

IOWA STATE UNIVERSITY

CASR

FAA Center for Aviation Systems Reliability

**CASR FPI – Engineering
Studies:
Emulsification Studies**



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<http://www.cnde.iastate.edu/faa-casr/fpi/index.html>



- When working with hydrophilic emulsifiers, current industry specifications provide allowable concentration ranges for immersion and spray application of each approved chemical, and limit the total contact time for the process.
- However, with complex parts, particularly those with cavities, ensuring that all surfaces are adequately covered, and that the emulsification process is stopped within the time limit can be quite challenging.



Four maximum emulsifier concentration ranges are listed in AMS 2647B

- 5% = 5% max
- 10% = 7-10% concentration
- 20% = 17-20% concentration
- 30% = 27-30% concentration

Three representative Level IV sensitivity hydrophilic PE penetrant families were chosen based on their manufacturer's recommended concentrations. They will be referred to as:

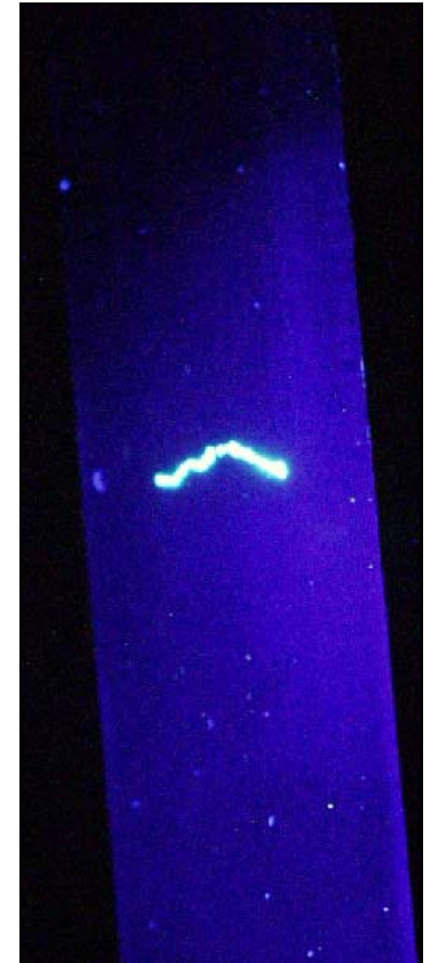
- PL-10 = 10% max
- PM-20 = 20% max (BASELINE MATERIAL)
- PH-30 = 30% max

Note: this study is not intended to be an exhaustive comparison of penetrant products, nor is it a qualification process study. Rather its purpose is to provide data from representative products which are typical of aerospace use.



This work monitored the change in FPI indication brightness while varying:

1. Concentration
 - Lower than recommended
 - Within the recommended range
 - Above the specified range
2. Application Method
 - Immersion
 - Spray
3. Agitation
 - No agitation
 - Periodic agitation
 - Constant agitation
4. Duration
 - Short emulsifier time
 - Maximum emulsification time allowable
 - Twice the maximum emulsification time

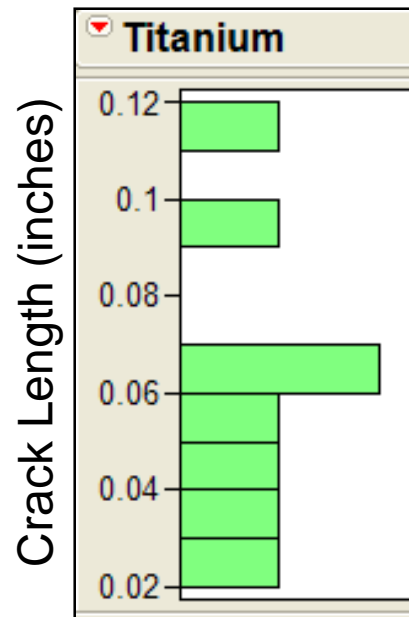
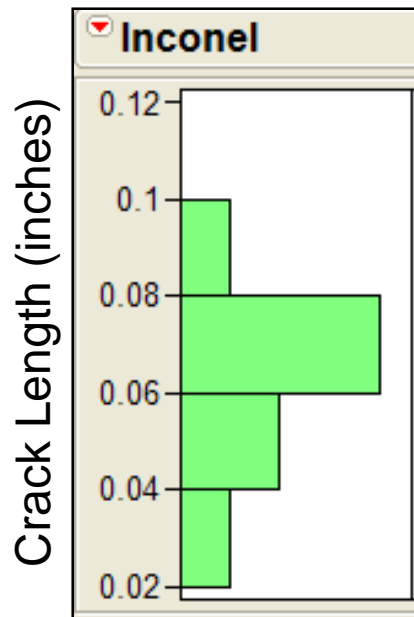


Example Indication



Samples were low-cycle fatigue (lcf) crack blocks

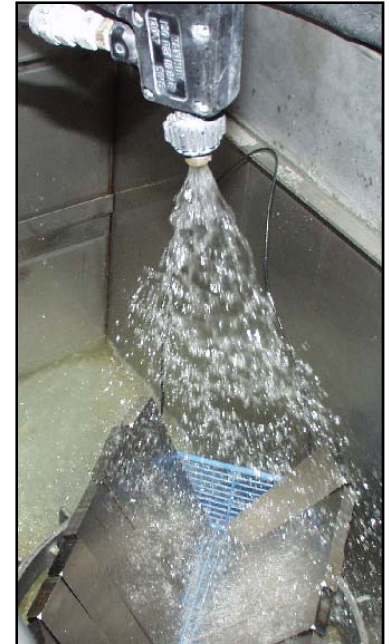
- (8) Inconel-718 and (8) Titanium 6-4
- EDM starter defect grown in 3-point bending
- Crack lengths ranged from 0.021" to 0.118" (0.060" aver.)
- Surface finish ranged between 7 and 20 μin (Ra)
- Aspect ratio ($a/2c$) \approx 0.4 - 0.5 (*based on past results*)





Inspection Process

- 20 minute penetrant dwell
- 90 second pre-wash
- Emulsification (varied process)
- 90 second post-wash
- 10 minute dry @ 155°F
- 10 minute development (dry powder, dip/drag)
- Photometer brightness and UVA microscope imaging
- 45 minute UT-agitated acetone clean
- 60 minute dry @ 155°F





Emulsification Methods

Immersion using a 5-gallon tub

- Varied concentration
- Varied emulsification time
- Varied agitation rate

Spray emulsification using a Hudson Bak-Pak[®] (*model 63184*)

- Constant concentration
- Varied emulsification time



Spray emulsification



Spray emulsification using a Hudson Bak-Pak® Sprayer (*model 63184*)

- 5% maximum concentration
- 60, 120, or 240 second spray
- flat fan spray nozzle
- ~80° spray angle
- regulated to 20 psi
- Approximately 1,200 mL/minute
- 12" stand-off distance
- 1 spray pass every 2 seconds



