

Atomic Emission Spectroscopy with Spark- or Arc Excitation

Experiments with the DADOS Spectrograph
and Simple Makeshift Tools

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1 Introduction

It may be little known that even amateur astronomers can generate "laboratory spectra" with very simple means. This document is intended to demonstrate simple ways, how one can detect chemical elements in certain materials, liquids and gases, with a spectrograph and easily available electronic and mechanical components.

The strongly simplified procedures, presented in this document, of course do not allow any kind of real "Analytical Chemistry". However at least qualitatively, for many substances, the main components of elements or molecules can be shown. For this purpose, the basic principle of *Optical Emission Spectroscopy* (OES) is applied, which is nowadays in use in commercial, chemical analysis equipments. For this, a sample is excited to such an extent, that it emits a spectroscopically analysable plasma. The wavelength range of professional applications reaches mostly from about 2,000 – 10,000 Å. With simple amateur means, analogous to the Astro Spectroscopy, some 3,800 – 8,000 Å can be covered.

My personal motivation was to reproduce the link between the stellar and laboratory spectra - quasi on the "traces" of Fraunhofer, Bunsen and Kirchhoff. If amateurs should be encouraged to conduct their own experiments, the purpose of this work would be fully met!

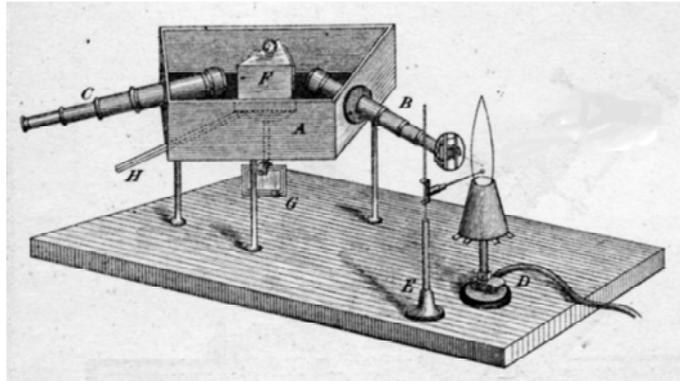
Many thanks to Urs Flükiger, who supported me with metallurgical know-how and arc welding experiments, carried out with equipment and personnel of his company [62].

2 Excitation of Emissions Spectra

Emission lines can not only be generated in stellar atmospheres and gas discharge lamps, but also under the influence of flames, electrical sparks and arcs. In the following these three principles are presented, which are very easy to implement with improvised means. In the professional area numerous other methods exist. Each of these processes is specifically suited for certain types of samples.

2.1 Flame Excitation

This is certainly the oldest and also simplest method. Generally known is the "legendary" Bunsen Burner, which is omnipresent in most of the laboratories (picture from [20]). Further also the fact that common table salt, strewed into the flame, generates the characteristic yellow emission of the sodium doublet D1, D2. Also many gases can be analysed this way. The intense combustion of hydrocarbon gases, such as Butane, produces directly the emission of the famous molecular Swan bands (C_2), as shown in [13].



The disadvantages of this method are the relatively low excitation energy, the latent fire hazard, and the damage or even destruction of the sample.

2.2 Spark Excitation

This is also a very old method, already applied by Bunsen and Kirchhoff [20]. An electric spark generates plasma from a tiny fraction of "vaporized" electrode material, as well as from the struck gas mixture between the electrodes. Thereby temperatures of some 4000 – 5000 K are achieved, corresponding to the stellar atmospheres of the spectral class K. This excitation energy is sufficient even for non-destructive analysis of metals and alloys. Performed with improvised means like piezoelectric push-button gas igniter eg for BBQ grill or *spark inductors*, this is a relatively harmless procedure.

In current commercial analytical equipment the spark excitation of the sample material usually takes place in an inert argon atmosphere. Picture: portable analyse equipment with pistol grip from *Angstrom Inc.* The electrodes consist partly of a special graphite to minimise unwanted intrinsic lines in the spectrum.



2.3 Arc Excitation

A little more sensitive, but also more complex, is the excitation of spectra with arcs. In addition, here an impairment or "consumption" of the sample material is an issue. Here mostly carbon or tungsten electrodes are used and the temperatures reach some 3000 – 4000 K, as found in the stellar atmospheres of late-K and all M-Class.

3 Tests with Spark Excitation

3.1 Equipment

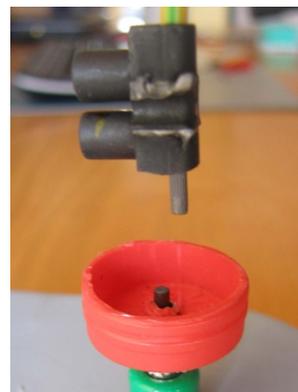
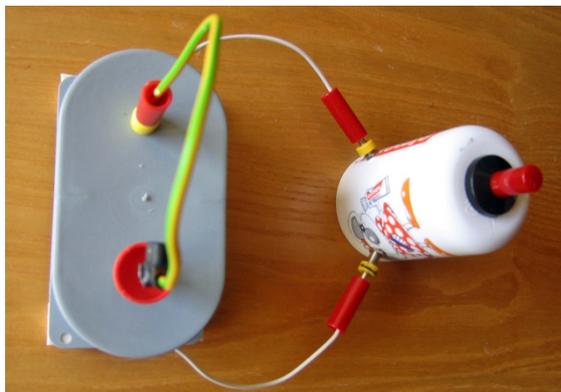
The easiest and cheapest means for first attempts are certainly the already mentioned piezoelectric push-button gas igniters. I use for this purpose the „SUPI V01“, from *Conrad Electronics*, (spare part for BBQ gas grill). It generates per push of a button a voltage surge of about 15 kV, which allows a spark length of >1 cm.



Here mounted into a plastic mug, it is important to keep the two wires at a sufficient distance to avoid an unwanted arcing (short cut). To attenuate the noise level, the upper part of the cup is finally stuffed with foam.



The spark gap is mounted on the bottom of an upside down positioned plastic box, designed for electrical installations. The whole is interconnected by plugs to stay as versatile as possible. The top electrode consists of a short piece of graphite pencil lead, which is attached with a lustre terminal to a thick, stiff but still formable copper wire. Such a graphite electrode is of course only an imperfect substitute for so-called "spectrally pure" graphite. Anyway relatively "soft" molecular C_2 Swan bands are in any case better than sharp narrow atomic emission lines of Fe I, which for example would be produced by an iron nail! The lower electrode consists of the sample holder. For the fastening of different types and shapes of samples I've created a whole set of different versions. In the right picture a makeshift container for loose material or liquids can be seen (eg salt).





Who plans more extensive tests will probably better use a spark inductor. This may be constructed for example using a simple car ignition coil and some additional electronics, which nowadays replaces the former terribly rattling mechanical interruptor. Under these keywords numerous construction guidances are to find on the Internet.

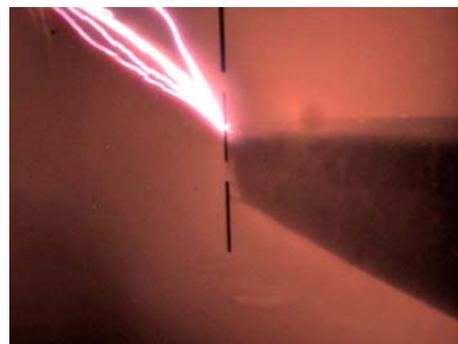
3.2 Design and Implementation of the Experiment

The DADOS spectrograph with CCD camera is mounted on a tripod. Absolutely necessary is a focus lens that produces a sharp image of the sample on the slit plate. For this purpose I use an ancient VIVITAR macro zoom lens. This requirement could also be met using a simple close-up lens of appropriate focal length. The spectra are recorded here with a monochrome camera Meade DSI III Pro. The line identification was performed using the listed references, the "elements" or "lineident" tool of the Vspec software [52], as well as the NIST Atomic Spectra Database [31].



Logically, during the spark discharge the part of the generated plasma is recorded, which is located just on the slit of the spectrograph. Therefore, the impact point of the spark on the sample must be centered on the slit as precisely as possible. Preferred impact points are sharp tips, corners or edges.

The necessity of this procedure shows a simple experiment. If the upper graphite electrode is positioned on the slit, one obtains a spectrum of Swan emission bands with molecular C_2 , which is typical for carbon compounds (Table 1). Aimed on the center of the arc-over between the two electrodes results in the characteristic spectrum of a lightning strike, with lines of ionised air components, ie above all, O II and N II (Table 2). Focused on the sample appears its emission spectrum, possibly superimposed by the spark spectrum of the air molecules, because the test is carried out here without the protection of any argon or another inert gas. In this case, a previously recorded spark spectrum of the air must be subtracted or be displayed together with the sample spectrum. Thus, it becomes immediately clear which emissions originate



from the sample and which from the air molecules. Should only the spark spectrum of the air be visible, in most cases, the impact point of the spark was not positioned accurately enough on the slit of the spectrograph. Therefore, it is always worth to take several shots with a slightly altered perspective. The spark should hit diagonally from the side onto the sample and not run in the slit axis – such as it is shown on the picture above.

For the 200L grating in a darkened room, exposure times <15 seconds have been proven. During this time, the spark was triggered about 10 times. With the 900L grating about 30 seconds, combined with approximately 20 – 30 spark strikes are necessary. Here own experiments are essential. Who is tired of the noise from manually triggered sparks should soon change to a spark inducer. Computer monitors should be turned off during the recordings or turned away because they produce themselves relatively intense emission lines.

The following experiments were carried out with electrically conductive samples, which were used directly as electrodes. Non-conductive materials must first be prepared accordingly. In the literature, e.g. the coating of the surface with graphite powder, etc. is recommended. For liquids, [21] for example suggests the impregnation of coal pills, available in pharmacies.

3.3 Results

Table 1:

Table 1 shows the spectrum of the graphite electrode, i.e. the typical C₂ Swan bands, produced for example by comets and carbon stars [13]. Superimposed is here the spark spectrum of the air molecules, which consists of emissions of ionised oxygen O II, -nitrogen N II and the hydrogen line H α . The strong sodium emission (Fraunhofer D1 and D2) shows up in most such material spectra as a dominant byproduct or "contamination" due to the low excitation energy of Na. This was already noticed by Bunsen and Kirchhoff – excerpts from [20], translated here from German to English:

...Of all the spectral responses the one of sodium is most sensitive. The yellow line, the only one which sodium exhibits in the spectrum, coincides with the Fraunhofer D line and is characterized by a particularly sharp boundary and its exceptional brightness. If the flame temperature is very high and the amount of the applied substance very large a continuous spectrum in the immediate vicinity of the line becomes evident. Intrinsically weak neighbouring lines of other substances appear to be even more weakened and some of them remain often invisible until the reaction of sodium expires...

...Already Swan has advised to the just small amounts of salt, which are necessary to generate very intense sodium lines. The following experiment shows that in the field of chemistry no other reaction can be compared, not even remotely, with the sensitivity of spectroscopic determination of sodium. We deflagrated in the furthest corner of the observation room, which holds about 60 cubic meters of air, 3 milligrams sodium chlorate with milk sugar. After a few minutes a strong sodium line appeared, which completely disappeared just after 10 minutes.

To a somewhat lesser extent than the sodium, also calcium lines (Ca I and Ca II) may appear as a byproduct in such material spectra.

