

# **Australian Soil Testing: quality assurance in measurement, interpretation and recommendation**

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## **ABSTRACT**

Particularly across the past 50 years, soil analysis has increasingly helped guide the assessment of soil health and the need for fertilisers. Despite many successes, the profession has been drawn into the science quality debate, due to variations in measurements and interpretations between laboratories. This paper reviews the past and present status of the soil testing profession, inclusive of efforts by the Australasian Soil and Plant Analysis Council Inc (ASPAC) to improve measurement performance and to assist with the interpretation of results. The presentation makes use of data from a contemporary survey of soil testing laboratories in Australia.

## **INTRODUCTION**

A prominent Australian rural newspaper *The Weekly Times*, in its Wednesday December 17, 2003 edition, led with the headlines "*Tests Soiled*". Across five pages in that edition, articles (and graphics) by journalist Peter Hunt used words such as "*One soil sample, 18 results; Farmers warned on soil test competency; Testing criticised; One soil sample, 19 results; and, Cut-price testing is proving far too costly*".

Those articles, and a subsequent series of short reports on soil laboratory accuracy and related topics by the Kondinin Group (Nugent 2004), were both a public service and a serious assault on a profession that has been working with limited resources for close to two decades to quantify and enhance the quality of laboratory data and to disseminate information on soil test interpretations and recommendations. Certainly, those who have come to rely on soil (and plant) chemical analysis for guidance on the need for fertilisers and amendments, and to monitor soil condition and trend, need to be assured that results they purchase are fit-for-purpose and capable of meaningful and transparent interpretation. Measurement uncertainty is ubiquitous, but needs to be held within acceptable limits.

This paper provides a little history, an update on contemporary soil testing in Australia, and perspectives on service quality. It takes an informed position, mentions initiatives of ASPAC, and outreaches beyond the measurement laboratory to include a call for transparency and rigour in soil test interpretations and recommendations. The paper includes selected findings from a comprehensive laboratory survey in Australia undertaken by the author in November 2003.

## **BUILDING THE BUSINESS**

Soil chemical testing in Australia had its genesis over 100 years ago, when Australia's natural diversity challenged early European settlers keen to exploit the land's potential for grazing,

cropping and other rural pursuits. State-based approaches to soil testing became entrenched as local knowledge of the link between particular soil tests and soil fertility expanded. Early calls from South Australia (Prescott and Piper 1928) for coordination between the various soil testing laboratories in Australia were mostly ignored in practice. Indeed, the profession in Australia retains a strong legacy from those formative years, reinforced by limited Australian and international agreement on preferred equipment and analytical methods.

The Queensland sugar industry pioneered in the 1930s a field-calibrated, advisory soil testing service (Kerr and von Stieglitz 1938), the remnants of which still operate to-day. It took a further 25 years for “modern” commercial soil testing to emerge. That service commenced in New South Wales in 1963 as an aid to the marketing of fertilisers. The aim was to offer a “speedy, accurate and moderately priced soil test to farmers” (Fogliati 1967). Concurrently, interest in soil test calibration studies accelerated, particularly in wheat cropping (e.g. Colwell 1963), in pasture lands (e.g. McLachlan 1965; Spencer *et al.* 1969) and for citrus (Craddock and Weir 1963). Commercial soil (and plant) testing services within the Australian fertiliser industry commenced in Queensland in 1967 and 1968, and in Western Australia for soils in 1971 (plants in 1975).

Those commercial initiatives and their associated publicity, which included well-attended public meetings and eminent guest speakers (e.g. Anon 1968), stimulated allied investment in government laboratories in the Australian states and Northern Territory. Soil testing was at the frontier of an exciting period of growth, opportunity and popularity: the golden years had arrived.

Soil testing laboratories took advantage of rapidly improving instrumentation that permitted semi-automation of soil tests such as phosphorus (P), chloride (Cl), nitrate ( $\text{NO}_3$ ), ammonium ( $\text{NH}_4$ ), sulfate ( $\text{SO}_4$ ), boron (B) and silicates (Si). Concurrently, flame emission and flame adsorption technology transformed and simplified the measurement of elements such as calcium (Ca), magnesium (Mg), sodium (Na), potassium (K), iron (Fe), copper (Cu), zinc (Zn) manganese (Mn) and other metals, allowing these to be easily incorporated into routine soil testing “packages”. Likewise, electrode technologies for pH, electrical conductivity (EC) and fluoride (F) advanced and became relatively cheap, effective and reliable.

Whereas the fertiliser industry largely pioneered soil test automation, government organisations, CSIRO and university researchers dominated in the areas of soil test method development and soil test calibrations, so essential in providing meaning for the measurements. Unfortunately, there has been a trend towards professional stagnation in these areas across the past two decades, largely due to limited research and development funding, coupled with pressure on laboratory managers to recover full operational costs. That said, tests for soluble silicate [mainly monosilicic acid,  $\text{Si}(\text{OH})_4$ ] are now available for sugarcane (Berthelsen *et al.* 1999; Hurney 1973), which was a Bureau of Sugar Experiment Stations (BSES) Ltd initiative. ASPAC has endorsed a new P sorption index test (Burkitt *et al.* 2002), and improved soil tests for extractable B,  $\text{SO}_4$  and cadmium (Cd) have been investigated with some success. Mehlich 3 (a universal soil test that has wide use in North America) is now operating in parts of Western Australia, and has also been cross-correlated with “conventional” soil tests in eastern Australia, including catchments that drain to the Great Barrier Reef.

While Australian scientists across 25 years from 1960 were innovative in adopting complex mathematical approaches to the calibration of soil chemical tests and fertiliser requirements (e.g. Colwell 1967, 1983), the calibrations typically took little or no account of off-site losses and harm to the downstream environment. Moreover, many of these correlations still guide present-day soil test recommendations.

Finally, attention to laboratory measurement quality was either ignored or was embryonic, until a systematic effort commenced in 1985 as the Australian Soil Quality Assurance Program

(Greenhill and Peverill 1988). Examples from that program (Table 1), which took no account of field sampling errors, highlighted the need for improvement. This need stimulated completion of the *Australian Handbook of Soil and Water Chemical Methods* (Rayment and Higginson 1992), the present “bible” for chemical soil testing in Australia. It also led to the formation of ASPAC in 1991.

**Table 1. Examples of expected uncertainty in the mid-1980s of results reported by soil testing laboratories in Australia and Papua New Guinea, based on data from up to 25 laboratories reported independently for 10 well-prepared Australian soils; data of Rayment (1988a,b).**

Details	Soil pH		Soil extractable P (mg P/kg)	
	1:5 soil / water (4A1) <sup>A</sup>	(1:5 soil / 0.01M calcium chloride (4B)	Colwell (9B)	Olsen (9C)
No. of laboratories	21	14	16	5
Expected value	6.3	5.6	26	12
Likely range of reported results (95% probability)	5.9–6.7	5.2–6.0	14–38	5–19

<sup>A</sup> Soil method codes are those of Rayment and Higginson (1992), which also contains references and full details of the methods.

## CONTEMPORARY SOIL TESTING

Table 2 provides summary details of present numbers of Australian laboratories known to engage in soil chemical analyses, at least to some degree. Annual average soil sample numbers (2001–2003), based on responses from a national survey in November 2003 (Rayment 2004) are included, noting that the survey attracted a 64% response (34 laboratories). Laboratories with only minor roles in “traditional” soil testing were the main defaulters.

