

INFILTRATION AND PERCOLATION STUDIES
OF SULFIDES AND SEWAGE CARBONACEOUS MATTER

by
James S. Kumagai

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Principal Investigators: L. Stephen Lau and Nathan C. Burbank, Jr.

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ABSTRACT

The laboratory study of the infiltration and percolation of sulfides and sewage carbonaceous matter was conducted in two phases: Phase I utilized simulated cesspool lysimeters and Phase II considered the generation of sulfides and the infiltration and percolation of sulfides through soil and sand columns.

Results from Phase I dictated a need for further study owing to free percolation of certain odorous compounds and excellent COD removals under presumably anaerobic conditions contrary to findings of similar studies in the literature.

In Phase II the soil column was more effective for sulfide removal than the sand column which allowed continuous breakthrough of an odorous percolate. Organic removal indicated by TOC, COD, and BOD under anaerobic conditions was insignificant with BOD data indicating an increase in organics during percolation. The progressive movement of a black precipitate through the sand bed indicated that the filtering action of the sand was not as effective as the soil column. Under acid conditions, sulfide breakthrough was clearly demonstrated in both the sand and soil columns. The flow rate significantly improved in both columns after percolation of acidified fluids.

All columns exhibited the characteristic non-linear relationship between filtration and percolation rates and the hydraulic gradient. The results of the flow study can be described by the relationship of Hansbo (1960):

$$v = \lambda i^n$$

where V = velocity, i = hydraulic gradient, n = constant and λ = constant describing permeability.

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INTRODUCTION

Because of the significance of hydrogeology in ground water recharge and because of the uniqueness of the geology of different geographic locations, specific problems for any particular situation and locality still need study in spite of the growing volume of research results related to ground water pollution.

No widespread reports of ground water pollution have been noted in Hawaii. However, to conclude that pollution is not occurring or will not occur from surface waste disposal may be premature especially since analysis for micro-contaminants, as far as is known, has not been performed. The changes in water quality may be subtle and may have escaped subjective detection. Further, the uniqueness of Hawaiian hydrogeology and environment makes localized research in ground water pollution necessary.

However, one opinion of a possible solution to ground water pollution was expressed by Geraghty (1965): "In the long run, there is probably only one solution — and that is to prevent disposal of wastes into reservoirs that are tapped for water supply." The solution may not be practical, but the statement expresses caution.

PURPOSE AND SCOPE

It is the purpose of this report to (i) to investigate the degree of organic removal during percolation of anaerobic effluents through kaolinite clayey soils, and (ii) to study the transmission and the clogging characteristics of sulfides in kaolinite clayey soils and quartz sands.

It is intended that this work aid and stimulate further work in this direction.

SULFIDES

The review of the literature indicated that total sulfides were significant in ground water recharge systems because of the odor problems and of the clogging action of the ferrous sulfide precipitates. The results of the Warner (1966) and ASCE (1977) studies with ferric chloride indicated its contribution to porous media clogging, having adhesive properties more pronounced than other precipitates. These results strongly suggested that research directed at specific precipitates or clogging agents was necessary until a general theory can be developed to relate fundamental porous media and particulate material properties. For this reason, the study of sulfides and their significance in infiltration and percolation was initiated.

Significance of Sulfides

Sulfide precipitates in septic tank effluents clog infiltration and percolation systems. In other sanitary engineering processes and in the environment, sulfides, as hydrogen sulfide, are a definite health hazard.

In municipal water supplies, sulfides impart taste and odor while exerting high chlorine and oxygen demand. When sulfides are present in significant amounts, disinfection by chlorine may be uneconomical due to the high demand for oxidation of sulfides. A secondary effect in sewage chlorination may be experienced by oxygen consuming sulfides, creating and maintaining anaerobic conditions under which the resulting organics may impose a high chlorine demand (Rhines, 1965).

Some significant aspects of sulfides in environmental systems are summarized in Table 1. The significance of sulfides can readily be seen to be far-reaching in many phases of environmental and sanitary engineering systems. For this reason, the results of sulfide studies are expected to have widespread application.

In ground water recharge with sewage sources, clogging by sulfide precipitates has been reported. A significant factor not well documented is the transmission of soluble sulfides in porous media. This aspect is especially significant because of the taste and odor characteristics of sulfides in low concentrations in water supplies. The clogging property of metallic sulfide precipitates implies retention of sulfides in the

TABLE 1. SUMMARY OF SULFIDE SIGNIFICANCE IN ENVIRONMENTAL SYSTEMS

ITEM	CONCENTRATION	REFERENCE
TASTE AND COLOR	0.05 mg/l	MCKEE AND WOLF (1963)
ODOR	LESS THAN 1 mg/l	STANDARD METHODS (1965)
CLOGGING	APPROX. 3 mg/l IN SEPTIC TANK EFFLUENT	WINNEBERGER <i>et al.</i> 1960)
SEWER CORROSION	APPROX. 3 mg/l REPORTED IN SEWER CORROSION PROBLEMS	LAWRENCE (1965)
FISH TOXICITY	0.5-1.0 mg/l CRITICAL	MCKEE AND WOLF (1963)
BENTHIC DEOXYGENATION OF EUTROPHIC LAKES	MINIMUM NOT ESTABLISHED 1400-3300 mg/l TOTAL S- FOR LAKE MENDOTA. (FERROUS IRON SIGNI.)	GARDNER AND LEE (1965)
CHLORINE DEMAND	STOICHIOMETRIC OXIDATION	SAWYER (1960)
TOXIC TO MAN	AS TOXIC AS HCN	ZOBELL (1963)

porous media, and thus their removal from the percolating fluids.

pH Dependence

Deposition or transmission of sulfides in porous media depended on whether the sulfide was a precipitate or a solute. Sulfide precipitates, especially ferrous sulfides, are soluble under acid conditions unlike other metallic forms. The deposition or transmission of sulfides in porous media is, therefore, pH dependent. Since sulfides readily form metallic sulfides and, perhaps, precipitates, the reaction of sulfides with porous media minerals appears to be a possibility. But it can be speculated also that soluble sulfides can be transmitted to and through ground water aquifers.

Occurrence of Sulfides

A review of reports of the occurrence of sulfides in ground water support the possibility of the transmission of sulfides and consequent sulfide contamination of ground water.

McKee and Wolf (1963, p. 200) reported that hydrogen sulfide was present in many municipal supplies as a result of anaerobic decomposition of crude ground deposits. McMichael and McKee (1965) in reporting on the Whittier Narrows project speculated that anaerobic conditions

TABLE 2. REPORTED SULFIDE OCCURRENCES

LOCATION	CONCENTRATION	REMARKS	REFERENCE
VOLCANIC GASES	40 TO 42.4%	H ₂ S IN GAS AND TRACES OF MERCAPTAN	ZOBELL (1963)
GROUND WATER	NOT GIVEN	NOT REFERENCED	MCKEE & WOLF (1963) STANDARD METHODS (1965)
NORWEGIAN FJORDS & BLACK SEA	14 mg/l	H ₂ S	ZOBELL (1963)
BIG SODA LAKE, NEVADA	786 mg/l	TOTAL SULFIDES	ZOBELL (1963)
OILFIELD WATERS	2,400 mg/l	H ₂ S	ZOBELL (1963)
DIGESTED SLUDGE			
EAST BAY MUNICIPAL UTILITY DISTRICT	825 mg/l	TOTAL SULFIDES	WINNEBERGER, <i>et al.</i> (1960)
STEGE SANITARY DISTRICT	91 mg/l	"	"
SAN PABLO SANITARY DIST.	130 mg/l	"	"
COMPOST PILES	NOT GIVEN	BLACK COLOR	WINNEBERGER, <i>et al.</i> (1960)
SEPTIC TANK EFFLUENT	3.1 mg/l	TOTAL SULFIDES	WINNEBERGER, <i>et al.</i> (1960)
SEWAGE, SAN DIEGO BAY AREA	2.8-3.8 mg/l	CORROSION PROBLEMS	LAWRENCE (1965)
SEWER FORCE MAIN, TRINITY RIVER AUTHORITY, TEXAS	12 mg/l	CORROSION PROBLEMS	LAUGHLIN (1964)
SEWAGE, FORCE MAIN	10 mg/l	SEA WATER INFILTRATION ORP-320	BACHMEYER & DRAUTZ (1963)
LAKE MENDOTA, BOTTOM DEPOSITS	1,400-3,300 mg/l	TOTAL SULFIDES IN EUTROPHIC LAKE	GARDNER & LEE (1963)

were undesirable in the recharge system because of the possibility of sulfide formations entering ground water supplies and polluting the municipal water supply. Zobell (1963) noted that sulfides were found in concentration up to 2400 mg/l in oil field formation waters. *Standard Methods* (1965, p. 293) stated that sulfides were found in many well waters, lakes, and even in water distribution systems because of the presence of organics under anaerobic conditions. Although these reports were not referenced or documented they nevertheless support speculation that generation and transmission of sulfides in ground water aquifers are possible under anaerobic conditions.

The occurrence of sulfides in sewage and anaerobic treatment processes has been long recognized. Some examples of sulfides in sewage and natural waters are listed in Table 2.

The formation and occurrence of sulfides are usually characterized by black deposits of ferrous sulfides or detected by the distinct odor of hydrogen sulfide. Winneberger *et al.* (1960) reported that it was long

believed that the black color of digested sludge and other effluents from anaerobic processes was due to humus-like material indicating the "richness" of the sludge. It was later found that the black color was due to ferrous sulfide precipitates.

Zobell (1963) presented examples of sulfides in natural water bodies, including lakes and oceans. Gardner and Lee (1965) reported a high sulfide content in bottom deposits in Lake Mendota, an eutrophic lake. These authors further reported that sulfides as well as ferrous iron may be contributing to the deoxygenation of the lake bottom and presented laboratory oxygen uptake studies of lake bottom sediments.

Sulfides are readily detectable in sewers as hydrogen sulfide by the distinctly characteristic rotten egg odor. Lawrence (1965), Laughlin (1964), and Bachmeyer and Drautz (1963) described significant sewer sulfide concentrations. A common effect of sulfides in sewers is corrosion.

Temperatures

Lawrence (1965) in a study of sewer corrosion potential of sewage in far Western States observed that sewer corrosion problems were found in areas below the 39th parallel or approximately south of Sacramento, California. Laughlin (1964) reported sewer main sulfides up to 12 mg/l in Trinity River Authority, Texas. Bachmeyer and Drautz (1963) reported concentrations of 10 mg/l in sewer force main experiencing sea water infiltration in Miami, Florida. These reports indicated problems located in southern areas of the continental United States where warmer temperatures prevail for longer periods. The reported high concentrations of sulfides in the South and in Southern oil field waters (Zobell, 1963) can be explained by the biological reaction rate increases with rising temperatures. But high sulfides found in colder climate zones, such as the Norwegian Fjords and Lake Mendota (Table of Occurrences), indicated that under adverse climatic conditions, the long-term action of standing waters produced equivalent, or greater concentrations of sulfides than for shorter-termed higher temperature action in sewers. It is apparent that the time-temperature relationship depicts extremes where either time or temperature may be the predominant factor. Furthermore, when both are optimum, higher sulfides can be expected provided other factors are

not limiting.

Bachmeyer and Drautz (1963) reported sea water infiltration with a significant corresponding increase of sulfides in sewer mains. The significance of sea water infiltration into sewers is the addition of sulfates that occur in ample quantities in sea water as its second major anion. Although sulfides occurring in sewage have been reported to be largely derived from proteinaceous material, sulfides due to sulfate reduction can become significant as Bachmeyer and Drautz (1963) indicated. The problems related to sulfides in the environmental systems can become acute at higher concentrations and with increasing temperatures.

Sulfides from Sulfate Reduction

Zobell (1963) presented a thorough review of the literature on sulfides, its occurrences in geologic formations, action of sulfate reducing bacteria, and other general considerations of the sulfur cycle.

The sulfate reducers are a hardy group of bacteria as indicated by the ecological factors summarized by Zobell (1963). They have been isolated from sea floor samples at depths exceeding 30,000 ft., from oil brines at depths exceeding 10,000 ft., and also in significant numbers in marine deposits, soil, sewage, and stagnant waters.

Sulfate reducers were found to tolerate wide extremes in temperatures. Zobell (1963) gave an example of sulfate culture growth at 104°C at 1000 atm to 85°C at 1 atm. However, most strains grew best at 20°C to 45°C. They can tolerate a pH range of 4.5 to 10.0 with optimum at 6.5 to 8.0, but were strict anaerobes deriving energy from organic matter or free hydrogen. Utilizing many forms of organic matter, and the sulfate reducers can either work alone or with other forms of bacteria (Zobell, 1963).

The sulfide literature, summarized by Zobell (1963), describes the versatility of the sulfate reducers and their widespread existence in ground, ocean, lake, and sewage environments. Sulfate reducers are present in environmental systems where sufficient nutrition and energy sources are available under anaerobic conditions.

Summary

The review of the literature presented the following significant factors for optimum sulfide generation:

- (1) Anaerobic environment
- (2) High organic matter concentration
- (3) Sulfate concentration
- (4) Temperature (20°-45°C)
- (5) Proper seeding of sulfate reducers initially
- (6) Time of activity.

Under long-term activity in relatively stagnant waters such as in lakes, ponds, and ground water reservoirs, sulfide concentrations may become significant. Ample evidence supports these fundamental findings and at optimum conditions sulfide generation occurs in significant concentrations regardless of the physical or geologic location.

METHODS AND PROCEDURES

General Conduct of Laboratory Study

Laboratory study of the treatment of sewage in soil lysimeters was undertaken as an extension of the work initiated by Koizumi (1965). The purpose of this initial phase of study was the characterization of the percolate in general chemical classes as proteins, carbohydrates, and COD and the determination of the order of treatment efficiency by these parameters. Nitrogen forms as ammonia nitrogen, kjeldahl organic nitrogen, and nitrate nitrogen were determined in relation to the organic parameters. Head measurements were not made during this phase except for the notation of positive or negative pressures indicated by the manometers. Koizumi's lysimeter study lasted approximately 3 months. The second phase included the infiltration and percolation of sulfides as well as the transmission of sewage organics under anaerobic conditions in lucite soil columns. The parameters observed during the second phase of the study included total organic carbon (TOC), total carbon (TC), BOD, COD, and total sulfides.

The laboratory lysimeter study for the two phases differed in these aspects; (i) type of sewage used, (ii) hydraulic head, and (iii) lysimeter geometry.

The First Phase. The first phase of the study was performed with soil lysimeters designed and constructed by Koizumi in 1965, and operated in a similar manner. The soil lysimeters simulated cesspools and enabled the study of sidewall and bottom area flow. The side sections of the soil lysimeters allowed oxygen diffusion into the soil mass, but the bottom areas of the soil mass became unsaturated during the course of the study as indicated by negative manometer readings. Similar occurrences were reported by others in lysimeter studies (Orlob and Butler, 1955; McGauhey and Winneberger, 1963). The unsaturated conditions in the lysimeters provided an opportunity for soil aeration. In column studies, the middle sections can be unsaturated but the bottom section must be saturated, or must be maintained above field capacity or specific retention for flow to occur under influence of gravity alone against atmospheric pressure. With the creation of an opportunity for aeration,

the time of exposure of percolate to air and the flow rate becomes critical since laboratory aerobic action becomes significant in the soil system.

