Article

Plasma Spectroscopic Study of Dergaon Meteorite, India

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Abstract: Meteorites represent the recoverable portions of asteroids occurring between Mars and Jupiter within the solar system that reach the surface of the Earth. Meteorites are rare extraterrestrial objects studied extensively to improve understanding of planetary evolution. In this work, calibration-free laser-induced breakdown spectroscopy (CF-LIBS) evaluates quantitative elemental and molecular analysis of the Dergaon meteorite, an H 4-5 chondrite fall sample, Assam, India. Spectral signatures of H, N, O, Na, Mg, Al, Si, P, K, Ca, Ti, Cr, Mn, Fe, Co, Ni, Ir, are measured. Along with the atomic emission, this work reports as well molecular emission from FeO molecules. The concentration of the measured elements obtained using CF-LIBS are in close agreement with earlier reports. The elements H, N and O and their concentrations are estimated using CF-LIBS for the first time. This study applies laser spectroscopy to establish presence of Ni, Cr, Co, and Ir in meteorites. Elemental analysis forms the basis for establishment of potential molecular composition of the Dergaon meteorite. Moreover, the elemental analysis approach bodes well for in-situ analyses of extraterrestrial objects including applications in planetary rover missions.

Keywords: Dergaon Meteorite; calibration-free laser-induced breakdown spectroscopy; atomic spectroscopy; molecular spectroscopy; planetary diagnosis.

1. Introduction

Meteorites are the recoverable portions of asteroids that reach the surface of the Earth. The meteorites that are observed to fall and subsequently collected are classified as "falls". The remaining extraterrestrial objects of unknown age are grouped under "finds." Only less than 2 % of all meteorites (iron, stony and stony-irons) collected to date are classified as "falls". The Dergaon meteorite is one such "fall" observed on March 2, 2001, at 16:40 hrs (Meteoritical Bulletin, 2001) around Dergaon (96°46'48"; 26°46'32") village, Assam State, India. It is an ordinary chondrite (H 4-5) representing shock stage S5 [1] and comprises about ten different types of chondrules [2-3] with variable chondrule:matrix ratio (60:40-80:20;)[3].The minerals that are present include: olivine (Ol: ~Fo80-Fa20), orthopyroxene (Opx: ~En82-Fs18), clinopyroxene (Cpx: ~Wo47-En46-Fs7), plagioclase (Plag: Ab87-An13), chlorapatite, chromite, merrillite, kamacite-taenite and troilite [3]. In addition to about 92 volume % of mineral phases, glass constitutes about 8 % of the total volume [3]. It is a relatively well studied chondritic meteorite [3-10] possessing some interesting characteristics, such as the potassium-depletion associated with vesiculated feldspar, the presence of aliphatic hydrocarbons [8-9] and nano-diamonds [10] observed using conventional analytical techniques (including Gamma Ray, FTIR and Laser Raman, etc.). Meteorites were successfully analyzed by laser-induced breakdown spectroscopy (LIBS) [11-16]. For the quantitative elemental analysis of the meteorite, calibration-free LIBS (CF-LIBS) approach is used to determine the elemental composition of Dergaon meteorite. CF-LIBS method is used for analyzing samples such as soils and rocks [17]. Furthermore, CF-LIBS can also analyze meteorite samples that are not easy to be treated by traditional analytical techniques.

In this approach, the traditional calibration curve is not required to quantify the elements present in the sample. The results obtained from CF-LIBS are compared with the results obtained from Instrumental Neutron Activation Analysis (INAA), Atomic Absorption Spectroscopy (AAS) and Inductively Coupled Plasma – Atomic Emission Spectroscopy (ICP-AES) data (Shukla *et al.*) [2] and X-ray fluorescence (XRF) data (Saikia *et al.*) [9] from the past literature. Thus, our experiment demonstrates that CF-LIBS is a rapid and fast technique for quantitative analysis showing the presence of heavy and light elements rapidly while conserving the valuable extraterrestrial sample for the posterity. An attempt is also made in this study to establish `the extraterrestrial affinity of Dergaon sample using high nickel/chromium content [18] and the presence of iridium (Ir) [18] based on LIBS spectral signatures [19]. We have quantified the presence of characteristic elements in the above meteorite using CF-LIBS technique.

