Test Monitoring Center
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MEMORANDUM: 04-054

DATE: June 14, 2004

TO: Joe Franklin, Chairman, CBT Surveillance Panel

FROM: Jeff Clark

SUBJECT: HTCBT Solvent Matrix Results

BACKGROUND

At the request of the CBT Surveillance Panel, the TMC designed a matrix to study the effects of changing the HTCBT (D 6594) coupon cleaning solvent from tetrahydrofuran (THF) to acetone. This change is considered desirable by the panel due to toxicity and environmental concerns associated with THF. If the results of the study suggest that the cleaning solvent can indeed be changed to acetone, then the panel intends to pursue a similar solvent change for the CBT test (D 5898).

MATRIX DESIGN

The matrix design involves five oils, three labs, and the two solvents. Each oil was run four times at each lab, twice with each solvent. This yields a total of sixty tests which is sufficient to examine all the necessary factors in the design (lab, oil, solvent; and all permutations of two and three factors). The oils chosen for the matrix were TMC reference oils 42 and 1005, and three candidate oils that were designated as NO3, NO4, and NO5. The table below summarizes the matrix design.

	Lab	Solvent	Oil	Runs	Total Tests
	A B G	Acetone THF	42 1005 NO3 NO4 NO5	2	
Total	**3**	**2**	**5**	**2**	**60**

TEST RESULTS

The test data are shown in Attachment 1. These results are also available in spreadsheet format from the TMC website.

ANALYSIS OF RESULTS

Statistical analysis of the results on copper corrosion indicated that differences in oil proved to be significant ($p < 0.05$) and lab differences proved to be marginally significant ($0.05 < p < 0.10$). The differences between solvents were not significant ($p = 0.49$).

Analysis of the lead corrosion results shows a significant ($p = 0.0013$) interaction between oil, lab, and solvent. This makes the evaluation difficult because the presence of the three-way interaction indicates that lead corrosion does not respond consistently to any individual factor or combination of two factors. Spot examination of overall lab averages, which include results on both solvents, suggests that each lab's data set needs to be evaluated separately; refer to Attachment 2. This second level analysis yields different significant factors among the labs; again refer to Attachment 2. Labs A and G show a solvent-oil interaction to be significant for lead (Lab A also shows this for copper). Lab B shows no significant factors other than oil. To better understand these differences, averages and least squares means plots for lead (by lab) are shown in Attachment 3. From these plots it appears that Lab A shows a difference for lead on oil NO4 as does Lab B, though at much different severity levels. Lab G shows a difference for oils 42 and NO3. For each of the cases where a difference is visually noted, the acetone results are mild of the THF results.

CONCLUSIONS

The results of this experiment suggest that for copper corrosion, a change to using acetone as the cleaning solvent would not result in any significant differences. Unfortunately, the results for lead corrosion for this solvent change are not so easily judged due to the three-way interaction previously mentioned. In general though, the difference between labs in lead corrosion performance appears to be so great as to obscure the effect, if any, that a solvent switch would have on lead corrosion. This neither supports nor refutes making a change in cleaning solvent. It simply means that the surveillance panel needs to consider the varying effects at each laboratory (Attachment 3). If these differences do not cause discomfort, then the change in cleaning solvent can be made; otherwise caution would suggest that the solvent not be changed or that another possible solvent be examined. However, the strongest suggestion of the data from this experiment is that real lab differences exist on lead corrosion, and the surveillance panel should investigate these differences whether or not they choose to change cleaning solvents.

JAC/jac

Attachment

c: ftp://ftp.astmtmc.cmu.edu/docs/bench/htcbt/memos/mem04-054.pdf
CBT Surveillance panel
JLZ
FMF

