated in figure 10. This discharge, which occurs inside the cylindrical cavity, can carry currents of the order of several amperes at a cathode fall of several hundred volts and in a relatively cold carrier gas. The distribution of the various zones seen in the planar cathode discharge mode is present in the hollow cathode mode also. The zones are disposed in a circular manner inside the hollow cathode.

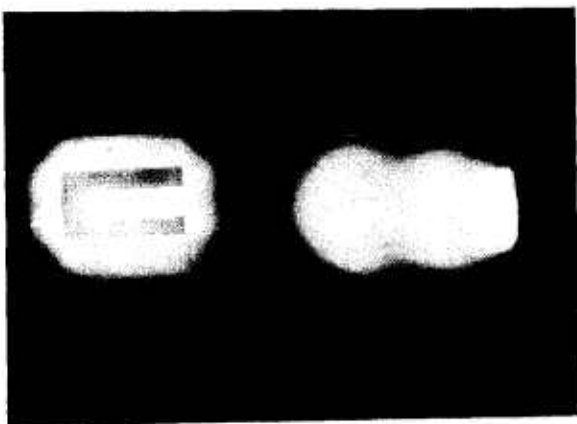


Figure 9 – Same discharge as shown in figure 8 with the two cathodes separated by a gap of 8 mm.

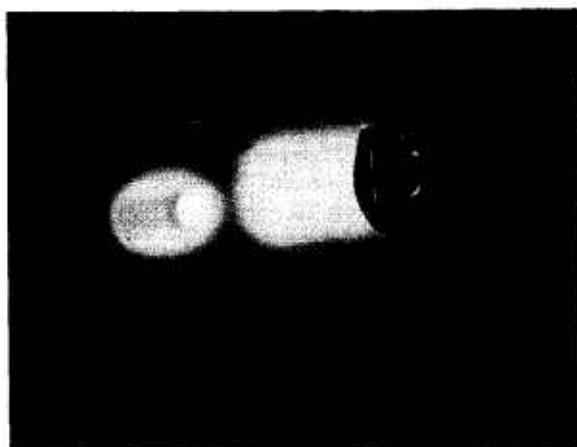


Figure 10 – Same discharge as shown in figure 9 with a cylindrical cavity as a cathode.

The low pressure hollow cathode discharge source, first described by Paschen in 1916 [253], consisted of a metallic rectangular hollow cathode and a cylindrical anode sealed in a glass vessel filled with helium at a pressure of several Torr (Fig. 11). It was adapted by Schüler and Gollnow to analytical measurements in 1937 [315], and is described in figure 12. It consists of a water cooled anode and cathode unit separated by a glass tube. The gap between the interchangeable hollow cathode and the anode electrode is of 1 mm. The hollow cathode sources used today are all derived from this basic form which was modified to a smaller or larger extent to satisfy the particular requirements of the analyst.

3.2.2 Description of an Experimental Hollow Cathode Discharge Source

One of the forms developed and used in our laboratory is illustrated schematically in figures 13 and 14. It consists of a water cooled circular anode and cathode units made of brass. The anode is provided with an inlet for the carrier gas, a fused silica window, and a terminal. An adjustable conical ring permits the distance between the anode and cathode to be varied. The cathode unit can accept an interchangeable cylindrical hollow cathode 19 mm long, with an external diameter of 6 mm, provided with a 14 mm deep and 4 mm diameter bore. The anode and cathode are separated by a ring made of 99.7 percent pure alumina provided with a lateral alumina tube for connection to a vacuum pump. The anode, alumina ring and the nylon sleeve are assembled with six nylon screws. The nylon sleeve holds the cathode unit in place, and "O" rings are used to provide a vacuum tight assembly.

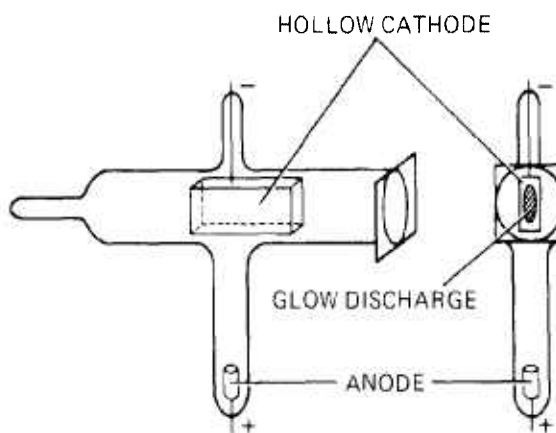


Figure 11 – Schematic description of the first low pressure hollow cathode discharge developed by Paschen [253].

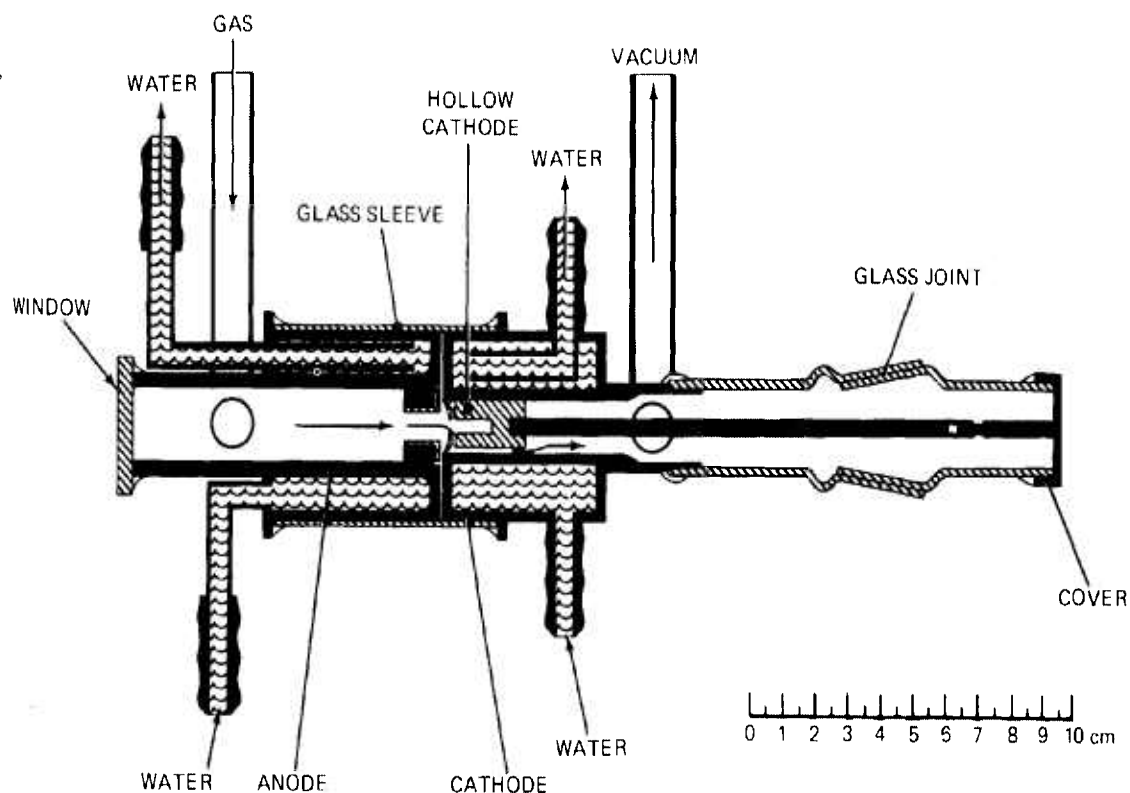


Figure 12—A hollow cathode low pressure glow discharge source developed by Schüller and Gollnow for analytical measurements [315].

The nominal dimensions are: length and diameter of anode unit, 65 mm and 76 mm, respectively; length of alumina insulator, 12 mm; and length and diameter of cathode unit, 38 mm and 36 mm, respectively.

The cathode from (b) of figure 14 consists of a conical shaped part provided with an opening of 3 mm, and is made of pure copper in this case. Other pure materials could be used including graphite. The bottom of this conical unit is closed with a pellet 2 mm thick made from the analytical sample. This can be a solid metallic pellet or a pressed disc made of a metallic powder or a nonconducting sample mixed with a conducting powder such as pure copper. This type of hollow cathode was developed and used by Spectroscandia (Nagu, Finland) [339].

An exploded view of the unit is illustrated in figure 15 and the assembled unit is shown in figure 16. Figure 17 shows the unit in front of a Czerny-Turner 1 m grating universal spectrometer which can be operated as a scanning monochromator, a spectrograph, or a multichannel direct reading spectrometer.

The hollow cathode is connected, through a resistance of 1000 Ω and 2000 w, to a dc power supply capable of providing a current of 2.5 A and 2000 V.

When the spectrometer is used in the scanning mode, the signal measuring system consists of a photomultiplier followed by a lock-in amplifier, an analog recorder and digital voltmeter. A scaler and timer unit is also available for integration of the signal over a chosen time interval. These parts are contained in the cabinet from figure 17. left. An 8-channel integrating electronic system is used in conjunction with the multichannel functioning mode, and is illustrated in figure 18 at left.

