X-ray powder diffraction investigations of Ruthenian-Byzantine frescoes from the royal Wawel Cathedral (Poland)

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Laboratory X-ray powder diffraction was applied to investigate the pigments used by medieval artists in Ruthenian-Byzantine frescoes in the royal Wawel Cathedral in Krakow (Poland). It was found that red fragments contained cinnabar (PDF 06-0256), yellow contained goethite (PDF 29-0713), and blue pieces contained azurite (PDF 02-0153). The green pigment, quite difficult to identify, was finally established as green earths—pigments commonly used in Byzantine wall paintings. Calcite (PDF 24-0027) was detected in all the samples. Small amounts of quartz (PDF 46-1045) in the plaster samples were also detected. The absence or presence of only minute amounts of quartz is a characteristic feature of the Ruthenian-Byzantine frescoes. Malachite was not detected in the green parts of the frescoes, in contrast to earlier investigations of wall paintings in Poland. Experimental details and the results obtained in this study are described and discussed. © *2010 International Centre for Diffraction Data.* [DOI: 10.1154/1.3478748]

Key words: X-ray powder diffractometry, pigments, conservation science, frescoes

I. INTRODUCTION

Compromise and exchange of ideas were observed in Byzantine and Western arts (frescoes, wall, and panel paintings) even though the efforts to unify the Latin and Greek churches in the late Byzantine period were unsuccessful. In the icon *Christ Bearing the Cross* painted by Nicolaos Tzafouris (end of the 15th century) in Crete (Greece), the artist signed his name in Latin and wrote the title of the scene in Greek; the rock formation depicted was Byzantine while the iconography was typically Italian (Labatt, 2000). On the other hand, the Byzantine style can also be found in non-Byzantine countries. Several churches and chapels in Poland (e.g., the chancel of the Gothic cathedral in Sandomierz, the chapel of the Holy Trinity in Lublin, and the Collegiate church in Wiślica) were decorated in Ruthenian-Byzantine style during the reign of the king Ladislaus Jagiello (1386 to 1434), who was brought up in Lithuania. His successor, King Casimir Jagiellonian (1447 to 1492), also funded the Ruthenian-Byzantine frescoes in the Świętokrzyska Chapel of the Wawel Cathedral in Krakow (Poland). These examples indicate that this type of decoration was funded by rich royal families and can be encountered in non-Byzantine regions.

Remains of Ruthenian-Byzantine frescoes were also discovered during the restoration of St. Mary's Chapel in the same royal Wawel Cathedral. The history of the chapel dates back to the first half of the 14th century. Beginning in 1421 the chapel was the burial place of the royal family. When the third wife of Ladislaus Jagiello died, he ordered the chapel to be adorned with paintings in Ruthenian-Byzantine style. However, some historians claim that it was painted earlier at the end of the 14th century (Jakubczyk, 2006).

The frescoes in St. Mary's Chapel have not survived (except for small fragments) to the present day. The only bomb dropped on Krakow during World War II destroyed the chapel. Further restoration work uncovered overpainted frescoes. Some new fragments of the wall paintings and numerous fragments of painted plaster hidden under the floor were also discovered. These fragments, mixed with sand and stone, had been used as ballast. The conservators collected them as valuable samples for investigations of ancient Ruthenian-Byzantine wall paintings in Poland (Jakubczyk, 2006). The results of X-ray powder diffraction investigations of these painted fragments of plaster are described and discussed below.

II. EXPERIMENTAL

The aim of the studies was to determine the materials used in Poland by 14th and 15th century artists in wall paintings. The historical material obtained from the restorers was first cleaned of dust with swabs moistened in distilled water and then divided into groups according to color: red, yellow, dark blue, green, black, and white. Before the measurements, the fragments of particular paint layers were removed from the plaster with a scalpel and additionally powdered in an agate mortar if needed. Then, the powdered paint samples were placed into a shallow or nonreflecting sample holder prior to the measurements.

