





WP 3 – Applicability assessment WP 6 – Long Term Behavior

I. CONSOLIDATION

Gema Art Group, a. s.

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VENICE, 27 November 2014

Materials for consolidation

Nano-suspension	Original concentration	Applied concentration
CSGI Nanorestore 1P35	10 g/l	10 g/l
CSGI Nanorestore E 35	10 g/l	10 g/l
MBN Nf064	$20-26 \mathrm{\ g/l}$	10 g/l
ZFB 695p	ca 120 g/l	10 g/l

Nano-suspension	"Solvent"	Concentration [g/liter]	Particle size [nm]
Nanorestore E35	Ethanol	10	~100
Nanorestore 1P	Propanol	10	~450
MBN	Propanol	25	~300; small amount ~4000
MBN-R	Propanol	10	~300; small amount ~4000

The Particle size was measured by dynamic light scattering methods (Zetasizer Nano ZSP device) after re-dispersing in US bath

Experimental areas and sites for consolidation of stone and stucco

Stone

limestone: Kutná Hora (lab and on site tests)

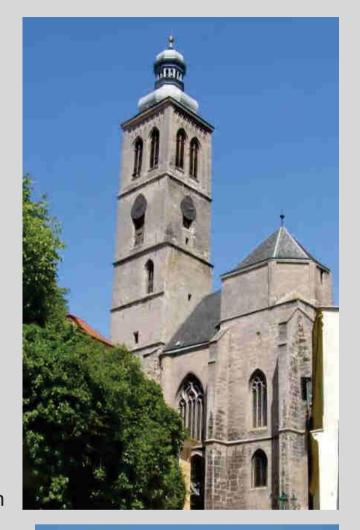
sandstone: Hořice (lab tests)

Plaster

Karlštejn Castle, late 18th Cent. Plaster (lab and on site tests)



St James church





Hořice Quarry

Way of treatment:

5 consecutive treatments with interruptions up to 1 month between applications



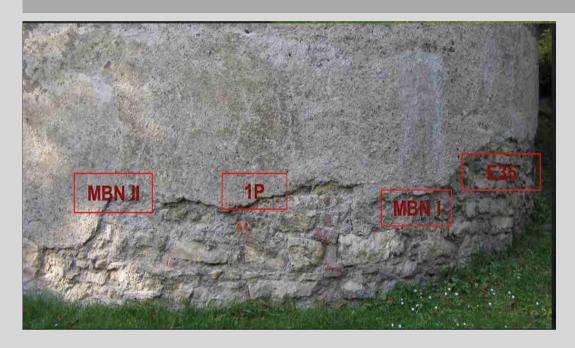


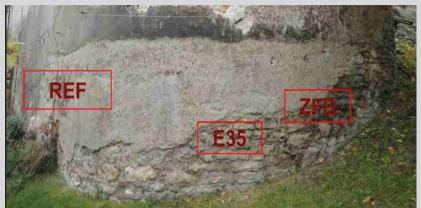
Karlštejn Castle (Central Bohemia), built in 14th cent., regotized in 2nd half of 19th cent., **mortars from 1890**





Distribution of application areas



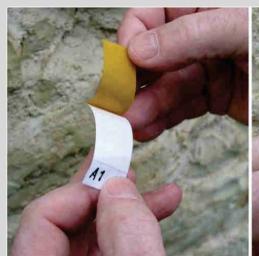


Below: surface 1 year after the last application



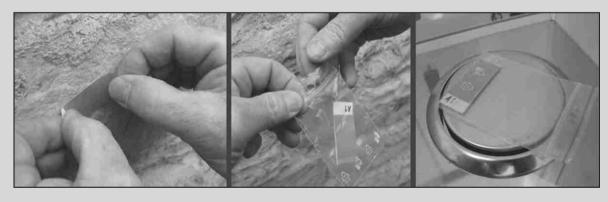


Peeling Test





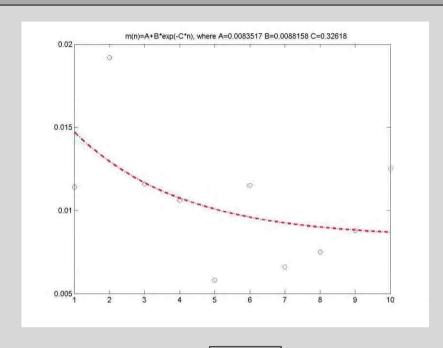


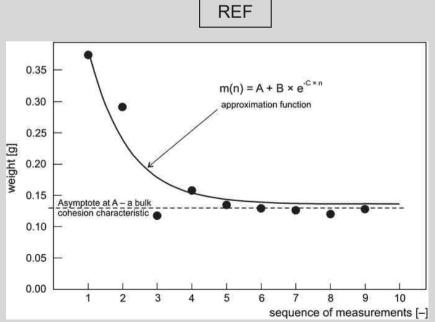


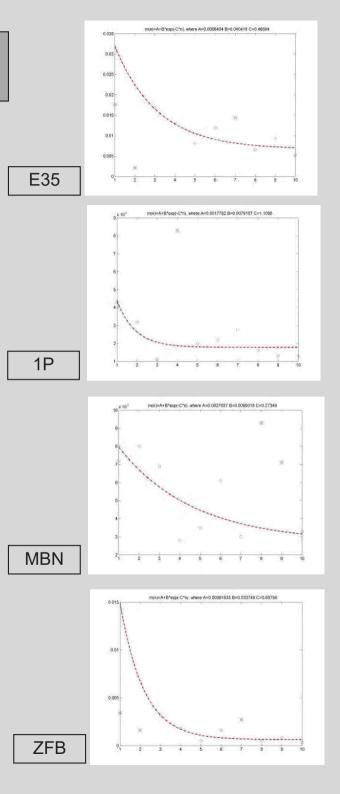


Peeling test evaluates the coherence of the surface material and is used to assess the effectiveness of the strengthening consolidation agents applied to the degraded material.

Peeling Test



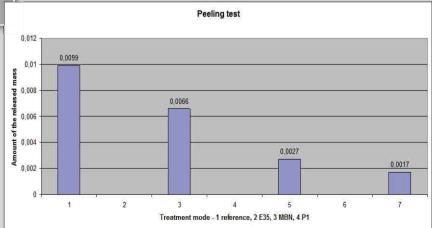




Peeling test - evaluation

	Weight	of plaster (g) released	by tearing	of the tape
Peeling No	REF 1	REF 2	E 35	MBN II	P1
1	0,0114	0,0233	0,0176	0,0072	0,0043
2	0,0192	0,0387	0,0021	0,008	0,0032
3	0,0116	0,0145	0,0168	0,0069	0,0011
4	0,0106	0,0232	0,0129	0,0028	0,0083
5	0,0058	0,0093	0,0081	0,0035	0,002
6	0,0115	0,0077	0,0119	0,0061	0,0022
7	0,0066	0,0143	0,0144	0,003	0,0028
8	0,0075	0,0152	0,0065	0,0093	0,0016
9	0,0088	0,0144	0,0093	0,0071	0,0013
10	0,0125	0,0147	0,0053	0,0033	0,0013
Konst. A	0,0083	0,0114	0,0066	0,0027	0,0017
EVALUATION			3	2	1
Decrease					
cons./ref.			33% rel.	73% rel.	83% rel.

Peeling test results of untreated reference area and areas treated with different nanolimes



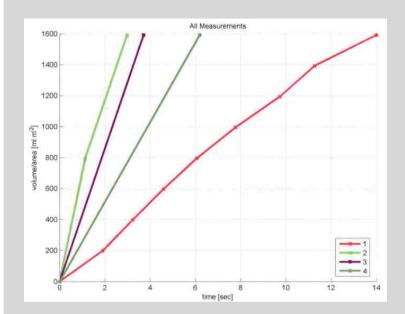
Water uptake by Microtube system





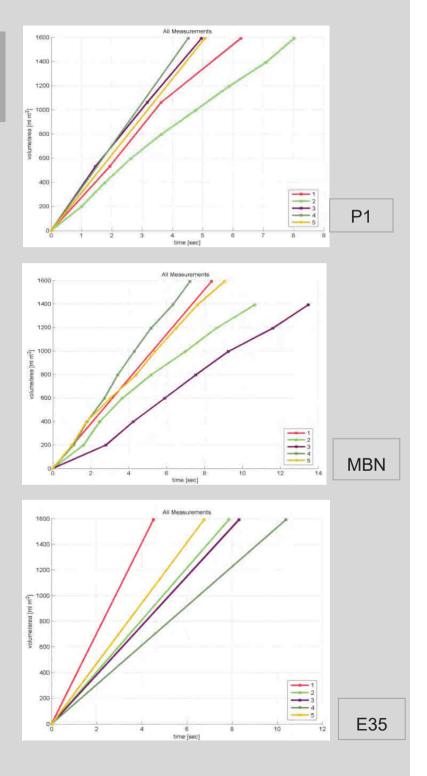


Velocity of absorbtion of water into plaster



REF

Consoildant	wac [kg.m ⁻² .s ^{-0.5}]	Rating of velocity of absorbtion	Decrease wac cons./ref.
reference	0,70	1 (fastest)	0
P1	0,67	2	4 % rel.
E35	0,60	3	14 % rel.
MBN	0,50	4 (slowest)	29% rel.



