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EVALUATING THE ACCURACY OF BIOCHEMICAL OXYGEN DEMAND AND SUSPENDED SOLIDS ANALYSES PERFORMED BY WISCONSIN LABORATORIES

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ABSTRACT

Wisconsin Pollutant Discharge Elimination System (WPDES) permits require that municipal and industrial dischargers in the state self-monitor wastewater flows for certain parameters. However, similar self-monitoring data in many states has often been of questionable accuracy.

This study assessed the accuracy of a representative number of Wisconsin laboratories analyzing effluent samples from Wisconsin wastewater treatment plants. For such an assessment their results were compared to those from an Environmental Protection Agency (EPA) approved reference laboratory, the State Laboratory of Hygiene (SLH).

As of January 1978, 8% of Wisconsin's 634 municipal wastewater treatment plants employed at least one full-time analyst (major municipal labs); 43% had the plant operator do the analyses in addition to other duties (minor municipal labs); 8% contracted with a neighboring major municipal lab; 11% used a nearby minor municipal lab. The remaining 30% had their testing done by a commercial (private) laboratory. Labs were defined as major if they tested for more than two communities; a commercial laboratory was classified as minor if it tested for two or fewer communities.

During the study wastewater treatment plant operators split their BOD and suspended solids effluent samples, having half a sample analyzed by the SLH and half by their usual laboratories. DNR field personnel explained the split sampling procedure to be used to the cooperating plant operator. Results from the regular lab analyses were recorded by the operators and sent to the study coordinator. Approximately 150 plants and operators were involved in the study.

Given the data accuracy criterion adopted (for each parameter at least 80% of a lab's data had to fall within two standard deviations above and below the SLH results), 80% of the major municipal, 72% of the minor municipal, 60% of the major commercial labs with sufficient data to study, and 100% of the minor commercial labs generated unacceptable data for BOD. Twenty percent of the major municipal, 35% of the minor municipal, 70% of the major commercial and 33% of the minor commercial labs produced unacceptable data for suspended solids. Sixteen percent of the major municipal, 30% of the minor municipal, 40% of the major commercial, and 33% of the minor commercial labs generated unacceptable data for both parameters.

Follow-up visits by DNR personnel indicated that general causes for unacceptable municipal lab data include use of malfunctioning, inadequate, outdated, or improperly maintained equipment, as well as poor analytical technique employed by analysts.

Alternatives for dealing with the problem include the establishment of at least a voluntary quality assurance program for labs, as well as mandatory certification programs for labs and individuals performing WPDES self-monitoring analyses.

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by

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BACKGROUND

How accurate are self-monitoring lab results from Wisconsin wastewater treatment plants?

The performance of municipal wastewater treatment plants is of increasing concern to federal, state, and local officials as well as to the public at large. Millions of tax dollars have been spent to build or upgrade these facilities. As these plants are placed on-stream, the question in many minds is how well are plants now performing their function of purifying wastewater?

The regular reporting of self-monitoring data on biochemical oxygen demand and suspended solids has been required of all Wisconsin municipal wastewater treatment plants since 1974. The object of this particular study has been to assess the accuracy of a representative sample of Wisconsin commercial and municipal wastewater treatment plant laboratories in analyzing wastewater flow samples for these two parameters. For such an assessment their results were compared to those from a reference lab, in this case the State Laboratory of Hygiene (SLH) in Madison.

TABLE 1. *Village of Milladore wastewater system effluent split sample results: DNR lab and Village contract lab.**

Sampling Date	Results (mg/l) DNR** BOD	SS	Contract Lab BOD	SS
27 Oct 1976	3.7	3	19	8
8 Nov 1976	4.3	1	12	12
22 Nov 1976	8.2	7	49	26
6 Dec 1976	4.9	4	21	32
20 Dec 1976	5.1	1	29	10
Average	5.2	3	26	18

*Unpublished data submitted by Robert Dirksen, District Environmental Engineer, DNR-Rhinelander.
**State Laboratory of Hygiene, Madison.

The State has the responsibility to enforce the requirements of a Wisconsin Pollutant Discharge Elimination System (WPDES) permit

In 1972, Congress passed the Federal Water Pollution Control Act Amendments (PL 92-500). The amendments established the National Pollutant Discharge Elimination System (NPDES), "a national permit program for controlling the discharge of pollutants" (Comptroller General of the United States [CGUS] 1977:2). Under PL 92-500, it became illegal to discharge pollutants into navigable waters of this country without a NPDES permit.

The State of Wisconsin accepted primary enforcement responsibility for the 1972 amendments and in 1974 began issuing the first Wisconsin Pollutant Discharge Elimination System (WPDES) permits to municipal and industrial dischargers in the state. WPDES permits require that dischargers meet specified effluent limitations and submit to the state regulatory agency, the Wisconsin Department of Natural Resources, monthly reports of self-monitoring results for certain pollutant parameters in the influent and effluent.

Self-monitoring data from other states has often been of questionable accuracy

From the start of the NPDES program the monthly self-monitoring data have often been of questionable accuracy (CGUS 1977:36). The United States Environmental Protection Agency (EPA) has documented operation and maintenance problems at municipal wastewater treatment plants throughout the country since 1970. In a 1975 study of 100 plants EPA reported that ". . . our (EPA's) analysis . . . showed that 53 of the plants had inadequate laboratory controls. Laboratory controls and/or testing procedures were also inadequate at 11 of the 28 plants which we reviewed in detail. In some cases the plants visited lacked adequate equipment needed for laboratory testing. At one plant which had the necessary laboratory equipment, the operator informed us he did not know how to perform all the required tests" (CGUS 1977:13). In 1976 a Georgia state official told federal reviewers "that less than 50 percent of the data received from municipal waste treatment plants was considered reliable" (CGUS 1977:36).

A 1976 comparison revealed considerable discrepancies in results from two laboratories analyzing split samples from a Wisconsin municipal wastewater treatment plant at Milladore (Table 1). In this prior study, the samples were collected and split by an experienced DNR field person. The "DNR lab" referred to is the State Laboratory of Hygiene (SLH) in Madison. The "contract lab" is a private commercial laboratory which performs the required analyses for a number of Wisconsin municipal wastewater treatment plants. The WPDES limits for Milladore are 20 mg/1 each for BOD and suspended solids (all BOD's referred to in this study are five-day, 20°C BOD's). Given the considerable difference between the values reported by the two laboratories, which lab is more accurate in its analyses?

Proper operation of a wastewater treatment plant is increasingly dependent on reliable data

This question of data accuracy is important for *all* wastewater treatment plants in the state: as the days of operating by intuition, smell, and whim come to an end, the proper operation of a wastewater treatment plant is increasingly dependent on reliable data; as they design new wastewater treatment plants or additions to existing plants, consulting engineers are depending more and more on the self-monitoring data now available; such data provide a basis, in part, for decision-making by the regulatory agency relative to enforcement of the limits imposed on a discharger by its WPDES permit. In cases such as Milladore, it makes a substantial difference which laboratory results are used by these individuals and groups in evaluating the plant. As the SLH is neither equipped nor funded to do the monitoring for every municipal wastewater treatment plant in the state, both commercial laboratories and laboratories at the wastewater treatment plants regularly perform the required analyses.

