APPENDIX 7.3

Survey of Mechanical Filtration From 50% Sodium Hydroxide Solutions

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Mechanical Separation Optimization Team

Butch Carman	Coordinator
Harvey Cochran	Analytical Resource
Jim Heron	Process Resource
Dr. Leonard Scott	Technology Resource
Jorge Vilanova	Process Resource

Edited By Bob Brooker

Acknowledgement

We thank our fellow amalgam cell Chlor Alkali manufacturers who participated in making this survey study possible. Without their open and thoughtful disclosures, this effort could not have been successfully completed.

Mechanical Separation Optimization Team

Abstract

The objective of this study was to identify the optimum equipment organization and operating controls necessary to consistently achieve 30 PPB to 50 PPB mercury levels in 50% Sodium Hydroxide product from a amalgam cell Chlor Alkali plant filtration system.

Six amalgam cell Chlor Alkali plants, having seven cell rooms, were visited in order to determine the best operating practices. Individual plant equipment data, and analytical methods were gathered to provide a basis of comparison for the study. It was agreed, prior to the survey, that the data obtained be presented to the Chlorine Institute for public use as deemed appropriate by member companies.

Key features for the caustic filtration system were identified to consistently produce low mercury in caustic product and are as follows.

1. Caustic from the decomposer should be in the 104 °C to 106 °C range.
2. Ensure that a flux rate of 0.30 gallons per minute per square foot through the R. P. Adams filters is not exceeded.
3. Recycle filtered product back to the caustic receiver at a rate equal to or greater than the production rate.
4. Poro-carbon 200 filter elements should be used in the filters.
5. The poro-carbon elements should be “conditioned” prior to their first use. This conditioning should be done with a fine carbon pre-coating material. The particle size of the carbon should be 325 mesh or smaller.
6. Future element pre-coating using only carbon or at least using a carbon base coat is the preferred method.
7. The maximum pressure drop across the filter elements should be 15 pounds per square inch.
8. A constant filter outlet pressure should be maintained. This minimizes down stream pressure changes caused by storage tank changes or valve switching.

The above filtration system recommendations are based solely on a compilation of all sites visited.

I. Objective

The objective of this study was to identify the optimum equipment arrangement and operating controls necessary to consistently achieve 30 PPB to 50 PPB mercury levels in 50% Sodium Hydroxide product from a amalgam cell filtration system. The study approach was to visit Chlor Alkali industry plants that produce 50% Sodium Hydroxide using amalgam cell technology to gather the necessary information. The plants selected have published mercury levels in caustic with the Chlorine Institute, which meet the objective criteria. The conclusions and recommendations will be published through the Chlorine Institute.

II. Summary

A caustic filtration system is detailed that will produce mercury in caustic in the range of 30 PPB to 40 PPB. At the time of this study, the range of 30 PPB to 40 PPB mercury in caustic was believed to be the best sustainable quality in the Chlor Alkali industry using only mechanical separation equipment. The caustic filtration system recommended by this report is a composite system of the plants surveyed.

The final equipment sizing and optimum equipment operating procedures will require an investment in a pilot system. The entire study team recommended the purchase of this verification system.

The composite filtration system involves, first, use of a recycle stream from the discharge of the existing filters back to the caustic surge tank or receiver. This stream is to equal the normal caustic production flow forward to storage. The second part of the recommended system is to achieve a filter area flux rate of 0.25 gallons per minute per square foot. The third process feature is the use of a finely ground carbon pre-coat for the filter tubes. The fourth process optimization reduces the allowable pressure drop to a maximum of 15 PSI. The fifth process enhancement is incorporation of backpressure control on the filter discharge to eliminate the negative impact of switching caustic storage tanks. The sixth process adaptation is a procedure by which fine carbon is body fed to the filter upon elevated or mercury break-through to prolong the filter cycle.

III. Background

One of Olin Chlor Alkali Divisions' goals is to reduce the release of mercury from all sources. It is also a division goal to produce products in a fashion that minimizes mercury discharges from our plants in the air, water, solids, and products so that there is no harm to human health or the environment as a result of emissions or exposure. Specifically, the reduction of mercury in caustic soda has focused on three areas. The first area focuses on the decomposer and its operation. The second area focuses on the mechanical separation of elemental mercury from the 50% caustic soda solution. The third area is focusing on achieving the next generation of filtration / separation of mercury from the filtered and cooled product.