Laboratory tests

Bulk density, open porosity and water absorption

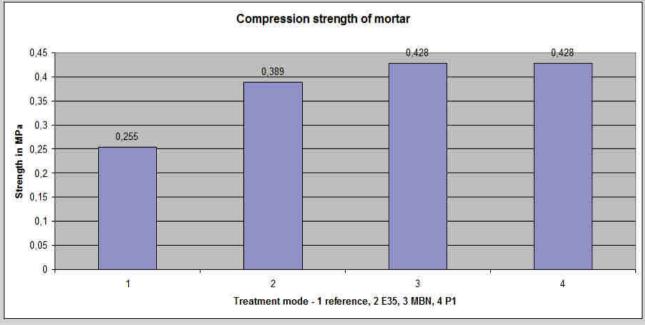


	Bulk density	Open porosity	Decrease of values	Water absorbtion
SAMPLE	[kg.m ⁻³]	[%]	of open porosity cons./ref.	[%]
REF	1622	30,1		18,6
MBN	1661	26,8	11 % rel.	16,1
E 35	1696	27,3	9 % rel.	16,1
P1	1672	27,8	8 % rel.	16

Compressive strength



Sample	depth a [mm]	width b [mm]	height h [mm]	Maxiimum power F [N]	Compre ssive strength R _e [MPa]	Equivalent average cubic strength R _c [MPa]	Increase of strength cons./ref.
K_P1_t1*	28.6	36.5	40.2	1238.50	0.84		00.0/
K_P1_t2	22.6	37.1	42.2	1317.90	0.84	0,428	68 % rel.
K_MBN_t1	28.6	36.0	40.4	939.99	0.65		
K_MBN_t2	27.0	29.0	33.8	495.07	0.51	0,428	68 % rel.
K_MBN_t3	30.9	34.3	39.3	702.58	0.52		00 70 101.
K_E35_t1	32.8	28.8	33.4	288.18	0.30		0/
K_E35_t2	34.3	35.2	37.9	669.52	0.50	0,389	53 % rel.
K_ref_t1	30.6	40.0	44.2	750.10	0.42	0,255	



Flexural strength

Results were calculated according to the formula

$$R_t = \frac{1.5Fl}{bh^2}$$

R_t flexural strength of morar in MPa

F maximum strength needed for failure in N

I distance between supports in mm

b,h width and heigth of cross section in the point of failure

TESTATRON instrument, capacity100 kN. Loading force measured by force transducer LUKAS with capacity up to 500 N. bending measured with LVDT sensor with susceptibility below 0,00015 mm, range ±1 mm. Speed of movement 0,15 mm/min.

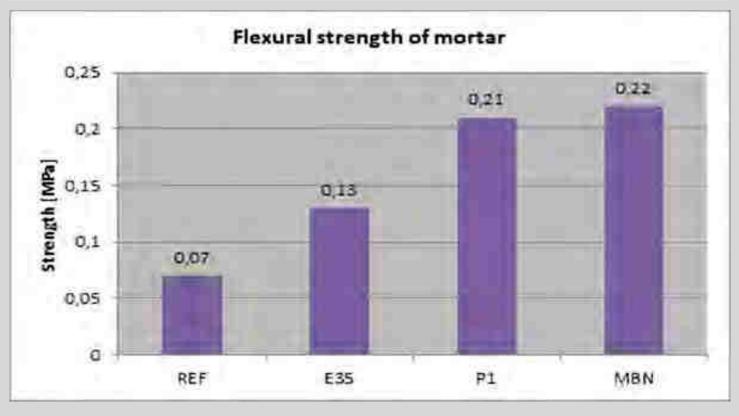




Sample with and without prosthesis

Flexural strength

Sample	height h [mm]	width b [mm]	b. support s I [mm]	Maximu mstrengt h F [N]	Flexural strength R _t [MPa]	increase of strength cons./ref.
K_P1_1	30.0	22.4	80	27.18	0.16	
K_P1_2	31.6	21.6	80	46.53	0.26	300 % rel.
K_MBN_1	22.8	33.2	80	40.41	0.28	
K_MBN_3	20.1	26.6	120	9.80	0.16	314% rel.
K_E35_1	16.3	18.2	120	3.63	0.14	
K_E35_2	14.3	21.0	120	2.82	0.12	186 % rel.
K_ref_1	24.6	20.0	120	4.01	0.06	
K_ref_2	27.5	29.2	120	9.88	0.08	



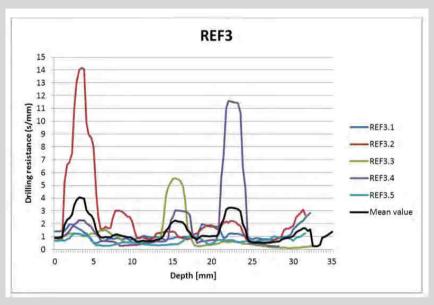
Drilling Resistance

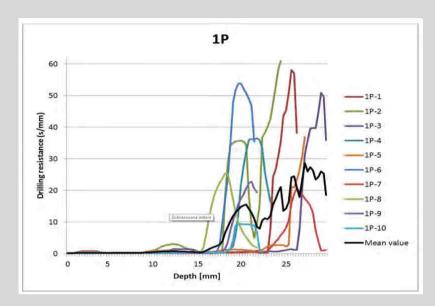


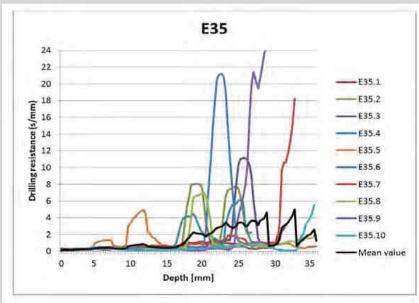


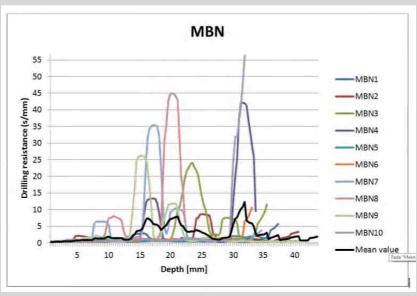


Drilling resistance results









CONCLUSION FROM LABORATORY TESTS

- Compressive strength of treated plasters increased by 50-70 % in comparison with the reference and vary between 0,30 0,84 MPa.
 Strength increase: E 35 > P1 ≥ MBN
- Flexural strength of treated plaster increased by up to 300 % BUT the total value of flexural strength vary only around 0,20 MPa
- Peeling test showed good consistency in plaster treated by ZFB, P1 and MBN. Worse results, but still positive reviews, are found if the plaster is treated by E 35
- Open porosity of the treated plaster is lower by 8 to 11 % rel.
- Results of water absorption show that the plaster retains the ability to absorb water after treatment. Measured rate reduction vary between 4-29 %.
- Results of drilling resistance confirmed the slight consolidation effect already after 5 applications

Consolidation of mortars - General conclusions

HISTORIC MORTARS

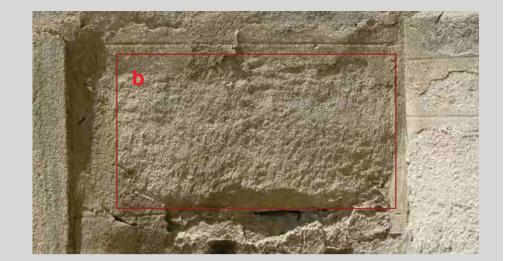
- White haze which appears soon after application gradually disappears within the time if exposed to rain
- Consumption approx. 2,5 I /m2 per first application
- Consumption considerable decreases after 2nd application
- Overall consumption can be estimated up to be 3,5 4 l/m2 after 5 applications
- Consumption decreases in the course of applications
- It can be estimated that approx.15 applications would be needed to reach comparable consolidation effect with standard techniques
- Consolidation of weathered plaster by nanolime was evaluated as an effective option within restoration projects but one must consider limits and side effects of this method

Consolidation of stone



Salinity of samples

No.	Sample	CI - %	NO ₃ - %	SO ₄ ²⁻ %
2	Coarse limestone	0,20	0,98	3,35
1	Fine limestone	0,01	0,03	0,28