The carrier gas, usually pure argon, helium, or a mixture thereof, is supplied to the hollow cathode from corresponding high pressure bottles. The continuous flow is controlled through pressure regulators at the bottles and is limited by an individual glass capillary tube; the flow is monitored by flowmeters. A duo-seal oil vacuum pump provides the necessary vacuum which is monitored through a sensitive Bourdon-type mechanical gauge, and the hollow cathode source is connected to the gas and vacuum through off-on vacuum valves. The monitoring gauges and the valves and vacuum pump are contained in a cabinet as seen in figure 18. The current-monitoring meters—milliampmeter, voltmeter, and wattmeter—are located at the top of this cabinet which also contains the 1000 Ω , 2000 w resistor.

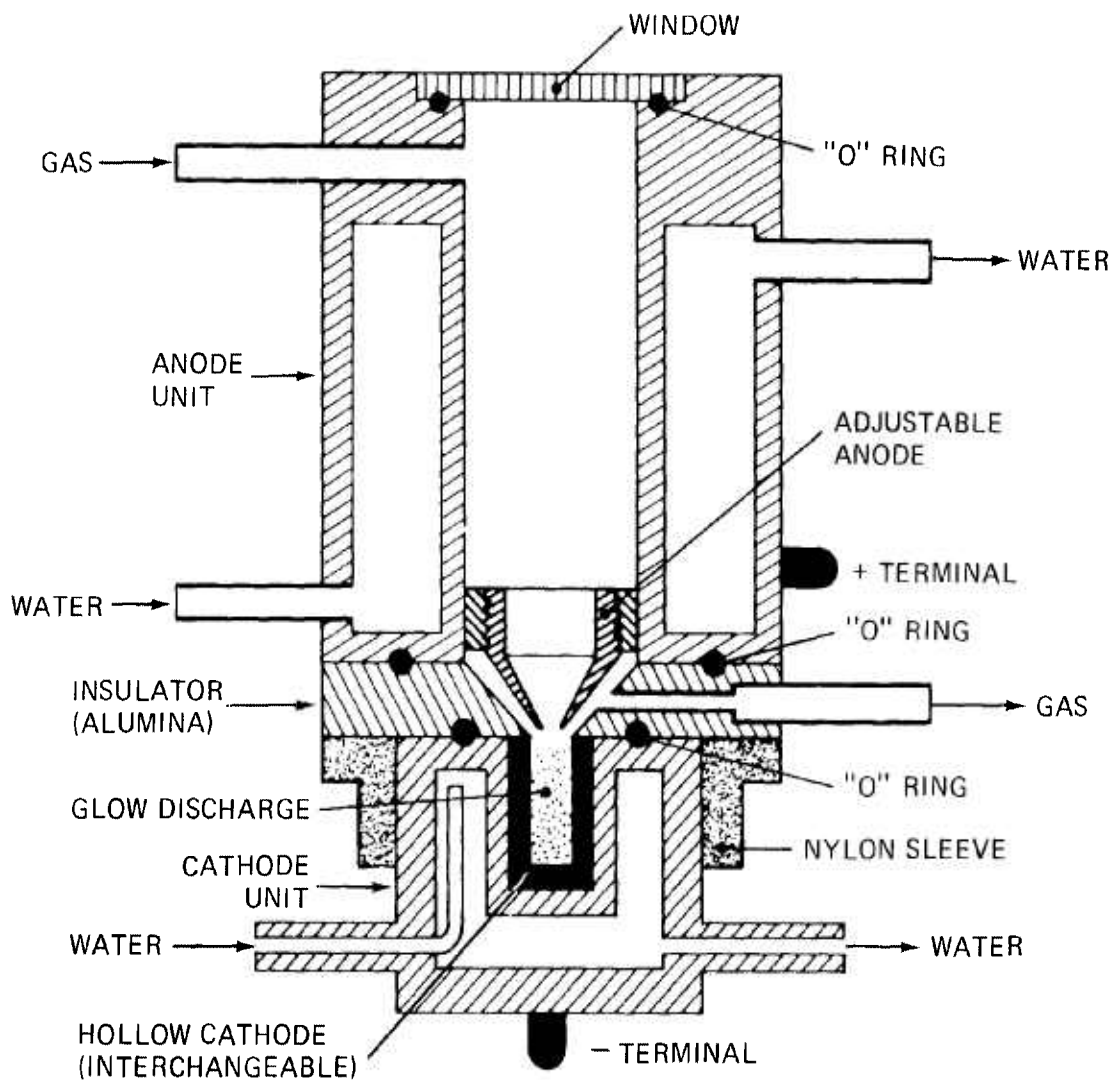
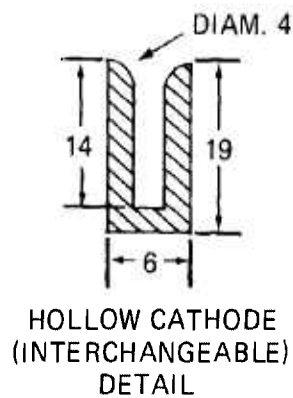


Figure 13--Experimental hollow cathode source developed in the Center for Analytical Chemistry, National Bureau of Standards. (Dimensions of detail, right, in mm.)



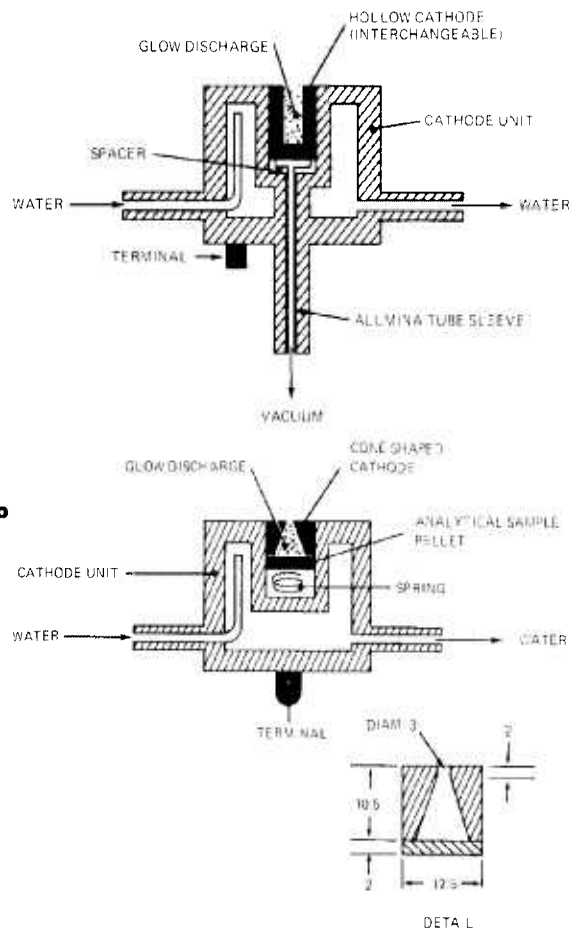


Figure 14—Alternate cathode unit (a) used in conjunction with the anode unit of figure 13. The hollow cathode proper is made from the metallic analytical sample and is interchangeable. Another alternate cathode unit, (b), also is used in conjunction with the anode unit from figure 13. The hollow cathode proper is made from two parts: a conical body, provided with a 3 mm orifice, closed by a pellet at the bottom. This pellet is the analytical sample, and can be made from a solid conducting material (metal) or pressed from a metallic powder. Non-conducting materials can be pelleted with a conducting powder such as pure copper, silver or graphite. Dimensions, right, in mm [339].

3.2.3 Characteristics of the Hollow Cathode Discharge Source

The voltage-current characteristics of the discharge were determined using an iron hollow cathode of 3.5 mm inside diameter, at various pressures, in helium, argon, and helium-argon mixture, and the results are given in table 5. From these measurements it can be seen that for a large change in current, from 50 mA to 1600 mA, the corresponding change in voltage is small, from 260 V to 430 V, when argon is used at a pressure of 10 Torr. This current-voltage relation of hollow cathode discharges is responsible for the stability of this excitation source.

Hollow cathodes can be operated at currents from several milliamperes to several amperes. Since the intensity of the radiations excited in these sources is a function of this current, the hollow cathodes used in analytical applications are supplied with currents varying from about 100 mA to about 2 A, the first hollow cathode operated at such high current being described in 1933 by Paschen and Ritschel [256]. Figure 19 describes this source which consists of a metallic cathode made, in this case, of a thick aluminum block 200 mm long provided with a transversal bore of 5 mm. A fused silica container is adapted at each end of this block through conical ground joints, and each is provided with a cylindrical anode, a quartz window, and side tubes for the carrier gas and vacuum connections; the cathode is water cooled. Under these conditions currents of up to 3 A were supplied to the hollow cathode producing an extremely brilliant source of radiation. More recently, high-current hollow cathodes were designed and used for analytical purposes by Maierhofer and associates [196], and by Thornton [362]. The current used with the source illustrated in figures 13 and 17 varied from about 200 mA to 1800 mA according to the experimental conditions.

