

The Fertilizer (Control) Order, 1985

UNION OF INDIA

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Rule THE-FERTILIZER-CONTROL-ORDER-1985 of 1985

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The Fertilizer (Control) Order, 1985 Published vide Notification Gazette of India, Extraordinary, Part 2, Section 3(1), dated 25th September, 1985, (w. 53).G.S.R. 758(E), dated 25th September, 1985. - In exercise of the powers conferred by Section 3 of the Essential Commodities Act, 1955 (10 of 1955), the Central Government hereby makes the following Order, namely -

1. Short title and commencement.

(1) This Order may be called the Fertilizer (Control) Order, 1985. (2) It shall come into force on the date of its publication in the Official Gazette.

2. Definitions.

- In this Order, unless the context otherwise requires, -(a) "Act" means the Essential Commodities Act, 1955 (10 of 1955) ;(b) "Certificate of source" means a certificate given by a State Government, Commodity Board, manufacturer, pool handling agency or, as the case may be, wholesale dealer indicating therein the source from which fertilizer for purpose of sale is obtained ;(c) "Commodity Board" means the Coffee Board constituted under Section 4 of the Coffee Act, 1942 (7 of 1942), or the Rubber Board constituted under Section 4 of the Rubber Act, 1947 (24 of 1947), the Tea Board constituted under Section 4 of the Tea Act, 1953 (29 of 1953), or, as the case may be, the Cardamom Board constituted under Section 4 of the Cardamom Act, 1965 (42 of 1965) ;(d) "compound or complex fertilizer" means a fertilizer containing two or more [* * *] [The words 'essential plant' omitted by S.O. 725(E), dated 28th July, 1988 (w.e.f. 28th July, 1988).] nutrients during the production of which chemical reaction takes place ;(e) "controller" means the person appointed as Controller of Fertilizers by the Central Government and includes any other person empowered by the Central Government to exercise or perform all or of the powers, or as the case may be, functions of the Controller under this Order ;(f) "dealer" means a person carrying on the business of selling fertilizers, whether wholesale or retail, [or industrial use] [Inserted by S.O. 795(E), dated 22nd November, 1991.] and includes a manufacturer and a pool handling agency carrying on such

business and the agents of such person, manufacturer or pool handling agency ;(g)[* * *] [Clause (g), omitted by S.O. 725 (E), dated 28th July 1988, (w.e.f. 28th July, 1988).].(h)["fertiliser" means any substance used or intended to be used as a fertiliser of the soil and/or crop and specified in Part A of Schedule I and includes a mixture of fertilizer and a special mixture of fertilizers ;] [Substituted by S.O. 354(E), dated 3rd June, 1993.](i)"Form" means a form appended to this order ;(i)["industrial dealer" means a dealer who sells fertilisers for industrial purposes. [Inserted by S.O. 795(E), dated 22nd November, 1991.](ii)"industrial purpose" means the use of fertilizer of purpose other than fertilisation of soil and increasing productivity of crops;](j)["grade" means the nutrient contents in the Fertilizers, expressed in percentage;] [Substituted by S.O. 725(E) dated 28th July, 1988 (w.e.f. 28th July, 1988).](k)"granulated mixture" means a mixture of fertilizers [* * *] [Clause (nn), omitted by S.O. 725(E), dated 28th July, 1988 (w.e.f. 28th July, 1988).] made by intimately mixing two or more fertilizer with or without inert material and granulating them together, without involving any chemical reaction ;(kk)["importer" means a person who imports fertilizer in accordance with the Export and Import Policy of the Central Government, as amended from time to time;] [Inserted by S.O. 397(E), dated 18th June, 1993.](l)"inspector" means an Inspector of Fertilizers appointed under Clause 27;(m)"manufacturer" means a person who produces fertilizers or mixtures of fertilizers [* * *] [Omitted by S.O. 354(E), dated 3rd, June 1993.] and the expression 'manufacture' with its grammatical variations shall be construed accordingly ;(n)["mixture of fertilizers" means a mixture of fertilizer made by physically mixing two or more fertilizers ; with or without inert material in physical or granular form and includes a mixture of NPK fertilizers, a mixture of micronutrient fertilizers and a mixture of NPK with micronutrient fertilizers ;] [Substituted by S.O. 354(E), dated 3rd June, 1993](nn)[* * *] [Omitted by S.O. 354(E), dated 3rd June, 1993.].(o)"offer for sale" includes a reference to an intimation by a person of a proposal by him for the sale of any fertilizer, made by publication of a price list, by exposing the fertilizer for sale indicating the price, by furnishing of a quotation or otherwise howsoever ;(p)"physical mixture" means a mixture of fertilizers [made by physically, mixing two or more] [Substituted by S.O. 725(E), dated 28th July 1988 (w.e.f. 28th July, 1988).] fertilizers with or without inert material necessary to make a required grade, without involving any chemical reaction ;(q)"prescribed standard" means, -(i)in relation to fertilizer included in column I of Part A of Schedule I, the standard set out in the corresponding entry in column 2, subject to the limits of permissible variation as specified in Part B of that Schedule ; and(ii)in relation to a mixture of fertilizers, the standard set out in respect of that mixture under sub - clause (1) of Clause 13 by the Central Government, subject to the limits of permissible variation as specified in Part B of Schedule I;(iii)[] [Inserted by S.O. 725(E), dated 28th July, 1988 (w.e.f. 28th July, 1988).] in relation to a mixture [mixture of fertiliser] [Substituted by S.O. 354(E), dated 3rd, July, 1993.] the standard set out in respect of that mixture under sub - clause (2) of Clause 13 by the State Government, subject to limits of permissible variation as specified in Part B of Schedule I.(r)"pool handling agency" means an agency entrusted by the. Central Government with functions relating to handling and distribution of imported fertilizers ;(s)"registering authority" means a registering authority appointed under Clause 26 ;(t)"retail dealer" means a dealer who sells fertilizers to [to farmers or plantations for agricultural use such as for fertilisation of soil and increasing productivity of crops] [Substituted by S.O. 795(E), dated 22nd June, 1991.];(u)"Schedule" means a Schedule appended to this order ;(v)"special mixture of fertilizers" means any mixture of fertilizers prepared for experimental purposes in pursuance of a requisition made by any person (including a person engaged in the cultivation of tea, coffee or rubber) for sale to that person in such

quantity and within such period as may be specified in such requisition ; and(w)"wholesale dealer" means a dealer who sells fertilizers otherwise than in retail [for agricultural use such as for fertilisation of soil and increasing productivity of crops] [Added by S.O. 795(E), dated 22nd, June, 1991.].II. Price Control

3. Fixation of prices of fertilizers.

(1)The Central Government may, with a view to regulating equitable distribution of fertilizers and making fertilizers available at fair prices, by notification in the Official Gazette, fix the maximum prices or rates at which any fertilizer may be sold by a dealer, manufacturer or a pool handling agency.(2)The Central Government may having regard to the local conditions of any area, the period of storage of fertilizers and other relevant circumstances, fixed different prices or rates for fertilizers having different periods of storage or for different areas or for different classes of consumers.(3)No dealer, manufacturer or pool handling agency shall sell or offer for sale any fertilizers at a price exceeding the maximum price or rate fixed under this clause.

4. Display of stock position and price list of fertilizers.

- Every dealer, who makes or offers to make a retail sale of any fertilizers, shall prominently display in his place of business. -(a)the quantities of opening stock of different fertilizers held by him on each day ;Explanation. - The actual stocks at any point of time during the day may be different from that of the displayed opening stocks to the extent of sale and receipt of such fertilizers up to the time of inspection during that day.(b)a list of prices or rates of such fertilizers fixed under Clause 3 and for the time being in force.

5. Issue of cash/credit memorandum

. - Every dealer shall issue a cash or credit memorandum to a purchaser of a fertilizer in [Form M] [Substituted by S.O. 261(E), dated 16th April, 1991 (w.e.f. 16th April. 1991).].III. Control On Distribution Of Fertilizers By Manufacturer

6. Allocation of fertilizers to various States.

- The Central Government may, with a view to securing equitable distribution and availability of fertilizers to the farmers in time, by notification in the Official Gazette, direct any manufacturer to sell the fertilizers produced by him in such quantities and in such State or States and within such period as may be specified in the said notification.IV. Registration Of Dealers

7. Dealers to be registered.

- No person, including a manufacturer, a pool handling agency [a wholesale dealer, a retail dealer and industrial dealer] [Substituted by S.O. 795(E), dated 22nd November, 1991.] shall offer for sale or carry on the business of selling fertilizers at any place except under and in accordance with the

terms and conditions of a certificate of registration granted to him under Clause 9: Provided that a State Government may, by notification in the Official Gazette, exempt from, the provisions of this clause any person selling fertilizers to farmers in such areas and subject to such conditions as may be specified in that notification.