Distribution: Email

ATTACHMENT 1
SOLVENT MATRIX TEST RESULTS

CMIR	LAB	BATH	RUN	OIL	SOLVENT	COPPER	LEAD
49807	B	3	563	42	THF	28.0	87.0
49808	B	3	563	42	ACETONE	28.0	90.0
52071	B	3	563	1005	THF	11.0	22.0
52072	B	3	563	1005	ACETONE	10.0	22.0
52217	B	3	563	NO3	THF	6.0	48.0
52218	B	3	563	NO3	ACETONE	7.0	52.0
52221	B	3	563	NO4	THF	3.0	77.0
52222	B	3	563	NO4	ACETONE	4.0	58.0
52225	B	3	563	NO5	THF	131.0	57.0
52226	B	3	563	NO5	ACETONE	114.0	59.0
49809	B	2	564	42	THF	91.0	88.0
49810	B	2	564	42	ACETONE	81.0	87.0
52074	B	2	564	1005	THF	11.0	25.0
52075	B	2	564	1005	ACETONE	11.0	24.0
52219	B	2	564	NO3	THF	7.0	52.0
52220	B	2	564	NO3	ACETONE	7.0	59.0
52223	B	2	564	NO4	THF	4.0	94.0
52224	B	2	564	NO4	ACETONE	4.0	84.0
52227	B	2	564	NO5	THF	132.0	55.0
52228	B	2	564	NO5	ACETONE	119.0	51.0
49371	G	1	353	42	THF	47.6	111.6
49372	G	1	353	42	ACETONE	40.0	92.2
49373	G	1	353	42	THF	37.8	123.9
49374	G	1	353	42	ACETONE	33.0	85.0
52043	G	1	353	1005	THF	9.6	26.7
52044	G	1	353	1005	ACETONE	10.0	24.7
52045	G	1	353	1005	THF	10.2	33.0
52046	G	1	353	1005	ACETONE	10.2	29.5
52229	G	1	353	NO3	THF	5.4	63.4
52230	G	1	353	NO3	ACETONE	6.2	54.2
52231	G	1	353	NO3	THF	6.2	72.2
52232	G	1	353	NO3	ACETONE	5.4	53.8
52233	G	1	353	NO4	THF	3.6	56.8
52234	G	1	353	NO4	ACETONE	3.7	61.4
52235	G	1	353	NO4	THF	3.8	70.6
52236	G	1	353	NO4	ACETONE	3.9	64.0
52237	G	1	353	NO5	THF	96.0	80.6
52238	G	1	353	NO5	ACETONE	73.4	82.5
52239	G	1	353	NO5	THF	115.4	84.6
52240	G	1	353	NO5	ACETONE	121.2	79.4
49811	A	7	35	42	THF	32.0	149.0
49812	A	7	35	42	THF	32.0	140.0
49813	A	7	35	42	ACETONE	34.0	132.0
49814	A	7	35	42	ACETONE	34.0	140.0
52056	A	7	35	1005	THF	11.0	42.0
52057	A	7	35	1005	THF	12.0	44.0
52058	A	7	35	1005	ACETONE	12.0	46.0
52059	A	7	35	1005	ACETONE	11.0	44.0
52241	A	7	35	NO3	THF	8.0	136.0
52242	A	7	35	NO3	THF	7.0	136.0
52243	A	7	35	NO3	ACETONE	8.0	126.0
52244	A	7	35	NO3	ACETONE	9.0	142.0
52245	A	6	12	NO4	THF	5.0	233.0
52246	A	6	12	NO4	THF	5.0	238.0
52247	A	6	12	NO4	ACETONE	5.0	197.0
52248	A	6	12	NO4	ACETONE	5.0	186.0
52249	A	6	12	NO5	THF	106.0	78.0
52250	A	6	12	NO5	THF	102.0	91.0
52251	A	6	12	NO5	ACETONE	101.0	86.0
52252	A	6	12	NO5	ACETONE	100.0	84.0