Laboratories in public ownership included those of state departments (particularly New South Wales, Queensland, Victoria and Western Australia), public research organisations (e.g. CSIRO), and government funded universities. Private laboratories included but were not limited to major operations in Western Australia and Victoria linked to the fertiliser industry. To the extent possible, samples were allocated to the states where the samples were collected. Moreover, what is now BSES Ltd was classified as a laboratory in public ownership, as it only became a public company in June 2003.

Soil testing was the main business (from a laboratory perspective) of seven responders. Most soil samples were from Western Australia, Queensland, New South Wales and Victoria (in that order), while the ratio of soil samples analysed (includes extracts of soils) in commercial laboratories relative to public-funded laboratories averaged around 2.6:1 at the time of survey and has probably widened since then. Without question, commercial soil testing laboratories have market leadership in routine soil testing for diagnostic purposes. The quality of the product in the eyes of end users (and media) will undoubtedly impact on whether growth in sample numbers continues, stagnates or declines. Another factor is a perception by some that the fertiliser industry in Australia provides soil-testing services mainly to encourage the sale of fertilisers.

**Table 2. Summary details at November 2003 of the geographic distribution and ownership of soil-testing laboratories in Australia and the number of soil samples analysed by private and publicly funded laboratories for rural, natural resource management and related research purposes (annual average for years 2001, 2002 and 2003). The statistics are conservative.**

Location	No of soil laboratories (2003)		Annual average soil samples ('000) 2001–2003	
	Private	Public	Private	Public
ACT	No data	2	No data	17
NSW	4	5	46	17
NT	No data	1	No data	1
Qld	4	4	73	33
SA	1	3	13	6
Tas	1	1	6	5
Vic	2	2	42	17
WA	2	2	89 <sup>A</sup>	9

<sup>A</sup> Annual sample numbers received for analysis in WA (multiple states) in 2003–04 rose to ≈150,000 (G. Proudfoot, personal communication)

Nowadays, most of the larger soil-testing services offer a range of tests to “comprehensively” characterise the samples submitted for analysis. However, sub-sets of these tests, typically including soil pH, EC, Cl, NO<sub>3</sub>, extractable P and exchangeable K may be offered as a lower-cost option that covers the “basic” soil properties that are susceptible to short-term chemical changes. Table 3 outlines (as an example) a suite of contemporary chemical tests that might be used on surface soils from coastal southern Queensland to assess the fertility status and likely responsiveness to fertilisers for improved pastures, when little is known about the site. Follow-up soil sampling and analysis can be less comprehensive, or might focus on deeper segments of the soil profile to assess things such as sub-soil acidity, soil sulfur status, the presence of pyritic layers, the likelihood of Ca deficiency, and nutrient movement and accumulation.

For contaminated land, the British Standard (BS100175: 2001) states that confidence in the assessment can be achieved by increasing the number of samples taken and analysed, particularly because large differences in contamination can occur across relative small distances or depth (Thompson and Nathanail 2003). Similarly for soil fertility assessment, it is often better to analyse more samples by simple (or cheap) methods than fewer samples by more sophisticated (or costly) methods. This is exemplified by introduction of the Mehlich 3 universal test in Western Australia by the State Chemistry Centre, supported by in-field research (Bolland *et al.* 2003). Recent studies in eastern Australia comparing Mehlich 3 results with those of “traditional” soil chemical tests, could be precursors to the wider use in this country of this quick, relatively cheap, soil chemical test, noting that while it is far from perfect, it already has wide use on acidic and near-neutral soils in North America.

There has been little movement across the years on soil sampling patterns, sampling equipment, and sampling depth. Zigzag and grid patterns of sampling dominate, while guidelines warn of the need to ensure the operator’s hands and clothing and all equipment are free of relevant contamination. Unfortunately, uniformity across Australia on sampling depths is lacking due to historical pressure and an absence of incentives to take a national approach. In

Queensland, usual sampling depths are 0–100 mm for most field crops and pastures, 0–250 mm for assessing the P and K (and silicate) fertility of sugar cane soils, 0–150 mm for horticultural and tree crops, and sampling to 600 or 900 mm for NO<sub>3</sub>, SO<sub>4</sub> and salinity assessment. Samplers are warned to avoid recent fertiliser applications because errors are likely to be greater due to incomplete reaction of some fertilisers with the soil. The time between sampling and laboratory processing commonly takes one to three weeks, although this varies widely at the upper end and is influenced by the types of tests undertaken.

**Table 3. A contemporary, comprehensive suite of soil chemical tests (with method codes<sup>A</sup>) suited to assessing the soil fertility status and likely responsiveness to fertilisers of improved pastures in southern Queensland on soils (0–100 mm) known to be unaffected by pyritic minerals.**

Common	Optional to desirable
EC (3A1), pH (4A) Chloride – 1:5 soil/water extract (5A) Water soluble nitrate (7B) Bicarbonate-extractable P – Colwell (9B); Acid-extractable P (9G) Calcium phosphate-extractable S (10B) Exchangeable bases (Ca <sup>2+</sup> , Mg <sup>2+</sup> , Na <sup>+</sup> , K <sup>+</sup> ) - 1M ammonium chloride at pH 7.0 (15A); Exchange acidity (hydrogen and aluminium) by 1M potassium chloride (15G1) if soil is strongly acidic; Effective cation exchange capacity, ECEC (15J1) DTPA-extractable Cu, Mn, Zn and Fe (12A1); Calcium chloride extractable B (12C1) Lime requirement – Mehlich single buffer (16C1)	pH of 1:5 soil/0.01M calcium chloride (4B) Phosphate sorption index (PSI) Organic carbon – W & B (6A1)

<sup>A</sup> Soil method codes are those of Rayment and Higginson (1992)

Most laboratories prefer samples in the vicinity of 0.5 to 1.0 kg. Accordingly, soil cores / clods usually need to be downsized and well mixed in the field, which is a possible source of unquantified error. Protocols to avoid or minimise chemical transformations due to oxidation and biological activity between sampling and receipt at the laboratory typically recommend rapid transport and/or keeping the samples well sealed and cool, but the extent to which these guidelines are followed in commercial practice is elusive.

The laboratory survey revealed that 40°C was (correctly) the most common soil drying temperature for unprocessed samples submitted to assess soil fertility. A few laboratories claimed to dry at 30°C, 35°C, between 35–40°C, at 45°C and from 40–60°C, while one laboratory reported drying soils at up to 300°C for some unknown reason. In addition, several laboratories mentioned drying at 100, 105 and 110°C for total elemental analysis. While there is need for greater uniformity in sample drying prior to soil chemical testing, there is no national initiative at present to overcome the diversity apparent from the survey. Implementation costs would be mostly quite low.

## QUESTIONS OF QUALITY

### Interlaboratory Proficiency Programs

ASPAC was established to represent individuals, laboratories and organisations with a special interest in soil and plant analysis (doing and using). Arguably, one of ASPAC's most significant contributions has been its interlaboratory proficiency programs and related efforts to improve measurement performance. So far, ASPAC has completed eight discrete interlaboratory proficiency programs for soils (1993, 1995, 1997, 1998, 1999, 2000, 2001–2, 2003), and a 2004 soil proficiency program is current. The new program now operates at a frequency of three soil “rounds” annually, each of four samples, which is superior to times past (Rayment *et al.* 2000), when the maximum frequency was one “round” annually of six samples.

