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Comparison of Optogalvanic and Laser-Induced Fluorescence Spectroscopy

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Abstract: Within the spectra of lanthanide atoms quite often overlapping spectral lines (blends) are observed. On the example of complicated blend situations within Pr I and La I it is shown that in such cases a combination of optogalvanic and laser-induced fluorescence detection is necessary to find all transitions contributing to the observed spectral signature.

Ketwords: Lanthanide spectroscopy; Optogalvanic detection; Laser-induced fluorescence detection

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1. Introduction

Within the spectra of atoms having a huge number of energy levels, like the atoms of the lanthanide series, quite often overlapping spectral lines are observed, so-called blend situations. On the example of Pr and La it is shown that for such cases a combination of optogalvanic (OG) and laser-induced fluorescence (LIF) detection is necessary to find all blend lines and to determine which energy levels are involved in the formation of the lines.

Investigations of the group in Graz was directed on one hand to the determination of hyperfine (hf) structure constants of several chemical elements, on the other hand to the discovery of previously unknown energy levels.

For the element Pr now about 1200 even-parity levels and 1600 odd-parity levels are known. The databank contains more than 9000 Pr I – lines. The manifold of La is a little bit smaller; now about 700 even-parity La I – levels and 600 odd-parity levels are known, and in the databank approximately 6000 La I – lines are contained. For the handling of these data the program "Elements" [1,2] is used which provides for a certain wavelength classification suggestions and predict hf patterns, if the hf constants of the combining levels are known. Nevertheless, for all investigated chemical elements there exist also a huge number of spectral lines which up to now can not be classified, thus by far not all energy levels are known.

In the present artice it is shown that OG and LIF detection must be both used to identify all lines which contribute to a complicated blend situation.

2. Experiment

Hollow cathode lamps were and are widely used as light sources in classical and Fourier-transform (FT) spectroscopy. For performing laser spectrocopy, we use a special home-made see-through hollow cathode lamp, which is based on ideas of H. Schüler [3] and was further modified by the group of Prof.Dr. G.H. Guthöhrlein in Hamburg [4,5]. This type of hollow cathode lamp was used by the group in Graz and is further used by groups at University of Technology Poznan (Poland), University of Gdansk (Poland), and University of Istanbul (Turkey).

The lamp consist of a grounded cathode, ca. 20 mm long, with an inner diameter of 3 to 4 mm, and two anodes, with an axial distance of 0.8 to 1.2 mm, on both sides of the cathode (see Figure 1a; ceramic holders and voltage pins are not shown). The cathode is made either from the material to be investigated (e.g. Ta, [6]) or from copper. In the latter case the bore is bushed with a thin layer of the metal of interest (e.g. Pr or La) or is prepared with a powder containing a stable chemical compond of it (e.g. Ta_2O_5, [7]). The discharge region is cooled with help of liquid nitrogen in order to minimize Doppler broadening of the spectral lines. The discharge is operated usually in a constant current

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mode (20 to 90 mA), the voltage is ca. 350 V. First the hollow athode lamp is evacuated to ca 10^{-3} mbar, then noble gas (Ar or Ne, ca. 0.2 to 1.5 mbar) is filled and electric voltage applied. After ignition, the plasma burns in the noble gas, and the lamp emits weak light characteristic for Ar or Ne. After some minutes, a sputtering process begins, and the discharge is mainly carried by metal atoms, which have much lower ionization limit than the noble gases. Finally, the discharge becomes stable, is burning in the matal vapor, and emits bright light characteristic now for the element under investigation (e.g. green for Pr and white for La).

A sketch of the laser spectroscopic apparatus is shown in Figure 2b. Narrow-band tunable laser light is provided by a suitable laser system. Here a home-made cw dye laser, based on the optical scheme od a Coherent 699-21 laser, but with a scan range slightly more than 45 GHz was used. A small part of the laser light was provided to a temperature-stabilized confocal Fabry-Perot interferometer (marker etalon). Its transmission signal (free spectral range 367.33(6) MHz) was used to convert the records, made by scanning the laser frequency, from signal versus time to signal versus offset frequency. The main part of the laser light, modulated by a mechanical chopper, intersected the hollow cathode. The discharge light, emitted in the direction opposite to the incoming laser beam was captured by a mirror with a hole and a lens and focused to the entrance slit of a monochromator. At the exit slit of the monochromator a photomultiplier detected the transmitted light. A Lock-In amplifier, synchronized with the chopper, was used to amplify the changes of the transmitted light due to action of the laser beam with the plasma (laser-induced fluorescence, LIF). The Lock-In amplifier could be also used to amplify changes of the voltage drop on a resistor in the discharge curcuit, in order to record so-called optogalvanic (OG) signals. One can quickly switch between OG and LIF signals, even during a laser scan. With this arrangement, Doppler-limited spectra can be recorded. By changing the optical path of the exciting laser light, also Doppler-reduced spectra (via saturation spectroscopy) can be obtained.

