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IDENTIFYING AND CONTROLLING ODOR IN THE MUNICIPAL WASTEWATER ENVIRONMENT PHASE II:

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ABSTRACT

This project was undertaken in response to the wastewater treatment industry's need to better understand the generation of odors from biosolids produced by wastewater treatment plants (WWTPs). Its primary objective is to begin to establish relationships between WWTP process parameters and biosolids odors, so that more effective techniques for minimizing biosolids odors can be developed.

The project consisted of a detailed field study involving extensive sampling and analyses at 11 WWTPs across North America with capacities from 13 to 350 million gallons per day. Biosolids samples were collected from the WWTPs at a number of sampling points, which were chosen to represent a complete snapshot of biosolids generation and handling at each facility. The sampling points started with influent wastewater, proceeded through primary and secondary clarification, through digestion, dewatering, and onsite storage of dewatered biosolids cake.

Laboratory-scale anaerobic storage tests were conducted to simulate odor development of biosolids in storage prior to their beneficial reuse or disposal. A battery of analyses was performed on the biosolids samples by the participating utility laboratories, commercial laboratories, and specialized university laboratories. The analytical data were evaluated and compared with process and operation parameters at each participating WWTP.

Results indicate that the anaerobic digestion process, including its impacts on achieving stability and minimizing odors in the final biosolids product, are not yet completely understood. A significant finding was that biosolids odors after digestion and dewatering correlate with the amount of bio-available protein in the biosolids. Possible causes for increased bio-available protein and increased odor generation from dewatered biosolids begin with the primary and secondary sludge handling, mixing, and liquid storage steps, and continue through the anaerobic digestion process to post-digestion processes, such as dewatering, conveyance, and cake storage.

A list of future research needs that was developed based on the study findings centered on the need for more controlled experiments to identify and quantify the impacts of different biosolids handling and stabilization processes on biosolids odor generation.

BENEFITS AND KEYWORDS

Benefits:

- ◆ Helps the wastewater treatment industry understand and manage biosolids odor and its impacts on surrounding communities by understanding more completely the chain of events involved in the generation of biosolids odors.
- ◆ Identifies gaps in scientific knowledge regarding mechanisms of odor generation in WWTP biosolids.
- ◆ Shows that biosolids stability parameters may be misleading with respect to their impact on odors produced from biosolids.
- ◆ Provides a reference guide for the wastewater treatment industry and a starting point in identifying the causes of biosolids odors.
- ◆ Emphasizes the importance of whole plant management for reduction and control of biosolids odors.

Keywords: anaerobic digestion, biosolids, odors, olfactometry, wastewater, WWTP

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LIST OF ACRONYMS

Al	Aluminum
APHA	American Public Health Agency
ASTM	American Society for Testing and Materials
AVSR	Additional volatile solids reduction
BOD	Biological oxygen demand
BRC	Biosolids Recycling Center
°C	Degrees Celsius
Ca	Calcium
CEN	European Committee for Standardization
CLP	Contract Laboratory Program
CO ₂	Carbon dioxide
COS	Carbonylsulfide
CS	Combined sludge
CS ₂	Carbondisulfide
DAF	Dissolved air flotation
DAFT	Dissolved air flotation thickener
DMDS	Dimethyldisulfide
DMS	Dimethylsulfide
DQO	Data quality objectives
DS	Dry solids
DSWA	Damon S. Williams and Associates
DT	Detection Threshold
D/T	Dilutions-to-Threshold
EMC	Environmental Management Consulting
Fe	Iron
FID	Flame ionization detector
g	Grams
GBT	Gravity belt thickener
GC	Gas chromatograph
GC/MS	Gas chromatograph/mass spectrophotometer
H ₂ S	Hydrogen sulfide
HCl	Hydrochloric acid
HP	Hewlett Packard
HPLC	High Performance Liquid Chromatography
HT	Hedonic tone
I	Odor intensity
ICP	Inductively coupled plasma
ID	Internal diameter
K	Potassium
kg	Kilogram
L	Liter
LACSD	Los Angeles County Sanitation Districts

EXECUTIVE SUMMARY

ES.1 Introduction

The project summarized in this report, *Impacts of In-Plant Parameters on Biosolids Odor Quality*, represents Phase II of a larger project by the Water Environment Research Foundation (WERF) called *Identifying and Controlling Odor in the Municipal Wastewater Environment* (WERF 00-HHE-5). The project to date has been comprised of two major study phases. Phase I was a review of literature related to odors in the wastewater industry and has been published separately. Phase II was a field and laboratory study of plant parameters related to odors from biosolids and is the subject of this report.