On all occasions, participants of annual programs received comprehensive data compilations incorporating statistical evaluations (e.g. Maheswaran and Peverill 1994; Johnstone *et al.* 2003). They also received a *Certificate of Certification* signed by ASPAC's Chairperson and the Convenor of the ASPAC Laboratory Proficiency Committee. These certificates contained the laboratory name and the methods for which satisfactory performances were confirmed. Apart from these certificates, high levels of confidentiality applied, with numbers used to identify laboratories in the published reports. Laboratory confidentiality was also applied when data from the various programs were reviewed nationally and internationally (e.g. Rayment *et al.* 2000; Rayment, in press).

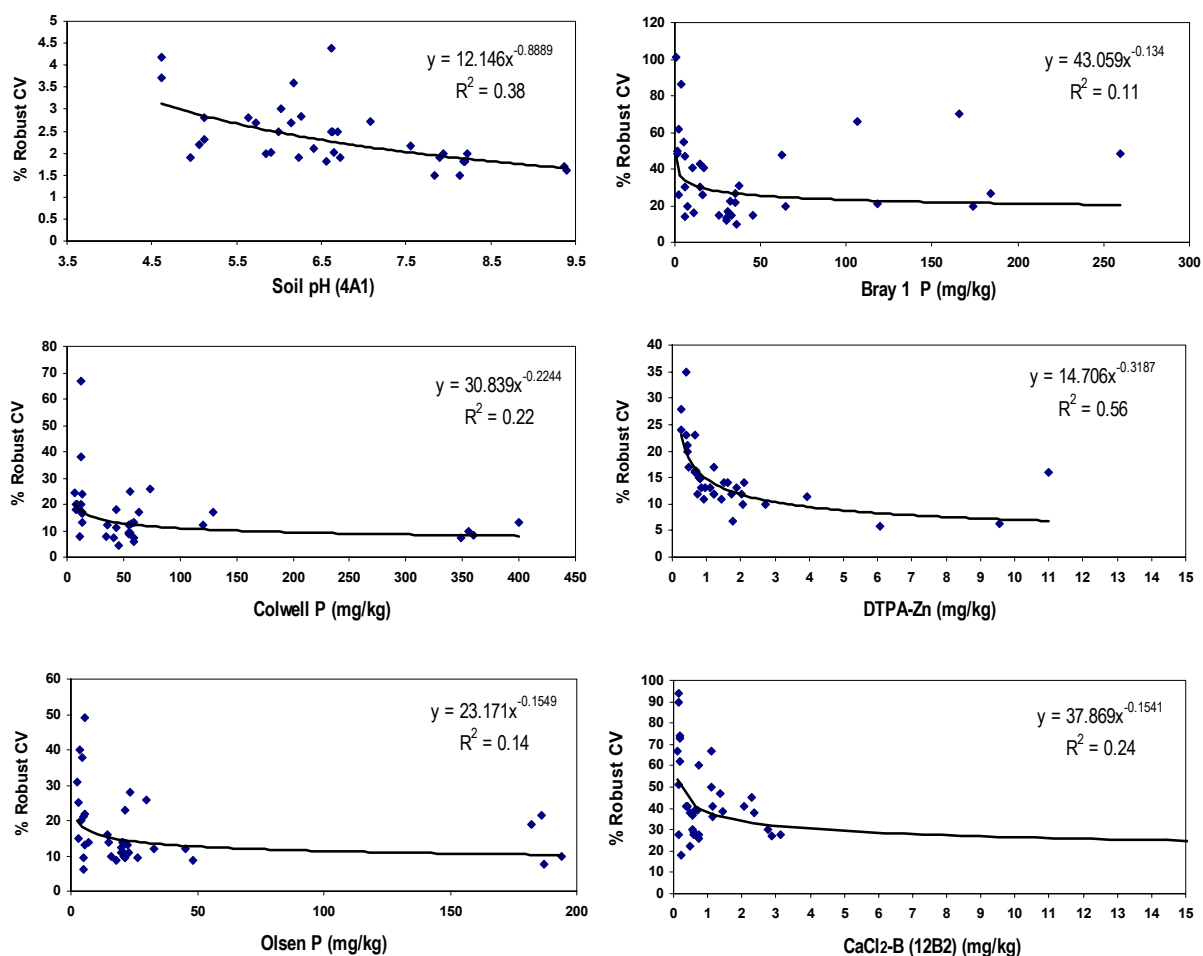
The Horwitz equation [ $CV\% = \pm 2^{(1-0.5 \log C)}$ , where CV is the coefficient of variation and C is the concentration expressed as an exponent of 10] (Horwitz, 1982a,b) provides a mathematical expression of the expected variation in test measurements obtained from interlaboratory studies involving all sorts of analyses from extremely low concentrations to 100%. Typically, %CVs increase rapidly and in a predictable way as analyte concentrations approach trace to ultra-trace concentrations, when performed by experienced analysts using well-tested methods. Indeed, concentrations of 10,000, 100, 1, 0.1, and 0.01 mg/kg have predicted %CVs of 4%, 8%, 16%, 23% and 32%, respectively. It follows that the Horwitz equation provides laboratory managers and end users with an independent guide to expected analytical measurement performance.

This form of benchmarking has been undertaken by the profession in recent years, using results from ASPAC's interlaboratory proficiency programs as well as international datasets (e.g. Rayment *et al.* 2000; Rayment and Peverill 2002). In most cases %CVs for annual soil test data for particular methods were higher than those predicted as typical by the Horwitz equation at corresponding analyte concentrations. Moreover, there has been little sign of collective improvement across the decade, despite occasional training workshops and offers of assistance. Unfortunately, program participants from South-East Asia and the Pacific Islands have not had the resources to attend these workshops, even though they contribute to the Australian public record of measurement performance.

Despite the above, the ASPAC interlaboratory proficiency program datasets are now large enough to provide insights into how soil test results tend to vary with concentrations across laboratories on a method-by-method basis. Fig. 1 provides examples relevant to soil test methods used widely in Australia for routine soil testing purposes.