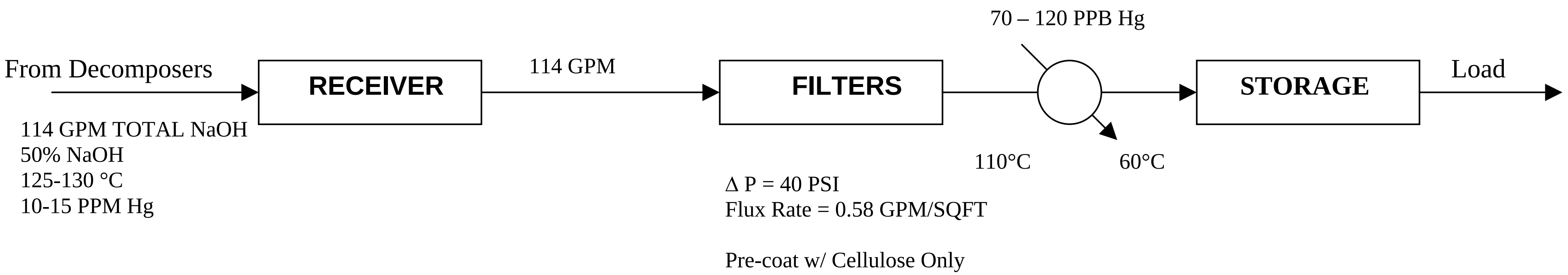
This report deals only with the second area of mercury removal efforts, namely, mechanical separation. The goal is to achieve the minimum mercury in caustic levels possible by visiting and understanding the techniques used by the industry leaders in caustic filtration. The published literature indicates that 30 to 50 PPB levels are consistently achievable. Levels of 10 PPB have been demonstrated but have not been consistently repeated.

A team of five people was assembled to conduct a study of the current techniques within the Chlor Alkali Industry. The Team's approach was to visit each plant to gather equipment, operating, and analytical data. The data would then be used to formulate the optimum equipment sizing & arrangement, operating procedures, pre-coat material, and analytical procedures. These visits occurred between April 5th and April 17th of 1998.

IV. DATA

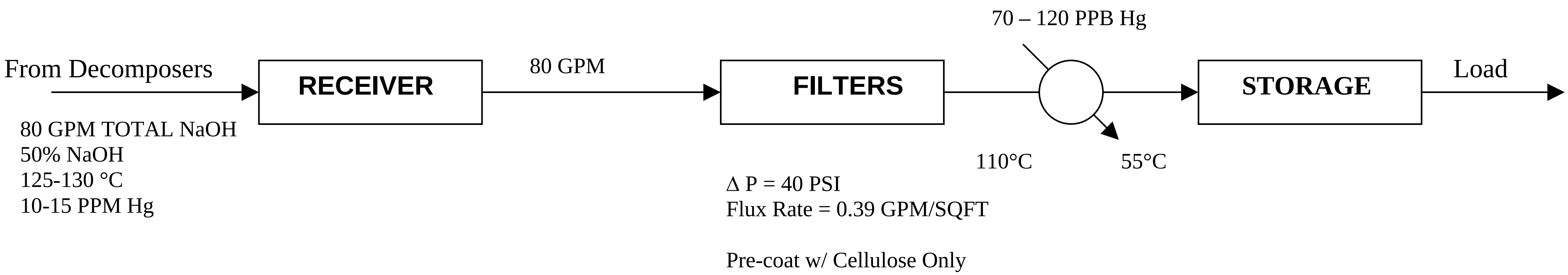
Data collected from five manufacturers forms the basis for the conclusions and recommendations contained within this report. The contained tables compile the data collected. Also included are brief schematic representations of the individual caustic filtration systems studied.