Backpack sprayer for
emulsification

Spray emulsification





Immersion using a 5-gallon tub

- Concentration
 - PL-10 material
 - 5%, 10%, 15%, 20%
 - PM-20 material
 - 15%, **20%**, 25%
 - PH-30 material
 - 20%, 25%, 30%, 35%
- Time
 - 60, **120**, and 240 seconds
- Agitation
 - none, **15 second intervals**, and constant

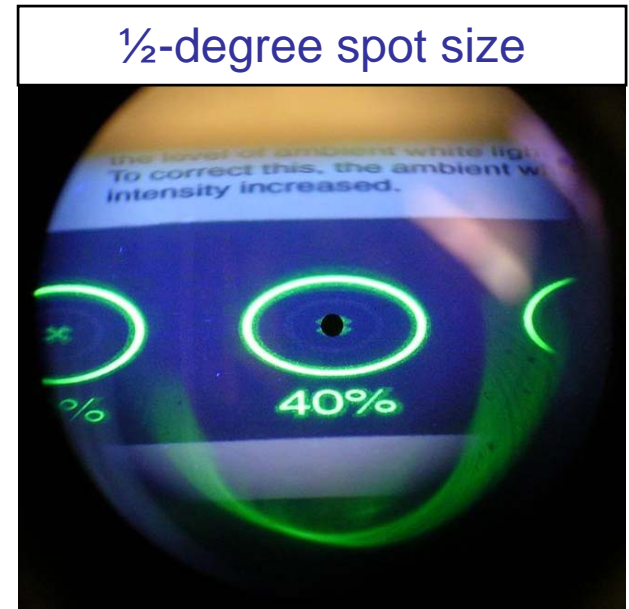
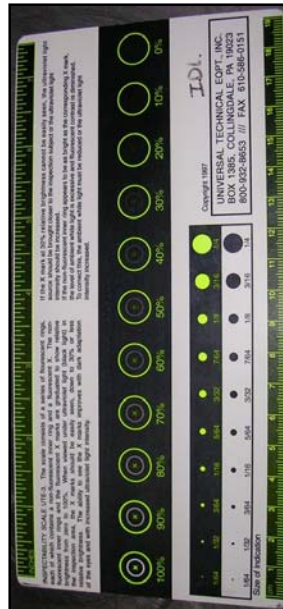
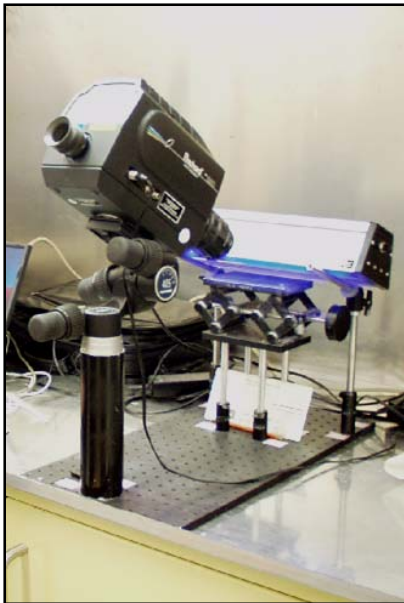
=Baseline Procedure



Emulsifier immersion



- Brightness measurements were made with a Pritchard PR-880 photometer by Photo Research
- UVA intensity measured with Spectroline DSE-100X and broadband DIX-365 sensor
- UVA illumination provided by twin 40W fluorescent bulbs
- Indication images captured using a Leica MZFLIII UVA binocular microscope and QImaging Retiga 1300 cooled camera



How Was It Performed



Run #	Penetrant	Emulsifier concentration	Emulsifier agitation	Emulsifier contact time
1	PM-20	17 - 20%	lifting every 15 sec	120 sec
2	PM-20	17 - 20%	lifting every 15 sec	120 sec
3	PM-20	17 - 20%	lifting every 15 sec	60 sec
4	PM-20	17 - 20%	lifting every 15 sec	240 sec
5	PM-20	17 - 20%	lifting every 15 sec	120 sec
6	PM-20	25%	lifting every 15 sec	120 sec
7	PM-20	15%	lifting every 15 sec	120 sec
8	PM-20	17 - 20%	lifting every 15 sec	120 sec
9	PM-20	17 - 20%	lifting every 15 sec	120 sec
10	PM-20	17 - 20%	continuous agitation	120 sec
11	PM-20	17 - 20%	no agitation	120 sec
12	PM-20	17 - 20%	lifting every 15 sec	120 sec
13	PM-20	5%	spray application	120 sec
14	PM-20	5%	spray application	120 sec
15	PM-20	5%	spray application	240 sec
16	PM-20	17 - 20%	lifting every 15 sec	120 sec

 = Baseline Procedure

How Was It Performed



Run #	Penetrant	Emulsifier concentration	Emulsifier agitation	Emulsifier contact time
17	PH-30	27 - 30%	lifting every 15 sec	120 sec
18	PH-30	27 - 30%	lifting every 15 sec	120 sec
19	PH-30	27 - 30%	lifting every 15 sec	60 sec
20	PH-30	27 - 30%	lifting every 15 sec	240 sec
21	PH-30	27 - 30%	lifting every 15 sec	120 sec
22	PH-30	20%	lifting every 15 sec	120 sec
23	PH-30	20%	lifting every 15 sec	240 sec
24	PH-30	25%	lifting every 15 sec	120 sec
25	PH-30	35%	lifting every 15 sec	120 sec
26	PH-30	27 - 30%	lifting every 15 sec	120 sec
27	PH-30	27 - 30%	continuous agitation	120 sec
28	PH-30	27 - 30%	no agitation	120 sec
29	PH-30	27 - 30%	lifting every 15 sec	120 sec
30	PH-30	5%	spray application	120 sec
31	PH-30	5%	spray application	240 sec
32	PH-30	27 - 30%	lifting every 15 sec	120 sec
33	PM-20	17 - 20%	lifting every 15 sec	120 sec
34	PM-20	17 - 20%	lifting every 15 sec	120 sec

= Baseline Procedure

How Was It Performed



Run #	Penetrant	Emulsifier concentration	Emulsifier agitation	Emulsifier contact time
35	PL-10	7%-10%	lifting every 15 sec	120 sec
36	PL-10	7%-10%	lifting every 15 sec	120 sec
37	PL-10	7%-10%	lifting every 15 sec	60 sec
38	PL-10	7%-10%	lifting every 15 sec	240 sec
39	PL-10	7%-10%	lifting every 15 sec	120 sec
40	PL-10	5%	lifting every 15 sec	120 sec
41	PL-10	15%	lifting every 15 sec	120 sec
42	PL-10	20%	lifting every 15 sec	120 sec
43	PL-10	20%	lifting every 15 sec	240 sec
44	PL-10	7%-10%	lifting every 15 sec	120 sec
45	PL-10	7%-10%	continuous agitation	120 sec
46	PL-10	7%-10%	no agitation	120 sec
47	PL-10	7%-10%	lifting every 15 sec	120 sec
48	PL-10	5%	spray application	120 sec
49	PL-10	5%	spray application	240 sec
50	PL-10	7%-10%	lifting every 15 sec	120 sec
51	PM-20	17 - 20%	lifting every 15 sec	120 sec
52	PM-20	17 - 20%	lifting every 15 sec	120 sec