The scatter of data in Fig. 1 is quite wide, and while the associated trend lines are not all significant at the 5% level, the plots do provide insights into the level of measurement uncertainty that can be expected in the real world. For example, the Olsen-P and Colwell-P soil tests have similar uncertainty, which is less than the uncertainty associated with the Bray 1-P test at concentrations <30 mg P/kg. At 100 mg P/kg, the Horwitz equation anticipates a CV of

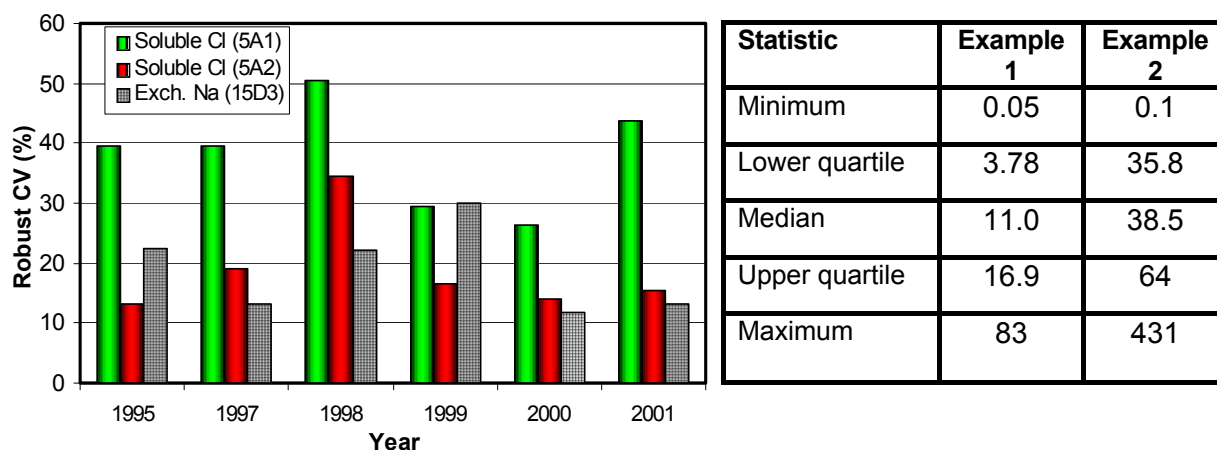
8%, whereas predictions from the equations in Fig. 1 suggest values of 11%, 11.3% and 9.9% for Colwell, Olsen and Bray 1-P, respectively. Most analytical improvement needs to occur at extractable P concentrations <100 mg/kg and particularly <50 mg/kg.



**Figure 1.** Continuous relationships between percentage coefficients of variation (robust) and concentrations for a selection of routine soil chemical tests obtained from ASPAC interlaboratory proficiency programs from 1997 to 2003. Soil methods and method codes are those of Rayment and Higginson (1992). Two data points for soil boron method 12B2 are not shown because of their high concentrations (41.7 and 52 mg B/kg).

Fig. 1 also provides an alert on two micronutrient soil tests, namely DTPA-Zn and hot CaCl<sub>2</sub> extractable B. Clearly, the variance in measurements between laboratories is much more predictable and smaller for DTPA Zn than it is for the soil B test, which is 2.5 times more variable at 1 mg/kg concentration. Rayment (in press) and Rayment and Lyons (2004) have already reported on the need to improve the measurement of salinity in Australasia (see Fig. 2).

The Kondinin Group (Nugent 2004) recently published analytical “performance scores” for up to six soil tests across six laboratories that operate in southern Australia. While the rigour of their assessment protocols and assumption that the “reference laboratory” always provided accurate results could be questioned, the 2003 Kondinin Group’s survey does reveal that measurement differences sufficient to affect recommendations for fertilisers and amendments are occurring in day-to-day practice.



**Figure 2. Robust coefficients of variation as indicators of the Australasian performance of laboratory methods for soil salinity used to support Australia’s National Action Plan for Salinity and Water Quality. The accompanying table provides two examples from 2001 for the potentiometric titration method (5A1) for water-soluble soil chloride, based on results (mg Cl/kg air dry soil) from 11 laboratories.**

### Other Measurement Quality Indicators

From the November 2003 questionnaire of the author’s, it was established that all Australian soil-testing laboratories use control charts or equivalent internal quality control procedures on a routine basis, which is good laboratory management practice. In addition, 68% of responders claimed they routinely complied with intra- and inter-state regulations pertinent to the movement of soil samples in Australia, which are designed to prevent the accidental movement of pests and diseases. Biosecurity compliance needs to rise to 100% without delay.

The November 2003 questionnaire established that seven “traditional” soil testing laboratories had National Association of Testing Authorities (NATA) accreditation, with one claiming their accreditation covered 65 tests. Two laboratories indicated that NATA accreditation was “in progress”, while 12 laboratories reported they were not NATA accredited for soil chemical tests. In addition, 65% of all Australian soil testing laboratories had ASPAC certification for one or more methods but only five Australian laboratories (16%) reported certification in that year for all methods submitted for assessment. This is low, and makes one wonder if ASPAC’s policies contributed to this outcome.

### Measurement Enhancement

Perhaps the high level of confidentiality applied by ASPAC to laboratories in its proficiency programs has been partly responsible for the slow rate of analytical improvement observed in those programs. Others do it differently, such as South Africa’s AgriLASA (Agricultural Laboratory Association of South Africa). This group publishes regular summaries of laboratory analytical performance in agricultural and associated press. AgriLASA also encourages potential users of analytical services to refer to this information. In Australia, the National Cadmium Management Committee is providing information on Australasian laboratories shown by ASPAC to be proficient at measuring Cd in plant materials, which is a step in the right direction. In the United States, relevant data from North American Interlaboratory Proficiency Testing are supplied to those state governments who monitor the performance of soil testing laboratories. For example, in the State of Idaho, where the

addition of farmyard manure to land containing >40 mg/kg of Olsen-extractable P in surface soil is not permitted, poor performing soil testing laboratories are asked by government regulators to exit the industry.

In the absence of any enforcement process, ASPAC placed the onus on participating laboratories to request technical assistance / support, typically through state representatives and/or members of ASPAC's Laboratory Proficiency Committee. Few laboratory managers have accepted this sustained offer of support. Consequently, the main opportunity to improve measurement performance, other than self-motivation within the laboratory, was via attendance at occasional ASPAC-arranged analytical workshops.

Arguably, proficiency certificates issued by ASPAC to laboratories on a program-by-program basis (to record the methods for which satisfactory performance was achieved) have contributed to the present situation. This "reward" only mentions successes and not failures (if any). ASPAC is presently reviewing its protocols for issuing proficiency certificates, and has already acted to increase the frequency of its interlaboratory proficiency programs from annual to three times each year, with double the number of samples in that period. Secondary reference materials are also made available at low cost to member laboratories.

Attitudinal change by laboratory staff is another area warranting attention. In particular, a culture that promotes continuous improvement needs to be encouraged, implemented and periodically reviewed. New equipment and new ideas may need to be introduced, and reinforced by relevant competency training. Rewards at the team level that recognise and reinforce positive actions and initiatives warrant serious consideration but they may not always achieve their purpose if equipment and in-service training is neglected. Interestingly, staff in a large soil-testing laboratory in New Zealand have named all their key analytical instruments: they treat them as vital members of their team.

## INTERPRETATIONS AND RECOMMENDATIONS

Interpretations and recommendations are areas of soil testing that have escaped detailed scrutiny in the quest for quality, yet they have a major bearing on the outcome. Indeed, there is a persuasive case to require these steps to be as rigorous and transparent as soil sampling and laboratory measurements. The view of many that local agronomists are generally in the best position to interpret soil test results and make recommendations is only supported when the agronomist has an in-depth awareness of the science that associates the particular test or tests with likely responsiveness and the likelihood or otherwise of environmental harm.