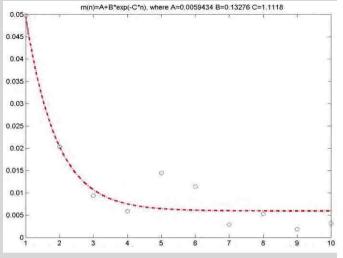




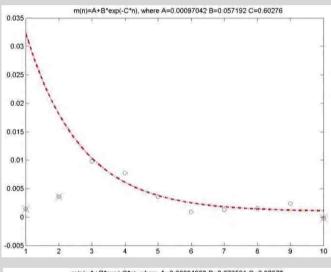
Testing area: St. James Chuch in Kutna Hora - coarse and fine limestone ashlars

Peeling test I - Coarse stone

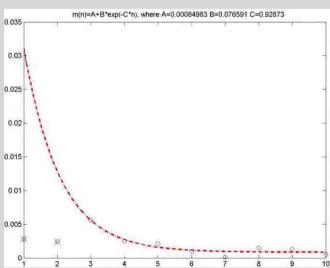
	Weight of released stone (g)				
No.	REF 1-hr.v.	E 35 – hr.v.	1P-hr.v.		
1	0.0497	0.0014	0.0028		
2	0.0203	0.0036	0.0024		
3	0.0094	0.0098	0.0056		
4	0.0059	0.0077	0.0025		
5	0.0145	0.0036	0.0021		
6	0.0114	0.0009	0.0010		
7	0.0029	0.0013	0.0001		
8	0.0053	0.0015	0.0015		
9	0.0019	0.0024	0.0013		
10	0.0032	0.0002	0.0005		
konst. A	0,00594	0,00097	0,00085		
evaluation		2	1		
decrease					
cons./ref.		84 %	86 %		



REF

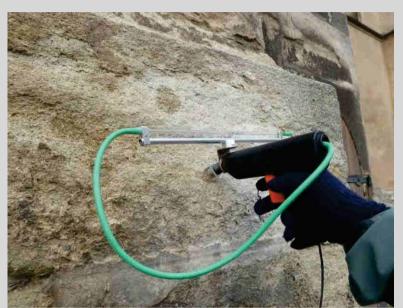


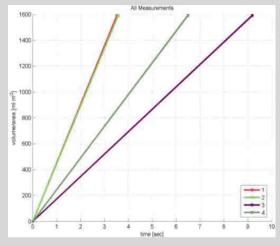
E35

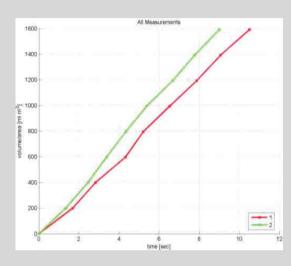


1P

Water uptake









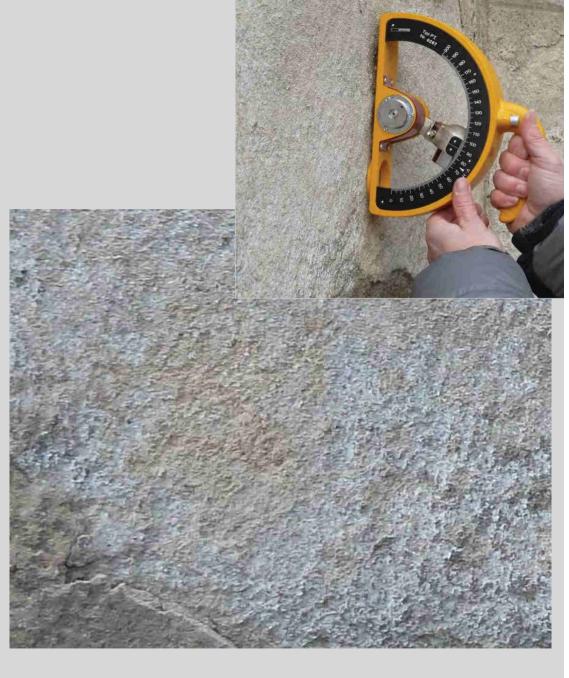
REF E35

stone	consolidant	wac [kg.m ⁻² .s ^{-0.5}]	wac decrease kons./ref.
coarse grained	reference	0,396	
	E35	0,304	23 %
fine grained	reference	0,710	
	E35	0,511	28 %

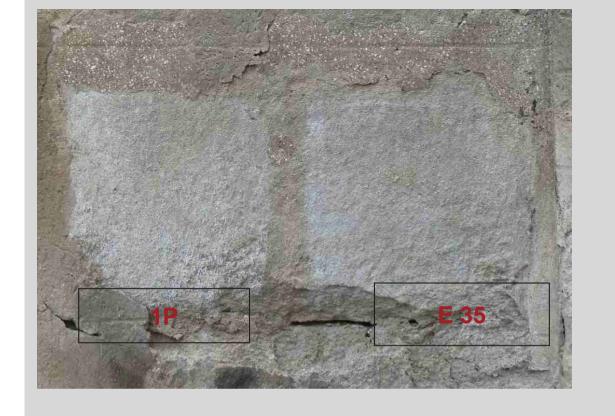




Grinding test confirmed only surface consolidation



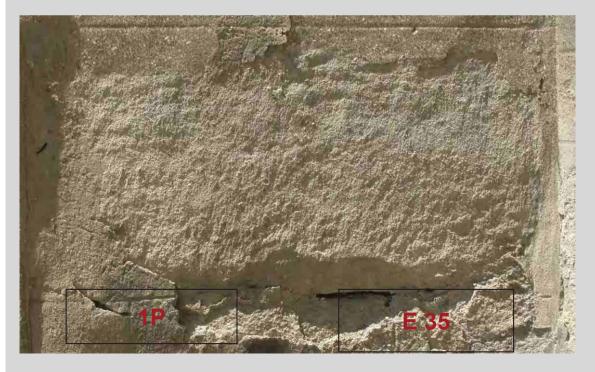
Schmidt hammer test unsuccessful



Fine limestone

Extent of white haze of both consolidants after 4 applications and complete drying

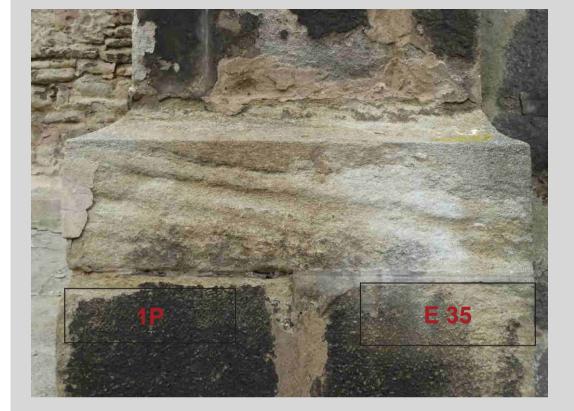
Photo: December 2013

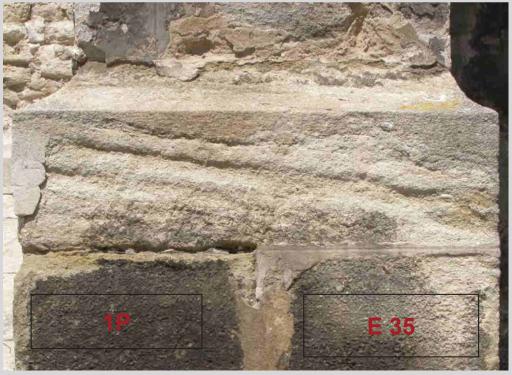


Appearance of the same block before treatment

Photo: June 2013

PARTLY PROTECTED AREA





Coarse Limestone

Extent of white haze of both consolidants after 4 applications and complete drying