Plant #1

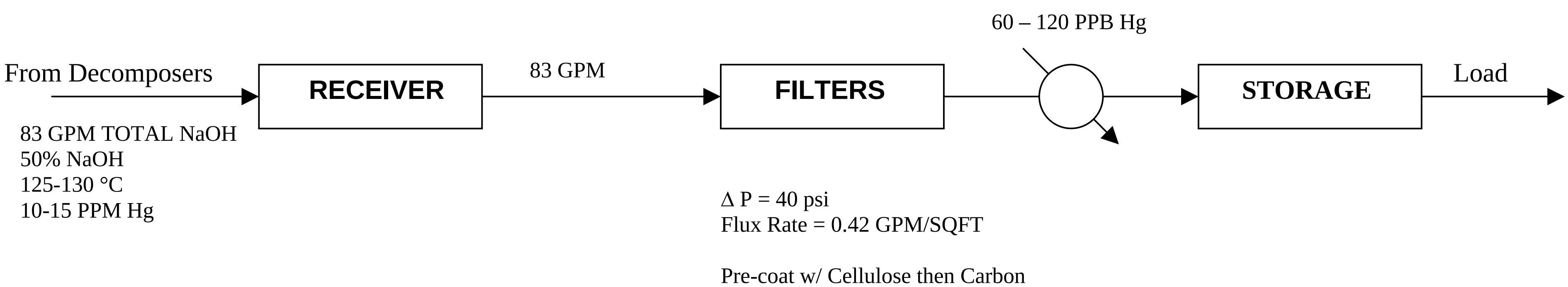


70 – 120 PPB Hg
From Decomposers
RECEIVER
114 GPM
FILTERS
STORAGE
Load
114 GPM TOTAL NaOH
50% NaOH
125-130 °C
10-15 PPM Hg
110°C
60°C
Δ P = 40 PSI
Flux Rate = 0.58 GPM/SQFT
Pre-coat w/ Cellulose Only

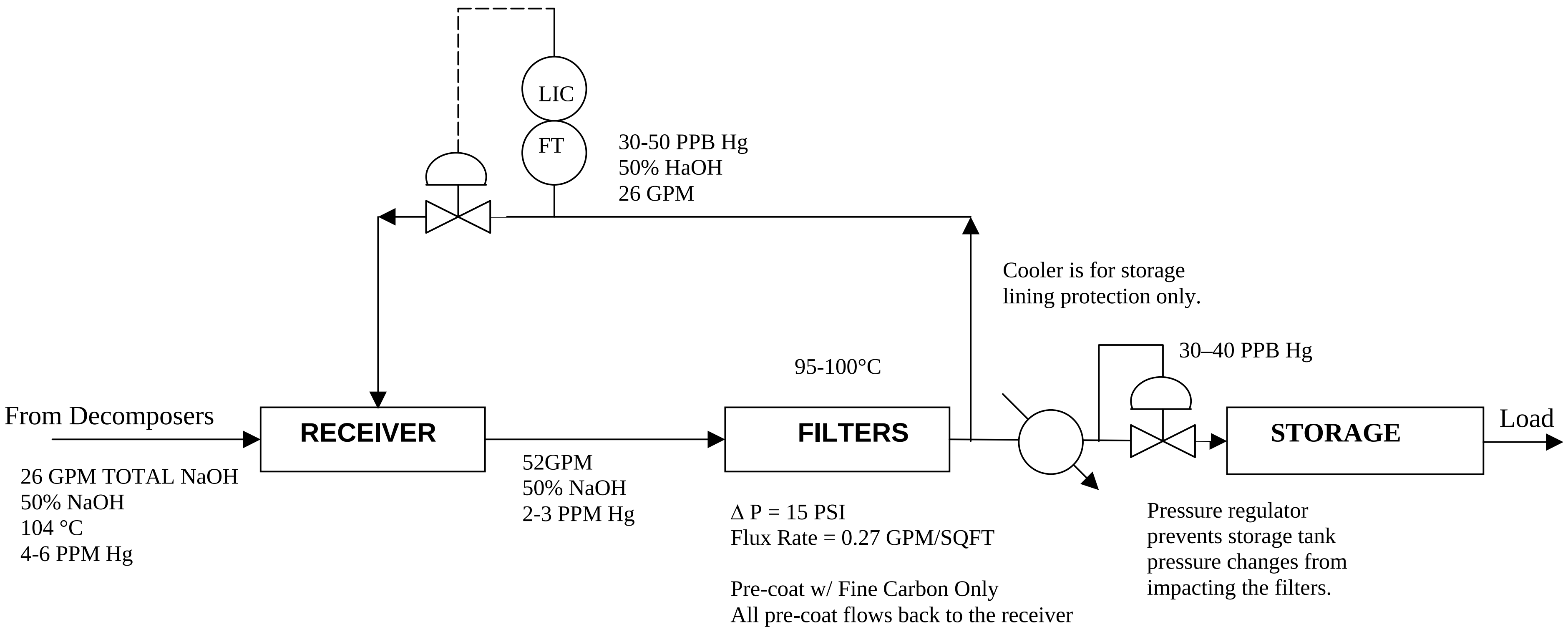
Plant #2


70 – 120 PPB Hg
From Decomposers
RECEIVER
80 GPM
FILTERS
STORAGE
Load
80 GPM TOTAL NaOH
50% NaOH
125-130 °C
10-15 PPM Hg
110°C
55°C
Δ P = 40 PSI
Flux Rate = 0.39 GPM/SQFT
Pre-coat w/ Cellulose Only

