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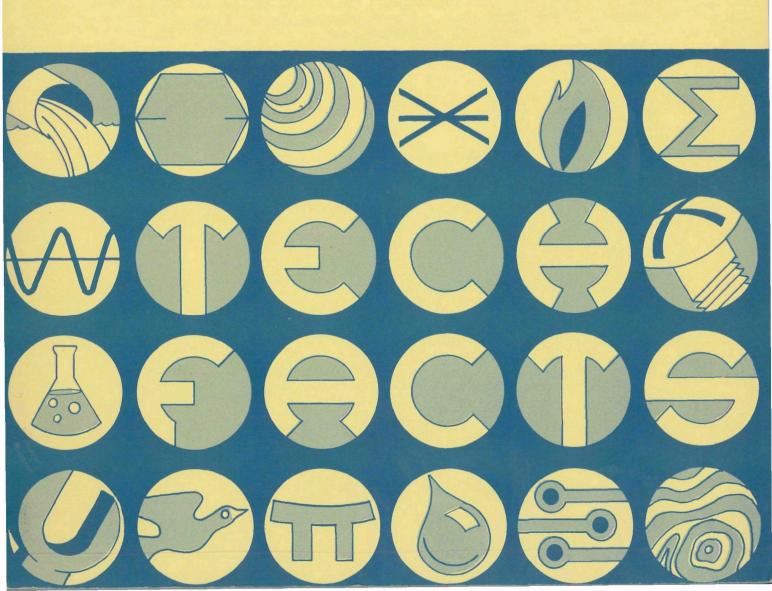
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Solid Waste



# Municipal Sludge Agricultural Utilization Practices

An Environmental Assessment Volume I



## MUNICIPAL SLUDGE AGRICULTURAL UTILIZATION PRACTICES

## An Environmental Assessment

Volume I

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#### **ABSTRACT**

An environmental assessment was performed at nine study sites across the United States to investigate the effects of utilizing municipal wastewater treatment plant sludge for agricultural purposes. The sites represented a wide range of sludge application rates, sludge characteristics, cropping practices, soils, population densities, and climatological and geographical conditions. The assessment included evaluative criteria such as chemical, physical, and microbiological constituents of sludges; surface and subsurface soil properties including presence and accumulation of heavy metals, pesticides, nutrients, bacteria, and parasites; plant characteristics including heavy metal and pesticide uptake, and potential contamination by bacteria and parasites; public attitudes; landspreading costs (capital and operational); current and past operating procedures; and management practices at each of the nine sites.

The project was divided into two phases. Phase I involved a literature search and an extensive screening program to select the nine study sites. Phase II involved the sampling and chemical, physical, and microbiological analyses of over 600 soil, plant, and sludge samples to assess those criteria mentioned above.

Volume I presents an in-depth analysis of the data including summary, conclusions, and recommendations. Volume II presents detailed write-ups of each study site along with a complete record of all the analytical data.

Volume II is available from the National Technical Information Service, U.S. Department of Commerce, Springfield, Virginia, publication number PB-279 357.

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## CHAPTER I

#### SUMMARY

Agricultural utilization is one of several accepted disposal methods for municipal sewage treatment plant solids (sewage sludge). Several university- and federally-sponsored research projects have been conducted to study the effects of long-range sludge applications to croplands, but these have generally been limited to the controlled conditions of test plots. The Environmental Protection Agency (EPA) Office of Solid Wastes, to advance the knowledge of actual practices, contracted with SCS Engineers (SCS) to undertake an environmental assessment of sewage sludge utilization on cropland at nine study sites. These sites were to be diverse --encompassing as many types of crops, soils, and sludge characteristics as possible, and representative of sludge spreading activities as currently practiced.

The project was divided into two phases. The first involved the establishment of site selection criteria, contact with approximately 100 municipalities and/or sewage treatment plants, and the selection of 16 preliminary sites. Field visits were made for five major reasons:

- To obtain detailed information on sludge spreading programs at each location,
- To assess the availability of historical records.
- To interview the owners/managers of the farms receiving sludge,
- To select suitable sludge-treated and control plots for preliminary sampling, and
- To obtain sludge, soil, and plant samples for chemical and physical analyses.

From this assessment and the analytical data, nine sites were selected for in-depth study in Phase II of the project. Table 1 provides a summary of site characteristics at the nine locations. All sites shared the following:

 Only municipal sewage treatment plant solids were utilized.

TABLE 1. COMPARISON OF PHYSICAL PARAMETERS FOR NINE STUDY SITES

Site Location	Agricultural Crop	Food Crop Use	Surface Soil Description*	Surface Soi pH†
Macon, GA	Cheatgrass	Non-dairy cattle feed	Sandy loam	3.7
Las Virgenes, CA	Ryegrass	Non-dairy cattle feed	Clay loam	6.5
Wilmington, OH	Alfalfa	Non-dairy cattle feed	Silt loam	6.5
Springfield, MO	Fescue	Non-dairy cattle feed	Silt loam	7.0
Chippewa Falls, WI	Soybeans	Open market <sup>#</sup>	Sand	5.5
Hopkinsville, KY	Fescue	Non-dairy cattle feed	Silt loam	6.2
Frankfort, IN	Wheat	Flour mill	Silt loam	6.7
Kendallville, IN	Alfalfa	Non-dairy cattle feed	Clay loam	6.4
Columbus, IN	Corn	Distillary	Sandy loam	6.6

<sup>\*</sup>USDA Classification to.01 m CaCl<sub>2</sub> method \*Crop not harvested because of poor yield

TABLE 1 (continued)

Site and Location	STP Average Capacity (cu m/day)	Population Equivalent	Type of Secondary Treatment	Estimated Industria Contribution (percent flow)
Macon, GA	45,400	125,000	Trickling filter	30
Las Virgenes, CA	17,000	75,000	Activated sludge	10
Wilmington, OH	7,600	19,000	Activated sludge	25
Springfield, MO	75,700	164,000	Activated sludge with Kraus modification	15
Chippewa Falls, WI	13,200	51,000	Activated sludge	65
Hopkinsville, KY	8,300	28,710	Trickling filter	15
Frankfort, IN	11,700	12,900	Trickling filter	30
Kendallville, IN	6,000	22,200	Trickling filter	25
Columbus, IN	24,400	50,000	Activated sludge	67

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TABLE 1 (continued)

Site Location	Reported Years of Sludge Spreading	Estimated Annual Sludge Application Rate** (m tons/ha)	Cumulative Total Sludge† (m tons/ha)
Macon, GA	11	28 <sup>##</sup>	308 <sup>#</sup>
Las Virgenes, CA	7	50	149
Wilmington, OH	17	6.8	116
Springfield, MO	15	15.8	237
Chippewa Falls, WI	6	16##	80 ##
Hopkinsville, KY	9	22##	66 <sup>##</sup>
Frankfort, IN	12	30##	360 ##
Kendallville, IN	13	19.7	81
Columbus, IN	5	65	326

<sup>\*\*1975</sup> † Through Dec. 31, 1975 ##Estimated

4

- All applied sludge had been stabilized by conventional aerobic or anaerobic processes.
- Crops grown in the sludge amended soils were used for animal or human food.
- Sludge had been applied for a minimum of four years.
- All fields were under active farm management.

Phase II encompassed data gathering at each site and sampling of sludge, surface and subsurface soils, and plants for physical, chemical, and bacteriological analyses. These data provided the basis for the ensuing assessment of environmental impacts at each of the sites. The prescreening and selection of nine case study sites resulted in an investigation of a wide variety of different sludges, soils, and plants, although two of the sites were planted to alfalfa and two to fescue. Because of these diverse conditions, no direct comparisons between any of the sites are presented.

Communications between the consultant and responsible officials at each site were maintained throughout the project. As conclusions evolved from the assessments, these officials were afforded the opportunity to review and comment on the findings.

## HIGHLIGHTS OF PROJECT FINDINGS

- The case study sites were located in seven states. Three states (Wisconsin, Illinois, and Ohio) had published guidelines for agricultural utilization of sewage sludge. The states of Georgia, California, and Indiana were in the process of adopting similar guidelines, while neither Missouri nor Kentucky had initiated guidelines programs.
- Record keeping by sewage treatment plants for sludge utilization practices was found to be incomplete. Several maintained records of sludge quantities transported to farms but could not specify spreading areas within the receiving fields, and one site kept virtually no sludge distribution records whatsoever. Farm owners/managers receiving sludge had not recorded sludge quantities received nor the locations for spreading on their farms.
- In general, the public officials interviewed were not fully cognizant of the sludge utilization practices within their respective communities. None expressed concern for ongoing sludge utilization practices, and local health officials reported no instance of health problems attributed to sludge utilization practices at any of the nine sites.

- Sludge utilization practices at two sites had minimal adverse environmental impacts. Ironically, however, sludge spreading at both sites has been halted. Spreading at one site ceased because of an isolated odor complaint arising from the field-drying process for the sludge. (Other utilization sites within the near vicinity were not affected.) The other site was located on marginal agricultural land and was recently sold to the city for park development.
- Of the nine case study sites, only two exhibited the potential for significant adverse environmental impact: one utilized a sludge containing high concentrations of Cd; the other disposed of sludge on highly-acidic, coarse textured soil in close proximity to the groundwater.
- Table 2 presents an analysis of the remaining useful life of the sites, based upon the following assumptions:
  - The chemical composition of each sludge will remain constant at the levels found during the study.
  - Guidelines proposed by the North Central Regional Committee NC 118 (1976), based on CEC and total metal loadings, are used as evaluating criteria.

TABLE 2. USEFUL REMAINING LIFE OF CASE STUDY SITES BASED ON TOTAL METAL LOADINGS AND SLUDGE CHARACTERISTICS\* †

			Years		
Site	Cd	Cu	Ni	Pb	Zn
Macon, GA	49	8	40	106	9
Las Virgenes, CA	33	8	77	997	12
Wilmington, OH	246	162	963	248	0
Springfield, MO "	7.5	24	27	829	4
Chippewa Falls, WI#	84	6	308	620	21
Hopkinsville, KY	117	38	149	1,007	28
Frankfort, IN	0	0	1	254	4
Kendallville, IN	16	31	123	9	3
Columbus, IN	49	15	14	251	16

<sup>\*</sup>CEC of all surface soils, except Chippewa Falls, WI, was  $\geq$  15. †As of January 1, 1976. #CEC  $\leq$  5.

From the table, the disposal sites at Wilmington, Ohio, and Frankfort, Indiana, have surface metal concentrations exceeding

the proposed guidelines. Using NC 118 criteria, then, these two sites have no useful remaining life for sludge utilization.

• In general, there was close analytical agreement between soil and plant composites and the means of the five individual section samples from which the composites were derived. This indicates that the samples accurately represented field conditions. Statistically significant differences at the 90 percent confidence level (treated versus control) in terms of Cd, Cu, Ni, and Zn concentrations contained in surface and subsurface soils are shown in Table 3.

TABLE 3. STATISTICALLY SIGNIFICANT DIFFERENCES FOR METAL CONCENTRATIONS IN SOILS\*

Site	Cd	Cu	Ni	Zn
Macon, GA	S†	S	S	5,1,2,3,4#
Las Virgenes, CA		S,1	S	5
Wilmington, OH		S		S
Springfield, MO	S		S,1	S,1,2
Chippewa Falls, WI		S,1		S
Hopkinsville, KY	S,1	S,1,2	S,1	S
Frankfort, IN	5,1,2	S	S	S,1
Kendallville, IN	-	5,1	5,1,2,3,4	s,1
Columbus, IN	S	S,3,4	S,4	5,3,4

<sup>\*</sup>Significant at the 90 percent confidence level.

- Similarly, statistically significant differences at the 90 percent confidence level (treated versus control plots) were observed for Cd, Cu, Ni and Zn concentrations in plants and grains. These data are presented in Table 4 (on the following page). In general, however, the concentration of metals reported were within ranges not considered hazardous to health or phytotoxic to plants except for Zn at Macon, GA (phytotoxic range) and Cd at Frankfort, IN (potential health concern).
- o Liquid sludge spreading costs in the study locations ranged from \$7.98 to \$79.08 per dry m ton. One site employing sludge dewatering incurred costs of \$128.07 per dry m ton.
- o The study results tend to confirm that with proper site management and crop selection, as well as careful attention to soil, crop, and sludge compatibility, little or no adverse environmental impacts will result from sludge utilization for agricultural purposes.

<sup>+</sup>Surface soil.

<sup>#</sup>Depths (1 - shallowest; 4 - bottom depth to 122 cm).

TABLE 4. STATISTICALLY SIGNIFICANT DIFFERENCES FOR METAL CONCENTRATIONS IN PLANTS AND GRAINS\*

Site	Plant/Grain	Cd	Cu	Ni	Zn
Macon, GA	Cheatgrass	+	+	+	+
Las Virgenes, CA	Ryegrass	_	_	+	_
Wilmington, OH	Alfalfa	_	_	_	+
Springfield, MO	Fescue	_	-	+	+
Chippewa Falls, WI	Soybean petioles Soybeans	+ +	++	- +	++
Hopkinsville, KY	Fescue	+	+	_	+
Frankfort, IN	Wheat - Immature bud, stalk, leaves Wheat - Grains	++	+	-	<u>-</u>
Kendalville, IN	Alfalfa	+	-	_	+
Columbus, IN	Corn - leaves Corn - grain	+	+	+	+

<sup>\*</sup>Significant at the 90 percent level. +Denotes significant difference.

#### CHAPTER II

#### CONCLUSIONS

Conclusions, as presented in this chapter, have been divided into the following categories:

- General,
- Sludge Related,
- Soil and Plant Related,
- Site Specific, and
- Sludge Management.

The site specific category is a capsulation of conclusions to be found in Chapter VIII, Section I. The other categories present statements, tables, and data excerpted and condensed from Chapter VIII, Sections I and II. The reader is advised to review Chapter VIII in its entirety for an in-depth analysis of all supportive data.

#### GENERAL

- Interviews with local health officials and site personnel revealed no reported health problems associated with the sludge utilization practices at the study sites.
- No adverse comments on the sludge utilization practices were received from state, county, or city officials contacted during the project. However, some officials were not fully cognizant of local practices.
- Two sites had no useful remaining life for receipt of sludge based on cumulative metal loadings to date and guidelines proposed by the North Central Regional Committee NC 118 (1976). They were Wilmington, Ohio (excess Zn loadings), and Frankfort, Indiana (excess Cd and Cu loadings).
- Concentrations of DDT in sludge ranged from 0.9 to 160 ppb. The highest DDT residual found in the soil was 0.29 ppb.
- Detectable concentrations of dieldrin, ranging from 0.4 to 114 ppb, were found in the sludges from five sites.

- Detectable concentrations of dieldrin were found in surface soils at six sites. The maximum concentration detected was 0.84 ppm.
- In the respective sludges, polychlorinated biphenyls (PCB's) were detected in measurable quantities at all locations except one. The maximum concentration measured was 5,872 ppb with five others exhibiting concentrations above 3,300 ppb.
- PCB's were detected in the surface soil at all but two sites. A significant decrease in concentration occurred between sludge and soil values observed.
- PCB's were found in or on plants at two locations. The highest plant PCB concentration level was 0.4 ppb which is near the detection limit.
- An economic analysis of the sludge disposal costs at eight sites utilizing liquid sludge showed a range of \$7.98 to \$79.08 per m ton (dry-weight basis). At the Las Virgenes site, where sludge was dewatered, the cost was \$128.07 per m ton (dry-weight basis).
- The microbiological findings (total aerobes, salmonella, shigella, fecal coliforms, and fecal streptococci) revealed no significant deviations from occurrence or concentration levels commonly observed in municipal sludges. The soil and plant data in general compared well to similar studies. Where results were atypical, they could in part be attributed to the limited number of analyses performed.
- Salmonella sp. organisms were not isolated in any of the stabilized sludges. This finding supports other related research which has shown that <u>Salmonella sp.</u> and <u>Shigella sp.</u> organisms do not normally survive sludge stabilization processes.
- Salmonella sp. (Salmonella paratyphi C.) was isolated in one of 88 composite soil samples examined.
- No <u>Salmonella</u> <u>sp.</u> or <u>Shigella</u> <u>sp.</u> organisms were detected in any of the plant samples.
- The failure to detect parasitic helminth ova in some of the sludge samples appeared to be atypical based on similar studies. This can be attributed to the random sampling, the small number of samples, and the tendency for the helminth ova to sediment out during the sludge stabilization residence time.

- Similarly, no parasitic helminth ova were detected in any of the composite surface soil samples.
- The percent DTPA extractable metals (based on total metals) was, in almost every instance, greater in surface than subsurface soils, suggesting that metals in sludges are more soluble in DTPA than those naturally present in the soil.
- In most cases, the percent of Cd extracted with DTPA was higher than those of Cu, Ni, or Zn.
- The percent of total metals extracted by DTPA, in most cases, increased in proportion to the organic carbon concentration of the soil.

## SLUDGE RELATED

- In terms of nitrogen (N) and phosphorus (P), the sludges represented an unbalanced but significant fertilizer resource. Calculated equivalent fertilizer values ranged from a low of \$11.79 per m ton (dry basis) to a high of \$40.18, assuming 50 percent of the ammonia-N and 20 percent of the organic-N are available in any one year, and that 100 percent of the P is plant available. All sludges were more valuable in terms of P than N.
- Sludges at all sites were distributed over the soil surface resulting in ammonia loss by volatilization.
   If the sludge were incorporated into the soil, it would have an increased value as a source of N.
- The cumulative total addition of Cd ranged from 0.5 to 13.3 kg/ha at eight of the nine sites, while annual Cd loading rates at these sites ranged from 0.08 to 1.0 kg/ha. The ninth site had a cumulative total Cd loading of 540 kg/ha, and an annual Cd loading rate of 45 kg/ha. If sludge application rates are based on 200 kg/ha available N, two locations would add Cd in excess of maximum levels proposed by the North Central Regional Committee, NC 118 (1976).
- Based on 1975 actual application rates, and the annual maximum Cd loading of 2.0 kg/ha as recommended by the EPA Municipal Sludge Management Technical Bulletin, utilization practices at one of the nine sites resulted in Cd quantities in excess of this maximum.

## SOIL AND PLANT RELATED

• The Cd concentration in the soil profile (treated and control plots) at the Las Virgenes, California, site was unusually high (approximately 4  $\mu$ g/g). However, the source of Cd was considered to be of natural origin and not the result of sludge spreading.

- Small differences in metal concentrations between treated and control subsurface soil sections and within depth increments were in some instances statistically significant in a 90 percent confidence level. However, in all of these cases, the concentrations determined were within the ranges typically reported for soils (see Table 40) and, in most cases, considerably less than those of the treated surface soil. The data show that metals applied have remained in the depth of incorporation with the following exceptions:
  - Macon, Georgia Total and DTPA Zn concentrations indicate that Zn has migrated to the 122 cm depth.
  - Frankfort, Indiana Total and DTPA Cd concentrations indicate that Cd has migrated to a depth of 61 cm and possibly 91 cm.
- The depth of metal movement (Zn) was greatest in coarsetextured, acid soils.
- Metal accumulations in soil at the surface of the treated plots as a result of sludge spreading were significantly higher (90 percent confidence level) than the control plots at all sites with the following exceptions:

Las Virgenes, CA - Cd Wilmington, OH - Cd, Ni Springfield, MO - Cu Chippewa Falls, WI - Cd, Ni Kendallville, IN - Cd

- Similarly, subsurface metal accumulations at varying depths of the treated plots were significantly higher (90 percent confidence level) than those of the control plots as shown in Table 5 (see next page).
- The subsurface soil data suggest that potential groundwater contamination from surface sludge spreading operations is remote except at Macon, Georgia, where Zn has migrated to a soil depth of 122 cm. The groundwater table at this site is located between 120 and 150 cm.
- Statistically significant differences (90 percent confidence level) were observed in a majority of plant and grain Cd, Cu, Ni, and Zn concentrations. In general, however, the concentrations of metals reported were within ranges considered normal except for the following:

TABLE 5. STATISTICALLY SIGNIFICANT DIFFERENCES IN SUBSURFACE SOIL METAL ACCUMULATIONS\*

Site	Cd	Cu	Νi	Zn
Macon, GA				1,2,3,4†
Las Virgenes, CA		1		
Wilmington, OH				<del>-</del> -
Springfield, MO			7	1,2
Chippewa Falls, WI		1		
Hopkinsville, KY	1	1.2	1	
Frankfort, IN	1.2			7
Kendallville, IN	**	1	1,2,3,4	ĺ
Columbus, IN		3,4	4	3,4

<sup>\*90</sup> percent confidence level. †Depths (1 - shallowest; 4 - bottom depth to 122 cm).

- Macon, Georgia Zn concentrations in cheatgrass (Bromus Tectorum) ranged from 341 to 455  $\mu g/g$  (averaging 383  $\mu g/g$ ), a level considered phytotoxic to some plant species.
- Frankfort, Indiana Cd concentrations in wheat grains ranged from 0.58 to 1.89  $\mu g/g$  (averaging 1.26  $\mu g/g$ ). Based on an average daily human consumption of 100 g of this wheat, the maximum amount of Cd recommended by the World Health Organization would be exceeded.

#### SITE SPECIFIC

Site specific conclusions regarding acceptability of sludges, soils, and plants are based on recommendations as outlined in the North Central Regional Committee NC 118 Report (1976); see Table 35.

## Macon, Georgia

- With the possible exception of Se concentrations, the sludge was suitable for agricultural utilization. However, excessive annual sludge applications, site soil properties, shallow depth to groundwater, and high rainfall have created conditions that greatly enhance plant uptake and movement of heavy metals (e.g., Cu and Zn) to lower soil depths.
- The data suggest that Zn may be migrating through the soil, and that groundwater contamination from Zn and other anions (NO<sub>3</sub>, Cl, SO<sub>4</sub>, etc.) may be occurring.

## Las Virgenes, California

- The utilization of sludge has had little or no impact on trace metal uptakes in plants.
- Large amounts of nitrogen, considerably in excess of those needed by the crop, have been applied via irrigation using wastewaters. It is possible that groundwater NO<sub>3</sub>-N contamination may occur, if these applications continue.

## Wilmington, Ohio

- The limiting factor to agricultural utilization of this sludge is probably the exceedingly high Zn content.
- The treated plot soil has been substantially enriched with Zn, but this condition has not yet resulted in significant increases in the Zn levels of alfalfa.
- DTPA Zn indicates possible Zn movement to a depth of 61 cm in the soil.

## Springfield, Missouri

The sludge did not contain excessive concentrations of heavy metals and appears to be a good source of plant nutrients for the fescue grown at the sludge-treated site.

# Chippewa Falls, Wisconsin

The possibility of groundwater contamination appears remote, in view of the restricted heavy metal movement and the low total sludge loadings.

# Hopkinsville, Kentucky

- The landspreading practices have not resulted in elevated concentrations of heavy metals in the fescue forage.
- DTPA analyses suggest limited movement of Cu and Zn to a depth of 30 cm in the soil.

## Frankfort, Indiana

- The high Cd concentration of the sludge indicates that it is not suitable for utilization on agricultural land.
- Total and DTPA-extractable analyses indicate that Cd and possibly Zn have migrated to at least the 61-cm level in the soil.

• The high application rates and elevated Cd in the sludge and soil pose an undesirable public health condition.

## Kendallville, Indiana

- The high concentrations of Zn in the soils have resulted in elevated Zn concentrations in the alfalfa crop.
- The Pb enrichment of the treated soils has not resulted in elevated Pb alfalfa levels.
- Cu and Zn results indicate possible limited movement to a soil depth of 30 cm.

## Columbus, Indiana

- No significant heavy metal increases were observed in corn grains.
- Variations noted in subsurface metal concentrations may have resulted from extensive earth moving in the field in early 1975.
- The concentrations of Cd in corn leaves obtained from the treated plot were significantly higher than the control plot leaves, the difference being 0.88 µg/g. Similarly, Ni and Zn differences showed statistically significant differences of 3.44 and 18.94 µg/g, respectively.

## SLUDGE MANAGEMENT

- The maintenance of sewage treatment plant records for various aspects of sludge management was found to be lacking or incomplete. Several plants had maintained records of sludge quantities transported to farms, but could not identify spreading areas within the receiving fields. One plant kept virtually no sludge distribution records whatsoever. None of the receiving farm owners/managers had ever recorded the sludge quantity received nor the spreading locations on their farms.
- The study results tend to confirm that proper site management and crop selection, as well as careful attention to soil, crop and sludge compatibility, can ensure that little or no adverse environmental impacts will occur.

#### CHAPTER III

#### RECOMMENDATIONS

The following recommendations are a summary of the finding of the environmental assessment at each site. They do not address the widely recognized parameters of soil pH, and annual or cumulative metal additions used to limit the movement of metals from sludge to plants. These controls are not discussed because the study was not designed to compare the impacts at one site to the other sites. EPA has published a Municipal Sludge Management - Technical Bulletin (Nov. 2, 1977; FR 57420) which does give guidance in these areas, specific criteria and guidelines for landspreading of sludge are currently under development which will give specific operational controls.

- Maintenance of historical documentation by a responsible party should be considered for sludge utilization programs. Considerations should include:
  - Quantities of sludge spread, on both wet and dry weight basis, with dates and field locations
  - Crops grown on treated fields
  - Results of all analyses performed. (e.g. sludge, soils, etc.)

The analyses to be performed on soils and sludges are site specific, but in general, sludges should be characterized as to percent solids, NH<sub>4</sub>-N, total kjeldahl N (TKN), and other constituents as suggested by industrial discharges to the wastewater treatment plant.

Surface and subsurface soil characteristics of interest include pH, heavy metal concentrations (Cd, Cu, Ni, Pb, Zn, and other metals of concern known to be in the sludge), TKN, and P. Plant characteristics of interest include plant yield and heavy metal uptake.

 Communities contemplating agricultural utilization of sludges should consider industrial discharge standards to mitigate concentrations of undesirable pollutants in the sludge.

- Consideration should be given to a program to identify soil types throughout the United States which have naturally high Cd concentrations and to determine if crops grown on these soils pose potential health problems. This recommendation is made because the control plot soil at the Las Virgenes site had an unusually high Cd concentration 3 to 4  $\mu g/g$  compared to a typical U.S. range of 0.1 to 0.5  $\mu g/g$ . A literature review showed that Burea, et al., 1973, reported that soil series in California similar to those sampled at Las Virgenes have Cd concentrations as great as, or even greater.
- Good judgement should be exercised in implementing an agricultural utilization program for sludge to assess such factors as groundwater elevations and soil properties (texture, pH, water holding capacity, organic matter, etc.) prior to the implementation of any program.

#### CHAPTER IV

#### INTRODUCTION

The disposal of municipal sewage sludge is a major environmental and technological problem of our time. Increasing quantities of sewage sludge must be disposed of in an environmentally acceptable manner, as a direct result of many factors. Foremost among these is the Federal Water Pollution Control Act of 1972 (PL 92-500) which mandates higher wastewater treatment removal efficiencies of various pollutants. The oil embargo and shortage of natural gas, which resulted in increased costs for energy and has caused many communities to minimize use of sludge conditioning, pretreatment, or incineration facilities, is another factor in this problem.

To add to this dilemma, the disposal of sewage sludge to the oceans or other water bodies, long the primary disposal method for many metropolitan areas, is currently being phased out. Other expedient disposal methods previously used include:

- Subsurface disposal techniques;
- Evaporative and percolation lagoons/ponds, followed by land disposal;
- Landspreading on non-productive land;
- Landspreading as a soil amendment for agricultural and/or forest land; and
- Incineration.

