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Assessment of the Application of an Ecotoxicological Procedure to Screen Illicit Toxic Discharges in Domestic Septic Tank Sludge

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ABSTRACT

An innovative screening procedure has been developed to detect illicit toxic discharges in domestic septic tank sludge hauled to the Montreal Urban Community wastewater treatment plant. This new means of control is based on an integrative approach, using bioassays and chemical analyses. Conservative criteria are applied to detect abnormal toxicity with great reliability while avoiding false positive results. The complementary data obtained from toxicity tests and chemical analyses support the use of this efficient and easy-to-apply procedure.

This study assesses the control procedure in which 231 samples were analyzed over a 30-month period. Data clearly demonstrate the deterrent power of an efficient control procedure combined with a public awareness campaign among the carriers. In the first 15 months of application, between January 1996 and March 1997, approximately 30% of the 123 samples analyzed showed abnormal toxicity. Between April 1997 and June 1998, that is, after a public hearing presentation of this procedure, this proportion dropped significantly to approximately 9% based on 108 analyzed samples.

The results of a 30-month application of this new control procedure show the superior efficiency of the ecotoxicological approach compared with the previously used chemical control procedure. To be able to apply it effectively and, if necessary, to apply the appropriate coercive measures, ecotoxicological criteria should be included in regulatory guidelines.

IMPLICATIONS

An integrative control procedure would help wastewater treatment plant (WTP) managers to detect illicit toxic waste that could be shipped to their facilities and that could pollute the environment and/or interfere with the treatment process. This powerful cost-effective procedure requires minimal investment and could be easily established in a typical WTP control laboratory and carried out by staff of average skill. The response is fast and reliable and allows a quick intervention.

Montreal Urban Community existing regulations will need to be modified to include not only chemical but also ecotoxicological criteria. This control procedure has to be applied on both local and regional scales; otherwise, carriers may tend to ship illicit sludge to peripheral areas where control is less rigorous.

INTRODUCTION

Since 1989, the Montreal Urban Community (MUC) wastewater treatment plant has handled annually an average of 20,000 m^3 of sludge collected from domestic septic installations located within the MUC territory, as well as in neighboring municipalities (Figure 1). Carriers must obtain permits before discharging sludge and comply with the conditions of the permits, especially those forbidding the discharge of toxic substances. The sludge is mixed with the wastewater inflow before the primary chemical treatment process. This treatment removes about 80% of suspended solids and total phosphorus, 60% of the BOD_5, and 50% of the heavy metals.

One of the major concerns of the MUC authorities is for appropriate measures to be taken to ensure that no toxic waste is mixed with domestic sludge.[1] Such a practice would be attractive to hazardous waste producers because of the high cost of treatment and/or elimination of toxic waste. In the past, the control procedure was based on identifying abnormally high levels of contaminants by comparing the results of 12 chemical parameters with the data obtained through a characterization program that involved 125 samples analyzed over a two-year period (Table 1). If the chemical analysis results deviated significantly

Table 1. Physical and chemical characterization of septic tank sludge (1989–1990).

Parameter	Descriptive Statistics (n = 125)		
	Median	Minimum	Maximum
pH	6.5	5.0	8.4
Total solids (g/100 g)	1.26	0.06	26
Total volatile solids (g/100 g dry weight)	66	6.7	92
Total phosphorus (mg/kg)	82	0.57	525
BOD_5 (mg/kg)	4800	11	80000
Total oil and grease[a] (mg/kg)	1255	<3	15000
Hydrocarbons[a] (mg/kg)	268	<3	4170
Phenols (mg/kg)	0.60	<0.01	11
Total cadmium (mg/kg)	0.04	<0.02	0.36
Total chromium (mg/kg)	0.4	<0.1	3.8
Total copper (mg/kg)	4.1	0.17	49
Total nickel (mg/kg)	0.3	<0.2	8.0
Total lead (mg/kg)	1.1	<0.2	27
Total zinc (mg/kg)	9.4	0.12	84

[a]Not analyzed on a regular basis during the period of the study.

from the historical expected value, then administrative action was initiated. However, this approach seemed limited because it could not ascertain, with rapidity and confidence, the acceptability of sludge shipments that could possibly contain thousands of toxic compounds. For this reason, the MUC developed an integrative procedure to screen illicit discharges in domestic septic tank sludge by the use of quantitative toxicity tests. Toxicity thresholds were established during the developmental phases of this procedure to define the normal toxicity of sludge. High efficiency was attained (detection of 93% of added toxic substances to non-contaminated sludge) while avoiding false positives, that is, falsely accusing a carrier of illicit toxic waste discharging.[2]

