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Measurement of number density of lead and thallium see-through hollow cathode discharges with a high resolution Fabry–Pérot spectrometer and by conventional atomic absorption

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ABSTRACT A see-through hollow cathode lamp, or galvatron, is investigated. A novel method is presented for the measurement of an atomic absorption profile using a quasi-continuum source created by the combination of two line sources and a high-resolution Fabry–Pérot interferometer coupled to a spectrometer. Number densities are calculated from the resulting absorption profiles by the peak absorption coefficient relationship and compare well with results obtained from high-resolution emission measurements. Number densities are also determined for the lead $^3P_1$ metastable state and thallium $^2P_{1/2}{}^{\mathrm{o}}$ ground state by conventional atomic absorption. A hollow cathode lamp is used as an emission source and is set at a relatively low current to approximate as a line source relative to the galvatron. Due to the relative line widths of the source and absorber, only the lead metastable state results compare to results obtained by saturated fluorescence.

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1 Introduction

The application of atomic line filters to the detection of low levels of light has been investigated by several authors [1–5]. Their high background rejection and high spectral resolution make them ideal filters for applications in environments of high solar background. Many atom reservoirs have been used as atomic line filters, such as heated sealed cells [6], flames [7], and ICP [8] plasmas. Hollow cathode lamps are also capable of producing an atomic vapor which can be easily controlled by the applied current. They also offer a low quenching environment, which results in quantum efficiencies limited only by the transition probabilities. A see-through hollow cathode lamp, or galvatron (Hamamatsu), is now commercially available. The galvatron and hollow cathode lamp both operate by the hollow cathode effect; however, the cathode of a galvatron is oriented in a T-shape and the cathode is open on both ends. This allows optical access for direct absorption measurements on a commercially available hollow cathode discharge. Since few spectroscopic data have been collected on these discharges, absorption profiles need to be measured and number densities determined at various currents in order to evaluate them as potential atomic line filters.

In order to evaluate the resolving power of this atomic line filter, high resolution absorption measurements are needed. High resolution absorption measurements on atomic vapors are typically done with narrow band diode lasers capable of scanning across the absorption profile. However, most atomic transitions of interest are in the UV region not easily accessible by diode lasers. Another common approach is the use of a continuum source with a high resolution monochromator; however, typical conventional grating and echelle monochromators [9, 10] have shown to give inadequate resolution and throughput, necessary for these measurements.

Fabry–Pérot interferometers have also been used in conjunction with continuum sources. In this method, radiation from a continuum source is sent through an absorbing medium and then to a Fabry–Pérot interferometer. The interferometer output is sent to a monochromator for cross dispersion and detection. As the Fabry–Pérot is tuned, or scanned, the result will be a line source capable of scanning across the absorption profile of interest; however, several problems are associated with this approach. Kirkbright et al. [11] applied this method to the measurement of calcium in a flame using a monochromator with a spectral bandpass of 2 nm. In those measurements, the monochromator had such a poor resolution absorption measurements were only possible with an extremely high concentration of the salt solution. This was due to a large and unknown number of non-absorbing channels being detected simultaneously with the resonant absorbing channels, which resulted in a very weak, or diluted, absorption signal and was only overcome by averaging multiple data along with a very high absorber concentration. Wagenaar et al. [12] also applied this method to the measurement of calcium, but with a high resolution monochromator capable of resolving one free spectral range. This approach also required a high concentration of calcium in order to obtain absorption profiles. The profiles that were obtained yielded a poorly defined baseline due to the monochromator bandpass which resulted in a loss in the wings of the profile. This makes it difficult for accurate absorption coefficients to be assigned. Another problem encountered was the limited temperature stability of the monochromator, which caused the selected bandpass of the monochromator to slowly drift away from the absorption profile.

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In 1970, Bazhov and Zherebenko [13] introduced a method using two electrodeless discharge lamps to create a quasi-continuum source over the absorption profile of interest. This was achieved by setting one emission source to be self-reversed and another emission source to be self-absorbed. These two emission sources superimposed onto one another resulted in a broadened, flat topped emission profile centered over the absorption profile as shown in Fig. 1. This approach retains the resolving power of a Fabry–Pérot interferometer and greatly relaxes the restrictions of the monochromator, only needing the resolution capable of isolating the broad quasi-continuum from neighboring spectral lines. This was easily achieved with commercially available monochromators. Since line sources are used, the emission profile will be centered directly over the absorption profile. This approach provides excellent wavelength stability without the need to worry about wavelength fluctuations and drift. There was no need for unrealistically high absorber concentrations and the thermal stability of the monochromator was unimportant. The relaxation of the slit width restrictions of the monochromator greatly improved the throughput of the system and allowed for a much higher signal to noise ratio. In addition, the signal to noise ratio will be further enhanced by the use of an electrodeless discharge lamp and hollow cathode lamp rather than a Xe-arc lamp. Xe-arc lamps have significant low frequency flicker noise due to arc wander, and since the noise is generated from the source, the use of a lock-in will have no effect. This is important when measuring the absorption profile of an atomic vapor with a relatively low number density. This approach allowed the determination of the number density measurements through the measurement of the peak absorption coefficient.

