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REINJECTION WATER AT THE NORTH BOUNDARY OF RMA WILL BE S DRINKING WATER STANDARDS. IN RESPONSE TO THIS DIRECTIVE ENGAGED BY RMA TO PERFORM A FEASIBILITY STUDY DURING SEE DOCUMENTED THAT USING THE ACTIVATED ALUMINA TREATMENT ME FLUORIDE CAN BE REMOVED FROM THE CARBON TREATED WATER AT AT THE CONCLUSION OF THE FEASIBILITY STUDY, IT WAS DETER TESTING WOULD BE NECESSARY TO OPTIMIZE THE CAUSTIC REGEN MAXIMUM LONG-TERM ECONOMY. THEREFORE, THIS PILOT TEST F THE FLUORIDE REMOVAL PILOT TEST RUNS HAVE DEMONSTRATED T FLUORIDE REMOVAL WATER TREATMENT PLANT, WITH RAW WATER F RELIABLY REMOVE 1700 GRAINS OF FLUORIDE PER CUBIC FOOT O OPERATING TREATMENT COST PROJECTION FOR THE FULL SCALE F CAPITAL COST ESTIMATE FOR THE 1000 GPM TREATMENT SYSTEM	E, RUBEL AND HAGER WAS PTEMBER 1978. THE STUDY ETHOD, THE EXCESS I THE REINJECTION SITE. RMINED THAT FURTHER PILOT NERATION PROCEDURE FOR PROGRAM WAS ESTABLISHED. IHAT A FULL SCALE PH ADJUSTMENT, CAN DF ACTIVATED ALUMINA. PLANT IS 15/1000 GALLONS.
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Standard Form 298 (Rev. 2-89 Prescribed by ANSI Std. 239-18 298-102

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FINAL REPC

ON

PILOT TEST PROGRAM

FOR

REMOVAL OF EXCESS FLUORIDE

FROM

ACTIVATED CARBON EFFLUENT

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NTIS DTIC Unanno Justific								
By Distrib	By Distribution /							
A	Availability Codes							
Dist	Dist Avail and / or Special							
A-1								

Rocky Mountain Arsenal Information Center Commerce City, Colorado

FOR

THE DEPARTMENT OF THE ARMY ROCKY MOUNTAIN ARSENAL (REF. CONTRACT #DAAA05-79-C-006)

Ву

FILE COPY

Rubel and Hager, Inc. 4400 E. Broadway, Suite 710 Tucson, Arizona 85711

November 30, 1979

Copies

1 - 6: Carl G. Loven

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FINAL REPORT ON PILOT TEST PROGRAM FOR REMOVAL OF EXCESS FLUORIDE FROM ACTIVATED CARBON EFFLUENT FOR THE DEPARTMENT OF THE ARMY ROCKY MOUNTAIN ARSENAL (REF. CONTRACT #DAAA05-79-C-006)

> by Rubel and Hager, Inc. November 30, 1979

INTRODUCTION

In accordance with a directive from the Colorado Department of Health, the reinjection water at the north boundary of the Rocky Mountain Arsenal will be subject to drinking water standards established by the U. S. Environmental Protection Agency and the Colorado Department of Health. In addition to organic limitations for which purpose the granular activated carbon system was installed, there is also a specific limit of 2.4 mg/l of fluoride.

In response to this directive, this firm, Rubel and Hager, Inc. was engaged by the Rocky Mountain Arsenal to perform a feasibility study¹ during September 1978. The results of that study documented that using the activated alumina treatment method the excess fluoride can be removed from the carbon treated water at the re-That study, consisting of two complete treatment injection site. cycles including chemical regeneration, demonstrated the removal of fluoride from levels of 4-5 mg/l to an average of 1 mg/l. А capacity of more than 2,000 grains of fluoride per cubic foot of activated alumina was achieved in both cycles. That fluoride removal level is far below the maximum contaminant level requirement for fluoride of 2.4 mg/l established by the EPA and the Colorado Department of Health. At the conclusion of the feasibility study, it was determined that further pilot testing would be necessary to optimize the caustic regeneration procedure for maximum long term economy. Therefore, this pilot test program was established.

The purpose of this program was to determine whether there are any interferences present in the activated carbon effluent that may reduce the efficiency of the process. An optimum alumina regeneration process will be developed to minimize operating costs. The working capacity of the activated alumina along with chemical consumption upon which a full-scale plant can be designed will be determined.

TEST APPARATUS

The test apparatus is a fully assembled skid-mounted treatment system (see Figure 1 for Schematic Flow Diagram). The system is designed to treat the activated carbon effluent at the rate of 5 gpm at 50 psig maximum working pressure. There are two treatment vessels (fourteen-inch diameter by ninety six inches high) each contains 3.75 cubic feet of Alcoa (grade F-1, - 28+48 mesh) activated alumina. This manually operated system is piped to permit one treatment unit to perform design flow treatment individually, or two units can be operated in either series or parallel. The system contains 1" PVC schedule 80 threaded pipe and fittings with manually operated full port ball valves. Accessories for each treatment unit include pH sensors with panel mounted indicators at inlet and outlet, sample points at inlet and outlet, a pressure gauge, an air vent, a flow totalizer, injection points for acid and caustic, and an in-line static chemical mixer.

The system also includes a feed water pump, a caustic feed system, an acid feed system, a zeolite softener, and sample points. The caustic feed system includes one twelve gallon solution tank and one metering pump for 50% sodium hydroxide. The caustic is employed during regeneration only; therefore, one pump serves both treatment units. The acid feed system includes one fifty gallon solution tank and two metering pumps for dilute sulfuric acid. One acid pump is required for pH adjustment for each treatment unit. The zeolite softener is an optional feature which can be used to pretreat raw water during caustic regeneration. There are also provisions for adding other pretreatment equipment, or additional treatment units.

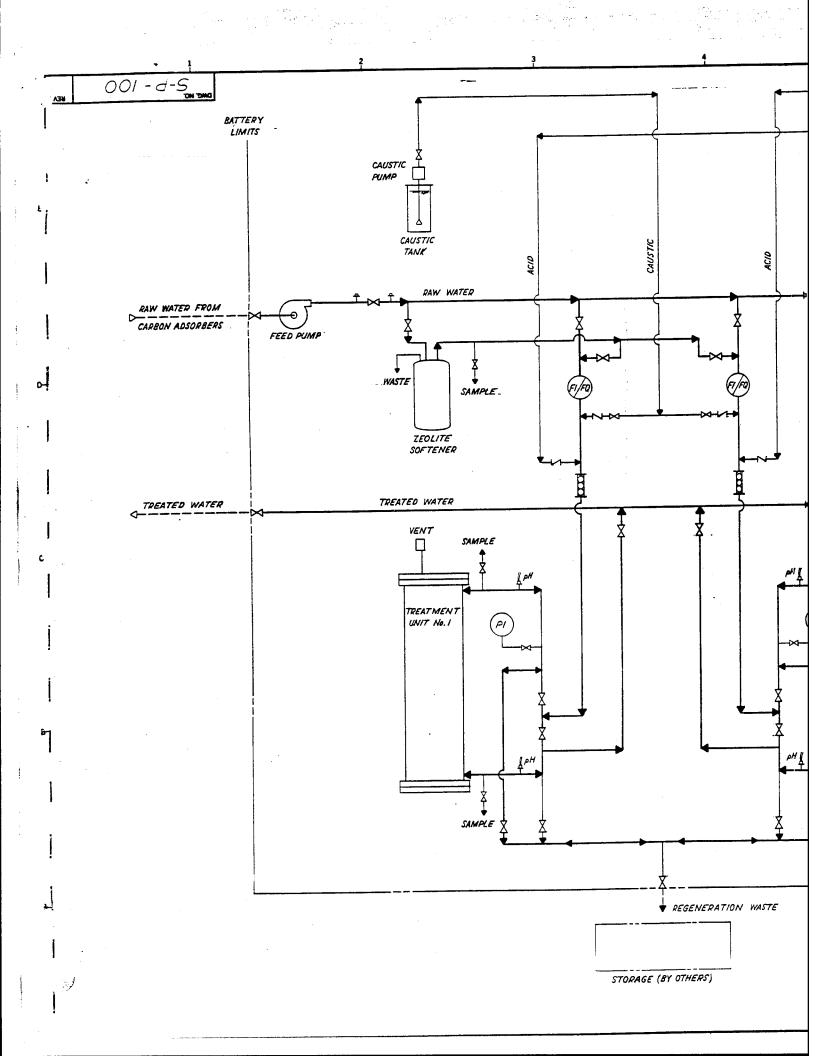
The entire treatment system is mounted on a steel skid (six feet wide by ten feet long) which is elevated approximately eighteen inches above the floor. The skid is located adjacent to the activated carbon system in the north boundary treatment building. Raw water is piped from the carbon adsorbers to the feed water pump suction. The treated water is piped to a designated discharge point. Regeneration wastewater is collected in 50 gallon drums for analysis and waste treatment process development.

