

# ELEMENTAL ANALYSIS OF WELDS WITH AN X-RAY FLUORESCENCE ANALYSER

## ELEMENTARNA ANALIZA ZVARNEGA SPOJA Z RENTGENSKO FLUOROSCENTNIM ANALIZATORJEM

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**Keywords:** HSLA steel, elemental analysis, welds, x-ray fluorescence analyser

### **Abstract**

High-strength low-alloyed (HSLA) steels are often used as an advanced material for the construction of multi-pass welded joints. Chemical analyses of welds are performed with X-ray fluorescence spectrometry (XRF) using an X-ray fluorescence spectrometer (Thermo Scientific Niton XL3t GOLD+).

The aim of this paper is to design a measuring table for the x-ray fluorescence analyser. At the end of the research, the experimental results measured with the table are compared with the values obtained from manual measurements.

### **Povzetek**

Visokotrdnostna mikrolegirana (VTML) jekla so pogosto uporabljena kot moderna jekla za gradnjo večvarkovnih zvarnih spojev. Kemijska analiza zvarov je izvedena z rentgenskim fluorescentnim analizatorjem XRF (Thermo Scientific Niton XL3t GOLDD+).

Namen članka je konstrukcija merilne mizice za rentgenski fluorescentni analizator. Na koncu raziskave so eksperimentalni rezultati, izmerjeni na mizici, primerjani z vrednostmi, ki so bile izmerjene ročno.

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## 1 INTRODUCTION

Welding is a process in which two or more pieces of material are joined and is achieved by pressure, heat, with a combination of both, with or without added material, [1-8].

During the process of welding, various problems occur due to weather and mechanical influences. Due to the growing needs and requirements of the market, improvements and the provision of perfect welds are constantly needed, [8-16].

To ensure complete welds, the x-ray fluorescent analyser can be used, which also aids in the analysis of welds. In the survey, the Niton Gold+ analyser will be used, which is a manual, compact, high-performing, portable device for the x-ray analysis of elements. With the help of the device, the welds will be analysed with two procedures. One is that the device is handheld; the second procedure is the creation of a small support panel installed in the device, thus enabling the performance of the measurements. After completion of the measurement, the results of the chemical analysis will be compared.

## 2 EXPERIMENTAL PROCEDURE

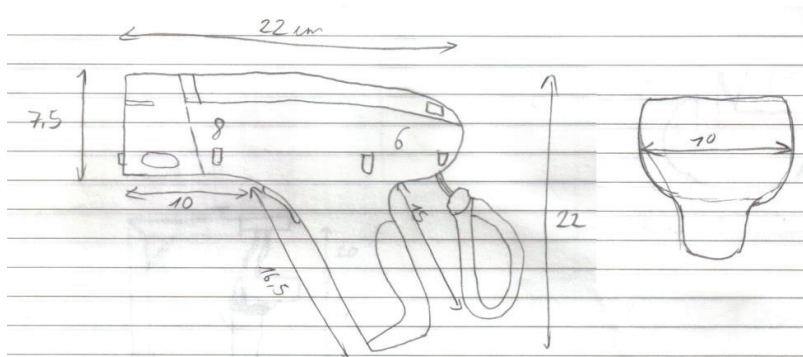
Our goal was to make the product (support panel or table) for the Niton device so that we would be able to make hands-free measurements. The idea was to make the product more useful and as reliable as possible so that it could be used by anyone. We also wanted the panel to have a benchmark so that measurements could be performed several times, ensuring minor deviations. Our purpose was that with the help of a panel we could control the device with a PC, which would be significantly easier for the user and for the subsequent processing of the results.

To make this product, we first needed the dimensions of the Niton apparatus. With the help of clay and the case of the apparatus (Figure 1), we attempted to obtain the shape of the device, but it turned out that the case was bigger and of a different shape than the device, so this approach was unhelpful.

