MONITORING OF ON-SITE SYSTEMS - REGULATORS, INSPECTORS AND OPERATORS BEWARE!

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Abstract

The usual methods for site evaluation, and later the determination of effective operation of an on-site wastewater system, whether a traditional septic drainfield or a modern aerated wastewater system, are for soil and effluent samples to be taken and compared against some arbitrary ideal. Regulators seem keen for check lists of soil, water and waste constituents for analysis either at the planning phase or later to 'check' on the operational performance and sustainability of the system. Whether these activities have any real role in risk assessment and environmental security, the variability between and within samples needs to be considered prior to preparation of monitoring routines.

This paper examines potential variability in soil sampling and analysis depending upon the location of the sample, the preservation of the sample, deterioration of samples and the analytical results. While it is recognised that in-soil variability is high, there are erroneous expectations that sample analyses are precise and accurate. Within and between sample results and within and between laboratory results often fall outside an expected range of variability. This paper uses data from national and international inter-laboratory proficiency testing programs in soil and water analysis to examine typical variability for identical samples. The consequences of assuming high levels of accuracy may be misconceived and the end user needs to be conscious of possible natural variations. When the variability of sampling is added to this assessment, wide ranges in typical values of contaminants, pollutants and nutrients can be expected.

Keywords

accreditation, certification, detection limits, practical limits of quantification

1 Introduction

It is not usual to come across a long list of 'pollutants' (many would use the term nutrients) to be measured in order to comply with some state or local regulation, or against which to verify performance. Trade waste connections to sewer require approval by the local authority operating the sewage treatment plant (STP) while licences to discharge from the STP into the waterway or onto land are regulated at the state level. Septic tanks, aerated wastewater treatment plants, sand filters and the numerous other combinations of treatment, and their soil dispersal areas, come under the scrutiny of local authorities. In NSW, The Local Government (Approvals) Regulations 1999 applies to all on-site systems. Similar regulations exist in other states, some under the local environment protection agency (Victoria and Queensland). Health departments invariably are co-conspirators in developing requirements for monitoring and surveillance, while the possibility of reuse of treated effluent gathers a myriad of likely (and competing) regulators, depending upon the location.

With the plurality of the 'nice people' that oversee the day to day operation of the regulations, it is not without 'good reason' that each has an input into what should be measured. Just because a 'pollutant' or 'nutrient' can be measured, does not mean that it should be measured. A long list of analyses does not necessarily make a valuable array of data. Because technology has the ability to discriminate concentrations in the parts per billion (µg/L or µg/kg), there is often no evidence to support the reason why the lowest detectable value should be sought. In many cases, analysing a time series of an on-site system is closer to 'bucket chemistry' than it is to high-tech instrumental analysis. It is the interpretation of reliable data that is most important and often poorly done.

The assumption that samples can be split and sent to two reputable laboratories and the results will be 'the same' does not always hold true and considerable variation is shown in the results of proficiency testing that occurs from inter-laboratory programs and the numerous methods available to the analyst. This paper will give an insight into the variability one may expect.

Triple Bottom Line assessment should cover monitoring operations and in particular the avoidance of 'waste' of collecting data that are irrelevant.

2 Laboratory Procedures

International and national practices for analysing soil, water and wastewater have been in place for many years. *Standard Methods for the Examination of Water and Wastewater 19th Edition* (APHA, 1998) is the current reference for water analysis as set out in NSW Department of Environment and Conservation (DEC) *Approved Methods for the Sampling and Analysis of Water Pollutants* in NSW. A range of methods from Standards Australia and United Stated Environment Protection Agency ((USEPA) are also used. That does not mean that all the tests are routinely performed by Australian laboratories. Some tests are labour intensive and therefore expensive, or require technically intricate equipment to run a particular test. Whether a laboratory conducts a particular routine test depends upon equipment, expertise and a reasonable throughput of samples to warrant such dedicated resources. Therefore, for a regulator to specify particular test methods that are not performed routinely in the local area, it may mean that many samples from the same source have to be sent to several laboratories, adding to sample collection and freight costs. These additional costs may be significant and should be considered in the monitoring program.

