Rocky Mountain Arsenal
Information Center
Commerce City, Colorado

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29 SEP 1965

U.S.A.R.M.:

SUBJECT: Report of Industrial Waste Survey, Rocky Mountain Arsenal,
Denver, Colorado

TO:

The Surgeon General

ATTN: PUB-PO

Department of the Army

Washington, D.C. 20315

Commanding General

US Army Materiel Command

ATTN: ACPT-11

Washington, D.C. 20315

Inclusive herewith are nine copies of the report of subject study,

performed by Captain Lefroy H. Reuter, MC and 1st Lieutenant William F. Milbury,
MC, this Army, during the period 9-21 February 1965.

RICHARD J. PHILLIPS

1st Colonel, MC

Commanding

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original document which is located in Central Files, TRAP.

VIRGINIA RAY

VIRGINIA RAY, Sec Mgt Ofc, Custodian

Carol Filer

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4. TITLE AND SUBTITLE
   REPORT OF INDUSTRIAL WASTE SURVEY, PROJECT NUMBER 3471E19-59/66, ROCKY
   MOUNTAIN ARSENAL, DENVER, COLORADO

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)
   ARMY ENVIRONMENTAL HYGIENE AGENCY
   ABERDEEN PROVING GROUND, MD

13. ABSTRACT (Maximum 200 words)
   THIS SURVEY WAS CONDUCTED TO DETERMINE THE PHYSICAL AND CHEMICAL
   CHARACTERISTICS OF LIQUID WASTES ENTERING THE INDUSTRIAL WASTE
   DISPOSAL SYSTEM AT RMA FOR A 12 DAY PERIOD. THE SURVEY WAS ALSO
   PERFORMED TO EVALUATE THE EFFECTIVENESS OF TREATMENT OF THE
   INDUSTRIAL WASTE AND TO DOCUMENT THE CHEMICAL NATURE OF THE WASTE
   INJECTED INTO THE DEEP WELL DISPOSAL SYSTEM.

14. SUBJECT TERMS
   PESTICIDE PRODUCTION, CHEMICAL ANALYSIS
CLASS II ACTIVITY OF THE SURGEON GENERAL

SERVING THE ARMY IN ITS PREVENTIVE MEDICINE PROGRAM

REPORT OF INDUSTRIAL WASTE SURVEY
PROJECT NO. 3471E19-59/66
ROCKY MOUNTAIN ARSENAL
DENVER, COLORADO
9-21 FEBRUARY 1965
HEADQUARTERS
U.S. ARMY ENVIRONMENTAL HYGIENE AGENCY
UNITED STATES ARMY MEDICAL SERVICE

CLASS II ACTIVITY
OF
THE SUBCOM GENERAL

EDGEMOOD ARSENAL
MARYLAND 21010

22 SEP 1963

REPORT OF INDUSTRIAL WASTE SURVEY
PROJECT NO. 3471E19-59/66
ROCKY MOUNTAIN ARSENAL
DENVER, COLORADO
9-21 FEBRUARY 1965.
ABSTRACT

Sanitary Engineering Division personnel conducted the survey to determine the physical and chemical characteristics of liquid wastes entering the industrial waste disposal system at Rocky Mountain Arsenal for a twelve-day period. The survey was also performed to evaluate the effectiveness of treatment of the industrial waste and to document the chemical nature of the waste injected into the deep well disposal system.

The average flow of liquid wastes into the industrial waste system was 266.6 gallons per minute. Less than one sixth of the waste flow was injected down the deep well and the remainder cumulated to the existing volume of Lake F. This average influent flow was more than double that recorded in a survey conducted in 1960 by this Agency. A comparison of chemical properties indicate that the current waste is much more dilute than that of 1960. The chemical results also showed the current inlet stream exhibits wide variations in properties which are greatly stabilized by the treatment system, which consists of a one million-gallon sedimentation lake and pressure filtration. Pesticide analyses indicated significant concentrations throughout the system, with the current treatment methods reducing the incoming pesticides concentration by one-half before deep well injections.

The disposal of all currently produced industrial wastes through the deep well system or some other adequate treatment system is an essential step in the elimination of Lake F. Greater effectiveness of the sedimentation lake can be obtained by relocation of the inlet and elimination of stagnant zones.
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REPORT OF INDUSTRIAL WASTE SURVEY
PROJECT NO. 3471E19-59/66
ROCKY MOUNTAIN ARSENAL
DENVER, COLORADO
9-21 FEBRUARY 1965

1. AUTHORITY.
   a. Para 4-6b(3)(d), AR 40-5, dated 10 April 1964.

2. REFERENCES.
   d. US Army Chemical Corps, "Staff Study Ground Water Contamination, Rocky Mountain Arsenal, 18 February 1960".

3. OBJECTIVES. The objectives of this study were to:
   a. Determine the quantities and characteristics of the waterborne industrial waste generated by all activities at Rocky Mountain Arsenal.
b. Evaluate the existing waste disposal facilities.

4. BACKGROUND.

a. General Background. Rocky Mountain Arsenal is located in the northeast section of Denver, Colorado. Original construction was completed in 1943 and the facilities were operated at full production during World War II producing Lewisite, mustard, arsenous chloride, and chlorine gas and filling munitions with white phosphorus. Industrial wastes were stored in a number of clay bottom evaporation lakes. The east plant facilities were leased in 1947 by Julius Hyman Company and used for the production of insecticides. In 1952, the Shell Chemical Company took over the operation of the insecticide facilities. The GB Plant was constructed during 1952 and production continued from 1953 to 1956. Filling of munitions with CW-agents, the decontamination of mustard-filled munitions and the blending of hydrazine have been the principle military operations contributing industrial waste since 1956. Figure 1 shows the physical arrangements of Rocky Mountain Arsenal industrial facilities.

b. Crop damage in the area adjacent to and northeast of Rocky Mountain Arsenal was first noticed in 1951 and the damage was associated with the use of the shallow well irrigation water. The US Corps of Engineers concluded in a 1955 report that the Arsenal waste lakes were the main source of contamination of these shallow wells.

c. In a report by the Ralph M. Parsons Engineering Company in September 1955 it was recommended that sealed evaporation-storage lakes be provided and an acid treatment plant be built. Acid and salt were to be reclaimed and sold, and the remaining waste was to be retained and evaporated in the sealed lakes.

d. Waste disposal remained unchanged, however, until October 1955 when complete retention of the wastes produced was initiated to eliminate additional ground water contamination. Lake F, the largest of the evaporation-storage lakes (96 acres and approximately 200 million gallons at full capacity) was sealed with an asphalt membrane. For additional protection, the membrane was covered with approximately 12" of compacted soil. Sealing Lake F was only an interim measure since the continuing waste flow exceeded the anticipated losses through solar evaporation.