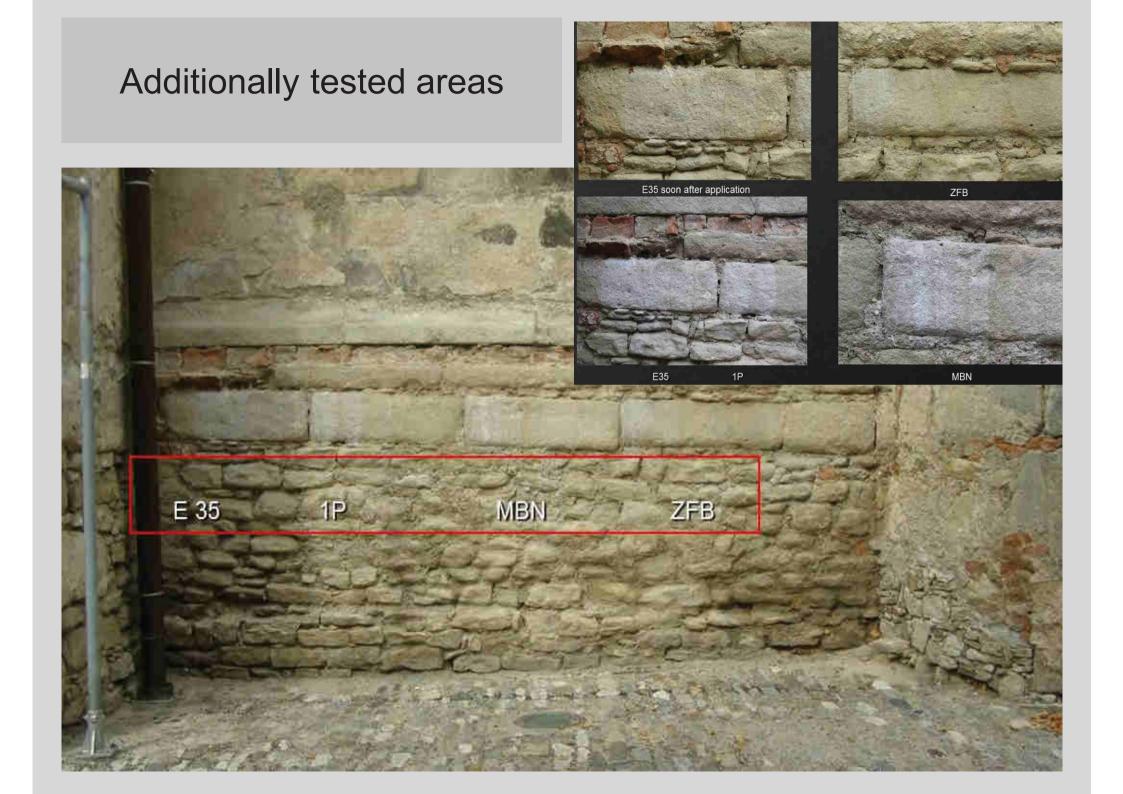
Photo: December 2013

Appearance of the same block before treatment

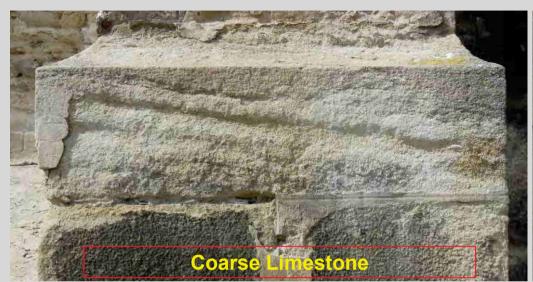
Photo: June 2013

Calculated consumption was roughly 1,5 I per square meter

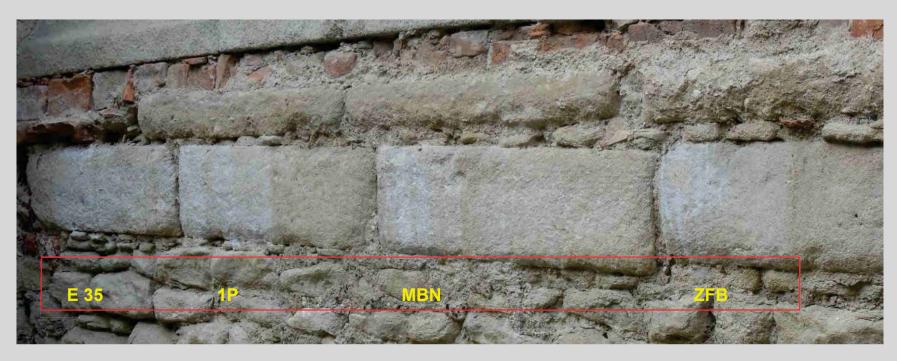
WASHED AREA



Testing areas 1 year after the last application of nanomaterials







Laboratory tests - Stone

Stone	Properties	Color
Kutná Hora	Sandy limestone, larger grains and higher porosity	White
Hořice	Sandstone without CaCO ₃ , fine grain size and lower porosity	Yellowish, ochre



Sandstone Hořice



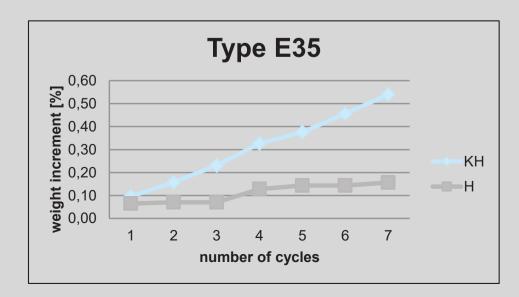
Limestone Kutna hora

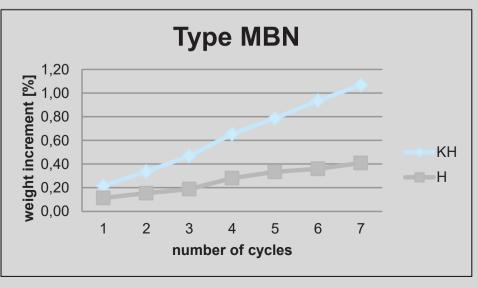
EXPERIMENTAL CONDITIONS

- Stone samples 4×4×4 cm
- Dried to constant weight at 105 °C
- Nano-suspensions applied by brush to 5 sides
- Each side painted 5 times in one cycle
- 7 cycles (at least 24 hours between cycles)
- Weight changes observed in each cycle
- Evaluation of penetration depth by phenolphthalein on cross-section

Results

Nano- suspension	conc. (g/L)	Stone	Depth of penetration [mm]	Surface vailing
E35	10	Kutná Hora	6 to 20	-
E33	10	Hořice	Only surface	+
1D	40		total cross-section	-
1P 10	10	Hořice	1 to 2	+
MDNI	0.5	Kutná Hora	total cross-section	-
IVIDIN	MBN 25		2 to 8	+
MBN-R	10	Kutná Hora	Slight coloration on all cross-section, intensive coloration in depth 3 to 10	-
		Hořice	Only surface	+



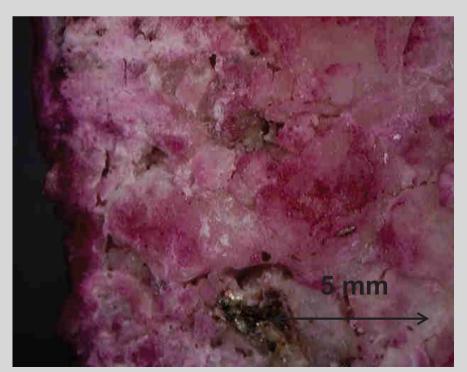






Limestone Kutná Hora

Surface before treatment
Surface after treatment
Cross-section after treatment



Depth of penetration in cross-sections staining by phenolphthalein



MBN

E35

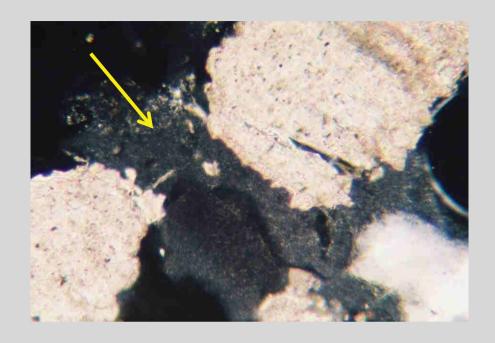


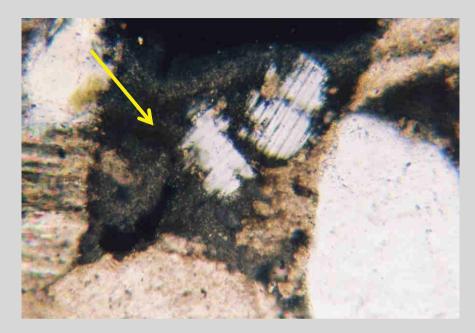


Sandstone Hořice



Limestone Kutná Hora

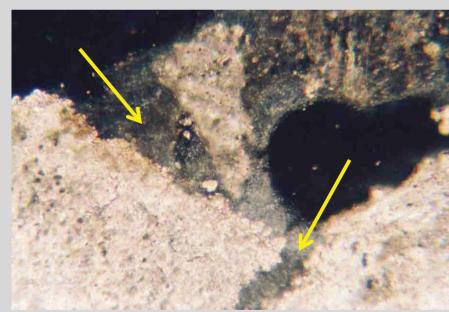




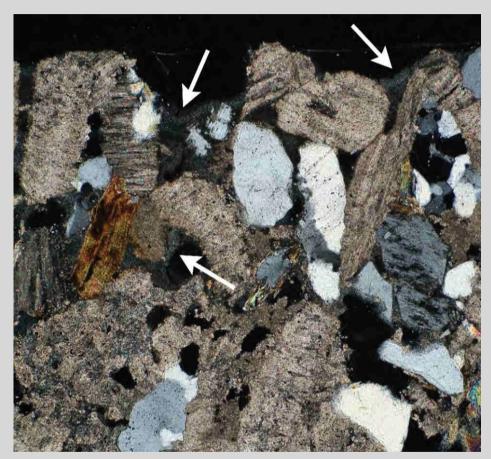
Limestone Kutná Hora (macrophoto, cross polars)