The accuracy problem can be stated in terms of quality assurance (QA) of the data produced by laboratories which do analyses required by the WPDES permits. As Delfino pointed out, state QA programs are currently conducted on a voluntary basis. As a result, the quality of data generated by many laboratories has been variable and subject to a variety of undetected or underestimated errors. This fact has been verified through performance audits conducted by various agencies and organizations (Delfino 1977).

The performance audits referred to by Delfino were round-robin studies in which one state sample (either simulated or actual wastewater) was sent to a number of laboratories, and their results on that same sample were then compared. A review of the literature indicated that, to date, except for the previously cited DNR study, no accuracy study has been reported using actual wastewater samples collected for self-monitoring at municipal wastewater treatment plants and analyzed by their respective laboratories.

STUDY DESIGN

Laboratories doing WPDES analyses were categorized as municipal or commercial

DNR field personnel determined which laboratory did the analyses for each Wisconsin municipal wastewater treatment plant. As of January 1978 a little over half (324) of the 634 municipal wastewater treatment plants with WPDES permits performed their own laboratory testing (internal analysis). The remaining 310 plants had their testing done by neighboring municipal wastewater laboratories or by commercial laboratories (external analysis).

All the wastewater treatment plant laboratories were categorized as municipal or commercial, major or minor. Major municipal labs employed at least one person full-time to perform the required analyses. At minor municipal labs the treatment plant operator, in addition to other duties, performed the analyses. (Note: EPA and DNR define major and minor municipal *dischargers* in terms of the quantity of flow.) Commercial laboratories are private operations that contract with municipalities to perform their analyses. A major commercial laboratory was defined as one which tested for more than two communities, while a minor commercial lab was defined as one which tested for two or fewer communities. Eleven major and 10 minor commercial labs were identified as performing WPDES analyses in this study. Table 2 displays the numbers of labs in each of the five categories.

TABLE 2. *Distribution of internal/external analyses for Wisconsin municipal wastewater treatment plants.*

	Lab Source of Analyses					
	Internal		External			
DNR Field	Municipal		Municipal		Commer-	
District	Major	Minor	Major	Minor	cial	Totals
WCD	6	25	14	5	87	137
LMD	10	82	0	22	33	147
SD	11	83	8	15	20	137
SED	17	40	20	9	14	100
NCD	2	27	5	10	19	63
NWD	2	18	1	12	17	50
TOTALS	48	275	48	73	190	634

Effluent samples of BOD and suspended solids were split for a representative number of labs

The quality assurance study decided upon, a split sampling procedure, was limited to analyses for BOD and suspended solids of effluent samples made by the laboratories in each category. The State Laboratory of Hygiene was chosen as the reference laboratory because it serves as the primary wastewater laboratory for the regulatory activities of the State of Wisconsin. It has been inspected and audited by EPA.

Three constraints shaped this program: economics, seasonal workload, and capacity of the SLH. The economic constraint meant that only 800 samples could be analyzed by the reference lab. As spring is the busy period at the SLH, the sampling needed to be essentially completed by the time of the first spring runoffs and the subsequent submission of nonpoint source pollution samples. The sample capacity of the SLH at the time of this study limited the number of BOD and suspended solids samples which could be submitted to 35 of each per day on Wednesdays, Thursdays, and Fridays. More than one sample from each laboratory was needed in order to make

statements about the accuracy of each in performing these analyses. It was decided by the author, based on the advice of statisticians on the faculty of the University of Wisconsin-Madison, to have five samples from each laboratory studied. Since only 800 samples could be analyzed, only 152 laboratories could be studied using 5 samples for each to give a total of 760 samples. The remaining 40 samples were used for a quality assurance (QA) study of the SLH.

The numbers of laboratories to be studied in each category were determined from the following conditions. Since most of the municipal wastewater discharged in Wisconsin comes from the 48 treatment plants served by the 48 major municipal laboratories, it was important that a large percentage of these laboratories be studied. (The larger the percentage, the more certain the conclusions of accuracy of data submitted.) Consequently, 25 (or 52%) of these major municipal labs were randomly selected. While the minor municipal laboratory category is by far the largest (275), in terms of flow the wastewater treatment plants served by these labs are the smaller ones in the state; 41% (113) were randomly chosen. As the few laboratories in the major commercial category (11) tested for about 175 communities, all (or 100%) of these labs were sampled. However, since most of the laboratories in the minor commercial category (10) tested for only one community, only 3 (or 33%) of these labs were randomly sampled (Table 3). Those labs selected were chosen by a computer program that randomly selected 25 out of 48 numbers, 113 out of 275 numbers, and 3 out of 10 numbers for the major municipal, minor municipal, and minor commercial categories respectively. (All of the major commercial labs were selected.) Figure 1 displays the distribution of municipal wastewater treatment plants in the state as well as the number of plants which participated in the study.

TABLE 3. *Categories of laboratories serving Wisconsin wastewater treatment plants.*

Lab Category	Total No. Labs	No. Plants Served	No. Labs Sampled
Major Municipal	48	48	25(51)*
Minor Municipal	275	--**	113(41)
Major Commercial	11	175	11(100)
Minor Commercial	10	--	3(33)

*Percent of total number of labs.
**Information not obtained.

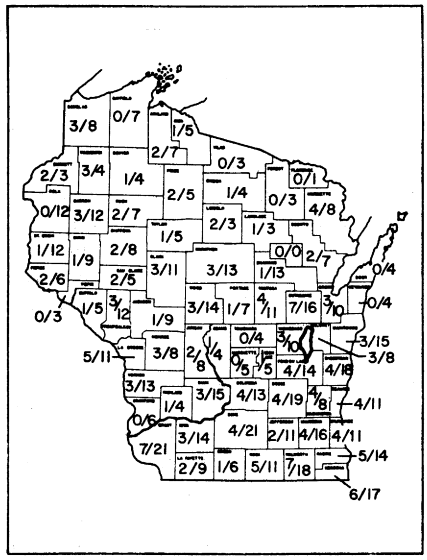

FIGURE 1. *Wisconsin municipal wastewater treatment plants*

Plant operators collected and split the samples for regular and SLH analyses

DNR field personnel visited each treatment plant selected, carefully explained the split sampling program to the operator, and requested his or her cooperation in this voluntary study. Every operator who was asked agreed to participate in the study. By having the cooperating operators split the WPDES self-monitoring samples this study differed from previous studies of data accuracy that simply sent known or check samples to the laboratories being studied.

To minimize the extra effort on the operator's part, he or she was given a styrofoam shipper for each sample containing a one-liter sample bottle, a 250-ml bottle for ice, and a lab slip identifying the sample to the SLH. The operator was also given the sampling data sheet (Fig. 2) and a postage prepaid envelope.