FIGURE 1
INDUSTRIAL WASTE COLLECTION SYSTEM, ROCKY Mt. ARSENAL
Deep well injection of the waste was concluded to be practicable if the wastes were injected in a low suspended solids condition. Dilution of the waste with surface water was concluded to be unacceptable, because of non-availability and water reuse policies.

f. This Agency performed an industrial waste survey in June 1960 to provide information on the sources, volume of flow, and composition of the waste for use in the evaluation of waste treatment methods (para 2a).

g. E. A. Polumbus, Jr., and Associates, working as Consulting Engineers for the US Army, concluded in a January 1960 report that a geological investigation indicated that with a reasonable degree of certainty, 800 gallons per minute (gpm) of waste water could be disposed of in a deep well drilled on Arsenal property. It was estimated that the cost of drilling the deep well to an 11,400' proposed depth, plus all necessary facilities for treating and injecting the waste at a rate of 800 gpm would be approximately $1,000,000. This recommended solution was ultimately accepted and a deep injection well designed to handle 800 gpm at a surface pressure of 2,500 pounds per square inch (psi) was drilled to a depth of 12,075 feet. Surface pretreatment facilities included a clarifier and asbestos-diatomaceous earth coated pressure filters designed to reduce suspended solids concentration to 20 milligrams per liter (mg/l) or less. The system was placed in operation early in 1962.

h. After a few months of operation, removal of suspended solids from the Lake F waste became very expensive as coagulation became more difficult and filter runs shorter. Most of the difficulties were attributed to an unfilterable bacterial slime. The use of the deep well was discontinued in mid-1963 because of these high pretreatment costs. To offset the increasing waste volume in Lake F, a spray evaporation raft was placed in operation. The combined evaporation rate from the surface of Lake F and the spray system reportedly average approximately 140 gpm during the eight months of the year when climatic conditions are most favorable for operation.

i. Plant Processes.

(1) Shell Chemical Company Operations. The Shell Chemical Company has manufactured the following pesticides at the Arsenal: Aldrin, azodrin, bidrin, ciodrin, dibrom, dieldrin, endrin, parathion, methyl parathion, vapon, phosphrin, nemagon C and Compound 4072. Azidrin, bidrin, ciodrin, phosdrin and Compound 4072 were not produced during this survey and Shell anticipates reducing the production of aldrin, dieldrin and endrin.
(a) Aldrin. Aldrin, produced in Building 422, is a highly chlorinated cyclic hydrocarbon produced by the Diels-Alder diene reaction. Production involves peroxide oxidation in agitated, jacketed kettles. Waste was primarily wash water combined with a CaCl₂ brine solution and organic solvents. The brine was derived from dryers at an estimated rate of 2 gpm. Additional wastes include diatomaceous earth from filters which is diluted with water and trucked out to Lake F for disposal.

(b) Dieldrin. Dieldrin, produced in Building 516, is an epoxide of aldrin formed by reacting peracetic acid with aldrin. Manufactured by a batch operation, the primary wastes were acetic acid, wash water and hydrogen peroxide. Estimated quantity of waste was 500 gallons per batch of highly concentrated acetic acid and 1 to 2 gpm from a continuous centrifuge.

(c) Endrin. Isodrin, an isomer of aldrin, is converted to endrin by reaction with peracetic acid in Buildings 515 and 516. Wastes included acetic acid, sodium chloride, suspended carbon, and calcium chloride from dryers.

(d) Methyl Parathion and Parathion. Production facilities for these products are located in Building 514. Both are made in batches from p-nitro sodium phenolate (PNSP) and dimethyl phosphoro chloro-iodophosphate. The wastes which are discharged to the sewer after hydrolysis are p-nitro phenol and diethyl ortho-thiophosphonic acid plus excess caustic. The estimated waste quantity which requires hydrolyzation is about 1000 gallons per batch.

(e) Vapona, Nemagon C and Dibrom. All three products are manufactured in Building 471. Vapona is the addition product of chloral (Trichloro aldehyde) and trimethyl phosphate. Before the reaction chloral is treated in concentrated H₂SO₄ to remove polymers, producing an acid waste stream. The estimated waste flow is approximately 400 pounds of concentrated H₂SO₄ and 400 pounds of chlorinated hydrocarbon per batch of Vapona plus an additional dilute waste flow of about 50 gpm from steam jets. Dibrom is made by the addition of bromine to the unsaturated bond of Vapona. A steam jet with a 48 gpm discharge is used for this production. Nemagon C, a soil fumigant, is the addition product of bromine and allyl chloride, which produces 15 gpm dilute waste flow from a condensor jet. In addition to the above waste flows, this system also produces 60 gpm flow from the hot well dilution condensor. These condensor jets would not be expected to be in operation more than an average of 8 hours per day.
(2) Rocky Mountain Arsenal Operations. The industrial waste producing facilities of the Arsenal are located in all of the installation plants areas.

(a) Mustard Demilitarization. The demilitaring facilities are located in Building 538 of the east plants area. A waste stream is derived from cooling water used on the incinerated bombs. During the study period, the facilities were used to demilitarize 155 mm shells which were air dried so that only a very dilute waste was contributed to the industrial waste sewer.

(b) GB Plant. The GB Plant facilities for manufacturing and filling of munitions are located as indicated in the general layout sketch (Figure 1). The manufacturing facilities were not in operation during the study period; however, GB was being redistilled during the survey and munitions were being filled with GB and Vx. Waste consisted primarily of the hydrolysis products of the CW agents, caustic solution and wash down. Spray paint activities are also included in Building 1601 in the GB Plant area; however, the water used for the screen in the booths is recirculated and waste flow is negligible. Total average waste flow from the GB plant area was approximately 3 gpm.

(c) Hydrazine Blending Plant. Hydrazine blending facilities are located in Building area 755. Waste is produced from scrubbers for vapors produced during the blending operation. Approximately average flow is 3 gpm. Waste flow is into a sump with gravity flow into the industrial waste line.

(d) Laundry. Laundry facilities located in Building 314 generate a waste composed of wash water, detergents and dyes. Average waste flow from the laundry during the survey was approximately 3000 gallons per day.

(e) Quality Survey Lab. Small quantities of laboratory wastes are generated from this activity which is located in Building 313. Control analyses are performed on GB and other CW agents which produce small quantities of waste which goes into the industrial sewer. Rocky Mountain Arsenal officials estimate this industrial waste flow to be 10,000 gallons per month.

j. Industrial Waste Disposal Facilities.

(1) General. All industrial waste water flows into Lake F-1, a "diked off" portion of Lake F. Shell's parathion wastes are hydrolyzed...
before discharge into the sewer lines but all other wastes flow from
their point of generation without treatment into a trunkline which flows
approximately two miles by gravity to Lake F-1. Part of the waste from
Lake F-1 overflows into Lake F while the remainder is discharged down
the deep well. Waste discharged down the well flows from either Lake F
or F-1 through a clarifier to a wet well, is pumped through pressure
filters to a storage tank and then down the deep well. A dry chemical
feeder and flocculator, although available, have not been in use as
stated in para 4b. No waste from Lake F has been injected down the deep
well since 1963.

(2) Shell Chemical Company's Waste Hydrolysis Tank. The
treatment procedure used for parathion wastes consists of main-
taining a 140°F temperature for approximately 16 hours with a caustic
supply in excess of at least 10 times the stoichiometric requirement.
Mixing is achieved by injecting 20 percent NaOH into the suction side
of a circulating pump which discharges into the hydrolysis tank.

(3) GB Plant Sump. All industrial wastes from the GB plant
flow into a collecting sump equipped with a float-activated pump having a
reported maximum capacity of approximately 200 gpm at the float activated
head level which pumps waste into a sewer line leading to Lake F-1. Within
the plant, detoxification tanks are provided in which rejected batches of
agent may be dumped, and detoxified with Cl₂ and NaOH before being dumped
into the contaminated sump.