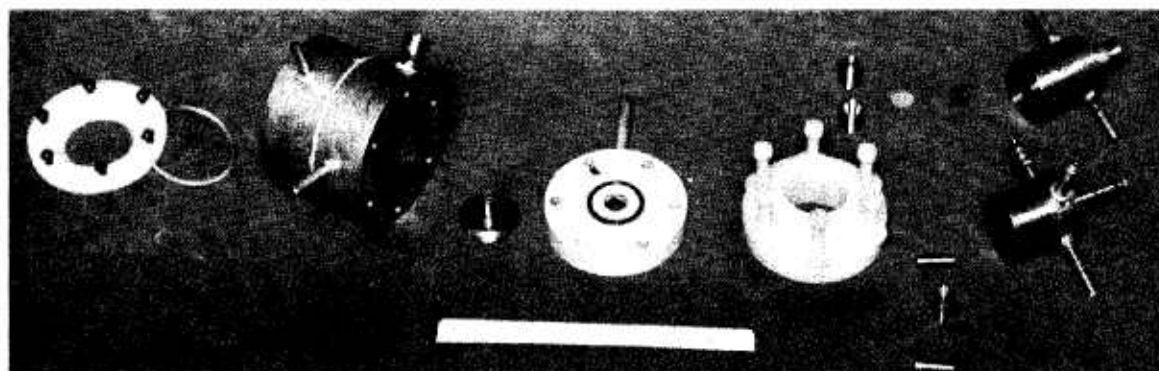


Figure 15—Exploded view of the hollow cathode source described in figures 13 and 14. From left: fused silica window with nylon retaining ring, anode unit, adjustable anode electrode, alumina insulator, nylon sleeve with six nylon assembling bolts, three cylindrical interchangeable hollow cathodes (Fe; Cu; Ag), conical hollow cathode with sample pellet, and two cathode units.

Table 5. Voltage-current characteristics for an iron hollow cathode 3.5 mm diam. in helium and argon at various pressures.

Current A	Volts							
	Helium at			Argon at			Helium-Argon mixture 1:1	
	3 Torr	6 Torr	10 Torr	3 Torr	6 Torr	10 Torr	10 Torr	13 Torr
0.050	230	240	300	240	280	260	220	280
0.10	230	240	300	240	300	300	230	300
0.20	230	240	300	400	310	310	220	315
0.30	260	240	300	480	315	330	200	340
0.40	320	230	300	540	340	330	200	360
0.50	540	230	320	620	340	360	210	370
0.60	580	240	310	700	360	380	220	370
0.70	620	240	300	760	380	380	240- 320	380
0.80	640	260	300	820	390	380	340	380
0.90	680	270	300	870	400	380	360	390
1.00	720	400	300	920	410	380	370	400
1.10								
1.20	770	440	310	1000	440	400	400	400
1.30				Arc				
1.40	820	460	320		480	420	410	400
1.50								
1.60		490	340		510	430	440	400
1.70								
1.80		Arc	360		540	440	440	400

The development and use of high intensity hollow cathodes is described in references 2, 20, 28, 29, 151, 184, 239, 245, 266, and 279.

The radiation intensities originating from the atoms of various elements, excited in a hollow cathode, are for a given set of experimental conditions a function of the current. This dependence was determined for the source described in figures 13 through 18, using a silver hollow cathode in helium at 3, 6, and 10 Torr pressure, and the results obtained are given in table 6. These data indicate that an increase of current from 50 mA to 1200 mA corresponds to an increase in the relative radiation intensity (PMV) of the Ag λ 3280.68Å emission from 0.30 V to 11.75 V, or a factor of about 40, tapering off from about 1100 mA.

The spatial distribution of this radiation inside the hollow cathode is illustrated in figure 20, after Büger and Fink [55]. The figure shows the relative intensity profiles obtained in helium at a pressure of 5 Torr and for currents from 40 mA to 1000 mA. The highest intensity was observed in the middle of the negative glow discharge, while close to the walls of the hollow cathode, the radiation intensity decreased to practically zero. This is to be expected since the cathode dark space is located between the wall of the hollow cathode and the negative glow. Similar observations were made earlier by Berezin [23].

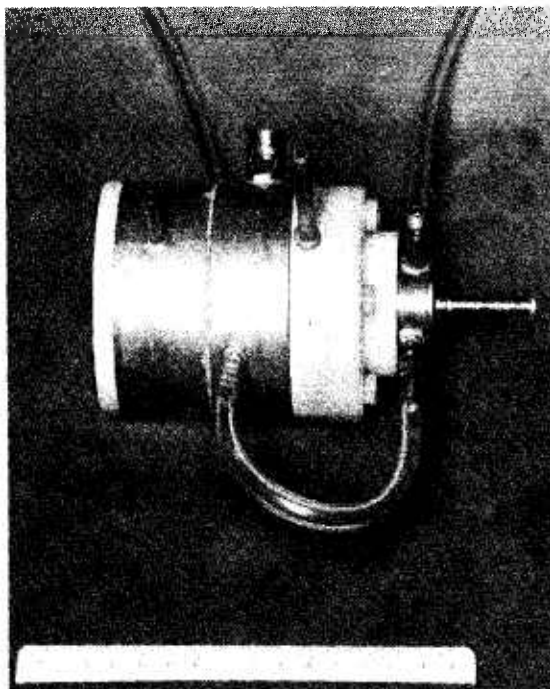


Figure 16—Assembled hollow cathode source.

Table 6. Relative intensity, expressed as photomultiplier voltage (PMV), for Ag λ 3280.68Å as a function of the current (mA) supplied to the hollow cathode source (HC) in helium at various pressures (P_{He}).

HC, mA	PMV		
	P_{He} 3 Torr	P_{He} 6 Torr	P_{He} 10 Torr
50	0.11	0.25	0.30
100	0.31	0.67	0.74
150	0.51	0.86	1.50
200	0.78	1.11	1.71
250	1.03	1.32	1.86
300	1.34	1.63	2.08
350	1.75	1.94	2.39
400	2.11	2.26	2.68
450	2.45	2.61	3.04
500	2.86	3.04	3.38
550	3.20	3.48	3.76
600	3.62	3.93	4.25
650	4.07	4.37	4.57
700	4.70	4.70	4.80
750	5.32	5.21	5.38
800			5.90
850			6.30
900			6.82
950			7.44
1000			9.05
1100			10.15
1200			11.75

The radiation stability of the hollow cathode source was determined using copper, silver, and iron hollow cathodes in helium at 10 Torr and for currents of 200, 100, and 200 mA at λ 3273.96Å, 3280.68Å, and 3719.94Å, respectively. The results obtained are assembled in table 7.

The table 7 data are expressed as averages of photomultiplier voltages. These data were obtained from the tracings produced with the 1 m spectrometer described in figures 17 and 18 and operated in the analog recording mode. A preburn of 10 min was used in all cases. The stability of the photomultiplier with the associated electronics was initially determined using a stable radiation source consisting of a tritium-activated phosphor (half-life 12.5 years). Under these constant illumination conditions, a variation of the photomultiplier signal of 0.3 percent was observed over a period of 60 min. The noise observed during each measurement, expressed as photomultiplier voltage, was on the order of ± 0.2 V.

The table 7 data demonstrate the excellent stability of the hollow cathode discharge which is capable of producing and maintaining a radiation intensity over a time period of 20 min with a relative standard deviation of not more than 1.6 percent for a single measurement.