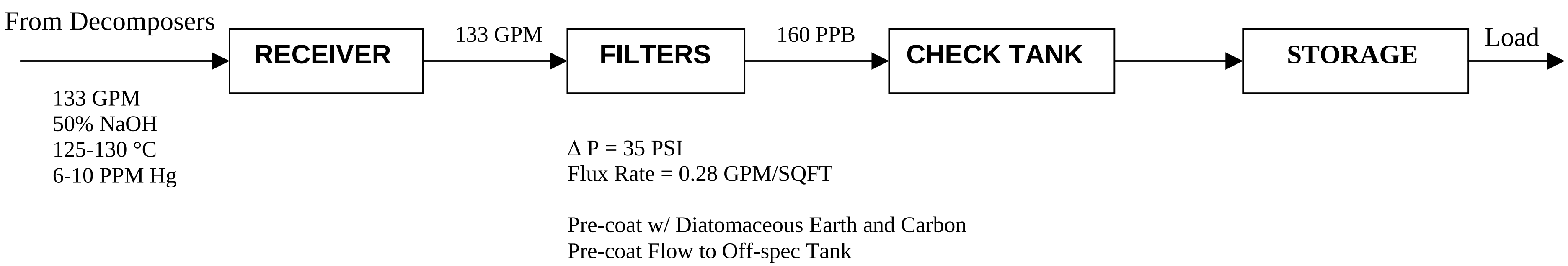
Plant #3


60 – 120 PPB Hg
From Decomposers
RECEIVER
83 GPM
FILTERS
STORAGE
Load
83 GPM TOTAL NaOH
50% NaOH
125-130 °C
10-15 PPM Hg
Δ P = 40 psi
Flux Rate = 0.42 GPM/SQFT
Pre-coat w/ Cellulose then Carbon