2. Materials and methods

The LIBS study was conducted on a sliced Dergaon fall meteorite sample and five polished sections were used for petrographic study. Figure 1 illustrates the Dergaon sample. The LIBS spectra were obtained on a freshly cut smooth surface.

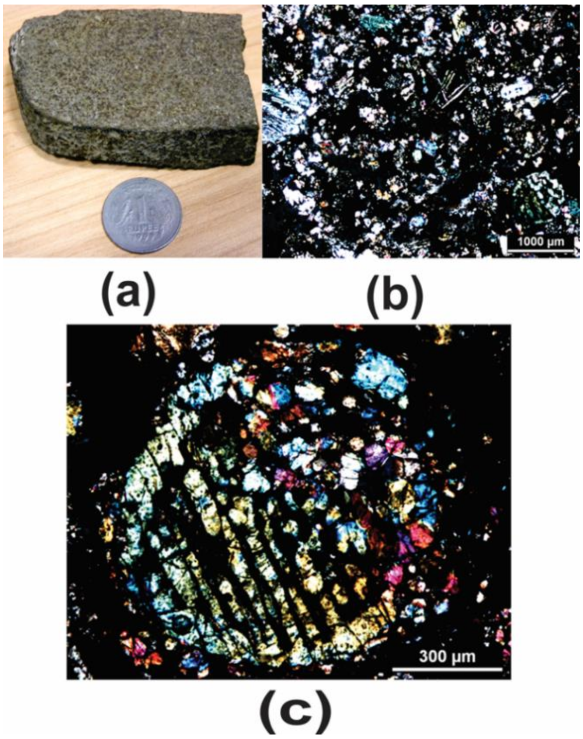


Figure 1. (a) Photograph of Dergaon fall meteorite sample (KD). **(b)** Photomicrograph of Dergaon meteorite showing chondrules of various typesand exhibiting porphyritic texture under crossed polars. **(c)** A barred olivine chondrule from the Dergaon meteorite showing the olivine bars and single rim in optical continuity. The glass phase is optically isotropic under crossed polars. Metallic phases are optically opaque.

2.1. Experimental arrangement

The experimental setup is similar to our recent study [19]. The Nd:YAG laser radiation (Continuum Surellite III-10) is adjusted to an energy/pulse of 15 mJ at 532 nm. The pulse duration amounted to 4 ns. A repetition rate of 10 Hz was selected for recording of LIBS spectra from the meteorite. Every spectrum presented in this study is the average spectra of 10 laser shots. The laser beam is directly focused on the flat, sliced surface of the Dergaon meteorite sample (see Figure 1) using a convergent lens of focal length 15 cm to produce laser-induced plasma at the surface of the sample. Care is taken to avoid crater formation on the sample surface. The meteorite sample is placed on the translation stage and is continuously moved to get a fresh surface for each laser shot. The emiited light from the laser-induced plasma is collected and fed to the Mechelle spectrometer (ME5000, Andor Technology, US) equipped with an intensified charge-coupled device (ICCD) (model iStar 334, Andor Technology, US). The gate delay and gate widths are 0.7 µs and 4 µs, respectively. With these choices, contributions from background continuum are suppressed while sustaining reasonable signal to background and signal to noise ratios.