8. Application for registration.

- Every person desiring to obtain a certificate of registration under this Order for selling fertilizers, whether in wholesale or retail or both shall make an application for registration to the registering authority [or, as the case may be, for industrial use through registered post to the controller] [Inserted by S.O. 795(E), dated 22nd November, 1991.] in Form A, in duplicate, together with the fee prescribed under Clause 36 and a certificate of source [in Form O] [Added by S.O. 354(E), dated 2nd June, 1993.] : Provided that where the applicant is a State Government, a manufacturer or a pool handling agency, it is not necessary to enclose a certificate of source along with the application : Provided further that separate certificates of registration shall be obtained for selling fertilizer in wholesale and retail, [and in case of a State Government, a manufacturer and a pool handling agency, also for selling fertilizer for industrial use] [Added by S.O. 795(E), dated 22nd November, 1991]: Provided also that where fertilizers are obtained for sale from different sources, a certificate of source from each such source shall be furnished [provided also that for obtaining a certificate for registration for industrial dealer, a certificate of source obtained only from the State Government or a manufacturer or pool handling agency shall be furnished] [Added by S.O. 795(E), dated 22nd November, 1991.]: [Provided also that a wholesale dealer, excepting manufacture, a pool handling agency or a State Government, shall not issue the certificate of source to another wholesale dealer. A wholesale dealer shall issue a certificate of source only to a retail dealer] [Added by S.O. 354(E), dated 3rd June, 1993.].[* * *] [Proviso, omitted by S.O. 489(E), dated 22nd May, 1995.].

9. Grant or refusal of certificate of registration.

- The registering authority [or, as the case may be, the controller] [Inserted by S.O. 795(E), dated 22nd November, 1991.] shall grant a certificate of registration in Form B within thirty days of the receipt of application to any person who applies for it under CI 8 : Provided that no certificate of registration shall be granted to a person. -(a)if his previous certificate of registration is under suspension ; or(b)if his previous certificate of registration has been cancelled within a period of one year immediately precedent the date of application ; or(c)if he has been convicted of an offence under the Act, or any Order made thereunder within three years immediately preceding the date of making the application ; or(d)if he fails to enclose with the application a certificate of source ; or(e)if the application is incomplete in any respect ; or(f)[if he makes an application for obtaining the certificate of registration for industrial dealer and, excepting if he is a manufacturer or pool handling agency, holds a valid certificate of registration for wholesale dealer or retail dealer or both, and as the case may be, the vice versa.] [Added by S.O. 795(E), dated 22nd November, 1991.]

10. Period of validity of certificate of registration.

- Every certificate of registration granted under Clause ["as case may be renewed under Clause 11"] [Added by S.O. 795(E), dated 22nd November, 1991.] shall, unless suspended or cancelled, be valid for a maximum period of three years, from the date of issue.

11. Renewal of certificate of registration.

(1) Every holder of a certificate of registration desiring to renew the certificate of registration granted under Clause 9 shall, before the date of expiry of such certificate of registration make an application for renewal to the registering authority [or, as the case may be, the controller"] [Inserted by S.O. 795(E), dated 22nd November, 1991.] in Form C, in duplicate, together with the fees prescribed under Clause 36 for such renewal and a certificate of source as required under Clause 8.(2) On receipt of such application, together with such fee and certificate of source, the registering authority ["or, as the case may be, the controller"] [Inserted by S.O. 795(E), dated 22nd November, 1991.] may renew the certificate of registration :Provided that a certificate of registration shall not be renewed, if the holder of the certificate of registration did not sell any fertilizer during the period of one year immediately preceding the date of expiry of the period of validity of the certificate of registration sought to be renewed.(3) If any application for renewal is not made before the expiry of the period of validity of the certificate of registration but is made within one month from the date of such expiry, the certificate of registration may be renewed on payment of such additional fee as may be prescribed by the State Government, ["or, as the case may be, the controller"] [Inserted by S.O. 795(E), dated 22nd November, 1991.] in addition to the fee for renewal of the certificate of registration.(4) Where the application for renewal is made within the time specified in sub - clause (1) or sub - clause (3) the application shall be deemed to have held a valid certificate of registration, until such date as the registering authority ["or, as the case may be, the controller"] [Inserted by S.O. 795(E), dated 22nd November, 1991.] passed orders on the application for renewal.(5) If an application for renewal of a certificate of registration is not made within one month of the date of expiry of the period of validity of the certificate of registration, the certificate of registration shall be deemed to have lapsed on the date on which its validity expired and any business carried on after that date shall be deemed to have been carried on in contravention of Clause 7.V. Manufacture Of Mixtures Of Fertilizers

12. Restriction on preparation of mixtures of fertilizers.

- No person shall carry on the business of preparing any mixture of fertilizers [* * *] [Omitted by S.O. 354(E), dated 3rd June, 1993.] or special mixture of fertilizers except under and in accordance with the terms and conditions of a certificate of manufacture granted to him under Clause [15 to 16] [Substituted by S.O. 725(E), dated 28th July, 1988 (w.e.f. 28th July, 1988).].

13. [Standards of mixtures of Fertilizers] [Substituted by S.O. 725(E), dated 28th July, 1988 (w.e.f. 28th July, 1988).]. - (1) Subject to the other provisions of this order no person shall manufacture any mixture of fertilizers unless

such mixture conforms to the standards set out in the notification to the issued by the Central Government in the Official Gazette.

(2)[Subject to the other provisions of this Order, no person shall manufacture any mixture of fertilisers unless such mixture conforms to the standards set out in the notification to be issued by the State Government in the Official Gazette ;Explanation. - For the purposes of this sub - clause, mixture of fertilisers shall not include liquid fertilisers and 100% water soluble fertilisers, containing N.P.K.] [Substituted by S.O. 329(E), dated 12th May, 1999 (w.e.f. 12th May, 1999).](3)Notwithstanding anything contained in his Order, those units which are already manufacturing [mixture of fertilisers] [Substituted by S.O. 354(E), dated 3rd June, 1993.] as on the date of publication of this order, for which standards have not yet been specified under 13(2), may continue to manufacture for sale, sell, offer for sale, stock or exhibit for sale or distribute such mixtures of micro - nutrient fertilizers for a period of [upto 27th July, 1992] [Substituted by S.O. 530 (E), dated 16th August, 1991 (w.e.f. 16th August, 1991).] from such date or till the standards are prescribed by the State Governments whichever is earlier subject to the condition that before the expiry of said period, they will follow the standards prescribed by State Government and obtain Certificate of Manufacture under Clause 15.(4)No Certificate of Manufacture shall be granted in respect of any mixture of fertilizers or [* * *] [Omitted by S.O. 354(E), dated 3rd June, 1993.] which does not conform to the standards set out in the notification referred in sub - clause (1) or (2) ;(5)Nothing in this clause shall apply to special mixtures of fertilizers.

14. Application for certificate of manufacture of mixtures of fertilizers.

(1)Every person desiring to obtain a certificate of manufacture for preparation of any mixture of fertilizers or special mixture of fertilizers [* * *] [Omitted by S.O. 354(E), dated 3rd June, 1993.] shall possess such qualification as may be prescribed for this purpose by the State Government or shall employ a person possessing such qualification for the preparation of such mixture [and possess the minimum laboratory facility as specified, in Clause 21 - A of this order.] [Inserted by S.O. 261(E), dated 16th April, 1991 (w.e.f. 16th April, 1991).](2)An applicant for a certificate of manufacture for preparation of mixture of fertilizers or special mixture of fertilizers [* * *] [Omitted by S.O. 354(E), dated 3rd June, 1993.] shall make an application to the registering authority -(a)if he is an applicant for a certificate of manufacture for any mixture of fertilizers, [* * *] [Omitted by S.O. 354(E), dated 3rd June, 1993.] in Form D, in duplicate, together with the fee prescribed therefor under Clause 36, or,(b)if he is an applicant for a certificate of manufacture for any special mixture, in Form E, in duplicate, together with the fee prescribed therefor under the said Clause 36 and an attested copy of the requisition of the purchaser.

15. Grant or refusal of certificate of manufacture for preparation of mixtures of fertilizers [* **] [Omitted by S.O. 354(E), dated 3rd June, 1993.]