Other methods are commonly developed to suit particular proprietary equipment. Pallintest™ and Lovibond™ have a range of simplified colorimetric tests that use hand held equipment. Hanna Instruments have similar tests that use hand-held and benchtop equipment while Merck have tests that use small spectrophotometers. While these measuring devices may be 'simplistic', they are based upon more elaborate laboratory testing procedures, the reliability of the crude data may be sufficient to identify performance of the on-site system.

The Australasian Soil and Plant Analysis Council (ASPAC) relies upon soil testing methods documented in Rayment & Higginson (1992) *Australian Laboratory Handbook of Soil and Water Chemical Methods* for its proficiency program. Not all nutrient analyses are covered and other references such as *Methods of Soil Analysis* by the American Society of Agronomy (1982) are used. It is interesting that no reference is made to soil testing methods by NSW DEC in its site and soil requirements for development planning. The importance of acceptable methods is that they offer reliability to reproduce consistent results that reflect what they are supposed to measure. For that reason it is often not appropriate to take water testing methods and apply them to soils or soil solutions. Measuring phosphorus in water is relatively simple – filter the water and by a colorimetric method develop a blue colour that can be matched

against known calibration standards. However, that method cannot be used with soil unless the phosphorus is first displaced from the soil, filtered and measured. It is the removal from the soil that requires specific reagents and procedures. Results will depend upon extracting solution and measuring technique.

3 Certified, Accredited or Proficiency Tested

The Macquarie Dictionary defines the verb "accredit - (1) to furnish (an officially recognised agent) with credentials; or (2) to certify as meeting official requirements. One would assume that an accredited person, laboratory or body would hold some recognition as to meeting certain requirements set by some 'official' body. A term that is used to convey a similar meaning is 'certification'. The Macquarie Dictionary defines "certify – (1) to guarantee as certain; give reliable information of; and (2) to testify to or vouch for in writing.

So what gives a body the right to accredit or certify an organisation as: (1) meeting some formal requirements; and (2) the certificate it issues gives reliable information. Notice that neither term uses 'accurate' or 'precise', each of which has a scientific implication.

Only the Joint Accreditation System of Australia and New Zealand (JAS-ANZ) provides accreditation of certification bodies, inspection bodies and auditor training course providers. It is not unusual that a professional organisation to accredit members who meet minimum rules of entry. So where does it get the right? Simply from its own Charter! But how legitimate is that accreditation? It is usual for governments, the courts and others to accept that when a professional body accredits a member, that the professional body has checked that members credentials against predefined criteria and certified that the member meets the criteria (which may be academic, experience and/or ethics oriented). The most well understood certification is that to practise medicine, one must be recognised by the Australian Medical Association (AMA). AS ISO/IEC 17010-2003 *"General requirements for bodies providing accreditation of inspection bodies"* requires that the accreditation body meets organisational criteria, has a quality system in place, audits its system and has suitably qualified personnel.

The question arises "can a government department or local authority proclaim itself an accrediting authority?" There are many examples where a government agency has just done that and used regulations to enforce compliance. But does that give credibility to the accreditation process? The author believes that agencies often make a mockery of scientific endeavour because the "certification' is less than rigorous and often based upon incomplete scientific and statistical scrutiny. An example is the 'accreditation' of aerated wastewater treatment systems that have been tested on secondary effluent at a municipal sewage treatment works, rather than on a statistically valid sample of actual working domestic systems. A second example is NSW Workcover which has regulated its accreditation role through the Occupational Health and Safety Regulation 2001 (NSW Government, 2001).

The National Association of Testing Authorities (NATA), an organisation set up as an accreditation body, is recognised by the Commonwealth Government for the accreditation of laboratories conducting tests and measurements in all technical fields and as a peak authority for the accreditation of inspection bodies (Standards Australia, 2004). There are other professional bodies that provide accreditation roles but avoid the term 'accreditation' because of the conflict with NATA accreditation. One such body is ASPAC that undertakes inter-laboratory proficiency testing program for soil and plant material in Australasia, and certifies laboratories that meet specific statistical criteria for the analyses they perform. NATA accepts these proficiency programs as meeting its own proficiency requirements. Therefore, from an Australasian perspective these ASPAC certified laboratories perform equally as well as

NATA accredited facilities in testing Australian soils. In the USA, the American Association for Laboratory Accreditation performs a similar task (AALA, 2005). Both NATA and AALA, and European countries use the ISO/IEC 17025:2000 standard as their criteria for accreditation. However, there are major differences between methods in these countries.