ES.2 Objectives of this Study

In Phase II the project team established as its primary goal determining how process conditions (storage, anaerobic digestion, and mechanical dewatering) affect odor emissions from biosolids in wastewater treatment facilities. In accomplishing this goal, the project team set the following objectives:

- ◆ Produce a consistent set of general testing protocols to be followed at identical testing events at every facility in the study.
- ◆ Use established and new sampling and analytical methods to measure odor precursors in the liquid and gaseous phases of the biosolids, which were produced under a variety of set process conditions.
- ◆ Enter process and operational data from all plants in the study for the week and month prior to the testing date into a Request for Information database.
- ◆ Draw correlations between the process conditions and the measured odor precursors to provide a better understanding of the conditions that produce more odorous biosolids.

Phase II of the project was developed to be an *observational study* of biosolids odor characteristics, summarizing detailed field work and laboratory analyses of samples collected from 11 wastewater treatment plants (WWTPs) across North America. The main purpose of the study was to observe and document relationships and correlations found among wastewater characteristics, plant operations, and biosolids odor characteristics.

ES.3 Hypotheses Supported, Rejected, or Found Inconclusive

Various hypotheses concerning the origins of odors in anaerobically digested biosolids have been put forth as a result of prior research (see References). The research findings for this study have been linked to these hypotheses and grouped as being supported with conclusive evidence, rejected with conclusive evidence, or inconclusive, depending on the sufficiency of information from the study.

ES.3.1 Hypotheses Supported Based on Study Results

The list below contains hypotheses derived from previous research and experience that were found to be conclusively supported based on the results and correlations developed as part of the study:

1. Higher amounts of bio-available (labile) protein in biosolids cake create more odors.
2. Different dewatering practices affect bio-available protein differently; some dewatering practices tend to increase odors in the biosolids cake.
3. Volatile sulfur compounds (VSCs) are the major sources of odors in digested biosolids. This relationship was shown by a high correlation between odor detection threshold (DT) and concentration of VSCs, indicated by a multiple regression equation having a correlation coefficient of 0.90, which describes this relationship.
4. Odor concentrations in mesophilicly digested biosolids cake rise and then decline over time during storage.
5. Based on comparison of results from the one WWTP in the study with thermophilic anaerobic digestion and 10 WWTPs with mesophilic digestion, odors from thermophilicly digested biosolids cake have different characteristics and patterns of time release than mesophilicly digested biosolids cake.
6. Iron in sufficient concentrations binds bio-available protein in biosolids cake and thus reduces odor production from dewatered biosolids.

ES.3.2 Hypotheses Rejected Based on Study Results

The list below contains hypotheses that were developed because of a collective belief in the industry that a potential relationship exists, as reported in the literature. However, the results of the study indicated that no relationship exists, and therefore the hypotheses were rejected based on the data collected from 11 WWTPs studied and the correlations that were produced.

1. The study findings showed no positive correlation between high influent sulfate concentrations and odors in biosolids cake.
2. The study findings provided no evidence that WAS has a higher odor potential following digestion than primary sludge.
3. The study findings provided no evidence that enzyme activity can be used as an indicator of biosolids odor production.

ES.3.3 Hypotheses Found to be Inconclusive Based on Study Results

The list below contains hypotheses developed because the project team believed, based on prior research and experience, that potential relationships exist between biosolids processes or

CHAPTER 4.0

RESEARCH FINDINGS

Chapter 4.0 summarizes the findings of the Phase II research and presents conclusions and recommendations. The chapter begins with a discussion of the chemical compounds that create odors in biosolids (Subsection 4.1) and then discusses the sampling and analytical methods that were used in the study for testing the odorous compounds emitted from the biosolids (Subsection 4.2). Following the discussion of biosolids odors and how they are measured, the chapter covers the constituents in wastewater and biosolids that are precursors to odors (Subsection 4.3).

Subsection 4.4 discusses the potential impacts of wastewater and biosolids processes on odors in general order of the flow of wastewater and biosolids through a typical wastewater treatment facility, as follows:

- ◆ Subsection 4.4.1 – Impacts of processes upstream of anaerobic digestion
- ◆ Subsection 4.4.2 – Impacts of anaerobic digestion
- ◆ Subsection 4.4.3 – Impacts of biosolids dewatering and conveyance processes
- ◆ Subsection 4.4.4 – Impacts of biosolids cake storage

Each of the subsections follows the same general format: 1) a hypothesis; 2) a summary of results pertaining to the hypothesis; 3) a discussion of the results; and 4) conclusions and recommendations with respect to the original hypothesis. During the preparation of this chapter many potential relationships between treatment parameters and observed sensory and chemical odor measurements were considered, but only a fraction of these are presented in this part of the report. The ones omitted did not show any correlation.

Throughout Chapter 4.0 the numerical identifiers (1 through 11) associated with various data points refer to their respective WWTP sources. Alpha character identifiers (A through I) refer to their respective Sample Locations. Generalized Sample Location identifiers are shown in Figure 2-1 in Chapter 2.0, and specific Sample Location identifiers for each WWTP tested are shown on each WWTP schematic presented in Chapter 3.0.