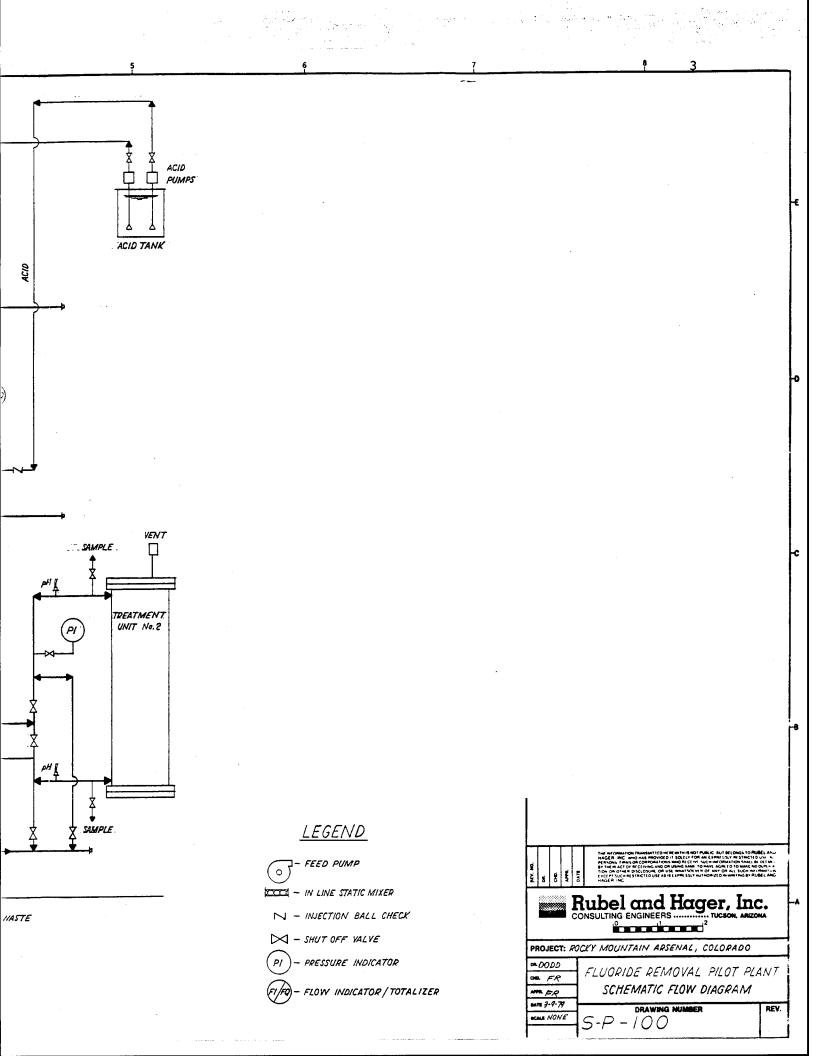
TEST PROGRAM

The scope of the test program for the 5 gpm pilot included six complete treatment cycles. Each cycle consisted of a treatment run through exhaustion of the treatment bed followed by a chemical regeneration.

Treatment Unit No. 1 was designated as the primary treatment unit; Treatment Unit No. 2 was designated as the standby treatment unit. All testing took place in the primary unit.

Upon completion of the installation of the pilot plant test





apparatus, initial start-up procedures began. This entailed placing the treatment media in the vessels and properly backflushing. Concurrently all instruments were calibrated and mechanical equipment checked out. The EPA Technical Report, "Removal of Excess Fluoride from Drinking Water" by Rubel and Woosley² elaborates upon start-up and operating procedures which apply to this type of treatment plant.

During all treatment runs, operating personnel conducted pilot plant surveillance and operating functions which included the following:

- Monitor raw water flow rate, maintain at 5 gpm ± 1/4 gpm (unless directed otherwise)
- 2. Monitor raw water pH, maintain at 5.3-5.5 (unless directed otherwise)
- 3. Sample raw and treated water for analysis at six hour intervals. Record pH and fluoride levels at time of sampling. Collect samples for laboratory analysis.
- 4. Maintain record at pilot plant of flow rate, total flow, pH and fluoride levels.
- 5. Maintain supply of dilute acid in solution tank. Record all concentrated acid additions.

Treatment runs were continuous until terminated upon saturation of the treatment bed. Saturation was defined as the point at which the average fluoride level in the treated water exceeded 1.20 mg/l (optimum level for this climate.) In any operational fluoride removal plant equipped with two or more treatment units and/or treated water storage facilities, blending is achieved resulting in constant distribution of optimum levels of fluoride.

The Regeneration Mode included backwash at 7-1/2 gpm for ten to twenty minutes. The bed was then drainned, followed by an upflow regeneration with 100 gallons of 1% sodium hydroxide using softened raw water to dilute 50% sodium hydroxide at a flow rate of 2.5 gpm for forty minutes. This was followed by an upflow soft water rinse of 5 gpm for 120 minutes followed by another draining of the liquid level down to the top of the bed. Finally, a downflow regeneration identical to the upflow described above was accomplished. This was followed by downflow neutralization using soft raw water adjusted to pH 2.5 for 120 minutes. At this point raw water was switched over to unsoftened for the duration of the neutralization and treatment run. When the pH of the treated effluent dropped to 7.5, the pH of the raw water was adjusted to 3.5. When the pH of the treated effluent reached 6.5, the pH of the raw water was adjusted to 5.5 where it remained for the duration of the run.

Wastewater from the regeneration and neutralization was collected in 50 gallon batches for study of ultimate disposal methods. The regeneration described above was not modified during the six test runs.

TEST RESULTS

The fluoride level in the carbon treatment effluent, designated as raw water, remained in the range of 3.6-4.0 mg/l throughout the duration of the pilot plant test program.

Initial tests indicated loss of efficiency due to interference from competing ions in the raw water. However, the interfering ions were regenerated from the bed; and process efficiency experienced no degradation. The bed capacity was directly affected by the quantity of interfering ions present in the raw water. Increased fluoride removal capacity was experienced during treatment runs with lower levels of competing ions. The interfering ions were recovered in the regeneration wastewater and were identified as organics designated as Total Organic Carbon (T.O.C.).

The first five (5) treatment runs employed the planned fluoride removal treatment and regeneration processes. A plot of the data for a typical treatment run (No. 4) is illustrated in Table No. 1; a plot of the test data for a typical regeneration (No. 4) is illustrated in Table No. 2. The only exceptions took place in treatment run number 5 when the flow rate was lowered from 5 gpm to 4 gpm. The change of flow rate had no effect upon the process.

The treatment process was altered during the sixth treatment run to illustrate the raw water pH adjustment effect upon the fluoride removal process. During this run pH adjustment was eleiminated. The treatment bed was neutralized according to the normal process to the raw water pH of 7.3 where it remained during the duration of the run. The result was a loss of two thirds of the fluoride removal capacity during this run. Figures No.2 and No.3 illustrated the comparative performance of treatment runs with and without pH adjustment.