Composite Spectrum: Graphite Electrode + Air DADOS 200L/mm

TABLE 1

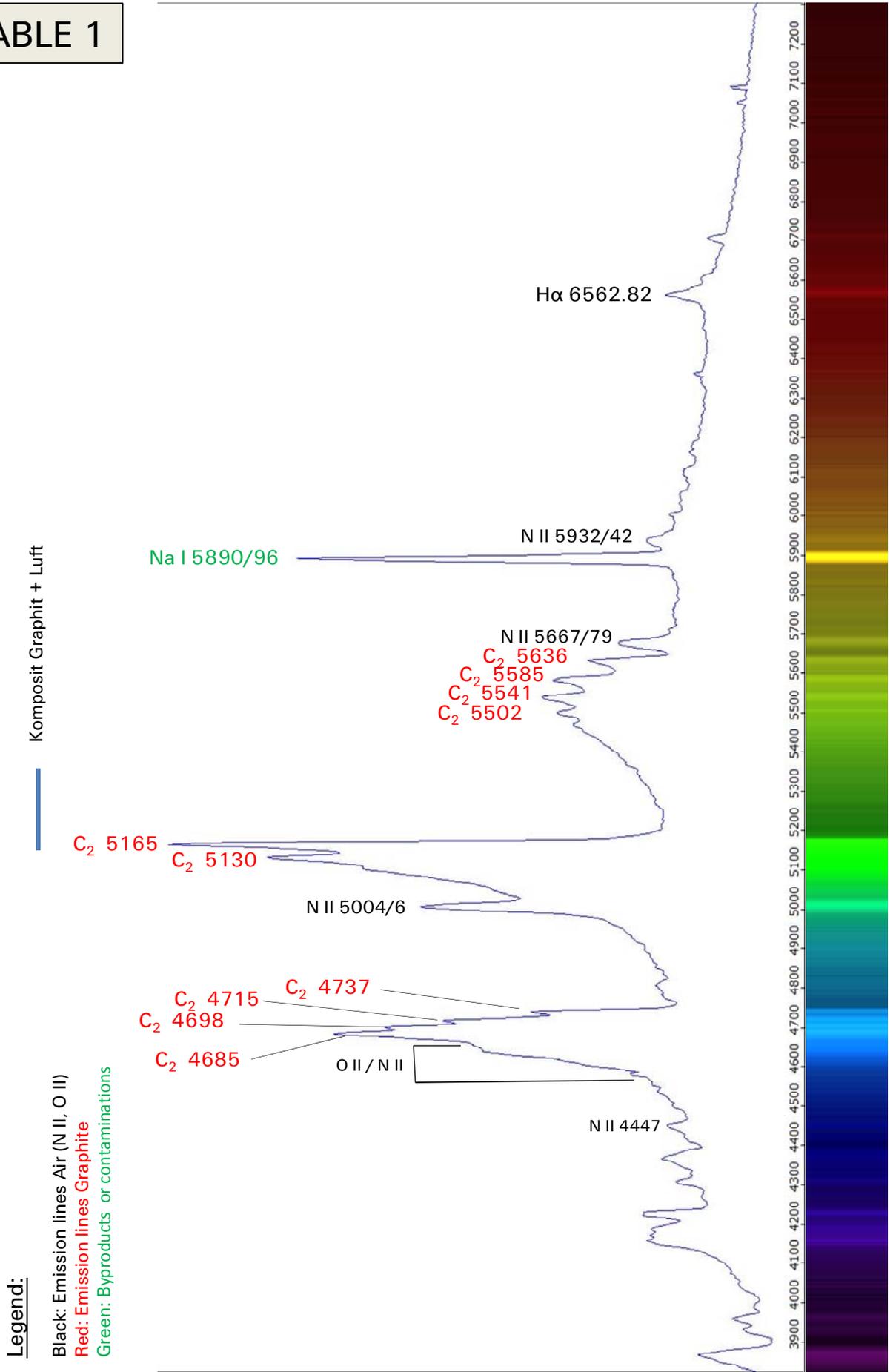


Table 2:

Table 2 shows the spark spectrum of the air molecules, recorded in the middle between the two graphite electrodes. Here just emission lines of the air and no remnants of C_2 Swan bands or any other carbon molecules can be detected.

This profile shows, not surprisingly, striking similarities with a lightning spectrum (see table 2a), which is dominated by the emissions of singly ionised nitrogen (N II) and oxygen (O II). The most intense lines are the cause for the bluish-white color of the lightning or spark. Only the usually strong hydrogen emissions, typically observed in the lightning spectra, appear here just weak in this experimental setup. Interestingly, however, they are more or less intensely visible in most material spectra, even if the assessed samples hardly contain any hydrogen.

Table 2a:

Table 2 shows a lightning spectrum, which was recorded via cloud reflection. The C8 telescope was set up in the living room at night with DADOS and Atik314L+, aiming through the closed window on the approaching thunderstorm. Three shots have been processed, each 180 seconds in 2x2 binning mode. Per exposure the reflected light of about 5-10 lightning strokes has been recorded. Since the cloud base was very low, on all shots the light pollution had to be subtracted. Striking is the intense CN emission at about 3900Å.

Already since the beginning of spectroscopy in the 19 century, it was attempted to gain spectra of lightning discharges. To the beginning of the 20th century also well known astronomers have been involved like Pickering and Slipher. The lightning research today is particularly interested in the course of intensity, plotted against time, of the individual emission lines during a lightning strike. This allows conclusions about the physical processes that are active during the various phases of this phenomenon. The following diagrams from [15], taken with high-speed cameras, show the intensity profile of a lightning spectrum in a time span of 0–50 μ sec and plotted in the range of 4,000-5,000 Å.

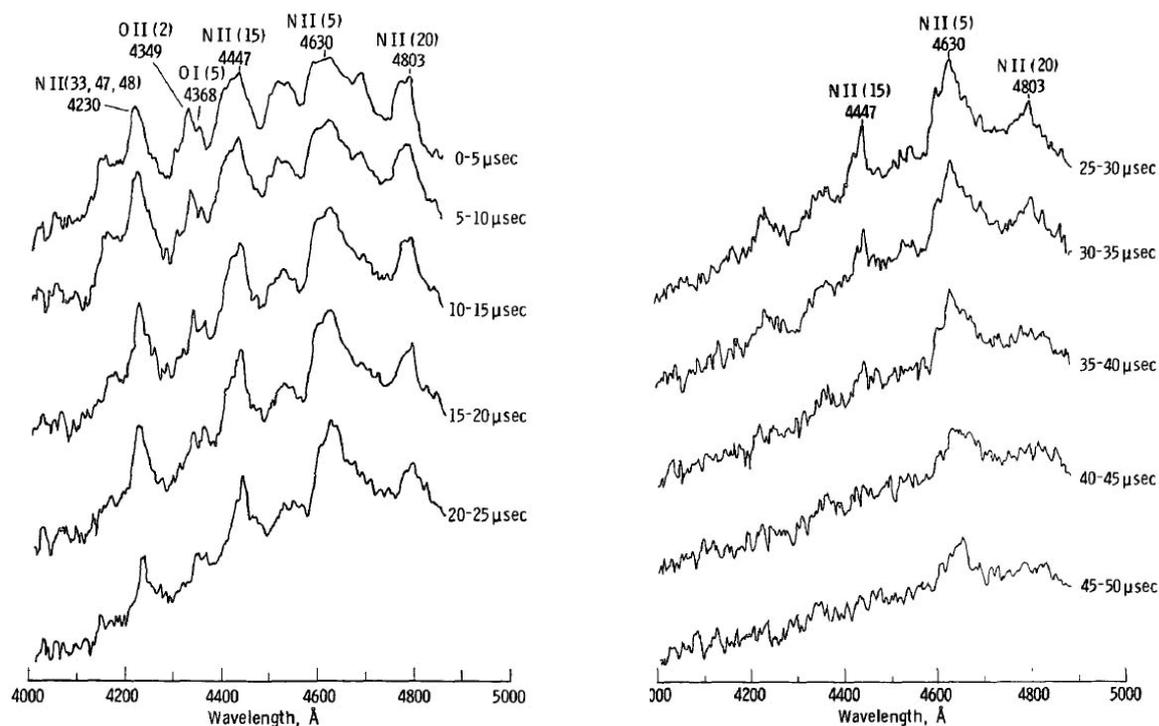
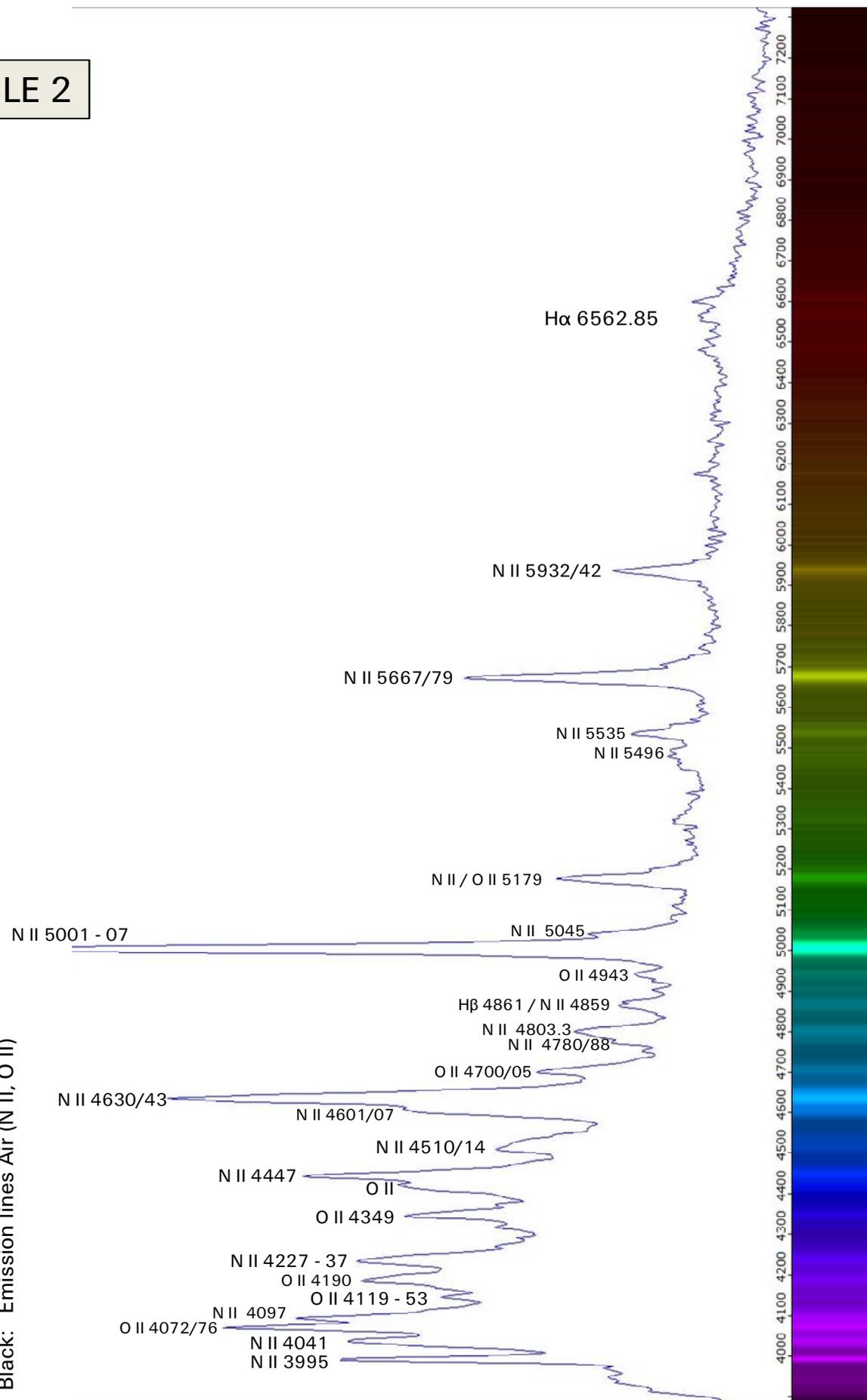


TABLE 2

Spark spectrum of air DADOS 200L/mm

Legend:

Black: Emission lines Air (N II, O II)



Lightning Cloud Reflection

C8, DADOS: Grating 200L mm⁻¹, Slit width 25μm 4.8.2012
 Atik 314L+ -2°C, 3x180s, 2x2 Binning (ca. 30 Lightning strokes)