The split sampling data sheet specified the dates on which the operator was to follow his/her usual routine in collecting the WPDES self-monitoring samples and then split the effluent sample, sending one half in the above shipper to the SLH and having the other half analyzed in the usual way. As it was important that the sample be split properly, the DNR field person demonstrated the proper technique to the operator: two bottles were placed in the sink, the sample shaken for about two minutes and then poured rapidly back and forth between the two bottles, filling them simultaneously.

Arrangements were made with United Parcel Service (UPS) to pick up each shipper on specified dates at the wastewater treatment plant, or wherever in town the operator preferred, and to deliver each shipper on the following day in Madison. Collection dates had to be determined in advance for each treatment plant so that UPS could be notified to pick up the samples on specific days and to ensure that no more than 35 samples/day would be shipped to the SLH. Where possible a representative sampling for each major commercial lab was set up by obtaining one wastewater sample from each of five communities that regularly submitted samples to that lab. In most cases it was possible to schedule the split sampling dates so they were on different days of the week for

the five samples for each laboratory studied. The purpose was to have the study simulate as much as possible the usual pattern of self-monitoring data analyzed on different days of the week often by different analysts each assigned to perform the tests on a given day. Such scheduling was a somewhat difficult task and required some disturbance, however minimal, in the operator's routine.

At the conclusion of the split sampling program for a given wastewater treatment plant, the operator filled in the required data, including the BOD and suspended solids results obtained by his or her laboratory for the samples split. The data were then mailed to the study coordinator in Madison. The two right hand columns of the sheet were at that time filled in with the SLH results on those same samples. A copy of the completed split sampling data sheet was mailed at the end of the study to the operator, another to the DNR district headquarters.

Operators who sent their monitoring samples to commercial laboratories were asked not to inform their laboratory of the split sampling program. For this to be a "blind" study every laboratory participating would have been unaware at any given time that it was analyzing a split sample also sent to a reference laboratory. However, the municipal laboratory operators who collected the samples also usually performed the analyses. For the most part, the commercial laboratories and analysts did not know about this study nor about their participation in it.

WISCONSIN DEPARTMENT OF NATURAL RESOURCES

Municipality _____

Lab Testing Done By _____

Data Sample Collected & Split	Split By Whom?	Date Sample Analyzed	BOD mg/l	SS mg/l	Do not fill in. DNR Use Only. BOD	SS

When completed with all SS and BOD data, please mail this form to: Wisconsin Department of Natural Resources, Box 7921, Madison, Wisconsin 53707, Attn: Susan Weber, using the envelope provided. Thank you for your help in this study.

FIGURE 2. *Split sampling data sheet.*

Accuracy criteria required that at least 80% of a lab's data fall within two standard deviations above and below SLH results

The criteria used by the author for acceptable data accuracy were based on precision data given in *Standard Methods* (American Public Health Association [APHA] 1975): two standards deviations (SD) above and below the SLH results. For the BOD analysis, *Stardard Methods* states that SD = 15%. Thus, a laboratory's result for BOD was considered acceptable if it was within ±30% of the SLH result. Precision changes with concentration for suspended solids. At 15 mg/1, SD = 33%; at 242 mg/1, SD = 10% (APHA 1975). A laboratory result for suspended solids was acceptable if it was within ±67% of the reference laboratory result at values from zero through 15 mg/1. For values between 15 and 242 mg/1, criteria were based on an assumed decrease in the size of the SD with increasing concentrations. (For example, at 40 mg/1, SD = 25%; at 60 mg/1, SD = 20%; at 100 mg/1, SD = 15%.) Finally, suspended solids results at values of 242 mg/1 and above were considered acceptable if they were within ±20% of the SLH results. According to statistical probabilities, 97.7% of all the data *should* fall within 2 SD above and below the true value (here the value found by the SLH). To allow for some variability within the reference laboratory itself, one out of the five results (or 20% of the data) for each laboratory could lie outside the above criteria for each parameter. Data falling outside the 2 SD were considered "outliers."

As stated in *Standard Methods* (APHA 1975) there are no criteria for accuracy established in the areas just discussed. The author created the criteria based on advice from statisticians at the University of Wisconsin-Madison.

The quality assurance study of the SLH used EPA reference samples for BOD

Use of the State Laboratory of Hygiene as the reference laboratory in this study needed to be justified in terms of the accuracy and variability of its data. That is, how would one know to what degree SLH was accurate in its results. A quality assurance (QA) study was conducted using known reference samples for analysis by the SLH. The reference samples were pro-

vided by EPA with instructions for dilution to the known concentrations.

Each day when municipal wastewater samples for this study were received at the SLH for analysis, two to four QA samples were included. At least one QA sample for suspended solids and one QA sample for BOD were submitted, and usually there were duplicates of each. The QA samples were identified as municipal wastewater effluents (chlorinated) with the usual BOD estimate given on the laboratory slip by the author and the person who daily prepared the EPA check samples. *Note:* A BOD estimate is required on the lab slip so that the SLH can use the proper series of dilutions in setting up the test. All of the labs studied knew the history of BOD results in the past on their samples. Therefore they had an estimate as to what BOD to expect as they analyzed a sample. These QA samples were prepared each morning by an experienced laboratory technician in the sanitary engineering laboratories of the Civil and Environmental Engineering Department of the University of Wisconsin-Madison, placed with ice in the same type of shipping containers used in the rest of the study, and mixed in with the municipal wastewater samples each day in the SLH receiving area. Care was taken to affix the same shipping labels to these QA shippers so in every way the QA samples appeared to be municipal wastewater samples like all the others received there each day. However, to facilitate identification and retrieval of QA data generated by the SLH, mythical Wisconsin community names (Meadowville, Culver, Stockton, etc.) were assigned to the QA samples.

During the course of the study many major municipal laboratories obtained lower BOD results than the SLH. Two major municipal labs demonstrating this tendency were asked to analyze five EPA reference samples for BOD, analyses which served as an additional QA check in this study. Each of the five samples was of a different concentration and was analyzed on a different day.

The operator of each municipal wastewater treatment plant which participated in this study was asked to use the extra lines on the split sampling data sheet for replicate testing (that is, testing the same sample in the same way a second time) on both BOD and suspended solids and for next-day analysis of BOD if possible. This last test was an important aspect of the overall study. Almost all Wisconsin wastewater treatment plant laboratories and some of the commercial laboratories begin the BOD analyses of samples on the same day on which they are collected. Shipping samples to the reference laboratory meant that one day elapsed before the SLH could begin its analysis. It was suspected that even though samples were iced for the shipping period (shipper with ice maintained temperature close to 4°C), the analysis results for BOD might differ between the two laboratories solely due to a difference as to when the respective analyses were initiated. For these reasons, treatment plant analysts were asked to begin their BOD tests the same day the sample was taken (same-day testing) as they usually do, but then to refrigerate the sample overnight and to start another BOD test on that same sample the next day (next-day testing). This procedure would simulate the reference laboratory sample being iced and shipped to the SLH overnight. (Note: In some cases the sample was placed outside overnight; in other cases it was placed in a refrigerator close to 4°C.) It was not possible for the commercial laboratories to do such replicate or next-day BOD testing and remain unaware of this study. For this reason all of the next-day testing data for BOD were submitted by municipal wastewater laboratories.

