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STUDIE

THE EFFECT OF PREVIOUS MATERIAL RESEARCH INTO THE *MANUSCRIPT OF DVŮR KRÁLOVÉ* AND THE *MANUSCRIPT OF ZELENÁ HORA* ON THEIR CURRENT STATE*

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Abstract: In 2017, the National Museum commemorated the bicentenary of the discovery of the Manuscript of Dvůr Králové and the Manuscript of Zelená Hora by further material research into both works and especially by an exhibition of their originals. The main aims of this research into the manuscripts included the documentation and evaluation of their current physical condition and the mapping of the effect of the microchemical analyses performed in the context of the disputes over the authenticity of the manuscripts between the middle of the 19th century and the 1970s. For the achievement of these objectives, a detailed documentation of all the pages of the manuscripts in different types of lighting (visible direct, lateral, transmitted, ultraviolet, infrared), optical microscopy, and the identification of the degradation productions of damaged places by means of X-ray fluorescence analysis and Raman spectroscopy were used. This provided new information on the current physical condition of the manuscripts and documentation of the damage caused by historical microchemical testing. In addition, some previously unpublished historical tests were identified, thus offering a new perspective on some current damage of the two manuscripts.

Keywords: Manuscript of Dvůr Králové – Manuscript of Zelená Hora – microscopy – Raman spectroscopy – X-ray fluorescence analysis

Introduction

Since 2017, the National Museum has conducted new material research into the *Manuscript of Dvůr Králové* (hereinafter abbreviated as the *RK*; in Czech *Rukopis královédvorský*) and the *Manuscript of Zelená Hora* (*RZ*; in Czech *Rukopis zelenohorský*) – the two are jointly referred to as the *RKZ*. The research has been consulted with and commented on by an expert committee,[1] which has also approved the individual steps proposed. The main aims of this research are to document and evaluate the current physical condition of both manuscripts and to contribute, using imaging and non-invasive analytical methods, to the clarification of some technological questions of their origin. An important component goal is also the mapping of the microchemical analyses performed in the context of the disputes over the authenticity of the manuscripts between the middle of the 19th century and the 1970s.

This article summarises some of the research findings regarding the physical condition of the *RKZ* with a particular focus on the evaluation of the effect of historical microchemical analyses. Attention is paid to the description and documentation of the most serious damage, which is placed in the context of historical laboratory reports and sources. The results obtained may contribute to responsible monitoring of the damaged places and to addressing the need for conservation treatment wherever necessary.

An Introduction to the History of the *RKZ*

The *RKZ*[2] served as a significant encouragement in the second phase of the National Revival and had an impact on Czech science as well as society, but they also became an ideological means of political struggle and their ancient motifs inspired (and still inspire) numerous artists. In 2017, the National Museum commemorated the bicentenary of the

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[1] The members of the committee are: Mgr. Martin Sekera, Ph.D. (NML Director), doc. Dr. Ing. Michal Ďurovič (the head of the Department of Chemical Technology of Monument Conservation, University of Chemistry and Technology, Prague), RNDr. Karel Nesměrák, Ph.D. (Department of Analytical Chemistry, Faculty of Science, Charles University), Ing. Hana Paulusová (Department of the Care of the Physical Condition of Archival Materials, National Archives of the CR), PhDr. Jan Novotný (Restoration Department, NL CR).

[2] The literature written on the *RKZ* over the 200 years of their history is truly abundant. In the 1960s, the bibliography of the *RKZ* was summarised in: OTRUBA 1969. Some texts were reedited in: DOBIÁŠ 2014. Numerous materials can be found on the website of the Czech Manuscript Society: *Rukopisy královédvorský a zelenohorský* [online]. Česká společnost rukopisná [retrieved on 9 November 2017]. Available at: http://www.rukopisy-rkz.cz/rkz/.

discovery of the *RKZ* by further material research into both works and by an exhibition of their originals (Fig. 1).[3]

The *Manuscript of Dvůr Králové*[4] was discovered by the famous patriot Václav Hanka at the church of Saint John the Baptist in Dvůr Králové nad Labem in 1817. It comprises five parchment bifolios and two folios with so-called strips,[5] containing 14 epic, lyric-epic and lyric poems, which the Slavicist Josef Dobrovský dated between 1290 and 1310. Václav Hanka asked the city of Dvůr Králové to give him the manuscript. He then owned it until 1819, when he donated it to the Patriotic (National) Museum, where he became employed as a librarian. Within that time, he managed to have the manuscript rebound twice.[6] As recently claimed by Lubomír Sršeň, Hanka affected the appearance of the *RK* not only at the time when the manuscript was in his possession but also later – he kept improving and gilding the original plain initials.[7] Along with the *RK*, he found a few more parchment fragments, the best known of which are a fragment of an astronomical text and the *Decretum Gratiani*, both from the 14th century.[8]

The *Manuscript of Zelená Hora*[9] was allegedly discovered at the castle at Zelená Hora in West Bohemia already in 1817 as well, but it was sent anonymously to Count Franz Anton von Kolowrat as a representative of the Patriotic Museum as late as one year later. Two parchment bifolios[10] contain two incomplete poems (*Sněm* [The Diet] and *Libušin soud* [Libuše's Trial]), purportedly dated to the 9–10th century. Josef Dobrovský stated that the manuscript was a forgery and thus placed the *RZ* among other finds of the time: *Píseň pod Vyšehradem* [The Song of Vyšehrad],[11] *Milostná píseň krále Václava* [The Love Song of King Wenceslas][12] and others. Nevertheless, the *RZ* was defended by the young generation led by Josef Jungmann, which resulted in the famous controversy over the authenticity of the work.

In 1858, Václav Hanka was first accused of having forged the *RK*. This culminated in a defamation trial, which Hanka won. The *RK* and *RZ* began to be considered as one whole (the *RKZ*) at the end of the 19th century, when the alleged cryptogram 'V. Hanka fecit' was discovered in the *RZ*. This find was proved to be a mistake in 1911. Hardly any other works of literature were so frequently studied over 200 years by both social and natural sciences – whereas social sciences consider the *RKZ* to be forgeries from the 19th century, natural-science research has not clearly confirmed this hypothesis.

A Brief Overview of Historical Invasive Research

Invasive research is a term referring to any direct penetration into the substance of the original object (in this case a parchment manuscript fragment) that has caused even the slightest change in its physical condition. Such an intervention is irreversible. In dependence on the substance applied, it is evaluated with respect to the manuscripts as destructive or non-destructive. Invasive research thus includes all historical testing in which the surface of the manuscripts came into direct contact with the reagents or the original material was physically sampled.

A comparison of the current state of the manuscripts with the literature related to earlier research reveals that not all interventions were consistently recorded in reports (if there were any reports at all). Consequently, not all visible traces of previous research can be reliably assigned to extant records. An overview of existing material research into both manuscripts in question was prepared by Karel Komárek[13] and then again by Karel Nesměrák.[14]

The first invasive test was performed in 1835 in the presence of František Palacký and Pavel Josef Šafařík.[15] During this single test, the word *pogubi* on the *RZ* was moistened with tannin tincture in order to prove the presence of iron in the writing.

After a relatively long break, further, more extensive research was carried out as late as in 1880. The so-called rasped-text committee was established,[16] focusing on the places in the text of the *RK* that gave any reason to doubt and different interpretations. Vojtěch Šafařík performed microchemical tests on approximately sixty letters, or the whole words.

In 1886, the *RK* was examined even twice using microchemical tests. Independently of each other, the analyses were performed by Antonín Bělohoubek[17] and Vojtěch Šafařík.[18] Bělohoubek performed almost 70 tests, which he recorded in detail. This has been the most comprehensive material research ever, focusing not only on the main text but also on colour layers. In this research, Šafařík limited himself to twelve tests, only one of which focused on an initial.

The last invasive research to be comprehensibly recorded was conducted by so-called Ivanov's team in 1967–1971.[19] This team examined both the *RK* and *RZ*. During the re-

[3] The exhibition *RKZ: Rukopisy královédvorský a zelenohorský* [RKZ: The Dvůr Králové and Zelená Hora Manuscripts] was organised as part of the exhibition *Fenomén Masaryk* [Masaryk as a Phenomenon] in the New Building of the National Museum on 15 September – 2 October 2017. It presented the originals of both manuscripts (this was the first time that visitors had the chance to see both sides of the folios) and some conclusions of *Hmotný průzkum RKZ 2017* [The Material Research into the *RKZ* in 2017].
[4] NM, NML, CMF, shelf mark 1 A b 6. BRČÁK et al. 2014, No. 203.
[5] Dimensions: 12 × 2–8 cm.
[6] Two loose (separated) book bindings: NM, NML, CMF, shelf mark 1 C b 3/1.
[7] SRŠEŇ 2009, pp. 134–142.
[8] The fragments discovered by Hanka along with the *RK* are deposited in: NM, NML, CMF, shelf marks 1 C b 2/7/1, 1 C b 2/18. BENEŠ et al. 2015, No. 816, 832.
[9] NM, NML, CMF, shelf mark 1 A b 1 α. BRČÁK et al. 2014, No. 187.
[10] Dimensions: 16 × 12 cm.
[11] NM, NML, CMF, shelf mark 1 A b 4. Ibidem, No. 201.
[12] NM, NML, CMF, shelf mark 1 A b 5. Ibidem, No. 202.
[13] KOMÁREK 1971.
[14] NESMĚRÁK 2013.
[15] ŠAFAŘÍK – PALACKÝ 1840. According to Matoušek, the author of the test was Augustin Corda. MATOUŠEK 1938, p. 26.
[16] REPORT 1881. A rasped text is the part of the parchment from which the original text has been removed, either to a lesser extent in order to correct the text or on a larger scale to 'recycle' the whole parchment folios.
[17] BĚLOHOUBEK 1887.
[18] ŠAFAŘÍK 1886.