This approach has an advantage over diode lasers since it has a much larger scanning spectral range, limited only by the reflection coefficient of the mirrors. This allows the investigation of many atomic transitions within the spectral range of the mirrors, whereas diode lasers have a very narrow scanning range and typically are only useful for a single atomic transition. In addition, for some absorption profiles, such as in high temperature, high pressure plasmas, diode lasers may not be able to scan over the entire absorption profile which can decrease the accuracy of their measurements. The present method described above is introduced as an alternative method for obtaining number densities and absorption profiles when a diode laser system is not available in the lab.

Conventional absorption spectroscopy can also be applied for number density measurements. It is well known that as the current applied to a hollow cathode lamp (HCL) is increased, the width of the emission profile is also increased. If an HCL is held fixed at a low current relative to that applied to the galvatron, then the emission from the HCL should be roughly approximated as a line source. In the line source approximation, the source is assumed to have an infinitely narrow width, or delta function, so the frequency dependence on the absorption coefficient is removed. It is also assumed the line source is centered on the transition, $k(\nu) = k_0$. Since both lamps are low pressure discharges, the profiles are limited by Doppler broadening. Therefore, there will be no shifting in the peaks of the emission or absorption profiles. Thus, it can be assumed that the emission profile from the hollow cathode lamp will be centered on the absorption profile of the galvatron.

If the line source approximation holds true, the absorption measurements can be directly related to the number density of the atomic vapor created, if the path length, absorption profile width, and oscillator strength are known [14, 15]. In this work, the 276.79 nm thallium transition will be used to determine the ground state number density of the thallium galvatron. For lead, the 405.8 nm transition will be used to determine the number density of the metastable state.

2 Experimental

2.1 *Combining two line emission sources*

2.1.1 Optics. The 535.046 nm light from a thallium EDL (Perkin Elmer) and thallium hollow cathode lamp (Jarrell Ash) is collimated and combined with a 50/50 beam splitter as shown in Fig. 2. The light is focused through the bore of the see-through hollow cathode discharge followed by a lens to re-collimate the light. Due to the dimensions of the optical table, the light is reflected 90° with a mirror and sent to the interferometer.

2.1.2 The scanning Fabry–Pérot interferometer. The interferometer (Coherent Optics Inc. 370) is composed of a static mirror and a translating mirror. A high voltage ramp generator (EXFO RG-91) applies a voltage from 0–1000 V in a saw tooth waveform. As the voltage is ramped, the piezoelectric crystal linearly translates the mirror and therefore, scans across the profile. The resulting interference pattern is collected and focused onto an aperture placed in front of the monochromator (Acton 500i). The aperture has a 300 μm diameter and allows the passing of only the central portion of the interference pattern. Therefore, as the mirror translates, the circular interference pattern collapses onto the aperture and the transmitted light is filtered by the monochromator tuned to the 535.046 nm transition. The monochromator used had a geometric spectral bandpass of 0.85 nm which was more

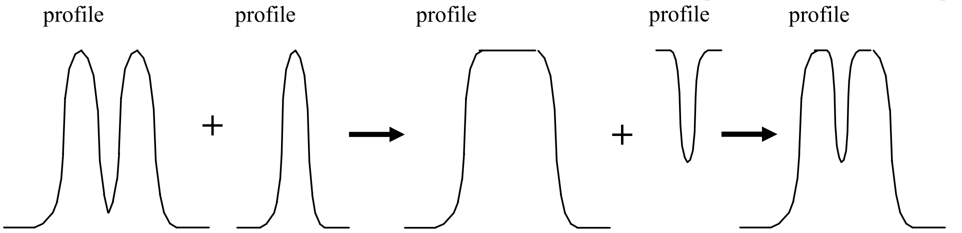

FIGURE 1 Illustration of the production of a quasi-continuum for the measurement of high resolution absorption measurements