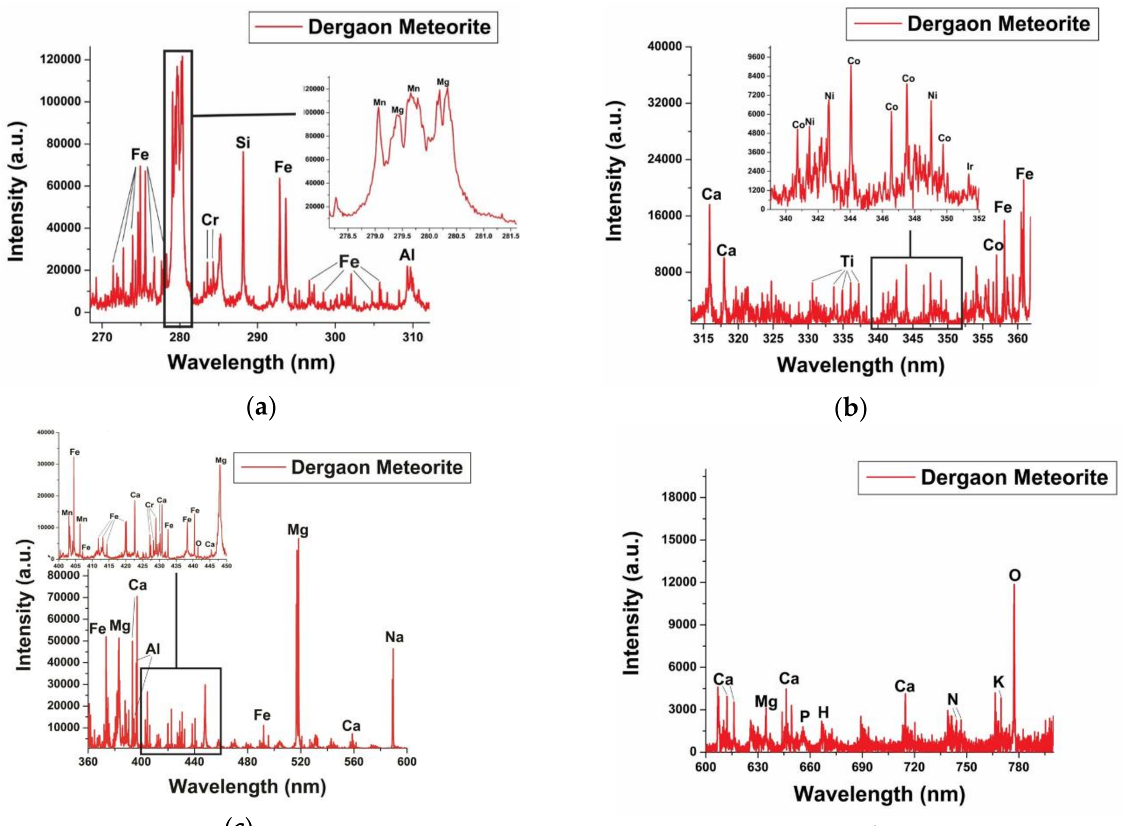
3. Results and discussion

The spectra of Dergaon chondrite are recorded in the range of 200 to 900 nm. The spectral lines for various elements are identified using NIST spectral database [20] and are enumerated in Table 1.

Table 1. Spectral wavelengths observed with laser spectroscopy of Dergaon meteorite.

Elements	Major lines (nm) present in Dergaon meteorite
H (1)	656.3(I)
N (7)	744.2(I), 746.8(I), 868.3(I)
O (8)	777.4(I), 844.6(I), 926.6(I)
Na (11)	588.9(I), 589.5(I)
Mg (12)	279.0(II), 279.5(II), 280.2(II),285.2(I), 382.9(I), 383.2(I), 383.8(I), 448.1(II), 516.7(I), 517.2(I), 518.3(I)
Al (13)	308.2(I), 309.2(I), 394.3(I), 396.1(I)
Si (14)	220.7(I), 221.0(I), 221.6(I), 250.6(I), 251.4(I), 251.6(I), 251.9(I), 252.8(I), 288.1(I), 390.5(I), 413.1(II),557.6(II), 634.7(II)
P (15)	645.9(II), 650.3(II), 650.7(II)
K (19)	766.4(I), 769.8(I)
Ca (20)	315.8(II), 317.9(II), 370.6(II), 373.6(II), 393.2(II), 396.7(II), 370.5(II), 422.6(I), 430.2(I), 442.5(I), 443.9(I), 445.4(I), 501.9(II), 518.8(I), 558.7(I), 610.2(I), 612.2(I), 616.2(I), 645.6(II), 646.2(I), 649.5(I), 647.2(I), 720.1(I)
Ti (22)	334.9(II), 336.1(II)
Cr (24)	283.5(II), 284.3(II), 284.9.(II), 302.0(I), 369.5(I), 381.5(I), 385.5(I), 428.9(I)
Mn (25)	257.6(II), 279.4(I), 279.8(I), 280.1(I), 380.6(I), 403.0(I), 403.3(I), 403.4(I)
Fe (26)	234.3(II), 238.2(II), 239.5(II), 240.4(II), 249.3(II), 250.7(II), 252.4(I), 254.3(II), 256.7(II), 258.5(II), 259.8(II), 259.9(II), 260.7(II), 261.1(II), 273.9(II), 274.9(II), 275.5(II), 293.6(I), 305.7(I), 305.9(I), 344.0(I), 358.0(I), 358.1(I), 364.7(I), 371.9(I), 373.4(I), 373.7(I), 374.5(I), 374.9(I), 375.8(I), 382.0(I), 385.9(I), 404.1(I), 404.5(I), 406.3(I), 432.5(I), 438.3 (I)
Co (27)	340.5(I), 344.3(I), 347.3(I), 349.5 (I)
Ni (28)	338.0(I), 341.3(I), 342.3(I), 349.2(I),352.4(I)
Ir (77)	351.3(I)

