



# MINEX<sup>®</sup>

Functional Filler

## Evaluation of MINEX<sup>®</sup> in clear coatings

### Part I: Performance studies in an aqueous, UV-curable polyurethane formulation

#### Abstract

A multiphase study was conducted with an independent test laboratory to evaluate the performance of MINEX products (MINEX 7, 10, and 12, the latter a new ultra fine grade), in clear wood coating formulations. In this phase of study, MINEX 7, 10, and 12 were evaluated in a standard UV-curable water based polyurethane, commonly used as a clear coat over wood cabinetry, at 6, 12, 18 and 24 % loading. The addition of MINEX at all levels demonstrated significantly improved block and abrasion resistance. MINEX had minimal impact on crosshatch adhesion, while improving scrape adhesion at an optimal 12% loading. Evaluations for optical clarity, gloss, hardness and package stability confirmed there were significant additional advantages for the ultra fine MINEX 12, offering the best optical clarity, least haze development, least settling, and a more uniform increase in hardness with increased loading. For example, an 18% loading of MINEX 12 showed only a slight decrease in finish clarity (i.e. haze increase < 5%). Combined phase 1 results show that MINEX, and especially MINEX 12, offers numerous performance enhancements over un-pigmented systems in clear coatings applications.

#### Objective

Determine the effect of MINEX additions on the optical and physical properties of a UV-curable, waterbased poly urethane clear coating system. Compare the performance of alternative MINEX grades with different particle size distributions.

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## Materials and methods

This phase of study utilized an aqueous UV cure formulation based on Bayhydrol UV VP LS 2317, a polyurethane resin containing acrylic functional groups. Performance of MINEX 7, 10, and 12 in this binder system as a function of mineral loading from 0 – 24% by weight based on coating clear solids was evaluated. Coating volume solids were held constant for the various evaluations discussed in this report. Table 1 provides details on the composition and method of preparation of the samples. No defoamers or wetting/dispersing agents were used in any of the formulations.

### 1. Evaluation of haze and gloss

Gloss – samples from Table 1 were stirred for a minimum of 10 minutes and then applied to a Leneta chart, Form 2A with a 30 RDS rod. Samples were allowed to air dry for 10 minutes, followed by forced drying at 49C for 10 minutes. Samples were then exposed to 3 passes through an American Ultraviolet UV reactor equipped with a medium pressure mercury lamp housed in an elliptical reflector. The lamp wattage was set at 300 WPI, and the belt speed at 25 FPM. The cure energy per pass was measured at 330 mj/cm<sup>2</sup> and 0.674 W/cm<sup>2</sup> UVA. 60 degree gloss was determined over the black portion of the chart with a BYK Gardner Tri-Gloss instrument.

Haze – samples from table 1 were stirred for a minimum of 10 minutes and re-filtered through 150 mesh silk. Samples were then applied to a cleaned 1 X 3 microscope slide with a 30 RDS rod. Coatings were dried and cured as above. Haze of the films was characterized by ASTM D1003-61 utilizing a Cary 100 spectrophotometer equipped with a 73mm diameter diffuse reflectance integrating sphere. A blank slide was utilized when obtaining the baseline and scatter of the instrument.

### 2. Determination of pencil hardness

Samples from table 1 were stirred for a minimum of 10 minutes and then applied to plate glass with a 30 RDS rod. The coated glass was allowed to air dry for 10 minutes, followed by forced drying at 49°C for 10 minutes. Coatings were UV cured as described above. Gouge pencil hardness was determined using ASTM D3363.

### 3. Determination of scratch resistance and film adhesion

Panel development – samples from table 1 were stirred for a minimum of 10 minutes and adjusted to 25% vehicle solids for spray application to cherry veneer wood panels.

Topcoats were dried (10min @41°C, 14% RH, 100L/min air movement; 10min @49°C) and cured (3 passes, UV unit equipped with medium pressure mercury lamp, at 300 WPI, elliptical reflector, approx. 361 mj/cm<sup>2</sup>, .686 w/cm<sup>2</sup> per pass). Topcoat dry film thickness averaged between 15 and 18 microns.

Abrasion measurements – approximately 1 inch wide strips of each panel were cut for evaluation. The panels were subjected to a total of 50 #1-grade steel wool double rubs. Change in gloss after 0, 10, 25, and 50 double rubs for each sample was determined.

Crosshatch and scrape adhesion measurements – tape crosshatch was determined using ASTM D3359. Each panel was evaluated for scrape adhesion by pushing the coated panel under a loop stylus over which is mounted a given amount of weight. The greatest weight at which the coating shows no to only a slight amount of release, whether by fracture, scraping, chatter, or some other type of film failure, was recorded as the adhesion value in KG. Weights were determined in 0.5KG increments. Test scrape areas were evaluated by stereoscopic observation.