Soil Lysimeter Operation. Soil lysimeters used in Phase I were described by Koizumi (1965) and Koizumi, Burbank and Lau (1966). The lysimeter designation and manner of operation for the Phase II study are shown in Table 3.

Second Phase. In Phase II, gradients up to 4.6 were used and columns were operated as falling head permeameters. The apparatus, however, was not as versatile as Koizumi's lysimeters (1965) but permitted greater control in maintaining anaerobic and aerobic conditions in the soil mass. Another reason for the selection of columns in the second phase was their simplicity in studying variable gradient effects. Complete saturation was ensured by raising the end of the discharge tube above the soil mass. Because of reported instances of oxidation near the liquid surfaces of sulfide samples, greater depths were used to minimize the effect of oxygen diffusion from the surface into the liquid body and sulfide diffusion to the surface.

Both phases of study were considered to be short-term when compared to reported lysimeter studies maintained for one-year periods or longer. It was also expected, as reported in many past studies, that under anaerobic conditions organic contaminants such as ABS percolated freely

TABLE 3. DESCRIPTION OF THE SOIL LYSIMETERS

LYSIMETER	POROUS MEDIA	FLUID	LOADING
1	SOIL	TAP WATER	FALLING HEAD PERMEAMETER
2	SOIL	ANAEROBIC SEWAGE	"
3	SOIL	"	INTERMITTENT LOADING
4	SAND	TAP WATER	FALLING HEAD PERMEAMETER
5	SAND	ANAEROBIC SEWAGE	"
6	SAND	"	INTERMITTENT LOADING

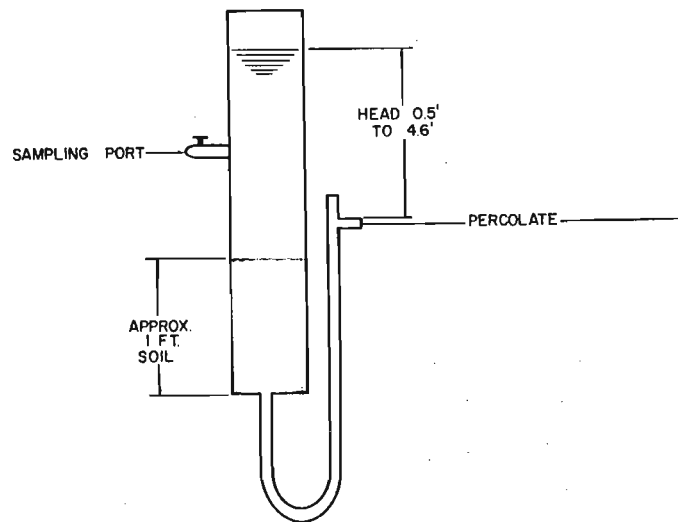


FIGURE 1: SOIL COLUMN CONSTRUCTION

through soil lysimeters. Because a soil with high clay content was used for these lysimeters studies, it was felt that exploratory studies on the possible soil related mechanisms of organic removals such as adsorption would be significant. The soil columns (Phase II) were 2 inches in diameter by 6-foot long lucite tubes filled with approximately a 1-foot section of soil or sand and operated as falling head permeameters with depths on the order of 0.5 to 4.6 feet (Figure 1).

The soil samples selected were Low Humic Latosols significant in Hawaii for agricultural purposes and possessing good drainage characteristics. The soil was sieved to remove extraneous matter and large clumps. The columns were backwashed and later compacted by drawing tap water through the samples under a vacuum of 30 in. Hg. The soil samples were analyzed for clay, silt, and sand percentages along with soil organics and free iron as Fe_2O_3 .

Quartz sand sizes were between 0.8 mm and 0.4 mm. The soil and sand columns were run in parallel. The sand columns had a higher permeability due to the larger grain size than the soil samples. The infiltration and percolation rates through the sand columns were regulated to correspond approximately to that of the soil columns by pinching the discharge tube.

Sulfide Generation

The study of infiltration and percolation of sulfides was preceded by a sulfide generation study in incubation bottles. This procedure minimized possible inhibition of sulfide generation and concentration increase by aerobic conditions created by oxygen diffusion through containers or lysimeters exposed to the atmosphere. The anaerobically treated sewage samples from the incubation bottles were then used to dose the soil columns. (The lysimeters in Phase I were dosed with raw sewage.)

Under laboratory conditions of Phase II, anaerobic sewage containing maximum sulfides was desired to measure sulfide concentration changes in the order of magnitude far greater than changes occurring due to experimental error. For this reason, sulfide generation was allowed to continue until steady-state condition was reached before concluding the bottle incubation tests.

The sulfide generation study was performed initially by utilizing sewage samples obtained from the Hawaii Kai primary treatment plant, the Ala Moana Pumping Station, Wahiawa sewage treatment plant, and the Pacific Palisades subdivision sewage treatment plant. The sewage samples were incubated in open polyethylene containers at $22^{\circ} \pm 2^{\circ}\text{C}$ and were followed by incubation tests in one gallon bottles similar to the single dilution method for long-term BOD tests described by Orlorb, *et al.* (1955). This technique minimized exposure of sulfide samples to atmospheric oxygen. The unstable sulfide oxidizes readily forming white precipitates of colloidal sulfur as reported by many workers (Zobell, 1963). A similar reaction was observed during this study with exposed containers.

Sewage grab samples were collected from the Hawaii Kai primary treatment plant and from the Pacific Palisades sewage treatment plant which employed the trickling filter process as the secondary treatment unit. The location and pertinent remarks of field sample collection are summarized in Table 4. Samples were collected and transported to the laboratory in polyethylene containers and allowed to settle for 24 hours for deposition of some suspended solids. Sulfates were added in predetermined amounts as magnesium sulfate. The sewage samples with conditions summarized above were incubated at room temperature in a dark cabinet to suppress algae growth. When the sulfide generation rate decreased to

TABLE 4. SAMPLE COLLECTION LOCATIONS

	COLLECTION TIME AND DATE	TREATMENT UNIT SAMPLED	REMARKS
HAWAII KAI PRIMARY TREATMENT PLANT	1600 HR 20 JUNE '66	A. GRIT CHAMBER B. CHLORINE CONTACT TANK	SEA WATER INFILTRATION. PLANT SULFIDES PRESENT.
PACIFIC PALISADES TREATMENT PLANT	1400 HR 20 JUNE '66	A. PRIMARY CLARIFIER B. SECONDARY CLARIFIER	

TABLE 5. DESCRIPTION OF SAMPLE

SAMPLE NO.	DESCRIPTION	SULFATES ADDED mg/1 AS $MgSO_4$
1	PACIFIC PALISADES PRIMARY SEWAGE	0
2	"	100
3	"	200
4	HAWAII KAI RAW SEWAGE (GRIT CHAMBER)	0
5	HAWAII KAI PRIMARY TREATED SEWAGE (CHLORINE CONTACT TANK)	0
6	PACIFIC PALISADES SECONDARY SEWAGE (SECONDARY CLARIFIER)	0
7	"	100
8	"	300

steady-state condition, 500 mg/l dextrose was added to the incubated samples to observe the effect of increased organic concentration on sulfide generation and their corresponding sulfate reduction. The parameters observed during the sulfide generation tests were these: total sulfides, sulfates, total organic carbon pH, and ORP. At the conclusion of the sulfide generation study, incubated samples containing high sulfides were used to dose the soil columns.

SOIL LYSIMETER OPERATION

Percolate Collection and Handling

Samples were collected on alternate days for sulfides and then for organic analyses as BOD, COD, total carbon, and total organic carbon. The sulfide samples were fixed by allowing the sample to drip into a collection cylinder containing a zinc acetate solution. Analyses were performed of both the sewage infiltrating into the soil sample and the resulting percolate.

Field samples analyzed for sulfides were collected with a DO sampler containing a 300 ml BOD bottle. Samples were fixed in the field with zinc acetate solution upon collection.

Analytical Methods

All analyses were performed according to *Standard Methods* (1965) unless otherwise noted.

Proteins. Proteins were performed by Folin's method described by Woods (1965). The standard used was casein and results expressed as mg/l casein. Absorbance measurement was performed with the Bausch and Lomb spectronic 20 with red filter.

Carbohydrates. Carbohydrates were determined by the phenol-sulfuric acid method of DuBois, *et al.* (1956) using sucrose as the standard carbohydrate. All carbohydrates were expressed as sucrose.

Sulfides. Total sulfides were determined by the titrimetric method outlined by *Standard Methods* (1965) using volumes from 100 to 1000 ml depending on the concentration of sulfides and available collected sample volume. For low sulfides of about 1 mg/l, sample volumes of approximately 1 liter were used. For samples containing sulfides up to 40 mg/l, a 100-ml volume was used. The order of sample volume was determined by the intensity of the sulfide odor (measured qualitatively).

Carbon dioxide was used as the flushing gas, but some samples were flushed with nitrogen when the CO₂ supply was depleted.

Total Organic Carbon: Instrumental Procedure. The procedure for total organic carbon was essentially the procedure recommended by the R. A. Taft Sanitary Engineering Center for the Beckman Carbonaceous Analyzer. Since difficulty with noise and drifting was experienced initially, the injection sample volume used was 40 microliters instead of the 20 microliter sample volume recommended by the manufacturer. This procedure allowed operation at low gain settings on the instrument allowing minimum drift and noise.

The standard used was potassium acid phthalate prepared as a stock solution of 100 mg/l carbon. The working solutions for the calibration curves were prepared in concentrations of 10 mg/l to 100 mg/l. When higher concentrations were necessary, calibration curves were extended to 200 mg/l. Samples beyond this carbon concentration were diluted to give concentrations within the calibration curve range.

Since it was noted that the recorder gave erratic results occasionally while the corresponding meter reading was consistent, greater reliance was placed on the meter readings. When the recorder was used, a standard solution of 100 mg/l carbon was injected after every fourth sample as a calibration check. (It was convenient to work with groups of four in sample preparation.) When deviations of recorder readings for the 100 mg/l standard varied more than 5%, a new calibration curve was prepared and used for subsequent samples. Normally, calibration curves were prepared daily with the carbon analyzer left on continuously during sampling periods.

Total Organic Carbon: Sample Preparation. The total organic carbon determined by the carbonaceous analyzer was that portion of total carbon measured after sample acidification to pH 2 with concentrated HCl, and ~~flushing with an inert gas, usually a high purity nitrogen gas.~~ Since the carbon analyzer measured all forms of carbon including carbonates, sample acidification and flushing was necessary to eliminate the carbonates and carbon as dissolved CO₂ resulting in the loss of some of the volatile organic compounds. A discussion of this aspect was given by Van Hall, *et al.* (1965). Carbon determination on the untreated sample, including all forms of carbon, is termed total carbon (TC) and carbon measured after treatment is referred to as total organic carbon (TOC). The results are expressed as mg/l TOC or mg/l TC in this report.

When particulate organic solids were significant, such as in raw sewage samples, they were blended for 15 minutes to obtain homogeneity. Some difficulty was experienced with samples containing high suspended solids. Since the sample volume used in the carbon determination was small (20-40 microliters), the selection of a homogeneous sample with respect to suspended solids was not practical and widely divergent values were obtained. The Taft Center procedure recommended that the average be taken from 5 determinations. For all other determinations the average of two determinations was reported.

Sulfates. Sulfates were determined by the turbidimetric method outlined in *Standard Methods* (1965). For water analysis, the precision was specified at $\pm 10\%$ for a range of applicability of approximately 10 to 40 mg/l sulfates. The turbidimetric method was listed as a method of sewage sampling but with a note of caution for possible organic interferences. No precision was specified for sewage samples. This method was selected for its simplicity and ease of performance. Apparently, other methods of sulfate determination are also subject to the same possible interferences found in sewage samples.

Soil Analysis. The soil parameters included in this study, clay, silt, and sand percentages along with free iron, soil organics, and clay mineral were identified by X-ray diffraction. These analyses were performed by Bernadino Cagauan, Jr. Soil Scientist, WRRRC, University of Hawaii.

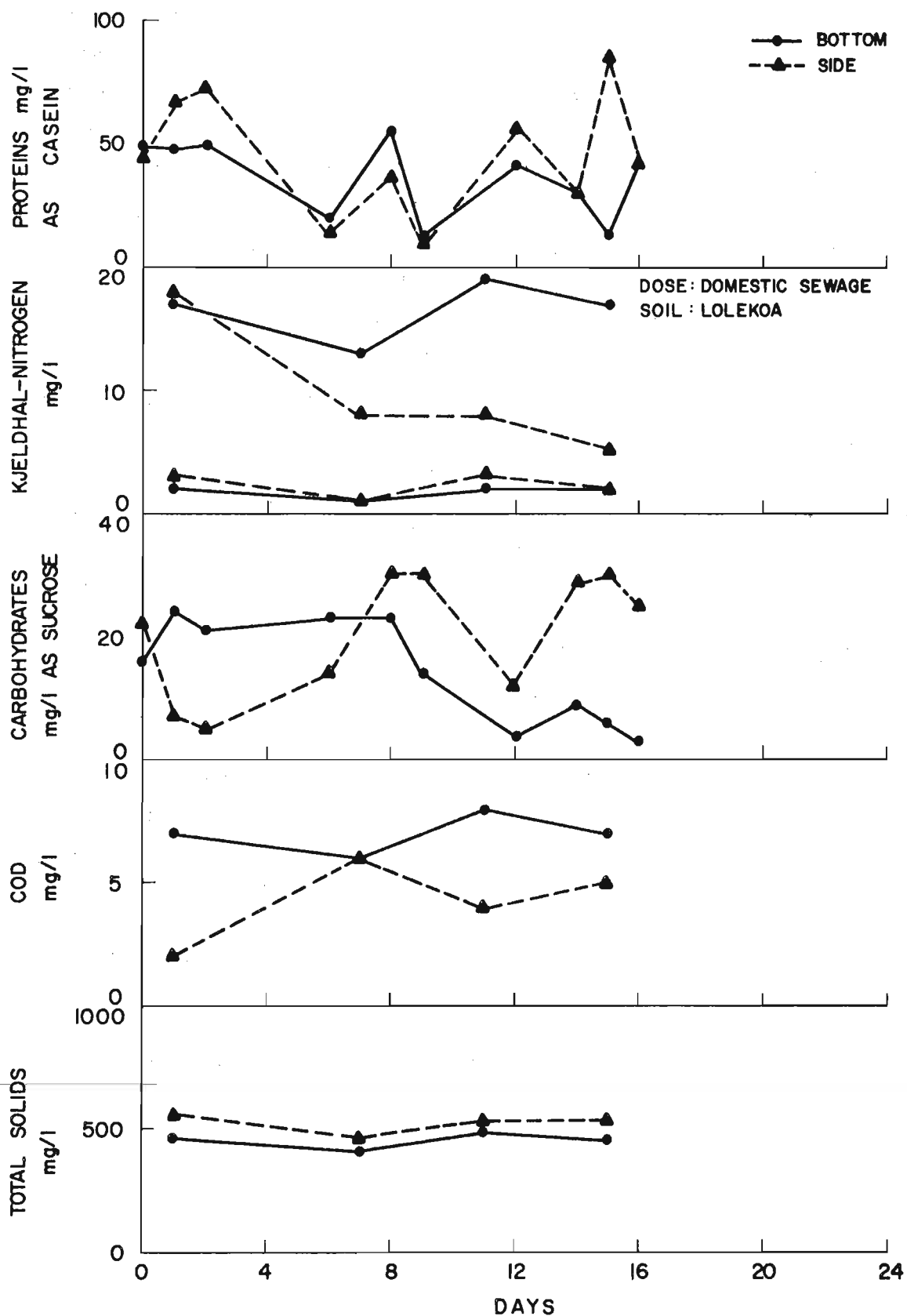


FIGURE 2: PERCOLATE CHARACTERISTICS FROM UNIT C2.