Summary of the results of the test runs are as follows:

Ru	n No.	pH Adjustment	Flow (gpm)	Fluoride Removal grains/ft ³	TOC Removal grains/ft ³
	1	yes	5	1350	375
	2	yes	5	1550	225
·7	3	yes	5	1650	225
	4.	yes	5	1750	200
	5	yes	4	1700	250
-	6	no	5	575	not measured

TABLE NO.1

TABULATED DATA TREATMENT RUN NO. 4

-								
				RAW	WATER		FREATED	WATER
		METER	TOTAL					FLUORIDE
DATE	TIME	READING	FLOW	рH	F (mg/l	рĦ	F mg/l	TOTAL GRAINS
		_						
7/16/79	1730	43820		2.5	3.8	11.6	3.4	
	1830	44120	300	2.5	3.8	9.4	0.65	13
	2000	44560	740	3.5	3.8	6.9	0.10	95
	2400	45730	1910	5.3	3.8	5.4	0.08	348
7/17/79	0600	47480	3660	5.3	3.8	5.4	0.08	727
	1200	49270	5450	5.4	3.8	5.4	0.08	1114
	1800	51030	7210	5.3	3.8	5.3	0.07	1495
	2400	52790	8970	5.3	3.8	5.3	0.08	1876
7/18/79	0600	54540	10720	5.4	3.8	5.3	0.11	2255
	1200	56280	12460	5.5	3.8	5.5	0.15	2631
	1800	57990	14170	5.4	3.8	5.5	0.3	2988
	2400	59700	15880	5.0	3.8	5.0	0.5	3326
7/19/79	0600	61370	17550	5.0	3.8	5.0	0.7	3636
	1200	63210	19390	5.2	3.8	5.5	1.1	3948
·	1800	65020	21200	5.6	3.8	5.6	1.4	4214
	2400	66740	22920	5.0	3.8	5.0	1.5	4450
7/20/79	0600	6854 0	24720	5.0	3.8	5.0	1.5	4692
	1200	70110	26290	5.4	3.8	5.4	1.6	4899
	1800	71880	28060	5.6	3.8	5.4	1.7	5121
•	2400	73600	29780	5.2	3.8	5.2	1.8	5327
7/21/79	0600	75450	31630	5.1	3.8	5.1	1.8	5543
	1200	77120	33300	5.4	3.8	5.5	1.9	5733
	1800	78740	34920	5.4	3.8	5.5	2.1	5904
	2400	70420	36600	5.4	3.8	5.4	2.1	6080
7/22/79	0600	82190	38370	5.1	3.8	5.2	2.2	6246
	1200	83920	40100	5.6	3.8	5.6	2.3	6402
	1600	85120	43100	5.4	3.8	5.5	2.3	6507

Total Grains per cubic foot - 1736

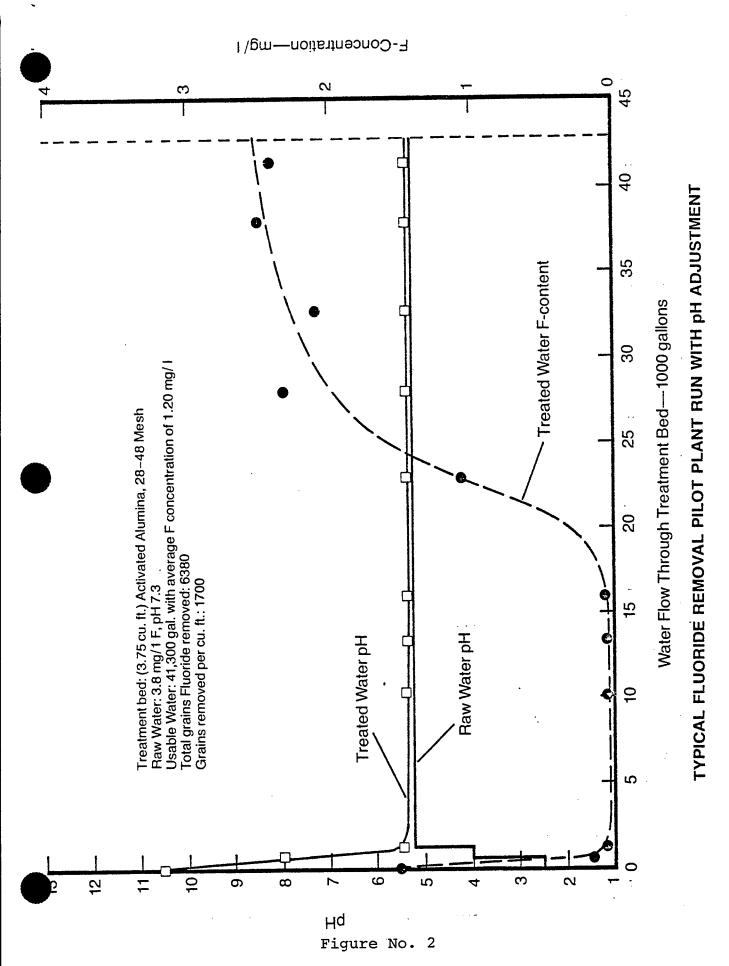
TABLE NO. 2

TABULATED DATA - REGENERATION NO. 4

50 GALLON BATCH NO.	pH	F(mg/1)	FLUORIDE TOTAL GRAINS	T.O.C. (mg/l)	T.O.C. TOTAL GRAINS
l	9.7	119	348	55	160
2	12.5	617	2152	21	221
3	12.6	541	3734	76	444
	12.2	263	4503	88	701
4 5	11.9	114	4836	11	733
6	11.6	57	5004	5.5	750
7	11.4	32	5097	-	-
8	11.2	27	5176	-	-
8 9	11.0	21	5237	-	-
10	10.9	17	5287		-
11	10.7	16	5333	-	-
12	10.3	13	5371	-	-
13	11.1	102	5669	-	-
14	13.1	239	6367	-	-
15	12.4	59	6539		-
16	12.1	21	6601	_	-

Total Grains Fluoride per cubic foot Activated Alumina - 1760 Total Grains T.O.C. per cubic foot Activated Alumina - 200

2.92 110 30 2



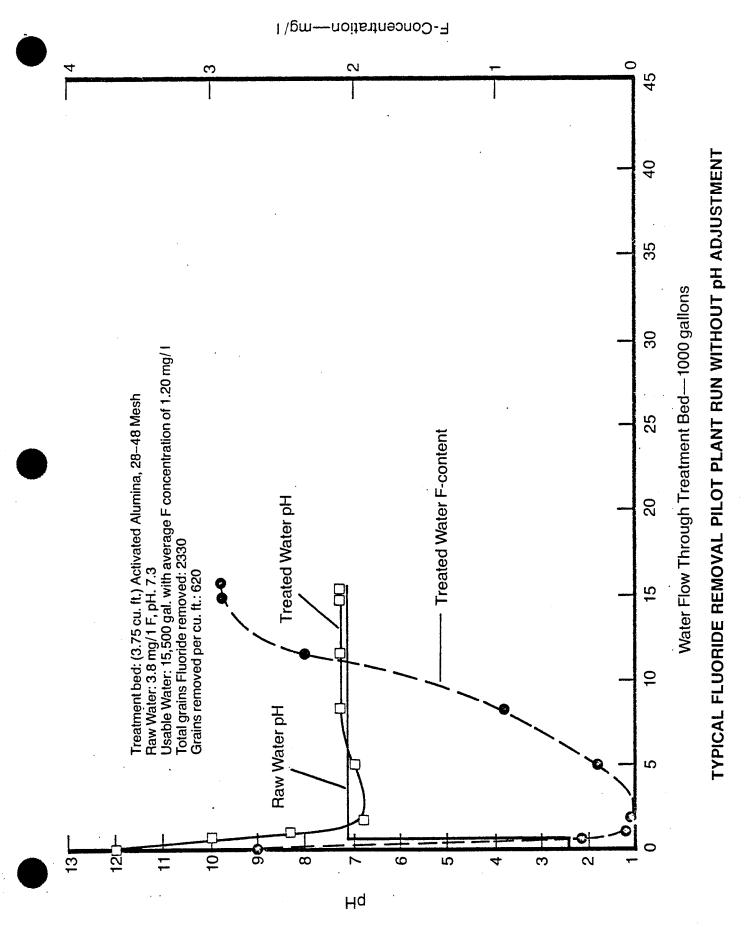


Figure No. 3

ECONOMICS

During the pilot plant treatment runs and regenerations exact figures on chemical consumption were maintained. Figure 4 illustrates chemical consumption and related chemical costs for pilot plant runs 3, 4, 5 and 6. Chemical costs are current and include delivery to the Arsenal. One can see from this data that when raw water pH adjustment is not employed, chemical costs and wastewater volumes requiring treatment and/or handling will double.

From the pilot test data the operating cost for the full scale fluoride removal water treatment plant have been projected. This cost is approximately 15¢/1000 gallons; see Figure 5.

In the following section design criteria is established for the full scale fluoride removal water treatment plant process equipment. The estimated cost for the installed system is \$270,000, see Figure 6.