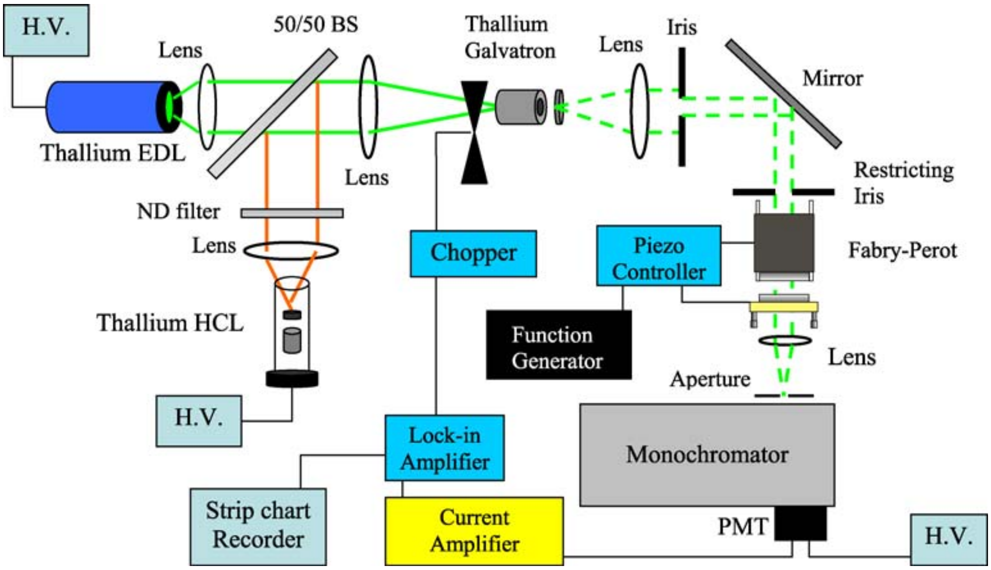

FIGURE 2 Experimental setup used for high resolution absorption profile measurements

than sufficient to suppress detection of neighboring spectral lines. The scanning Fabry–Pérot was found to have an experimentally determined finesse of 130. A mirror separation of 6.50 mm resulted in a free spectral range of 22.0 pm. This arrangement yielded an instrument FWHM of 0.17 pm, which should be more than capable of resolving the absorption profiles. Alignment of the interferometer was accomplished with a He-Ne laser.

2.1.3 Detection. Since the see-through hollow cathode discharge is also a thallium emission source, it will produce 535.0 nm emission. For this reason, a mechanical chopper is used to modulate the combined line sources. The resulting signal from the PMT is then amplified with a current amplifier and sent to a lock-in amplifier for demodulation. Since the shortest time constant of the lock-in is 1 ms, the scanning of the interferometer must be considerably slower, otherwise a distortion of the true signal will result. For this reason, a function generator is used to control the ramp generator. A Tektronix function generator capable of producing a triangle waveform with a 2×10^{-4} Hz frequency is used. Scanning the profile at this frequency allowed the use of a 1 s time constant on the lock-in amplifier. This resulted in the best signal to noise ratio without distortion of the recorded profile.

2.2 *Conventional atomic absorption*

Absorption measurements are carried out using neon filled, thallium (Jarrell Ash) and lead (Fisher) hollow

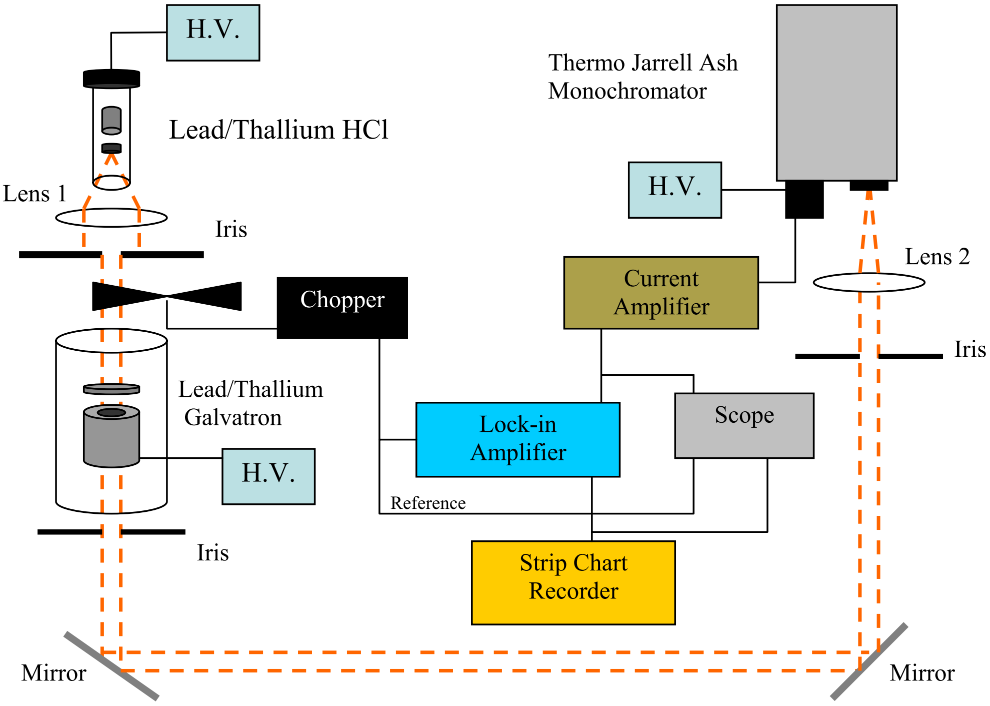

FIGURE 3 Experimental arrangement for absorption measurements. Lens 1 and lens 2 are both fused silica with focal lengths of 10 cm. Both irises had a diameter of 2 mm. Not drawn to scale