As indicated, "tradition" and local experience have influenced which soil tests and soil-sampling depths have proven to be the most useful in a given region. However, much of the science that underpins relationships between particular soil tests and crop and pasture responses to fertilisers and amendments are many years old. As such, they often involve responses on soils that had received little fertiliser, while varieties and forms of fertiliser may have changed. Nowadays, much greater attention needs to be taken to limit opportunities for off-site losses. This implies that fertilisers (and organic by-products) will not be used at rates well in excess of requirements, or applied to sites with large reserves of plant-available nutrients. That is, mathematical functions used to describe a fertiliser response "surface" or used to derive a soil critical value or range, should optimise in favour of the lowest reasonable values.

When experimental design permits, appropriate analysis of variance will provide information on the significance of any apparent yield trends and interactions. Quite commonly there may be no

significant difference between two or more of the fertiliser rates employed, even though yields may appear to increase slightly as fertiliser rates for nutrients such as nitrogen increase.

It has been common in Australia to derive soil critical values corresponding to the yield or relative yield asymptote or something like 95% of the maximum, although economics and other externalities might moderate the recommended rate to some extent. The goal should be something like “the lowest possible fertiliser rate or soil test critical level/range to just achieve maximum yield and/or profit and/or use of capital”.

Linear, quadratic, square-root quadratic and similar least-squares models have found wide use because they are “easy to calculate”, but often have significant bias at key points on the response surface. For example, the quadratic equation often overestimates the measured yield at zero rate and the first few rates beyond the control, whereas the Mitscherlich equation (e.g.  $Y = B_0 + B_1 e^{-B_2 X}$ ) may overestimate maximum yield (or the soil critical value) when the response to fertiliser is gradual. Also, the Mitscherlich equation is inappropriate when yields or relative yields decline sharply beyond the maximum.

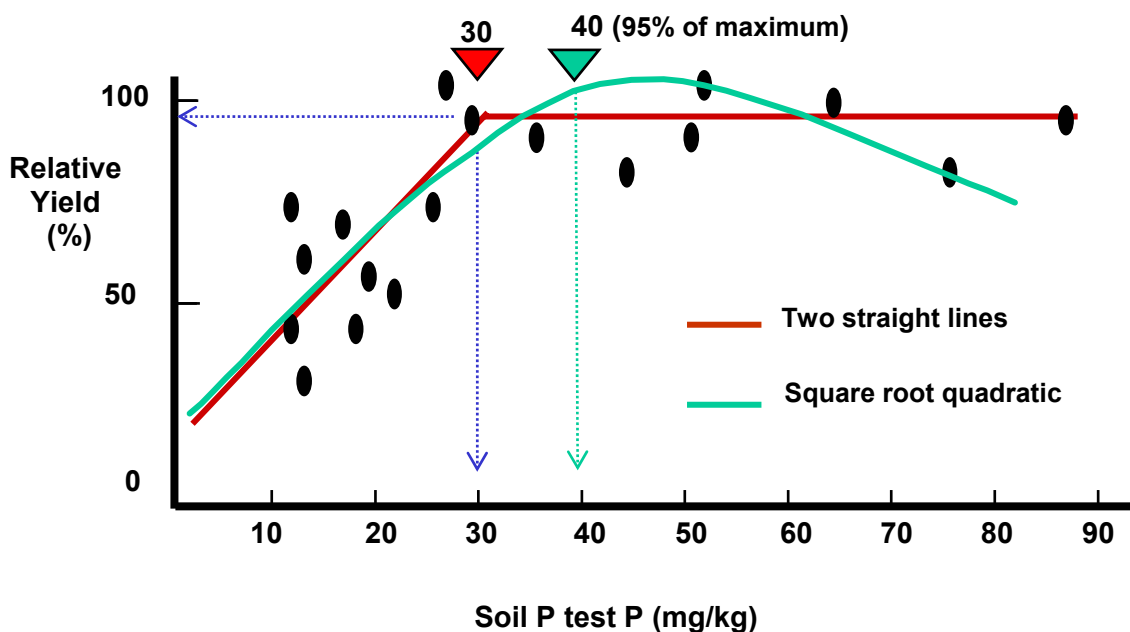
Often a superior option is the Linear Response and Plateau Model (LRP Model), which relates to Liebig’s law of constant returns. Variants include the Two Intersecting Straight Lines Model (TISL Model), the Non-rectangular Hyperbola Model and the Powered Inverse Quadratic Model at its limiting case (Sparrow 1979; Rayment 1985). Typically, such models provide lower estimates (with at least equal confidence) of the optimum fertiliser rate or critical level than those corresponding to 90–95% of the maximum derived from quadratic and related models (see Fig. 3) or from common forms of the Mitscherlich relationship. Moreover, the LRP and TISL models provide an estimate of error at the critical point of intersection. The qualification is that there must be at least five and six data sets, respectively, as the LRP Model has three parameters, while the TISL Model has four parameters.

The author is unaware of any published Australian study where soil samples go to competing laboratories for analysis and fertiliser recommendation, and those recommendations are then adopted and subsequent crop performance assessed under field conditions. This has been done in western Canada by Karamanos and Cannon (2002). These authors used six laboratories and recorded recommendations from each for two crops (wheat and canola) and four locations. They also reported for wheat the various yields, protein and revenue less fertiliser input expenses, achieved for each recommendation. For canola they measured and reported yield, protein, oil content and the same economic data. The recommended N fertiliser application rates for wheat varied from approximately 50–110 kg/ha at one site, from 55–129 at a second site, 0–70 at a third site, and 28–120 at a fourth site. Corresponding recommendations for P fertiliser were approximately 20–30, 20–24, 30–40, and 0–20 kg/ha. Moreover, significant differences in wheat yields were subsequently recorded at three of the four sites. These differences combine sampling, laboratory and interpretative error, in addition to the effects of agronomy and weather on the final outcome.

In the late 1960s and early 1970s, the profession experienced a period when a major provider treated their soil test interpretations and recommendations as commercial-in-confidence. Growers were both confused and suspicious when competing services provided different recommendations for the same crop and paddock. Following rationalisation, policies changed and secrecy was removed from the process of interpretation – recommendation. Moreover, there was close collaboration between research providers and representatives of soil testing laboratories in the development of “best-bet” soil test interpretation charts.

This era of collaboration, transparency and agreement on “best-bet” recommendations continued for several years, but now seems to have withered, partly because good information on soil test interpretation in Australia is available (Pevevill *et al.* 1999), and new

commercial arrangements now operate in this area. Computer-aided packages such as *NU logic* and *Nutrient Advantage* are examples. Despite this, a sound case can be made to reinvigorate the consultation – participative process regarding soil test interpretations and recommendations, to ensure the best possible soil test interpretations are operating in the public and private sectors, particularly in nutrient-sensitive areas such as catchments that drain into the Great Barrier Reef World Heritage Area.



**Figure 3.** Hypothetical examples of the influence of model on soil test “critical” level from the same data set.

Ultimately, it is just as valid to corroborate the “robustness” and quality of the derivation of fertiliser recommendations from particular soil test levels or ranges, as it is to monitor the measurement performance of soil (and plant) testing laboratories operating in the same marketplace. Both influence the eventual outcome. The challenge is to ensure such studies are adequately resourced and peer reviewed.