Since these disposal methods were utilized with little thought given to their environmental effects, they are now being investigated and/or re-evaluated. This report represents one of those investigations: an environmental assessment of the landspreading of municipal wastewater treatment sludge for agricultural purposes.

Nine landspreading sites representing a wide range of sludge application rates and characteristics, cropping practices, soils, climatological and geological conditions, and population densities were selected for study. Sludge characteristics, plant uptake and accumulation of heavy metals in soils, past

and current operating procedures, public attitudes, and landspreading costs were assessed at each location.

The study consisted of two separate phases. Phase I involved identification and preliminary screening of potential sites for inclusion in Phase II. Site visits were made to 16 locations meeting desired selection criteria: to validate reported information; sample sludge, surface soils, and plant life; and more accurately assess the desirability of each site for inclusion in Phase II.

Phase II involved a comprehensive and much more detailed on-site investigation at 9 locations selected from 16 Phase I sites. Data obtained during these investigations are summarized for each site in Volume II. These data provide the basis for the detailed individual and comparative site analyses for environmental impact to follow in this volume.

Guidelines can be developed from the results of this and other OSW projects to aid communities in selecting sludge management methods that are both cost effective and environmentally acceptable.

#### CHAPTER V

## CASE STUDY SITE METHODOLOGY

#### SITE SELECTION CRITERIA

At project initiation, various criteria were identified for use in selecting and screening prospective case study sites. Criteria are listed below:

- 1. The site must be 2 ha (5 ac) or larger in area;
- 2. The site must have received sludge for at least five years and must have been used within one of the past three years;
- 3. The sludge solids applied must
  - have originated from a municipal treatment plant,
  - have been stabilized prior to application,
  - have been applied at a rate greater than 11 m tons/ha/yr (5 tons/acre/yr), and
  - not be heat dried;
- Septic tank wastes and industrial sludges were not allowed unless incorporated into the municipal wastewater stream prior to treatment by the municipal facility;
- 5. Several sites receiving dewatered sludge of 15 to 30 percent solids were preferred;
- The site must have been used for agricultural purposes since the sludge had been applied;
- 7. Proprietors of the site had to be willing to cooperate throughout the study;
- 8. Collectively, sites were to represent a wide range of sewage solids application rates, soil characteristics, farming practices, geographical locations, and actual or potential environmental impacts; and

9. A control site with soil and crop characteristics, similar to the study site but not receiving sludge, must have been available.

In addition, the availability of historical information and operating records for each prospective site was desirable. This included:

- Farming practices;
- Types of soils;
- Uses of land prior to application of sludge;
- Number of years, relative frequency, sources and quantities of sewage sludge applied over the years;
- Crop yields; and
- Ultimate use planned for the land.

If a site was or had been part of a demonstration research, or evaluation program, it was not considered for this study. The intent of the study was to have relatively unknown sites investigated in order to further the knowledge of soil amendment through sludge spreading.

## SITE SCREENING METHODOLOGY

Several sources were used to develop a prospective site listing, including: EPA Report No. 670/2-75.049 (Review of Landspreading of Liquid Municipal Sludge by Battelle Research) dated June 1975; literature about related projects; and knowledge of the project team. Letters were sent to state solid waste management and wastewater quality control agencies requesting their assistance in identifying prospective sites.

A tentative site listing was prepared and telephone calls were made to 95 sewage treatment plants and/or communities in 24 states to further determine whether or not the prospective site met the selection criteria. Primarily, the basic eligibility of the study site was established by telephone. Information requested from these telephone contacts included:

- Amount of industrial waste received by the treatment plant;
- Amount of sludge applied to agricultural land;
- Type of sludge processing or pretreatment performed at the STP;

- Availability of sludge spreading data, e.g., cost of spreading, chemical analyses of sludge, etc.;
- Number of years that sewage sludge had been applied to the land;
- Method of sewage sludge application;
- Identification of farmers receiving sludge;
- Distance from STP to farms; and
- Willingness to cooperate in the study.

Sites continuing to appear promising were identified, and telephone calls were made to the farmers receiving sludge for the following information:

- Willingness to cooperate in the study;
- General soil types;
- Distance of treated area(s) from the nearest well, groundwater, and surface water;
- Number of acres to which the sludge had been applied;
- Number of years the sludge had been applied;
- Method of sludge application and any problems associated therewith;
- Rate of application (tons/acre/yr);
- Frequency of application;
- Type of crops grown; and
- Availability of a control site.

If the prospective sites remained within the selection criteria, additional telephone calls were made to the U.S. Department of Agriculture, Soil Conservation Service (SCS) offices to request:

- Soil survey maps for the areas under consideration;
- Specific information about soils and topography of the potential study site and a nearby control site.

The 16 candidate sites ultimately selected met, to the maximum degree, the selection criteria and responded more favorably to the questions than those which were not considered. These sites were:

Las Virgenes, CA
Marshall, MO
Springfield, MO
Chippewa Falls, WI
Dixon, IL
Litchfield, IL
Columbus, IN
Frankfort, IN

Kendallville, IN
Hopkinsville, KY
Wilmington, OH
Xenia, OH
Bethlehem, PA
Easton, PA
Macon, GA
Danville, VA

#### PHASE I SITE VISITATION AND SAMPLING

Personal visits were made to each of the 16 selected sites. In-depth questioning made possible a more complete description of each site, further substantiating the telephone data, and facilitated an assessment of the prospective site relative to the selection criteria.

Specific inquiries were made to obtain:

- 1. Detailed records from the farmer pertaining to
  - Crops,
  - Yields.
  - Fertilizer and pesticide application, and
  - Sludge spreading;
- 2. Detailed records of the treatment processes at the STP, sludge analyses, and data available relative to agricultural utilization of the sludge;
- Personal knowledge of both STP and farms which correlated the new data with the telephone interviews and established a basis for later evaluation of site adequacy for inclusion in Phase II;
- 4. A further verbal commitment from STP and farmers to cooperate through later phases of the project; and
- 5. Sludge, surface soil, and plant tissue samples for an initial assessment of sludge spreading effects.

Samples were obtained as follows:

## Sludge

A single composite sludge sample consisting of several grab subsamples was taken at each STP and analyzed for:

```
calcium (Ca)
                                      chromium (Cr)
moisture content
                                     cobalt (Co)
copper (Cu)
iron (Fe)
volatile solids (V.S.)
chlorides (C1)
sulfates (SO<sub>4</sub>)
                                      lead (Pb)
organic nitrogen
                                     magnesium (Mg)
manganese (Mn)
nitrate-nitrogen (NO<sub>3</sub>-N)
ammonia-nitrogen (NH4-N)
boron (B)
                                      mercury (Hg)
                                     molybdenum (Mo)
nickel (Ni)
potassium (K)
phosphorus (P)
                                      selenium (Se)
sodium (Na)
arsenic (As) cadmium (Cd)
                                      silver (Ag)
                                      zinc (Zn)
```

Initial analysis used emission spectroscopy; Cd, Cu, Ni, Pb, and Zn were also analyzed using flame atomic absorption (AA) techniques.

#### Soils

Fifty representative soil borings were taken with a 2.5 cm (l in) diameter stainless steel split-tube sampler to a depth of approximately 20.3 cm (8 in) from plots in both treated and control fields, composited, and analyzed for pH, Cd, Cu, Ni, Zn, and Pb (metals by AA).

## Plants

Approximately 1.8 kg (4 lbs) of representative plant tissues (where available) were obtained from the treated and control fields and analyzed by AA techniques for Cd, Cu, Ni, Pb, and Zn.

## PHASE II SITE SELECTION

As a result of the in-depth questions, sampling, and personal observations, additional information was now available such as:

- Evaluative data including the results of sludge, surface soil, and plant chemical analyses;
- Calculations of soil accumulation and plant uptake of heavy metals in relation to total sludge loadings;

- Personal observations of the quality of the record keeping by farm and STP management, as well as the degree of cooperation that could be expected; and
- Brief but comprehensive descriptions and reports of each study site.

At a December 1975 meeting of all project participants (contractor, EPA, and project consultants), the 16 Phase I sites were reviewed and the nine sites listed below chosen as best representing diverse crops, management practices, climate, and soil conditions desired for study in this project.

Las Virgenes, CA
Macon, GA
Columbus, IN
Frankfort, IN
Kendallville, IN
Hopkinsville, KY
Springfield, MO
Wilmington, OH
Chippewa Falls, WI

#### PHASE II SITE VISITATION AND SAMPLING

Site characterization, site analysis, and case study development were performed in Phase II. The information sought was similar to that identified in Phase I, but more comprehensive and expanded to include public opinion of sludge management practices in the case study locations.

## Interviews

Interviews conducted with farmers, STP personnel, Soil Conservation Service officials, and others confirmed information obtained in Phase I and gathered more details concerning the following:

#### Farmer--

- Field preparation techniques:
  - Soil preparation for planting,
  - Equipment used for field preparation, and
  - Tilling depths for soil;
- Field additives:
  - Use of fertilizers, pesticides, herbicides, and

- Incorporation of field additives into/onto the soils/plants;
- Crop rotation practices;
- Irrigation techniques, if any;
- Yields, historical and current, when available;
- Complaints from neighbors and/or other farmers concerning the use or handling of sludge;
- Health problems either personal or related to the handling of sludge or with ingestion by people or animals of crops grown on the sludge amended fields: and
- End use of the crop(s) grown on the treated land.

## Treatment Plant (STP)--

- Detailed descriptions of the treatment process;
- Records of amounts of sludge distributed monthly and/or annually to each study site for each farm's sludge-spreading history;
- Copies of the last 12 month's STP operating records;
- A history of capital equipment purchases necessary for the sludge spreading operation (including age, date of purchase, and use);
- Records of labor, operation and maintenance (0&M), energy, chemicals, and other costs incurred for sludge disposal to the land; and
- Any physical, health, transportation, regulatory, or environmental problems associated with the spreading of sewage solids.

## Soil Conservation Service (SCS) --

- Knowledge of soil conditions in the general area of both treated and control fields:
- Verification of the soil series of both plots; and
- Opinions of environmental effects of the sludge spreading operation.

#### Other--

Local newspapers, city and county sanitarians, county departments of health, and other city and county officials were queried regarding:

- General public opinion of the use of sewage sludge as a soil amendment;
- Knowledge of any problems (environmental, health, etc.) associated with or assumed to be the result of the practice; and
- Existence of any newspaper articles, pro or con, on the spreading of sewage sludge.

#### Sampling Program

At the same time information was being assembled on the above, an extensive sludge, soil, and plant sampling program was being performed as described in detail in Chapter VI; analytical procedures are described in the appendix of this volume.

#### CHAPTER VI

#### SUMMARY DESCRIPTION OF CASE STUDY SITES

The selected case study sites encompassed a wide range of study conditions, including geographical location; climate; crops; STP operating techniques; and sludge generation, spreading, and disposal practices. Volume II of this report contains a detailed description of each case study site, and the results of the sampling program performed at each location.

#### LOCATION

Figure 1 indicates the geographical location of each of the nine case study sites in the U.S. Three sites were located in Indiana, and one each in California, Georgia, Kentucky, Missouri, Ohio. and Wisconsin.

#### CLIMATE

The climatological conditions at the case study locations showed wide variation, ranging from a semi-arid southern California site (Las Virgenes) to severe winter conditions at Chippewa Falls, Wisconsin, and southern humidity at Macon, Georgia. Selected climatological factors for each location are presented on Table 6.

#### CROPS

Comparative information on crops grown at each of the nine sites is shown in Table 7. Two of the sites were planted in alfalfa and two in fescue; the remaining five were in wheat, cheatgrass, corn, soybeans, and ryegrass. Eight sites had identical treated and control crops; however, at Macon, Georgia, the treated crop was cheatgrass and the control crop, oats.

Three of the sites produced crops for human consumption: wheat at Frankfort, Indiana; corn at Columbus, Indiana; and soybeans at Chippewa Falls, Wisconsin. The remaining six sites produced animal feed crops, none for dairy cattle use.

#### STP INFORMATION

Comparative STP information is presented in Table 8. Average daily influent to the STP's ranged from a low of 6,000

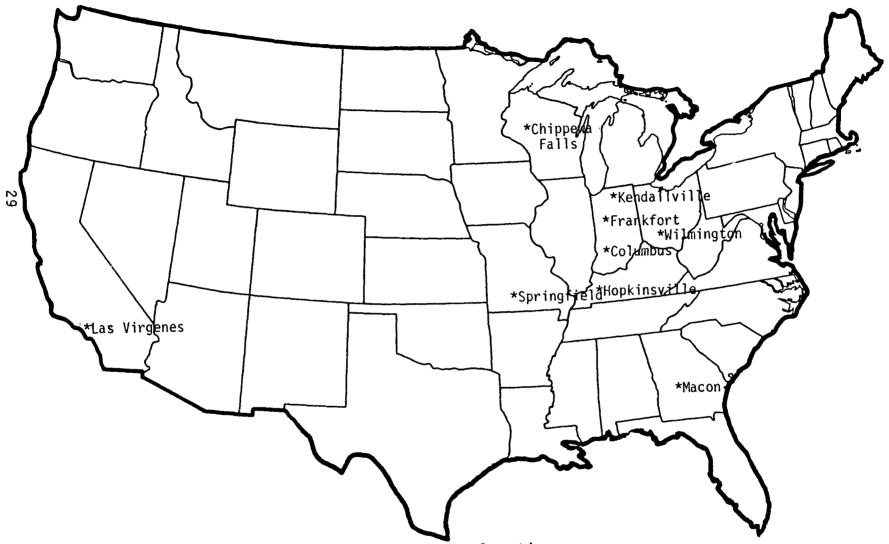


Figure 1. Study site locations.

TABLE 6. COMPARATIVE WEATHER DATA FOR 1975

	Pre	ecipitat	ion (in	)			Tem	peratur	е		
	Snow, Water Equiv. Ice Pellets			Averages ( <sup>O</sup> F)		Me Maxi		of Days Mini			
Site	Total	Max 24 hr	Total	Max 24 hr	Daily Max.	Daily Min.	Monthly	90° & Above	32 <sup>0</sup> & Below	32 <sup>0</sup> & Below	0 <sup>0</sup> & Below
Macon, GA	55.48	3.23	0	0	75.9	53.5	64.7	46	0	37	0
Las Virgenes, CA	10.70	1.84	0	0	7.19	54.1	63.0	10	0	0	0
Wilmington, OH	44.23	2.25	16.6	3.9	64.9	44.9	54.9	35	9	99	0
Springfield, MO	53.94	4.83	35.2	11.9	67.0	44.4	55.7	47	13	105	0
Chippewa Falls, WI	26.44	n.d.*	63.6	n.d.*	55.9	36.4	46.2	28	81	156	22
Hopkinsville, KY <sup>†</sup>	47.67	6.30	10.7	12.0	68.9	45.5	57.2	53	13	98	2
Frankfort, IN <sup>#</sup>	37.92	4.45	21.2	8.0	61.4	40.0	50.7	23	37	138	8
Kendallville, IN	36.73	1.92	27.1	4.9	59.4	41.4	50.4	10	39	137	2
Columbus, IN	46.72	3.73	34.8	5.3	62.2	43.4	52.8	8	25	120	2

<sup>\*</sup>No data

<sup>†</sup>Means - revised 1972 #Means - revised

TABLE 7. COMPARATIVE STUDY SITE INFORMATION

Site	Treated Plot Crop	Crop Rotation Cycles	Crop Use	Average Annual Precipitation (cm)
Macon, GA	cheatgrass*	none	non-dairy cattle feed •	112.9
Las Virgenes, CA	ryegrass	none	non-dairy cattle feed	35.7
Wilmington, OH	alfalfa	alfalfa-corn oats	non-dairy cattle feed	99.2
Springfield, MO	fescue	various	non-dairy cattle feed	100.8
Chippewa Falls, WI	soybeans	none	open-market	73.9
Hopkinsville, KY	fescue	none	non-dairy cattle feed	121.3
Frankfort, IN	wheat	wheat-corn	flour mill	96.3
Kendallville, IN	alfalfa	corn-soybeans- oats-alfalfa	non-dairy cattle feed	90.9
Columbus, IN	corn	none	distillery	98.4

<sup>\*</sup>Control plot crops from all sites were the same as the treated plot except Macon, GA where control crop was oats.

TABLE 7 (continued)

			Sludge Application					
Site	Surface Soil Type*	Depth to Groundwatert (m)	Total Sludge Treated Area (ha)	Rates Annual <sup>#</sup> Total** (m tons/ha)		Years of Sludge Spreading**		
Macon, GA	sandy loam	1.5	50.6	28 ‡	308‡	11		
Las Virgenes, CA	clay loam	213	7.3	50	149	7		
Wilmington, OH	silt loam	18	22.7	6.8	116	17		
Springfield, MO	silt loam	41	12.6	15.8	237	15		
<sup>ω</sup> Chippewa Falls, WI	sand	23	56.7	16 ‡	<b>80</b> ‡	6		
Hopkinsville, KY	silt loam	10	21	22 ‡	6 <b>6</b> ‡	.9		
Frankfort, IN	silt loam	21	18.5	30 ‡	360 <del>†</del>	12		
Kendallville, IN	clay loam	30	40.5	19.7	81	13		
Columbus, IN	sandy loam	5	14.2	65	326	5		

<sup>\*</sup>USDA Classification +All ground water depths reported as estimated static levels # 1975

<sup>\*\*</sup> Through Dec. 31, 1975 †Estimated

TABLE 8. COMPARATIVE SEWAGE TREATMENT PLANT (STP) INFORMATION

STP	Average Flow Capacity (cu m/day)	Population (1975)	Population Equivalent*	Type of Secondary Treatment	Estimated Industrial Contribution (percent)
Macon, GA	45,400	107,000	125,000	Trickling filter	30
Las Virgenes, CA	17,000	40,000	75,000	Activated sludge	10
Wilmington, OA	7,600	10,000	19,000	Activated sludge	25
Sprinafield, MO	75,700	135,000	164,000	Activated sludge	15
				w/Kraus modificatior	1
Chippewa Falls, WI	13,200	12,500	51,000	Activated sludge	65
Hopkinsville, KY	8,300	21,250	28,710	Trickling filter	15
Frankfort, IN	11,700	15,000	12,900	Trickling filter	30
Kendallville, IN	6,000	9,200	22,700	Trickling filter	25
Columbus, IN	24,400	26,500	50,000	Activated sludge	67

<sup>\*</sup>Based on 0.17 lbs  $BOD_5/capita/day$ 

<sup>+</sup>Combined sewage and storm drains

TABLE 8 (Continued)

STP	Sludge Generated In 1975 (dry metric tons/year)	Sludge Generated/ Capita/day (kg)	Sludge Digestion Process	Average Solids Content (%)	Disposal Costs (\$/dry m ton)
Macon, GA	642	0.03	2-stage anaerobic	11.4	7.98
Las Virgenes, CA	1120	0.15	aerobic	10.0	128.07
Wilmington, OH	81	0.04	anaerobic	5.0	26.92
Springfield, MO	1815	0.07	2-stage anaerobic	3.0	19.58
Chippewa Falls, WI	254	0.11	anaerobic	3.0	79.08
Hopkinsville, KY	178	0.05	2-stage anaerobic	10.0	22.28
Frankfort, IN	320	0.12	anaerobic	6.5	19.04
Kendallville, IN	795	0.47	anaerobic	4.0	26.48 <sup>#</sup>
Columbus, IN	2174	0.45	ana erobic	5.0	17.65

<sup>#</sup>City employees, private hauling firm.

cu m/day (1.6 mgd) at Kendallville, Indiana, to a high of 75,700 cu m/day (20 mgd) at Springfield, Missouri.

All of the STP's provided secondary treatment: four utilized trickling filters, the remaining five providing either conventional or modified activated sludge processes. Las Virgenes, California provided aerobic digestion and dewatering of sludge; the other eight sites simply employed anaerobic digestion and did not dewater. Three of the sites (Macon, Georgia; Springfield, Missouri; and Hopkinsville, Kentucky) employed two-stage anaerobic digestion.

Industrial input to the STP's ranged from an estimated low of 10 percent at Las Virgenes, California to a high of 67 percent at Columbus, Indiana. Population equivalents ranged from a low of 12,900 at Frankfort, Indiana to a high of 164,000 at Springfield, Missouri.

SLUDGE

#### Generation

Based on 1975 STP sludge generation data and average sludge solids concentrations (either estimated by STP personnel or calculated from STP or contractor data), the daily generation of dry sludge solids ranged from a low of 0.03 and 0.04 kg/capita/day at Macon, Georgia and Wilmington, Ohio, to highs of 0.45 and 0.47 kg/capita/day at Columbus and Kendallville, Indiana. The latter sites precipitate phosphorus by chemical addition; the former do not utilize any chemical treatment.

#### Spreading

The duration of sludge spreading varied from five years at Chippewa Falls, Wisconsin to seventeen years at Springfield, Missouri. Annual sludge application rates ranged from 6.8 to 65 m tons/ha at Wilmington, Ohio and Columbus, Indiana respectively, while total amounts of sludge applied ranged from 66 to 360 m tons/ha at Hopkinsville, Kentucky and Frankfort, Indiana.

Spreading at two of the sites - Las Virgenes, California and Chippewa Falls, Wisconsin - stopped as of the 1976 harvest, the former due to an odor complaint and impending sale, and the latter following sale of the property to the city for a park.

# Disposal

Disposal costs ranged from a low \$7.98 to a high \$128.07/dry m ton at Macon, Georgia and Las Virgenes, California, respectively. Table 9 presents additional STP comparative information concerning items such as types of transportation

TABLE 9. COMPARATIVE SEWAGE TREATMENT PLANT DATA

Site	Haul Vehicles	Type of Discharge	No. of Disposal Sites	One-way Haul Distance to Study Site	1975 O&M Cost* (\$/dry m ton)
Macon, Ga	GMC 5 cu yd dump truck body w/5.2 cu m tank	gravity - splash pan	1	(km) on STP property	1.09
Las Virgenes, CA	1971 Chevrolet truck body modified to hold two 3 cu yd Dumpsters	dewatered sludge piled & spread for field-drying	6	3	15.70
Wilmington, OH	1975 Ford 1 ton truck body w/3.8 cu m tank	gravity - splash plate	1	10	3.74
Springfield, MO	1965 Chevrolet truck body w/ll.4 cu m tank	gravity - spread- ing bars	11	0.5	3.13
Chippewa Falls, WI	1967 5 ton Army truck body w/8.7 cu m tank	gravity - no splash plate	2	8	40.84
Hopkinsville, KY	1966 l 1/2 ton Chevrolet truck body w/3.8 cu m tank	gravity - no splash plate	4	0.3	3.81
Frankfort, IN	W.W. II Army truck w/6.6 cu m tank W.W. II Army truck w/5.7 cu m tank	gravity - no splash plate	1	1.5	2.05

<sup>\* 0&</sup>amp;M - Operation and Maintenance

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TABLE 9 (continued)

Site	Haul Vehicles	Type of Discharge	No. of Disposal Sites	One-way Haul Distance to Study Site	1975 O&M Cost† (\$/dry m ton)
Kendallville, IN <sup>#</sup>	Private contractor truck w/9.5 cu m tank	gravity - no splash plate	1	(km) 3	none
Columbus, IN**	1965 Dodge truck body w/9.1 cu m tank 1975 Marmon truck body w/9.8 cu m tank	gravity - ten spreaders	4	0.2	3.67

+ 0&M - Operation and Maintenance

# Sludge disposal contracted to private hauler @ \$10/load

\*\*88% of the sludge was delivered to the study site via irrigation pipe

vehicles, haul capacities, one-way distances to the study sites, and methods of discharge. The final column presents normalized 1975 0&M costs.

#### PUBLIC AWARENESS

Interviews were conducted at each site with representatives of the local newspapers, city and/or county health departments, and various other agencies to assess public awareness and acceptance of sludge disposal to nearby agricultural land. • The responses ranged from complete unawareness to a working knowledge of the practice. Those officials who were cognizant of sludge spreading supported the program; no opposition was voiced.

Newspaper representatives at seven of the sites were not aware of any articles, pro or con, relative to sludge spreading, although two articles which discussed the Phase I and II contractor visits appeared in the local Frankfort, Indiana newspaper. Several syndicated column articles dealing with farm and sludge spreading equipment appeared in the local Chippewa Falls, Wisconsin newspaper.

Numerous articles pertaining to the Las Virgenes Water District's effluent disposal program have appeared in the local newspaper which serves the Las Virgenes Valley in Southern California. This community expresses a high degree of opposition to effluent discharge and has been instrumental in thwarting the Las Virgenes Water District's attempts to obtain a discharge permit. The editor could not recall publishing any articles dealing with sludge disposal.

#### ENVIRONMENTAL PROBLEMS

Table 10 presents a summary of environmental and health-related items compiled from other interviews. Odor complaints were reported at three of the sites; two were not considered serious, and no further action was taken by any of the involved parties. At Las Virgenes, California, as was mentioned previously, the complaint was serious enough to stop the spreading operation at the study site.

Only two of the states (Ohio and Wisconsin) had published sludge spreading guidelines during the interview phase of this project (summer 1975), although California, Georgia, Indiana, and Missouri have documents and drafts in various stages of development. None of the sites were disposing of sludge under permit.

No health problems were reported. As can be seen from Table 11, some of the STP's offered voluntary innoculations; others offered none. At Las Virgenes, California, Chippewa Falls, Wisconsin, and Columbus, Indiana, innoculations were mandatory.

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TABLE 10. SUMMARY OF REPORTED ENVIRONMENTAL PROBLEMS AND EXISTING STATE REGULATIONS

Site	Reported Environmental Problems	Existing State Landspreading Regulations
Macon, GA	None	None
Las Virgenes, CA	Odor complaint has stopped spreading at study site. Other STP disposal sites were not affected.	In working draft stage as of 2/1/76
Wilmington, OH	None	Bulletin #598, "Ohio Guide for Land Application of Sewage Sludge," revised May 1976
Springfield, MO	Some odor complaints - none serious	In development stage
Chippewa Falls, WI	None	Technical Bulletin #88, "Guidelines for the Application of Wastewater Sludge to Agricultural Land in Wisconsin," 1975
Hopkinsville, KY	None	None
Frankfort, IN	One odor complaint - not serious	In development stage
Kendallville, IN	One complaint about possible "runoff" contamination - complaint not investigated, and dismissed by County Sanitarian	In development stage
Columbus, IN	None	In development stage

### TABLE 11. STP EMPLOYEE INNOCULATION PROGRAM

Macon, GA Voluntary -	influenza,	tetanus,	typhoid
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Las Virgenes, CA Mandatory - typhoid, tetanus, polio

Wilmington, OH None

Springfield, MO Voluntary - various innoculations offered

Chippewa Falls, WI Mandatory - typhoid, tetanus, polio

Hopkinsville, KY None

Frankfort, IN None

Kendallville, IN None

Columbus, IN Mandatory - typhoid, tetanus, polio

#### CHAPTER VII

# FIELD SAMPLING PROGRAM SCOPE AND OBJECTIVES

The Phase I sampling program provided preliminary data sufficient to select the nine case study sites; the program for Phase II sampling was designed to provide an expanded data base, so that the environmental impact of the sludge landspreading at each site could be properly assessed, conclusions drawn, and recommendations made. A detailed sampling program was developed and executed for the chemical, physical, and microbiological examination of sludges, soils, and plants at each site.