. - (1) The registering authority may, for reasons to be recorded in writing, refuse to grant a certificate of manufacture in respect of any mixture of fertilizers, or special mixture of fertilizers [* * *] [Omitted by S.O. 354(E), dated 3rd June, 1993.] and shall furnish to the applicant a copy of the

order so passed.(2)Where an application for a certificate of manufacture for mixture of fertilizers [* * *] [Omitted by S.O. 354(E), dated 3rd June, 1993.] is not refused under sub - clause (1), the registering authority shall grant a certificate of manufacture in Form F and where an application for a certificate of manufacture for a special mixture is not refused under that sub - clause, such authority shall grant a certificate of manufacture to the applicant in Form "G".

16. Conditions for grant of certificate of manufacture in respect of special mixture of fertilizers and period of validity of such certificate.

(1)No certificate of manufacture in respect of any special mixture of fertilizers shall be granted to an applicant unless he holds a valid certificate of manufacture under this Order for any mixture of fertilizers.(2)Every certificate of manufacture granted in respect of any special mixture of fertilizer's shall be valid for a period of three months from the date of its issue ;Provided that the registering authority may, if it is satisfied that it is necessary so to do extend the said period to such further period or periods as it may deem fit, so however, that the total period or periods so extended shall not exceed six months.

17. Period of validity of certificate of manufacture for preparation of mixtures of fertilizers.

- Every certificate of manufacture granted under Clause 15 for preparation of a mixture of fertilizers shall, unless suspended or cancelled, be valid for a period of three years from the date of issue.

18. Renewal of certificate of manufacture for preparation of mixtures of fertilizers [* * *] [Omitted b S.O. 354(E), dated 3rd June, 1993.].

(1)Every holder of a certificate of manufacture for preparation of a mixture of fertilizers' [* * *] [Omitted b S.O. 354(E), dated 3rd June, 1993.] desiring to renew the certificate, shall, before the date of expiry of the said certificate of manufacture make an application to the registering authority in Form D in duplicate, together with the fee prescribed for this purpose under Clause 36.(2)On receipt of an application for renewal as provided in sub - clause (1), and keeping in view the performance of the applicant and other relevant circumstance, the registering authority may, if he so decides, renew the certificate of registration by endorsement on Form F and in case the certificate of registration is not renewed, the registering authority shall record in writing his reasons for not renewing the certificate of registration.(3)If an application for renewal is not made before the expiry of the certificate of registration but is made within one month from the date of expiry of the certificate of registration the certificate of registration may be renewed on payment of such additional fee as may be prescribed by the State Government for this purpose.(4)Where the application for renewal is made within the time specified in sub - clause (1) or sub - clause (3), the applicant shall be deemed to have held a valid certificate of registration until such date as the registering authority passes order on the application for renewal.(5)If an application for renewal of a certificate of manufacture is not made within the period stipulated under sub - clause (1) or, as the case may be, under sub - clause (3), the certificate of manufacture shall be deemed to have expired

immediately on the expiry of its validity period, and any business carried on after that date shall be deemed to have been carried on in contravention of Clause 12.VI. Restrictions On Manufacture, Sale, Etc. Of Fertilizers

19. Restriction on manufacture, sale and distribution of fertilizers.

- [* *] [Omitted by S.O. 163(E), dated 14th February, 1994.] No person shall himself or by any other person on his behalf -(a)manufacture for sale, sell, offer for sale, stock or exhibit for sale or distribute any fertilizer which is not of prescribed standard ;(b)manufacture for sale, sell, offer for sale, stock or exhibit for sale, or distribute any mixture of fertilizers [mixture of fertilizers] [Substituted by S.O. 354(E), dated 3rd June, 1993.] which is not of prescribed standard [(subject to such limits of permissible variation as may be specified from time to time by the Central Government)] [Inserted by S.O. 725 (E), dated 28th July, 1988 (w.e.f. 28th July 1988).] or special mixture of fertilizers which [* * *] [Omitted by S.O. 725(E), dated 28th July, 1988 (w.e.f. 28th July, 1988)] does not conform to the particulars specified in the certificate of manufacture granted to him under this Order in respect of such special mixture ;(c)sell, offer for sale, stock or exhibit for sale or distribute(i)any fertilizer the container whereof is not packed and marked in the manner laid down in this Order ;(ii)any fertilizer which is in limitation or a substitute for another fertilizer under the name of which it is sold ;(iii)any fertilizer which is adulterated ;Explanation. - A fertilizer shall be deemed to be adulterated, if it contains any substance the addition of which is likely to eliminate or decrease its nutrient contents or make the fertilizer not conforming to the prescribed standard.(iv)any fertilizer the label or container whereof bears the name of any individual, firm or company purporting to be manufacturer of the fertilizer which individual, firm or company is fictitious or does not exist ;(v)any fertilizer, the label or container whereof anything accompanying therewith bears any statement which makes a false claim for the fertilizer or which is false or misleading in any material particular ;(vi)any substance as a fertilizer which substance is not, in fact, a fertilizer ; or(vii)any fertilizer without exhibiting the minimum guaranteed percentage by weight of plan nutrient.

20. Specifications in respect of imported fertilizers.

- Notwithstanding anything contained in this Order, the Central Government may, by an order published in the Official Gazette, fix separate specifications in respect of imported fertilizers.

21. Manufacturers/pool handling agencies to comply with certain requirements in regard to packing and making, etc.

- [* * *] [Omitted by S.O. 163(E), dated 14th February, 1994.] Every manufacturer and pool handling agency shall in regard to packing and making of containers of fertilizers, comply with the following requirements, namely :(a)[every container in which any fertilizer is packed shall bear only such particulars and nothing else as may from time to time be specified by the controller in this behalf, and] [Substituted by S.O. 163(E), dated 14th February, 1994.](b)every container shall be so packed and sealed that the contents thereof cannot be tampered with without breaking the seal :Provided

that where fertilizer manufactured in India are packed in bags stitched in hand, such bags shall bear lead seals, so that the contents thereof cannot be tampered with without breaking the seals. Provided further that lead sealing shall not be necessary, -(i) if such bags are machine stitched in such a manner that contents thereof cannot be tampered with without a visible break in the stitching ; and (ii) in the case of fertilizers imported from abroad and packed in bags stitched in hand, in such a manner that the contents thereof cannot be tampered with without visible break in the stitching. [21A. Manufacturers to comply with certain requirements for laboratory facilities. - Every manufacture shall, in order to ensure quality of their product, possess the minimum laboratory facility, as may be specified from time to time, by the Controller] [Inserted by S.O. by S.O. 261(E) dated 16th April, 1991 (w.e.f. 16th April, 1991).].

22. Bulk sale of fertilizers.

- Notwithstanding anything contained in the Order, -(a) a retail dealer may retain at any time one bag or container of each variety of fertilizer in an open and unsealed condition for the purpose of sale ; (b) a manufacturer may sell the fertilizer manufactured by him in bulk to a manufacturer of mixture of fertilizers, compound/complex fertilizers or special mixture of fertilizers ; and (c) the Central Government may by notification published in the Official Gazette in this behalf authorise a manufacturer to sell any fertilizer manufactured by him in bulk also direct to farmers for such period as may be specified in that notification: Provided that a certificate indicating the minimum guaranteed percentage of plant nutrients is issued by the manufacturer to each former at the time of such sale.

23. Disposal of non - standard fertilizers.

(1) Notwithstanding anything contained in this Order, a person may sell, offer for sale, stock or exhibit for sale or distribute any fertilizer which, not being an adulterated fertilizer, does not conform to the prescribed standard (hereinafter in this Order referred to as non - standard fertilizer) subject to the conditions that, -(a) the container of such non - standard fertilizer is conspicuously superscribed in red colour with the words "non - standard" and also with the sign "X", and (b) an application for the disposal of non - standard fertilizers in Form H is submitted to the registering authority to grant a certificate of authorisation for sale of such fertilizers and a certificate of authorisation with regard to their disposal and price is obtained in Form I ; (c) such non - standard fertilizer shall be sold only to the manufacturers of mixtures of fertilizers or special mixtures of fertilizers or research farms of Government or universities of such bodies. (2) The price per unit of the non - standard fertilizer shall be fixed by the registering authority after satisfying itself that the sample taken is a representative one, and after considering the nutrient contents in the sample determined on the basis of a chemical analysis of the non - standard fertilizer. (3) The Central Government may, by notification in the Official Gazette and subject to the conditions, if any, laid down in that notification, and subject to guidelines issued in this regard by the Central Government exempt such pool handling agencies, as it deems fit, from complying with conditions laid down in paragraphs (a) and (b) of the sub - clause (1).

24. Manufacturers/pool handling agencies to appoint officers responsible with compliance of the order.

- Every manufacturing organisation [importer] [Inserted by S.O. 163(E), dated 14th February, 1994.] and pool handling agency shall appoint in that organisation and in consultation with the Central Government, an officer, who shall be responsible for compliance with the provisions of this Order.