All measurements were carried out with the use of an X'PERT PRO MPD diffractometer. Cu $K\alpha$ radiation at 40 kV and 30 mA, a diffracted-beam graphite monochromator,

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TABLE I. Powder diffraction data for red fragments of the investigated Ruthenian-Byzantine frescoes from St. Mary's Chapel of Wawel Cathedral.

Mineral name Formula PDF No.			Calcite $CaCO_3$ 24-0027		Cinnabar HgS 06-0256	
d (Å)	2*θ* (deg)	*I* (%)	*d* (Å)	*I* (%)	*d* (Å)	*I* (%)
3.87	22.97	10	3.85	29		
3.36	26.49	24			3.36	100
3.17	28.12	8			3.16	30
3.05	29.31	100	3.03	100		
2.87	31.14	24			2.86	95
2.50	35.91	10	2.50	7		
2.38	37.77	2			2.38	10
2.29	39.37	17	2.28	18		
2.10	43.17	16				
2.08	43.61	14	2.09	27	2.07	25
2.03	44.54	26			2.03	12
1.98	45.74	6			1.98	35
1.92	47.42	17	1.93	4		
1.88	48.43	15	1.87	34		
1.78	51.70	3			1.77	20
1.74	52.67	3			1.74	25
1.68	54.57	4			1.68	25
1.61	57.31	6	1.60	15		
1.59	58.17	2			1.58	6
1.52	60.77	3	1.52	3		
1.48	63.00	1	1.51	3		
1.44	64.58	3	1.44	5	1.43	8
1.42	65.60	2	1.42	3		
1.36	69.19	1			1.36	6

TABLE II. Powder diffraction data of yellow fragments of the Ruthenian-Byzantine frescoes from St. Mary's Chapel of Wawel Cathedral.

Name Formula PDF No.			Calcite $CaCO_3$ 24-0027		Goethite FeOOH 29-0713	
d (Å)	2*θ* (deg)	*I* (%)	*d* (Å)	*I* (%)	*d* (Å)	*I* (%)
4.20	21.15	3			4.18	100
3.86	23.01	9	3.85	29		
3.35	26.60	5			3.38	10
3.04	29.38	100	3.03	100		
2.85	31.44	2	2.83	2		
2.69	33.27	1			2.69	35
2.57	34.89	1			2.58	12
2.50	35.97	12	2.50	7	2.49	10
2.44	36.72	3			2.45	50
2.35	38.34	28			2.31	1
2.29	39.38	17	2.28	18		
2.24	40.23	1			2.25	14
2.20	41.07	1			2.19	18
2.09	43.12	12	2.09	27		
2.03	44.60	12			2.01	2
1.93	47.15	5	1.93	4		
1.91	47.53	14	1.91	17		
1.88	48.55	15	1.87	34		
1.82	50.15	1			1.80	6
1.72	53.18	1			1.72	20
1.69	54.26	0.5			1.69	6
1.63	56.55	2	1.63	2		
1.61	57.34	5	1.60	15		
1.59	59.28	1			1.60	4
1.51	61.44	3	1.52	3		
1.49	62.24	1			1.51	8
1.47	63.10	0.5			1.47	2
1.44	64.75	4	1.44	5		
1.42	65.72	2	1.42	3		
1.36	69.25	1			1.36	3
1.34	70.33	1	1.34	3		
1.29	73.69	2			1.29	1
1.24	76.37	1			1.24	1
1.22	77.97	2			1.20	1

and a scintillation or X'Celerator detector were used. The divergence of the incident X-ray beam was 0.5° or 1° for samples available in very small amounts. The measurements were performed in the 2θ range from 5° to 80° with a step size of 0.02°. The obtained diffraction patterns were interpreted with the use of diffractometer software (X'MENU: Philips Diffraction Software) and PDF-2 or PDF-4 databases.

III. RESULTS AND DISCUSSION

Two of the main advantages of powder diffractometry are its nondestructive nature and the possibility of analyzing multiphase samples containing crystalline components. Mainly for these reasons, this technique is increasingly used for investigations of objects of cultural heritage (Rogóż, 2009; Aucouturier and Darque-Ceretti, 2007). Because the potentially invaluable frescoes we studied had been destroyed and thus deprived of their aesthetic values, it was possible to use larger samples than those normally used in this kind of study.