Since the beginning of 1996, the MUC Environmental Department has applied this new control procedure while participating in an interlaboratory study with the Quebec Ministry of Environment to validate the threshold values and to test the efficiency of the procedure to detect other toxic substances. Meanwhile, information describing this procedure and its implications was diffused among the carriers through a public hearing session of the MUC Environment Commission held in April 1997.[3]

The purpose of this study is to assess the efficiency of this new ecotoxicological control procedure after a 30-month period of application.

MATERIALS AND METHODS

Sludge Reception and Sampling

Septic sludge was hauled to the MUC wastewater treatment plant in tanker trucks with an average capacity of about 12 m^3. It was mixed with the incoming wastewater that flows at an average of about 30 m^3/sec. Samples were collected by the carrier directly from the dumping valve connected to the truck. A representative sample of the sludge from the truck was obtained by making a composite of samples collected at the beginning, middle, and near the end of the discharge process. These portions were well mixed and transferred to a 1-L high-density polyethylene bottle before being stored at 4 °C. Sampling was carried out under the supervision of the treatment plant's operator. The pH was measured on-site, and if the pH was outside the normal range or if there was evidence of abnormal physical characteristics (i.e., oily film, color, and odor), the sample was then shipped to the laboratory for further analysis. Otherwise, samples were chosen randomly for analysis according to an established frequency (e.g., one out of 10 shipments).

Description of the Ecotoxicological Procedure

The control procedure involved three standardized bioassays. The first, the lettuce root elongation test, involved 5 days exposure of lettuce seeds (Lactuca sativa) to the sludge sample. Toxicity was indicated by an inhibition of root growth.[4] The second, the inhibition of respiration test (after a 3-hr aeration period), was carried out after the addition of activated sludge to the sample. The presence of toxicants was reflected by an inhibition of the oxygen consumption rate.[5] The third bioassay was based on the inhibition of bacterial bioluminescence (Vibrio fischeri) using the Microtox system, in which the microorganisms were exposed to a contaminated sample.[6] For this test, salinity was adjusted to 2% with reagent-grade sodium chloride. The results obtained by these bioassays were compared with

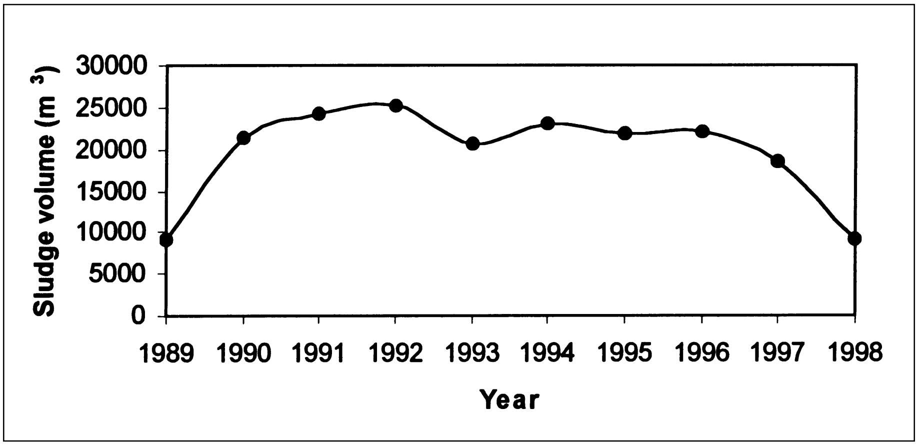

Figure 1. Sludge volume received at wastewater treatment plant.

the threshold values (Table 2) established in earlier studies.[1,2] Data outside these values indicated anomalous toxicity. The endpoint used to define the threshold for the root elongation test was expressed as the median inhibition concentration (IC_{50}; i.e., the concentration of sludge estimated to cause 50% inhibition of measured biological response of the tested organism). The oxygen consumption rate was used as the endpoint for the respiration test. In the case of the Microtox test, the large variability of toxicity among the samples diminished the test's sensitivity to detect added contaminants.[2] The toxicity of inorganic substances measured by the Microtox test tends to increase with exposure time.[7-9] A previous study[2] demonstrated that, by expressing the results as the ratio between the IC_{50}s after 5 and 30 min of incubation, the Microtox test became sensitive to some inorganic toxic substances. As a result, tested organic compounds could not be detected by the Microtox test but could still be detected by the other two bioassays.