(4) Lake F-1. Lake F-1, which has an approximately capacity
of one million gallons, receives the total industrial waste flow. A
parshall flume located on the effluent channel provides flow measurement.
Lake F-1 is divided approximately into two equal volumes (see Figure 2)
by an old dirt road which serves as a dike. The overflow, allowing waste
to flow from F-1 to F, is located at the canal neck connecting the two
sections of the lake. Lake F-1 is lined with an asphalt membrane covered
with approximately 12 inches of compacted soil.

(5) Dry Chemical Feeder and Flocculator. A dry chemical
vibrating type feeder with rated capacity from 0 to 300 lbs/day is available
but has not been used. The unit has a dissolving chamber, mechanical
mixer, and 4½ gpm at 16' head injection pump.

(6) Clarifier. The circular unit is 38' in diameter and
depth to the notched weir is 9 feet. The clarifier is equipped with a
rake type sludge collector, influent diffuser, and surface scum skimmer.
Influent is fed into the center of the unit. This EDMCO-Process
Equipment was designed for an overflow rate of 500 gals/ft²/day.
FIGURE 2
INDUSTRIAL WASTE LAKES, ROCKY MT. ARSENAL
Filters. Two pressure filter units manufactured by United States Filter Company are operated in parallel. They are designed to remove suspended solids by means of an asbestos filter-diatomaceous filter coat from 400 gpm waste flow under 65 psi working pressure. The filters are cylindrical, multiple screen type with center discharge and bottom sludge removal. The filters are cylindrical, multiple screen type with center discharge and bottom sludge removal. The filter leaves are made up of five layers of screening. The screen area per filter is approximately 400 square feet and can be increased to 500 square feet. The filter leaves are cleaned by high pressure water jets.

Storage Tank. Filtered waste is pumped to a 50,000 gallon capacity circular storage tank, 24' in diameter and 15' high.

Deep Well. The deep well was designed to accommodate a waste volume of from 400 to 800 gpm at 2500 psi surface injection pressure. To effectively isolate the injection reservoir a 20" casing was set at 135 feet; a 13-3/8" casing at 2020 feet, an 8-5/8" casing at 11,171 feet and a 5-1/2" liner placed from 1,007 to 11,975 feet. To further insure complete isolation, each string of casing was sheathed with cement for its entire length. The annual volume between the 5-1/2" injection tubing and the 8-5/8" casing is filled with fresh water and a pressure sensing and recording device at the well head is activated if any leaks occur from the closed chamber. The geological formations and the depths at which they were encountered are:

<table>
<thead>
<tr>
<th>Geological Formation</th>
<th>Depth to Top of Formation (ft)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fox Hills</td>
<td>1,250</td>
</tr>
<tr>
<td>Pierre Shale</td>
<td>1,480</td>
</tr>
<tr>
<td>Hygiene Zone</td>
<td>5,448</td>
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<tr>
<td>Niobrara</td>
<td>7,710</td>
</tr>
<tr>
<td>Carlile Shale</td>
<td>8,078</td>
</tr>
<tr>
<td>Greenhorn Limestone</td>
<td>8,120</td>
</tr>
<tr>
<td>Granerous Shale</td>
<td>8,345</td>
</tr>
<tr>
<td>&quot;J&quot; Sandstone</td>
<td>8,485</td>
</tr>
<tr>
<td>Dakota Sandstone</td>
<td>8,633</td>
</tr>
<tr>
<td>Lakota Sandstone</td>
<td>8,730</td>
</tr>
<tr>
<td>Morrison Formation</td>
<td>8,786</td>
</tr>
<tr>
<td>Lykins Formation</td>
<td>8,972</td>
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<tr>
<td>Lyons Formation</td>
<td>9,582</td>
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<tr>
<td>Fountain Formation</td>
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<tr>
<td>Regolith</td>
<td>11,880</td>
</tr>
<tr>
<td>Ordovician Cambrian</td>
<td>11,895</td>
</tr>
<tr>
<td>Pre-Cambrian Schist</td>
<td>11,950</td>
</tr>
<tr>
<td>Pre-Cambrian Gneiss</td>
<td>11,970</td>
</tr>
</tbody>
</table>
Although not in use during the survey, four .95 gpm TDH 4650' reciprocating Multiplex plunger pumps are available to inject waste down the deep well.

5. PROCEDURES. To obtain the required data, the US Army Environmental Hygiene Agency sent out a survey party which included two sanitary engineers and four enlisted technicians.

   a. Sample Collection. The sample period for the survey extended from 0700 hours, 9 February 1965, to 0700 hours, 20 February 1965. The sample day ran from 0700 hours one day to 0700 hours the following day.

   (1) Sample Point Locations. The locations of the sample points are shown in Figure 1.

   Sample Point 1 - Influent to Lake F-1
   Sample Point 2 - Effluent from Lake F-1
   Sample Point 3 - Effluent from pressure filters

   (2) Grab Samples.

       (a) Grab samples, 1000 ml volume, were taken at points 2 and 3 every two hours.

       (b) Flows at points 2 and 3 were measured at time of sampling from readings on the flow meter at the filters. Since the pressure and head on the filters were relatively constant, flow was considered uniform throughout any two hour period. The flow meter had been calibrated before the survey.

   (3) Composite Samples.

        (a) An automatic sampler was installed at point 1 and adjusted to collect a sample every 15 minutes. The sample volume from eight consecutive 15-minute interval periods was collected at each odd numbered hour of the sampling day and was retained. At the end of the sample day the two-hour samples were composited in proportion to flow at point 1. Portions of each composited daily sample were returned to USAEHA for additional analyses.

        (b) Flows at point 1 were measured by an existing Parshall Flume equipped with an automatic recording device.

        (c) Composite samples for points 2 and 3 were made by proportioning 350 ml volumes from each grab sample at these points. Portions of each daily composited sample were returned to USAEHA for additional analyses.
(4) Special Samples and Studies.

(a) Waste samples from the aldrin process still bottom were collected and returned to USAEHA for analysis.

(b) Samples from the ice cover on Lake F and Lake F-1 were collected and analyzed.

(c) Samples of bottom sludge from Lake F and F-1 were collected and returned to USAEHA for analysis.

(d) Tracer Studies. Congo red waste from the Rocky Mountain Arsenal laundry and a commercial fabric dye were used as tracers in determining the flow pattern in Lake F-1. In addition to visual observations, samples were manually collected periodically at five locations and analyzed for tracer concentration.

b. Methods of Analysis.

(1) Laboratory facilities in the treatment plant and in a section of the Quality Surety Laboratory at Rocky Mountain Arsenal were utilized. Where noted, certain samples were returned and analyses completed at USAEHA and USA Chemical Research and Development Laboratories (USACRDL).

(2) Grab Samples. Analyses were in accordance with the schedule shown in Table 1. The temperature readings were made in situ, while pH, specific conductance, acidity/alkalinity, and turbidity were measured in the treatment plant laboratory. Turbidity measurements were made with the Hellige turbidimeter calibrated with a standard SiO₂ solution.