Our examination of the materials used by the artists in the 15th century in St. Mary's Chapel has revealed calcite (PDF 24-0027) in all investigated samples. This was consistent with the technical aspects of the *buon fresco* technique in which pigments are diluted in water and then applied to the still-wet plaster. Usually lime mortar is mixed with sand as a filler, but we have detected only small amounts of quartz (PDF 46-1045) in the plaster samples, a characteristic for Ruthenian-Byzantine frescoes where sand was not widely used.

Apart from calcite the red fragments contained cinnabar (PDF 06-0256) and yellow fragments contained goethite (PDF 29-0713). Their X-ray diffraction results are shown in Tables I and II and Figures 1 and 2.

In dark-blue fragments of the investigated frescoes we have detected azurite (PDF 02-0153). Some authors claimed that blue pigments such as azurite $[2CuCO_3 xCu(OH)_2$ or $Cu_3(CO_3)_2(OH)_2]$ and lapis lazuli [lazurite $(NaCa)_8[(AlSi)_{12}O_{24}](S,SO_4)]$ (Eastaugh *et al.*, 2008) were unstable in the high alkaline environment of lime mortar (Procacci and Cione, 1969). However, the results of our X-ray powder diffraction investigations of dark-blue parts of the St. Mary's Chapel frescoes clearly show the presence of azurite (see Table III and Figure 3). In some cases blue parts of frescoes were prepared according to the *fresco secco* technique, in which the painting is executed on a dry wall or wall moistened to simulate the fresh plaster. In *fresco secco* the

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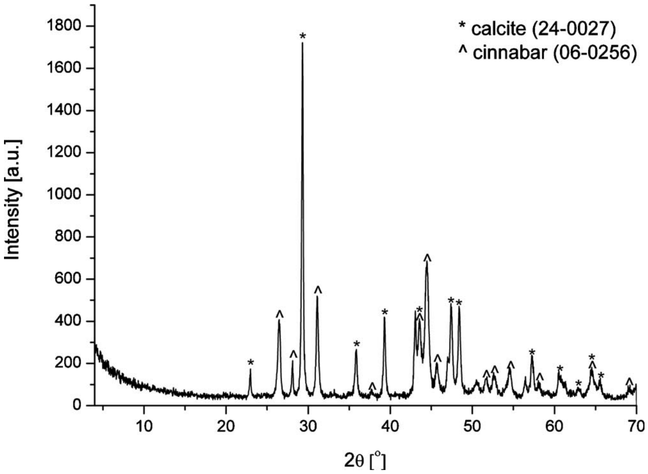

Figure 1. XRD pattern for red fragments of Ruthenian-Byzantine frescoes from St. Mary's Chapel of Wawel Cathedral.

pigments are mixed with binders such as egg, oil, glue, or casein. These binding materials, however, are usually investigated by methods other than powder diffractometry, which is why we have not been able to determine if the blue fragments were painted using *buon fresco* or *frescoe secco* techniques.

The powder diffraction patterns of the white and black fragments were quite similar. They are shown in Tables IV and V and Figures 4 and 5, respectively. The only detected phases were calcite with a small amount of quartz. We have not detected any maxima of black pigments in the black parts of frescoes. Plant black, vine black, ivory black, or black earths (known also as black chalk, containing graphite, quartz, and other minerals) were the black pigments used in mural paintings in medieval times. We have detected neither $Ca_3(PO_4)_2$, the main component of bone black (whose other components are carbon and calcium carbonate), nor any earth pigments. By burning a piece of wood we have prepared a sample of plant black and obtained its XRD pattern. It has no diffraction maxima. These results suggest that amorphous black pigment, most probably plant black, was used in the black parts of the frescoes.