The spectral lines of the elements present in the LIBS spectra of Dergaon meteorite are shown in Figures 2(a) – (d). The distinct advantage of LIBS lies in its unique ability to detect heavy as well as light elements, as indicated in Figure 2.



(**a**) (**b**)

(**c**) (**d**)

Figure 2. Typical LIBS spectra of Dergaon meteorite in the wavelength range **(a)** 270-310 nm; **(b)** 315-360 nm; (c) 360-560 nm; and **(d)** 600-800 nm.

The spectral lines of characteristic trace elements observed in the LIBS spectra of Dergaon chondritic meteorite include Cr, Co, Ni, and Ir as shown in Figure 2(b). It is essential to mention that Cr, Co and Ni are also found in mafic-ultramafic terrestrial rocks. But, iridium (Ir), a platinum group element, very rarely occurs in ultramafic rocks on Earth and is used to trace extraterrestrial signature [18]. However, the extraterrestrial samples, like chondritic meteorites, show Ni/Cr>1 and contain substantial Ir. Furthermore, wavelengths of spectral lines for characteristic elements found in the Dergaon meteorite include Ni I (338.05, 341.34, 342.37, 349.29 and 352.454 nm), Co I (340.51, 344.364, 347.39and 349.56 nm) and Ir I (351.364 nm). Identical spectral lines of these elements are also observed in meteorites analyzed with LIBS recently [16]. An earlier study on Dergaon meteorite reported the presence of noble gases and some light elements as well [2]. At a suitable gate delay the signature of the molecular emission are also expected to be observed with LIBS [21, 22]. Our experimental result reveal that the concentration of Fe and O in the present sample is in appreciable amount i.e. ≅ 28% and ≅ 34% respectively. The high concentration of the elements may result in formation of molecular bands in the recorded spectra (23). Thus, we have tried to identify the molecular band of FeO in the LIBS spectra of Dergaon meteorite. Figure 3 displays signatures of molecular bands observed in the range 558 nm to 564 nm.The molecular spectra belong to the orange band system of the FeO molecule [24].

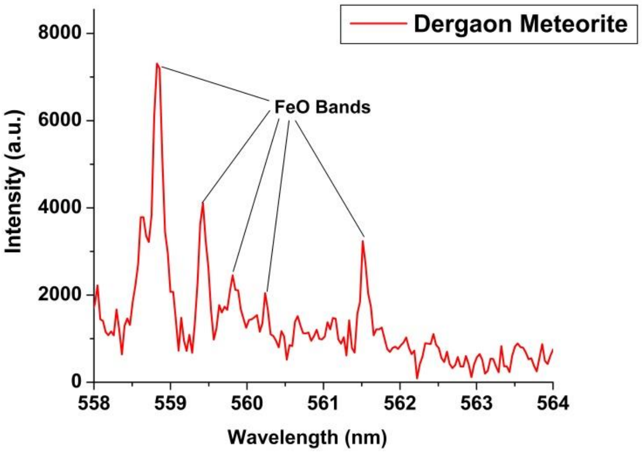


Figure 3. FeO molecular bands recorded with LIBS of Dergaon meteorite.