The effect of the sputtering process on the surface of

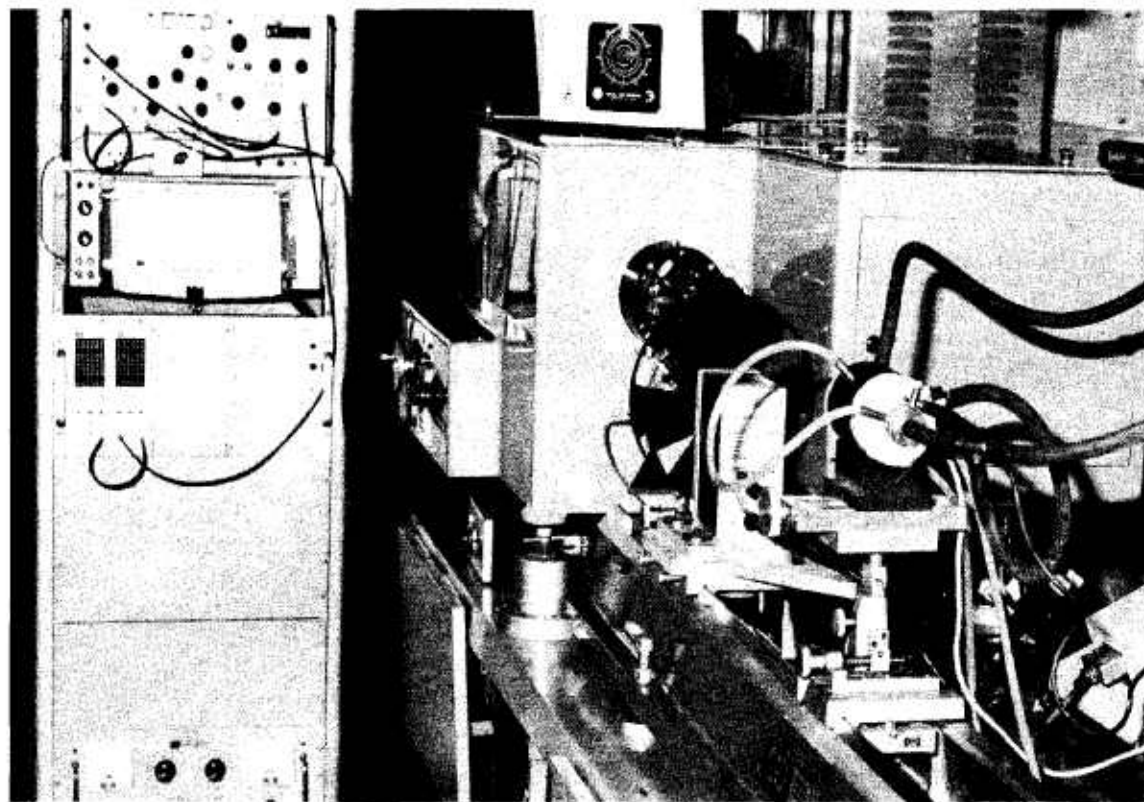


Figure 17—Hollow cathode unit from figure 16 under functioning conditions on an optical bench in front of a 1 m Czerny-Turner spectrometer. From left: console with power supply for the photomultiplier tube; the preamplifier, amplifier and lock-in amplifier; and the digital voltmeter, analog recorder and time-scaler unit.

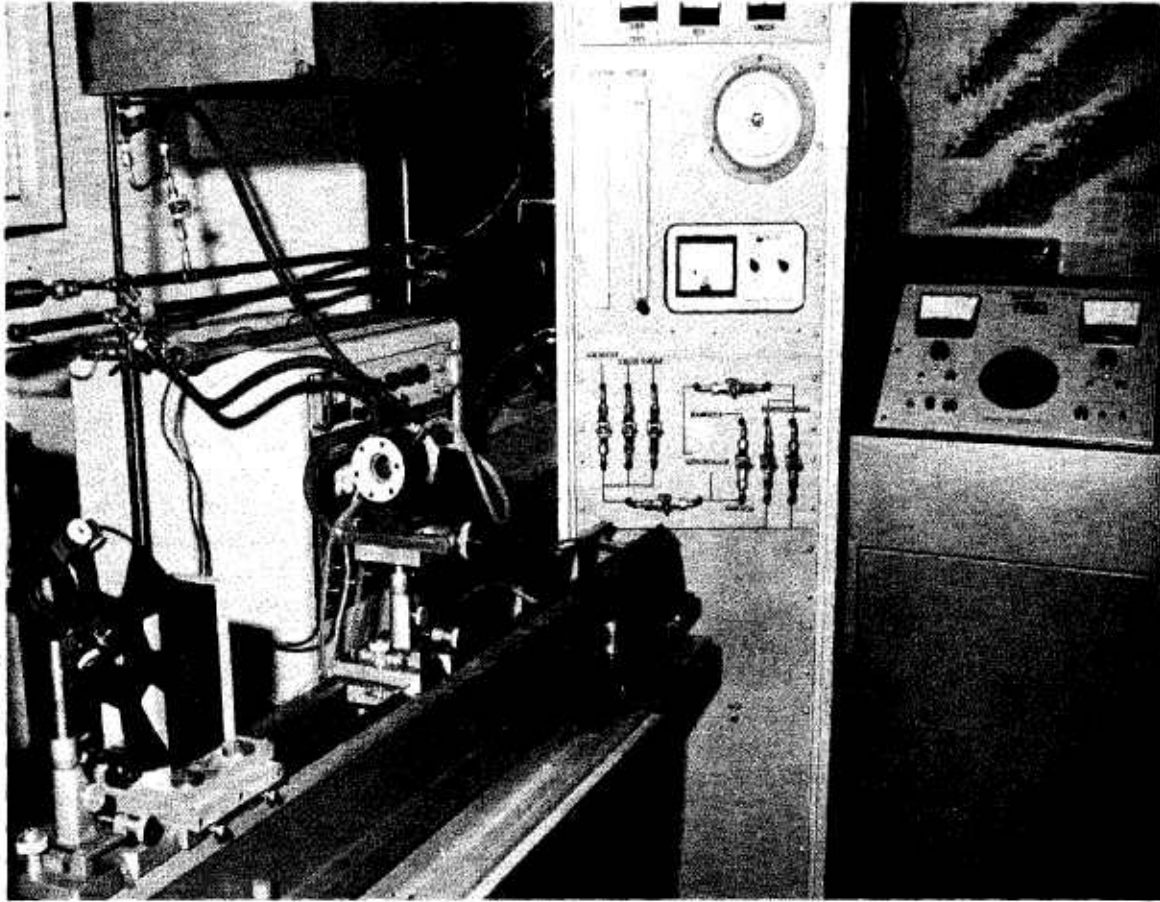


Figure 18—Front view of the same hollow cathode unit from figure 17. From left: Lock-in light chopper, hollow cathode unit, 8-channel simultaneous direct reading system, console with current and gas monitoring meters and vacuum control valves, and dc power supply for the hollow cathode.

Table 7. Stability of the radiation intensity, expressed as photo-multiplier signal average (V), determined for a copper (Cu), silver (Ag), and iron (Fe) hollow cathode operated in helium at 10 Torr and a current of 200, 100, and 200 mA. The measurements were taken at time intervals of 100 s after a preburn of 10 min.

Time, s	V		
	Cu	Ag	Fe
0	8.43	8.13	6.00
100	8.43	8.15	6.05
200	8.33	8.15	5.97
300	8.20	8.17	6.00
400	8.17	8.20	5.95
500	8.23	8.20	5.90
600	8.19	8.20	5.95
700	8.09	8.19	5.87
800	8.07	8.17	5.85
900	8.10	8.27	5.85
Aver	8.22	8.18	5.94
σ	0.13	0.04	0.07
% σ	1.6	0.5	1.2

the cathode was studied recently by Jäger and Blum [156] and by Harrison and associates [72] who used copper, stainless steel, and graphite discs in helium, neon, and argon, at various gas pressures and discharge currents and for various sputtering times. After exposure to the discharge, the metal surface was examined with a scanning electron microscope using magnifications up to 5000 times. Different erosion patterns were observed for helium, neon, and argon, indicating a more pronounced change for argon which produced a surface having a "conical structure 20 to 30 microns high," and a heavier sputtering. When the sputtering time was extended to 15 hours in argon and under a current of 200 mA, the initial cylindrical shape of the hollow cathode was changed progressively into one or several successive bulb-like cavities (also see references 170 and 388). Examination of various parts of the cavity revealed different erosion patterns. It is interesting to compare these results with those obtained by White who studied the potential distribution in an ideal spherical hollow cathode cavity [388].

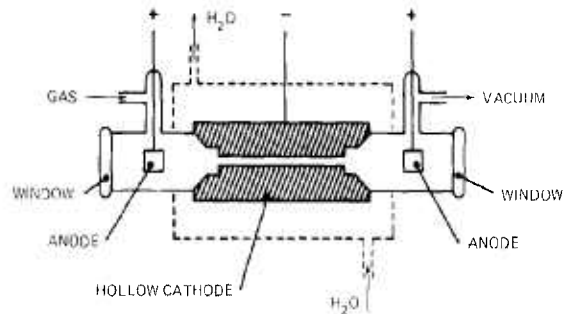


Figure 19—High-current hollow cathode source of Paschen-Ritschel [256].

Jäger and Blum have observed the conical structure mentioned above and have concluded from their study [156], using gold and brass cathodes, that the removal of the sample material depends on crystal structure and orientation, and that there are no signs of surface melting. They also concluded that the preburn time is an essential parameter for obtaining an equilibrated sputtering surface and good analytical results. The preburn time should be established experimentally for every type of sample.

Due to the excitation conditions prevailing in the low pressure glow discharges, the spectra excited in the hollow cathode discharge are characterized by radiations with narrow spectral bandwidth and are practically free of self-absorption under the experimental conditions used to produce and observe the radiations. An example of the spectra excited with the hollow cathode from figures 13 and 14, using a copper and a silver cathode, is illustrated in figure 21 in comparison with the spectra from the same metals excited in a dc arc. The hollow cathode was operated in argon at a pressure of 4 Torr, a current of 500 mA, and an exposure time of 60 s. The dc arc was operated at a current of 8 A in air and the exposure time was 5 s. Both spectra were recorded with a 3 m concave grating Eagle spectrograph, using a 6 step rotating sector, on Eastman Kodak 33 plates. The self-reversal of the resonance lines of copper and silver is clearly visible for the radiations excited in the arc, and the lines are wide and ill-defined. The same lines obtained with the hollow cathode are narrow, well-defined, and free of measurable self-absorption.