#### SLUDGE

The sampling at each sewage treatment plant was performed as follows:

- At six-hour intervals, five prepared l-l plastic "cubitainers" were filled with grab samples of combined primary and secondary sludge (before they entered the digester). These samples were labeled "raw-frozen."
- Another five cubitainers were filled, over a 24-hour period, with grab samples of stabilized sludge. These samples were taken when the sludge entered the tank truck for field distribution and were labeled "stabilized-frozen."
- Eight of the cubitainers were placed in a freezer and frozen for a minimum of 16 hr.
- One cubitainer sample each, of the raw and stabilized sludge taken from the same sampling points, was preserved by chilling in a refrigerator at approximately 2 to 4°C. These samples were labeled "raw-chilled" and "stabilized-chilled," respectively.

Each of the cubitainers was then wrapped in several layers of newspaper, placed in a styrofoam-lined cardboard box to which three or four frozen "blue-ice" packets had been added (to maintain sample integrity) and shipped air freight to the contractor. Upon arrival, all samples were immediately transferred to either chilled or frozen storage.

Each set of four frozen "raw" grab samples was allowed to thaw at 2 to  $4^{\circ}\text{C}$  and then composited in four  $1-\ell$  beakers which had been acid-cleaned in the same manner as the cubitainers; these were stirred thoroughly and poured back into the original cubitainers, each of which then held a composite. This procedure was repeated for the "stabilized-frozen" sludge, and all eight samples then returned to the freezer prior to shipment to the contractor.

Four cubitainers (one each of chilled and frozen, raw and stabilized sludge) were analyzed for:

- Helminths,
- Protozoans,
- Total aerobes,
- Salmonella,
- Shigella,
- Fecal coliforms, and
- Fecal streptococci.

The remaining frozen stabilized samples were analyzed for:

```
рΗ
                                    Arsenic (As)
Dieldrin
                                    Cadmium (Cd)
DDT
                                    Calcium (Ca)
PCB's
                                    Chromium (Cr)
Moisture content
                                    Cobalt (Co)
Volatile solids (V.S.)
                                    Copper (Cu)
Chlorides (C1)
                                    Iron (Fe)
Sulfates ($0<sub>4</sub>)
Organic-nitrogen
                                    Lead (Pb)
                                    Magnesium (Mg)
Nitrate-nitrogen (NO_3-N)
                                    Manganese (Mn)
Ammonia-nitrogen (NH<sub>4</sub>-N)
                                   Mercury (Hg)
Boron (B)
                                    Molybdenum (Mo)
Potassium (K)
                                    Nickel (Ni)
Phosphorus (P)
                                    Selenium (Se)
Sodium (Na)
                                    Silver (Aq)
```

As previously indicated, 1- $\ell$  plastic "cubitainers" were used for the sludge sampling. The cubitainers and caps were prepared by:

- Rinsing with hot water,
- Allowing to cool.
- Removing all traces of contaminants with a reagent grade hydrochloric acid diluted 1:1 with distilled water,
- Rinsing several times with cold tap water,

- Rinsing several more times with ultra pure (18 megohm) water,
- Collapsing the cubitainers, and
- Securing the caps tightly.

#### SOIL SAMPLES

Commercially available 1- or 2-gal polyethylene bags were used for soil containers; all samples were double-bagged.

At each study site, both the treated and control plots were divided into five rectangular sub-plots of approximately equal area; the Las Virgenes control plot was divided into only three sections because of its shape and size.

# Surface Soils

Surface soil samples were taken to a depth ranging from 15 to 30 cm (6 to 12 in); this corresponds to the plow depth or "cultivation zone" (Ap) defined by the farmer. Twenty to forty soil corings were taken from each of the five sections in both the treated and control plots at each site. A 2.5 cm (1 in) diameter stainless steel split tube sampler was used. The sampling was performed in such a manner that cores were drawn from all areas within each section, except at Las Virgines, California, where samples were withdrawn on the section line as opposed to within an area. The number of cores withdrawn varied directly with the depth of the Ap zone; the deeper the Ap zone, the fewer the corings.

Any miscellaneous non-soil debris was removed from each coring prior to the soil being placed in the sample bag; each section was ultimately represented by approximately 2.3 kg (5 lbs) of soil samples.

The soil sample representing each section was analyzed for total Cd, Cu, Ni, and Zn.

A composite sample, consisting of a representative amount from each of the five section samples, was analyzed for:

pH
Moisture content
NO3-N
NH4-N
Organic N
Organic carbon
Cation exchange capacity
Textural composition

PCB's
Total Cd, Cu, Ni, Pb, and Zn
DTPA-extractable Cd, Cu, Ni, and Zn
Total aerobes
Salmonella
Shigella
Fecal coliforms
Fecal streptococci

DDT Dieldrin

#### Helminths Protozoans

One bulk sample of approximately 115 kg (250 lbs) of soil from the Ap zone of both the treated and control plots of each site was sent to the EPA Municipal Environmental Research Laboratory, Cincinnati, Ohio, for greenhouse studies.

## Subsurface Soil

Subsurface soils were considered to be those below the Ap zone; samples were obtained from treated and control plots at all nine sites. A 7.6 cm (3 in) dia stainless steel bucket auger was used to obtain corings except at Kendallville, Indiana, where a 5.1 cm (2 in) dia stainless steel pneumatic coring tool mounted on a vehicle was used.

Discussions between the farmer, local SCS representatives, and the contractor's soil consultant, with the aid of soil survey maps, defined the layers (horizons) of the samples.

The A horizon typically was the zone between 20 and 40 cm (8 and 16 in), the B horizon between 40 and 61 cm (16 and 24 in), the C horizon between 61 and 91 cm (34 and 36 in), and the D horizon between 91 and 122 cm (36 and 48 in). However, at Kendallville, Indiana, the A horizon was between 20 and 30 cm (8 and 12 in), and the B horizon between 30 and 61 cm (12 and 24 in). At Springfield, Missouri, the C horizon was between 61 and 81 cm (24 and 32 in), the D horizon between 81 and 122 cm (32 and 48 in); and at Las Virgenes, California, the two lowest horizons, between 61 and 91 cm (24 and 36 in) and 91 and 122 cm (36 and 48 in), were labeled B and C.

Samples were withdrawn from the four horizons in each of the five subplots at all sites; each sample was placed in a bag specific for the soil horizon and depth and labeled horizon A, B, C, or D. Thus, each plot was represented by twenty subsurface samples, one from each horizon of each section. Total sample size for each composited horizon was approximately 2.5 kg (5 lbs).

The uppermost portion of soil, that from the Ap zone, as well as miscellaneous non-related debris sloughed from the sides of the hole or the surface, was discarded from the auger before the samples were bagged.

Small subsamples were withdrawn in the field from each of the surfaces and four horizon subsurface samples to form plot composites for both the treated and control plots. These were immediately placed in refrigerated storage (2 to 4°C) and marked for bacteriological and parasite examinations; the remaining samples were stored under ambient conditions.

Each of the chilled soil samples was boxed in a styrofoam-lined cardboard container to which three or four frozen "blue-ice" packets had been added; all of the soil samples were then sent via air freight to the contractor. The chilled samples were immediately transferred to 2 to 4°C storage.

The unchilled soil samples were unpacked and spread in discrete piles on a clean, tarped floor. As each sample bag was opened, a small subsample was withdrawn into a whirl-pak bag to determine field moisture. The samples were then air dried from 7 to 14 days, depending on the original moisture level. Each sample was then ground thoroughly (to less than 2 mm) in an Iler Disk Pulverizer.

The series of cores taken from each horizon of each section were analyzed individually for total Cd, Cu, Ni, and Zn.

Four composite samples representing the four distinct depths throughout all five sections of each plot were prepared from the above samples and analyzed for:

pH
Moisture content
NO3-N
NH4-N
Organic N
Organic carbon
Cation exchange capacity
Textural composition

Total aerobes
Salmonella
Shigella
Fecal coliforms
Fecal streptococci
Helminths
Protozoans

as well as Total Cd, Cu, Ni, Zn, and Pb, and for DTPA extractable Cd, Cu, Ni, and Zn.

All of the analyses were performed for treated and control plots.

#### PLANT SAMPLES

Plant samples were taken from treated and control plots of all nine sites. Care was taken to ensure that the portion of the plant sampled was not contaminated with dried or adhering sludge. This was performed by sampling aerial portions of the various plants and careful visual inspection.

Wheat and grasses were sampled by shearing and retaining the aerial portion; approximately 10 to 30 bunches of grasses and wheat with 10 to 100 individual stalks or blades were taken in each section. Since several plots contained more than one type of grass, the desired plants were hand sorted from the others. Corn and soybean leaves were handpicked. The crops at Frankfort and Columbus, Indiana and Chippewa Falls, Wisconsin produced edible grains of wheat, corn and soybeans, respectively;

these were sampled just before harvesting, the wheat with a pair of shears, the corn by hand picking, and the soybeans with a combine.

Samples were taken from the entire area of each section except as noted in Volume III.

Each of the section samples resulted in approximately 2 kg (4.5 lbs) of plant tissue. Samples were double-bagged in 1- or 2-gal polyethylene bags which were refrigerated at 2 to 4°C until shipment. All samples were placed in styrofoam-lined cardboard boxes to which three or four frozen "blue-ice" packets had been added and were air freighted to the contractor, where they were again refrigerated at 2 to 4°C. The edible grains were shipped under ambient conditions.

Two composites of leaves were prepared for each plot by mixing equal weight portions. One of the composites was analyzed for:

- Organic-N
- DDT
- Dieldrin
- PCB's
- Total Cd, Cu, Ni, Zn, and Pb.

The other composite was analyzed for:

- Total aerobes,
- Salmonella.
- Shigella.
- Fecal coliforms, Fecal streptococci, •
- Helminths, and
- Protozoans.

The edible grain samples were composited as above, except for the wheat which was air-dried for seven days and threshed before compositing. The composites were chemically analyzed for total Cd, Cu, Ni, Zn and Pb. Bacteriological and parasite examinations were not made of the edible grains.

# CHAPTER VIII DATA ANALYSIS

#### INTRODUCTION

The data analysis is presented in two sections as follows:

- Section I Individual Study Site Analyses.
   The sludge, soil, plant, and microbiological findings from the field sampling program and the information gathered at each study site are summarized and analyzed in this section. The raw data supporting the analyses are presented in Volume II for each study site.
- Section II General Evaluation.
   The site data are collectively examined and compared to data obtained during related studies in this section.
   Observations and conclusions are drawn within the framework of the study constraints.

The following were utilized in completing the data analysis:

- Reference is made to "reported ranges" and "median" concentration values when discussing sludge characteristics. These data are from Sommers (1977) and Furr, et al. (1976), and are presented in Table 12, along with sludge analytical data from all nine case study sites.
- Calculation of available N in the sludges was based on an assumed annual availability of 50 percent of the ammonia-N and 20 percent of the organic-N. It was assumed that organic-N was unavailable in the following year. NO<sub>3</sub>-N levels were so low that they were not considered in the calculations.
- The current (1977) fertilizer equivalent value of N was assumed at \$0.44/kg.
- The plant availability of sludge-borne P is not precisely known. Therefore, it was assumed that 100 percent of the sludge P was plant available.

TABLE 12. CHEMICAL COMPOSITION OF STABILIZED SLUDGES FROM ALL STUDY SITES

Parameter*	Macon, Georgia	Las Virgenes, California	Wilmington, Ohio	Springfield, Missouri	Chippewa Falls Wisconsin
Vol. Solids (%)	36.9	74.7	52.4	59.2	56.4
Total N	4.55	4.84	3.81	8.48	4.58
NO3-N	7.80	0.41	13.7	51.4	13.0
NH4-N (%)	1.10	0.32	1.01	4.18	2.12
Org-N (%)	3.45	4.52	2.80	4.30	2.46
P (%)	1.23	1.71	3.55	1.74	1.34
K (%)	0.22	0.16	0.21	0.54	0.22
Na (%)	0.39	0.20	0.20	0.80	0.69
Ca (%)	2.78	2.65	3.14	5.92	1.39
Mg (%)	1.21	0.49	0.92	0.63	0.52
S0 <sub>4</sub>	348	81.7	105	111	66.0
C1 (%)	0.10	0.15	0.28	0.50	1.09
Ag	1.58	3.45	2.42	0.95	5.92
As	2.21	4.13	1.13	6.21	2.52
В	72	71.6	37.5	40.5	42.5
Cd	11.9	11.1	11.2	56.1	7.03
Cr	1,130.	150	2,240	2,430.	1,270.
Co	21.3	6.39	6.96	9.07	6.17
Cu	960.	<b>950</b> .	410.	820	1,370
Fe	23,600	6,920	13,600	13,500	13,300
Hg	16.4	7.27	1.93	10.6	24.1
Мn	600.	480.	250.	450	220
Мо	14.5	10.2	45.5	43.4	6.60
Vi	140.	50.	30.	300	20.
Pb	610.	40	1,110.	150.	100
Se	13.7	7.02	4.23	8.17	7.60
<sup>2</sup> n	1,770.	1,350	45,800	3,310	1,190
	91.3	85.6	95.8	98.3	97.5
1 <sub>2</sub> 0 off	7.50	6.60	7.80	8.30	7.80

TABLE 12 (continued)

Parameter*	Hopkinsville, Kentucky	Frankfort, Indiana	Kendallville, Indiana	Columbus, Indiana	Comparative S Range	ludge Data† Median
Vol. Solids (%)	41.1	48.3	36.1	55.0		
Total N	2.37	3.04	1.76	3.57	0.5-7.6	4.8
N03-N	10.2	13.4	4.05	60.0	2-4,900	140
NH4-N (%)	0.54	0.74	0.28	1.27	0.005-6.76	0.09#
Org-N (%)	1.83	2.30	1.48	2.30	< 0.1-10.8	3.21
P (%)	1.56	1.57	1.41	1.33	< 0.1-14.3	2.3
K (%)	0.13	0.22	0.53	0.32	0.02-2.64	0.3
Na (%)	0.20	0.62	0.65	0.61	0.01-3.07	0.24
Ca (%)	6.25	4.76	2.58	2.26	0.1-25.0	3.9
Mg (%)	0.36	0.55	1.23	0.59	0.03-1.97	0.45
S0 <sub>4</sub>	10.0	10.0	8.60	118	0.6-1.1	0.8
C1'(%)	0.03	0.12	0.67	0.32	0.05-1.02**	0.29*
Ag	1.16	1.95	10.2	1.23	5-150**	20 <del>†</del>
As	1.14	3.06	5.11	2.19	6-230	10
В	32.1	43.6	43.8	38.7	4-760	33
Cd	7.60	1,500	50.0	5.70	3-3,410	16
Cr	380	3,990	2,010	410	10-99,000	890
Со	9 <b>.6</b> 8	16.2	24.9	24.1	1-18	4.0
Cu	550	7,140	730	390	84-10,400	850
Fe	7,460	21,400	48,100	10,200	< 1,000-153,000	11,000
Нд	9.82	4.41	3.08	2.60	0.5-10,600	5.0
Mn	200	320	9,870	470	18-7,100	260
Mo	11.1	6.77	7.54	21.1	5-39	30
Ni	60	500	80	160	2-3,520	82
Pb	90	250	7,480	100	13-19,700	500
Se	6.36	5.68	17.2	6.64	1.7-8.7+	2.7 <sup>†</sup>
Zn	1,470	2,150	7,190	740	101-27,800	1,740
H <sub>2</sub> 0	87.8	93.8	91.0	96.0	•	
рĤ	7.5	7.3	7.5	8.2		

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# TABLE 12. (continued)

\*All units in  $\mu g/g$  (oven-dry weight basis) unless otherwise noted.

†Dewatered and dried sludges included.

#Results from analyses of more than 250 sewage sludge samples from approximately 150 STP's in the north central and eastern regions, U.S. (Sommers, 1977). The S values are for total S, not SO4.

\*\*Data of 42 sewage sludges from locations in England and Wales (Berrow and Webber, 1972).

†Data of sewage sludges from 16 American cities (Furr, et al., 1976).

- The current fertilizer equivalent value of P was assumed at \$1.00/kg.
- The potential for groundwater contamination was based on a combination of the chemical and physical properties of the sludges and soils to a depth of 122 cm. The analyses were based only on the potential migrations of Cd, Cu, N, Zn, and Pb in the soil profile.

#### SECTION I - INDIVIDUAL STUDY SITE ANALYSES

# Macon, Georgia

Sludge Characteristics --

Table 12 shows a comparison of Macon sludge composition with Sommers' data for 150 STP's.

Nitrogen and phosphorus--Total N (4.55 percent), organic-N (3.45 percent), NO<sub>3</sub> (7.8  $\mu$ g/g), and NH<sub>4</sub>-N (1.1 percent) from the Macon STP were within the ranges previously reported for sludges.

The Macon STP method of spreading liquid sludge onto the surface without immediate incorporation into the soil probably results in a substantial loss of ammonia to the atmosphere. Assuming that 20 percent of the organic-N and 50 percent of the NH4-N is available to plants, 1 m ton of Macon sludge would contain 12.4 kg of available N and have a value of \$5.46 (at current fertilizer prices).

The P concentration (1.23 percent) was somewhat less than the reported median concentration (2.3 percent). How much of the sludge-borne P was available to plants is not precisely known, since the Macon sludge contained considerable Fe which can tie up P as an insoluble complex. If, however, all of the P in the sludge is considered equivalent to commercial sources of P, 1 m ton of the Macon sludge would supply the equivalent of 12.3 kg and have a value of \$12.30. In terms of both N and P, the sludge would have a value of \$17.76/m ton.

The annual estimated application rate of the Macon STP sludge was approximately 28 m tons/ha, equivalent to 347 kg/ha of available N and 344 kg/ha of total P, and was in excess of yearly plant requirements. However, the availability of both N and P is probably considerably less than the calculated figures due to various factors:

- Volatilization of NH3 from surface spreading,
- Inhibition of nitrification by low soil pH, and
- Fixation of P by Al and Fe compounds.

Other elements—All of the elements analyzed were within the normal concentration ranges reported for sludges. Mg and Concentration ranges reported for sludges. Mg and Concentration ranges reported for sludges. Mg and Concentration the sludge was particularly beneficial to both soil and crops at the Macon site, since most soils in the southeastern U.S. are acid, sandy, and deficient in Mg. The trace elements B, Fe, Hg, Ni, and Se, too, were present at concentrations higher than the median values customarily reported in the

literature. Conversely, Ag, As, Cd, Cu, and Zn were either less than or comparable to normal median concentrations. Selenium, in particular, was of possible concern at 13.7  $\mu g/g$ , when compared to the median value of 2.7  $\mu g/g$ . The strongly acidic, sandy soil has never been limed. Consequently, the trace elements, in concentrations which would not normally cause problems if applied to properly managed soils, would be likely to cause plant phytoxicity or groundwater contamination.

#### Soil Analysis--

Total metals--Table 13 presents total and DTPA-extractable results for treated and control surface and subsurface soil samples. The data show that the treated surface soil was greatly enriched with Cd, Cu, Ni, Zn, and Pb, and the concentrations of Cd, Cu, Zn, and Pb in the treated surface soil were greater than soil concentrations normally reported. However, as noted under sludge characteristics, the sludge metal levels were either below or at comparable reported median values, which suggests that repeated applications of a sludge over a long time may result in a significant accumulation of metals in the surface soil.

The data also indicate downward migration of Cd, Cu, and especially Zn in the treated plot; Cd and Cu moved into the 20-to 46-cm depth layer, while Zn migrated down to 122 cm. This movement of the heavy metals was attributed to the high acidity and coarse texture of the soil and high precipitation in the area.

The metal concentrations at the 91- to 122-cm depth were considerably higher than the other subsurface layers, in both treated and control plots, due in part to the abrupt change from sand to clay at this depth.

Selenium values for surface and subsurface treated soil samples ranged from 0.26 to 1.2  $\mu g/g$ . Control samples were all reported as 0.016  $\mu g/g$ .

DTPA-extractable metals--DTPA-extractable metal concentrations (Cd, Cu, Ni, and Zn) evidenced trends similar to those observed for total metals. There was some indication of movement of the metals beyond the depth of incorporation (20 cm) and significant downward migration of Zn. The DTPA-extractable Cu and Zn levels were quite high, while the Cu level was below phytotoxic concentration. Based on the NCR 118 (1976) report, the Zn level was sufficiently elevated to be in the phytotoxic range for some plant species.

The percent of total metals extracted by DTPA was higher for the treated soils than for the control, and also higher for the surface soil than for subsurface layers. These observations

TABLE 13. CONCENTRATIONS OF TOTAL AND DTPA-EXTRACTABLE METALS IN COMPOSITE SURFACE AND SUBSURFACE SAMPLES FROM MACON, GEORGIA\*

Soil			Tota	†			DTPA-Extra	ctable	
Depth	Cd	Cu	Ni	Zn	Pb	Cd	Cu	Ni	Zn
- cm -			- µg/g				- <b>-</b> μg/g	9	
Treated:									
0-20	5.89	267	67.6	475	325	1.42(24)#	64.3 (24)	13.7 (20)	269 (56.6)
20-46	0.96	13.3	17.1	80.4	35.4	0.22(23)	4.10(31)	2.55(15)	37.80(47)
46-61	0.47	10.1	9.44	39.0	19.4	0.10(20)	1.63(16)	0.94(10)	16.40(42)
61-91	0.33	6.93	12.5	27.5	13.9	0.06(18)	1.20(17.3)	0.66(5.3)	9.59(35)
91-122	0.99	19.6	24.4	88.5	49.6	0.10(10)	2.40(12)	1.70(7)	27.60(31)
Control:									
0-20	0.76	11.9	19.1	27.7	35.0	0.08(10.5) <sup>#</sup>	1.23(10.4)	0.72(3.8)	1.14(4.1)
20-46	0.63	10.5	13.6	25.1	28.5	0.03(4.8)	0.74(7)	0.40(3)	0.87(3.5)
46-61	0.51	6.0	12.3	15.5	16.8	0.04(8.0)	0.62(10.3)	0.27(2.2)	0.59(3.8)
61-91	0.40	6.56	13.4	15.3	13.1	0.06(15)	0.72(11)	0.29(2.2)	1.03(6.8)
91-122	0.95	20.4	24.0	52.4	38.9	0.08(8)	3.31(1.6)	0.55(2.3)	1.02(2.0)

<sup>\*</sup>Oven-dry weight basis.

 $<sup>\</sup>mbox{tHNO}_3\mbox{-HClO}_4$  digestion.

<sup>#</sup>Percent of total concentration.

suggest (1) a relationship between organic matter content and solubility of metals in DTPA, and (2) that sludge-borne metals are more readily extracted by DTPA than metals occurring naturally in soils. The high percentages of Zn extracted by DTPA in the treated plot (31 to 57 percent) strongly suggest that the movement of Zn in the treated plot is real, and the Zn occurring at various depths apparently is sludge-borne.

# Plant Analyses--

Total metal (Cd, Cu, Ni, Zn, and Pb) concentrations in the treated plot cheatgrass and in the control plot oats are given in Table 14.

The Cu and Ni concentrations in the cheatgrass forage were relatively high, but not high enough to cause health problems to livestock (CAST, 1976). However, the concentration of Zn in the cheatgrass was sufficiently high so that most agronomic crops would be expected to suffer phytotoxic effects. Heavy metal uptake by any future crop at the Macon sludge-spreading site can be expected, because of the soil pH and texture.

The data exhibit excellent agreement between composite metal concentrations and the mean concentrations of the five sections constituting the composite. Selenium analysis on treated and control plant composites showed levels of < 0.016  $\mu\,g/g$ .

# Microbiology--

Stabilized sludge samples taken in April 1976 contained four species of intestinal parasite ova, one of human and three of animal origin. Salmonella sp. or Shigella sp. were not found in the raw or stabilized sludge. This is somewhat surprising because the poultry processing plant which discharges wastewater to the system should contain a high titre of Salmonella sp. organisms. (Poultry, and chicken in particular, are a known reservoir of salmonella-type organisms.) The fecal coliform (FC) and fecal streptococci (FS) groups of organisms in both the raw and digested sludge were represented in slightly anomalous ratios of concentration, as compared to the other eight sludges studied. No reason is apparent for this difference.

The soils in the treated and control plots had roughly the same content of FC and FS organisms. As with other test sites where these data correspond, homeostatic conditions in the soil are the probable explanation.

The soil sample from the treated plot, at a depth of 20 cm, contained Ascaris lumbricoides ova in low concentration. The viability of the ova could not be conclusively determined because of the low concentration and difficulty in embryonating

TABLE 14. METAL CONCENTRATIONS IN CHEATGRASS AND OATS FROM MACON, GEORGIA\*

Sample		Cheatgrass					Oats					
	Cd	Cu	Ni	Zn	Pb †	Cd	Cu	Ni	Zn	РЬ†		
		μg/g					μg/g					
Treated:												
Composite	0.77	18.5	16.4	375	4.86							
Section 1	0.84	30.0	17.0	455								
Section 2	0.77	19.8	16.7	404								
Section 3	0.58	24.1	15.1	392								
Section 4	0.73	17.2	21.6	325								
Section 5 Mean Std. Dev. Control:	0.62 0.71 0.11	18.8 22.0 5.16	16.6 17.4 2.46	341 383 52								
Composite						0.31	5.93	3.90	30.8	4.38		
Section 1						0.36	6.02	3.50	31.5			
Section 2						0.22	5.69	3.28	28.0			
Section 3						0.31	6.34	3.83	33.3			
Section 4						0.39	5.31	3.70	31.3			
Section 5 Mean Std. Dev.						0.29 0.31 0.07	4.70 5.61 0.64	3.83 3.63 0.24	31.9 31.2 1.95			

<sup>\*</sup>Oven-dry weight basis.

<sup>†</sup>Lead was analyzed on composite only.

the low numbers of immature eggs. Therefore, the possibility that the ova were viable exists.

The cheatgrass in the treated plot and oats from the control plot were both negative for FC, but did show the presence of FS in low numbers. The ratio of FC/FS concentrations on the plant tissue follow roughly the low FC/FS ratios exhibited by the raw and stabilized sludge. Since there is no indication in the history of the site of livestock grazing, that influence must be discounted.

Sludge, Soils, and Plants Conclusions--

- Although the sludge from the Macon STP is acceptable for agricultural utilization, and site soil properties, depth to groundwater, and high rainfall have created conditions that greatly enhance plant uptake and movement of heavy metals (e.g., Cu and Zn) to lower soil depths.
- Such factors as low soil pH, coarse soil texture, and high precipitation in the area all favor the downward movement of heavy metals in addition to other easily leached anions. The soil data indicate some movement of Cd, Cu, Ni, and Zn beyond the depth of incorporation. There is a good likelihood that Zn is already entering the groundwater, and the possibility of groundwater contamination from Zn or anions such as NO3, Cl, SO4, etc., is a reality.
- The sludge-spreading operation has resulted in adverse effects on crops, soil, and probably groundwater quality.
- Selenium has accumulated in the treated surface and subsurface soils but was not detected in the plant tissues. This was consistent with results reported by others (Cast, 1976).

# Las Virgenes, California

Sludge Characteristics--

Nitrogen and phosphorus--As shown on Table 12, concentrations of total N (4.84 percent), organic N (4.52 percent), NH<sub>4</sub>-N (0.32 percent), and NO<sub>3</sub>-N (0.41 ug/g), were within the ranges previously reported.

The spreading and drying of the sludge prior to application probably resulted in almost complete loss of the NH4-N. Based

on previous assumptions, 1 m ton of the sludge would supply the equivalent of 10.6~kg of available N, with a current value of \$4.66/m\$ ton.