A small committee of Rayment, Lyons and Shelley is presently developing a new Australian Handbook of Soil Chemical Methods. While the handbook will be considerably larger than the Rayment and Higginson (1992) handbook it supersedes, it is likely only to guide rather than to recommend the suitability of methods for particular purposes. The developers envisage that informed panels of soil chemists, agronomists and commercial interests would make such decisions, perhaps coordinated by ASPAC. Ideally, there would be at least three mega-regional panels, one covering subtropical and tropical Australia, a second dealing with Southern Central and South-Eastern Australia, and a third dealing with the bottom half of Western Australia. It is further envisaged that these panels would overview the interpretative criteria and generic recommendations for fertiliser for a range of cropping and pastoral situations, based on the recommended methods of analysis. Annual or two-yearly meetings are suggested, to ensure that new knowledge and experience is incorporated with minimum delay. Major laboratories, end users, agri-business and possibly R&D Corporations may be willing to contribute funds to allow these panels to operate, initially for a five-year trial period. This type of information could then feed into the *FERTCARE* accreditation process.

## CHOOSING THE RIGHT LABORATORY

Loyalty, speed of service, cost, the attractiveness of reports, proximity and whether or not an interpretative service is available are reasons why clients often choose one soil testing service in preference to another. Thompson and Nathanail (2003) recorded six basic principles for a testing laboratory that warrant attention by potential clients. These are:

- i. Analytical measurements should be made to satisfy an agreed requirement.
- ii. Analytical measurements should be made using methods and equipment that have been tested to ensure they are fit-for-purpose.
- iii. Staff making analytical measurements should be both qualified and competent to undertake the task.
- iv. There should be regular independent assessment of the technical performance of the laboratory.
- v. Analytical measurements made at one location should be consistent with those made elsewhere.
- vi. Organisations making analytical measurements should have well-defined quality control and quality assurance procedures.

Ideally, the client should specify what tests are required on the samples they submit for analysis, but laboratories may simplify the decision by offering different suites of analyses at specified prices, perhaps with batch-size discounts.

The ASPAC web site provides local guidance on how to choose a laboratory that can be expected to give good results<sup>1</sup>. A copy of their advice is provided as Appendix 2. Certainly, questions such as how did the laboratory perform in ASPAC's interlaboratory programs should be asked, particularly for the most important tests. The ability of the laboratory or another reliable provider to interpret the test results is a further consideration. In addition, laboratories that play little or no heed to the biosecurity of the samples they receive and process should be avoided.

The Kondinin Group (Nugent 2004) suggests giving the laboratories "the third degree". Knowing the right questions to ask will help farmers make informed decisions about fertiliser needs. Their checklist includes: is the laboratory NATA accredited; does the laboratory participate in proficiency programs and if so, did it perform well; does the laboratory run 'check' samples; and who else uses that laboratory, such as universities and other research organisations? The Group also suggests asking for tests to be repeated if results are in doubt.

Benefit-cost is another question, and a 'driver' of change. Many would argue that soil testing is too expensive and certainly prices for tests vary among laboratories. Laboratory automation has helped achieve operational efficiencies, but Australian laboratories have been slow to trial and adopt "universal" extractants such as Mehlich 3 in favour of "traditional" soil chemical tests. This test is now offered in Western Australia, and there are sufficient comparative data now emerging to suggest wider use in other parts of Australia is possible in the short term. While some interpretative adjustments will need to be made, present

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<sup>1</sup> <http://www.aspac-australasia.com/aspac/jsp/choose.jsp>

indications are that test results for Mehlich 3 are likely to be more repeatable within and across laboratories than is the case for several "traditional" tests. The future is in the hands of laboratory managers, agronomists and end users.

## REFERENCES

- Anon. (1968). *Proceedings of the A.C.F. Soil Testing Seminar*. Lennons Hotel, Toowoomba, 16 August 1968. (Papers by D. Hauck, J.S. Russell, E.J. Britten, J.K. Leslie, R.J. Esdaile, and N.J. King).
- Aitken, R.L., Moody, P.W. and Dickson, T. (1995). Field calibration of lime requirement soil tests. In *Proc. 3rd Int. Symp. on Plant/Soil Interactions at Low pH*. Ed. D. Date, N.J. Grundon, G.E. Rayment and M.E. Probert, pp 479-84. (Kluwer Academic Publishers, The Netherlands.)
- Berthelsen, S. Noble, A.D. and Garside, A.L. (1999). An assessment of soil and plant silicon levels in north Queensland. *Proceedings of the Australian Society of Sugar Cane Technologists* **21**, 92-100.
- Bolland, M.D.A., Allen, D.G. and Walton, K.S. (2003). Soil testing for phosphorus: comparing the Mehlich 3 and Colwell procedures for soils of south-western Australia. *Australian Journal of Soil Research* **41**, 1185-1200.
- Burkitt, L.L., Moody, P.W., Gourley, C.J.P. and Hannah, M.C. (2002). A simple phosphorus buffering index for Australian soils. *Australian Journal of Soil Research* **40**, 497-513.
- Colwell, J.D. (1963). The estimation of the phosphorus fertilizer requirements of wheat in southern New South Wales by soil analysis. *Australian Journal of Experimental Agriculture and Animal Husbandry* **3**, 190-198.
- Colwell, J.D. (1967). Calibration and assessment of soil tests for estimating fertilizer requirements. I. Statistical models and tests of significance. *Australian Journal of Soil Research* **5**, 275-293.
- Colwell, J.D. (1983). Fertilizer requirements. In: *Soils: an Australian Viewpoint*, Division of Soils, CSIRO, pp 795-815. (CSIRO: Melbourne / Academic Press: London.)
- Craddock, F.W. and Weir, R.G. (1963). Citrus leaf analysis. *The Agricultural Gazette of New South Wales* **74**, 1425-1431.
- Fogliati, G. (1967). The commercial adaptation of soil testing. *The Journal of the Australian Institute of Agricultural Science*, December 1967, pp 336-339.
- Greenhill, N.B. and Peverill, K.I. (Editors). (1988). *Standardisation of Methods of Soil Analysis*. (Department of Agriculture and Rural Affairs Technical Report, Melbourne.)
- Horwitz, W. (1982a). Evaluation of analytical methods used for regulation. *Journal of the Association of Official Analytical Chemists* **65**, 525-530.
- Horwitz, W. (1982b). The problems of utilizing trace analysis in regulatory analytical chemistry. *Chemistry in Australia* **49**, 56-63.
- Hurney, A.P. (1973). A progress report on calcium silicate investigations. *Proceedings of the Queensland Society of Sugar Cane Technologists* **40**, 109-113.
- Johnstone, P., Shelley, B. and Kitching, M. (2003). *Soil Proficiency Testing Program Report 2003*. 231 pp. (ASPAC, c/- State Chemistry Laboratory: Victoria, October 2003.)
- Karamanos, R.E. and Cannon, K.R. (2002). Virtual soil testing: is it possible? *Communications in Soil Science and Plant Analysis* **33**, Nos. 15-18, 2599-2616.