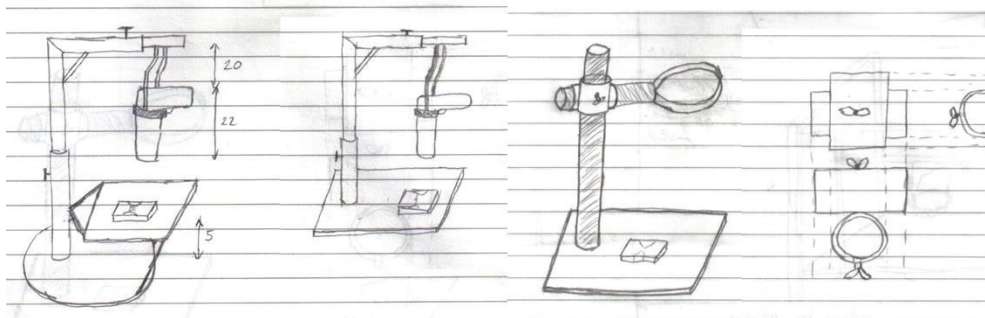


**Figure 1:** Model obtained by using the clay

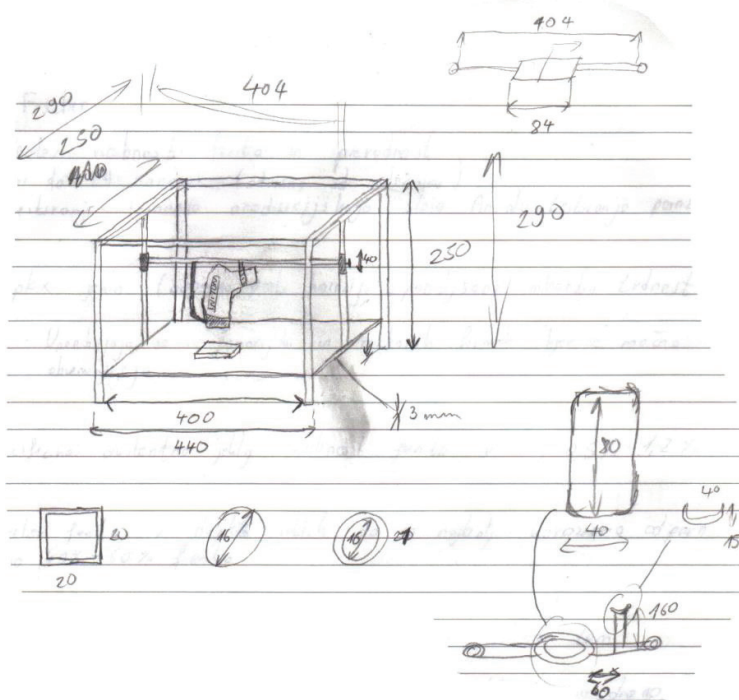
Next, we obtained the dimensions directly from the device, using a calliper (Figure 2). Due to the unusual shape of the device, we had difficulties obtaining measurements. Next, we copied the shape of the device. Initially, the idea was to make a stand, following the idea from Figure 3. After a couple of considerations, this concept turned out to be inappropriate, since the device would be attached to only one side, and therefore would not be sufficiently stable. After consultation, we had the idea of making the product similar to as shown in Figure 4. Thus, we have achieved greater stability of the device, since the device was attached to two sides. We had to take additional measurements for brackets for a perfect fitting of the device (Figure 5).



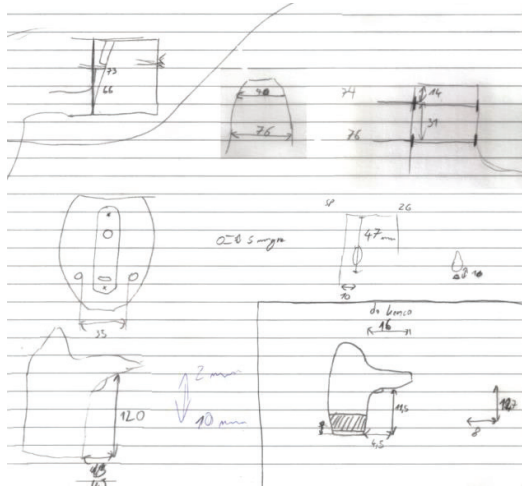
**Figure 2:** Sketch of dimensions of the Niton apparatus