25. Restriction on sale/use of fertilizers.

(1) No person shall, except with the prior permission of the Central Government and subject to such terms and conditions as may be imposed by such Government, sell or use fertilizer, for purposes other than fertilization of soils and increasing productivity of crops : Provided that the price of fertilizers permitted for sale for industrial use shall be no profit no loss price, excluding all subsidies at the production, import, handling or on sale for agricultural consumers : Provided further that wherever customs or excise duties are chargeable, these may be added to the price so fixed : Provided also that in the case of non - standard fertilizers, reductions shall be made from the no profit no loss price, indicated above, proportionate to the loss of nutrient contents. (2) [Notwithstanding anything contained in sub - clause (1), no prior permission for use of fertiliser for industrial purposes shall be necessary when the fertiliser for such purpose is purchased from the industrial dealer possessing a valid certificate of registration granted under Clause 9. (3) Any person possessing a valid certificate of registration for industrial dealer, unless such person is a State Government, a manufacturer or a pool handling agency, shall not carry on the business of selling fertilisers for agricultural purposes, including a wholesale dealer or a retail dealer. However, in case of a State Government, a manufacturer or a pool handling agency possessing a valid certificate of registration of sale of fertiliser for industrial use and also for sale of fertiliser for agriculture use, whether in wholesale or retails or both, shall not carry on the business of selling fertilisers both for industrial use and agricultural use in the same premises.] [Added by S.O. 795(E), dated 22nd November, 1991.] VII. Enforcement Authorities

26. Appointment of registering authority.

- The State Government may, by notification in the Official Gazette, appoint such number of persons, as it thinks necessary, to be registering authorities for the purpose of this Order, ["excepting for grant or renewal of certificate of registration for industrial dealer"] [Added by S.O. 795(E), dated 22nd November, 1991.] and may, in any such notification, define the limits of local area within which each such registering authority shall exercise his jurisdiction.

27. Appointment of inspectors.

- The State Government, or the Central Government may, by notification in the Official Gazette, appoint such number of persons, as it thinks necessary, to be inspectors of fertilizers for the purpose of this Order, and may, in any such notification, define the limits of local area within which each such inspector shall exercise his jurisdiction. [27A. Qualifications for appointment of fertilizer

inspectors. - No person shall be eligible for appointment as fertilizer inspector under this order, unless he possesses the following qualifications, namely : -(1)graduate in Agriculture or Science with Chemistry as one of the subjects, from a recognised University, and(2)training or experience in the quality control of fertilizers and working in the State or Central Department of Agriculture.]

28. Powers of Inspectors.

(1)An Inspector may, with a view to securing compliance with this Order, -(a)require any manufacturer, pool handling agency, wholesale dealer or retail dealer to give any information in his possession with respect to the manufacture, storage and disposal of any fertilizer manufactured or, in any manner handled by him;(b)draw samples of any fertilizer in accordance with the procedure of drawal of samples laid down in Schedule II :Provided that the Inspector shall prepare the sampling details in duplicate in Form J, and hand over one copy of the same to the dealer or his representative from whom the sample has been drawn ;(c)enter upon and search any premises where any fertilizer is manufactured or stored or exhibited for sale, if he has reason to believe that any fertilizer has been or is being manufactured, sold, offered for sale, stored, exhibited for sale or distributed contrary to the provisions of this Order ;(d)seize or detain any fertilizer in respect of which he has reason to believe that a contravention of this Order has been or is being or is about to be committed ;(e)seize any books of accounts or documents relating to manufacture, storage or sale of fertilizers, etc, in respect of which he has reason to believe that any contravention of this Order has been or is being or is about to be committed :Provided that the inspector shall give a receipt for such fertilizers or books of accounts or documents so seized to the person from whom the same have been seized :Provided further that the books of accounts or documents so seized shall be returned to the person from whom they were seized after copies thereof or extracts therefrom as certified by such person, have been taken.[Provided also that the Inspector shall give the stop sale notice in writing, to the person whose stocks have been detained and initiate appropriate actions as per the provisions of this Order within a period of twenty - one days. If no action has been initiated by the Inspector within the said period of twenty - one days from the date of issue of the said notice, the notice of stop sale shall be deemed to have been revoked.] [Added by S.O. 140(E), dated 12th February, 1990 (w.e.f. 12th February,1990).](2)Subject to the proviso to paragraphs (d) and (e) of sub - clause (1), the provisions of the Criminal Procedure Code, 1973 (2 of 1974), relating to search and seizure shall, so far as may be, apply to searches and seizures under this clause.(3)Where any fertilizer is seized by an Inspector under this clause he shall forth with report the fact of such seizure to the Collector whereupon the provisions of Sections 6 - A, 6 - B, 6 - C, 6 - D and 6 - E of the Act, shall apply to the custody, disposal and confiscation of such fertilizers.(4)Every person, if so required by an inspector, shall be bound to afford all necessary facilities to him for the purpose of enabling him to exercise his powers under sub - clause (1).VIII. Analysis Of Samples

29. Laboratory for analysis.

- A fertilizer sample, drawn by an Inspector, shall be analysed in accordance with the instructions contained in Schedule II in the Central Fertilizer Quality Control and Training Institute [Faridabad or Regional Fertilizer Control Laboratories at Bombay, Madras or Kalyani (Calcutta)] [Inserted by S.O. 498(E), dated 29th June, 1989 (w.e.f. 29th June, 1989).] or in any other laboratory notified for

this purpose by the State Government.[29A. Qualifications for appointment of fertilizer analyst in the fertilizer quality control laboratories.] [Inserted by S.O. 261(E), dated 16th April, 1991 (w.e.f. 16th April, 1991).] - No person shall be eligible for appointment as fertilizer analyst or analysis of fertilizer samples in the laboratories notified under Clause 29 of the order, unless the possesses the following qualifications, namely :(1)graduate in Agriculture or Science with Chemistry as one of the subject from a recognised University, and(2)training in fertilizer quality control and analysis at Central Fertilizer Quality Control and Training Institute, Faridabad.Provided that the fertilizer analysts appointed before the commencement of this order, who do not possess the requisite training, shall undergo prescribed training, within a period of three years, in the Central Fertilizer Quality Control and Training Institute. Faridabad, from the date of commencement of this order.

30. Time - limit for analysis and communication of result.

(1)Where sample of a fertilizer has been drawn, the same shall be despatched, along with a memorandum in Form K to the laboratory for analysis within a period of seven days from the date of its drawal.(2)The laboratory shall analyse the sample and forward the analysis report in Form "L" within 60 days from the date of receipt of the sample in the laboratory to the authority specified in the said memorandum.(3)The authority to whom the analysis report is sent under sub - clause (2) shall communicate the result of the analysis to the dealer/manufacturer/pool handling agency from whom the sample was drawn within 30 days from the date of receipt of the analysis report of the laboratory.IX. Miscellaneous

31. Suspension/Cancellation of registration certificate.

(1)A registering authority ["or, as the case may be, the controller"] [Inserted by S.O. 795(E), dated 22nd November,1991.] may, after giving the holder of a certificate of registration or a certificate of manufacture or any other certificate granted under this Order, an opportunity of being heard, suspend or cancel such certificate on any of the following grounds, namely :(a)that such certificate has been obtained by misrepresentation as to material particulars.(b)that any of the provisions of this Order or any of the terms and conditions of such certificate has been contravened or not fulfilled :Provided that while cancelling the certificate the holder thereof may be allowed a period of 30 days to dispose of the balance stock of fertilizers if any, held by him :Provided further that the stock of fertilizer lying with the holder after the expiry of the said 30 days shall be confiscated.(2)Where the contravention alleged to have been committed by a person is such as would, on being proved, justify cancellation of the certificate of registration or, as the case may be, certificate of manufacture or any other certificate granted under this Order to such person the registering authority ["or, as the case may be, the controller"] [Inserted by S.O. 795(E), dated 22nd November,1991.] may, without any notice, suspend such certificate, as an interim measure :Provided that the registering authority ["or, as the case may be, the controller"] [Inserted by S.O. 795(E), dated 22nd November,1991.] shall immediately furnish to the person details and the nature of contravention alleged to have been committed by such person and, after giving the person an opportunity of being heard, pass final orders either revoking the order of suspension or cancelling the certificate within fifteen days from the date of issue of the order of suspension :Provided further that where no final order is passed within the period as specified above, the order of interim