The general aspect of the spectra excited in low pressure glow discharges also exhibit a different intensity distribution when compared with those obtained in conventional arcs and sparks. These differences have led E. W. Salpeter to produce, in collaboration with the RSV Company, an atlas of spectra of the noble gases, He, Ne, Ar, Kr, and Xe from λ 500Å to λ 4000Å, and that of the following chemical elements: Fe, Co, Cr, Mo, Nb, Ni,

Ta, Ti, Cu, Be, Ca, Si, Hg, Zn, Pb, Bi, Sb, S, Sn, As, Mg, Cd, Ag, Au, Ir, Pd, Pt, Rh, Ru, Al, Hf, Mn, Se, Te, V, W, and Zr, from λ 1500 Å to λ 4000Å. The excitation source used to produce the spectra was the Grimm glow discharge lamp manufactured by RSV. The atlas was produced in 1971 to 1973 and is divided into five parts. It can be obtained from the Specola Vaticana, Cita di Vaticano, Italy [305].

An atlas of the emission spectrum of uranium excited in a hollow cathode discharge is given in reference 172.

3.2.4 Analytical Applications

The energy, expressed in electron volts (ev), required to excite radiations from the neutral atoms of all known chemical elements, varies from one of the lowest values of 1.426 ev for uranium to the highest value of 21.215 ev for helium. Helium is one of the supporting gases used currently to produce low pressure glow discharges, and is strongly excited in these discharges to emit the radiations from its neutral and the singly ionized atoms (40.811 ev). Hence, the energy available in a hollow cathode discharge is largely sufficient to excite all known chemical species and produce radiations origi-

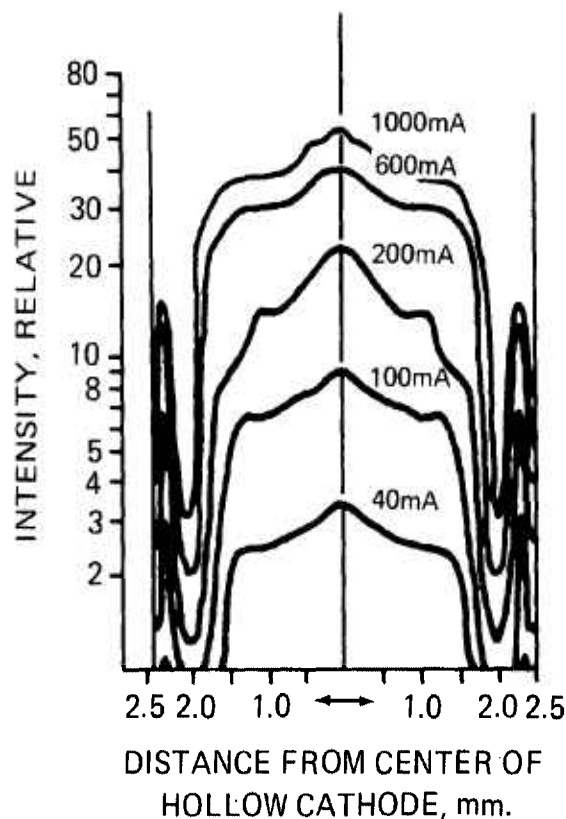


Figure 20—Radiation intensity distribution in a hollow cathode. After Büger and Fink [55].

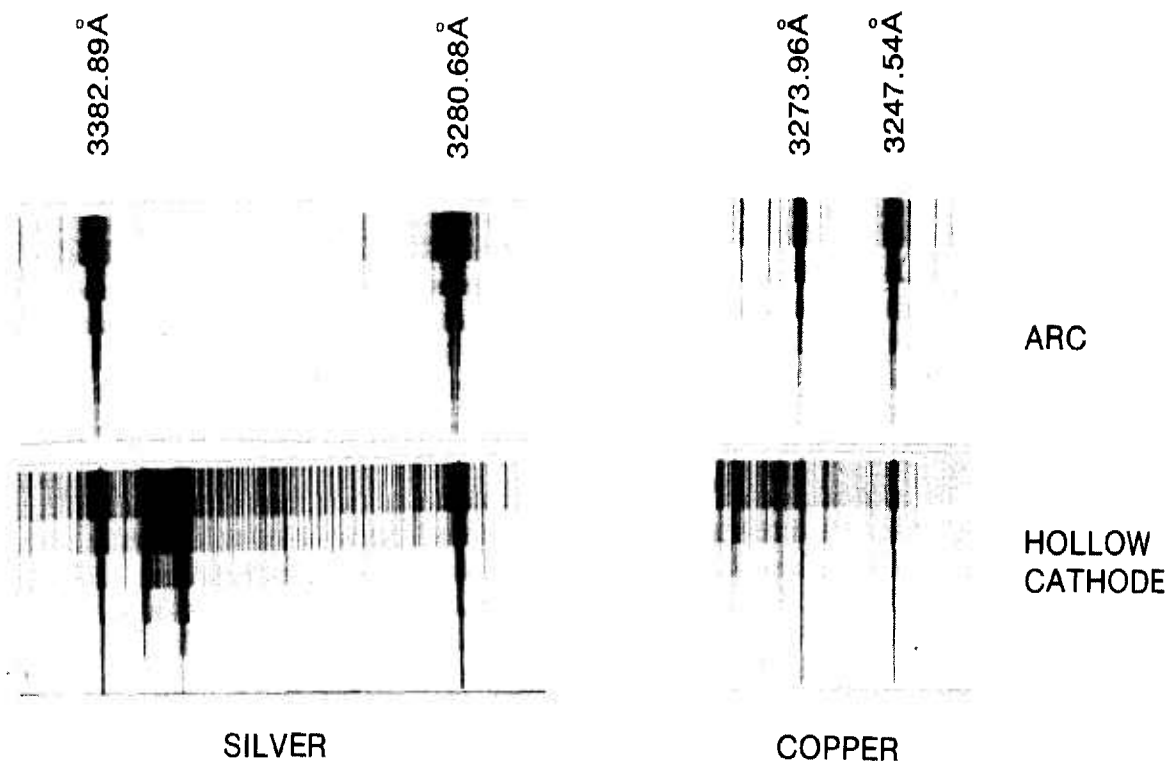


Figure 21--Spectra of Ag and Cu excited in a dc arc and a hollow cathode.

nating from the neutral and ionized atoms as well as from their molecules.

The hollow cathode discharge was initially used by Paschen to produce and study the fundamental characteristics of the spectra of aluminum [254]. Schüler and associates have extended these investigations to the examination of the hyperfine structure of the spectra from rare earths, and of elements available in milligram quantities such as the artificially produced radioactive materials [311, 312, 314, 315, 317, 318, and 322]. The excellent spectral characteristics of the discharge are related to the fact that the experimental parameters of the discharge, such as the nature, pressure, and flow of the sustaining gas, current, discharge tube and analytical sample geometry, can all be controlled. This has caused Schüler to recommend its use as a source of excitation in analytical spectroscopy.

Since then, the hollow cathode discharge has been applied extensively to various analytical problems including isotopic analyses and trace element analyses for practically all chemical species, including the halogens, in various matrices, in solid, liquid, and gaseous form, and for electrically conducting and non-conducting materials.

Some of these applications will be discussed here using data available from the scientific literature. Results obtained in our laboratories will also be presented. Fur-

ther analytical use of the hollow cathode will be found in the numerous publications assembled in Section 5, Collection of References to Works on Low Pressure Glow Discharges, and to its addendum, Subsection 5.1.

The detection sensitivities obtainable with hollow cathodes depend greatly on the experimental parameters; therefore, the data given here should be considered only as an order of magnitude. The detection limits for a number of chemical elements are given in table 8 after Korovin [175]. Further measurements, published by Zilbershtein and associates [398, 399, 400], indicate that an absolute sensitivity of 3 to 5×10^{-10} g can be obtained for Ag, Mn, and Cu; 6×10^{-10} g for Ga and In; 3 to 5×10^{-9} g for Al and Ni; and 6 to 7×10^{-9} g for Mg and Fe, in a silicon matrix, using an excitation current of 800 to 900 mA for a burn of 2 min.

Webb and Webb [381, 382] have used a hollow cathode to determine gases in metals. Their results are given in table 9; the detection limits are given in table 10.

A hollow cathode was used by Birks [29] to determine the halogens, fluorine, chlorine, bromine, and iodine, for which the following detection limits were found: 0.25 μ g, 1 μ g, 5 μ g, and 2.5 μ g respectively.

Thornton has used a high temperature hollow cathode source similar in design to that described by Webb and Webb [381, 382] to analyze steels and high temperature alloys for trace elements [362]. The samples

Table 8. Absolute detection limits for 22 chemical elements, expressed in nanograms, after Korovin [175].