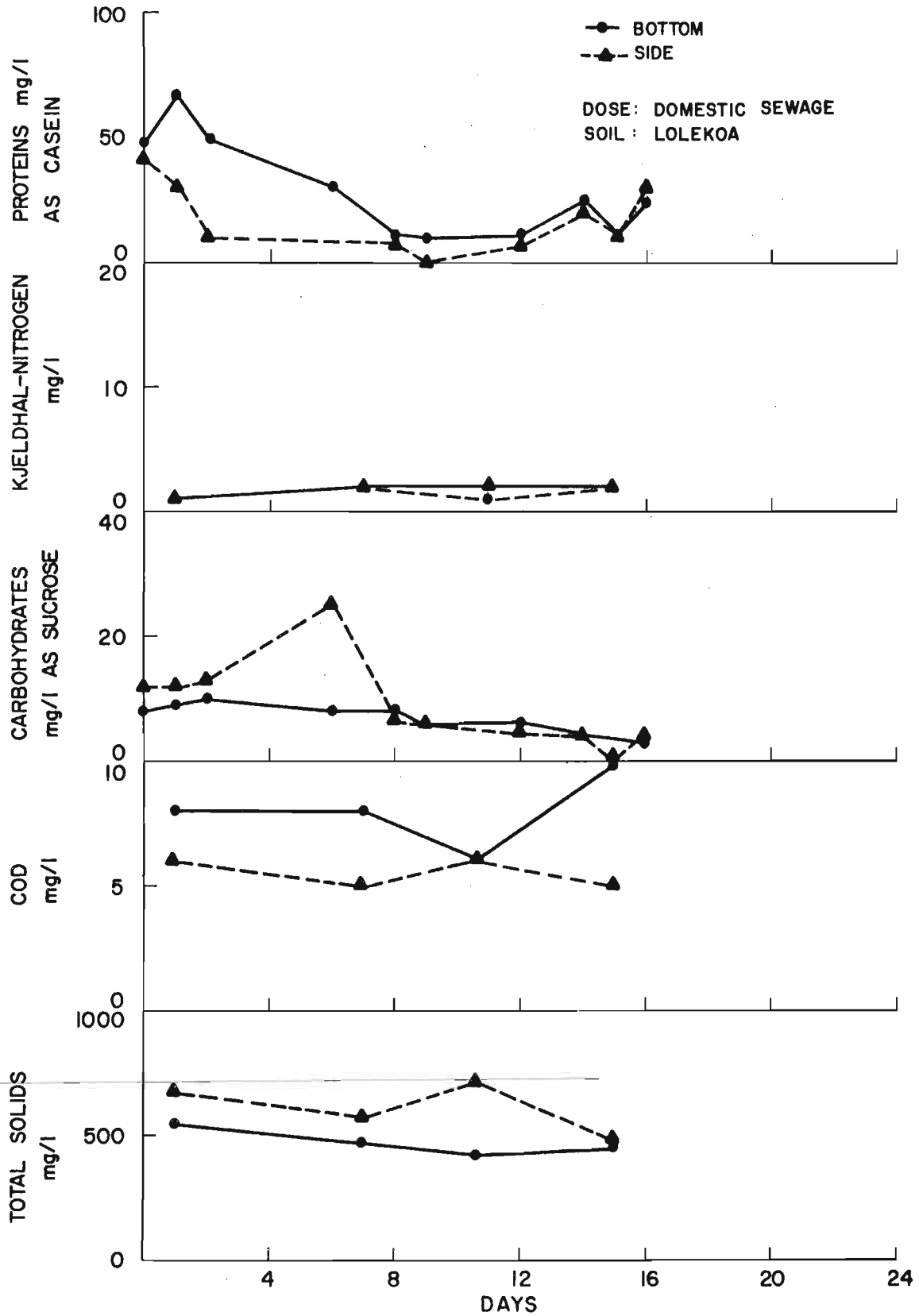


FIGURE 3: PERCOLATE CHARACTERISTICS FROM UNIT C3.

RESULTS AND CONCLUSIONS

Phase I: Laboratory Simulated Cesspool Lysimeters

The Phase I study was conducted with emphasis on percolate characterization as an extension of the work initiated approximately 45 days prior to this study by Koizumi (1965). Two lysimeters, containing Lolekaa soil, were selected, but they differed in the history of sewage loading prior to this study. Koizumi (1965) loaded unit C2 (Figure 2) with a synthetic sewage while unit C3 (Figure 3) was loaded with domestic sewage.

Percolate Quality. The percolate was odorous in some instances, but the odor was not usually detectable after cold storage. The results of the percolate characterization as proteins, kjeldhal-nitrogen, carbohydrates, COD, and total dissolved solids are shown in Figures 2 and 3. These results indicated a marked improvement in percolate quality over the results of the earlier study (Koizumi, 1965). Although both units produced different results quantitatively, the order of apparent treatment efficiency of the units were as follows:

- (1) Protein removals of approximately 90%.
- (2) Carbohydrate removals of approximately 50%.
- (3) COD removals of approximately 90%.

These results further indicated that the percolate consisted of measurable amounts of proteins and carbohydrates with a total dissolved solids concentration on the order of 500 mg/l, a 2-fold increase over Oahu tap water values.

Hydraulic Data. The flow rate through the lysimeters decreased to approximately 0.3 ft/day or 1/3 of the final flow rate found at the conclusion of the study by Koizumi (1965). Pressure readings were negative in all but the uppermost manometers indicating unsaturated conditions in the major portion of the soil mass.

Need for Additional Study. Two significant observations made during the Phase I study indicated the need for further laboratory study:

- (1) Odor in some percolate samples indicated that certain odorous compounds percolated freely through soil.
- (2) Excellent COD removal under presumably anaerobic conditions was not consistent with the reported performance of similar studies in the literature. The fact that unsaturated conditions were indicated by observed negative manometer pressures and nitrate values (Koizumi, 1965) suggested that aerobic conditions prevailed in significant portions of the lysimeter soil mass.

Phase II: Generation of Sulfides and the Infiltration and Percolation of Sulfides and Sewage Organic Matter

Since the probable odor compounds in sewage are generally known to include sulfur compounds, notably sulfides, the laboratory study was directed in greater detail toward sulfide generation in sewage and the infiltration and percolation of sulfides and sewage organics.

Sewage samples from selected plant locations were incubated anaerobically to simulate anaerobic sewage ponding while allowing greater experimental control during study of sulfide generation and identification of significant parameters. Following the sulfide generation studies, sewage samples were used to dose soil columns.

Sulfide Generation from Sewage Samples. The concentration of sulfides from sewage samples typical of Honolulu, Hawaii was determined. Laboratory experiments were performed initially with sewage samples collected from low lying areas near beach fronts: Hawaii Kai primary treatment plant, and Ala Moana Pumping Station; and samples collected from highlands: Pacific Palisades sewage treatment plant, and Wahiawa sewage treatment plant. (Figure 4).

The Hawaii Kai sewage (1500 - 8000 mg/l chlorides) and Ala Moana pumping station sewage (3000 mg/l chlorides) were known to contain sea water infiltration from sewers. Since sea water contains significant concentrations of sulfates as a major anion, sewage samples were expected to yield high sulfides due to sulfate reduction. The technique of incubating sewage samples at liquid depths of 12 inches in containers allowing surface exposure of the sewage to the atmosphere represented the approximate ponding depth in the soil lysimeter studies conducted in Phase I and also approximated lysimeter ponding in studies reported in

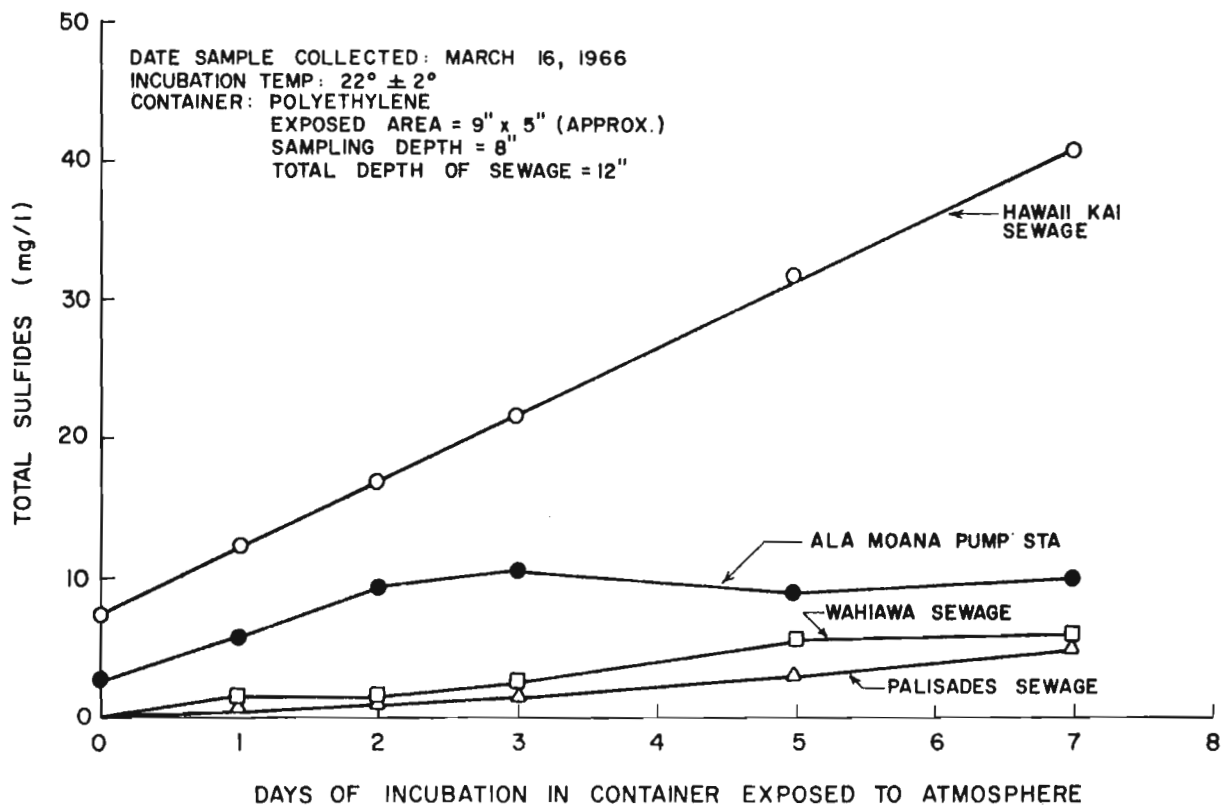


FIGURE 4: TOTAL SULFIDE GENERATION FROM SELECTED SEWAGE SAMPLES IN CONTAINERS EXPOSED TO THE ATMOSPHERE.

TABLE 6. SUMMARY OF RESULTS: SULFIDE GENERATION IN SEWAGE FROM SELECTED TREATMENT PLANTS AND INCUBATED IN OPEN CONTAINER.

TEMPERATURE $22^{\circ}\text{C} \pm 2^{\circ}$
 SAMPLE COLLECTED MARCH 16, 1966

DATE	DAY	HAWAII KAI	ALA MOANA PUMP STA.	WAHIAWA	PALISADES
mg/l TOTAL SULFIDES					
MARCH 16, 1966	0	7.2	2.6	0.0	0.0
17	1	12.2	5.6	1.5	0.4
18	2	17.0	9.1	1.5	1.0
19	3	21.6	10.4	2.5	1.5
21	5	31.8	9.0	5.3	3.0
23	7	40.7	10.0	6.0	5.0

the literature. The results of the laboratory sulfide generation study are plotted in Figure 4 and tabulated in Table 6.

As expected, Hawaii Kai Sewage and Ala Moana sewage yielded higher sulfide values than samples from Wahiawa and Pacific Palisades. Since the samples were exposed to the atmosphere, some oxidation of sulfides was expected. The white precipitates in incubated samples were consistent with reports of similar observations by others (Zobell, 1963).

Although a quantitative estimate of the significance of sulfide oxidation was not possible here, relative laboratory rates of sulfide generation under lysimeter ponding conditions with different sewage samples were demonstrated. Inspection of plotted results of various samples (Figure 4) indicated significant and linear increase for Hawaii Kai sewage while Ala Moana sewage samples showed a steady state condition after 2 days of incubation. Results from these two samples when compared with results from Wahiawa and Palisades sewage samples demonstrated that sulfates in sewage from sea water infiltration was a significant factor in increased sulfide generation rates and in maximum sulfide concentrations.

Additional and detailed laboratory study of sulfide generation was performed for Palisades sewage and Hawaii Kai sewage. Sample incubation was performed by a technique used in the single dilution method of BOD by Orlorb, *et al.* (1955) to prevent sample exposure to air and to insure anaerobic conditions in the incubated samples. Sample withdrawal for analysis was conveniently accomplished by this technique. Principal parameters observed were total sulfides, total organic carbon (TOC), sulfates, ORP, and pH. The results of this study are plotted in Figures 5, 6, and 7, and the data appear in Tables 2 to 12.