- Kerr, H.W. and von Stieglitz, C.R. (1938). The laboratory determination of soil fertility. Technical Communication No. 9, Bureau of Sugar Experiment Stations, Queensland.
- Kingston, G. (1999). A role for silicon, nitrogen and reduced bulk density in yield responses to sugar mill ash and filter mud/ash mixtures. *Proceedings of the Australian Society of Sugar Cane Technologists* **21**, 114-121.
- Maheswaran, J. and Peverill, K. (1994). National Plant Quality Assurance Program Report 1994. (ASPAC, State Chemistry Laboratory, Victoria.)
- McLachlan, K.D. (1965). The nature of available phosphorus in some acid pasture soils and a comparison of estimating procedures. *Australian Journal of Experimental Agriculture and Animal Husbandry* **5**, 125-132.
- Nugent, T. (2004). Survey digs up the dirt on lab accuracy (and related articles). *Farming Ahead* No. 148, May 2004, pp 22-35.
- Peverill, K.I., Sparrow, L.A. and Reuter, D.J. (Editors). (1999). *Soil Analysis: an interpretation manual*. 369 +18 pp. (CSIRO Publishing, Collingwood, Australia.)
- Prescott, J.A. and Piper, C.S. (1928). Methods for the examination of soils. Pamphlet No. 8. Council for Scientific and Industrial Research, Commonwealth of Australia, Melbourne. 52 pp.
- Rayment, G.E. (1985). Calibration and interpretation of soil chemical analyses. In: *Identification of Soils and Interpretation of Soil Data*. pp 81-101. (ASSSI, Queensland Branch, Brisbane.)
- Rayment, G.E. (1988a). Report on soil pH data – Australian Soil Quality Assurance Program, 1985. In: *Standardisation of Methods of Soil Analysis*. Eds. N.B Greenhill and K.I Peverill. 19 pp. (Department of Agriculture and Rural Affairs, East Melbourne.)
- Rayment, G.E. (1988b). Report on soil phosphorus data - Australian Soil Quality Assurance Program, 1985. In: *Standardisation of Methods of Soil Analysis*. Eds. N.B Greenhill and K.I Peverill. 17 pp. (Department of Agriculture and Rural Affairs, East Melbourne.)
- Rayment, G.E. (in press). Statistical aspects of soil and plant test calibration. *Communications in Soil Science and Plant Analysis*.
- Rayment, G.E. (2004). Soil and plant testing in Australia – the players, clients, methods and issues. In: Proceedings of the Workshop “Tools for Nutrient and Pollutant Management - applications to agriculture and environmental quality”. (Eds: L.D. Currie and J.A. Hanly). pp. 9-23. Fertilizer & Lime Research Centre (in conjunction with ASPAC) Occasional Report No. 17 (ISSN 0112-9902), Massey University, New Zealand.)
- Rayment, G.E. and Higginson, F.R. (1992). *Australian Laboratory Handbook of Soil and Water Chemical Methods*. 330 pp. (Reed International Books Australia P/L, trading as Inkata Press, Port Melbourne.)
- Rayment, G.E. and Lyons, D.J. (2004). Australasian performance of laboratory methods for soil and plant salinity. In: “Salinity under the sun – investing in the prevention and rehabilitation of saline lands in Australia”. Proceedings of 9th PUR&L National Conference – 29th September - 2nd October, 2003, Yeppoon. Theme 1 (Sharing a common understanding) poster paper, 3 pp. (CD ROM). ISBN 1 920920 31 5. QNRME 04090.
- Rayment, G.E., Miller, R.O. and Sulaeman, E. (2000). Proficiency testing and other interactive measures to enhance analytical quality in soil and plant laboratories. *Communications in Soil Science and Plant Analysis*. **31**, 1513-1530.
- Rayment, G.E. and Peverill, K.I. (2002). Proficiency testing for soil and plant analysis in Australasia. *Communications in Soil Science and Plant Analysis* **33**, Nos. 15-18, 2441-2455.

Sparrow, P.E. (1979). Nitrogen Response Curves of Spring Barley. *Journal of Agricultural Science, Cambridge* **92**, 307-317.

Spencer, K., Bouma, D. and Moye, D.V. (1969). Assessment of the phosphorus and sulfur status of subterranean clover pastures. 2. Soil tests. *Australian Journal of Experimental Agriculture and Animal Husbandry* **9**, 320-328.

Thompson, K.C. and Nathanail, C.P. (2003). The risk assessor as the customer. Ch. 1 In: *Chemical Analysis of Contaminated Land*. Eds. K.C. Thompson and C.P. Nathanail. 290+15 pp. (Blackwell Publishing, CRC Press, Oxford.)

## APPENDIX 1

**Various soil methods and soil method codes described by Rayment and Higginson (1992), together with brief comments on recommended applications of those methods. Refer to Rayment and Higginson (1992) for more details on these and other methods.**

<i>Soil method</i>	<i>Code</i>	<i>Comment</i>
EC of 1:5 soil/water extract	3A1	Suited to use on all soils, irrespective of whether acidic or alkaline. May underestimate the soluble salt status of soils with high natural gypsum content (>1%). Such soils could have an EC of about 2 dS/m.
pH of 1:5 soil/water suspension	4A	Suited to use on all soils, irrespective of whether acidic or alkaline. Values may be lower than expected if the soil was fertilised prior to sampling, as this typically causes a rise in the ionic strength of the soil solution.
pH of 1:5 soil/0.01M calcium chloride extract	4B	Suited to use on all soils, irrespective of whether acidic or alkaline. Values are usually unaffected if the soil was fertilised prior to sampling, as the soil's ionic strength is masked by the ionic strength of the calcium chloride solution. This soil pH test is popular in southern Australia, particularly Victoria. There is value in using both water and calcium chloride to measure soil pH.
pH of 1:5 soil/1M potassium chloride extract	4C1	Suited to use on all soils, irrespective of whether acidic or alkaline, but mainly used in the sugar industry and to estimate $\Delta$ pH.
pH of sodium fluoride suspension	4D1	A quick test to indicate the presence of active aluminium in the soil. The test is positive if there is a pH elevation, indicating the release of hydroxyl ions.
pH of hydrogen peroxide extract	4E1	A pyritic soil can be inferred if the soil pH drops to around 3 or lower following oxidation with 30% hydrogen peroxide. The test would usually be specified if the sampling site was < 3 AHD and there was evidence of prior inundation by sea water.
$\Delta$ pH	4F1	When $\Delta$ pH [pH (KCl) – pH (H <sub>2</sub> O)] is positive to zero, the soil colloids are likely to be dominated by variably charged surfaces.
Chloride - 1:5 soil/water extract	5A	Suited to use on all soils and should be specified if soil salinity is a possibility.
Organic carbon - W & B	6A1	This simply-performed measure of soil organic carbon usually yields a lower figure than the true total carbon value. Nowadays it is less preferred than method 6B.
Total organic carbon - high frequency induction furnace	6B3	The preferred method for total organic carbon in soils.
Total nitrogen	7A	An essential test if there is need to report the soil's C:N ratio. Also essential in mass-balance studies involving N.
Water soluble nitrate - automated colour	7B1	This test is environmentally important and may correlate with the responsiveness of the site to N fertiliser, such as for wheat on the Darling Downs. In some highly weathered soils with a measurable anion exchange capacity, water does not extract all of the nitrate-N that may be present. Such soils exist in the Wet Tropics of Queensland.
Mineral nitrogen with 2M KCl, - steam distillation	7C1	The most accurate (but slow and tedious) procedure for measuring the various components of mineral-N present in the sample. Preferred for research studies involving soil N.