**Figure 3:** Sketch of the stand



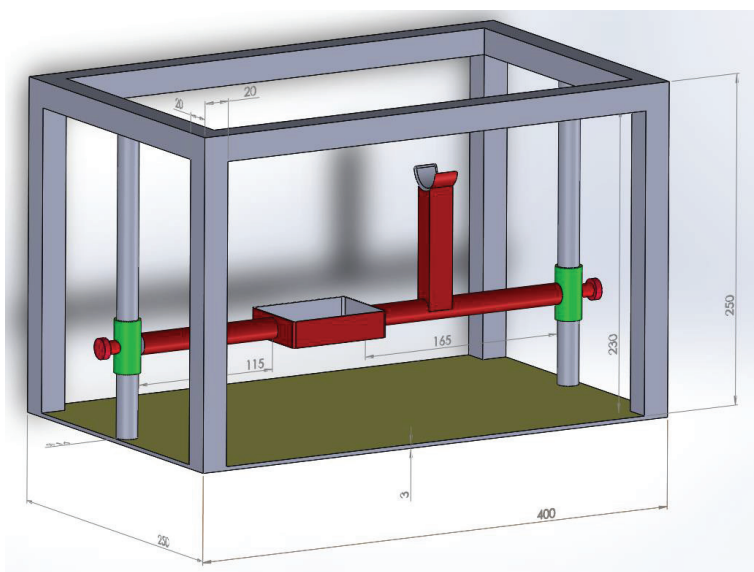
**Figure 4:** Sketch of the support panel with dimensions



**Figure 5:** Dimensions of the device where it is attached to the stand

Our technological requirements were that the support panel should be stable and rigid and perfectly fit the device. We also wanted it to be as easy as possible to use. In choosing the material, we focused on strength. We opted for stainless steel, because it is solid and does not require additional protection against oxidation. Parts of the support panel, where the device and bracket are in contact, were cushioned for the device to fit better into the panel. Thus, the device was also protected against unwanted abrasions. On the bottom of the panel is a 3-mm-thick plate of stainless steel, with an attached benchmark that enables more accurate positioning of the measured object.

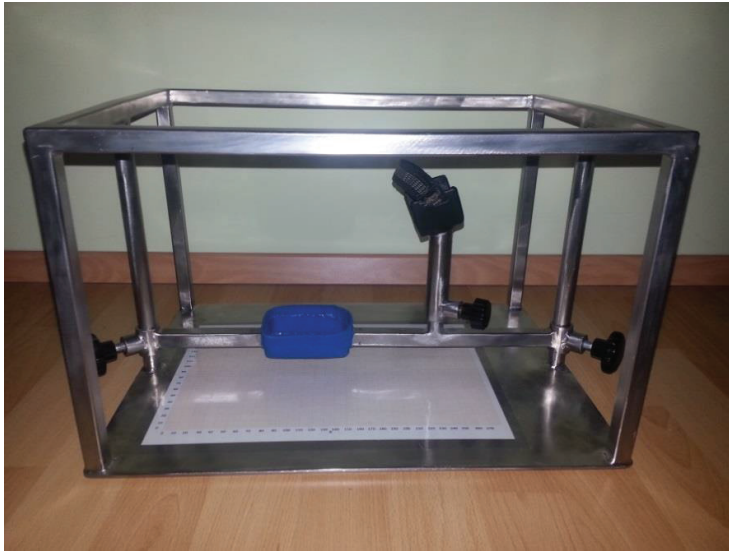
When the support panel was complete, welds were cleaned with Antox 71 E acid and rinsed with water. In the end, the panel was polished with polish paste. At the bottom of the panel, we attached an A4 millimetre scale with printed units, which was protected with self-adhesive foil. The complete panel is shown in Figure 8.



**Figure 6:** Plan of the panel made with the program SolidWorks



**Figure 7:** Welded panel



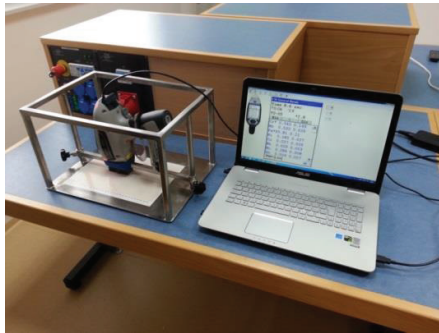
*Figure 8: Completed panel*

### 3 RESULTS AND DISCUSSION

In the research, we used two different measurement procedures. Measurements were carried out manually and with the use of the support panel presented in the previous chapter. Each measurement was repeated three times, and later the average value calculated. Each measurement was done for one minute. The manual measurement is shown in Figure 9, and the measurement with the help of the support panel is shown in Figure 10.