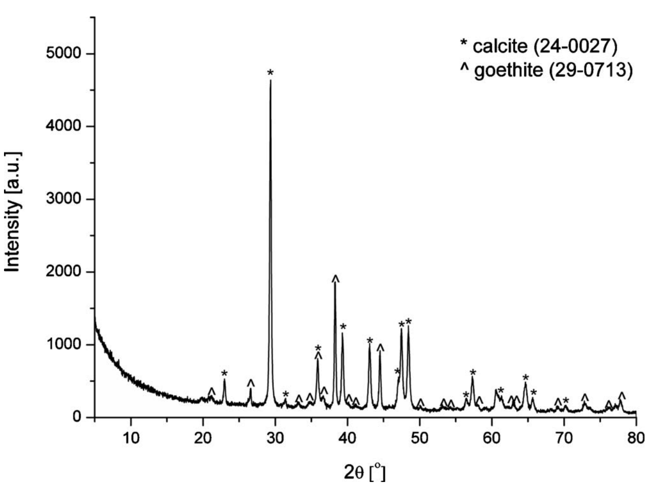

Figure 2. XRD pattern for yellow fragments of Ruthenian-Byzantine frescoes from St. Mary's Chapel of Wawel Cathedral.

TABLE III. Powder diffraction data of blue fragments of the Ruthenian-Byzantine frescoes from St. Mary's Chapel of Wawel Cathedral.

Name Formula PDF No.			Calcite $CaCO_3$ 24-0027		Azurite $2CuCO_3 \cdot Cu(OH)_2$ 02-0153		Quartz SiO_2 46-1045	
d (Å)	2*θ* (deg)	*I* (%)	*d* (Å)	*I* (%)	*d* (Å)	*I* (%)	*d* (Å)	*I* (%)
5.47	16.2	5					[a]	
5.18	17.11	28			5.20	100		
4.27	20.83	11					4.25	16
3.86	23.04	14	3.85	29				
3.68	24.18	16			3.67	100		
3.54	25.18	17			3.53	100		
3.34	26.67	94					3.34	100
3.04	29.40	100	3.03	100				
2.54	35.27	5			2.54	100		
2.51	35.72	12	2.50	7				
2.46	36.56	6					2.46	9
2.35	38.21	4			2.35	60		
2.29	39.42	18			2.28	80		
2.27	39.77	19	2.28	18			2.28	8
2.23	40.44	5			2.24	80	2.24	4
2.17	41.55	2			2.18	40		
2.13	42.51	4			2.12	40	2.13	6
2.10	43.15	10	2.09	27				
1.95	46.50	9			1.95	100		
1.91	47.53	9	1.93	4				
1.88	48.50	9	1.87	34				
1.82	50.11	5			1.83	80	1.82	13
1.79	51.15	2			1.79	60		
1.67	54.86	2			1.65	20	1.67	4
1.61	57.38	4	1.60	15				
1.52	59.94	4	1.52	3				
1.53	60.57	2					1.54	9
1.52	61.04	2			1.51	60		
1.44	64.72	2	1.44	5				
1.42	65.69	1	1.42	3	1.43	40		
1.38	68.11	6			1.38	40	1.37	7

[a]This maximum may suggest the presence of lutecite (d=5.47 Å) PDF (46-1441)—a fibrous variety of quartz whose diffraction pattern is very similar to quartz. Lutecite, however, is not a mineral approved by IMA.

Particularly difficult to identify was the green pigment. According to some authors malachite was not used in *buon fresco* techniques. However, this pigment has been detected quite often in Russian frescoes (Naumova *et al.*, 1990) and mural paintings in Poland. In the investigations of 16th century wall paintings (Kondratow, Kurowa, and Świerzawa regions), malachite was often detected in the samples (Trąbska, 2001). However, in the green parts of frescoes from Wawel Cathedral, we have not found malachite.