(3) Composite Samples. Analyses were in accordance with the schedule shown in Table 1. All determinations were those as described in Standard Methods for the Examination of Water and Wastewater, Eleventh Edition, 1960, with the following exceptions:

(a) Fluoride - "Ion-Exchange Method for Determination of Fluorides in Potable Waters", Kelso, Toymae S., Mathews, John M. and Kramer, Harry P., Analytical Chemistry, Vol 36, 1964, p 577. This method was used because of its simplicity, reproducibility and freedom from interference.

(b) COD - "Elimination of Chloride Interference in the Chemical Oxygen Demand Test", Dobbs, R. A., and Williams, R. T., Analytical Chemistry, Vol 35, 1963, p 1064. This method was used to avoid high salt concentration interferences.
### TABLE 1

**SCHEDULE FOR ANALYSES**

<table>
<thead>
<tr>
<th>Analyses</th>
<th>Sample Point Number</th>
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<tbody>
<tr>
<td></td>
<td>Sample 1</td>
</tr>
<tr>
<td>Two-Hour Interval Samples</td>
<td></td>
</tr>
<tr>
<td>1. Acidity/Alkalinity</td>
<td>2H</td>
</tr>
<tr>
<td>2. pH</td>
<td>2H</td>
</tr>
<tr>
<td>3. Specific Conductivity</td>
<td>2H</td>
</tr>
<tr>
<td>4. Temperature</td>
<td>2H</td>
</tr>
<tr>
<td>5. Turbidity</td>
<td>2H</td>
</tr>
</tbody>
</table>

| 24-Hour Composite Samples|          |          |          |
|---------------------------|          |          |          |
| 1. Acidity/Alkalinity     | D         | D         | D         |
| 2. Aluminum               | D         | D         | D         |
| 3. Ammonia Nitrogen       | D         | D         | D         |
| 4. Arsenic                | D         | D         | D         |
| 5. Calcium                | D         | D         | D         |
| 6. Chemical Oxygen Demand | D         | D         | D         |
| 7. Chloride               | D         | D         | D         |
| 8. Fluorides              | D         | D         | D         |
| 9. Insecticides           | D         | D         | D         |
| 10. Iron                  | D         | D         | D         |
| 11. Kjeldahl Nitrogen     | D         | D         | D         |
| 12. Magnesium             | D         | D         | D         |
| 13. pH                    | D         | D         | D         |
| 14. Phenols               | D         | D         | D         |
| 15. Solids:               | D         | D         | D         |
| Dissolved                 | D         | D         | D         |
| Volatile                  | D         | D         | D         |
| Volatile Settleable       | D         | D         | D         |
| Total                     | D         | D         | D         |
| Total Volatile            | D         | D         | D         |
| 16. Specific Conductance  | D         | D         | D         |
| 17. Sulfates              | D         | D         | D         |
| 18. Sulfides              | D         | D         | D         |
| 19. Total Phosphorus      | D         | D         | D         |
| 20. Turbidity             | D         | D         | D         |

2H - Every two hours  
D - Daily
Iron - The standard tripyridine method was not reproducible because of the high phosphate concentration of the waste. The actual procedure used is described in Appendix I.

Arsenic - The Robert A. Taft Sanitary Engineering Center, Cincinnati, Ohio, silver diethylthiocarbamate method was used to provide a limit of sensitivity low enough for the anticipated concentrations. The procedure used is described in Appendix I.

Sulfide - "Sulfur by Evaluation as Hydrogen Sulfide", Snell, F. D., and Snell, C. T., *Colorimetric Methods of Analysis, Vol I*, Sixth Printing, 1945, p 598. This method was used because of its simplicity and the interferences to other applicable procedures.

Phosphorus - The phosphorus method is described in Appendix I and was used because it is less sensitive to interferences than the method in *Standard Methods for the Examination of Water and Wastewater*.

Special Analyses.

(a) Pesticide Analyses. Gas chromatography was used to measure the presence of the following pesticides: Aldrin, dieldrin, endrin, methyl parathion, parathion, nemagon C and vapona. These determinations were performed on selected daily composite samples from sample points 1, 2 and 3 and mud samples taken from the bottom of Lake F-1. Complete instrumental data for the gas chromatography unit used for these determinations is given in Appendix I. Each liquid sample was pretreated by extraction with spectrograde hexane in a liquid-liquid apparatus as described in *Analytical Chemistry, Vol 36*, pp 1340-3, June 1964. A one liter waste sample was extracted two times with 250 ml amounts of hexane for 3½ hours each. The two extractions were combined, dried with 2 gm of K₂SO₄ and concentrated to 30 ml by distillation prior to analysis by the gas chromatograph. Mud samples were extracted by taking 50 ml of the sample, diluting to 1 liter with distilled water and adding 100 ml of hexane. The two liquid layers were mixed together with a magnetic stirrer for one hour and then the hexane layer decanted. This procedure was repeated and the two hexane extracts were combined and then dried and concentrated in the same manner as in the pretreatment of the liquid waste samples.

(b) Spectrographic Analyses. Spectrographic methods were used for the analysis of calcium, aluminum and magnesium. Spectrographic plates were run in the 2300-3400 Angstrom range in duplicate for
each specimen. Calcium was evaluated by flame spectrophotometry to
serve as an internal standard. Aluminum and magnesium were read on the
film plates and negative log ratios were set up as absorbance ratios.
The ratios were then multiplied by plate factors and by the value taken
for the calcium standard. Plate factors were found by running six spectra
of mixtures of calcium, aluminum and magnesium salts at various volumes
to determine relative concentration responses on the electrodes, and then
averaging these responses to obtain factors for calcium:aluminum and
calcium:magnesium ratios.

6. FINDINGS.

a. Production Activities.

(1) Shell Chemical Company Pesticide Production. A list
of the production activities of Shell during the survey is shown in
Table 2. For each sample day the amount of pesticide made is listed
by number of batches and, as indicated, the production runs of certain
pesticides were discontinued during the survey. For the two weeks of
the survey, it is estimated that the Shell Company building complex consumed
a total of 94,000,000 gallons of water which is an average of 4,630 gpm.
(This is about 97 percent of water consumed on the Arsenal.) This
figure includes all water uses such as steam production, cooling, and other
process water.

(2) Other RMA Industrial Waste Producing Activities.
During the period of 8-21 February 1965, the following approximate waste
flow estimates have been provided by Rocky Mountain personnel. These
estimates are based on a five day per week operation.

<table>
<thead>
<tr>
<th>ACTIVITY</th>
<th>WASTE FLOW</th>
</tr>
</thead>
<tbody>
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<td>GB Plant</td>
<td>3 gpm</td>
</tr>
<tr>
<td>Mustard DeMiliterization</td>
<td>1.5 gpm</td>
</tr>
<tr>
<td>Hydrazine Blending</td>
<td>3 gpm</td>
</tr>
<tr>
<td>Laundry</td>
<td>2 gpm</td>
</tr>
<tr>
<td>Quality Surety Lab.</td>
<td>1 gpm</td>
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### TABLE 2

**PESTICIDE PRODUCTION**

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<th>Wed</th>
<th>Thur</th>
<th>Fri</th>
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</table>

**Product**

- **Aldrin**
- **Dieldrin**
- **Endrin**
- **Nemagon C**
- **Dibrom**
- **Vapona**
- **Parathion**
b. Industrial Waste Flow Rates.