4.1 Odorous Compounds in Biosolids

Volatile sulfur compounds (VSCs) are known to contribute significantly to odor problems of digested biosolids cake produced by centrifuges (Higgins et al., 2003; Murthy et al., 2002; Novak et al., 2002). Trimethylamine (TMA) is a nitrogen-based compound often associated with a fishy odor in limed sludges. Indole and skatole are odorous aromatic amines (also nitrogen-based) that were first found in mammalian feces and could cause a fecal odor scent. Fatty acids are common in biosolids and produce a rancid smell.

Odor is defined in this study as a human perception that can be quantified by olfactometry in odor units of dilutions-to-threshold (D/T) and in quantitative terms of Detection Threshold (DT), the number of Odor Units (D/T) at which an odor is detected, or Recognition Threshold (RT), the number of Odor Units (D/T) at which an odor is recognizable by descriptive terms. Odor can also be qualified in descriptive terms, such as pungent, rancid, fecal, and rotten.

The odors of selected biosolids samples have been both quantified and qualified at each of the 11 test WWTPs. The Phase II research began with the hypothesis that the odor of biosolids is caused by volatile chemicals that can be measured in the headspace of biosolids in bottles. The project team also hypothesized a correlation between the concentration of odorous compounds and quality and quantity of odors. The hypothesized odor-causing compounds analyzed by chemical odor methods (GC/MS) were as follows:

- ◆ The sulfur compounds H₂S, methanethiol or methyl mercaptan (MT), dimethylsulfide (DMS), dimethyldisulfide (DMDS), dimethyltrisulfide (DMTS), carbonylsulfide (COS), carbondisulfide (CS₂). As a group, they are referred to as VSCs.
- ◆ The nitrogen compound TMA.
- ◆ The nitrogen compounds indole and skatole, which are aromatic amines.
- ◆ Fatty acids, which are odorous but difficult to measure in headspace. These were measured by direct liquid analysis at Bucknell University to compare these results with olfactometry measurements.

Odors themselves were measured by olfactometry, using human test panels that worked with the same headspace gas samples used for chemical analysis. However, olfactometry was undertaken only with headspace gas taken at Day 6 of incubation, which was indicated by prior research as the time period required to generate maximum odor levels from biosolids samples.

4.1.1 Results and Discussion

Olfactometry data for the 11 WWTPs that took part in this study are listed in Table 4-1.

**Table 4-1. Results of Olfactometric and Chemical Measurements
Performed on Sample Bottle Headspace Gas Samples.**

WWTP No.	Sample Location	Detection Threshold ¹ (DT)	Recognition Threshold ¹ (RT)	Peak Total Sulfur mg S/m ³	Peak Total Nitrogen mg N/m ³
1	F2 Digested Biosolids	360	230	4.0	ND ⁵
	G Fresh Low-Solids Centrifuge Biosolids Cake	17,000	11,000	2020 ²	ND
	I2 Low-Solids Centrifuge Cake after about 7-10 days storage	18,000	14,000	1774 ³	ND
2	F2 Digested Solids After Holding Tank	390	230	4.0	ND
	G Low-Solids Centrifuge Biosolids Cake	6,100	4,300	352	ND
2	I High-Solids Centrifuge Biosolids Cake	21,000	13,000	787	ND
3	F Digested Biosolids	460	270	4.8	ND
	G Dewatered Biosolids	9,600	7,300	416	ND
	I Stored Biosolids Cake	4,800	4,200	173	ND
4	F2 Digested Sludge	230	120	5.0	ND
	G Lagoon Top Biosolids Sample	3,700	1,600	60	ND
	I Lagoon Top Biosolids Sample	3,500	2,000	27	ND
5	F Digested Biosolids	270	140	2.7	ND
	G High-Solids Centrifuge Biosolids Cake	6,100	3,500	494	1.1
	I High-Solids Centrifuge Cake after about 2 days storage	7,400	4,300	394	1.7
6	F1 Digested Biosolids (DS)	95	70	8.0	ND
	F2 DS After Holding Tank	120	75	1.0	ND
	G Fresh Biosolids Cake	5,100	3,100	139	1.0
	I Stored Biosolids Cake	2,900	1,700	131	1.0
7	F Digested Biosolids	1,300	830	19.3	ND
	G Centrifuge Biosolids Cake	19,000	14,000	2408 ⁴	4.34
	I Drying Bed Biosolids Cake	1,900	1,400	67	0.56
8	F2 DS Post-Screening	120	65	2.8	1.0
	G Centrifuge Biosolids Cake	9,100	5,000	621	2.23
	I1 Cake Post-Conveyance	2,500	1,400	578	3.33
	I2 Cake Post-Storage	8,900	6,100	304	2.85
9	F1 Digested Sludge	2,500	1,400	14.3	ND
	F2 Digested Sludge Post-Storage	95	70	1.8	ND
	G Plate & Frame Filter Press Cake	1,700	1,100	19	0.97
	I Cake Stored for Two Days	2,200	1,300	130	0.85
10	F2 Train #2 Digested Sludge	1,300	730	4.3	ND
	G2 Train #1 Centrifuge Cake	12,000	8,100	874	0.91
	G3 Train #2 Centrifuge Cake	8,700	5,700	632	1.13
	I Stored Biosolids Cake	21,000	11,000	1160	0.87
11	F2 Primary Digested Sludge	100	65	0.7	ND
	G Centrifuged Biosolids	15,000	8,700	819	0.54
	I1 Cake Post-Conveyance	13,000	8,700	983	0.72
	I2 Cake Post-Storage	1,300	730	19	2.14