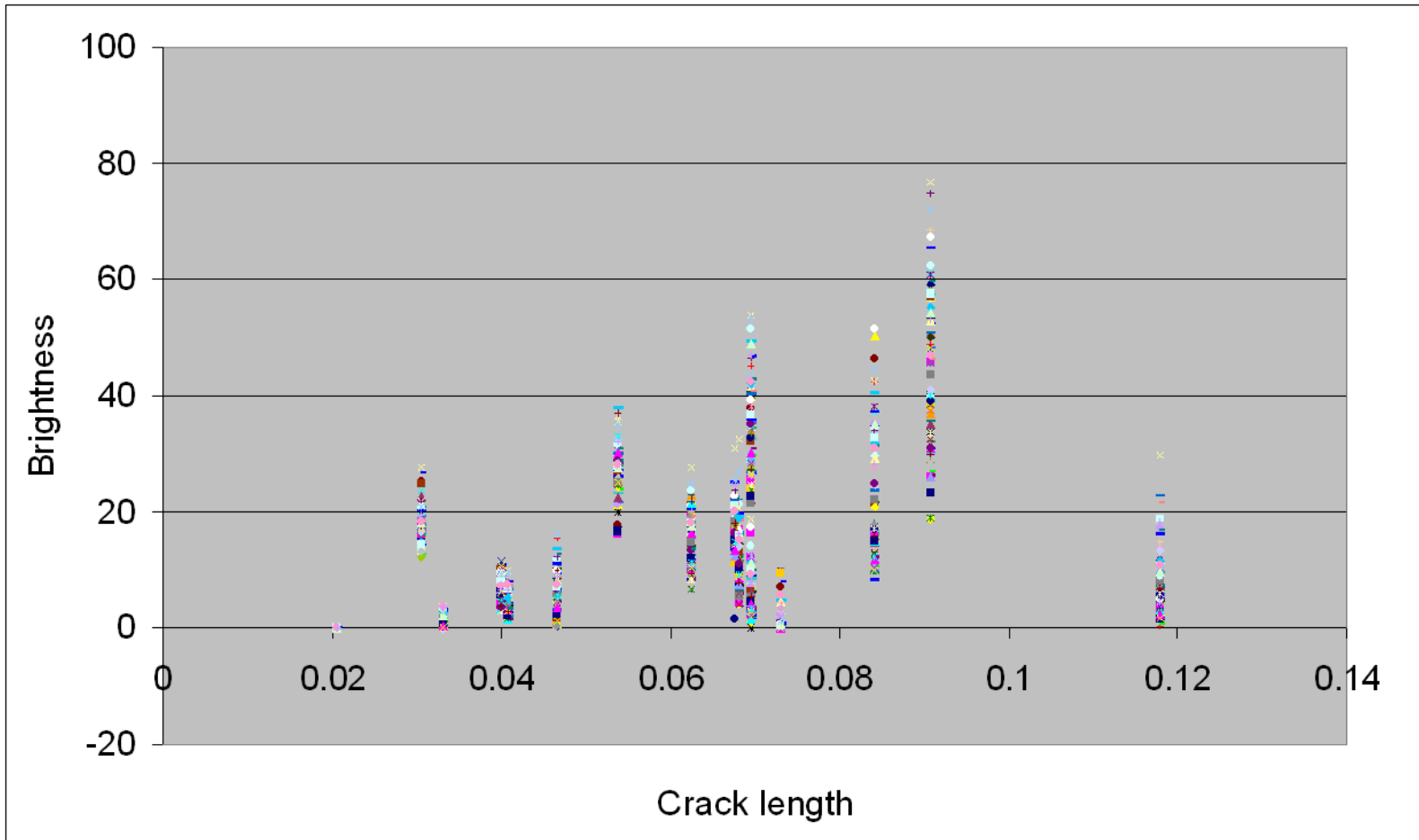
= Baseline Procedure

How Was It Performed

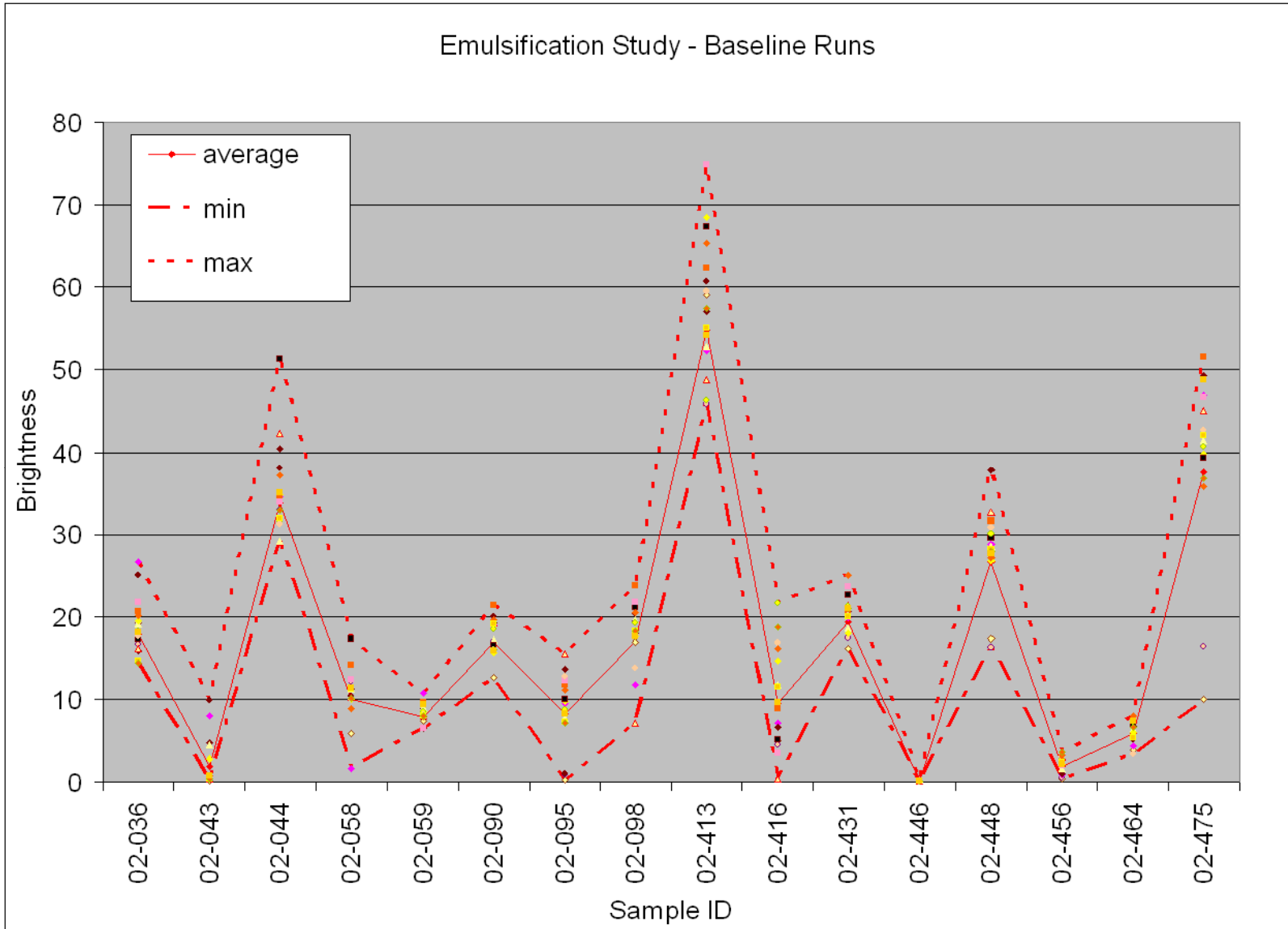


53	PM-20	17 - 20%	lifting every 15 sec	120 sec
54	PM-20	17 - 20%	lifting every 15 sec	240 sec
55	PM-20	17 - 20%	lifting every 15 sec	480 sec
56	PH-30	27 - 30%	lifting every 15 sec	120 sec
57	PH-30	27 - 30%	lifting every 15 sec	240 sec
58	PH-30	27 - 30%	lifting every 15 sec	480 sec
59	PL-10	7%-10%	lifting every 15 sec	120 sec
60	PL-10	7%-10%	lifting every 15 sec	120 sec
61	PL-10	7%-10%	lifting every 15 sec	120 sec
62	PM-20	17 - 20%	lifting every 15 sec	120 sec
63	PM-20	17 - 20%	lifting every 15 sec	120 sec
64	PM-20	17 - 20%	lifting every 15 sec	120 sec
65	PM-20	17 - 20%	lifting every 15 sec	120 sec
66	PM-20	17 - 20%	lifting every 15 sec	120 sec
67	PH-30	27 - 30%	lifting every 15 sec	120 sec
68	PH-30	27 - 30%	lifting every 15 sec	240 sec
69	PL-10	7%-10%	lifting every 15 sec	120 sec
70	PL-10	7%-10%	lifting every 15 sec	240 sec