In the event that anomalous toxicity was detected by one or more of these bioassays, chemical analyses were then carried out according to standardized methods[10] in order to identify the contaminants by comparing the results with the historical data (Table 1). Other parameters could also be analyzed if other substances (e.g., oil and grease) were suspected. Results deviating considerably from median values were identified as chemical anomalies.

Quality Control

Details of quality control procedures were described in a previous article.[2] Briefly, all series of chemical analyses included a sample blank (deionized water, ASTM type II). Within a series of chemical analyses, at least one sample was analyzed in duplicate. A sample was also spiked with a standard to verify method recovery. Certified aqueous and dried sludge standards were used to validate the chemical analyses.

Negative controls (no sample or toxic substances added) were included. For these assays, American Chemical Society reagent-grade substances were also used as reference toxicants (positive controls), and the IC_{50} values of each were determined. For this purpose, phenol for the Microtox test, 3,5-dichlorophenol for the respiration test, and mercuric chloride for the root elongation test were used. The validation of toxicological analyses was assured, since all these quality control data were considered acceptable according to control charts[11] and other established criteria (e.g., response in the negative control). For the results to be valid, the IC_{50} of phenol had to be within the range of 17.1 ± 2.2 mg/L as measured by the Microtox test; the IC_{50} of 3,5-dichlorophenol had to be within the range of 22.6 ± 4.7 mg/L as measured by the inhibition of respiration test; and the IC_{50} of mercuric chloride had to be within the range of 25.2 ± 5.3 mg/L as measured by the root elongation test.

Table 2. Thresholds for bioassays.

Bioassay	Endpoint	Anomaly Zone
Root elongation	IC_{50}-5 days	<21%
Respiration inhibition	Oxygen consumption rate	<39 mg $O_2.L^{-1}.hr^{-1}$
Microtox	IC_{50}-5 min/ IC_{50}-30 min	>2.1

Data Analysis

The linear interpolation method developed by the U.S. Environmental Protection Agency[12] was used to estimate the IC_{50} values for the root elongation test. For the respiration test, the IC_{50} of the toxic reference substance was calculated using the Trimmed Spearman-Karber method.[13,14] The IC_{50} values for the Microtox test were calculated using the Microtox computer-assisted program, version 7.8.[15]

RESULTS AND DISCUSSION

During the period of this study (from January 1, 1996 to June 30, 1998), 1889 shipments were transported to the wastewater treatment plant by 16 different carriers. An analysis according to the ecotoxicological procedure was carried out on 231 sludge samples (Table 3). Of the 47 samples presenting anomalous toxicity, 24 were detected by the root elongation test only, 13 by the respiration test only, and one by the Microtox test only (Table 4). In nine cases, anomalies were confirmed by both the root elongation and the respiration tests. Results presented in Table 4 indicate that chemical analyses were able to confirm the possible source of anomalous toxicity in 11 samples, while one sample presented a dubious physical characteristic (odor of hydrocarbons).

Figure 2 shows that most of the anomalous samples were detected during the first half of the study (i.e., between January 1996 and March 1997). Anomalies dropped from approximately 30% (37 anomalies detected in 123 analyzed samples) in the first half of the study to approximately 8% (10 anomalies detected in 108 analyzed samples) in the second half (Table 3). The public hearing session of the MUC environment commission held in April 1997, to which all permit-holding carriers were invited and during which the procedure was presented, may have had a deterrent effect on less scrupulous carriers. Note that in September 1996, MUC authorities instructed carriers not to mix chemical toilet sludge with domestic sludge because the relatively high toxicity of the disinfectants used in the chemical toilets would interfere with the control procedure. This action was justified, since mixing the two distinct kinds of sludge was a common but unacceptable practice, as observed in the case of the carriers coded L and M (Table 4). The impact of that measure, however, is not tangible (Figure 2).

The performance of each individual carrier was also studied. Of the 16 permit-holding carriers, only two (carriers coded B and E) had mediocre results over the whole study period and four (carriers coded A, H, M, and O) significantly improved the quality of their shipments after the public awareness campaign (Table 3). Accordingly, sampling and analysis frequency should be established based on the historical records of each carrier.