DESIGN CRITERIA

The design of the Fluoride Removal Water Treatment Plant for the Activated Carbon Effluent (raw water) at the North Boundary of the Rocky Mountain Arsenal, Commerce City, Colorado employing the activated alumina treatment process with caustic regeneration and acid neutralization is to be based on the following criteria:

A. Fluoride levels

- 1. Raw Water 3.5-4.0 mg/1
- 2. Treated Water 1.2 mg/l (avg.)

B. Treatment

- 1. Material Spec. Alcoa Activated Alumina Grade F-1, -28 + 48 mesh
- 2. Bed Design
 - Number of beds (treatment units) 2
 - Bed depth 5'-0"
 - Bed expansion during backwash 50% = 2'-6"
 - Tank free board 6"
 - Superficial residence time of raw water flowing through bed 5 minutes (min)
 - Treatment unit flow rate 7 gpm/ft.² (max)

Pilot Plant Treatment Chemical Cost and Wastewater Summary

-:;)

* With acid neutralization but without pH adjustment during run

Pilot Plant Run I Total Treated W Acid Consumed Acid Consumed Gallons of Acid (1000 gal. trea Cost of Acid/ 1000 gal. trea @ 441/2¢/gal. (**Caustic Consur** Gallons of Caus 1000 gal. treat Cost of Caustic/ 1000 gal. treai @ 88[¢]/gal. of Softener Salt Cc Lb. of Softener S 1000 gal. treat Cost of Softener 1000 gal. treat @ 2.9[¢]/lb. of s Total Chemical (1000 gal. of tre

Recycle (backwa neutralization % Treated Wa

High Fluoride Waste Water % Treated Wa

Plant Run Number	3	4	5	*6]
Treated Water Flow/gallons	42,600	41,300	41,300	15,500	
Consumed (ml)	27,300	23,800	21,600	7,000	
Consumed gallons	7.21	6.29	5.71	1.85	
ns of Acid Consumed/ 00 gal. treated water of Acid/	0.17	0.15	0.14	.12	
0 gal. treated water, 141⁄₂¢/gal. of acid	<u>075</u> ¢	<u>.068</u> ¢	<u>.061¢</u>	<u>.053</u> ¢	
ic Consumed gallons ns of Caustic Consumed/	2.67	2.67	2.67	2.67	
0 gal. treated water	.063	.065	.065	.172	
0 gal. treated water, 38⊄/gal. of caustic	<u>.055</u> ⊄	<u>.057</u> ¢	<u>.057</u> ¢	<u>.152</u> ¢	
her Salt Consumed/lbs. Softener Salt Consumed/	25	25	25	25	
0 gal. treated water of Softener Salt/	.587	.605	.605	1.61	
0 gal. treated water, 2.9⊄/lb. of softener salt	<u>.017¢</u>	<u>.018</u> ¢	<u>.018</u> ¢	<u>.047¢</u>	
Chemical Cost/ 0 gal. of treated water	<u> 14.7¢</u>	<u>14.3</u> ¢	<u>13.6¢</u>	<u>25.2</u> ¢	
le (backwash + tralization water-gallons) reated Water	300 1%	500 1%	400 1%	200 1%	
luoride ste Water reated Water	1000 2½%	900 2%	700 1¾%	600 4%	

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	Cost ga treate	Cost per 1000 gallons treated water
1. Chemicals		
a. 66° Baume H ₂ SO₄ at 44.5⁴/gallon		\$.061
b. 50% NaOH at 88⁴/gallon		.057
c. Salt at 2.9¢/lb.		.018
	Subtotal	\$.136
2. Operator (existing)		
Alumina Replacement		
3% per year @ 40⁴/lb.		\$.002
4. Miscellaneous supplies and services		200.
Electric Utility @ 5⁴/kwh		.012
	Total	\$.157

Rocky Mountain Arsenal Fluoride Removal Water Treatment Plant Capital Cost Estimate

Process Equipr Treatment Vessels Process Piping an Treatment Media Chemical Storage Chemical Pumps, Regeneration Soft

Process Equipr Mechanical Electrical Miscellaneous

Miscellaneous I Slabs, Foundation Pre-Engineered St

Miscellaneous Freight & Taxes Contingencies

Fees Engineering Other

	میں جو ایس اور	
ss Equipment		
nent Vessels		\$ 28,000
s Piping and Installation		24,000
nent Media		17,000
cal Storage Vessels		14,000
cal Pumps, Piping and Accessories		7,000
eration Softener		20,000
	Subtotal	\$110,000
ess Equipment Installation		
nical		\$ 20,000
cal		15,000
aneous		15,000
	Subtotal	\$ 50,000
llaneous Installed Items		:
Foundations, Earthwork, Site Work		\$ 25,000
gineered Steel Building		25,000
	Subtotal	\$ 50,000
llaneous		- ·
t & Taxes		\$ 8,000
gencies		17,000
	Subtotal	\$ 25,000
ering		\$ 25,000
		10,000
	Subtotal	<u>\$ 35,000</u>
	Total	<u>\$270,000</u>

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- Treatment unit backwash flow rate - 10 gpm/ft.² (max)

3. Treatment Unit Design

- 50 psi ASME unfired pressure vessel code design
- Rubber lined
- Access manway at top with platform, drain at bottom
- Lifting lugs
- Skid mounted
- Air bleed vent/vacuum break
- Internal header and lateral collection pipe system
- Distribution baffle
- Inlet and outlet flanges
- 4. Piping System Design
 - Liquid velocities (except backwash) 10 ft./sec. (max)
 - Liquid velocity backwash 13 ft./sec. (max)
 - Material suitable for dilute acid and dilute caustic service
 - Manual valves butterfly, gate
 - Measure flow rate into unit, total flow into unit
 - Sampling system to monitor pH and Fluoride
 - Control treatment system flow rate
 - Control treatment system pressure

C. Quantity and Quality of Treated Water for Distribution

- 1. Raw water flow rate- 1000 gpm (max). Treatment unit design. Flow rate 300 gpm each, or 600 gpm.
- Treated water total flow 97% raw water flow -840,000 GPD.
- 3. Waste water 3% raw water flow 26,000 GPD.
- 4. Measure fluoride content of treated water.

- 5. Measure pH level of treated water. Include high and low pH alarms interlocked with raw water pump power supply; thereby plant will sound alarm and will shut down in the event of high or low pH. Include provision to flush system to waste in the event of high or low pH shut down. Manual startup only after shutdown.
- 6. Incorporate provisions to lower raw water pH and raise treated water pH.

D. Regeneration and Neutralization

- 1. Regeneration material 1% NaOH
 - Blend of 50% NaOH and softened raw water in "mixing T" at treatment unit
 - 50% NaOH procured directly from caustic manufacturer, delivered to plant in tank trucks
 - Provide 6000 gallon insulated caustic storage tank (horizontal) with manway, immersion heater (incl. controls) to maintain 70° F min., lifting lugs, vent w/overflow, fill piping, drain piping, level measurement capability and outlets to service.
- 2. Regeneration process
 - Flow rate through treatment unit 2-1/2 gpm/ft.² (max)
 - Residence time in treatment bed 40 minutes (min)
 - Incorporate provision for upflow or downflow through bed
- 3. Regeneration Equipment
 - Chemical pump Metering pumps with precision flow control. Corrosion resistant material - Interlock pump controls with well pump power supply so that chemical pump can't operate when pump is off. Pump mounted upon steel stand above Day Tank. Day Tank filled via gravity from Caustic Storage
 - Plug valves, check valves, pressure relief valves and back-pressure valves as required for total operational control and good maintenance.
 - Provide emergency shower and eyewash

4. Neutralization material - 0.04% H₂SO₄

- Blend of 93% H₂SO₄ and softened water in "mixing T" at treatment unit
- 93% H₂SO₄ procured directly from acid manufacturer, delivered to plant in tank trucks
- Provide 6000 gallon acid storage horizonizontal tank with manway, lifting lugs, vent w/overflow, fill piping, drain piping, level measurement capability and outlet to service
- 5. Neutralization process
 - Flow rate through treatment unit 7 gpm/ft.² (max)
 - Amount of acid rinse required sufficient to adjust pH within acceptable pH limits 6.5 - 8.5
 - Incorporate provision for upflow or downflow through bed
- 6. Neutralization equipment
 - Chemical pumps 2 Metering pumps with precision flow control. Corrosion resistant material. Interlock pump controls with well pump supply so that chemical pumps cannot operate when well pump is off. Pumps mounted upon steel stand above Day Tank. Day Tank filled via gravity from Acid Storage Tank.
 - Plug valves, check valves, pressure relief valves and back-pressure valves as required for total operational control and good maintenance.
- 7. Zeolite softener complete with brine tank and automatic regeneration. Size softener to remove hardness ions from 300 gallons (min) of raw water per cubic feet of activated alumina in individual treatment unit. Hardness level during pilot testing was 30 grains per gallon. Include manual isolation valves to employ softened water only during regeneration.
- E. Treatment Building
 - 1. Provide facility to house Treatment Units, Chemical Handling Systems, and Sampling System.
 - 2. House operator's tools, supplies, records, etc.
 - 3. House chemical test equipment with lab sink, counter and cabinets.