cathode lamps both set at a fixed current of 2.0 mA and modulated with a mechanical chopper (EG&G 5207) at a frequency of 625 Hz. Neon filled thallium and lead galvatrons (Hamamatsu) are used as the see-through hollow cathode discharges. All lenses used are fused silica with one inch diameters. A monochromator (Thermo Jarrell Ash) is used with a 500 mm focal length, 1200 grooves mm^{-1} grating, and slit widths of 0.5 mm. The detector is a photomultiplier tube (R928 Hamamatsu) with -1000 V applied from a high voltage power supply (Bertan model 342A). The anode current is sent to a low-noise current preamplifier (Stanford Research model SR570) with a gain of 200 nA V^{-1} and a 6 dB high pass filter at 10 kHz. The amplified signal is sent to a lock-in amplifier for phase sensitive detection with a gain of 50 mV V^{-1}, a time constant of 1 s, and a phase of 153.4°. The demodulated signal is monitored on a 100 MHz oscilloscope (Tektronix TDS 3012B) and recorded with a strip chart recorder.

The experimental design used for the collection of absorption data is shown in Fig. 3. Since the galvatron is also a spectral line emission source, reduction of background emission is required. This is accomplished by using a lens placed 10 cm from the cathode face of the HCL to collimate the emission. The emission was spatially filtered with an iris having a diameter of 2 mm. The collection lens used to focus the emission onto the slits of the monochromator is placed approximately one meter from the galvatron. This is done in order to remove as much of the emission from the galvatron as possible, since the galvatron emission is diverging and the HCL emission was collimated. The galvatron emission is further reduced with a 2 mm iris placed before the collection lens. The monochromator is tuned to the 276.79 nm emission line of thallium and 405.8 nm emission line of lead.

It is important to note that by placing the galvatron with the anode facing the HCL, rather than facing the detector (seen in Fig. 3) less emission from the galvatron is observed. Orientation in this manner aided with the reduction of background emission noise.

3 Results and discussion

3.1 *High resolution absorption measurements*

The combination of the two line sources results in a quasi-continuum source for the thallium 535.046 nm transition as shown in Fig. 4. The profiles of the EDL and HCL are also measured individually.

Figure 5 show the results of spectral scans at four currents in the galvatron. The results obtained are similar to those obtained by Wagenaar et al. [12], with a poorly defined baseline. An improved baseline could be obtained by broadening each profile; however, the width of the profile is already slightly too broad for the selected free spectral range of the interferometer. Increasing the emission profile would result in severe order overlap which would distort the true profile. The free spectral range could be adjusted to accommodate for order overlap but would also result in a broader instrument FWHM, which would also cause a distortion of the true profile.

The measurements in Fig. 5 are the result of absorption from the $F_2 \rightarrow F_1$ transition for the two isotopes, ^{203}Tl and ^{205}Tl. The number density of each isotope can be calculated using the peak absorption coefficient. This method assumes

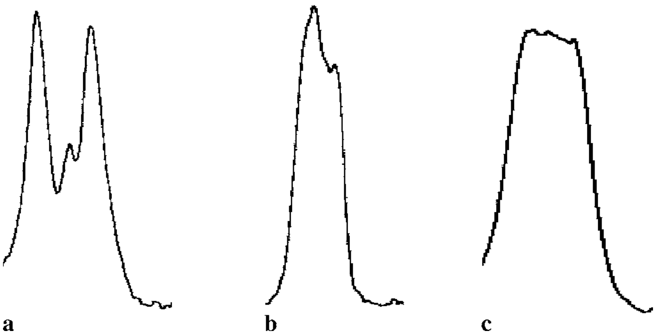

FIGURE 4 (**a**) Scan of the EDL profile self-reversed. (**b**) Scan of the HCL profile self-absorbed. (**c**) Resulting profile of the combined profiles yielding a quasi-continuum source over the absorption profile