TABLE 2a

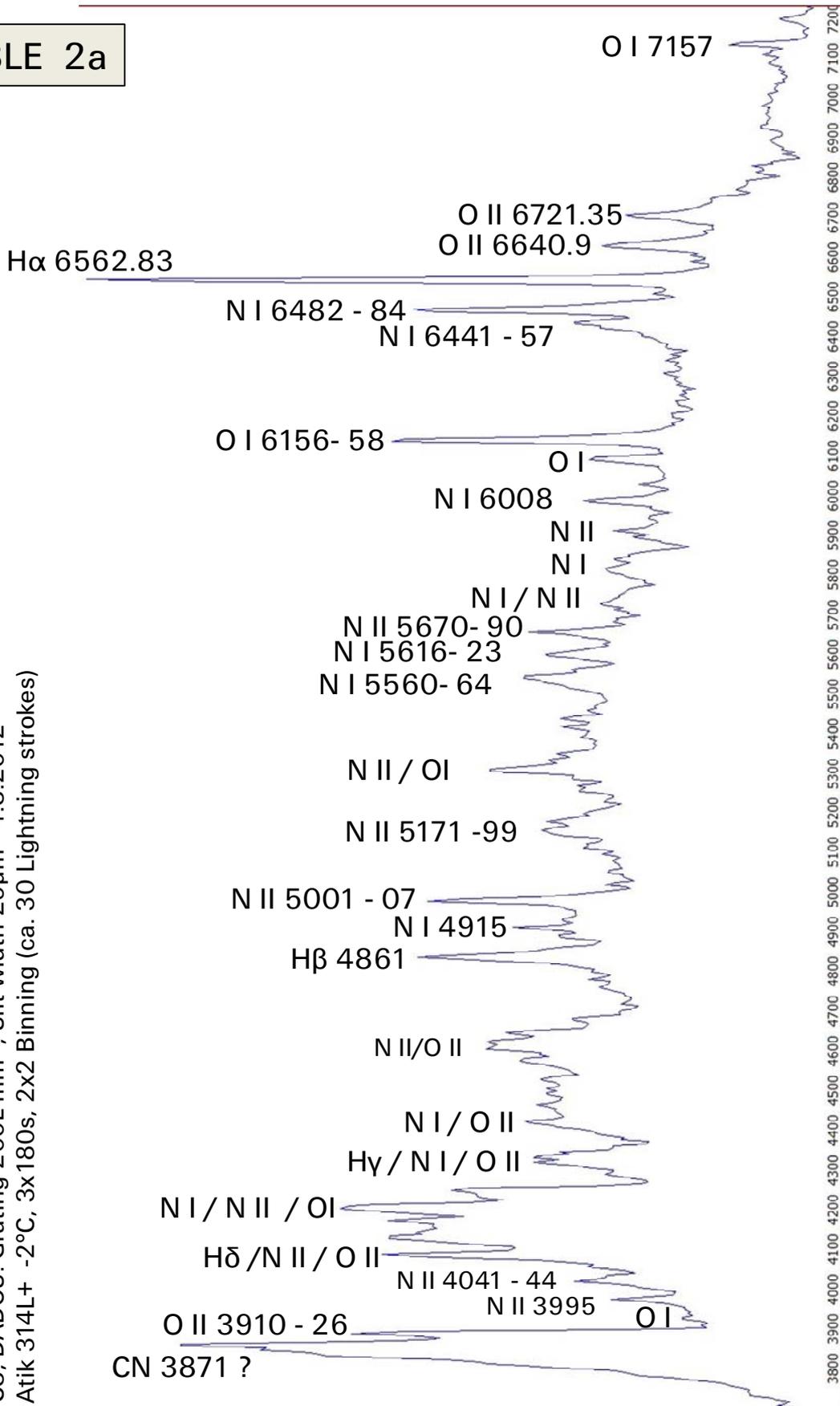


Table 3 to 7:

The tables 3 to 7 show the spectrum of *C15E* steel/materialnumber 1.1141 (hereafter called "*C15E*"). Chemical additives in [%] according to [62]:

| | | |
|----|------------|-------------|
| C | carbon: | 0.12 – 0.18 |
| Si | silicium: | max. 0.40 |
| Mn | manganese: | 0.30 – 0.60 |
| P | phosphor: | max. 0.035 |
| S | sulfur: | max. 0.035 |

Besides the main component *Fe* in the higher-resolution spectra only Mn (0.4%) is detectable. The numerous *Fe* lines could theoretically be used to calibrate a spectrum. Since there are smarter ways however, as to fire up the spark coil each time. The profiles are unnormalised and uncorrected pseudocontinua.

Table 3: Low-resolution (200L) composite spectrum:
C15E steel + spark spectrum of air.

Table 4: 900L composite spectrum in the range 3900 – 4800 Å of *C15E* steel + spark spectrum of air (blue profile). See also *Table 20*, where this spectrum is compared with the profile of the sun. Shown here is also the separately recorded air spectrum without iron lines (red curve).

Table 4a: *C15E* steel spectrum from *Table 4* after subtraction of the air spectrum. Nevertheless, here some weaker emission lines of the air can still be seen. The iron lines show up here more intensive (subtraction with the *Vspec* function "*Subtract Profile by Profile*").

Table 5: dito *Table 4*, here in the range of 4650 – 5550 Å

Table 6: dito *Table 4*, here in the range of 5550 – 6450 Å and without a superposed air spectrum, because almost no *Fe* I lines show up here.

Table 7: dito *Table 6*, here in the range of 6450 – 7400 Å

TABLE 3

Composite Spectrum: C15E Steel + Air DADOS 200L/mm

Legend:
 Black: Emission lines Air (N II, O II)
 Red: Emission lines steel C15E
 Green: By-products or contaminations

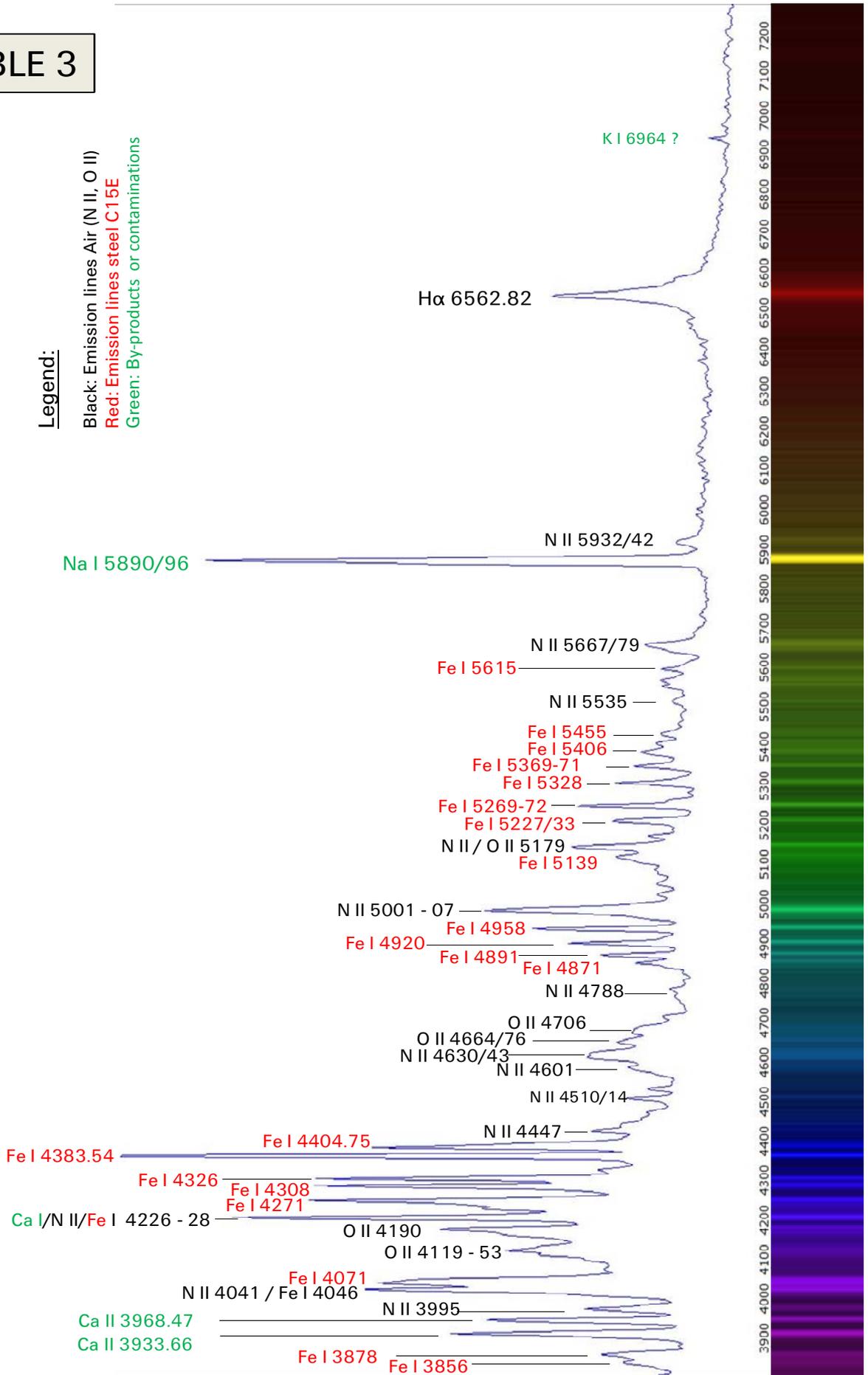
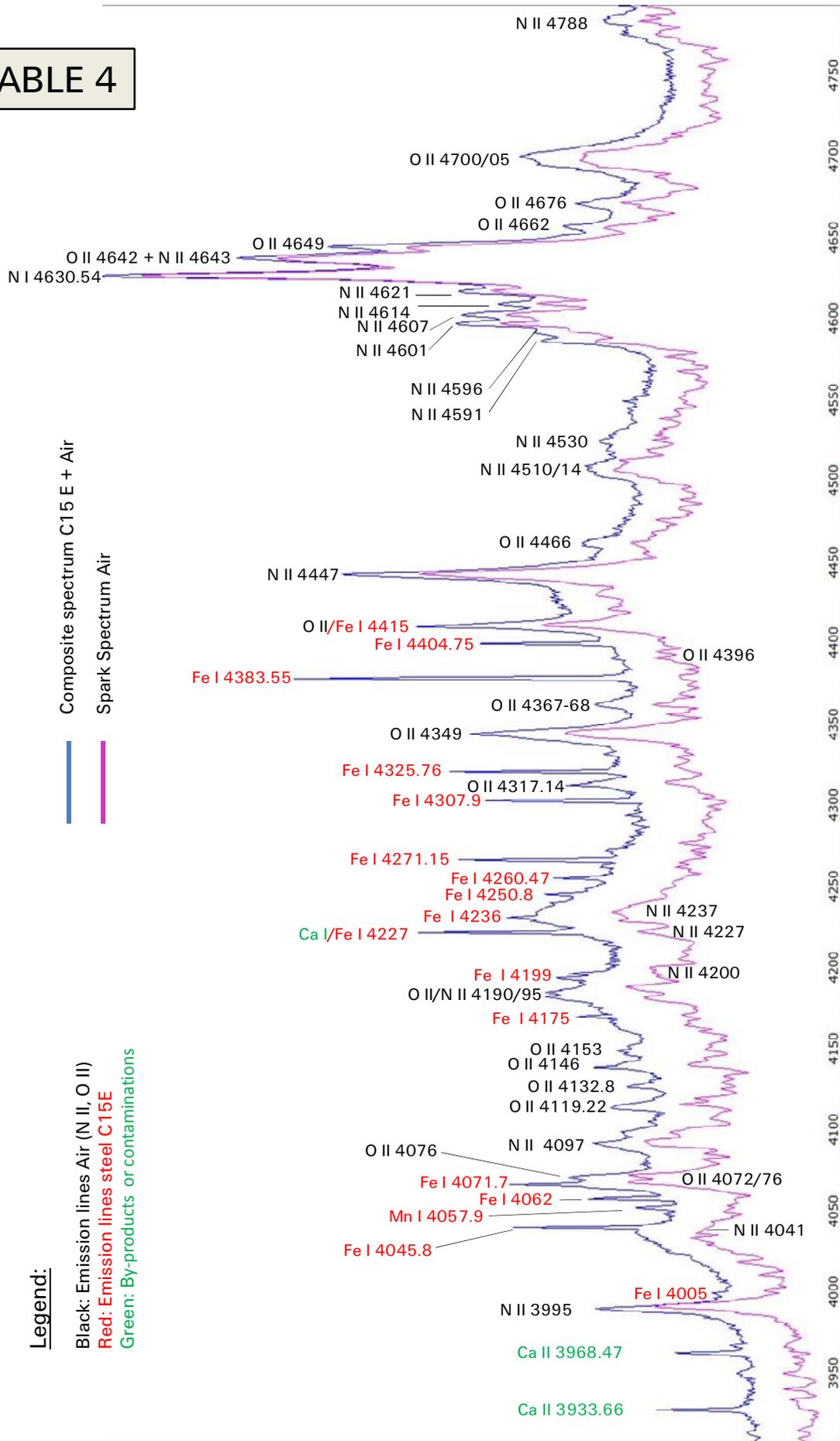