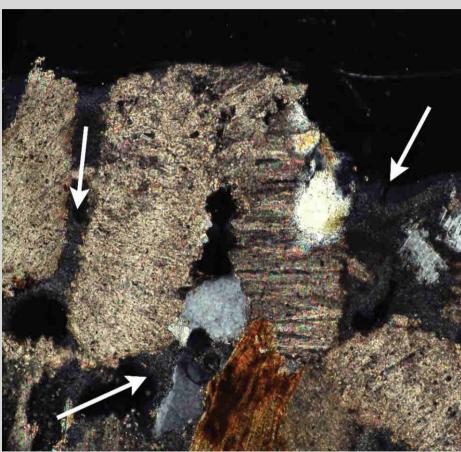
Distribution of "lime" in porous system

Penetration of nano-suspension MBN from surface into narrow crack



Kutna Hora limestone





Consolidation of stone - General conclusions

STONE IN EXTERIOR

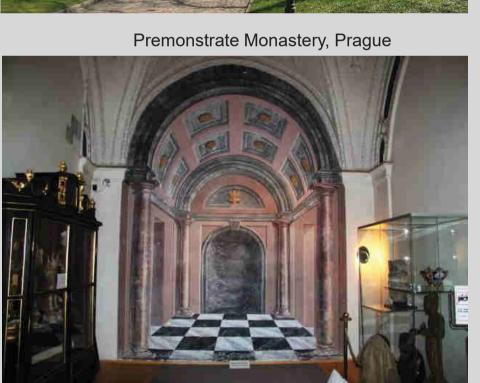
- White haze appears soon after application and remains unchanged within a time in sheltered areas
- Consumption up to 2 I /m2
- Consolidation effect recognizable already after the first application
- Loosed surface particles of stone are visibly consolidated
- Consolidation is effective only on the surface layer
- Internal humidity prevents consolidation in deeper layers

DRY STONE

- Very promissing results
- Method effective only if stone is absolutely dry
- Reccomendable for indoor sculptures not for the outdoor objects and/or architecture

Testing areas for consolidation of wall paintings





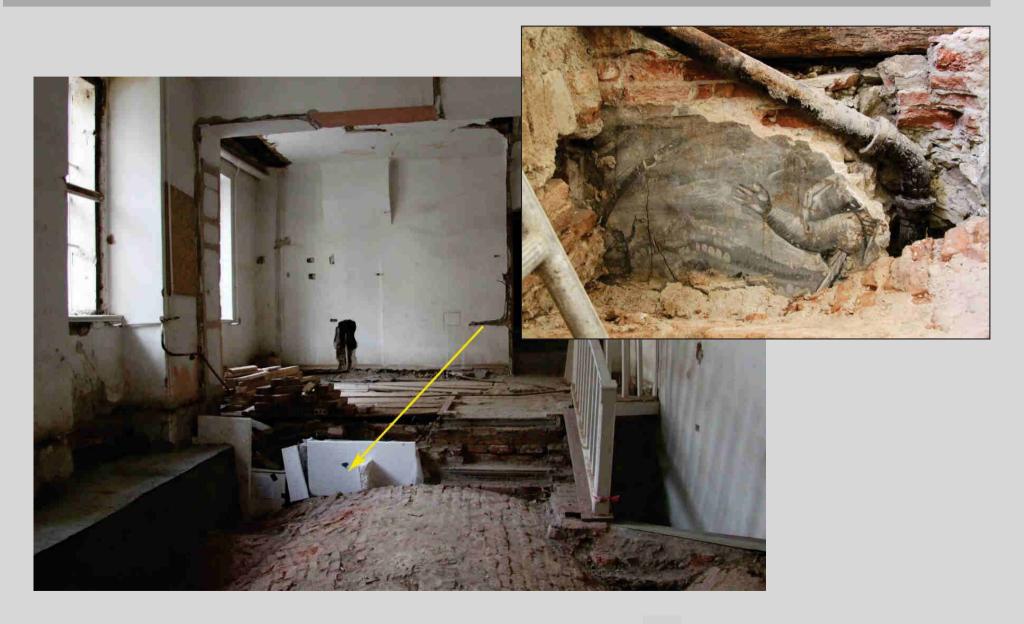


Slikovsky palace, Prague



Šlikovský palace

uncovered fragment of the wall painting found in backfilling of the arch

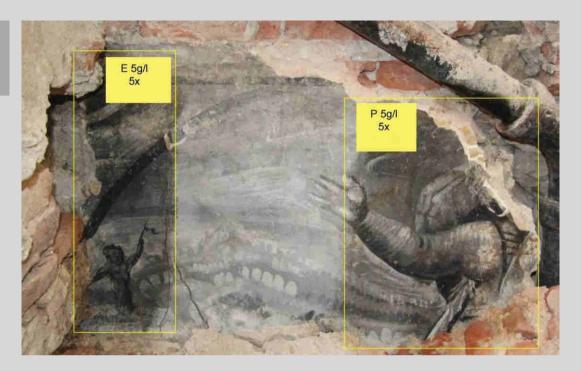


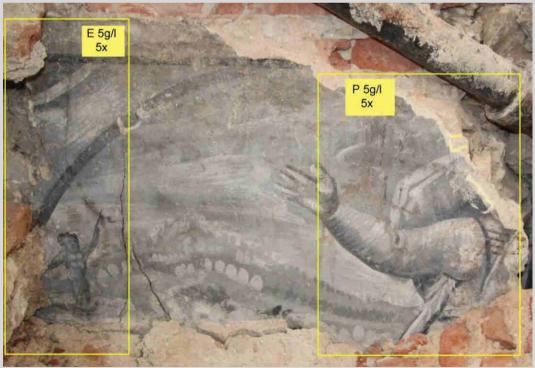
Fragment dated 1590, has never been treated ever since

Fixation of partially powdered color layer with high absorbency.

- Consolidation treatment was repeated 5 times
- White haze appeared slightly on darker areas

when wet





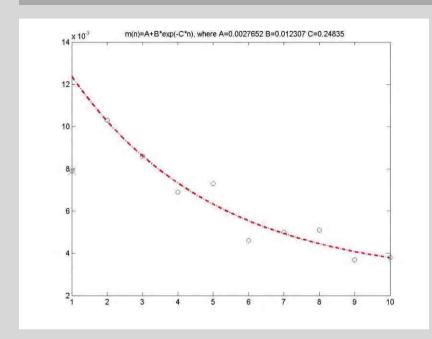
Slikovsky palace inner courtyard re-discovered part of wall painting – lime secco applied into wet mortar



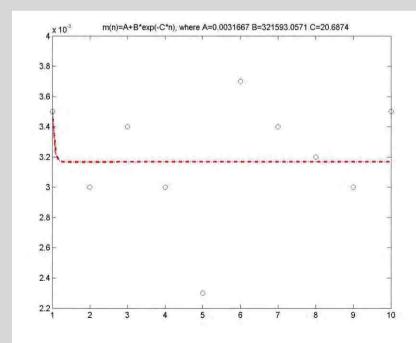


Smooth plaster with low absorbency showed negligible white haze effect, particularly in case of 1P.

Consolidation test of color layer – peeling test proved the efficiency of treatment

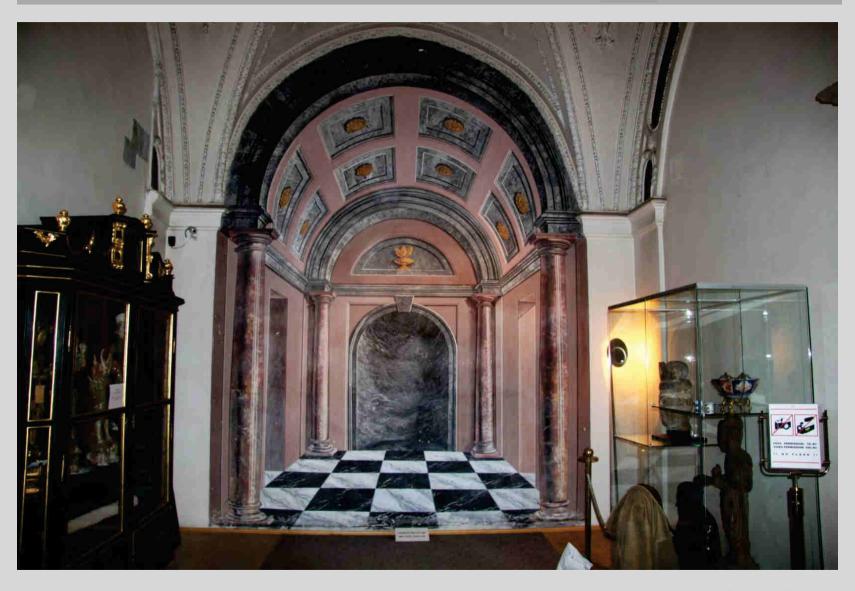


	Slikovsky palace			
	Weigh of plaster (g) detached material by peeling of tape			
no.	REF	E 35		
1	0,0079	0,0035		
2	0,0103	0,003		
3	0,0086	0,0034		
4	0,0069	0,003		
5	0,0073	0,0023		
6	0,0046	0,0037		
7	0,005	0,0034		
8	0,0051	0,0032		
9	0,0037	0,003		
10	0,0038	0,0035		
Const. A	0,00277	0,00317		