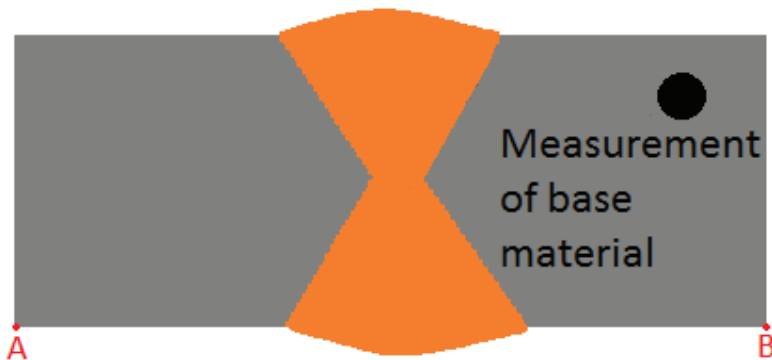


*Figure 9: Manual measurement*



**Figure 10:** *Measurement with the help of the panel*

Figure 11 shows the weld and location of the first measurement, designated with a black dot. The first measurements were done on basic material, which did not change its properties after welding. Points A and B represent the location of the measured object on the instrument when measuring with the aid of a panel. With the help of these points, we can later duplicate the measurements. Point A has coordinates (80 mm, 85 mm), and Point B (147 mm, 85 mm).



**Figure 11:** *Measurement of base material*

Tables 1 and 2 show the results of the first measurements of the basic material. Table 1 shows the results obtained with the help of the panel; the results in Table 2 were obtained with manual measurements. Each measurement was repeated three times in a row. We made excerpts from both tables and presented them as a percentage. From all three measurements, we then calculated the average of each element and the maximum and minimum deviation from those values. In Table 3, we copied the deviated values for a better overview. Thus, it is clearly shown with which measurement technique the deviations were smaller. Those deviations that were smaller in the manual mode of measurement have been tagged in red.

**Table 1:** Results of measurements of the basic material obtained with the panel

| With the panel | Mo (%) | Nb (%) | Cu (%) | Ni (%) | Fe (%)  | Mn (%) | Cr (%) | Si (%) |
|----------------|--------|--------|--------|--------|---------|--------|--------|--------|
| Measurement 1  | 0.2730 | 0.0300 | 0.2220 | 0.1450 | 97.7240 | 0.4960 | 0.5530 | 0.3820 |
| Measurement 2  | 0.2720 | 0.0300 | 0.2410 | 0.1250 | 97.8570 | 0.5140 | 0.5460 | 0.3960 |
| Measurement 3  | 0.2710 | 0.0280 | 0.2430 | 0.1710 | 97.8810 | 0.4440 | 0.5480 | 0.4010 |
| Average        | 0.2720 | 0.0293 | 0.2353 | 0.1470 | 97.8207 | 0.4847 | 0.5490 | 0.3930 |
| Deviations +   | 0.0010 | 0.0007 | 0.0077 | 0.0240 | 0.0603  | 0.0293 | 0.0040 | 0.0080 |
| Deviations -   | 0.0010 | 0.0013 | 0.0133 | 0.0220 | 0.0967  | 0.0407 | 0.0030 | 0.0110 |

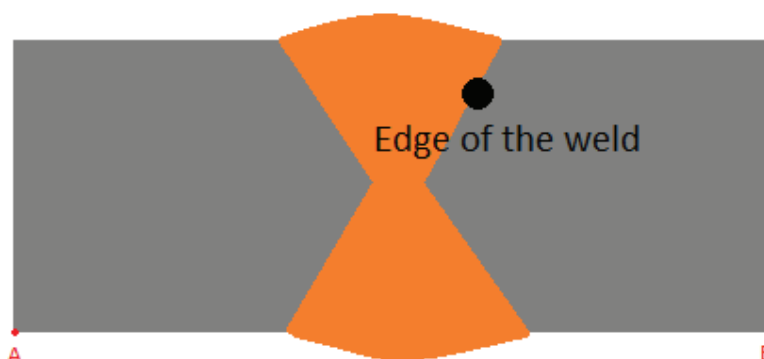
**Table 2:** Results of measurements of the basic material obtained by manual measurements

