FERTMARK CODE

Fertmark Code of Practice:

For the sale of Fertiliser in New Zealand

www.fertqual.co.nz

TABLE OF CONTENTS

Page

Appendices

INTRODUCTION

The Fertmark programme was established in 1992 to give New Zealand farmers confidence in the quality of fertilisers and the associated advertising. It is also constructed to include the requirements of the Agricultural Compounds and Veterinary Medicines Act which requires fertilisers to be supplied in such a way as to minimise risks to food safety and animal welfare.

This Code of Practice relates to all fertilisers made and sold under the Fertmark programme.

Farmers can be confident that fertilisers supplied from Fertmark registered companies are reliable and, if used properly, do not pose hazards to food safety or animal welfare.

Jim Galloway
Chairman
Fertiliser Quality Council

DOCUMENT CONTROL

The Fertmark Code of Practice is a controlled document.

Changes to the Code will only be made if they are approved at the Fertiliser Quality Council.

Each part of the Code will have a name, version date and page number. The name of each document will be shown on every page. A control sheet for each document will record its status and a brief comment about the changes that have been made to it.

The Fertmark Code will not normally be issued as a paper manual. It will be maintained electronically on the website of the Fertiliser Quality Council: www.fertqual.co.nz

Rules for the Use of the Fertmark Trademark

The following document has been lodged with the Intellectual Property Office of New Zealand. If changed, the amended copy must be lodged.

Date	**Version**	**Description of Revision**
Undated	1	First edition
March 2000	1.1	Uncontrolled draft, ACVM modifications
April 2000	1.2	Uncontrolled second draft
May 2000	2	Second edition
October 2002	3	Third edition, Fertiliser Quality Council
November 2012	4	Fourth edition, use of the terms ‘accreditation’ and ‘registration’.

RULES FOR THE USE OF THE FERTMARK TRADEMARK

1. Federated Farmers is the sole authority by which licences to use the mark of the Federation may be granted. Applications for a licence should be addressed to the Executive Director, Fertiliser Quality Council, c/- Federated Farmers of NZ (Inc), PO Box 715, Wellington.

2. Licences will be granted to Applicants by the Executive Committee of the Fertiliser Quality Council who, firstly, satisfy the Fertmark auditor that the product in respect of which the certification is sought meets the Fertmark Product Classification Standards and, secondly, undertakes to comply with the Fertmark Code of Conduct and thirdly, pay the prescribed fee. Upon satisfaction of these requirements the Executive Committee of the Fertiliser Quality Council shall grant the application.

3. The Executive Committee shall be entitled to refuse to grant a licence provided that the refusal is in accord with the rules and standards as laid down by the Fertiliser Quality Council.

4. The Executive Committee shall be entitled to revoke a licence, product certification and company accreditation where:

4.1 The product does not meet the certification specification as determined by the quality auditor and the Licensee is unable to attain that specification or has not registered a new composition for that product; or

4.2 A complaint has been made in respect to breach of the Code of Conduct and such complaint has been upheld by either the Advertising Standards Complaints Board or the Executive Committee and the breach has not been remedied within the specified time; or

4.3 The Executive Committee considers that there has been a breach of the Code of Conduct and that breach remains un-remedied after the expiration of the due notice period; or

4.4 The Licensee defaults in paying the requisite certification fees, annual audit fees or the promotion levy and remains in default after the expiration of the due notice period.

5. Without prejudice to the generality of their powers to revoke a licence the Executive Committee will normally consider revoking a licence if the Licensee:-

5.1 Uses the mark in connection with any goods not complying with the appropriate standard; or

5.2 Claims the right to use the mark in a form that is in the opinion of the Board misleading to the public; or

5.3 Commits a breach of or does not comply with these Rules; or

5.4 Becomes subject to the Bankruptcy Laws or makes any arrangement or composition with his creditors or if a company enters into liquidation (but not including voluntary liquidation for the purpose of reconstruction) or has a Receiver appointed of its business.

6. A Licensee shall:-

6.1 Conform with the Fertmark Product Classification and with any amendments to it as notified to him; and

6.2 Comply in all respects with the scheme of supervision and control which shall have been approved by the Executive Committee; and

6.3 Give the auditor appointed by the Executive Committee reasonable access during working hours to the premises in which the goods the subject of the licence are manufactured, mixed or stored, for the purpose of examining materials, finished articles, methods of test and records; and

6.4 Pay the fees fixed by the Fertiliser Quality Council for its licence and for its services in inspecting, testing, administration, and promotion, if any; and

6.5 Discontinue any use of the mark to which the Executive Committee may reasonably take exception and similarly discontinue any form of statement with reference to the authority of the Licensee to use the mark or otherwise in relation thereto which in the opinion of the Executive Committee may be misleading to the public and;

6.6 Comply with such requests for information including the results of quality tests that are made by the auditor.

6.7 Upon the termination of the licence (however determined) forthwith discontinue all use of the mark and all advertising matter which contains the mark or any reference thereto. Any goods not already disposed of by the Licensee which bear the mark shall if the Executive Committee so requires be treated so as to erase the mark.

7. The Licensee may only use the mark by implication that he is licensed to use the mark in respect of the goods which are the subject of the licence and which are manufactured in accordance with the appropriate Product Classification. In the event of any dispute as to whether the goods have been manufactured by the Licensee or in accordance with the appropriate Product Classification the decision of the Executive Committee shall be final.

8. A list of the Licensees and their products and analyses certified shall be kept by the Executive Director and be open to inspection by the public.

9. Any notice or other communication required to be given or sent thereunder shall be deemed to be duly given or sent if sent by post to the address of the Licensee last known to the Executive Director and shall be deemed to be given at the time when the same would have been delivered in the ordinary course of post.

Operational Rules

Date	Version	Description of Revision
Undated	1	First edition
March 2000	1.1	Uncontrolled draft
April 2000	1.2	Uncontrolled second draft
May 2000	2	Second edition
October 2002	3	Third edition, Fertiliser Quality Council
November 2010	4	Fourth edition, updated complaints procedure
November 2012	5	Fifth edition, expansion of complaints section to include non-Fertmark companies. Inclusion of Agronomic Expert Panel.

OPERATIONAL RULES

1. OBJECTIVE

The objectives of the Fertmark programme are:

- to give consumers confidence in fertiliser quality and product advertising; and
- to prevent and manage food residue and animal welfare risks associated with the use of fertilisers.

2. OWNERSHIP

Fertmark is a certification trade mark owned by Federated Farmers of New Zealand, acting as agent for all New Zealand primary producers. The administration of the Fertmark Certification Trade Mark is undertaken by Fertiliser Quality Council (Incorporated) at the discretion of Federated Farmers of New Zealand.

3. USE

The Fertiliser Quality Council (Incorporated) will grant the use of the Fertmark Certification Trade Mark in accordance with "Section 4.2 Rules for the Use of the Fertmark Trademark".

4. ORGANISATION

The Fertiliser Quality Council (Incorporated) will be organised as detailed in "Constitution of The Fertiliser Quality Council (Incorporated) a Registered Society under the Incorporated Societies Act 1908". For information purposes, this covers "Members", the "Executive Committee" and the "FQC Forum".

5. OPERATION

5.1 The scheme shall be administered by an Executive Director appointed by the Chairman of the Fertiliser Quality Council after consultation with the Executive Committee and Federated Farmers.

5.2 The Executive Committee shall have the responsibility to manage the scheme but on the following matters must have its decision ratified by the FQC Forum:

- the setting of fees;
- changes to the Operational Rules;
- appointment of auditor.

5.3 In addition the FQC Forum will meet no less than annually to review the scheme and make recommendations to the Executive Committee.

5.4 The Executive Committee will from time to time appoint an Expert Panel to advise on matters of a technical nature. The membership of this panel shall be approved by the Executive Committee in consultation with the FQC Forum.

6. CERTIFICATION

6.1 Applications for product certification will be in writing on the official application form and must be accompanied by the application fee. The application fee is not refundable in the event that the application proceeds to the pre-certification audit.

6.2 Upon receipt the Executive Director shall verify that the application is in the correct form and that the product fits the Fertmark classification. The Executive Director shall notify the applicant within 5 working days of the acceptability of the application. (This is not to be construed as provisional certification).

6.3 The Executive Director shall forward the application to the Auditor who will undertake a pre-certification audit. The auditor will then make a recommendation to the Executive Director on the applicants' ability to meet the audit requirements as a Fertmark accredited company.

6.4 The Executive Director shall accept the application provided the auditors' approval is given and the applicant agrees to abide by the Fertmark Code of Conduct and to pay the appropriate fees.

6.5 The applicant shall be notified of the final decision regarding becoming an accredited Fertmark company with Fertmark certified product(s) within 40 working days of lodging a correct application.

6.6 Any accredited company may apply to change the declared composition of its own certified product. This application is to be in writing to the Executive Director. The Executive Director will refer the matter to the auditor who will investigate the reasonableness of the proposal and who, if satisfied, will approve the change in the declared composition of the fertiliser.

If the change in the declared values or the process is such that the original classification is no longer met, then the application to change the declared composition will be dealt with as a new application.

6.7 The Executive Committee's decision on accreditation and certification or removal of accreditation or certification shall be final.

7. COMPLAINTS

The procedure for making and resolving complaints made by one Fertmark accredited company [or member of the Fertiliser Quality Council] (the complainant) against any other Fertmark accredited company (the respondent) [collectively with “the parties”] or any other complaint or investigation undertaken by the Executive Committee with respect to an alleged breach of any Fertmark rule is:

7.1 Before making a formal approach to the Executive Committee it is expected that the complainant will have made contact with the respondent, in an effort to resolve the dispute. A complaint about a non Fertmark fertiliser product should be made in writing to the Executive Director, define the reasons for the complaint and advise measures taken to resolve the matter with the respondent.

7.2 When placing a complaint before the Executive Director, the written submission from the complainant, duly signed by that member or accredited company's Chief Executive in New Zealand, should define the clauses of the Fertmark Rules or alleged breaches of the Fertmark field experiments protocol considered to be breached, and advise measures taken to resolve the matter with the respondent.

7.3 On receipt of the complaint, the Executive Director [or their nominees] will call a mediation meeting of the parties in an endeavour to reach a resolution within a period of no more than 20 working days.

7.4 Should this measure be declined or fail to settle the complaint, the Executive Director [or their nominee] will present the case, in confidence, for resolution by the members of the Executive Committee, none of whom shall be representatives of any of the parties. The resolution meeting shall be held within 20 working days of the mediation failing. It may be held by a physical meeting or teleconference. The decision will be made on the written evidence supplied by both parties unless the Executive Committee decides otherwise. If this alternative action is taken, then both parties must be consulted and treated equally. Where commercially sensitive information has been supplied in confidence to the Executive Committee by any party to a complaint, such information must be made available under privilege to legal counsel. If the Executive Committee considers that there are matters of a technical nature to be resolved, these may be referred to the Expert Panel established under Rule 5.4 of the Fertmark Operational Rules within a period of no more than 20 working days. It is expected that the Expert Panel will refer to "Managing and Planning Agricultural and Ecological Experiments" Johnston (Peter), Stanley Thornes Publishers, Cheltenham, United Kingdom, 1998 and Conduct of Field Experiments, P B Lynch, Bulletin No.399, NZ Department of Agriculture, Revised in 1966 (refer to Appendix 2).

7.5 When the Executive Committee has reached a decision, both parties will be advised, together with any intention of the Executive Committee with respect to publicity that it proposes to make. This would be done, if in the Executive Committee's opinion, there has been a breach sufficiently serious to damage the integrity of Fertmark or mislead fertiliser users.

7.6 If the Executive Committee proposes to make a public statement the defendant or accredited company shall be advised in writing by registered mail and by fax of the proposed publicity and be given at least 5 working days to respond. The five days will be from the date of receipt of the registered letter which will be deemed to be two working days after its dispatch. The response will be considered by the Executive Committee before it issues its public statement.

7.7 If either of the Parties are unwilling to accept the decision of the Executive Committee made under clause 7.5, they may appeal that decision to a sole arbitrator.

The appeal will take place pursuant to the Arbitration Act 1996, save that clauses 4 (determination of preliminary point of law) and 5 (appeals on questions of law) of Schedule 2 shall not apply. The place of arbitration shall be Wellington, New Zealand. The jurisdiction of the sole arbitrator will be limited to questions of law and procedural fairness. The sole arbitrator may, in its discretion, vary, revoke or remit a decision of the Executive Committee only in the event (and to the extent) that the sole arbitrator concludes that the Executive Committee has made an error of law or has acted in a procedurally unfair manner.

Any appeal must be lodged by the appellant serving a notice of appeal on the other Party and the Fertiliser Quality Council (naming both entities as respondents to the appeal), within fourteen (14) days of the date on which the decision appealed against was communicated to the appellant. The notice of appeal must specify the grounds of the appeal in sufficient detail to identify the issues raised by the appeal.

Within seven (7) further days, the other Party and the Fertiliser Quality Council must each indicate whether they intend to oppose the appeal, in which case they must do so by serving on the appellant and the other Party or the Fertiliser Quality Council (as the case may be) a notice of opposition to the notice of appeal.

The appellant and (provided they have filed a notice of opposition) the other Party and the Fertiliser Quality Council shall endeavour to jointly agree upon the identity of a sole arbitrator. If they cannot jointly agree within seven (7) days following the service of the notice of opposition, any party to the appeal may request the President of the Arbitrators' and Mediators' Association of New Zealand to appoint the sole arbitrator.

Within fourteen (14) days following the service of the notice of appeal, the Fertiliser Quality Council must make available to all Fertmark accredited companies [and members of the Fertiliser Quality Council] a copy of the notice of appeal and, if applicable, a copy of any notices of opposition to the appeal.

Within seven (7) further working days, any Fertmark accredited company [or member of the Fertiliser Quality Council] which considers itself affected may elect to join the appeal as a third party by serving on the parties to the appeal a notice of third-party joinder. Any such notice must specify that entity's interest in the issues raised by the appeal and the position of the entity in respect of those issues. All Fertmark accredited companies [and members of the Fertiliser Quality Council] agree and accept that a third party shall have no right to appoint the sole arbitrator. The nature and extent of a third party's participation in the arbitration proceedings shall be determined by the agreement of all parties to the appeal or, failing this, by the sole arbitrator.

To the extent practicable, the appeal shall proceed expeditiously with the objective of the sole arbitrator producing a short written and reasoned decision within four (4) months of the date of the notice of appeal.

The decision of the sole arbitrator shall be final and no party (including any third party) shall challenge, or seek appeal or review of, such decision, save under article 34 of Schedule 1 of the Arbitration Act 1996.

7.8 If the Executive Committee considers that there has been a breach of the Fertmark rules it may begin the process of withdrawing the Fertmark certification of that product or products. In doing so it will follow the procedure outlined in ‘De-certification’, Rule 8 of the Fertmark Operational Rules.

8. DE-CERTIFICATION

The certification for a product may be withdrawn by the Executive Committee in accordance with Clause 4 of the Rules governing the use of the Certification Trademark and under the following circumstances:

8.1 On the recommendation of the auditor when a product is found not to meet registered specification and the supplier has been unable to reach the certified specification and has not certified a new composition for that product.

8.2 When a complaint is made against matters covered in the Code of Conduct and such complaint is upheld either by the Advertising Standards Complaints Board or the Executive Committee and the matter complained of has not been rectified within the specified time.

8.3 When the Executive Committee considers there has been a breach of the Fertmark Rules or an accredited company is in default with the payment of certification fees. In such cases the registrant must be given 20 working days following notice of the perceived breach in which to respond with a proposed plan of action to remedy the matter. A further 20 working days will then be allowed to enable the matter to be put right. If the accredited company fails to respond during the initial period of 20 working days or, having responded, then fails to put the matter right to the satisfaction of the Executive Committee, within the second period of 20 working days, the Executive Committee may then withdraw certification and accreditation.

The Executive Committee's decisions on de-certification shall be final.

9. PROMOTION

It will be the responsibility of the Executive Committee, to undertake promotional activities to ensure that consumers are aware of the obligations of accreditation and the benefits of purchasing products so certified.

10. GROWTH PROMOTANTS

Where Fertmark certification is granted to a product that contains both fertilising elements and growth promotants, certification is to cover only the fertiliser component.

11. OPERATIONAL RULES, PROTOCOLS, DEFINITIONS AND CLASSIFICATIONS

The Council may from time to time set such operational rules, protocols, definitions, classifications of fertilisers and acceptable tolerances as are required to operate the scheme. These shall comprise schedules to be attached to these rules.

Policies and Protocols

This section of the Fertmark Code of Practice contains:

1. Fertmark Promotional Levy Policy;
2. Fertmark Confidentiality Protocol; and
3. Non-specification Fertiliser Line Protocol.
4. Reporting Physical Properties of Fertilisers at Depots Protocol

Date	Version	Description of Revision
July 1996	1	First edition
March 2000	1.1	Uncontrolled draft, ACVM modifications
April 2000	1.2	Uncontrolled second draft
May 2000	2	Second edition
November 2012	3	Third edition, use of the terms ‘accreditation’ and ‘registration’.
May 2025	4	Reporting Physical Properties of Fertilisers at Depots Protocol added.

PROMOTIONAL LEVY POLICY

1. The Promotional Levy will be based on sales value of certified Fertmark products, levied at an agreed percentage of sales value ratified at the Council meeting held before the commencement of the Fertmark financial year.

2. If the levy collected is greater than that required for Fertmark to operate on a break even basis, then accredited companies will have the surplus credited to them on a pro rata basis for the following year.

3. The sales value is deemed to be income to the accredited company (so excludes merchant's margin etc.).

4. The promotional levy is a payment in advance (at the start of the coming year), not in arrears.

5. It is based on sales of products which are Fertmark certified at the end of Fertmark's financial year, to fund the activities of Fertmark and its promotion for the following year.

6. Although collected at the start of the Fertmark year (1 July), it applies to the accredited company's previous financial year which currently range from 31 December to 30 June.

7. Companies which certify products during the Fertmark financial year will not face the promotional levy on those products until the new year (1 July).

8. The foundation principle of Fertmark is protection and quality assurance for the fertiliser consumer and therefore this should be used as a guide as to which products, mixes or blends the promotional levy applies to:

(a) on-sold Fertmark certified products destined for direct landholder consumer use are levied once to the company holding the Fertmark certification, even if they on-licence the use of the Fertmark certification to other companies that market the product;

(b) if a Fertmark certified product has some sales for non-landholder consumer purposes or is exported, and the Fertmark certification is not used for promotional purposes in these market segments, then no levy from those sales will be required;

(c) when an accredited company mixes or blends two or more of its Fertmark certified products for sale to the fertiliser consumer, the levy will be paid on each of those components, although Fertmark has made no assurances on the mix or blend;

(d) if two (or more) certified products make up another product which in itself is also Fertmark certified, double levying will be avoided by the levy applying only to the two (or more) components, but at their normal individual sales value; and

(e) when an accredited company mixes or blends an uncertified product with a certified product for sale to the fertiliser consumer, then a levy will be paid on the certified product portion, where benefit is still derived in the market from the certification of that component as part of the final product. Fertmark makes no assurances on the mix or blend.

9. In July of each year, accredited companies will supply the Executive Director of Fertmark with sales value of each specific Fertmark certified product for that company's proceeding financial year.

10. Such information will remain strictly confidential to the Executive Director.

11. The Executive Director will invoice the accredited company at the agreed percentage plus GST on the total value of Fertmark products sold on the basis of the information supplied. This is to be payable by the FQC Forum.

12. Any situations outside of the above policies, or where there is disagreement will be worked through with the company by the Executive Committee and the Executive Director, but only in a way that is consistent across all accredited companies.

13. Regardless of the above policies, the promotional levy system will still be dependent on the goodwill and co-operation of Fertmark accredited companies.

CONFIDENTIALITY POLICY

1. Certification, Monitoring and Audits

1.1 After an application for certification of a product is made by any company the details of the application will only be known to the Executive Director and the auditor. Neither the identity of the company or the product will be made available to any other party unless expressly allowed by the applicant company.

1.2 If any products fail to gain initial certification on the advice of the auditor, that information and the reasons for the failure to certify including analytical results, will be held by the auditor and the company concerned, and available exclusively to the Executive Director. No other party will have access to that information, being the name of the company, the product(s) involved, or the reasons for certification being declined.

1.3 If a product receives certification, then Fertmark will publicly declare that the product from the accredited company is certified.

1.4 The analytical values declared by the company at the time of certification (or subsequently modified), as well as the product description under the Fertmark Product Classification will be publicly available from the Executive Director.

1.5 All information and data collected from a company by the auditor in carrying out the obligations to Fertmark is confidential to that company and the auditor apart from the following exceptions:-

1.5.1 the normal recommendations (exclusive of exact data) from the auditor to the Executive Committee about product certification, de-certification and amendments.

1.5.2 requests from the Executive Director (and privy only to the Executive Director) to the auditor for information needed for the efficient functioning of the Fertmark accreditation scheme. Such requests will generally be sought only in the following circumstances:-

- (a) when a product is not meeting its declared values, or product classification as advised by the auditor;
- (b) when there is controversy or confusion over testing methods; or
- (c) when tolerances allowable under Fertmark accreditation, and other operational matters are under review.

1.6 All information held by the auditor on a company or its products is available to that company.

2. De-certification and Amendment of Declared Values

All actions taken by Fertmark in relation to de-certification and amendments of declared values will be undertaken in accordance with the Fertmark Operational Rules.

2.1 If a company significantly amends its declarations so as to avoid de-certification, Fertmark reserves the right to make it publicly known that the declaration has changed, particularly if the company does not take appropriate steps itself to inform the market.

2.2 If a company chooses to remove a product from the Fertmark certification scheme, Fertmark reserves the right to make it publicly known that the product is no longer certified.

2.3 If a product is de-certified on the recommendation of the auditor, Fertmark reserves the right to make it publicly known that the product is no longer certified, due to failure to meet declared values, but will not specify which values have not been met.

2.4 If a product or an accredited company with certified products is de-certified because of failure to comply with the advertising code of conduct, Fertmark reserves the right to make it publicly known that the product(s) is (are) no longer certified due to failure to comply with the advertising code of conduct.

3. Public Requests for Information

3.1 Any requests to the Executive Director for specific audit results for a certified product will only be granted upon receiving written confirmation from the company involved that they are prepared to have such results released to the party requesting the information.

4. Contaminant Content Reporting

4.1 The NZ fertiliser industry contaminant reporting system is operated by the auditor and funded by Fertmark. All specific data will be privy to the auditor only. The Executive Director or the Executive Committee will not have access to the data the auditor will report to Fertmark every six months in a manner which does not reveal individual company data. This report will have circulation agreed to by the Fertmark accredited companies who contribute the data.

5. Promotional Levy

5.1 The annual sales value of each Fertmark certified product that is supplied to the Executive Director for the purposes of calculating the promotional levy, and the promotional levy total per company will be strictly confidential to the Executive Director.

NON-SPECIFICATION FERTILISER LINE PROTOCOL

INTRODUCTION

With Fertmark certified products, heavy emphasis is given to those products meeting declared chemical and/or physical specifications and originating from a quality-controlled system. With both manufactured and imported lines, there is the potential for products to fall outside of the normal Fertmark specifications and tolerances. This protocol is not concerned with deviations, such as over specification, where there is no detrimental effect to the customer from the change in specification.

These events can be rare and outside of the control of the accredited company. Sometimes the deviations may have little agronomic significance, despite breaching Fertmark tolerances. De-certification of such products for a short period of time can be an overly severe step, and potentially confusing to the market. Yet the market still deserves to expect protection from such non-specification lines through the Fertmark programme. Of primary concern to Fertmark is the potential impact of the deviation and that the market is adequately informed of such an event and the relevant changes affecting declared values. This is essential for the integrity of the Fertmark programme.

A company in such a position has two options:-

A. To sell the product as a non-Fertmark certified line, which does not use the same product name as the certified product and is not called by a name on the Fertmark Product Classification if the actual analysis misrepresents that classification.

or

B. Continue to sell as a Fertmark certified product but with adequate market information (and possibly a price differential) so that it is clear that the product is not the normal line.

Fertmark is not concerned or involved if an accredited company adopts option A. Fertmark does need to be involved if option B is chosen and the following protocol should be adopted as soon as a non-specification line is detected.