(1) Flow into Lake F-1.

(a) Figure 3 presents graphically the two-hour average flows recorded at sampling point 1 for the twelve sampling days. A complete listing of flow data for this plot is tabulated in Appendix II.

(b) Table 3 lists the two-hour maximum, minimum, and daily average flow into Lake F-1. The overall average, 266.6 gpm, results in a total waste flow of 4.61 million gallons during the twelve-day survey. This is a substantial increase in flow over the value found in the 1960 USAEHA survey of 131 gpm average for the period 9-22 June 1960.

### TABLE 3
AVERAGE FLOWS INTO LAKE F-1

<table>
<thead>
<tr>
<th>Sample Day No.</th>
<th>Day of Week</th>
<th>Min 2 hr-Avg (gpm)</th>
<th>Max 2 hr-Avg (gpm)</th>
<th>Daily Avg (gpm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Tuesday</td>
<td>202*</td>
<td>242</td>
<td>219</td>
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<tr>
<td>2</td>
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<td>215</td>
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<td>3</td>
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<td>12</td>
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<td>247</td>
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Average Daily Average 266.6

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**Minimum 2 hour-flow recorded during survey**

**Maximum 2 hour-flow recorded during survey**

(2) Rate of Injection into Deep Well.

(a) A plot of the two hour average flow is shown in Figure 4. Changes in filter operating conditions are also indicated in Figure 4 at the appropriate times. One filter was on line at a time for
FIGURE 3  INDUSTRIAL WASTE FLOW CHART
ROCKY MT. ARSENAL, 0700 9 FEB TO 0700 21 FEB 65
UPPER CURVE: FLOW INTO LAKE F-1
OVERALL AVERAGE = 266.0 gpm
LOWER CURVE: FLOW DOWN DEEP WELL
OVERALL AVERAGE = 41.5 gpm
NOTE: AVERAGE FLOW TO LAKE F = 225.1 gpm
KEY: — TWO HOUR AVERAGE FLOW
—— SAMPLE DAY AVERAGE FLOW (0700 TO 0700)

TIME — DAYS (TWO HOUR SUB-UNITS)
Figure 4: Record of Industrial Waste Flow Down Well

Key:
- Two Hour Average Flow
- Sample Day Average Flow
runs of approximately 2.5 days. During each run, filter cakes were
patched when analysis indicated leaks. The down time for switching or
patching filters corresponds to the lower average injection rates.

(b) Table 4 lists the 2 hour maximum, minimum and
average daily flow down the deep well. The overall average rate of
41.5 gpm indicates 0.717 million gallons of industrial waste were
injected down the deep well for the twelve day survey period. This is
15.5 percent of the amount of industrial waste produced during the
period.

TABLE 4

AVERAGE FLOWS DOWN INJECTION WELL

<table>
<thead>
<tr>
<th>Sample Day No.</th>
<th>Min 2 hr-Avg (gpm)</th>
<th>Max 2 hr-Avg (gpm)</th>
<th>Daily Avg (gpm)</th>
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AVERAGE DAILY AVG 41.5

*Maximum 2 hour flow recorded during survey
**Minimum 2 hour flow recorded during survey

c. Standard (On Site) Chemical Analyses.

(1) Two Hour Grab Samples. Figure 5 shows plots of the results
of the analyses of grab samples from sample point 1. Waste properties that
were measured are: Temperature, pH, turbidity, alkalinity and conductivity.
The conductivity sensing electrodes became inoperative on sample day 9,
terminating the conductivity results. Figure 6 graphically presents the
results of analyses of grab samples from sample points 2 and 3. The
analyses reported are the same as those in the previous figure.
<table>
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<th>DATE</th>
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<th>ALKALINITY mg/l CaCO₃</th>
<th>TURBIDITY mg/l SiO₂</th>
<th>pH</th>
<th>TEMPERATURE °F</th>
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**LEGEND**

- Sample Point 2
- Sample Point 3
(2) Daily Composite Results. A summary of the analyses performed on the composite samples is presented in Table 5. The average, standard deviation, maximum and minimum of the twelve day sampling period are tabulated for all three sampling points. Complete analytical data for composite samples is presented in Appendix II.

(3) Precision Tests. A test of the reproducibility of analytical methods was performed on eight of the standard chemical determinations. The results are presented in Table 6. Eleven consecutive analyses were performed on the same sample by each method. The laboratory work was done in the USAEHA laboratory at Edgewood Arsenal and completed on 16 April 1965.

d. Special Analyses.

(1) Pesticides. A summary of the results of pesticide analyses are shown in Table 7. The average, minimum and maximum for each sample point and analysis are indicated. From sample point 1, ten of the composite samples were tested and from sample points 2 and 3, only three composite samples were tested (sample days 4, 7 and 11) to make up the averages. As indicated, no detectable amounts of Milbrom and nemagon C were found in the samples. Complete data on individual pesticide analyses results are shown in Appendix II.

(2) Also included in Table 7 are the results of a grab sample taken from Lake F at the end of the pumphouse pier. Although they are the results from a single determination, they indicate significant pesticide concentrations in the liquid of Lake F.

(3) Solid Samples. Bottom deposit samples were taken at two locations, sample points A and B (as indicated in Figure 7), and analyzed for pesticide concentrations. The results are shown in Table 8 and given as grams of pesticide per liter volume of mud. Also results are shown of the analysis of the still bottoms and production filter cake waste which the Shell Company dumps into Lake F along the east bank.

(4) General Observations.

(a) Visual observation of the flow patterns of the congo red dye used by the laundry indicated a considerable amount of short circuiting from the inlet of Lake F-1 to the overflow into Lake F, as indicated in Figure 7. Flow to the treatment building inlet proceeded across sample point A (Figure 7) directly along the north side of the Lake F-1. At sample point B (Figure 7), the lake appeared stagnant. These observations were qualitatively confirmed by observation of rate of
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**TABLE 7**

**SUMMARY OF PESTICIDE ANALYSIS RESULTS**

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<td>Max</td>
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<td>Min</td>
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<td>Min</td>
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<td>Aldrin</td>
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<td>None Detected</td>
<td>None Detected</td>
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surface ice formation and melting. Approximate estimates of the Lake F-1 effective detention time were four days as compared to a calculated theoretical detention time of 9.8 days. These figures are based on the average flow into Lake F-1 and average overflow into Lake F.

(b) General observations were also made of the effect that surface storage of toxic industrial waste in Lakes F and F-1 has on the environment. Many sightings were made of the toxic effect on naturally occurring wild life. Along the banks of Lake F-1 and Lake F were found remains of dead ducks, rabbits, birds and mice.

7. DISCUSSION.

a. Production Activities.

(1) Shell Company Pesticide Production.

(a) Table 2, which lists the pesticide production by batches, indicates that three pesticide runs were terminated during the survey. Although the runs of individual pesticide may have been discontinued on a given day, residuals of the pesticide may continue to flow into the industrial waste stream for several more days because of washdown and clean-up procedures. The survey was conducted during the period 8-21 February 1965 because the large variety and amount of pesticide production scheduled during this period represented the maximum production activity of Shell for any two week period during the year. Table 2 also indicates that although Shell operates seven days a week, their production on weekends is greatly reduced.