ATTACHMENT 2

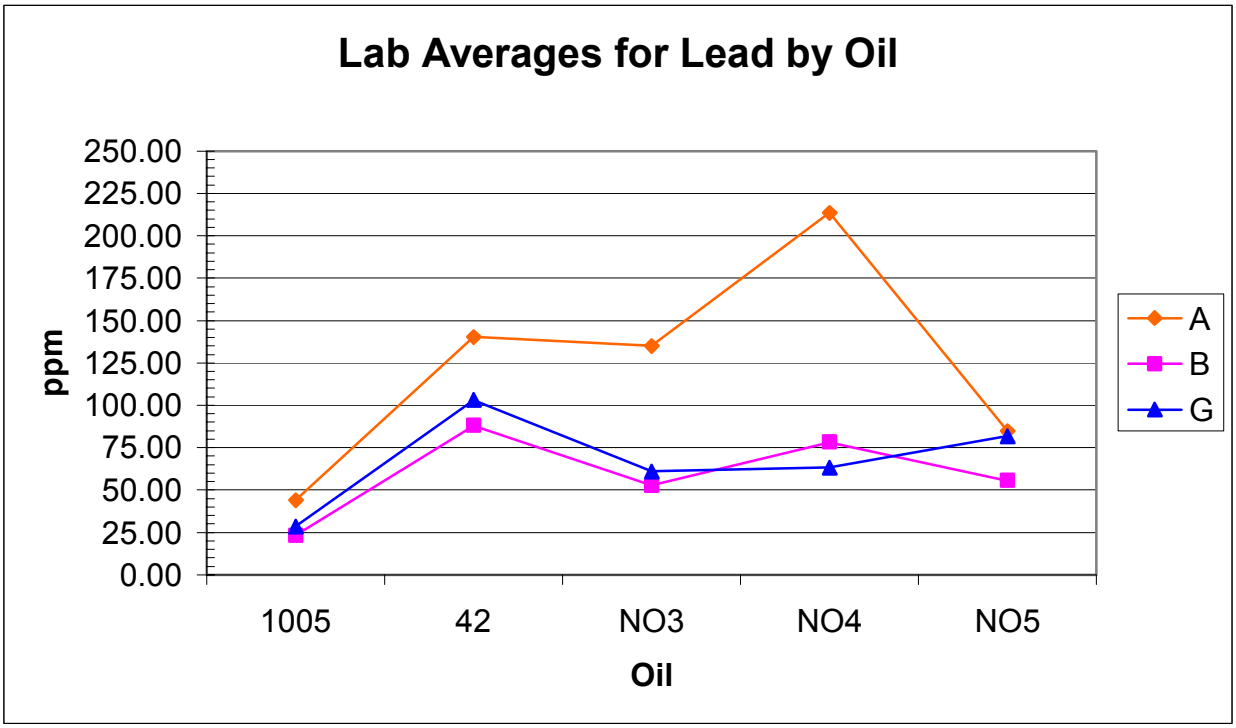
Solvent Matrix – Overall Lab Averages

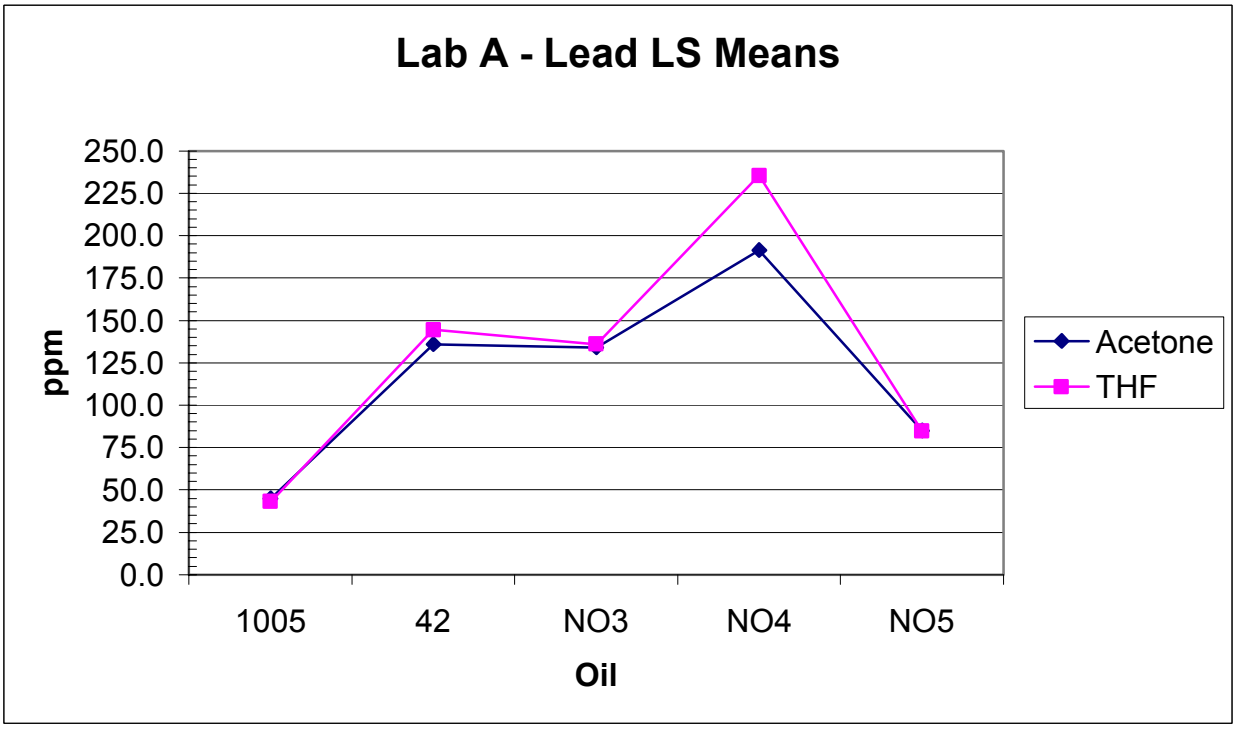
Lab	Copper (ln units)	Lead (ppm)
A	2.850	123.5
B	2.861	59.6
G	2.730	67.5