Table 1

Formulation, wt/grams					
Item					
% Wt MINEX on clear solids	0	6	12	18	24
Charge items in order listed under agitation					
Bayhydrol UV VP LS 2317	100	100	100	100	100
Irgacure 500	1.5	1.5	1.5	1.5	1.5
Mix 20 minutes, then add					
Acrysol RM 825	1.0	1.0	1.0	1.0	1.0
Mix well, then sift in					
MINEX		2.34	4.68	7.03	9.37
Mix 15 minutes, hock blade, high speed, approx. 2200 rpm; evaluate grind; then let down with the following					
DI Water	23.33	25.31	27.34	29.38	31.41
BYK346	0.62	0.62	0.62	0.62	0.62
Mix 10 minutes at approx. 800 rpm; Filter 150 mesh					
pH MINEX 12	7.13	7.11	7.04	7.06	7.21
pH MINEX 10	7.13	7.06	7.13	7.27	7.19
pH MINEX 7	7.13	7.27	7.16	7.31	7.47
Viscosity, cps, RVDVE, sp6, 50ml Grad Cylinfer, 78F					
50 rpm MINEX 7	540 @ 2.7%T	970 @ 4.8%T	690 @ 3.3%T	420 @ 2.0%T	470 @ 2.2%T
100 rpm MINEX 7	565 @ 5.6%T	825 @ 8.2%T	645 @ 6.4%T	415 @ 4.1%T	445 @ 4.5%T
50 rpm MINEX 10	540 @ 2.7%T	730 @ 3.5%T	360 @ 1.8%T	300 @ 1.5%T	360 @ 1.9%T
100 rpm MINEX 10	565 @ 5.6%T	670 @ 6.7%T	395 @ 3.9%T	330 @ 3.3%T	370 @ 3.7%T
50 rpm MINEX 12	540 @ 2.7%T	330 @ 1.5%T	400 @ 2.1%T	170 @ 0.9%T	320 @ 1.6%T
100 rpm MINEX 12	565 @ 5.6%T	355 @ 3.5%T	420 @ 4.2%T	205 @ 2.1%T	350 @ 3.5%T

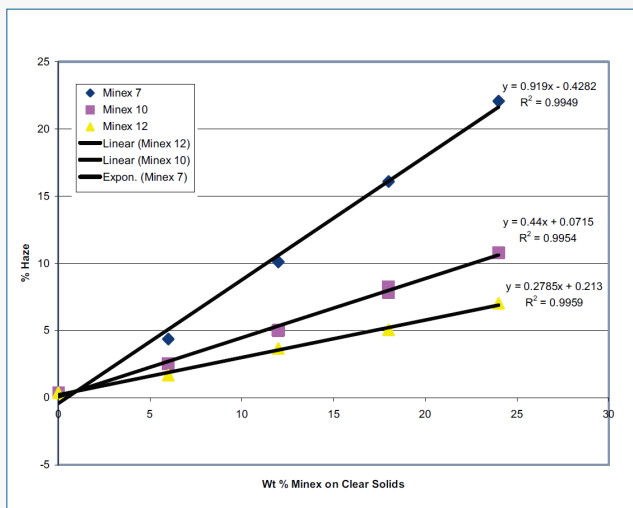
## Results and conclusions

### A. Haze and gloss

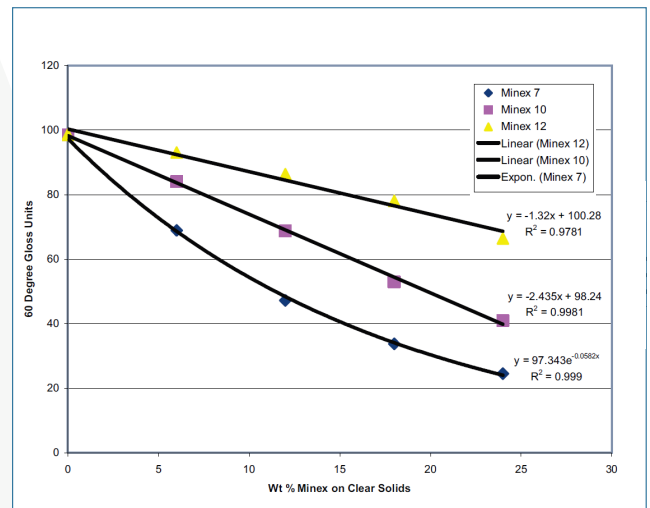
Graph 1 compares and summarizes the effect of mineral type and concentration on haze development. The data indicate that 18% MINEX 12 may be utilized in this coating with a minimal (5%) increase in haze. An 18% loading of MINEX 12 results in a gloss decrease of about 20 points relative to an unmodified coating (Graph 2). MINEX 10 additions decrease gloss more dramatically (although the effect of MINEX 10 additions on haze is not as significant).