PROTOCOL

1. The accredited company to immediately contact both the Executive Director and the Fertmark auditor and disclose in writing the following (referred to as the Proposal):-

1.1 The circumstances which resulted in the non-specification fertiliser line.

1.2 The chemical and/or physical analysis for the Fertmark declared values of the line, and the magnitude and effect of the discrepancies, such as if it breaches the Product Classification, or its potential agronomic effect.

1.3 The size of the line, relative to annual sales of that product and the likely time required to market the line.

1.4 Other relevant factors, such as if the line is mixed with other existing lines.

1.5 The Company's proposal of how to handle the line and the particular emphasis on how (or if) the company intends to inform potential purchasers of the Fertmark certified product as to its short comings or deviations.

2. Failure of a Company to submit a Proposal after the detection of such a non-specification fertiliser line that would undermine the Fertmark accreditation would be deemed to be a breach of the Fertmark Code of Conduct.

3. The Executive Director and the Fertmark auditor will jointly consider the Company's Proposal and where necessary will seek further clarification from the Company.

This will lead to the following outcomes:-

3.1 Accept the Company's Proposal, with no modification to Fertmark certification.

3.2 Recommend a modified Proposal to the Company and liaise directly with the Company over those modifications.

3.3 Decline the Company's Proposal and either seek an amendment of declared values or consider temporary or permanent de-certification of the product.

3.4 Where matters of a technical nature are making resolution difficult the Executive Director and Fertmark auditor may refer this to an expert panel established under rule 5.4 of the Fertmark Operational Rules.

4. If 3.1 occurs, Fertmark reserves the right to check compliance with the Company's Proposal, particularly in relation to informing the market. Failure to comply with the Proposal would be deemed to be a breach of the Fertmark Code of Conduct.

5. If 3.2 occurs, after liaison with the Executive Director and the Fertmark auditor, the Company would resubmit its Proposal, which would then lead to either of the outcomes discussed in 3.1 or 3.3.

6. If 3.3 was to occur, or the Code of Conduct breached as considered in clause 2 and 4 of this protocol, then this would be dealt with in accordance with Fertmark Operational Rules Clauses 6.6 and 6.7, and the relevant provisions of the Introduction of this Code of Conduct.

7. In their assessment of the Proposal, the Executive Director and the Fertmark auditor will give primary consideration to the impact of the non-specification line in the marketplace, the degree of information given to the market, and to the integrity of the Fertmark programme.

REPORTING FERTILISER PHYSICAL PARAMETERS AT DEPOTS PROTOCOL

INTRODUCTION

To assist in enabling spreaders to adequately spread any given material, a comprehensive set of product physical characteristics will be made available at despatch points.

The intention is to provide detailed current information on the physical characteristics of fertiliser and lime to the spreader operator at the depot. This is to enable the spreader operator to proactively adjust their spreading bout widths, or other machine parameters, so as to adequately apply the product that the farmer has engaged the spreader to apply or are applying themselves.

For clarity, it is not intended that such a report is a guarantee that the product will meet the ideal guideline at all times. The intent of such reporting is to be clear about the physical characteristics of the product at that point in time, so as to allow the spreader operators to make adjustments to the ensure evenness of spreading. The reporting will not be subject to FQC audit.

PROTOCOL

1. Companies selling Fertmark product are to display information at the depot about the physical characteristics of the products against an agreed guideline specification. Initially, such reporting would be for DAP, SoA and Urea. It is intended that reporting standards for other products will be developed over time.

2. The information that will be displayed is:

Product	% of product <1.9 mm	*% of product within 1.9-4.7 mm*	*% of product > 4.7 mm*	Bulk density t/m^3	Granule Strength, kg-f

3. The results will be reported on a notice board visible to spreader operators at depots supplying the Fertmark products. These numbers represent the line of product and do not absolve the spreader operator from measuring their individual load.

4. The 'best practice guidelines' in Fertmark defines the target physical characteristic ranges for the following fertilisers as:

Product	Particle size	Bulk density	Granule Strength
Ammonium Sulphate (SoA)	> 90% between 1.9 to 4.7 mm	0.95 – 1.02 t/m^3	>3kg-f
Diammonium Phosphate (DAP)	> 90% between 1.9 to 4.7 mm	0.90 – 1.10 t/m^3	>5 kg-f

Urea	> 90% between 1.9 to 4.7 mm	0.7 – 0.8 t/m^3	>3 kg-f

5. Information will be taken from source data from each new shipment on arrival, or new domestically manufactured fertiliser pile. Sampling will be undertaken when there is a batch change (new shipment or new pile).

6. In addition, sampling will be completed if there is a noticeable change in the condition of fertiliser within the pile, regardless of batch or shipment, or where there is a known change in batch (e.g. moving from brown to cream DAP), regardless of which batch is the most recent.

7. The displayed notice should be refreshed when there is a change in the line of product supplied.

8. A record will be kept of each set of results.

Code of Conduct

Date	**Version**	**Description of Revision**
June 1996	1	First edition
March 2000	1.1	Uncontrolled draft, ACVM modifications
April 2000	1.2	Uncontrolled second draft
May 2000	2	Second edition
November 2012	3	Third edition

CODE OF CONDUCT

1. INTRODUCTION

1.1 This Code sets out standards for the marketing and production of fertiliser.

1.2 Observance of the Code is a condition of Fertmark certification.

1.3 It is necessary for Fertmark certified companies, operating as they do in a keenly competitive industry, to draw attention to the existence and nature of a particular product by the use of advertising and other promotional measures. It follows that the marketing methods employed should be centred on the provision of standards of ethics and be in good taste. These precepts are embodied in the detailed provisions of the Code as set out hereunder.

1.4 This Code seeks to secure the acceptance and adoption of high standards of conduct in the marketing and production of fertilisers.

1.5 This Code will be administered by the Executive Committee. Complaints by one certified company against another for alleged breaches of this Code of Conduct will follow the Complaints Procedure outlined in the Fertmark Operational Rules.

1.6 This Code will be kept under constant review and amended from time to time where necessary to clarify it and bring it up to date. Notes for the guidance of accredited companies will be issued periodically to keep them informed of the rulings and recommendations of the Executive and of any alterations to the Code.

1.7 Fertmark certification entitles companies to use the Fertmark logo in the marketing of certified products.

2. DEFINITIONS OF CERTAIN TERMS

2.1 Marketing covers the representation, advertising and general promotion, distribution and sale of products.

2.2 Production covers importation, basic manufacture, blending, formulation, labelling and packaging.

3. QUALITY ASSURANCE

3.1 Participation in Fertmark company certification will be conditional upon the certification of fertiliser[s], subject to such rules and regulations that shall be set from time to time by the Executive Committee of the Fertiliser Quality Council.

4. MARKETING

4.1 A product must not be marketed with any direct or indirect reference to Fertmark in New Zealand unless it complies with all relevant statutory legislation and Fertmark requirements.

4.2 Where companies market a product that is not certified, there must be no suggestion in any marketing information that the Fertmark certification covers such a non-certified product.

4.3 Methods of marketing must never be such as to incite unfavourable comment or bring discredit upon either the Fertiliser Industry or companies with Fertmark certified products.

4.4 Production information furnished must be accurate and balanced and must not be misleading, either directly or by implication.

4.5 Products, services or personnel of other companies should not be disparaged either directly or by implication.

5. CLAIMS AND COMPARISONS

5.1 All claims and/or comparisons whether written or verbal as representation or advertisement shall abide by the Advertising Standards Authority Codes of Practice as well as the following:

5.2 Claims for the usefulness of Fertmark certified products must be based on agronomic trials conducted within industry recognised trial standards.

An Appendix describing the Agronomic Expert Group criteria is included as Appendix 2 to this Code.

5.3 The word "*safe*" must not be used without qualification and it must not be stated categorically that a product has no side effects, toxic hazards or environmental impact.

5.4 Comparisons of products must be factual, fair and capable of substantiation. In presenting a comparison, care must be taken to ensure that it does not mislead by distortion, by undue emphasis, or in any other way.

5.5 Fertmark accredited companies shall avoid price or savings claims which are misleading, or which do not offer provable bargains or savings.

6. ADVERTISING AND PROMOTION

6.1 All promotional and advertising material must comply with statutory requirements and the codes of practice of the Advertising Standards Authority.

6.2 Any complaint regarding advertising can be dealt with as described in Section 7 Operational Rules.

6.3 Advertisements must be clearly distinguishable from editorial matter; where there could be doubt, the word “*advertisement*” is required.

6.4 Promotional material, such as mailings and journal advertisements, must not be designed to disguise its real nature.

6.5 Promotional material should conform, both in text and illustration, to canons of good taste.

6.6 Promotional material should not imitate the devices, slogans or general layout adopted by other companies in a way that is likely to mislead or confuse.

6.7 Advertisements which make use of scientific data should clearly state the source of that data, which must not be used out of context or in such a manner that does not accurately reflect or portray the overall conclusions of that research. In addition, such data is to be scientifically robust. Wherever possible, previously unpublished data, including verbal communications on a subject, may not be used for advertising purposes unless specific written consent is obtained from the originating organisation, or the individual(s) concerned, after they have viewed the advertisement in question.

7. PUBLIC RELATIONS

7.1 Information furnished on any aspect of the industry generally should be accurate and in no way derogatory to Fertmark or any of its certified companies.

8. LABELING AND MARKETING

8.1 The following written material will be available for all Fertmark certified fertilisers: trade name, name and address of producer/manufacturer, batch number/order number or date of delivery, nutrient content, details of precautions to be taken and directions for use. Nutrient content, precautions and directions for use may be supplied as Product Information Sheets or Product User Guides.

8.2 Nutrient content information is to show the concentration of significant elements as registered with Fertmark.

8.3 Precautions for Use information should include appropriate fertiliser management information so that a user is able to minimise any risk to animals when using the product. It should also include any precautions that need to be taken to avoid food residue contamination problems

9. REPRESENTATIVES

9.1 Representatives should be adequately trained and possess sufficient knowledge to present information on their company’s products in an efficient manner and in accordance with other provisions of this code.

10. MARKET RESEARCH

10.1 Methods used for market research must never be such as to bring discredit upon or reduce confidence in the industry. The following provisions apply whether such research is carried out directly by the manufacturer or by an organisation acting on its behalf.

10.2 Interviews must not be gained by subterfuge.

10.3 Any incentives given should be kept to a minimum and be commensurate with the work involved.

10.4 Questions intended to solicit disparaging references to competing products or manufacturers must be avoided.

Market research must not be used as a form of disguised sales promotion.

11. PREVENTION AND MANAGEMENT OF RISKS

11.1 A product may not be certified under Fertmark, or a certified product may be de-certified, if as a result of using that product as recommended, residues are produced in food products that exceed NZ or overseas food residue standards. In addition, members will import, manufacture, formulate, blend, package and distribute their products in such a manner as to minimise any chances of environmental contamination.

11.2 A product may not be certified under Fertmark, or a certified product may be de-certified, if as a result of using that product as recommended animal health or animal welfare problems may be caused.

11.3 A product may not be certified under Fertmark, or a certified product may be de-certified, if it contains micro-organisms at pathogenic levels or any other plant or animal pest that is likely to promote disease or pest transmission.

11.4 For all Fertmark certified products there must be a traceable link between products delivered and their manufacture.

12. PRODUCTION

12.1 Certified companies involved in importing, manufacturing, blending, formulating and packaging will ensure that these activities are at all times carried out in accordance with the best currently accepted practices.

12.2 Certified companies shall ensure that proper records are kept of the manufacturing process, by batch if appropriate, and of the product testing programme.

12.3 Certified companies will fully comply with relevant codes as may be applicable to the industry.

12.4 Certified companies will comply with relevant legislation and in particular will enforce safety precautions for the health of employees engaged in any procedures involving the industry’s products.

12.5 Certified companies will ensure that the exterior of all outer containers, crates, drums or other packaging used for the distribution of technical, intermediate or finished products, conforms to the highest standard and is adequately marked with such precautionary information as may minimise the effects of accidents during transportation and storage.

12.6 Where appropriate, labels should be so printed and fixed to containers as to remain legible and permanently attached under all climatic, transport and other conditions likely to be experienced.

13. DISTRIBUTION AND STORAGE

13.1 Certified companies will ensure that packaged products are contained in packages suitable for the purpose under any storage and climatic conditions likely to be met.

13.2 Products are to be transported in a safe manner by responsible carriers who have been informed of the nature of their goods carried.

13.3 Adequate product information should be available to all interested parties, with particular respect to ingredients, toxicity, first aid and methods of handling spills.

13.4 Certified companies will ensure that resellers and their clients are provided with adequate information on storage and handling of the industry’s products and specific advice on products with special storage requirements and encourage compliance.

14. RESPONSIBILITIES TO EMPLOYEES

14.1 Certified companies will ensure that within their particular fields of authority and responsibility employees or sub-contractors will comply with all statutory requirements concerning the manufacture, packaging, handling, storage, distribution and application of their products.

14.2 Certified companies will ensure that all employees or sub-contractors who may have reason to handle any industry products are adequately trained in the interests of both themselves and the ultimate user or consumer.

Product Classifications

Date	Version	Description of Revision
July 1996	1	First edition
March 2000	1.1	Uncontrolled draft, ACVM modifications
April 2000	1.2	Uncontrolled second draft
May 2000	2	Second edition
June 2002	3	Third edition
October 2002	3.1	Addition of Poultry Fertiliser
May 2006	3.2	Included definition of the basis for Declared Values, altered limits.
November 2009	4	Fourth edition
November 2010	5	Fifth edition – clarifying the definitions of serpentine superphosphate, reverted superphosphate, dicalcic superphosphate, calcium chloride and magnesium oxide.
November 2012	6	Sixth edition.
February 2014	7	Addition of aerial agricultural lime classification, amendment of agricultural lime requirements and the clarification of the 'fertiliser' definition.
August 2016	7.1	Addition of the requirement for fertilisers not named in the table of Fertmark Product Classifications to have meaningful Declared Values.
February 2017	7.2	Addition of requirements for 'novel fertilisers'.
June 2017	7.3	Addition of specific requirement for the demonstration of compliance with the ACVM Act and Regulations. Addition of definitions of: Controlled Release, Slow Release and Stabilised Nitrogen Fertilisers.
January 2018	7.4	Amendment to the definitions of controlled release, slow release and stabilised nitrogen fertilisers.
October 2021	7.5	The oversize lime limit for agricultural lime and aerial lime was changed from 95% to 90%.
May 2024	7.6	Requirements regarding the strengthening of residue controls in fertilisers added.
November 2024	7.7	Amendment to the definition of monoammonium phosphate (MAP) and addition of Best Practice Guideline PSDs for SOA, Urea and DAP.
May 2025	7.8	Cross references added regarding Acceptable Tolerances and the requirement to have a Residue Risk Management Programme.

FERTMARK PRODUCT CLASSIFICATIONS

The purpose of these Product Classifications is to protect the existing meanings of terms used in the fertiliser and farming industries.

The Agricultural Compounds and Veterinary Medicines Act 1997 describes fertilisers as a subset of the agricultural chemicals that are used to sustain or increase the growth, productivity, or quality of plants or, indirectly, animals through the application of nutrients. The Act also notes that fertilisers must be 'fit for purpose'.

The ACVM Act defines 'fertiliser' as follows:

'Fertiliser' (a) means a substance or mix of substances that is described or held to be for, or suitable for, sustaining or increasing the growth, productivity, or quality of plants or, indirectly, animals through the application to plants or soil of –

- (i) Nitrogen, phosphorus, potassium, sulphur, magnesium, calcium, chlorine, sodium as major nutrients; or
- (ii) Manganese, iron, zinc, copper, boron, cobalt, molybdenum, iodine, selenium as minor nutrients; or
- (iii) Fertiliser additives; and

(b) includes non-nutrient attributes of the materials used in fertiliser.

(c) does not include substances that are plant growth regulators that modify the physiological functions of plants.

The Fertiliser Quality Council considers that, in order to be 'fit for purpose' as a fertiliser, products must:

- Comply with the ACVM Act definition of 'fertiliser', and
- Contain in aggregate, not less than three percent of nitrogen, phosphorus, potassium, sulphur, magnesium or calcium or contain such other elements at such levels as may be determined by the Executive Committee of the Fertiliser Quality Council.
- That the methods of analysis for nitrogen, phosphorus, potassium, magnesium and calcium be a water- or citric acid-solubility test that has been validated with the current Industry Agreed Test Method (refer to Appendix One of this Code).

In order for a product to be registered by Fertmark the supplier must supply sufficient information about the product and directions for use and for the supplier to demonstrate that it is "Fit for Purpose" in accordance with the ACVM Act, Regulation 7 of the ACVM (Exemptions and Prohibited Substances) Regulations 2011, and the ACVM Notice: Fertilisers, Plant Biostimulants and Soil Conditioners 2024. In order to demonstrate that this requirement is met the supplier shall provide an acceptable Risk Management Plan that demonstrates that, when the product is used as recommended, there is no accumulation of residues in primary produce that exceed the limits prescribed in applicable food residue standards or that there is toxicity to animals exposed to the compound to an extent that causes unnecessary or unreasonable pain or distress.[1] Refer to Appendix Four of this Code for guidance.

Where it is intended to describe a product using a name in the following table of Fertmark Product Classifications, then the product must conform to the appropriate description and quality standard. Similarly, where products are described using a name from the Fertmark Product Classifications table they will have values declared for each of the defined characteristics.

Where the fertiliser product is not named as one in the following table of Fertmark Product Classifications, the Declared Value Declarations made shall properly reflect the product attributes claimed by the vendor and be a generally recognisable attributes e.g. phosphatic fertilisers will generally have a citric-soluble P or water-soluble P declaration so as to make the declaration agronomically meaningful.

Where fertilisers belonging to a novel type are introduced, the Fertmark Auditor will consult on the proposed Declared Value Declarations for products of that class. It is intended that this consultation will generally be with all Fertmark-registered companies (and those with an interest in fertiliser). However, where the party wishing to gain Fertmark registration for a product belonging to a novel fertiliser type does not wish to alert the rest of the industry to their proposed entry, the consultation will be limited to the FQC Executive and such other parties as they may determine, providing always that confidentiality can be assured.

Declared Values will be stated, advertised and monitored on an ‘as delivered’ basis – not on a ‘dry basis’. The Acceptable Tolerances to the Declared Values can be found in the Fertmark Auditor Protocol.

The Declared Values for each of the Fertmark certified phosphatic fertilisers (Super, Sulphur Super, RPR, PAPR, DAP, MAP, TSP and any other fertiliser with more than a total P of 3.0% will have “Cadmium, not to exceed 280 mg Cd/kg P” as a Declared Value. If the voluntary industry cadmium limit changes, this Declared Value will be altered accordingly.

Any product conforming to the above description of a fertiliser and meeting the Fertmark quality standards will be eligible for Fertmark certification.

It is not intended that Fertmark certification be restricted solely to products described in the attached classification.

---

[1] *Guidance note for clarity: Suppliers of products which are exempt from registration must comply with all ACVM (Exemptions and Prohibited Substances) Regulations, 2011. Requirements for ‘Fitness for Purpose’ are stipulated in Regulation 7. Information requirements are stipulated in Regulation 12.*

Name of Material	Meaning	Declarations
Aerial Agricultural Lime	Ground limestone rock with a minimum lime equivalent of 65% by weight of $CaCO_3$. In addition, the material shall meet the CAA requirement that not less than 80% of the load shall be able to be dumped from a conventional agricultural aviation hopper in less than 5 seconds. Ground limestone which has not less than 90% by weight able to pass through a 2.00 mm sieve, and not less than 40% by weight able to pass through a 1.0 mm sieve, and not more than 2.5% able to pass through a 0.5mm sieve shall be deemed to meet this requirement.	Neutralising value percentage. Total Ca. Amount of material as a percentage by weight that will pass through a 2.00 mm, 1.00 mm and a 500 micron sieve.
Agricultural Lime	Ground limestone rock with a minimum lime equivalent of 65% by weight of $CaCO_3$. Not less than 90% by weight shall be able to pass through a 2.00 mm sieve, and not less than 30% by weight shall be able to pass through a 0.5 mm sieve.	Neutralising value percentage. Total Ca. Amount of material as a percentage by weight that will pass through a 2.00 mm and a 500 micron sieve.
Agricultural Sulphur	Products containing not less than 50% and up to 100% elemental sulphur (S) in finely particulate form with the balance being largely inert matter. The product may be in powder form or in the form of granules or prills which disperse upon absorbing water.	Total S. Amount of dispersed material percentage by weight that will pass through a 500 micron, 250 micron and 150 micron sieve.
Ammonium Nitrate	Chemically obtained product containing ammonium nitrate as its essential ingredient. The nitrogen (N) content must be not less than 33% and the nitric nitrogen and ammoniacal nitrogen fractions shall each account for about half the nitrogen present.	Total N.
Ammonium Sulphate (Sulphate of Ammonia)	Chemically obtained product with ammonium sulphate as its essential ingredient and containing not less than 20% ammoniacal nitrogen (N) and not less than 23% sulphur. The Best Practice Guideline for this product's particle size distribution is to ensure that at least 90% falls within the 1.9 to 4.7 mm fraction.	Total N. Total S.
Calcined Magnesite (See Magnesium Oxide)		

Name of Material	**Meaning**	**Declarations**
Calcium Ammonium Nitrate (C.A.N)	Chemically obtained product containing ammonium nitrate as its essential ingredient. The total nitrogen (N) content must be not less than 26% and the nitric nitrogen and ammoniacal nitrogen fractions shall each account for about half the nitrogen present. The product may contain, in addition to ammonium nitrate, only calcium carbonate (limestone) and/or magnesium carbonate and calcium carbonate (dolomite).	Total N. Total Ca.
Calcium Chloride	Chemically obtained material having calcium chloride as the essential ingredient. The material must contain at least 36% total calcium.	Total Ca. Water-soluble Ca.
Calcium Nitrate (Nitrate of Lime)	Chemically obtained product containing calcium nitrate as its essential ingredient and possibly ammonium nitrate, containing not less than 15% total nitrogen (N), a maximum ammoniacal nitrogen content of 1.5%, and minimum of 19% calcium.	Total N. Total Ca.
Chicken litter (see Poultry Fertiliser)		
Controlled Release Nitrogen Fertilisers	A fertiliser in which nutrient release is controlled. These are often fertilisers with a physical mechanism such as a coating or an occlusion.	Principle nutrient/s concentration. Type of coating e.g. resin or sulphur. Assessment of release profile as described in IFA 2014.
Diammonium Phosphate (DAP)	Product obtained chemically by reaction between ammonia and phosphoric acid and in which most of the phosphorus (P) is present as the divalent HPO_4 ion. The product shall contain not less than 17% total nitrogen (N) and 19% total phosphorus (P) and 17% water soluble phosphorus. The Best Practice Guideline for this product's particle size distribution is to ensure that at least 90% falls within the 1.9 to 4.7 mm fraction.	Total P P soluble in 2% citric acid. Water-soluble P. Total N.
Dicalcic Superphosphate	The product made by reacting mature superphosphate with limestone (or other basic calcium reverting agent). The water-soluble P content shall not be more than 2%. The total P content shall be not less than 4%, of which at least 80% shall be soluble in 2% citric acid solution.	Total P. P soluble in 2% citric acid. Water-soluble P. Total S. Total Ca.