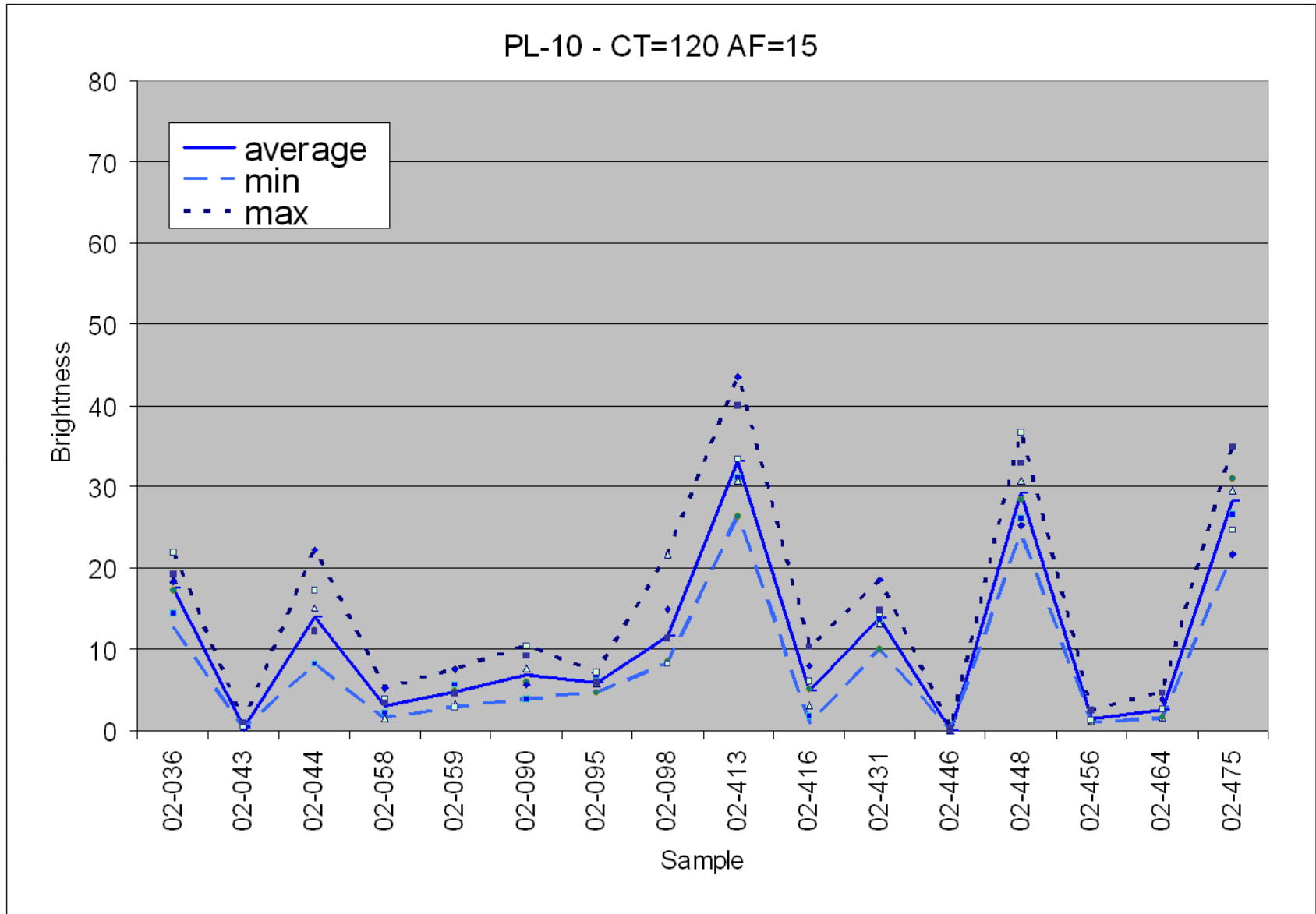
 = Baseline Procedure



- Variation in brightness for all samples, all runs

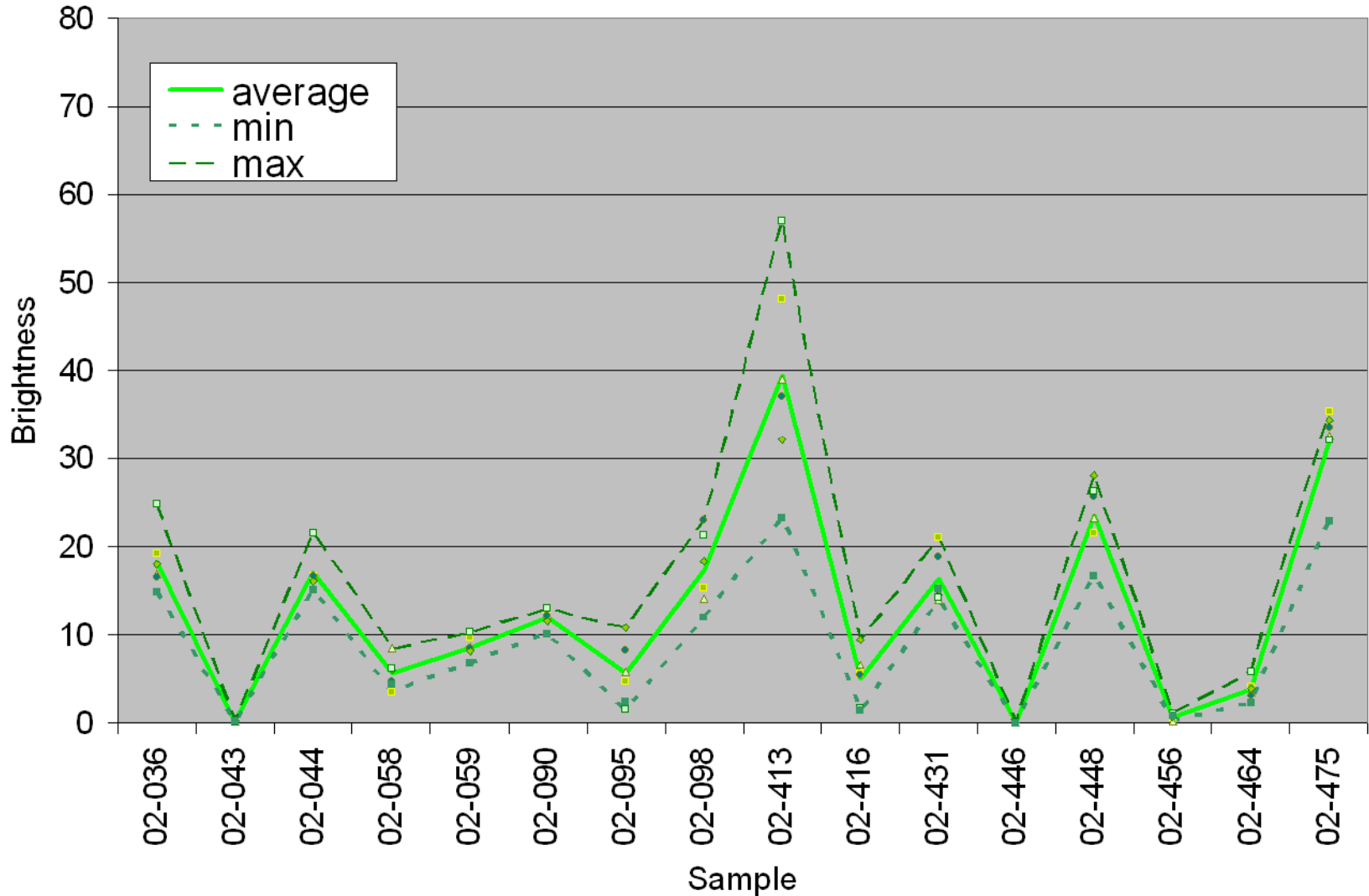


PL10 - CT=120 - AF=15





PH-30 - CT=120 AF=15



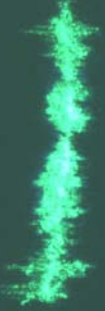
Sample 044 – PM20



CT: 60 sec

B: 50.2

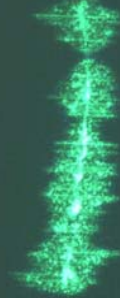
R4



CT: 120 sec

B: 38.7

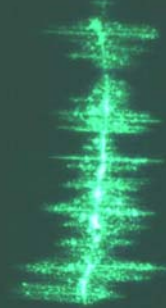
R6



CT: 240 sec

B: 37.3

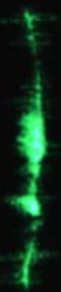
R5



CT: 120 sec

B: 31.6

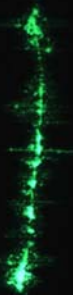
R53



CT: 240 sec

B: 23.6

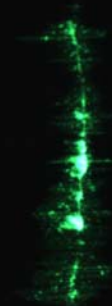
R54



CT: 480 sec

B: 24.7

R55



Sample 058 – PM20



CT: 60 sec

B: 7.84

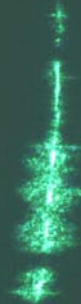
R4