For verification, the concentration ratio of Ni and Cr (i.e. Ni/Cr>1) are further investigated using a quantitative analysis of elemental constituents present in the Dergaon meteorite [18]. In the CF-LIBS approach, the intensity of the spectral lines of all the elements present in the material is used and its application involves three assumptions:

(i) optically thin plasma,
(ii) stoichiometric ablation, and
(iii) thermal equilibrium.

The three assumptions (i) to (iii) are discussed in the subsequent paragraphs.

3.1. Optically thin plasma

In CF-LIBS, measurement of the absolute intensity of spectral lines is required, i.e., the area under of the spectral lines profile needs to be determined. The spectral lines used for the measurement of intensity should be free from self-absorption. Self-absorption of a spectral line causes distortion of the line profile that usually causes errors in the determined area. These errors may leads to the wrong values of electron density and temperature. Therefore, laser-induced plasma should be optically thin to avoid self-absorption. The plasma is optically thin if the intensity ratio of two interference-free spectral lines of the element having nearly the same upper energy level is equal to the multiplication of transition probability, statistical weight and the inverse of the wavelength of these spectral lines. The intensity ratios, I/I_0 for spectral lines of Ca (315.8/317.9) and Fe (374.9/375.8) present in the LIBS spectra of Dergaon meteorite are calculated. It is found that these ratios are close to $A_{ki}g_k\lambda'/A'_{ki}\,g'_k\lambda$. The results are summarized in Table 2. Consequently, the selected lines for analysis of the laser-induced plasma are optically thin [25].

Table 2. Intensity ratios of two atomic lines in the recorded LIBS spectra.

Elements	$A_{ki}g_k\lambda'/A'_{ki}\,g'_k\lambda$	Intensity ratio, I/I′
Fe-I (374.9/375.8)	1.59	1.43 ± 0.02
Ca-II (315.8/317.9)	0.57	0.55 ± 0.01

3.2. Stoichiometric ablation

The intensity of the spectral line present in the laser-induced plasma is directly related to the concentration of the element present in the sample. This is true when the composition of the laser-induced plasma is representative of the target material, i.e., ablation is stoichiometric. It is already experimentally established [25] that when laser irradiance at the focal spot of the sample surface is higher than 10^9 Wcm^{-2}, nearly nanogram material from the focused spot is exploded before the surface layer can vapourize. Therefore, the rapidly heated exploded material in the plasma has the same composition as the target which leads to stoichiometric ablation [26]. In the present experiments, laser energy is 15 mJ and diameter of the focal spot is about ≈ 12 μm and calculated irradiance at the focal spot is found to be ≈ 3.7 × 10^{12} Wcm^{-2}whichcorresponds to the condition of stoichiometric ablation and suggest that the laser-induced plasma is stoichiometric as laser irradiance is greater than 10^9 Wcm^{-2}.

3.3. Local Thermodynamic Equilibrium (LTE)

The intensity of a spectral line, I_λ^{ki} , corresponding to the transition from the upper level 'k' to the lower level 'i' can be calculated using

$$I_\lambda^{ki} = F\, C_s A_{ki} \frac{g_k exp\left\{-\frac{E_k}{k_B T}\right\}}{Q} \, . \tag{1}$$

Here, A_{ki} is the transition probability in sec^{-1}, g_k is degeneracy factor (dimensionless), C_s is the concentration of the emitting atomic species, Q is the partition function of that species at plasma temperature, k_B is the Boltzmann constant, E_k is the energy of the upper level, λ is the wavelength of the spectral line, and F is an experimental parameter which takes into account the optical efficiency of the collection system. Equation 1, may be written as,

$$ln \frac{I_\lambda^{ki}}{A_{ki} g_k} = -\frac{E_k}{k_B T} + ln \frac{C_s F}{Q} \, . \tag{2}$$

For the determination of the plasma temperature, a graph of $ln \frac{I_\lambda^{ki}}{A_{ki} g_k}$ vs. E_k is drawn, *viz.* a Boltzmann plot is constructed as illustrated in Figure 4.