Phosphorus concentration (1.71 percent) was slightly below the reported median of 2.3 percent. If all of the P in the sludge is considered equivalent to commercial sources, 1 m ton of sludge would supply the equivalent of 17.1 kg P and have a value of \$17.10/m ton.

In terms of N and P, the sludge would have a fertilizer value of \$21.76/m ton. The annual estimated application rate of Las Virgenes sludge was approximately 50 m tons/ha, equivalent to 530 kg/ha of available N and 855 kg/ha of total P, in excess of plant requirements. The nitrogen equivalent was possibly less than stated, depending on NH3 losses from the spreading operation.

Other elements—Concentrations of all of the elements were within the normal ranges reported for sludges, Cd slightly less, B, Co, and Cu slightly greater, and Hg (49  $\mu g/g$ ) and Se (7  $\mu g/g$ ) considerably greater than median concentrations reported.

Effluent characteristics--Irrigation water at the study site was supplied through reclaimed secondary effluent from the STP. The quantities of metals N and P applied to the test plot at Las Virgenes were consequently influenced by the amount of secondary effluent irrigation water used. Records of the quantities of effluent water applied were not kept; an estimate based upon normal irrigation practices for the region (1.2 m irrigation water per year) is presented in Table 15.

TABLE 15. NITROGEN, PHOSPHORUS, AND METALS APPLIED IN SEWAGE TREATMENT PLANT EFFLUENT AND SLUDGE - LAS VIRGENES, CALIFORNIA

Element	Total Amount Applied in Effluent* kg/ha	Total Amount Applied in Sludge† kg/ha	Total Applied kg/ha	
N	1,010	6,920	7,930	
Р	1,800	2,620	4,410	
Cd	0.42	1.69	2.1	
Cu	3.0	145	148	
Ni	2.2	7.6	9.8	
РЬ	3.0	6.1	9.1	
Zn	6.6	207	214	

<sup>\*</sup>Assuming effluent has been applied at the rate of 1.2 m/yr for five years and the composition of the effluent is as given in Table 16, Volume II.

<sup>&</sup>lt;sup>†</sup>Assuming 153 total m tons/ha applied and the composition of the sludge is as given in Table 12.

The analyses showed that the use of effluent water had made a substantial contribution to N, P, Cd, Ni, and Pb loadings. In terms of the total mass loading estimate (from both sludge and effluent), the effluent had contributed about 15 percent of the total N applied, 41 percent of the total P, 20 percent of the total Cd, 29 percent of the total Ni, and 33 percent of the total Pb. The amounts of Cu and Zn applied in the form of effluent were very small in comparison to amounts applied in the form of sludge.

# Soil Analysis--

Total metals--Table 16 presents total and DTPA-extractable metal analyses of treated and control surface and subsurface soils. Except for Cd, the total soil metal concentrations for both treated and control plots were in the range considered normal. However, Cu and Zn concentrations in the surface 30 cm of treated soil were greater than those of the control plot, a result of these metals being added in the form of sludge.

Total metal concentration profiles for both the treated and control soil columns, except for Cu and Zn in the treated surface soils, were fairly consistent and almost identical. The relatively constant metal concentrations throughout the soil column confirmed that the sludge applied metals had not migrated in measurable amounts below the depth of incorporation.

Total treated and control plot soil Cd concentrations were approximately the same at all depths and unusually high (3 to 4  $\mu g/g$ ). However, Burau, et al., 1973, reported that soil series in California similar to those sampled at Las Virgenes have Cd concentrations as great, or even greater. It is reasonable, therefore, to conclude that the high Cd levels observed in the soil are of natural origin and not derived from sludge disposal or recycling operations.

DTPA-extractable metals--The DTPA data tended to confirm the interpretation of the data presented for total analyses. DTPA data showed accumulations of both Cu and Zn in the surface soil of the treated plot and further indicated that the metals applied in the form of sludge had remained in the surface layer and not migrated to lower depths.

The percent of total metals extracted by DTPA varied with soil depth. Again, the sludge-borne metals in the surface soil were more readily extracted than were the metals occurring naturally in the surface soil of the control plot. More Cd and Cu than Ni or Zn were extracted by DTPA. Analyses of composite samples of the surface soil correspond closely to the mean of each of the samples which made up the composite.

TABLE 16. CONCENTRATIONS OF TOTAL AND DTPA-EXTRACTABLE METALS IN COMPOSITE SURFACE AND SUBSURFACE SAMPLES FROM LAS VIRGENES, CALIFORNIA\*

Soil Depth			Tota	1†		DTPA-Extractable					
	Cd	Cu	Ni	Zn	Pb	Cd	Cu	Ni	Zn		
- cm -		_	- μg/g			μg/g					
Treated:											
0-30	3.58	45.4	52.9	120	21.7	1.48(41) <sup>#</sup>	5.41(12)	2.51(4.8)	8.12(6.8)		
31-61	3.79	29.1	50.3	93.4	19.3	1.21(32)	2.06(7.1)	2.30(4.6)	0.42(0.5)		
61-91	3.41	28.5	49.5	93.8	19.0	1.23(36)	2.47(8.7)	2.99(6.0)	0.31(0.3)		
91-122	3.53	28.5	50.2	90.7	18.7	1.35(38)	2.89(10)	3.07(6.1)	0.80(0.4)		
Control:											
0-30	3.77	27.7	48.0	98.1	19.9	1.37(36)#	1.69(6.1)	1.45(3.0)	1.01(1.0)		
30-61	3.75	27.6	50.5	94.2	19.7	1.37(37)	2.31(8.4)	2.37(4.7)	0.89(0.9)		
61-91	3.22	28.0	50.2	90.2	19.9	1.53(48)	2.50(8.9)	3.22(6.4)	0.85(0.9)		
91-122	4.11	28.1	52.8	90.9	20.6	1.65(40)	2.37(8.4)	3.15(6.0)	0.60(0.7)		

<sup>\*</sup> Oven-dry weight basis.

 $<sup>+</sup> HNO_3 - HC1O_4$  digestion.

<sup>#</sup> Percent of total concentration.

# Plant Analyses--

As is apparent from Table 17, which presents total metal analysis of the ryegrass from the treated and control plots, the concentrations of Cu, Ni, and Zn were in the normal range for this type of forage (Chapman, 1966). Concentrations of Cd were slightly greater, and Pb considerably greater, than those usually observed. The slightly elevated concentrations of Cd were probably a result of the naturally high concentration of Cd in the soil. Since the treated plot contained less Cd than the control plot, it appears that application of sludge has not been responsible for the higher Cd levels in the rye grass.

Concentrations of Cu and Zn in rye grass for the control plot were not significantly different from those of the treated plot. Nickel in the treated plot was low for the foliage, but greater than its concentration in the control plot.

#### Microbiology--

No intestinal parasite ova were found in any of the sludge samples. However, it should not be inferred from this finding, or other sites with negative results, that no parasite ova are associated with waste sludge, since the absence of intestinal parasite ova in sludge as reported by Aiba, Sudo, and Liebman (Aiba, et al. 1965, Liebman 1965) is atypical. The high economic levels and health standards of inhabitants of the area tributary to the Tapia Water Reclamation Plant may suggest a low or negligible incidence of helminth infection. The absence of salmonella or shigella species was not unexpected in view of the small sample base and the relatively low numbers of these organisms usually found in sewage solids (Kenner, 1972). However, the absence of fecal coliforms in the August 4, 1976, raw sludge sample was not readily explainable; the FC population in the stabilized sludge for the same date was in excess of seven logs greater. One possible explanation could be the aerobic digestion process of the Tapia plant.

The soils and associated microflora in the treated and control plot showed some evidence of being homeostatic. The positive detection of fecal organisms in the treated plot may have resulted from the incidental pasturing of livestock (beef cattle) in the field, subsequent to rye grass harvest. Further support to this thesis was provided by the preponderance of FS organisms in the soil (Mara, 1977).

Both the treated and control plots of rye grass also were positive for FS. The control plot plant specimens, too, appeared to be influenced by livestock grazing.

TABLE 17. METAL CONCENTRATIONS IN RYEGRASS FROM LAS VIRGENES, CALIFORNIA\*

		Grains											
Sample	Cd	Cu	Ni	Zn	Pb †	Cd	Cu	Ni	Zn	Pb			
		µg/g						μg/g					
Treated:													
Composite	0.72	12.5	7.80	60.0	14.6								
Section 1	0.44	13.9	9.30	60.4									
Section 2	0.52	9.53	10.50	51.0									
Section 3	0.89	13.3	8.48	77.0									
Section 4	0.78	10.4	7.38	58.6									
Section 5 Mean Std. Dev. Control:	0.72 0.67 0.19	9.68 11.4 2.08	9.37 9.01 1.16	45.5 58.5 12.0									
Composite	1.56	11.5	4.25	70.0	8.40								
Section 1	1.79	11.1	5.43	71.3									
Section 2	1.11	10.3	5.69	57.7									
Section 3	1.34	10.2	4.13	54.2									
Section 4													
Section 5 Mean Std. Dev.	1.41 0.35	10.5 0.49	5.08 0.84	61. <b>1</b> 9.03									

<sup>\*</sup>Oven-dry weight basis.

 ${\sf tLead}$  was analyzed on composite only.

Sludge, Soils, and Plants Conclusions --

- The use of sludge as a soil amendment has had little or no impact on uptake of those trace metal elements tested in plants.
- Total and DTPA-extractable metal analyses suggest that metals have not moved beyond the depth of incorporation.
- Large amounts of nitrogen, considerably in excess of those needed by the crop, have been applied via irrigation waters. It is possible that groundwater NO<sub>3</sub>-N contamination may occur, if these large N applications continue.
- The possibility of heavy metals contamination of groundwater as a result of sludge spreading at the site is remote.
- The Cd concentrations in the soil profile (treated and control plots) were unusually high (approximately 4  $\mu g/g$ ). The source of Cd was not the result of sludge spreading, but rather was of natural origin.

## Wilmington, Ohio

Sludge Characteristics--

Nitrogen and phosphorus--The concentrations total N (3.81 percent), organic-N (2.8 percent), NH<sub>4</sub>-N (1.01 percent), and NO<sub>3</sub>-N (13.7  $\mu g/g$ ) were within the ranges previously reported for sludges.

The liquid sludge was spread on the surface at the site and allowed to dry, resulting in a substantial loss, by volatilization, of NH4-N. Based on previous assumptions, this sludge was capable of supplying 10.65 kg of available N/m ton. At current fertilizer prices, the equivalent value of this N fertilizer sludge is 4.68/m ton.

The P concentration of the Wilmington sludge (3.55 percent) fell into the range commonly reported, but was substantially greater than the median concentration (2.3 percent). If all the phosphorus was considered plant available or equal to commercial fertilizer sources, the sludge would be an excellent source of fertilizer phosphorus; each m ton of sludge would supply the equivalent of 35.5 kg P which, at current fertilizer prices, has a value of \$35.50/m ton. In terms of N and P fertilizer equivalent, the value of the Wilmington sludge was \$40.18 per m ton.

The annual estimated application rate was approximately 6.8 m tons/ha, equivalent to 73 kg/ha of available N and 241 kg/ha of total P, in excess of plant requirements. However, the nitrogen equivalent was possibly less than stated, depending on NH $_3$  losses from the spreading operation.

Other elements--Concentrations of all metals, except Mo and Zn, were in the normal range and near median concentrations observed for other sludges. However, Mo and Zn concentrations were higher, Zn in particular (45,800  $\mu g/g$ ) compared to the highest value reported by Sommers (27,800  $\mu g/g$ ).

Molybdenum is an essential element for animals at low concentrations, but is toxic at higher concentrations; concentrations of Mo as low as lo  $\mu g/g$  in forage (Cast, 1976) can cause a disorder in animals called molybdenosis. The soil pH of the treated plot was neutral, which makes Mo more plant available; the alfalfa could absorb quantities of Mo sufficient to be toxic to animals.

Soil Analyses--

Total metals--Table 18 presents total and DTPA-extractable metals of treated and control surface and subsurface soil samples.

The range in Cd concentration throughout the soil profile of both the treated and control plots (0.77 and 1.0  $\mu g$  Cd/g) was greater than that commonly reported for agricultural soils (0.5  $\mu g/g$ ). Cadmium was statistically significantly higher in the control plot subsurface soils, suggesting that these high levels were due to natural sources.

Zinc, Cu, and Pb showed substantial enrichment in the treated surface soil. This can be attributed to the high concentrations of both metals in the sludge.

The concentrations of the five heavy metals in both the treated and control plot subsurface soils were nearly the same, indicating that the metals applied with the sludge were essentially retained in the surface 18 cm.

Molybdenum values of the treated surface and subsurface soil samples ranged from 2.0 to 3.25  $\mu g/g$ . Similarly, control samples showed a range of 1.97 to 3.10  $\mu g/g$ , indicating little or no appreciable difference between the plots.

DTPA-extractable metals--The DTPA-extractable metal results corresponded to the trends already discussed for total metals. The only observable difference between the treated and control plots was the amount of DTPA-extractable Zn, which had not only accumulated in the surface layer of the sludged soil, but was

TABLE 18. CONCENTRATIONS OF TOTAL AND DTPA-EXTRACTABLE METALS IN COMPOSITE SURFACE AND SUBSURFACE SAMPLES FROM WILMINGTON, OHIO\*

Soil			Total	†			DTPA-Extra	ctable	
Depth	Cd	Cu	Ni	Zn	Pb	Cd	Cu	Ni	Zn
- cm -		-	- µg/g				- <b>-</b> μg/g		
Treated:									
0-18	0.86	16.0	24.7	190	47.8	0.14(16.3)#	1.86(11.6)	0.70(2.8)	27.5 (14.5)
18-30	0.89	19.0	32.3	62.5	23.5	0.11(12.4)	0.94(5.0)	0.85(2.6)	1.77(2.8)
30-61	0.77	22.2	39.0	65.1	22.8	0.09(11.7)	0.74(3.3)	0.68(1.7)	1.08(1.7)
61-91	1.00	22.8	44.3	66.0	20.0	0.16(16)	0.66(2.9)	0.99(2.2)	0.81(1.2)
91-122	1.10	23.0	41.8	67.8	21.4	0.12(10.9)	0.71(3.1)	0.98(2.3)	0.83(1.2)
Control:									
0-18	0.87	14.6	24.9	49.5	24.6	0.13(14.9)#	1.38(9.5)	0.87(3.5)	1.20(2.4)
18-30	0.91	17.6	31.4	57.3	22.1	0.12(13.2)	0.98(5.6)	0.47(1.5)	0.63(1.1)
30-61	1.08	21.5	42.3	75.0	22.7	0.11(10.2)	0.79(3.7)	0.64(1.5)	0.60(0.8)
61-91	1.08	23.4	46.2	74.2	21.9	0.08(7.4)	0.69(3.0)	0.38(0.8)	0.57(0.8)
91-122	1.06	24.8	44.5	71.3	20.1	0.09(8.5)	0.79(3.2)	0.38(0.9)	0.62(0.9)

<sup>\*</sup>Oven-dry weight basis.

 $<sup>+</sup> HNO_3 - HC1O_4$  digestion.

<sup>#</sup>Percent of total concentration.

also greater than the control plot in each of the lower depths of the treated soil. The DTPA-extractable Zn data suggest the possible movement of sludge-borne Zn to depths as low as 61 cm.

### Plant Analyses--

Total metals in alfalfa from the treated and control plots are presented in Table 19. Concentrations of Cu, Ni, and Zn were in the range commonly reported for alfalfa, although Cd and Pb concentrations were slightly higher (Chapman, 1966). Except for Zn, mean concentrations of metals in treated plot plant materials were not statistically different from those of the control plot. Therefore, it would seem that sludge spreading has not resulted in increased concentrations of Cd, Ni, and Pb in the alfalfa plants.

Moreover, considering the concentration of Zn in the treated surface soil (190  $\mu g/g$ ), the enrichment of Zn in alfalfa was lower than would be expected. In contrast, the concentration of Zn in treated surface soil at Kendallville, Indiana, was less (113  $\mu g/g$ ), while the alfalfa had accumulated about twice as much Zn (83  $\mu g/g$ ) as that grown on the Wilmington treated plot (40.5  $\mu g/g$ ). Since the pH of the soil at both sites was approximately the same, these differences do not seem to be explainable in terms of the soil acidity. Since plant Zn concentrations from the control plots from the two sites were essentially the same, the differences in the treated plots apparently were not due to a plant-age factor. Other factors, e.g., long-term Zn sludge concentrations, fertility level, metal competition, etc., were probably involved.

The Mo concentration of composite treated plants was 1.99  $\mu g/g$  compared to a control of 1.34  $\mu g/g$ . Both of these values are considerably lower than the 10  $\mu g/g$  level which researchers have indicated to be the threshold animal toxicity level (Cast, 1976).

# Microbiology--

No intestinal parasite ova were detected in the Wilmington sludge samples, an atypical result based upon published data and larger sample bases. The absence of Salmonella sp. or Shigella sp. was not an unexpected result in view of the small sample base and the relatively low numbers of these organisms usually found in sewage solids (Kenner, 1972). The low number of fecal coliform organisms detected for the August 25, 1976, raw sludge sample was unexplainable; the stabilized sludge for the same sampling date was some four logs greater.

The treated and control soils contained like profiles of fecal coliform and fecal streptococci organisms.

TABLE 19. METAL CONCENTRATIONS IN ALFALFA FROM WILMINGTON, OHIO\*

			Leaves					Grains		
Sample	Cd	Cu	Ni	Zn	Pb †	Cd	Cu	Ni	Zn	Pb
		-	- μg/g -	-			-	- μg/g -	-	
Treated:										
Composite	0.90	8.91	6.43	37.8	6.30					
Section 1	1.02	9.10	7.62	43.3						
Section 2	0.83	8.51	7.24	42.4						
Section 3	0.84	7.62	5.90	37.6						
Section 4	0.93	8.41	8.35	38.1						
Section 5 Mean Std. Dev. Control:	0.79 0.88 0.09	7.77 8.28 0.60	7.76 7.37 0.92	41.3 40.5 2.56						
Composite	1.00	9.40	7.18	37.0	7.79					
Section 1	0.75	9.90	7.73	33.5						
Section 2	1.10	9.95	7.87	34.1						
Section 3	0.56	9.70	9.04	34.3						
Section 4	0.84	8.11	7.96	27.3						
Section 5 Mean Std. Dev.	1.01 0.85 0.21	8.46 9.22 0.87	8.75 8.27 0.59	32.8 32.4 2.91						

<sup>\*</sup>Oven-dry weight basis.

<sup>†</sup>Lead was analyzed on composite only.

The alfalfa plant specimens selected from the mixed perennial grasses were associated with fairly high counts of fecally associated organisms, in comparison to other sites with other crops. The data are very similar to the Kendallville, Indiana site, where sludge was applied directly to the low-lying alfalfa.

Sludge, Soil, and Plant Conclusions--

- The Wilmington sludge was an excellent source of nitrogen and phosphorus. It had a potential fertilizer value of \$40.19/m ton, the highest among the nine sites studied.
- The limiting factor in agricultural utilization of this sludge was probably the exceedingly high Zn content.
- The treated plot soil had been substantially enriched with Zn, but this had not yet resulted in significant increases in the alfalfa Zn levels.
- DTPA Zn indicated possible Zn movement to a depth of at least 61 cm.
- The results obtained indicated that, except possibly for Zn, the metals applied in the form of sludge had been almost completely retained by the soil within the depth of incorporation. Therefore, groundwater contamination from Cd, Cu, Ni, and Pb would not be expected to have occurred.
- Soil and plant molybdenum concentrations were low, indicating little or no accumulation as a result of sludge spreading.

# Springfield, Missouri

Sludge Characteristics--

Nitrogen and phosphorus--Concentrations of NO<sub>3</sub>-N (51.4  $\mu$ g/g) and organic-N (4.3 percent) were in the range reported by Sommers (Table 12). However, the total N (8.48 percent) and NH<sub>4</sub>-N (4.18 percent) concentrations in the Springfield sludge were substantially higher than the median concentrations of 4.8 and 0.09 percent, respectively, suggesting that this sludge can be an excellent nutrient when applied to agricultural land.

Spreading of liquid sludges onto the surface without incorporation into the soil, as currently practiced at Springfield, results in substantial loss of NH3 to the atmosphere. Based on previously stated assumptions, the sludge would supply 29.5 kg of available N/m ton and would have a current equivalent value of \$12.98/m ton.

The P content of the sludge (1.74 percent) was within the range reported by Sommers, but less than the median P concentration (2.3 percent). One m ton of the Springfield sludge would supply 17.4 kg of available P and have a current equivalent nutrient value of \$17.40. The value of the sludge based on the N and P content is high - \$30.38/m ton.

The K content of the sludge (0.54 percent) was considerably higher than the surveyed median concentration (0.3 percent). Based upon an annual application of 15.8 m tons/ha, this sludge would have supplied 466, 275, and 85 kg of available N, P, and K, respectively, per ha, for the fescue plants.

Other elements——In the Springfield sludge, concentrations of all metals except Mo were within the reported concentration ranges (Table 20). However, Ca, Cr, Cd, Ni, and Zn concentrations were considerably higher than the median reported concentrations. The sludge was alkaline (pH 8.3), which suggests that the solubility of the heavy metals was probably low and that the metals were less available for downward movement and plant uptake.

Total metals--Table 20 presents total and DTPA-extractable metal analyses of the surface and subsurface treated and control soils. Statistically significant Cd, Zn, Ni, and Pb surface soil enrichment as a result of repeated sludge applications was indicated. Concentrations of total metals throughout the soil profile were generally in the range reported for agricultural soils, indicating virtually no movement of metals beyond the surface 20 cm.

The Mo concentrations of the treated surface and subsurface soil samples ranged from 1.9 to 2.93  $\mu g/g$ . Similarly, control samples showed a range of 2.0 to 3.26  $\mu g/g$ , indicating no appreciable difference between the plots.

<u>DTPA-extractable metals--</u>There appeared to be no correlation between concentrations of DTPA-extractable and total metals. The data indicated accumulations of Cd, Zn, and probably Ni in the surface soil as a result of the sludge application.

The amounts of metals extracted by DTPA were higher on the surface compared to the subsurface soil and were particularly noticeable in the treated plot. The data suggest that sludge-borne heavy metals are, in large part, present as metal-organic complexes and more soluble in DTPA than are the metals indigenous to the soil.

TABLE 20. CONCENTRATIONS OF TOTAL AND DTPA-EXTRACTABLE METALS IN COMPOSITE SURFACE AND SUBSURFACE SAMPLES FROM SPRINGFIELD, MISSOURI\*

Soil			Tota	1+			DTPA-Extra	ctable	
Depth	Cd	Cu	Ni	Zn	Pb	Cd	Cu	Ni	Zn
- cm -		_	- μg/g				µg/g		
Treated:									
0-20	1.17	10.5	28.9	108	40.2	0.38(32.5)#	7.07(67.3)	3.18(11)	36.6 (34)
20-41	0.77	13.1	28.2	41.8	21.4	0.11(14.3)	1.80(13.8)	1.62(5.7)	3.03(7.3)
41-61	0.82	15.7	47.6	48.8	25.8	0.07(9.0)	1.47(9.4)	1.56(3.3)	2.00(4.1)
61-81	0.83	20.0	38.7	52.9	28.0	0.07(8.4)	1.87(9.4)	1.57(4.1)	2.30(4.4)
81-122	0.77	16.1	35.8	57.6	25.1	0.06(7.8)	1.12(7.0)	1.10(3.1)	1.00(1.7)
Control:									
0-20	0.73	23.6	19.9	36.8	34.0	0.11(15.1)	7.35(31.1)	1.49(7.5)	1.79(4.9)
20-41	0.68	14.3	22.7	34.8	24.1	0.09(13.2)	2.87(20.1)	1.11(4.4)	2.78(8.0)
41-61	0.70	12.0	24.4	39.2	22.1	0.07(10)	1.32(11)	0.95(3.9)	1.42(3.6)
61-81	0.82	13.2	28.8	45.7	20.6	0.06(7.3)	0.98(7.5)	0.96(3.3)	0.68(1.5)
81-122	0.89	15.0	34.4	48.0	26.1	0.06(6.7)	1.36(9.1)	0.97(2.8)	1.13(2.4)

<sup>\*</sup>Oven-dry weight basis.

 $<sup>\</sup>text{†HNO}_3\text{-HClO}_4$  digestion.

<sup>#</sup>Percent of total concentration.

## Plant Analysis --

Table 21 presents total metal analysis of fescue grass from the treated and control plots and shows concentrations of Ni and Zn significantly higher in the forage from the treated plot. The opposite was observed for Cu and Pb. The Cd concentration in section 2 of the control was inexplicable and affected the mean concentration of Cd in the control forage.

Generally, concentrations of Cd, Cu, Ni, Zn, and Pb in the forage were in the normal range for fescue (Chapman, 1966). Plant analysis indicated no apparent problems stemming from utilization of the forage as animal feed (CAST, 1976).

The Mo concentrations of composite treatment plants were 0.84  $\mu g/g$  compared to a control of 0.67  $\mu g/g$ . Both of these values were considerably lower than the 10  $\mu g/g$  level which researchers have indicated to be the threshold animal toxicity level (Cast, 1976).

## Microbiology--

The raw sludge contained ova of one animal parasite, the stabilized sludge, the ova of one human intestinal and one canine parasite. The anaerobic digestion process environment was not sufficiently adverse to inactivate these organisms.

Salmonella sp. was detected in one sample of raw sludge in very low numbers, while FC and FS organisms were found in numbers corresponding generally to those reported in the literature.

The treated soils indicated an almost complete absence of sludge-associated organisms; this was somewhat surprising because the grass cover crop was used as fodder for beef cattle. Moreover, the control plot exhibited a higher FS count than the treated plot, although the absolute numbers were very low. The soil may have supported only a limited microflora; the total aerobic bacteria count was some 3 logs below other values for the other study sites. The reason for this was not readily apparent.

Since the fescue hay from the treated plot showed a high FS count, it seems probable that the grazing livestock may have had some effect. It may be assumed with some confidence that with the absence of fecal organisms in the treated soil, the FS on the plant tissue was of animal origin, the leaf surface of a plant having higher rates of microbial inactivation (i.e., dessication, ultraviolet radiation) than soil.

Sludge, Soil and Plants Conclusions--

 The sludge from the Springfield STP did not contain excessive concentrations of heavy metals and appears

TABLE 21. METAL CONCENTRATIONS IN FESCUE FROM SPRINGFIELD, MISSOURI\*

			Leaves					Grains		
Sample	Cd	Cu	Ni	Zn	Pb+	Cd	Cu	Ni	Zn	Pb
		-	- μg/g -	-			-	- µg/g -	-	
Treated:										
Composite	0.25	4.79	4.27	26.0	2.71					
Section 1	0.36	4.22	4.15	28.4						
Section 2	0.41	4.68	4.71	32.8						
Section 3	0.32	4.76	4.21	32.8						
Section 4	0.33	4.50	3.76	28.7						
Section 5 Mean Std. Dev. Control:	0.29 0.34 0.05	4.97 4.63 0.28	5.05 4.38 0.51	33.7 31.3 2.52						
Composite	0.29	5.79	3.96	23.4	5.60					
Section 1	0.27	5.12	3.76	23.8						
Section 2	1.03	5.12	3.59	24.1						
Section 3	0.36	5.22	3.59	26.0						
Section 4	0.24	5.20	3.25	24.5						
Section 5 Mean Std. Dev.	0.25 0.43 0.34	5.61 5.25 0.20	3.37 3.51 0.20	24.9 24.6 0.86						

<sup>\*</sup>Oven-dry weight basis.

tLead was analyzed on composite only.

to be an excellent source of plant nutrients for the fescue grown at the sludge-treated site.