Notes: Olfactometry samples were collected on Day 6 of storage. Values for DT and RT are in Odor Units (D/T). Peak total sulfur and nitrogen concentrations were not necessarily measured on samples collected on the 6th day of storage.

¹ These measurements were performed on gas samples obtained from headspace bottles on Day 6 of storage and collected in Tedlar® bags, without any headspace losses during the 6-day storage period. Day 6 was chosen, since the days-to-peak values were not known at the time of sampling. The values represent a 1:50 dilution in samples.

² This value represents a 93% H₂S contribution to total sulfur concentration.

³ This value represents an 89% H₂S contribution to total sulfur concentration.

⁴ This value represents a 58% H₂S contribution to total sulfur concentration.

⁵ "ND" means "not detected," results were below the analytical detection limit.

Table 4-2 lists the olfactometry and headspace chemical odor measurement results ranked based on the odor detection thresholds measured in the dewatered cake (G-cake) odor values. It shows that WWTP No. 9 cake was least odorous and WWTP No. 2H (high-solids centrifuge train) was the most odorous based on the sensory odor measurements. Once the “Detection Threshold” and “Peak Total Sulfur” columns are compared, it is evident that the latter parameter follows the same order as the former. In other words, a strong relationship between the headspace odor and peak total sulfur is implicit from this data. The nitrogenous odor compounds measured in the headspace were not high enough to indicate a correlation.

Table 4-2. Results of Olfactometric and Chemical Measurements Performed on G-Cake Bottle Headspace Gas Samples Ranked According to Detection Threshold Values.

WWTP No.	Sample Location	Detection Threshold ¹ (DT)	Recognition Threshold ¹ (RT)	Peak Total Sulfur mg S/m ³	Peak Total Nitrogen mg N/m ³
2H	I High-Solids Centrifuge Biosolids Cake	21,000	13,000	787	ND ⁴
7	G Centrifuge Biosolids Cake	19,000	14,000	2408 ³	4.34
1	G Fresh Low-Solids Centrifuge Biosolids Cake	17,000	11,000	2020 ²	ND
11	G Centrifuged Biosolids	15,000	8,700	819	0.54
10	G2 Train #1 Centrifuge Cake	12,000	8,100	874	0.91
3	G Dewatered Biosolids	9,600	7,300	416	ND
8	G Centrifuge Biosolids Cake	9,100	5,000	621	2.23
10	G3 Train #2 Centrifuge Cake	8,700	5,700	632	1.13
2	G Low-Solids Centrifuge Biosolids Cake	6,100	4,300	352	ND
5	G High-Solids Centrifuge Biosolids Cake	6,100	3,500	494	1.1
6	G Fresh Biosolids Cake	5,100	3,100	139	1
4	G Lagoon Top Biosolids Sample	3,700	1,600	60	ND
9	G Plate & Frame Filter Press Cake	1,700	1,100	19	0.97

Notes: Olfactometry samples were collected on Day 6 of storage. Values for DT and RT are in Odor Units (D/T). Peak total sulfur and nitrogen concentrations were not necessarily measured on samples collected on the 6th day of storage.

¹ These measurements were performed on gas samples obtained from headspace bottles on Day 6 of storage and collected in Tedlar[®] bags, without any headspace losses during the 6-day storage period. Day 6 was chosen, since the days-to-peak values were not known at the time of sampling. The values represent a 1:50 dilution in samples.

² This value represents a 93% H₂S contribution to total sulfur concentration.

³ This value represents a 58% H₂S contribution to total sulfur concentration.

⁴ “ND” stands for “not detected,” results were below analytical detection limit.

Figures 4-1 and 4-2 show that the maximum concentration for volatile nitrogen in analyzed samples was generally on the order of 1,000 times lower than the maximum concentration for volatile sulfur in the same samples. These figures also demonstrate that most of the odor samples analyzed from the test WWTPs show a positive correlation between olfactometry measurements in terms of odor DT and volatile sulfur in concentration units of milligrams of sulfur per cubic meter (mg S/m³).