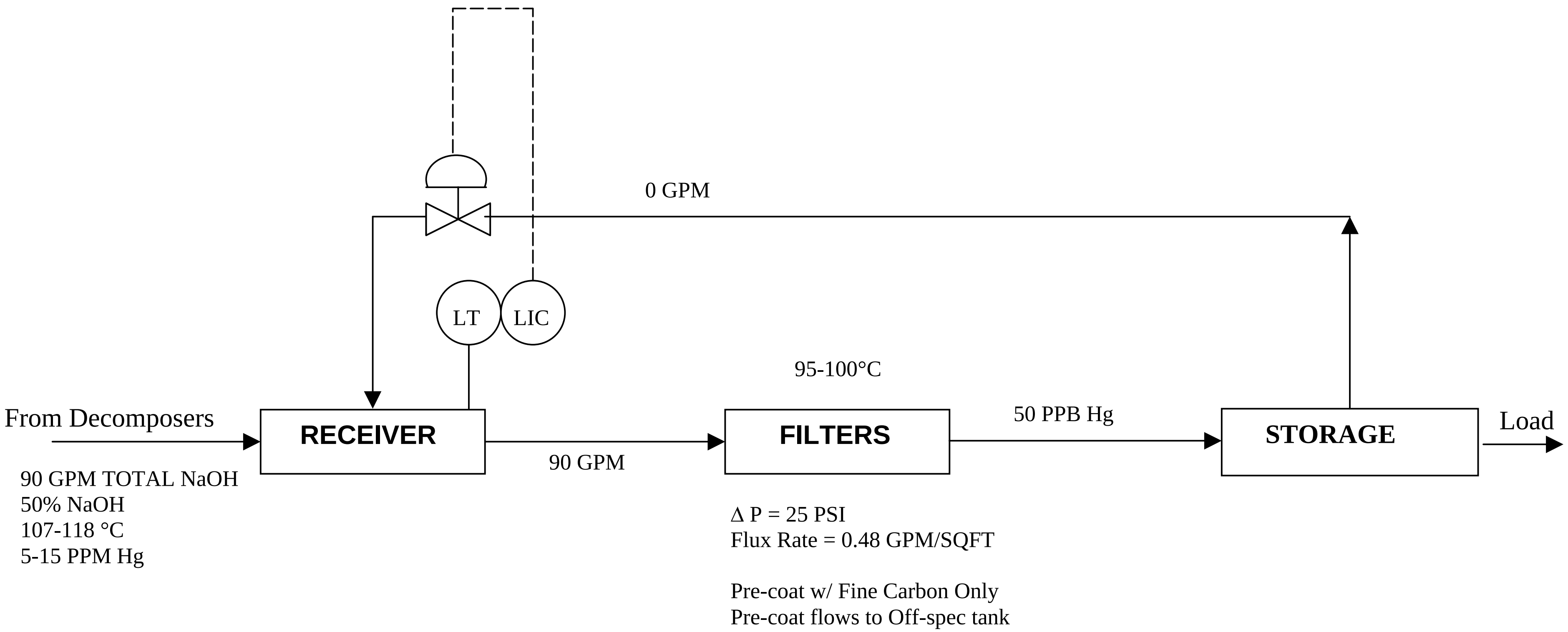
Plant #4


LIC
FT
30-50 PPB Hg
50% HaOH
26 GPM
Cooler is for storage
lining protection only.
30–40 PPB Hg
95-100°C
From Decomposers
RECEIVER
FILTERS
STORAGE
Load
26 GPM TOTAL NaOH
50% NaOH
104 °C
4-6 PPM Hg
52GPM
50% NaOH
2-3 PPM Hg
Δ P = 15 PSI
Flux Rate = 0.27 GPM/SQFT
Pre-coat w/ Fine Carbon Only
All pre-coat flows back to the receiver
Pressure regulator
prevents storage tank
pressure changes from
impacting the filters.

Plant #5

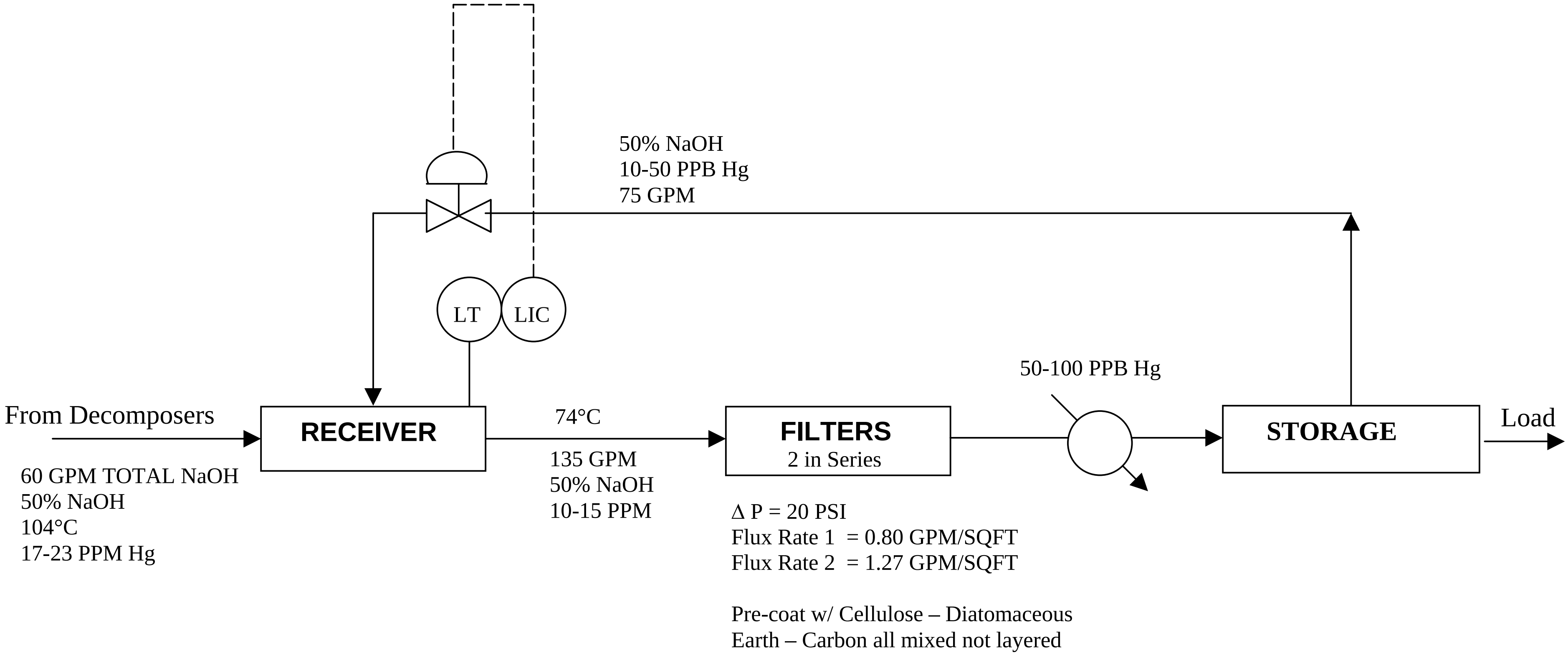

From Decomposers
133 GPM
50% NaOH
125-130 °C
6-10 PPM Hg
RECEIVER
133 GPM
FILTERS
160 PPB
CHECK TANK
STORAGE
Load
Δ P = 35 PSI
Flux Rate = 0.28 GPM/SQFT
Pre-coat w/ Diatomaceous Earth and Carbon
Pre-coat Flow to Off-spec Tank