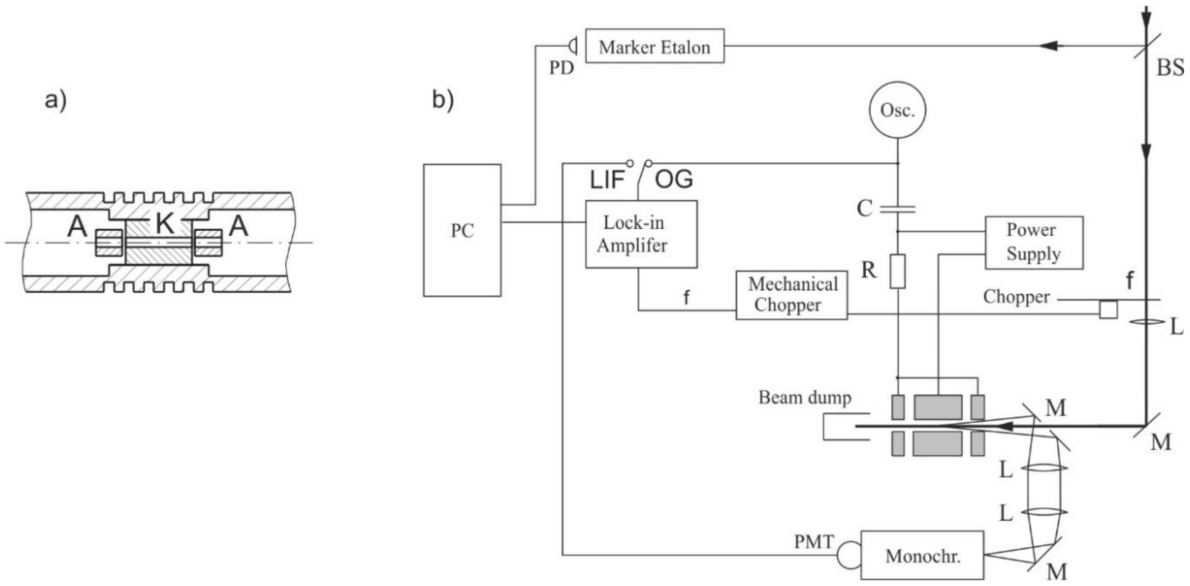

Figure 1. Experiment. a) Schematic cut through the hollow cathode. A … anodes, K … cathode (grounded). b) simplified sketch of the apparatus. BS … beam splitter, M … mirror, L … lens, PMT … photomultiplier tube, PD … photodiode, R … resistor, C … capacitor, Osc. … oscilloscope, f … chopper frequency, PC … personal computer.

The discharge plasma is a self-luminating source of free atoms, and the population of the energy levels follows a detailed thermodynamic equilibrium. This has the advantage that also levels having relatively high energy are sufficiently populated and can serve as lower levels of a laser-driven transitions. On the other hand, usually laser excitation does not influence very much the equilibrium population, thus the addititional LIF intensity caused by laser excitation may be small compared to

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the plasma emission intensity. At OG detection, laser excitation causes only very small changes of the discharge voltage (some mV versus hundreds of V). Thus usually small changes of a high signal must be detected what makes the use of phase-sensitive detection unavoidable. This high background signal causes additional noise.

3. A blend situation within the Pr I – spectrum

Praseodymium, atomic number 59, has one stable isotope with mass number 141 ans nuclear spin quantum number I = 5/2. Its magnetic moment is relatively large (4.2754(5) μ_N [8]), but its electric quadrupole moment is small (-0.077(6) b [8]). Thus the hyperfine (hf) structure of Pr lines is dominated by the magnetic hf structure constant A, only for some energy levels (small) values of the electric hf structure constant B can be determined from Doppler-limited recordings.

