Energy-Cured Coatings – KMnO4 UV Cure Stain Test

Scope

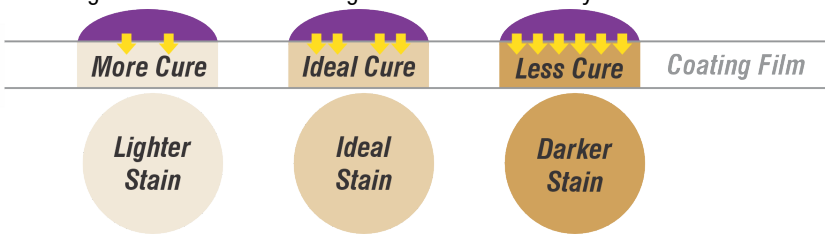
- Process control test to qualitatively and objectively measure the degree of cure for an Energy-Cured coating film applied to a substrate.
- KMnO4 (Potassium Permanganate) solution supplied by INXCAC produces a stain residue in the coating film that can be measured using a densitometer – actual density results can be compared to density reference targets to determine actual coating film cure condition.
- Ideal for use as an 'off-press' Operator quality test to verify the relative coating film cure condition and make necessary adjustments to optimize coating film cure – use to produce correct and consistent cure results.

Test Instruments

- 1% KMnO4 (Potassium Permanganate) Stain Solution Jar – sourced from INXCAC as a 'UV Cure Stain Test Kit'
  - Dispensing Pipette (supplied with kit)
  - Gloves (supplied with kit)
  - Soft Paper Towel/Rag (supplied with kit)
- Densitometer/Spectrophotometer
- Timer

Testing Considerations

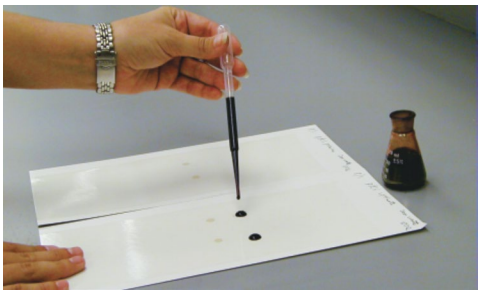
- KMnO4 stain solution has a shelf life of 1-year from DOM.
- If not sealed tightly during storage, the solution will oxidize in the jar and change from a deep purple color to a brown color – the solution is no good and not recommended for use if oxidized/brown.
- During the test, once the excess stain solution is blotted from the coating film surface, the resulting stain color can quickly fade and lose intensity contributing to an inaccurate result – density measurements should be made immediately and not longer than 5 minutes after stain solution removal.
- The KMnO4 solution absorbs into the coating film – the resulting stain residue and relative density measurement is observed as the degree of Energy-Cured coating film cure:
  - A more cured/cross-linked coating film will have better holdout with less absorption of the stain solution resulting in a lighter visual and lower measurable density stain result.
  - A less cured/cross-linked coating film will have less holdout with more absorption of the stain solution resulting in a darker visual and higher measurable density stain result.



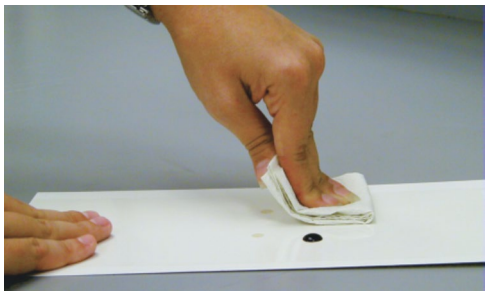
- Care should be taken to avoid contaminating skin, clothing or porous surfaces with the KMnO4 stain solution as exposure can result in staining that is difficult to remove – the use of gloves is recommended.

Test Procedure

1) Obtain the coated sample to be tested – age of the sample does not matter:
   - □ There must be ink-free areas that are coated for stain testing – stain testing cannot be conducted over ink areas.
   - □ Conduct testing on a flat surface – solution droplets will run on an uneven surface.
2) Shake the KMnO4 stain solution bottle prior to each use.
3) Using the supplied pipette, apply the stain solution in droplets to coated areas of the sheet without ink:
   - □ Apply enough solution in each test area to create a circle large enough for measuring with a densitometer – a droplet area the size of a dime is usually sufficient.



- Start the timer once the first droplets have been applied.
- Apply droplets across the sheet to survey the entire sheet width, edge to edge.
- Make sure there are no air bubbles in the droplets.
- Allow solution droplets to remain on the coated sample for precisely 5-minutes in duration prior to removal.



4) At the end of the 5-minute test time, carefully blot the droplet areas to remove excess solution using a soft towel/rag:
   - In the case of a severely under-cured/soft coating film, aggressive wiping of the droplet from the substrate surface can remove some of the stained coating film – this produces a false result as the stain appears lighter (more cured) than is actually the case due to some of the stained coating having been removed.
5) A light brown/orange stain will be observed in the test areas.
6) Observe the stain area for intensity/darkness:
   - Stain color intensity can be visually compared to a reference color standard.
   - For most precise interpretation, measure for density using a densitometer or spectrophotometer.

- When measuring density:
  - Must be a RELATIVE measurement – substrate white subtracted from the density result.
  - Must use the Yellow (Y) density filter.
  - Both settings can be applied automatically with most modern handheld densitometer/spectrophotometer systems.