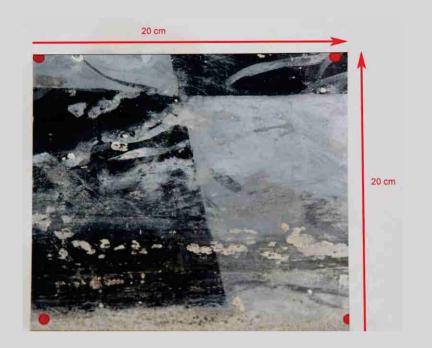




Premonstrate Monastery



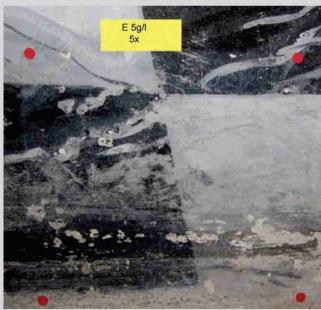
Illusive lime wall painting with advanced powdering of surface paint layer





The tests were carried out on squares 20 x 20 cm in the least important parts







Better result in case of E35

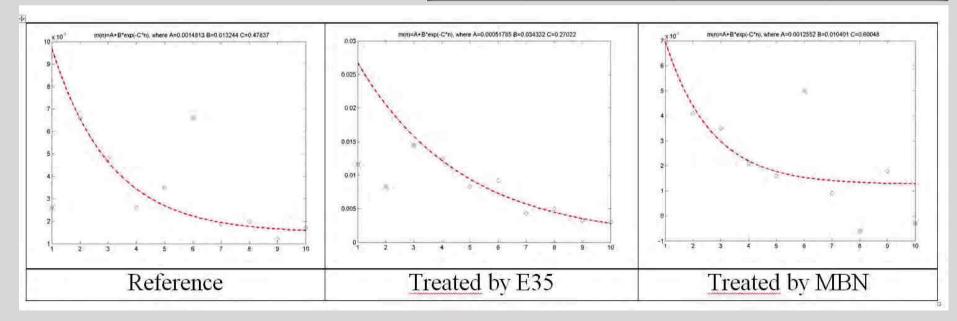
MBN did not show any traces of white haze

Visible white haze effect in case of 1P

Peeling test

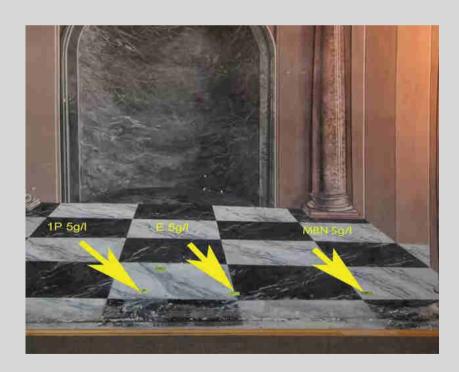


	Strahov Monastery				
	Weight of plaster (g) detached by peeling off				
Peeling no.	reference	MBN 5g/l	E35 5 g/l		
1	0,0026	0,007	0,0116		
2	0,0066	0,007	0,0083		
3	0,0048	0,0041	0,0145		
4	0,0026	0,0035	0,0124		
5	0,0035	0,0021	0,0083		
6	0,0066	0,0016	0,0092		
7	0,0019	0,005	0,0043		
8	0,002	0,0009	0,005		
9	0,0012		0,0032		
10	0,0017	0,0018	0,003		
Const. A	0,00148	0,00126	0,00052		
Decrease		15%	65%		



SPECTRORADIOMETRY

- Optical tests were carried approximately one year after the last application
- Color changes were measured by photometry and by reflected light intensity (Luminance).
- The method allows the analysis of the color in the range of 380 780 nm under the step of 1 nm.



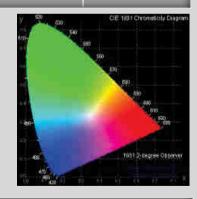


Points for Spectroradiometry measurements

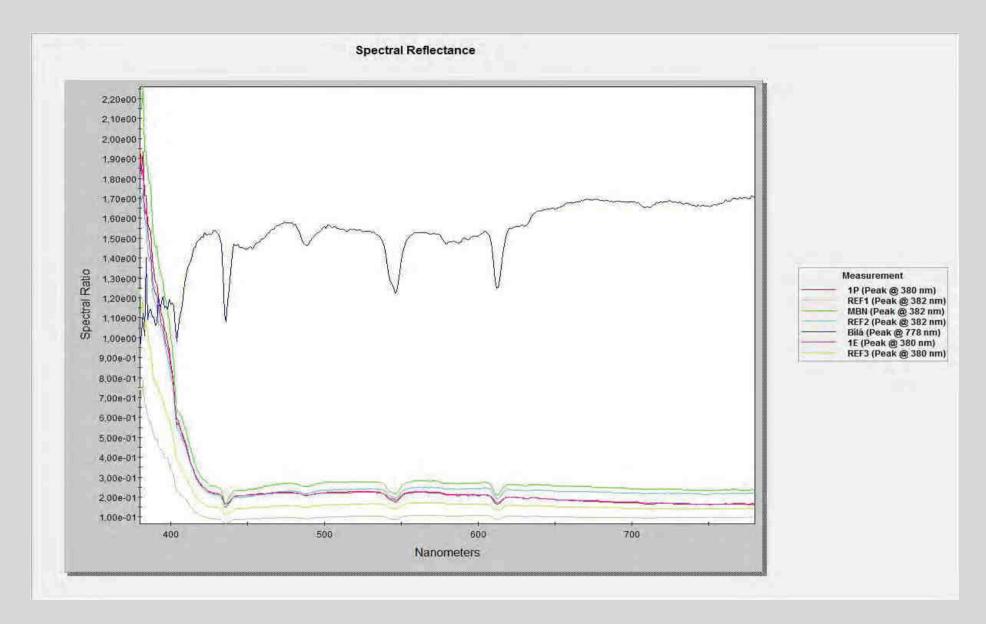
SpectraScan 740

Wave length	Spectral Ratio						
(nm)	REF3	E35	White	REF2	MBN	REF1	1P
646	1,541e-001	1,907e-001	1,645e+000	2,323e-001	2,591e-001	1,021e-001	1,904e-001
647	1,536e-001	1,905e-001	1,646e+000	2,322e-001	2,584e-001	1,018e-001	1,899e-001
648	1,535e-001	1,902e-001	1,650e+000	2,313e-001	2,581e-001	1,017e-001	1,891e-001
649	1,529e-001	1,896e-001	1,650e+000	2,309e-001	2,573e-001	1,013e-001	1,882e-001
650	1,518e-001	1,883e-001	1,645e+000	2,298e-001	2,562e-001	1,008e-001	1,871e-001
651	1,521e-001	1,877e-001	1,650e+000	2,298e-001	2,558e-001	1,007e-001	1,863e-001
652	1,515e-001	1,869e-001	1,652e+000	2,293e-001	2,554e-001	1,002e-001	1,862e-001
653	1,512e-001	1,869e-001	1,654e+000	2,289e-001	2,550e-001	1,001e-001	1,857e-001

- Consolidated areas were compared with reference areas.
- CIE standard was used for color changes of the painting.
- The results show differences between color hue of treated and reference area measured within the same painted field.



consolidant	wave length interval	average difference
MBN	646-653	0,26
E35	646-653	0,36
1P	646-653	0,88



Results confirmed the restorer's visual results and proved the smallest difference in color in case of treatment by MBN. The highest color shift occurs when using 1P reaching the average number of 0.88, where the slight white haze is visible by naked eye.

Reflected light intensity (Luminance)

Spectral	Photometry	Radiometry	Radiometry Chromaticity Setup Custom					
7.17		REF3	1E	Bílá	REF2	MBN	REF1	1P
3 2 7	erver: grees							
	nance (m2)	2,741e+000	3,584e+000	2,536e+001	3,991e+000	4,486e+000	1,762e+000	3,541e+000
1777	nance fl)	8,001e-001	1,046e+000	7,401e+000	1,165e+000	1,309e+000	5,142e-001	1,034e+000

- Measurement of reflected light should help in evaluation of the luminance of areas treated by different consolidants and have direct connection with the intensity of white hazing.
- Photometry reflectance shows clear differences between reference and treated areas.