Fig. 1. The *RK* at the exhibition *RKZ: Rukopisy královédvorský a zelenohorský* [RKZ: The Dvůr Králové and Zelená Hora Manuscripts].

search, some of Bělohoubek's tests were revised and new invasive tests were carried out, including around 25 tests performed on the *RK* and five tests on the *RZ*.[20]

As part of the new material research conducted in the National Museum in 2017, microsamples were taken for biochemical analysis of collagen. The collection of collagen fibres from the surface of the parchment on all bifolios of the *RK* as well as the *RZ* using soft rubber and their subsequent analysis provided i.a. information on the origin of the animal from which the parchment had been made.

The Methodology for the Evaluation of the Current State

For the evaluation of the current state of the manuscripts, the authors of the article primarily created a complete, detailed documentation[21] of all the pages of the *RKZ* in different types of lighting.[22] The physical condition of the parchment support was evaluated only visually and subjectively by touch.[23] The excitation of the pages by ultraviolet light[24] identified the places where some of the earlier chemical tests had been performed or indicated stains of biological character. The inspection of the folia in transmitted visible light[25] highlighted mainly bends, fractures, holes and cracks. Lateral illumination in visible light[26] revealed a high number of shape deformations in the parchment, which had either occurred naturally or as a consequence of the *in situ* microchemical tests performed in the past. For the documentation of characteristic details and the damage, extensive microscopic research was conducted.[27]

The basic characterisation of the damaged places in both of the manuscripts studied was done using X-ray fluorescence analysis,[28] which determined the elemental composition of the stains caused by previous invasive tests. For their precise spectroscopic identification, some of these spots were further studied by Raman spectroscopy.[29]

The Results

Because of the different physical conditions as well as microchemical testing history of the two manuscripts, it is better to present the obtained results separately. This provides a clearer overview of the description of the damage found and facilitates its interpretation using historical sources.

The Manuscript of Dvůr Králové

The Parchment

The structure of the parchment is not uniform. Microscope inspection reveals different degrees of porosity. Fig. 2 shows a detail of several lines of the main text with a line of a more open parchment surface. Nevertheless, the non-uniform structure is also evident on its surfaces, with varied parchment transparency in transmitted light. The light has i.a. revealed all previous folds and bends as well as holes or material losses. If the folds include bifolio folding in the middle, it is necessary to mention the visible crack in the bottom part of the bifolio 4–5, leading from the bottom margin of the page almost to the first sewing hole.

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[19] The examination was entrusted to the former Institute of Criminology of the Federal Crime Control Centre in Prague (IC). The members of the team included: Dobroslav Srnec, Jindřich Sitta, Jiří Josefík, Jaroslav Šonka and Miroslav Ivanov.

[20] REPORT 1969 and REPORT 1971.

[21] The pictures were taken with a Canon EOS 20D camera with a Canon EF-S 17–85mm f/4.0-5.6 USM IS lens in JPEG and RAW formats.

[22] For the evaluation of the physical condition, also earlier photographic editions may be helpful: VRŤÁTKO 1862; STŘEMCHA, Bohumil. *Snímky Rukopisu Zelenohorského* [Photographs of the Manuscript of Zelená Hora]. Praha 1913 (NM, NML, CMF, shelf mark 1 A b 7. BRČÁK et al. 2014, No. 204); VOJTĚCH – FLAJŠHANS 1930.

[23] The strictly non-invasive research proposal, approved by the expert committee, did not plan for parchment fibre sampling for the evaluation of its hydrothermal properties, which would have clearly determined the degree of degradation.

[24] Source of light: two Philips TL-D fluorescent tubes, 36 W, 365 nm.

[25] Source of light: PEL light pad, cool white light, 50 W.

[26] Source of light: True Light fluorescent tubes, 15W, 5500K.

[27] The microscopy was performed on a Leica MZ16FA stereomicroscope in connection with a Leica digital CCD video camera in the magnification range of 8–60x. Circular LED lighting was used in combination with lateral halogen lighting by flexible light guides.

[28] The measurement was performed using a Bruker Artax 400µ-XRF spectrometer with the following parameters: a Mo anode; collimator: 0.65 mm; voltage: 25 kV; current: 1mA; time: 30 s; protective atmosphere: He.

[29] Raman spectroscopy was carried out in cooperation with the Chemical-Technological Laboratory of the National Gallery in Prague on a mobile dispersive Raman spectrometer with an iRaman Plus fibre optic probe from BWTek. Degradation products were identified using an excitation laser with a wavelength of 780 nm (the maximum power: 420 mW, spectral range: 65–3200 cm^{-1}, spectral resolution: 4.5 cm^{-1}; incident laser radiation intensity: max. 5 %, time of measurement: 4–40 s). The fibre optic probe was 1.5 m long, connected via an Olympus microscope, which was mounted on a motorised x,y,z-shift tripod, with the minimum step of 5 µm. The measurement was contactless. The evaluation was performed using BWSpec and Omnic programs. The spectra were compared with the spectral library of the National Gallery in Prague. Due to the high fluorescence of the spots analysed, it was possible to detect only a small part of the measured spots.

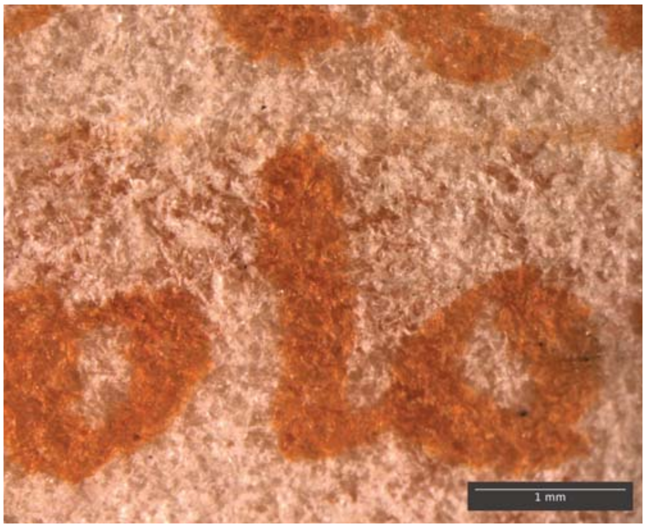

Fig. 2. The *RK*, fol. 5r. A handwriting detail with the parchment surface of varying porosity.

Fig. 3. The *RK*, fols. 9v–10r. The documentation of deformations mainly caused by previous microchemical tests.

A permanent trace in the parchment mass has been left by so-called gelatine or contact prints,[30] used by Ivanov's team. These prints are visible when the parchment surface is studied after UV-light excitation. They were applied onto the spaces on bifolio margins, which did not contain any writing, in order to provide evidence of the presence of an earlier text written with iron gall ink. In the pictures, the prints are easily discernible thanks to the proteins contained in the printed paper, which show yellow fluorescence. An inspection of all the folios has revealed that the reports of Ivanov's team did not list all the places tested in this way.[31]

Photographs in lateral light clearly showed strong corrugation caused by hydrothermal shrinkage either due to natural aging after historical microchemical tests or due to the washing of the chemicals used. Fig. 3 shows bifolio 9v–10r, on which numerous tests were performed, causing distinct shape deformations and changes.[32] Gelatine prints are likely to have fixed the margins of the parchment, thus preventing its natural movement according to the environment. Fig. 4 depicts a microscopic detail of a layer of the substance, probably gelatine,[33] left on the parchment after the prints.

Fig. 4. The *RK*, fol. 4v. A detail of the bottom left corner affected by several contact prints with a noticeable parchment crack in the upper part of the picture.