Name of Material	Meaning	Declarations
Dolomite	Naturally occurring rock consisting largely of calcium and magnesium carbonates and containing not less than 10% of magnesium (Mg). Not less than 95% will pass through a sieve of 2.00 mm and not less than 80% by weight shall pass through a 0.5 mm sieve.	Neutralising value. Total Mg. Total Ca. Amount of material as a percentage by weight that will pass through a 2.00 mm and a 500 micron sieve.
Dunite (see Serpentine)		
Epsom salts (see Magnesium Sulphate)		
Ground Rock Phosphate	Product not otherwise specified in this table obtained from mineral calcium phosphate deposits, to which no other matter has been added and containing not less than 8% total phosphorus (soluble in mineral acids).	Total P. P soluble in 2% citric acid. Water-soluble P. Amount of material as a % weight that will pass through a 150 micron sieve.
Kieserite	A naturally occurring mineral with magnesium sulphate monohydrate as its essential ingredient. The material shall contain not less than 15% by weight of total Mg and not less than 20% sulphur.	Total Mg. Total S.
Lime (see Agricultural Lime)		
Magnesium Oxide in powder or chip form	The product obtained by the calcination of magnesium carbonate minerals and containing magnesium oxide (MgO) as the essential ingredient. The product shall disperse upon absorbing water. The magnesium (Mg) content shall be not less than 45%.	Total Mg. Amount of material as a percentage by weight that will pass through a 2 mm and a 500 micron sieve.
Magnesium Oxide in prill or granule form	The product obtained by the calcination of magnesium carbonate minerals and containing magnesium oxide (MgO) as the essential ingredient. The product shall disperse upon absorbing water. The magnesium (Mg) content shall be not less than 40%.	Total Mg. Amount of material as a percentage by weight that will pass through a 2 mm and a 500 micron sieve.
Magnesium Sulphate (Epsom Salts)	Product obtained from crude magnesium salts with magnesium sulphate as its essential ingredient. The material shall contain not less than 9.5% by weight of water-soluble magnesium.	Total Mg. Total S.

Name of Material	Meaning	Declarations
Monoammonium Phosphate (MAP)	Product obtained chemically by reaction between ammonia and phosphoric acid in which most of phosphorus (P) is present as the monovalent H_2PO_4 ion and containing not less than 9% total nitrogen (N) and not less than 20% total phosphorus (P) and at least 78% of the total phosphorus shall be soluble in water.	Total P P soluble in 2% citric acid. Water soluble P. Total N.
Muriate of Potash (see Potassium Chloride)		
Nitrate of Lime (see Calcium Nitrate)		
Nitrate of Potash (see Potassium Nitrate)		
Partially Acidulated Phosphate Rock (PAPR)	Product obtained by partial acidulation of reactive rock phosphate with sulphuric acid or phosphoric acid or a mixture thereof, with monocalcium phosphate, tricalcium phosphate and calcium sulphate (present only if sulphuric acid is used in the acidulation process) as essential ingredients, and containing not less than 9% total phosphorus (P) (soluble in mineral acids), at least 40% of the declared total phosphorus being soluble in water and not less than 50% soluble in 2% citric acid.	Total P. Water-soluble P. P soluble in 2% citric acid. Total Ca.
Potassium Chloride (Muriate of Potash)	Product obtained from crude potassium salts with potassium chloride as its essential ingredient and containing not less than 48% water-soluble potassium.	Total K.
Potassium Nitrate (Nitrate of Potash)	Potassium nitrate for fertilising purposes. Containing not less than 12% total N and not less than 38% K.	Total N. Total K
Potassium Sulphate (Sulphate of Potash)	Product obtained chemically from potassium salts, with potassium sulphate as its essential ingredient, and containing not less than 40% water-soluble potassium.	Total K. K soluble in water. Total S.
Poultry Fertiliser	Products derived from chicken litter solely obtained from approved poultry houses using identified feed systems for which a Risk Assessment Analysis and traceability system is provided.	Total N Total P Total K

Name of Material	Meaning	Declarations
Reactive phosphate rock for direct application (RPR)	Soft mineral phosphates, containing not less than 10% total phosphorus (soluble in mineral acids), at least 30% of the declared total phosphorus being soluble in 2% citric acid.	Total P. P soluble in 2% citric acid. P soluble in 2% formic acid by weight. Amounts of material as a percentage by weight that will pass through a sieve with a mesh of 1.0 mm and 500 microns. Total Ca.
Reverted Superphosphate	The product obtained by reverting immature superphosphate with ground limestone or other suitable alkaline material so that the total P content is not less than 7%. No less than 60% of the total P content shall be soluble in 2% citric acid solution and no more than 35% of the total P content shall be soluble in water.	Total P. P soluble in 2% citric acid. Water-soluble P. Total S. Total Ca.
Serpentine or Dunite	Ground magnesium silicate minerals containing not less than 18% magnesium (Mg). Not less than 90% of the material by weight shall be able to pass through a sieve with a mesh of 1.0 mm and not less than 75% by weight shall be able to pass through a sieve with a mesh size of 0.5 mm.	Total Mg. Total Ca. Amount of material as a percentage by weight that will pass through a 1 mm and a 500 micron sieve.
Serpentine Superphosphate	The product obtained by adding serpentine rock or dunite to immature superphosphate so that the total P content is not less than 6%. No less than 70% of the total P content shall be soluble in 2% citric acid solution.	Total P. P soluble in 2% citric acid. Water-soluble P. Total Ca. Total Mg. Total S.
Slow Release Nitrogen Fertilisers	Fertiliser products that release (convert to a plant-available form) their nutrients at a slower rate relative to a “reference soluble” product. This may be accomplished by biodegradation and/or by limited solubility and/or by hydrolysis or other recognised chemical or biochemical means.	Principle nutrient/s concentration or form. Nutrient solubility test as described in IFA 2014.
Stabilised Nitrogen Fertilisers	A nitrogenous fertiliser where the rate of conversion of the nutrient is inhibited or slowed by the addition of a compound/s.	Principle nutrient/s concentration. Amount of active ingredient using certified or validated methodology. Inhibition type.
SOA (see Ammonium Sulphate)		
Sulphate of Ammonia (see Ammonium Sulphate)		

Name of Material	Meaning	Declarations
Sulphate of Potash (see Potassium Sulphate)		
Sulphur (see Agricultural Sulphur)		
Sulphur Superphosphate (see wet-mix sulphur superphosphate)		
Superphosphate (Super)	Product obtained by fully acidulating ground phosphate rock with sulphuric acid with monocalcium phosphate and calcium sulphate as essential ingredients and containing a minimum of 8% total P. At least 85% of the total P shall be soluble in 2% citric acid, and at least 78% of the total P shall be soluble in water.	Total P. P soluble in 2% citric acid. Water-soluble P. Total S. Total Ca.
Triple Superphosphate (triple super, TSP)	Product obtained by fully acidulating ground mineral phosphate with phosphoric acid, with mono-calcium phosphate as its essential ingredient, and containing not less than 19% total phosphorus. At least 85% of the total phosphorus shall be soluble in 2% citric acid, and at least 78% of the total phosphorus shall be soluble in water.	Total P. P soluble in 2% citric P. Water-soluble P. Total Ca.
Urea	Chemically obtained product with carbonyl diamide (carbamide) as its essential ingredient and containing not less than 44% total ureic nitrogen (N) (including biuret). The Best Practice Guideline for this product's particle size distribution is to ensure that at least 90% falls within the 1.9 to 4.7 mm fraction.	Total N. Biuret.
Wet-mix sulphur Superphosphate	Compound prepared by the addition of elemental sulphur to superphosphate during the manufacturing process of the latter. The proportion of elemental sulphur shall be not less than 4% and not greater than 50%.	Total P. P soluble in 2% citric acid. Water-soluble P. Total Elemental S. Total Ca.

CLASSIFICATION OF PHYSICAL FORMS OF MATERIALS WHEN USED IN ASSOCIATION WITH FERTMARK CERTIFIED PRODUCTS

1. SOLIDS

Products intended for application in solid form without dilution and which may be sold in any of the forms listed below.

1.1 Compound

A material containing two or more of the primary nutrients (N, P, K, S, Mg, Ca) in a form or manner such that the components cannot be separated by physical means.

1.2 Blend

A material made by dry mixing of several components of similar particle size range. There is no chemical reaction during or following mixing and the various components could be physically separated.

1.3 Mixture

A material made by dry mixing several components which do not have a similar particle size range. There is no chemical reaction between the components, which could be separated by physical means.

2. NUTRIENT SALTS

Mixtures of readily water-soluble salts sold as dry or liquid concentrates requiring dilution prior to application.

3. FLUIDS

Products intended for application in liquid form with or without prior dilution and which may be sold in any of the forms listed below.

3.1 Suspension

Liquid product containing fine solid particles which are dependent on agitation or stirring to keep them in suspension. Dissolved components may also be present.

3.2 Slurry

Liquid product containing solid particles which are dependent on agitation or stirring to keep them in suspension. Dissolved components may also be present.

3.3 Solution

Liquid product consisting of completely dissolved mineral and/or organic constituents and free of particulates or solid crystalline matter.

3.4 Foliar Feed

Liquid product consisting of completely dissolved constituents and intended for application to the foliage of plant after dilution to an appropriate concentration.

Application Form

Date	Version	Description of Revision
December 1996	1	First edition
March 2000	1.1	Uncontrolled draft, ACVM modifications
April 2000	1.2	Uncontrolled second draft
May 2000	2	Second edition
November 2009	3	Third edition
November 2012	4	Fourth edition.
June 2017	4.1	Specific requirements regarding the demonstration of compliance with the ACVM Act and Regulations added.
May 2024	4.2	Changes to Declaration section to strengthen residue control.

APPLICATION FOR FERTMARK CERTIFICATION

Please complete one copy of this form for each product for which certification is sought (photocopy if necessary). If the product is manufactured at more than one site in the company, then complete a form for each site. When complete, attach appropriate fees and send to:

The Executive Director
Fertiliser Quality Council
c/- Federated Farmers Inc
PO Box 715
WELLINGTON

- Company Name...…….
- Postal Address: ....……..
- Street address of factory or warehouse:..……
- Contact Person: ....(position)
- Phone Number: ....………
- Fax Number: ……..………

- Product Name: ....…………………
- Product Classification: .... liquid/solid (*delete one*)
  (See Product Classification attached). Note: The schedule is not exhaustive, but if you use a term in the scheme it should comply with that definition.

1. AGRICULTURAL RISK DECLARATIONS

I declare that the fertiliser which is the subject of this application:

- meets the requirements of the ACVM Group of MPI and a copy of evidence of this is attached.
- does not contain microorganisms at pathogenic levels or any other plant or animal pest that is likely to promote pest or disease transmission.
- that when used as recommended it will not result in residues in food products that violate New Zealand or overseas limits.
- that when used as recommended it will not result in animal health or animal welfare issues.

………………………………………… …………………………………………..
(Signature) (Name)

…………………………………………
(Date)

2. ACVM ACT AND REGULATIONS

In order for a fertiliser to be registered by Fertmark the supplier must supply sufficient information about the product and directions for use, for the user to ensure it is “Fit for Purpose” in accordance with Regulation 7 of the ACVM (Exemptions and Prohibited Substances) Regulations 2011.[2]

3. TOTAL NUTRIENT CONTENT

This product contains the following essential nutrient elements at the concentrations stated:

3.1 **Major Nutrient Elements:** Express the % total element as w/w to nearest 0.1%.

Nitrogen (N)	Phosphorus (P)	Potassium (K)	Sulphur (S)	Magnesium (Mg)	Calcium (Ca)	Sodium (Na)	Chlorine (Cl)

3.2 **Micronutrient Elements:** Express as ppm

Boron (B)	Cobalt (Co)	Copper (Cu)	Iron (Fe)	Manganese (Mn)	Molybdenum (Mo)	Zinc (Zn)	Selenium (Se)

The Declared Value is not a guaranteed minimum but the average value which must be achieved by the companies own product testing and Fertmark audit samples.

If no claim is being made about the content of a particular element in the product, enter the concentration of that element as “N/A”. Only declared values will be tested. Declarations made beyond those specified in the Product Classification will be tested at the discretion of the Fertmark auditor and may incur extra charges. (Failure to meet any of the values declared can result in a product certification being rejected).

4. SPECIFIC DECLARATIONS: (% W/W) TO NEAREST 0.1%

(Refer to Fertmark Product Classification and complete the applicable declarations).

Nitrogen (N): Water soluble N ……………………………………………………%
for Urea state Biuret content …………………………………… %

Phosphorus (P): Water soluble P ……………………………………………………%
2% Citric acid soluble P …………………………………………… %
(See also “Physical Parameters”)

---

[2] *Guidance note for clarity: Suppliers of products which are exempt from registration must comply with all ACVM (Exemptions and Prohibited Substances) Regulations, 2011. Requirements for ‘Fitness for Purpose’ are stipulated in Regulation 7. Information requirements are stipulated in Regulation 12.*

Potassium (K): Water soluble K ……………………………………………………%

Sulphur (S): S as Sulphate S ………………………………………………… %
Water soluble S ………………………………………………… %
S as Elemental S ………………………………………………… %
(See also “Physical Parameters”)
Magnesium (Mg): Water soluble Mg (20°) ……………………………………… %
(100°) ………………………………………… %

Calcium (Ca): Water soluble Ca ………………………………………………… %
For Liming Materials, e.g. Agricultural Limes,
Dolomite and others -
State neutralising value ……………………………………… %

Any other specific declarations require………………………………………………
(see also “Physical Parameters”)

5. FORM OF NUTRIENT ELEMENTS PRESENT

State the common names of the components used during manufacture and the proportions used. Describe the nature and origin of the materials used and a brief description of manufacturing process, where applicable.

____

____

____

____

(attach additional pages if necessary)

Is the product to be diluted prior to use? Yes / No *(Delete one)*

6. PHYSICAL PARAMETERS

If you are registering any of the materials listed in the left-hand column below, please complete the appropriate details.

	Sieve Size (mm) ***(% w/w material passing through sieve)****				
	2.00	1.00	0.50	0.25	0.15
Agricultural Lime		▨		▨	▨
Agricultural Sulphur	▨	▨			
Basic Slag	▨	▨	▨	▨	
Calcined Magnesite		▨		▨	▨
Dolomite		▨		▨	▨
Ground Phosphate Rock	▨	▨	▨	▨	
Reactive Phosphate Rock					
Serpentine or Dunite	▨			▨	▨
Wet-Mix Sulphur Superphosphate (elemental sulphur component)	▨	▨	▨		

* to the nearest 1%

7. IMPORTED PACK - ONLY PRODUCTS

The following additional application details are required for products certified under this Fertmark category.

7.1 Specific Declarations

For each of the Specific Declarations listed on page 2 of this application form declare the range (tolerances) around each declared value.

<u>Element</u> **<u>Range (tolerance)</u>**

.... %

.... %

.... %

.... %

.... %

(if more than 5 declarations have been made please attach an additional sheet)

7.2 Fertmark Auditor's Interpretation of Declarations

7.2.1 The declared value is not a guaranteed minimum but the average value which must be achieved by the companies own product testing and Fertmark audit samples.

7.2.2 For Imported Pack-Only Products the tolerances are assessed on those quoted in the declaration above.

7.3 Further Information Requirements

Please provide the following numbered attachments:-

- Attachment 1 - Statistical information on adherence to declared values for lines supplied.
- Attachment 2 - Details of purchase Specifications (so the continuity of standards for declared values can be determined regardless of the international supply source). (Include below if space sufficient).

....

....

....

....

Note: Companies who have only certified Imported Pack-Only Products will be required to regularly furnish the Fertmark auditor with shipping data and statistical information for the product, and any subsequent New Zealand testing undertaken by the company.

8. FERTMARK FEE CHARGES

Audit Fees

These fees are subject to change. Please contact the Fertmark Executive Director for an update.

Promotional Levy

The Fertmark Promotional Levy is set each year. Refer to the Promotional Levy Policy for details.

9. DECLARATION

We agree that upon being granted Fertmark certification for this Product we:

- Will abide by the Fertmark Code of Conduct;
- Allow such re-audits as are requested by the auditor;
- Supply such analytical data as requested by the auditor
- Pay such fees as are by due to Fertmark and, if applicable, to the auditor;
- Allow such samples to be taken as are requested;
- Will operate and maintain a Risk Management Programme for potential contaminants and we will advise the auditor of any changes to the sourcing, process or formulation that might impact on the residues in our product,

- Will operate and maintain a Risk Management Programme for potential contaminants and we will advise the auditor of any changes to the process or formulation that could impact on the residues in our product, and
- Abide by such rules, protocols and policies as are made by the Council from time to time.

....…

Signature Name

Date …………...................................

Protocol for Mixing Plants

Date	**Version**	**Description of Revision**
May 1997	1	First edition
March 2000	1.1	Uncontrolled draft, ACVM modifications
April 2000	1.2	Uncontrolled second draft
May 2000	2	Second edition
November 2012	3	Third edition

PROTOCOL FOR THE CERTIFICATION OF FERTILISER MIXING PLANTS

---

1. INTRODUCTION

Fertmark, is a programme aimed at ensuring that fertiliser materials purchased by New Zealand farmers and growers meet specified quality criteria. The majority of companies supplying N.P.K. products are committed to the Fertmark scheme. Most of these products are straight lines, rather than mixes.

Mixes have primarily not been certified due to the high degree of variability that can occur, both due to segregation and product testing procedures. Because the components used in special mixes may segregate during transport, obtaining a representative sample may be difficult, if not impossible, once the mix has been loaded into a truck, railway wagon or conveyance, or dumped on a farm.

As a large proportion of the fertilisers sold in New Zealand are made up of mixtures of different fertiliser products, the Fertiliser Quality Council authorised the appointment of an expert panel to examine the issue of certification of mixing plants and to provide recommendations for examination by both the Executive and Council.

1.1 Reason for Adoption of Objective

Unless a mix is made from equal size, shape and mass particles, segregation will occur. This results in erratic analysis when the product is sampled. With some trace element mixes chemical detection methods also have limitations. The potential number of mixes able to be provided by a plant are infinite.

Consumers seek consistent mix analysis for the reasons given above. The Fertmark product certification system could not adequately serve this purpose. Therefore it was considered more prudent for Fertmark to certify the mixing plant and system itself. Mix products can still be certified separately under the existing Fertmark product certification system, but would have to meet the quality criteria that apply.

2. OUTLINE OF SCHEME FOR OBTAINING CERTIFICATION OF MIXING PLANTS (SITES)

2.1 The site and process used to make mixes must be certified for this purpose with the Executive Director of Fertmark.

2.2 The auditor shall have access for the purpose of monitoring mixing plants.

3. SITE CERTIFICATION

To obtain Fertmark certification for a mixing plant, the company needs to:

3.1 Have 95% or more of the total annual fertiliser products by weight, passing through the mixing plant being Fertmark certified products. Note this excludes products brought onto the site by a client to be mixed for their own use.

3.2 Have a reliable and accurate system to record inwards orders.

3.3 Have a system in place to provide written operational procedures to staff for the mixing of each order.

3.4 Make available such operational procedures for scrutiny on request by a client.

3.5 Store components so as to avoid cross-contamination.

3.6 Have a way of ensuring that the correct amounts of the components are drawn for each mix.

3.7 Be able to verify stock usage against orders despatched.

3.8 Be able to satisfactorily mix the components.

3.9 Have its own quality control data on the mixes it produces.

3.10 Show to the satisfaction of the auditor that where the complete mix is not despatched directly to the client, but is stored in bulk on site, and later drawn off at different times, that the final mix despatched conforms with the tolerances (refer to Table 1).

4. DOCUMENTATION OF MIXES

4.1 Each delivery of a mixture shall be accompanied with a certification document which states, inter alia:-

a) the quantity of the mixture comprised in the sale
b) the date of dispatch of the mixture
c) the common names of the components contained in the mixture and the weight of each component as a proportion of the weight of the mixture
d) the name of the site where the mixture was prepared
e) the product analysis for N.P.K.S. where appropriate and practical to do so.

5. TOLERANCE LIMITS

5.1 Tolerances cannot exceed the component maximums listed in Table 1. Where companies wish to specify more rigorous tolerances they may do so. Such tolerances are public information once a mixing plant has been approved for certification.

5.2 Companies will confirm that production settings can be supported by appropriate calibration checks and product verification.

5.3 Statistical analysis of the results for each mixing plant should show that, over time, test values agree with the claimed values (with a fair level of probability as assessed by the auditor).

5.4 Analysis of the results for each mixing plant should show that, for individual consignments, test results agree with the claimed value within the specified tolerances (with a fair level of probability as assessed by the auditor).

5.5 If standard mixes (such as those listed on a price list) alters in a way that significantly reduces its ability to perform (such as spreading or agronomic performance) then the company must take all necessary steps to inform the market of that change and its implications. An example of this might be a significant change in particle size specification.

TABLE 1 ***Tolerances for Certification***

The table relates to maximum acceptable statistical variations (expressed in percentage terms) of the components stated to be added to obtain the mix.

	Maximum Negative Variation for Each Component	Maximum Collective Variation for mixtures of more than two components
Macronutrients (N,P,K,S,Mg)	5%	10%
Other Major Elements	7%	12%
Trace Elements	10%	12%*

Maximum Collective Negative Variation is determined by adding all the negative variations, but not offsetting these with positive variations. A negative variation is deemed to be a variation contrary to what the market would wish to see in the final product.

* Where two or more trace elements are involved, a further negative tolerance of 5% can be added for each additional trace element but the further variation must only apply to the trace element component.

Example

Assuming a theoretical mix result as follows

(Based on 3 components at 25/25/50 to give the desired N-P- K)

		Component			**Sum of Negative Variation**
		1	**2**	**3**	
(A)	%	26.25	24.75	49.00	
	Maximum Component Variation	(+5%)	(-1%)	(-2%)	(-3%)

(B)	%	29.00	23.50	47.50		
	Maximum Component Variation	(+16%)	(-6%)	(-5%)		(-11%)
(C)	%	24.00	24.00	48.50	3.50	
	Maximum Component Variation	(-4%)	(-4%)	(-3%)	(X)	(-11%)

For A, each component is within the 5% variation tolerance and collectively within the 10% criteria (for More than Two Products). Therefore it is acceptable.

For B, it fails on 2 counts. Component 2 exceeds the maximum tolerance for a component, and the maximum collective negative variation of 10% is exceeded.

C fails on one criteria, which is the exceedence of the maximum collective negative variation of 10%. (X) must have been an unauthorised fourth product, which created this effect.

6. AUDITING FREQUENCY

6.1 The frequency of re-audit is based on the performance of the mixing plant as assessed by the auditor, giving due recognition to ISO registration or equivalent systems that are third party audited.

6.2 At 12 monthly intervals the company will send to the auditor statistical analysis from the mixing plant that relates to the previous 12 month period.

6.3 If the process as detailed in the initial audit is changed, the auditor should be notified and a re-audit may be required.

7. COSTS

7.1 Audit fees are subject to change. Please contact the Fertmark Executive Director for an update.

The Fertmark auditor deals with both product certification and mixing plant certification. Whenever possible audits will be conducted in tandem so as to reduce charges. This may not always be possible, particularly for the initial audit.

Charges are based on real time taken for conducting the audits, and the direct charges incurred.

The auditor can be approached directly for an estimate of the likely time required to conduct the audit.

In the event of certification not being granted by the auditor, the costs of the audit are still payable.

8. OPERATION OF THE FERTMARK PROGRAMME FOR CERTIFICATION OF MIXING PLANTS

All Constitutions, Operational Rules, Policies and Protocols applying to the Fertmark programme for product certification will apply to mixing plant certification. This includes provision for the de-certification of mixing plants.

9. AUDITING ASSESSMENT CRITERIA FOR MIXING PLANT CERTIFICATION

9.1 The process of mixing should be properly described in a document containing:

a) a graphic flow diagram showing the entire process.
b) inputs and outputs including flow rates or weight measurements of fertiliser components.
c) any critical points in the process of mixing that if control is lost (manually/electronically) will result in out-of-spec product.
d) any in-process sampling or examination points within the process.
e) use of log books, diaries, electronic data retrieval.

9.2 The mixing process equipment should have regular planned maintenance especially where the sensitive parts of the equipment are likely to fail.

This will include:

- weigh feeders
- mass load cells
- screens and sieves
- conveyor drive frequency control or variable drives
- rotary drums, ribbon blenders, bucket chains etc.
- electronic monitoring and interception

9.3 The staff must be trained and fully understand the complexities of the products and the process.

9.4 Product that fails mix recipes, e.g.: products left out, or wrong products introduced must be segregated and prevented from being inadvertently despatched to the customer.

The company’s operating procedure should specify how the out-of-spec product should be disposed of.

9.5 Where the company already has an accredited quality assurance system (such as ISO 9001), which specifically covers the mixing plant, the Fertmark auditor will assess whether such procedures adequately meet Fertmark requirements, to avoid dual auditing.

9.6 Over time the auditor will work to establish further details on tolerance limits.

MIXING PLANT APPLICATION FORM

APPLICATION FOR THE CERTIFICATION OF FERTILISER MIXING PLANTS BY FERTMARK

Please complete one copy of this form for each site containing a mixing plant or plants (photocopy if necessary). This form is to be read in conjunction with the “Protocol for Fertmark Certification of Fertiliser Mixing Plants”. When completed, send to:

The Executive Director
Fertiliser Quality Council
c/- Federated Farmers Inc
PO Box 715
WELLINGTON

Company Name : ……………………………………………………

Postal Address:

…………………………………………………………………………………..