Plant #6



0 GPM
LT
LIC
95-100°C
From Decomposers
RECEIVER
FILTERS
50 PPB Hg
STORAGE
Load
90 GPM
90 GPM TOTAL NaOH
50% NaOH
107-118 °C
5-15 PPM Hg
Δ P = 25 PSI
Flux Rate = 0.48 GPM/SQFT
Pre-coat w/ Fine Carbon Only
Pre-coat flows to Off-spec tank

Plant #7


50% NaOH
10-50 PPB Hg
75 GPM
LT
LIC
50-100 PPB Hg
From Decomposers
RECEIVER
74°C
FILTERS
2 in Series
STORAGE
Load
60 GPM TOTAL NaOH
50% NaOH
104°C
17-23 PPM Hg
135 GPM
50% NaOH
10-15 PPM
Δ P = 20 PSI
Flux Rate 1 = 0.80 GPM/SQFT
Flux Rate 2 = 1.27 GPM/SQFT
Pre-coat w/ Cellulose – Diatomaceous
Earth – Carbon all mixed not layered

PROCESS INFORMATION	Plant #1	Plant #2	Plant #3	Plant #4	Plant #5	Plant #6	Plant #7
GENERAL							
Chlorine Capacity - TPD	455	305	330	99	540	366	250
Cautic Capacity - TPD	513	344	372	113	610	413	275
Cell Type	E-812	E-510	E-510	Denora	UHDE	Denora	Denora
Cell Number	48	58	60	24	52	36	32
				11 Na			
DECOMPOSER							
Decomposer NaOH outlet temp.	130 C	130 C	130 C	102-104 C	125-130 C	107-118 C	104 C
Decomposer NaOH flow	114	76	83	26	133	90	60
Outlet NaOH seal leg (Y/N)	No	No	No	Yes	No	No	No
Hydrogen cooling (direct/indirect)	Direct	Direct	Direct	Indirect	Indirect	Indirect	Indirect
Hydrogen condensate temp. (C)	60-80	60-80	60-80	13-27	29	30-60	N/A
Deionized Water Cooling (Y/N)	No	No	No	Yes	No	No	No
Deionized Water Temp. (C)	60-80	60-80	60-80	49	19	N/A	N/A
Hg in NaOH at Decomposer PPM	10 to 15	10 to 15	10 to 15	4 to 6	6 to 10	5 to 15	17 to 23
Hg in NaOH into filters PPM	10 to 15	10 to 15	10 to 15	2 to 3	6 to 10	5 to 15	10 to 15
Hg in NaOH out of filters PPB	**70-120**	**70-120**	**60-120**	**30-40**	**160**	**50**	**50-100**
CAUSTIC RECEIVER							
Caustic Receiver Volume - Gal.	2280	2280	1385	1500	4000	4200	2000
Residence Time (min.)	8	9	9	19	15	23	8
Recycle to Receiver (Y/N)	No	No	No	Yes	No	Yes	Yes
Recycle from: (filters / storage)	---	---	---	Filters	---	Storage	Storage
Recycle rate (gpm)	0	0	0	25	0	0	75
Mercury level in recycle - PPM	---	---	---	0.03	---	---	0.03
Operated on level control (Y/N)	Yes	Yes	Yes	Yes	Yes	Yes	Yes
yes - (pump discharge or recycle)	pump flow	pump flow	pump flow	pump flow	pump flow	pump flow	recycle flow
CAUSTIC TRANSFER PUMP(S)							
Capacity (GPM)	120	80	100	50	133	90	130
Manufacturer	Goulds	Goulds	Goulds	Durco	Durco	Durco	Durco
Type	Mag-drive	Centrif.	Centrif.	Centrif.	Centrif	Centrif	Centrif
Pump Discharge Pressure (PSIG)	85	85	80	72	95	N/A	42
Filter Inlet Pressure (PSIG)	50-80	50-80	50-75	60	78-95	30-60	22

PROCESS INFORMATION	Plant #1	Plant #2	Plant #3	Plant #4	Plant #5	Plant #6	Plant #7