The areas that were exposed to chemicals in the past no longer have good mechanical properties. The corners with dark spots are probably the most affected; based on XRF analysis results, they are coloured by iron compounds (Fig. 5). In these areas, applied gelatine prints overlapped even three times in some cases. X-ray fluorescence analysis detected increased chlorine content in all the measured areas where gelatine prints had previously been applied. This element is probably represented here in the form of chloride ions,[34] which remained in the parchment after the contact with printed gelatine papers impregnated with hydrochloric acid. This seems to be the reason for the low pH value of the parchment, which was determined to be around 3 by Karel Komárek in 1971.[35] Iron compounds and acid residues catalyse the hydrolytic degradation of collagen here. The parch-

[30] 'The paper, coated on one side with a layer of gelatine, was soaked in a 5% HCl solution for 30 min before use. Strips of wet (not damp) prepared gelatine paper were applied, specifically with its gelatine layer facing down, onto the parchment margins, which did not contain any text. The whole system was inserted between filtration papers and pressed firmly for 1 min. After removal from the press, the gelatine layer of the paper was detected by a solution of the yellow prussiate of potash (potassium ferrocyanide), whose saturated solution was diluted in distilled water in the ratio of 1:3. The developed and perfectly washed contact prints were then photographed. Both standard and mirror-reversed photographs were made from the negatives.' (REPORT 1971, p. 50) The reports say nothing about the subsequent removal of free acid from the parchment.

[31] The report lists only 19 contact prints (REPORT 1971, p. 50). Nevertheless, it is evident from the pictures made with UV and lateral lighting that there were at least 21 prints.

[32] The margin of the parchment fol. 9v (the area around the initial, Fig. 3) is affected by the test performed by Bělohoubek, during which the drying of the paper caused its strong local shrinkage (BĚLOHOUBEK 1887, p. 331).

[33] Sampling for the identification of the substance has not been performed.

[34] Raman spectroscopy has not been able to identify precisely the degradation products formed in the parchment.

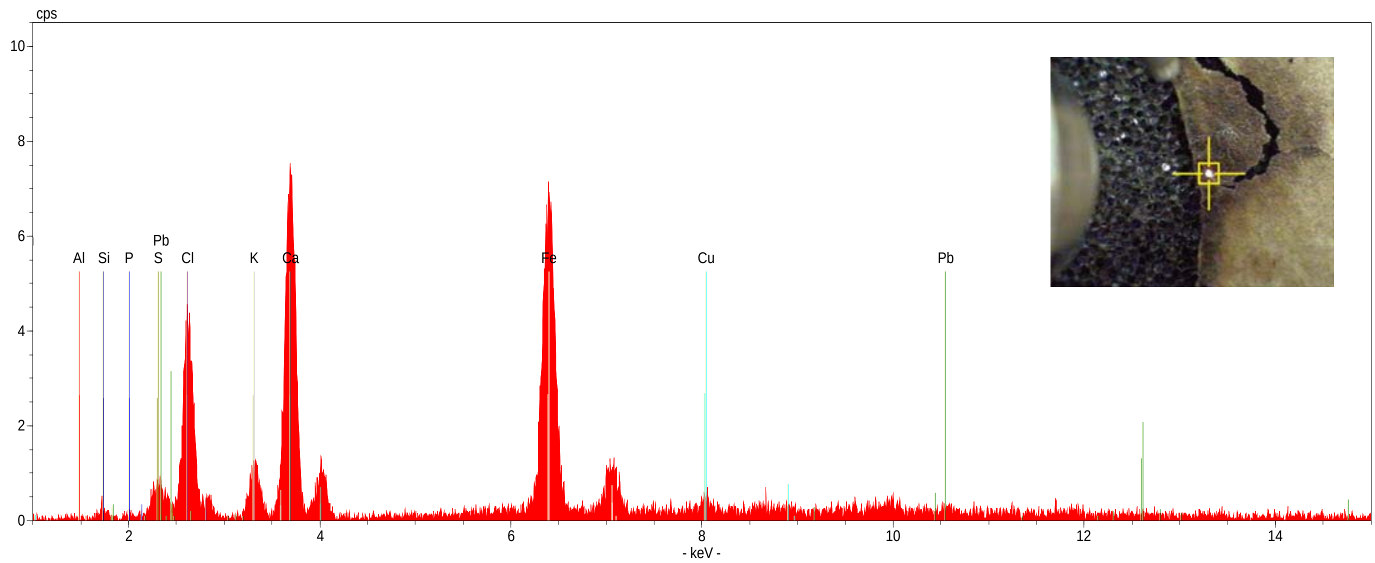

Fig. 5. The *RK*, fol. 12v. The XRF spectrum of the left margin of the parchment with a dark stain and a crack.

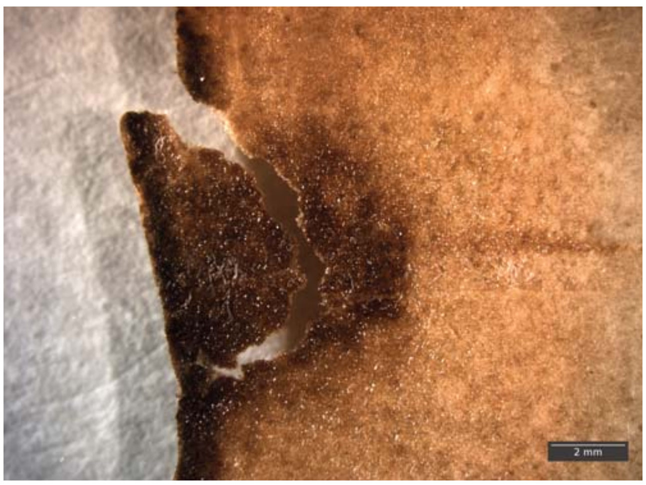

Fig. 6. The *RK*, fol. 12v (left corner). A detail of the damage of the bottom corner with contact prints.

ment is less elastic, becomes fragile and is prone to mechanical damage. Consequently, there are visible cracks in some of these locations. Fig. 6 shows a detail of the most severe damage recorded – a torn corner of fol. 12.[36] Similar damage, evidently connected with the prints, but already with a significant loss of the parchment mass in fol. 3, was described, along with the course of the degradation, by Karel Nesměrák in 2010.[37]

Microscopic analysis i.a. mapped the frequent staining and mechanical damage of the parchment. On the bifolios, it is possible to see usual surface impurities, resulting from their manipulation in the past or associated e.g. with their repeated binding into a book block. One can mention here at least the greater amount of glue on the spines of the outer bifolios with numerous sewing-needle prick holes.[38] Nevertheless, the parchments also show staining that can hardly be considered as a natural patina. XRF analysis has proved here the presence of iron compounds with trace amounts of copper and zinc (Fig. 7). This concerns some small dark spots at the upper margin of bifolio 2–7 (the centre of the bifolio) or fol. 9 (the upper right-hand corner), the soiling of the edges of some folios and a layer of apparently glutinous mass on the edges of so-called strips, evident in Fig. 8. Most of this soiling has already been described by Ivanov's team.[39]

Writing Media

The most significant ink damage includes the colour changes resulting from microchemical testing in the past. The distinctive blue spots on fol. 1r (l. 7), soaked through onto fol. 1v (l. 7 – Fig. 9); fol. 4r (l. 6 – Fig. 10);[40] fol. 6r (l. 1 – the right line), soaked through onto fol. 6v (l. 1 – the left line); fol. 8r (l. 1 – the right line); fol. 14v (l. 9) – now discernible both on the handwriting and ruling in the *RK* – were formed by reaction with the yellow prussiate of potash[41] in an acidic en-

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[35] NM, NMA, NMLA, KOMÁREK, p. 4.

[36] Before the manuscript was displayed at the exhibition *RKZ*: *Rukopisy královédvorský a zelenohorský*, the torn corner was fixed by the NML restorer Karel Křenek.

[37] NESMĚRÁK 2010, p. 111.

[38] The inspection of these sewing holes will be the subject of further research.

[39] The issues of the 'treatment' applied to the edges of the strips as well as the soiling of the edges and margins are dealt with in detail by Ivanov's team (REPORT 1971, pp. 56–60).

[40] None of the reports available to the authors mention the significant stain on fol. 4 (l. 6) or a test performed there.

[41] This is potassium ferrocyanide with the formula $K_4[Fe(CN)_6]$. Historical sources also refer to it as Gioberti's tincture (REPORT 1881, p. 147; BĚLOHOUBEK 1886). Šafařík worked with the substance called ferrocyanide (ŠAFAŘÍK 1886, p. 319), probably meaning ferrocyanic acid $K_4[Fe(CN)_6]$. The author describes the substance as water-soluble white crystalline matter that immediately reacts with ferric oxide to form dark-blue ferric ferrocyanide, also called Prussian (or Berlin) blue (ŠAFAŘÍK 1886, p. 326). Urban and Nesměrák (URBAN – NESMĚRÁK 1996, p. 42) speculated that the use of directly ferrocyanic acid was unlikely, but that the more common potassium ferrocyanide was utilised instead also in these cases.