The ground level of Pr has odd parity, and higher odd-parity levels start at energy 1376 cm-1. The lowest even-parity has an energy of only 2793 cm-1, thus the ladders of even and odd levels run nearly parallel. This fact and the existence of a huge number of even and odd levels causes a very rich spectrum of Pr; at nearly each laser frequency more than one transition is excited since usually some differences of energies between pairs of combining levels coincide. All excited transitions contribute to the OG signal, and the relative strengths of the signals are dependent on their influence on the discharge current. This influence is – among others - dependent on the transition probability between the combining levels, the population of the lower level of the excited transition, and the position of the upper level relative to the ionization limit.

On the other hand, LIF detection is selective to the excited levels. But when investigating a certain region of the Pr spectrum, it is difficult to find all possible LIF lines. This must be done for each laser wavelength by scanning the monochromator transmission wavelength. Thus one can benefit from a highly resolved FT spectrum or an OG spectrum for setting appropriate excitation wavelengths and then searching for LIF signals.

A smal part of the Pr spectrum, between 5838.04 and 5837.47 Å, is discussed now as an example of combination of FT-, OG and LIF spectroscopy.

In Figure 2, trace a), the OG spectrum is shown in dependence on the offset frequency. Start wavelength of the laser scan is 5838.04 Å (offset frequency 0), and the scan direction is to higher laser frequencies (and thus lower wavelengths).

In the investigated region, one strong spectral line is known, classified already by A.Ginibre [9,10]. This line is clearly visible in an FT spectrum [11], trace c), with signal-to-noise-ratio (SNR) of 140. A simulation of the hf pattern of this line is shown in trace b), directly within trace a), using the known hf constants of the involved levels.

A. Ginibre noticed also the peak at ca. 27500 MHz as a separate line, but could not classify it. At the left side of the strong line another line with SNR of 10 can be identified. The hf pattern of this line is shown in Figure 2 as trace h). But the peak at ca. 27500 MHz offset frequency is too high in the FT spectrum, thus another line must be involved. By use of the classification program "Elements" this peak was identified and recorded as trace g). The lower level of line h) is higher than that of line g) and thus a little bit less populated. Nevertheless, the LIF signal of line g), recorded at LIF wavelength 4934 Å (SNR in the FT spektrum 12) has a relatively low SNR of 15, while the LIF signal of line h), recorded at LIF wavelength 5468 Å (SNR in the FT spektrum 10) has a much higher SNR of 50. A possible reason may be different branching ratios of the upper, laser-populated levels of lines g) and h). Nevetheless, not all structures visible in the OG spectrum at offset frequencies < 20000 MHz are explained by lines g) and h). Apparently, some up to now not detected lines contribute to the OG signal.

Comparing traces a) and b) in Figure 2, it is clear that at the high frequency part of the strong line some other lines must contribute to the OG signal of trace a). Outside the laserspectroscopic investigated range line d) is present, having two weak components in this range. Two further lines were found by laser excitation and detection of LIF, shown in Figure 2 as e) and f). Lower and upper levels of both lines are different only by ca. 10 cm-1. Despite of this fact, the SNR