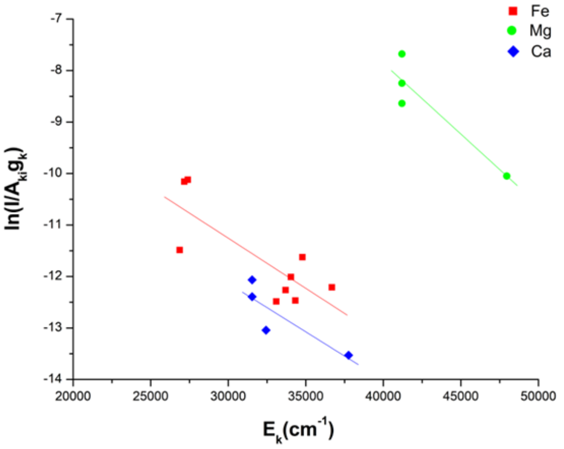


Figure 4. Boltzmann plot for different emission lines of elements for Dergaon meteorite.

According to the McWhirter criterion [27], plasma is in LTE if the value of electron density satisfies

$$N_e\,(\mathrm{cm}^{-3}) > 1.6 \times 10^{12}\,[T\,(K)]^{1/2}\,[\Delta E\,(\mathrm{cm}^{-1})]^{3} \, . \tag{3}$$

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In the present experiment, the electron density is determined from the FWHM of Stark broadened lines given,

$$\Delta\lambda_{1/2} = 2\ w\ (N_e\ /\ 10^{16}). \quad (4)$$

Figure 5 illustrates the recorded calcium line at 422.6 nm. The inferred electron density, N_e, is of the order of $N_e \approx 10^{18}$ cm^{-3}, which is approx. 100 × larger than the lower limit of $N_e \approx 10^{16}$ cm^{-3} for LTE. Therefore, the experimentally determined value of N_e ($\approx 10^{18}$ cm^{-3}) is larger than the limit set by the necessary McWhirter criterium, thus, the laser-induced plasma is in LTE.

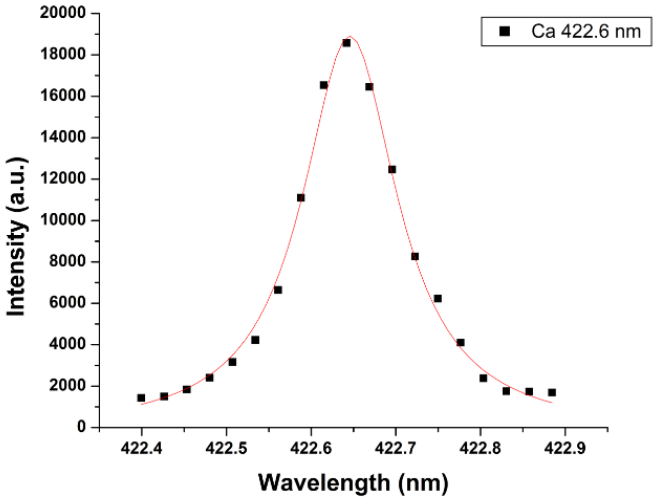

Figure 5. Measured and fitted (Lorentzian) Ca (422.6nm) spectra of Dergaon meteorite.