Nano-suspension	MBN	E35	1P
Difference in reflectance in (%)	3	5	11

Difference in reflectance between reference area and tested nano-suspensions

Conclusion to the consolidation of mural paintings

- True fresco painting show very good results after consolidation
- MBN was evaluated as the best applicable material followed by Nanorestore E35
- Nanorestore 1P was evaluated as visually less acceptable from point of view of their optical properties
- When treated secco painting then consolidation effect is limited only to the paint layer with negligible anchoring to the substrate
- Painting separates from the substrate during cohesion test which can be affected by lime surface coating underneath

General conclusion to the consolidation tests

- Tested products show insufficient stability
- The sedimentation is rather fast without regular shaking
- Larger particles strongly influence application and behavior of consolidant within the substrate.
- Sedimentation cause filtering effect and cause general decrease of depth of penetration leaving white haze on the surface.
- Promising results from the laboratory were not fully confirmed on authentic objects in exteriors.
- Humidity of substrate may considerably influence the proper penetration

II. CLEANING

Micro-emulsions used for cleaning

EAPC	XYL	MEB
Water 82%	Water 85%	Water 85%
Surfactant SDS* 4%	Surfactant SDS 4%	Surfactant Brij L4 6%
1-pentanol 4%	1-pentanol 8%	2-butanone 3%
Propylene-carbonate 5%	p-xylene 3%	Ethylacetate 3%
Ethylacetate 5%		Butylacetate 3%

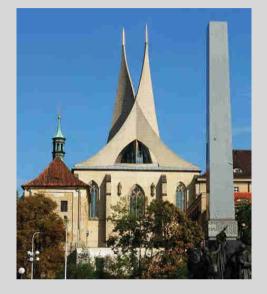
Experimental areas and sites – wall paintings

Wall painting cleaning fresco/secco paintings in various locations









Stone cleaning

limestone tombstones in Old Jewish Cemetery, Prague



Wall painting: Grabštejn

Gothic castle of Grafenstein founded in the 13th century. Rebuilt in 1566 - 1586 in Renaissance style Chapel of St Barbara decorated with murals in fresco/secco technique







1980's treatment: fixation with acrylic dispersion 2008 treatment

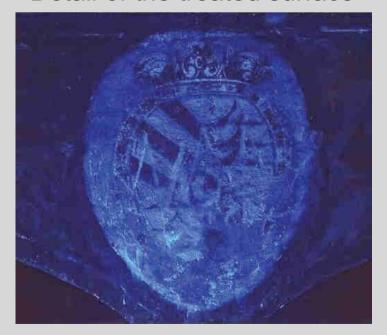
Removal of fixative: aceton-ethanol 1:1 in Arbocell 200

Removal of overpaints: toluene- ethylacetate 1:3 in Arbocell

New fixative: : 5%- Klucel M, 2% Primal AC 35



Detail of the treated surface



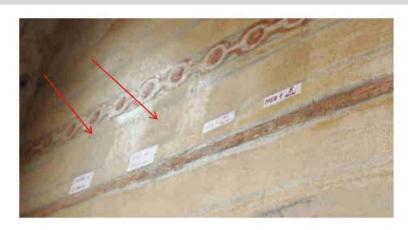


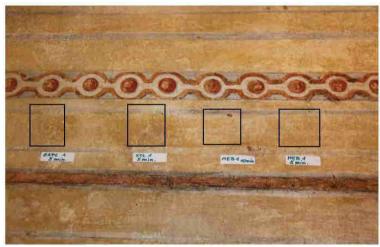
Surface after cleaning in 2008



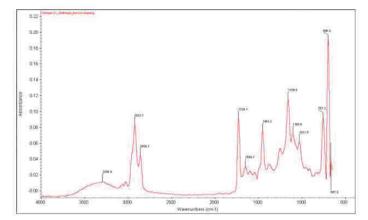
Surface of murals in UV light

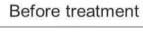
Results of identification of organic compounds (acrylics) by FTIR Analysis

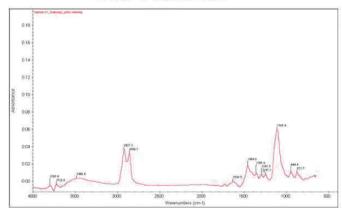




(FTIR Analysis Protocol by K.Bayer, 2013)



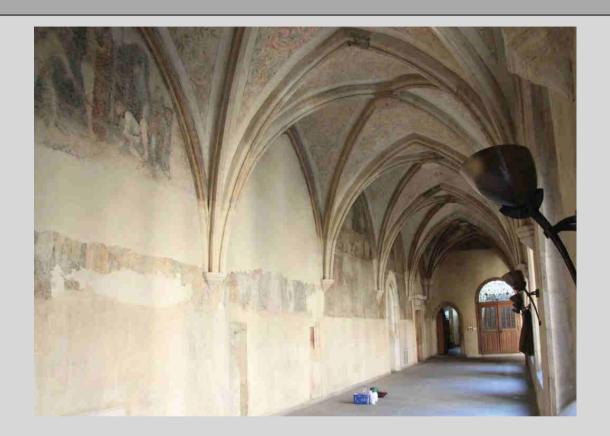


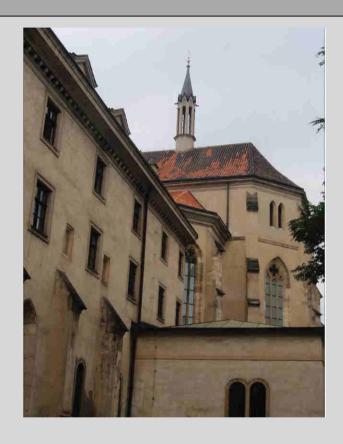


After treatment

material	exposure period	efficiency	est. damage to painting
XYL	5 minutes	96%	does not occur
EAPC	5 minutes	80%	minor damage
MEB	7 minutes	90%	larger damage

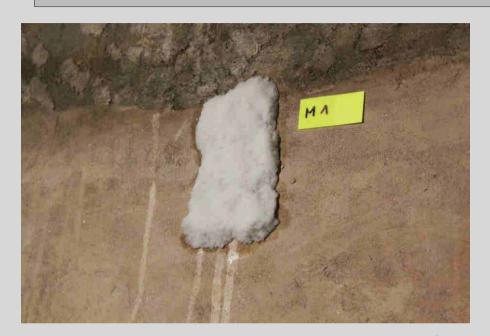
Wall painting: Cloister of Benedictine Monastery, Prague





- The best results show XYL with exposure period of 5 minutes.
- The fixation layers were removed up to approximately 90% without traces of visible damages to the painting.
- EAPC and MEB shows also satisfactorily results, nevertheless minor damages of paint layer observed under detailed observation.

Cleaning effect of XYL and MEB on heavily covered surface with acrylic dispersion





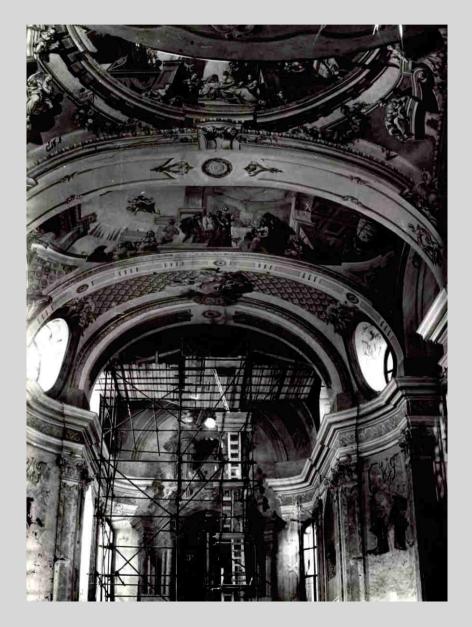
Application of cleaning poultices in Arbocel

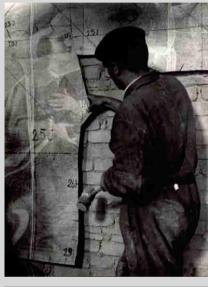


Both products proved to be satisfactory for the removal of aged acrylic dispersions after 5+5 min In cases of thicker layer the additional mechanical cleaning was easily applicable

Wall painting: Transferred panels from St Elisabeth Church in Doupov

church demolished in 1960's, walls decorated with mural paintings in 1777 by J.Kramolin







- The designated interior paintings were fixed with Disapol (locally produced acrylic emulsion)
- Subsequently covered by three layers of gauze (light fabric with the weave of 10 x 10 threads per cm²).
- A mixture of Lovosa (kind of CMC) and Disapol in the ratio of about 10:1 was used as glue

The least important panel was given for testing
The major damage was caused by the insufficient fixing of paintings before gluing
Powdered paint layer was coming off in many cases.