Street address of factory or warehouse:

…………………………………………………………

Contact Person: ……………………………………………………………….. (position)

Phone Number: ……………………………………………………………

Fax Number: ………………………………………………………………

Site Name : ……………………………………………………………………….

Number of mixing plants at the site: ……………………………………………………..

Name or identification number of each mixing plant on site:

1. ……………………………………………………………………………………….
2. ……………………………………………………………………………………….
3. ……………………………………………………………………………………….
4. ……………………………………………………………………………………….
5. ……………………………………………………………………………………….

MIXING PLANT DOCUMENTATION

For each mixing plant listed above, please provide the following information:

a) a graphic flow diagram of the mixing process

b) documentation on:
   (i) inputs and outputs including flow rates or weight measurement of fertiliser components.
   (ii) critical points in the process of mixing
   (iii) in-process sampling or examination points (Refer to Appendix 1 of the Protocol for Fertmark Certification of Fertiliser Mixing Plants). Attach this information to this application form and label it “Attachment 1.

The Fertmark auditor deals with both product certification and mixing plant certification. Whenever possible audits will be conducted in tandem so as to reduce charges. This may not always be possible, particularly for the initial audit.

Charges are based on real time taken for conducting the audits, and the direct charges incurred. The current charge out rate from the auditor is as follows:)

The auditors can be approached directly for an estimate of the likely time required to conduct the audit.

In the event of certification not being granted by the auditor, the costs of incurred are still payable.

Is the Site and Mixing Plant(s) part of an independently audited Quality System	**YES / NO**
The appropriate deposit is attached	**YES / NO**

We agree that upon being granted Fertmark certification for these mixing sites we:

- Will adhere to the protocol for Fertmark Certification of Mixing Plants;
- Will abide by the Fertmark Code of Conduct;
- Allow such re-audits as requested by the auditor;
- Will supply such analytical data as requested by the auditor;
- Will abide by such rules, protocols and policies as are made by the Council;
- Will pay such fees as set by Fertmark from time to time.

....
Signature Name

Date ....…………........

Auditor Protocol

Date	Version	Description of Revision
February 2000	1	Uncontrolled draft
April 2000	1.1	Uncontrolled second draft
May 2000	2	First edition
October 2002	3	Second edition, Micronutrient limit
November 2012	4	Third edition, use of the terms ‘accreditation’ and ‘registration’
May 2025	5	Amendment to align Fertmark Acceptable Tolerances with the ACVM Notice (section 7.2).

FERTMARK AUDITOR PROTOCOL

1. INTRODUCTION

1.1 This protocol sets out the role and responsibilities of the Fertmark auditor.

1.2 The Fertmark auditor shall be appointed by the Executive Committee for such a term as the Executive Committee shall determine.

The Fertmark auditor shall have received appropriate auditor training, shall be familiar with the fertiliser industry and familiar with Fertmark and ACVM requirements.

2. COMPANY CONTACTS

2.1 The auditor will maintain a register of nominated accredited company contact people and site people. The nominated company contact people are to be the primary points of contact for the auditor with the company.

3. AUDIT FREQUENCY

3.1 After the initial pre-certification audit, site audits will normally be carried out every two years.

3.2 Accredited companies may elect to have additional special audits more frequently than this at their own cost.

4. NOTIFICATION

4.1 The auditor will be notified by the Executive Director each time a valid application is received.

4.2 The auditor will negotiate with the nominated company contact person and the site contact person for a suitable time to conduct pre-certification audits, site re-audits and to collect product monitoring samples.

5. DECLARED VALUES

5.1 The auditor will maintain a register of Declared Values for each product registered.

5.2 Where a company wishes to change any of its Declared Values the auditor will investigate and keep in close contact with the Executive Director.

6. PRODUCT MONITORING

6.1 The auditor will take, or cause to be taken; routine product monitoring samples for each product certified. The auditor will obtain the company’s own test results for that product, where practicable.

6.2 Product monitoring samples will be competently taken and will be taken every six months except where the company has ISO registration, with a relevant scope, when they will be taken every twelve months.

6.3 Product monitoring samples will be tested in a proficient laboratory. Proficiency will be assessed by the Auditor. Participation in an interlab comparative testing programme (ILCP) will be taken into account.

6.4 Product monitoring sample test results will be compared to the Declared Value for that product and the company's own test results.

If the average of the Company's own test results for the last year is lower than the Declared Value for that fertiliser then the company and the Executive Director will be advised of this problem.

If any product monitoring results are lower than the Declared Value by more than the Acceptable Tolerance then the company and the Executive Director will be advised. The company may elect to have special additional samples taken and tested by the auditor at their own cost. The normal number of special samples taken is five, taken together.

7. ACCEPTABLE TOLERANCES

7.1 The term Fertmark Acceptable Tolerances refers to the maximum allowable difference between the Declared Values for a fertiliser and the results from the analysis of a Fertmark Monitoring Sample.

7.2 The Acceptable Tolerances are:

Macronutrients: For macronutrients the Fertmark Acceptable Tolerances are:

- Less than 5% of the DVs for all nutrient levels at or above 10 % and,
- Less than 0.5% as an absolute tolerance value, for all nutrient levels between the DVs at or above 5% and below 10% and,
- Less than 10% of the DVs for all macronutrient levels at or above 1% and below 5% and,
- Less than 30% of the DVs for all macronutrient nutrient levels below 1%

Nutrient Level	Limits
0.5%	0.35% to 0.65%
1%	0.9% to 1.1%
5%	4.5% to 5.5%
10%	9.5% to 10.5%
20%	19% to 21%
60%	57% to 63%

Micronutrients: Less than 20% of the Declared Value unless the product being declared is a primary source of the micronutrient (e.g., copper sulphate) when the tolerances as for macronutrients will apply.

7.3 The Fertmark Auditor will actively monitor the Acceptable Tolerances and will report to the Executive Director at least once a year on whether the limits remain appropriate.

8. SITE AUDITS

8.1 During site audits the auditor will assess both systems and technical operations. Where a site has ISO registration, due recognition and use is to be made of this. The purpose of the site audit is to assess the capability of the site to supply fertiliser of the quality defined in their application that is consistent with the requirements of the ACVM Group of MAF. There will be a specific assessment of the availability of product use information to farmers and growers. In addition, the audit will cover:

- written operational procedures;
- inwards goods quality;
- control of process; and
- avoidance of contamination.

8.2 During the site audit the auditor will examine the company product monitoring programme and assess its suitability with regard to methods, staff competence and performance in an appropriate fertiliser laboratory proficiency programme.

8.3 After the audit is complete a site audit report will be sent to the nominated company contact person. It will include a summary of findings and a statement of the extent to which the company systems and practices meet the Spreadmark Code of Practice.

9. STATUS REPORT

9.1 The auditor shall maintain a status report showing the current status of each certified product, when it is next due to be sampled and any current issues that relate to it.

9.2 The status report shall be supplied to the Fertiliser Quality Council Executive Director every six months or within 10 days of it being requested.

10. RECORDS

10.1 The auditor shall maintain proper records. These records shall include audit reports, records of product monitoring analyses and comparison information, status reports and correspondence.

10.2 Records, or copies of records, shall be supplied to the Executive Director upon request in accordance with the Fertmark Confidentiality Protocol.

11. CONFIDENTIALITY

11.1 The auditor will not communicate information about any company or its products to anyone other than the company itself through its nominated contacts or the Executive Director. Requests for information are to be referred to the Executive Director.

11.2 All information held by the auditor on a company or its products are to be made available to that company on request by the nominated company contact people.

FERTMARK CODE OF PRACTICE

APPENDIX ONE

INDUSTRY AGREED REFERENCE TEST METHODS

The material recorded here are the Industry Agreed Reference Methods that have been recognised by the Fertiliser Quality Council as being appropriate for the testing of fertiliser and lime.

The purpose of this collection of test methods is to provide the standardization of reliable reference methods which can either be used directly or which can be used to validate bench methods.

The constructive contributions of the Fertiliser Quality Council Laboratory Test Methods Expert Group continues to be very much appreciated. We also wish to recognize the Fertiliser Association of New Zealand (FertResearch) for their generosity in providing their “Analytical Methods for Fertiliser Materials” to the Expert Group.

Suggestions for improvements to the tests are welcomed.

There is on-going work on the development of Industry Agreed Reference Test Methods for lime and fertiliser testing and parties with an interest in this matter are invited to contact:

The Executive Director
Fertiliser Quality Council
c/- Federated Farmers Inc
PO Box 715
WELLINGTON

Date	Version	Description of Revision
December 2006	1	First edition, liming equivalent test in agricultural lime established.
August 2007	2	Cadmium method for soils and fertiliser added.
June 2008	3	Reference methods for sulphate sulphur, total phosphorus and moisture in fertilisers established.
November 2009	4	Reference methods for elemental sulphur, CSP and WSP, free phosphoric acid in fertilisers established. Guide for sampling solid fertiliser and lime materials and for the preparation of the analytical sample established.
November 2010	5	Reference methods for wet sieve analysis, granule strength, granule attrition and nitrogen analysis of fertilisers are established and the phosphorus test method is amended.
November 2012	6	Reference Methods for the preparation of the analytical sample (amended), Statistical Interpretation (new), Moisture in fertilisers method (amended), sulphate sulphur in superphosphate (amended), Cadmium in fertiliser (amended) and TSP, CSP and WSP (amended).

February 2015	7	Reference Methods for the wet sieve test method (amended), the dry sieve test method (amended), the biuret method for urea (amended), the granule strength method (amended), granule attrition method (amended), moisture in fertilisers method (amended), heavy metals in fertilisers (amended), formic soluble phosphorus (new), elemental sulphur in fertilisers (amended) and sulphate sulphur in fertilisers (amended).
February 2016	8	Reference Methods for Total Fluoride in fertilisers (new), Water Soluble Fluoride (WSF) in fertilisers (new), Mercury in Fertilisers (new), Total Sulphur (non-soxhlet) in Fertiliser (new) and Moisture in Sulphur (amended).
February 2017	9	Reference Methods for Controlled Release Nitrogen Fertilisers and Boron if Fertilisers added, Common Cations in Fertilisers Method amended and extended and Wet Sieve Method amended.
January 2018	9.1	Reference Methods for Granule Strength, Total Fluoride, Abrasion Resistance (re-named) and Cadmium in Fertilisers were amended.
January 2019	9.2	Methods for sulphate sulphur in superphosphate, elemental sulphur in fertiliser, and the statistical guide amended and the Watkinson Dissolution test for RPR added.
January 2020	9.3	Methods for the Preparation of the Analytical Sample, the Dry Sieve Method and the Liming Equivalent Method were amended.
January 2021	9.4	The new Lime Reactivity Test Method, the new NBPT Test Method, the new Fluoride in Soils Test Method, the revised Biuret Test Method, the revised Dry Sieve Test Method and the revised Preparation of the Analytical Sample Guide were all added.
October 2021	9.5	The Granule Strength, Moisture in Fertilisers and Moisture in Sulphur Test Methods were amended.
November 2022	9.6	The Fertiliser Sampling Protocol was amended.
May 2024	9.7	The Free Phosphoric Test Method, the Total Phosphorous (TP), Citric Soluble Phosphorous (CSP) and the Water Soluble Phosphorous (WSP) Test Methods and the Formic Soluble Phosphorous (FSP) Test Methods were amended.
November 2024	9.8	The Elemental Sulphur in Fertilisers Test Method and the Sulphate Sulphur in Fertilisers Test Method were amended.

INDUSTRY AGREED REFERENCE TEST METHODS

CONTENTS

SECTION ONE: GENERAL

1.1 Sampling of Solid Fertiliser and Lime Materials

Important Note
For the purposes of comparison or compliance, a single grab sample of any fertiliser or lime material (especially if blended) must not be regarded as representative of the whole consignment. To collect a representative sample, this guideline should be followed.

Scope and Application
Methods are described for obtaining a primary sample of fertiliser (or fertiliser raw material) from a large-scale source held either in bulk or in bags.

Careful technique is necessary to obtain a completely representative sample from a bulk source of fertiliser (or raw material) as segregation problems will result from any lack of homogeneity. It is important that, where practicable, a sample be taken from a moving stream during transfer or dispatch when the whole of the bulk source is available for sampling. It is essential that samples are taken with care and in a manner which provides confidence that the primary sample collected is truly representative of the batch of fertiliser or lime being sampled.

It is essential that samples are taken with care and in a manner which provides confidence that the primary sample collected is truly representative of the batch of fertiliser or lime being sampled.

If reduction in size of the primary sample is necessary, mechanical methods of subdivision (e.g. riffle) are recommended rather than manual methods (e.g. cone and quartering etc.) to reduce operator bias. These methods should only be used as a last resort and with extreme care.

Summary of Method

1. Bulk material is sampled from a moving stream during intake, processing or dispatch, either from the conveyor belt or a suitable transfer point. If belt conveying is not in use an alternative is to take samples from front end loader buckets.

2. Where the material is held in bags, a representative number of these is sampled by riffling or less desirably by use of a sampling spear.

3. If the material in a bulk heap is known to be homogeneous shovel samples may be taken if the recommended sampling method is not available.

Health and Safety Considerations
Sampling procedures should be taken in compliance with internal company HS&E policies and procedures. Particular care should be taken when sampling from the cut faces of large fertiliser piles.

Key Points/Critical Steps

1. By far the greatest errors in sampling occur at the primary sampling

stage, and if the derived laboratory specimen is not completely representative of the original source much careful analytical work may be completely negated. Recommended sampling techniques must therefore be followed as closely as possible, particularly with heterogeneous materials.

2. Mechanical (e.g. riffle) methods of sample reduction should be used wherever possible to reduce operator bias.

3. Care should be taken to avoid contamination from other sources during the sampling and sample size reduction processes.

4. When sampling from conveyors, samples should only be taken when product uniformity is expected to be optimum (e.g. avoid the initial and final portions of a dispatch run).

Interferences

The major interference results from the non-homogeneity of the material being sampled, either in chemical composition or size range. The resulting segregation can lead to major sampling errors.

Reagents and Equipment

1. Suitable belt formers, sampling boxes, shovels and scoops as required by the sampling procedure used.

2. Riffle (20 to 25 mm apertures) for primary sample size reduction.

3. Clean bags or other suitable containers.

Standards

Not applicable.

Procedures

<u>Sampling from conveyor belts (intake, process or dispatch)</u>

1. Mechanical sampling from conveyor belts is the safest and most reliable method of getting a representative sample. However, it needs to be recognised that, increasingly, conveyors are being fenced off which makes manual sampling difficult and so any sampling system has to be mechanised.

2. If samples are to be collected manually, use a narrow mouth sample box at regular intervals to traverse at a uniform rate the complete stream of material being discharged from the moving belt. Bulk the individual samples (10 to 15 kg) and reduce by riffle to 1 to 2 kg.

3. From a stationary conveyor belt by removing complete a transverse section of the material using two parallel formers (sheet metal or wood) to define the sample area. Only a limited number of replicate samples can be taken because of the impracticability of repeated belt stops. Bulk samples and reduce as above.

4. By taking selected portions across a moving belt. If no suitable discharge point is available and it is not possible to stop the belt remove a series of scoop samples at

points across the width of the moving belt, the number of scoops taken at each point being proportional to the depth of material on the belt at that point. Insert scoop into the material as deeply as possible. Repeat at regular intervals, bulk the samples and reduce as above. This method is most appropriate for homogeneous materials and should not be used for inhomogeneous products (e.g. blends).

<u>Sampling from mechanical loaders</u>

1. Whilst a truck or rail wagon is being loaded directly by means of a front-end loader, one shovelful of material should be collected (by digging deeply, or at tipping) from each of at least six loads spread at random throughout the full batch. Bulk samples and reduce as above.

<u>Sampling from bags</u>

1. Select a number of representative bags from the batch. Take one sub-sample from each bag – to a maximum of ten samples – using a sampling spear to draw several portions from each bag. The bag should be laid flat and the spear inserted to its full extent in several directions.

2. Bulk the samples and reduce as above.

<u>Sampling from bulk heaps or bins</u>

1. This method should only be used when others are not practicable and should only be attempted for homogeneous materials such as potash salts, superphosphate, nitrogen salts, sulphur and imported NPK granular products.

2. Using a shovel or scoop, collect at least six portions of the material from different parts of the heap – as widely separated as is practicable. Avoid taking samples of obviously contaminated product. It is preferable to take samples from ‘cut’ faces of heaps, if possible.

3. Bulk the samples and reduce as above.

Calculations/Uncertainty of Measurement

Quality Control

Future Work

References

The following references were used in the development of this guideline:

1. FMRA “Analytical Methods for Fertiliser Materials” Method 1.1.
2. Ravensdown SOP, ‘Unbiased Sampling’.
3. Ballance SOP, ‘Bulk Product Sampling Procedures’.

1.2 Preparation of the Analytical Sample

Scope and Application

Recommendations are given for the reduction of a primary sample of fertiliser (or fertilizer raw material) of maximum size 10 to 15 kg to an appropriate size and condition for chemical and physical analysis.

Although problems due to non-homogeneity are not as serious as in primary sampling considerable care is still necessary with other than single component fertilizers (and raw materials) if a satisfactory analytical sample is to be obtained.

Summary of Method

1. The primary sample (10 to 15 kg) is reduced by riffling to about 2 kg, and after breaking up any large lumps or aggregates is well mixed and further reduced to about 500 g.

2. After intermediate crushing and remixing the sample is further riffled to about 250 g and ground to provide the final analytical specimen.

Health and Safety Considerations

1. If preparing manure samples, extra care should be taken as animal manures may contain disease pathogens and parasitic organisms that can pose a health risk to humans under certain circumstances. The common exposure pathways are through dust inhalation or hand-to-mouth contact. Particular care should be taken with operations that generate dust or aerosols, such as grinding or weighing dried manures.

Key Points/Critical Steps

1. To minimize segregation effects in multi-component mixtures attention to detail is necessary, particularly the remixing and particle size reduction steps involved.

2. Care should be taken to avoid contamination from other sources throughout the process.

3. With some materials fine grinding may lead to the loss or gain of moisture, so precautions are necessary to minimize this. Grinding should be carried out as rapidly as possible and unnecessary exposure to the atmosphere avoided. In extreme cases it may be necessary to dry moisture sensitive samples before final preparation, with measurement of moisture content on a portion of the primary sample, depending on the desired method of reporting analytical results (i.e. ‘dry’ or ‘as received’ bases).

4. Where practicable a laboratory mill should be used rather than a pestle and mortar for grinding to reduce exposure to the atmosphere and extended working of the sample.

5. It is desirable that sub-sampling be performed mechanically with a suitable riffle rather than manually to reduce operator bias.

Interferences

As in primary sampling, the major interference results from non-homogeneity of the material being sampled.

Reagents and Equipment

1. Laboratory mill.

2. Small riffle (6 to 12 mm apertures) or similar mechanical divider.

3. Sieves, 200 mm diameter, with apertures of 1.00 mm, 500 µm and 150µm (i.e. 16, 30 and 100 mesh BS).

Standards

Procedure for Organic Solid Fertilisers

1. Record obvious visible contamination of the sample and, if possible, quantify and define the contaminant.

2. Where the material to be tested is very non-homogeneous (chicken litter, biosolids etc) then any matted or clumped portions will be broken up or the material subjected to such other treatment as to allow it to be mixed as far as is possible into a uniform state. If the material is such that it separates into a fibrous part and a part consisting substantially of powder, then care must be taken that the sample drawn has the same relative proportions as the original material.

3. Reduce the primary sample by riffling to about 2 kg.

4. Break up any large lumps or aggregates, mix well and further riffle to a sample small enough for the next stages (100 to 500 grams).

5. If milling is required, dry the sample. To dry the sample, air dry at 30°C ± 4°C overnight or until manageable.

6. Pass this sample through a suitable sieve, crushing any oversize as required, returning the crushed oversize material, mix thoroughly and reduce by riffle to the final sample size of 250 g.

7. Grind the analytical sample to pass the following sieves, depending on the type of material involved:

a.	Fertilisers containing organic matter	2.00 mm
b.	Crystalline salts	1.00 mm
c.	Dry powdered and granular fertilisers	500 µm
d.	Lime and phosphate rocks	500 µm

8. Mix thoroughly and store in a suitable stoppered non-corrodible container.

9. Identify the sample (with adequate details) on the container, with reference to the laboratory records.

10. Digest high organic substrate samples using one of the methods described in “Recommended Methods of Manure Analysis”, available from www1.uwex.edu/ces/pubs/. These methods are:
    - dry ashing (adapted from AOAC 985.01)
    - microwave/acid digest (EPA 3051)
    - nitric acid/hydrogen peroxide digest (EPA 3050).

Note: If the sample is required for physical analysis the final size reduction stages in steps 3 to 4 above may not be necessary.

Procedure for General Solid Fertilisers

1. Record obvious visible contamination of the sample and, if possible, quantify and define the contaminant.

2. Where the material to be tested is very non-homogeneous then any matted or clumped portions will be broken up or the material subjected to such other treatment as to allow it to be mixed as far as is possible into a uniform state. If the material is such that it separates into a fibrous part and a part consisting substantially of powder, then care must be taken that the sample drawn has the same relative proportions as the original material.

3. Reduce the primary sample by riffling to about 2 kg.

4. Break up any large lumps or aggregates, mix well and further riffle to a sample small enough for the next stages (100 to 500 grams).

5. If milling is required, dry the sample. To dry the sample, air dry at 30°C ± 4°C overnight or until manageable.

6. Pass this sample through a suitable sieve, crushing any oversize as required, returning the crushed oversize material, mix thoroughly and reduce by riffle to the final sample size of 250 g.

7. Grind the analytical sample to pass the following sieves, depending on the type of material involved:

a.	Fertilisers containing organic matter	2.00 mm
b.	Crystalline salts	1.00 mm
c.	Dry powdered and granular fertilisers	500 µm
d.	Lime and phosphate rocks	250 µm

8. Mix thoroughly and store in a suitable stoppered non-corrodible container.

9. Identify the sample (with adequate details) on the container, with reference to the laboratory records.

10. Digest high organic substrate samples using one of the methods described in “Recommended Methods of Manure Analysis”, available from www1.uwex.edu/ces/pubs/. These methods are:
    - dry ashing (adapted from AOAC 985.01)

- microwave/acid digest (EPA 3051)
- nitric acid/hydrogen peroxide digest (EPA 3050).

Note: If the sample is required for physical analysis the final size reduction stages in steps 3 to 4 above may not be necessary.

Procedure for Suspensions

1. If the sample container is full, decant some into another container and mix both containers thoroughly.

2. Recombine and homogenize with Waring-type blender.

3. Immediately sample as required by the method. Ensure that the sample remains homogeneous until sampling is completed and that the sampling device does not introduce bias.

Procedure for Clear Liquids

1. Ensure that the sample is homogeneous and no crystallization has occurred.

2. Shake and warm if appropriate.

3. Sample.

Calculations/Uncertainty of Measurement

Quality Control

Future Work

References

The following references were used in the development of this method:

1. FMRA analytical methods for fertiliser materials 1982 Draft.
2. ‘Recommended Methods of Manure Analysis’ by John Peters, University of Wisconsin–Madison.

1.3 Statistical Interpretation

The confidence with which analytical data can be stated can be described in a number of ways. Where available, the reference test methods will describe the Repeatability and reproducibility values that can be expected.

Reproducibility (R) values apply between laboratories and repeatability (r) values apply when samples are tested in the same laboratory "Under repeatability conditions". Note that these values are the maximum difference that should exist between two tests, 95% of the time.

Individual laboratories can use the 'repeatability' of the method to assess if the method is optimised in their laboratory environment, either by comparing the repeatability (r) with the range between two values, or by comparing the standard deviation of repeated measurements under repeatability conditions with the repeatability standard deviation (S_r).

The reproducibility standard deviation (S_R) can be used to make Uncertainty of Measurement (UoM) statements of tolerance as:

$$\text{UoM} = S_R \times 1.96 \quad \text{to a 95\% confidence level}$$

Inherent variability within methods, and between laboratories is unavoidable, therefore it is important to have some information on which to base expectation of method variation. Where available, the reference test methods describe the Repeatability and reproducibility values that can be expected.