Element	Wavelength, Å	Hollow cathode	Copper spark	dc arc carrier distillation
Al	3092.7	10	10	500
Ag	3280.7	0.03		5
B	2497.7	1	10	1
Be	3130.4	0.03	0.2	10
Cd	2288.0	30	200	7
Co	3453.5	0.3	50	100
Cr	2835.6	1	5	300
Cu	3247.5	0.03		30
Fe	2599.4	3	50	100
Ga	2943.6	0.03	100	
K	7664.9	10	10	200
Li	6707.8	0.1	0.2	10
Mg	2852.1	0.3 (~0.0001)	1(0.1)	50
Mn	2794.8	0.03	2	100
Na	5889.9	0.03	10	50
Ni	3050.8	1	10	200
P	2535.6	30	2000	5000
Pb	2833.1	10	5	100
Sb	2528.5	100	500	1000
Si	2881.6	1	10	300
Sn	2840.0	10		100
Zn	3345.0	3	200	2000

Table 9. Determination of nitrogen, oxygen, and hydrogen in metals [382].

Gas	Steel, NBS Standards				Uranium carbide				Copper		Tungsten			Zirconium	Zircaloy	
	8i	101E	343	125	A	B	C	D	1	2	1	2	3		A	B
Nitrogen, certified	0.018	0.039	0.074	0.002	0.051	0.037	0.037	0.022								
percent found	0.0175	0.034	0.072	0.0055	0.051	0.048	0.035	0.018								
Oxygen, certified					0.09	0.20	0.07	0.026	290	150	5.5	35	22			
percent found					0.12	0.20	0.06	0.024	295	160	7.3	25	20			
Hydrogen, certified														46	140	150
ppm found														46	133	143

were loaded in graphite hollow cathodes and were excited in helium with currents from 0.2 A up to 1.4 A and an exposure time of 5 min. The results obtained in the determination of trace elements at the part-per-million level in various metals and alloys are given in tables 11 and 12, in comparison with accepted values. It is interesting to note that the internal standard used in these determinations was helium at λ 2945.11 Å. Various factors which affect the sensitivity and precision as well as matrix interferences are also discussed by Thornton in the same paper.

The hollow cathode discharge source described in

Table 10. Limit of detection of gases in metals in μg [382].

Material	Oxygen	Nitrogen	Hydrogen
Steel	0.35	0.55	
Uranium Carbide	1.5	2.5	
Tungsten	0.65		
Copper	0.75		
Zirconium			0.05

Table 11. Analysis of various samples with constant trace-element additions[362].

Sample No.	Material		Element concentration found, percent										
			Bi	In	Ga*	Sn	Pb	Tl	Sb	Zn*	Ag	Te*	As*
R3393	Nickel	Accepted value	0.0009	0.0010	0.001	0.006	0.0014	0.0014	0.0013	0.001	0.0011	0.001	0.01
		Hollow cathode	0.0012	0.0010	0.0012	0.006	0.0013	0.0016	0.0018	0.0009	0.0009	0.0012	0.014
R3394	Cobalt	Accepted value	0.0008	0.0011	0.001	0.006	0.0014	0.0004	0.0020	0.001	0.0010	0.001	0.01
		Hollow cathode	0.0010	0.0009	0.0013	0.006	0.0012	0.0006	0.0021	0.0007	0.0018	0.0014	0.012
R3395	Iron	Accepted value	0.0008	0.0008	0.001	0.007	0.0009	0.0008	0.0033	0.001	0.0010	0.001	0.01
		Hollow cathode	0.0010	0.0009	0.0019	0.007	0.0013	0.0010	0.0038	0.0008	0.0011	0.0011	0.013
R3396	Nickel 80%; chromium 20%	Accepted value	0.0009	0.0008	0.001	0.006	0.0014	0.0008	0.0016	0.001	0.0012	0.001	0.01
		Hollow cathode	0.0011	0.0009	0.0009	0.005	0.0012	0.0012	0.0018	0.0010	0.0010	0.0010	0.009
R3400	Nickel 40%; iron 40%; chromium 20%	Accepted value	0.0008	0.0009	0.001	0.006	0.0010	0.0008	0.0025	0.001	0.0012	0.001	0.01
		Hollow cathode	0.0011	0.0011	0.0012	0.006	0.0012	0.0010	0.0028	0.0009	0.0013	0.0012	0.010
R3401	Iron 50%; nickel 30%; cobalt 20%	Accepted value	0.0008	0.0009	0.001	0.007	0.0011	0.0007	0.0019	0.001	0.0011	0.001	0.01
		Hollow cathode	0.0009	0.0010	0.0014	0.006	0.0011	0.0010	0.0026	0.0007	0.0010	0.0011	0.010

*The "accepted" values given are the nominal additions; all other elements were determined by spectrochemical methods.

Table 12. Hollow-cathode discharge analysis of high temperature alloys [362].

Sample	Material		Element concentration found, percent										
			Bi	In	Ga*	Sn	Pb	Tl	Sb	Zn*	Ag	Te*	As*
R3385	Nickel-base alloy†	Accepted value	0.0001	0.0001	0.0002	0.009	0.0001	0.0001	0.0004	0.0001	0.0001	0.0001	0.01
		Hollow cathode	0.0002	0.0002	0.0003	0.011	0.0002	0.0002	0.0006	0.0001	0.0001	0.0002	0.0053
R3386	Nickel-base alloy†	Accepted value	0.0002	0.0002	0.0005	0.0050	0.0002	0.0002	0.0009	0.0002	0.0002	0.0002	0.005
		Hollow cathode	0.0003	0.0003	0.0006	0.0056	0.0003	0.0003	0.0011	0.0002	0.0002	0.0003	0.0051
R3387	Nickel-base alloy†	Accepted value	0.0009	0.0009	0.002	0.0014	0.0009	0.0009	0.0048	0.001	0.0009	0.001	0.001
		Hollow cathode	0.0012	0.0011	0.0026	0.0015	0.0012	0.0011	0.0056	0.0010	0.0009	0.0013	0.0016
R3388	Nickel-base alloy†	Accepted value	0.0005	0.0005	0.001	0.0026	0.0005	0.0004	0.0019	0.0005	0.0005	0.0005	0.002
		Hollow cathode	0.0006	0.0006	0.0012	0.0025	0.0006	0.0006	0.0022	0.0004	0.0004	0.0007	0.0023

*The "accepted" values given are the nominal additions, all other elements were determined by spectrochemical methods.

†Basic composition, percent: nickel 60, chromium 15, cobalt 15, molybdenum 5, titanium 2 1/2, aluminum 2 1/2.

figures 13, 14, 15, and 16 was used in conjunction with a 3 m concave grating Eagle spectrograph and the gas monitoring and power supply, illustrated in figure 18, to determine Mn, Ni, Co, Cr, Al, Si, and Cu in a series of four steel standards certified by NBS as Standard Reference Material (SRM) 1261 to 1264.

Hollow cathodes machined from each sample were submitted to a discharge of 0.8 A in argon at a pressure of 4 Torr, with a preburn of 60 s followed by an ex-

posure of 120 s. The spectra of the four samples were recorded on a photographic plate (Kodak 33) together with an "unknown" sample which was selected from the four standard samples. The plate was processed in the usual manner and was read on a microdensitometer. Calibration curves were then established from the values found for the standard samples using iron as internal standard, and the values measured for the "unknown" sample were obtained by interpolation from these

curves. The resulting data are given in table 13 for the seven elements determined, and indicate that the average uncertainty of the analysis of about 5 percent is within the expected values which can be obtained when photographic plates and procedures are used as photo-detectors and data acquisition means.

Table 13. Determination of seven elements in SRM 1262 using a hollow cathode discharge source.

Element	Certified value, percent	Found, percent
Mn	1.04	0.95
Ni	0.59	0.58
Co	0.30	0.30,
Cr	0.30	0.28
Al	0.095	0.087
Si	0.39	0.39,
Cu	0.50	0.51

The results from the stability tests discussed earlier indicate that an appreciably smaller uncertainty should be obtained when direct measurements are made with multichannel spectrometers using photomultipliers with integration and adequate data acquisition and processing methods available today. Such measurements are being performed at the present time, in association with J. Norris, using a 2 m concave grating multichannel spectrometer and the preliminary results seem to confirm the above statement. However, before the hollow cathode discharge can be used as a routine source of excitation in analytical spectroscopy, further investigations are needed. This author believes that the area requiring detailed studies is that of the sputtering processes occurring in the circumstances characteristic of the hollow cathode discharge mode of operation. The works cited in Section 5 under the title "Sputtering" should provide useful information on this subject.

4. Future Developments

Future developments in the field of low pressure hollow cathode discharges should aim in particular at increasing the sensitivity of analytical measurements. This sensitivity could be increased by increasing the current supplied to the hollow cathode. Currents from 20 A to 80 A were used by Ahsmann and van Benthem [6] in conjunction with a tantalum hollow cathode of special design. The ion temperature measured in the hot plasma beam produced with this source was of 20000 K to 30000 K for argon and 8000 K for neon.

The properties of high intensity hollow cathodes are discussed in a number of papers [2, 20, 28, 29, 151, 184,

239, 266, 279], and to our knowledge this type of discharge was not used in analytical spectroscopy.