CT: 120 sec

B: 10.3

R6



CT: 240 sec

B: 9.69

R5



CT: 120 sec

B: 11.2

R53



CT: 240 sec

B: 9.79

R54



CT: 480 sec

B: 9.32

R55



Sample 446 – PM20



CT: 60 sec

B: 7.84

R4



CT: 120 sec

B: 10.3

R6



CT: 240 sec

B: 9.69

R5



CT: 120 sec

B: 11.2

R53



CT: 240 sec

B: 9.79

R54



CT: 480 sec

B: 9.32

R55



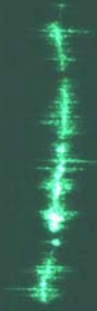
Sample 044 – PH30



CT: 60 sec

B:

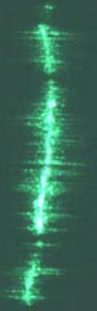
R19



CT: 120 sec

B:

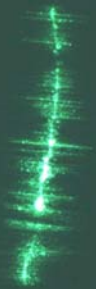
R18



CT: 240 sec

B:

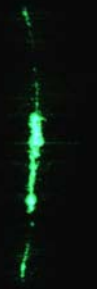
R20



CT: 120 sec

B:

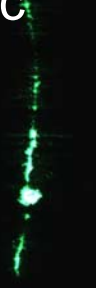
R56



CT: 240 sec

B:

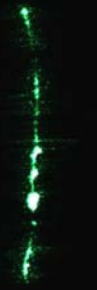
R57



CT: 480 sec

B:

R58



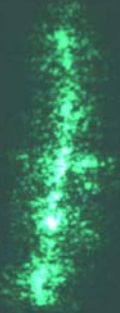
Sample 044 – PL10



CT: 60 sec

B:

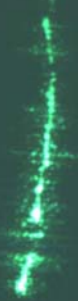
R37



CT: 120 sec

B:

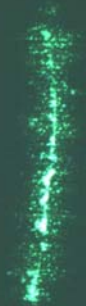
R36



CT: 240 sec

B:

R38



CT: 120 sec

B:

R59



CT: 240 sec

B:

R60



CT: 480 sec

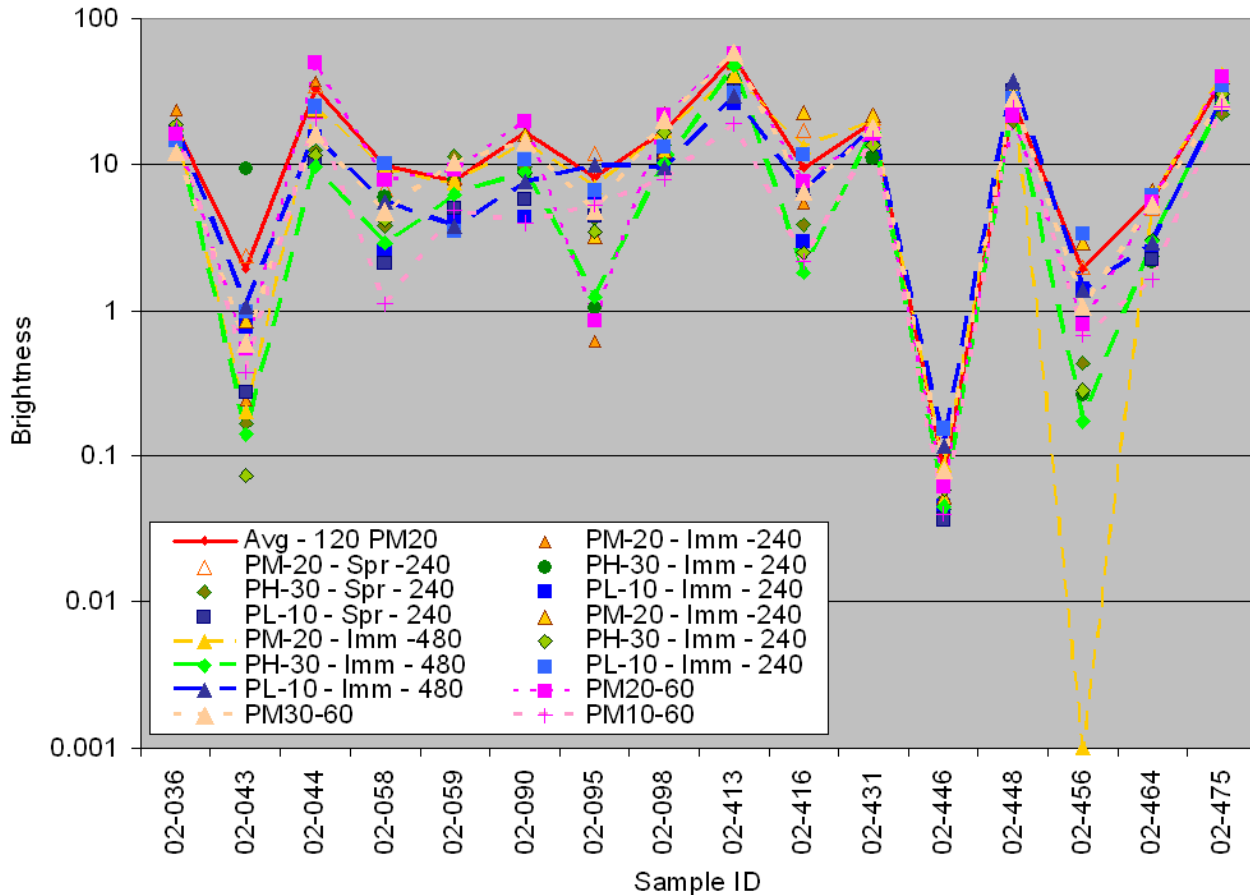
B:

R61





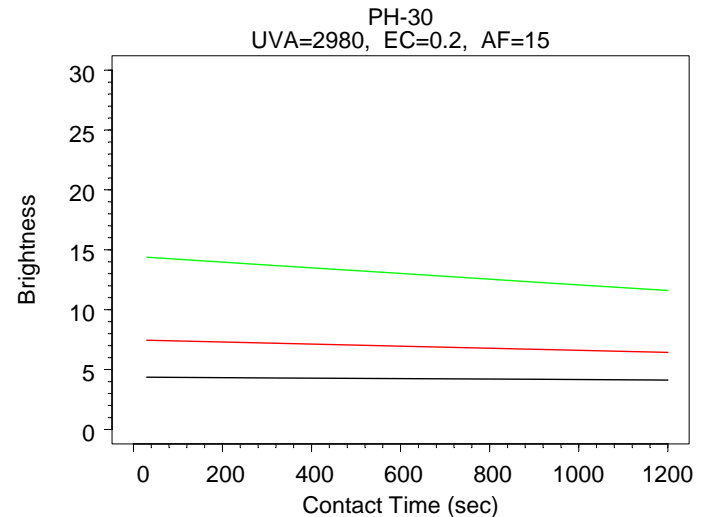
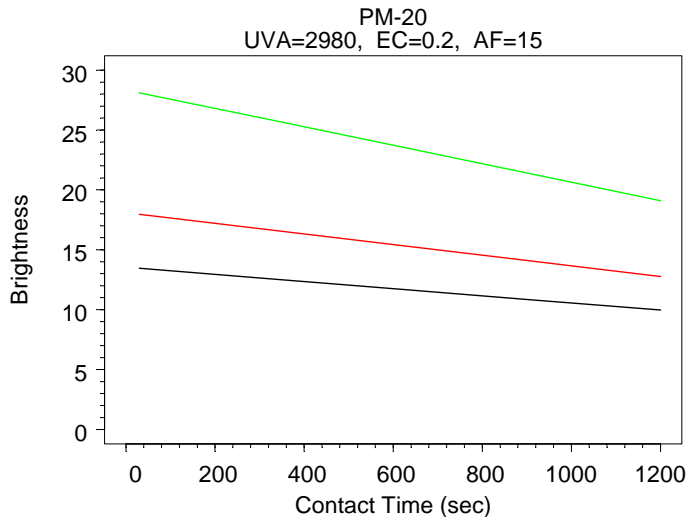
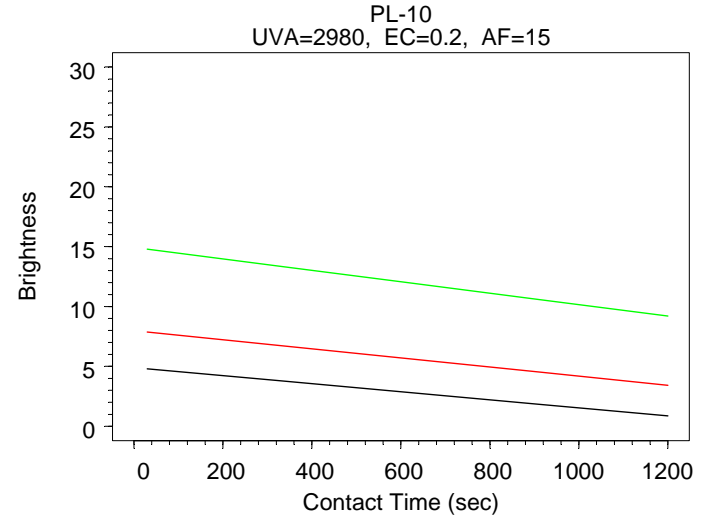
Emulsification Study - Contact Time Comparison