suspension shall be deemed to have been revoked without prejudice, however, to further action which the registering authority ["or, as the case may be, the controller"] [Inserted by S.O. 795(E), dated 22nd November, 1991.] may take against the holder of the certificate under sub - clause (1).(3)Wherever a certificate is suspended or cancelled under this clause, the registering authority ["or, as the case may be, the controller"] [Added by S.O. 795(E), dated 22nd November, 1991.] shall record a brief statement of the reasons for such suspension or, as the case may be, cancellation and furnish a copy thereof to the person whose certificate has been suspended or cancelled.(4)[Wherever the person alleged to have committed the contravention is an industrial dealer, the registering authority may also take action against the holder of such certificate of registration, under sub - clauses (1) and (2) :Provided that where such certificate is suspended or cancelled the registering authority shall within a period of fifteen days from the date of issue of such order of suspension or cancellation, furnish to the Controller also, besides sending the same to the person whose certificate has been suspended or cancelled, a detailed report about the nature of contravention committed and a brief statement of the reasons for such suspension or, as the case may be, cancellation :Provided further that the Controller shall, in case of the order for suspension passed by the registering authority, on receipt of the detailed report and after giving the person an opportunity of being heard, pass final order either revoking the order of suspension or cancelling the certificate of registration, within fifteen days from the date of receipt of the detailed report from the registering authority, falling which the order of interim suspension passed by the registering authority shall be deemed to have been revoked without prejudice, however, to further action which the Controller may take against the holder of certificate under sub - clause (1) :Provided also that the order of cancellation passed by the registering authority shall remain effective as if it has been passed by the Controller till such time the Controller, on receipt of the detailed report from the registering authority, and if deemed necessary, after giving the person a fresh opportunity of being heard, pass the final order either revoking or confirming the order of cancellation.] [Added by S.O. 795(E), dated 22nd November, 1991.]

32. Appeal.

- [Any person, excepting an industrial dealer or, as the case may be, the person desiring to obtain a certificate of registration for industrial dealer, aggrieved by] [Substituted by S.O. 795(E) dated 22nd November, 1991.]: -(1)an order -(a)refusing to grant, amend or renew a certificate of registration for sale of fertilizers ; or(b)refusing to grant a certificate of manufacture for preparation of mixture of fertilizers or special mixture of fertilizers ; or(c)suspending or cancelling a certificate of registration or manufacture ; or(2)non - issuance of certificate of registration to him within the stipulated period - may within sixty days from the date of receipt of such order or, as the case may be, from the date of expiry of such stipulated period, appeal to such authority as the State Government may specify in this behalf, and the decision of such authority shall be final.

33. Grant of duplicate copies of certificate of registrations, etc.

- Where a certificate of registration or a certificate of manufacture or any other certificate granted or, as the case may be, renewed under this Order is lost, the registering authority ["or, as the case may be, the controller"] [Inserted by S.O. 795(E), dated 22nd November, 1991.] may, on an

application made in this behalf, together with the fee prescribed for this purpose under Clause 36, grant a duplicate copy of such certificate.

34. Amendment of certificate of registration.

- The registering authority ["or, as the case may be the controller"] [Inserted by S.O. 795(E), dated 22nd November, 1991.] may, on an application made in this behalf, together with the fee prescribed for the purpose under Clause 36, amend a certificate of registration.

35. Maintenance of records and submission of returns, etc.

(1)The Controller may by an order made in writing direct the dealers, manufacturers, and pool handling agencies -(a)to maintain such books of accounts, records, etc. relating to their business [in Form N] [Substituted by S.O. 261(E), dated 16th April, 1991 (w.e.f. 16th April, 1991).] ; and(b)to submit to such authority, returns and statements in such form and containing such information relating to their business and within such time as may be specified in that order.(2)Where a person holds certificates of registration for retail sale and wholesale sale of fertilizers, he shall maintain separate books of accounts for these two types of sales made by him.(3)[Where a State Government, a manufacturer and a pool handling agency holds valid certificates of registration for sale of fertilisers in wholesale or retail or both and also for sale for industrial use, he shall maintain separate books of accounts for these two, or three types of sales made by him.] [Added by S.O. 795(E), dated 22nd November, 1991.](4)[Every importer shall inform the Director of Agriculture of the State in which he intends to discharge the imported fertiliser, under intimation to the Central Government, before the import is made or within a period of fifteen days after an indent for import as placed, the following details, namely : -(i)name of fertiliser,(ii)name of the country of importer,(iii)name of manufacturer,(iv)quantity to be imported,(v)date of arrival of the consignment,(vi)name of the discharge port,(vii)other information.]

36. Fees.

(1)The fees payable for grant, amendment or renewal of a certificate of registration or certificate of manufacture a duplicate of such certificates or renewal thereof under this Order shall be such as the State Government may, with prior approval of the Controller, from time to time, fix, subject to the maximum fees fixed for different purposes by the Central Government and different fees may be fixed for different purposes or for different classes of dealers or for different types of mixtures of fertilizer or special mixture.Provided that no fee shall be payable by a person holding a valid certificate of manufacture for preparation of mixture of fertilizers or special mixture of fertilizers issued under the Fertilizer (Control) Order, 1957 and making an application for grant of a fresh certificate of manufacture for preparation of such mixture after the commencement of this Order.(2)The authority to whom and the manner in which the fee fixed under sub - clause (1) shall be paid shall be such as may be specified by the State Government by notification in the official Gazette.(3)Any fee paid under sub - clause (1) shall not be refundable unless the grant or renewal of any certificate of registration or certificate of manufacture or duplicate copy of such certificate or renewal under this Order has been refused.(4)[The fees payable for grant, amendment, renewal or

duplicate copy of certificate of registration for industrial dealer and the authority to whom and the manner in which such fee shall be paid, shall be such as may be specified by the Controller, from time to time by notification in the Official Gazette.] [Added by S.O. 795(E), dated 22nd November, 1991.]

37. Service of orders and directions.

- Any order or direction made or issued by the Controller or by any other authority under this order shall be served in the same manner as provided in sub - section (5) of Section 3 of the Act.

38. Advisory Committee.

(1)The Central Government may by notification in the Official Gazette and on such terms and conditions as may be specified in such notification, constitute a Committee called the Central Fertilizer Committee consisting of a Chairman and not more than ten other persons, having experience or knowledge in the field, who shall be members of the Committee, to advise the Central Government regarding -(i)inclusion of a new fertilizer, under this Order ;(ii)specifications of various fertilizers(iii)grades/formulations of physical granulated mixtures of fertilizers that can be allowed to be prepared in a State ;(iv)requirements of laboratory facilities in a manufacturing unit, including a unit manufacturing physical/granulated mixtures of fertilizers ;(v)Methods of drawal and analysis of samples ;(vi)any other matter referred by the Central Government to the Committee.(2)The Committee may, subject to the previous approval of the Central Government, make bye - laws fixing the quorum and regulation its own procedure and the conduct of all business to be transacted by it.(3)The Committee may co - opt such number of experts and for such purposes or periods as it may deem fit, but any expert so co - opted shall not have the right to vote.(4)The Committee may appoint one or more sub - committees, consisting wholly of members of the Committee or wholly of co - opted members or partly of the members of the Committee and partly of co - opted members, as it thinks fit, for the purpose of discharging such its functions as may be delegated to such sub - committee or sub - committees by the Central Fertilizer Committee.(5)[The State Government may by notification in the Official Gazette and on such terms and conditions as may be specified in such notification, constitute a Committee called the State Fertilizer Committee consisting of a Chairman and not more than 5 other members, having experience or knowledge in the field, including a representative from State Agricultural University, the Fertilizer Industry and Indian Micro Fertilizers Manufacturers' Association to advise the State Government regarding the grades/formulations of [mixture of fertilizer] [Inserted by S.O. 725(E), dated 28th July, 1988 (w.e.f. 28th July, 1988).] and their specifications] [Added by S.O. 397(E), dated 18th June, 1993].

39. Repeal and saving.

(1)The Fertilizers (Control) Order, 1957 is hereby repealed except as respects things done or omitted to be done under the said Order before the commencement of this Order.(2)Notwithstanding such repeal, an order made by any authority, which is in force immediately before the commencement of this Order and which is consistent with this Order, shall continue in force and all appointments made, prices fixed, certificates granted and directions issued under repealed Order and in force

immediately before such commencement shall likewise continue in force and be deemed to be made, fixed, granted or issued in pursuance of this order till revoked.