Figure 4-2 indicates no discernible relationship between odor DT and volatile nitrogen (milligrams of nitrogen per cubic meter [mgN/m³], measured as TMA, indole, and skatole). This has two implications: nitrogen compounds are secondary odor producers compared to sulfur compounds, and the GC/MS method needs to be improved to better capture the N-bearing odorous compounds.

- ◆ The storage condition in the headspace of the storage bottles was anaerobic, simulating the anaerobic core of full-scale cake piles or storage vessels, since biosolids mixing during storage was not part of the sample bottle handling schedule.
- ◆ The headspace of the storage bottles allows the odor consumption of the cakes and the odor production-consumption cycles to be measured. The headspace method is a simple laboratory test to track the changes in sulfur odor that occur during a period of storage before transport. Odor complaints from biosolids often occur during, or as a result of, cake storage.

4.2.4 Recommendations

The following recommendations are based on the results and conclusions of this subsection:

- ◆ The bottle headspace method should be considered as part of the odor test protocols for biosolids to make odor measurement simpler and comparable on a plant-by-plant basis.
- ◆ For best results, headspace parameters in sample bottles should be controlled, based on:
 - Mass of biosolids and headspace volume of the sample bottle.
 - Oxygen (prevention of unwanted air leakage for anaerobic experiment or wanted addition of pure oxygen for aerobic experiment).
 - Incubation temperature and time, including control during sample shipment.
 - The material of the bottle (leakage, overpressure, and other safety aspects).
- ◆ A bench-scale prediction of VSC production and consumption in digested biosolids by anaerobic cake storage should be used for a period that simulates full-scale storage conditions. If biosolids cake cannot be transported from the WWTP within the first day or two, reduction of odors through longer cake storage times might be advised until VSC emissions start to decrease.

4.3 Wastewater Constituents Affecting Biosolids Odors

4.3.1 Role of Protein

4.1.3.1 Hypothesis

The central hypothesis for this research is that bio-available protein is the main substrate for the formation of VSCs associated with odors in biosolids cake. Sulfur-containing amino acids can be degraded to form VSCs. For example, methionine can be degraded to form MT, and cysteine can be degraded to form H₂S (Oho et al., 2000; Persson et al., 1990; Persson, 1992; Higgins et al., 2003). Both MT and H₂S can be methylated to form DMS and MT, respectively (Drotar et al., 1987; Bak et al., 1992; Lomans et al., 2001). In addition, MT can be oxidized to form DMDS (Higgins et al., 2003). The bio-transformations that are mediated by bacteria demonstrate that protein, specifically sulfur-containing amino acids, are the likely substrate for formation of VSC-associated odors. Therefore, greater amounts of bio-available protein should result in greater VSC-associated odors.

4.1.3.2 Results

As part of this study, three different fractions of protein were measured: one soluble and two bound fractions that were extracted from centrifuged pellets of the liquid samples or directly from the cake samples (Subsection 2.3.5). The soluble fraction was measured on filtrate of the

4.1.3.5 Recommendations

The following recommendations are based on the conclusions of this subsection:

- ♦ Reducing the bio-available protein concentration in biosolids cake could lead to a reduction in the odors associated with cake storage.
- ♦ The mass of bound methionine in biosolids cake samples can be used as an indicator of the odor production potential during storage.
- ♦ Further research should be conducted to investigate the impact of protein on nitrogen and sulfur-bearing odorous compounds, testing different types of cake samples processed by different types of biosolids handling equipment.

4.3.2 Role of Enzyme Activity

4.3.2.1 Hypothesis

A fraction of the protein in biosolids is made up of enzymes that are responsible for breaking down protein and producing odorous compounds. The project team hypothesized that enzyme activity may also play a role in producing odor and that greater enzyme activity would be associated with greater odors. If samples had more enzyme activity, this could be an indication of poor digestion performance. As a result, enzyme activity also has potential.

$$RTD(t) = w_1 RTD1(t) + w_2 RTD2(t) \quad (4.3.2.1-1)$$

4.3.2.2 Results

Protein degrading (or proteolytic) enzyme activity was characterized by l-leucine aminopeptidase (LLAP) activity, which is a common enzyme used for this purpose (Teuber and Brodisch, 1977). A summary of the LLAP activity measured in the bound fraction of the cake and digester samples is given in Table 4-5. The LLAP activity was measured on samples.

$$RTD(t) = w_1 RTD1(t) + w_2 RTD2(t) \quad (4.3.2.2-1)$$

Hypothesis Number 1: “Good digestion” (usually defined by VS destruction and methane gas production) leads to minimized odors in digested biosolids, and conversely, poor digester performance exacerbates odors in the digested biosolids.

Hypothesis Number 2: Thermophilic digestion can create a different time pattern of odor release and a different odor quality than mesophilicly digested biosolids.