- There was some indication of Cd, Ni, Zn, and Pb enrichment of the surface soil as a result of the sludge spreading. However, these soil concentrations were still within the range normally reported.
- The metals Cd, Cu, Zn, Ni, and Pb applied in the sludge over the past 18 years have remained essentially in the surface 20 cm of soil.
- Based on the metal contents of the fescue, the forage is safe for feeding.
- Soil and plant Mo concentrations were low, indicating no accumulation as a result of sludge spreading.

## Chippewa Falls, Wisconsin

Sludge Characteristics --

Nitrogen and phosphorus--As shown in Table 121 concentrations of total N (4.58 percent), organic N (2.46 percent), NH<sub>4</sub>-N (2.12 percent) and NO<sub>3</sub>-N (13.0  $\mu$ g/g were within the ranges previously reported.

The technique of spreading liquid sludge from the Chippewa Falls STP onto land and allowing it to dry, prior to incorporation, probably resulted in substantial loss of NH<sub>4</sub>-N. One m ton of the sludge would supply the equivalent of 15.5 kg of available N and have a value of \$6.82.

The P concentration of the Chippewa Falls sludge (1.34 percent) fell within the range reported for comparative sludge, but was considerably less than the reported median concentration (2.3 percent). One m ton of the Chippewa Falls sludge would supply 13.4 kg of P and have a value of \$13.40. Thus, in terms of N and P, 1 m ton of this sludge would be worth \$20.22.

The annual estimated application rate of the Chippewa Falls sludge was approximately 16 m tons/ha, equivalent to 248 kg/ha of available N and 214 kg/ha of total P, in excess of plant requirements. The nitrogen equivalent was possibly less than stated, depending on NH $_3$  losses from the spreading operation.

Other elements—Concentrations of all of the elements were within normal ranges. Only Hg, at 24.1  $\mu g/g$ , and Se, at 7.6  $\mu g/g$ , were significantly higher than the median values reported by Sommers. Since Hg in soil is generally present in an

insoluble form, thus unavailable for plant uptake, there is probably little reason for concern.

## Soil Analyses --

Total metals—As is apparent from Table 22, which presents total and DTPA—extractable metals for treated and control surface and subsurface plots, the concentrations of total metals Cd, Cu, Ni, Zn, and Pb in soils from both the treated and control plots were in the normal range, although Cu and Zn concentrations in the surface 20 cm of the treated soil were greater than the control.

The soil at this site was sandy and strongly acidic, so some metal migration would be expected. However, because of the low metal content in the sludge and relatively low amount spread (80 m tons/ha) at this site to date, no migration of metals beyond the depth of incorporation had occurred.

DTPA-extractable metals--Data on DTPA-extractable metals showed Cu, Ni, and Zn enrichment of the surface soil as a result of the sludge application. The percent of total metals extracted by DTPA from the surface soil of the treated plot was higher than from the control, indicating that sludge-borne metals are more soluble in DTPA than are native metals. The results indicate little or no migration of Cd, Cu, Ni, and Zn to lower soil depths.

# Plant Analyses--

Concentrations of Cd, Cu, Ni. Zn. and Pb in the leaves and grains of sovbeans grown on the treated and control plots are presented in Table 23.

Except for Ni concentration in leaves, mean concentrations of the metals analyzed for both leaves and grains were significantly higher in plants grown in the treated plot than those in the control. The increases of metal concentrations in the grains and leaf tissue appeared to be parallel, although the soybean grains contained less Cd, Ni, and Pb than the leaves. In the treated plot, Cu concentration appeared to be higher in the grain than in the leaves, while Zn concentrations showed no difference.

Although sludge application at Chippewa Falls resulted in significant increases in metal concentrations in soybean grains and leaves, the concentrations were within the range normally reported for soybeans. The concentration of Cd, the element of most concern in health problems, was very low in the soybean seeds; the soybeans harvested at this site may be considered safe for consumption by animals or man.

TABLE 22. CONCENTRATIONS OF TOTAL AND DTPA-EXTRACTABLE METALS IN COMPOSITE SURFACE AND SUBSURFACE SAMPLES FROM CHIPPEWA FALLS, WISCONSIN\*

Soil			Total	†			DTPA-Extra	ctable	
Depth	Cd	Cu	Ni	Zn	Pb	Cd	Си	Ni	Zn
- cm -		_	- μg/g				µg/g		
Treated:									
0-20	0.42	17.4	14.2	49.3	9.90	0.06(14.3)#	3.44(19.8)	1.00(7.0)	4.28(8.7)
20-46	0.38	10.5	16.7	29.4	4.40	0.03(7.9)	0.55(5.2)	0.36(2.2)	0.61(2.1)
46-61	0.35	11.3	16.1	21.8	4.00	0.02(5.7)	0.57(5.0)	0.39(2.4)	1.00(4.6)
61-91	0.31	13.0	14.6	22.1	2.40	0.02(6.5)	0.34(2.6)	0.41(2.8)	0.89(4.0)
91-122	0.44	18.5	14.4	24.7	2.70	0.03(6.8)	0.31(1.7)	0.68(4.7)	1.17(4.7)
Control:									
0-20	0.46	7.6	15.2	31.3	8.50	0.06(13)#	0.42(5.5)	0.33(2.2)	1.54(4.9)
20-46	0.34	7.6	14.3	24.2	5.70	0.03(8.8)	0.23(3.0)	0.33(2.3)	0.39(1.6)
46-61	0.48	12.2	18.3	30.2	5.60	0.02(4.2)	0.17(1.4)	0.41(2.2)	0.79(2.6)
61-91	0.31	15.7	19.6	27.8	4.00	0.03(9.7)	0.36(2.3)	0.33(1.7)	1.12(4.0)
91-122	0.33	13.8	16.3	19.5	2.10	0.02(6.1)	0.23(1.7)	0.21(1.3)	0.89(4.6)

<sup>\*</sup>Oven-dry weight basis.

 $7HNO_3-HC1O_4$  digestion.

<sup>#</sup>Percent of total concentration.

TABLE 23. METAL CONCENTRATIONS IN SOYBEANS FROM CHIPPEWA FALLS, WISCONSIN\*

			Leaves					Grains		
Sample	Cd	Cu	Ni	Zn	Pb †	Cd	Cu	Ni	Zn	Pb†
			- μg/g -				-	- μg/g ·		
Treated:										
Composite	0.97	11.9	10.8	87.5	5.50	0.12	18.5	4.01	93.1	0.74
Section 1	2.31	14.9	14.5	149		0.12	18.3	3.09	86.2	
Section 2	0.95	15.1	10.4	113		0.09	19.9	5.18	106	
Section 3	0.92	12.5	10.7	80.5		0.15	19.8	4.41	99.8	
Section 4	1.48	11.5	17.8	111		0.19	18.9	3.57	92.6	
Section 5 Mean Std. Dev. Control:	1.24 1.38 0.57	12.6 13.3 1.59	13.3 13.3 3.04	89.9 109 26.4		0.11 0.13 0.04	19.0 19.2 0.67	4.06 4.06 0.80	99.5 96.8 7.60	
Composite	0.82	10.4	10.4	60.4	8.20	0.07	11.4	1.18	74.3	0.60
Section 1	1.21	11.1	15.5	65.2		0.03	11.7	1.20	71.3	
Section 2	0.66	10.0	8.7	51.6		0.06	10.8	2.25	72.6	
Section 3	0.72	11.0	9.1	56.4		0.06	11.5	1.03	69.2	
Section 4	0.80	9.6	9.6	52.5		0.12	10.7	1.88	67.6	
Section 5 Mean Std. Dev.	0.77 0.83 0.22	10.6 10.5 0.65	9.9 10.6 2.80	60.8 57.3 5.72		0.09 0.07 0.03	11.0 11.1 0.44	2.74 1.82 0.71	67.1 69.6 2.36	

<sup>\*</sup>Oven-dry weight basis.

tLead was analyzed on composite only.

### Microbiology--

No intestinal parasite ova, salmonella, or shigella organisms were detected in the sludge samples. FC and FS organisms were found in numbers generally corresponding to those reported in the literature.

The soils had not been treated with sludge for some three months prior to the sampling date, August 3, 1976. Both treated and control plots exhibited similar microbiological character indicating homeostasis and the achievement of some equilibrium condition.

The soybean tissue contained about the same concentration of FC and FS organisms as did vegetation of similar size and associated cultural practices. The organisms can most likely be traced to aeolian transport in association with dust particles, the result of tillage or other man-induced or natural activity in the field.

Sludge, Soils, and Plants Conclusions --

- Copper, Ni, and Zn enrichment of the surface-treated soil was observed to be the direct result of repeated sludge applications.
- The data suggest no migration of any metals through the soil profile.
- Cd was statistically significantly (treated vs. control) higher in the petioles; the difference was  $0.55 \mu g/g$ .
- The possibility of groundwater contamination is remote, in view of the restricted heavy metal movement and the low total sludge loadings.

### Hopkinsville, Kentucky

Sludge Characteristics --

Nitrogen and phosphorus--The concentrations of NO<sub>3</sub>-N (10.2  $\mu$ g/g), organic-N (1.83 percent), total N (2.37 percent), and NH4-N (0.54 percent) in the Hopkinsville sludge were in the range reported for sludges.

The practice of spreading liquid sludges on the surface at Hopkinsville and allowing them to dry prior to incorporating into the soil, probably resulted in significant loss of NH3, greatly reducing the nutrient value of the sludge. Based upon

previous assumptions, each m ton of sludge would supply 6.30 kg of available N and have a value of \$2.80.

The concentration of phosphorus in the sludge (1.56 percent) was in the range, but slightly below the median concentration reported (2.3 percent). If all of the P in the sludge were considered plant available, 1 m ton of the Hopkinsville sludge would supply the equivalent of 15.6 kg P, and have a value of \$15.60. If only N and P were considered, the value of the sludge would be \$18.40/m ton.

The annual estimated application rate was approximately 22 m tons/ha, equivalent to 140 kg/ha of available N and 343 kg/ha of total P, the approximate requirements of fescue. The N equivalent was possibly less than stated, because of NH $_3$  losses from the spreading operation.

Other elements—Concentrations of all of the elements were within the normal ranges reported, although the trace element Hg was somewhat higher and Cd, Cr, Cu, Mn, Pb, and Zn less than median concentrations reported. In view of heavy metal content, the Hopkinsville sludge is suitable for application onto agricultural land.

## Soil Analyses--

Total metals—Table 24 presents total and DTPA-extractable metal analyses of treated and control surface and subsurface soils. Total concentrations of the metals Cd, Cu, Ni, Zn, and Pb analyzed in the HNO3-HClO4 digests were within the ranges normally reported for soils; Cd, Cu, Zn, and Pb enrichment was noted in the surface soil as a result of sludge applications. The data indicated virtually no evidence of metal movement below the depth of incorporation; the application rate was not heavy, and the sludge did not contain large quantities of any heavy metal.

<u>DTPA-extractable metals--Cadmium</u>, Cu, and Zn enrichment in the surface soil has apparently resulted from sludge application practices over the years (Table 24). Limited Cu and Zn migration to 30 cm was noted.

The DTPA-extractability, as expressed by percent of total metals extracted, was greater in the surface than the subsurface layers. The sludge-borne metals were more effectively extracted by DTPA than the metals occurring naturally in the soil.

# Plant Analyses--

Metal analyses on fescue grass of the treated and control plots are presented in Table 25. Although concentrations of Cu, Cd, Zn, and Pb were higher in the treated plot than the control,

TABLE 24. CONCENTRATIONS OF TOTAL AND DTPA-EXTRACTABLE METALS IN COMPOSITE SURFACE AND SUBSURFACE SAMPLES FROM HOPKINSVILLE, KENTUCKY\*

Soil			Tota	1+			DTPA-Extra	ctable	
Depth	Cd	Cu	Ni	Zn	РЬ	Cd	Cu	Ni	Zn
- ċm -		-	- µg/g				- + μg/g	)	<del></del>
Treated:									
0-15	1.33	26.3	24.8	109	30.2	0.30(22.6)#	6.12(23.3)	1.59(6.4)	17.0 (15.6)
15-30	0.95	17.7	27.0	68.0	17.7	0.08(8.4)	1.20(6.8)	1.10(4.1)	1.49(2.2)
30-61	0.69	18.2	39.2	55.0	23.2	0.07(10.1)	0.55(3.0)	1.03(2.6)	0.48(0.9)
61-91	0.84	15.4	26.3	60.4	16.0	0.05(6.0)	0.54(3.5)	0.80(3.0)	0.46(0.8)
91-122	0.63	15.4	35.4	60.1	24.2	0.06(9.5)	0.47(3.1)	0.70(2.0)	0.44(0.7)
Control:									
0-15	0.83	9.8	21.5	50.2	19.5	0.09(10.8)#	0.79(8.1)	1.20(5.6)	0.94(1.9)
15-30	0.77	13.7	25.6	62.3	18.0	0.05(6.5)	0.48(3.5)	1.10(4.3)	0.42(0.7)
30-61	0.78	16.8	37.2	64.0	39.4	0.08(10.3)	0.40(2.4)	1.40(3.8)	0.39(0.6)
61-91	1.02	15.4	26.8	71.6	15.3	0.05(4.9)	0.50(3.3)	1.30(4.9)	0.40(0.6)
91-122	0.68	15.4	36.4	78.0	23.6	0.05(7.4)	0.60(3.9)	1.30(3.6)	0.49(0.6)

<sup>\*</sup>Oven-dry weight basis.

 $<sup>+</sup>HNO_3-HCTO_4$  digestion.

<sup>#</sup>Percent of total concentration.

TABLE 25. METAL CONCENTRATIONS IN FESCUE FROM HOPKINSVILLE, KENTUCKY\*

			Leaves					Grains		
Sample	Cd	Cu	Ni	Zn	₽b÷	Cd	Cu	Ni	Zn	РЬ
			- μg/g -	_			-	- μg/g -	-	
Treated:										
Composite	0.39	5.39	3.10	31.3	5.08					
Section 1	0.30	4.63	3.65	27.6						
Section 2	0.18	4.83	3.89	26.7						
Section 3	0.39	11.7	6.38	55.3						
Section 4	0.28	7.50	3.65	40.7						
Section 5 Mean Std. Dev. Control:	0.22 0.27 0.08	7.80 7.29 2.87	3.83 4.28 1.18	37.6 37.6 11.6						
Composite	0.23	4.64	2.70	20.1	4.20					
Section 1	0.25	4.34	2.92	21.4						
Section 2	0.19	4.83	3.81	21.7						
Section 3	0.08	3.64	2.92	17.9						
Section 4	0.14	4.14	3.79	18.4						
Section 5 Mean Std. Dev.	0.23 0.18 0.07	4.98 4.39 0.54	5.10 3.71 0.89	23.2 20.5 2.28						

<sup>\*</sup>Oven-dry weight basis.

tLead was analyzed on composite only.

these concentrations were within the range normally reported for fescue (Chapman, 1966). Moreover, mean concentrations in the forage were in most analyses in close agreement with the composite sample representing the five sections.

Microbiology--

The raw sludge sampled on May 20, 1976, contained two species of ova; one of human intestinal parasites and one of animal origin. The second sample sequence, obtained on August 25, 1976, contained the ova of a canine parasite. The presence of these ova was not unexpected.

The raw sludge for this site contained  $\frac{Salmonella}{Salmonella}$  sp. (seratyped as,  $\frac{Salmonella}{Salmonella}$  paratyphi C.). Concentrations of other index bacteria were of the right order of magnitude in comparison to published data on the microbiological character of typical sewage sludges.

As sludge was applied to the test plot only two weeks before sampling, the stabilized sludge microbiological data can be considered to be fairly representative. The chronology of short-term inactivation of microorganisms demonstrated in the soil-sludge matrix was of special interest.

Microbial populations of index organisms in the treated soil profile were two to three logs higher than comparable sites, where sludge had been applied at a much earlier date, (prior to the actual field sampling). The concentration of organisms in a vertical dimension from surface to 122 cm was also relatively constant. The ratio of FS/FC organisms (3:1 to 30:1) suggested the influence of livestock (Mara, 1977), in the treated plot, as reported for this site under Farming Practices.

No intestinal parasite ova were recovered from the soils. The fescue hay (61 cm during sampling) was high enough to have escaped direct contamination from the sludge applied two weeks previously. The low numbers of index organisms detected in the plant tissue support this; the findings compare well to the other sites for which "tall" crops exhibited a low level of sludge-associated organisms.

Aeolian transport in association with dust particles is a likely source of the low-level fecal contamination.

Sludges, Soils, and Plant Conclusions --

 Accumulation of Cd, Cu, Zn, and Pb in the surface soil of the sludge-treated plots was evidenced, but the concentrations of these metals were within the ranges normally found in soils.

- DTPA analyses suggest limited movement of Cu and Zn to a depth of 30 cm.
- Since no depth of groundwater table was given for the Hopkinsville site, it was difficult to estimate the possibility of groundwater pollution. However, since the metals analyzed showed practically no movement beyond the depth of incorporation, and the soil properties at this site restricted downward migration of heavy metals, it is reasonably safe to assume that the land-spreading practices have not resulted in contamination of the groundwater with heavy metals.
- The fescue Cd concentrations from the treated plot were statistically significantly higher than those from the control (difference of 0.09  $\mu g/g$ ), but were within the range normally reported for fescue.

# Frankfort, Indiana

Sludge Characteristics--

Nitrogen and phosphorus—The concentrations of total N (3.04 percent),  $NO_3-N$  (13.4 µg/g),  $NH_4-N$  (0.74 percent), and organic—N (2.3 percent) in sludge from the Frankfort STP were within the reported ranges.

The practice of spreading liquid sludges onto the surface without incorporation into the soil at Frankfort probably resulted in considerable loss of ammonia to the atmosphere. Based on previous assumptions, the Frankfort sludge contained 8.3 kg available N and had a value of \$3.65/m ton.

The P concentration of the sludge (1.57 percent) was in the range reported for sludges, but less than median concentrations (2.3 percent). One m ton of the Frankfort sludge would supply 15.7 kg of available P and have a value of \$15.70. In terms of N and P contents, the sludge was worth \$19.35/m ton; the sludge value was based principally on its P content.

The annual estimated application rate was approximately 30 m tons/ha, equivalent to 249 kg/ha of available N and 471 kg/ha of total P, in excess of plant requirements. The N equivalent was possibly less than stated depending on NH $_3$  losses from the spreading operation.

Other elements--Concentrations of all metals were comparable to median concentrations normally reported for sludges except for Cd (an excessively high 1,500  $\mu$ g/g). Based on the Cd concentration and high Cd/Zn ratio (0.698), the Frankfort sludge is not suitable for agricultural utilization.

## Soil Analyses--

Total metals--Table 26, which presents total and DTPA-extractable metal analyses of treated and control surface and subsurface soils, shows significant accumulations of Cd, Cu, Ni, Zn, and Pb in the surface soil as a result of sludge spreading over the years. However, except for Cd, concentrations of metals in the surface soil from the sludge-treated plots were within the range normally reported for soils.

There is indication that Cd has moved to at least the 61-cm depth in the treated plot. Some movement of Zn into the 15- to 40-cm depth is also suggested.

DTPA-extractable metals--Concentrations of DTPA-extractable metals were higher in the surface than the subsurface soils for both treated and control plots. Accumulations of heavy metals were more evident from the DTPA extraction data than the total metal data. Again, this indicates movement of Cd to at least a depth of 61 cm.

DTPA appeared to extract considerably higher percentages of the total Cd than Cu, Ni, or Zn. The percentage of total metals extracted by DTPA was greater in the surface soil than in the subsurface soil from the treated plot.

### Plant Analyses--

There were no major differences in concentrations of Ni, Zn, or Pb in the leaf tissue of wheat from both treated and control plots, as shown in Table 27. This was not in agreement with the soil data. However, since the DTPA-extractable concentrations of these three metals were relatively low in the sludged soil, variations of this type can be expected.

Concentrations of Cu and noticeably Cd in wheat leaves increased as a result of sludge application. However, these Cu concentration levels were considered normal and should not present a health hazard.

Except for Cd, the metal concentrations in wheat grains from the treated plot were slightly lower than the control plot. The relatively low concentrations of these metals in the DTPA extracts could in part account for this observation.

Probably, the most significant finding was the elevated concentrations of Cd in the wheat grains of the treated plot. Cadmium values varied from 0.58 to 1.89  $_{\mu}$ g/g with a mean of 1.26  $_{\mu}$ g/g. The difference in Cd content between the treated and control plot was about 1.1  $_{\mu}$ g/g. Considering that wheat grown at this sludge-treated site is used for flour, the Cd level in the grains could pose a health hazard. Assuming a per-capita

TABLE 26. CONCENTRATIONS OF TOTAL AND DTPA-EXTRACTABLE METALS IN COMPOSITE SURFACE AND SUBSURFACE SAMPLES FROM FRANKFORT, INDIANA \*

Soil			Total	†			DTPA-Extra	ctable	
Depth	.Cd	Cu	Ni	Zn	Pb	Cd	Cu	Ni	Zn
- cm -		-	- μg/g				- <b>-</b> μg/g		
Treated:									
0-15	12.9	30.0	25.5	88.0	27.8	7.70(59.7)#	5.75(19.2)	2.78(10.9)	8.10(9.2)
15-40	2.17	15.1	24.5	61.6	16.9	0.87(40.1)	1.60(10.6)	1.70(6.9)	1.74(7.0)
40-61	1.05	19.1	34.6	57.0	17.9	0.28(26.7)	1.15(6.0)	1.80(5.2)	1.00(1.8)
61-91	0.92	19.1	34.3	56.1	19.9	0.17(18.5)	1.21(6.3)	1.70(5.0)	0.69(1.2)
91-122	0.83	22.7	38.5	59.1	18.4	0.13(15.7)	0.91(4.0)	1.70(4.4)	0.61(1.0)
Control:									
0-15	0.92	10.6	19.9	49.2	17.2	0.18(19.6)#	1.24(11.7)	1.87(9.4)	1.62(3.3)
15-40	0.75	15.5	32.6	54.0	21.0	0.08(10.7)	0.92(5.9)	1.50(4.6)	0.60(1.1)
40-61	0.80	20.9	37.8	60.8	21.0	0.07(8.8)	0.90(4.3)	1.50(4.0)	0.54(0.9)
61-91	0.79	22.0	42.0	57.2	17.3	0.07(8.9)	0.70(3.2)	1.50(3.6)	0.38(0.7)
91-122	0.84	22.7	44.5	55.3	18.2	0.07(8.3)	0.60(2.6)	1.20(2.7)	0.38(0.7)

<sup>\*</sup>Oven-dry weight basis.

<sup>†</sup>HNO3-HCT:04 digestion.

<sup>#</sup> Percent of total concentration.

TABLE 27. METAL CONCENTRATIONS IN WHEAT FROM FRANKFORT, INDIANA\*

			Leaves					Grains		
Sample	Cd	Cu	Ni	Zn	Pb†	Cd	Cu	Ni	Zn	РЬ
			- μg/g -	-			-	- μg/g -		
Treated:										
Composite	0.87	6.50	3.61	30.4	1.78	1.24	4.64	1.34	44.8	0.67
Section 1	0.85	6.93	3.99	32.3		0.58	4.16	1.08	40.4	
Section 2	0.85	6.32	2.85	29.8		1.34	4.68	1.20	45.7	
Section 3	0.56	6.68	3.80	28.7		1.38	4.83	1.27	51.4	
Section 4	1.01	6.81	2.47	29.3		1.13	4.55	1.18	44.4	
Section 5 Mean Std. Dev. Control:	1.32 0.92 0.28	6.99 6.75 0.27	3.80 3.38 0.68	28.7 29.8 1.49		1.89 1.26 0.47	5.01 4.65 0.32	1.45 1.24 0.14	43.5 45.1 4.04	
Composite	0.28	5.81	3.99	29.1	1.68	0.20	5.95	1.87	57.0	0.73
Section 1	0.27	6.32	5.38	26.7		0.22	6.66	1.88	61.9	
Section 2	0.37	5.95	3.99	33.3		0.19	6.00	1.85	55.9	
Section 3	0.20	5.95	3.80	27.1		0.16	5.99	1.57	52.6	
Section 4	0.20	5.53	3.99	25.8		0.20	5.90	2.18	59.9	
Section 5 Mean Std. Dev.	0.34 0.28 0.08	5.77 5.90 0.29	3.61 4.15 0.70	26.7 27.9 3.04		0.21 0.20 0.02	6.28 6.17 0.31	2.41 1.98 0.32	57.3 57.5 3.60	

<sup>\*</sup>Oven-dry weight basis.

thead was analyzed on composite only.

consumption of 100 g of wheat/day, and that all of the Cd in this wheat is carried through the milling processes, the daily intake of Cd from this source could be 126  $\mu g$  or, roughly, double the total average daily intake.

The Zn concentration of the control wheat grains was significantly higher than those from the treated plot. The immature leaves (stalk and grain) from the treated and control plots, however, had Zn concentrations approximately the same. The treated surface soil Zn levels (88  $\mu g/g$ ) were indicative of enrichment due to sludge utilization when compared to the control plot (49.2  $\mu g/g$ ). Since the Cd levels in the grains were as high as shown, possible suppression of Zn uptake is indicated.

### Microbiology--

The raw and stabilized sludge contained ova of three species, two of the common human parasitic helminths and one of a canine parasite. Viability tests for these ova were inconclusive because of the low density of the eggs for which incubation and embryonation could be attempted.

Fecal coliform concentrations in the raw sludge were unusually low. There is no rational explanation for this anomaly; fecal streptococci concentrations in the raw sludge appeared to be of the right order of magnitude in comparison to other published data. No salmonella or shigella species were detected.

Populations of index bacteria in the test and control plots were almost identical, suggesting that both microbial populations were homeostatic at some desirable equilibrium condition.

The spring wheat tissue suggested some evidence of sludge contamination probably associated with aeolian transport. The numbers of FC were very low to none, while the FS concentration was of the same order of magnitude as the corn leaf tissue from Columbus, Indiana.

Sludge, Soil, and Plants Conclusions--

- The high Cd concentration of the Frankfort sludge indicates that it is not suitable for utilization on agricultural land.
- Repeated sludge applications have resulted in surface accumulations of all 5 metals measured.
- Total and DTPA-extractable analyses indicate that Cd has migrated to at least the 61-cm level.

- The high application rates and elevated Cd in the sludge have resulted in elevated Cd wheat grain levels posing a possible public health hazard.
- Section variations in surface soil Cd concentrations in the treated plot relate well to the respective Cd wheat grain values from those same sections.
- The Zn concentrations in wheat grains from the treated plot were significantly lower than the control, suggesting a possible suppression as a result of the high Cd uptake.

## Kendallville, Indiana

Sludge Characteristics--

Nitrogen and phosphorus--The concentrations of total N (1.76 percent), organic-N (1.48 percent), NH<sub>4</sub>-N (0.28 percent) and NO<sub>3</sub>-N (4.05  $\mu g/g$ ) in sludge from the Kendallville STP were in the ranges previously reported.

Spreading liquid sludge onto the surface without incorporation into the soil probably resulted in considerable loss by volatilization of ammonia-N. Based on previous assumptions, the sludge contained 4.34 kg of available N and had a market value of 1.91/m ton.

The P concentration of the sludge (1.41 percent) was in the range reported by Sommers but considerably less than the reported median concentration (2.3 percent). One m ton of the Kendallville sludge would supply 14.1 kg of available P and have a current value of \$14.10. In terms of N and P contents, the sludge was worth \$16.01/m ton, a value principally based on its P content.