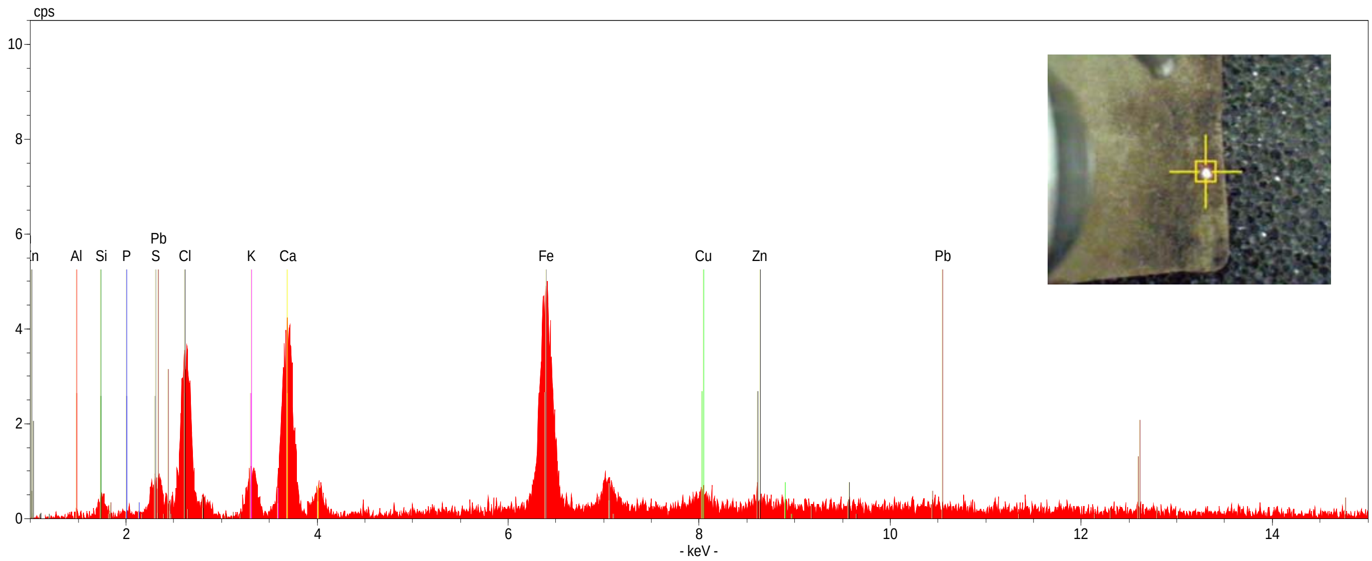

Fig. 7. The *RK*, fol. 6r. The XRF spectrum of the staining on the edge of the parchment.

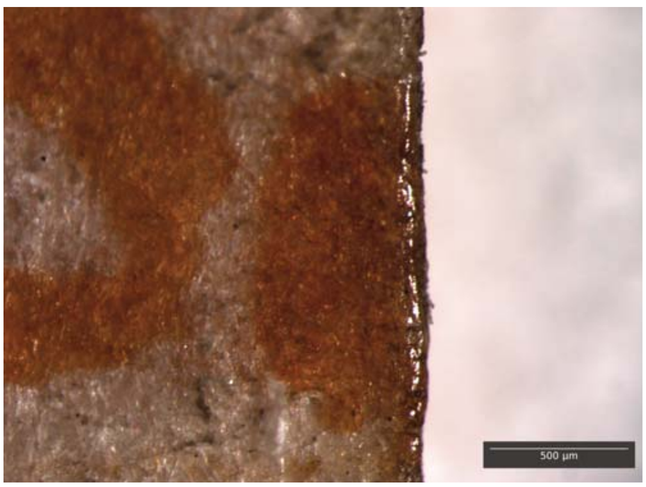

Fig. 8. The *RK*, fol. 2r (right margin). The margin closed by the glutinous mass applied.

vironment during a reaction[42] to prove the presence of ferric ions in ink. This reaction, also labelled as 'Bělohoubek's revival test',[43] proceeds according to the equation below, resulting in the formation of the blue compound of ferric hexacyanoferrate, i.e. a pigment called Prussian blue.[44] The presence of this substance in the stain spots on fols. 1r (l. 7), 4r (l. 6 – see Fig. 11), 6r (l. 1) and 8r (l. 1) was identified spectroscopically. The presence of iron in the analysed stains was confirmed by XRF analysis.

$$3\ K_4[Fe(CN)_6] + 4\ Fe^{3+} \rightarrow Fe_4[Fe(CN)_6]_3 + 12\ K^+$$

Fig. 9 shows the substantial damage to the parchment at the place of the blue stain on fol. 1r (l. 7), where the test caused a hole.[45] As mentioned by Karel Nesměrák,[46] the basic difference between this test performed in 1971 and the others was the concentration of the acid used[47] and the addition of the acid also to a saturated solution of the yellow prussiate of potash diluted in the ratio of 1:3. In this place, hydrochloric acid is likely to have caused hydrolytic damage to the parchment, catalysed by ferric ions, which finally resulted in the hole.

Three places in the manuscript (fols. 5v |l. 8|, 11v |l. 32| and 13r |l. 33|) also exhibit slight changes in the colour of the handwriting after treatment with 'ammonia water' following 'Bělohoubek's test'. These spots were washed with water, dried and then 'leached with ammonia water', which was rinsed after two minutes.[48] As mentioned by Nesměrák,[49] the principle of the test is based on the conversion of Prussian blue (formed during the 'Bělohoubek's test') into iron oxides through the effect of ammonium hydroxide.

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[42] This chemical proof test was performed by Bělohoubek (BĚLOHOUBEK 1887, p. 400), Šafařík (ŠAFAŘÍK 1886, p. 319) and Ivanov's team (REPORT 1971, p. 66) using hydrochloric acid according to slightly different procedures.

[43] The term was used by Ivanov's team (REPORT 1971, p. 66) and subsequently adopted by Nesměrák (NESMĚRÁK 2013, p. 197).

[44] It is a pigment also referred to as Berlin blue, Paris blue and Chinese blue (ŠIMŮNKOVÁ – BAYEROVÁ 1999, pp. 86–87).

[45] URBAN – NESMĚRÁK 1996, p. 106. The authors mention here a blue stain with a hole of ca 1 mm on fol. 1r (l. 7), which is not mentioned in the Report, and speculate that the stain was not formed until after 1969. Nevertheless, it is evident on l. 7 and clearly documented by Ivanov's team (REPORT 1971, p. 69). Nesměrák has already located the stain correctly to l. 7, cf. NESMĚRÁK 2010.

[46] NESMĚRÁK 2010, p. 110.

[47] During the test performed in 1886 (so-called Bělohoubek's test), the concentration of the acid was 10 %. In the testing in 1971, it was ca 19 % (NESMĚRÁK 2010, p. 110).

[48] BĚLOHOUBEK 1887, pp. 354–355. No water was applied onto the place on fol. 5v (l. 8) before the leaching with ammonia water. The same test procedure was also used on fol. 7r (l. 2), where, however, the letter tested does not exhibit any change in its hue. The reactant used was a 25% ammonia solution (NESMĚRÁK 2013, p. 198). In 1971, Ivanov's team subjected the discussed place on fol. 1r (l. 7) to a similar test using a slightly modified procedure, but it remained blue even after the treatment with ammonia.

[49] NESMĚRÁK 2013, p. 198.

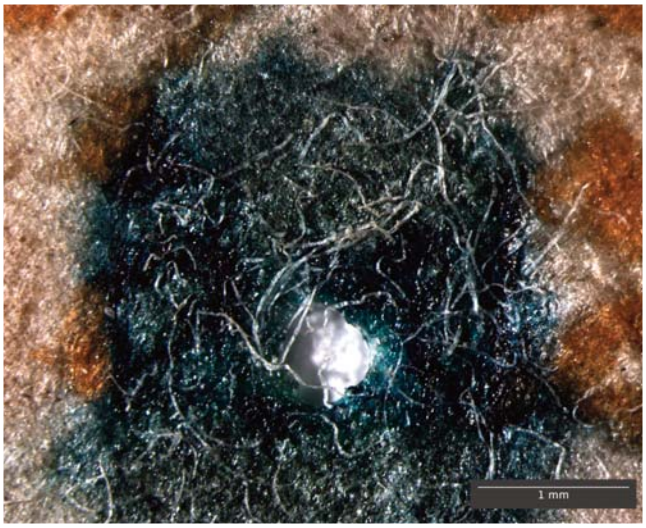

Fig. 9. The *RK*, fol. 1r (l. 7), a microscopic detail. The blue stain with the hole in the parchment at the place of the test performed by Ivanov's team in 1971.