Reproducibility (R) values provide the maximum range, within which the difference of two results from two different laboratories testing the same sample using the same method should fall, 95% of the time.

Likewise repeatability (r) values provide the maximum range, within which the difference of two results from the same laboratory testing the same sample using the same method should fall within, 95% of the time.

Individual laboratories can use the 'repeatability' of the method to assess if the method is optimised in their laboratory environment, either by comparing the repeatability (r) value with the range between two values, or by comparing the standard deviation of repeated measurements under repeatability conditions with the repeatability standard deviation (S_r).

In addition to the more precise procedures for calculating uncertainty of measurement (UoM) given in ISO Guide 98-3 (GUM), the reproducibility standard deviation (S_R) can also be used to make somewhat broader UoM statements of test results:

$$\text{UoM} = S_R \times 1.96 \quad \text{(at a 95\% confidence level)}$$

Example
When this test method is used to determine a total phosphorus result on a sample of superphosphate, the Uncertainty of Measurement (UoM) can be estimated from the S_R (0.077 g/100g. see table below).

Therefore, a result of 9.1% total phosphorus could reasonably be expressed as:

TP = 9.1 ± 0.15%

Reproducibility and Repeatability Values

A fertiliser industry project was carried out in 2022 to determine Reproducibility and Repeatability values for a range of tests.

The "Reproducibility Value" represents what duplicate determinations between different laboratories should be within.

The "Repeatability Value" is what duplicate determinations within the same laboratory ("under repeatability conditions") should be within.

The values determined for New Zealand laboratories were:

Test	**Repeatability SD (S_r)**	**Reproducibility SD (S_R)**	**Repeatability value (r)**	**Reproducibility value (R)**
TP	0.106 g/100g	0.152 g/100g	0.293 g/100g	0.42 g/100g
CSP	0.058 g/100g	0.085 g/100g	0.162 g/100g	0.235 g/100g
WSP	0.036 g/100g	0.056 g/100g	0.099 g/100g	0.154 g/100g
Moisture	0.088 g/100g	0.345 g/100g	0.245 g/100g	0.957 g/100g
Free Acid (as P)	0.021 g/100g	0.077 g/100g	0.058 g/100g	0.213 g/100g
SO_4^{2-} sulphur	0.068 g/100g	0.139 g/100g	0.188 g/100g	0.386 g/100g
Ca	0.312 g/100g	1.23 g/100g	0.864 g/100g	3.41 g/100g
Cd	0.295 mg/kg	1.25 mg/kg	0.817 mg/kg	3.46 mg/kg
Mg	0.041 g/100g	0.119 g/100g	0.114 g/100g	0.331 g/100g
K	0.328 g/100g	0.516 g/100g	0.909 g/100g	1.430 g/100g
F	0.016 %	0020 %	0.044 %	0.055 %
Elemental S	0.141 g/100g	0.313 g/100g	0.391 g/100g	0.866 g/100g

References

The following material was used in the development of this protocol:

1. "FertChek Method Validation Trial (September 2022) by Lana Pears, LabChek.

SECTION TWO: FERTILISERS

2.1 Determination of Cadmium in Fertilisers

The New Zealand fertiliser industry agreed test method for the determination of the cadmium content in fertilisers shall be:

- the latest version of the American Organisation of Analytical Chemists (AOAC) Official Method 2006.03 “Arsenic, Cadmium, Cobalt, Chromium, Lead, Molybdenum, Nickel and Selenium in Fertilizers”.

- the latest version of either EPA Method 3050B or EPA Method 3051 for high organic substrates. Examples of high organic substrates to which these methods are applicable are: biosolids or sewage sludges, manures, composts, soil conditioners and blood and bone.

- the AOAC method for determination, noted above, using the extract obtained from the Industry Agreed Method for the Determination of Total Phosphorous, Citric Soluble Phosphorus and Water Soluble Phosphorous (TP, CSP and WSP).

References

2.2 Determination of Heavy Metals in Fertilisers

The New Zealand fertiliser industry agreed test method for the determination of the arsenic, cobalt, chromium, lead, molybdenum, nickel, selenium, uranium and thallium content in fertilisers is the latest version of the American Organisation of Analytical Chemists (AOAC) Official Method 2006.03 “Arsenic, Cadmium, Cobalt, Chromium, Lead, Molybdenum, Nickel and Selenium in Fertilizers”.

2.3 Determination of Elemental Sulphur in Fertilisers

Scope and Application
This method is suitable for determining the elemental sulphur content of fertilizers.

Summary of Method
Elemental sulphur is isolated from the fertiliser matrix by dissolution in toluene, methylene dichloride (dichloromethane) or chloroform using a Soxhlet apparatus. Subsequent quantitation is via gravimetric determination of the isolate.

Health and Safety Considerations

1. Toluene, methylene dichloride (dichloromethane) and chloroform are hazardous chemicals. They are highly flammable (they burn similar to petrol), are readily absorbed through skin or by inhalation and are skin sensitizers.

2. Long term exposure to toluene, methylene dichloride (dichloromethane) or chloroform have a number of negative health effects. Always carry out this method in a fume cupboard and in a way that minimizes exposure.

3. Ensure that a dry powder fire extinguisher is available and should a fire start do not hesitate to use it.

4. Use safety glasses if the toluene, methylene dichloride (dichloromethane) or chloroform is removed under vacuum.

Key Points/Critical Steps

1. Do not use grease on joints.

2. Remember to use bumping granules and ensure that the sample has a rolling boil before being left unattended.

3. Cotton wool previously extracted with solvent to remove residues can be used to ‘plug’ the thimble.

Interferences

1. Very fine particles not retained within the Soxhlet thimble will cause an overestimate of the elemental sulphur content of the sample.

2. Fertilisers containing solvent soluble material (e.g. organic fertilisers containing waxes or oils) are not suitable for analysis.

Reagents and Equipment

1. Toluene, methylene dichloride (dichloromethane) or chloroform (tested to show the residue after drying 100 ml is less than 5 mg)

2. Soxhlet extraction equipment.

3. 30 mm x 80 mm Cellulose Soxhlet Thimbles. Grade: Advantech #84 or Whatman #2800 or equivalent. Thimbles must have a particle retention of less than 10 μm and

wall thickness of 1 mm or greater. Ensure that the thimble is at least 10 mm higher than the solvent inlet on the Soxhlet apparatus.

4. Heating mantle.

5. Condenser

Standards

Procedures

1. Accurately weigh approximately 5 grams (to three decimal places) into the extraction thimble and record the result.

2. Oven-dry the sample in the thimble at 70°C for two hours.

3. Add sufficient toluene, methylene dichloride (dichloromethane) or chloroform into the pre-weighed round-bottom flask, with bumping aid, and assemble the soxhlet setup with the thimble and sample in place.

4. Turn on heating mantle and make sure water is flowing through the condenser.

5. Allow the extraction to take place. This will take at least 15 siphon cycles to ensure adequate extraction. Ensure condenser is going.

6. When extraction has finished, turn off the power to the heating mantle and transfer the round bottom flask to the rotary evaporator.

7. Evaporate the toluene, methylene dichloride (dichloromethane) or chloroform with the rotary evaporator until dryness.

8. Place the flask in the 70˚C oven for at least 1 hour, then cool in a desiccator and weigh the flask.

9. Calculate the % of elemental sulphur using the formula below.

Calculations

The percentage of elemental sulphur in the fertiliser can be determined using the formula below:

$$\text{Sulphur \% (elemental)} = \frac{\text{weight of S(el) found}}{\text{Initial weight}} \times 100$$

$$\text{or} = \text{weight of S (el)} \times 20$$

Quality Control

Run an internally generated Check Sample with each batch of analyses.

Future Work

References
The following references were used in the development of this method:

1. FMRA analytical methods for fertiliser materials 1982 Draft (toluene, methylene dichloride (dichloromethane) or chloroform substituted for carbon tetrachloride for safety reasons).

2. Sulphur Data book (McGraw-Hill-1954).

2.4 Determination of Total Sulphur (non-soxhlet) in Fertilisers

Scope and Application

When sulphur is required but is present in various forms and when elemental sulphur soxhlet extraction is unsuitable due to sample matrix interference. For example, this method is suitable for the determination of the total sulphur content of sulphur-coated urea or DAP/sulphur mixes.

Summary of Method

Elemental sulphur is converted by sodium hydroxide and peroxide into polysulphides and thiosulphate; these, along with any sulphites that may be present are then oxidised with hydrogen peroxide to form sulphate sulphur that is determined by ICP-OES.

Health and Safety Considerations

Take care with concentrated acid and when dissolving sodium hydroxide (exothermic reaction).

Key Points/Critical Steps

Interferences

Reagents and Equipment

1. Hot plate.

2. ICP-OES.

3. Diluted hydrochloric acid. This can be prepared by mixing one volume of concentrated hydrochloric acid (SG = 1.16) with one volume of water. Stir to mix.

4. Sodium hydroxide solution. NaOH 30% minimum (SG = 1.33). Make up required amount, for example 30 g NaOH + 70 g RO water. Caution: very hot, exothermic reaction.

5. Hydrogen peroxide solution, w = 30%.

Procedures

1. Where S is > 15%, weigh 1.000 g ± 0.001 g of sample into a 400 ml beaker.

   Where S is < 15%, weigh 2.500 g ± 0.001 g.

2. Add 20 ml of sodium hydroxide solution and 20 ml of water. Cover with a watch glass. Boil on a hot plate for 5 minutes. Rinse any sulphur that has gone up the side of the beaker back into the solution with a small amount of water and boil for 20 minutes. Allow to cool.

3. Add 2 ml increments of hydrogen peroxide solution until no reaction is observed (6 to 8 ml of hydrogen peroxide will be necessary). Allow oxidation to continue for one hour at ambient room temperature, and then bring to the boil for 30 minutes.

4. Remove from boil and allow to cool. Add approximately 50 ml of water and 50 ml of hydrochloric acid solution and boil the solution for 15 minutes or down to a volume of 100 ml.

5. Cool and transfer to a 500 ml volumetric flask and dilute to volume and mix, filter if required through Whatman #2 or equivalent filter paper discarding the first 10 ml.

6. Depending on expected sulphur content further dilute the sample with ICP-OES standard matrix matching solution to achieve approximately 50 ppm S in solution.

Calculations/Uncertainty of Measurement
Calculations are performed as follows:

$$\% \text{ Element} = \frac{(\text{ICP read} - \text{blank}) \times 500 \times \text{dilution}}{\text{Weight}}$$

Quality Control

Future Work

References
The following references were used in the development of this method:

1. Eurofins ‘Total Sulphur in Various Forms Method’, Revision 1 (15 April 2015). Based on European Standard I. S. EN 15925:2011, ‘Fertilisers – Extraction of total sulphur present in various forms’.

2.5 Determination of Sulphate Sulphur in Fertilisers

Scope and Application

This method is suitable for all fertilizers.

Summary of Method

The fertiliser sample is dissolved in acid and the residue filtered off.

Barium chloride solution is added to precipitate the sulphate and this precipitate is dried and weighed.

Health and Safety Considerations

Note that this test uses and produces soluble barium salts. These are toxic and care should be taken with their disposal. Consider adding around 20 ml of 5% sulphuric acid to the washings to precipitate the soluble barium salts and make them non-toxic for disposal.

Key Points/Critical Steps

Interferences

Precipitation of barium sulphate in the presence of large amounts of interfering ions such as phosphate and calcium is rather empirical. The following procedure gives reasonably complete recovery of sulphate with adequate precision, probably due to a compensation of errors. Slight variation of condition may give reproducible results, but recoveries may be consistently less than or greater than 100%, necessitating the use of an empirical correction factor. The method should be checked regularly against a synthetic fertiliser of accurately known acid/rock ratio or against sulphuric acid solutions of known content, in case a correction should be necessary.

Reagents and Equipment

1. 5% barium chloride solution. Dissolve 58.64 g of $BaCl_2.2H_2O$ in 900 ml water, make up to 1 litre and filter through Whatman No 42 paper.

2. Water bath or hot plate.

Standards

Procedures

1. Dissolve 2.500 g of superphosphate-type fertilisers (or 1.000 g of fully soluble fertilisers, like ammonium sulphate) by boiling in a 250 ml beaker with 12 ml concentrated HCl and 50 ml water. Cover with a watch glass and stir occasionally. When dissolved, dilute to approximately 100 ml with water for superphosphate-type fertilisers (or 50 ml of fully soluble fertilisers, like ammonium sulphate) and boil briefly. Cool and make to volume in a 250 ml volumetric flask.

2. Dry filter (Whatman No 1) and take a 50 ml aliquot for analysis.

3. Dilute to c. 225 ml in a 400 ml beaker and bring to the boil. Add 12 ml of 5% $BaCl_2$ solution in discrete drops (using a burette), over a period of about two minutes, allowing the mixture to maintain a rolling boil. Test for complete precipitation.

4. Transfer to a boiling water bath for 1 to 1½ hours. Filter directly through a previously weighed and dried #4 sintered glass crucible. Wash with cold water 10 to 12 times.

5. Discard washings, rinse vessel, do a final rinse, add a few drops of silver nitrate solution, if cloudy continue to wash. Repeat until no precipitate is formed to prove that washing has been to completion.

6. Dry the crucible at 100°C to constant weight (for about 15 minutes). Weigh as $BaSO_4$.

Quality Control

Future Work

References

The following references were used in the development of this method:

1. FMRA “Analytical Methods for Fertiliser Materials” Method 13.3.

2.6 Determination of Moisture in Fertilisers

Scope and Application

This method is suitable for the determination of the moisture content of fertilisers that do not contain elemental sulphur. Fertilisers containing elemental sulphur should have their moisture content determined using the ‘Determination of Moisture in Sulphur’ method.

It is important to note that measured moisture levels are a combination of free water and water of hydration. The measured moisture content of a fertiliser can change over time and any comparisons made need to take this into account. The results of a moisture test are influenced by: factors surrounding the original sample, time and physical factors, storage factors, ambient atmospheric temperature and humidity and the hydroscopicity of the sample.

By suitable choice of oven temperature and drying time this method is used for the routine determination of free and/or total moisture in phosphate rocks and other fertiliser materials (including superphosphate) where no other volatile substances are given off at the temperature and duration of drying.

The number of samples that can be processed at one time is only limited by the capacity of the drying oven.

Free Moisture associated with the aqueous superphosphate phase commences evolution at slightly above room temperature, reaches a peak at 45°C, and is virtually completely evolved by 50-60°C. Heating to constant weight at 60°C (or for 2 hours at 50°C in a vacuum oven according to the AOAC) will give a reasonable indication of free moisture. Laboratories are unlikely to use the more rapid AOAC variant as this requires a suitable vacuum oven.

Water of Hydration associated with calcium phosphates, calcium sulphate and other superphosphate components is evolved from 60 to 70°C, reaches peak rate at 110 to 120°C, and is virtually completely evolved by 130°C. Heating to constant weight at 150°C gives a reliable estimate of total (i.e. free plus hydrate) water.

Heating at 100°C for 5 hours gives a reproducible but empirical value for superphosphate, which includes not only free moisture, but also a proportion (usually approximately one seventh) of the water of hydration.

Water of Constitution is formed by pyrolysis of orthophosphate, producing pyrophosphate and water. Reaction rate for this is insignificant below 160°C and will not affect assessment of total water in superphosphate by drying at 150°C.

Since during maturing and storage of superphosphate fertilisers there is interchange of water between free and hydrate forms, attempts to compare fertiliser analyses by conversion to a standard moisture basis are seldom reliable if made on 100°C oven drying data. Total moisture values (150°C) should be used for this purpose.

Summary of Method

1. A suitable weighed quantity of material is dried in the oven for 5 hours (or to constant weight, depending on requirements) and reweighed after cooling. Moisture content is reported as the percentage loss in weight at the temperature and heating time used.

Health and Safety Considerations

Key Points/Critical Steps

Sampling and sampling preparation operations should be performed rapidly with minimal energy input to avoid possible water losses from high moisture materials. If only a moisture value is required the standard of sampling need not be as high as that required for chemical analysis.

Uniformity of temperature in the drying oven is essential, especially if a large number of samples are to be processed concurrently. Fan circulation rather than air convection is recommended.

Interferences

Samples which evolve volatile substances other than moisture (e.g. ammoniacal products, like ammonium sulphate) at the temperature of drying will give erroneously high moisture values. Other methods (e.g. vacuum desiccator methods) should be used for such materials.

Note the discussion under ‘Scope and Application’ on the limitations of this method for superphosphate analysis.

Reagents and Equipment

1. Thermostatically controlled oven – preferably a fan circulation design – capable of maintaining the selected drying temperature.

2. Desiccator for cooling hot samples.

3. Suitable desiccant, such as concentrated H_2SO_4 or silica gel.

Standards

Procedures

1. Drying at 100°C for 5 hours
    a. Weigh to the nearest milligram about 5g of the sample and heat for 5 hours in a flat dish at 99 to 101°C.

    b. Cool the dried sample in a desiccator and reweigh. Report the percentage weight loss as moisture at the temperature and heating time used. Note that for superphosphates this empirical result is not the true free moisture value (refer to ‘Scope and Application’ above). The result is total moisture for phosphate rock and most other minerals and fertiliser salts (except as noted below).

2. Drying at 130°C

    If the material consists of sodium nitrate, ammonium sulphate or potassium salts heat at 129 to 131°C to constant weight. Proceed as above, reheating for periods of 2 hours until a constant weight is attained. Report the final percentage loss in weight as moisture at the temperature used.

3. Total Water in Superphosphate
   Proceed as in ‘Drying at 130°C’, above, at a temperature of 148 to 152°C. The initial heating period should be 16 to 18 hours, with subsequent increments of 2 hours to constant weight.

Quality Control

Future Work

References

The following references were used in the development of this method:

1. FMRA “Analytical Methods for Fertiliser Materials”, Method 2.1.

2.7 Determination of Moisture in Sulphur

Scope and Application

Summary of Method
A suitable weighed quantity of material is dried in the oven and reweighed after cooling. Moisture content is reported as the percentage loss in weight at the temperature and heating time used.

Health and Safety Considerations

Key Points/Critical Steps
Hydrocarbon volatilization may be significant with dirty sulphurs, and it may be necessary to correct the observed weight loss for this. This can be done by determining the carbon content before and after drying and multiplying the difference by the factor 1.17. This loss may be subtracted from the value obtained in the moisture determination to obtain a corrected moisture content.

Interferences
Prolonged drying or overheating can cause vapourisation of sulphur and hydrocarbons volatile at 105°C ± 2°C will be driven off.

Reagents and Equipment
1. Thermostatically controlled oven.

2. Desiccator.

3. Suitable desiccant such as concentrated H_2SO_4 or silica gel.

Procedures
1. Weigh 5.0 ± 0.01g of sulphur into a previously dried, cooled and weighed 100 by 15mm Petri dish and place in an oven at 105°C for 1 hour.

2. Alternatively, the drying may take place at 80°C for 12 to 16 hours.

3. Remove the dried sample, cool in a desiccator and reweigh.

4. Calculate and report the percentage weight loss at the temperature and heating time used.

Calculations/Uncertainty of Measurement

Quality Control

Future Work

References
The following references were used in the development of this method:

1. FMRA “Analytical Methods for Fertiliser Materials”, Method 13.11.

2.8 Determination of Total Phosphorus, Citric Soluble Phosphorus (CSP) and Water Soluble Phosphorus (WSP)

Scope and Application

This method is used for the routine determination (as orthophosphate) of total phosphorus in phosphate rocks, superphosphate and other fertilizers and of phosphorus soluble in water and 2% citric acid.

The colour of the phosphovanadomolybdate complex is more stable than that of the reduced molybdenum blue complex and generally applies to a more realistic range of concentrations for fertilizers. It is preferred if ferric and silicate ions are present, both of which interfere more in the molybdenum blue method.

When testing RPR; citric soluble phosphorus (CSP) and water-soluble phosphorus (WSP) are to be analysed on an as-received representative sample.

Summary of Method

The phosphorus (as orthophosphate) for analysis is obtained by dissolving the sample in boiling oxidizing acid or “soluble P” extractant, or by dissolving a fusion product. Aliquots suitable for measurement are prepared by dilution to contain about 1 mg P.

When an excess of molybdate solution is added to an acidified solution of a vanadate and an orthophosphate a stable yellow coloration develops within 5 to 10 minutes.

The spectrophotometric method compares the amount of light, at a wavelength of 420 nm, absorbed by the developed colour relative to that by a phosphorus standard colour.

Health and Safety Considerations

If perchloric acid is used, adhere closely to standard laboratory safety procedures. There may be interference using perchloric acid on high elemental sulphur samples.

Key Points/Critical Steps

Follow standard sampling procedures, particularly with respect to particle size of analytical sample as this is important for extraction methods to measure P solubility. Rocks should be reduced to minus 250 µm (preferably 150 µm) and superphosphates and mixtures to minus 500 µm if possible.

Analyse all samples on an “as received” basis and correct to “dry basis” or “standard moisture basis” as required by means of measured moisture content.

Extraction step. It is important that the filtration step start immediately after the shaking extraction is completed.

Reagent blanks. It is important to measure solutions versus reagent blanks (not water). For maximum precision standards should be treated similarly to measured samples. This is normally unnecessary, except following neutral ammonium citrate extraction.

Standards. Standards should be prepared from the same batch of colour reagent as is used for the test sample The standard and the test sample should be analysed together and within two hours of the colour addition.

Interferences

Acidity

Some excess of acid should be present to prevent the appearance of the orange-yellow colour which the reagent forms in near-neutral solutions. Care should be taken that the solution is not too acidic as this slows the colour development and may lead to error. The optimum range is 0.6 to 0.9M (with respect to HNO_3) in the final solution. As the reagent contributes 0.5M the aliquot contribution can be up to 0.4M (equivalent to a maximum of 1 mL conc. H_2SO_4, c. 3mL 60% HNO_3 or $HCl0_4$). If acid in the aliquot is greater than this it is advisable to neutralize and add a small excess (c. ½ mL) HNO_3 before reagent addition.

Organics

Organic matter can impart a yellow colour to the solution, but this may be removed by calcining the rock before analysis or adding sufficient oxidising acid to remove it during the dissolution process.

Citrate ions

Citrate ions suppress colour development, but the relative error is less than 1% for citrate levels below 100 mg in the final 100 mL 2% citric acid extract aliquots would contain 20 to 80 mg citrate ion and would interfere little, but ammonium citrate extracts (Neutral, Peterman or Joulie) contain up to 15 times this amount, and the excess must be removed by oxidation etc.

Ferric ions

Ferric chloride has a characteristic yellow colour, but interference is minimized by measuring at 420 nm. Interference by large amounts of ferric ion (greater than 10 mg in final 100 mL) can be overcome by converting it to the perchlorate or by the addition of sodium fluoride. (Note that fluoride retards colour development). In most rock or fertilizer systems the maximum ferric ion would not exceed 1 mg in the final 100 mL

Silica and silicates

Dissolved SiO_2 (or silicate ions) in large amounts may interfere because of formation of yellow silicomolybdic acid. Colour development for this, however, is very slow and normally there is no interference if SiO_2 is below 1 mg in the 100 mL final solution. This is unlikely to occur in acid extracts, but alkaline fusions of high SiO_2 minerals should preferably be treated with perchloric acid or suitable alternative to dehydrate the silicic acid making it easy to filter off rather than to rely solely on acidification with HNO_3.

Note

Use of perchloric acid for extraction and oxidation purposes has advantages in reducing interferences due to organics, ferric ions and silica. However, because of safety and cost factors it is considered inadvisable to use straight $HClO_4$ as a routine P extractant. Wherever its use is essential it is safer to use it in “ternary acid” combination with appropriate precautions.

Reagents and Equipment

1. A grating or prism spectrophotometer (visible range) capable of selecting a 420 nm wavelength.

2. An end-over-end shaker or flatbed orbital shaker to accommodate 200 to 250 mL stoppered conical flasks (for water and 2% citric acid extractions). ~~The~~ If an end-over-end shaker is used it should operate at 40 RPM for 30 minutes. If a flatbed orbital shaker is used it should operate at 140 to 150 RPM for 30 minutes on superphosphate samples and 300 RPM for phosphate rock samples.