The annual estimated application rate was approximately 19.7 m tons/ha. This is equivalent to 85 kg/ha of available N and 278 kg/ha of total P, excessive for alfalfa requirements. The N equivalent was possibly less than stated depending on NH $_3$  losses from the spreading operation.

Other elements—Concentrations of all metals, except Se, Mn, and Co were within the reported ranges. However, concentrations of Mg, Cd, Cr, Pb, Se, and Zn were greater than median concentrations. Additions of Mg to the soil via sludge application would probably not have an adverse effect on either yield or quality of the crop (alfalfa), while additions of Cd, Cr, Pb, Se, and Zn might.

The sludge from Kendallville contained an unusually high concentration of Se (17.2  $\mu g/g)$ , which when present in an alkaline soil (such as the soil in Kendallville) at concentrations much above a few  $\mu g/g$ , can be absorbed by certain forage crops in amounts considered unsafe for animal consumption. Based on metal concentrations in the sludge and the total quantity of sludge applied over the years, Zn would be the limiting element in determining application rates.

### Soil Analyses--

Total metals--Table 28 presents total and DTPA-extractable metal analyses of the treated and control surface and subsurface soil samples. The total concentrations of Cd, Cu, Ni, Zn, and Pb were in the range commonly reported.

In the treated plot, the concentrations of Cu, Ni, Zn, and Pb in surface soils were greater than in the subsurface soils, while metal concentrations in subsurface soils were reasonably uniform throughout the soil column. Essentially all of the metals applied to the surface of the treated plot were concentrated in the surface soil. The elevated concentrations of Cu, Ni, Zn, and Pb in the treated surface soil were caused by repeated sludge applications.

Selenium concentrations for surface and subsurface composite treated and control soils were all reported as being <0.016 ug/g.

DTPA-extractable metals--The trends for DTPA-extractable metals were somewhat comparable to those for the total metals (Table 28); concentrations in the treated surface soil were greater than in each of the successive subsurface layers. The treated plot DTPA Zn and Cu results indicated possible limited movement to a depth of 30 cm. The control plot showed metal concentrations similar in relation to depth to those for the treated plot, although absolute concentration differences between the control and treated soils were appreciable. DTPA also appeared to extract a considerably higher percentage of the total Cd, than Cu, Ni, or Zn.

Concentrations of metals in the surface soil composite compared very well with the mean of the samples making up the composite, verifying the relative accuracy of the sample taking, compositing, and analysis of surface soil samples.

#### Plant Analysis --

Total metal analysis of alfalfa from the treated and control plots is presented in Table 29. The concentrations of Cd in alfalfa plants for both plots (0.6 to 0.9  $\mu g \text{Cd/g})$  were somewhat greater than those commonly observed for plants grown on soils from other regions (0.05 to 0.2  $\mu g \text{Cd/g})$ . The only

TABLE 28. CONCENTRATIONS OF TOTAL AND DTPA-EXTRACTABLE METALS IN COMPOSITE SURFACE AND SUBSURFACE SAMPLES FROM KENDALLVILLE, INDIANA\*

Soil		_	Total	ተ			DTPA-Extrac	table	
Depth	Cd	Cu	Ni	Zn	Pb	Cd	Cu	Ni	Zn
~ cm -			- µg/g				- ¬ μg/g		
Treated:									
0-20	0.52	21.8	26.5	113	32.9	0.27(51.9) <sup>#</sup>	5.92(27.1)	2.29(8.6)	27.2 (24.2)
20-30	0.16	19.6	37.3	64.8	12.9	0.14(87.5)	2.46(12.6)	1.40(3.8)	4.63(7.2)
30-61	0.25	21.0	44.9	67.0	13.0	0.12(48)	1.10(5.2)	1.28(2.9)	1.02(1.5)
61-91	0.46	21.3	44.6	65.7	13.0	0.11(23.9)	0.95(4.5)	1.27(2.9)	1.06(1.6)
91-122	0.60	19.7	43.4	63.9	14.5	0.11(18.3)	0.95(4.8)	1.27(2.9)	0.92(1.4)
Control:									
0-20	0.29	11.5	20.6	55.0	11.3	0.17(58.6) <sup>#</sup>	1.50(13)	1.82(8.8)	2.81(5.1)
20-30	0.22	14.6	28.2	60.3	11.0	0.16(72.7)	1.46(10)	1.55(5.5)	1.66(2.8)
30-61	0.28	19.9	38.0	66.9	12.2	0.13(46.4)	1.21(6.1)	1.16(3.1)	1.08(1.6)
61-91	0.55	19.4	40.6	61.9	12.3	0.13(23.6)	0.95(4.9)	1.21(3.0)	0.59(1.0)
91-122	0.51	18.5	37.7	57.6°	12.8	0.11(21.6)	0.89(4.8)	1.22(3.2)	0.89(1.6)

<sup>\*</sup>Oven-dry weight basis.

 $tHNO_3-HCTO_4$  digestion.

<sup>#</sup>Percent of total concentration.

TABLE 29. METAL CONCENTRATIONS IN ALFALFA FROM KENDALLVILLE, INDIANA\*

			Leaves					Grains		
Sample	Cd	Cu	Ni	Zn	Pb+	Cd	Cu	Ni	Zn	Pb
		-	- µg/g -	-			-	- μg/g -	-	
Treated:										
Composite	0.89	8.48	6.44	78.1	5.42					
Section 1	0.74	9.77	6.63	61.9						
Section 2	0.89	10.70	7.76	145						
Section 3	0.65	8.98	7.28	57.8						
Section 4	0.68	8.23	6.78	73.9						
Section 5 Mean Std. Dev. Control:	0.74 0.74 0.09	8.88 9.31 0.95	6.63 7.02 0.49	76.6 83.0 35.5						
Composite	0.74	8.83	5.37	37.6	5.86					
Section 1	0.62	7.77	6.95	31.5						
Section 2	0.63	8.46	6.84	37.2						
Section 3	0.57	9.72	6.78	40.0						
Section 4	0.61	8.28	6.63	37.8						
Section 5 Mean Std. Dev.	0.57 0.60 0.03	8.08 8.46 0.75	6.29 6.70 0.26	35.9 36.5 3.15						

<sup>\*</sup>Oven-dry weight basis.

tLead was analyzed on composite only.

other metal constituent which exceeded normal concentration levels in alfalfa was Zn.

The mean concentration between treated and control Cu and Ni values in the alfalfa was not significantly different. Mean plant Cd concentrations from the treated plot (0.74  $\mu g/g$ ) were slightly higher than the mean for the control plot (0.60  $\mu g/g$ ). The most notable difference occurred with Zn; mean Zn concentrations for alfalfa grown on the treated plot (83  $\mu g/g$ ) were substantially higher than for the control (36.5  $\mu g/g$ ). Phytotoxic concentrations of Zn for alfalfa are not well established, but are suspected to occur at about 150  $\mu g/g$ ; this level had not been approached except in section 2 of the treated plot.

Treated and control composite plant samples showed Se levels of <0.016  $\mu g/g$ .

### Microbiology--

No intestinal parasite ova were detected in any of the sludge samples from Kendallville. The small difference in numbers of FC and FS in both raw and stabilized sludge at this facility can be attributed in part to inadequate digester capacity and, during certain periods of high solids loading, the partial stabilization of the sludge before its application to the land. The high concentration of indicator organisms in the applied sludge did not appear to alter the percent inactivation occurring in the soil-sludge environment. As measured by the concentration of FC and FS organisms in the soil, die-off in excess of 99 percent was indicated.

In the crop rotation scheme for the treated plot, the alfalfa is cut one year and grazed the next; the sampling year was the year for cutting. Had livestock been grazing the treated field in the sampling year, higher densities of FS would likely have been detected in the soil.

Leaf tissue contained relatively high concentrations of both FC and FS organisms. This is readily explained: the perennial alfalfa was in the soil and established during the periods when the liquid sludge was applied. It is probable that not even the frequent summer rainfall at this site would wash the vegetative structure free of sludge-associated microbes; the low-lying nature of the alfalfa plant and its dense leaf structure would also favor entrapment of sludge-index bacteria. No helminth ova were found on the leaf tissue, although from the sparse evidence because of the operational peculiarities of this sludge application system and cover crop. it should not be inferred that they are not in fact present.

Sludge, Soil, and Plant Conclusions --

- Copper, Ni, Zn, and Pb concentrations were statistically significantly greater on the treated surface soil. This was the direct result of repeated sludge applications.
- The high concentrations of Zn in the soils of the treated plot have resulted in similarly elevated Zn concentrations in the alfalfa plants.
- The Pb enrichment of the treated soils has not resulted in elevated Pb alfalfa levels.
- Total metal analyses indicate no significant movement of metals in the surface profile, while DTPA Cu and Zn results indicate possible limited movement to 30 cm.
- The impact on groundwater is probably minimal.
- Cd concentrations in the alfalfa grown on the treated plot were statistically significantly greater than Cd levels of the control crop; the difference was 0.14 µq/q.
- Selenium had not accumulated in the surface and subsurface soil.

## Columbus, Indiana

Sludge Characteristics--

Nitrogen and phosphorus--The concentrations of total N (3.57 percent), NO<sub>3</sub>-N (60  $\mu$ g/g), NH<sub>4</sub>-N (1.27 percent), and organic N (2.3 percent) in the Columbus sludge were in the ranges previously reported.

The spreading of liquid sludges onto the surface without soil incorporation has probably resulted in considerable volatilization of NH<sub>4</sub>-N to the atmosphere. Based on previous assumptions, the Columbus sludge contains 10.95 kg available N and has a value of \$4.82/m ton at current fertilizer prices.

The P concentration of the sludge (1.33 percent) was in the range normally reported for sludges, but less than the reported median value (2.3 percent). If all of the P in the sludge is considered available, 1 m ton of the Columbus sludge would supply 13.3 kg of available P and have a market value of \$13.30. Combining the N and P contents, the sludge has a value of \$18.12/m ton.

The annual estimated application rate is approximately 32.5\* m tons/ha, equivalent to 356 kg/ha of available N and 865 kg/ha of total P, which is in excess of plant requirements. The nitrogen equivalent is possibly less than stated depending on NH3 losses from the spreading operation.

Other elements--Concentrations of heavy metals and other constituents, except Co analyzed in the Columbus sludge fell within typical reported ranges, although the concentrations of Ni and Se were slightly higher than reported median concentrations.

Soil Analyses--

Total metals--Table 30 presents total and DTPA-extractable metal analyses of the treated and control surface and subsurface soils. The total concentrations of Cd, Cu, Ni, Zn, and Pb in the surface soil were very high and reflected the substantial sludge applications made to this site over the past several years.

There were indications that Ni and probably Pb had moved to a depth of at least 46 cm in the treated plot. Cadmium at the two lowest depths of the treated plot was high (0.78 and 1.30  $\mu g/g$ , respectively) but lower than the control, indicating high Cd levels of natural origin. Further interpretation of analytical data relative to metal movement through the soil column was difficult due to the extensive earth moving required prior to replanting the plot to corn (spring 1975).

<u>DTPA-extractable metals</u>—Concentrations of DTPA-extractable metals showed trends similar to those observed for total metals Concentration of DTPA-extractable Ni in the surface soil of the treated plot was considerably greater than that normally reported for sludge amended soils. Movement of Ni to the 30-to 46-cm depth or lower was suggested by the DTPA data.

The DTPA extractability of heavy metals in the treated plot was not as high as expected despite the heavy sludge applications and high organic-C in the surface soil; generally, more Cd was extracted by DTPA than Cu, Ni, or Zn. However, the extractability of metals varied with soil depth; more Cu, Ni, and Zn were extracted from the surface than subsurface soils. While the percent of total Ni and Zn extracted was higher in the surface soils of the treated plot compared to the control plot, the opposite was true for Cd and Cu. It must also be noted that, for reasons unknown, a higher percentage of total Cd was

<sup>\*</sup>It was assumed that continuing application rates would be at half the annual average.

TABLE 30. CONCENTRATIONS OF TOTAL AND DTPA-EXTRACTABLE METALS IN COMPOSITE SURFACE AND SUBSURFACE SAMPLES FROM COLUMBUS, INDIANA \*

Soil Depth			Total	†		DTPA-Extractable				
	Cd	Cu	Ni	Zn	Pb	Cd	Cu	Ni	Zn	
- cm -	μg/g					μg/g				
Treated:										
0-30	4.37	185	521	625	190	0.74(16.9)#	21.40(11.6)	60.4 (11.6)	59.1 (9.5)	
30-46	0.39	19.7	41.1	70.0	24.9	0.22(56.4)	2.25(11.4)	2.67(6.5)	2.02(2.9)	
46-61	0.41	17.9	31.9	57.0	16.4	0.17(41.5)	1.44(8.0)	1.51(4.7)	1.54(2.7)	
61-91	1.46	21.7	38.4	57.0	21.6	0.13(8.9)	1.84(8.5)	2.85(7.4)	1.79(3.1)	
91-122	1.75	19.4	42.0	51.3	51.0	0.20(11.4)	1.64(8.5)	2.72(6.5)	2.11(4.1)	
Control:										
0-30	0.78	20.3	30.0°	80.6	21.9	0.19(24.4) <sup>#</sup>	2.58(12.7)	1.73(5.8)	1.94(2.4	
30-46	0.49	18.8	34.5	75.0	19.8	0.14(28.6)	1.90(10.1)	1.46(4.2)	0.80(1.1	
46-61	0.49	15.7	32.4	61.7	15.8	0.13(26.5)	1.55(9.9)	1.20(3.7)	0.84(1.4	
61-91	1.57	9.65	32.8	35.5	19.5	0.12(7.6)	0.70(7.3)	0.93(2.8)	0.49(1.4	
91-122	2.00	5.91	32.1	22.3	22.6	0.10(5)	0.40(6.8)	0.69(2.2)	0.42(1.9	

<sup>\*</sup>Oven-dry weight basis.

 $<sup>+</sup>HNO_3-HC1O_4$  digestion.

<sup>#</sup>Percent of total concentration.

extracted from the 30- to 46- and 46- to 61-cm depth than from other soil depths.

### Plant Analyses--

Total metal analyses of corn leaves and grain from the treated and control plots at Columbus are presented in Table 31. These analyses indicate significant increases in the concentration of Cd, Cu, Ni, and Zn in corn leaves apparently due to sludge applications over the years while, except for slightly elevated Cd levels, metal concentrations in corn grain showed practically no increase. The metal concentrations were relatively low and fell well within the ranges reported for edible crops.

## Microbiology--

No intestinal parasite ova, <u>Salmonella</u> <u>sp.</u> or <u>Shigella</u> <u>sp.</u> were detected in any of the sludge samples. Concentrations of FC and FS appeared typically to those associated with similar sewage sludges.

In the treated plot, approximately one log reduction of FS appeared per 20-cm depth increase; no particular significance can be attached to the absence of FC organisms in the treated and control soils for the Columbus site.

The leaf tissue was not positive for either Salmonella sp. or Shigella sp. As with the soils, the presence of FS in low concentration may indicate animal contamination by aeolian transport of dust containing the organisms. No helminth ova would be expected on leaf tissue unless mature corn was sprayed with sludge or there was incidental avian dispersal of sludge.

Sludge, Soil, and Plant Conclusions --

- Sludge spreading practices have resulted in significant enrichment of surface soils with Cd, Cu, Ni, Zn, and Pb.
- The heavy metals (Cu, Cd, Zn, and Pb) have not moved in the soil profile much beyond the depth of incorporation.
- Limited Ni movement is indicated by total and DTPAextractable analyses.
- No significant heavy metal increases were observed in corn grains.
- Although there is indication of downward migration of Ni, soil analyses show that other metals have not moved in measurable concentrations to depths in the soil below 30 cm. Thus, metal contamination of groundwater caused by sludge spreading practices has probably not occurred.

TABLE 31. METAL CONCENTRATIONS IN CORN FROM COLUMBUS, INDIANA\*

		Grains								
Sample	Cd	Cu	Ni	Zn	Pb †	Cd	Cu	Ni	Zn	Pb <sup>†</sup>
		_	- μg/g -	-			-	- µg/g -		
Treated:										
Composite	2.49	11.8	9.13	53.8	2.98	0.67	2.57	4.01	24.7	1.34
Section 1	2.01	11.5	10.8	61.7		0.23	2.33	4.90	27.5	
Section 2	2.25	11.7	9.52	63.9		0.52	2.19	6.29	29.7	
Section 3	2.35	12.2	9.81	61.7		0.42	3.05	5.66	34.0	
Section 4	1.68	12.4	7.79	44.2		0.48	1.62	3.77	27.4	
Section 5 Mean Std. Dev. Control:	3.27 2.31 0.59	13.5 12.3 0.78	8.17 9.22 1.23	63.4 59.0 8.32		0.48 0.43 0.12	2.62 2.36 0.53	4.03 4.93 1.07	27.4 29.2 2.86	
Composite	1.25	9.81	5.67	38.9	2.93	0.26	2.58	6.64	29.80	1.40
Section 1	1.25	12.1	6.15	45.9		#				
Section 2	2.09	11.4	5.58	42.9						
Section 3	1.32	10.6	6.41	39.6						
Section 4	1.20	10.3	5.19	38.1						
Section 5 Mean Std. Dev.	1.27 1.43 0.37	9.93 10.9 0.88	5.58 5.78 0.49	33.7 40.0 4.65						

<sup>\*</sup>Oven-dry weight basis.

tLead was analyzed on composite only.

<sup>#</sup>No individual samples.

- The Cd corn grain levels were slightly elevated relative to the control, but still within previous reported values considered normal.
- The Cd corn leaf concentrations were statistically significantly higher than the control leaves, the difference being 0.88  $\mu g/g$ . Similarly, Ni and Zn showed significant differences of 3.44 and 18.94  $\mu g/g$ , respectively.

TABLE 32. CHEMICAL COMPOSITIONS OF SLUDGES FOR ALL SITES

Element	Range 	Mean %	Median
Vol. Solids NH4-N Organic-N P K Na Ca Mg Cl Cr Cu Fe Mn Ni Pb Zn H20	36.1 - 74.7 0.276 - 4.18 1.48 - 4.50 1.23 - 3.55 0.131 - 0.535 0.195 - 0.798 1.39 - 6.25 0.359 - 1.23 0.25 - 1.09 0.015 - 0.399 0.039 - 0.714 0.692 - 4.81 0.020 - 0.987 0.002 - 0.05 0.004 - 0.748 0.074 - 4.58 85.6 - 98.3	51.1 1.28 2.83 1.72 0.283 0.481 3.53 0.700 0.36 0.156 0.148 1.76 0.143 0.015 0.110	52.4 1.01 2.46 1.56 0.222 0.611 2.78 0.585 0.275 0.127 0.082 1.35 0.045 0.008 0.015 0.177 93.8
NO <sub>3</sub> -N SO4 Ag As B Cd Co Cr Cu Fe Hg Mn Mo Ni Pb Se Zn pH Cd/Zn	0.41 - 60.0 8.6 - 348 0.95 - 10.2 1.13 - 6.21 32.1 - 72.0 5.70 - 1500 6.17 - 24.9 150 - 3990 390 - 7140 6920 - 48100 1.93 - 49.0 200 - 9870 6.60 - 45.5 20 - 500 40 - 7480 4.23 - 17.2 740 - 45800 6.3 - 8.3 0.0002 - 0.698	19.3 95.4 3.21 3.08 46.9 185 13.9 1560 1480 11760 13.6 1430 18.5 150 1110 8.51 7220 7.58 0.084	13.0 81.7 1.95 2.52 42.5 11.2 9.68 1270 820 13500 9.82 450 11.1 80 150 7.02 1770 7.50 0.007

### SECTION II - GENERAL EVALUATION

# Sludge Characteristics

A summary of information on the sewage treatment plants supplying sludges to the case study sites was presented in Table 8. The size of the secondary treatment plants ranged from small to moderately large; the largest served a population of 135,000 with an average flow of 75,700 cu m/day and the smallest, a population of 9,200 with an average flow of 6,000 cu m/day.

Constituent concentration levels found in the sludges analyzed are thought to be influenced by a host of synergistic factors such as:

- Background water supply,
- Type and percent of contributions by industry to the treatment plant,
- Degree and nature of pretreatment by industry,
- Wastewater treatment processes and chemicals used, and
- Method of sludge stabilization.

In several instances, there appeared to be an inverse relationship between treatment plant size and chemical composition of sludge produced. For example, the sludge from Frankfort, Indiana (population 15,000) had a much higher concentration of Cd (1,500  $\mu g/g$ ) than did that from Springfield, Ohio (population of 135,000). Similarly, the Zn concentration of sludge from Wilmington, Ohio with a population of 10,000 was 45,800  $\mu g/g$ , much greater than other treatment plants serving larger populations. This relationship is believed due to the larger impact of a major heavy metal industrial contributor on a small system as contrasted to its impact on a larger sewerage system.

There were no discernible differences in metal concentrations in the sludge from an activated sludge plant as opposed to a trickling filter treatment facility. The chemical composition of the sludges obtained from the nine treatment plants (Table 32) fall within the broad range of those normally reported for sludges (Page, 1974; and Sommers, 1977). Median concentration results reported herein are reasonably representative of median concentrations for sludges in general, although the mean concentrations of Co, Cd, Hg, Mo, Se, and Zn at the case study sites were considerably greater. This is due to the high concentrations of:

 Co from Macon, Georgia (21.3 μg/g); Kendallville (24.9 μg/g) and Columbus, Indiana (24.1 μg/g);

- Cd from Frankfort, Indiana (1500  $\mu$ g/g);
- Hg from Las Virgenes, California (49 μg/g); and Chippewa Falls, Wisconsin (24.1 μg/g);
- Mo from Springfield, Missouri (43.4 μg/g); and Wilmington, Ohio (45.5 μg/g);
- Se from Macon, Georgia (13.7  $\mu$ g/g); and Kendallville, Indiana (17.2  $\mu$ g/g); and
- Zn from Wilmington, Ohio (4.58 percent).

As shown in Table 33, the Cd to Zn ratio of the sludges ranged from 0.0002 to 0.698.

TABLE 33. SLUDGE Cd/Zn RATIOS - ALL SITES

	Cd/Zn
Macon, Ga	0.007
Las Virgenes, CA	0.008
Wilmington, OH	0.0002
Springfield, MO	0.017
Chippewa Falls, WI	0.006
Hopkinsville, KY	0.005
Frankfort, IN	0.698
Kendallville, IN	0.007
Columbus, IN	0.008

There are a number of federal and state guidelines which specify limits on the quantities of sludge which can be applied to agricultural lands; quantity limits are based upon constituents in sludge considered hazardous to the health of humans and animals, which may adversely affect the growth of crops, or which may degrade the quality of surface or groundwaters. The components in sludges usually considered detrimental are N, P, B, soluble salts, trace metals (principally Cd, Cr, Cu, Pb, Zn, Ni, and Hg), certain organics, and pathogenic bacteria and viruses.

For agricultural utilization, annual loading rates are usually based upon providing an adequate supply of available nitrogen for the crop grown. Only part of the organic nitrogen in sludge is mineralized to available forms during the cropping season; in addition, a portion of the ammoniacal-N is volatilized and lost to the atmosphere. After application to land, estimates of available N in sludge are based upon some assumed values of

the organic-N mineralization rate and NH<sub>4</sub>-N volatile loss. The data in Table 34 show the quantities of each sludge needed to supply nitrogen at an annual rate equivalent to 200 kg available N/ha, assuming 1) 20 percent availability of organic-N, and 2) a 50 percent loss of NH<sub>4</sub>-N. In terms of this particular criterion for the sludges obtained for this study, the annual sludge application rates would vary from a low of 6.8 to a high of 46.1 dry m tons/ha.

The data in Table 34 also show the quantities of P and metals which would be applied each year if the sludges were applied at the above-mentioned rate of 200 kg available N/ha under the assumed conditions. The data show considerably more phosphorus would be applied than normally needed; rates of phosphorus applied in commercial agriculture rarely exceed 80 kg P/ha. Quantities of metals applied vary greatly, e.g., Cd from 0.07 to 36.2, and Zn from 9.2 to 568 kg/ha. Guidelines for Cd normally specify a maximum annual loading rate somewhere between 0.2 and 2 kg Cd/ha. Of the sludges surveyed, two treatment plants (Frankfort and Kendallville, Indiana) would add more Cd annually than is considered permissible if application rates were based upon available N.

Recommended maximum loading rates for metals (Table 35) have been suggested (USDA, 1974, and Knezek and Miller, 1976). Table 36 presents the maximum quantity of each sludge which could be applied and not exceed suggested metal loading limits. table also shows the number of years each sludge could be applied at the assumed N rates before cumulative metal loading would be considered limiting. Utilizing the limits recommended by two groups of researchers (Knezek and Miller, 1976) as a point of reference, the data show maximum quantities of sludge which could be applied are limited in certain cases by Cd, Cu, Ni, and Zn. In would first become limiting for sludges from Springfield, Missouri; Wilmington, Ohio; Hopkinsville, Kentucky; and Kendallville, Indiana; Cu for sludges from Macon, Georgia; Las Virgenes, California; and Chippewa Falls, Wisconsin; Cd for sludge from Frankfort, Indiana; and Ni for sludge from Columbus, Ohio. These same calculations indicate that sludges from Wilmington, Ohio, Kendallville and Frankfort, Indiana, could only be applied to the same parcel of land for 2, 3, and 0 years, respectively, according to these guidelines. The Frankfort, Indiana, sludge seems unfit for utilization on agricultural land. The remaining communities' sludges based upon the above N requirements would be considered acceptable for agricultural use on the same parcel of land for periods of 24 years or more.

Data presented in Table 37 show estimates of the total and annual quantities of sludge and metals applied to each of the soils from the selected sites. The computations for total metal

TABLE 34. QUANTITIES OF SLUDGE, P, AND METALS APPLIED, BASED UPON AVAILABLE N FOR CROPS

					Annua 1	Applicat	ion Rate	•		
	Annual Loading Rate*	P	Cd	Cr	Cu	Ni	Pb	Мо	Se	Zn
	m tons/ha					kg/ha				
Macon, GA	16.7	205	0.2	18.9	16.0	2.3	10.2	0.24	0.23	29.6
Las Virgenes, CA	18.9	323	0.21	2.8	18.0	0.94	0.76	0.19	0.14	25.5
Wilmington, OH	12.4	440	0.14	27.8	5.1	0.37	13.8	0.56	0.05	568
Springfield, MO	6.8	118	0.38	16.5	5.6	2.0	1.0	0.30	0.06	22.5
Chippewa Falls, WI	12.9	173	0.09	16.4	17.7	0.26	1.3	0	0.10	15.4
Hopkinsville, KY	31.4	490	0.24	11.9	17.3	1.9	2.8	0.35	0.20	46.2
Frankfort, IN	24.1	378	36.2	96.2	172	12.0	6.0	0.16	0.14	51.8
Kendallville, IN	46.1	650	2.3	92.7	33.6	3.7	344	0.31	0.79	331
Columbus, IN	12.5	166	0.07	5.1	4.9	2.0	1.3	0.26	0.08	9.2

<sup>\*</sup>To supply the equivalent of 200 kg available N per hectare.

TABLE 35. TOTAL AMOUNT OF SLUDGE METALS ALLOWED ON AGRICULTURAL LANDS\*

	Soil Cat	ion Exchange Capacity (me	q/100 g) <sup>†</sup>
Metal	0 - 5	5 - 15	> 15
	Maxir	num Amount of Metal (lb/a	cre)
Pb	500	1000	2000
Zn	250	500	1000
Cu	125	250	500
Ni	50	100	200
Cd	5	10	20

†Determined by the pH 7 ammonium acetate procedure.