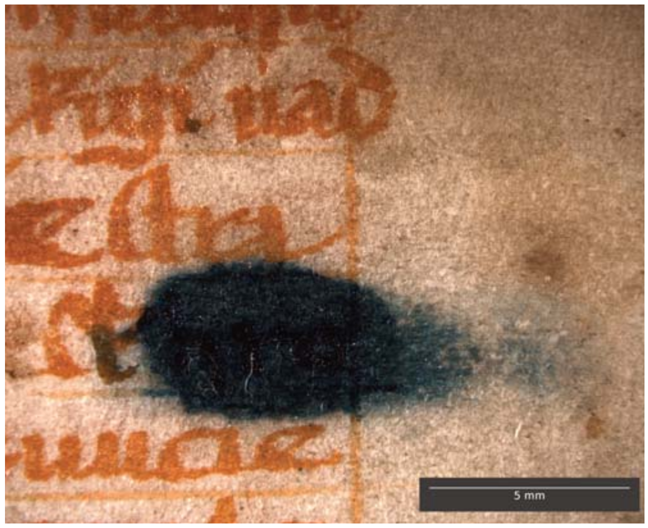

Fig. 10. The *RK*, fol. 4r (l. 6), a microscopic detail. The blue spot at the place of an unrecorded test.

Another significant change in the colour observed in the ink is the local fading or almost complete disappearance of the text on pages 4r (l. 6 – Fig. 12, l. 7), 4v (l. 5), 6r (l. 27), 6v (l. 12), 11r (l. 20), 12r (l. 26), 13r (l. 17) and 13v (l. 32).[50] This is a consequence of the testing[51] in 1880 by the so-called rasped-text committee, when water and then 'ammonium sulphide' were applied onto the selected areas with suspected presence of rasped text.[52] This was a reaction to prove the presence of iron ions in the ink. A positive reaction in the presence of iron ions produces black iron sulphides. At the places concerned, however, probably the use of the mentioned reagents only led to the washing of the text. Text darkening after the test with ammonium sulphide can now be observed in the word *bodrost* on fol. 9v (l. 26) after the test performed by prof. Šafařík in 1886.[53]

At the places of the treatment by 'ammonium vanadate' during the tests performed by Bělohoubek in 1886, three yellow spots have been left on pages 3v (l. 9), 5r (l. 1) and 8r (l. 27 – Fig. 13; l. 8[54]).[55] It was a test to prove the presence of tannin derivatives in ink, whose positive reaction was supposed to be manifested through the darkening of the letters treated. As stated by the author himself, he has no evidence on how this reaction works.[56] The only results were yellow stains from the reactants used at the places tested.

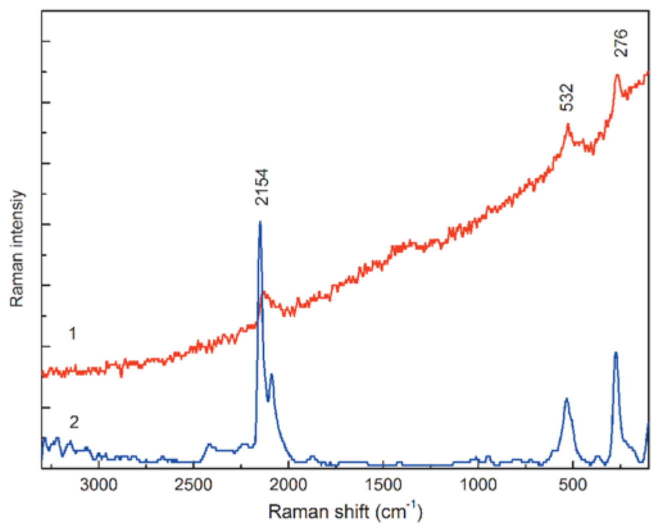

Fig. 11. The *RK*, fol. 4r (l. 6). The measured Raman spectra of the blue spot (1) and Prussian blue as the benchmark (2). Based on the characteristic positions of Raman bands, the substance at the edge of the letter has been identified as ferrocyanide.

[50] REPORT 1881. According to the rasped-text committee, the places 3v (l. 3), 7r (l. 2), 7v (l. 22), 8r (l. 1 and 2), 8v (l. 25, 31 and 32), 9r (l. 14 and 32), 9v (l. 29) and 11v (l. 1 and 4) were tested on the parchment mostly behind the words. A faintly visible letter can thus be regarded as a revived part of the text that had been rasped.

[51] In some cases, the mere reaction with ammonium sulphide was used without prior application of water – REPORT 1881, pp. 140, 142 and 145. In several other cases, the only testing consisted of the wiping of the pigment with distilled water. REPORT 1881, pp. 142, 144 and 146.

[52] In the same year, Bělohoubek applied ammonium sulphhydrate and hydrogen sulphide solution onto several places, but without an apparent impact on the places tested (BĚLOHOUBEK 1887, pp. 339–340, 349 and 352).

[53] ŠAFAŘÍK 1886, p. 319. No change is observable on other tested places with ink now. Only on fol. 3r (l. 17), the ink stroke under letter *A* can be regarded as a revived rasped text.

[54] Concerning the stain in the last letter on fol. 8r (l. 8), none of the reports available to the authors mention a test that would have been performed there.

[55] The tested place was first moistened with water and dried. After that, a solution of 'ammonium vanadate' was applied onto it and left to act for 2 minutes. Subsequently, the solution was sucked off and rinsed with water. Finally, the whole place was dried again. BĚLOHOUBEK 1887, pp. 357 and 399.

[56] BĚLOHOUBEK 1887, pp. 399–400.

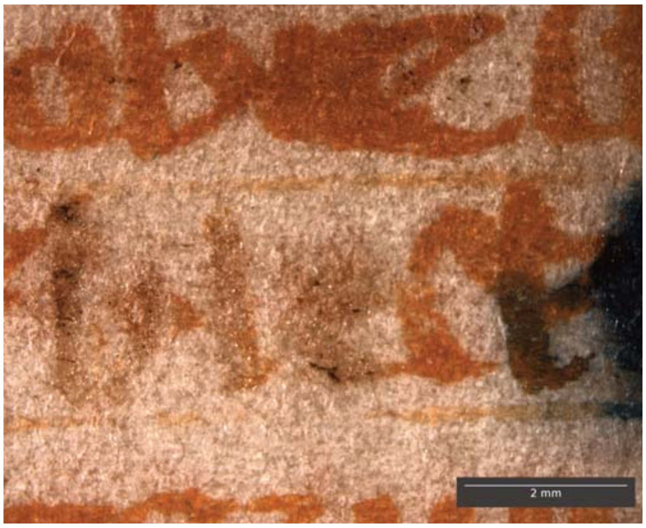

Fig. 12. The *RK*, fol. 4r (l. 6), a microscopic detail. The ink washing done by the so-called rasped-text committee in 1880.

Fig. 13. The *RK*, fol. 8r (l. 27), a microscopic detail. A yellow stain in a place where a test was performed by Bělohoubek in 1886.

Fig. 14. The *RK*, fol. 10v (l. 5), a microscopic detail. The brown stain at the place of the test performed by Bělohoubek in 1886.

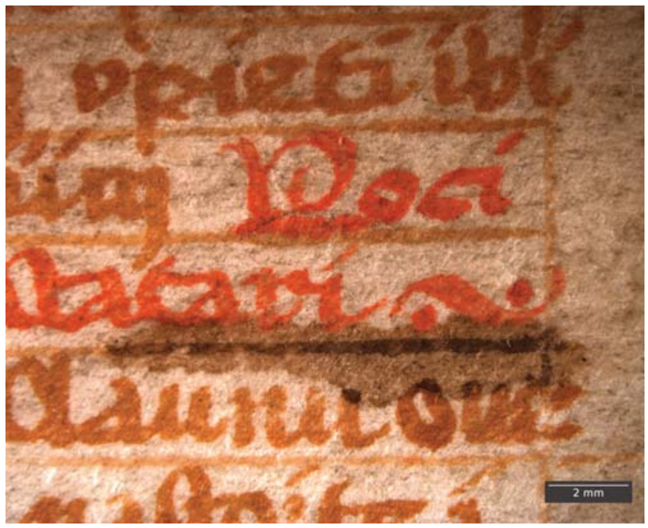

Fig. 15. The *RK*, fol. 4r (l. 21), a microscopic detail. The change in the colour of the line and the dark spot at the place of the test performed by Bělohoubek in 1886.

Two brown stains were a result of the testing performed by prof. Bělohoubek also after the reaction with ferric chloride[57] on fols. 10v (l. 5 – Fig. 14) and 12v (l. 28). This again was a reaction to prove the presence of tannins in ink according to the reactions below. A positive reaction in the presence of the polyhydroxy phenols contained in tannins produces a black complex compound of ferric tannate. Nevertheless, the brown spot in the *RK* is more likely to be a stain left from the testing sample, because no blackening has been observed at the places tested.

The test to prove free ferric ions in ink with tannin tincture was based on the same principle. The testing performed on fols. 4r (the line below the text on l. 21 – Fig. 15), 6r (the line between the text on l. 3 and 4) and 11r (l. 33) in 1886 resulted in the blackening of the places tested[58] and the formation of dark stains caused by the leakage of the reactant there.