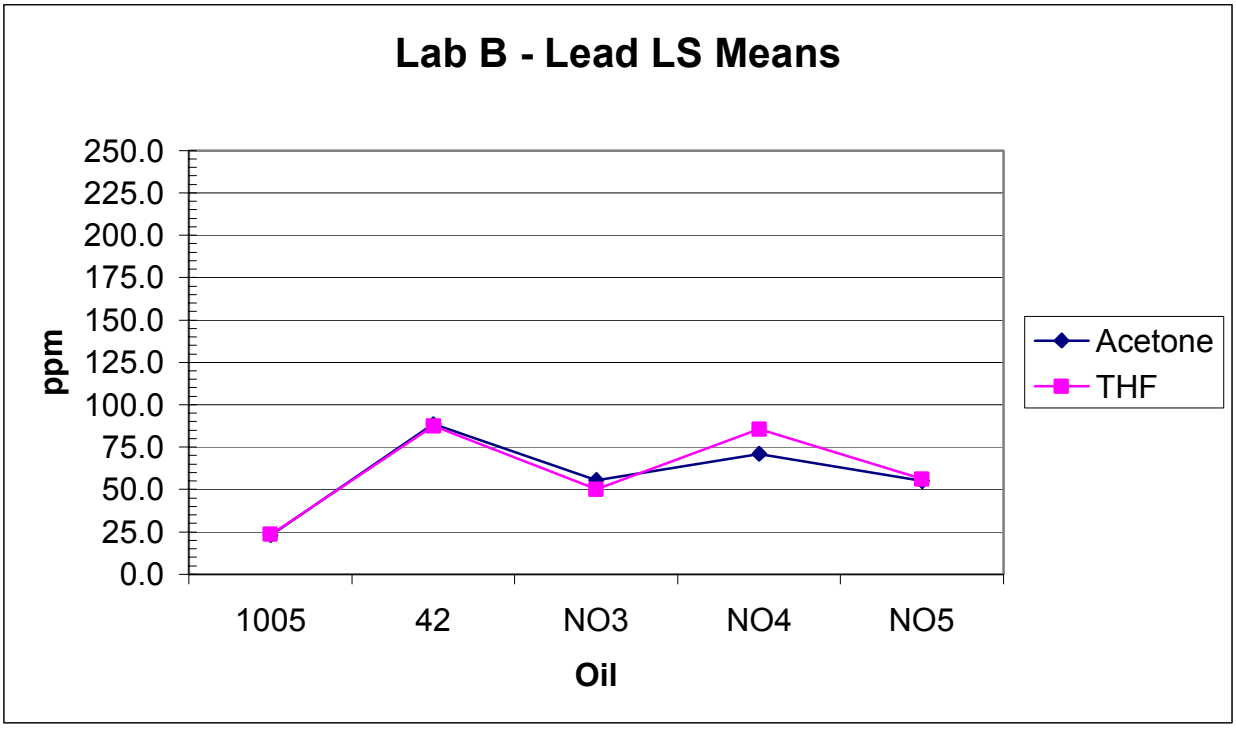
Solvent Matrix – Significant Factors by Laboratory