TABLE 4

Composite Spectrum: C15E Steel + Air DADOS 900L/mm



C15E-Steel Spectrum after Subtraction of the Air Spectrum 900L/mm

TABLE 4a

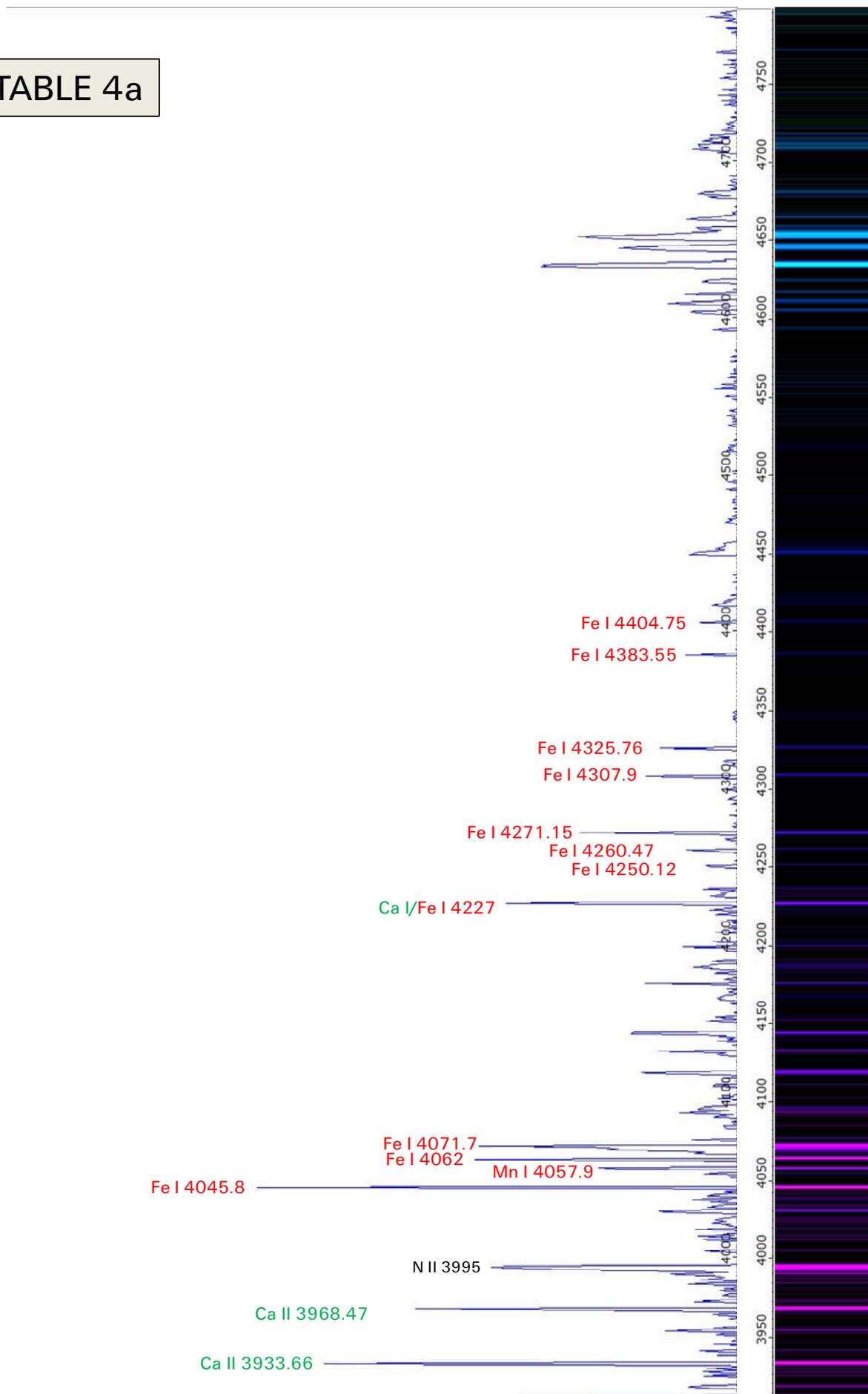


TABLE 5

Composite Spectrum: C15E Steel + Air DADOS 900L/mm

Legend:

Black: Emission lines Air (N II, O II)

Red: Emission lines steel C15E

Green: By-products or contaminations

Composite spectrum C15 E + Air

Spark Spectrum Air

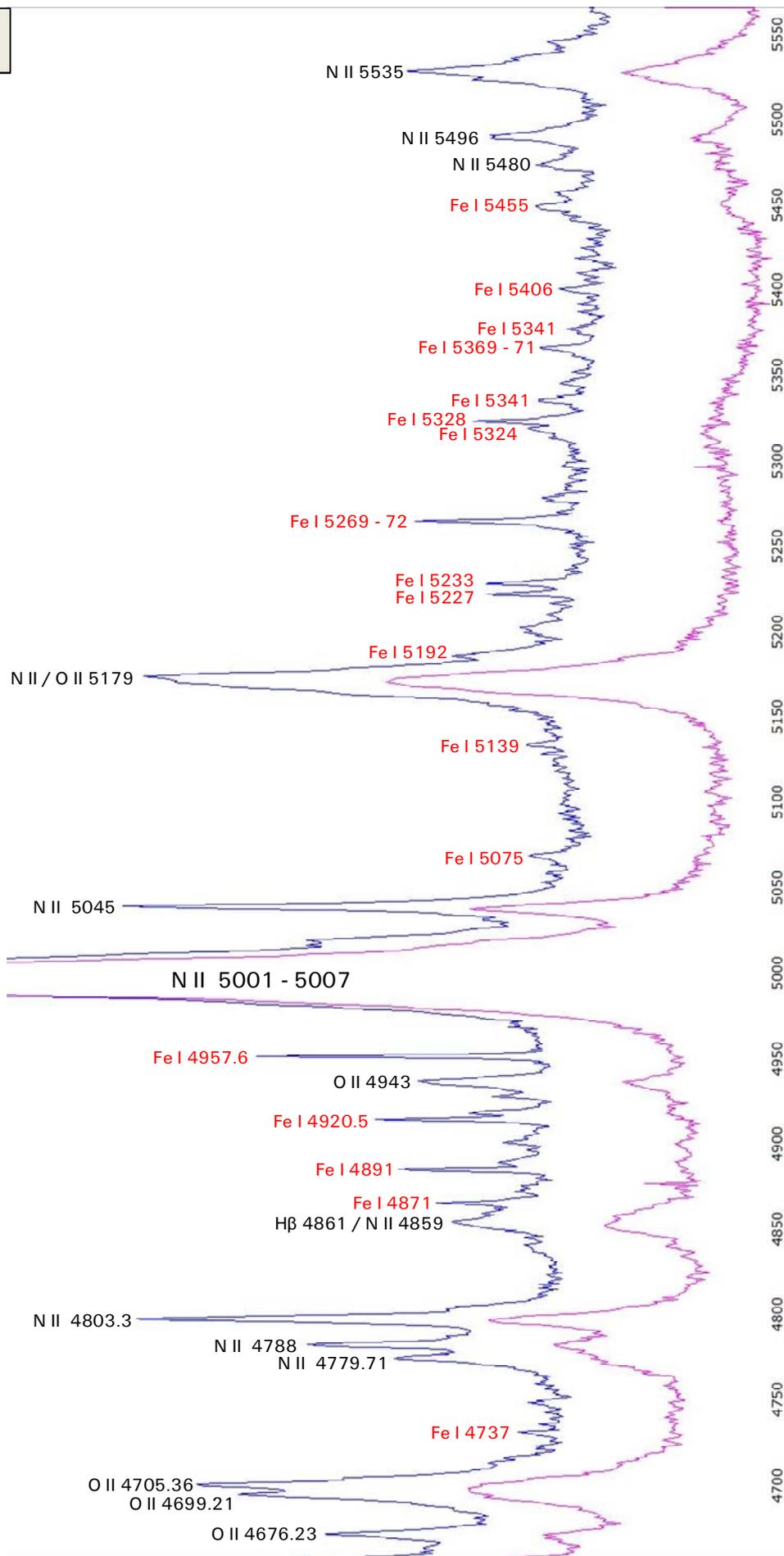
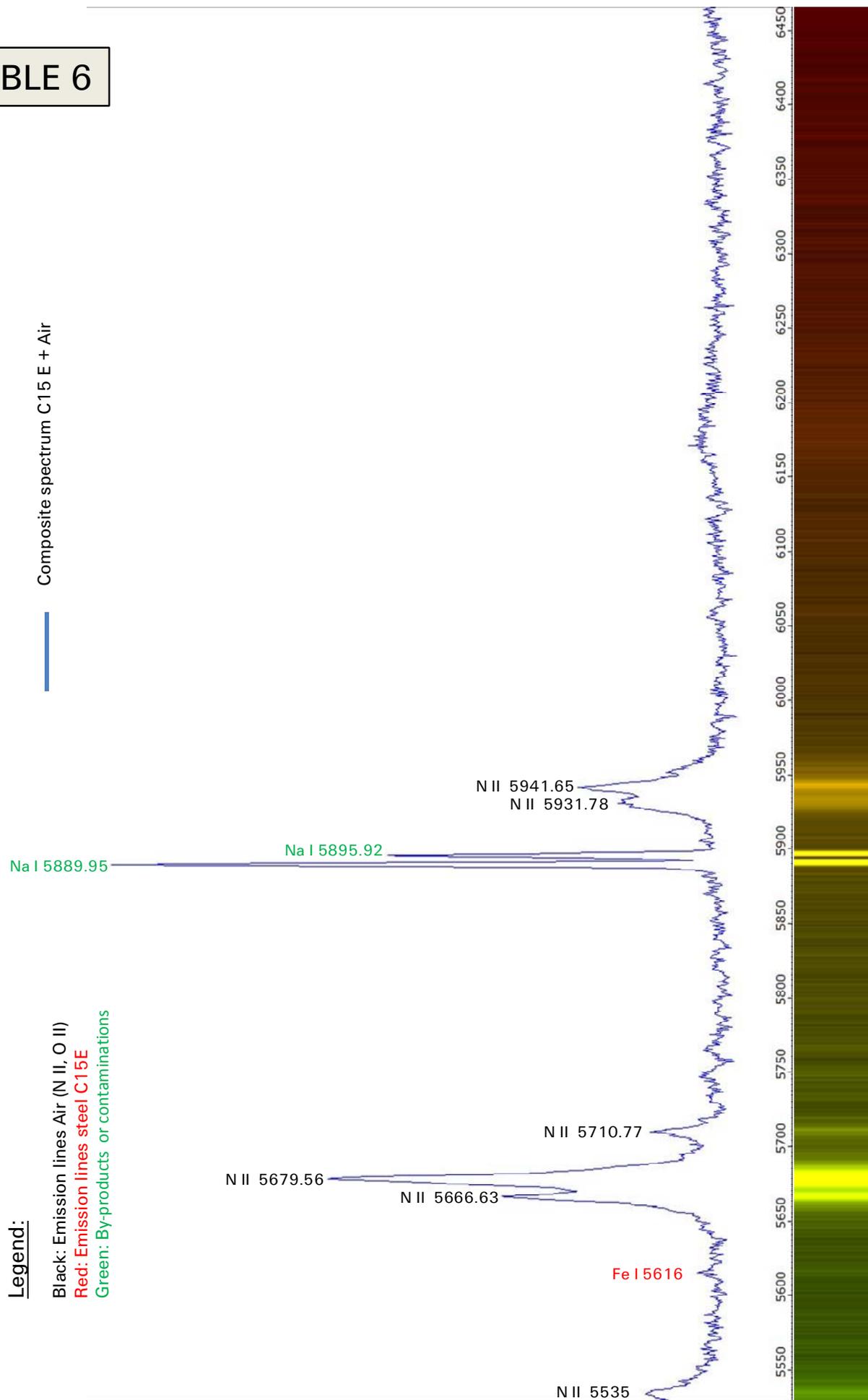


TABLE 6

Composite Spectrum: C15E Steel + Air DADOS 900L/mm



Composite Spectrum: C15E Steel + Air DADOS 900L/mm

TABLE 7

Legend:

Black: Emission lines Air (N II, O II)