Further increase in the current can be achieved by operating the hollow cathode in the pulsed discharge mode using pulses of the order of microseconds with amplitudes of several thousand amperes and with a repetition rate from several discharges to several hundred discharges per second. Such operating conditions have been described by Kielkopf [166] who used pulse amplitudes between 30 and 1500 A and gas pressures of 5 to 50 Torr in helium. This discharge was used to study the spectrum of triply ionized iron, aluminum, and the triply and quadruply ionized rare earths. Ion temperatures of 12000 K and electron temperatures of 15000 K have been measured for such a discharge. A similar high current pulsed discharge hollow cathode is used currently at the National Bureau of Standards by V. Kaufman to study the emission spectra of various chemical elements in the vacuum ultraviolet.

Pulsed hollow cathode discharges are also described in references 24, 60, 64, 65, 66, 67, 69, 122, 135, 141, 158, 163, and 259.

Superdense hollow cathode discharges have been studied also by Klyarfeld and associates [169] and by Abramovich and associates [1]. Further references to works in this field are assembled in Section 5.

The radiation intensity of a hollow cathode can be increased by providing additional energy to the discharge in the form of an electrical or magnetic field. Thus, van Gelder [114] has designed a cylindrical hollow cathode open at both ends as illustrated in figure 22. Additional excitation is provided by a stream of electrons generated by an auxiliary emitting cathode. Supplementary excitation was achieved also by Human and associates [146, 147, 148] to increase the emission from hollow cathodes, using superimposed direct current and a high frequency field. An intensity gain of two orders of magnitude was obtained by Bodretsova and associates [31] when a high frequency current was supplied to a hollow cathode. See also reference 35, 36, and 72.

The effect of magnetic fields on hollow cathode discharges was explored by Popovici and Somesan [271, 337, 338]. The hollow cathode used by these authors is illustrated schematically in figure 23. It consists of two opposite flat circular discs 20 mm in diameter separated by a gap of 4.5 mm and surrounded by a cylindrical anode 10 mm wide and 30 mm in diameter. The magnetic field, from zero to 3000 Oe, was applied as shown.

The intensity of the lead radiation at λ 4057.83Å was measured, under a constant current intensity (15 mA/cm²), in Ne, Ar, Kr, Xe, and He, as a function of the magnetic field, and the results from figure 24 show that an increase by a factor of 10 was observed.

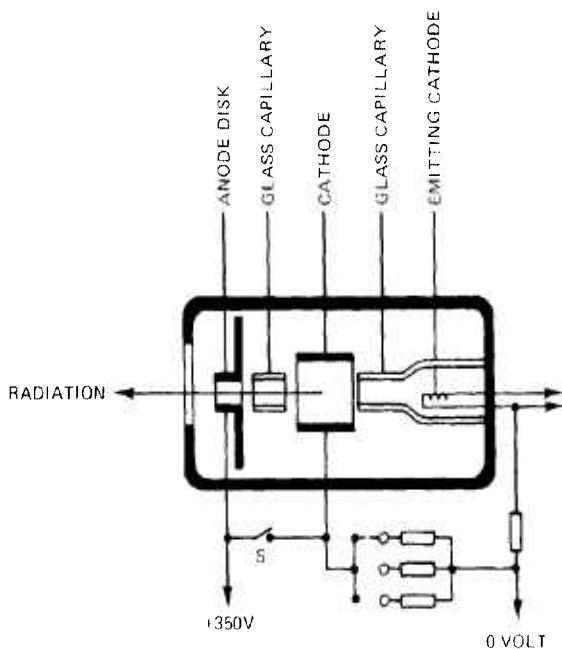


Figure 22 – High intensity hollow cathode source after van Gelder [114]

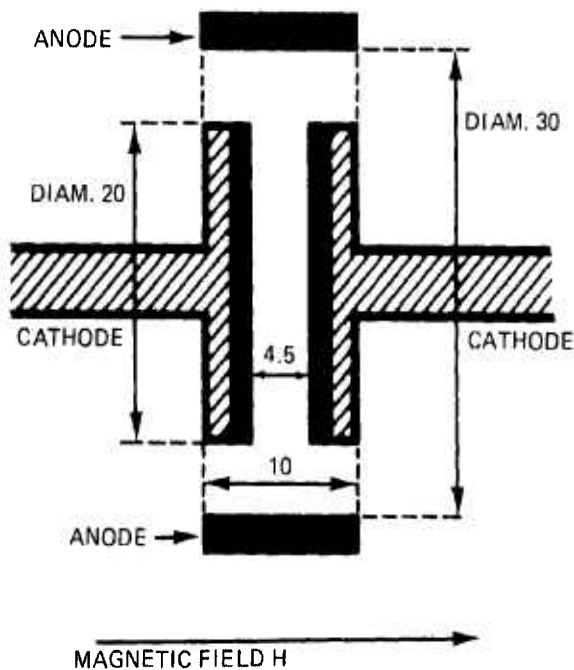


Figure 23 – Description of a low pressure hollow cathode discharge with superimposed magnetic field. After Popovici and Somesan [271, 337, 338]. Dimensions in mm.

This enhancement is significant and, if proved valid for other chemical species, should increase the use of hollow cathode discharges to the analysis of trace elements at the sub-nanogram level.

Further investigations on the effects of magnetic fields on the plasma generated in hollow cathode discharges are discussed in the Addendum to Section 5, Paragraph 15.

The author gratefully acknowledges the permission received from the various scientific journals and publications to use several illustrations and tables reproduced in this work. The sources from which the material was taken are indicated in the text. Further acknowledgment is given to the Center for Analytical Chemistry Text Editing Facility for typing the manuscript.

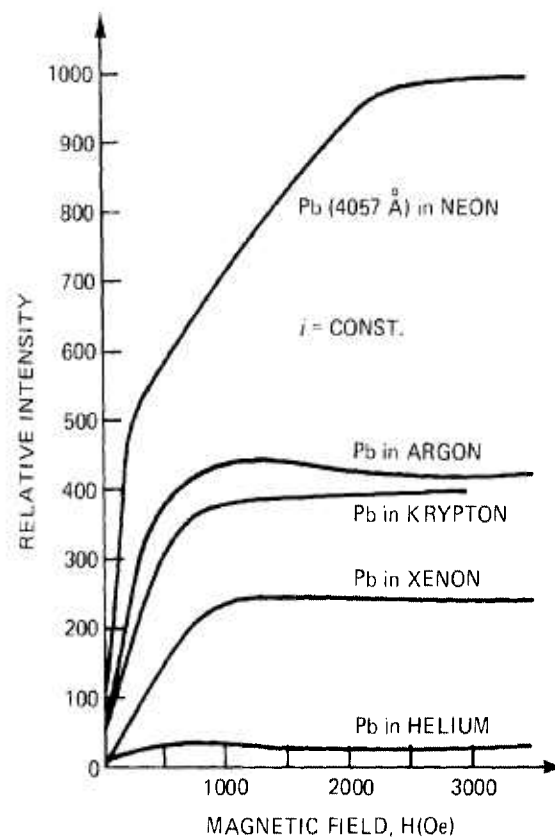


Figure 24 – Intensity of the lead radiation at λ 4057.83 Å excited, under constant current, in a magnetic field and in different noble gases, using the hollow cathode source from figure 23 [271, 337, 338].

5. Collection of References to Works on Low Pressure Glow Discharges

The references in the bibliography that follows were selected with an eye toward analytical measurements.

Preceding the alphabetically arranged references is a Brief Subject Index in which the cited works are grouped under 20 subject categories. This index is intended to assist the reader in preliminarily selecting papers in his field of interest. The reader is also furnished a List of Chemical Elements.

The 400 works of the bibliography cover, roughly, the period from 1916 to 1975. In addition to this listing, a second listing offers another 290 citations. Essentially, this addendum covers the years 1975 to 1983.

In all, the base and addendum bibliographies contain 690 citations.

5.1 Brief Subject Index

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2. **Atlas of Glow Discharge Spectra:** 305.
3. **Wavelength Standard:** 71, 341.
4. **Bibliography:** 159, 207, 208.
5. **Sputtering:** 20, 41, 72, 136, 156, 161, 232, 344, 383, 387.
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11. **Excitation of Chemical Elements:** 5, 10, 25, 26, 27, 28, 32, 40, 41, 53, 67, 69, 97, 98, 117, 118, 124, 125, 173, 175, 190, 193, 194, 203, 218, 262, 263, 264, 266.
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16. **Pulsed Hollow Cathodes:** 18, 76, 77, 78, 166, 167, 195, 275, 287, 329.
17. **Hollow Cathode in a Magnetic Field:** 271, 337, 338.
18. **Hollow Cathode-RF Discharge Association:** 31, 146, 147, 148.
19. **High Pressure Glow Discharge:** 95, 111, 361.
20. **Lasers and Hollow Cathodes:** 4, 10.