In the literature one may read that malachite under certain conditions may be transformed into brochantite (copper sulfate hydroxide [$Cu_4SO_4(OH)_6$]) (PDF 43-1458), nantokite [copper chloride-CuCl (PDF 06-0344) or copper trihydroxychloride polymorphs: botallackite (PDF 08-0088), atacamite (PDF 25-0269), or paratacamite (PDF 25-1427) with the common formula $Cu_2(OH)_3Cl$ (Naumova and Pisarewa, 1994; Trąbska *et al.*, 2000)]. Accordingly, we have carefully checked each of the obtained XRD patterns of green fragments of frescoes in a search for malachite corrosion products. We have found that some maxima present in the pattern

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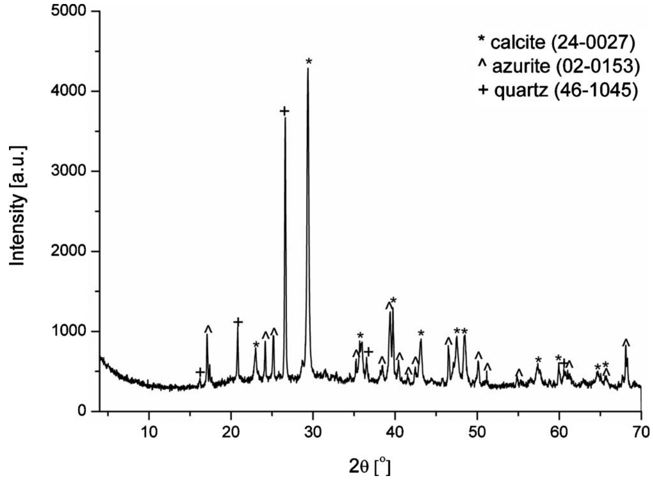

Figure 3. XRD pattern for blue fragments of Ruthenian-Byzantine frescoes from St. Mary's Chapel of Wawel Cathedral.

of botallackie were also present in the powder patterns of the investigated samples. Due to the absence of the strongest maximum of botallackite in all of these patterns, this result was not conclusive.

We have also compared the XRD patterns of green samples with the PDF data of green earths. Historical green earths are composed of either celadonite $K(Mg,Fe^{2+})\times(Fe^{3+},Al)[Si_4O_{10}](OH)_2$ or glauconite $(K,Na)\times(Fe^{3+},Al,Mg)_2(Si,Al)_4O_{10}(OH)_2$ (Eastaugh *et al.*, 2008). In PDF4+2008, one can find nine entries for celadonite and ten for glauconite. We have found some evidences in the diffraction patterns that green earths were used for the green parts of the investigated frescoes, but the results were not entirely convincing. Due to the complex composition of this pigment (Hradil *et al.*, 2003; Ospitali *et al.*, 2008; Rafalska-Lasocha *et al.*, 2010) it is not easily detectable in multiphase samples. Its identification was additionally hindered by the fact that for hundreds of years, it was mixed in the fresco with other chemical compounds—pigments, binders, fillers, or mortar.

To confirm or exclude the presence of green earths, investigations were undertaken for the green parts of the Ruthenian-Byzantine frescoes with the use of scanning electron microscopy/energy-dispersive X-ray (SEM/EDX) techniques. Several measurements were carried out on a JEOL JSM 5500LV scanning electron microscope combined with an energy-dispersive X-ray spectroscopy equipment. Results of all measurements indicate that Fe, K, Al, Mg, and Si are present in the green layer of the samples. To be sure that glauconite was the pigment used in the investigated frescoes, we have collected a larger amount of the green paint layer and dissolved it in a diluted hydrochloric acid to remove calcite and in this way to enrich the sample in glauconite or

TABLE IV. Powder diffraction data of white fragments of the Ruthenian-Byzantine frescoes from St. Mary's Chapel of Wawel Cathedral.