Letter *r* on fol. 8r (l. 6) is significantly darker now. In 1886, a test[59] with glacial acetic acid was performed there. Based on the different solubility of tannates and iron oxide, it was supposed to determine the age of the handwriting according to the

[57] The tested place was moistened and covered with a solution of ferric chloride for 1 minute, after which the solution was sucked off and rinsed with water. Finally, the entire place was dried again. BĚLOHOUBEK 1887, pp. 353–354.

[58] BĚLOHOUBEK 1887, pp. 352–353. Nesměrák (2013, p. 197) mentions the use of 'gall tincture' by the so-called rasped-text committee, but it was actually used by prof. Bělohoubek six years later.

[59] BĚLOHOUBEK 1887, p. 353. During the test, the letter was moistened with water, followed by the application of a drop of diluted acetic acid for half a minute. After that period, the acid was rinsed off with water, dried, and the test was repeated with another dose of the same acid.

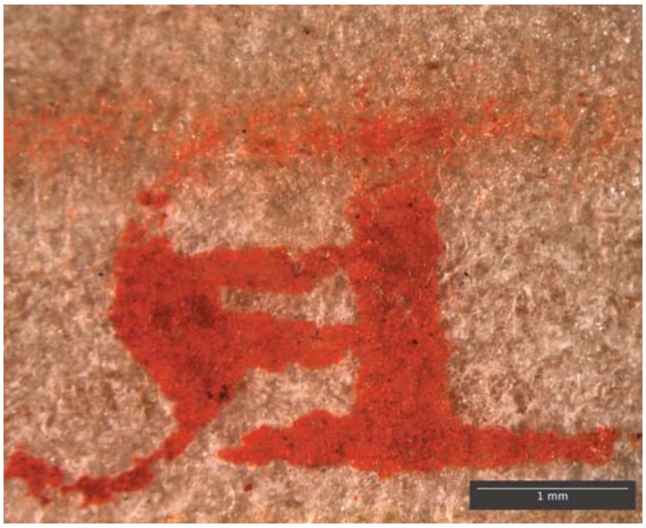

Fig. 16. The *RK*, fol. 4r (l. 1), a microscopic detail of the capital *A*. The damage caused by the rinsing of the pigment by Bělohoubek in 1886.

ink colour after the treatment.[60] As stated in Bělohoubek's report, however, the test had not had any effect on the letter.

In the past, microchemical tests were also applied on the red pigment of capital letters and the main text. The visible damage by washing has caused several colour changes. When the rasped-text committee inspected the rasped text before the capital *I* on fol. 6r, l. 21 in 1880, it partly washed off the pigment with water.[61] The red pigment in the upper part of the capital *A* on 4r (l. 1 – Fig.16) was significantly washed off by water and then aqua regia[62] when prints were made[63] by prof. Bělohoubek for the reaction to prove vermilion in 1886. The same test also caused the lightening of the word *pobití* on fol. 10v (l. 14), which had been moistened with water and brushed for the preparation of contact prints. The work is lighter than the red text around it now. This colour change was caused by the brushing away of the soiled top layer when the print was being made.

Initials

All initials in the manuscript are affected by previous tests. In the green volutes of the initials *Z* (fol. 1r), *Z* (fol. 4r – Fig. 17), *N* (fol. 7v), *S* (fol. 10v – Fig. 18) and *P* (fol. 13r), the green pigment has visibly darkened, which is a consequence of the microchemical testing performed by Bělohoubek in 1886. This darkening was caused by a reaction with potassium ferrocyanide (i.e. the yellow prussiate of potash) during the reaction to prove[64] the presence of copper ions in the green pigment. This reaction proceeds according to the following equation, resulting in the formation of the brown-red compound of copper hexacyanoferrate:

$$4\,Cu^{2+} + 2\,K_4[Fe(CN)_6] \rightarrow Cu_4[Fe(CN)_6]_2 + 8\,K^+$$

Some places that have been treated in this way are brown-red now (see e.g. Fig. 18), others rather blue (see e.g. Fig. 17). In these places, XRF analysis has detected an increased amount of iron and potassium. The content of iron in the green pigment may hence be the reason for its dark colour after the test, caused by the formation of not only copper hexacyanoferrate but also ferric hexacyanoferrate.

Fig. 17. The *RK*, fol. 4r, a microscopic detail of the volute of the initial *Z*. A change in the colour of its part at the place where Bělohoubek performed his test in 1886.

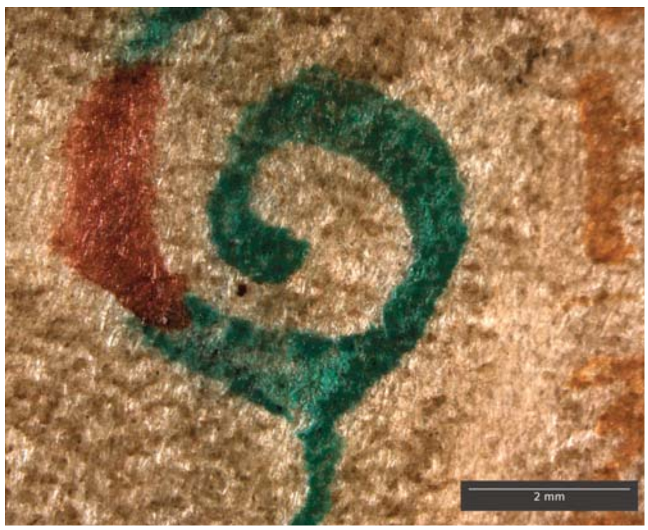

Fig. 18. The *RK*, fol. 10v, a microscopic detail of the volute of the initial *S*. A change in the colour of its part at the place where Bělohoubek performed his test in 1886.

[60] BĚLOHOUBEK 1887, p. 399. Nesměrák wrote: 'The old handwriting is not diluted in it. On the contrary, it becomes darker (which is related to the different solubility of tannates and iron oxides in the acid and to the fact that iron hydroxides and oxides become less soluble in acids with age).' NESMĚRÁK 2013, p. 198

[61] REPORT 1881, p. 141. For the description of the test, see Note 45.

[62] i.e. a mixture of the concentrated nitric acid and hydrochloric acid in the molar ratio of 1:3.

[63] BĚLOHOUBEK 1887, p. 374.

[64] This test of proof was used by Bělohoubek (BĚLOHOUBEK 1887, pp. 382–383, 386, 388–390, 394 and 395). The pigment was first washed with water and then with diluted hydrochloric acid. Finally, the yellow prussiate of potash was applied.

Fig. 19. The *RK*, fol. 9v, a microscopic detail of the initial *Z*. The rinsing of the red pigment at the place of the test performed in 1886 after the interventions by Bělohoubek and Šafařík.

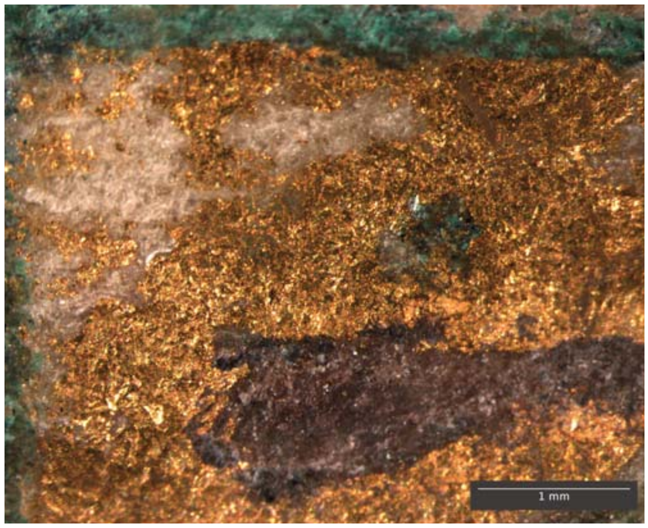

Fig. 20. The *RK*, fol. 4r, a microscopic detail of the initial *Z*. The place of the sampling of the gilding above the letter by Bělohoubek in 1886.

What is unique is the damage in the left part of the frame of the initial *S* (fol. 10v), where the blue pigment was subjected to testing in 1886. Bělohoubek claims[65] that the place was only rinsed with water, followed by the application of a drop of hydrochloric acid, which caused vigorous effervescence and the dissolution of the blue colour. The resulting solution was transferred onto a glass plate without any other findings. These reagents alone, however, could not cause such significant changes in pigment colour. It is likely that another reagent was added there at that time or later, but no mention of it has been preserved in any of the inspected historical sources.

A significant microchemical intervention has also occurred on both red initials. The rinsing of the red pigment of the volutes and frames of the initials *A* (fol. 3v) and *Z* (fol. 9v – Fig. 19) by Bělohoubek in 1886 is clearly perceptible. For the preparation of the prints,[66] the pigment was washed first with water and then with diluted hydrochloric or nitric acid. In the same year, the red pigment of the initial *Z* (9v) was also tested by Vojtěch Šafařík[67] using diluted ammonia. In the case of this initial (Fig. 19), its poor condition and legibility are caused by the combination of these two tests. The degree of damage is further documented by the results of XRF analysis – the parchment underneath the initial contains significantly less calcium than the undamaged parts.