(b) Over 90 percent of the water used, excluding water for power production, is consumed by Shell's industrial operations. All liquid production wastes except uncontaminated cooling water of Shell are discharged to the industrial waste disposal system.

(2) Other Rocky Mt. Ars Industrial Waste-Producing Activities. Since physical conditions render it impossible to meter the flow from individual activities into the industrial waste disposal system, only estimates can be made of these flows. The GB plant was not producing agent during the survey and its only activities were filling projectiles and some agent redistillation. The estimated flow reported was provided by the operating staff at the GB plant. The mustard demiling activity was operating, but there was no waste flow since the shells were air cooled. Other flow estimates listed are approximate but indicate a level of magnitude.

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b. Industrial Waste Flow Rates.

(1) Flow into Lake F-1.

(a) The two-hour average flows illustrated in Figure 3 point out considerable variation indicating many "slug" discharges. Within a single day as much as 60 percent variation in flow was observed with the highest flows occurring during the daylight hours of week days. On sample days 4 and 10, the peak discharges, occurring between 1500 and 1700 hours, appear to be related to high flows from the EB plant.

(b) The total industrial waste flow rate was over two times that found in the 1960 survey by this Agency. This increase is primarily due to Shell's increased production, alterations in the cooling system and the discharge of condenser jets to the industrial waste sewer.

(c) The Parshall Flume used to measure the flows produced reliable results. The manufacturer's specification state an accuracy of 1 ± 3 percent. The continuous depth recorder was manually recalibrated every twenty-four hours.

(2) Rate of Injection down Deep Well. The variations in the expanded plot of average injection flows shown in Figure 4 correspond directly to alterations in filter operation. Whenever there was down time for filter repair or change-over, the average flow was reduced. Also Figure 4 shows that for a given filter, the longer the running time the lower the flow rate. A comparison between the flow down the deep well and the total industrial waste flow is shown in Figure 3. With about 85 percent of the waste flowing into Lake F, this clearly emphasizes that increased injection rates or decreased incoming industrial flows are necessary if the accumulation in Lake F is to be stopped.

c. Standard Chemical Analyses.

(1) Two-hour Grab Samples and Composites.

(a) In general, the physical and chemical analytical results of the inlet stream into Lake F-1 shown in Figure 5 showed definite stabilization on weekends. The increased variation during week days emphasizes the batch nature of the discharges and the increased production activity. Figure 5 shows the pH as significantly stabilizing on sample day 5. The periodic increases of pH on days 7 through 11 correspond to periods of high flow shown on Figure 3. It can be noted that the alkalinity very closely follows the pH.
(b) The dampening effects of Lake F-1 on industrial waste characteristics can clearly be seen by comparing the results from sample point 1 in Figure 5 with those from sample points 2 and 3 shown in Figure 6. Since the only difference between sample points 2 and 3 is passage through a diatomaceous filter, all properties shown in Figure 6 other than turbidity closely follow one another. Differences in temperature reflect filter alterations and changes. The pH levels indicate about a five day lag between sample points 1 and 2. Average turbidity removal across the filter was about 25 mg/l and all of the waste going down the deep well and a turbidity of >20 mg/l.

(2) Daily Composite Results.

(a) Acidity and Alkalinity. Only maxima and minima are reported in Table 5 because of the characteristics of these analyses. Results from sample point 1 are quite variable, but those from sample points 2 and 3 are more uniform.

(b) Arsenic. Results were reasonably consistent and similar at all three sample points. The values are in the range of the background level in the water supply system. At sample point 1 the results agree closely with the findings in this Agency's 1960 survey.

(c) Chemical Oxygen Demand (COD). From Table 5 the lower standard deviations of COD results from sample points 2 and 3 clearly indicate the stabilizing effect of Lake F-1. Data from sample point 1 showed significantly lower values on weekends when compared with week day figures. Comparison of this data with the results from the 1960 USAEHA survey show the present waste to contain a much lower concentration of organics.

(d) Chlorides. Chloride values from sample points 2 and 3 were higher than those from sample point 1 but they showed a general downward trend during the survey. This could be accounted for if there had been previous high concentrations of chlorides in the waste stream just before the survey started. Again, comparison between chloride results of this and the previous survey indicate that the waste stream was about four times as concentrated in 1960.

(e) Fluorides. Although the average results from all three sample points were similar, the tenfold decrease in magnitude of the standard deviations for sample points 2 and 3 indicate a much greater consistency in values at these points. The maximum value at sample point 1 of 40 mg/l on the tenth day appear to be related to a slug of waste pumped from the GB plant sump. These results indicate that there is much more fluoride in the waste stream than when the 1960 survey was performed. This can be attributed to agent redistillation during the survey.
(f) Iron. The values of iron concentration found at sample point 1 were much higher than those at sample points 2 and 3. This removal could occur by precipitation or sedimentation. Also the inlet waste iron values were much lower on weekends. The iron concentration in the 1960 survey was about seven times higher.

(g) Ammonia Nitrogen. The procedure used in this determination was quite variable and appeared to measure not only ammonia but also all volatile organic bases present. The very high values at sample point 1 occurred on the last two days of the survey.

(h) Kjeldahl Nitrogen. Within the variability of the analytical method used all three sample points showed similar results. Data show a definite upward trend in the latter half of the survey.

(i) Total Phosphorus. Average results for sample point 1 were substantially lower than those at sample points 2 and 3. The latter two sample points produced results showing a definite downward trend which would account for this difference. Sample point 1 showed greatly reduced concentrations on weekends. The average level found in the 1960 survey was much lower than the current survey. This can be accounted for by increased production of organic phosphorus pesticides.

(j) Phenols. Concentrations found at sample point 1 were much more variable than those at sample points 2 and 3, which were very similar. All positions exhibited a slight downward trend after sample day 4. Again sample point 1 results showed a definite decrease during weekends. The 1960 survey phenol results were just slightly higher than those discussed above.

(k) pH. The pH results from the daily composites are very similar to those reported earlier for the two-hour grab samples. The data clearly show the dampening effect of Lake F-1 and that sample points 2 and 3 exhibit a lag time of about four days behind changes at sample point 1. The average pH of the waste in 1960 was very close to that found during this survey.

(l) Specific Conductance. Only a limited amount of data were obtained because the instrument became inoperative during the survey. However, the values obtained were much higher than those found in 1960.

(m) Sulfates. As seen by a comparison of the standard deviations among the three sample points on Table 5, the dampening effect of the system is clearly observed. Values for sample point 1 were much
lower on weekends. Also, the levels of sulfates are about double that found in the 1960 survey. Vapona production can be related to the increase in sulfates.

(o) Sulfides. The method used to measure sulfides was not very precise. However, results indicate a significant increase across Lake F-1. This may be due to the presence of some sulfide producing action.

(o) Solids. Results presented in Table 5 indicate that most of the solids present in the waste are in dissolved form. The total solids appeared to increase across Lake F-1 while the suspended solids were greatly reduced by sedimentation. Because of the cold weather and ice cover, little evaporation occurred to concentrate the dissolved solids. The solids results from the 1960 survey indicate that at the time the waste was about five times more concentrated than it was during this survey. Because there are many volatile organics present in this waste, it is important to point out that solid results will not include these substances.