Palisades Settled Sewage. Sulfide generation from settled Palisades sewage (Figure 5) approached a steady state condition at a concentration of 12 mg/l after 7 days of incubation. Corresponding Palisades settled sewage with added sulfates continued to increase in sulfides up to 20 mg/l total sulfides with 11 days of incubation. Continued increase in sulfides was observed for the sulfate-added samples which indicates that 12 mg/l for settled Palisades sewage was the concentra-

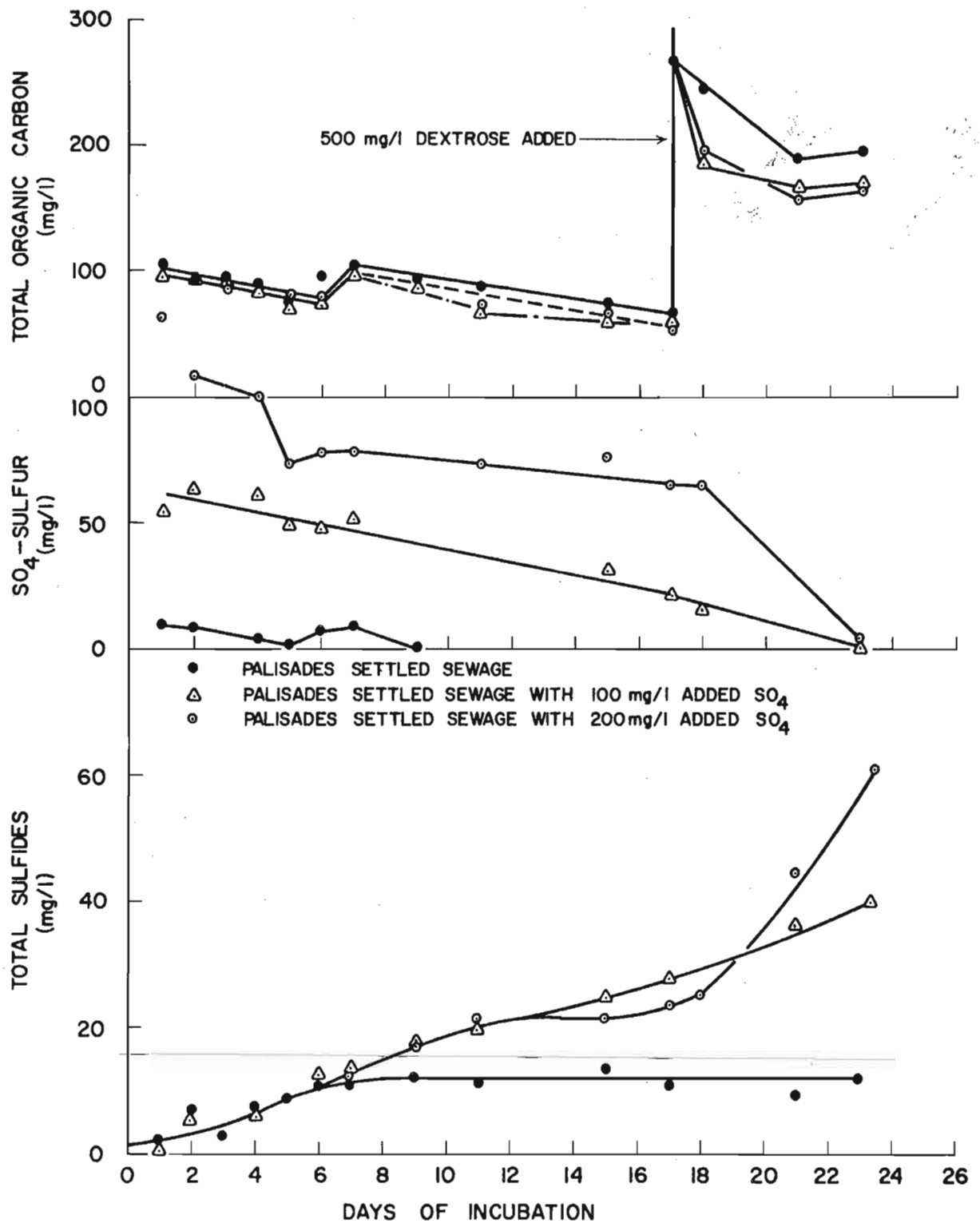


FIGURE 5: SULFIDE GENERATION FROM PALISADES SETTLED SEWAGE WITH DECREASES IN CORRESPONDING SULFATE - SULFUR AND TOC CONCENTRATIONS.

tion to be expected from settled sewage alone with additional increases from sulfate reduction to sulfides.

The sulfate and sulfide data for a 200 mg/l sulfate-added sample showed steady state conditions in sulfide generation rates from 11 to 17 days. On the 17th day, 500 mg/l dextrose was added to the sample. A substantial increase in sulfide generation resulted, suggesting that the organic matter was the limiting factor in sulfide generation. The TOC at the 17-day incubation time was 54 mg/l.

The 100 mg/l sulfate-added sample, although obtained from the same plant sample, did not behave similarly. In contrast, sulfate and sulfide data indicated that sulfide generation was not limited by organic matter at 59 mg/l TOC, and the data did not show a significant increase in sulfides with the addition of 500 mg/l dextrose to the sample.

Palisades Treated Sewage. The Palisades secondary effluent (Figure 6) with 75 mg/l TOC resulted in sulfide generation up to approximately 9 mg/l at 9 days of incubation. Addition of sulfates to the Palisades sample did not result in greater sulfide generation until the 15th day. The addition of 500 mg/l dextrose resulted in a significant increase in sulfides indicating that organic matter was a limiting factor.

One sample, exposed to the atmosphere during the study after leakage from the incubation bottle, left an insufficient volume to keep the bottle full. Tests were continued in this exposed condition to represent sulfide generation under conditions of aeration by diffusion. The data obtained indicated that exposure of treated sewage sample to the atmosphere resulted in inhibition of sulfide accumulation. Near-constant sulfate concentrations in the sample during the study indicated that ~~sulfates were not being reduced and that anaerobiosis (ORP -125 mv to -160 mv)~~ was probably significant in limiting activity of sulfate reducers in the sample.

Hawaii Kai Sewage. The data from Hawaii Kai sewage samples (Figure 7) indicated high initial sulfates from sea water infiltration into sewers. Two samples, primary effluent and raw sewage, showed marked differences, however, in initial sulfate values. This difference was considered to

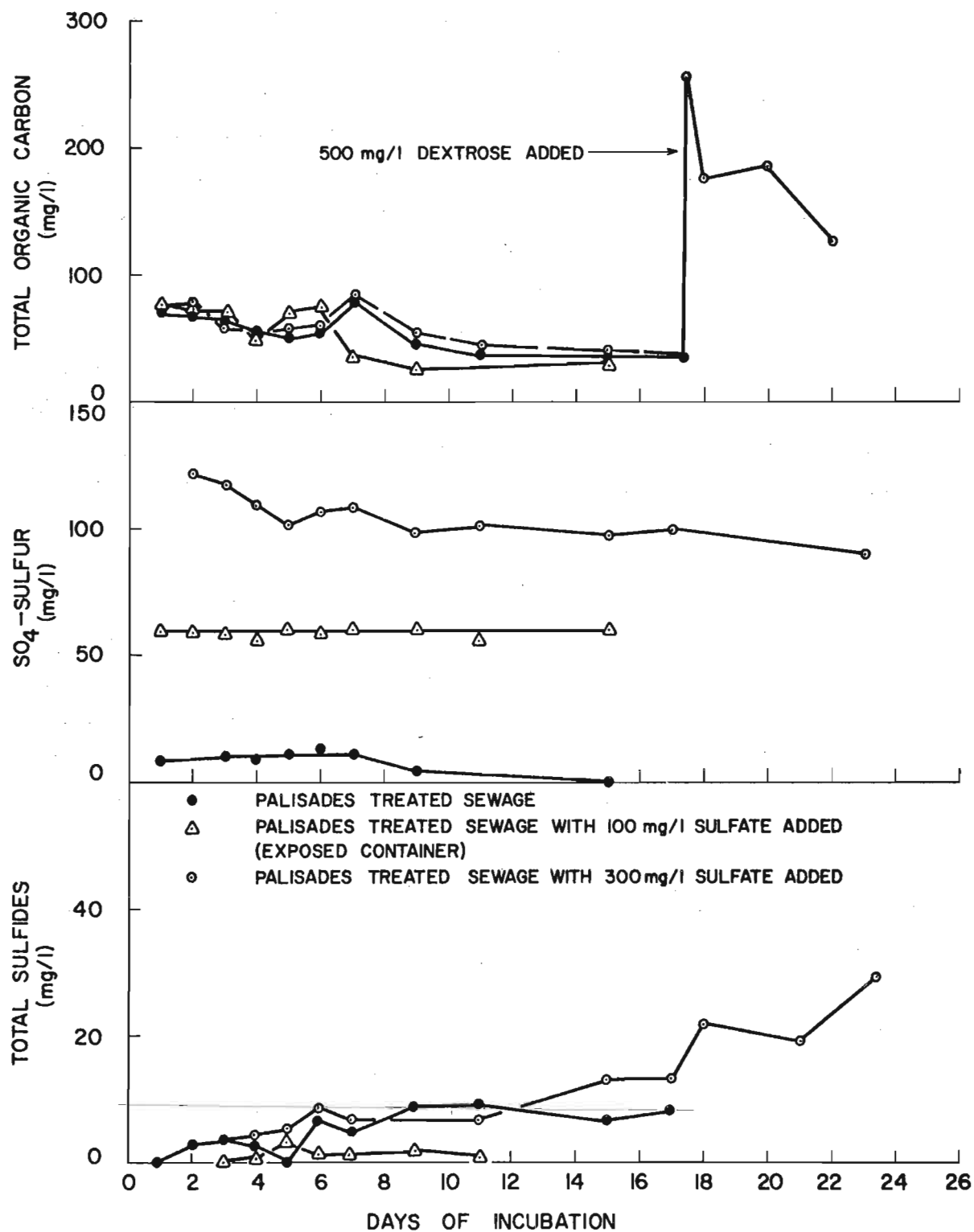


FIGURE 6: SULFIDE GENERATION FROM PALISADES TREATED SEWAGE WITH CORRESPONDING SULFATE-SULFUR AND TOC CONCENTRATIONS.

TABLE 7. SUMMARY OF RESULTS: PALISADES SETTLED SEWAGE.

DATE	DAY	TOTAL SULFIDES mg/l	TOTAL CARBON (TC) mg/l	TOTAL ORGANIC CARBON (TOC) mg/l	SULFATE mg/l	SO ₄ - S mg/l	ORP (mv)	pH
JULY	20	0	-	-	-	-	-	-
	21	1	2.0	-	105	30	-200	6.5
	22	2	6.4	110	92	27	-200	6.8
	23	3	2.8	134	93	ND	-200	6.8
	24	4	7.0	129	90	11	-210	6.9
	25	5	8.8	125	74	4	-	-
	26	6	11.2	123	98	22	-	-
	27	7	10.4	131	104	27	-200	6.8
	29	9	12.0	126	92	0	-220	-
AUG	1	11	11.2	125	77	0	-225	6.8
	5	15	13.2	119	75	0	-265	6.9
	7	17	10.8	123	66	0	-330	6.7
500 mg/l DEXTROSE ADDED								
AUG	8	18	-	246	214	0	-275	5.2
	11	21	9.2	292	190	0	-300	4.8
	13	23	11.6	224	194	0	-300	5.3

TABLE 8. SUMMARY OF RESULTS: PALISADES SETTLED SEWAGE
WITH 100 mg/l SO₄ ADDED.

DATE	DAY	TOTAL SULFIDES mg/l	TOTAL CARBON (TC) mg/l	TOTAL ORGANIC CARBON (TOC) mg/l	SULFATE mg/l	SO ₄ - S mg/l	ORP (mv)	pH
JULY	20	0	-	-	-	-	-	-
	21	1	1.6	-	164	55	-215	6.7
	22	2	4.8	109	191	64	-215	6.6
	23	3	3.2	132	94	ND	-205	6.9
	24	4	6.0	129	84	158	-225	6.6
	25	5	9.6	122	74	147	-	-
	26	6	12.4	116	75	142	-	-
	27	7	13.6	124	99	153	-	-
	29	9	17.2	120	87	ND	-250	-
AUG	1	11	19.6	120	68	ND	-250	6.8
	5	15	24.4	119	60	93	-265	7.0
	7	17	26.8	121	59	62	-375	6.9
500 mg/l DEXTROSE ADDED								
AUG	8	18	-	220	182	46	-330	5.5
	11	21	36.0	336	166	ND	-350	5.6
	13	23	40.0	214	170	0	-350	5.9

TABLE 9. SUMMARY OF RESULTS: PALISADES SETTLED SEWAGE
WITH 200 mg/l SO₄ ADDED.

DATE	DAY	TOTAL SULFIDES mg/l	TOTAL CARBON (TC) mg/l	TOTAL ORGANIC CARBON (TOC) mg/l	SULFATE mg/l	SO ₄ - S mg/l	ORP (mv)	pH
JULY	20	0	-	-	-	-	-	-
	21	1	1.2	-	181	60	-220	6.9
	22	2	4.8	106	327	109	-225	6.9
	23	3	4.0	129	88	300	-225	6.7
	24	4	7.0	127	88	ND	-205	6.8
	25	5	9.2	129	81	223	-220	6.5
	26	6	12.0	119	79	234	-	-
	27	7	13.2	120	97	235	-	-
	29	9	17.2	127	ND	ND	-300	-
AUG	1	11	20.4	121	71	218	-290	6.6
	5	15	21.6	121	63	229	-295	6.9
	7	17	22.8	130	54	195	-350	6.7
500 mg/l DEXTROSE ADDED								
AUG	8	18	25.6	229	195	65	-300	5.4
	11	21	44.2	364	158	ND	-350	5.7
	13	23	61.3	246	166	8	-325	5.6

TABLE 10. SUMMARY OF RESULTS: PALISADES TREATED SEWAGE.

DATE	DAY	TOTAL SULFIDES mg/l	TOTAL CARBON (TC) mg/l	TOTAL ORGANIC CARBON (TOC) mg/l	SULFATE mg/l	SO ₄ - S mg/l	ORP (mv)	pH
JULY	20	0	-	-	-	-	-	-
	21	1	0	70	-	7	-210	7.0
	22	2	3.2	69	-	-	-245	7.3
	23	3	4.0	65	-	10	-225	7.0
	24	4	3.0	55	-	9	-200	6.7
	25	5	0.0	50	-	11	-	-
	26	6	6.8	53	-	13	-	-
	27	7	5.2	80	-	11	-250	6.6
	29	9	8.4	54	-	4	-250	-
AUG	1	11	9.2	36	1	0	-275	7.0
	5	15	6.4	36	5	1	-250	7.0
	7	17	8.4	33	0	0	-355	6.5

TABLE 11. SUMMARY OF RESULTS: PALISADES TREATED SEWAGE WITH 100 mg/l SULFATE ADDED.
(SAMPLE EXPOSED TO ATMOSPHERE.)

DATE	DAY	TOTAL SULFIDES mg/l	TOTAL CARBON (TC) mg/l	TOTAL ORGANIC CARBON (TOC) mg/l	SO ₄ - S mg/l	ORP (mv)	pH
JULY	20	0	-	-	-	-	-
	21	1	0.4	75	59	-200	6.9
	22	2	3.2	70	59	-225	7.2
	23	3	0	68	58	-200	6.9
	24	4	1.0	50	56	-145	6.7
	25	5	3.6	52	60	-130	6.4
	26	6	1.2	60	58	-	-
	27	7	1.6	65	60	-	-
	29	9	2.0	35	60	-125	-
AUG	1	11	1.2	25	56	-150	6.8
	5	15	-	31	60	-160	6.8

TABLE 12. SUMMARY OF RESULTS: PALISADES TREATED SEWAGE WITH 300 mg/l SULFATE ADDED.