CAUSTIC FILTERS

Manufacturer	Adams IWF	Adams IWF	Adams IWF	Adams IWF	Adams IWF	Adams IWF	Adams IWF
Filter Element	C200	C200	C200	C200	C200	C200	C200
Primary Filter Area (ft2)	197	197	197	94	238	189	169
Number of Primary Filters	1	1	1	2	2	1	1
Primary Filter Arangement	Single	Single	Single	Parallel	Parallel	Single	Single
Secondary Filter Area (ft2)	197	197	NA	NA	NA	189	53
Number of Secondary Filters	1	1	---	---	---	1	2
Secondary Filter Arangement	Single	Single	---	---	---	Single	Parallel
Flux rate on Primary filter- gpm/ft2	0.58	0.39	0.42	0.27	0.28	0.48	0.80
Flux rate-Secondary filter- gpm/ft2	0.58	0.39	NA	NA	NA	0.48	1.27

FILTER OPERATION

Precoat Material (1)	Solka-floc BW40	Solka-floc BW40	Solka-floc BW40	Darco Premium	E.P. Diat. FW-60	E.P. PB33	Celite
Precoat Material (2)	---	---	Norit D-10	---	Norit AZO	(Eagle Picher)	Nuchar SA
Precoat Material (3)	---	---	---	---	---	---	E.P. PB33
Loading (1) lb/ft2	0.254	0.254	0.127	0.005	0.084	0.132	0.296
Loading (2) lb/ft2	---	---	0.254	---	0.084	---	0.266
Loading (3) lb/ft2	---	---	---	---	---	---	0.237
Precoat Fluid	Water	Water	50% Hot NaOH	50% Hot NaOH	DI Water	DI Water	DI Water
Precoat Quality Assesment	*1	*1	*1	*2	*3	*1	*1
Filter Tube Pre-conditioning	No	No	No	.096 lbs/ft2	No	No	Yes
Scheduled Replacement	As needed	As needed	As needed	As needed	24months	18 months	48 months

*1 -- Filter discharge sent to off-spec tank until lab verification of acceptable Hg levels in filtered caustic.
*2 -- Filter discharge recycled to caustic receiver until lab verification of acceptable Hg levels in filtered caustic.
*3 -- Filter discharge sent to check tank until lab verification of acceptable Hg levels in filtered caustic.

BACKWASH CRITERIA

Hg concentration	>0.25 ppm	>0.25 ppm		Body feed	>0.5 ppm	>0.09 ppm	>0.30 ppm
Pressure drop across filters	40 PSIG	40 PSIG		15 PSIG	35 PSIG	25 PSIG	20 PSIG
Time on-line	5 days	7 days		4 weeks	3 weeks	3 weeks	5-10 days
Backwashes	2 min - 4 max	2 min - 4 max		1	3 pulse/2 wash	3	1
Bodyfeed	No	No	No	High Hg	No	No	No
Amount (lbs)	---	---	---	0.5	---	---	---

PROCESS INFORMATION	Plant #1	Plant #2	Plant #3	Plant #4	Plant #5	Plant #6	Plant #7
Misc.							
Off Spec NaOH	Separate	Separate	Separate	Blend/Product	Blend/Product	Blend/Product	Blend/Product
Off Spec Filter at Off Spec Tank	No	No	No	Yes	No	No	No
Check Tank	No	No	No	No	Yes	No	No
Inlet Cooler Temperature (C)	110	110		93	125	110	77
Outlet Cooler Temperature (C)	60	55		54	60	77	60

LABORATORY ANALYSIS

Analytical Instrumentation	LDC Milton Roy 920404 & P.S. Analytical	LDC Milton Roy 920404 & P.S. Analytical	LDC Milton Roy 920404 & PSA	Bacharach, Coleman; Model 50D & Leeman Labs PS200	Varian, M-6000A & Perkin-Elmer M 3100	Jerome Model 411	Methods for analysis were not available at time of visit.
Analytical Method	CVAA, & CVAF	CVAA, & CVAF	CVAA, & CVAF	CVAA & CVUV	CVAA & CVUV	Gold Film	
Digestion Sample Size (grams)	10	10	20	5	2	1.5	
Neutralization Acid	Conc. HNO3	Conc. HNO3	Conc. HNO3	1:4 H2SO4	Conc. H2SO4	Conc. HNO3	
Aliquot Sample Size (grams)	0.05	0.05	0.2	5	2	0.3	
Oxidation Solution	5% Acid KMnO4	5% Acid KMnO4	5% Acid KMnO4	5% Acid KMnO4	5% Acid KMnO4	5% Acid KMnO4	
Reduction Solution	SnCl2	SnCl2	SnCl2	SnCl2 or SnSO4	SnCl2 (10%)	SnCl2	
Calibration	Verification Once per Shift & Multi-Point Quarterly.	Verification Once per Shift & Multi-Point Quarterly.	Verification Once per Shift	Multi-Point Calibration Monthly & Verification Daily.	Verification Once per Shift & Multi-Point Monthly.	Multi-Point Calibration Monthly	
Operators Analysis Frequency	N/A	N/A	Hourly	Hourly	Four times/day	Every 4 Hours	
Laboratory Analysis Frequency	Every 4 Hours	Every 4 Hours	Every 8 Hours	Daily 24 hour Composite & 0800 confirmation	Daily 24 hour Composite ,0800 confirmation & Check Tank.	Daily 24 hour Composite ,0800 hrs. confirmation.	