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[See Clause 2(h)& (q)]

Part A – Specifications of fertilisers

Name of the Fertiliser	Specifications
1	2
1.(a) Straight Nitrogenous Fertilisers :	
1. Ammonium Sulphate	(i) Moisture per cent by weight, maximum 1.0
(ii) Ammoniacal nitrogen percent. by weight, minimum	20.6
(iii) Free acidity (as H ₂ SO ₄) per cent.by weight maximum (0.04 for material obtained from by product ammonia and by - product gypsum)	0.025
(iv) Arsenic as (A S ₂ O ₃) per cent. byweight, maximum	0.01
2. Urea (46% N) (White free flowing)	(i) Moisture per cent. by weight maximum 1.00
(ii) Total nitrogen, per cent. by weight, (on dry basis)minimum	46.00
(iii) Biuret per cent. By' weight, maximum	1.5
(iv) Particle size - 90 per cent. of the material shall passthrough 2.8 mm IS sieve and not less than 80 per cent. by weightshall be retained on 1 mm IS sieve.	1.5
3. Urea (coated) (45% N) (White free flowing)	(i) Moisture per cent. by weight, maximum 0.5
(ii) Total nitrogen per cent. by weight content with coatingminimum	45.0
(iii) Biuret per cent. by weight maximum	15
(iv) Particle size - 90 per cent. of the material shall passthrough 2.8 mm IS sieve and not less than 80 per cent. by weightshall be retained on i mm IS sieve.	
4. Ammonium Chloride	(i) Moisture per cent. by weight, maximum 2.0
(ii) Ammoniacal nitrogen per cent. by weight, minimum	25.0

(iii) Chloride other than ammonium chloride (as NaCl) percent. by weight (on dry basis) maximum	2.0	
(iv)[* * *] [Omitted by S.O. 1079(E), dated 11th December, 1987 (w.e.f. 11th December, 1987).]		
5. Calcium Ammonium (Nitrate 25%N)	(i) Moisture percent. by weight, maximum	1.00
(ii) Total ammoniacal and nitrate nitrogen	25.0	
(iii) Ammoniacal nitrogen per cent. by weight, minimum	12.5	
(iv) Calcium nitrate per cent. by weight.maximum	0.5	
(v) Particle size - 80 per cent. of the material shall passthrough 4 mm IS sieve and be retained on 1 mm IS sieve. Not morethan 10 per cent. shall be below 1 mm IS sieve.		
6. Calcium Ammonium Nitrate (26% N)	(i) Moisture per cent, by weight, maximum	1.0
(ii) Total ammoniacal and nitrate nitrogen per cent. byweight, minimum	26.0	
(iii) Ammoniacal nitrogen per cent. by weight, minimum	13.0	
(iv) Calcium nitrate per cent. by weight, maximum	0.5	
(v) Particle size - 90 per cent. of the material shall passthrough 4 mm IS sieve and be retained on 1 mm IS sieve. Not morethan 5 percent shall be below 1 mm IS sieve.		
7. Anhydrous Ammonia	(i) Ammonia per cent. by weight, minimum	99.0
(ii) Water per cent. by weight, maximum	1.0	
(iii) Oil content by weight, maximum 20 ppm		
["8. Urea Super Granulated.] [Inserted by S.O. 271(E), dated 29th March, 1990 (w.e.f. 29th March, 1990).]	(i) Moisture, percent, by weight, maximum	1.00
(ii) Total nitrogen, per cent. by weight, (on 46.00 dry basis)minimum	46.00	
(iii) Biuret per cent. by weight, maximum	1.5	
(iv) Particle size, 90 per cent. of the material shall passthrough 13.2 mm IS sieve and not less than[80] [For 39 per cent, corrected by S.O. 332(E), dated 18th April, 1990.]per cent.by weight shall be retained on 9.5 mm IS sieve		

["9. (Urea granular)] [Added by S.O. 354(E), dated 3rd June, 1993.]		(i) Moisture per cent. by weight, maximum	1.0
(ii) Total nitrogen, per cent. by weight, minimum	1.5		
(iii) Biurate, per cent, by weight, maximum	1.5		
(iv) Particle size 90 per cent. of the material shall 15 passthrough 4 mm IS sieve and be retained on 2 mm IS sieve. Not morethan 5 per cent. shall be blow 2 mm IS sieve."	1.5		
1.(b) Straight Phosphatic fertilisers :			
1. Single Super - phosphate (16% P ₂ O ₅ powered)		(i) Moisture percent. by weight, maximum	12.0
(ii).Free phosphoric acid (as P ₂ O ₅) percent. by weight, maximum	4.0		
(iii) Water soluble phosphates (as P ₂ O ₅)per cent. by weight, minimum	16.0		
2. Single Super - phosphate (14% P ₂ O ₅ powered)		(i) Moisture per cent. by weight, maximum	12.0
(ii) Free phosphoric acid (as P ₂ O ₅) percent. by weight, maximum	4.0		
(iii) Water soluble phosphates (as P ₂ O ₅)per cent. by weight, minimum	14.0		
3. Triple Super - phosphate		(i) Moisture per cent. by weight, maximum	12.0
(ii) Free phosphoric acid (as P ₂ O ₅) percent. by weight, maximum	3.0		
(iii) Total phosphates (as P ₂ O ₅) percent. by weight, minimum	46.0		
(iv) Water soluble phosphates (as P ₂ O ₅)per cent. by weight, minimum	42.5		
4. Bone meal, Raw.		(i) Moisture per cent. by weight, maximum	8.0
(ii) Acid insoluble matter per cent. by weight, maximum	12.0		
(iii) Total phosphates (as P ₂ O ₅) percent. by weight, maximum	20.0		
(iv) 2 per cent. citric acid soluble phosphates (as P ₂ O ₅)per cent. by weight, minimum	8.0		
(v) Nitrogen content of water insoluble portion per cent. byweight, minimum			
(vi) Particle size—The material shall pass whollythrough 2.36 mm IS sieve of which not			

more than 30 per cent. shall be retained on 0.85 mm IS sieve.

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|--|---|------|
| 5. Bone meal, Steamed | (i) Moisture per cent. by weight, maximum | 7.0 |
| (ii) Total phosphates (as P ₂ O ₅) percent. by weight (on dry basis), minimum | | 22.0 |
| (iii) 2 per cent, citric acid soluble phosphates (as P ₂ O ₅) per cent. by weight (on dry basis), minimum | | 16.0 |
| (iv) Particle size—Not less than 90 per cent. of the material shall pass through 1.18 mm IS sieve. | | 1.18 |

6. Rock - phosphate

- | | |
|---|------|
| (i) Particle size—minimum 90 per cent. of the material shall pass through 0.15 mm IS sieve and the balance 10 per cent. of material shall pass through 0.25 mm IS sieve. | |
| (ii) [Total phosphate (as P ₂ O ₅) per cent. by weight, minimum] [Substituted. by S.O. 822(E), dated 14th October, 1987 (w.e.f. 14th October, 1987).] | 18.0 |

7. [Single Super Phosphate (16% P₂O₅ Granulated)] [Added by S.O. 252(E), dated 11th March, 1988 (w.e.f. 11th March, 1988).]

- | | |
|---|-----|
| (i) Moisture per cent, by weight, maximum | 5.0 |
| (ii) Free phosphoric acid (as P ₂ O ₅) percent. by weight, maximum | 4.0 |
| (iii) Water soluble phosphates (as P ₂ O ₅) per cent. by weight, maximum | |
| (iv) Particle size - Not less than 90 per cent. of the material shall pass through 4 mm IS sieve. Not more than 5 per cent. shall pass through 1mm IS sieve.] | |

8. [* * *] [Omitted by S.O. 140(E), dated 12th February, 1990 (w.e.f. 12th February, 1990).]

1. (c) Straight potassic fertilisers :

- | | | |
|---|--|-----|
| 1. Potassium Chloride (Muriate of potash) | (i) Moisture per cent. by weight, maximum | 0.5 |
| (ii) Water soluble potash content (as K ₂ O) percent. by weight, minimum . | 60. [o] [Substituted. by S.O. 252 (E), dated 11th March, 1988 (w.e.f. 11th March, 1988)] | |
| (iii) Sodium as NaCl per cent. by weight (on dry basis), maximum | | |
| (iv) Particle size - [minimum 95 per cent.] [Substituted. by S.O. 1068(E), dated 4th | | |

November, 1999 (w.e.f. 4th November, 1999),
for the figures and word "95 per cent.".]of
thematerial shall pass through 1.7 mm IS sieve
and be retained on 0.25 mm IS sieve.