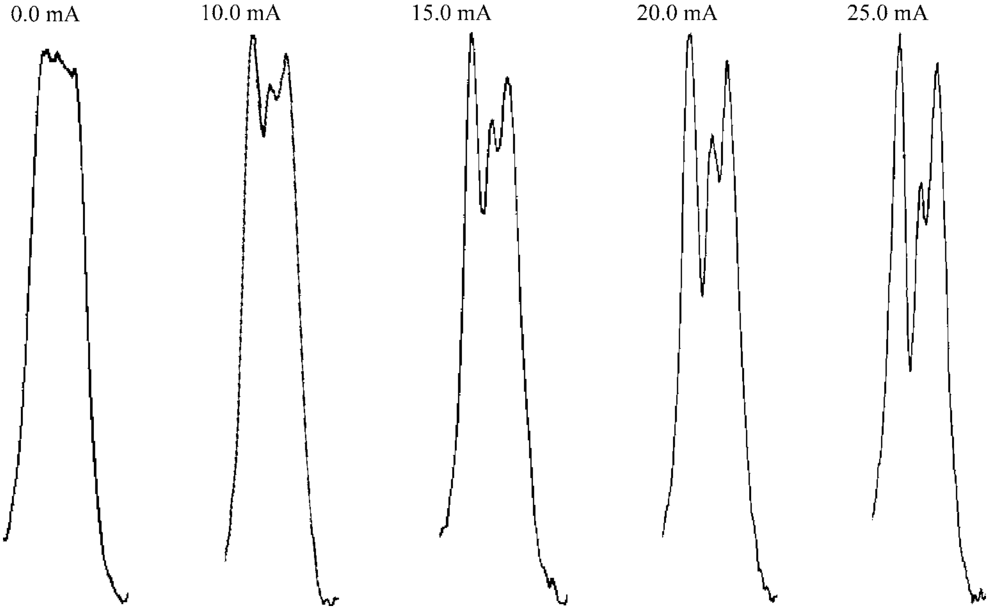

FIGURE 5 Resulting absorption profiles of the thallium $6\,^2P_{3/2}{}^{\circ}$ metastable state of thallium due to various currents applied to the see-through hollow cathode discharge. The blue wing on the quasi-continuum profile is cut off due to a small amount of order overlap from an adjacent order. This order overlap did not cause significant distortion of the observed absorption profiles

that the spectral line is fully resolved which is a good approximation considering an instrument FWHM of 0.17 pm was used. Calculation of the number density by this method can be done using the following equations [14],

$$n = \frac{k_0}{(0.4697)\left(\frac{2}{\Delta\nu_D}\right)(2.65\times 10^{-2})(f_{12})} \tag{1a}$$

$$\Delta\nu_D = 7.16\times 10^{-7}\nu_0\sqrt{\frac{T}{M}} \tag{1b}$$

$$k_0 = \frac{-\ln\left(\frac{I}{I_0}\right)}{l}\,, \tag{1c}$$

where I_0 is the intensity of incident light and I is the intensity after absorption. The absorption path length, l, is assumed to be 2 cm, the length of the cathode bore. The FWHM of the Doppler broadened absorption profile, $\Delta\nu_D$ (Hz), can be calculated from (1b), where ν_0 is the central frequency of the transition (Hz), T is the Doppler temperature (K), and M is the atomic mass (g/mole). The Doppler temperature values used for this calculation are obtained by high resolution emission measurements [16]. Since the hyperfine structure is measured, the oscillator strengths, f_{ij}, for each hyperfine component must be determined. This is accomplished by the following relationship and is calculated as follows [17],

$$\frac{I_{kl}}{I_{mn}} = \frac{f_{kl}}{f_{mn}}\cdot\frac{2F_k+1}{2F_m+1} \tag{2}$$

$$f_{11}+f_{21}+f_{10} = f_{\text{total}} \tag{3}$$

$$f_{\text{total}} = 0.15$$

$$\begin{aligned}
\frac{I_{11}}{I_{21}} &= \frac{f_{11}}{f_{21}}\frac{[(2\times 1)+1]}{[(2\times 2)+1]}\,, &\quad \frac{f_{11}}{f_{21}} &= \left(\frac{0.20}{1}\right)\left(\frac{5}{4}\right)\\
\frac{I_{11}}{I_{10}} &= \frac{f_{11}}{f_{10}}\frac{[(2\times 1)+1]}{[(2\times 1)+1]}\,, &\quad \frac{f_{11}}{f_{10}} &= \left(\frac{0.20}{0.40}\right)\left(\frac{4}{4}\right)\\
\frac{I_{21}}{I_{10}} &= \frac{f_{21}}{f_{10}}\frac{[(2\times 2)+1]}{[(2\times 1)+1]}\,, &\quad \frac{f_{21}}{f_{10}} &= \left(\frac{1}{0.40}\right)\left(\frac{5}{4}\right)\\
f_{11} &= 0.0214\,,\quad f_{21} = 0.0955\,,\quad f_{10} = 0.0331\,,
\end{aligned}$$

where I_{11} (0.20), I_{21} (1), and I_{10} (0.40) are the relative intensities of the $F_1 \to F_1$, $F_2 \to F_1$, and $F_1 \to F_0$ hyperfine transitions for each thallium isotope [18]. If all calculated values are combined and substituted into (1a), the number densities for each isotope are determined.

$$n_{205} + n_{203} = n_{\text{total}}\,. \tag{4}$$

From (4), the total number density of the 6 $^2P_{3/2}{}^{\text{o}}$ metastable state can be calculated by high resolution absorption measurements. The results obtained from the calculation of the number density are in agreement with values obtained by high resolution emission measurements [16] shown in Fig. 6.

It is also interesting to note that from the number densities of each isotope, the relative abundance of each isotope present could be determined. Based on the calculated number densities, the relative abundance of each isotope was found to be 31.2% for ^{203}Tl and 68.8% for ^{205}Tl. This is in fair agreement to that of the natural abundance of 29.5% and 70.5% for ^{203}Tl and ^{205}Tl respectively.