RESULTS AND DISCUSSION

The SLH met the accuracy criteria for both BOD and suspended solids

State Laboratory of Hygiene results on the BOD reference samples are displayed in Table 4 and plotted in Figure 3. The solid line shown at a 45° angle to the axes in Figure 3 represents where the points would ideally fall if the SLH results agreed exactly with the theoretical values. The dotted lines represent the criteria for acceptable data, for example ±30% of the theoretical BOD values. Out of 42 points four (9.5%) lie outside the criteria lines and are unacceptable (outliers). Since 20% or less of the data may consist of outliers, the SLH met the criteria of accuracy for BOD data.

When EPA first developed its QA samples for BOD it conducted a study in which 73 laboratories analyzed these BOD reference samples. For a theoretical value of 194 mg/1, the mean result was 175 mg/1 (90% recovery); the standard deviation was 15% (Winter 1971). Since the standard deviation is a measure of uncertainty, most statisticians would say a laboratory should average 90% ±15% recovery on these BOD reference samples. With an average recovery of 88% the SLH results are well within the uncertainty limitations.

Results from the SLH on the suspended solids QA samples are shown in Table 5. Splitting the BOD sample and submitting the two duplicate samples for analysis did not seem to affect the BOD results. However, splitting the EPA suspended solids reference sample and subsequent analysis of the two "duplicate" samples presented difficulties, difficulties that may be due to the fact that the suspended solids samples were nylon fibers diluted to yield the known concentrations. These fibers do not very accurately simulate wastewater other than in a gravimetric way. (All of the BOD was in a soluble form.) Due to the early discrepant results obtained, the suspended solids QA samples submitted for SLH analysis after April 13, 1978 were no longer split after preparation.

Since the early suspended solids QA data was considered unreliable, only the SLH results for April 13 through the end of the study are plotted in Figure 4. Again the solid line represents where the points would ideally fall if there were perfect agreement of SLH results with the known values. The dotted lines represent the criteria for acceptable suspended solids data defined previously. With no outliers for suspended solids, the SLH meets the criteria for data accuracy for suspended solids analyses. To date EPA has not conducted a study of laboratory performance on its QA samples for suspended solids (Harold Clements, pers. comm. 1978). However, the standard deviation of the SLH data (SD = 14%) is well within the uncertainty of this analysis as reported by *Standard Methods* (APHA 1975).

TABLE 4. *State Laboratory of Hygiene (SLH) results on BOD reference samples.**

1978 Date	BOD (mg/l) Theoretical	BOD (mg/l) SLH Results	% Recovery*	1978 Date	BOD (mg/l) Theoretical	BOD (mg/l) SLH Results	% Recovery
3 Mar	31	30	96.8	29 Mar	33	29	87.9
	31	27	87.1				
				30 Mar	26	23	88.5
8 Mar	31	36	116		26	22	84.6
	31	26	83.9				
				31 Mar	37	49	132
9 Mar	16	15	93.8		37	57	154
	16	15	93.8				
				5 Apr	21	16	76.2
10 Mar	29	27	93.1		21	19	90.5
	29	25	86.2				
				6 Apr	36	34	94.4
15 Mar	24	20	83.3		36	34	94.4
	24	17	70.8				
				12 Apr	32	24	75.0
16 Mar	43	29	67.4		32	30	93.8
	43	24	55.8				
				13 Apr	58	49	84.5
17 Mar	36	26	72.2		58	49	84.5
	36	25	69.4				
				14 Apr	44	40	90.9
22 Mar	38	29	76.3		44	40	90.9
	38	22	57.9				
				19 Apr	13	12	92.3
23 Mar	22	17	77.3		13	11	84.6
	22	17	77.3				
				20 Apr	64	53	82.8
24 Mar	26	23	88.5		64	62	96.9
	26	23	88.5				
				21 Apr	88	76	86.4
29 Mar	33	30	90.9		88	78	88.6

Mean = 31 mg/l; s = 16 mg/l.
**Average overall recovery = 87.7%.

One of two municipal labs analyzing EPA reference samples for BOD had acceptable data

As many major municipal laboratories have shown a tendency to obtain BOD results that were consistently lower than those obtained by the SLH, two ("Lab A" and "Lab B") were each asked to analyze five EPA reference samples for BOD.

In checking the results for Lab A given in Table 6, the average recovery of BOD (56.7%) seems low. Figure 5 represents these same data plotted with the data criteria lines for BOD imposed. This figure shows all five points as outliers: not one result of acceptable accuracy. Follow-up visits have found that at the time of the study Lab A was cleaning its BOD dilution water carboy and delivery tubing with acid dichromate. If not thoroughly eliminated by rinsing, the dichromate serves as a toxic agent for the BOD analysis. Since the study acid dichromate cleaning has been discontinued. It was later found that an improperly prepared reagent was being used for the analysis. However, Lab A is still being studied to account for its low BOD results.

Results obtained by Lab B on the BOD reference samples (Table 6) show a reasonable average recovery of 88%. Figure 6 indicates that when these data are plotted with the BOD criteria lines there are no outliers; all of the BOD data for Lab B are of acceptable accuracy. The wastewater treat-

ment plant which this major municipal laboratory serves has fairly recently started nitrification. It is postulated that a difference in seed materials between Lab B and the SLH may account for differences in their results on split samples. To date, this possibility is still being investigated.

Many labs had unacceptable data

Five split sampling results for BOD and suspended solids were obtained for almost all of the laboratories: 25 major municipal, 90 minor municipal, 10 major commercial, and 3 minor commercial.

As displayed in the example given in Figure 7, these data were also plotted for each individual laboratory. (These results and graphics are in the author's files.) Figure 7 shows the BOD and suspended solids results obtained by "Lab X" plotted against the results obtained by the SLH on the split samples. The graph contains the solid line at a 45° angle to the horizontal; that again represents where the points would fall should the results be in complete agreement. Criteria lines for BOD are values 30% above and below the 45° line. Criteria lines for suspended solids are curved above and below the 45° line with values ranging from 67% at 15 mg/l to 20% at 242 mg/l; above 242 mg/l the lines parallel the 45° line. The values for the criteria lines between 15 and 242 mg/l are as defined earlier. (Note: The criteria lines are *not* mirror images above and below the 45° line. At each point on the horizontal axis the criteria lines are equal distances vertically above and below the 45° line.)

Each graph was generated by a computer program which adjusted the scale to be 1.1 times the highest value point to be plotted. In a few cases, this resulted in a point with an extremely large value causing most of the other points to be clustered near the origin. In these cases supplementary plots were made on a much smaller scale to show those previously obscured points.

Lab X in Figure 7 has two outliers out of five results for BOD (40% of the data). Two of the five suspended solids results are clearly outliers. A third is on the upper criterion line (where points fell on criteria lines they were judged as being within the acceptable range). Thus, Lab X has two suspended solids outliers out of five results (or 40%). Since only up to 20% of the data can be outliers, Lab X was found to generate unacceptable data for both BOD and suspended solids analyses.