2. Potassium Sulphate	(i) Moisture per cent. by weight, maximum	1.5
(ii) Potash content (as K ₂ O) per cent. by 6.[weight] [Substitued, by S.O. 261(E), dated 16th April, 1991 (w.e.f. 16th April, 1991).], minimum	50. [00] [Substitued. by S.O. 261(E), dated 16th April, 1991 (w.e.f. 16th April, 1991).]	
(iii) Total chlorides (as Cl) as per cent. by weight (on drybasis), maximum		
(iv) Sodium as NaCl per cent. by weight (on dry basis),maximum	2.0	
3. Potassium Schoenite	(i) Moisture per cent. by weight, maximum	1.5
(ii) Potash content (as K ₂ O) per cent. by weight(on dry basis), minimum	23.0	
(iii) Magnesium oxide (as MgO) per cent. by weight, maximum	10.0	
(iv) Total chlorides (as Cl) (on dry basis) per cent. byweight, maximum	2.5	
(v) Sodium (as NaCl) (on dry basis) per cent. by weight,maximum	1.5	
4. [PotassiumChloride (Murriate of Potash (Grnular)] [Added by S.O. 354(E), dated 3rd June, 1993.]	(i) Moisture per cent. by weight, maximum	0.5
(ii) Water soluble potash (at K ₂ O) per cent. by weight minimum	60.9	
(iii) Sodium (as NaCl) per cent. by weight maximum	3.5	
(iv) Magnesium (as MgCl ₂) per cent. by weightmaximum	1.0	
(v) Particle size - 90 per cent of the material shall passthrough 3.35 mm IS sieve and be retained on 1 mm IS sieve. Notmore than 5 per cent. shall be blow 1 mm IS sieve.]		
1.(d) N.P. Fertilisers :[* * *] [Omitted by S.O. 377(E), dated 29th May, 1992.]		
1. Diammonium Phosphate (18 - 46 - 0)	(i) Moisture per cent. by weight maximum	1.0
(ii) Total nitrogen all in ammoniacal form per cent. byweight, minimum	18.0	
	46.0	

(iii) Total phosphates (as P ₂ O ₅) percent. by weight, minimum		
(iv) Water soluble phosphates (as P ₂ O ₅) per cent. by weight, minimum]	41.0	
2. Diammonium Phosphate[* * *] [Omitted by S.O. 377(E), dated 29th May, 1992.](18 - 46 - o)	(i) Moisture per cent. by weight maximum	1.5
(ii) Total nitrogen per cent. by weight, minimum	18.00	
(iii) Ammoniacal nitrogen form per cent by weight, minimum	15.5	
(iv) Total nitrogen in the form of urea per cent. by weight, maximum	2.5	
(v) Neutral ammonium citrate soluble phosphates (as P ₂ O ₅) per cent. by weight, minimum		
(vi) Water soluble phosphates (as P ₂ O ₅) per cent. by weight, minimum	41.0	
(vii) Particle size - 90 per cent. of the material shall passthrough 4 mm IS sieve and be retained on 1 mm IS sieve. Not more than 5 per cent. shall be below than 1 mm size.		
3. Ammonium, phosphate Sulphate (16 - 20 - o)	(i) Moisture per cent. by weight, maximum	1.0
(ii) Total ammoniacal nitrogen per cent by weight, minimum	16.0	
(iii) Neutral ammonium citrate soluble Phosphates (as P ₂ O ₅) per cent by weight, minimum	20.0	
(iv) Water soluble phosphates (as P ₂ O ₅) per cent. by weight, minimum	19.5	
(v) Particle size - 90 per cent. of the material shall passthrough 4 mm IS sieve and shall be retained on 1 mm IS sieve. Not more than 5 per cent. shall be below 1 mm IS sieve.		
4. Ammonium phosphate Sulphate (20 - 20 - o)	(i) Moisture per cent. by weight, maximum	1.0
(ii) Total nitrogen per cent. by weight, minimum	20.0	
	18.0	

(iii) Ammoniacal nitrogen percent. by weight, minimum		
(iv) Nitrogen in the form of urea per cent. by weight, maximum	2.0	
(v) Neutral ammonium citrate soluble phosphates (as P ₂ O ₅) per cent. by weight, minimum	20.0	
(vi) Water soluble phosphates (as P ₂ O ₅) per cent. by weight, minimum	17.0	
(vii) Particle size - 90 percent. of the material shall passthrough 4 mm IS sieve and shall be retained on 1 mm IS sieve. Notmore than 5 per cent shall be below 1 mm IS sieve.		
5. Ammonium phosphate Sulphate nitrate (20 - 20 - 0)	(i) Moisture per cent. by weight, maximum	
(ii) Total nitrogen per cent. by weight minimum	20.0	
(iii) Ammoniacal nitrogen per cent by weight, maximum	17.0	
(iv) Nitrate nitrogen per cent by weight maximum	3.0	
(v) Neutral ammonium citrate soluble phosphates (as P ₂ O ₅) per cent. by weight, minimum	20.0	
(vi) Water soluble phosphates (as P ₂ O ₅) per cent. by weight, minimum	17.0	
(vii) Particle size - 90 per cent. of the material shall be passthrough 4 mm IS sieve. Not more than 5 per cent. shall be below 1mm IS sieve.		
6. Ammonium phosphate Sulphate (18 - 9 - 0)	(i) Moisture percent. by weight, maximum	1.0
(ii) Ammoniacal nitrogen per cent by weight, minimum	18.0	
(iii) Nitrate ammonium citrate soluble phosphates (as P ₂ O ₅) by weight minimum	9.0	
(iv) Water soluble phosphates (as P ₂ O ₅) per cent by weight, minimum	8.5	
(v) Particle size - - 90 per cent of the material shall passthrough 4 mm IS sieve and be retained on 1 mm IS sieve. Not morethan 5 per cent. shall be below 1 mm IS sieve.		
7. Nitrogen Phosphate (20 - 20 - 0)	(i) Moisture per cent. by weight, maximum	1.5

(ii) Total nitrogen per cent. by weight, minimum	20.00	
(iii) Nitrogen in ammoniacal form per cent by weight, minimum	10.0	
(iv) Nitrogen in nitrate form per cent by weight, maximum	10.0	
(v) Neutral ammonium citrate soluble phosphates (as P ₂ O ₅) per cent by weight, minimum		
(vi) Water soluble phosphates (as P ₂ O ₅) per cent by weight, minimum 1. Substituted. by S.O. 942(E), dated 10th December, 1993.	12. [0] [[Substitued. by S.O. 942(E), dated 10th December, 1993.]]	
(vii) Calcium Nitrate per cent by weight, maximum	1.0	
(viii) Particle size - 90 per cent of the material shall passthrough 4 mm IS sieve and be retained on 1 mm IS sieve. Not morethan 5 per cent shall be below 1 mm IS sieve.		
8. Urea Ammonium Phosphate (28 - 28 - 0)	(i) Moisture per cent. by weight, maximum	1.5
(ii) Total nitrogen per cent by weight, minimum	28.0	
(iii) Ammoniacal nitrogen per cent by weight, minimum	9.0	
(iv) Neutral ammonium citrate soluble phosphate (as P ₂ O ₅) per cent by weight, minimum		
(v) Water soluble phosphates (as P ₂ O ₅) per cent. by weight, minimum	25.2	
(vi) Particle size - 90 per cent. of the material shall passthrough 4 mm IS sieve and be retained on 1 mm IS sieve. Not morethan 5 per cent shall be below 1 mm IS sieve.		
9. Urea Ammonium Phosphate (24 - 24 - 0)	(i) Moisture per cent by weight, maximum	15
(ii) Total nitrogen per cent by weight, minimum	24.0	
(iii) Ammoniacal nitrogen per cent, by weight, minimum	7.5	
(iv) Nitrogen in the form of urea per cent by weight, maximum	16.5	
(v) Neutral ammonium citrate soluble phosphates (as P ₂ O ₅) per cent by weight,	24.0	

minimum

(vi) Water soluble phosphate (as P ₂ O ₅) per cent by weight, minimum	20.4	
(vii) Particle size - 90 per cent of the material shall passthrough 4 mm IS sieve. Not more than 5 per cent shall be below 1mm IS sieve.(Note.—The products contain inertfiller material such as sand or dolomite to the extent of 20% byweight, maximum	20.4	
10. Urea Ammonium Phosphate (20 - 20 - 0)	(i) Moisture per cent by weight, maximum	1.5
(ii) Total nitrogen per cent by weight, minimum	20.0	
(iii) Ammoniacal nitrogen per cent by weight, minimum	6.4	
(iv) Neutral ammonium citrate soluble phosphates (as P ₂ O ₅) per cent by weight, minimum	20.0	
(v) Water soluble phosphates (as P ₂ O ₅) per cent by weight, minimum	17.0	
	(vi) Particle size - 90 per cent of the material shall passthrough 4 mm IS sieve and be retained on 1 mm IS sieve. Not morethan 5 per cent shall be below 1 mm IS sieve.(Note.—Thisproduct contains filler materials (inert soil) to the extent of 30 per cent by weight).	
11. Mono Ammonium Phosphate (11 - 52 - 0)	(i) Moisture per cent by weight, maximum	1.0
(ii) Total nitrogen all in ammoniacal for per cent weight, minimum	11.0	
(iii) Neutral ammonium citrate soluble phosphates (as P ₂ O ₅) per cent by weight, minimum	52.0	
(iv) Water soluble phosphates (as P ₂ O ₅) per cent by weight, minimum Particle size - 90 per cent of the material shall passthrough 4 mm IS sieve and be retained on 1 mm sieve. Not morethan 5 per cent shall be below 1 mm IS sieve.	44.2	
12. [Nitrophosphate (23 - 23 - 0)] [Inserted by S.O. 724(E), dated 28th July, 1988 (w.e.f. 28th July, 1988).]	(i) Moisture per cent by weight, maximum	1.5