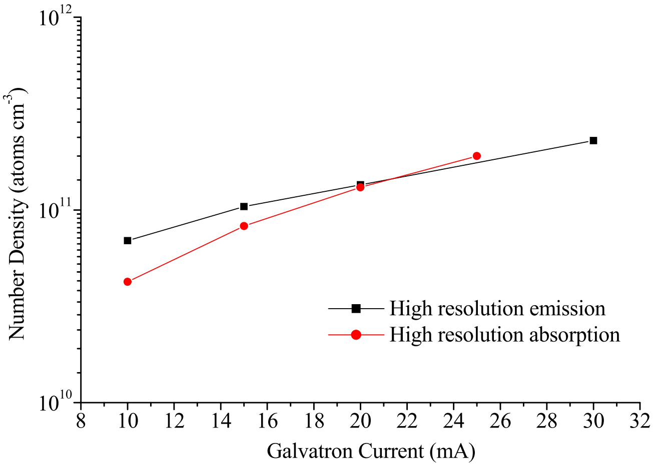

FIGURE 6 Number density measurement of the $6\,^2P_{3/2}{}^{\text{o}}$ metastable state of thallium by absorption and emission measurements. A path length of 2 cm was assumed for each method

3.2 *Conventional absorption measurements*

3.2.1 Number density measurements. The results obtained from the strip chart recorder are used to calculate the ground state number density for each current using (1a)–(1c). Temperature values used to calculate the Doppler width of the absorption profiles are obtained from results obtained by high resolution emission measurements [16]. Figure 7 shows that the absorption data obtained for thallium are not in agreement with values obtained from saturated fluorescence measurements [19], whereas the lead data shows excellent agreement. In order to explain this discrepancy, our assumptions must be reviewed. Two major assumptions are made; the hollow cathode lamp acts as a line source and that the absorption path length is constant at each current. The method for calculating the number density is based on the assumption that an infinitely narrow line source is used; however, this is known to not be true since the emission of the hollow cathode lamp will have some inherent width due to Doppler broadening and possibly a small amount of broadening due to self-absorption. This will result in number density errors at lower galvatron currents. Therefore, one must know what the relative widths of the absorption and emission profiles are in order to accurately apply the line source approximation. In addition, because the galvatron is also a line source the detector detects the D.C. emission from the galvatron. At low currents, the background emission noise will be reduced by the lock-in amplifier; however, as the galvatron current is increased the background emission noise will grow to the point where the signal becomes buried in noise.

3.2.2 Line source measurements. In order to validate that the HCL can be approximated as a line source, emission profiles of the HCL and galvatron were measured at similar currents. This is done by use of a scanning Fabry–Pérot interferometer as described in reference [16]. Because of the spectral range of the interferometer mirrors, a thallium ground state transition

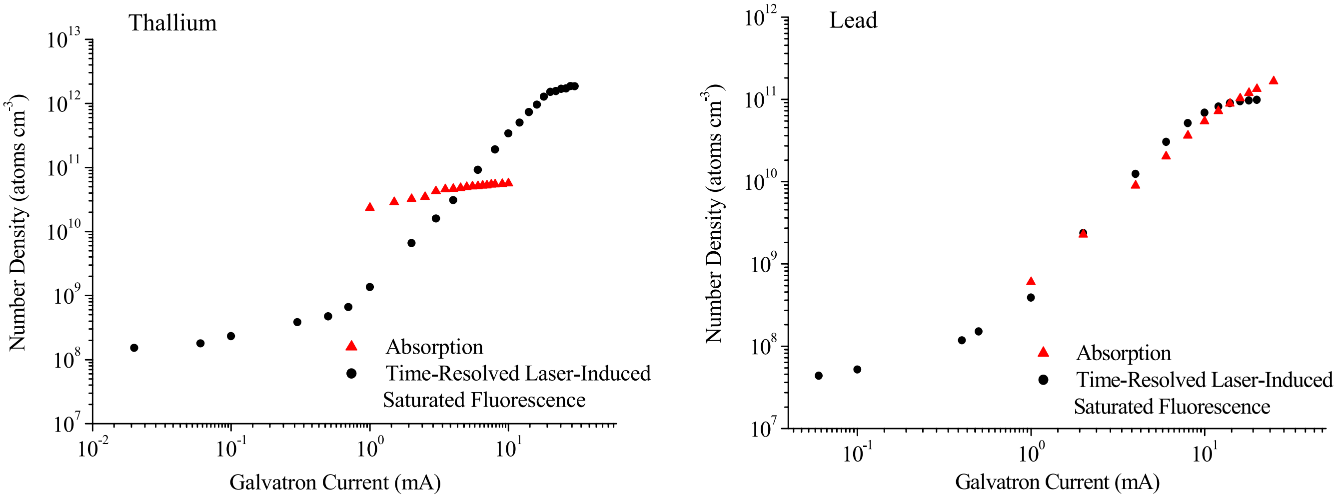

FIGURE 7 Number density measurements by absorption. The results from saturated fluorescence data are also plotted. For the absorption number density calculation, temperature values were obtained from high resolution emission measurements [16]. The emission source (HCl) current was fixed at 2.0 mA