(p) Turbidity. These results are similar to those from the two hour grab samples and show the effectiveness of sedimentation in Lake F-1 and pressure filtration in producing an effluent of an average turbidity of about 4 mg/1 for injection down the deep well. Strong winds increase the turbidity level at sample point 2.

(q) Calcium, Magnesium and Aluminum. Flame spectrophotometry showed the calcium content in the daily composite to run about 300 mg/1 at all sample points so this value was used as an internal standard for magnesium and aluminum determinations. Average results from sample points 1 and 2 for magnesium and aluminum are very similar and establish the concentration levels present. The reason for the higher values reported for sample point 3 is related to the use of calcium as an internal standard and its obvious increase across a diatomaceous filter.

(3) Precision Tests. The precision tests of some of the standard chemical analytical methods are shown in Table 6. A quantity of waste from sample point 2 was used for these tests but it was five weeks after sample collection before these tests were completed. Significant visual change had taken place in the sample during that time. Due to the complex nature of this waste and interferences present, more variability was found than anticipated. However, these results do give an indication of the reproducibility of the analytical methods employed.

d. Special Analyses.

(1) Pesticides
(a) Aldrin. Results from sample points 2 and 3 agree quite closely and were about half the average value found at sample point 1. Although the production schedule of aldrin was quite constant throughout the survey, the concentrations in the inlet stream at sample point 1 were greatly reduced on weekends.

(b) Dieldrin. The production of this pesticide was stopped the second day of the survey, at which time the highest concentration was found in the inlet waste stream at sample point 1. However, lower peak inlet concentrations were also observed on sample days 4 and 8 with the weekend showing very reduced values. Results from sample points 2 and 3 exhibited much less variability, had an average of about half that at the inlet and showed about a four day lag time of the effect of peaks at sample point 1.

(c) Endrin. Similar to the other two chlorinated pesticides, Lake F-1 was shown to have about a 50 percent attenuation effect on inlet concentrations. The production of endrin was terminated on sample day 2. There was a general downward trend of inlet results after that time, but similar to dieldrin, there was a definite peak on sample day 8. This may have been caused by washing activities on that day. The closeness of the average results from sample points 2 and 3 indicate the free passage of the pesticides through the filter and the approximate precision of the analytical methods used.

(d) Parathion. The highest pesticide concentrations found were those of parathion ranging up to 1000 μg/l. Although the production was consistent throughout the survey (as shown in Table 2), the inlet concentration showed distinct peaks on sample days 3, 7 and 11. These periodic discharges are probably due to the Shell’s policy of accumulating and hydrolyzing the parathion waste before discharging it into the industrial waste stream. Results from sample points 2 and 3 were much less variable and about half the average inlet concentrations. The differences in averages between sample points 2 and 3 are felt to be insignificant considering the number of samples and analytical procedures.

(e) Methyl Parathion. Results of analyses for this pesticide were fairly similar for all sample positions and sample times. Since no methyl parathion was produced during the survey period, the concentrations are either due to residuals from previous runs or from certain breakdown products of parathion. The data did not reflect any of the variations shown by the parathion results.

(f) Other Pesticides. The particular analytical methods used showed no detectable levels of dibrom or nemagon C present. More
sensitive procedures might establish that low levels of these pesticides are present; however, considering the nature of production by Shell, no significant concentrations of these pesticides are expected in the waste stream.

(2) Solid Samples. The results in Table 8 indicate very high pesticide concentrations in solid samples taken from the bottom of Lake F-1. Values from the sample taken in the direct line of flow (Location A, Figure 1) are very high. The lower levels at Location B confirm the visual observation that there is a somewhat stagnant zone in that portion of the lake. Most of the pesticide concentrations were reduced about 50 percent between the inlet and outlet of Lake F-1. The pesticides apparently settle out accounting for the high concentration in the bottom material.

(3) General Observations. Chemical results have shown that Lake F-1 does have a significant dampening effect to variations in the inlet waste stream; however, greater buffering effect could be achieved if the stagnant areas were eliminated. This could be done by relocation of the inlet, the overflow to Lake F, and the removal of the center dike. Attaining the maximum retention will become more important when the overflow to Lake F is eliminated. If there were no flow into Lake F, the retention time in Lake F-1 for the 266 gpm average flow would be approximately 2.6 days.

8. CONCLUSIONS.

a. A high volume of waste is still being contributed to Lake F and evaporation rates will not keep up with the rate of excess waste flow.

b. Chemical and physical properties of the wastes at the inlet of Lake F-1 were highly variable. Lake F-1 does exert some dampening effect on the variability of the waste properties; however, increased detention time would be desirable.

c. Wastes analyzed during this survey were more dilute than those analyzed during the 1960 survey conducted by this Agency. The addition of slightly contaminated cooling water to the industrial waste system accounts for the increased volume and more dilute nature of the waste. The greater volume means total cost of disposal has increased.

d. Industrial wastes from still and process tank bottoms, which are trucked out and emptied in Lake F, are high in pesticide content.

e. The toxic effects on the environment of surface storage of this industrial waste in Lake F-1 and Lake F are quite obvious, particularly on wildlife.
f. There were high pesticide concentrations in Lake F-1 liquid and a significant build-up in bottom deposits. Lake F also contained significant pesticide concentrations.

9. RECOMMENDATIONS.

a. Dispose of all currently generated industrial wastes through the deep well disposal system and eliminate the build-up of waste volume in Lake F.

b. Change the overflow and inlet location of Lake F-1 and modify the peninsula in Lake F-1 to attain greater settling and equalizing effects. The new inlet location can be achieved by surface diversion or surface piping.

c. Investigate waste quantity reduction steps and alternate disposal methods for the industrial wastes currently being produced.

d. Discontinue the current policy of disposal of still and process tank bottom wastes into Lake F or Lake F-1.

e. Take steps to eliminate Lake F, as soon as possible, and thereby remove much of the present environmental hazard of exposed surface storage of toxic wastes.

10. ACKNOWLEDGMENTS. Appreciation is expressed to Mr. Charles Bushey, DEIS Representative at Rocky Mountain Arsenal, for assistance and cooperation during the survey. Acknowledgment is also made to Mr. Samuel Sass, Chemical Research Division of the Chemical Research and Development Laboratories, Edgewood Arsenal, for the very capable performance of the pesticide analyses. The following personnel of the Sanitary Engineering Division Laboratory are to be commended for their technical skill, cooperation, and perseverance in developing the analytical techniques and in conducting the survey.