An examination of each of the indi-

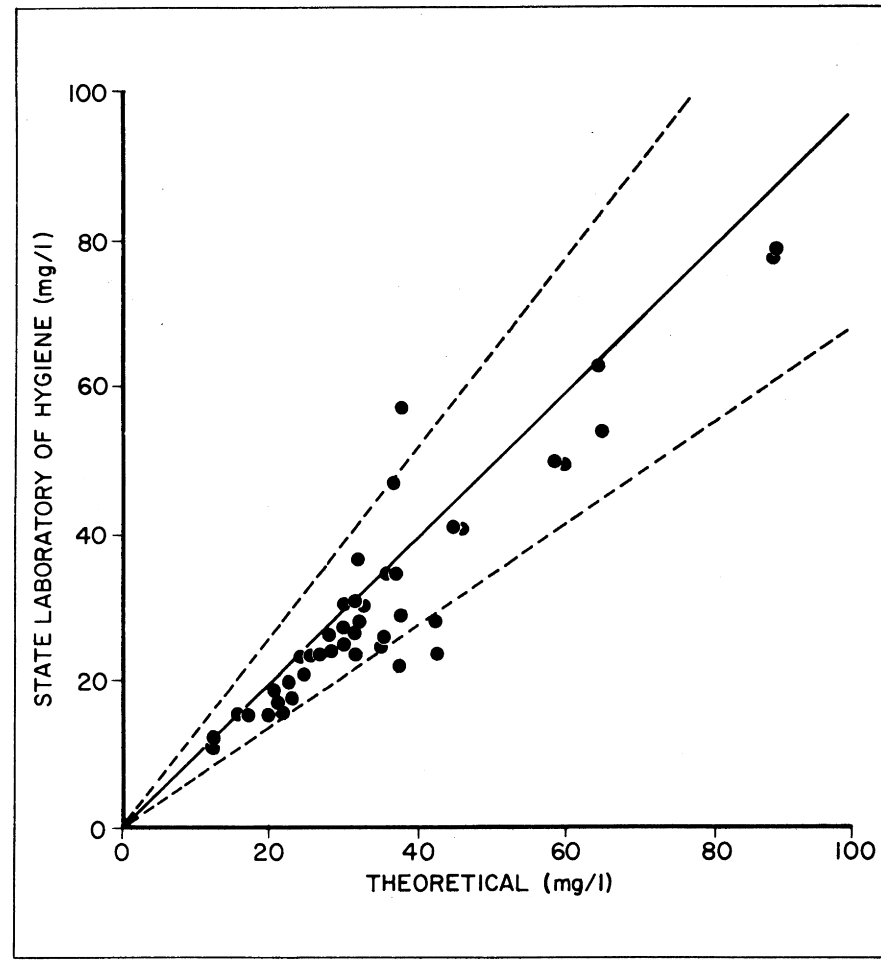

FIGURE 3. *State Laboratory of Hygiene results for BOD reference samples.*

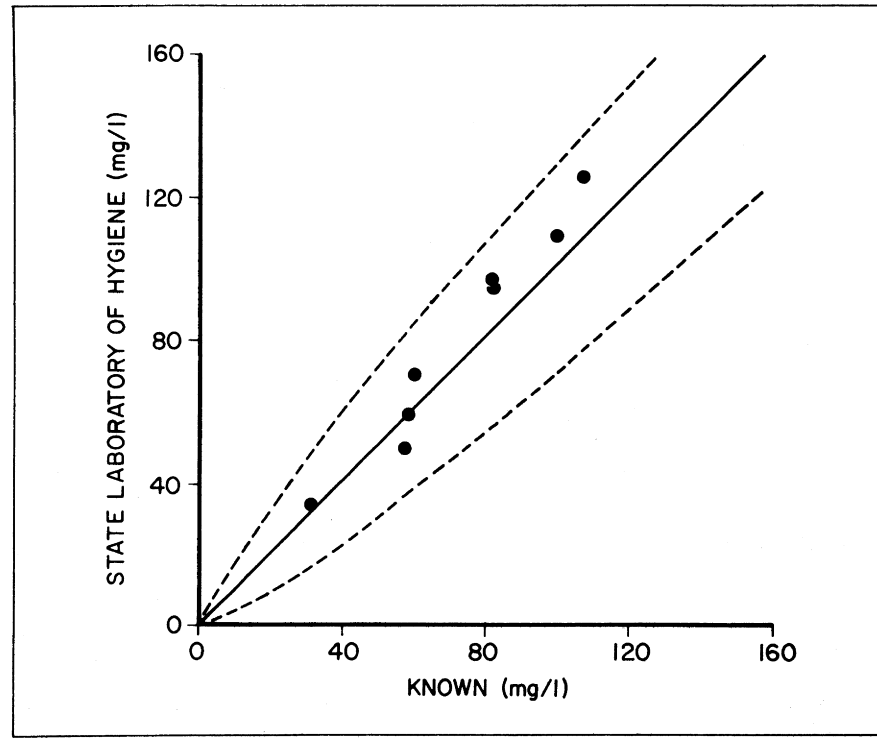

FIGURE 4. *State Laboratory of Hygiene results for suspended solids reference samples.*

TABLE 5. *State Laboratory of Hygiene (SLH) results on reference samples for suspended solids (SS).*

1978 Date	SS (mg/l) Known	SS (mg/l) SLH Results	% Recovery	1978 Date	SS (mg/l) Known	SS (mg/l) SLH Results	% Recovery
3 Mar	32.9	32	97.3	30 Mar	30.33	23	75.8
	32.9	16	48.6		30.33	30	98.9
8 Mar	32.3	18	55.7	31 Mar	29.22	18	61.6
	32.3	38	118		29.22	26	89.0
9 Mar	29.8	32	107	5 Apr	31.22	25	80.1
	29.8	21	70.5		31.22	29	92.9
10 Mar	29.0	36	124	6 Apr	31.22	26	83.3
	29.0	21	72.4		31.22	34	109
15 Mar	30.9	22	71.2	12 Apr	30.55	26	85.1
	30.9	30	97.1		30.55	23	75.3
16 Mar	30.8	25	81.2	13 Apr*	60.88	70**	115[1]
	30.8	36	117				
				14 Apr	58.88	60	102
17 Mar	32.4	41	126				
	32.4	28	86.4	19 Apr	31.77	34	107
22 Mar	30.9	34	110	20 Apr	82.77	98	118
	30.9	21	68.0				
				21 Apr	100.88	110	109
23 Mar	29.6	20	67.6				
	29.6	28	94.6	26 Apr	108.66	126	116
24 Mar	28.6	36	126	27 Apr	83.98	96	114
	28.6	20	69.9				
				28 Apr	58.66	50	85.2
29 Mar	28.2	21	74.5				
	28.2	22	78.0				

*QA samples taken after April 13 were no longer split after preparation due to early discrepant results. See text for discussion.
**Mar 13-28 mean = 80.5 mg/l; s = 31.9 mg/l.
[1]Mar 13-28 ave. recovery = 108%.

vidual laboratory graphs yielded the summary in Table 7, indicating that 80% of the major municipal, 72% of the minor municipal, 60% of the major commercial with sufficient data to study, and 100% of the minor commercial labs generated unacceptable data for BOD. Twenty percent of the major municipal, 35% of the minor municipal, and 70% of the major commercial labs produced unacceptable data for suspended solids. Sixteen percent of the major municipal, 30% of the minor municipal, 40% of the major commercial, and 33% of the minor commercial labs generated unacceptable data for both parameters.