3. Vanadomolybdate colour reagent:
    i. Dissolve 80 g ammonium molybdate {$(NH_4)_6Mo_7O_{24}.4H_2O$} in 800 mL hot water (1 litre beaker), cool and make up to volume with water in a 2 litre volumetric flask.
    ii. Dissolve 4 g ammonium metavanadate (NH_4VO_3) in 1200 mL hot water (2 litre beaker), cool and add 560 mL concentrated (69% to 70%) nitric acid. Make up to 2 litres with water in a volumetric flask.
    iii. Mixed reagent: Slowly add while stirring, one volume of reagent (i) to one volume of reagent (ii). The mixture will keep for about one week, so that the volume required to be mixed will depend on the number of samples anticipated (25 mL mixed reagent per sample).

4. Standard phosphate solutions:
    i. Stock standard phosphate solution: Weigh out 2.197 g potassium di-hydrogen phosphate (preferably containing at least 99.8% KH_2PO_4, previously dried at 105°C for an hour) and dissolve in 500 mL hot distilled water. Cool and make accurately to 1 litre in a volumetric flask. This solution contains 0.50 mg P/mL If the exact batch analysis of the KH_2PO_4 is known, modify the weighed quantity accordingly (i.e., 99.8% = 2.201 g).
    ii. Working standard solution: Dilute 50 mL (pipette) stock solution to 500 mL in a volumetric flask with distilled water. This working standard will contain 0.05 mg P/mL.

5. Extraction ternary acid:
    i. Mix 50 mL of concentrated (36% to 38%) hydrochloric acid and 50 mL concentrated (69% to 70%) nitric acid and add to this 70 mL of the 60 to 70% perchloric acid. Store in fume cupboard when freshly made up. TAKE CARE.

6. 2% Citric acid solution:
    i. Dissolve 40 g ~~commercial~~ analytical grade crystalline citric acid monohydrate in water and dilute to 2 litres. Produce in smaller quantities and renew at least monthly ~~frequently~~.

7. Decomposition ternary acid:
    i. Add 20 mL of concentrated (98%) H_2SO_4 to 100 mL concentrated nitric acid and then add 40 mL of 60-70% perchloric acid. TAKE CARE.

8. Strontium solution:
    i. Dissolve 60 g $Sr(OH)_2$. $8H_2O$ in the minimum amount of $HClO_4$ 60 to 70% (40 to 50 mL) and dilute to 1 litre in a volumetric flask. This solution contains 20 g/l Sr so that 10 mL addition to the 100 mL measured P solution

gives a 2,000 ppm Sr level, suitable for measurement by atomic absorption of Al, Fe and Ca.

Standards

1. Pipette into 100 mL volumetric flasks: 0mL, 5 mL, 10 mL, 15 mL, 20 mL, 25 mL and 30 mL working standard solution (as appropriate) and dilute to about 50 mL with distilled water. Use at least three standards and a blank. Add 25 mL (pipette or tilt measure) mixed reagent to each flask and make to volume with distilled water. This gives “reagent blank”, 0.25, 0.50, 0.75, 1.00, 1.25, 1.50 mg P standards in 100 mL volumes.

2. Measure the absorbances of these solutions in 10 mm optical cells in the spectrophotometer, at 420 nm against the “reagent blank”. A graph of absorbance against mg P is generated.

3. In routine analysis (when the calibration curve has been obtained) it is only necessary to include at least one standard sample (say 1.0 mg P) with each batch of determinations, taking care to keep all measured solutions at the same temperature. If the absorbance of this standard differs from the plotted value, all absorbances for the particular batch must be corrected proportionately. The absorbance of a 1 mg P standard in 100 mL volume should be about 0.5 in a 10 mm cell.

Procedures

1. Phosphate rocks – Total Phosphorus

   1.1 Low R_2O_3 (up to 15%) rocks with little organic matter:

      a. Weigh 2.50 g phosphate rock into a 250 mL conical flask or beaker and add 20 mL of concentrated (36% to 38%) hydrochloric acid and 5 mL of concentrated (69% to 70%) nitric acid.

      b. Add water, boil the solution for about ten minutes.. Cool, filter and transfer to a 500 mL volumetric flask and make to volume.

      c. Prepare a 1:100 dilution with the final solution to be made up to 100 mL. Dilute with water to about 50 mL, add 25 mL mixed vanadomolybdate reagent and make to volume.

      d. Measure the absorbances as described in the Standards section. Note that 1 mg P is equivalent to 20% P in the sample.

      e. In the final 100 mL there is a 25 mL aliquot (a_2) of the 250 mL sample (V_2) which contained a 10 mL aliquot (a_1) of the original 500 mL solution (V_1). There is:

$$\frac{a_1}{V_1} \times \frac{a_2}{V_2} = \frac{1}{500}$$

of the sample weight (W_1) in the final solution. If this contains W_2 g of P it follows that P in the sample

$$= \frac{W_2}{W_1} \times \frac{V_1V_2}{a_1a_2} \times \frac{100}{1} \ \%$$

For W_2 = 1.0 mg, P in sample =

$$\frac{0.001}{2.5} \times \frac{500 \times 250}{10 \times 25} \times \frac{100}{1} \ \% = 20\%$$

1.2 Low R_2O_3 (up to 15%) rocks with appreciable organic matter:

a. Repeat the above procedure but use 20 mL of concentrated (69% to 70%) nitric acid and 5 mL of concentrated (36% to 38%) hydrochloric acid in the initial dissolution step.

b. When determining other elements by atomic absorption (e.g. Fe, Al, Ca) on the same solution as P it is necessary to dissolve the phosphate rock in a ternary acid mixture and add strontium to the system. Weigh 1.000 g phosphate rock into a 100 mL conical flask, add 20 mL “extraction” ternary acid (see above) and cover the flask with a small filter funnel. Heat the solution until dense white fumes persist. Add about 25 mL water and re-boil. Cool and add 10 mL strontium solution (see above) and make to 100 mL in a volumetric flask. Dilute ten-fold, transfer a 5 mL aliquot to a 100 mL volumetric flask and add water and vanadomolybdate reagent to complete the determination as above.

1.3 High R_2O_3 (over 15%) rocks – uncalcined and low temperature (500 to 600°C) calcined:

a. Weigh 5.0 g fusion mixture (Na_2CO_3 + K_2CO_3, 1+1) into a small, covered platinum crucible (10 to 15 mL) and heat over a Meker gas burner until a clear melt is obtained. Remove and cool and then weigh 0.250 g rock on top of the melt. Heat over the burner until a clear melt is obtained and then cool rapidly. Immerse the crucible in 20 mL HCl + 5 mL HNO_3 in a 150 mL beaker. When the effervescence stops, remove the crucible rinsing with a minimum of water and boil the solution until clear. Cool, transfer to a 250 mL volumetric flask and make to volume. Without further dilutions, transfer a 5 mL aliquot to a 100 mL volumetric flask and add water and vanadomolybdate reagent completing the determination as above.

1.4 High temperature (1100°C) calcined rocks:

1. Weigh 7.0 g NaOH (AR) into a covered 50 mL nickel crucible and heat in a furnace at 400°C until melted (about ½ hour). Remove and cool. Weigh 0.500 g rock on top of the NaOH and return to the furnace at 550°C for 1½ hours, remove and cool.

Cover the crucible with water in a 500 mL tall-form beaker and heat until the melt dissolves. Remove the crucible, rinsing with water and allow to cool. Add 25 mL concentrated nitric acid and dilute to 500 mL in a volumetric flask. Transfer a 5 mL aliquot to a 100 mL volumetric flask and add water and vanadomolybdate reagent completing the determination as above.

6. Total Phosphorus in fertilizers

    1. Weigh 5.00 g fertilizer (such as superphosphate) into a 250 mL conical flask or beaker and dissolve in 20 mL of concentrated (36% to 38%) hydrochloric acid, 5 mL of concentrated (69% to 70%) nitric acid and 100 mL water, filter if necessary and continue as above. Note that 1 mg P = 10% P in the sample.

7. 2% Citric Soluble Phosphorus in fertilizers

    1. Proceed as above, using 200 mL of 2% citric acid solution at a temperature of 20° C (± 2° C) (prepared as W/V of monohydrate) in place of water. Note that 1 mg P = 10% of 2% citric acid soluble P in the sample.

8. Water Soluble Phosphorus in fertilizers

    1. Weigh 2.00 g fertilizer into a 250 mL conical flask, add 200 mL water at a temperature of 20° C (± 2° C) and shake continuously as per Note 2 in the Reagents and Equipment section. Dry filter (i.e., do not pre-wet filter or funnel) through No. 1 Whatman or similar grade paper. Filtration is to begin immediately after the completion of the shaking step. Ensure collection of a clear filtrate. This may require discarding the first 10 mL of filtrate. Dilute 10 mL (pipette) aliquot to 250 mL (volumetric flask). Transfer 25 mL aliquot of this to a 100 mL volumetric flask and add water and vanadomolybdate reagent completing the determination as in part 1.1 (e), above. Note that 1 mg P = 10% water soluble P in the sample.

Quality Control

Future Work

References

The following references were used in the development of this method:

1. FMRA “Analytical Methods for Fertiliser Materials”, Method 11.1.

2.9 Determination of Free Phosphoric Acid in Fertilisers

Scope and Application

This method is suitable for determining the amount of free phosphoric acid remaining in superphosphate fertilizers.

Summary of Method

Use a water-soluble extract prepared from the WSP test.

The resultant solution is titrated with standardized sodium hydroxide solution to a pH 4.5 indicator end-point.

Health and Safety Considerations

Key Points/Critical Steps

The titration should be done with care as some operators find the end-point indistinct.

Interferences

Extraction with water can hydrolyse some of the monocalcium phosphate present in superphosphate to phosphoric acid giving slightly high results.

Reagents and Equipment

1. 250 ml conical flasks (stoppered).

2. Burette (preferably no larger than 10 ml.).

3. Standardised 0.1M sodium hydroxide.

4. BDH pH 4.5 indicator or pH meter.

Standards

Procedures

1. Pipette a 50 ml aliquot from the water-soluble extract prepared for the WSP test into another flask, use a pH meter or add 4 to 5 drops BDH pH 4.5 indicator and titrate with standard 0.1M sodium hydroxide. The colour change is from orange-red to grey.

Calculations/Uncertainty of Measurement

The percentage of free phosphoric acid in the fertiliser can be determined using the formula below:

$$\% \ H_3PO_4 = 19.6 \times V \times M$$

Where: V = volume of NaOH used (ml)
M = molarity of NaOH used.

Note: to express % H_3PO_4as %P use the formula:

$$\% \ P = \frac{\% \ H_3PO_4}{3.16}$$

Quality Control

Future Work

References

The following references were used in the development of this method:

1. FMRA analytical methods for fertiliser materials 1982 Draft.

2.10 Determination of Formic Soluble Phosphorus in Fertilisers

Scope and Application

This method is suitable for the determination of the formic acid soluble phosphorus content of fertilizers.

Summary of Method

This method uses a 30-minute extraction using formic acid solution at 20°C, followed by a colorimetric analysis.

Health and Safety Considerations

Use a Type A (organic vapour) respirator, eye protection and gloves when weighing formic acid.

Key Points/Critical Steps

Ensure 2% formic acid solution is used.

Interferences

Reagents and Equipment

1. End-over-end shaker.
2. Calibrated thermometer.
3. 2% w/v formic acid solution. This is prepared by diluting 37 mL of formic acid (‘AnalaR Grade – 90%, SG = 1.2) to 2 litres. Refer to the ‘Quality Control’ section below for guidance on assessing the purity of formic acid.

Standards

Procedures

1. Weigh out 2.000 ± 0.001 g of sample into a dry 250 mL Screw Top Erlenmeyer flask (note: for phosphate rock sample, use 2.000 g of ‘as received’ material.
2. Accurately dispense 200 mL of 2% formic acid solution (at 20 ± 2°C) to the flask.
3. Place the flask on an end-over-end shaker and rotate at approximately 40 rpm for 30 minutes.
4. Immediately filter through Whatman No. 2 (or equivalent) filter paper, discarding the first few mLs (note: when filtering the extract, the filter funnel must be filled once only to avoid further extraction beyond the 30 minute time period.
5. Determine the phosphate content of the extract colorimetrically.

Calculations/Uncertainty of Measurement

Quality Control

The following guidance is offered for testing the purity of formic acid.

Assay

Dilute 2 g with 25 ml of water and titrate with N sodium hydroxide using phenolphthalein as indicator.

$$1 \text{ ml N NaOH} = 0.04603 \text{ g } CH_20_2$$

Identity

Confirm by IR Spectroscopy.

Colour

Compare with a 10 Hazen unit standard.

Water-insoluble Matter

Dilute 25 ml with water to produce 100 ml and set aside for 1 hour. The solution is clear and colourless.

Non-volatile Matter

Evaporate 41 ml (50 g) to dryness on a steam bath. Not more than 1 mg of residue remains.

Acetate

Determine by Gas-Liquid Chromatography.

Stationary phase/support: Porapak Q 80-100 mesh
Column: glass, length 1 m, diameter 4 mm
Column temperature: 150°
Detector: FID
Carrier gas: nitrogen
Flow rate: 30 ml/min
Sample size: 1 µl

Chloride

Dilute 16.4 ml (20 g) with 33 ml of water, add 1 ml of dilute nitric acid and 1 ml of silver nitrate solution. Any opalescence produced is minor.

Sulphate

To 4.1 ml (5 g) add 5 mg of sodium carbonate anhydrous and evaporate to dryness on a steam bath. Dissolve the residue in 20 ml of water, add 1 ml of dilute hydrochloric acid and mix. Apply the Sulphate Test.

Sulphite

Dilute 33 ml (40 g) with 40 ml of water and add 0.1 ml of 0.1 N iodine. The mixture is coloured yellow.

Future Work

The following work is suggested for this method:

1. A comparison between an end-over-end shaker and an orbital shaker at different speeds could be run to determine the sensitivity of the method to the shaking method used.
2. The effects of filtering time to be investigated if the above aspect fails to narrow results sufficiently.
3. The effect of temperature on the reaction could be investigated if the above matters again fail to narrow the results sufficiently. Three temperatures: 15, 20 and 25 °C would be sufficient to establish the effect.
4. The effect of extraction time could be examined.

References

The following references were used in the development of this method:

1. Eurofins ‘Formic Acid Extraction Method’, revision 2.
2. BDH ‘AnalaR’ Standards for Laboratory Chemicals: Formic Acid, 98 to 100%.

2.11 Watkinson Dissolution Test for RPR

Important Note: At the January 2021 meeting of the Fertiliser Quality Council, it was determined that this method should be annotated with: “This Method Is Under Review As It May Not Produce Reproducible Results”

Scope and Application

The use of RPR as a direct application fertiliser is confronted by the difficulty of not knowing the reactivity of a specific product and site-specific factors affecting release of P from RPR. Total P content, particle size, citric and formic acid solubility are individually insufficient parameters for predicting reactivity. To provide a reactivity index, Watkinson (1994) developed a model based on particle size distribution, total P content and dissolution of RPR to predict RPR reactivity expressed as % P potentially released during the first year.

This test is suitable for RPR products in their pure form.

Summary of Method

Dissolution of P from RPR is measured as mg/L P released over a period of 3 hours into a salt solution of 5 mMol ionic strength containing 0.5 mMol Ca^{2+}, and maintained at pH 5.5 while being stirred vigorously. Constant stirring is required to prevent development of elevated Ca concentrations around RPR particles since that would prevent further dissolution of P. This concentration value of P is taken as representative of the equilibrium solubility value (C_R) at the RPR surface.

Health and Safety Considerations

Handle all acids with care.

Key Points/Critical Steps

Interferences

Other fertiliser products co-mixed with RPR are likely to interfere with the analysis by altering the ionic strength of the test solution.

Reagents and Equipment

Reagents

1. Sodium acetate tri-hydrate ($CH_3COONa.3H_2O$) or anhydrous sodium acetate (CH_3COONa)
2. NaCl
3. Calcium chloride dehydrate ($CaCl_2.2H_2O$)
4. Acetic acid 0.1 – 1 % v/v
5. pH 4 and 7 buffer solutions
6. 0.1 mol/L HCl

Equipment

Auto titrator in pH stat mode fitted with a pH electrode and propeller style stirrer. Stirring speed to be sufficient to create a significant vortex ensuring dispersion of RPR particles through the testing solution. To reduce air entrainment and bubble formation, avoid surface splashing by partially submerged impeller, and ensure that a deep vortex does not reach an impeller. Calibrate the pH electrode with pH 4 and 7 buffers.

Standards

Dissolution Solution

Dissolve NaCl (0.7305 g), sodium acetate trihydrate (0.6804 g) OR anhydrous sodium acetate (0.4103 g) and Calcium chloride dihydrate (0.3675 g) in 4.8 L of RO water. Adjust the pH to 5.5 with dilute acetic acid and make up to 5L.

Procedure

1. Add 0.5 g RPR to 250 ml dissolution solution.
2. Activate auto-titrator stirrer.
3. Maintain pH at 5.5 ± 0.1 by addition of 0.1 mol/L HCl solution.
4. Stop the process after three hours and remove the reaction vessel from the Auto titrator.
5. Filter sufficient solution through a 0.45µm membrane filter for analysis.
6. Measure dissolved phosphate in solution on FIA utilizing colour reaction described by Murphy & Riley (1962), and report result as mg/L.

Calculations/Uncertainty of Measurement

Quality Control

Future Work

It is noted that development work on this method is underway and amendments can be expected.

References

The following references were used in the development of this method:

1. Watkinson, JH, 1994: A test for phosphate rock reactivity in which solubility and size are combined in a dissolution rate function. Fertilizer Research 39: 205-215, 1994.
2. Murphy & Riley (1962): A modified single solution method for the determination of phosphate in natural waters, ANALYTICA CHIMICA ACTA 27, pp 31-36.

2.12 Determination of Total Nitrogen in Fertilisers

The New Zealand fertiliser industry agreed test method for the determination of the total nitrogen content of fertilisers is the latest version of the AOAC Standard Reference Method 993.13

2.13 Biuret in Urea

Scope and Application
This method determines the biuret content in urea.

Summary of Method

1. Biuret forms a blue coloured compound with copper sulphate in the presence of alkaline potassium sodium tartrate.

2. A large excess of ammonium salts gives rise to a positive error. Insoluble material is removed by flocculation with aluminium sulphate.

Health and Safety Considerations

1. Copper complex reagent contains sodium hydroxide. Wear goggles and latex gloves when transferring copper complex. If contamination occurs flush well with running water.

2. Wear latex gloves and goggles to transfer sodium hydroxide solution. Avoid contact with eyes and skin.

3. Wear heat resistant gloves when placing or removing beakers from the waterbath.

Key Points/Critical Steps

1. Heating urea above 90°C may cause biuret formation. Heating on a hot plate should be done with gentle swirling. If milling temperatures exceed 90°C then it should be avoided.

2. Colour blanks are analysed due to colour interferences from contaminants in urea (mainly oil).

Interferences

Regents and Equipment
Equipment:

1. Visible spectrophotometer.

2. Hot plate.

3. Filtration apparatus.

4. 4.7 cm GFC Glass Fibre Filter Papers.

5. 40 ml volumetric pipette.

6. 100 ml beakers (one per sample).

7. 100 ml volumetric flasks (one per sample).

8. 400 ml beaker (one per pre-treated sample).

9. Top pan balance.

Reagents:

1. 2% Aluminium sulphate
   Dissolve 10 g of aluminium sulphate ($Al_2(SO_4)_3.18H_20$) and dilute to 500 ml with demineralised water. Make this solution fresh fortnightly.

2. 0.2 M Sodium hydroxide
   Dissolve 4.0 g of sodium hydroxide and dilute to 500 ml with demineralised water. Make this solution fresh fortnightly.

3. Copper complex reagent
   To a 10 litre reagent bottle add about 5 litres of demineralised water. Dissolve 80 g of sodium hydroxide in a reagent bottle. Dissolve 400 g of potassium sodium tartrate in a reagent bottle. Dissolve 100 g of copper sulphate pentahydrate in a separate quantity of 1,500 ml of demineralised water. Add the dissolving copper complex slowly to the reagent bottle with regular mixing. Dilute to the 10 litre mark with demineralised water and mix and leave overnight. The copper complex reagent is stored in an amber bottle.

Standards

1. Biuret QC standard
   Dry approximately 4.4 g of Biuret in the oven for two hours at 100°C. Cool in a desiccator. Accurately weigh 4.0000 g of biuret on an analytical balance. Dissolve in 1,000 ml of warm demineralised water with stirring. Cool when dissolved. Transfer to a 2,000 ml volumetric flask and dilute to the mark with demineralised water when the solution is at 20°C. Expiry: three months.

2. 2 mg/ml Biuret standard
   Dry approximately 2.4 g of Biuret in the oven for two hours at 100°C. Cool in a desiccator. Accurately weigh 2.0000 g of biuret on an analytical balance. Dissolve in 500 ml of warm demineralised water with stirring. Cool when dissolved. Transfer to a 1,000 ml volumetric flask and dilute to the mark with demineralised water when the solution is at 20°C. Record the weight of biuret (on powder bottle) and the volume of the solution that was made up to on the standard bottles using a sticky label. Expiry: three months.

Calculation:

$$\text{mg /ml biuret} = \text{wt used (g)} \times \frac{\%\ \text{purity}}{100}$$

Procedure

1. If a urea sample contains ammonia or ammonium carbamate it requires pre-treatment to remove these interfering ions. This is not required for granular urea as the free ammonia content is low (removed in the granulation process).

2. For urea samples not requiring pre-treatment, weigh out two portions of approximately 5 to 6 grams into a 100 ml beaker using a top pan balance (each weight to be within ± 0.02 grams of each other). One is the sample (record weight) and the second is the colour blank (do not record weight).

3. Add 20 ml of demineralised water to the beaker.

4. Add 0.2 ml of aluminium sulphate solution.

5. Add 0.2 ml 0.2 M sodium hydroxide solution.

6. Dissolve the samples by warming gently over a hot plate with constant swirling. Excessive heating of the sample will form biuret.

7. Filter each sample through a Whatman GFC Glass Fibre filter paper in a filtration apparatus and flush the beaker and paper with demineralised water into separate 100 ml volumetric flasks. Pierce the filter paper to release the vacuum and completely drain washings into the flask. The final volume of filtrate should be about 50 ml. Rinse all apparatus with demineralised water.

8. Top up the colour blank (don't add copper complex reagent) to 100 ml with demineralised water.

9. Prepare a sample blank by adding about 40 ml of demineralised water directly to a 100 ml volumetric flask.

10. Add 40 ml of copper complex reagent to each flask using a dispenser. Swirl while adding reagent.

11. Dilute to the mark with demineralised water and store the sample and blank in the dark for 15 minutes. Only remove them from the dark as they are ready to be read off. If they are left in the light too long the intensity of the copper complex reaction products is reduced which will give a lower result.

12. Set the spectrophotometer to 530 nm and zero against the blank.

13. Measure the blank, colour blank and sample absorbance against the laboratory demineralised water and note the readings.

14. Rinse the spectrophotometer cell with demineralised water when finished.

Calculations

a) Net absorbance (abs) = (Sample abs – Blank abs – Colour Blank abs)

b) $$\%w/w\ \text{Biuret} = \frac{F\ (mg/Abs) \times \text{Net Abs}}{wt\ (g) \qquad 1{,}000} \times \frac{1}{1{,}000} \times 100$$

$$= \frac{F \times Abs}{wt \times 10}$$

$$\text{Casale biuret }\%w/w = \frac{F\ (mg/Abs) \times Abs}{Wt\ (g)} \times \frac{1}{1{,}000} \times \frac{100}{1} \times \frac{1{,}000\ (ml)}{100\ (ml)}$$

$$= \frac{F \times Abs}{wt}$$

Where:

Wt = sample weight in grams

Abs = absorbance reading at 530 nm

F = calibration factor for spectrophotometer

Quality Control and Calibration

For calibration purposes:

1. Use Biuret calibration standard (S33) to calibrate the spectrophotometer.

2. Because the biuret standard is not necessarily 100% pure we need to correct the concentration based on the certified purity of the standard powder.

   $$\text{mg/ml biuret} = \frac{\text{wt of std (g)}}{\text{vol (ml)}} \times \frac{\%\ \text{purity}}{100} \times \frac{1{,}000}{1}$$

   $$= \frac{\text{wt of std (g)} \times \%\ \text{purity} \times 10}{\text{vol (ml)}}$$

3. Into 100 ml volumetric flasks pipette the following aliquots of standard solution (S33):

   Volume of standard (ml) 0, 10, 20, 40, 50

4. Calculate the mg of biuret in each flask by using

   mg biuret = mg / ml std x volume (ml)

5. Proceed from step 9 of the Procedure.

6. Plot a graph of mg biuret versus absorbance.

7. F (mg / ml) = slope of the above graph.

Quality control:

1. Analyse a quality control standard with each batch of samples.
2. Pipette 40 ml of QC Biuret Standard (Q2) into a 100 ml volumetric flask.
3. Proceed from step 9 of the Procedure.
4. mg biuret = F(mg/Abs) x Abs
5. Ensure result is within standard range. If not, repeat.
6. Record standard value.