5.2 Listing of Chemical Elements

References	Element
[5]	Se, As, P, S, I
[9]	Pu
[11]	nuclear fuels
[21]	alloys
[22]	S, Cl, F, Br, I
[25]	S, Cl, F
[26]	S, Cl, F
[27]	deuterium
[29]	F, Cl, Br, I
[48]	Li
[52]	F

[56] He, Ar, N, CO₂
 [57] I
 [66] Cl, F
 [73] B
 [86] Cu, Fe, Mg, Mn, Si, Ti, Zn
 [93] Si, Ca, Mg, Mn, Fe, Al, Ti, Na, K, P, S, C
 [97] Mg, Zn, V, Cr
 [98] F, Cl, As
 [116] F, Cl, B, Li, Na, K, Rb, Cs
 [123] Mo, Ru, Rh, Pd
 [129] Be, Si, Fe, Mg, Cr, Mo, Al, Pb
 [140] Au
 [142] Pb, Cu, B, Sn
 [151,153] Zn, Cu, Mg, Mn, Ca, Sr, Cr, Fe, Co, Ni, Al, Ag, Ga, In, Tl
 [154,155,157] Au
 [163] F, Cl
 [171] Ar, N
 [176] Cl, F
 [194] F, Cl, S
 [197] As
 [198] I
 [203] Na, K, Tl, Hg, Fe, Co, Ni
 [205] Cd, Mn, Sb, Fe, Mg, Pb, Sn, Ni, Bi, Al, Cu
 [206] Li
 [210] F, Cl
 [219,220,221] I
 [228] N
 [234] Ag, Al, As, B, Be, Bi, Cd, Cr, Cu, In, Mg, Mn, Na, Pb, Sb, Sn, Tl, Zn, Ni, Si
 [238] Ag, Cu, Pb, Bi, Cd
 [239] Ag, Bi, Cu, As, Ni
 [259] Cu, Ag, Mn, Mg, Pb, Bi, Ga, Zn, Cr, Sn, Ni, Sb
 [260] Al, Fe, Ca, Co, Si, Mg, Mn, Cu, Na, Ni, Ag, Cr
 [262] Cu, In, Sn, Al, Ga, Mg, Ni, Cr, Pb, Mn
 [266] V, Mo, Ta, Zr, Ti
 [267] Al, Ge, Fe, Bi, Au, In, Co, Mn, Cu, As, Ni, Pb, Ag, Sn, Tl, Cr, Zn, Ga, Sb
 [283] P
 [289,290,291] O
 [295] Cd, Zn
 [296] Ge, Si
 [298] As, Sb

[299] Se, Zn
 [300] S, Cd
 [301] Bi, Al, B
 [303] In, Ge, Cd, Tl, As, Sb, Pb, Sn, Fe, Cu, Ni, Co, Bi, Ag
 [332] Bi, Pb, Sn, Cd, Zn, Sb, Cu, Mg, Mn, Fe, Al, Cr, Ni
 [335] N, O
 [362] Bi, In, Ga, Sn, Pb, Tl, Sb, Zn, Ag, Te, As
 [370] N, O
 [381] O
 [382] O, H, N
 [393] N, O, H
 [398] Ag, Mn, Cu, Ga, In, Al, Ni, Mg, Fe
 [399,400] Al, Ga, Fe, Ag, Mn, Cu

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5. **Excitation Phenomena in Hollow Cathodes:** 9, 14, 16, 17, 19, 25, 29, 39, 40, 41, 45, 48, 49, 52, 53, 54, 55, 56, 57, 69, 71, 74, 78, 81, 85, 87, 88, 89, 90, 92, 94, 95, 96, 103, 105, 109, 110, 112, 113, 116, 119, 124, 125, 127, 140, 149, 150, 153, 155, 157, 164, 166, 168, 170, 178, 182, 183, 189, 194, 200, 207, 210, 211, 212, 213, 221, 222, 229, 230, 235, 237, 238, 239, 242, 244, 246, 247, 249, 250, 251, 254, 261, 262, 265, 271, 272, 273, 275, 280, 281, 282, 283, 285, 287, 288, 289, 290.
6. **Excitation of Molecular Species:** 96, 106, 216, 218, 259, 263, 268, 270.
7. **Isotopic Analysis and Fine Structure:** 3, 15, 22, 58, 79, 80, 225, 233, 234, 277.
8. **Instrumental Characteristics:** 4, 6, 7, 9, 18, 21, 32, 33, 37, 39, 40, 41, 43, 47, 68, 84, 98, 106, 107, 108, 120, 121, 131, 138, 145, 188, 223, 232, 255, 260, 264, 269, 273, 286.
9. **Excitation of Chemical Elements:** 1, 3, 6, 26, 34, 64, 65, 73, 86, 102, 118, 166, 167, 170, 172, 173, 252.
10. **Analytical Applications:** 33, 34, 38, 42, 44, 45, 50, 104, 120, 126, 128, 132, 133, 134, 136, 139, 147, 148, 152, 154, 174, 175, 176, 179, 189, 190, 191, 192, 193, 195, 196, 197, 198, 199, 201, 202, 203, 204, 205, 206, 208, 209, 214, 215, 223, 224, 228, 234, 245, 270, 274, 276, 288.
11. **Planar Cathode (Grimm Discharge):** 4, 37, 43, 44, 223.
12. **Hollow Cathodes in Atomic Absorption:** 32, 51, 62, 70, 77, 82, 83, 99, 156, 181, 227, 231, 240.
13. **High Intensity Hollow Cathodes:** 2, 20, 28, 29, 151, 184, 239, 245, 266, 279.
14. **Pulsed Hollow Cathodes:** 24, 55, 60, 63, 64, 65, 66, 67, 69, 122, 135, 141, 158, 163, 259.
15. **Hollow Cathodes in a Magnetic Field:** 11, 13, 31, 111, 130, 137, 142, 143, 146, 185, 187, 191, 192, 193, 196, 197, 199, 200, 201, 205, 206, 207, 224, 229, 230, 242, 248, 278, 279, 284.
16. **Hollow Cathode—RF Discharge Association:** 35, 36, 72.
17. **Microhollow Cathode:** 50.

5.4 Addendum—Preface

The 290 references assembled in the addendum for the most part cover the period from 1975 to 1983. They are in alphabetical order and are numbered in italic type to differentiate them from the 400 references of the base bibliography. Several references dating before 1975 escaped the base bibliography and are included in the addendum. The addendum, like the base listing, is preceded by a Brief Subject Index and a Listing of Chemical elements.

5.5 Brief Subject Index (Add.)

1. **Glow Discharges, General Characteristics, and Reviews:** 10, 177, 180, 226, 241, 252.
2. **Atlas of Glow Discharge Spectra:** 172.
3. **Sputtering:** 53, 129, 230.
4. **Fundamental Characteristics:** 5, 12, 23, 27, 30, 46, 59,

5.6 Listing of Chemical Elements (Add.)

Reference	Element
[1]	Al
[3]	H
[6]	Bi, In
[16]	Li
[17]	I

[19]	Ca	[179]	Graphite
[25]	Rare Earths	[189]	Cd, Na
[26]	Rare Earths	[190]	W, S, P
[34]	Cu, Al	[191]	Alkali Metals
[38]	Ga	[192]	Alkali Earth Elements
[49]	He, Cd	[194]	In, Ga
[51]	As, Se	[198]	W, S, P, Zn, Cd
[56]	Ba, Sr	[199]	Cr
[58]	H	[201]	Cd
[64]	Cu, Fe	[202]	Si, Ge
[65]	Zn	[203]	S, Cd, Se
[66]	Al	[204]	Si
[67]	Al	[205]	Rare Earths
[69]	Cu	[206]	Rare Earths
[70]	Mo, Fe, Si	[208]	B
[73]	Sr	[209]	Ge
[75]	Ar	[214]	Si
[76]	Ar	[216]	Cr, Ti
[77]	As	[218]	Pt
[79]	Li	[221]	He, Cu, Ag
[81]	U	[222]	Cu
[87]	Cu	[223]	Cu
[90]	Cs	[224]	Si
[102]	Hydride	[228]	Ga
[108]	He	[233]	U-235/U-238
[109]	He	[234]	U-235
[113]	N, Co	[236]	Ne, Zn
[118]	Mg	[237]	Cu, Ag
[119]	Mg	[239]	Cu
[122]	Sb, Bi, Ag, Cu	[244]	Au
[123]	He	[245]	Steels, Nickel-base and Ferroall
[128]	Te	[259]	Xe F, Xe Cl
[133]	Ni, Co	[262]	Sr, Ba
[135]	He	[263]	Xe Cl
[136]	Au	[268]	Cl, Xe Cl
[137]	Ti	[270]	Al, Cu, Ni hydrides and deuterides
[139]	Co, N	[271]	Cu
[140]	He	[273]	Am, Cm
[142]	Alkaline Elements, Mg	[274]	Am, Cm
[143]	Group IV Elements	[277]	H
[147]	Ge, Se, Cd	[283]	Cu
[149]	Cd	[285]	Cu
[152]	F, U		
[164]	Ba, Cd, Cu		
[166]	Dy		
[167]	Zn		
[172]	U		
[173]	Cd		
[174]	Al-ammonium Alums		
[175]	Al		
[176]	Ti		
[178]	U		

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