The author is concerned that when authorities and courts require accreditation of service providers, then the service they provide should address accredited regulators. That is far from the case as is clear from the numerous 'nonsense' guidelines and check lists one must satisfy.

4 Practical limits of quantification

Under the NSW Load Calculation Protocols (DEC, 2005), the practical quantification limit (PQL) are "the lowest level at which a substance can be routinely quantified and reported by a laboratory". A licensee may use half the PQL when the PQL is reported or where over a licence period more than 50% of the samples are less than the PQL, then zero may be used. In scientific terms the use of 'zero' is to be avoided since we can only detect down to the noise levels of the instrument. A different laboratory may have more sensitive instruments able to measure to lower concentrations. For example, an inductively coupled plasma-optical emission spectrometer (ICP-OES) in radial mode may have detection limits for aluminium in water at 1.5 µg/L, an axial ICP the limit is 0.30 µg/L and an ICP mass spectrometer (ICP-MS) can detect to 0.027 µg/L. The difference in price of instruments is substantial.

The 'bucket chemistry' that is still the mainstay of much soil and wastewater analysis has detection limits in the mg/L range, however, just because an element can be measured at ultra-low levels, such as the example used above, does not mean that the result has any more validity than that derived by wet chemistry. Therefore, an understanding of the detection limits used by the analyst and reported by the laboratory is critical to interpreting the results.

Two questions arise: (1) "What is an acceptable detection limit for each analyte?" (2) What detection limit (therefore method) gives sufficient precision on which to make a meaningful interpretation? I believe that these two areas are poorly understood by regulators and forcing additional precision does not always achieve a more valuable number for better interpretation. It will be seen that when NATA results have a significant inter-laboratory variation, the precision of measuring has only minor relevance in monitoring on-site system.

6 Sample Collection

Perhaps the most abused component of environmental monitoring is the actual collection of the representative sample. Before you attempt to take the sample, you need to ask - Why? Who? When? How? Where? The answer to each will indicate a sampling method, the container, the timing, the location of the sampling point and who is competent to collect.

In Figure 1, there are many possible sampling locations. A sample for biochemical oxygen demand (BOD_5) determination can be taken on the windward or the leeward side, from just on the surface or at some predetermined depth. Does taking numerous samples and mixing them together produce a better result? How does one account for various volumes at each depth? Well, what **IS** a better result? One that passes the licence limit or one that more truly reflects what is actually in the lagoon? The answer is NOT simple and requires considerable thought as to what purpose will the result be put. If you are after the likelihood of environmental consequences from overflows, then the sampling point is near the potential discharge point. If it is one of irrigation water quality, the sample is taken near the intake to the pump. Taking

numerous samples from various points or depth may actually distort or mask 'real' water quality. You need to have a sampling protocol in place.

Figure 1 Numerous possible locations for water sampling to determine 'average' quality

7 Sample Containers and Preservatives

The AS/NZS 5667-1998 (ten parts) sets out the acceptable methods for sampling equipment, sample collection, sample preservation, identification and the number of samples required. Table 1 sets out some of the considerations needed to collect routine wastewater samples. It is clear from the table that to take samples for all the required analytes at Figure 1, you would require equipment to analyse on-site (residual chlorine and nitrite-N), a separate 1 L bottle for BOD_5, a 50 mL sample filtered (0.45 um) on-site and refrigerated for dissolved phosphorus, a 100 mL bottle completely filled for EC, a 500 mL sample for ammonia-N and a 100 mL acidified sample for metals. How often is only one sample taken and sent to the laboratory?