| Manually      | Mo (%) | Nb (%) | Cu (%) | Ni (%) | Fe (%)  | Mn (%) | Cr (%) | Si (%) |
|---------------|--------|--------|--------|--------|---------|--------|--------|--------|
| Measurement 1 | 0.2770 | 0.0280 | 0.2260 | 0.1700 | 97.7230 | 0.4870 | 0.5520 | 0.3660 |
| Measurement 2 | 0.2750 | 0.0300 | 0.2410 | 0.1450 | 97.8470 | 0.5350 | 0.5230 | 0.3910 |
| Measurement 3 | 0.2680 | 0.0290 | 0.2320 | 0.1670 | 97.8690 | 0.5060 | 0.5220 | 0.3760 |
| Average       | 0.2733 | 0.0290 | 0.2330 | 0.1607 | 97.8130 | 0.5093 | 0.5323 | 0.3777 |
| Deviations +  | 0.0037 | 0.0010 | 0.0080 | 0.0063 | 0.0560  | 0.0257 | 0.0197 | 0.0133 |
| Deviations -  | 0.0053 | 0.0010 | 0.0070 | 0.0157 | 0.0900  | 0.0223 | 0.0103 | 0.0117 |

**Table 3:** Deviations of measurements of the basic material

|                |          | Mo (%) | Nb (%) | Cu (%) | Ni (%) | Fe (%) | Mn (%) | Cr (%) | Si (%) |
|----------------|----------|--------|--------|--------|--------|--------|--------|--------|--------|
| With the panel | Deviat + | 0.0010 | 0.0007 | 0.0077 | 0.0240 | 0.0603 | 0.0293 | 0.0040 | 0.0080 |
|                | Deviat - | 0.0010 | 0.0013 | 0.0133 | 0.0220 | 0.0967 | 0.0407 | 0.0030 | 0.0110 |
| Manually       | Deviat + | 0.0037 | 0.0010 | 0.0080 | 0.0063 | 0.0560 | 0.0257 | 0.0197 | 0.0133 |
|                | Deviat - | 0.0053 | 0.0010 | 0.0070 | 0.0157 | 0.0900 | 0.0223 | 0.0103 | 0.0117 |

Figure 12 shows the weld and the location of the second measurement marked with a black dot. Other measurements were performed at the edge of the weld where basic and added material are blended and are thermally treated. Points A and B represent the location of measurement with the use of the panel. With the help of these points, we were able to repeat the measurements. Point A coordinates are (68 mm, 78 mm), Point B (135 mm, 78 mm).

**Figure 12:** Measurement at the edge of the weld

In Tables 4 and 5, the results of measurements on the edge of the weld are shown. In Table 4, the results are obtained with the help of the panel; in Table 5, the results are obtained by manual measurements. Each measurement was repeated three times in a row. From both tables, we excerpted the results of the measured elements that are presented as a percentage. From all three measurements, we then calculated the average of each element and the maximum and minimum deviations from those values. In Table 6, we copied deviated values for a better overview. Thus, it is clearly shown at which measurement technique deviations were smaller. We have tagged those deviations that were smaller in the manual mode of measurement in red.

**Table 4:** Results of measurements on the edge of the weld obtained with the panel

| With the panel | Mo (%) | Nb (%) | Cu (%) | Ni (%) | Fe (%)  | Mn (%) | Cr (%) | Si (%) |
|----------------|--------|--------|--------|--------|---------|--------|--------|--------|
| Measurement 1  | 0.1680 | 0.0180 | 0.1650 | 0.1290 | 97.3930 | 0.9630 | 0.3440 | 0.4140 |
| Measurement 2  | 0.1680 | 0.0200 | 0.1570 | 0.1470 | 97.7240 | 0.9490 | 0.3490 | 0.4620 |
| Measurement 3  | 0.1650 | 0.0180 | 0.1470 | 0.1230 | 97.3870 | 0.9660 | 0.3430 | 0.4370 |
| Average        | 0.1670 | 0.0187 | 0.1563 | 0.1330 | 97.5013 | 0.9593 | 0.3453 | 0.4377 |
| Deviations +   | 0.0010 | 0.0013 | 0.0087 | 0.0140 | 0.2227  | 0.0067 | 0.0037 | 0.0243 |
| Deviations -   | 0.0020 | 0.0007 | 0.0093 | 0.0100 | 0.1143  | 0.0103 | 0.0023 | 0.0237 |