Abide by Applicable Portions of the Following Codes and Standards

- ASME Boiler & Pressure Vessel Code Section VIII, Division 1
- 2. ASTM
- 3. AWWA
- 4. American Concrete Institute Building Code 318
- 5. National Electric Code
- 6. National Sanitation Foundation Standards
- 7. Manufacturing Chemists Association
 - a. Chemical Safety Data Sheet SD-9 Caustic Soda
 - b. Chemical Safety Data Sheet SD-20 Sulfuric Acid
- 8. OSHA
- 9. Uniform Building Code

CONCLUSIONS

The fluoride removal pilot test runs have demonstrated that a full scale fluoride removal water treatment plant with raw water pH adjustment, can reliably remove 1700 grains of fluoride per cubic foot of activated alumina. Operating treatment cost projection for the full scale plant is 15¢/1000 gallons. Capital cost estimate for the 1000 gpm treatment system is \$270,000.

Interfering ions present in the raw water are competing fluoride for adsorption sites on the granular activated alumina surfaces. The interfering ions have been identified as T.O.C. Although the T.O.C. is easily removed during regeneration, its presence does reduce the fluoride removal capacity of the activated alumina to the above mentioned 1700 grains per cubic foot.

The use of softened raw water during regeneration and neutralization has increased the efficiency of the regeneration process.

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- 1. Rubel and Hager, Inc., "Feasibility Study for the Removal of Excess Fluoride from Activated Carbon Effluent", The Deaprtment of the Army, Rocky Mountain Arsenal (Ref. #DAAA05-78-M-0914), ITARMS Project No. 1.05.15, September 30, 1978
- Rubel, Jr., Frederick and Woosley, R. Dale,
 "Removal of Excess Fluoride from Drinking Water",
 Technical Report EPA 570/9-78-001, Jan. 1978

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FINAL REPC

ON

PILOT TEST PROGRAM

FOR

REMOVAL OF EXCESS FLUORIDE

FROM

ACTIVATED CARBON EFFLUENT

Rocky Mountain Arsenal Information Center Commerce City, Colorado

FOR

THE DEPARTMENT OF THE ARMY ROCKY MOUNTAIN ARSENAL (REF. CONTRACT #DAAA05-79-C-006)

By

FILE COPY

Rubel and Hager, Inc. 4400 E. Broadway, Suite 710 Tucson, Arizona 85711

November 30, 1979

Copies

1 - 6: Carl G. Loven

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FINAL REPORT ON PILOT TEST PROGRAM FOR REMOVAL OF EXCESS FLUORIDE FROM ACTIVATED CARBON EFFLUENT FOR THE DEPARTMENT OF THE ARMY ROCKY MOUNTAIN ARSENAL (REF. CONTRACT #DAAA05-79-C-006)

> by Rubel and Hager, Inc. November 30, 1979

INTRODUCTION

In accordance with a directive from the Colorado Department of Health, the reinjection water at the north boundary of the Rocky Mountain Arsenal will be subject to drinking water standards established by the U. S. Environmental Protection Agency and the Colorado Department of Health. In addition to organic limitations for which purpose the granular activated carbon system was installed, there is also a specific limit of 2.4 mg/l of fluoride.

In response to this directive, this firm, Rubel and Hager, Inc. was engaged by the Rocky Mountain Arsenal to perform a feasibility study¹ during September 1978. The results of that study documented that using the activated alumina treatment method the excess fluoride can be removed from the carbon treated water at the reinjection site. That study, consisting of two complete treatment cycles including chemical regeneration, demonstrated the removal of fluoride from levels of 4-5 mg/l to an average of 1 mg/l. Α capacity of more than 2,000 grains of fluoride per cubic foot of activated alumina was achieved in both cycles. That fluoride removal level is far below the maximum contaminant level requirement for fluoride of 2.4 mg/l established by the EPA and the Colorado Department of Health. At the conclusion of the feasibility study, it was determined that further pilot testing would be necessary to optimize the caustic regeneration procedure for maximum long term economy. Therefore, this pilot test program was established.

The purpose of this program was to determine whether there are any interferences present in the activated carbon effluent that may reduce the efficiency of the process. An optimum alumina regeneration process will be developed to minimize operating costs. The working capacity of the activated alumina along with chemical consumption upon which a full-scale plant can be designed will be determined.

TEST APPARATUS

The test apparatus is a fully assembled skid-mounted treatment system (see Figure 1 for Schematic Flow Diagram). The system is designed to treat the activated carbon effluent at the rate of 5 gpm at 50 psig maximum working pressure. There are two treatment vessels (fourteen-inch diameter by ninety six inches high) each contains 3.75 cubic feet of Alcoa (grade F-1, - 28+48 mesh) activated alumina. This manually operated system is piped to permit one treatment unit to perform design flow treatment individually, or two units can be operated in either series or parallel. The system contains 1" PVC schedule 80 threaded pipe and fittings with manually operated full port ball valves. Accessories for each treatment unit include pH sensors with panel mounted indicators at inlet and outlet, sample points at inlet and outlet, a pressure gauge, an air vent, a flow totalizer, injection points for acid and caustic, and an in-line static chemical mixer.

The system also includes a feed water pump, a caustic feed system, an acid feed system, a zeolite softener, and sample points. The caustic feed system includes one twelve gallon solution tank and one metering pump for 50% sodium hydroxide. The caustic is employed during regeneration only; therefore, one pump serves both treatment units. The acid feed system includes one fifty gallon solution tank and two metering pumps for dilute sulfuric acid. One acid pump is required for pH adjustment for each treatment unit. The zeolite softener is an optional feature which can be used to pretreat raw water during caustic regeneration. There are also provisions for adding other pretreatment equipment, or additional treatment units.

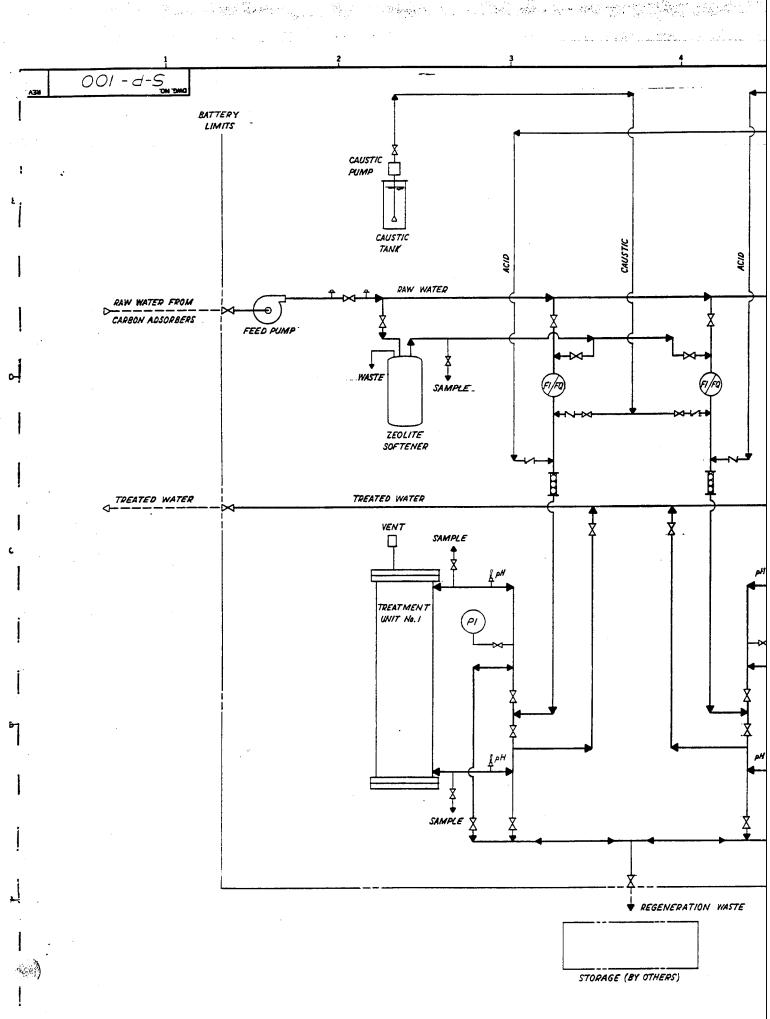
The entire treatment system is mounted on a steel skid (six feet wide by ten feet long) which is elevated approximately eighteen inches above the floor. The skid is located adjacent to the activated carbon system in the north boundary treatment building. Raw water is piped from the carbon adsorbers to the feed water pump suction. The treated water is piped to a designated discharge point. Regeneration wastewater is collected in 50 gallon drums for analysis and waste treatment process development.