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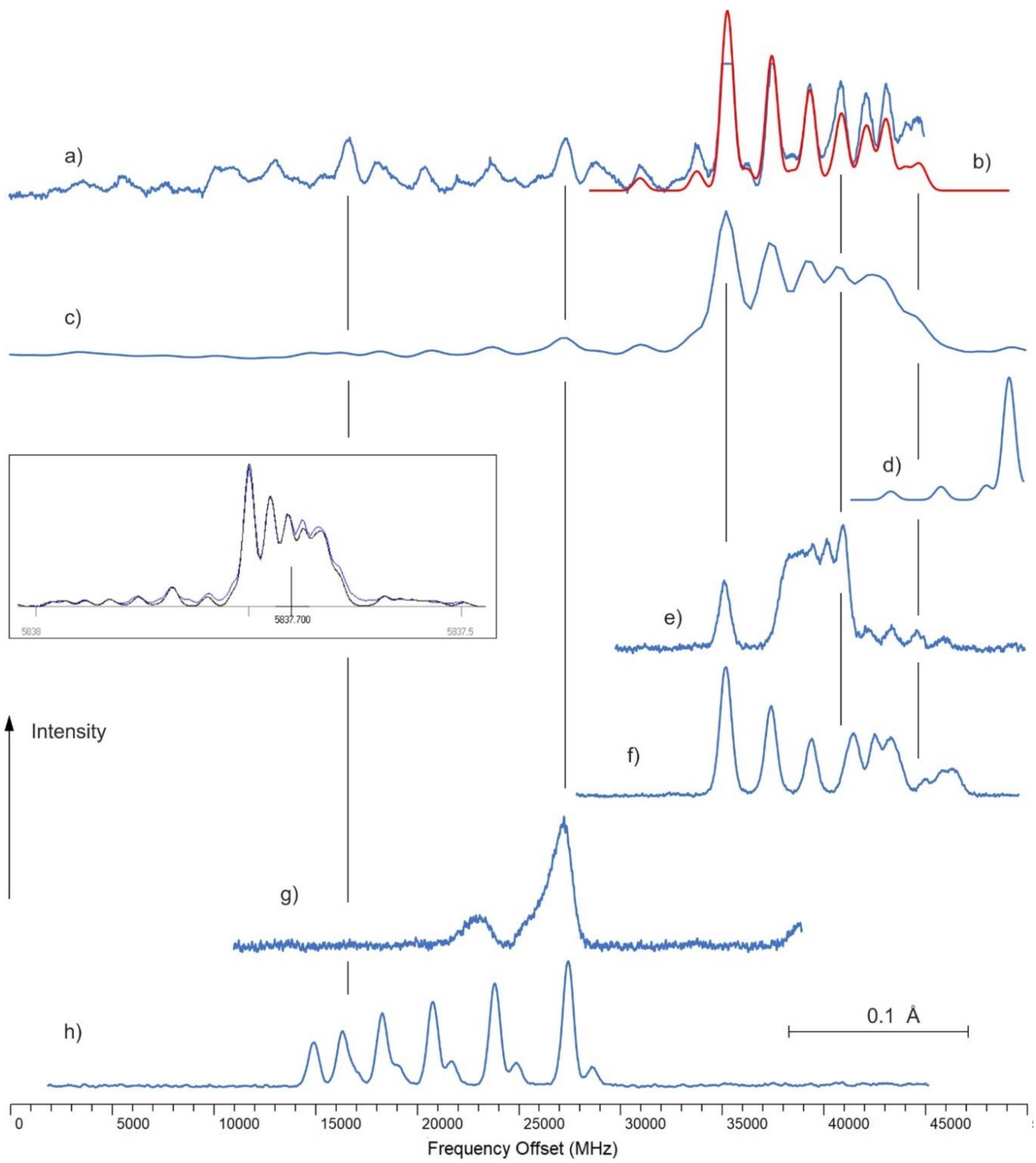

Figure 2. Optogalvanic, FT and LIF spectra between 5838.04 and 5837.47 Å. Classification of lines: see Table 1. FWHM … full width at half maximun, cg … center of gravity, wl … wavelength. The vertical lines should guide the eye of the reader. The inset shows the FT spectrum and a simulated spektrum (black) taking into account lines d), e), g), and h). FWHM = 1300 MHz close to the resolution of the FT spectrum.

a) Optogalvanic record

b) Simulation of the strong line at cg wl 5837.7101 Å using hf constants known from literature assuming FWHM = 700 MHz.

c) FT spectrum. FWHM of the observed structures 1300 MHz. Here only the strong line b) and line h) are visible.

d) This line (cg wl 5837.5607 Å) has some weak hf components in the investigated region. Shown is a simulation assuming FWHM = 700 MHz.

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e), f) Hidden under the strong line at 5837.7101 Å we found two further transitions, detected via LIF

g), h) The peak at ca. 27500 MHz offset frequency, visible in the FT spectrum and the OG spectrum, is caused by the overlap of two further lines, detected via LIF.

of both detected LIF lines differs by a facto of ca. 2. But at offset frequencies < 20000 MHz, lines e) and f) do not sufficiently explain the OG signal. The used classification program does not suggest a line with suitable cg wavelength and hf structure which can explain the remaining deviation. Thus most probably a transition to an up to now not known Pr level is excited when recording the OG signal.

The classification of all Pr lines shown in Figure 2, including the hf constants of the combining levels, is given in Table 1. A level scheme, showing the lines dicussed is shown in Figure 3.

Table 1. Pr-transitions shown in Figures 2 and 3. SNR FT … signal-to-noise-ratio in the FT spectrum, wl … wavelength, E … energy, J angular momentum, A,B … hf structure constants, Ref … reference to values of A,B.