Red: Emission lines steel C15E

Green: By-products or contaminations

Composite spectrum C15 E + Air

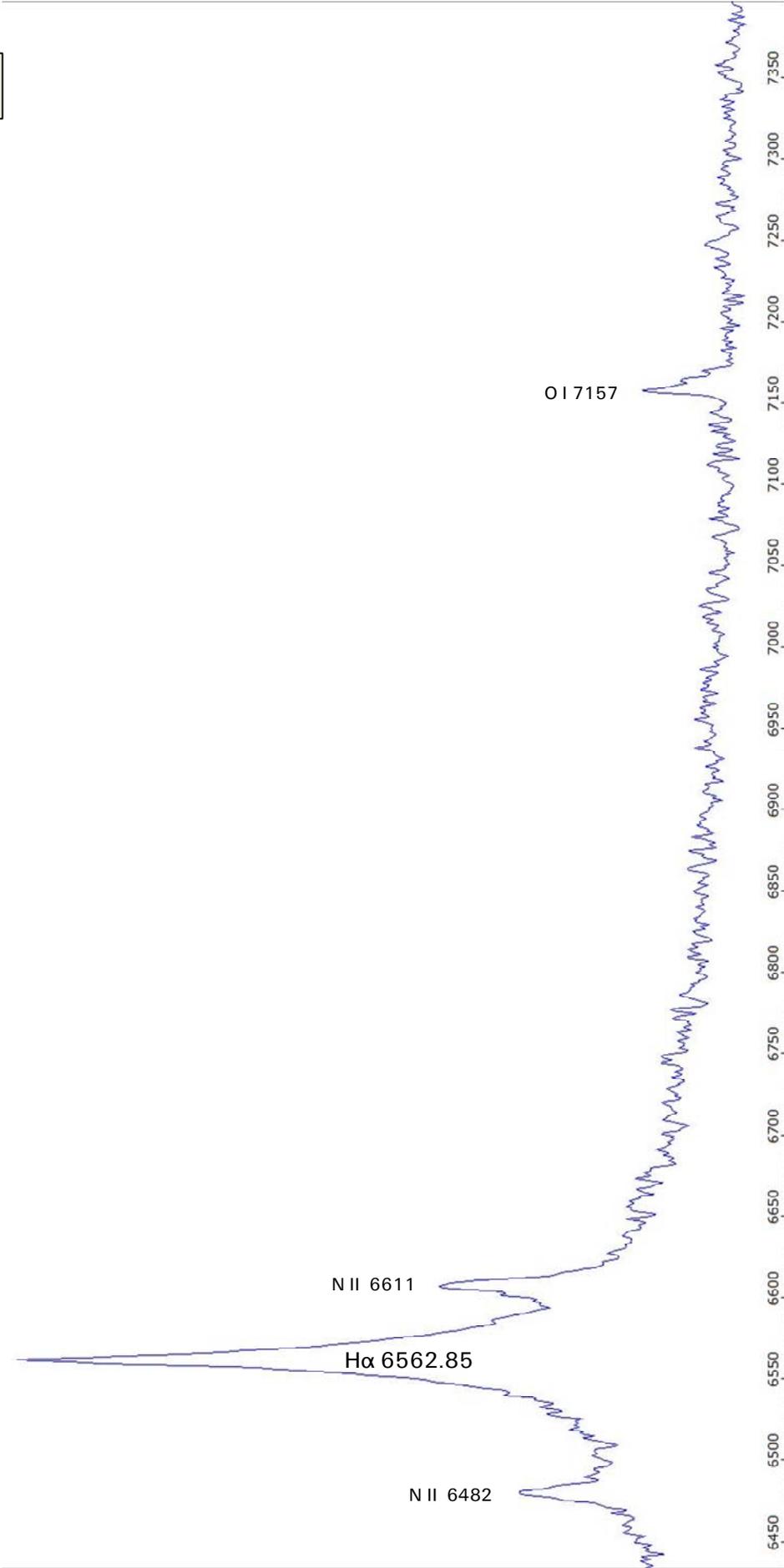


Table 8: Low-resolution (200L) composite spectrum:
Galvanized steel + spark spectrum of air.

Striking are here, as expected, the emission lines of zinc. "Front runner" is clearly the line at 4810.53 Å. In this profile it is clearly noticeable that the emission lines of the air show up relatively modest, particularly the otherwise intense line, N II 5001 – 5007 Å, is missing here even totally. There is also no strong continuum visible. The reason for this effect remains unclear until now. Perhaps the spectrograph slit was accidentally aligned here very precisely to the impact site of the spark.

Table 9: Low-resolution (200L) composite spectrum: aluminum + spark spectrum of air.

Here three aluminum emissions in the blue region of the spectrum are dominant. Also in this profile, it is striking that the emission lines of the air appear relatively modest and a strong continuum is missing. Two clusters with emission lines in the range of 4600 – 4750 Å and 5100 – 5200 Å, can currently not be clearly identified.

Table 10: Low-resolution (200L) composite spectrum: copper wire + spectrum of air.

At first the insulation varnish was removed from the copper wire. Also here a strong continuum is missing. Striking are here three dominant copper lines in the range of 5100 - 5300 Å. Several others are blended with N II and O II emissions, which appear here relatively intensive. Noteworthy are also the by-products, i.e. Na I and Ca II lines.

Table 11: Low-resolution (200L) composite spectrum, recorded with Urs Flükiger in his company in Oberburg, Switzerland. Sample: Tool steel coated with titanium oxide (Ti O₂).

Well visible are here emission bands of diatomic titanium monoxide molecules (TiO) and atomic Ti lines. Titanium dioxide (Ti O₂) at such high temperatures > 3000 K, dissociates to titanium monoxide (Ti O) like in the stellar atmospheres, down to late M-Class.

The air spectrum appears here only weak. See also Table 21, where a section of this spectrum is shown in a montage with the profile of Ras Algethi (α Her). In the area of 5000 Å, however, the leading line of the air spectrum, the intense emission of N II, is blended with the TiO bands.



Because in this experiment the spark had to penetrate the titanium oxide layer, it was correspondingly weaker. Therefore to improve the recognisability of the absorption bands, the exposure time was increased here to 1 minute and the spark was triggered approximately 150 times. If the spark hits an edge or corner of the sample, there is a risk that the coating is here too thin or no longer present. Therefore it has proven to be ideal, when the spark hits the center of the sample at a right angle and the picture is recorded approximately in the surface plane. So it does not matter if the impact point slightly moves.

Remark: Several attempts failed so far, to generate emission lines of gold. In each case only the spectrum of the air molecules was obtained. The reason is still unknown.

Composite Spectrum: Galvanised Steel + Air DADOS 200L/mm

TABLE 8

Legend:
 Black: Emission lines Air (N II, O II)
 Red: Emission lines galvanised steel
 Green: By-products or contaminations