Among the bioassays used in this study, the root elongation test proved to be the most efficient (Table 4). The results also show that the respiration test detected about 28% of the anomalous toxicity of sludge that the root elongation test failed to detect. This indicates that data from these tests are complementary and that a multi-test procedure is necessary to improve the overall efficiency. The apparent lack of efficiency of the Microtox test (detection of only 2% of the anomalous toxicity of sludge samples) can be explained by the presence of contamination of organic origin. Background toxicity of sludge, as measured by the Microtox test, was relatively high. Consequently, samples had to be diluted about 50 times, which made this test unable to detect the potentially low concentration of inorganic contamination that could be encountered in the sludge. Root elongation and respiration tests did not suffer from this disadvantage, since the background toxicity measured by these tests was relatively low, probably because of the high concentration of organic nutrients that favored the growth of the organisms used. Thus, the procedure could be simplified while maintaining high efficiency by retaining only the two most reliable bioassays (i.e., the inhibition of respiration and root elongation tests).

The main purpose of developing an integrative means of controlling the quality of sludge samples is to improve the efficiency of detecting permit violators. Results demonstrate that about 74% of the samples presenting an abnormal toxicity did not show any anomaly in their chemical composition (Table 4). If chemical analyses failed to identify the contaminants, administrative action (i.e., investigation and enhanced monitoring) was initiated. If the contaminant responsible for the abnormal toxicity could be identified, the carrier was issued a notification of the offense; if the violation was repeated, the discharge permit was suspended or revoked. Since the beginning of 1997, there was a significant decrease in the sludge volume received at the wastewater treatment plant (Figure 1). Carriers may have shipped sludge to treatment sites where sludge control was less rigorous, which also demonstrates

Table 3. Number of discharges, samples analyzed and anomalies detected for each of the carriers.

Code of the Carriers	Number of Discharges		Number of Analyzed Samples (Anomalies Detected)	
	January 1, 1996, to March 31, 1997	April 1, 1997, to June 30, 1998	January 1, 1996, to March 31, 1997	April 1, 1997, to June 30, 1998
A	71	12	13 (7)	2 (0)
B	265	311	31 (3)	46 (4)
C	9	1	3 (1)	0 (0)
D	81	41	8 (0)	4 (1)
E	50	32	3 (1)	3 (1)
F	26	5	2 (0)	0 (0)
G	14	11	3 (0)	0 (0)
H	108	26	10 (7)	4 (0)
I	9	0	0 (0)	0 (0)
J	16	21	1 (0)	4 (2)
K	37	34	1 (0)	2 (0)
L	5	7	1 (1)	0 (0)
M	222	204	33 (13)	23 (0)
N	15	38	4 (0)	3 (1)
O	79	124	9 (4)	16 (1)
P	1	14	1 (0)	1 (0)
Total	1008	881	123 (37)	108 (10)

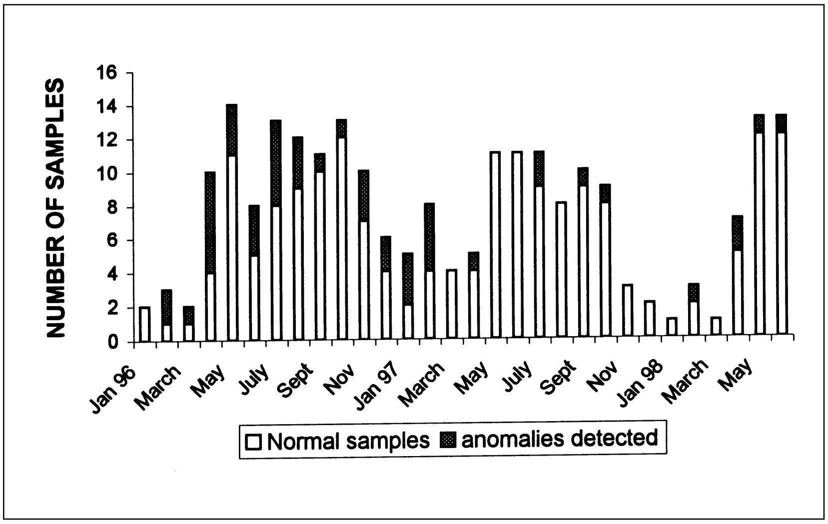

Figure 2. Number of normal samples and anomalies detected for the period between January 1, 1996, and June 30, 1998.