DATE	DAY	TOTAL SULFIDES mg/l	TOTAL CARBON (TC) mg/l	TOTAL ORGANIC CARBON (TOC) mg/l	SO ₄ - S mg/l	ORP (mv)	pH
JULY	20	0	-	-	-	-	-
	21	1	0	75	-	-200	6.8
	22	2	2.8	79	121	-225	6.9
	23	3	0.8	59	117	-225	6.9
	24	4	4.0	51	109	-220	7.0
	25	5	5.2	56	101	-250	6.7
	26	6	8.4	59	107	-	-
	27	7	6.4	81	109	-	-
	29	9	8.4	54	98	-250	-
AUG	1	11	6.8	42	101	-275	6.9
	5	15	12.8	40	98	-255	7.1
	7	17	13.6	33	100	-	-
500 mg/l DEXTROSE ADDED							
AUG	8	18	22.0	178	-	-350	6.7
	11	21	19.6	188	-	-250	5.9
	13	23	29.6	128	90	-265	4.5

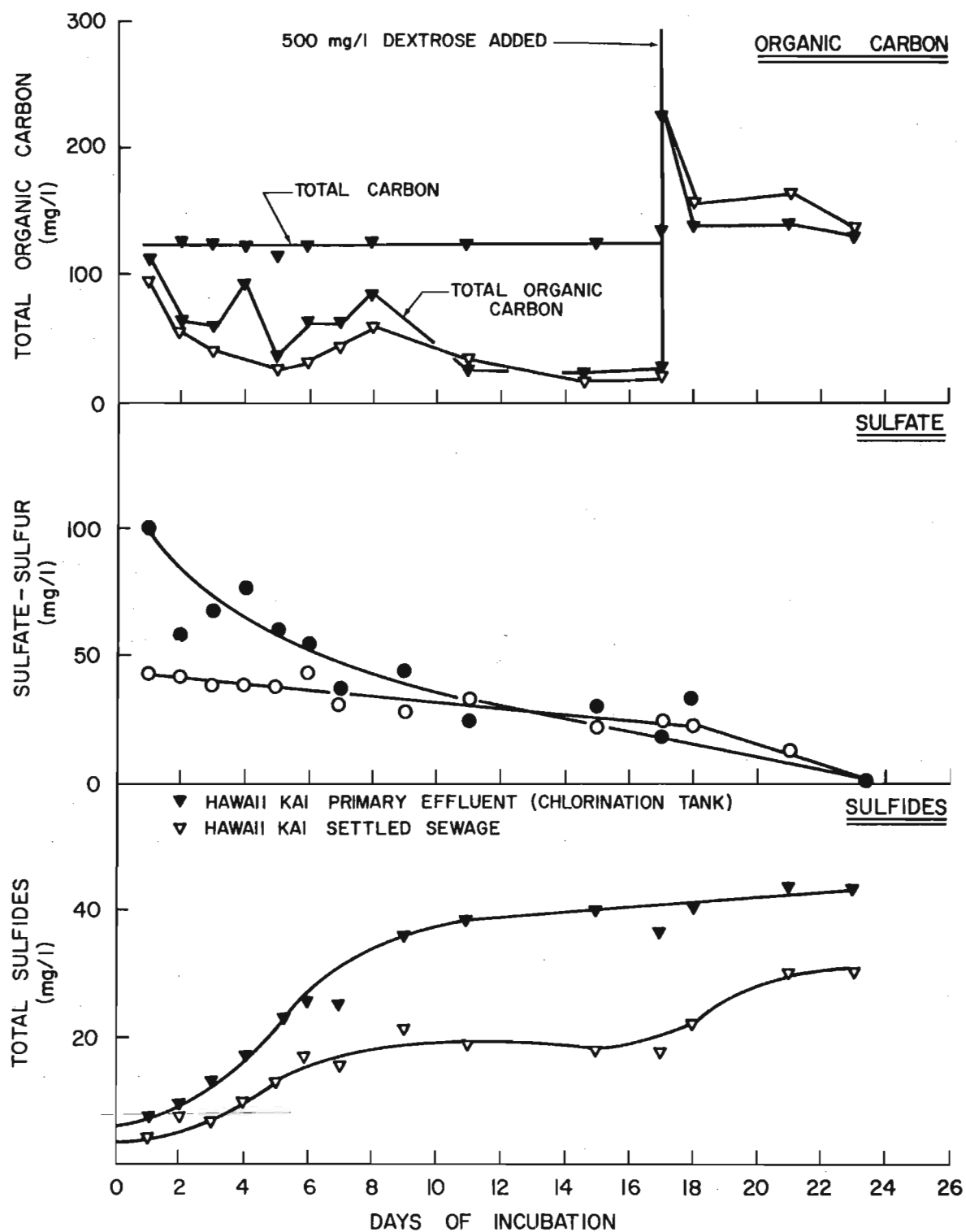


FIGURE 7: SULFIDE GENERATION FROM HAWAII KAI SEWAGE WITH CORRESPONDING DECREASES IN SULFATE SULFUR AND TOC CONCENTRATION.

be reasonable because of large detention times through the plant which was not yet operating at design capacity. Sulfide generation rates exhibited typical logarithmic increase curves often associated with biological oxygen uptake and biological growth.

The Hawaii Kai primary effluent sample showed increases in total sulfide concentrations up to approximately 40 mg/l after 14 days of incubation. Sulfate value decreases paralleled sulfide generation. Addition of 500 mg/l dextrose did not result in an increase in sulfide generation rates, indicating that sulfate concentration was a limiting factor.

Hawaii Kai settled sewage yielded lower steady state sulfide concentration. When compared to primary effluent sulfides, the ratio of initial sulfates (0.42) was in close agreement to the ratio of corresponding steady state sulfides (0.45). The addition of 500 mg/l dextrose to a settled sewage sample resulted in a significant increase in sulfides, indicating that organic matter was the limiting factor. This estimate, however, was not consistent with settled sewage data which was nearly comparable in TOC.

Comparison of Sulfide Generation Rates between Hawaii Kai and Palisades Samples. Sulfide generation rates and maximum sulfide concentrations observed depended on TOC and sulfate concentrations. The significance of microbial inoculum and acclimation was evident by comparison of results from Palisades sewage and Hawaii Kai sewage samples.

Initial rates of sulfide generation between Palisades sewage (sulfate added) and Hawaii Kai primary effluent sample indicated that although both samples contained similar concentrations of sulfates and TOC, the ten-day data showed sulfides of nearly 40 mg/l for the Hawaii Kai sample as compared to sulfides of nearly 20 mg/l for the Palisades sample. This difference in rates of generation can be explained by a difference in an active sulfate reducing microbial population present in the samples. Further, the distinct exponential increase in generation rate of sulfides for Hawaii Kai samples in contrast to the nearly linear increase in generation rate for Palisades sewage samples was consistent with the estimate that rate of sulfide generation was dependent on acclimated microbial seeding as well. As a result, data

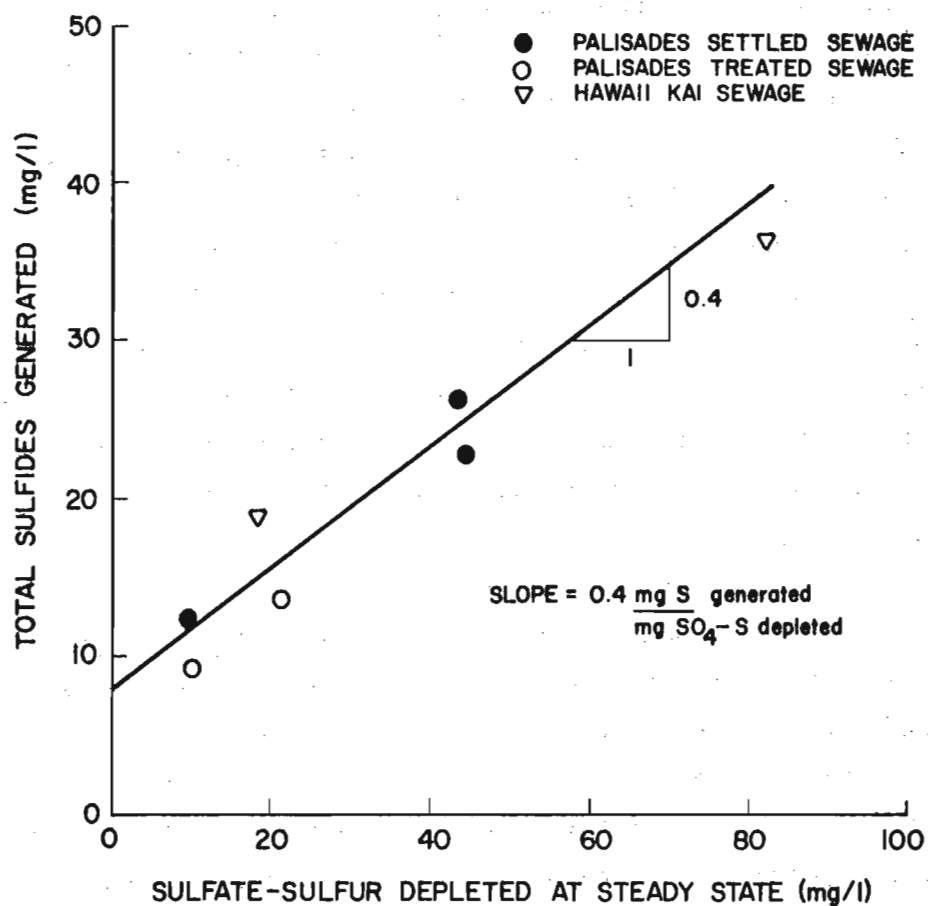


FIGURE 8: TOTAL SULFIDES GENERATED VS. TOTAL SULFATE-SULFUR DEPLETED AT STEADY STATE GENERATION RATE FOR PALISADES, AND HAWAII KAI SAMPLES.

from the Hawaii Kai sewage samples provide a more realistic basis for estimating sulfide generation rates from sulfate reduction under field condition.

Limiting concentration of TOC in sulfate reduction by sulfate reducing bacteria was not determined for this report, but it was estimated from data obtained that this value was 30 mg/l, or less. However, relationship between total sulfides found at steady state condition and depletion of sulfates is shown in Figure 8, and the data are summarized in Table 10. The average amount of sulfide generated per milligram of sulfate-sulfur depleted is represented by the slope of the line in Figure 8; and the slope was determined graphically to be approximately 0.4 milligram total sulfide per milligram sulfate-sulfur depleted in the sample. This value indicates that reduction of sulfate-sulfur to sulfides is not 100%, but a fraction of this amount. Results shown in Figure 8 demonstrate that a definite correlation exists between sample sulfates and total sulfides generated in the sample under anaerobic incubation.

Comparison of field sulfide samples with laboratory results. Selected field grab samples were analyzed for concentrations of sulfides found under field conditions where organics and sulfates were considered to be significant. Further, data obtained from samples subjected to varying field conditions of time, temperature, mixing, and concentration of organics and sulfates were felt to be necessary for adequate documentation of sulfide concentrations. Grab samples from selected cesspools requiring frequent pumping were characterized (Table 16). In addition, samples from Kapalama stream, an estuary, along with Hawaii Kai treatment Plant sedimentation tank samples were analyzed for total sulfides (Table 17). ~~All samples with possible exceptions of upper-valley cesspool samples~~ were estimated to contain sufficient organics and sulfates from sea water to produce significantly high sulfide concentrations under anaerobic conditions. Field incubation times for cesspool samples and Hawaii Kai sedimentation tank samples were considered to be far less than laboratory incubation times.

Kapalama stream samples receive waste waters from pineapple canneries which contribute to organic concentration and together with the

estuarine location explain the significant sulfate concentration. Effective time of incubation however, was considered to be far in excess of incubation times for laboratory samples, and the stream bottom samples from the Kapalama stream indicated significant accumulation of total sulfides. Field samples analyzed indicated that laboratory results were reasonable, and with time even greater concentrations than found in the laboratory may result.

Effect of Aeration of Anaerobically Incubated Samples. Anaerobically incubated sewage samples containing significant sulfides produced a milky white precipitate after exposure to the atmosphere. Samples collected in 200-ml beakers developed noticeable white precipitates within 5 minutes of exposure, and precipitate accumulation became more pronounced with time. Two-liter samples developed a white scum layer at the liquid surface upon standing for periods greater than one day. These results indicated that sulfides were being readily oxidized, probably to colloidal sulfur (Zobell, 1963).

The effect of bubble aeration was studied with Hawaii Kai sewage. Aeration was performed with a gallon-size sample. Air flow rate was measured with a rotameter and the exhaust air was bubbled through zinc acetate traps to precipitate sulfides volatilized during sample aeration. The results of this test are plotted in Figure 9.

Total sulfides remaining were determined by withdrawing samples at selected intervals and analyzing for sulfides. The zinc acetate trap was removed for analysis and replaced with fresh solution. Two-hour aeration at 200 ml/min/gal was sufficient to reduce total sulfides from a concentration of 40 mg/l to 1 mg/l. Approximately one-half of the total sulfides were volatilized and the other one-half was apparently oxidized. ~~Milky white precipitates were observed almost immediately upon aeration.~~

Aeration of Black Precipitates. The effect of aeration of black precipitates found in incubated samples was qualitatively described and observations are summarized in Table 13. The black precipitates were shaken loose from the bottom of the sample container and a spatula-full was placed in aeration tubes and diluted to 100 ml with distilled water. Aeration was begun and the appearance of the solution observed. Obser-

vations indicated that black precipitates were oxidized but at a much slower rate than for total sulfides in solution. Approximately 5 hours of aeration was necessary for nearly complete oxidation as judged by disappearance of the black color. A similar observation was made in experiments with digester sludge.

Reduction of Total Organic Carbon with Incubation. Reduction in TOC with incubation of settled sewage as summarized in Table 10 indicated that TOC reduction (Figures 5, 6, and 7) was generally poor for Palisades sewage samples as compared to Hawaii Kai sewage. TOC reduction was 32 to 38% at 17 days of incubation for Palisades settled sewage while 56% reduction was observed for secondary effluent after the same period of incubation. In contrast, Hawaii Kai sewage samples consistent with data reported in the literature showed better TOC reduction with removal of 75 to 79% in 17 days. Of significance, however, was the improved efficiency of TOC reduction for Hawaii Kai samples containing active populations of sulfate reducers. The improvement coincided with reports suggesting that sulfate reducers were responsible in satisfying BOD in addition to sulfate reduction (Zobell, 1963).

Results of Organic Characterization of Sewage Samples. Sewage and cesspool grab samples were included in this study to obtain an estimate of sulfide concentration found in sewage samples from various locations. Three different parameters for organics in sewage were used to demonstrate their inter-relationship.