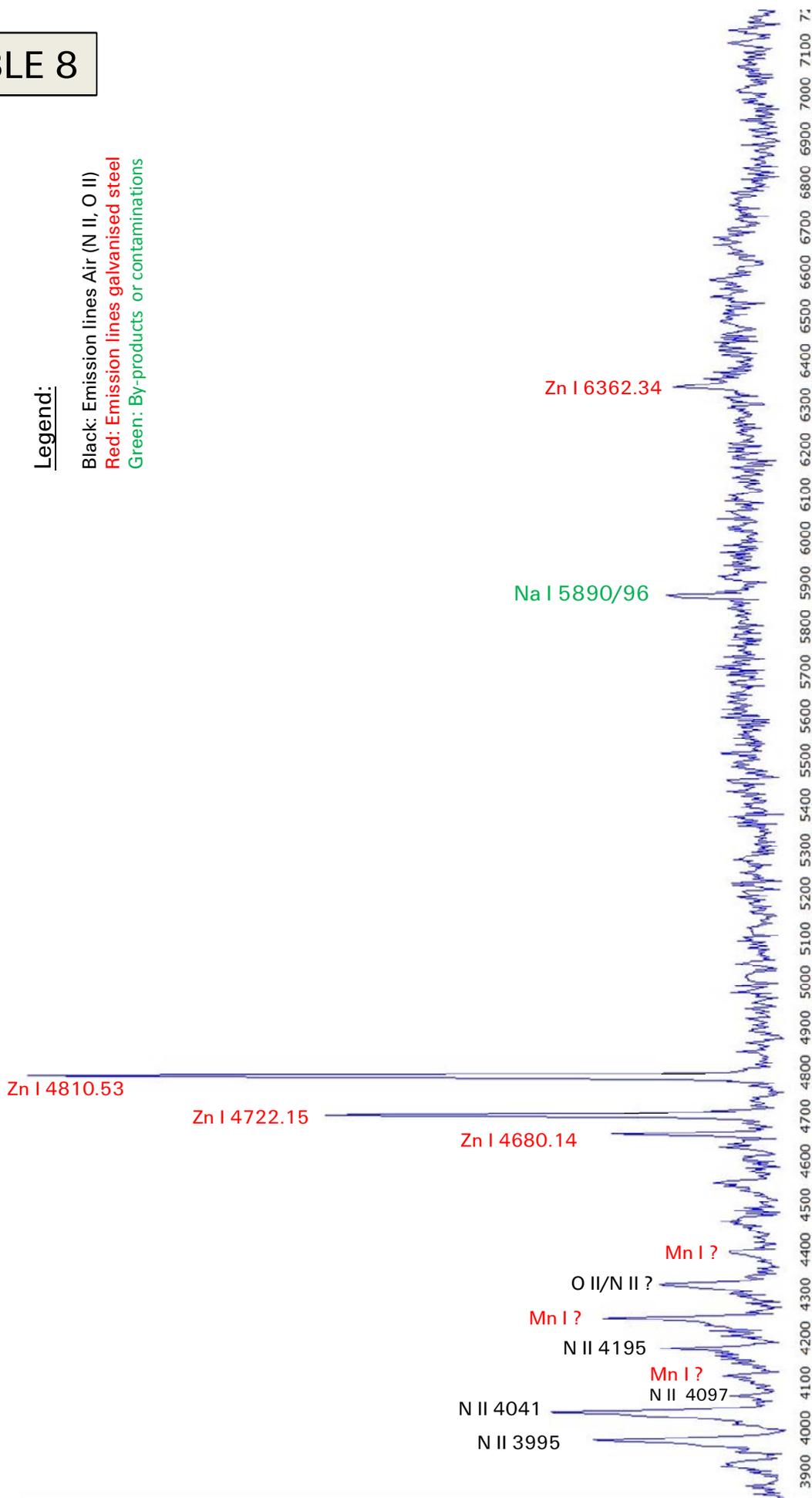


TABLE 9

Composite Spectrum: Aluminum + Air DADOS 200L/mm

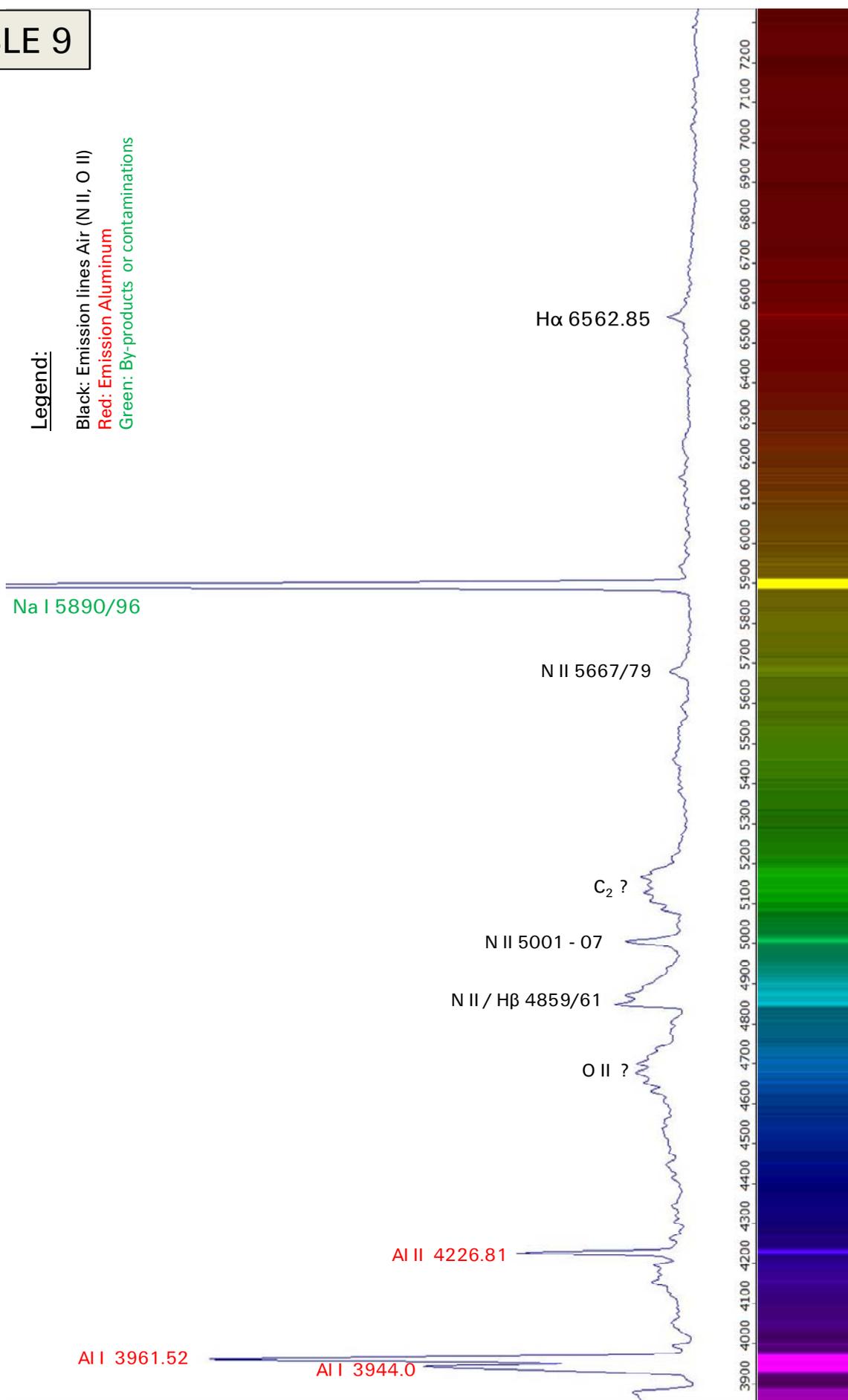


TABLE 10

Composite Spectrum: Copper Wire + Air DADOS 200L/mm

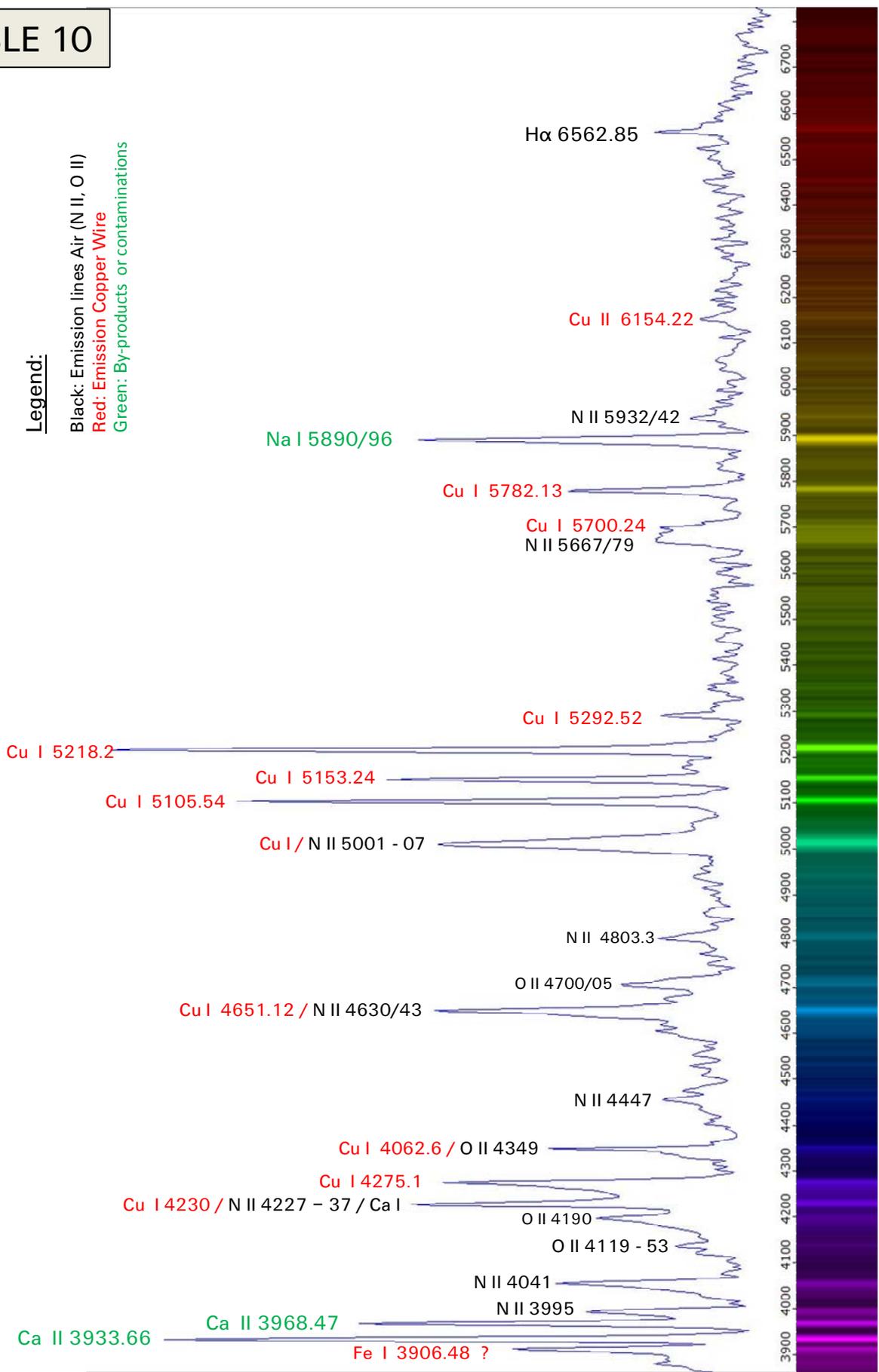


TABLE 11

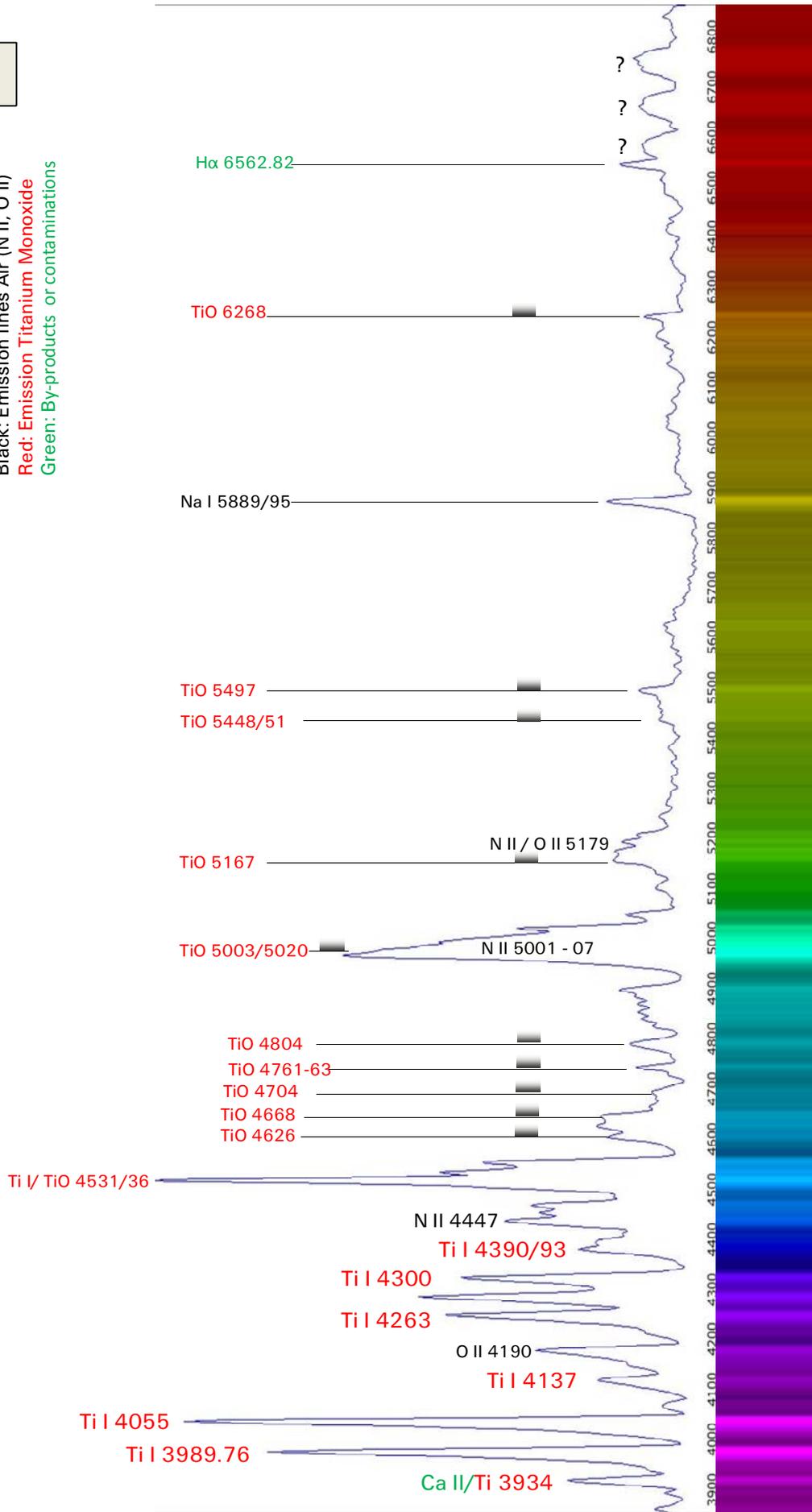
Composite Spectrum: Titanium Monoxide (Ti O) Coating + Air
DADOS 200L/mm

Legend:

Black: Emission lines Air (N II, O II)

Red: Emission Titanium Monoxide

Green: By-products or contaminations



4 Tests with Arc Excitation

4.1 Design and Implementation of the Experiment

Professionally arc spectra are obtained in an inert protective gas atmosphere with electrodes, made of tungsten or spectrally pure high density graphite. In the factory of *Urs Flückiger* in Oberburg Switzerland [61] we have attempted to simulate this rudimentary with an ordinary electric welding machine and a rutile-acide type electrode (Ti O₂). An additional test was carried out with a TIG electrode, combined with argon gas (TIG: Tungsten-Inert-Gas).

Picture: Electric welding with rutile coated electrode. DADOS spektrograph and old VIVITAR macro zoom lens on an AYO mount, distance ca. 3m. Urs Flückiger (left picture in the background), recording the spectra on the laptop.



TIG welding with Argon protective gas (Mr. Baumgartner)



4.2 Results

Table 15: Electric arc, simulated with electric welding machine and rutile-acide type electrode (Ti O₂). Low-resolution (200L) composite spectrum: *steel 37*, materialnumber 1.0037, (herafter called "steel 37") + spectrum of air. The comparison with the *spark spectrum* shows that the *arc spectrum* is resolved somewhat higher. In addition to the lines of Fe I atoms and air molecules here very numerous atomic and molecular emissions of titanium appear (Ti / TiO), obviously originating from the mineral rutile (Ti O₂) of the welding electrode. The distinction between Fe I and Ti I has proven to be difficult and uncertain. These lines are often separated only by a fraction of 1 Å. Also striking is the broad *Na I* emission with a central self-absorption dip, as can be observed in the spectra of high-pressure sodium vapor lamps (see [13]).

Table 16: Electric arc, simulated with electric welding machine, TIG electrode, combined with argon gas. Low-resolution (200L) composite spectrum: *steel 37* + spectrum of air.

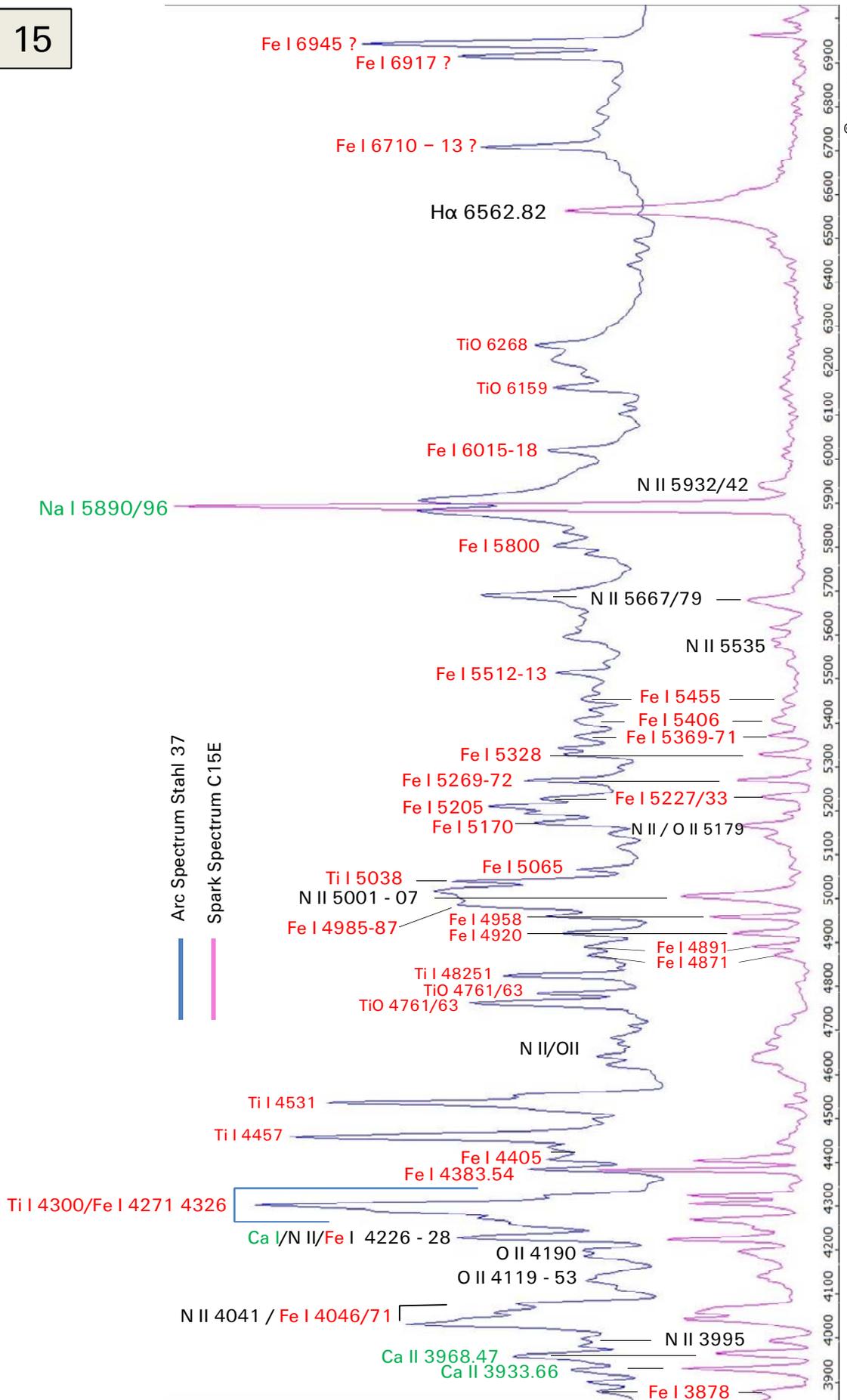
In older welding systems radioactive thoriumoxide improved the ignition. This tungsten electrode is free of radioactive thorium here because Flükiger & Co Ltd applies modern welding equipment and high-voltage ignition with appropriate control system. Consequently, such emission lines are her absent in the spectrum.