Figure2 trace	wl (Å)	SNR FT	odd E (cm-1)	J	A (MHz)	B (MHz)	Ref.	even E (cm-1)	J	A (MHz)	B (MHz)	Ref.	LIF detected at wl (Å)
b)	5837.7101	140	0.000	9/2	926.209	- 11.878	[12]	17125.256	9/2	614.0(3)	-4(4)	[13]	
d)	5837.5607	10	28675.293	9/2	831(5)		[14]	11549.602	9/2	1064(2)		[15]	
e)	5837.675	-	12108.867	5/2	1272(2)		[16]	29234.227	3/2	1038(4)		[17]	5525.571
f)	5837.70	8	29243.323	13/2	638(5)		[18]	12118.039	13/2	554(1)	- 45(30)	[15]	5268.121
g)	5837.85	9	29742.537(20)	9/2	777(8)		[19]	12617.700	7/2	883(2)		[20]	4934.694
h)	5837.903	10	30485.194(20)	9/2	684(3)		[19]	13360.511	11/2	151(3)		[21]	5468.668

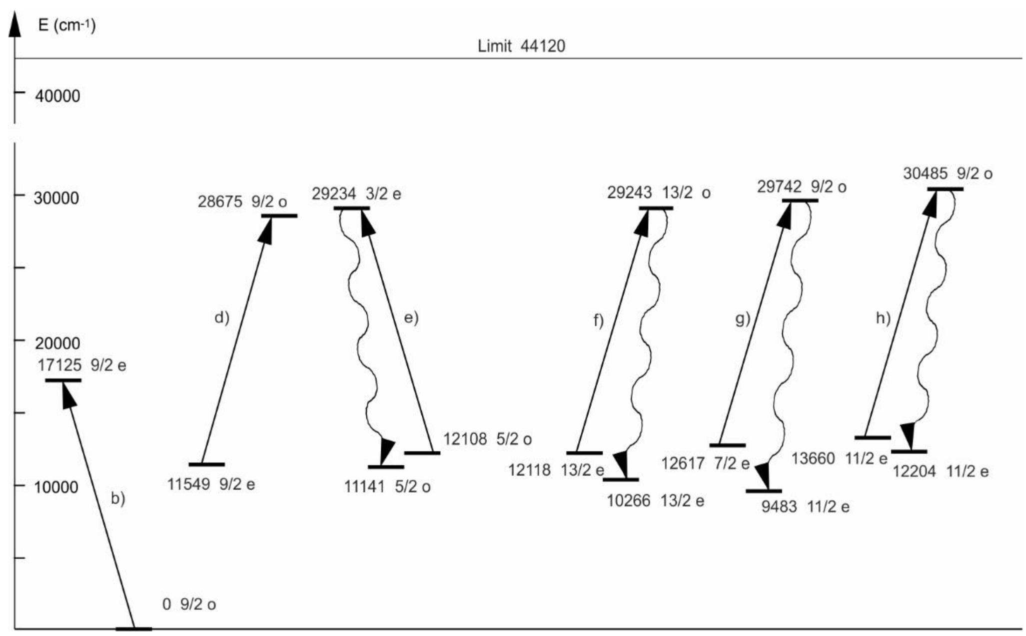

Figure 3. Level scheme of Pr I corresponding to Figure 2. The transitions are labelled according to Table 1. b) strong line dominating the FT spectrum; d) line at the high-frequency border of Figure 2;

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e) to h) lines recorded with LIF. The LIF signals have the same phase as the OG signal; the upper level of the LIF lines is populated via laser light.

4. A blend situation within the La I – spectrum

Lanthanum has atomic number 57, and its natural composition is dominated to 99.91 % by a stable isotope, with mass number 139 and nuclear spin quantum number I = 7/2. Like in Pr, the magnetic moment is relatively large (+2.7830455(9) µN [8]), but its electric quadrupole moment is small (0.200(6) b [8]). The hyperfine (hf) structure of La lines is again dominated by the magnetic hf structure constant A.

Also La I has a huge number of energy levels. The ground level has even parity, the higher even-parity levels start at an energy of 1053 cm-1. But here the lowest odd-parity level has an energy of 13260 cm-1. Thus transitions even-odd and odd-even are not as strongly mixed as in Pr, and the number of lines is smaller. Nevertheless, beside a huge number of separated lines also for this element complicated blend situations are observed, one of it discussed here.