The significant difference in next-day BOD results from the SLH and municipal laboratories was not due to shipping time to SLH

The municipal wastewater laboratories began the BOD analyses on the same day the samples were taken (same-day testing), their usual practice. However, 134 of the samples were refrigerated overnight at the municipal laboratories and BOD analyses conducted again the next day (next-day testing). Thus, for each of these 134 samples there were three BOD results: the "same-day" result, the "next-day" result (both generated by the municipal laboratory), and the SLH result. These data are in the author's files.

The purpose of this aspect of the study was to assess any discrepancy in the results of the BOD analysis conducted on the same day the sample was

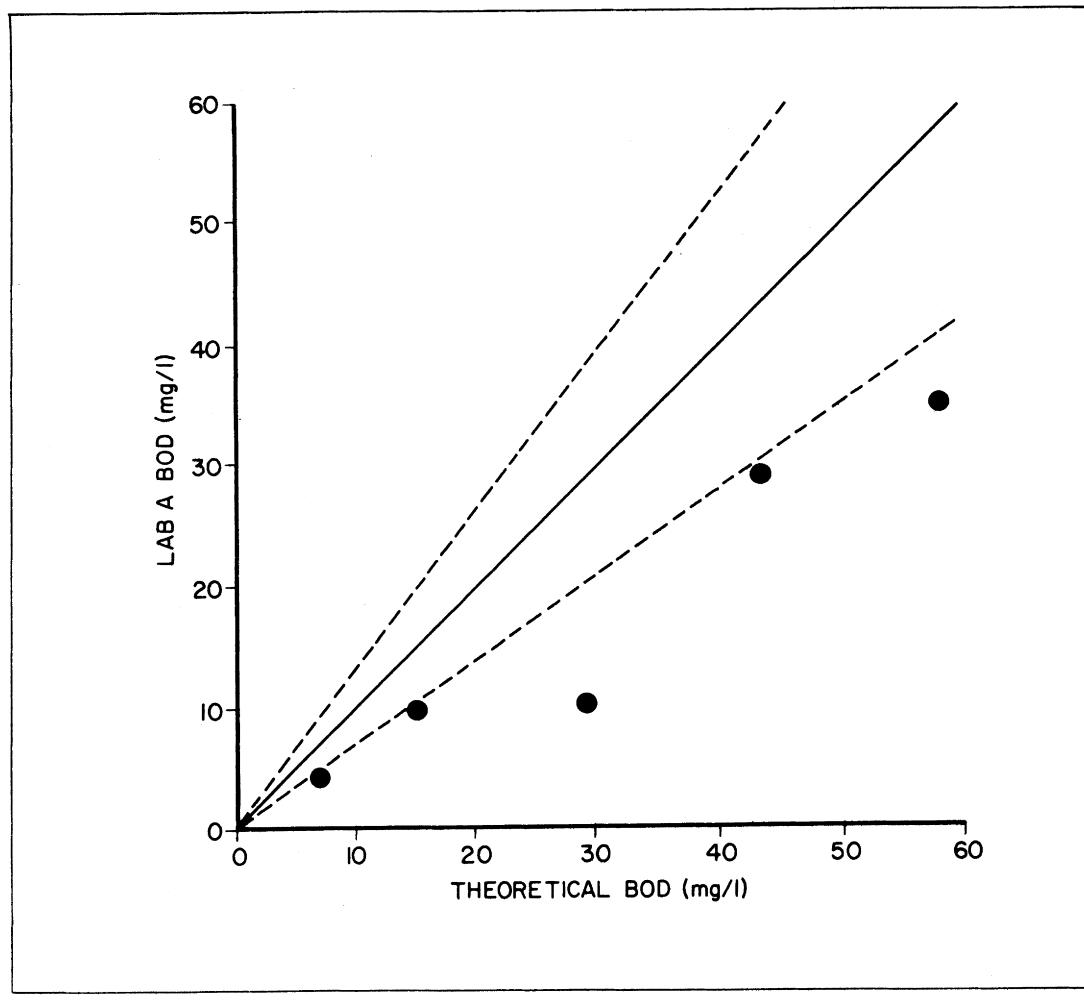

FIGURE 5. *Lab A's results for BOD reference samples.*

taken compared to the next day (when the SLH performed the analysis) that might be caused solely by the difference in when the respective analyses were initiated. The null hypothesis (that there would be no difference) was tested by means of a *t-test* (Table 8). For a 95% confidence interval and 133 df, the null hypothesis can be rejected at *t*-values above 1.98. Thus the null hypothesis holds for same-day vs. next-day testing of BOD within a municipal wastewater laboratory; there is no significant difference in these two results. However, the null hypothesis is rejected when the SLH result is compared to either of the other two. That is, there is a signifcant difference in results between the SLH and municipal laboratories for the BOD analysis.

The data were also examined in terms of correlation coefficients, average percent differences ($\bar{d}\%$), and the uncertainty of those mean differences. The correlation coefficient; *r*, is a mea-

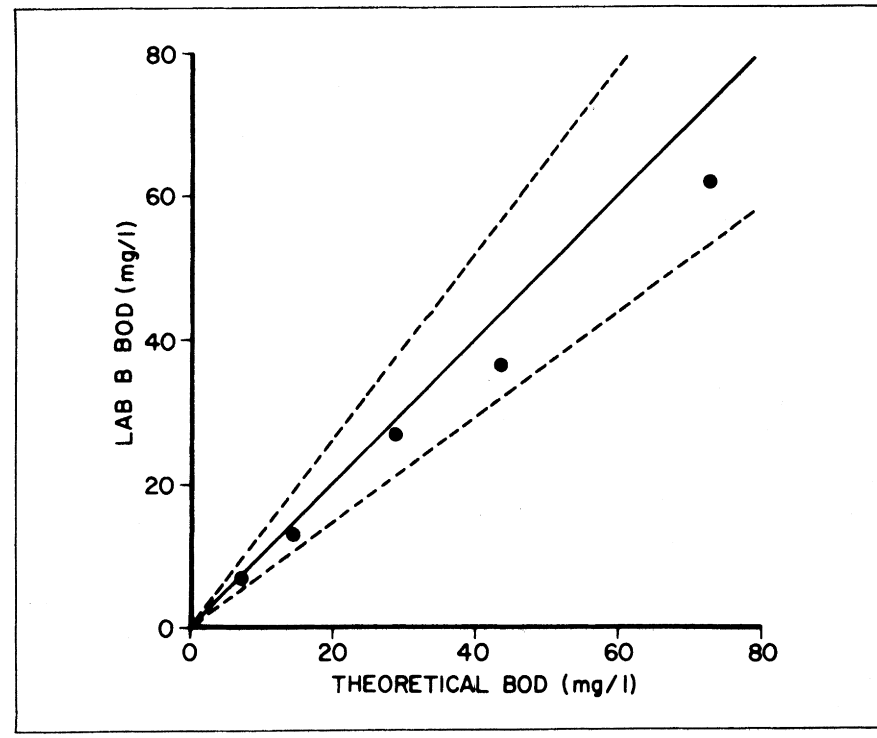

FIGURE 6. *Lab B's results for BOD reference samples.*

TABLE 6. *Reference sample BOD analysis results from two major municipal laboratories.*