Table 1. Sample containers and preservation of water samples (After AS/NZS 5667-1998)

Determinand	Type of container	Typical volume (mL)	Filling technique	Preservation	Maximum recommended holding time
Ammonia	Plastic or glass	500		Refrigerate	6 h
				Filter to 0.45um and refrigerate	24 h
BOD_5	Plastic or glass	1000	Do not pre-rinse, fill completely to exclude air	Refrigerate, store in dark	24 h
Copper	P(AW), G(AW)	100		Acidify with nitric acid to <pH 2	1 month
Chlorine residual	P or G	500		Keep out of direct sunlight	5 min
Nitrite-N	P or G	250		Immediate analysis	immediate
Phosphorus, dissolved	Plastic or glass	50		Filter to 0.45 um, refrigerate	24 h
Electrical conductivity	Plastic or glass	100	Fill completely to exclude air	None	24 h
				Refrigerate	1 month

8 Timing of Sample Dispatch

A water sample collected in Armidale on Monday afternoon, packed in an esky with an ice brick, cannot be dispatched until Tuesday, simply because there are no freight transfers out of Armidale on Mondays. By the time the sample leaves on Tuesday, 12 noon for road transport, or 5 pm for air dispatch to Sydney, the sample may be up to 24 h old and a further 24 h before receipt in the laboratory, usually about 10 am the following morning. Under these conditions the sample may not conform to AS/NZS 5667-1998 for its holding period. The alternative is to collect the sample on the same day as dispatch to conform to less than 24 h holding.

While some water samples may not vary significantly throughout the day, others can change quite rapidly depending upon the upstream events or in-house activities. Septic tank effluent may change considerably throughout the day because of the use of water in the house. Patterson (2003) shows a two-day measurement of pH from the outlet of a single dwelling septic tank, measurements taken at 15 minute intervals and matched against water use (Figure 1). A grab sample may represent the greater volume of water discharged from the tank if taken on one of the peaks or troughs of the curve. Numerous samples may be required, but more importantly, understanding the temporal variation will allow one to sample while avoiding the extremes. Time of sampling must be stated, or at least repetitive samples taken at the same time of the day. Perhaps we are measuring the wrong variable!

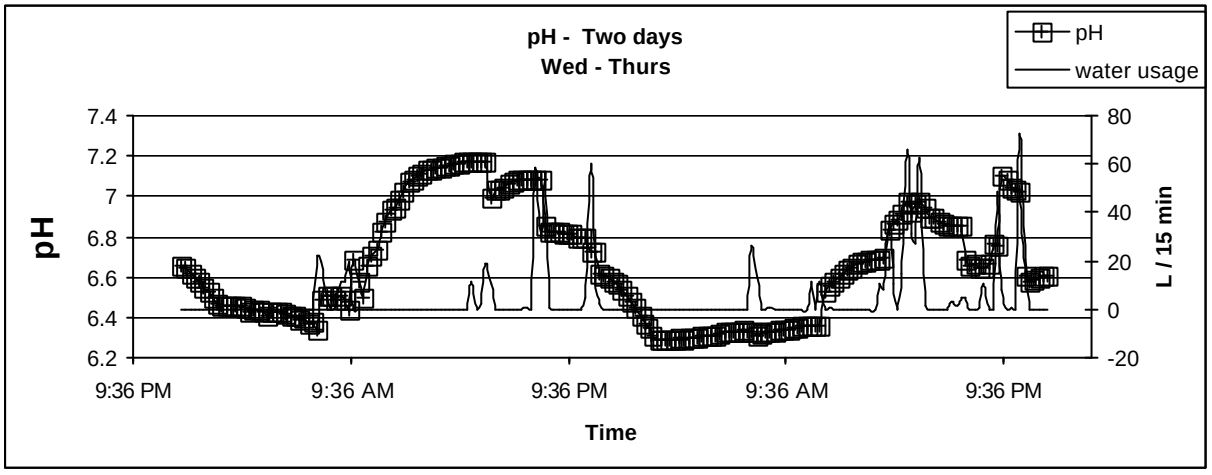

Figure 1 Daily pH variation in septic tank over two days: D-1 laundry, D-2 no laundry

9 Statistics

You do not have to be a statistician to realise that when you have more than one sample, the results may vary from sample to sample. The more samples you have, the wider may be the spread of sample results rather than the narrower the spread. Simple averages, standard deviations, coefficients of variation, confidence intervals are all measures of uncertainty. You are after some reasonably definitive value of your soil or water sample, therefore you need to consider sufficient samples to be able to perform a statistical assessment. It is important to consider that the volume in storage in Figure 1 exceeds 2 ML, yet the sample we take is usually one litre or less and the volume analysed by the laboratory may be only 1 mL. Therefore, the measure of uncertainty can be high. Cumulative errors can become significant unless some rationale is applied to their interpretation. However, if the sample collection was fouled, then the laboratory's quality system cannot rectify the problem. Again, perhaps we are measuring and reporting concentration when load (concentration times volume) is more valid.