In Figure 4 we treat the spectral region between 5649.48 and 5649.08 Å, corresponding to an offset frequency of 50 GHz. In trace a) the FT spectrum [22] is shown. Only one peak, at ca. 12000 MHz offset frequency, is noticable larger than noise (SNR ≈ 2). But this peak allows to find an accurate cg wavelength value (5649.3797 Å) of the corresponding spectral line.

Trace b) shows the same region recorded with help of the OG signal. The line at 5649.3797 Å shows up now with SNR = 80, and at its high-frequency side a complicated blend situation appears with SNR = 25. This part is enlarged shown as trace c). The strong line has a very narrow hf structure with overlapping components (transition Jlow = 9/2 to Jup = 9/2), while the main structure of the blend could be easily classified as transition Jlow = 3/2 to Jup = 3/2 with widely splitted well separated components. A simulation of both lines (the intensities normalized to the same height, FWHM = 1100 MHz) is shown in trace d).

Then the laser wavelength was set to regions in which the OG signal of the blend was not explained by the 3/2 – 3/2 – transition, and a search for LIF signals was performed by scanning the monochromator transmission wavelength. No LIF signals with the same phase as the OG signal were observed. Apparently the upper laser-excited levels are depopulated maily by collisions which lead to ionization. Thus a relatively strong OG signal, but no LIF-signal is observed. Such behavior is observed for most of Li I - levels with energies higher than 40000 cm-1.

But at some wavelengths LIF-signals with opposite phase occurred. Such a "negative" LIF line (nLIF) occurs if the laser light depletes the population of the upper level of the observed LIF line; this is at the same time the lower level of the laser-driven transition. During all investigations it turned out that nLIF-lines are strong, well-known La transitions to low-lying even-parity levels. Thus observation of a nLIF-line tells the upper, odd-parity level of the observed nLIF-line. In this way the lower level of the laser excited transition is identified, and the unknown upper level must have even parity. J and hf-constants of such a level then can be determined from the hf pattern of the excited line.

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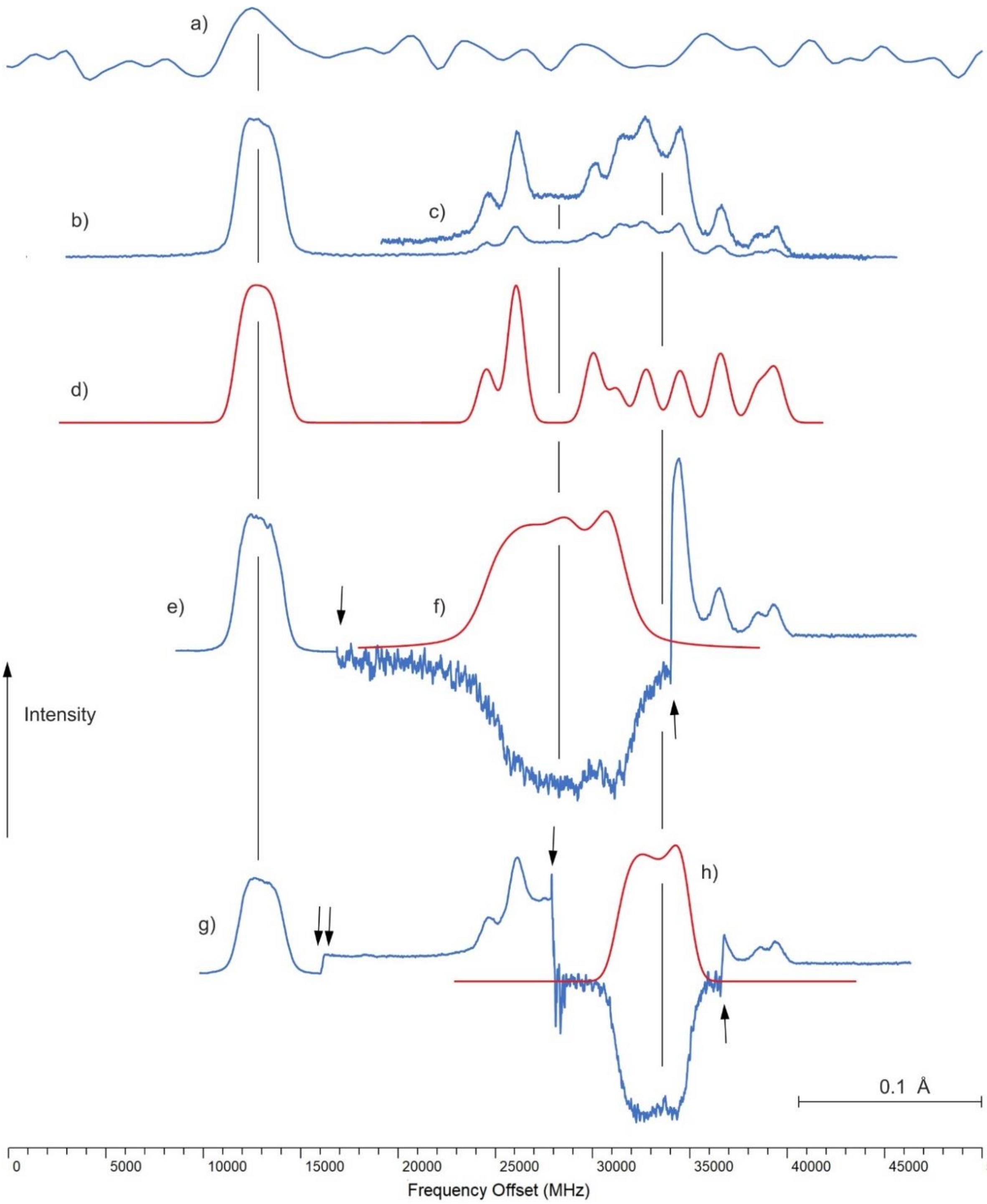