	Laboratory A				Laboratory B		
	BOD (mg/l)				BOD (mg/l)		
1978 Date	Theoretical	Reported	% Recovery	Day	Theoretical	Reported	% Recovery
May 3	14.6	10	68.5	1	14.6	12.9	88
May 4	43.8	29	66.2	2	43.8	36.7	84
May 5	7.3	4	54.8	3	7.3	6.5	89
May 10	58.4	35	59.9	4	73	62.3	85
May 11	29.2	10	34.2	5	29.2	27.2	93
Average	30.7	17.6	56.7	Average	33.6	29.1	88

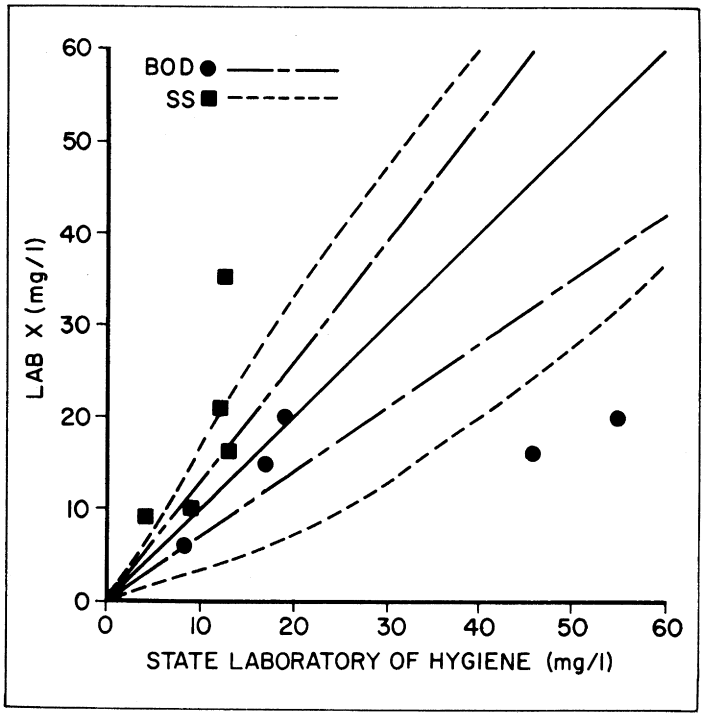

FIGURE 7. *Split sampling lab results for Lab X.*

TABLE 7. *Percent of laboratories studied generating unacceptable data.*

Laboratory Category	BOD	Suspended Solids (SS)	Both BOD and SS
Minor municipal	72	35	30
Major municipal	80	20	16
Minor commercial	100	33	33
Major commercial	60	70	40

TABLE 8. *Results for next day analysis for BOD.*

Comparison	*t*-Test	Null Hypothesis	Correlation Coefficients (r)	Mean Percentage Differences ($\bar{d}$,%)	Uncertainty (%)
Same-day vs next-day	1.94	Accepted	0.716	7.1	7.2
SLH* vs next-day	3.73	Rejected	0.795	17	9.1
SLH vs same-day	3.38	Rejected	0.816	13	7.5

* State Laboratory of Hygiene.

sure of the strength of a linear relationship between two sets of data. If there is a perfect correlation, as ideally there should be with these data, $r = 1$. The percent uncertainties were calculated again using a 95% confidence interval (Table 8). The best correlation obtained was between SLH results and the same-day results of the municipal laboratories. The mean percent difference with its uncertainty suggests that 95% of the time the difference between the same-day and next-day BOD results will be 7.1% $\pm$7.2% with a 7.1% difference being the best guess (Table 8).

Minor data collection problems included late pickups and changes in labs serving plants

The laboratory split-sampling was carried out from February through May 1978. Problems experienced in receiving samples the day after collection at the SLH laboratory included pickups not made when scheduled and samples not delivered the next day to SLH. From early in this study samples more than one day old were rejected, a decision based on EPA guidelines and the experience of the staff at the SLH water chemistry laboratory. The above problems necessitated rescheduling of some sampling to obtain five split samples for each laboratory selected to participate in the study.

The study revealed that some small treatment plants which had been thought to do their own testing (minor municipal) actually did not. Instead, these treatment plants sent their analyses to commercial laboratories. As a result, data were received only from 90 of the 113 minor municipal laboratories selected for the study. In other cases it was found that wastewater plants had switched from one commercial laboratory to another. In these instances, other treatment plants served by the commercial laboratory in question were asked to submit the missing samples. Due to these two adjustments, the number of wastewater treatment plants in each column of Table 2 changed slightly from the initial aspect of the overall study.

There was also some minor difficulty in receiving and interpreting all the data from the treatment plants. Some operators needed to be reminded to send in their results. Still others recorded results which were difficult to decipher. However, most of the data was submitted promptly and clearly by the operators who participated in the study.

Unacceptable data from municipal laboratories stem from problems with equipment and techniques

Some of those municipal laboratories having gross data accuracy problems have received follow-up visits from DNR field staff. Findings from the Southern and Lake Michigan DNR districts indicate that use of the following equipment, procedures, or personnel can be possible causes for unacceptable laboratory data (not listed in order of importance or frequency):

(1) malfunctioning BOD incubators;

(2) outdated manometric procedure for the BOD analysis;

(3) dissolved oxygen probes improperly calibrated and maintained;

(4) improper dilution factors for the BOD analysis;

(5) improperly stored and dispensed dilution water that exerts a substantial demand;

(6) improperly cleaned BOD bottles;

(7) BOD bottle water seals that evaporated;

(8) analytical balances not capable of weighing to 0.1 mg or malfunctioning;

(9) improper filters for the suspended solids test;

(10) drying oven not capable of maintaining temperature 103$\pm$2°C;

(11) analyst unable to properly perform required calculations.

DISCUSSION

Quality assurance programs and mandatory certification are possible long-term solutions

Some alternatives which can be considered for long-term results in correcting quality assurance problems at laboratories analyzing municipal wastewater are as follows:

(1) Design a quality assurance (QA) program for waste treatment laboratories (CGUS 1977:30).

(2) Establish a mandatory wastewater laboratory certification program applicable to all laboratories doing WPDES self-monitoring analyses.

(3) Require each laboratory analyst to be certified for each WPDES analysis that he or she performs.

(4) Require each wastewater laboratory that performs analyses of WPDES self-monitoring samples to submit regular reports of its QA data.

(5) Conduct a study of industrial WPDES dischargers for laboratory data quality and, if results warrant it, initiate a QA program similar to (1).