### B. Pencil hardness

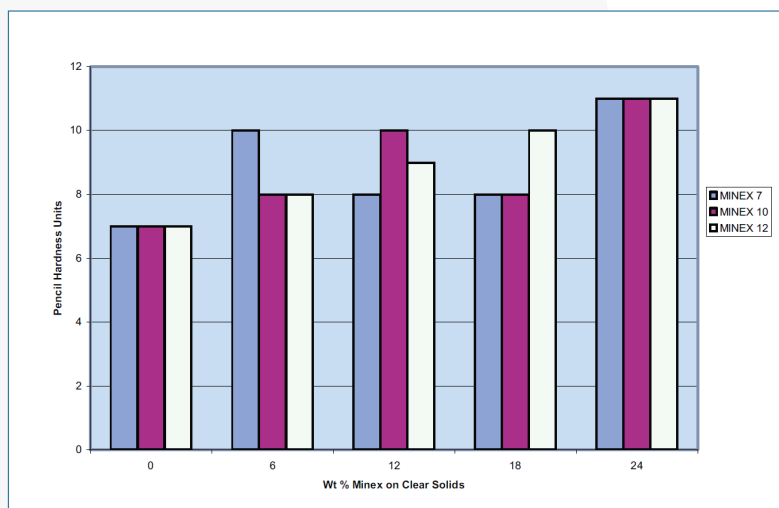
Graph 3 summarizes the hardness determinations. All MINEX grades result in equal hardness development at 24% loading. The data suggest that MINEX 7 results in the greatest hardness at the lowest mineral loading (6%), while the rate of hardness increase with concentration is more consistent with MINEX 12 additions.



Graph 1. % haze (ASTM D1003-61) as a function of MINEX type and concentration



Graph 2. 60 degree gloss as a function of MINEX type and concentration



Graph 3. Gouge hardness as a function of MINEX type and concentration

## Results and conclusions

### C. Scratch resistance, adhesion performance and block resistance

All samples containing MINEX demonstrated dramatically improved scratch resistance as compared to the unmodified control measured by #1-grade steel wool double rubs under a 1500 gram load over approximately 0.5in<sup>2</sup>. Graph 4 compares the scratch resistance of the MINEX modified samples compared to an unmodified sample after 50 double rubs. Photo 1 shows an image comparing the steel wool scratch resistance of MINEX 12 modified samples compared to an unmodified sample.

All coatings containing MINEX gave acceptable cross hatch adhesion at all levels evaluated. 12% MINEX 10 and 12 were found to be an optimum concentration for Gardner scrape adhesion performance, resulting in greatly improved scrape adhesion compared to other samples (including the sample without MINEX.)

MINEX addition resulted in significantly improved block resistance compared to the sample with no mineral present. Whereas the unmodified sample required a force of 1.43kgf to separate a face-to-face sample, the coatings modified with MINEX were able to be separated with forces ranging between 0.32kgf to 0.61kgf with a 1000 gram load spread over approximately 2 in 2.

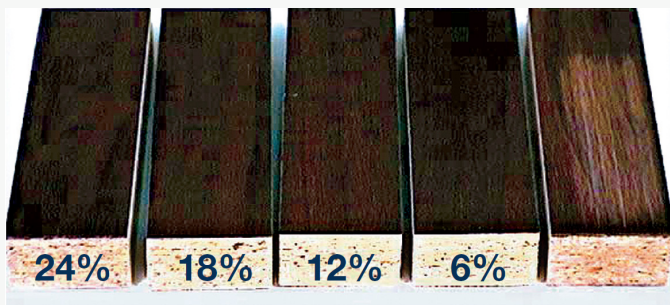
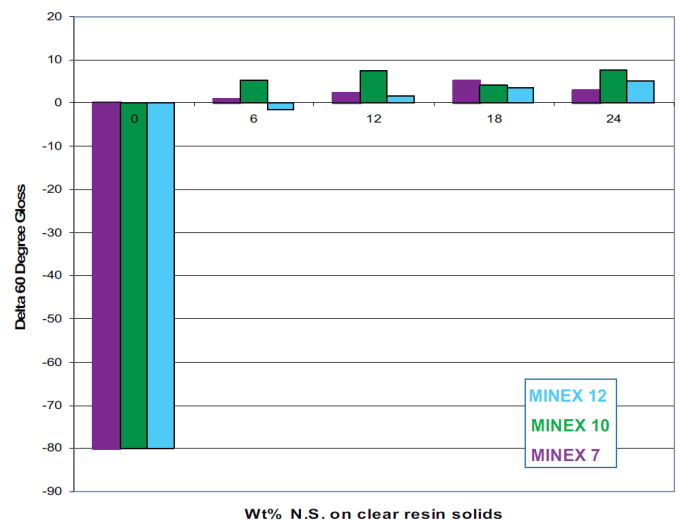


Photo 1 Steel wool double rub resistance of MINEX 12-modified aqueous UV cured coating compared to an unmodified coating. MINEX 12 loading levels as indicated.



Graph 4. Gloss change for 50 #1 grade steel wool double rubs as a function of MINEX type and concentration (1500 g load)

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