- Compare CT to average brightness of PM120
- CT60 ~ > CT120
- CT240 ~ < CT120
- CT480 < CT120

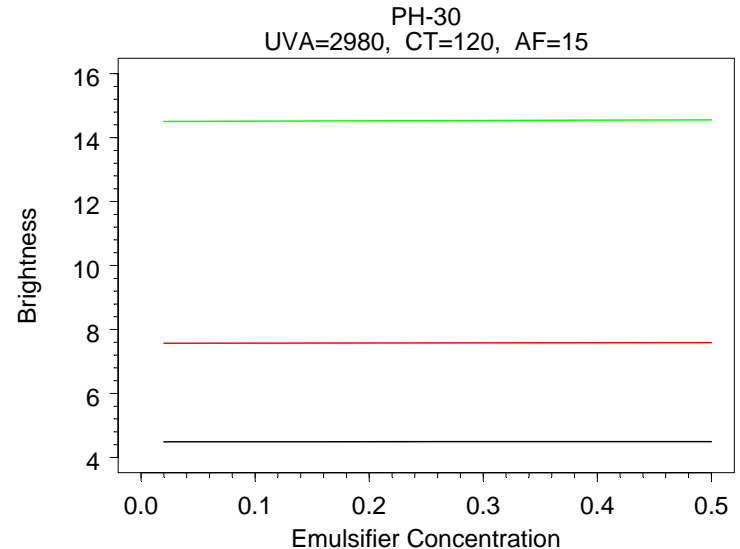
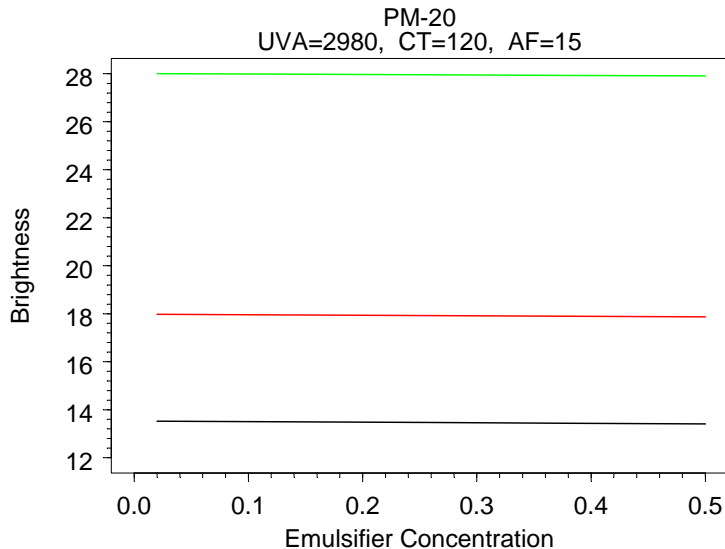
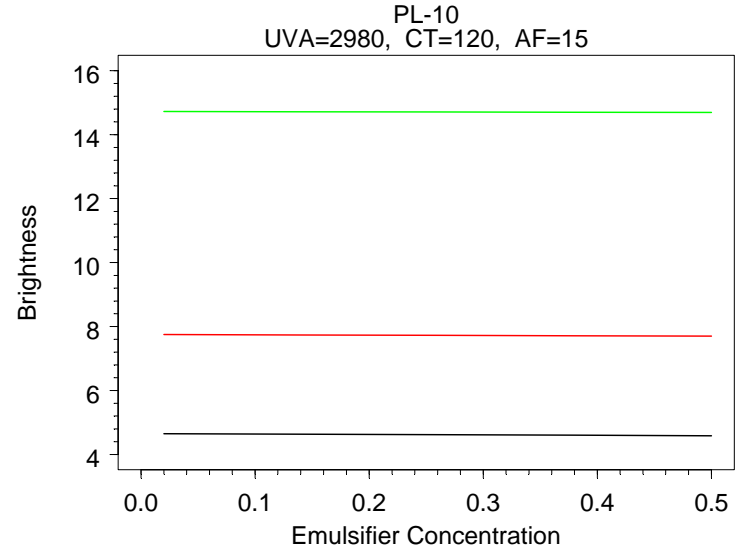


- Regression model used to predict effect of emulsifier contact time on brightness as a function of original brightness
- Brightness decreases with increasing contact time (note predictions beyond 480 minutes are extrapolations of the data)