Under a microscope, it is possible to see the mechanical damage in the gilding of the initials *Z* (fol. 4r – Fig. 20) and *N* (fol. 7v), caused by the sampling of the gilding by Bělohoubek in 1886. The samples were further subjected to microchemical testing to prove the presence of gold. Also this destructive sampling distorted the integrity of the initials and left irreversible traces on them.

The Manuscript of Zelená Hora

The Parchment

At first glance, the parchment of the *RZ* is entirely different from that of the *RK*.[68] The structure of the parchment, whose thickness ranges between 0.16 and 0.29 mm, is more porous. Just like in the case of the *RK*, transmitted and lateral light reveals the varied character of its surface mass here as well (see Fig. 21). Besides the originally selected part of the animal skin, these phenomena are undoubtedly affected also by the method of treatment of the surface during the preparation as well as other fates of the parchment. In the manipulation with the bifolios, its low elasticity and high fragility are evident.

The entire surface of the parchment has been stained. This may have been caused by the wiping of the found folios with a wet sponge, as mentioned in the anonymous letter accompanying the find of the *RZ*.[69] The whole manuscript is darkened, with lots of different stains, spread all over the parchment surface without apparent logic. Fols. 1v–4r contain numerous dark stains, much yellower after excitation by UV light, thus probably of organic or biological origin (see Fig. 22).

Both parchment bifolios of the *RZ* are not only folded in the middle, but they are also folded horizontally on the

[65] BĚLOHOUBEK 1887, p. 394.
[66] BĚLOHOUBEK 1887, pp. 384–385 and 391–392.
[67] ŠAFAŘÍK 1886, p. 323. The author demonstrates that the diluted ammonia washed the vermillion off, but the dirty pink contour neither faded nor turned purple.
[68] Probably the most complete description of the character of the parchment of the *RZ* and its material research was provided by Corda in 1840. ŠAFAŘÍK – PALACKÝ 1840.
[69] The most recent Czech translation of the letter about the discovery of the *RZ* was published by Dana Menzlová: *Nález Rukopisu zelenohorského* [online]. [retrieved on 13 October 2017]. Available at: http://rkz.wz.cz/nalez-rukopisu-zelenohorskeho/.

Fig. 21. The *RZ*, fol. 3r. The open surface structure of the parchment.

Fig. 22. The *RZ*, fol. 4r. The documentation in UV light with visible yellow stains.

bottom edge, which implies that they used to be arranged differently. The horizontal folding has numerous holes caused by mechanical wear as well as by biological pests and sewing needles. There is a significant, 40-mm-long crack on fol. 3, leading from the edge of the horizontal fold, probably caused by common use. It is also worth noting the double crack running horizontally along the bottom fold (Fig. 23), most likely a result of the already discussed contact prints. Although Ivanov's team did not describe the print precisely,[70] we are able to locate it based on the pictures made (Fig. 24).

It is also necessary to mention another gelatine print, which was not described in the report from 1969 at all. This print on fol. 1 was applied from 1v along the folding in the middle. It even covered ca five lines of the text. It is again visible in lateral light, which shows the contours of the printed paper and the smoothed surface of the parchment after pressing (Figs. 25 and 26). An interesting change occurred in the colour of the handwriting studied, which is clearly visible in incident light (Fig. 25a), but also in transmitted light (Fig. 25b). After UV-light excitation, the places of the prints exhibit yellow fluorescence again. The presence of the gelatine print at this place has been clearly proved by XRF analysis. As shown by Fig. 27, the analysis detected increased chlorine content also here, like in the other places in the *RKZ* where the gelatine prints had been applied.

Other destructive interventions made by Ivanov's team in 1971 include the excision of a parchment strip. The strip of 0.2 mm in width was cut out of the 'rectilinear side' of the semi-circular hole on fol. 3.[71]

Fig. 23. The *RZ*, fol. 4v. A detail of the bottom margin in transmitted light with subtle horizontal cracks, indicating the width of the contact print.

Fig. 24. The *RZ*, fol. 4 (l. 1 – Fig. 16). A detail of the right-hand bottom corner in lateral light, clearly indicating the approximate size of the contact print.

[70] REPORT 1969, p. 19.

[71] Afterwards, the excised strip was moistened in its cross-section by the yellow prussiate of potash. Consequently, the parchment mass turned brown-red and the parchment margins blue. The team reached the conclusion that the ferric salt only penetrated into the surface of the parchment (REPORT 1969, p. 20).

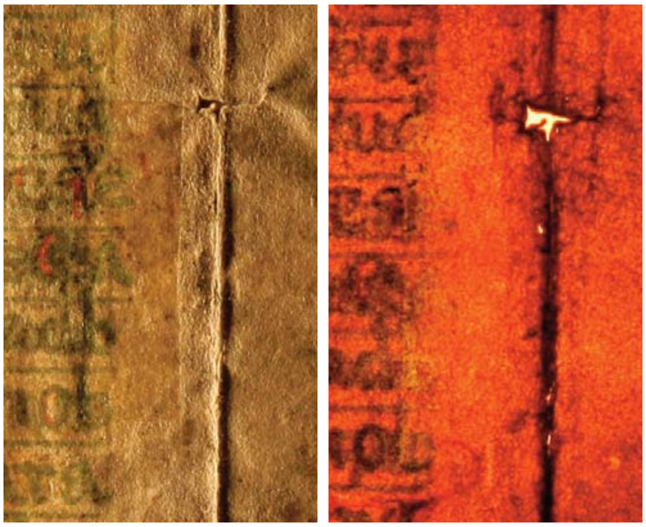

Fig. 25. The *RZ*, fol. 1v. An unreported contact print in visible incident light (a) and transmitted light (b).

Fig. 26. The *RZ*, fol. 1v. A detail of the print with a change in the colour of the handwriting and distinct lustre after contact with the gelatine.

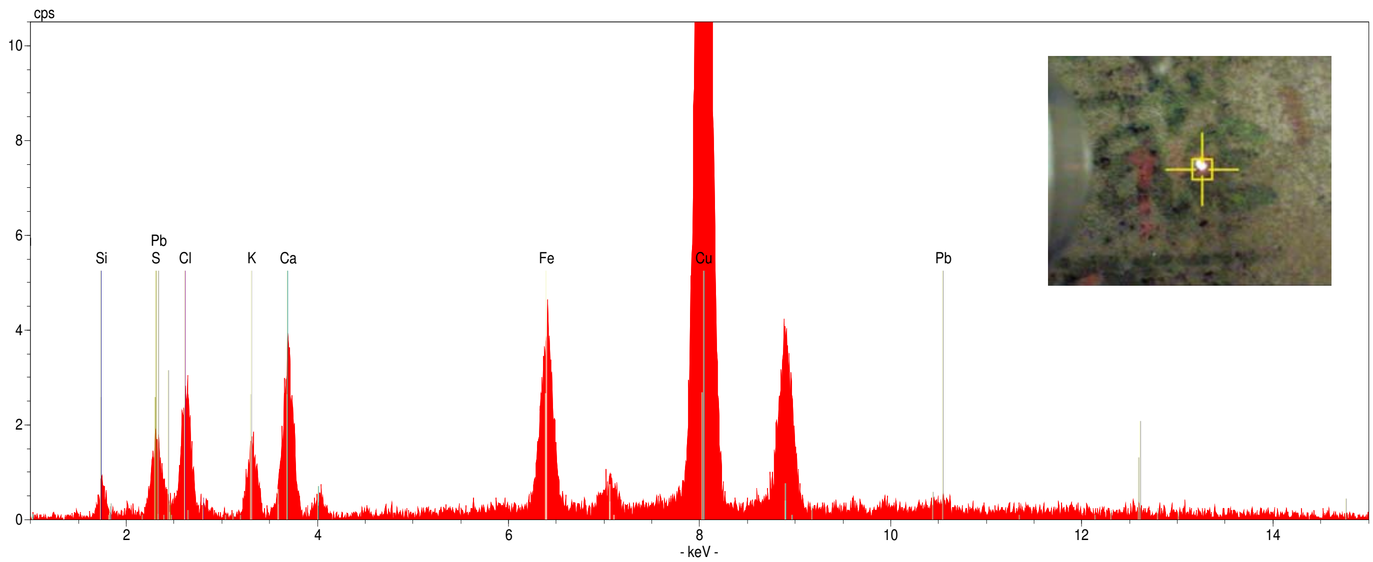

Fig. 27. The *RZ*, fol. 1b (l. 6). An XRF spectrum of the handwriting at the place of an unreported gelatine print.

Writing Media

The dark stain on fol. 2v (at the beginning of l. 1 – *pogubi*, Fig. 28) is a consequence of the first chemical testing performed by František Palacký and Pavel Josef Šafařík in 1835.[72] The word was moistened with tannin tincture, which, according to Nesměrák,[73] only caused the darkening of the parchment by tannin degradation products.