(6) Include the following as topics for further study and/or a more refined approach to the original problem this study has investigated: the variability of BOD analyses for samples with low concentrations (e.g. 5-10 mg/l); standard deviation sizes that differ from those cited in studies reported by *Standard Methods* (APHA 1975); the effect of inaccurate analyses on monthly mean BOD; and suspended solids concentrations at wastewater treatment plants.

A valid question at this point is whether a continuing QA program for waste treatment laboratories should be a voluntary reference sample program or a mandatory lab certification program. In January of 1978 EPA recommended "establishing at least a voluntary check sample quality assurance program" with all municipal permittees (CGUS 1977:52). The Agency suggested that a voluntary program could be implemented sooner than a certification program. EPA also stated that certification of wastewater laboratories "may be an attractive means for

assuring data quality" (CGUS 1977:52). EPA has not pursued laboratory certification because it lacks legislative authority to do so. However, it has made available its QA reference samples at no cost to any laboratory which requests them (i.e., a voluntary check sample QA program). Within the State of Wisconsin only a handful of wastewater laboratories have participated in this program. Of these laboratories only a few have used, and are currently using, the reference samples on any regular basis.

Another alternative possible is a laboratory certification program. As of April 1977 "three states had mandatory certification programs for wastewater laboratories, six states had voluntary programs, eight other states were planning to establish certification programs, and one state had established a task force to study laboratory certification" (CGUS 1977:37). The states which had mandatory laboratory certification differed as to whether or not in-plant laboratories were included. The State of Connecticut has an existing certification program which uses reference samples. Oklahoma requires an inspection of laboratory facilities as well as analysis of reference samples. Laboratories in that state are certified by parameter if they meet certain standards in terms of their performance on the reference samples. Oklahoma does not accept self-monitoring data reports from uncertified laboratories (Madden 1978:70).

Any mandatory certification program that would be applicable to all laboratories doing WPDES self-monitoring analyses could be administered by the Department of Natural Resources. However, statutory responsibility for laboratory certification currently rests with the Department of Health and Social Services (Section 143.15 Wis. Stats.). To date this Department has not implemented laboratory certification for wastewater parameters. As the Department of Health and Social Services does not deal with wastewater treatment plants nor with the WPDES program, such implementation does not seem likely in the future.

A laboratory certification program could require an inspection of laboratory facilities and procedures together with the performance of reference sample analyses. Laboratory certification could be granted if the reference sample data quality met certain criteria. The new Wisconsin Administrative Code, NR 114, relates to certification of operators by process, including laboratory testing. However, this code only requires the certification of wastewater treatment plant laboratory personnel. It does not require certification for those analysts employed by commercial laboratories. Also, NR 114 only requires an initial quality assurance evaluation; there is no required periodic follow-up to check quality assurance after initial certification.

SUMMARY AND CONCLUSIONS

Compared to a proven reference lab, the State Laboratory of Hygiene (SLH), most labs in this study produced unacceptable BOD data, one-third unacceptable suspended solids data, and one-third unacceptable data for both parameters due to malfunctioning or improper equipment and/or improper techniques. Quality assurance and mandatory certification programs are considered for labs/individuals performing WPDES analyses

Approximately one-half of the municipal wastewater treatment plants in Wisconsin conducted their own WPDES self-monitoring analyses for BOD and suspended solids. In most of these plants, the analyses were performed by the operators, who have other duties. Commercial laboratories did the analyses for about one-third of the treatment plants. The remaining plants had their analyses performed by neighboring municipal laboratories.

To assess the accuracy of data submitted by these laboratories, a split sampling program was conducted with a number of laboratories in each of the above categories; half of the major municipal laboratories (where at least one person was employed full-time to perform the analyses); about 40% of the minor municipal laboratories (where the operator did the testing); all of the major commercial laboratories (conduct analyses for more than two communities); and 30% of the minor commercial laboratories (conduct analyses for two or less municipalities). Except for the major commercial category, where all were studied, the laboratories were chosen at random. Based on a properly designed study of these laboratories, some conclusions and generalizations can be made about the BOD and suspended solids data generated by *all* Wisconsin laboratories.

Before the central issue of the accuracy of BOD and suspended solids data reported by Wisconsin laboratories could be addressed, three questions were investigated: (1) What criteria should be used to judge data accuracy for the BOD and suspended solids analyses; (2) How did one know the reference laboratory, the SLH, was accurate in its own analyses; (3) Did it make a difference in the BOD results that the reference laboratory analyzed the samples one day after the laboratories being studied did.

The question of data accuracy criteria was dealt with by making some statistically based decisions. Since 97.7% of the data should fall within two standard deviations (SD) of the correct result, it was decided that criteria to be used were two standard deviations (as SD is defined by *Standard Methods* for each parameter, APHA 1975) above and below the reference laboratory result. To allow for variability within the reference laboratory it was determined that as much as 20% of any other laboratory's data could be outside the criteria (outliers). A laboratory was judged to produce data of unacceptable quality for that parameter if more than 20% were outliers. Decisions made based on these criteria were quite generous in judging a lab's data acceptable and conservative in judging it unacceptable.

The question of reference laboratory accuracy was answered by means of a quality assurance study of the SLH using known samples. The reference laboratory met both the criteria for data accuracy as defined above. In addition, an EPA study and knowledgeable opinion support the conclusion that the SLH is a credible reference in determining the accuracy of BOD and suspended solids analysis results produced by other laboratories.

The question of the importance of a day's delay in initiation of the BOD analysis at the reference laboratory was also investigated. It was found that within a given municipal laboratory there is no significant difference in results obtained by analyzing a sample on the same day it is collected (same-day testing) versus analyzing the refrigerated sample one day later (next-day testing). Thus the delay in the start of the BOD analysis due to shipping to the reference laboratory does not seem to be responsible for a difference in results with a laboratory being studied.

In assessing the accuracy of BOD and suspended solids data reported by Wisconsin laboratories, this study revealed that most laboratories produced unacceptable BOD data, about one-third generated unacceptable data for suspended solids, approximately one-third produced unacceptable data in both analyses. As groups, the minor commercial labs seem to be the least accurate for BOD analyses (all had unacceptable data), major commercial labs the most (40% had unacceptable data). However, when compared to the other laboratory categories, the major commercial labs were quite inaccurate on the suspended solids analyses (70% had unacceptable data). Since no accuracy standards at all have been used to date for the BOD and suspended solids analyses data, it is not surprising to find that many Wisconsin wastewater laboratories do not meet even the generous criteria used in this study.

The following conclusion is based on follow-up visits to municipal laboratories which produced poor quality BOD and suspended solids data in this study: Most Wisconsin laboratories are not meeting the requirement that analysts should adhere to procedures described in *Standard Methods* (APHA 1975) in performing BOD analyses. One-third of the labs are not adhering to *Standard Methods* for suspended solids determinations. For both analyses unacceptable data seems to be the result of improper or malfunctioning equipment, poor analytical technique, and/or improper calculations.

Alternatives for correcting the problems include establishing at least a voluntary quality assurance program for wastewater treatment labs, as well as mandatory certification programs for labs and individuals doing WPDES self-monitoring analyses.

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