TEST PROGRAM

The scope of the test program for the 5 gpm pilot included six complete treatment cycles. Each cycle consisted of a treatment run through exhaustion of the treatment bed followed by a chemical regeneration.

Treatment Unit No. 1 was designated as the primary treatment unit; Treatment Unit No. 2 was designated as the standby treatment unit. All testing took place in the primary unit.

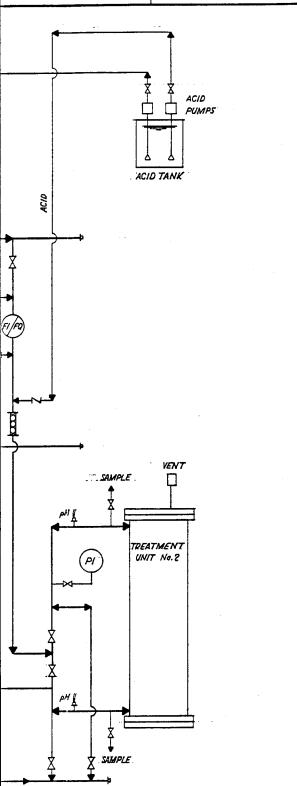
Upon completion of the installation of the pilot plant test



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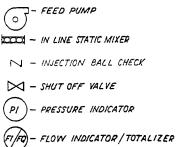
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ON WASTE

LEGEND



ţ and Hager, Inc. ING ENGINE PROJECT: ROCKY MOUNTAIN ARSENAL, COLORADO .DODD FLUORIDE REMOVAL PILOT PLANT one FR SCHEMATIC FLOW DIAGRAM ME FR DATE 3-9-79 DRAWING NUMBER REV. SCALE NONE S-P-100

apparatus, initial start-up procedures began. This entailed placing the treatment media in the vessels and properly backflushing. Concurrently all instruments were calibrated and mechanical equipment checked out. The EPA Technical Report, "Removal of Excess Fluoride from Drinking Water" by Rubel and Woosley² elaborates upon start-up and operating procedures which apply to this type of treatment plant.

During all treatment runs, operating personnel conducted pilot plant surveillance and operating functions which included the following:

- Monitor raw water flow rate, maintain at 5 gpm ± 1/4 gpm (unless directed otherwise)
- 2. Monitor raw water pH, maintain at 5.3-5.5 (unless directed otherwise)
- Sample raw and treated water for analysis at six hour intervals. Record pH and fluoride levels at time of sampling. Collect samples for laboratory analysis.
- Maintain record at pilot plant of flow rate, total flow, pH and fluoride levels.
- 5. Maintain supply of dilute acid in solution tank. Record all concentrated acid additions.

Treatment runs were continuous until terminated upon saturation of the treatment bed. Saturation was defined as the point at which the average fluoride level in the treated water exceeded 1.20 mg/l (optimum level for this climate.) In any operational fluoride removal plant equipped with two or more treatment units and/or treated water storage facilities, blending is achieved resulting in constant distribution of optimum levels of fluoride.

The Regeneration Mode included backwash at 7-1/2 gpm for ten to twenty minutes. The bed was then drainned, followed by an upflow regeneration with 100 gallons of 1% sodium hydroxide using softened raw water to dilute 50% sodium hydroxide at a flow rate of 2.5 gpm for forty minutes. This was followed by an upflow soft water rinse of 5 gpm for 120 minutes followed by another draining of the liquid level down to the top of the bed. Finally, a downflow regeneration identical to the upflow described above was accomplished. This was followed by downflow neutralization using soft raw water adjusted to pH 2.5 for 120 minutes. At this point raw water was switched over to unsoftened for the duration of the neutralization and treatment run. When the pH of the treated effluent dropped to 7.5, the pH of the raw water was adjusted to 3.5. When the pH of the treated effluent reached 6.5, the pH of the raw water was adjusted to 5.5 where it remained for the duration of the run.

Wastewater from the regeneration and neutralization was collected in 50 gallon batches for study of ultimate disposal methods. The regeneration described above was not modified during the six test runs.

TEST RESULTS

The fluoride level in the carbon treatment effluent, designated as raw water, remained in the range of 3.6-4.0 mg/l throughout the duration of the pilot plant test program.

Initial tests indicated loss of efficiency due to interference from competing ions in the raw water. However, the interfering ions were regenerated from the bed; and process efficiency experienced no degradation. The bed capacity was directly affected by the quantity of interfering ions present in the raw water. Increased fluoride removal capacity was experienced during treatment runs with lower levels of competing ions. The interfering ions were recovered in the regeneration wastewater and were identified as organics designated as Total Organic Carbon (T.O.C.).

The first five (5) treatment runs employed the planned fluoride removal treatment and regeneration processes. A plot of the data for a typical treatment run (No. 4) is illustrated in Table No. 1; a plot of the test data for a typical regeneration (No. 4) is illustrated in Table No. 2. The only exceptions took place in treatment run number 5 when the flow rate was lowered from 5 gpm to 4 gpm. The change of flow rate had no effect upon the process.

The treatment process was altered during the sixth treatment run to illustrate the raw water pH adjustment effect upon the fluoride removal process. During this run pH adjustment was eleiminated. The treatment bed was neutralized according to the normal process to the raw water pH of 7.3 where it remained during the duration of the run. The result was a loss of two thirds of the fluoride removal capacity during this run. Figures No.2 and No.3 illustrated the comparative performance of treatment runs with and without pH adjustment.

Summary of the results of the test runs are as follows:

Ru	n No.	pH Adjustment	Flow (gpm)	Fluoride Removal 	TOC Removal grains/ft ³
	1	yes	5	1350	375
	2	yes	5	1550	225
17	3	yes	5	1650	225
	4'	yes	5	1750	200
	5	yes	4	1700	250
•	6	no	5	575	not measured

TABLE NO.1

TABULATED DATA TREATMENT RUN NO. 4

				RAW	WATER	ŗ	FREATED	WATER
		METER	TOTAL					FLUORIDE
DATE	TIME	READING	FLOW	pН	Ý(mg/l	рH	F mg/l	TOTAL GRAINS
7/16/79	1730	43820	_	2.5	3.8	11.6	3.4	-
//10//9	1830	44120	300	2.5	3.8	9.4	0.65	13
	2000	44560	740	3.5	3.8	6.9	0.10	95
	2400	45730	1910	5.3	3.8	5.4	0.08	348
7/17/79	0600	47480	3660	5.3	3.8	5.4	0.08	727
1/ 1/ 12	1200	49270	5450	5.4	3.8	5.4	0.08	1114
	1800	51030	7210	5.3	3.8	5.3	0.07	1495
	2400	52790	8970	5.3	3.8	5.3	0.08	1876
7/18/79	0600	54540	10720	5.4	3.8	5.3	0.11	2255
1/ 10/ / 2	1200	56280	12460	5.5	3.8	5.5	0.15	2631
	1800	57990	14170	5.4	3.8	5.5	0.3	2988
	2400	59700	15880	5.0	3.8	5.0	0.5	3326
7/19/79	0600	61370	17550	5.0	3.8	5.0	0.7	3636
.,,	1200	63210	19390	5.2	3.8	5.5	1.1	3948
	1800	65020	21200	5.6	3.8	5.6	1.4	4214
	2400	66740	22920	5.0	3.8	5.0	1.5	4450
7/20/79	0600	68540	24720	5.0	3.8	5.0	1.5	4692
.,,	1200	70110	26290	5.4	3.8	5.4	1.6	4899
	1800	71880	28060	5.6	3.8	5.4	1.7	5121
· ·	2400	73600	29780	5.2	3.8	5.2	1.8	5327
7/21/79	0600	75450	31630	5.1	3.8	5.1	1.8	5543
	1200	77120	33300	5.4	3.8	5.5	1.9	5733
	1800	78740	34920	5.4	3.8	5.5	2.1	5904
	2400	70420	36600	5.4	3.8	5.4	2.1	6080
7/22/79	0600	82190	38370	5.1	3.8	5.2	2.2	6246
	1200	83920	40100	5.6	3.8	5.6	2.3	6402
	1600	85120	43100	5.4	3.8	5.5	2.3	6507