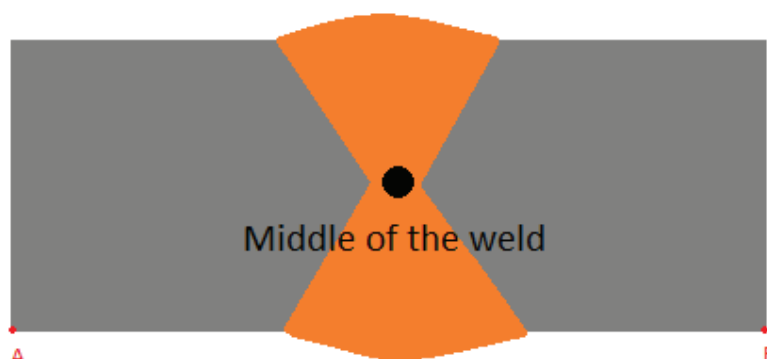
**Table 5:** Results of measurements on the edge of the weld obtained by manual measurements

| Manually      | Mo (%) | Nb (%) | Cu (%) | Ni (%) | Fe (%)  | Mn (%) | Cr (%) | Si (%) |
|---------------|--------|--------|--------|--------|---------|--------|--------|--------|
| Measurement 1 | 0.1680 | 0.0180 | 0.1600 | 0.1640 | 97.3390 | 0.9860 | 0.3290 | 0.4160 |
| Measurement 2 | 0.1840 | 0.0180 | 0.1760 | 0.1550 | 97.3280 | 0.8990 | 0.3730 | 0.4360 |
| Measurement 3 | 0.1770 | 0.0180 | 0.1820 | 0.1570 | 97.3200 | 0.9150 | 0.3670 | 0.4330 |
| Average       | 0.1763 | 0.0180 | 0.1727 | 0.1587 | 97.3290 | 0.9333 | 0.3563 | 0.4283 |
| Deviations +  | 0.0077 | 0.0000 | 0.0093 | 0.0053 | 0.0100  | 0.0527 | 0.0167 | 0.0077 |
| Deviations -  | 0.0083 | 0.0000 | 0.0127 | 0.0037 | 0.0090  | 0.0343 | 0.0273 | 0.0123 |

**Table 6:** Deviations of measurements on the edge of the weld

|                |          | Mo (%) | Nb (%) | Cu (%) | Ni (%) | Fe (%) | Mn (%) | Cr (%) | Si (%) |
|----------------|----------|--------|--------|--------|--------|--------|--------|--------|--------|
| With the panel | deviat + | 0.0010 | 0.0013 | 0.0087 | 0.0140 | 0.2227 | 0.0067 | 0.0037 | 0.0243 |
|                | deviat - | 0.0020 | 0.0007 | 0.0093 | 0.0100 | 0.1143 | 0.0103 | 0.0023 | 0.0237 |
| Manually       | deviat + | 0.0077 | 0.0000 | 0.0093 | 0.0053 | 0.0100 | 0.0527 | 0.0167 | 0.0077 |
|                | deviat - | 0.0083 | 0.0000 | 0.0127 | 0.0037 | 0.0090 | 0.0343 | 0.0273 | 0.0123 |

Figure 13 shows the weld and the location of the third measurement marked with a black dot. The third measurements were performed in the middle of the weld. Points A and B represent the location of the measurement with the use of the panel. With the help of these points, we were able to repeat the measurements. Point A coordinates are (64 mm, 84 mm) and Point B coordinates are (131 mm, 84 mm).

**Figure 13:** Measurement in the middle of the weld

In Tables 7 and 8, the results of the third measurement in the middle of the weld are shown. In Table 7, the results obtained with the help of the panel are shown; in Table 8, the results obtained with manual measurements are shown. Each measurement was repeated three times in a row. From both tables, we excerpted the results of the measured elements that are presented as a percentage. From all three measurements, we then calculated the average of each element and the maximum and minimum deviations from those values. In Table 9, we copied deviated values for a better overview. Thus, it is clearly shown at which measurement technique deviations were smaller. In red, we have tagged those deviations that were smaller in the manual mode of measurement.