Domestic sewage was collected from selected Oahu sewerage and compared with the results of cesspool grab samples which represented individual household wastes. Results of this organic characterization are summarized in Table 19 and are also expressed as BOD/COD, COD/TOC, and BOD/TOC ratios. The BOD of settled sewage grab samples ranged from 96 to 405 mg/l while BOD range for treated sewage samples was 147 mg/l to 210 mg/l. The 147 mg/l value determined from an extended aeration plant effluent was believed to be due to nitrification. Corresponding COD values for treated sewage were 38 and 78 mg/l while the TOC was 17 and 22 mg/l, respectively. Cesspool BOD was spread over a range of 18 to 185 mg/l for three samples. However, BOD/COD, COD/TOC,

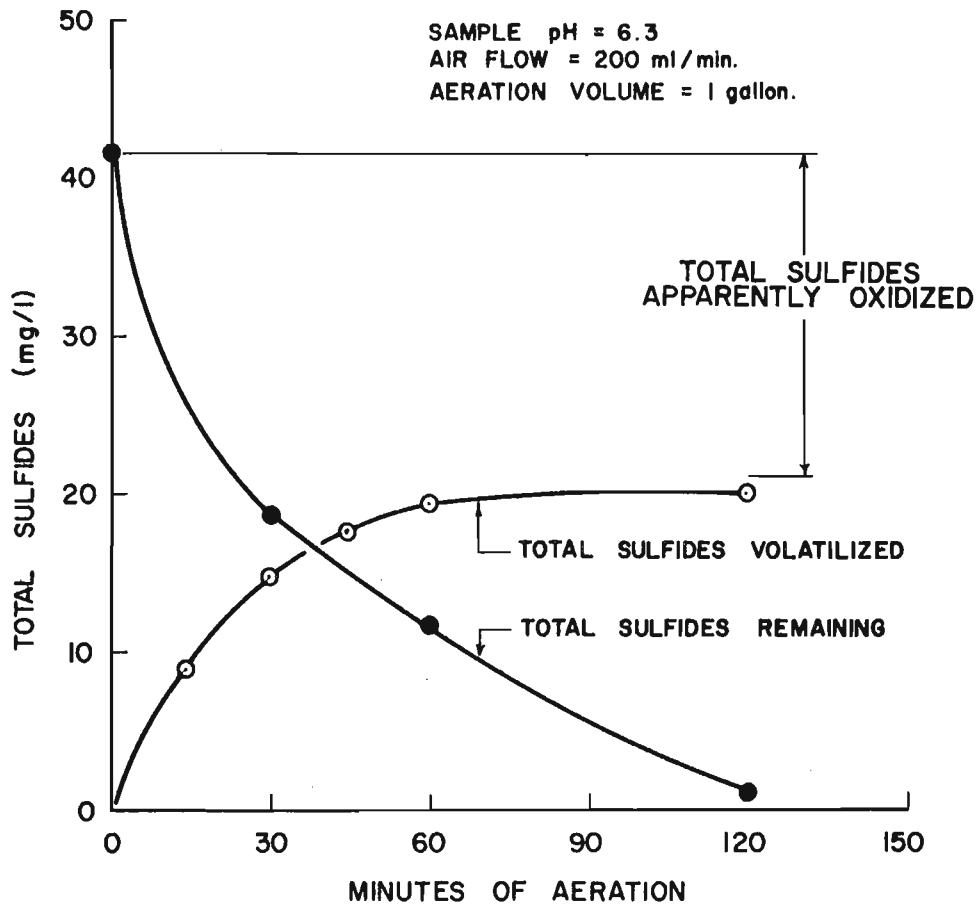


FIGURE 9: EFFECTS OF AERATION OF SULFIDE SAMPLES FROM HAWAII KAI SEWAGE.

TABLE 13. SUMMARY OF RESULTS: HAWAII KAI PRIMARY EFFLUENT

DATE	DAY	TOTAL SULFIDES mg/l	TOTAL CARBON (TC) mg/l	TOTAL ORGANIC CARBON (TOC) mg/l	SO ₄ - S mg/l	ORP (mv)	pH
JULY	20	0	-	-	-	-	-
	21	7.2	-	112	100	-285	6.7
	22	9.6	112	64	59	-280	6.8
	23	12.8	127	60	67	-225	6.9
	24	17.0	125	91	76	-300	6.9
	25	22.4	122	37	59	-	-
	26	25.6	119	62	53	-	-
	27	25.2	122	64	36	-	-
	29	36.8	127	85	44	-250	-
AUG	1	38.0	122	25	24	-325	6.9
	5	39.6	126	22	28	-310	7.1
	7	36.4	137	27	18	-400	7.0
500 mg/l DEXTROSE ADDED							
AUG	8	18	40.4	226	140	34	-325 5.9
	11	21	43.2	368	140	7	-350 5.8
	13	23	43.0	254	130	0	-340 5.9

TABLE 14. SUMMARY OF RESULTS: HAWAII KAI
RAW SEWAGE.

DATE	DAY	TOTAL SULFIDES mg/l	TOTAL CARBON (TC) mg/l	TOTAL ORGANIC CARBON (TOC) mg/l	SO ₄ - S mg/l	ORP (mv)	pH
JULY 20	0	-	-	-	-	-	-
	21	4.0	-	97	42	-280	7.1
	22	7.6	100	53	41	-275	7.1
	23	6.4	117	40	38	-275	7.1
	24	10.0	114	38	38	-260	7.2
	25	13.6	110	31	38	-	-
	26	18.4	109	44	44	-	-
	27	17.6	111	60	31	-	-
	29	20.8	108	32	29	-310	7.0
AUG	1	11	19.2	106	22	-350	7.1
	5	15	18.8	115	18	-340	7.3
	7	17	18.8	124	20	-375	7.0
500 mg/l DEXTROSE ADDED							
AUG	8	18	22.0	268	154	22	-300 5.6
	11	21	30.0	220	164	11	-350 5.6
	13	23	30.0	260	132	0	-275 5.9

TABLE 15. SUMMARY OF RESULTS OF SULFIDE
GENERATION AT STEADY STATE RATE
FOR PALISADES AND HAWAII KAI
SAMPLES.

SAMPLE	INITIAL SO ₄ - S (mg/l)	SO ₄ - S (mg/l)	INITIAL TOC (mg/l)	STEADY STATE TOC (mg/l)	TOC DEPLETED (mg/l)	% REDUCTION	TOTAL SULFIDES (mg/l)	PERIOD OF INCUBATION REPRESENTED BY DATA
PALISADES SETTLED SEWAGE	10	10	105	92	13	12	12.0	9
	64	43	95	59	36	38	26.8	17
	109	44	96	65	31	32	22.8	17
PALISADES TREATED SEWAGE	10	10	70	36	34	49	9.2	11
	121	21	75	33	42	56	13.6	17
HAWAII KAI								
PRIMARY	100	82	112	27	85	75	36.4	17
RAW	42	18	97	20	77	79	18.8	17

and BOD/TOC ratios of cesspool samples were consistent with values for domestic sewage samples and indicated that organic constituents of cesspool samples were similar to those of domestic sewage samples.

The Ala Moana pumping station sample indicated higher values of BOD, COD, and TOC than for other samples. Computed ratios indicated that BOD and TOC data were reasonable but the COD data was not. This suggested the presence of noncarbonaceous substances that interfered with the COD test. Of the three parameters for organic matter in sewage, the TOC was considered to be most reliable and rapid analytical measure.

Infiltration and Percolation Studies. Infiltration and percolation studies were performed on soil and quartz sand columns in parallel and transmission of sulfides and sewage organics observed. Hydraulic characteristics are documented in the Appendix.

Soil and Sand Characteristics. The soil used in the columns were samples obtained from the soil group characterized as Low Humic Latosol, with the clay fraction identified by X-ray diffraction as Kaolinite. Results of the soil analysis are shown in Table 20.

Quartz sand used for sand columns was relatively uniform with a size range of 0.4 to 0.8 mm.

**Percolation of Sewage Organics through the Soil Columns.* Column percolation study results are shown in Figures 10 and 11 and data summarized in Tables 16 and 17. The plots are specified as "percolate" and "column" data. Percolate data represented the effluent, and the column data represents anaerobic sewage ponded in the column to a depth of 4.5 ft.

Measures of organic matter percolating through the soil column (Figure 10) are indicated by BOD, COD, and TOC. Ready comparison is possible between the organic matter infiltrating (column data) and the organic matter percolating through the soil sample under anaerobic conditions. The difference between these two parameters is a measure of addition or removal of organics in the percolate. The data demonstrated that organic removals under anaerobic conditions were insigni-

* See Appendix for additional data (Tables 21 - 23).

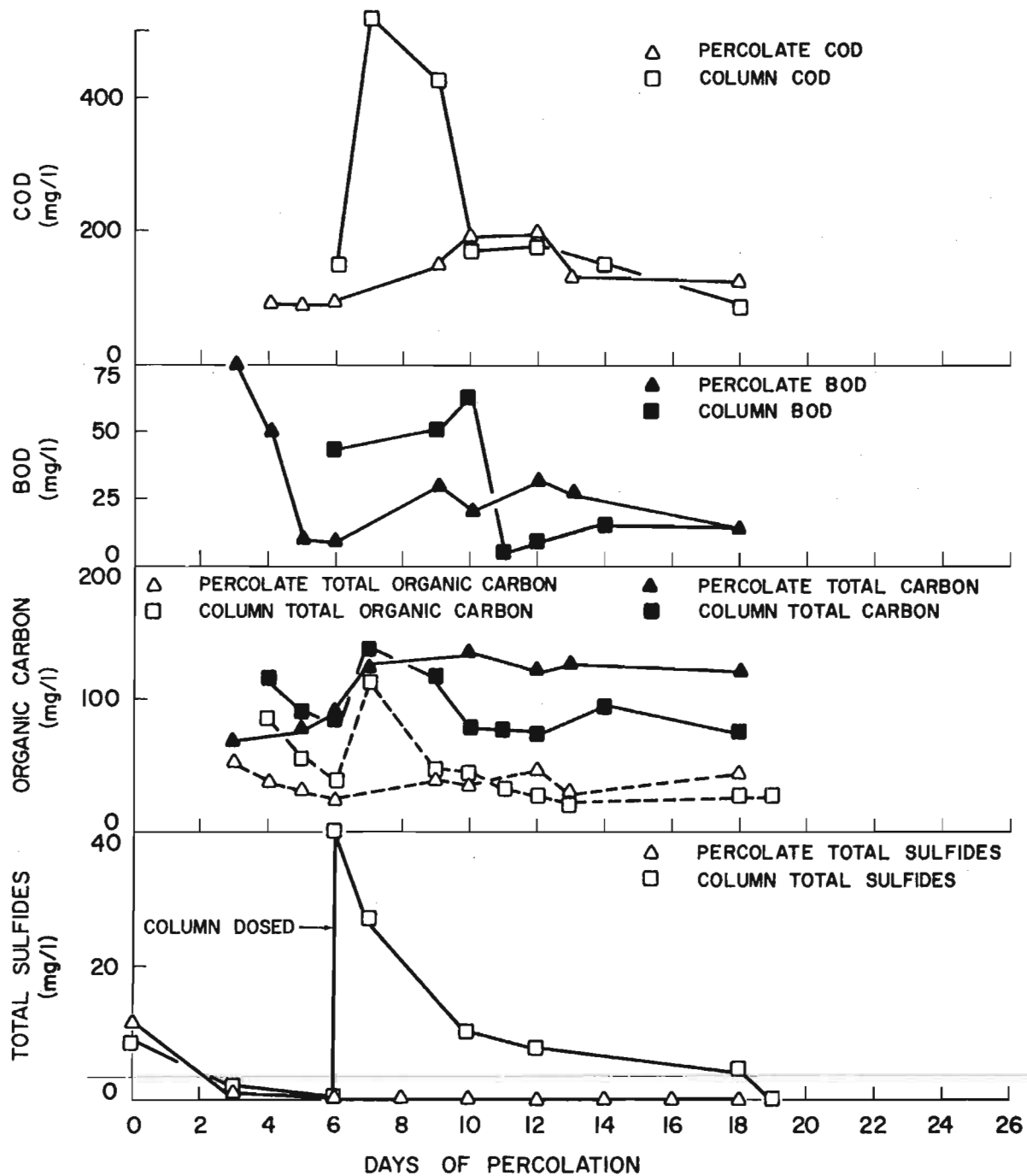


FIGURE 10: COLUMN AND PERCOLATE QUALITY FROM SOIL COLUMN PERCOLATION OF ANAEROBIC SEWAGE.

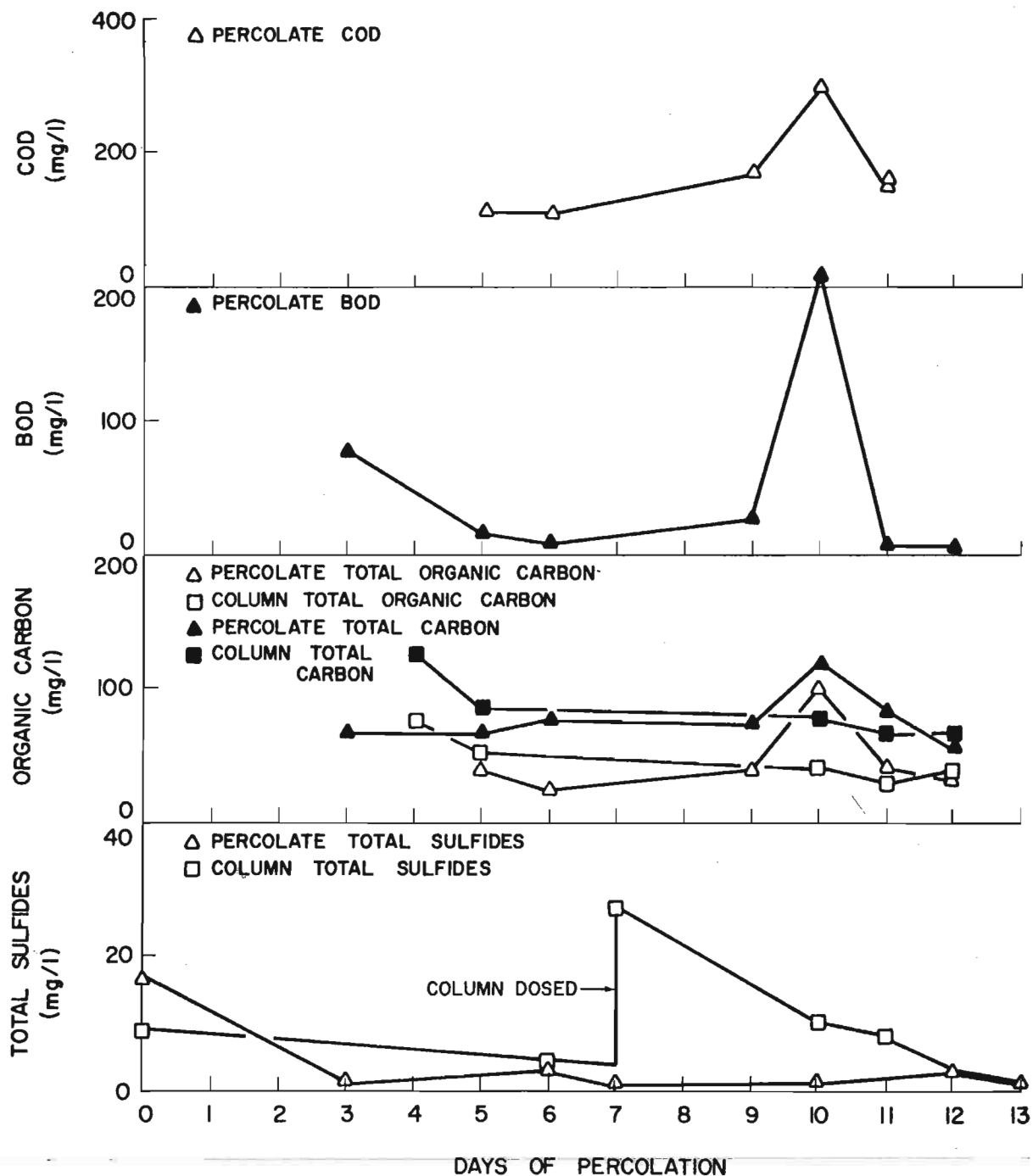


FIGURE II: COLUMN AND PERCOLATE QUALITY FROM QUARTZ SAND COLUMN PERCOLATION OF ANAEROBIC SEWAGE.