<sup>\*</sup>Knezek and Miller, 1976.

TABLE 36. MAXIMUM QUANTITIES OF SLUDGE PERMITTED, BASED ON RECOMMENDED METAL LOADING LIMITS\*

			imum Total I on Metal			No. of Annual Sewage Applications Permitted†						
Source	Cd	Cu	Ni	РЬ	Zn	Cd	Cu	Ni	Pb	Zn		
			. m tons/	na								
Macon, GA	1,848	573 <sup>#</sup>	1,571	3,607	621	111	34 <sup>#</sup>	94	216	37		
Las Virgenes, CA	1,982	579 <sup>#</sup>	4,400	55,000	815	105	31 <sup>#</sup>	233	2,910	43		
Wilmington, OH	1,964	1,341	7,333	1,982	24#	158	108	591	160	2 <sup>#</sup>		
Springfield, MO	392	671	733	14,667	332 <sup>#</sup>	58	99	108	2,157	49 <sup>#</sup>		
Chippewa Falls, WI	3,129	401#	11,000	22,000	924	242	31 <sup>#</sup>	853	1,705	72		
Hopkinsville, KY	2,895	1,000	3,667	24,444	748 <sup>#</sup>	92	32	117	778	24#		
Frankfort, IN	14.6#	77	440	8,800	512	0#	3	18	365	21		
Kendallville, IN	441	753	2,750	294	153#	10	16	60	6	3 <sup>#</sup>		
Columbus, IN	3,860	1,410	1,375#	22,000	1,486	309	113	110#	1,760	119		

<sup>\*</sup>Knezek and Miller, 1976 (see Table 35).

<sup>†</sup>Based upon loading rates required to yield 200 kg available N per hectare, and limits on metal loading suggested by NC 118 Regional Technical Committee (see Table 35).

<sup>#</sup>Limiting value.

TABLE 37. ANNUAL AND TOTAL LOADING RATES OF SLUDGE AND HEAVY METALS

	Average Annual Sludge Application Rate-	Annı	Average Annual Metal Loading Rate			Total Sludge Application		Total	Meta	l Load	ling	
	Dry Weight (m tons/ha)	Cd	Cu (Kg/	Ni	Zn	Pb	Dry Weight (m tons/ha)	Cd	Cu (	Ni Kg/ha	Zn )	Pb
Macon, GA	28*	0.3	27	3.9	50	17	308*	3.3	297	43	550	189
Las Virgenes, CA	50†	0.6	48	2.5	68	2	149	1.6	142	7.5	201	6
Wilmington, OH	6.8*	0.08	2.8	0.2	311	7.5	116*	1.3	47	3.4	5290	128
Springfield, MO	15†	0.88	13	4.7	52	2.4	237	13.3	194	71	784	36
Chippewa Falls, WI	16*	0.11	22	0.3	19	1.6	80*	0.56	110	1.6	95	8
Hopkinsville, KY	22*	0.17	12	1.4	32	2.0	66*	0.5	36	4.2	96	ε
Frankfort, IN	30+	45	214	15	65	7.5	360*	540	2570	180	774	90
Kendallville, IN	19.7#	1.0	14.4	1.6	142	147.	81*	4.1	59	6.5	582	605
Columbus, IN	65†	0.37	25	10	48	7.8	326	1.9	125	<sub>\$</sub> 50	239	39

<sup>\*</sup>Estimated

 $<sup>^{+}</sup>$ Annual amount computed from multi-year average.  $^{-}$ Data shown represents last 3 years of spreading.

<sup>&</sup>lt;sup>#</sup>1975

loading are based upon the assumption that the composition of sludge in prior years was comparable to that in 1975. In most cases, more sludge has been applied than would be needed to supply sufficient available N (compare Tables 36 and 37). Quantities of Cd applied annually, except for the Frankfort, Indiana site, fall within the range of 0.08 to 1.0 kg/ha; in terms of total metal loading, recommended limits have already been exceeded for Zn applied at Wilmington, Ohio and Cd and Cu applied at Frankfort, Indiana. As has been discussed, there is no perceivable impact due to Zn at the Wilmington site, and there is some question about the validity of the extrapolated Zn value. The sludges have been applied for periods which range from 5 to 17 years; and with the exception of those noted above, total maximum metal loadings are still within suggested limits.

Table 38 presents a comparison of sewage sludges evaluated in terms of current fertilizer costs. The sludges from Wilmington, Ohio and Springfield, Missouri had the greatest economic value at \$40.18 and \$30.38/dry m ton, respectively; the Kendallville, Indiana sludge, the least at \$16.01/dry m ton.

# Soil

#### Total Metals--

A summary of the trace metal concentrations of surface and subsurface soils is presented in Tables 39 and 40. For all sites surveyed, the data show statistically significantly greater concentrations of Zn in the treated surface soil compared to the control and significantly higher surface than subsurface concentrations. This is due no doubt to the much higher concentrations of Zn in sludge than in soil. Cu concentrations, except for the Springfield, Missouri site, and Ni concentrations, except for the Wilmington, Ohio and Chippewa Falls, Wisconsin sites, are likewise significantly greater in the surface than in subsurface soil. Five of the nine sites surveyed (Macon, Georgia; Springfield, Missouri; Hopkinsville, Kentucky; Frankfort, Indiana; and Columbus, Indiana) showed significantly higher concentrations of Cd in surface as compared to subsurface soils.

It should be noted, however, that while these soil metal concentrations are elevated, in most instances they do not exceed normal ranges for soils in the U.S. The total concentrations of metals in surface soils from all plots are in the range considered normal for soils in general. Sludge spreading operations at Macon, Georgia and Columbus, Indiana have produced atypically high concentrations of Zn in the surface soil. Similarly, concentrations of Cd in the surface soils from the treated plots at Macon, Georgia, Las Virgenes, California, Frankfort and Columbus, Indiana; of Cu at Macon, Georgia and Columbus, Indiana; and of Ni at Columbus, Indiana are greater than those usually observed (Table 41). Although the concentration of Cd in the surface soil from the Las Virgenes site is

TABLE 38. COMPARATIVE ECONOMIC VALUES FOR SEWAGE SLUDGE AS A FERTILIZER SOURCE\*

N† kg/m ton	\$/m ton	p# kg/m ton	\$/m ton	Total \$/m ton
12.4	5.46	12.3	12.30	17.76
10.6	4.66	17.1	17.10	21.76
10.7	4.68	35.5	35.50	40.18
29.5	12.98	17.4	17.40	30.38
15.5	6.82	13.4	13.40	20.22
6.36	2.80	15.6	15.60	18.40
8.30	3.65	15.7	15.70	19.35
4.34	1.91	14.10	14.10	16.01
10.95	4.82	13.30	13.30	18.12
	kg/m ton  12.4  10.6  10.7  29.5  15.5  6.36  8.30  4.34	kg/m ton     \$/m ton       12.4     5.46       10.6     4.66       10.7     4.68       29.5     12.98       15.5     6.82       6.36     2.80       8.30     3.65       4.34     1.91	kg/m ton     \$/m ton     kg/m ton       12.4     5.46     12.3       10.6     4.66     17.1       10.7     4.68     35.5       29.5     12.98     17.4       15.5     6.82     13.4       6.36     2.80     15.6       8.30     3.65     15.7       4.34     1.91     14.10	kg/m ton         \$/m ton         kg/m ton         \$/m ton           12.4         5.46         12.3         12.30           10.6         4.66         17.1         17.10           10.7         4.68         35.5         35.50           29.5         12.98         17.4         17.40           15.5         6.82         13.4         13.40           6.36         2.80         15.6         15.60           8.30         3.65         15.7         15.70           4.34         1.91         14.10         14.10

<sup>\*</sup>kg/m ton units\_expressed on an oven-dry weight basis.

<sup>†</sup>N calculated at a current value of \$.44/kg-N

<sup>&</sup>lt;sup>#</sup>P calculated at a current value of \$1.00/kg-P

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<sup>\*</sup>Indicates, with 90% confidence, that means of treated vs control plots have significant statistical differences.

 $tHNO_3-HClO_4$  Digestion: all data expressed on oven dry weight basis.

TABLE 39 (continued)

		(	Cd		Cu		Ni		Zn
Site	Depth	Treat.	Contr.	Treat.	Contr.	Treat.	Contr.	Treat.	Contr.
				• • • • •	μg,	/g			
Hopkinsville, KY	Surface	1.18*	0.78	27.7*	9.5	25.1*	20.7	110*	49.2
	15-30	0.96*	0.75	17.7*	12.6	27.5*	25.3	68.3	64.4
	30-61	0.68	0.72	18.4*	17.0	39.6	38.4	57.2	60.0
	61-91	0.84	0.89	15.4	15.2	25.7	26.4	60.7*	73.0
	91-122	0.62*	0.70	15.7	15.9	36.3	36.4	60.4*	80.1
Frankfort, IN	Surface	13.1 *	0.92	29.8*	10.7	26.1*	20.2	85.3*	50.5
	15-40	2.36*	0.78	16.0	16.5	25.0*	32.5	63.9*	55.1
	40-61	1.08*	0.77	19.3	20.4	35.5	36.9	58.8	57.1
	61-91	0.92	0.91	20.2	21.5	36.5	40.2	58.6	56.3
	91-122	0.82	0.87	22.3	23.6	37.0*	47.3	58.6	56.4
Kendallville, IN	Surface	0.36	0.20	22.9*	11.7	28.7*	22.3	118*	58.5
	20-30	0.15*	0.26	20.8*	15.3	37.2*	28.7	70.7*	62.5
	30-61	0.30	0.29	20.8	20.2	46.4*	38.5	67.3	66.8
	61-91	0.47	0.68	21.0	19.6	48.5*	44.1	67.8	63.6
	91-122	0.73	0.61	19.4	18.2	49.5*	41.8	65.7	58.4
Columbus, IN	Surface	3.52*	0.70	162*	19.5	496*	30.7	555*	81.8
	30-46	0.43*	0.66	19.9	20.8	38.0	33.6	68.1*	76.4
	46-61	0.47	0.48	17.2	16.3	32.7	31.3	57.8	62.4
	61-91	0.78*	1.54	20.3*	10.8	38.3	32.8	54.3*	36.0
	91-122	1.30*	1.80	16.5*	7.2	38.2*	33.1	43.1*	23.2

<sup>\*</sup>Indicates, with 90% confidence, that means of treated vs control plots have significant statistical difference.

		Macon, Georgia Mean	Las Virgenes, California Mean	Wilmington, Ohio Mean	Springfield, Missouri Mean	Chippewa Falls Wisconsin Mean
		Std. Deviation	Std. Deviation	Std. Deviation	Std. Deviation	Std. Deviation
Ele	ment/Plot	• • • • • • • •		μg/g		
Cd	Treated	4.98 0.66	4.00 0.69	0.95 0.19	1.12 0.10	0.44 0.06
	Control	0.55 0.18	3.88 0.29	0.87 0.04	0.75 0.06	0.44 0.06
Cu	Treated	283 33.3	45.9 8.06	15.9 1.13	21.1 2.04	18.0 2.69
1	Control.	12.1 2.91	27.2 0.85	14.3 1.37	25.2 4.71	7.42 0.31
Ni	Treated	68.8 10.9	53.3 0.90	24.3 1.97	30.2 5.54	14.6 0.77
	Control	18.7 3.72	49.8 0.87	25.1 2.22	21.8 0.83	14.2 0.64
Zn	Treated	536 46.5	128 11.2	139 37.7	103 5.58	48.0 1.84
	Control	31.7 8.02	97.1 3.68	49.2 2.59	39.3 1.31	31.4 1.26

 $<sup>{\</sup>rm *HNO_3\text{-}HC1O_4}$  Digestion; all data expressed on oven dry weight basis.

TABLE 40 (continued)

			Hopkinsville, Kentucky Mean Std. Deviation	Frankfort, Indiana Mean Std. Deviation	Kendallville, Indiana Mean Std. Deviation	Columbus, Indiana Mean Std. Deviation
	E 1 eme	ent/Plot			g/g	
_	Cd	Treated	1.18 0.44	13.1 7.47	0.36 0.21	3.52 0.56
		Control	0.78 0.07	0.92 0.02	0.20 0.08	0.70 0.08
	Cu	Treated	27.7 4.02	29.8 5.06	22.9 2.75	162 14.6
111		Control	9.52 0.93	10.7 0.50	11.7 0.28	19.5 1.16
1	Ni	Treated	25.1 0.42	26.1 3.03	28.7 1.85	496 64.2
		Control	20.7 1.65	20.2 1.01	22.3 1.04	30.7 1.24
	Zn	Treated	111 4.16	85.3 12.1	118 20.3	556 56.3
		Control	49.2 2.25	50.5 1.71	58.5 2.99	81.8 3.41

 $<sup>{\</sup>rm ^{\star}HN0_{3}\text{-}HC10_{4}}$  Digestion; all data expressed on oven dry weight basis.

TABLE 41. COMPARISON OF METAL CONCENTRATIONS IN SLUDGE TREATED SOILS WITH CONCENTRATIONS CONSIDERED TYPICAL FOR SOILS

	Concentra	tion in Trea	ted Plot Sur	face Soils f	rom All S	ites
Site	Cd	Cu	Ni	Zn	рН	CEC
	• • • • •			g/g		• • • •
Macon, GA	5.0	283	68.8	536	3.7	17
Las Virgenes, CA	4.0	45.9	53.3	128	6.5	30
Wilmington, OH	1.0	15.7	24.3	139	6.5	30
Springfield, MO	1.1	21.1	30.2	103	7.0	18
Chippewa Falls, WI	0.4	18.0	14.6	48.0	5.5	10
Hopkinsville, KY	1.2	27.7	25.1	111	6.2	31
Frankfort, IN	13.1	29.8	26.1	85.3	6.7	30
Kendallville, IN	0.4	22.9	28.7	118	6.4	27
Columbus, IN	3.5	162	490	556	6.6	30
Typical Range <sup>*,†</sup>	0.1-0.5	20-40	40-80	50-100		

<sup>\*</sup>Bowen, 1966

<sup>&</sup>lt;sup>†</sup>Allaway, 1968

higher than that considered typical for soils, the source is of natural origin, since Cd concentrations in soil from the control and treated plots are approximately the same, and the concentrations in treated and control plots are uniform at all depths sampled (Burau, et al. 1973).

The data in Table 39 also reflect significant differences between treated and control plots which are probably due to natural variation in soils rather than to sludge spreading operations. For example, concentrations of Cd in soils at two depths from the treated plot at Las Virgenes, California three depths at Wilmington, Ohio and Columbus, Indiana are markedly less than concentrations for soils at comparable depths from the control plots. Ignoring possible natural variation, the data at a few sites show evidence that metals may have migrated in the soil profile beyond the depth of incorporation. At the Macon, Georgia treated site, the data show evidence of movement on Zn to a depth of at least 1.2 m; the soil at this site is very sandy, and conditions would be most conducive to metal movement. It is generally thought that movement of metals is enhanced by acid soils and coarse texture. Since the water table is at 1.5 m, this site is probably one where sludge spreading operations should not be practiced. Limited movement of Zn at the Springfield, Missouri and Columbus, Indiana treated sites; of Ni at Springfield, Missouri, Hopkinsville, Kentucky, and Kendallville, Indiana treated sites; and of Cu at Las Virgenes, California, Hopkinsville, Kentucky, Kendallville and Columbus, Indiana, treated sites is also indicated. Overall, however, the data indicate that movement of metals in the soil profiles, except possibly for Zn at the Macon, Georgia site is quite restricted.

A summary of the range, median, and mean concentration for surface soils from the treated and control plots is presented in Table 42. The minimum and maximum soil concentrations of the metals Cd, Cu, Zn, and Pb from the treated plots exceed those for the control plots. Likewise, median and mean concentrations of metals in soils from the treated plots are greater than those from the control plots. Treated plot median metal concentrations in soils are, except for Zn, less than the maximum metal concentrations for the control plots. This affirms that sludge spreading operations, if properly designed and managed, need not exceed typical metal concentrations in soil.

Individual section soil composite samples, as well as five-section composites from the treated and control plots at each location, were analyzed. The data presented in Table 43 for plot composites are in general agreement with those for means of individual samples presented in Table 39. The data show that metal enrichment in the treated plots is, in most instances, restricted to the surface soil. There is an indication of some movement of Cd to the 30-cm depth sampled in the treated plots at

TABLE 42. TOTAL METAL CONCENTRATIONS IN COMPOSITE SURFACE SOILS FOR ALL SITES\*

Element	Plot	Range дд/д .	Median 	Mean
Cd	Treated	0.42 - 12.9	1.33	3.45
	Control	0.29 - 3.77	0.78	1.05
Cu	Treated	10.5 - 267	26.3	68.8°
	Control	7.60 - 27.7	11.9	15.3
Ni	Treated	14.20 - 521	26.5	87.3
	Control	15.2 - 48.0	20.6	24.3
Zn	Treated	49.3 - 625	113	208
	Control	27.7 - 98.1	49.5	53.2
Pb	Treated	9.90 - 325	32.9	80.6
	Control	8.50 - 35.0	19.9	21.3

 $<sup>*</sup>HN0_3-HC10_4$  Digestion.

			Cd		Cu		Ni		Zn		Pb
	Depth	Treat.	Contr.	Treat.	Contr.	Treat.	<u>Contr.</u>	Treat.	Contr.	Treat.	Contr.
Site	(cm)		• • • •	• • • • •			μ <b>g/g</b>				
Macon, GA	Surface	5.89	0.76	267	11.9	67.6	19.1	475	27.7	325	35.0
	20-46	0.96	0.63	13.3	10.5	17.1	13.6	80.4	25.1	35.4	28.5
	46-61	0.47	0.51	10.1	6.00	9.44	12.3	39.0	15.5	19.4	16.8
	61-91	0.33	0.40	6.93	6.56	12.5	13.4	27.5	15.3	13.9	13.1
	91-122	0.99	0.95	19.6	20.4	24.4	24.0	88.5	52.4	49.6	38.9
Las	Surface	3.58	3.77	45.4	27.7	52.9	48.0	120	98.1	21.7	19.9
Virgenes,	30-61	3.79	3.75	29.1	27.6	50.3	50.5	93.4	94.2	19.3	19.7
CA	61-91	3.41	3.22	28.5	28.0	49.5	50.2	93.8	90.2	19.0	19.9
	91-122	3.53	4.11	28.5	28.1	50.2	52.8	90.7	90.9	18.7	20.6
Wilmington,	Surface	0.86	0.87	16.0	14.6	24.7	24.9	190	49.5	47.8	24.6
0H	18-30	0.89	0.91	19.0	17.6	32.3	31.4	62.5	57.3	23.5	22.1
	30-61	0.77	1.08	22.2	21.5	39.0	42.3	65.1	75.0	22.8	22.7
	61-91	1.00	1.08	22.8	23.4	44.3	46.2	66.0	74.2	20.0	21.9
	91-122	1.10	1.06	23.0	24.8	41.8	44.5	67.8	71.3	21.4	20.1
Spring-	Surface	1.17	0.73	10.5	23.6	28.9	19.9	108	36.8	40.2	34.0
field, MO	18-30	0.77	0.68	13.1	14.3	28.2	22.7	41.8	34.8	21.4	24.1
	30-61	0.82	0.70	15.7	12.0	47.6	24.4	48.8	39.2	25.8	22.1
	61-81	0.83	0.82	20.0	13.2	38.7	28.8	52.9	45.7	28.0	20.6
	81-122	0.77	0.89	16.1	15.0	35.8	34.4	57.6	48.0	25.1	26.1
Chippewa	Surface	0.42	0.46	17.4	7.6	14.2	15.2	49.3	31.3	9.9	8.5
Falls, WI	20-46	0.38	0.34	10.5	7.6	16.7	14.3	29.4	24.2	4.4	5.7
	46-61	0.35	0.48	11.3	12.2	16.1	18.3	21.8	30.2	4.0	5.6
	61-91	0.31	0.31	13.0	15.7	14.6	19.6	22.1	27.8	2.4	4.0
	91-122	0.44	0.33	18.5	13.8	14.4	16.3	24.7	19.5	2.7	2.1

 $<sup>{\</sup>rm ^{\star}HNO_{3}\text{-}HC10_{4}}$  Digestion. All data expressed on oven dry weight basis.

TABLE 43 (continued)

			Cd		Cu		Ni		Zn		Pb
Site	Depth (cm)	Treat.	Contr.	Treat.	Contr.	Treat.	Contr. u g/g	Treat.	Contr.	Treat.	Contr.
Hopkins- ville, KY	Surface 15-30 30-61 61-91 91-122	1.33 0.95 0.69 0.84 0.63	0.83 0.77 0.78 1.02 0.68	26.3 17.7 18.2 15.4 15.4	9.8 13.7 16.8 15.4 15.4	24.8 27.0 39.2 26.3 35.4	21.5 25.6 37.2 26.8 36.4	109 68.0 55.0 60.4 60.1	50.2 62.3 64.0 71.6 78.0	30.2 17.7 23.2 16.0 24.2	19.5 18.0 39.4 15.3 23.6
Frankfort, IN	Surface 15-40 40-61 61-91 91-122	12.9 2.17 1.05 0.92 0.83	0.92 0.75 0.80 0.79 0.84	30.0 15.1 19.1 19.1 22.7	10.6 15.5 20.9 22.0 22.7	25.5 24.5 34.6 34.3 38.5	19.9 32.6 37.8 42.0 44.5	88.0 61.6 57.0 56.1 59.1	49.2 54.0 60.8 57.2 55.3	27.8 16.9 17.9 19.9 18.4	17.2 21.0 21.0 17.3 18.2
Kendall- ville, IN	Surface 20-30 30-61 61-91 91-122	0.52 0.16 0.25 0.46 0.60	0.29 0.22 0.28 0.55 0.51	21.8 19.6 21.0 21.3 19.7	11.5 14.6 19.9 19.4 18.5	26.5 37.3 44.9 44.6 43.4	20.6 28.2 38.0 40.6 37.7	113 64.8 67.0 65.7 63.9	55.0 60.3 66.9 61.9 57.6	32.9 12.9 13.0 13.0 14.5	11.3 11.0 12.2 12.3 12.8
Columbus, IN	Surface 30-46 46-61 61-91 91-122	4.37 0.39 0.41 1.46 1.75	0.78 0.49 0.49 1.57 2.00	185 19.7 17.9 21.7 19.4	20.3 18.8 15.7 9.65 5.91	521 41.1 31.9 38.4 42.0	30.0 34.5 32.4 32.8 32.1	625 70.0 57.0 57.0 51.3	80.6 75.0 61.7 35.5 22.3	190 24.9 16.4 21.6 51.0	21.9 19.8 15.8 19.5 22.6

Macon, Georgia and Hopkinsville, Kentucky; and to a depth of 61 cm at Frankfort, Indiana. No consistent evidence of movement of either Cu, Ni, or Pb beyond the depth of incorporation is apparent in any of the sludge treated soils. The data suggests limited movement of Zn beyond the depth of incorporation in soils from the treated sites at Macon, Georgia; Springfield, Missouri; and Frankfort and Kendallville, Indiana.

#### DTPA-Extractable Metals--

The concentrations of metals (Cd, Cu, Ni, and Zn) extracted from surface soils with diethylenetriaminepentaacetic acid (DTPA) are presented in Table 44. The data show results somewhat similar to those already discussed for total metals. metal concentrations extracted from soils of the treated plots exceed those extracted from soils of the control plots. Concentrations of Cu and Zn extracted from the treated soil at Macon, Georgia are unusually high and no doubt reflect Cu and Zn soil enrichment caused by sludge spreading. Although the Zn levels in soils at the Wilmington, Ohio, Springfield, Missouri, and Frankfort and Kendallville, Indiana treated plots were expected to exceed those at Macon, Georgia (see Table 39), the DTPA extractables from these sites were considerably less than those from the Macon, Georgia site. This may be due either to the high organic matter content or low soil pH or combination of these soil properties at the Macon, Georgia site. The range, median, and mean concentration of metals extracted with DTPA for the treated and control plots are summarized in Table 45. observed with total metals, the minimum and maximum, and median and mean concentrations for treated soils exceed those from the control plots. The median concentrations of DTPA-extractable Cd and Cu for treated soils are less than the maximum of the range for the controls, indicating, as previously stated for total metals, that metal enrichment beyond levels considered normal for soils can be controlled.

Bingham, et al. (1975) have shown that when the amount of Cd extracted with DTPA exceeds 2.4  $\mu g/g$ , a Cd phytotoxicity is possible, particularly with sensitive plants. Using this level as a reference point, the soil from Frankfort, Indiana at 7.70  $\mu g/g$  has probably become sufficiently enriched with Cd to adversely affect the growth of plants sensitive to excess Cd in soil.

## <u>Plants</u>

A summary of the types of plants, rotation cycles, and end uses of the crops from the nine sites was presented in Table 7. Crops grown at six of the nine sites (Macon, Georgia; Wilmington, Ohio; Las Virgenes, California; Springfield, Missouri; Hopkins-ville, Kentucky; and Kendallville, Indiana) were forages; all were used as non-dairy cattle feed.

TABLE 44. EFFECTS OF SLUDGE APPLICATIONS ON SOME SOIL PROPERTIES AND CONCENTRATIONS
OF DTPA-EXTRACTABLE HEAVY METALS IN SURFACE SOILS

	Organic			DTPA Extractables - Surface Soils			
C44.	Carbon	11	<b>T L</b>	Cd	Cu	Ni	Zn
Site	(percent)	рН	Texture	• • • •	(μg/	'g)	• • • •
Macon, GA			sandy loam/				
Treated	30.8	3.6	loamy sand	1.42	64.3	13.7	269
Control	1.2	4.1	sandy loam	0.08	1.23	0.72	1.14
Las Virgenes, CA							
Treated	1.58	6.5	clay loam	1.48	5.41	2.51	8.12
Control	1.40	7.1	clay loam	1.37	1.69	1.45	1.01
Wilmington, OH							
Treated	1.60	6.5	silt loam	0.14	1.86	0.70	27.5
Control	1.61	6.6	silt loam	0.13	1.38	.87	1.20
Springfield, MO							
Treated	1.53	7.0	silt loam	0.38	7.07	3.18	36.6
Control	1.83	6.9	silt loam	0.11	7.35	1.49	1.79
Chippewa Falls, WI							
Treated	0.74	5.5	sand	0.06	3.44	1.00	4.28
Control	1.05	5.4	loamy sand	0.06	0.42	0.33	1.54
Hopkinsville, KY							
Treated	1.41	6.2	silt loam	0.30	6.12	1.59	17.0
Control	1.43	6.2 5.2	silty clay loam/silt loam	0.09	0.79	1.20	0.94

31.1

TABLE 44 (continued)

	Organic Carbon			Cd	Extractable Cu	N1	Zn
Site	(percent)	рН	Texture	_	(μg		
Frankfort, IN							
Treated	1.48	6.7	silt loam	7.70	5.75	2.78	8.10
Control	1.22	6.0	silt loam	0.18	1.24	1.87	1.62
Kendallville, IN							
Treated	1.63	6.4	clay loam	0.27	5.92	2.29	27.2
Control	1.63	6.6	loam	0.17	1.50	1.82	2.81
Columbus, IN							
Treated	5.69	6.6	sandy loam	0.74	21.4	60.4	59.1
Control	1.63	6.4	clay loam	0.19	2.58	1.73	1.94

120

TABLE 45. CONCENTRATIONS OF DTPA-EXTRACTABLE METALS
IN COMPOSITE SURFACE SOILS FOR ALL SITES \*

Element	Plot	Range	Median	Mean			
Cd	Treated	0.06 - 7.70	0.38	1.39			
	Control	0.06 - 1.37	0.13	0.26			
Cu	Treated	1.86 - 64.3	5,92	13.4			
	Control	0.42 - 7.35	1.38	2.02			
Ni	Treated	0.70 - 60.4	2.51	9.79			
	Control	0.33 - 1.87	1.45	1.28			
Zn	Treated	4.28 -269	27.2	50.8			
	Control	0.94 - 2.81	1.54	1.55			

<sup>\*</sup>Data Expressed on Oven Dry Weight Basis

The following tables present the plant tissue and grain analyses and should be used as references when reviewing the observations that follow:

Table 46 - Mean Metal Concentrations in Leaf Tissue
Table 47 - Mean Metal Concentrations in Harvested Grains
Table 48 - Total Metal Concentrations in Plants

The Cd concentrations in forage crops grown in soils from the treated plots at Macon, Georgia, Hopkinsville, Kentucky, and Columbus and Kendallville, Indiana were significantly greater than similar crops grown on the soils of the control sites. Cadmium concentrations in ryegrass grown on the control plot at Las Virgenes, California were statistically significantly greater than those grown on the treated plot, probably due to the unusually high natural concentration of Cd in this soil. Fescue grown on soil from treated and control plots at Wilmington, Ohio and Springfield, Missouri did not contain statistically significant Cd concentration differences.