Name Formula PDF No.			Calcite $CaCO_3$ 24-0027		Quartz SiO_2 46-1045	
d (Å)	2*θ* (deg)	*I* (%)	*d* (Å)	*I* (%)	*d* (Å)	*I* (%)
3.86	23.03	10	3.85	29		
3.35	26.62	14			3.34	100
3.02	29.54	100	3.03	100		
2.84	31.53	3	2.83	2		
2.50	35.91	9	2.50	7		
2.29	39.36	14	2.28	18	2.28	8
2.10	43.08	11	2.09	27	2.13	6
1.92	47.46	11	1.93	4		
1.88	48.39	13	1.87	34		
1.63	56.62	2	1.63	2		
1.61	57.29	4	1.60	15		
1.53	60.47	3	1.52	3	1.54	9
1.44	64.53	2	1.44	5	1.46	5
1.42	65.52	2	1.42	3		
1.34	70.17	1	1.34	3		

TABLE V. Powder diffraction data of black fragments of the Ruthenian-Byzantine frescoes from St. Mary's Chapel of Wawel Cathedral.

Name Formula PDF No.			Calcite $CaCO_3$ 24-0027		Quartz SiO_2 46-1045	
d (Å)	2*θ*	*I* (%)	*d* (Å)	*I* (%)	*d* (Å)	*I* (%)
3.84	23.16	10	3.85	29		
3.33	26.75	10			3.34	100
3.03	29.54	100	3.03	100		
2.84	31.53	2	2.83	2		
2.49	36.04	12	2.50	7	2.46	9
2.29	39.36	17	2.28	18	2.28	8
2.10	43.08	13	2.09	27	2.13	6
1.92	47.46	13	1.93	4		
1.88	48.39	15	1.87	34		
1.63	56.49	2	1.62	2	1.66	2
1.61	57.28	6	1.60	15		
1.52	60.74	4	1.52	3	1.54	9
1.47	63.24	1			1.45	2
1.44	64.59	3	1.44	5		
1.42	65.52	2	1.41	3		
1.29	73.09	2			1.29	2

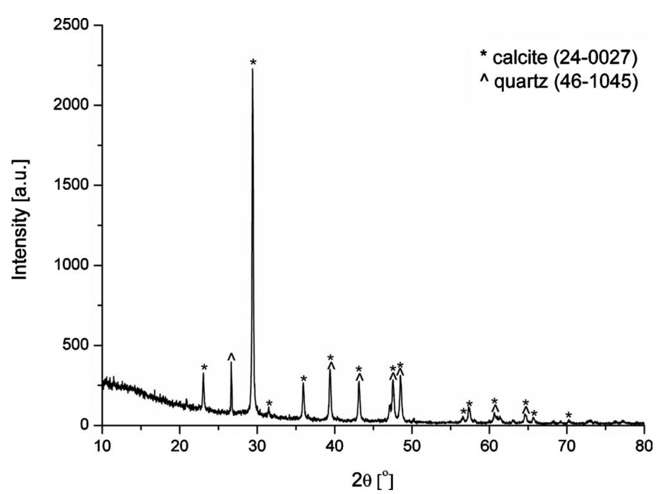

Figure 4. XRD pattern for white fragments of Ruthenian-Byzantine frescoes from St. Mary's Chapel of Wawel Cathedral.

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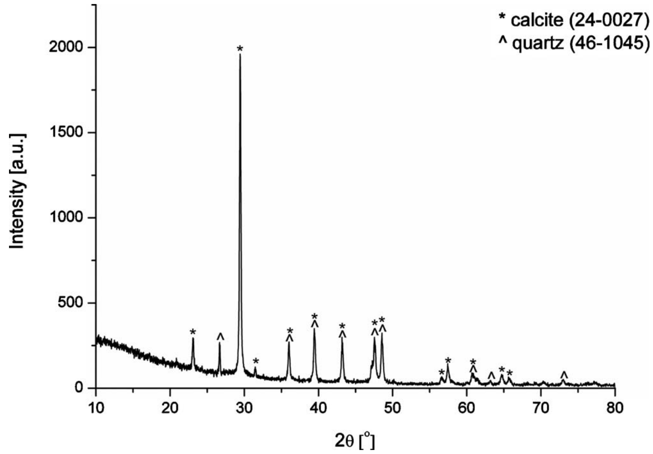

Figure 5. XRD pattern for black fragments of Ruthenian-Byzantine frescoes from St. Mary's Chapel of Wawel Cathedral.