Total Grains per cubic foot - 1736



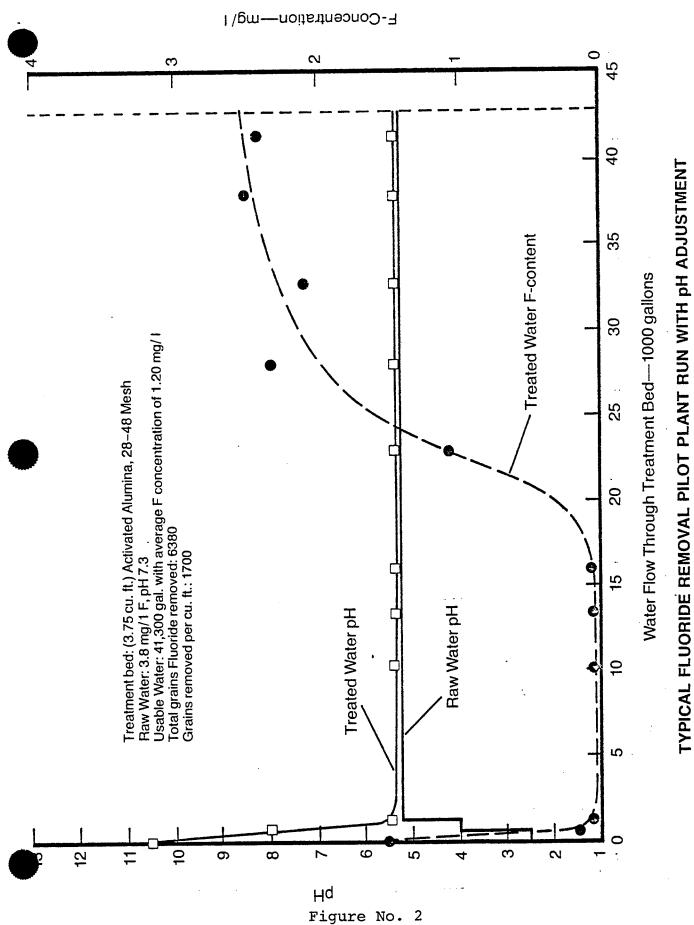
TABLE NO. 2

TABULATED DATA - REGENERATION NO. 4

50 GALLON BATCH NO.	рН	F(mg/l)	FLUORIDE TOTAL GRAINS 1	r.O.C. (mg/l)	T.O.C. TOTAL GRAINS
1	9.7	119	348	55	160
2	12.5	617	2152	21	221
3	12.6	541	3734	76	444
4	12.2	263	4503	88	701
5	11.9	114	4836	11	733
6	11.6	57	5004	5.5	750
7	11.4	32	5097	-	-
8	11.2	27	5176	-	-
9	11.0	21	5237	-	-
10	10.9	17	5287	-	-
11	10.7	16	5333	-	-
12	10.3	13	5371		-
13	11.1	102	5669	-	-
14	13.1	239	6367	-	-
15	12.4	59	6539	-	-
16	12.1	21	6601	-	-

Total Grains Fluoride per cubic foot Activated Alumina - 1760 Total Grains T.O.C. per cubic foot Activated Alumina - 200

2.92 110 34 8



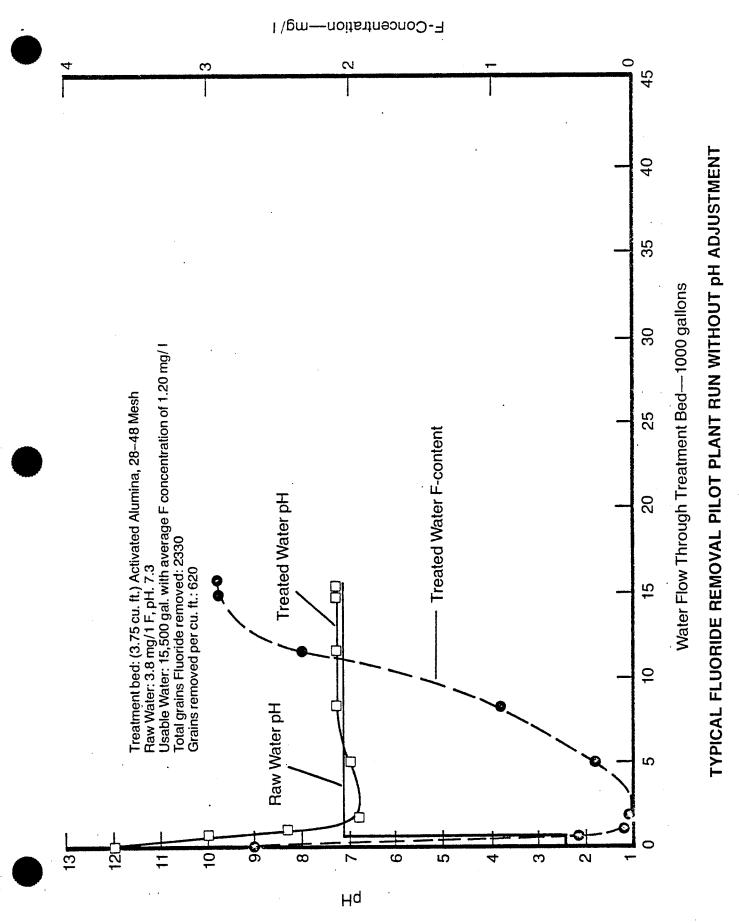


Figure No. 3

ECONOMICS

During the pilot plant treatment runs and regenerations exact figures on chemical consumption were maintained. Figure 4 illustrates chemical consumption and related chemical costs for pilot plant runs 3, 4, 5 and 6. Chemical costs are current and include delivery to the Arsenal. One can see from this data that when raw water pH adjustment is not employed, chemical costs and wastewater volumes requiring treatment and/or handling will double.

From the pilot test data the operating cost for the full scale fluoride removal water treatment plant have been projected. This cost is approximately 15¢/1000 gallons; see Figure 5.

In the following section design criteria is established for the full scale fluoride removal water treatment plant process equipment. The estimated cost for the installed system is \$270,000, see Figure 6.

DESIGN CRITERIA

The design of the Fluoride Removal Water Treatment Plant for the Activated Carbon Effluent (raw water) at the North Boundary of the Rocky Mountain Arsenal, Commerce City, Colorado employing the activated alumina treatment process with caustic regeneration and acid neutralization is to be based on the following criteria:

A. Fluoride levels

- 1. Raw Water 3.5-4.0 mg/l
- Treated Water 1.2 mg/l (avg.)

B. Treatment

- 1. Material Spec. Alcoa Activated Alumina Grade F-1, -28 + 48 mesh
- 2. Bed Design
 - Number of beds (treatment units) 2
 - Bed depth 5'-0"
 - Bed expansion during backwash 50% = 2'-6"
 - Tank free board 6"
 - Superficial residence time of raw water flowing through bed 5 minutes (min)
 - Treatment unit flow rate 7 gpm/ft.² (max)

Gallor 100 Cost o 100 @ Pilot Plant Caus Treatment Chemical Cost 100

and

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Wastewater Summary

* With acid neutralization but without pH adjustment during run Pilot Plant Run Total Treated W Acid Consumed Acid Consumed Gallons of Acid 1000 gal. trea Cost of Acid/ 1000 gal. trea @ 441/2¢/gal.