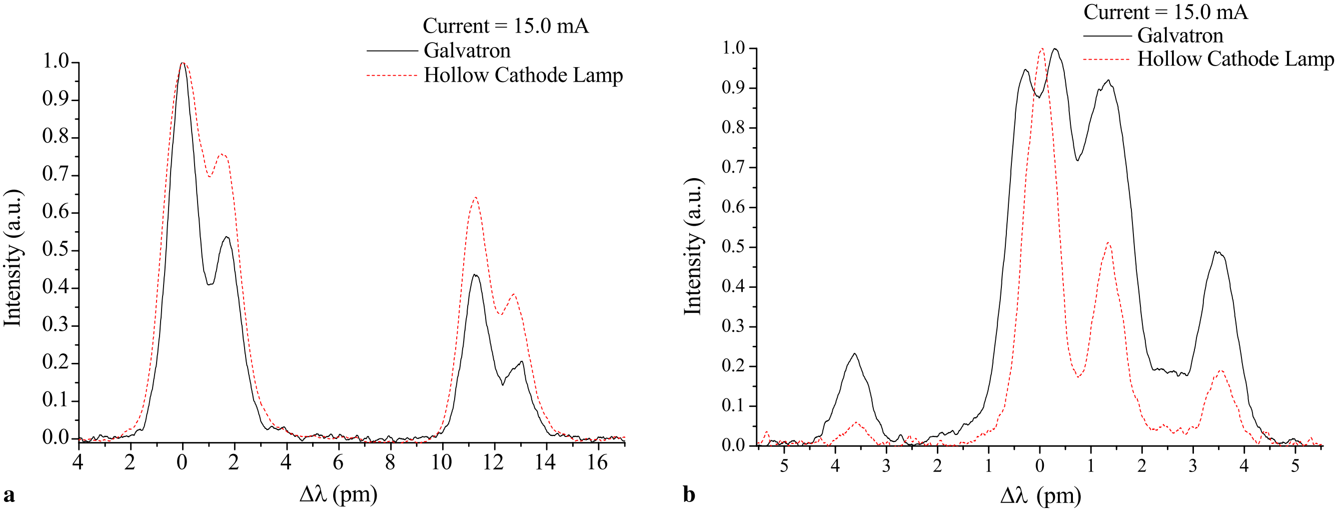

FIGURE 8 (**a**) High resolution emission measurements of the thallium 535.046 nm hyperfine structure in a galvatron and hollow cathode lamp at a current of 15.0 mA. (**b**) High resolution emission measurements of the lead 405.8 nm hyperfine structure in a galvatron and hollow cathode lamp at a current of 15 mA

could not be selected; however, the 535.046 nm transition was accessible and allowed relative conclusions to be drawn about the emission and absorption profiles of the ground state.

Measurement of the emission profiles from the thallium and lead hollow cathode lamps and galvatrons are shown in Fig. 8a and b. It can be seen that when applying similar currents to the HCL and the galvatron yields profiles of different widths. The thallium galvatron exhibits a slightly narrower profile than the HCL with noticeably less self-absorption. It is also important to note that the absorption profile is expected to be narrower than the emission profile observed in Fig. 8a due to the absence of the self-absorption broadening component. From the results in Fig. 8a, it cannot be concluded with any degree of certainty the assumption that a line source was used in the measurement of the number density for the thallium galvatron and would reflect in an error in the calculation. This explains the discrepancy between the fluorescence measurements and absorption measurements observed in Fig. 7. From the results of the lead emission profiles in Fig. 8b, it can be seen that the lead galvatron produces a broader emission profile than the lead hollow cathode lamp at similar currents. Therefore, it can be concluded that the hollow cathode lamp can be approximated as a line source relative to the absorption profile of the galvatron when operated at a low current, which explains the agreement in number density between the absorption measurements and fluorescence measurements.

The major reason the two galvatrons behaved so differently is due to the pressure of the buffer gas used in each lamp. Since the galvatrons are manufactured by Hamamatsu, the Pb HCL by Fisher, and the Tl HCL by Jarrell Ash, it is expected that each lamp will contain a different buffer gas pressure. This can affect the sputtering efficiency, number density, and Doppler temperature of each lamp operated at similar currents.