Concentrations of Cd in grains grown on soils from the treated plots at Chippewa Falls, Wisconsin (soybeans) and Frankfort, Indiana (wheat) were statistically significantly greater than the same grains grown on the control sites. The concentration of Cd in soil from the treated plot at Frankfort, Indiana is unusually high and is the cause of the elevated concentration of Cd in wheat grain grown at this site. grain levels are worthy of concern. Wheat is most common in the diet of humans in the U.S., with average daily consumption of approximately 100 gram per day (USDA 1959). Assuming that the Cd concentration is unchanged by the wheat milling process, daily Cd intake from this source would be 126 µg/day, which is in excess of the maximum daily intake of 57-72 µg Cd recommended by the World Health Organization (1972). Although there were significantly higher concentrations of Cd in plants grown on the treated plots, only in the case of wheat at Frankfort, Indiana concentrations of Cd above the normal range reported for similar plants grown on untreated soils.

Cu concentrations in plant material grown on treated and control plots were in the normal range and well below levels considered indicative of phytotoxicity. Concentrations of Cu were statistically significantly greater (treated vs control) in the cheatgrass (Macon, Georgia), soybeans and soybean petioles (Chippewa Falls, Wisconsin), immature wheat and wheat grains (Frankfort, Indiana), and corn leaves (Columbus, Indiana).

Generally, Ni concentrations of plant materials from both the treated and control plots were in the range normally found in plant materials. The Ni concentration in cheatgrass from the Macon, Georgia site may seem unusually high compared to plant

TABLE 46. MEAN METAL CONCENTRATIONS IN LEAF TISSUE\*

Site	Plant	Cd	Cu μg/	Ni	Zn
					<del></del>
Macon, GA	Cheatgrass† Oats	0.71 <sup>#</sup> 0.31	22.0 <sup>#</sup> 5.61	17.4 <sup>#</sup> 3.63	383 <sup>#</sup> 31.2
Las Virgenes, CA	Ryegrass	0.67 1.41 <sup>#</sup>	11.4 10.5	9.01 <sup>#</sup> 5.08	58.5 61.1
Wilmington, OH	Alfalfa	0.88 0.85	8.24 9.22 <sup>#</sup>	7.37 8.27	40.5 <sup>#</sup> 32.4
Springfield, MO	Fescue	0.34 0.43	4.63 5.25	4.38 <sup>#</sup> 3.51	31.3# 24.7#
Chippewa Falls, WI	Soybeans	1.38 <sup>#</sup> 0.83	13.3 <sup>#</sup> 10.5	13.3 10.6	109 # 57.3
Hopkinsville, KY	Fescue	0.27 <sup>#</sup> 0.18	7.29 <sup>#</sup> 4.39	4.28 3.71	37.6 <sup>#</sup> 20.5
Frankfort, IN	Wheat	0.92 <sup>#</sup> 0.28	6.75 <sup>#</sup> 5.90	3.38 4.15	29.8 27.9
Kendallville, IN	Alfalfa	0.74 <sup>#</sup> 0.60	9.31 8.46	7.02 6.70	83.0 <sup>#</sup> 36.5
Columbus, IN	Corn	2.31 <sup>#</sup> 1.43	12.3 <sup>#</sup> 10.9	9.22 <sup>#</sup> 5.78	59.0 <sup>#</sup> 40.0

 $<sup>^{*}\</sup>text{HNO}_{3}\text{-HC1O}_{4}$  digestion; oven-dry weight basis.

<sup>†</sup> For each metal, the first value is from the treated plot; the second, from the control plot.

 $<sup>^{\#}\</sup>mathrm{Indicates}$  a significant difference between the treated and the control with 90 percent confidence level.

TABLE 47. MEAN METAL CONCENTRATIONS IN HARVESTED GRAINS\*

Site	Plant	Cd	Cu	Ni	Zn
			μg/	g	
Chippewa Falls, WI	Soybeans†	0.13 <sup>#</sup> 0.07	19.2 <sup>#</sup> 11.1	4.06 <sup>#</sup> 1.82	96.8 <sup>#</sup> 69.6
Frankfort, IN	Wheat	1.26 <sup>#</sup> 0.20	4.65 6.17 <sup>#</sup>	1.24 1.98#	45.1 57.5#
Columbus, IN	Corn	0.43	2.36	4.93 	29.2

 $<sup>^*</sup>HNO_3-HClO_4$  digestion; data on oven-dry basis.

<sup>+</sup>For each metal, the first value is from the treated plot; the second, from the control plot.

 $<sup>^{\#}\</sup>mathrm{Indicates}$  a significant difference between the treated and the control with 90 percent confidence level.

TABLE 48. TOTAL METAL CONCENTRATIONS IN PLANTS\*

			S	ITE		
		Macon, Georgia	Las Virgenes, California	Springfield, Missouri	Hopkinsville, Kentucky	Wilmington, Ohio
		Mean Std. Deviation	Mean Std. Deviation	Mean Std. Deviation	Mean Std. Deviation	Mean Std. Deviatio
E16	ement/Plot			μg/g		
Cd	Treated	0.71 0.11	0.67 0.19	0.34 0.05	0.27 0.08	0.88 0.09
104	Control	0.31 0.07	1.41 0.35	0.43 0.34	0.18 0.07	0.85 0.21
Cu	Treated	22.0 5.16	11.4 2.08	<b>4.62</b> 0.28	7.29 2.87	8.28 0.60
	Control	5.61 0.64	10.5 0.49	5.25 0.20	4.39 0.54	9.22 0.67
Ni	Treated	17.4 2.46	9.01 1.16	4.38 0.51	4.28 1.18	7.37 0.92
	Control	3.63 0.24	5.08 0.84	3.51 0.20	3.71 0.69	8.27 0.59
Zn	Treated	383 52.0	58.5 11.9	31.3 2.52	37.6 11.6	40.5 2.56
	Control	31.2 1.95.	61.1 9.03	24.7 0.66	20.5 2.28	32.4 2.91

 $<sup>^*\</sup>mathrm{HNO_{3} ext{-}HC10_{4}}$  Digestion; all data expressed on oven dry weight basis.

TABLE 48 (continued)

			SITE		
		Frankfort, Indiana	Kendallville, Indiana	Chippewa Falls, Wisconsin	Columbus, Indiana
		Mean Std. Deviation	Mean Std. Deviation	Mean Std. Deviation	Mean Std. Deviation
E16	ement/Plot			g/g	
Cd	Treated	0.92 0.28	0.74 0.09	1.38 0.57	2.31 0.59
<b>1</b> 1	Control	0.28 0.08	0.60 0.03	0.83 0.22	1.43 0.37
Cu	Treated	6.75 0.27	9.31 0.95	13.3 1.59	12.3 0.78
	Control	5.90 0.29	8.46 0.75	10.5 0.65	10.9 0.88
Ni	Treated	3.38 0.68	7.02 0.49	13.3 3.04	9.22 1.23
	Control	4.15 0.70	6.70 0.26	10.6 2.80	5.78 0.49
Zn	Treated	29.8 1.49	83 35.5	109 26.4	59.0 8.32
	Control	27.9 3.05.	36.5 3.15	57.3 5.72 <sup>,</sup>	40 4.66

materials from other sites. However, the soil at the Macon site is quite acid, and Ni in plant materials grown on acid soils is commonly considerably greater than in plants grown on near neutral, neutral, and calcareous soils.

Except for the Macon, Georgia, site concentrations of Zn in the plant materials are in the range considered normal. At Macon, Georgia, the concentration of Zn in cheatgrass from the treated plot is 383  $\mu g/g$ . This is considered to be in or approaching the phytotoxic ranges for a number of plant species.

# Chlorinated Hydrocarbons

Tables 49, 50, and 51 present a summary of chlorinated hydrocarbons in stabilized sludges, soils, and plants, respectively Levels in the sludges generally correspond in magnitude to published values (Furr, et al. 1976). A summary of the findings is as follows:

#### PCB's--

Amounts of Aroclor 1248 were detected in the sludges at all sites except Columbus, Indiana, with the highest concentration at Springfield, Missouri (5087 and 2846 ppb). Aroclor 1221 was detected only in the sludges from Macon, Georgia (5872 ppb) and Hopkinsville, Kentucky (260 ppb).

Significant quantities of Aroclor 1248 were detected in treated surface soils at Wilmington, Ohio: Springfield, Missouri; and Columbus, Indiana. The concentrations were 99, 120, and 216 ppb, respectively. In each case, the PCB concentration of the control plot was at or below the detection level (0.01 ppb).

All plant data with the exception of fescue grass at Spring-field, Missouria and soybeans at Chippewa Falls, Wisconsin, show insignificant PCB levels. The concentration in these two crops, however, was very low - 0.27 and 0.8 ppb, respectively. The reason for the control PCB concentrations being higher than the treated plot levels at Springfield, Missouri is not clear.

#### DDT--

Five of the sludges (Macon, Georgia; Springfield, Missouri; Hopkinsville, Kentucky; and Frankfort, Indiana) contained quantifiable amounts of DDT.

The DDT levels in the treated surface soils at these sites indicate increased amounts at Macon, Georgia and Frankfort, Indiana.

Only the treated plot ryegrass at Las Virgenes, California showed a measurable quantity of DDT (0.28 ppb).

TABLE 49. CHLORINATED HYDROCARBON CONCENTRATIONS IN STABILIZED SLUDGES

,				PCB
Site	DDT	Dieldrin	Aroclor 1221	Aroclor 1248
			· · ppb · · · ·	
Macon				
4/5/76	5.85	<0.25	0.01*	42.4
10/20/76	<0.24	<0.04	5872	< 0.01
Las Virgenes				
4/15/76	<0.25	< 0.3	0.1	3302
8/4/76	< 0.06	<0.02	0.1	< 6
Field Dried	< 0.07	2.70	0.07	1626
Wilmington				
7/2/76	< 0.31	< 0.63	<b>≪</b> 0.1	1566
8/25/76	< 0.19	<1.35	< 0.1	4731
Springfield				
5/17/76	1.54	114	< 0.01	2846
9/7/76	<0.26	<0.09	<0.01	5087
Chippewa Falls				
8/3/76	<0.06	< 0.03	< 0.01	<1.9
11/6/76	< 0.05	0.4	< 0.01	335

<sup>\*</sup>Detection limits vary due to sample size, plant matter interference, and instrument sensitivity.

TABLE 49 (continued)

			.р	СВ
Site	DDT	Dieldrin	Aroclor 1221 ppb	Aroclor 1248
Hopkinsville 5/20/76 8/25/76	9.38 160-p,p <sup>1</sup> ; 0.4-o,p	14.9 <0.08*	260 < 0.08	1469 5837
Frankfort 5/24/76 8/24/76	6.6 <0.2	6.6 <1.35	< 1.0 1.0	1382 <13.5
Kendallville 5/27/76 8/26/76	<1.56 <1.90	5.78 <5.17	< 1.0 < 1.0	3672 449
Columbus 7/13/76 11/29/76	<0.4 0.9	<0.04 <0.18	< 0.4 < 0.1	< 15 < 27

<sup>\*</sup>Detection limits vary due to sample size, plant matter interference, and instrument sensitivity.

TABLE 50. CHLORINATED HYDROCARBON CONCENTRATIONS IN SURFACE SOIL COMPOSITES

			СВ	
Site/Plot	DDT · · ·	Dieldrin	Aroclor 1221 ppb	Aroclor 1248
Macon				0.0
Treated	0.11	0.56	1.6	0.2
Control	0.05	0.04	<0.02 *	<0.04
Las Virgenes				
Treated	0.03	0.20	0.09	<0.01
Control	0.15	<0.03	0.01	0.28
Wilmington				
Treated	< 0.01	0.84	<0.5	99
Control	<0.01	0.01	<0.5	<0.4
Springfield				
Treated	<0.01	<0.02	1	120
Control	0.12	<0.02	0.02	<1
Chippewa Falls				
Treated	<0.001	<0.001	10.5	0.001
Control	<0.001	<0.001	< 0.06	<0.06

\*Detection limits vary due to sample size, plant matter interference, and instrument sensitivity.

TABLE 50 (continued)

			СВ	
Site/Plot	DDT • • •	Dieldrin	Aroclor 1221 ppb	Aroclor 1248
Hopkinsville Treated Control	<0.007 * <0.007	0.47 0.007	<0.75 <0.75	<0.5 <0.5
Frankfort Treated Control	0.5 <0.007	0.004 <0.007	<0.75 <0.75	<0.5 <0.5
Kendallville Treated Control	0.29 <0.01	0.08 <0.02	300 <1	<1 <1
Columbus Treated Control	0.13 <0.01	<0.01 <0.01	<0.5 <0.5	216 <0.4

<sup>\*</sup>Detection limits vary due to sample size, plant matter interference, and instrument sensitivity.

TABLE 51. CHLORINATED HYDROCARBON CONCENTRATIONS
IN COMPOSITE PLANT SAMPLES

Site/Plot	DÐT	Dieldrin	Aroclor 1221	PCB Aroclor 1248		
Macon			¢.			
Treated	<0.07*	<0.03	<0.01	<0.01		
Control	<0.08	<0.03	<0.01	<0.01		
Las Virgenes						
Treated	0.28	0.8	<0.01	<0.01		
Control	<0.01	<0.01	<0.5	<0.4		
Wilmington						
Treated	<0.02	<0.04	<1	<1		
Control	<0.02	<0.04	<0.1	<0.11		
Springfield						
Treated	<0.1	<0.02	0.16	0.11		
Control	<0.2	<0.02	0.33	0.22		
Chippewa Falls						
Treated	<0.007	<0.007	<0.75	<0.5		
Control	<0.007	<0.007	<0.75	<0.5		
Chippewa Falls Soy Beans						
Treated	<0.001	<0.003	< 0 , 4	0.4		
Control	<0.001	< 0.003	< 0.06	<0.06		

\*Detection limits vary due to sample size, plant matter interference, and instrument sensitivity.

TABLE 51 (continued)

Site/Plot			PCB	
	DDT	Dieldrin	Aroclor 1221 ppb	Aroclor 1248
	• • •			
Hopkinsville	<0.01*	20	<1.17	<0.11
Treated Control	<0.01	<0.02	<0.83	<0.22
Control	<b>\0.01</b>	\0.0Z	(0.03	
Frankfort				_
_ Treated	<0.125	3+	<0.17	<0.11
Control	<0.25	3+	<0.17	<0.11
Frankfort Wheat Grains				
Treated	< 0.01	<0.02	<0.01	<0.01
Control	< 0.01	<0.02	<0.01	<0.01
Kendallville				
Treated	<0.02	< 0.04	<1	<0.11
Control	< 0.02	<0.04	<1	<0.11
Columbus				
Treated	< 0.02	<0.01	<0.25	< 0.75
Control	< 0.02	<0.01	< 0.75	<0.5
Columbus				
Corn Grains				
Treated	< 0.001	<0.001	<0.01	<0.01
Control	< 0.004	< 0.002	<0.01	<0.01

\*Detection limits vary due to sample size, plant matter interference, and instrument sensitivity. †Plant matter interference.

Dieldrin--

Six of the sludges (Las Virgenes, California; Springfield, Missouri; Chippewa Falls, Wisconsin; Hopkinsville, Kentucky; Frankfort, Indiana; and Kendallville, Indiana) show measurable amounts of Dieldrin. The highest recorded level was 114 ppb at Springfield. Of these, the treated surface soils at Las Virgenes, Hopkinsville, Macon, Wilmington, and Kendallville showed increased levels of Dieldrin with respect to their individual control plots.

# Microbiology

Sludge--

The sludges in general displayed microbiological identities for each process step (raw sludge to soil) similar to published data (Kenner, 1972). The failure to detect parasitic helminth ova in some of the samples analyzed would appear to be an atypical result. The random sampling, the small number of samples, and the tendency for the helminth ova to sediment-out during the anaerobic digester residence time, all contribute to the absence of helminths in a limited sampling program. Several investigators have reported helminth densities of 60 to 80 ova/ $\ell$  of sewage (Aiba et al., 1965, Liebman, 1965); human contribution is reported to be 10 percent with the remainder of animal origin.

Salmonella sp. or Shigella sp. were not detected at any point in the sewage treatment plant process except in the raw sludge at Springfield, Missouri, suggesting that these pathogens, generally low in numbers in raw sludge, do not survive the environment outside the human envelope, unless conditions are especially favorable. Viability tests for the various ova detected were all inconclusive due to the low density of the eggs for which incubation and embryonation could be attempted.

Soil--

The only parasite detected in soil samples was from Macon, Georgia, at the first depth below the surface. The presence of parasites in only four of the nine sludges is atypical, as mentioned above. The presence of these organisms in the sludge gives statistical weight to their probably being present in the soils given a larger sampling base for detection.

Salmonella sp. or Shigella sp. were found in soil samples (at Hopkinsville, Kentucky), a fact generally reported for sludge disposal projects with similar operations and practices for which microbiological monitoring data are published (Storm, et al., 1976, Storm, et al., 1977).

The approximate one-log reduction in fecal streptococci organisms per 20 cm of soil depth increase at Las Virgenes, California and Columbus, Indiana is to be expected, as physical entrapment of organisms in the upper zones of the soil matrix

occurs. Other biotic and abiotic factors which favor a decrease in numbers of organisms with depth are anaerobiosis for non-facultative species, competition with the resident soil flora, and enzymatic destruction of cells during further decomposition of the sludge-soil mixture.

The soil data from Wilmington, Ohio indicate a FC and FS profile closely resembling those in similar studies, wherein FC/FS ratios for feces excreted by man and animals differ significantly; for man the FC/FS ratio is 4.4:1 while for cattle it is 0.2:1 (Mara, 1974). Additionally, the history of the site shows a continual monthly application of sludge. The apparent inactivation of these organisms at 99.99 percent (nominal 30-day inactivation period) compares to published data for like methods of sludge disposal (Storm et al., 1976, Storm et al., 1977). These data suggest that the FS group of organisms find conditions for survival in soil more favorable than do the FC group.

Three of the sites (Las Virgenes, California; Springfield, Missouri; and Hopkinsville, Kentucky) have histories of grazing animals; the FC/FS data from Las Virgenes and Hopkinsville suggest that animal activity, and not the sludge, may have contaminated the soil.

#### Plants--

Neither parasites nor  $\frac{Salmonella}{plant}$  sp. or  $\frac{Shigella}{plant}$  sp. or  $\frac{Sh$ 

Alfalfa was the crop sampled at both Wilmington, Ohio and Kendallville, Indiana. Both crops had been planted in previous years and consequently had been present when sludge was spread. Their microbiological characters were remarkably similar in fecal organism count.

Perennial fescue was sampled from the Springfield, Missouri and Hopkinsville, Kentucky sites. The microbiological data of each also indicate a close similarity, probably reflecting a combination of direct contact with sludge, aeolian transport of dust particles, and the incidental microflora from pasture animals

No special significance can be attributed to either the total aerobic or FS counts on the soybean leaves, wheat stalks, or corn leaves from Chippewa Falls, Wisconsin; Frankfort and Columbus, Indiana, respectively. The FS counts can probably be attributed to aeolian transport of dust-containing organisms of animal origin.

#### CHAPTER IX

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### APPENDIX

### LABORATORY ANALYTICAL PROCEDURES

### **PROCEDURES**

### Moisture

Moisture was determined by placing the sample in an evaporation dish and drying at  $105^{\circ}$ C (Standard Methods, 13th Edition, Section 148A, p. 288).

## pН

All pH measurements were performed using an Orion Model 701 pH meter with glass electrode in combination with a saturated reference calomel electrode. The pH meter was standardized periodically under conditions of temperature and concentration as close as possible to those of the sample, using standard pH buffer solutions at pH 4, 7, and 10. Soils were run using 0.01m CaCl<sub>2</sub> at a soil/CaCl<sub>2</sub> ratio of 1/2.

## Nitrate Nitrogen

Nitrate nitrogen was determined by the brucine sulfate procedure (Standard Methods, Section 213C, p. 461).

### Ammonia Nitrogen

Ammonia nitrogen was analyzed by distilling procedure (Standard Methods, Section 216, p. 469).

### Volatile Solids

Volatile solids were determined by igniting a prepared filter disk of non-filtrable residue for 60 minutes at  $550^{\circ}$ C (Standard Methods, 224G, p. 46).

### Chloride

Chlorides were determined via the mercuric nitrate procedure (Standard Methods, Section 112B, p. 97).

### Sulfate

Sulfates were determined by the Turbidimetric Method (Standard Methods, Section 156C, p. 334).

## Sample Preparation

Soil-Total Extraction --

2.5 g finely ground (<1 mm particle size) air-dried samples were digested in 1.5 ml concentrated HNO $_3$  followed by 4 ml of 2:1 HNO $_3$ :HClO $_4$ . Samples were filtered and diluted to 25 ml.

DTPA Extraction --

10 g of soil were extracted with 20 ml (0.005 M DTPA, 0.01 M CaCl<sub>2</sub>, and 0.1 M Triethanolamine solution at pH 7.30), shaken for two hours, filtered through Whatman No. 42 filter paper, and Cd, Cu, Ni, and Zn determined by AA.

Sludge--

2.0 g of oven-dried (60°C) sludge solids were digested in 1.5 ml concentrated HNO3, followed by 4 ml of 2:1 HNO3:HClO4. Subsequent additions of the nitric-perchloric mixture were made until the digest became clear.

### Plants--

Plant tissues were placed in cheesecloth, bundled, and dipped repeatedly into a cold, dilute solution of Ivory soap flakes. The bundles were thoroughly rinsed with tap water, followed by several successive washes of ultra-pure water (18 megohm). The bundles were then dried in a forced-air oven at 60°C. The cheesecloth was removed and discarded. The dried tissue was finely ground in a Waring blender mill, placed into clean plastic vials, and stored under refrigeration.

The digestion procedure was essentially the same as that used for soils (Total), but with a smaller sample size of 2.0 g.

# Ni, Cd, Cu, Zn, Pb, Ag, Mg, Co, Mn, Mo, Fe, Ca, Cr

Elements were determined by atomic absorption techniques utilizing the following equipment:

- A. Jarrell-Ash Dialatom Atomic Absorption Spectrophotometer
- B. Perkin-Elmer Model 240 Atomic Absorption Spectrophotometer

Individual analyses were performed according to the specified operating conditions provided in the equipment manufacturer's analytical methods manual\*, and an EPA manual\*. Non-absorbing line techniques were utilized for Cd analysis to overcome background interferences.

#U.S. Environmental Protection Agency, 1974.

<sup>\*</sup>Perkin-Elmer Corp., 1971 †Jarrell-Ash Division, 1974.

## As, Se

Both As and Se were determined by chemical reaction to form the hydrides and subsequent AA analysis following standard procedures as referenced above.

# Hg

Mercury was determined by flameless AA (EPA, 1974, p. 118-126).

## K, Na

Potassium and sodium were determined by flame photometry as outlined in Section 147A, pp. 283-284 in Standard Methods (13th Edition).

## В

Boron was determined by the carmine spectrophotometric method as shown in Section 107B, p. 72, Standard Methods.

# P (Total)

Phosphorus was determined by the Ascorbic Acid Method as shown in Section 223F, pp. 532-534 of Standard Methods.

# Dieldrin, DDT (and analogs), PCB's

Techniques used included extraction with organic solvents (acetonitrile, hexane), solvent phase partitioning and gas chromatography with electron capture detector (detection and confirmation). These tests were followed by dehydrochlorination and electron capture GC for PCB's.

# Organic Carbon

Organic carbon was determined by the Walkley-Black Method as shown on pp. 1372-1376 in Methods of Soil Analysis, Part 2, American Society of Agronomy, 1965.

## <u>Texture</u>

Texture of soils was determined by the hydrometer method as shown on pp. 562-564 in Methods of Soil Analysis, Part 1, American Society of Agronomy, 1965.

# Cation Exchange Capacity

Cation exchange capacity was determined by extraction with sodium acetate, methanol, and ammonium acetate, followed by determination (sodium electrode) for sodium, as per Methods of Soil Analysis, Part 2, p. 899.

# Bacteriology

Each 100 g sample of sludge, soil, and plant was thoroughly mixed with 1  $\ell$  of distilled, sterile water (plant samples were homogenized). The mixture was strained through a U.S. No. 6 sieve into a 1-gal disposable reinforced paper container. Solids retained on the sieve were discarded. The remainder was poured through a No. 5 standard sieve using a sterile wooden tongue depressor to "screed" the material through the sieve. Solids retained on the sieve were discarded. The remainder was aspirated with a "Millipore" or similar suction unit through 6-to 14-u filters using approximately 133 + ml (1/6 of total) of the mixture for each aspiration. The remainder was then aspirated through 0.45u filters to collect organisms.

The 3-level test for <u>salmonella sp.</u>, fecal coliform and fecal streptococci was performed on all sludge soil and plant samples in accordance with Standard Methods for the Examination of Water and Wastewater (14th Edition) and the Laboratory Standards for Bacteriological Analysis prepared by the American Society of Microbiology. Identification was made of Salmonella sp. isolates. Quantitive analyses by the Most Probable Numbers (MPN) technique were made for total mesophilic aerobic forms, salmonella, shigella, fecal streptococci, and fecal coliform, and reported as number of organisms per gram.

## <u>Helminths/Protozoans</u>

Six 15-test tube samples for each sample were prepared using standard sedimentation and flotation techniques (saturated zinc sulfate and 10 percent buffered formalin, respectively). Slides were prepared from each aliquot sample and examined under a compound microscope. Quantitation was accomplished using the MacMaster's slide technique. Viability tests for embryonated ova were made by hamster inoculation and autopsy 30 days after inoculation (MacMasters's slide technique).

### Statistical Methods

The usual calculations for sample mean, sample standard deviation, and two-tailed student-T testing of significant difference between two sample means were performed. The probabilities of incorrectly accepting (Type I error) or of incorrectly reporting (Type II error) the null hypothesis - that the two sample means (and hence, the samples themselves) came from the same population - were kept within the reasonable limitations of the sampling techniques by using a 90 percent degree of confidence.