TABLE 16. SAMPLE OF ANALYSES ON
SELECTED CESSPOOL SAMPLES.

DATE OF SAMPLING: A.M. JULY 27, 1966.

LOCATION: NIU VALLEY

SAMPLE 1: BEACH FRONT PROPERTY.

SAMPLE 2: NEAR MIDDLE OF NIU VALLEY.

SAMPLE 3: NEAR UPPERMOST LOT OF NIU VALLEY.

SAMPLE	TOTAL SULFIDES** (mg/l)	ORP (mv)	pH	TOC mg/l	BOD mg/l	COD mg/l	REMARKS
1	6.0	>-400	6.4	112	185	595	PUMPED THE DAY BEFORE.
2	4.8	>-400	6.4	84	100	310	PUMPED DURING THE WEEK.
3	2.6	-360	6.6	40	18	85	PUMPED DURING THE WEEK.

**Sulfides average of bottom and middle depth samples.

TABLE 17. TOTAL SULFIDES FROM FIELD SAMPLES

NIU VALLEY CESSPOOLS		KAPALAMA STREAM		HAWAII KAI SEDIMENTATION TANK	
CESSPOOL NO.	TOTAL SULFIDES (mg/l)	DEPTH (ft)	TOTAL SULFIDES (mg/l)	DEPTH (ft)	TOTAL SULFIDES (mg/l)
1	6.0	1.3	3.0	1	17.7
2	4.8	2.3	2.1	2	19.1
3	2.6	3.3	104.0	5	19.1
		4.5	131.0	11	21.5
REMARKS		REMARKS		REMARKS	
SAMPLES OBTAINED JULY 27, 1966.		SAMPLES OBTAINED 1100, AUGUST 3, 1966. SAMPLED FROM CENTER OF RAILROAD BRIDGE NEAR NIMITZ HIGHWAY. NO OFFENSIVE ODORS. SAMPLES OBTAINED WITH D.O. SAMPLER.		SAMPLES OBTAINED 1250, AUGUST 2, 1966. LOCATION 15' FROM WEIR END OF SEDIMENTATION TANK. SAMPLE COLLECTED WITH D.O. SAMPLER.	
SAMPLE 1: BEACH FRONT PROPERTY PUMPED THE DAY BEFORE.		BLACK PRECIPITATES IN TWO BOTTOM SAMPLES.			
SAMPLE 2: MID-VALLEY LOT. LAST PUMPED WITHIN WEEK.					
SAMPLE 3: UPPER VALLEY LOT. LAST PUMPED WITHIN WEEK.					

ficant and BOD data indicated, instead, an increase in organics during percolation. The TOC data was in closer agreement with the COD than with the BOD data.

Total carbon (TC) increased in concentration with percolation (Figure 10), reflecting increases in alkalinity in passage through the column.

Percolation and retention of sulfides. Total sulfide data from the soil column (Figure 10) indicated that the soil column was effective in removing sulfides from the percolate while the sand column (Figure 11) allowed breakthrough of sufficient sulfides to create an odorous percolate. Breakthrough of sulfides was observed, however, during the initial run for the soil column. The reason for the initial breakthrough of sulfides was not immediately apparent. It was speculated that acid conditions with a pH 5.0 were significant in dictating the proportion of soluble sulfides and metallic precipitates of sulfides to percolate. It was reasonable to assume that a greater portion of soluble sulfides was present and an acid pH was a favorable condition for sulfide breakthrough. To gain greater insight into the problem of pH, percolation was carried out at an acid pH as well.

The quartz sand column, in contrast to the action in the soil column, showed progressive movement of a black layer through the sand bed until the entire sand bed was black. Breakthrough of sulfides in this column was continuous throughout the study. The penetration of the black precipitates into the sand bed demonstrated that the filtering action of the precipitates was not as effective as in the soil column. The relatively inert and large grained (0.45 - 0.8 mm) sand selected for this study, evidently did not effect a complete removal of sulfides from the percolate. The observation that a black precipitate layer was retained on the soil surface indicated that sulfide removal was in part attributable to filtration of metallic sulfide precipitates. The sand column's partial retention of sulfide precipitates within the sand bed indicated the probable sorption or sedimentation in the pores while allowing breakthrough of total sulfides.

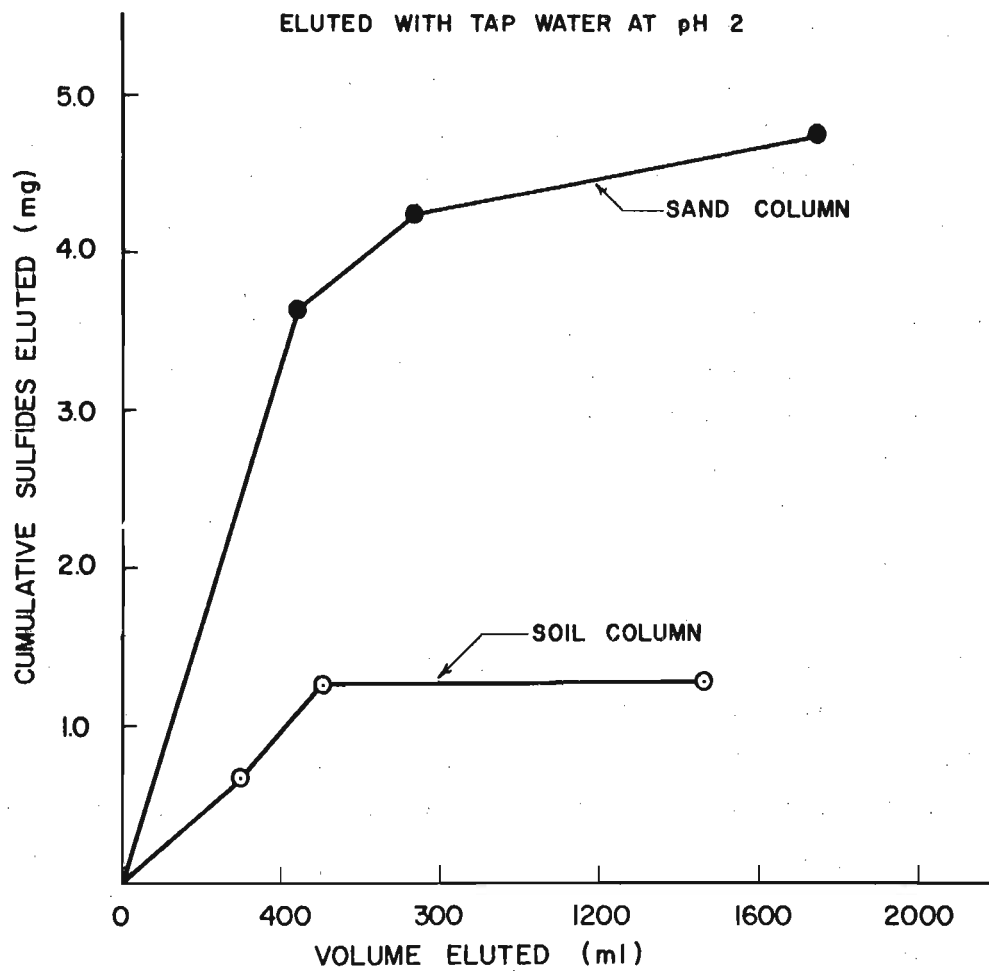


FIGURE 12: ELUTION OF SULFIDES FROM COLUMNS WITH ACIDIFIED TAP WATER.

TABLE 18. DESCRIPTION OF AERATION OF SETTLEABLE BLACK PRECIPITATES FROM ANAEROBIC INCUBATION OF SEWAGE.

HOURS OF AERATION	LIQUID COLOR	REMARKS
0	BLACK	PRECIPITATES DILUTED WITH DISTILLED WATER. RESULTING LIQUID BLACK.
0.50	BLACK	FOAMING ADDED OUT FOAM SPRAY. SPECKS OF GREY MATTER OBSERVED.
1.75	DARK GREY TO BLACK	LIQUID PORTION BLACK. PARTICULATE MATTER GRAY.
4.75	BROWN TO BLACK	BLACK AND BROWN PARTICULATE MATTER OBSERVED.
12.00	BROWN	NO BLACK PARTICULATE MATTER OBSERVED. BROWN SLURRY APPEARANCE.

TABLE 19. RESULTS OF BOD, COD, AND TOC OF SEWAGE GRAB SAMPLES FROM SELECTED TREATMENT PLANTS. (DATE OF SAMPLES: JULY 13, 1966.)

DESCRIPTION	TIME OF SAMPLE	BOD (mg/l)	COD (mg/l)	TOC (mg/l)	BOD/COD	COD/TOC	BOD/TOC
PALISADES SETTLED	1130	96	222	62	0.43	3.58	1.55
WAIPIO RAW	1200	162	392	82	0.41	4.78	1.98
WAIPIO TREATED	1200	147	38	17	3.87	2.23	-
WAHIAWA RAW	1220	150	377	68	0.40	5.53	2.21
WAHIAWA TREATED	1220	21	78	22	0.27	3.55	0.95
ALA MOANA	1030	405	1050	152	0.39	6.90	2.66
HAWAII KAI RAW	1400	72	207	49	0.35	4.23	1.47
<u>CESSPOOL SAMPLES**</u>							
1	A.M.	185	595	112	0.31	5.31	1.65
2	A.M.	100	310	84	0.32	3.69	1.19
3	A.M.	18	85	40	0.21	2.12	0.45

**Results from Table 11.

it yielded results which indicated that dissolved oxygen was partially depleted during elution of sulfides. Partial dissolved oxygen depletion suggested that only a part of the sulfides may have been oxidized during elution.

Percolation of acidified anaerobic sewage. Since sulfide percolation through soil under acid conditions was possible, further studies of percolation of sulfides were conducted with acidified anaerobic sewage. The acidity of anaerobic sewage samples was arbitrarily lowered to pH 2 by addition of concentrated sulfuric acid. Results, plotted in Figure 13 and tabulated in Table 25, demonstrate a sulfide breakthrough in soil under acid conditions. However, comparison of percolate and column sulfides indicate that a substantial portion was removed. As noted in previous tests, column sulfides decreased in concentration upon standing and after the second day of percolation. With the decrease in column sulfides, there was a corresponding decrease in percolate sulfides.

Upon completion of this study, the soil sample was removed from the soil column while still moist and analysis for sulfide retention started immediately. The results were negative for sulfides retained in the soil. The implication was that sulfides were either sorbed irreversibly or were involved in a chemical reaction which precluded measurement of sulfur as sulfide. The soil sample possessed a nauseating odor, but it was not characteristic of sulfides. The mechanism of sulfide removal, however, was not clearly understood.

Summary of column hydraulic data. The hydraulic performance of the soil columns are tabulated in the Appendix, and data from initial and final runs of tap water-loaded column (blank) and anaerobic sewage-loaded soil columns are plotted in Figure 14. The curves are numbered for discussion purposes and describe the column and order of run. Presentation of data from the first and final runs demonstrate significant hydraulic characteristics found throughout the laboratory study.

Deviation from Darcy's Law. All curves exhibited characteristic non-linear relationship between infiltration and percolation rates and hydraulic gradient. Similar occurrences were reported in the literature especially

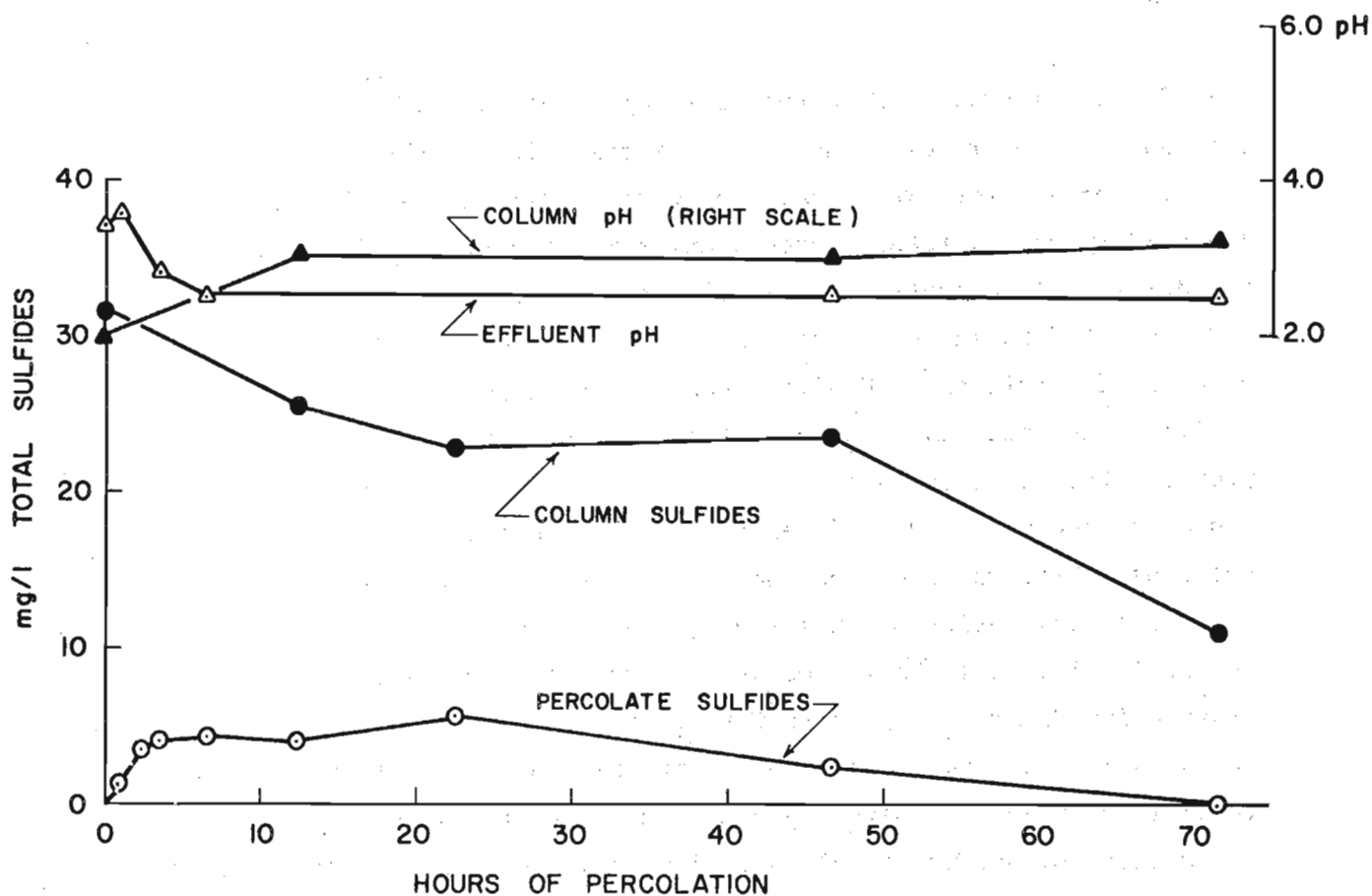


FIGURE 13: PERCOLATION OF SULFIDES FROM ACIDIFIED ANAEROBIC SEWAGE.