**Table 7:** Results of measurements in the middle of the weld obtained with the panel

| With the panel | Mo (%) | Nb (%) | Cu (%) | Fe (%)  | Mn (%) | Cr (%) | Si (%) |
|----------------|--------|--------|--------|---------|--------|--------|--------|
| Measurement 1  | 0.0210 | 0.0030 | 0.0670 | 97.1730 | 1.7270 | 0.0660 | 0.4050 |
| Measurement 2  | 0.0190 | 0.0040 | 0.0430 | 97.2700 | 1.7150 | 0.0650 | 0.4310 |
| Measurement 3  | 0.0210 | 0.0030 | 0.0460 | 97.2230 | 1.7420 | 0.0660 | 0.4100 |
| Average        | 0.0203 | 0.0033 | 0.0520 | 97.2220 | 1.7280 | 0.0657 | 0.4153 |
| Deviations +   | 0.0007 | 0.0007 | 0.0150 | 0.0480  | 0.0140 | 0.0003 | 0.0157 |
| Deviations -   | 0.0013 | 0.0003 | 0.0090 | 0.0490  | 0.0130 | 0.0007 | 0.0103 |

**Table 8:** Results of measurements in the middle of the weld obtained by manual measurements

| Manually      | Mo (%) | Nb (%) | Cu (%) | Fe (%)  | Mn (%) | Cr (%) | Si (%) |
|---------------|--------|--------|--------|---------|--------|--------|--------|
| Measurement 1 | 0.0130 | 0.0030 | 0.0660 | 97.1890 | 1.8040 | 0.0610 | 0.4310 |
| Measurement 2 | 0.0140 | 0.0040 | 0.0530 | 97.2420 | 1.7840 | 0.0570 | 0.4140 |
| Measurement 3 | 0.0150 | 0.0030 | 0.0380 | 97.2450 | 1.7440 | 0.0570 | 0.4330 |
| Average       | 0.0140 | 0.0033 | 0.0523 | 97.2253 | 1.7773 | 0.0583 | 0.4260 |
| Deviations +  | 0.0010 | 0.0007 | 0.0137 | 0.0197  | 0.0267 | 0.0027 | 0.0070 |
| Deviations -  | 0.0010 | 0.0003 | 0.0143 | 0.0363  | 0.0333 | 0.0013 | 0.0120 |

**Table 9: Deviations of measurements in the middle of the weld**

|                |              | Mo (%) | Nb (%) | Cu (%) | Fe (%) | Mn (%) | Cr (%) | Si (%) |
|----------------|--------------|--------|--------|--------|--------|--------|--------|--------|
| With the panel | deviations + | 0.0007 | 0.0007 | 0.0150 | 0.0480 | 0.0140 | 0.0003 | 0.0157 |
|                | deviations - | 0.0013 | 0.0003 | 0.0090 | 0.0490 | 0.0130 | 0.0007 | 0.0103 |
| Manually       | deviations + | 0.0010 | 0.0007 | 0.0137 | 0.0197 | 0.0267 | 0.0027 | 0.0070 |
|                | deviations - | 0.0010 | 0.0003 | 0.0143 | 0.0363 | 0.0333 | 0.0013 | 0.0120 |

By using Formulas 1, 2, 3, and 4, we calculated (in percentages) how much more repeatable measurements made with the panel are. In Formula 1, we inserted the number of cases for which the manual measurements were more accurate, and we multiplied the result by 100. Next, we divided this result with the number of all manual measurements that were made. The result was subtracted from 100, and thus we obtained the result of how repeatable the measurements with the panel are (expressed in percentages).

$$\text{Manual measurement} = \frac{\text{where manual measurements were more accurate} * 100}{\text{number of all manual deviations}} \quad (3.1)$$

$$\text{Manual measurement} = \frac{21 * 100}{46} = 45,65\% \quad (3.2)$$

$$\text{Measurement with the help of the panel} = 100\% - \text{manual measurement} \quad (3.3)$$

$$\text{Measurement with the help of the panel} = 100\% - 45,65\% = 54,35\% \quad (3.4)$$

With the help of measurements that have been carried out manually and measurements with the aid of the panel, we find that successive measurements with the panel are 54.35% more repeatable. With this, we proved that manual measurement is also reliable, despite the fact that the hand is not still during measuring. However, a measurement made with the panel is better since the results can later be repeated or checked, by referring to coordinates of previous measurements.

Tables 10, 11, and 12 show the results obtained by means of X-ray analysis and spectral analysis. X-ray data analysis was done with the Niton Analyzer. We used the average of the three measurements on the same spot. Spectral data analyses were provided by the company Spectro Martini d.o.o. The analyses are also an average of three measurements on the same spot. With

the help of these data, we calculated the difference or variation in each item. Measurements were carried out at three different places of the weld, as has already been mentioned in previous sections.