Caustic Consur Gallons of Caus 1000 gal. trea Cost of Caustic 1000 gal. trea @ 88¢/gal. of

Softener Salt C Lb. of Softener 1000 gal. trea Cost of Softene 1000 gal. trea

@ 2.9⊄/lb. of

Total Chemical 1000 gal. of tr

Recycle (backw neutralizatior % Treated W

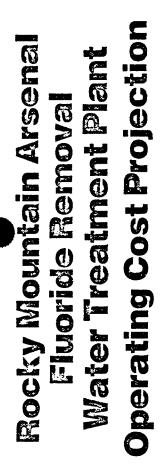
High Fluoride Waste Water % Treated W

			~		
ant Run Number reated Water Flow/gallons	3 42,600	4 41,300	5 41,300	*6 15,500	H2
pnsumed (ml)	42,000 27,300	41,300 23,800	21,600	7,000	
onsumed gallons	7.21	6.29	5.71	1.85	
of Acid Consumed/ gal. treated water Acid/	0.17	0.15	0.14	.12	•
gal. treated water, 11⁄2¢/gal. of acid	<u>.075</u> ¢	<u>.068</u> ¢	<u>.061¢</u>	<u>.053</u> ¢	
Consumed gallons	2.67	2.67	2.67	2.67	
gal. treated water Caustic/	.063	.065	.065	.172	
gal. treated water, ^{3¢} /gal. of caustic	_ .05 5⊄	<u>.057</u> ¢	<u>.057</u> ¢	<u>.152</u> ¢	
er Salt Consumed/lbs.	25	25	25	25	
oftener Salt Consumed/ gal. treated water Softener Salt/	.587	.605	.605	1.61	
gal. treated water, 9⊄/lb. of softener salt	<u>.017¢</u>	<u>018</u> ¢	<u>.018</u> ¢	<u>.047</u> ¢	
hemical Cost/ gal. of treated water	<u>14.7¢</u>	<u>14.3¢</u>	<u>13.6</u> ¢	<u>25.2</u> ¢	
e (backwash + alization water-gallons) eated Water	300 1%	500 1%	400 1%	200 1%	
uoride le Water eated Water	1000 2½%	900 2%	700 1¾%	600 4%	

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Process Equip Treatment Vessel Process Piping ar Treatment Media Chemical Storage Chemical Pumps, Regeneration Sof

Process Equip: Mechanical Electrical Miscellaneous

Miscellaneous Slabs, Foundatior Pre-Engineered S

Miscellaneous Freight & Taxes Contingencies

Fees Engineering Other

Rocky Mountain Arsenal Fluoride Removal Water Treatment Plant Capital Cost Estimate

	97 yan.	
ss Equipment		
ent Vessels		\$ 28,000
s Piping and Installation		24,000
ent Media		17,000
al Storage Vessels		14,000
al Pumps, Piping and Accessories		7,000
eration Softener		20,000
	Subtotal	\$110,000
ss Equipment Installation	,	
nical		\$ 20,000
al	:	15,000
aneous		15,000
	Subtotal	\$ 50,000
laneous Installed Items		
-oundations, Earthwork, Site Work		\$ 25,000
gineered Steel Building		25,000
	Subtotal	\$ 50,000
laneous		•
& Taxes		\$ 8,000
jencies		17,000
	Subtotal	\$ 25,000
ering		\$ 25,000
		10,000
	Subtotal	<u>\$ 35,000</u>
	Total	<u>\$270,000</u>
		

Seco

- Treatment unit backwash flow rate - 10 gpm/ft.² (max)

3. Treatment Unit Design

- 50 psi ASME unfired pressure vessel code design
- Rubber lined
- Access manway at top with platform, drain at bottom
- Lifting lugs
- Skid mounted
- Air bleed vent/vacuum break
- Internal header and lateral collection pipe system
- Distribution baffle
- Inlet and outlet flanges
- 4. Piping System Design
 - Liquid velocities (except backwash) 10 ft./sec. (max)
 - Liquid velocity backwash 13 ft./sec. (max)
 - Material suitable for dilute acid and dilute caustic service
 - Manual valves butterfly, gate
 - Measure flow rate into unit, total flow into unit
 - Sampling system to monitor pH and Fluoride
 - Control treatment system flow rate
 - Control treatment system pressure

C. Quantity and Quality of Treated Water for Distribution

- 1. Raw water flow rate- 1000 gpm (max). Treatment unit design. Flow rate 300 gpm each, or 600 gpm.
- Treated water total flow 97% raw water flow -840,000 GPD.
- 3. Waste water 3% raw water flow 26,000 GPD.
- 4. Measure fluoride content of treated water.

- 5. Measure pH level of treated water. Include high and low pH alarms interlocked with raw water pump power supply; thereby plant will sound alarm and will shut down in the event of high or low pH. Include provision to flush system to waste in the event of high or low pH shut down. Manual startup only after shutdown.
- 6. Incorporate provisions to lower raw water pH and raise treated water pH.
- D. Regeneration and Neutralization
 - 1. Regeneration material 1% NaOH
 - Blend of 50% NaOH and softened raw water in "mixing T" at treatment unit
 - 50% NaOH procured directly from caustic manufacturer, delivered to plant in tank trucks
 - Provide 6000 gallon insulated caustic storage tank (horizontal) with manway, immersion heater (incl. controls) to maintain 70° F min., lifting lugs, vent w/overflow, fill piping, drain piping, level measurement capability and outlets to service.
 - 2. Regeneration process
 - Flow rate through treatment unit 2-1/2 gpm/ft.² (max)
 - Residence time in treatment bed 40 minutes (min)
 - Incorporate provision for upflow or downflow through bed
 - 3. Regeneration Equipment
 - Chemical pump Metering pumps with precision flow control. Corrosion resistant material - Interlock pump controls with well pump power supply so that chemical pump can't operate when pump is off. Pump mounted upon steel stand above Day Tank. Day Tank filled via gravity from Caustic Storage
 - Plug valves, check valves, pressure relief valves and back-pressure valves as required for total operational control and good maintenance.
 - Provide emergency shower and eyewash

4. Neutralization material - 0.04% H₂SO₄

- Blend of 93% H₂SO₄ and softened water in "mixing T" at treatment unit
- 93% H₂SO₄ procured directly from acid manufacturer, delivered to plant in tank trucks
- Provide 6000 gallon acid storage horizonizontal tank with manway, lifting lugs, vent w/overflow, fill piping, drain piping, level measurement capability and outlet to service
- 5. Neutralization process
 - Flow rate through treatment unit 7 gpm/ft.² (max)
 - Amount of acid rinse required sufficient to adjust pH within acceptable pH limits 6.5 - 8.5
 - Incorporate provision for upflow or downflow through bed
- 6. Neutralization equipment
 - Chemical pumps 2 Metering pumps with precision flow control. Corrosion resistant material. Interlock pump controls with well pump supply so that chemical pumps cannot operate when well pump is off. Pumps mounted upon steel stand above Day Tank. Day Tank filled via gravity from Acid Storage Tank.
 - Plug valves, check valves, pressure relief valves and back-pressure valves as required for total operational control and good maintenance.
- 7. Zeolite softener complete with brine tank and automatic regeneration. Size softener to remove hardness ions from 300 gallons (min) of raw water per cubic feet of activated alumina in individual treatment unit. Hardness level during pilot testing was 30 grains per gallon. Include manual isolation valves to employ softened water only during regeneration.
- E. Treatment Building
 - Provide facility to house Treatment Units, Chemical Handling Systems, and Sampling System.
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Abide by Applicable Portions of the Following Codes and Standards

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- 6. National Sanitation Foundation Standards
- 7. Manufacturing Chemists Association
 - a. Chemical Safety Data Sheet SD-9 Caustic Soda
 - b. Chemical Safety Data Sheet SD-20 Sulfuric Acid
- 8. OSHA
- 9. Uniform Building Code

CONCLUSIONS

The fluoride removal pilot test runs have demonstrated that a full scale fluoride removal water treatment plant with raw water pH adjustment, can reliably remove 1700 grains of fluoride per cubic foot of activated alumina. Operating treatment cost projection for the full scale plant is 15¢/1000 gallons. Capital cost estimate for the 1000 gpm treatment system is \$270,000.

Interfering ions present in the raw water are competing fluoride for adsorption sites on the granular activated alumina surfaces. The interfering ions have been identified as T.O.C. Although the T.O.C. is easily removed during regeneration, its presence does reduce the fluoride removal capacity of the activated alumina to the above mentioned 1700 grains per cubic foot.

The use of softened raw water during regeneration and neutralization has increased the efficiency of the regeneration process.

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