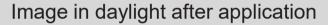












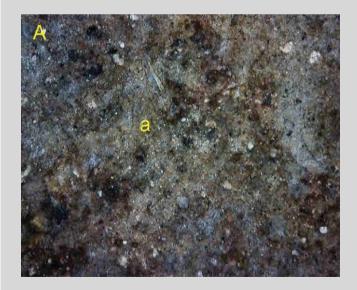
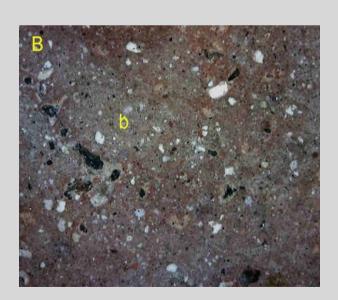




Image in UV light after appl.



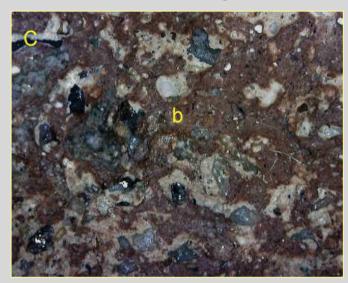
After 7 minutes The organic layer was removed except of minor fragments.

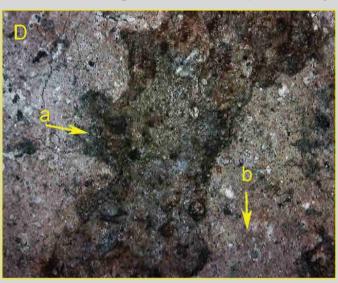
Surface before and after dispersion removal by XYL Macro-images

- A- detail before
- B- detail after the removal of the glue

Surface after dispersion removal by EAPC (C) and MEB (D)

While in case of using EAPC and MEB there were damages into the paint layer.





XYL material with exposure period of 7 minutes proved to be the gentlest and most effective.



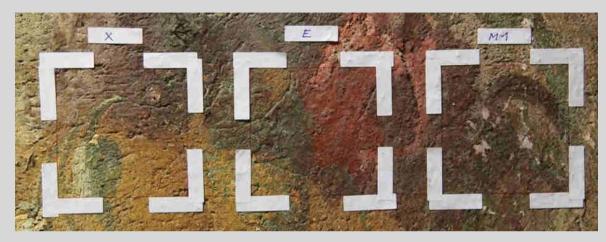
Although the color layer locally came off, it's clear becouse the color layer had not been properly fixed.



Dispersion has been removed from ca 95% of the test surface.(UV)

Mathematical evaluation of the effectivity of cleaning

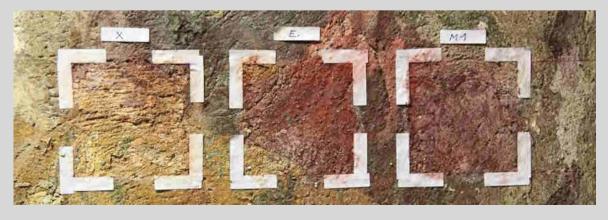
by Nikon Instrument software (NIS-Elements software)



Test areas before treatment, exact area required

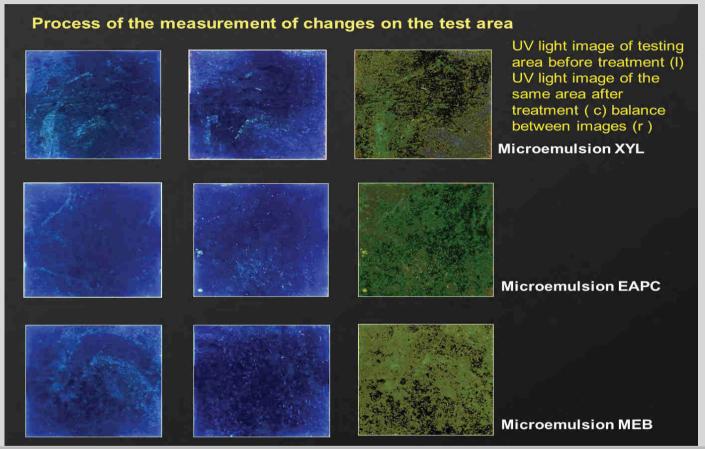


Materials XYL, EAPC, MEB in cellulose applied on test areas.



Test areas after treatment by microemulsions

The measurement method of changes between the reference image and the image of area treated by microemulsions consists in overlapping of pictures and substraction of their differences



Far right images show the balance after marking changes in the area.

Mathematical calculation declared the removal of acrylic coating in the extent of

57 % by XYL

68 % by MEB

63 % by EAPC

With increasing time of exposure the cleaning power increases. But one must consider that any further increase of the time of application may cause irreversible damage of the paint layer underneath.

material	time of application	efficiency	damage to painting
XYL	7 minutes	95%	minor damage only on secco layer
EAPC	10 minutes	90%	minor damage to secco as well as fresco layer
MEB	7 minutes	80%	larger damage to both layers

The table shows the best possible results after the application of micro-emulsions

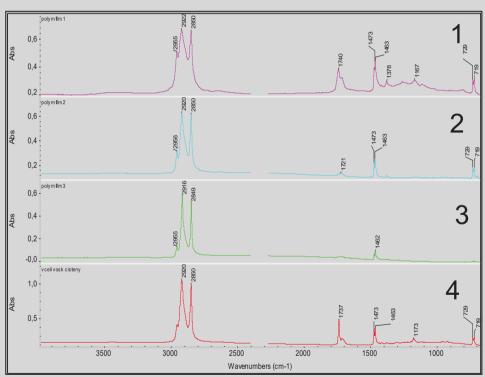
Conclusion on mural paintings cleaning

- All types of micro-emulsions showed very good and fast efficiency if used for the removal of acrylic dispersions tightly connected with the paint layers of mural paintings
- The effectiveness is highly dependent on the time of application which may bring difficulties in the control of the treated paint layer during the procedure
- Very good results were observed already after 5 minutes of the application
- The best results showed XYL product in all cases of testing
- Effectivity of safe cleaning is highly dependent on the technique and quality of paint layers

Stone Cleaning Tombstones in The Old Jewish Cemetery, Prague



Limestone with wax surface layer



FTIR

- 1, 2, 3 surface layers
- 4 beeswax



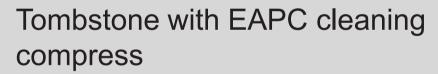


Results of preliminary testing



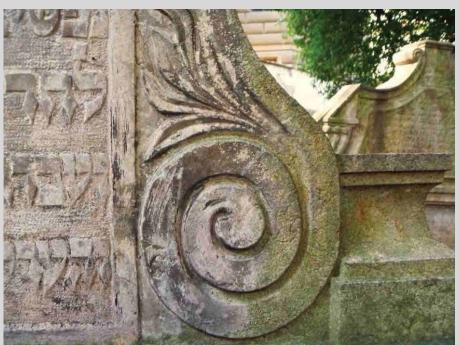
Detail of one of cleaned areas 14 months after cleaning





before application of nanoemulsion

after application of nano-emulsion

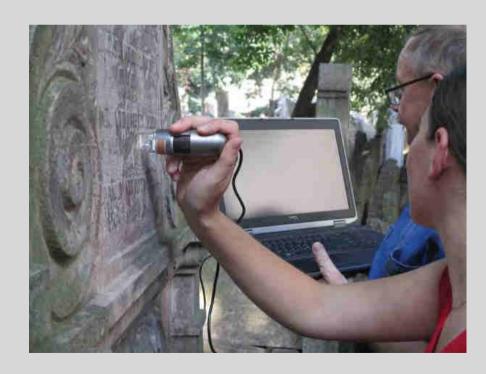






Microscopic photo of tested area cleaned with micro-emulsion

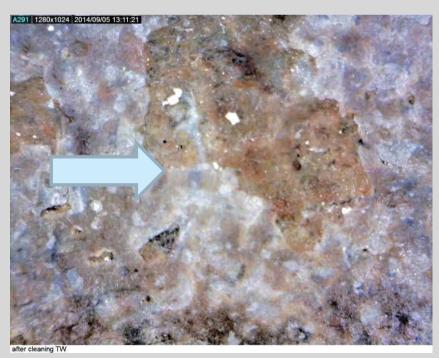






Cleaning with micro-emulsion as a selective process

tested area before cleaning



tested area after cleaning
new thin layer was uncovered

Conclusion on stone cleaning

- Detailed documentation confirmed excellent result of cleaning of aged organic layers for all tested nano-emulsions.
- Microscopy images show very good and sensitive removal of layers.
- Process of removal confirmed the ability to remove layers selectively
- Time of application was extended up to 24 hours as softening of the organic layers required much longer time to become removable
- XYL product was evaluated as the most effective product used for testing

In case of tombstone the first layer was washed away by rinsing while the other layer became softened to make the mechanical cleaning easily workable.



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