4.4.2.2 Results

In order to further examine the concept of anaerobic digestion and its impact on biosolids odor generation, the project team evaluated digester operation data collected from the 11 test WWTPs. Prior studies and some of the data from this study indicate that longer digestion SRT results in lower odor and sulfur emissions from liquid digested biosolids, when measured immediately downstream of digestion, as illustrated in stacked bar chart presented in Figure 4-27. However, when the project team investigated the odor potential of the digested biosolids in terms of the traditional digester performance indicators, such as VS reduction, the current study did not confirm this hypothesis. Digested biosolids odor quality prior to dewatering is not an indicator of dewatered biosolids odor quality. Digested biosolids quality and its changes during dewatering require further study, evaluating parameters such as:

- ◆ Digester effluent VFAs
- ◆ SRT
- ◆ RBA
- ◆ VS reduction
- ◆ NH₃ content in digester off-gas
- ◆ Methane content in digester off-gas

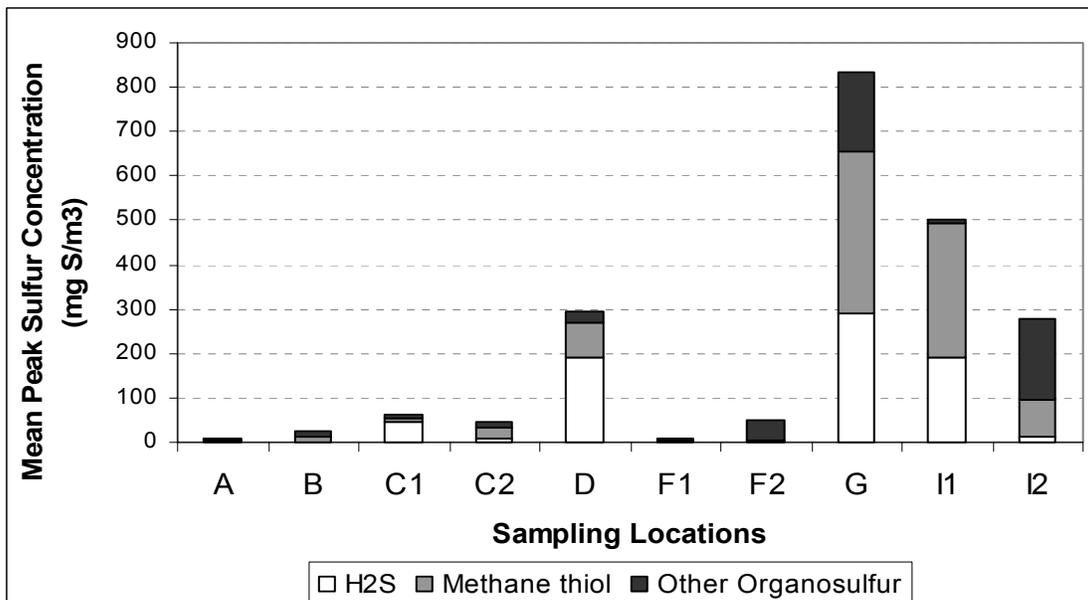


Figure 4-27. Sulfur Distribution at Different Points of Treatment Train Measured on Days 1, 3, 5, and 7 of Sample Storage (All WWTPs).

Digester Effluent VFAs: A high concentration of acetic acid (a short-chain VFA) in digested solids has historically been an indicator of poor digester performance, which is often thought of as a precursor to biosolids odors. However, as shown in Figure 4-28, no clear

relationship can be drawn from the data in this study. Even when the apparent outlier WWTPs No. 1 and No. 9 were omitted, the R^2 was 0.40. Figure 4-28 illustrates the variability in the dewatered cake olfactometry measurements (detection thresholds) when plotted against digester effluent acetic acid concentrations. All but two WWTPs had acetic acid concentrations below 200 mg/L, yet the odor (DT) in biosolids cake samples varied between 3,700 and 21,000 D/T. When the same correlation was plotted for WWTPs with medium-solids centrifuges only (not shown), the variation narrowed to between 5,100 and 12,000 D/T in biosolids cake when acetic acid concentration was below 200. WWTP No. 1 had the highest acetic acid concentration in its digester effluent (994 mg/L) and showed a relatively high odor level of 17,000 D/T. The high-solids centrifuge plants with better digester efficiencies (lower acetic acid levels in the digester effluent) still showed relatively high odor levels in the dewatered biosolids, indicating that other factors beyond digestion influence biosolids odor quality.