Figure 4. Optogalvanic, FT and LIF spectra of La between 5649.48 and 5649.08 Å. Classification of lines: see Table 2. SNR … signal-to-noise-ratio, FWHM … full width at half maximun, cg … center of gravity, wl … wavelength.

a) FT spectrum. Only the strong line at 5649.3797 Å is visible with SNR ≈ 2.

b) OG spectrum. The strong line is shown here with SNR ≈ 80. Additionally, a complicated blend structure is visible at higher frequencies.

c) The blend structure is shown with the same height as the strong peak. SNR ≈ 25. From the classification program it is easily to find out that the outermost structures of the blend belongs to a wide splitted Jup = 3/2 to Jlow = 3/2 transition.

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d) Simulation of the strong line at cg wl 5649.3797 Å and the Jup = 3/2 to Jlow = 3/2 transition (cg wl 5649.184 Å) using hf constants known from literature, assuming FWHM = 1000 MHz. Both structures are normalized to the same intensity.

e) For use as wavenumber marker, first the OG signal of the strong line was recorded. At ↓ we switched to detection of the LIF signal (monochromator wavelength 4494 Å), and after recording the line, at ↑ back to OG recording (with higher amplification).

f) Simulation of the LIF line, using a line profile which is a sum of 80 % Gaussian and 20% Lorentzian shape, both with FWHM = 2000 MHz.

g) Again, first the strong line is recorded using OG, at ↓↓ we switched to higher amplification. At ↓ we switched to detection of the LIF signal (monochromator wavelength 5145 Å), and after recording the line, at ↑ back to OG recording.

h) Simulation of the LIF line, using a Gaussian line with FWHM = 1200 MHz.

As can be seen, the LIF signals have opposite sign compared to the OG signals. This is an indication that the population of the upper level of the LIF line is lowered by the laser excitation. This level is at the same time the lower level of the excited transition.

In trace e) one of the two observed nLIF- signals, at wavelength 4648 Å, is shown. First the OG signal was recorded, at offset position the detection was switched to LIF detection, and at ↑ back to OG (with higher amplification). This allows not only to record the hf pattern of the unknown blend line, but also to determine the exact frequency position. Trace f) shows a simulation using the meanwhile known hf constants and a line profile which is a sum of 80% Gaussian and 20% Lorentzian shape, both with FWHM = 2000 MHz.

Trace g) shows the third line contributing to the recorded OG signal, recordet at nLIF-wavelength 5145 Å. At ↓ ↓ the Lock-in amplifier was switched to higher amplification, at ↓ to detecton of LIF signals, and at ↑ back to OG. The line has a small hf pattern and lies completely within the 3/2 – 3/2 – transition. A simulation is shown in trace h), using a Gaussian profile with FWHM = 1200 MHz.