The profile is impressively dominated by intensive emissions of argon protective gas Ar I. To make the Fe I lines at all visible, a strong zoom in to the intensity axis was necessary. In the red region of the spectrum, therefore most of the Ar I emission appear cutted. In reality, they are about two to three times as high! Clearly visible here is the protective effect of argon gas. The emissions of the air molecules are weak. The "front runner line" N II at 5000 – 5007 Å is here even completely missing!

TABLE 15

Legend:
 Black: Emission lines Air (N II, O II)
 Red: Emission steel 37/C15E
 Green: By-products or contaminations

Comparison Arc Spectrum Steel 37 versus Spark Spectrum C15E DADOS 200L/mm



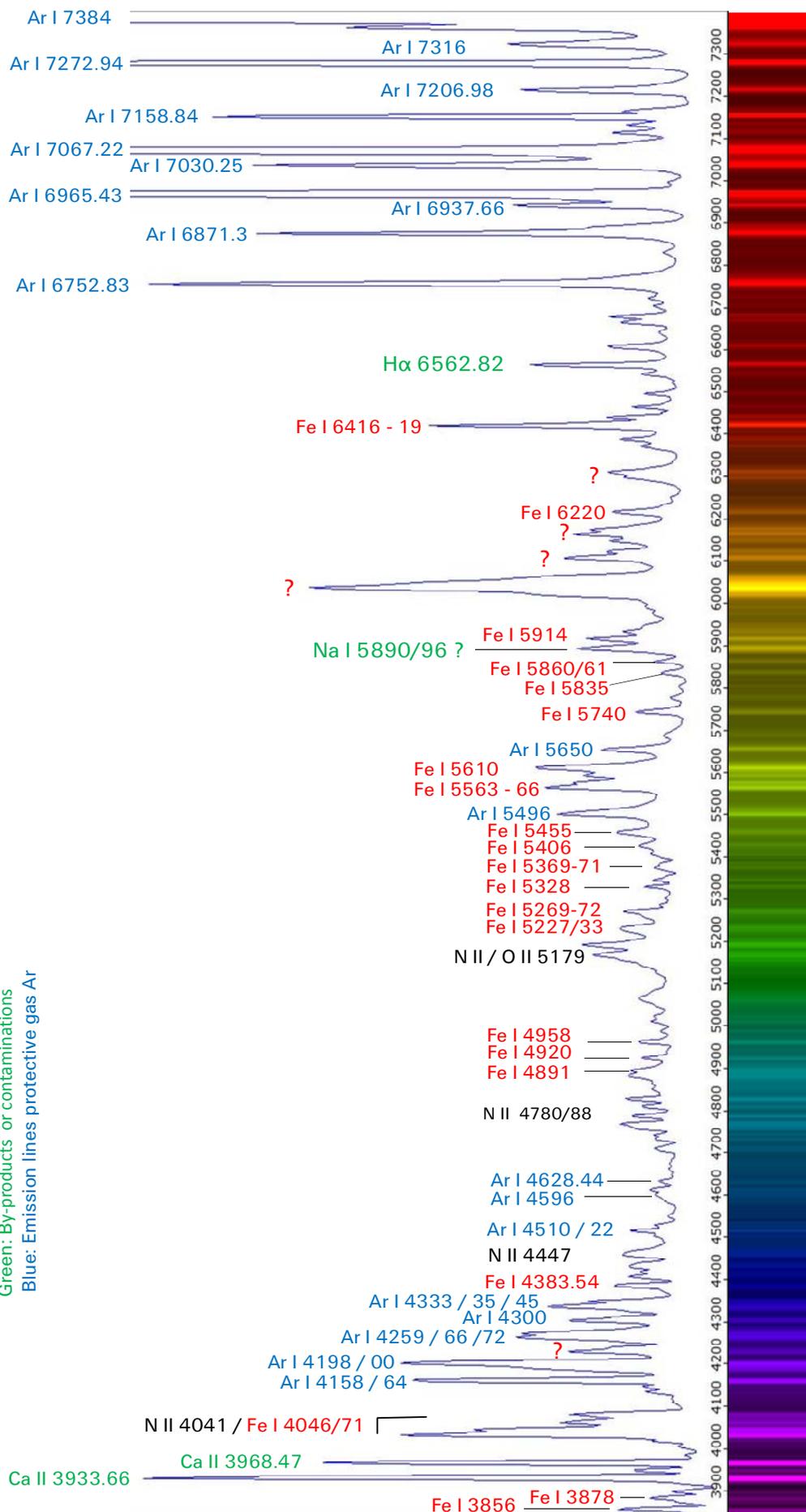
© Richard Walker 2011/03

TABLE 16

Composite Arc Spectrum: Steel 37 + Air DADOS 200L/mm

Legend:

- Black: Emission lines Air (N II, O II)
- Red: Emission steel 37
- Green: By-products or contaminations
- Blue: Emission lines protective gas Ar



5 The Link between the Stellar- and Laboratory Spectra

Table 20:

Shows in the blue range the comparison of the higher resolution (900L) composite spectrum of C15E (according to Table 4) and the solar spectrum. All Fe I and Ca II lines of the spark spectrum, can be clearly identified in the solar spectrum. Several iron lines in the spark spectrum are overprinted here by the much more intensive emissions of the air molecules (O II, N II) and therefore not available for a link to the solar spectrum.

Table 21:

Profile comparison, range from 4650 - 5900 Å, titanium oxide bands (TiO) (200L grating):

– Spark spectrum TiO emission bands of a surface coating (according to Table 11)

versus

– Stellar TiO absorption bands of *Ras Algethi* (α Her).

In this wavelength range in the spectrum of the surface coating appear clearly visible emissions of TiO bands. The intensity runs approximately mirror inverted to the characteristic absorption bands of the late M-class star α Her. The emissions of air molecules are here relatively weak. At 5000 Å however, N II forms an intense blend with the TiO bands.

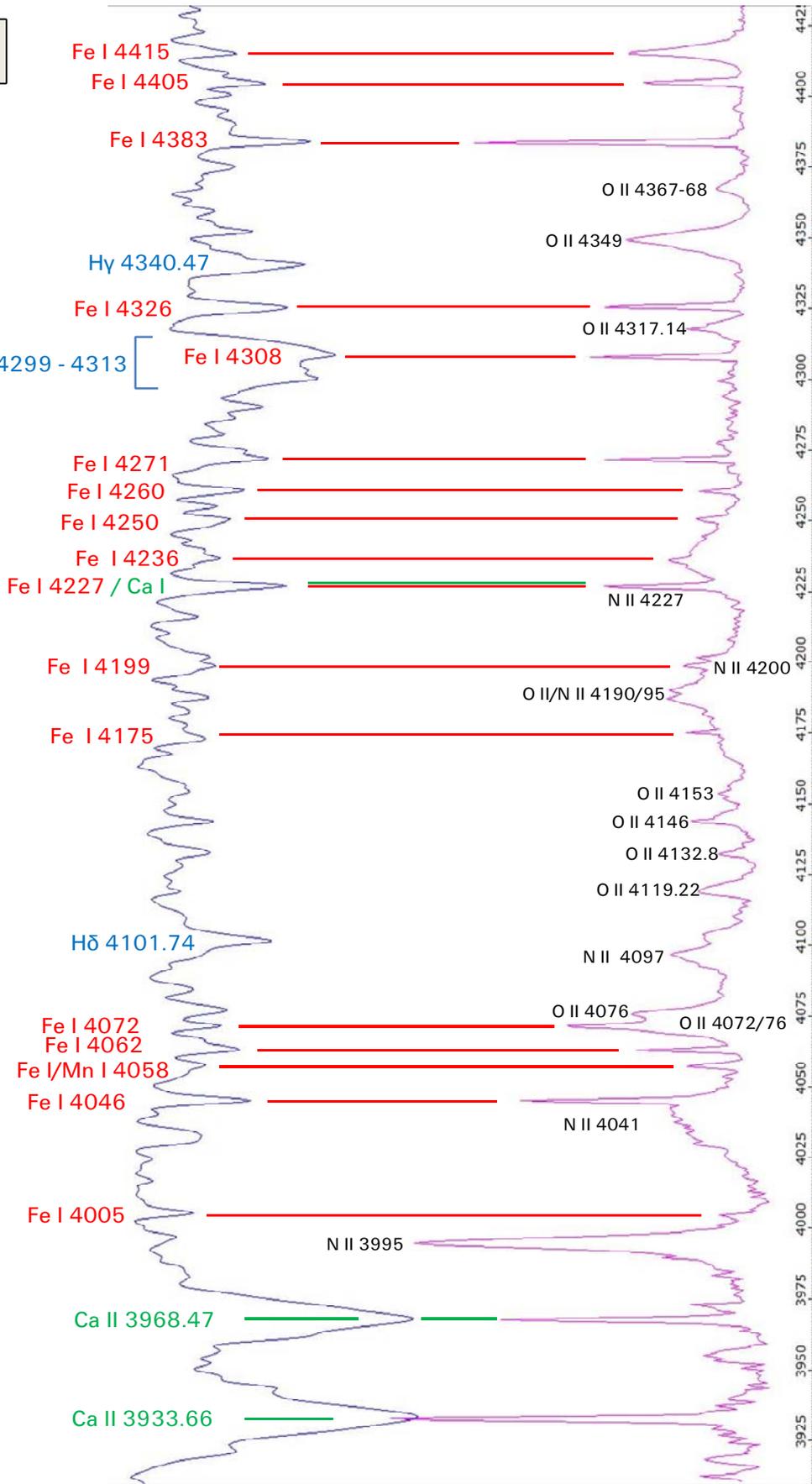
Comparison of Spectra: Sun versus C15E (+ Air) 900L/mm

TABLE 20

Legend:

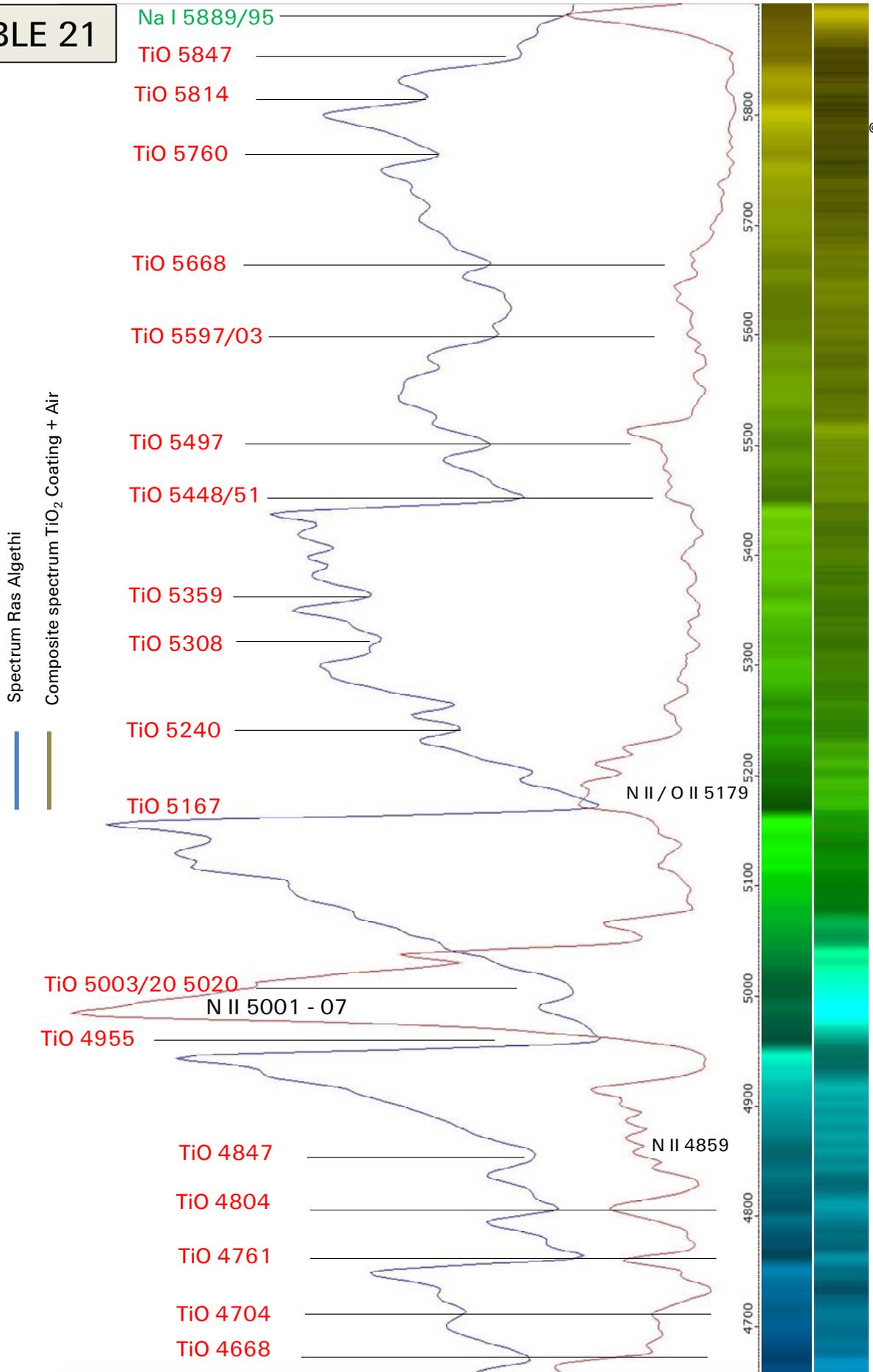
- Black: Emission lines Air (N II, O II)
- Red: Emission steel C15E
- Green: By-products or contaminations
- Blue: Absorption lines spectrum of the sun

— Spectrum of the Sun
 — Composite spectrum C15 E + Air



Comparison TiO₂ Coating and Ras Algethi (α Her M5 Ib-II) 200L/mm

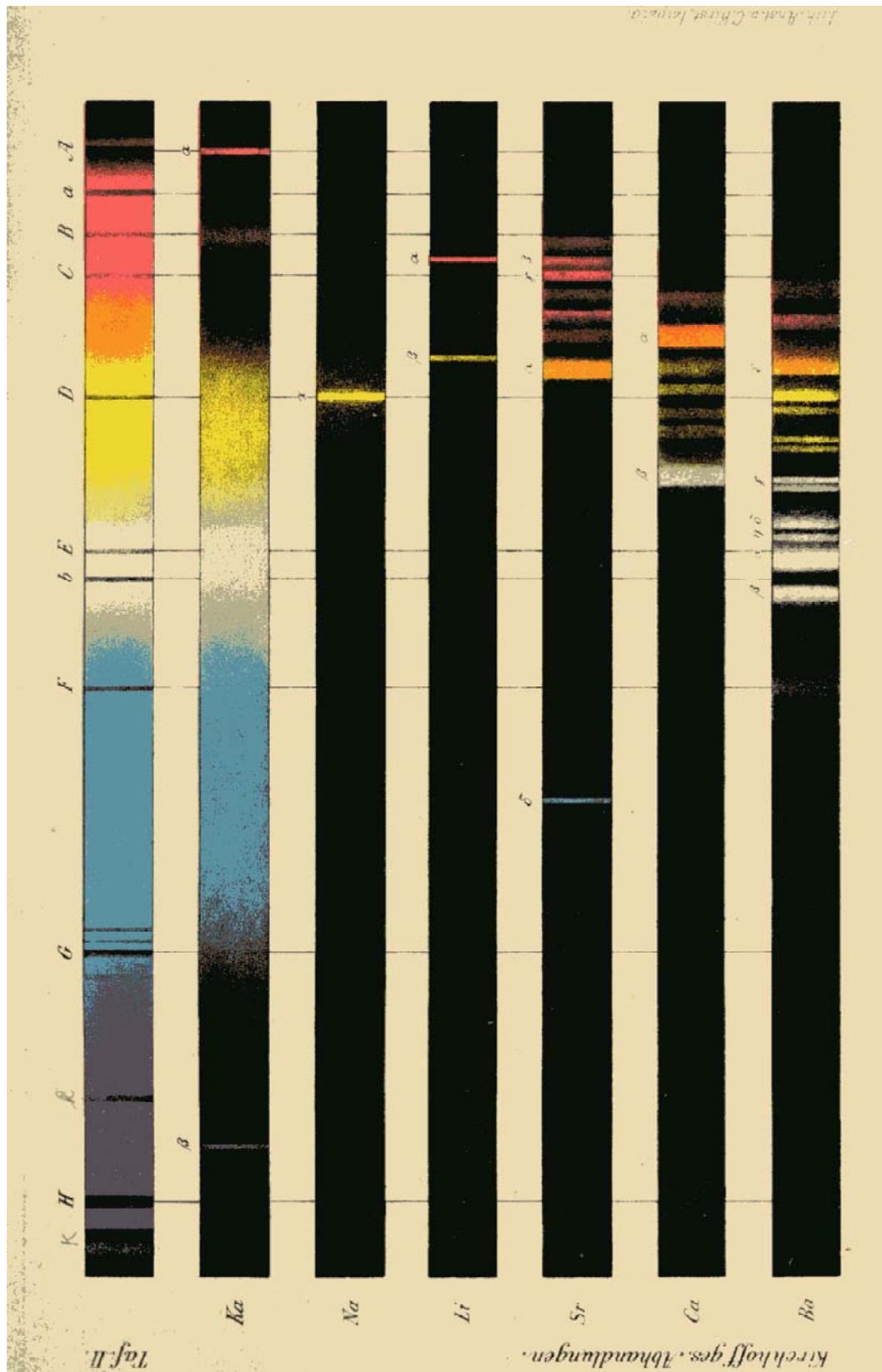
TABLE 21



6 Appendix

6.1 Kirchhoff's hand-drawn Spectra [20].

The Fraunhofer absorption lines of the sun (top line) compared to the emission spectra of the elements *Ka* (today *K*), *Na*, *Li*, *Sr*, *Ca* und *Ba*.



6.2 Vatican Spectral Atlases of Elements and Molecules

The Vatican Observatory in 1930-40 published several fundamental works with spark and arc spectra, continuing the tradition of the legendary Father Secchi, who wrote in the 19th century the first stellar spectroscopic atlas. The driving force behind this project was Father Alois Gatterer. The following pictures are from the antiquarian book *J. Ricerche Spettroscopiche*, from J. Junkes and E.W. Saltpeter [1].



ABB. 3. Spektrographensaal 1934 mit STEINHEIL-Spektrograph GH mit langer Kamera

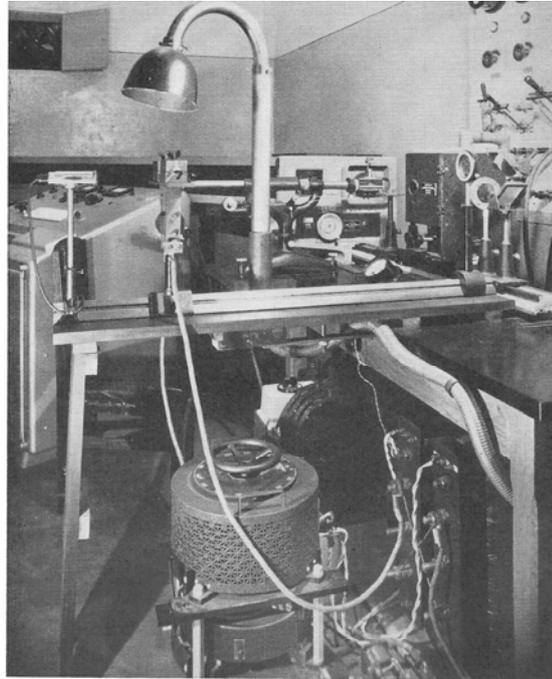


ABB. 20. Anordnung zur Aufnahme von Bandenspektren mit der Kohleflamme vor dem JARRELL-ASH-Spektrographen

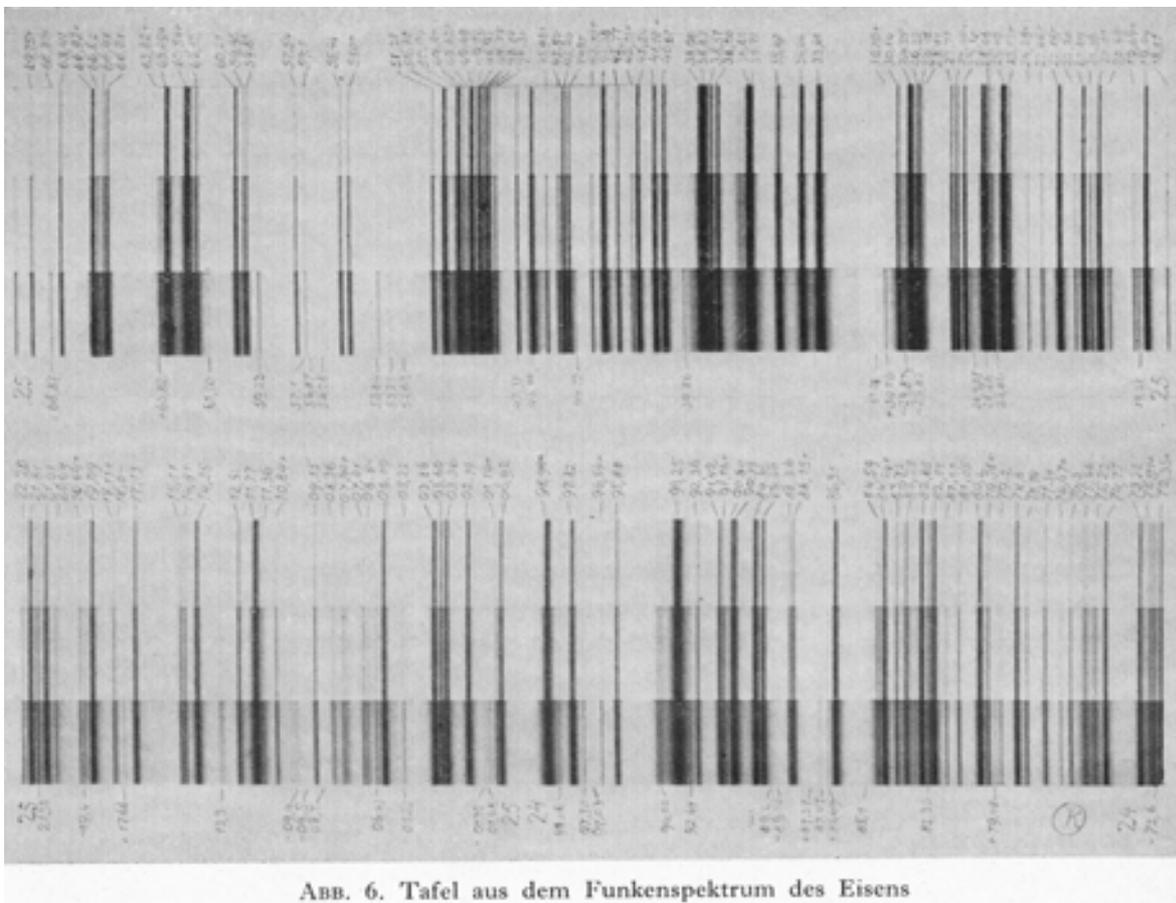


ABB. 6. Tafel aus dem Funkenspektrum des Eisens

7 Literature and Internet

Literature:

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Author:

The following scripts on the subject are downloadable under this link (some of them in german):
<http://www.ursusmajor.ch/astrospektroskopie/richard-walkers-page/index.html>

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<http://bmauclaire.free.fr/astronomie/spectro/simulation/noblet/index2.htm>

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<http://www.fluekiger.ch/>

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