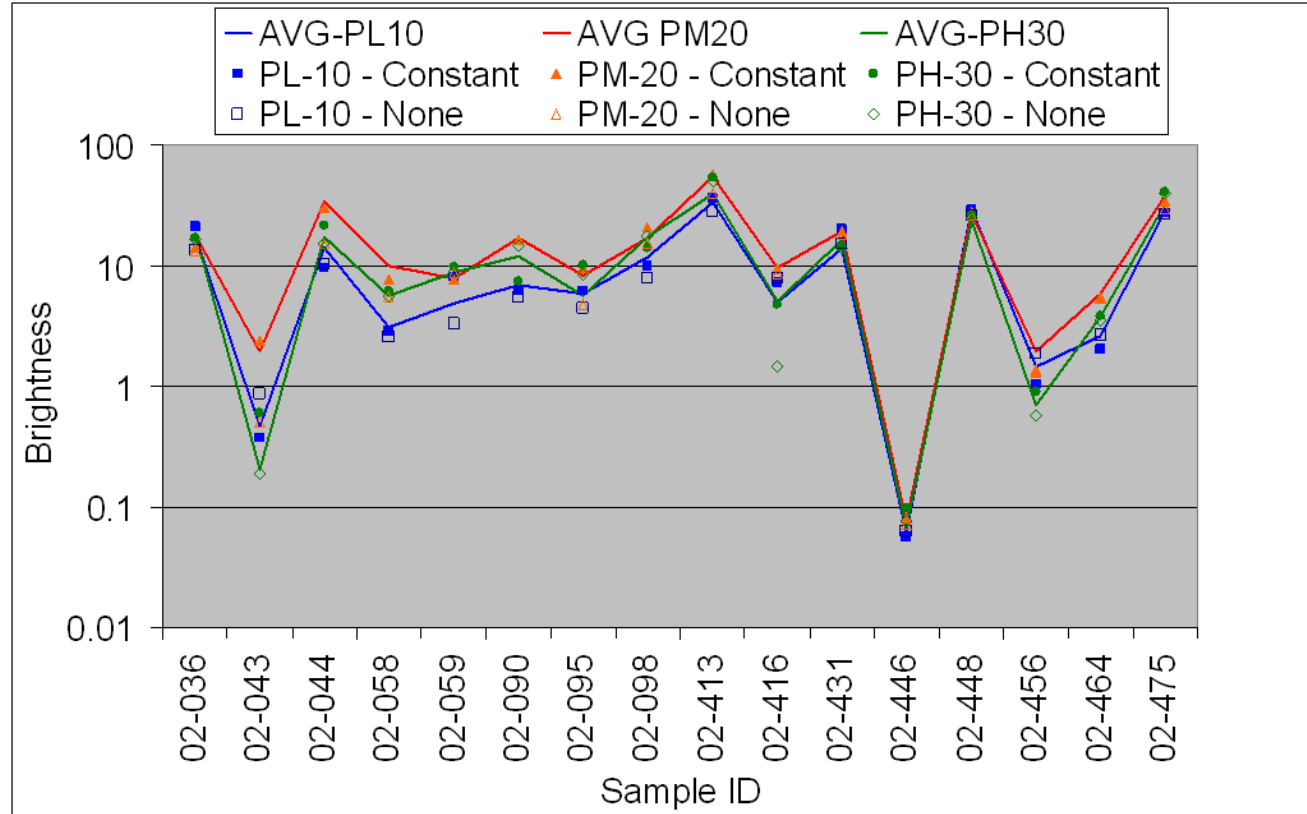
- Regression model used to predict effect of emulsifier concentration on brightness as a function of original brightness
- No significant changes in brightness observed at the concentrations measured



Agitation Frequency

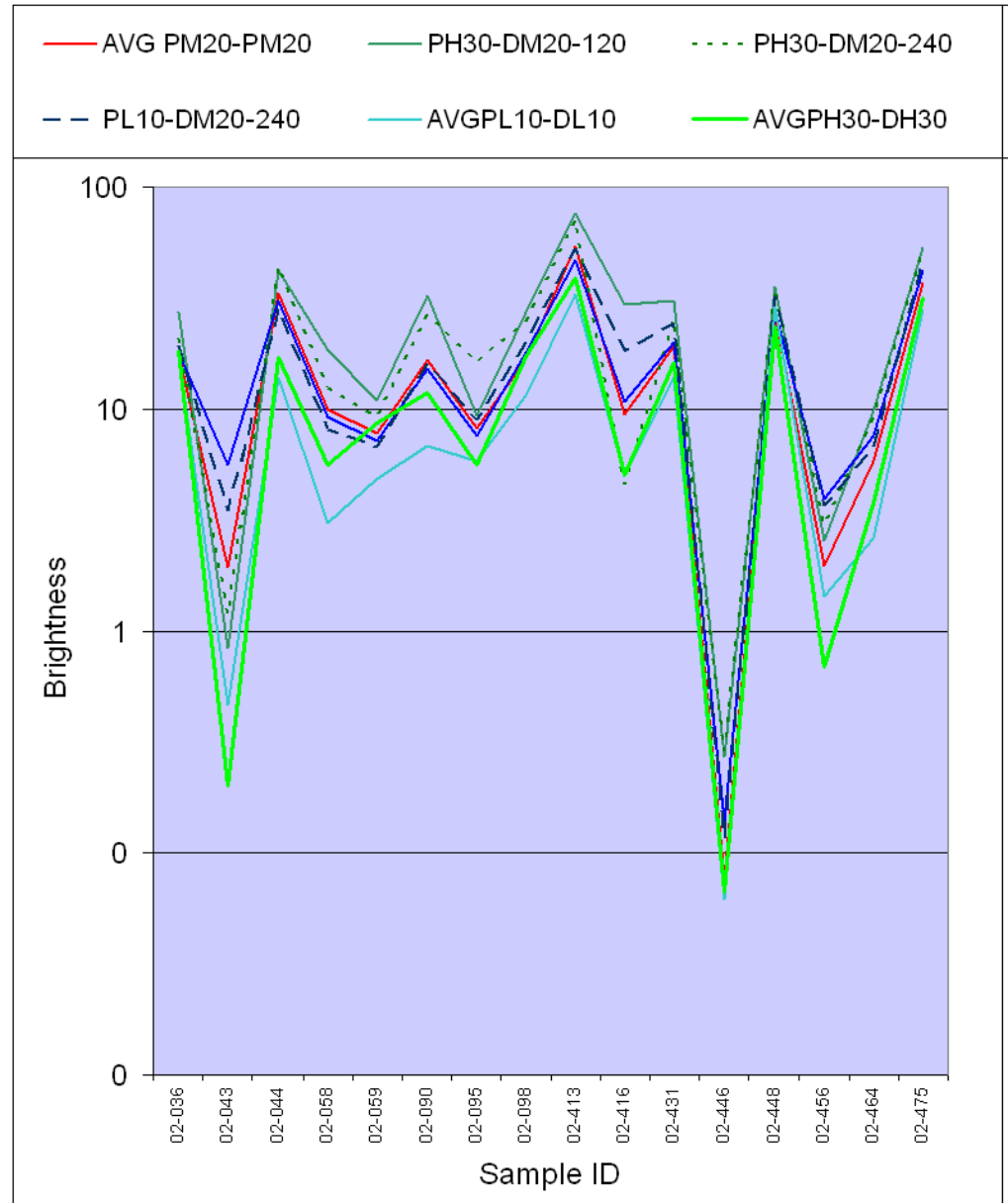


- Reduction in brightness occurs when no agitation is used for all three penetrants but the effect is only statistically significant for PM20
- Constant agitation essentially same as 15 sec agitation
- Conclusions based on one run and if viewed as important may warrant additional runs to verify trend





- Use of DM20 with PL10 and PH30 lead to improved brightness





- Emulsifier concentration has minimal impact on brightness when maintained at reasonable levels (+/- 5% of recommended concentration)
- Contact time has largest impact on brightness with brightness decreasing with increasing contact time
- Brightness decreases slightly when no agitation occurs but only statistically significant for one of the three developers (PM20)
- Use of DM20 with PL10 and PH30 lead to brightness improvements