Further irreversible changes are observed in several places of the *RZ* now. These include the colour change in the ruling on fols. 1r, 2r and 3v, where ink was examined. At the place of the test, the ink is blue-purple now. Another change is noticeable in the colour of the letters on fol. 3v (l. 2 – *n*, Fig. 29; l. 3 – *c*).[74] Raman spectroscopy has identified ferrocyanide on the edge of the letter *n* (Fig. 30). This indicates that the presence of ferric ions was tested here by the yellow prussiate of potash like in the case of the *RK*. The same method has also identified Prussian blue in the blue stain on fols. 3r and 1r. None of the historical records of the microchemical tests performed mention who and how implemented them. This may be a consequence of the microchemical tests performed by Vojtěch Šafařík in 1886.[75]

[72] No report on this research is available to the authors of the article.
[73] NESMĚRÁK 2013, p. 196.
[74] Ivanov's team mentions that they inspected these two places prior to their tests, because they had already been tested in the past. Therefore, the microchemical test must have been performed as part of one of the previous surveys (REPORT 1969, p. 19).
[75] A brief mention of the fact that Vojtěch Šafařík had performed microchemical tests on the *RZ* was published by NESMĚRÁK 2013, p. 199.

Fig. 28. The *RZ*, fol. 2v, a microscopic detail of the word *pogubi*. The dark stain at the place of the test performed in 1835.

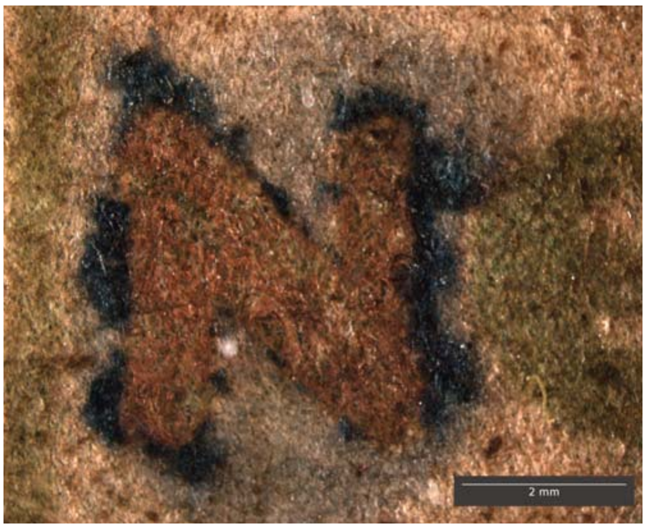

Fig. 29. The *RZ*, fol. 3r (l. 2), a microscopic detail of the letter *n*. The change in the colour of the letter at the place of an unrecorded test.

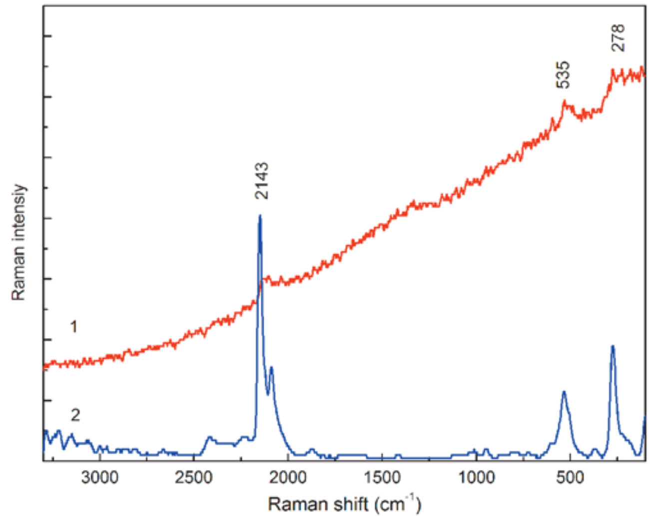

Fig. 30. The *RZ*, fol. 3r. The measured Raman spectra of the letter *n* (1) and Prussian blue as the benchmark (2). Based on the characteristic positions of Raman bands, the substance at the edge of the letter has been identified as ferrocyanide.

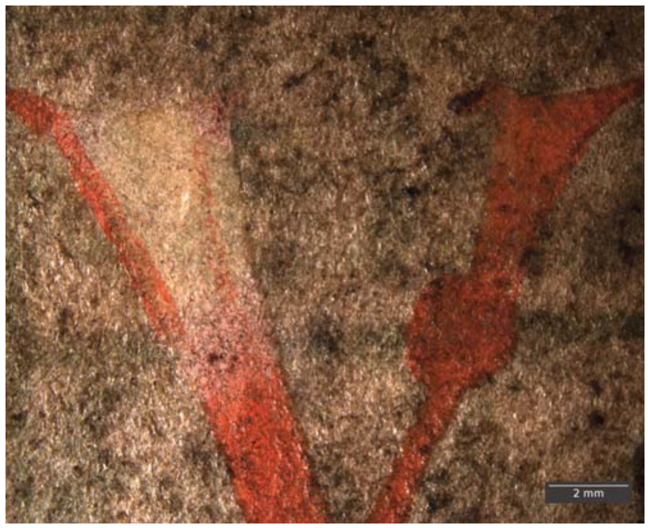

Fig. 31. The *RZ*, fol. 2r (l. 7), a microscopic detail of the initial *V*. The change in the colour at the place of an unrecorded test.

Initials

A significant colour change is noticeable in a part of the red initial *V* on fol. 2r (l. 7). The red pigment in the upper part of the left ascender has whitened (Fig. 31). This fact is also mentioned by Ivanov's team[76] – the authors state: 'It seems as if minium, after some chemical test, had been converted into Cremnitz white'.[77] A historical record of a chemical test performed on this initial is not known to the authors of the article either. Already in Střemcha's photograph from 1913,[78] just like later in the photographic edition of V. Vojtěch from 1930,[79] however, this colour change in the pigment is recorded. In the whitened place, XRF analysis detected the presence of not only lead but also a significant amount of chlorine.[80]

[76] REPORT 1969, p. 29.

[77] Cremnitz white (better known now as white lead) is the basic lead carbonate 2 $PbCO_3 \cdot Pb(OH)_2$ (ŠIMŮNKOVÁ – BAYEROVÁ 1999, pp. 30–32). The substance at the place tested has not been spectroscopically identified.

[78] NM, NML, CMF, shelf mark 1 A b 7. BRČÁK et al. 2014, No. 204. NM, NMA, NMLA, KOMÁREK.

[79] VOJTĚCH – FLAJŠHANS 1930.

[80] The white layer may also contain lead(II) chloride. The precise identification of the substance by Raman spectroscopy has not been successful.

Conclusion

An overall inspection of individual bifolios shows that the physical condition of the two manuscripts under study is not satisfactory. The work within new research includes the creation of the maps of previous interventions, showing the consequences of the invasive tests performed so far. The maximum number of the tests conducted on individual bifolios of the *RK* that have been recorded and are known to the authors of this article ranges between 11 (on fols. 11–14) and 24 (on fols. 4–5). Based on the results presented, it is evident that both manuscripts have undergone tests that have left irreversible traces on them. These include not only changes in the colour of the ink or pigments and stains on the parchment, visible at first glance, but, in terms of the long-term threat to the manuscripts, especially significant damage to the actual parchment support. In the *RZ*, the number of the listed tests is much lower, but not all of the tests applied were recorded here either in the past.

Perhaps the most serious finding is the currently clearly proven contamination of the parchment at the place of gelatine prints with chloride ions. These are an evident cause of the present-day fragility of the parchment structure and its direct destruction in the form of cracks and losses. In this connection, it has also been discovered that the official reports made by Ivanov's team do not include all the tests performed with gelatine prints. So far, it has been possible to identify two unreported prints on the *RK* and one on the *RZ*. Further research into the documentation made is likely to reveal also other damaged places. This fact is so important for the state of the *RKZ* that it will have to be resolved by a conservation intervention in the future.

It is equally important to continue to monitor any changes also at the places of the other tests performed. Because of the acidic environment, which was necessary for a large number of tests on the ink and pigments, further damage to the manuscripts in future cannot be excluded. The mapping of the implemented interventions has revealed some previously unpublished damage and thus indicated questions concerning their origin and dating. The facts discovered are important for any considerations about invasive methods of research and about their potential application to historical manuscripts.

A re-inspection of the state of the damaged places in the *RKZ* is an important part of ongoing material research. Its outcomes will be published in 2018 in a monograph being prepared.

Primary Sources:

NM, NML, CMF: National Museum, National Museum Library, collection of manuscript fragments, shelf marks 1 C b and 1 A b.
NM, NMA, NMLA, KOMÁREK: National Museum, National Museum Archives, National Museum Library Agenda, the typewritten manuscript *O hmotné podstatě RKZ s přílohami MPKV…* [The Material Substance of the RKZ with Supplements from The Love Song of King Wenceslas] by Václav Komárek from 1961, manual pagination 267.

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