Table 4. Anomalies detected by bioassays and chemical analyses during the period of this study.

Date of Reception	Code of the Carriers	Bioassays[a] Detecting the Anomalies	Chemical Anomalies and Observations
96.02.15	O	Respiration - Elongation	N.I.
96.02.19	A	Elongation	Zn = 53 mg/kg
96.03.06	B	Elongation	N.I.
96.04.09	M	Elongation	N.I.
96.04.13	H	Elongation	N.I.
96.04.18	O	Respiration - Elongation	Odor of hydrocarbons
96.04.26	O	Respiration - Elongation	Cu = 31 mg/kg, Cr = 4 mg/kg
96.04.26	M	Elongation	Cu = 140 mg/kg, Zn = 300 mg/kg, Pb = 24 mg/kg
96.04.30	M	Respiration - Elongation	Presence of chemical toilet sludge[2]
96.05.07	O	Elongation	N.I.
96.05.15	H	Elongation	N.I.
96.05.15	H	Elongation	N.I.
96.06.12	H	Elongation	N.I.
96.06.18	B	Respiration	N.I.
96.06.25	M	Elongation	Presence of chemical toilet sludge[b]
96.07.04	M	Elongation	Presence of chemical toilet sludge[b]
96.07.03	A	Respiration - Elongation	pH = 12.2
96.07.11	M	Respiration	Total oil and grease = 2.00%, Hydrocarbons = 1.56%
96.07.23	M	Elongation	Presence of chemical toilet sludge[b]
96.07.23	A	Respiration	N.I.
96.08.03	M	Elongation	N.I.
96.08.06	M	Elongation	Presence of chemical toilet sludge[b]
96.08.27	M	Elongation	Presence of chemical toilet sludge[b]
96.09.27	L	Respiration - Elongation	Presence of chemical toilet sludge[b]
96.10.22	H	Respiration - Elongation	N.I.
96.11.13	A	Elongation	N.I.
96.11.18	B	Respiration	N.I.
96.11.27	E	Elongation	N.I.
96.12.04	C	Respiration	N.I.
96.12.03	H	Elongation	Cr = 6.0 mg/kg, Cu = 40 mg/kg, Ni = 2.9 mg/kg, Pb = 7 mg/kg, Zn = 66 mg/kg
97.01.07	H	Elongation	Pb = 6 mg/kg
97.01.23	M	Elongation	N.I.
97.01.27	A	Elongation	Cu = 69 mg/kg
97.02.06	A	Respiration	N.I.
97.02.10	A	Respiration	N.I.
97.02.17	M	Elongation	N.I.
97.02.19	M	Respiration - Elongation	N.I.
97.04.28	J	Respiration	N.I.
97.07.10	B	Respiration	N.I.
97.07.31	O	Elongation	Pb = 7 mg/kg, Zn= 45 mg/kg
97.09.02	J	Respiration	N.I.
97.10.14	E	Respiration	N.I.
98.02.26	B	Respiration - Elongation	Phenols = 1.5 mg/kg, Cu = 24 mg/kg, Pb = 8 mg/kg, Zn = 39 mg/kg
98.04.08	B	Microtox	N.I.
98.04.25	D	Respiration	N.I.
98.05.25	B	Elongation	N.I.
98.06.10	N	Respiration	Total oil and grease =11.9%, Hydrocarbons = 0.79%

N.I. = Not identified; [a]See Materials and Methods; [b]As reported by the carrier.

that the most appropriate solution is still a coercive approach to regulatory control. This approach is opposite of the policies promoted by different government agencies that rely more and more on toxic waste producers to be responsible and self-controlled rather than on regulatory enforcement.

CONCLUSIONS

The results of a 30-month application of the new ecotoxicological control procedure show the deterrent effect that this powerful tool can have, especially when combined with a public awareness campaign among the sludge carriers. They also show the superior efficiency of the ecotoxicological approach compared with previously used chemical control procedures.

To be able to use the new procedure with full efficiency and, if necessary, to apply the appropriate coercive measures, ecotoxicological criteria should be included in future regulatory guidelines. Moreover, application of such a procedure could be extended on a regional basis to discourage carriers from shipping sludge to peripheral areas where control is less rigorous.

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