CVAA = Cold Vapor Atomic Adsorption
CVAF = Cold Vapor Atomic Florescence
CVUV = Cold Vapor UV-Vis Spectroscopy

V. Team Conclusions

1. Cooler decomposers reduce the level of mercury to be removed from the caustic product. Caustic from the decomposer should be in the 104 °C to 106 °C range.
2. Ensure that a flux rate of 0.30 gallons per minute per square foot through the R. P. Adams filters is not exceeded.
3. A recycle from the filter discharge to the caustic receiver should be used and it should equal to or greater than the production flow rate coming to the receiver.
4. The filter elements should be R. P. Adams poro-carbon 200 or equal.
5. The poro-carbon elements should be "conditioned" prior to their first use. This conditioning consists of having fine carbon distributed on the elements until the desired filter quality is achieved.. The particle size of the carbon should be 325 mesh or smaller.
6. Filter element pre-coating using only carbon or at least using a carbon base coat is the preferred method.
7. The pressure drop across the elements should not be allowed to exceed 15 pounds per square inch.
8. A constant filter outlet pressure should be maintained. This minimizes down stream pressure changes caused by storage tank changes or valve switching.

Specific elements of a composite caustic filtration system are detailed that will aid in achieving mercury in caustic in the range of 30 PPB to 40 PPB. At the time of this study, the range of 30 PPB to 40 PPB mercury in caustic was believed to be the best sustainable quality in the Chlor Alkali industry using only mechanical separation equipment. The caustic filtration system recommended by this report is a composite system based upon all of the manufacturing facilities visited.

The final equipment sizing and optimum equipment operating procedures will require an investment in a pilot system. The filtration system involves, first, use of a recycle stream from the discharge of the existing filters back to the caustic surge tank or receiver. This stream is at least equal to the normal caustic production flow forward to storage. The second part of the recommended system is to achieve a filter area flux rate of 0.25 gallons per minute per square foot. The third process feature is the use of a finely ground carbon pre-coat for the filter tubes. This pre-coat is to "fill" the pores of the filter element and not meant to provide the separation medium for the elemental mercury to be removed. This conditioning medium will make the filter elements less porous and thus more efficient. The fourth process optimization reduces the allowable pressure drop to a maximum of 15 PSI. The backwash of the filters would be done first on 15 PSI pressure drop, and second on high mercury. The fifth process enhancement is incorporation of backpressure control on the filter discharge to eliminate the negative impact of switching caustic storage tanks. The sixth process adaptation is a procedure by which fine carbon is body fed to the filter upon elevated or mercury break-through to prolong the filter cycle.

VI. Team Recommendations

The entire Team recommends purchase of a pilot filter system. The pilot program requires an investment in equipment, and commitment of our development resources to design and interpret the data from the proposed experimental plan. The Team recommends working with the Chlorine Institute to help us share the cost of this effort. However, the Team further believes that we must go forward with a pilot system on our own if necessary.

In addition to pilot testing, our Team makes recommendations in the following areas:

1. Continue to develop approaches to cooling the decomposer operation i.e. cooler caustic inside the decomposer and cooler caustic leaving the decomposer
2. Install 'Sir Galahad' on-line analyzers for final product and pre-coat recycle streams.
3. Optimize filter flux rates and caustic recycle rates.
4. Install filter recycle, pump capacity, and filter surface area capacity to match the results of the pilot testing.
5. Retain current lab techniques for mercury analysis.

A limited testing program with existing production equipment may provide a cost-effective and timely way to verify some of the conclusions drawn in this report. However, due to many factors only a limited amount of testing can be done without interruption of normal production, and would require equipment installation. Therefore, a combination of pilot testing and field-testing at one or both production sites provides the best approach for determining the exact requirements for our caustic filtration systems. The results from these studies would allow more precise capital assessments as well as providing a more through knowledge of caustic filter operation that is desperately needed in order to consistently achieve 30-40 PPB mercury, or lower, in caustic product.