Future Work

References

The following references were used in the development of this method:

1. Ballance Agri-Nutrients Ltd, Kapuni Test Method for Biuret Determination (Kapuni Ref UR-3).
2. Toyo Engineering Corporation Manual March 2010: Method Urea – 420, modified June 2000 (I257).
3. Japanese Industrial Standard JIS-K1485.

2.14 Controlled Release Nitrogen Fertilisers

The New Zealand fertiliser industry agreed test method for the determination of the ‘slow release nitrogen’ level in Coated Fertiliser Products is the European Union Method EN 13266:2001.

Note that ‘Coated Fertiliser Products’ are fertilisers where the granules are coated with sulphur or polymeric material.

References
The following material may be useful when considering this method:

1. The International Fertilizer Industry Association (IFA) publication ‘Review of Analytical Methods for Slow- and Controlled-Release Fertilizers’ (November 2014).

2.15 Determination of Common Cations in Fertilisers (Ca, Mg, K, Na and Cu, Fe, Mn, Zn, Co, Mo, Se supplementation)

Scope and Application

This method is suitable for the determination of calcium, magnesium, potassium and sodium in fertilisers and for fertilisers that have been supplemented with the following elements: copper, iron, manganese, zinc, cobalt, molybdenum, selenium and total extractable sulphur.

Summary of Method

Fertiliser samples are digested and the extract is run through an AA or ICP as defined in the relevant method.

Health and Safety Considerations

Key Points/Critical Steps

Interferences

Procedures

Inorganic materials

For inorganic materials, the New Zealand fertiliser industry agreed test method for the determination of calcium, magnesium, potassium and sodium in fertilisers and for fertilisers that have been supplemented with the following elements: copper, iron, manganese, zinc, cobalt, molybdenum, selenium and total extractable sulphur is the standard phosphate digest (HCl/HNO_3 digest) followed by ICP using the latest version of the relevant APHA method.

Organic materials

In the presence of organic matter, the New Zealand fertiliser industry agreed test method for the determination of calcium, magnesium, potassium and sodium in fertilisers and for fertilisers that have been supplemented with the following elements: copper, iron, manganese, zinc, cobalt, molybdenum, selenium and total extractable sulphur is the latest version of EPA Method 3051 microwave digest followed by measurement using the latest version of the relevant APHA method.

Calculations/Uncertainty of Measurement

Quality Control

Future Work

References

2.16 Determination of Granule Strength in Fertilisers

Scope and Application

Crushing strength is one of the parameters used to evaluate the physical quality of granular fertilisers. This is important in the evaluation of the stability and subsequent spreading characteristics of fertilisers. It is determined by applying a load to fertiliser granules.

Summary of Method

Selected fertiliser granules are crushed to estimate their crushing resistance.

Health and Safety Considerations

Key Points/Critical Steps

Interferences

Reagents and Equipment

1. 4.0 mm and 3.35 mm B.S. sieves together with a receiver pan and lid.

2. Automated digital force gauge instrument.

Standards

Procedures

1. Fertiliser granules are taken from a sample and run through a sieve series arranged as: Pan, 3.35 mm and 4.0 mm sieves. Collect the fraction retained on the 3.35 mm sieve.

2. Select the thirty spherical (or as nearly spherical as practicable) sample granules to be tested from those that are retained on the sieve. The particle size and shape of the granules are critical to obtaining a meaningful result. No agglomerates or chips or granules caught in the 3.35 mm sieve should be tested.

3. The crushing strength of the thirty representative granules is then individually determined. The surface in contact with the granules must be smooth. The granule strength is noted as the point where the granule first fractures or collapses.

4. Average the thirty results and record the average and the standard deviation.

Calculations/Uncertainty of Measurement

The granule strength of the sample is the mean result of the thirty tests carried out. The units are kilograms force (kg.f).

The standard deviation of the thirty tests gives an indication of the variation in Granule Strength within the sample.

Quality Control

Future Work

References

2.17 Determination of Abrasion Resistance (‘Granule Degradation’) in Fertilisers

Scope and Application

The ability of fertiliser to resist abrasion (or ‘granule degradation’ or attrition) is one of the parameters used to evaluate the physical quality of granulated fertilisers. Along with crushing strength, abrasion resistance is important in the evaluation of the stability and subsequent spreading characteristics of fertilisers.

Summary of Method

A representative sample of fertiliser is placed in a tumbler with steel balls and the effect on the sample is determined.

Health and Safety Considerations

Key Points/Critical Steps

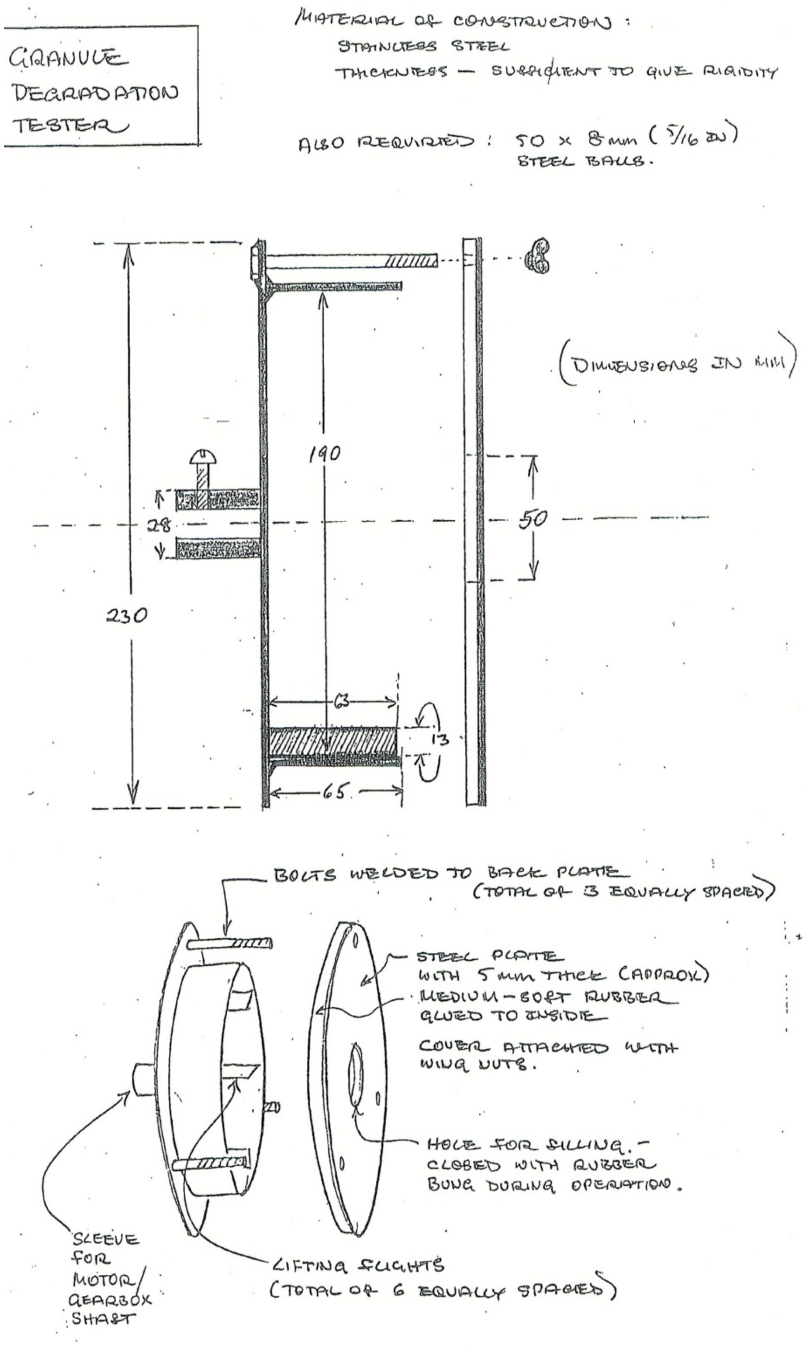
Check the number and total weight of the balls in the tumbler from time-to-time and ensure that they are clean and in good condition (not pitted).

Interferences

Reagents and Equipment

1. Suitable BS sieves together with a receiver pan and lid.

2. Incitec Abrasion Resistance Tester



Rotary drum used in abrasion resistance test

Standards

Procedures

1. Riffle the fertiliser sample down to approximately 1 kg. Sieve this sub-sample gently between a 5.6 mm and 1.4 mm sieve. Retain the portion on the 1.4 mm sieve. Riffle this portion down to give two samples for analysis:
    - one sample weighing between 60 and 70 grams.
    - one sample weighing between 80 and 90 grams.

2. Sieve gently by hand these two samples again to remove any product under 1.4 mm. Weigh the two samples accurately and record the weight.

3. Tumble each sample for 5.0 minutes at 30 rpm in the Granule Degradation Ball Mill containing 50 x 8mm diameter stainless steel balls weighing 100 ± 5 grams in total.

4. After 5 minutes brush the contents of the mill into a 5.6 mm sieve (to remove the ball bearings and unwanted sample) and 1.0 mm sieve with a pan. Brush gently to ensure all dust is included. Weigh the product retained on the 1.0 mm sieve and calculate the attrition percentage.

5. Calculate the Abrasion Resistance value as below.

Calculations/Uncertainty of Measurement
To calculate the Abrasion Resistance (AR) result, express the amount of product passing through the 1.0 mm sieve as a percentage of the original weight.

Plot the percentage Granule Degradation for the two samples on linear graph paper. Interpolate for GD at the 75 gram sample weight.

Quality Control
From time-to-time the stainless steel balls need to be inspected and checked as they wear away. Balls should be cleaned sufficiently to avoid fertiliser build-up.

Future Work

References

1. Abrasion Resistance of Fertilisers – Rotary Drum Method. Incitec Method QLM-07-341.

2.18 Wet Sieve Method for Lime and Fertilisers

Scope and Application

This method is used to determine the particle size distribution of fine, water-insoluble materials e.g. elemental sulphur.

Summary of Method

A representative sample of fertiliser or lime is placed in a stack of sieves and washed through. The collected material is then dried, and the appropriate calculations are performed.

Health and Safety Considerations

Key Points/Critical Steps

Prior to commencing the test, inspect the individual sieves to ensure that the mesh is clean and not corroded or otherwise damaged.

Interferences

Reagents and Equipment

1. Suitable BS sieves together with a receiver pan and lid. The usual sieve sizes are: 2.00 mm, 1.00 mm, 500 µ, 250 µ and 150 µ.

2. Balance with a sensitivity of 0.01 g.

3. Brush with soft bristles.

Standards

Procedures

1. Weigh the sieves and stack in order of progressing aperture with the finest sieve at the bottom.

2. Place the nest of sieves into a sink.

3. Weigh accurately 20 to 50 g of sample and transfer to the top sieve. The sample size required will depend on the proportion of fine fraction in the sample.

4. Thoroughly wet the sample with a solution of 1:5 diluted detergent.

5. Sample preparation for pastilles. Riffle to 200 g, add sufficient 1:5 detergent solution to cover, leave overnight.

6. Carefully wash the material through the sieves using a hose attached to tap water. Make sure the water is draining out the bottom.

7. Each successive sieve is removed and sprayed with ethanol.

8. Carefully place the sieves in an oven at 70° C for one hour or until constant weight is achieved. Reweigh each sieve containing dried retained material.

9. The retained sample weight on each sieve is worked out by difference between the gross dried weight and the initial sieve weight.
10. Retained weights are on a dried basis; therefore the initial sample weight used needs to be converted to a dry basis.

Calculations/Uncertainty of Measurement

% retained on sieve = ((weight of sieve + sample) – weight of sieve) ÷ initial weight x 100

% retained on pan = (initial wgt – sum of all sieve wgt fractions) x 100

Relative error = ± 2%

Quality Control

Future Work

References

The following references were used in the development of this method:

1. NZ Laboratory Services Ltd Fertiliser Chemistry Methods Manual Sieve Analysis Test Method, version 2 (23 January 2008).

2.19 Dry Sieve Method for Lime and Fertilisers

Scope and Application
Mechanically agitating a sample containing various particle sizes through a range of sieve openings will separate the sample into fractions that will allow an assessment of the particle size distribution. This is important in the evaluation of the stability and spreading characteristics of prilled fertilisers, and for the determination of the dissolution rate of agricultural lime.

Summary of Method
A representative sample of fertiliser or lime is placed in a stack of sieves and then placed on an appropriate mechanical vibrator. The collected material is then weighed and the appropriate calculations made.

Health and Safety Considerations
Avoid breathing dust when emptying and cleaning sieves. Use a dust mask if necessary. Wear hearing protection if continuously exposed to shaker noise.

Key Points/Critical Steps
Prior to commencing the test, inspect the individual sieves to ensure that the mesh is clean and not corroded or otherwise damaged.

Interferences

Regents and Equipment

1. Riffle for reducing the original sample.

2. Series of sieves together with a receiver pan and lid.

3. Mechanical sieve shaker.

4. 25 mm soft flat brush for cleaning finer mesh sieves.

5. A stiffer brush for cleaning the coarser mesh sieves.

6. Sensitive balance.

Standards

Procedures

1. For phosphate rock and lime, if required, air dry the sample at between 35°C and 40°C for at least two hours.

2. Riffle the sample of fertiliser or lime to give an appropriate sample size for sieving. The sample size should be small enough to avoid blinding of the sieves and large enough to collect a significant sample in each relevant sieve. Generally, about 100 to 200 grams is appropriate for finely ground fertilisers and lime and between 200 grams and 500 grams for prilled or coarse fertiliser. The objective is to obtain a representative sample but not so much as to 'blind' the screen. Riffling is usually only required for blended products.

3. Weigh the sample to be sieved and record to two decimal places.

4. Take the tare weight of each sieve and the pan and record the weights to two decimal places.

5. Connect up the range of sieves (biggest at the top, finest at the bottom) required for the analysis:

Sample Type	**Sieve series, from bottom to top**
Comprehensive lime analysis	Pan, 125µm, 250 µm, 500 µm, 1.0 mm, 2.0 mm
General particle size distribution	Pan, 250 µm, 500 µm, 1.0 mm, 1.4 mm, 2.0 mm, 2.8 mm, 3.35 mm, 4.0 mm, 5.6 mm, 6.7 mm
Direct Application Phosphate Rock (DAPR)	Pan, 75 µm, 150 µm, 250 µm, 500 µm, 1.0 mm, 1.4 mm
Rock shipments	Pan, 75 µm, 150 µm, 250 µm, 500 µm, 1.0 mm, 1.4 mm, 2.0 mm, 2.8 mm, 3.35 mm, 4.0 mm, 5.6 mm, 6.7 mm, 12.5 mm, 19.0 mm
Sulphur shipments	Pan, 150 µm, 250 µm, 500 µm, 1.0 mm, 1.4 mm, 2.0 mm, 2.8 mm, 3.35 mm, 4.0 mm, 5.6 mm, 6.7 mm

6. Add an appropriate amount of fertiliser or lime to the top sieve and put the lid on.

7. Place the sieves onto a sieve shaker and clamp the top plate to the top sieve.

8. Set the sieve shaker to shake for a pre-determined period. The appropriate shaking period should be determined for each material and for each setting to quantify the sieving end period. This occurs when the amount of material passing every sieve in a two-minute period is less than 0.2% by weight of the sample. It has been generally found that the following shaking times are appropriate:

- Lime	5 minutes
- Superphosphate	12 minutes
- Urea	1 minute

9. Set the shaker going. Best practice is reported to be to intermittently pulse the shaking programme (where this feature is available). After the shaking ~~period~~ is complete, record the weight of each sieve and the pan to two decimal places.

10. Empty each sieve and brush out to remove any residue.

11. Record the test results. The total of the weights collected shall be added together and subtracted from the weight of the sample taken. This gives the loss during sieving which shall be less than 0.5% of the original sample weight. If the loss is greater the test shall be discarded.

12. Calculate the percentage of the total sample weight retained and passed by each sieve. Report each faction percentage to one decimal place.

Calculations/Uncertainty of Measurement

Quality Control

Future Work

References

The following references were used in the development of this method:

1. B.S. 410-2:2000 “Methods for the Use of B.S. Fine-Mesh Test Sieves”.
2. Cunliffe, P. (1999), ed. ‘Official Methods of Analysis of AOAC International”, 18th edition, chapter 27.

2.20 Total Fluoride in Fertilisers

Scope and Application

This method can be used to determine the total amount of fluoride in phosphate rock and superphosphate fertiliser with total fluoride levels of less than 5%.

Summary of Method

Health and Safety Considerations

This method uses concentrated acids so take appropriate care.

Key Points/Critical Steps

Interferences

Reagents and Equipment

1. Conical flasks (100 ml).
2. Volumetric flasks (100 ml).
3. Beakers (100 ml).
4. Watch glass.
5. Pipettes (1, 3, 5 and 10 ml).
6. Fluoride electrodes and meter.
7. pH electrode and meter.
8. Total F extraction solution (2.4 M HCl, 10% NH_4Cl). This can be prepared by dissolving 50 g of NH_4Cl in approximately 250 ml of distilled water. Add 104 ml of concentrated hydrochloric acid (SG 1.18) and make up to 500 ml.
9. Total F buffer solution (sodium citrate 1.0 M, NaOH 0.5 M). This can be prepared by dissolving 74 g of tri-sodium citrate dehydrate and 5 g of NaOH in distilled water. Make up to 250 ml.
10. Standard fluoride solution (10,000 ppm).
11. Concentrated sulphuric acid.

Procedure

1. Prepare sample. Thoroughly mix sample and grind. Rock to pass 150 µm and fertiliser to pass 500 µm.
2. Weigh sample. Weigh 1.00 g sample into a 100 ml conical flask. It is usual to test in duplicate.

3. Extract samples. Add to the flask by pipette 20 ml of extraction solution. Add three drops of concentrated sulphuric acid. Cover the flask with a watch glass and heat gently until just boiling. Add 50 to 60 ml of water, taking care to wash into the flask any condensate on the watch glass.

4. Transfer sample. Cool and transfer to a 100 ml volumetric flask. Make up to the volume.

5. Dilute samples. Pipette 10 ml into another 100 ml volumetric flask. Add 10 ml of buffer. Make up to the mark. Mix.

6. Check/adjust pH. Pour about 50 ml of the buffered sample into a 100 ml beaker with a stir bar. Check that the pH is between 7.0 and 8.5. Adjust if necessary with either H_2SO_4 or NaOH.

7. Prepare Standard B. Pipette 10 ml of the 10,000 ppm F standard into a 100 ml volumetric flask. Make to the mark and mix well.

8. Prepare Working Standards. Pipette 1, 3 and 5 ml of Standard Solution B into each of three 100 ml flasks. Add 10 ml of buffer to each. Add 2 ml of extraction acid to each. Make to the mark These provide standards equivalent to 1 to 5% fluoride in a 1 g sample.

9. Transfer standards. Pour about 50 ml of each standard into separate 100 ml beakers.

10. Check/adjust pH. Check that the pH of the standards is between 5 and 9, preferably near 7. Adjust if necessary with either H_2SO_4 or NaOH.

11. Calibrate F meter. Calibrate the meter starting with Standard 3.

12. Read samples. Read each sample in turn. Between samples rinse electrodes thoroughly and blot dry. Do not wipe electrodes dry as this can impart a static charge.

13. Record data. Record data to two decimal points.

Calculations/Uncertainty of Measurement

Calculations are performed as follows:

Reading = % Total Fluoride (1 dp)

Quality Control

Future Work

References

The following references were used in the development of this method:

1. Ballance Total Fluoride Method, version 1 (8 May 2013). Based on FMRA Method 25.1 (1983).

2.21 Water Soluble Fluoride (WSF) in Fertilisers

Scope and Application

This method can be used to determine the amount of fluoride in fertilisers. It is suitable for the analysis of effluent water, stack gas, phosphate rock, DAP, MAP and stormwater where concentrations range from 0 to 30%.

Summary of Method

Health and Safety Considerations

Key Points/Critical Steps

Interferences

Reagents and Equipment

1. Metrohm 781 pH/ISE meter.

2. Fluoride electrode and reference electrode.

3. Beakers (100 ml) with magnetic stirrers.

4. Volumetric flasks and pipettes.

5. Citrate Buffer. This can be made by weighing out 147 g of tri-sodium citrate in one litre of distilled water. Once dissolved, adjust the pH of the solution to 6.5 with citric acid crystals. Add 1 ml of formaldehyde to the above solution to prevent algae growth.

6. F standard solution (10,000 ppm F). This can be made by weighing out 5.525 g of oven-dried and desiccated sodium fluoride (AR). Make up to 250 ml with distilled water in a volumetric flask. Transfer to a clean dry polythene reagent bottle.

Procedures

1. Set up meter. As soon as you know that fluoride analyses are to be run during the day, put the electrodes to soak in the 0.4 ppm F solution made without buffer. This improves the stability and speed of response of the electrode.

2. Prepare the working standards. These are:
    - 200 ppm. Pipette 10 ml of the 10,000 ppm F standard into a 500 ml volumetric flask. Make to the mark. The 200 ppm solution may be held 24 hours.
    - 20 ppm. Pipette 25 ml of the 200 ppm F standard into a 250 ml volumetric flask. Make to the mark.

- 2 ppm. Pipette 5 ml of the 200 ppm F standard into a 500 ml volumetric flask. Make to the mark.

3. Prepare calibration standards. These are:
   - Standards 100, 10 and 1. Pipette 25 ml of each standard (200, 20, 2 ppm) into a labelled100 ml beaker. To each beaker add 25 ml of Citrate Buffer.
   - Standard 0.4. Pipette 10 ml of the 2 ppm standard into a 50 ml volumetric flask. Add 25 ml of Citrate Buffer. Make to the mark with distilled water.

4. Prepare samples. Aim to have samples at around 20 ppm if practical.
   - Stormwater and dust tubes. Pipette 25 ml undiluted into 100 ml beaker. Add 25 ml Citrate Buffer. Normally <50 ppm.
   - Stack Gas. Pipette 25 ml undiluted into 100 ml beaker. Add 25 ml Citrate Buffer. Normally <50 ppm.
   - DAP and TSP. Pipette 20 ml undiluted to a 100 ml beaker. Pipette 5 ml of distilled water into the beaker. Add 25 ml Citrate Buffer. Normally 3,000 to 6,000 ppm.
   - Effluent and Salt. Pipette 5 ml sample into a 50 ml volumetric flask. Add 25 ml Citrate Buffer. Make to mark.

5. Calibrate meter. Place stir bars in the standard beakers. Run standards in the order 10, 1, 0.4, 100. Before running any samples or standards, they should be within ± 1°C of room temperature.

6. Record slope. After the calibration has run, record the slope on the graph above the meter. The optimal slope is -59.2 mV. If the slope is outside of ≤ 80% or ≥ 105% of this value:
   - Check the fluid levels in the reference electrode and change or refill as required.
   - Recalibrate.
   - Check the temperatures of the standards and solutions with regard to room temperature.
   - Remake the 0.4 standard and recalibrate.

   If the slope is still outside of the range, either electrode may be faulty. Use new electrodes and/or seek further help.

   Once an acceptable slope is established, the electrodes may be left in the 10 ppm standard.