After verifying the condition of stoichiometric ablation, the concentration of the element present in the Dergaon meteorite is estimated by measuring the intensity of the spectral line present in its LIBS spectra. Using equation 2, the Boltzmann plot as shown in Figure 4 is drawn for spectral lines of other elements. The experimental factor F in equation2 is calculated by assuming that the sum of the relative concentrations of all elements (species) present in the sample is 1, i.e., $\sum C_s = 1$. Finally, by measuring the intercept of the Boltzman plot, the concentrations of all the elements are calculated and the results are given in Table 3. Table 3 communicates that the concentration of Na, Mg, Al, Si, K, Ca, Ti, Cr, Mn, Fe, Co, and Ni, determined in this work with CF-LIBS, agree with values reported using other methods (viz. INAA, AAS and ICP-AES) (Shukla et al.) [2] and (XRF)(Saikia et al.) [9] with deviations of the values within 10%. The total percentage of the elements determined by Shukla et al. and Saika et al. [2,9] is ≈ 47% and ≈66%, respectively. The reported element concentration does not contain noble gases and light elements like (H, N and O). The LIBS spectra of Dergaon meteorite recorded in ambient air show presence of H, N and O (see Figure 2d) unlike previous reports, although minute atmospheric contribution cannot be ruled out. In the experiments, interference of ambient air is suppressed by adjusting the distance between the lens and sample. The H, N and O contents of the ambient air are 0.000055, 78.09 and 20.95%, respectively. However, the present data show that the concentration of O is 40 times larger than that of N (see Table 3) indicating small spectral interference from the ambient air in the spectra. Importantly, this work is the first report of presence of light elements in the Dergaon meteorite.

Table 3. Intensity ratios of two atomic lines for in the recorded LIBS spectra.

Elements (wt %)	INAA, AAS, ICP-AES [1]	XRF [2]	CF-LIBS [3]
H	-	-	0.27
N	-	-	0.69
O	-	-	33.50
Na	0.7061	0.670	0.77
Mg	13.6	14.26	13.44
Al	1.09	1.20	1.70
Si	-	17.30	17.42
K	0.03	0.067	0.09
Ca	1.08	1.19	1.77
Ti	-	0.04	0.07
Cr	0.3705	0.03	0.55
Mn	0.2329	-	0.31
Fe	27.3	27.73	27.90
Co	0.08	-	0.07

[1] Reference INAA, AAS, and ICP-AES data (P.N. Shukla *et al.* 2005) [2].

[2] Reference XRF data (B.J. Saikia et al. 2009) [9].

[3] This work.

The calculated concentrations of elements present in the laser-induced plasma of the Dergaon meteorite obtained by CF-LIBS are summarized in Table 3 and are in close agreement with the earlier data acquired by AAS, ICP-AES, XRF and INAA (Shukla *et al.* and Saikia *et al.*)[2,9].

4. Conclusions

The experimental investigation shows that laser-induced breakdown spectroscopy allows one to perform qualitative and quantitative elemental analysis of extraterrestrial materials with negligible sample loss. This is the first application of the calibration-free LIBS for quantitative analysis of Dergaon fall meteorite. Elemental concentration determination is similar to that obtained by conventional but time-consuming and expensive techniques including significantly more sample destruction than for minute ablation loss seen with LIBS. Identical to the previous study, the Dergaon meteorite shows depleted K concentration with Na >> K. It is an inherently K-depleted H chondrite (Shukla *et al.*) [2] rather than previously suggested impact-induced high-temperature alkali devolatilization process (Ray *et al.*) [3] since laser spectroscopy applied in the present study did not alter the inferred concentrations of alkali elements. The elevated Mg (13.9 wt %), Ni (1.90 wt %) and Cr (0.45 wt %) contents and confirmed presence of Ir (spectral line at 351.364 nm), analogous to previous studies (Shukla *et al.*, Saikia *et al.*, Senesi *et al.*) [2,9,16], and the petrographic observations establish the Dergaon sample as a chondrite meteorite. In addition to characteristic atomic lines of the elements that were recorded, the spectra also show molecular bands of molecules composed of elements that show relatively high concentration.

Author Contributions: The samples, sample photographs, thin-sections are provided by B.B., A.C.M. and K.D. of Gauhati University, Abhishek K.R. conducted the experimental investigations, and A.K.R., J.K.P, C.G.P., S.D., Awadhesh K.R. contributed to the writing of this article.

Funding: This research received no external funding.

Peer-reviewed version available at *Molecules* **2020**, *25*, 984; doi:10.3390/molecules25040984

Acknowledgments: One of the authors, Abhishek K. Rai, expresses thanks to the UGC (CRET) of India for financial assistance of his doctoral research at the University of Allahabad, UP, India.

Conflicts of Interest: The authors declare no conflict of interest.

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