10 Assessing Laboratory Results

Three considerations must be made when assessing laboratory results: (1) the method used and its detection limit; (2) units of measure; and (3) knowledge of the test and the relative meaning of the values reported. Table 2 shows the results from soil phosphorus tests for an ASPAC soil proficiency testing program. The numerical values have no correlation between tests and similarities between samples are not recognisable. The ranges shown are those values within which a laboratory would receive its certification (and satisfy NATA).

Table 2. Variation in extractable soil phosphorus (after ASPAC 2003)

Method	Units of measure	Soil 150	Soil 154
Colwell Extractable P	mg/kg	41.2 to 85.2	7.1 to 20.5
Olsen Extr. P	mg/kg	14.2 to 45.6	1.1 to 8.1
Bray Extr. P	mg/kg	32.0 to 58.6	1.2 to 3.8

A similar array of inter-laboratory variations can be seen in the results of tests performed by different NATA registered laboratories, as shown in Table 3. NATA (2003) suggested these differences can be accounted for by sample preparation, deterioration of sample between dispatch and analysis, operator error, and reporting error. So how do we interpret this variation? It simply means that sending a split sample to two NATA laboratories could derive two different values that have no correlation, even if the same test was used. The samples in Table 3 were identical but the laboratories were not told that when the samples were sent.

Table 3. Three bacteriological tests on identical samples (after NATA, Round 12 – 1998)

Test	Units	Sample 1	Sample 2
E. coli Membrane Filter Method (MF)	cfu/100 mL	14 to 66	16 to 64
Faecal coliform – Membrane Filter Method (MF)	cfu/100 mL	167 to 213	171 to 209
Total coliforms Most Probable Number (MPN)	cfu/100 mL	596 to 1118	589 to 1148

When comparing results or interpreting results based upon one's experience, that like tests are used for comparison. The most simple task of measuring pH could be done in two ways: (1) suspension of 1:5 soil/water (1:2 in New Zealand); or (2) suspension 1:5 soil/0.01M $CaCl_2$. The difference is that the former is usually 0.5 to 1.0 units higher than the latter. Unless the reader knows which test has been performed then interpretation is meaningless. Similarly, electrical conductivity can be measured in the saturated extract (EC_{se}) or in a 1:5 soil/water suspension ($EC_{1:5}$). The difference between the results from the two methods varies with soil texture and may lie somewhere between 5 and 8 fold difference, the latter being the lower. If you don't know the method –you don't know the result - you cannot assume!

11 Units of Measure

All results must be reported with their correct units of measure as well as the method used. The use of SI units avoids confusion, however, there are several units in general use for some analyses. Cations in soil may be reported in either mg/kg or cmol(+)/kg (same as old unit of meq/100g). It is the responsibility of the laboratory to report in correct units. For those who work with salinity issues, the units of electrical conductivity (EC) are either deciSiemens per metre (dS/m) or the older units (and smaller) microSiemens per centimetre (µS/cm) and never EC. There is no such unit as EC despite its use by government.

12 Nomenclature

Old terms take a long time to fade from use. Non-filterable residue (NFR) disappeared from Standard Methods in 1986 and has been replaced with total suspended solids (TSS), the weight of solids held on a glass fibre filter paper, reported in mg/L. The use of 'SS' for suspended solids should be avoided as it could be confused with settleable solids (SS).

13 CONCLUSION

There are numerous variations in sample collection, packaging, transportation and analysis that can confuse the real value of a sample, that is impinge upon the accuracy of the results. We must take all necessary steps to reduce these sources of error by having sampling protocols in place and following predetermined steps to regularly test these protocols. However, it is critical that regulators understand the difference in analytical methods, reporting units, likely ranges of possible results based upon principles of uncertainty. The cost of monitoring can be a significant impost upon a landowner. If the data are not important, don't measure them. If knowledge of a system can better be understood by more monitoring, perhaps a research project should be funded rather than imposing additional monitoring on individuals.

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