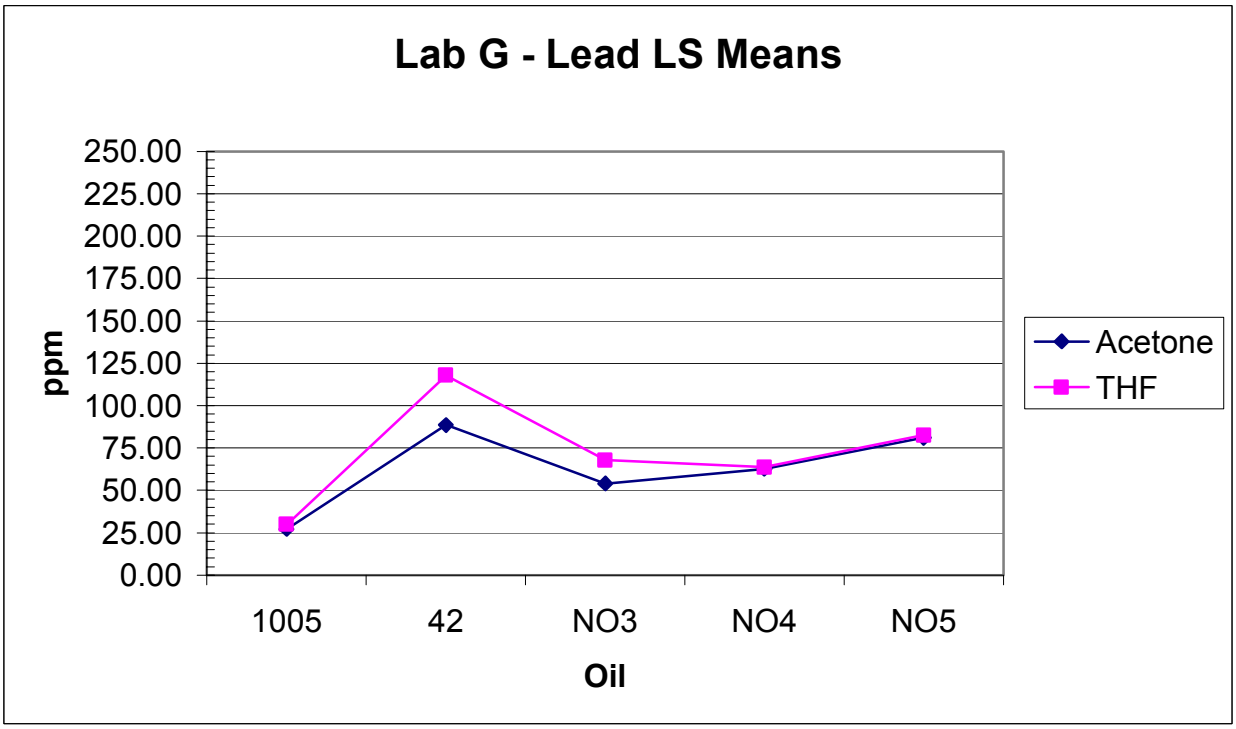
Lab	Parameter	Significant Factors
A	Copper	Oil
	Lead	Solvent-Oil interaction
B	Copper	Oil
	Lead	Oil
G	Copper	Solvent-Oil interaction
	Lead	Solvent-Oil interaction

ATTACHMENT 3






Lab B - Lead LS Means
250.0
225.0
200.0
175.0
150.0
125.0
100.0
75.0
50.0
25.0
0.0
ppm
1005
42
NO3
NO4
NO5
Oil
Acetone
THF


Lab G - Lead LS Means
250.00
225.00
200.00
175.00
150.00
125.00
100.00
75.00
50.00
25.00
0.00
ppm
1005
42
NO3
NO4
NO5
Oil
Acetone
THF