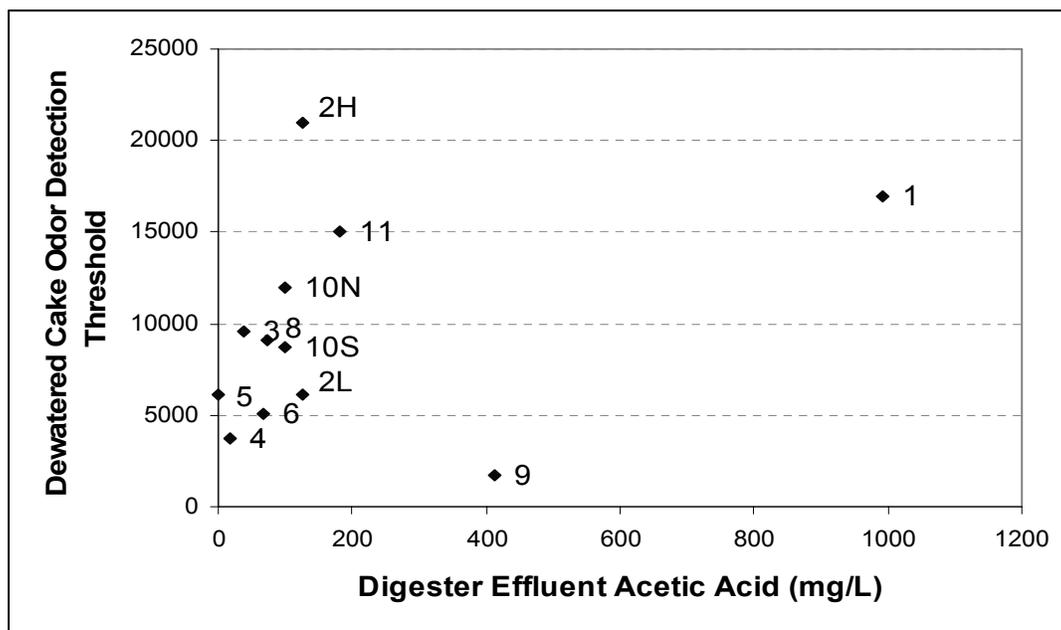


Figure 4-28. Correlation of Dewatered Cake Olfactometry Measurements (DT) with Digester Effluent Acetic Acid (All WWTPs).

Digester SRT: Figure 4-29, plotted for all WWTPs, does not indicate a relationship between digester SRT and cake odors ($R^2 = 0.06$). Data points in this figure included WWTPs without centrifuge dewatering (No. 4 and No. 9) and with thermophilic digestion (No. 8). WWTP No. 1 appears to be an outlier in Figure 4-28 due to very high H_2S concentrations in most biosolids samples. However, for low-solids centrifuge plants, no correlation was found.

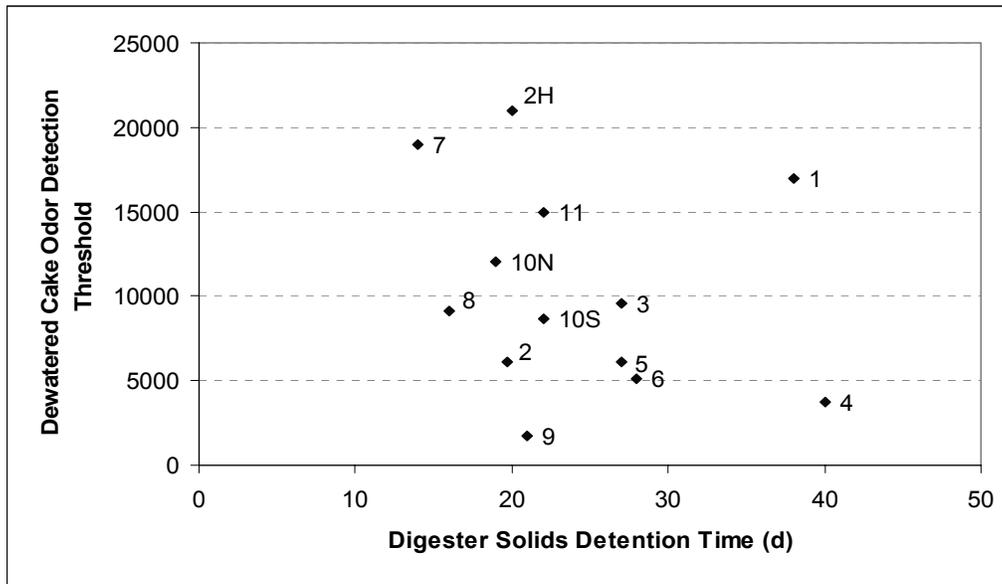


Figure 4-29. Impact of Digester Solids Detention Time on Dewatered Cake Olfactometry Measurements (DT).