SP4 Charles D. Thompson
SP4 Peter J. Berg
SP4 Larry P. McRae
SP4 Harrison J. Sheldon
USAHK-KE  Rept of Ind Waste Sv, Proj No 3471E19-59/66, Rocky Mt Ars,
Denver, Colo  (9-21 Feb 65)

LeRoy H. Reuter
LeROY H. REUTER
Captain, MSC
Sanitary Engineer

William F. Milbury
WILLIAM F. MILBURY
1st Lieutenant, MSC
Sanitary Engineer

APPROVED:

Richard J. Phillips
RICHARD J. PHILLIPS
Lt Colonel, MC
Commanding
APPENDIX I

SPECIAL LABORATORY PROCEDURES

1. **Iron Determination.**

   a. Because of the large amounts of interfering compounds expected in the waste, Standard Methods called for the use of the ether extraction technique. However, because of the large number of samples anticipated, a shorter, more simple technique was sought. Therefore, an investigation of modifications of the 2,2'-2''-tripyridine technique was performed in the SED laboratory utilizing known concentrations of expected interferences.

   b. Through an investigation of the procedural variables, the pH of the system prior to color development was found to be very critical. As stated by both Standard Methods and the original work, the volume of ethylenediamine to be used is 5 ml. This volume of base raised to pH to between 10.5 and 11. Although the references stated that the pH should be 9.6. It was found that careful addition of ethylenediamine to give a pH of 9.6 eliminated the error due to phosphate. Other interfering metals were added to water and found to produce negligible error when the pH was properly adjusted. Experiments with samples of the waste indicated that residual nitric acid was affecting color development and therefore had to be removed by boiling the sample to dryness. Finally the standard addition test was performed using samples of the waste and gave acceptable results.

   c. The procedure used was as follows:

      (1) Use 50 ml of sample, or a known value, containing less than 0.2 mg iron, dilute to 50 ml.

      (2) Add 3 ml HNO₃ and boil down to dryness.

      (3) Add 20 ml water and 2 ml HCl.

      (4) Heat and reduce volume by 30 percent.

      (5) Cool.

      (6) Add 1 ml Hydroxylamine-HCl.

      (7) Adjust pH to 9.6 using Ethylenediamine.

      (8) Add 5 ml Tripyridine and dilute to 100 ml.

      (9) Measure optical density at 555 μm.
Denver, Colo (9-21 Feb 65)

2. *Arsenic Determination.*

a. A determination with silver diethylthiocarbamate was used which was adopted from the US Public Health Service, Robert A. Taft Sanitary Engineering Center recommendations. The sensitivity and relative resistance to interferences made this determination favorable for the waste involved. A Gutsell apparatus is used. The arsenic is reduced to the trivalent form, evolved as arsine and passed through a lead acetate scrubber into an absorber containing the silver diethylthiocarbamate where the arsine forms a soluble red complex.

b. The procedure used was as follows:

1. Use 50 ml of sample, or a known volume, containing less than 15 ug As, diluted to 50 ml.

2. Add 5 ml of concentrated HCl, 2 ml of 15 percent KI solution and 8 drops 6% stannous chloride solution.

3. Mix well and allow to stand 15 minutes.

4. Impregnate the scrubber with lead acetate solution and place 4 ml of silver diethylthiocarbamate solution in the absorber tube.

5. Add 3 grams of granular zinc to the solution in the generator flask and connect the apparatus immediately.

6. Let stand 30 minutes until arsine evolution is complete.

7. Transfer the absorbing solution to 1 cm cells and measure absorbance at 560 mu in a spectrophotometer.

3. *Total Phosphorous Determination.*

a. For the determination of total phosphorus the procedures in Standard Methods were inadequate because of the organic nature of the waste. A literature search disclosed two techniques which appeared applicable for the anticipated waste. After preliminary laboratory analyses of organic phosphate ester compounds utilizing these two procedures, it was found that neither procedure yielded acceptable results, but that a modification of each combined into a third procedure described below proved to be satisfactory.

b. The sample is brought to a pH of 8-10 in order to hydrolize the organic phosphate esters. It is then digested over low heat with ammonium persulfate to convert the phosphorous to orthophosphate. After
completion of the oxidation process (resulting in a colorless solution), the excess ammonium persulfate is destroyed by the addition of sodium sulfite. The sample is then neutralized with nitric acid using paranitrophenol as the indicator. Acid molybdate solution and aminonaphtholsulfonic acid are added for color development, the volume is adjusted, and an aliquot is placed in the spectrophotometer. Readings are made at 690 nm and compared against a standard curve prepared in the same manner.

4. **Gas Chromatograph Analytical Specifications.**

**COLUMN:** 5'5" of 1/4" OD pyrex glass filled with a mechanical mixture of 1 part 5% DC-LSX-3+0295 on 60-80 mesh

Gas Chrom Z

1 part 20% SE-30 on 60-80 mesh

Gas Chrom P

**Temperature - Isothermal at 209°C**

**DETECTOR:** Electron Capture at 210°C

pulse internal - 1/150 Sec

pulse time - 0.75 sec

**Injection Port Temperature:** 225°C

**Carrier Gas Flow:** Argon-methane (95%-5%) - 85 ml/min

**Purge Gas Flow:** Argon-Methane (95%-5%) - 175 ml/min

**Type of Instrument Used:** F and M 810
BIBLIOGRAPHY (APPENDIX I)


2. Unpublished data of work performed by Sr4 L. P. McRae on file in the Sanitary Engineering Division Laboratory.


### Appendix II

**Flow Rate Data into Lake F-1**

*(Sample Point 1)*

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| Daily Average  | 219 | 255 | 250 | 300 | 234 | 245 | 276 | 333 | 295 | 310 | 259 | 223 |
**APPENDIX II (Cont)**

**FLOW RATE DATA DOWN DEEP-WELL**

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**Daily Average**

|          | 43.2| 41.6| 42.4| 41.8| 39.7| 43.1| 41.5| 40.9| 40.4| 40.8| 40.6|
## APPENDIX II (Cont)

### COMPOSITE RESULT DATA

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## COMPOSITE RESULT DATA

(Sample Point 3)

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<tr>
<td>Sulfates (mg/l)</td>
<td>153</td>
<td>135</td>
<td>500</td>
<td>427</td>
<td>460</td>
<td>493</td>
<td>378</td>
<td>467</td>
<td>491</td>
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<td>1.022</td>
<td>1.227</td>
<td>1.096</td>
<td>1.208</td>
<td>3.668</td>
<td>37.18</td>
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<td>35.37</td>
<td>28.88</td>
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<td>Solids, Sett. (mg/l)</td>
<td>&lt;0.01</td>
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<tr>
<td>Solids, Susp. (mg/l)</td>
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<td>3.0</td>
<td>3.2</td>
<td>4.0</td>
<td>2.4</td>
<td>2.4</td>
<td>2.0</td>
<td>1.0</td>
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<td>1.6</td>
<td>1.6</td>
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<td>Solids, Total (mg/l)</td>
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<td>1895</td>
<td>2910</td>
<td>1470</td>
<td>955</td>
<td>719</td>
<td>1600</td>
<td>1070</td>
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<td>1</td>
<td>8</td>
<td>7</td>
<td>&lt;1</td>
<td>3</td>
<td>&lt;1</td>
<td>2</td>
<td>8</td>
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<td>Aluminum (mg/l)</td>
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<td>367</td>
<td>320</td>
<td>168</td>
<td>306</td>
<td>326</td>
<td>251</td>
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<td>260</td>
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<tr>
<td>Magnesium (mg/l)</td>
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<td>205</td>
<td>193</td>
<td>216</td>
<td>213</td>
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<td>154</td>
<td>170</td>
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### APPENDIX II (cont)

**COMPOSITE PESTICIDE RESULTS**
(All concentrations in μg/ml)

<table>
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<tr>
<th>Sample Day</th>
<th>Aldrin</th>
<th>Dieldrin</th>
<th>Endrin</th>
<th>Parathion</th>
<th>Methyl Parathion</th>
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<td>2</td>
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