Table 10: X-ray and spectral analysis of the basic material

| Basic material    | Mo    | Nb    | Cu    | Ni    | Fe     | Mn    | Cr    | Si    |
|-------------------|-------|-------|-------|-------|--------|-------|-------|-------|
| X-ray analysis    | 0.272 | 0.029 | 0.235 | 0.147 | 97.821 | 0.485 | 0.549 | 0.393 |
| Spectral analysis | 0.300 | 0.033 | 0.240 | 0.160 | 97.400 | 0.540 | 0.520 | 0.420 |
| Difference        | 0.028 | 0.004 | 0.005 | 0.013 | 0.421  | 0.055 | 0.029 | 0.027 |

**Table 11:** X-ray and spectral analysis on the edge of weld

| On the edge       | Mo    | Nb    | Cu    | Ni    | Fe     | Mn    | Cr    | Si    |
|-------------------|-------|-------|-------|-------|--------|-------|-------|-------|
| X-ray analysis    | 0.167 | 0.019 | 0.156 | 0.133 | 97.501 | 0.959 | 0.345 | 0.438 |
| Spectral analysis | 0.082 | 0.010 | 0.130 | 0.091 | 97.400 | 1.380 | 0.170 | 0.410 |
| Difference        | 0.085 | 0.009 | 0.026 | 0.042 | 0.101  | 0.421 | 0.175 | 0.028 |

**Table 12:** X-ray and spectral analysis in the middle of weld

| In the middle     | Mo    | Nb    | Cu    | Fe     | Mn    | Cr    | Si    |
|-------------------|-------|-------|-------|--------|-------|-------|-------|
| X-ray analysis    | 0.020 | 0.003 | 0.052 | 97.222 | 1.728 | 0.066 | 0.415 |
| Spectral analysis | 0.016 | 0.006 | 0.060 | 97.100 | 1.950 | 0.057 | 0.430 |
| Difference        | 0.004 | 0.003 | 0.008 | 0.122  | 0.222 | 0.009 | 0.015 |

With the use of Formulas 5 and 6, we calculated an average deviation in elements when using two different measurement devices. In the formula above, we put the sum of all of the differences that we have specified in the above tables. The result was then divided by the number of differences. As a result, we obtain an average of differences.

$$\text{Average of the differences} = \frac{\text{sum of the differences}}{\text{number of differences}} \quad (3.5)$$

$$\text{Average of the differences} = \frac{1,851}{23} = 0,08\% \quad (3.6)$$

Using these measurements, we determined that the devices are comparable, as the average difference in elements was 0.08%. We also need to take into account that the spectral analysis measurement is made on the small spot, whereas x-ray analysis is measuring an area of 8 mm in diameter because of the larger lens. Nevertheless, the results are very similar. We must not forget that spectral analysis adds one percentage point of carbon, which is quite important to consider. In spite of the good qualities it has a downside: it is a breaking measurement method while the x-ray is a unbreaking method. Furthermore, the Niton device is more convenient for fieldwork, since it works on battery and does not need argon.

## 4 CONCLUSIONS

In the research, we were focused primarily on the study of welds using the Niton Analyzer Gold+ device. For this device, we made a support panel, so we were able to compare the measured data obtained by hand and with the help of the support panel. We assumed that the results obtained with the use of the panel would be more accurate, but also we found that the data obtained manually to be accurate, and there are no major discrepancies. We also have a comparison of spectral and X-ray analyses, and we found that both devices produce highly similar results.

With the panel, we have achieved more accurate and reproducible measurements. With it, we are also able to repeat the measurement the next time on the same spot via the coordinates. The panel also enables us to control the measurement device via PC, and we do not have to hold it in our hands. In the meantime, we can do something else: for example, preparing a report for the previous measurements.

We cannot influence the development of the device, as this is a matter of the company that manufactures it. Furthermore, the device is very advanced, because the company is constantly upgrading its features. Regarding further development, they could develop a new support panel, which could be managed with the help of a computer and appropriate software. Thus, we could enter coordinates, and the support panel would automatically place the device on designated spot. To protect against possible damage, the device would have a contact switch, so the panel would stop immediately if the device touched the measured object. Such a panel would ensure greater accuracy, ease of use, and safety of the device.

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