TABLE 20. ANALYSIS OF SOIL SAMPLES
(LOW HUMIC LATOSOLS) USED
IN PERCOLATION COLUMNS.

ITEM	QUANTITY	REMARKS & ANALYTICAL REFERENCES
ORGANIC MATTER (%)	1.16	WALKLEY, 1935
CLAY (%)	44.70	HYDROMETER METHOD
SILT (%)	50.40	
SAND	4.90	
FREE IRON (%)	14.53	KILMER, 1960
NATURE OF CLAY	KAOLINITE	X-RAY DIFFRACTION

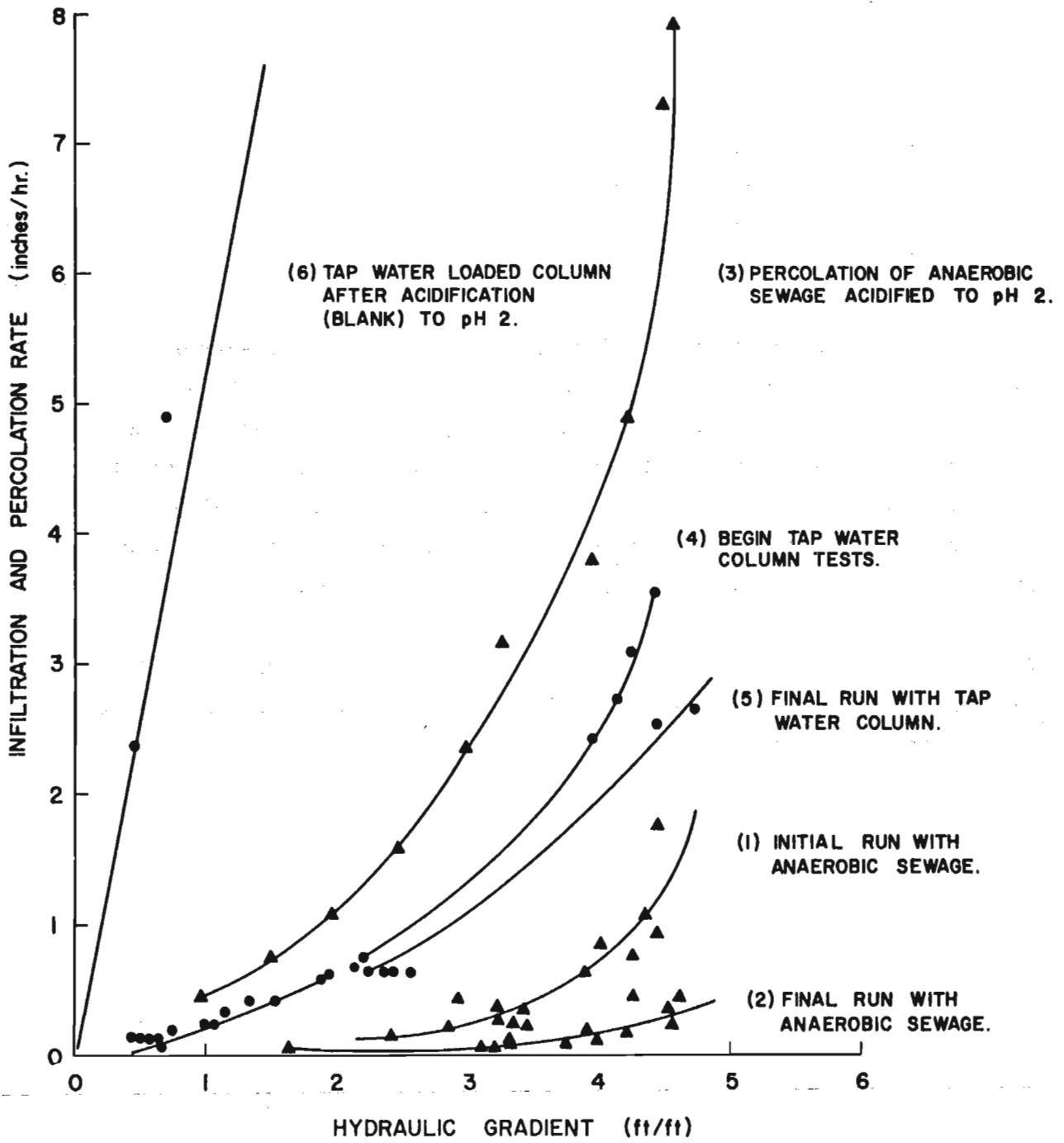


FIGURE 14: SUMMARY OF HYDRAULIC PERFORMANCE THROUGH SATURATED SOIL COLUMNS WITH TAP WATER AND ANAEROBIC SEWAGE LOADING.

for clayey soils. Results of this study can be described by the relationship by Hansbo (1960):

$$V = \lambda i^n$$

where V = velocity

i = hydraulic gradient

n = constant

λ = constant describing permeability

Clogging. Anaerobic sewage samples containing significant sulfides clogged the soil column (Curves 1 and 2, Figure 14) in less than 2 weeks of infiltration and percolation. The predominant factor contributing to early clogging was apparently an accumulation of sulfide precipitates as layers on the surface of the soil sample. The tap water column (Curves 4 and 5), however, behaved according to Darcy's Law at hydraulic gradients less than two, but deviated at higher gradients.

Improved flow rates after acidification. The flow rates were significantly improved after percolation of acidified fluids through tap water and sewage-loaded columns.

SUMMARY

Phase I of this study indicated need for further study due to the two significant observations made:

- (1) The odor in some percolate samples indicated that certain odorous compound percolated freely through the soil.
- (2) The excellent COD removals under presumably anaerobic conditions was not consistent with the reported performance of similar studies in the literature. The negative manometer pressures observed and the nitrate values (Koizumi, 1965) suggested that aerobic conditions prevailed in significant portions of the lysimeter soil mass.

In the sulfide generation study of Phase II, as expected the sewage collected from low-lying areas, Hawaii Kai and Ala Moana, yielded higher sulfide values than the samples from the highlands, Wahiawa and Pacific Palisades. This is due to the infiltration of seawater into the sewage since seawater contains significant concentrations of sulfates as a major anion.

In the 200 mg/l sulfate-added sample of Pacific Palisades settled raw sewage, organic matter was a limiting factor in sulfide generation. However, in the 100 mg/l sulfate-added sample, organic matter was not a limiting factor.

The treated sewage from Pacific Palisades indicated that organic matter was a limiting factor to sulfide generation. When exposed to the atmosphere, the incubated sewage did not reduce sulfates, indicating that anaerobiosis was probably limiting the activity of the sulfate reducers in the sample.

In the Hawaii Kai primary effluent sample, the sulfate values decreased with sulfide generation, with sulfate concentration being the limiting factor. On the other hand, the raw sewage obtained from this location was limited in its sulfide generation by the organic matter present.

The Hawaii Kai samples showed distinct exponential increase in generation rates of sulfides, in contrast to the nearly linear increase rate for the Pacific Palisades sewage.

The TOC concentration limiting sulfate reduction by sulfate

revealed that no detectable sulfides were retained in the soil, indicating that either (i) sulfides were completely desorbed under acidic condition, or (ii) a chemical reaction precluded the measurement of sulfur as sulfide. The removal mechanism was not clearly understood.

No significant removal of anaerobically treated sewage carbonaceous matter was observed during percolation through the soil and sand columns.

All curves exhibited the characteristic non-linear relationship between infiltration and percolation rates and the hydraulic gradient.

The predominant factor contributing to early clogging was the accumulation of precipitates as layers on the surface of the soil sample.

The tap water column behaved according to Darcy's law at hydraulic gradients less than 2, but deviated from Darcy's law at higher gradients.

Flow rates improved significantly after percolation of acidified fluids.

RECOMMENDATIONS

Percolation of significant amounts of pollutants through porous media demonstrates need for continuing study of controlling engineering parameters and their corresponding influence on organics-related water quality characteristics such as odor, taste, and toxicity. Although laboratory soil columns indicated that appreciable odor-significant sulfides were removed during percolation through Low Humic Latosols, the need for odor characterization exists. The nauseating odor imparted to soil samples during sulfide percolation, and the fact that soil samples were odorous without detectable sulfides suggests presence of other significant odorous compounds. Further, the relationship of the sulfide removal mechanism and the odorous sulfur compounds was not sufficiently understood to fully evaluate its significance to water quality. For these reasons, threshold odor determinations of percolating effluents are recommended to provide additional data necessary to realistically evaluate pollution possibilities of percolating sewage.

The results of this and other similar studies demonstrate percolation of soluble organics. The implications of this fact to water quality include the possibility of groundwater pollution from tastes and odor producing substances as well as from objectionable compounds which include the biocides used extensively in agricultural practice. Presence of sewage organics *per se* in water supplies is objectionable, yet the need for analyses to detect occurrence of these organics in groundwater supplies exists. Analyses are needed to ascertain possible groundwater contamination from sewage sources, to provide an early warning system of pollution from organics before they reach objectionable concentrations detectable as tastes and odors by consumers, and to monitor compounds considered injurious to public health. When taste and odor compounds, in particular, are detectable in groundwaters by consumers corrective measures are already too late. Groundwater movement and replenishment often occur in magnitudes of geologic times. Therefore, groundwater pollution can be considered an almost irreversible process. For these reasons, the development of procedures and facilities for microcontaminant analyses and continuing laboratory studies are especially significant in Hawaii where groundwater is the primary water resource.

Finally, implementation of recharge and ultimate ground disposal practices for sewage effluents should take cognizance of organic-sulfate-sulfide effects on water quality as well as recharge hydraulics which often dictate conditions for successful and economic performance of the recharge system.

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APPENDIX

TABLE 21. SUMMARY OF SOIL COLUMN DATA

PERCOLATE								COLUMN							REMARKS
DATE	DAY	pH	TC mg/l	TOC mg/l	COD mg/l	BOD mg/l	TOTAL SULFIDES mg/l	pH	TC mg/l	TOC mg/l	COD mg/l	BOD mg/l	TOTAL SULFIDES mg/l		
JULY	8	-	5.0	-	-	-	11.4	5.0	-	-	-	-	8.4	COLUMN #5	
	11	3	7.0	68	51	-	75	0.4	-	-	-	-	1.6		
	12	4	7.2	-	39	91	50	-	7.1	111	82	-	-	SOIL-SEWAGE	
	13	5	-	79	31	90	10	-	7.1	88	54	-	-		
	14	6	7.7	90	25	94	9	0	7.0	84	38	146	42	40.0	
	15	7	-	-	-	-	-	-	6.9	135	111	>460	-	27.4	
	17	9	8.1	122	40	155	30	0.0	7.0	115	47	424	50	-	
	18	10	8.1	134	67	186	20	0.0	7.0	77	41	174	63	10.0	
	19	11	-	-	-	-	-	-	7.1	77	31	-	5	-	
	20	12	8.0	120	46	180	33	0.0	7.2	72	29	198	9	8.0	
	21	13	8.2	124	39	132	27	0.0	7.3	-	-	-	-	-	
	22	14	-	-	-	-	-	-	7.0	93	22	151	15	-	
	26	18	8.0	120	43	123	14	0.0	7.0	74	26	94	14	4.4	
	27	19	-	-	-	-	-	-	7.0	67	26	-	10	0.0	

TABLE 22. SUMMARY OF QUARTZ SAND COLUMN DATA

PERCOLATE							COLUMN					REMARKS
DATE	DAY	TC mg/l	TOC mg/l	COD mg/l	BOD mg/l	TOTAL SULFIDES mg/l	TC mg/l	TOC mg/l	COD mg/l	BOD mg/l	TOTAL SULFIDES mg/l	
JULY 8	0	-	-	-	-	17.6	-	-	-	-	8.4	SETTLED SEWAGE
11	3	67	-	-	78	1.2	-	-	-	-	0.8	COLUMN #2
12	4	-	-	-	-	-	125	72	-	-	-	OTTOWA SAND
13	5	66	40	111	15	-	85	51	-	-	-	
14	6	73	25	111	8	3.2	-	-	-	-	4	
15	7	-	-	-	-	0.5	-	-	-	-	27.4	
17	9	72	40	173	29	-	-	-	-	-	-	
18	10	120	100	300	210	0.8	77	41	-	-	10	
19	11	82	41	144	6	-	68	30	180	5	8.0	
20	12	56	32	-	5	3.2	68	37	-	5	3.2	
21	13	-	-	-	-	1.3	-	-	-	-	1.0	

TABLE 23. SULFIDE ELUTED FROM SOIL AND SAND COLUMNS
WITH ACIDIFIED TAP WATER.TAP WATER ACIDIFIED TO pH₂ WITH CONCENTRATION H₂SO₄

SOIL COLUMN						SAND COLUMN					
DATE	TIME	VOL. ELUTED (ml)	SULFIDE CONC. ELUTED (mg/l)	CUMULATIVE VOLUME ELUTED (ml)	CUMULATIVE SULFIDE ELUTED (mg)	DATE	TIME	VOL. ELUTED (ml)	SULFIDE CONC. ELUTED (mg/l)	CUMULATIVE VOLUME ELUTED (ml)	CUMULATIVE SULFIDE ELUTED (mg)
JULY 28	2430	BEGIN TEST				JULY 28	2200	420	8.6	420	3.61
28	1000	300	2.2	300	0.66	29	0830	255	2.4	675	4.23
29	0830	248	2.4	548	1.26	30	1000	0.5	0.5	1675	4.73
30	1000		0.0	1548	1.26						

TABLE 24. SOME CHEMICAL CHARACTERISTICS
OF SOIL COLUMN PERCOLANT FROM
ACIDIFIED TAP WATER DURING SUL-
FIDE ELUTION.

PARAMETERS	SOIL COLUMN WITH SEWAGE DOSING	SOIL COLUMN WITH TAP WATER DOSING	SAND COLUMN WITH TAP WATER
COLOR	GRASS-GREEN	REDDISH BROWN	CLEAR
TOC mg/l	146.0	119.0	0.0
IRON mg/l Fe	52.0	40.0	0.5
pH	2.5	2.5	2.0
DISSOLVED OXYGEN* (mg/l)	4.0	ND	7.3

*Iron interference noted.

TABLE 25. TOTAL SULFIDES PERCOLATED WITH ACIDIFIED ANAEROBIC SEWAGE

DATE	TIME	HRS	EFFLUENT		INFFLUENT		CUMULATIVE VOLUME PERCOLATED (ml)
			pH	TOTAL SULFIDES mg/l	pH	TOTAL SULFIDES	
AUG 1	0955	0					
	1055	1	3.4	1.2	2.0	31.6	130
	1211	2.43	3.6	3.6	-	-	230
	1321	3.43	2.7	4.0	-	-	336
	1648	6.88	2.8	4.2	-	-	436
	2207	12.20	2.5	4.0	3.0	25.4	536
AUG 2	0830	22.58	-	5.5	-	22.8	666
3	0920	46.83	2.5	2.3	3.0	23.6	854
4	1000	71.50	2.5	0	3.2	11.0	954