As can be learned from Figure 4, for simulating the three lines of the blend one needs different line profiles. This can be explained by different broadening mechanisms occuring to the upper levels, as pointed out in ref. [23]. The SNR of the nLIF-lines is not very high (8 and 12, respectively). This is explained by the fact that the upper level of the strong nLIF-line is highly populated by the discharge, while the laser light depopulates the level only slightly.

A level scheme illustrating the description of Figure 4 is shown in Figure 5. All lines discussed are classified in Table 2.

Table 2. La-transitions shown in Figures 4 and 5. SNR FT … signal-to-noise-ratio in the FT spectrum, wl … wavelength, E … energy, J angular momentum, A,B … hf structure constants, Ref … reference to values of A,B.

Figure4 trace	wl (Å)	SNR FT	odd E (cm-1)	J	A (MHz)	B (MHz)	Ref.	even E (cm-1)	J	A (MHz)	B (MHz)	Ref.	nLIF detected at wl (Å)
b)	5649.3797	2	21383.990	9/2	33.9(30)		[24]	39080.131	9/2	94.9(10)	-20(15)	[25]	
c)	5649.184	-	17797.298	3/2	1335.0(10)		[26]	35494.057	3/2	301(1)	25(7)	[27]	
f)	5649.213	-	24173.826	3/2	-228.9(22)	30(11)	[28]	41870.494	5/2	165(5)		[29]	4648.639
h)	5649.168	-	22439.346	3/2	149.5(32)	-45(35)	[28]	40136.158	5/2	243.8(10)		[26]	5145.418

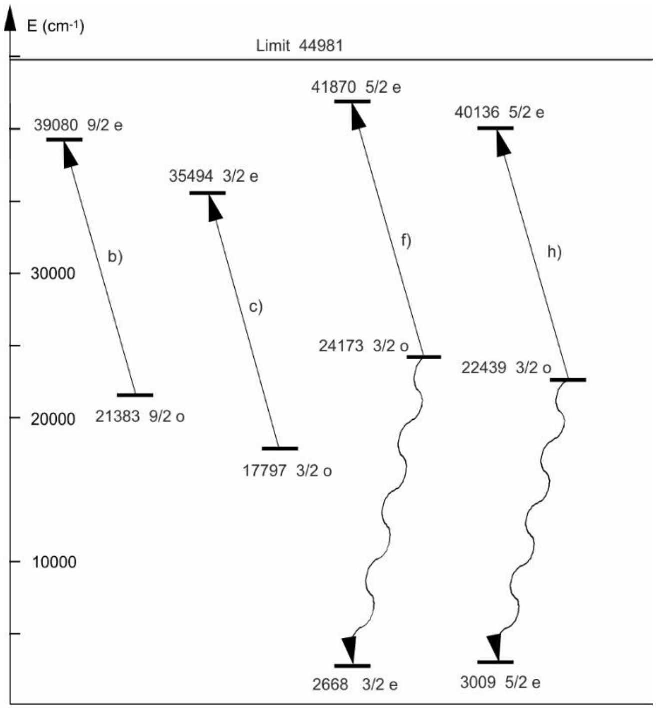

Figure 5. Level scheme of La I corresponding to Figure 4. The transitions are labelled according to Table 2. f) and h): lines recorded with LIF. The LIF signals have the opposite phase as the OG signal; the upper level of the LIF lines is de-populated via laser light, and their intensity is lowered. La I - levels higher than 40000 cm-1 are most probably ionized in the La-plasma, and no direct decay lines of such levels can be observed.

5. Conclusion

It is shown that a general decision between application of OG or LIF spectroscopy can not be made. In complicated blend situations, both methods have to be applied. FT spectra and OG spectra are well suited to set the laser wavelength to a value, where unexplained spectral features are observed. Finding of all lines contributing to a blend situation needs level-sensitive methods like recording of LIF signals.

If spectra recoded via LIF or OG show only one well separated line, in some cases the determination of the hf constants of both combining levels is possible, and at least one of the involved levels can be identified by its hf constants.

For the majority of investigated lines, and especially for blend situations, it is necessary to identify at least one of the levels involved in a transition by treating the manifold of LIF lines. LIF signals having positive phase (intensiy increased by laser interaction) mark the upper level of the laser-excited transition, like discussed at the example of the Pr-blend. If the intensity of a LIF line is lowered by laser interaction, the lower level of the driven transition is marked, as in the discussed La-blend.

Usually a combination of all observations is necessary to determine which transitions are observed, together with the introduction of previously unknown energy levels.

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