3.2.3 Path length measurements. Throughout the diagnostics of the thallium and lead galvatrons, it was noticed that the discharge extended beyond the ends of the cathode. Therefore, the optical path length is no longer constant with current or spatially uniform. In (1c), the path length, l, is assumed to be constant and not to vary with current. In order to validate this

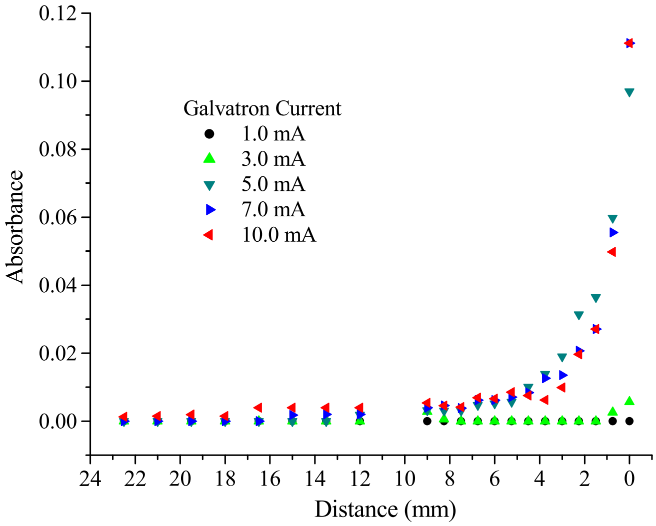

FIGURE 9 Absorption measurements as the cathode was translated through the emission beam. The HCl emission source was held constant at 10 mA throughout this experiment

assumption, the absorbance was measured across the face of the cathode as a function of distance from the face of the cathode. The experimental arrangement is similar to that shown in Fig. 3 except the galvatron is rotated 90° and placed on a translating stage in order to translate the cathode away from the intersecting beam.

A quartz shield surrounds the outer cylinder of the cathode in order to minimize sputtering of the cathode. As a result of sputtering on the face of the cathode, a small amount of metal vapor was deposited on this quartz shield. Therefore, a current of 10 mA was used in order to obtain a measurable signal. Figure 9 clearly shows that absorption occurs beyond the face of the cathode; however, it is only noticeable at higher currents. Even at higher currents, it can be seen that the absorption drops off dramatically past the face of the cathode and only extends out a few mm. This validates the assumption of a single value for the absorption path length with changing current.

4 Conclusions

It has been shown that the combination of two line sources to generate a quasi-continuum source is capable of measuring the absorption profile of a hollow cathode discharge. The results obtained are similar to these obtained by Wagenaar et al. [12], who had a poorly defined baseline; however, in these measurements a poor resolution monochromator was used which greatly relaxed the slit width requirement. This increased the throughput of the system resulting in a higher signal-to-noise ratio. Also, the use of two stable line sources, EDL and HCL, greatly reduced the low frequency flicker noise commonly associated with Xe-arc lamps.

Measurement of the absorption profile yielded number densities that are comparable to these obtained from high resolution emission measurements. The direct measurement of the absorption profile width would not yield an accurate Doppler temperature due to other broadening contributions such as unresolved hyperfine structure components. Despite this, the profile still appears to be Doppler limited and therefore, has a spectral resolution superior to a flame and ICP plasma, and comparable to a low pressure sealed metal vapor cell. The use of a diode laser system would provide an enhanced signal-to-noise ratio as well as provide a better baseline for interpretation of the data, however, such a system was not available in the lab.

It should be noted here that the quasi-continuum source could potentially be generated by another approach. If the EDL was subjected to a magnetic field, then the Zeeman effect would split these levels and the separation dependent on the magnitude of the applied magnetic field. If an appropriate polarizer is used, the combination of this source with a self-absorbed profile should result in a similar flat-topped quasi-continuum source for high resolution measurements. To this author's knowledge, the Zeeman approach has never been attempted. In fact, the only paper found that applied the combination of two line sources to create a quasi-continuum source was by Bazhov and Zherebenko [13] in 1970 and no follow-up papers or any variation of this method towards the application of absorption measurements could be found.

Number density measurements of the thallium and lead galvatrons by conventional absorption method yielded conflicting results when compared to the results obtained by saturated fluorescence. As shown in Fig. 7, thallium shows a very poor agreement to the saturated fluorescence measurements, whereas lead showed excellent agreement between the two methods. It was found that the poor agreement for the thallium galvatron and excellent agreement of the lead galvatron was due to the relative spectral widths of the emission source and absorbing medium. The thallium galvatron produced a narrower emission profile relative to the emission profile of the hollow cathode lamp at similar currents, whereas the lead galvatron produced broader emission profile relative to the emission profile of the lead hollow cathode lamp. From this, it can be concluded that the lead hollow cathode lamp behaved as a line source and in turn resulted in more accurate measurements. The thallium hollow cathode lamp did not satisfy the line source approximation and thus resulted in erroneous results as was demonstrated. The difference in the observed emission profiles is expected to be due to the different construction of each lamp. Each lamp would likely contain a different buffer gas pressure since each HCL came from a different manufacture than the galvatrons.

It has been shown that the use of a hollow cathode lamp can be applied to the measurement of number densities by absorption; however, the relative widths of the emission source and absorption profile should be known in order to correctly apply the line source approximation.

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