<b>Soil method</b>	<b>Code</b>	<b>Comment</b>
Total organic carbon/total nitrogen ratio	8A1	A commonly used ratio that can only be calculated if total C and total N data are available.
Total phosphorus	9A	Provides a measure of the soil's total P status to the depth sampled. Used in mass-balance studies. Typically has little bearing on whether or not the site will respond to P fertiliser.
Bicarbonate-extractable phosphorus (Colwell)	9B	Preferred for most crops and pastures when there is need to separate responsive from non-responsive soils to P fertiliser in most Australian States, including Queensland. Favours readily available and more slowly available forms of soil P.
Olsen-extractable phosphorus	9C	Widely used internationally. Preferred extractable P test in Victoria for most crops and pastures. Used to separate responsive from non-responsive soils to P fertiliser. Favours readily available forms of soil P.
Acid-extractable phosphorus (and silicate)	9G	Preferred for sugar cane and often useful for selected crops and pastures (Queensland) to separate responsive from non-responsive soils to P fertiliser. The test can also be adapted to assess the soluble silicate status of soils used for sugarcane, with a critical range of around 89-120 mg Si/kg soil (Kingston 1999).
Phosphate sorption index (PSI)	9I	This method was recently superseded by a new (ASPAC endorsed) phosphate sorption index test. The new test provides a quick, reliable indication of the P sorption status of the soil sample.
Phosphate sorption curve	9J	A tedious but informative test that provides superior information to that obtained by the new PSI test.
Total sulfur	10A	Provides a measure of the soil's total S percentage. Used in mass-balance studies. Typically has little bearing on whether or not the site will respond to S fertiliser. High values could indicate an acid sulfate (pyritic) layer.
Calcium phosphate-extractable S	10B	Provides a measure of the extractable sulfate-S status of soils with normal to deficient levels of plant-available S. The method is not guaranteed to extract all of the potentially available sulfate-S in soils containing gypsum, or in acid sulfate soils where sulfate is a dominant anion.
Total gypsum	11A1	Natural gypsum is present in sub-soils of many semi-arid soils in Australia, and can form following the oxidation of potential acid sulfate soils. It is an uncommon test.
DTPA-extractable Cu, Mn, Zn and Fe	12A1	A widely used trace element soil test with utility on acidic, neutral and alkaline soils.
Calcium chloride extractable B	12C1	A useful trace element soil test for soil B, for which local calibrations are available.
Saturation extracts	14	This method covers the estimation of a range of water-soluble salts. These tests are commonly applied to investigations pertaining to the presence of soil salinity and to specific ions. The method includes procedures for obtaining the saturation extract.
Exch. bases ( $\text{Ca}^{2+}$ , $\text{Mg}^{2+}$ , $\text{Na}^+$ , $\text{K}^+$ ) - 1M ammonium chloride at pH 7.0	15A	Suited for use on all soils, irrespective of whether acidic or alkaline, but preferred on acidic to weakly alkaline soils not dominated by solid-phase carbonates. Method 15A1 has no pre-treatment to remove soluble salts, with alternatives to remove them chemically (15A2) or to adjust for the presence of soluble sodium (15A3).
Exch. bases and CEC - alcoholic 1M ammonium chloride at pH 8.5, pretreat for soluble salts	15C1	Method is preferred if the soil is alkaline and calcareous and/or is strongly dominated by soils of constant negative surface charge. It incorporates a step to remove soluble salts. It overestimates the effective CEC of most acidic, highly weathered soils.

<b>Soil method</b>	<b>Code</b>	<b>Comment</b>
Exchangeable bases - 1M ammonium acetate at pH 7.0.	15D3	This rapid method for exchangeable cations in non-saline acidic through to slightly alkaline soils has no pre-treatment for soluble salts. It should yield similar data to those of method 15A1, except it can overestimate exchangeable Ca in soils containing calcium carbonate.
Exchange acidity (hydrogen and aluminium) by 1M potassium chloride	15G1	This is a preferred method for estimating the acidic cation status of acidic sub-tropical and tropical soils.
Effective cation exchange capacity (ECEC)	15J1	This is a preferred method for estimating the ECEC, typically acidic to slightly alkaline sub-tropical and tropical soils. ECEC represents the sum (on an equivalent basis) of the basic cations by method 15A or 15D3, plus the acidic cations (if any) by method 15G1.
Base saturation percentage (BSP)	15L1	A commonly used ratio that can only be calculated if exchangeable cations and CEC (or ECEC) data are available from the same sample.
Exchangeable sodium percentage (ESP)	15N1	A commonly used ratio that can only be calculated if exchangeable Na and CEC (or ECEC) data are available from the same sample. The value will be inflated if unadjusted /untreated for the presence of soluble Na salts.
Calculated lime rate	16A1	The calculation relies on exchangeable aluminium from 15G1.
Lime requirement - Mehlich single buffer	16C1	There are Queensland correlations for this test (Aitken <i>et al.</i> 1995).
Hydrochloric acid-extractable K	18B1	This is a quick test used for soil fertility purposes in Queensland. There are local interpretative guidelines for this test.

## APPENDIX 2

### **ASPAC Advice on: “How can I be sure that the lab is giving me the right results?”**

The Australasian Soil and Plant Analysis Council Inc (ASPAC) is an organisation that supports continuing improvement in laboratory performance, and also believes consumers need to be aware that laboratories do not all perform to the same, impeccable standard. When a farmer, extension officer or researcher sends soil and plant samples to a laboratory, how can they be sure the results are reliable? Analytical testing is like any other product – the quality can range from very good to the opposite extreme.

The following few simple questions can help ensure a wise and rewarding investment. These questions assume the client (e.g. farmer, extension officer, researcher) is certain of the method or methods to be used.

#### **Are you an ISO accredited lab?**

There is a special standard ([ISO 17025](#)) against which testing laboratories are audited. Independent assessors review the laboratory's Quality Assurance protocols and procedures, regularly inspect the laboratory, and ensure high standards are in place before awarding this accreditation. This does not guarantee nothing will ever go wrong (mistakes can always happen); it does provide proof the lab has good systems, appropriate staff and high standards.

#### **Do you compare well against other labs?**

Ask if they participate in [Laboratory Proficiency](#) programmes. These are also known as interlaboratory comparisons, or round robins. They involve a central coordinator preparing well-mixed samples, dividing these into homogenous subsamples and sending these periodically to participating laboratories for analysis. The proficiency program coordinator processes the reported values after a specified time to identify laboratories with acceptable and outlier results.

ASPAC runs such programmes for soils and plants. It also issues Certificates of Proficiency for each program on a method-by-method basis.

Ask the laboratory if they have an ASPAC Certificate of Proficiency for the test or tests of interest. Have confidence in the laboratory if the method is listed on the Certificate issued following the most recent ASPAC Program. Be cautious if the laboratory cannot or will not produce the ASPAC Certificate or if that Certificate does not list one or more of the tests of most interest to you.

#### **What do you do to be sure your results are right?**

This question is like doing a quick quality audit yourself, and obviously cannot be as comprehensive as a full ISO audit. But the lab's answers may either reassure you, or ring warning bells. They should be running check samples in every batch of analyses, and should be participating in some type of proficiency programme. If they are not doing these basics, be afraid; be very afraid.