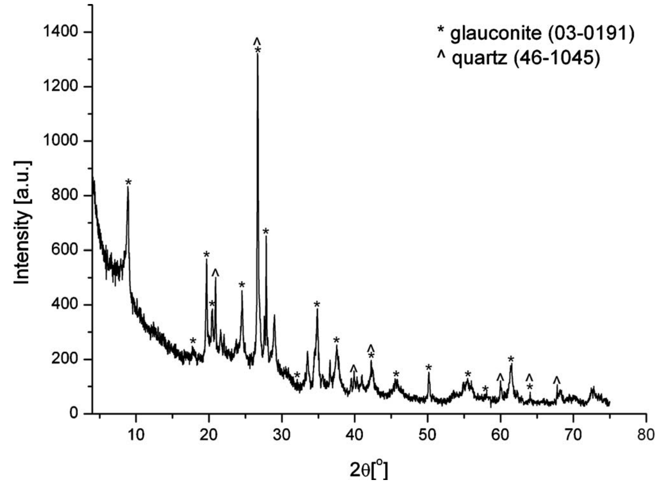

Figure 6. XRD pattern for green fragments of Ruthenian-Byzantine frescoes from St. Mary's Chapel of Wawel Cathedral after removal of calcite.

celadonite. The sample was then filtered off, the remained powder was dried in the air and powdered, and its XRPD pattern was obtained. In the data shown in Table VI and Figure 6 one can easily find the presence of glauconite (PDF 03-0191) which is a clear evidence that green earth pigments were used in the green parts of the medieval Ruthenian-Byzantine frescoes in St. Mary's Chapel of Wawel Cathedral.

TABLE VI. Powder diffraction data of green fragments of the Ruthenian-Byzantine frescoes from St. Mary's Chapel of Wawel Cathedral after removal of calcite.

Name Formula PDF No.			Glauconite $KMg(Fe,Al)_3(SiO_3)_6.3H_2O$ 03-0191		Quartz SiO_2 46-1045	
d (Å)	2*θ* (deg)	*I* (%)	*d* (Å)	*I* (%)	*d* (Å)	*I* (%)
10.025	8.821	29	10.00	75		
4.91	18.08	2	4.90	50		
4.46	19.90	30	4.46	10		
4.34	20.44	16				
4.24	20.90	27			4.25	16
4.12	21.61					
4.03	22.02	9				
3.62	24.56	22	3.62	75		
3.34	26.69	100	3.34	75	3.34	100
3.21	27.79	44	3.20	75		
3.08	28.97	19				
2.91	30.82	3				
2.84	31.43	10	2.84	50		
2.68	33.40					
2.56	34.92	24	2.56	100		
2.45	36.58	8				
2.39	37.50	13	2.39	75		
2.28	39.50	12			2.28	8
2.25	39.89	6	2.24	50*b*		
2.23	40.33	4				
2.20	40.97	4				
2.13	42.35	9	2.13	50*b*	2.13	6
1.98	45.67	4	1.97	50*b*		
1.81	50.25	8	1.81	50*b*		

IV. CONCLUSION

X-ray powder diffractometry was successfully applied to investigation of pigments used by medieval artists in Ruthenian-Byzantine frescoes in the royal Wawel Cathedral in Krakow (Poland). Mineral pigments known since antiquity [white-whitewash (calcite), yellow-yellow ochre (goethite), and red-cinnabar, blue-azurite] were easily detected with the use of a conventional powder diffractometer. The presence of green earths (glauconite), typically used in Byzantine art, was detected in this pigment sample of the green paint layer.

Amorphous plant black pigment probably gave some parts of the frescoes their greyish-black color. The obtained results confirmed that X-ray powder diffractometry is a good and complementary technique in investigations of valuable historical objects. Results obtained with the use of the powder diffraction technique may enrich our knowledge of the materials and technology used and applied in ancient and contemporary art.

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