7. Single point calibration. Just prior to reading the samples, run a single point calibration.

8. Read samples. Place stir bars in beakers. Read each sample, allowing enough time for the reading to stabilise. Between samples, rinse the electrodes well with distilled water and blot dry (do not wipe). Meters are subject to drift, so if there are more than six samples perform another single-point calibration to re-establish the 10 ppm standard.

Calculations/Uncertainty of Measurement

Calculations are performed as follows:

1. Stormwater and dust tubes: Reading x 2 = mg/l F
2. Stack gas: Reading x 2 = mg/l F
3. DAP and TSP: Reading x 250 = mg/l F
4. Effluent and salt: Reading x 10 = mg/l F

Quality Control

Future Work

References

The following references were used in the development of this method:

1. Ballance Fluoride Method, version 1 (4 December 2012). Based on APHA Method 4500-F C, 20th Edition.

2.22 Mercury in Fertilisers

Scope and Application

This method is used for the determination of the mercury content of all solid fertiliser types.

Summary of Method

The mercury contained within the fertiliser is extracted using concentrated nitric acid. The resultant acidic solution is analysed by ICP-MS.

Health and Safety Considerations

Personal protective equipment (PPE) should be worn when handling concentrated acids and mercury solutions.

Key Points/Critical Steps

Mercury is a contaminant in fertilisers, so very low levels are expected.

Interferences

There are no known ICP-MS interferences to the mercury mass 201. This should be used as the main detection mass.

Equipment

1. Closed vessel microwave digester (e.g. CEM MARS Xpress), with Teflon™ vessels
2. 50mL plastic tubes, with screw cap lids (e.g. SCP tubes)
3. 50mL glass test-tubes, with filter funnels
4. 14mL glass test-tubes
5. ICP-MS (e.g. Perkin Elmer NexION 300D)

Reagents

1. Concentrated nitric acid (minimum of 69%). Best purity possible
2. Concentrated hydrochloric acid (minimum of 34%). Best purity possible.
3. Type I water (>18 MΩ)
4. Gold Stability Solution
   Make a 5 mg/L gold solution by diluting 1 mL of 1,000 mg/L gold (Au) stock standard to 200 mL in a clean (rinsed) volumetric flask. Add 20 mL of concentrated hydrochloric acid and make to volume with Type I water. Mix well. Expiry: six months at room temperature.
5. Gold 'diluting' Solution
   Make a 200 µg/L gold solution for diluting the samples by diluting 20 mL of the Gold Stability Solution to 500 mL in Type I water. Ensure good mixing on a magnetic stirrer. Expiry: one month at room temperature.
6. ICP-MS Internal Standard Solution
   Prepare a 50 µg/L (ppb) Rhenium (Re) solution (from a stock solution) in a matrix of 2% nitric acid in Type I water. Mix well. Ensure sufficient solution for the whole run. Expiry: six months at room temperature.
7. ICP-MS Rinse Solution (to reduce sticking on instrument glassware)
   Prepare the following solution up to one litre of Type I water.
   Acids: 4% (40 ml/L) nitric acid, 1% (10 ml/L) hydrochloric acid.
   Solids: 0.1% mannitol (1g), 0.15% EDTA (pure, 1.5g).
   Mix well. Ensure sufficient solution for the whole run.
8. ICP-MS Carrier solution

2% nitric acid in Type I water.

Standards

Intermediate Standard

Make a 1 mg/L standard by diluting 0.5 mL of 1,000 mg/L mercury (Hg), to 500 mL, with 10% nitric acid in Type I water, using a glass volumetric flask. Mix well. Expiry: one month at room temperature.

Working Standards (prepare fresh working standards for every run)

Ref.	Vol of Intermediate standard (mL)	Vol of Nitric Acid (mL)	Vol of 5 mg/L Au solution (mL)	Final Volume (mL) (SCP tubes)	Final Standard Concentration (µg/L)
0	0.00	2.5	2	50	0
1	0.25	2.5	2	50	5
2	0.50	2.5	2	50	10
3	1.00	2.4	2	50	20

Procedures

1. Finely grind the sample and pass through a 500 micron (mesh #35) sieve, OR, if the sample is large, grind and sieve a representative subsample.
   NB: ENTIRE sample or subsample must pass through the sieve.

2. Weigh accurately approx. 0.500g of sample into a Teflon microwave tube.

3. Weigh a duplicate sample every 5th sample, for standard addition / recovery determination.

4. To ensure the sample is not on the upper sides of the tube, add 4mL of Type I water, via auto-pipette, to rinse sides of tube. Add 1mL of the Gold Stability solution and add 5mL of concentrated nitric acid, HNO_3. Mix well on a vortex mixer. Screw the lid to hand tightness, and place into microwave carousel.

5. Place the carousel into the microwave and the run method as stated below:-
   Instrument settings:-
   100% Medium power (approximately 600 Watts)
   Ramp to temperature
   - Ramp to 100°C, over 20 minutes
   - Hold at 100°C for 30 minutes
   - Allow to cool until below 50°C

6. Once the digestion is complete, allow the vessels to cool to below 50°C within the microwave cavity, then remove and cool further in a cold water bath.

7. Remove lid and add 15 mL of Type I water to achieve a total volume of 25mL. Mix very well.

8. Filter into a 50ml glass tube, rinsing the filter paper and tube with the initial 4-5ml of filtrate, which is discarded, before filtering the remaining solution.

9. Dilute the sample 2.5mL (using auto-pipette) to 10mL with the ‘Gold Diluting’ solution, and shake well (vortex).
10. Analyse on the ICP-MS (using the instrument specific conditions to achieve the best possible detection limits)

Quality Control

Recovery addition

To a sample replicate, add 0.5mL of the 1 mg/L intermediate standard before digestion. This is a standard addition of 5µg/L mercury in the final analysis solution (after dilution).

Also analyse blank samples, and check samples.

As mercury is a ‘sticky’ element in ICP-MS, analyse the samples and duplicates before analysing the recovery solutions. Always analyse a blank sample last to determine the level of any potential carryover.

NB: For any ‘unknown’ sample type, a rinse should be placed after the sample to minimise any carryover effects on the next sample.

Quality control sample results should be compared to the established acceptable range.

Calculations/Uncertainty of Measurement

Recovery of mercury from sample duplicate % =

$$\%\text{ recovery} = \frac{(\mu g/L\text{ sample + recovery}) - (\mu g/L\text{ sample}) \times 100}{\text{Amount of spike } \mu g/L\text{ (nominal: } 5\mu g/L)}$$

Acceptable recovery is between 85 to 105%

No adjustment for the recovery value should be made to the results.

$$\text{final mg/kg} = \frac{(\mu g/L\text{ in solution} - \mu g/L\text{ in blank}) \times (25 \times 4)}{\text{weight of sample g} \times 1000}$$

The (25 x 4) accounts for the final digest volume and the dilution of 2.5mL to 10mL to reduce the acid concentration in the sample solutions.

The results should be expressed to the nearest 0.01 mg/kg

Lower Reporting Limit (RL)

Nominally, with a recovery of 85% or more, the Reporting Limit would be expressed as less than (<) 0.05 mg/kg.

ICP-MS Mass parameters

Elemental Parameters	**Mass(amu)**

Mercury 1	200.97
Mercury 2	201.97
Internal Standard (Rhenium)	184.95

Always follow the manufacturer's recommendations for ICP-MS tuning.

Future Work

References

The following references were used in the development of this method:

1. ARL Test Methods and Procedures Manual. Determination of Mercury by Microwave Digestion and ICP-MS Detection. (m-C108; Issue 4)

2.23 Determination of Boron in Fertilisers

The New Zealand fertiliser industry agreed test method for the determination of boron in fertilisers is AOAC Method 982.01 'Boron, Acid and Water Soluble in Fertilisers Spectrophotometric Method'.

2.24 Determination of N-(n-butyl) thiophosphoric acid triamide (NBPT) in Fertilisers

The New Zealand fertiliser industry agreed test method for the determination of N-(n-butyl) thiophosphoric acid triamide (NBPT) in fertilisers is the German standard DIN 15688.

It should be noted that the dissolution process must be flexible enough to cope with variable sample types (e.g. if part of the sample won't dissolve in water).

SECTION THREE: LIME

3.1 Determination of Liming Equivalent (expressed as % w/w CaCO3)

Principle

This titrimetric method determines the amount of calcium carbonate (CaCO3) in lime. The lime sample is fully leached with an excess of standardised hydrochloric acid. The amount of hydrochloric acid consumed is determined by back-titration with standardised sodium hydroxide, and calculated as calcium carbonate % w/w.

Scope

Applicable to all lime samples or samples containing lime. Note that this method is also known as the ‘Neutralising Value’ method.

Reagents

1N HCl and 0.5N NaOH

Sample Preparation

Grind lime sample sufficiently to pass a 250 micron sieve opening. Coarser and drier samples will need longer grinding times than finer and damper samples.

Procedure

1. Accurately weigh 5 g ± 0.002 g of ground lime sample into a 250 mL conical flask.

2. Add 100 mL of 1N HCl using a bulb pipette.

3. Gently boil on a hot plate until the reaction is complete (generally around 5 minutes).

4. Remove the sample from the hot plate and allow to cool.

5. Using a pH meter and combination electrode, titrate the excess HCl with 0.5N NaOH with vigorous stirring (use a magnetic follower), to pH 4.5.

Calculation

Neutralising value (as $CaCO_3$) % w/w =

$$\frac{((100 \times \text{HCl normality}) - (\text{NaOH stop titre} - \text{start titre (mL)} \times \text{NaOH normality}) \times 5}{\text{sample weight (g)}}$$

Report results to one decimal place.

Quality Control

Analyse 5.000 g of AR grade $CaCO_3$. Acceptable range, 98.5-101.5 % recovery.

Analyse a blank with every batch by taking 25 mL of the 1 N HCl and following the procedure.

All samples are analysed in duplicate.

Agreement between duplicates should be within 0.6% (w/w) (Based upon validation data).

Reference

Cunniff, P (1999), ed: ‘Official Methods of Analysis of AOAC International’, 16th edition, 5th revision, chapter one, page 1 (955.01)

3.2 Wet and Dry Sieving Methods for Lime

For wet and dry sieving methods for lime, see the corresponding test methods in Section 2 of this Appendix.

3.3 Determination of Lime Reactivity

The New Zealand fertiliser industry agreed test method for the determination of Lime Reactivity is the latest version of European Standard Method EN 16357, ‘Determination of Reactivity of Carbonate Liming Materials.

This European Standard specifies a method for the determination of the reactivity of calcium carbonate and calcium magnesium carbonate liming materials. It assesses the speed and effectiveness of their neutralising potential by automatic titration with citric acid. The method is applicable only to liming materials with a maximum particle size of 6.3mm.

For liming materials coarser than 1mm or presenting a presumable heterogeneousness, determine the size distribution by dry sieving (sieve sizes 2mm, 1mm and 0.5mm) and store the different fractions. Select a maximum of three test sieves with different principal sizes to cover the range of the particle size expected. Compose the test portion of 5.00 g from the individual sieving fractions according to their proportion of the total test sample.

The titration is performed under stable pH conditions (pH 4.5) with an automatic titration apparatus. The citric acid consumption during a given time (15 min) is a direct measure for the reaction of the liming materials being tested.

Note that the final calculation requires the following tests:

- pH to check there are no hydroxides (pH should be <10).
- Lime Equivalence.
- Mg (to estimate any dolomite in sample as $MgCO_3$ reacts more slowly).
- Lime Reactivity Titration.

The final result produces an index for ranking different limes into slow, medium and fast reacting.

SECTION FOUR: SOILS

4.1 Determination of Cadmium in Soils

The New Zealand fertiliser industry agreed test method for the determination of the cadmium content of soils is the latest version of the US Environmental Protection Agency Method #200.2 ‘Sample Preparation Procedure for the Spectrochemical Determination of Total Recoverable Elements’, EPA Method #200.7 ‘Determination of Metals and Trace Elements by Inductively Coupled Plasma Mass Spectrometry’ and EPA Method #200.9 ‘Determination of Trace Elements by Stabilised Temperature Graphite Furnace Atomic Absorption Spectrometry’.

4.2 Determination of Fluoride in Soils

The New Zealand fertiliser industry agreed test method for the determination of fluoride in soils is the method published in ‘*Methods of Soil Analysis: Part 3. Chemical Methods’*, page 846. Published by Soil Science Society of America, Inc. (1996).

FERTMARK CODE OF PRACTICE

APPENDIX TWO

INDUSTRY AGREED AGRONOMIC TRIAL PROTOCOL_

The material recorded here is the Fertiliser Industry Agreed Agronomic Trial Protocol that has been recognised by the Fertiliser Quality Council as being appropriate for the conduct of trials to assess the agronomic performance of fertilisers.

The purpose of this protocol is to provide reliable methods for the conduct of trials.

Suggestions for improvements to the protocol are welcomed.

Parties with an interest in this matter are invited to contact:

The Executive Director
Fertiliser Quality Council
c/- Federated Farmers Inc
PO Box 715
WELLINGTON

Date	Version	Description of Revision
November 2012	1	First edition.

Fertmark Agronomic Trial Protocol

1.1 The Fertiliser Quality Council has determined that it should not establish specific experimental trial design beyond referring to “Conduct of Field Experiments” PB Lynch, Bulletin 399, New Zealand Department of Agriculture and ‘Planning and “Managing Agricultural and Ecological Experiments”, Peter B Johnstone, Stanley Thornes (Publishers) Ltd, Ellenborough House, Wellington Street, Cheltenham, GL501YW, United Kingdom. 1998. Refer to Rule 7.4 of the Fertmark Operational Rules.

1.2 When the Fertiliser Quality Council Expert Group is called on to advise the Executive Committee about a matter concerning the integrity of a fertiliser product or the methodology of the field trials for the fertiliser product, the Expert Group should be bound by the principles laid down in both the publications referred to in section 1.1 of this protocol.

FERTMARK CODE OF PRACTICE

APPENDIX THREE

CADMIUM EXCEEDANCE PROTOCOL

Reporting Protocol for Exceedance of Voluntary Limit for Fertiliser Cadmium Levels

Introduction:

In order to never exceed the voluntary limit of 280mg Cd/kg P the fertiliser companies source the rock phosphate from different locations and blend to ensure the resultant superphosphate remains within the required limit.

Monitoring is undertaken during the manufacture of superphosphate to ensure the voluntary limit is met. For imported manufactured products purchase criteria and monitoring provide for compliance.

As part of Fertmark fertiliser quality assurance an independent audit of fertiliser cadmium levels is conducted.

The audit process involves review of information supplied by the fertiliser companies themselves, plus independent sampling and testing of phosphate fertiliser products.

In the event that a sample audit result exceeds the voluntary limit, an agreed protocol for reporting is required. It is also likely that an exceedance will be discovered first by internal company systems. In the event that exceedance is anticipated by the company, a self-declaration process ensues.

Definitions:

Short-term: means the time frames for use and application of a phosphate fertiliser product pertains to just one shipment, or part of one shipment.

Long-term: means the time frames for use and application of a phosphate fertiliser product pertains to more than one shipment.

Exceedance: means a cadmium value in excess of 280 mg Cd/kg P when following the protocols and procedures for the ‘Determination of Cadmium in Fertilisers’ as described in the Fertmark Code of Practice.

Proposed response process

A) Fertmark audit discovery of exceedance

i. Exceedance observed in product available for dispatch for sale and use

ii. Contact laboratory and discuss result. Discuss pilot results and Inter-laboratory Comparison Programme (ILCP) performance. In addition, quality control limits, independent checks using an independent accredited laboratory and where relevant method detection limits should also be investigated at this time, in order to try to establish validity of result. Ensure that the remaining sample is retained.

iii. Verify lab sample against lab or sampling error
  - Retest sample within the same laboratory
  - Verify with inter-lab sample

Where it is confirmed through retesting that the product exceeds the voluntary limit:

iv. Verify the sample is representative of product held by the fertiliser company and, if the exceedance is detected by external audit, notify company concerned.

v. Determine the context/circumstances giving rise to exceedance
  - Sample other stock piles of the same manufacture batch but at different distribution depots.

vi. Establish the management protocols to prevent/limit distribution as required.

vii. Auditor notifies Fertiliser Quality Council, including details of extent of exceedance and steps being taken.

viii. Fertiliser Quality Council notifies MPI/Chair of the Cadmium Management Group, including details of extent of exceedance and steps being taken.

ix. Remedy the fertiliser batch through further blending with low cadmium product if possible.

x. Retest to verify compliance.

xi. If blending cannot resolve the exceedance and exceedance is a **short-term** issue;
  - manage distribution of high cadmium product to low intensity or low risk land use with formal withdrawal of Fertmark certification for this product (including label descriptors) and declaration and explanation to the end user (e.g. hill country pasture with low P requirements, or orchard/forestry application).
  - combination of blending and selective distribution strategies.
  - retain detailed records of management and use of the product.

xii. If the exceedance is a **long-term** issue;
  - In addition to xi above, convene the Cadmium Management Group to establish agreed management options.

(Management options may include revision of the maximum limit and/or adherence to agreed maximum soil loading rate, regardless of the Cd concentration per kg P. Managing to an agreed soil loading rate, based on current soil cadmium levels would be consistent with the Tiered Fertiliser Management System. The Cadmium Management Group may suggest alternative or additional management options).

B) Self-declaration of exceedance

The process is initiated with discovery in a product for dispatch by the fertiliser company, and declaration to Fertmark Auditor, detailing the nature and circumstances of exceedance, then protocols proceed from step vi as above.

vi. Determine the context/circumstances giving rise to exceedance.
  - quality control limits, independent checks using an independent accredited laboratory and where relevant method detection limits should also be investigated at this time.
  - Sample other stock piles of the same manufacture batch but at different distribution depot.

vii. Establish the management protocols to prevent/limit distribution as required.

xiii. Company notifies the Fertmark Auditor, including details of extent of exceedance and steps being taken. Fertmark Auditor informs the Fertiliser Quality Council.

xiv. Company notifies MPI/Chair of the Cadmium Management Group, including details of extent of exceedance and steps being taken.

xv. Remedy the fertiliser batch through further blending with low cadmium product if possible.

xvi. Retest to verify compliance.

xvii. Fertmark Auditor undertakes standard but targeted audits.

xviii. If blending cannot resolve the exceedance and exceedance is a short-term issue;
  − manage distribution of high cadmium product to low intensity or low risk land use with, formal withdrawal of Fertmark certification for this product (including label descriptors) and declaration and explanation to the end user (e.g. hill country pasture with low P requirements, or orchard/forestry application).
  − combination of blending and selective distribution strategies.
  − retain detailed records of management and use of the product.

xix. If the exceedance is a long-term issue;
  - In addition to xi above, convene the Cadmium Management Group to establish agreed management options.

(Management options may include revision of the maximum limit and/or adherence to agreed maximum soil loading rate, regardless of the Cd concentration per kg P. Managing to an agreed soil loading rate, based on current soil cadmium levels would be consistent with the Tiered Fertiliser Management System. The Cadmium Management Group may suggest alternative or additional management options).

FERTMARK CODE OF PRACTICE

APPENDIX FOUR

RESIDUE RISK MANAGEMENT PLAN GUIDE

RESIDUE RISK MANAGEMENT PLAN GUIDE

Background

The methodology outlined in this appendix is not meant to be prescriptive but it follows best practice. Other methodologies may be used.

Risk Assessment Methodology

The methodology to be used follows the generally accepted practice of:

- Defining the required outcomes for the product.
- Describing the manufacturing process in a step-by-step manner (to the extent possible), including identifying each component used.
- Identification of hazards and their causes at each step in the manufacturing process.
- Assessing the likelihood and severity (risk rating) for each hazard.
- Defining control and monitoring measures to reduce the identified hazards to acceptable levels.
- A Summary Table.

Updating the Risk Management Plan

The Risk Analysis and Mitigation Plan will be reviewed and, if appropriate revised, in response to new and changing circumstance and triggers. The reviews will be conducted at least annually or when there is a change in composition, source of components, or process. All reviews will be documented. The review frequency will be included in the Plan.

In addition, changing circumstances and triggers that may cause a review to be initiated include complaints, feedback from users or industry groups, Fertmark scheme changes, and regulatory changes.

When changes to the manufacturing process or formulation occur that may cause new residue risks to arise, the Risk Management Programme will be revised and the auditor will be advised.

Required Outcomes:

The required outcomes of the Risk Analysis and Mitigation Plan must include a provision to the effect that:

> When the product is used as recommended, there will be no accumulation of residues in primary produce that exceeds the limits prescribed in applicable food residue standards or that there is toxicity to animals exposed to the compound to an extent that causes unnecessary or unreasonable pain or distress.

In addition, the expected limits to specific residues or contaminants identified in the product Risk Assessment shall be defined.

Identification of Hazards:

For each step in the manufacturing process (as far as it is known), and for each component used in the manufacture of the product, there shall be a determination of the risks that could impact on the Required Outcomes (above).

Analysis of Risks:

Each of the risks identified will be analysed to determine their potential severity and the likelihood of their arising (from which their overall risk will be determined). In addition, proposed control and monitoring measures will be described.

The tables below are intended to assist with the assessment of Severity (the consequence if a particular hazard should occur and continue uncontrolled) and the Likelihood (the probability that a particular hazard will occur).

Severity		
1	Insignificant	Negligible real or perceived risk
2	Minor risk	Minimal risk
3	Moderate risk	A real or perceived risk
4	Major risk	Significant impact

Likelihood		
5	Almost certain	Common occurrence Happens quite frequently Might well be expected
4	Likely	Known to occur Quite possible
3	Possible	I’ve heard of it happening Realistically could occur Unusual but possible
2	Not likely	Not expected to occur Conceivable but unlikely to happen
1	Rare	Not known to have ever occurred Practically impossible

The hazard rating (Outcome Risk) is obtained by multiplying the probable frequency (likelihood) by the severity and comparing the numerical result with the table below:

Minor 1 to 5
Major 6 to 10
Critical 12 to 20

Major and Critical Outcome Risks will be regarded as unacceptable and additional controls will be accordingly implemented.

Likelihood	5	Common occurrence	5	10	15	20	25
	4	Known to occur	4	8	12	16	20
	3	Possible	3	6	9	12	15
	2	Unlikely	2	4	6	8	10
	1	Rare	1	2	3	4	5
			No risk	Minor	Moderate	Major	Fundamental
			1	2	3	4	5
			Severity				

The Analysis of Risks is normally recorded in a table with the following columns:

- Process step (note that each component used to manufacture the product needs to be included).
- Possible hazards introduced at each process step.
- Severity assessment
- Likelihood assessment.
- Risk rating (Severity x Likelihood).
- Control measures identified.
- Monitoring arrangements.

Summary Table:

Near the end of the Risk Analysis and Mitigation Plan, it is usual to have a Summary of Risks Table.

Validation:

Risk Analyses should be accompanied by the results of a validation trial that indicates, as far as is practicable, that the control measures are effective. The reports from these validation trials will be documented and included in the Risk Management Programme.

This, along with the Risk Analysis and Mitigation Plan constitutes what is required by the parts of the ACVM Notice that relate to fertiliser ‘Fitness for Purpose’, i.e. the ‘Requirements for Conditions of Exemption’, section 2.1 (1) requirement that: “*There must be documentary proof that the fertiliser will not cause chemical residue risks to public health or risks to trade and market access for primary produce arising from the use of the product”* and the ACVM Notice ‘Requirements for Conditions of Exemption’, section 2.1 (1), that “*There must be documentary proof that the fertiliser, when used as recommended, will not cause animal welfare risks”.*

In addition, there is an ACVM requirement (ACVM Notice ‘Requirements for Conditions of Exemption’, sections 2.1.4 (2)(a), (2)(b), (16), (23), (25), (28) and (30)), for fertiliser manufacturers to have “*a documented manufacturing system that includes:*

- *An outline of the overall manufacturing activities undertaken at the site and the sort of products that are routinely manufactured there. This should include an overall description of the equipment and premises.*
- *Manufacturing and quality process characteristics for each fertiliser – including a description of each manufacturing step (flow diagram), quality control steps throughout the manufacturing process and product release processes (that include the microbiological or chemical residue tests that are made on each batch before it is released) and how cross-contamination is managed”.*

It is not intended that Fertmark requirements will replace ACVM requirements, however by aligning the requirements, some degree of duplication is avoided for the applicant. As part of the application process, the applicant is required to declare and demonstrate that the product meets ACVM requirements.