Stain Result Interpretation

- The charts below can be used to correlate the measured density result of the stain and the actual degree of Energy-Cured coating film cure based on coating type – these are general references:

<Over-Cure			Optimum Cure			Under-Cure>
Gloss Coating						
<0.20	0.20 – 0.25	0.25 – 0.30	**0.30**	0.30 – 0.35	0.35 – 0.40	>.040
Satin Coating						
<0.40	0.40 – 0.45	0.45 – 0.50	**0.50**	0.50 – 0.55	0.55 – 0.60	>0.60
Matte/Dull Coating						
<0.60	0.60 – 0.65	0.65 – 0.70	**0.70**	0.70 – 0.75	0.75 – 0.80	>0.80

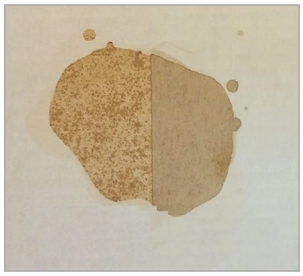
- For specific KMnO4 density targets related to a specific coating product and/or application, contact your INXCAC Technical Sales Representative.

Over-Cure

- Over-curing the coating film has been found to promote film brittleness, lack of pliability, fracturing/flaking, poor adhesion, cracking along scores and folds, and an overall loss of film flexibility.
- Over-curing the coating film will contribute to film hardness which can lower COF/AOS results to below target specifications.
- When using a contrast/migration 'strike-thru' varnish in conjunction with a gloss coating to create a gloss/matte contrast effect, over-curing the coating film can slow/impair the migration process and provide insufficient and undesirable contrast results – the matte varnish areas will not realize the full low-gloss potential.

Optimum-Cure

- Optimum cure of the coating film ensures that desired characteristics are achieved in respect to formulation and intended use.
- Proper coating film cure creates the highest probability of adequate adhesion being achieved while promoting desired attributes for gloss, COF/AOS, rub protection, chemical resistance, adhesion of additional surface applications and film flexibility.

	Under-Cure • Under-curing the coating film has been found to create a soft coating film, poor rub/scratch/mar resistance, clinging/sticking/bricking, poor chemical resistance and high COF/AOS results. • An under-cured gloss coating film will not achieve the formulated gloss potential.
Stain Result - Visual Inspection	- In addition to providing measurable qualitative guidance to the degree of Energy-Cured coating film cure, visual inspection/assessment of the stain result can provide details related to the quality of the applied coating film as it relates to: • Coating film coverage, uniformity • Foaming/entrained-air resulting in coating film defects – voids/pinholes • Anilox roll condition – insufficient coat-weight • Defects in beneath primer coating layer – voids/pinholes • Uneven/irregular substrate hold-out - When observing the stain, the result should be homogenous in color throughout the entire stain area. - In circumstances where 'freckles' are observed as darker spots within the stain test area, this is an indication that the stain solution has absorbed at an irregular rate within the stain area, with the stain solution becoming concentrated in areas or being absorbed by the substrate creating the darker 'freckle' spots – this can be due to: • The Energy-Cured coating film contains voids/pinholes which allows the stain solution to be concentrated or applied directly to the substrate and absorbed. • In cases where a waterbased primer coating is used beneath the Energy-Cured coating to impart hold-out to the substrate, voids/pinholes in the primer coating film can allow the stain solution to absorb into the substrate. • If the substrate surface offers very uneven/irregular hold-out of the Energy-Cured coating, the absorbency of the stain solution can result in a mottled stain appearance. • If the anilox is worn/clogged and the applied coat-weight is insufficient, the engraving pattern of the anilox may become visible in the stain area. - The below image shows the visual difference side-by-side between a stained test area that exhibits 'freckles' (left) versus a stained area that has a more homogenous stain result (right):  - When encountering a 'freckled' stain result, the coating should be examined for adequate coat-weight, coverage and lay. - If the coating is starved on the substrate or suffers from entrained-air/foaming which can create pinholes/voids in the applied film, measures should be taken to remedy these issues. - If the results are determined to be substrate related, the use of a primer coating to 'size' the sheet and improve the hold-out/barrier properties of the substrate surface prior to applying an Energy-Cured coating is recommended. - Changing to a different substrate with better surface characteristics may improve/eliminate the 'freckle' results. - When 'freckles' are observed in the stain area, this can result in a false/misleading density measurement as the darker stain spots can contribute to a higher measured density reading: • In this case, the darker 'freckles'/spots yielding a higher density reading would give a false indication of the coating film being under-cured. • When significant 'freckles' are observed in the stain area, the density measurement should not be considered for use – other methods should be used to determine proper film cure.
Stain Test Result - UV Curing System	- The stain test can be used as a qualitative method to determine the condition of the curing system as it relates to consistent output across the coated sheet, DS to OS:

Condition	• Measuring the stain result across the entire sheet width (edge to edge) can give an indication of lamp/reflector deficiencies which can appear as inconsistent stain density measurements. • In most cases, the outside edges of the sheet which correlate to the ends of the UV lamp/reflector may measure as being less cured compared to other areas within the sheet width due to lamp degradation or reflector contamination due to dust/debris. - Additionally, stain testing can be conducted from gripper to tail on the coated sheet to measure for inconsistencies that may be attributed to delivery gripper 'shadow' or sheet turbulence/flutter that might contribute to varying degrees of UV exposure across the sheet length. - Over the course of a UV lamp life, a slow drift in stain test results may be observed as the lamp loses efficiency – increased power/dwell is necessary to achieve the same stain result to compensate for UV system degradation.