Figure 4-30 indicates a fairly strong relationship ($R^2 = 0.62$) between digester SRT and peak organosulfur emissions measured on biosolids samples collected from WWTPs mesophilic digestion followed by centrifuge dewatering. Longer SRT values appear to result in lower peak organosulfur values in this restricted case. The olfactometry data did not show as good a correlation with digester SRT as the organosulfur compounds did, presumably because of the presence of odorous compounds other than those that were measured in this study. Also, results for WWTPs No. 2 and No. 9 indicate that the type of biosolids dewatering process has an impact on cake odors, a factor that needs to be further examined among WWTPs employing similar types of dewatering equipment.

Volatile Solids (VS): Based on prior research, the project team believed that digester feed VS content might play a role in the production of biosolids odors and VSC release. The relationship of VS concentration in the digester feed to dewatered biosolids odors found in this study is illustrated on Figure 4-31. There is no correlation between the two parameters ($R^2 = 0.001$).

The project team also believed based on prior research that higher VS destruction in the digester should have a beneficial impact on digested biosolids quality and dewatered biosolids odors. To investigate this potential relationship, dewatered biosolids odor levels were plotted against digester VS destruction, calculated from WWTP data (Figure 4-32). Odor levels from dewatered biosolids varied within a wide range (DT between 85,000 and 1,050,000 D/T) for a 42-67% VS-destruction range. No correlation was apparent from either this relationship or a plot of dewatered cake VS destruction and peak organosulfur (Figure 4-33), both at a 0.02 R^2 value.

APPENDIX B: SAMPLE CUSTODY PROTOCOL

Sample possession during all testing efforts must be traceable from the time of collection until the results are verified and reported. Sample custody procedures provide a mechanism for documentation of all information related to sample collection and handling to achieve this objective.

The WERF Study Team leader at the site will be responsible for seeing that the field team adheres to proper custody and documentation procedures for all sampling operations. Chain-of-Custody forms will be used as the primary documentation mechanism to ensure that information pertaining to samples is properly recorded. Copies of the Chain-of-Custody forms and the field logs will be retained in the project file.

B.1 Documentation Procedures

B.1.1 Field Records

Field personnel will be required to keep accurate written records of their daily activities in a bound logbook. All entries will be legible, written in waterproof ink, and contain accurate and inclusive documentation of an individual's field activities, including field data and observations, any problems encountered, and actions taken to solve the problem. The type of data recorded in the field logbook includes field measurements, ambient conditions, and any other information pertinent to sample collection.

Entry errors or changes will be crossed out with a single line, dated, and initialed by the person making the correction. Entries made by individuals other than the person to whom the logbook was assigned will be dated and signed by the individual making the entry. Field logbooks will be available for review by interested parties.

B.1.2 Sample Labeling

Each sample collected will receive a sample label that identifies the sample by a unique sample identification number. These labels are affixed to the sample container prior to sample collection. The sample label shall be sealed on the bottle with clear plastic tape. The sample labels will contain the following information:

Date sample was taken
Sample site
Sample Location ID
Analyte(s)
Sample Number

Examples of preprinted sample labels are provided in Figure B-2.

B.1.3 Sample Master Logbook

A sample master log will be maintained for all samples collected. Each sample will be assigned a unique identification number; a full description of the sample, its origin, and disposition will be included in the log entry.

B.2 Chain-of-Custody Procedures

After the samples are collected and documented in the master logbook, a Chain-of-Custody form will be completed and will accompany the samples to the laboratory (a sample form is provided in Figure B-1). Team members collecting the samples are responsible for the care and custody of the samples until they are transferred or dispatched to the appropriate laboratory. When transferring samples, the individuals relinquishing and receiving the samples will sign, date, and note the time on the record. This record documents sample possession from collection to the laboratory sample control center.

When the samples are received by the laboratory, the sample control officer will verify the Chain-of-Custody form against the samples received. If any discrepancies are observed, they will be recorded on the Chain-of-Custody form and the filed team leader will be notified to correct the problem.

B.2.1 Shipment

All sample shipments will be accompanied by the Chain-of-Custody record, which identifies the contents of each crate. The person relinquishing the samples to the laboratory will request the signature of a laboratory representative to acknowledge receipt of the samples. Sample collection and shipment will be coordinated to ensure that the receiving laboratory has staff available to process the samples according to method specifications. All shipping containers will be secured for safe transportation to the laboratory. The method of shipment, courier name(s), and other pertinent information is entered in the "Remarks" section when the samples are to be shipped (i.e., Federal Express, Express Mail, etc.).

B.2.2 Sample Handling Procedures

The objective of sample handling procedures is to ensure that samples arrive at the laboratory intact, at the proper temperature, and free of external contamination. Liquid and bag samples will be shipped via Federal Express to the appropriate laboratory by field sampling personnel. Each sample shipping container that contains samples for headspace analysis will have an enclosed temperature data logging device in it.

Once the samples have been collected, the methods specify preservation, storage requirements and holding time limitations. Table B-1 summarizes the types of sampling containers to be used and the preservation requirements for the types of analysis to be performed

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