	(ii) Total Nitrogen per cent by weight, minimum	23.0
(iii) Nitrogen in ammoniacal form per cent by weight, minimum	11.5	
(iv) Nitrogen in nitrate form per cent by weight, minimum	11.5	
(v) Neutral ammonium citrate soluble phosphate (as P ₂ O ₅) per cent by weight, minimum	23.0	
(vi) Water soluble phosphate (as P ₂ O ₅) per cent by weight, minimum	18.5	
(vii) Calcium nitrate per cent by weight, maximum	1.0	
(viii) Particle size - 90 per cent of the material shall passthrough 4 mm IS sieve and be retained on 1 mm IS sieve. Not morethan 5 per cent shall be below 1 mm IS sieve]		
13. [Ammonium Nitrate Phosphate] [Inserted by S.O. 140(E), dated, 12th February, 1990 (w.e.f. 12th February, 1990).]	(i) Moisture per cent by weight, maximum	1.5
(ii) Total nitrogen per cent by weight, minimum	23.0	
(iii) Nitrogen in ammoniacal form per cent by weight, minimum	13.0	
(iv) Nitrogen in nitrate form per cent by weight, maximum	10.0	
(v) Neutral ammonium citrate soluble phosphates (as P ₂ O ₅) per cent by weight, minimum	23.0	
(vi) Water soluble phosphates (as P ₂ O ₅) per cent by weight, minimum	20.5	
(vii) Particle size - 90 per cent of the material shall passthrough 4 mm IS sieve and be retained on 1 mm IS sieve. Not morethan 5 per cent shall be below 1 mm IS sieve.		
15. [AmmoniumPhosphates (14 - 28 - 0)] [Inserted by S.O. 573(E), dated 20th August, 1996.]	(i) Moisture per cent by weight, maximum	1.5
(ii) Total nitrogen per cent by weight, minimum	14.0	
	6.0	

- (iii) Urea nitrogen per cent by weight,
maximum
- (iv) Ammoniacal nitrogen per cent by weight, 8.0
minimum
- (v) Neutral ammonium citrate soluble
phosphates (as P₂O₅) per cent by weight, 28.0
minimum
- (vi) Water soluble phosphates (as P₂O₅) per
cent by weight, minimum 23.0
- (vii) Particle size - 90 per cent of the material
shall passthrough 4 mm IS sieve and be
retained on 1 mm IS sieve. Not morethan 5%
shall be below 1 mm IS sieve.

1. (e) N.P.K. Fertiliser :

1. Nitrophosphate with Potash (15 - 15 - 15) (i) Moisture per cent by weight, maximum 1.5
- (ii) Total nitrogen, minimum 15.0
- (iii) Ammoniacal nitrogen, per cent by weight,
minimum 7.5
- (iv) Nitrate nitrogen, per cent by weight,
maximum 7.5
- (v) Neutral ammonium citrate soluble
phosphates (as P₂O₅) per cent by weight,
minimum
- (vi) Water soluble phosphates (as P₂O₅) per
cent by weight, minimum 4.0
- (vii) Water soluble Potash (as K₂O) per cent
byweight, minimum 15.0
- (viii) Particle size - 90 per cent of the material
shall passthrough 4 mm IS sieve and be
retained on 1 mm IS sieve.
- (ix) Calcium nitrate, per cent by weight,
maximum
2. [N.P.K. (10 - 26 - 26)] [Substitued by GSR
201(E), dated 14th February, 1986 (w.e.f. 14th (i) Moisture per cent by weight, maximum 1.0
February, 1986).]
- (ii) Total nitrogen per cent by weight,
minimum 10.0
- (iii) Ammoniacal nitrogen per cent by weight,
minimum 7.0
- (iv) Nitrogen in the form of the urea per cent.
by weight,maximum 3.0

(v) Neutral ammonium citrate soluble phosphate (as P ₂ O ₅) per cent by weight, minimum	26.0	
(vi) Water soluble potash (as K ₂ O) per cent by weight, minimum	26.0	
(vii) Water soluble phosphates (as P ₂ O ₅) per cent by weight, minimum	22.1	
(viii) Particle size—Particle size of the material will be such that 90 per cent of the material will be between 1 mm and 4 mm IS sieve and not more than 5 per cent will be below 1 mm size.		
3. N.P.K. (12 - 36 - 16)	(i) Moisture per cent weight, maximum	1.0
(ii) Total nitrogen per cent by weight, minimum	12.0	
(iii) Ammoniacal nitrogen per cent by weight, minimum	9.0	
(iv) Nitrogen in the form of urea per cent by weight, maximum	3.0	
(v) Neutral ammonium citrate soluble phosphates (as P ₂ O ₅) per cent by weight, minimum	32.0	
(vi) Water soluble phosphates (as P ₂ O ₅) per cent by weight, minimum	27.2	
(vii) Water soluble potash (as K ₂ O) per cent by weight, minimum	16.0	
(viii) Particle size—Particle size of the material will be such that 90 per cent of the material will be between 1 mm and 4 IS sieve and not more than 5 per cent will be below 1 mm size.]		
4. N.P.K. (22 - 22 - 1)	(i) Moisture percent by weight, maximum	1.5
(ii) Total nitrogen, per cent by weight, minimum	22.0	
(iii) Ammoniacal nitrogen, per cent by weight, minimum	7.0	
(iv) Urea nitrogen, per cent by weight maximum	15.0	
(v) Neutral ammonium citrate soluble phosphates (as P ₂ O ₅) per cent by weight, minimum	22.0	
	18.7	

(vi) Water soluble phosphates (as P ₂ O ₅) per cent by weight, minimum		
(vii) Water soluble potash (as K ₂ O) per cent by weight, minimum	11.0	
(viii) Particle size - 90 per cent of the material shall be passthrough 4 mm IS sieve and be retained through 4 mm IS sieve and be retained on 1 mm IS sieve. Not more than 5 per cent shall be below 1 mm IS sieve.		
5. N.P.K. (14 - 35 - 14)	(i) Moisture per cent by weight, maximum	1.0
(ii) Nitrogen in ammoniacal form per cent. by weight. minimum'	14. [0] [Substituted by S.O. 1076(E) dated 11th December, 1987, (w.e.f 11th December, 1987 for the figures 12.0]	
(iii)[* * *] [Omitted by S.O. 1079(E), dated 11th December, 1987, (w.e.f. 11th December, 1987).]		
(iv) Neutral ammonium citrate soluble phosphates (as P ₂ O ₅) per cent by weight, minimum	35.0	
(v) Water soluble phosphates (as P ₂ O ₅) per cent by weight, minimum	29.0	
(vi) Water soluble potash (as K ₂ O) per cent by weight, minimum	14.0	
(vii) Particle size - 90 per cent of the material shall be passthrough 4 mm IS sieve and be retained on 1 mm IS sieve. Not more than 5 per cent shall be below 1 mm IS sieve.		
6. N.P.K.(17 - 17 - 17)	(i) Moisture per cent by weight, maximum	1.5
(ii) Total nitrogen, per cent by weight, minimum	17.0	
(iii) Ammoniacal nitrogen, per cent by weight, minimum	5.0	
(iv) Urea nitrogen, per cent by weight, maximum	12.0	
(v) Neutral ammonium citrate soluble phosphates (as P ₂ O ₅) per cent by weight, minimum	17.0	
(vi) Water soluble phosphates (as P ₂ O ₅) per cent by weight, minimum	14.5	
(vii) Water soluble potash as (as K ₂ O) per cent by weight, minimum	17.0	

(viii) Particle size - 90 per cent of the material shall be passthrough 4 mm IS sieve and be retained on 1 mm IS sieve. Not morethan 5 per cent shall be below 1 mm IS sieve.

7. N.P.K.(14 - 28 - 14)	(i) Moisture per cent by weight, maximum	1.5
(ii) Total nitrogen, per cent, by weight, minimum	14.0	
(iii) Ammoniacal nitrogen, per cent. by weight minimum	8.0	
(iv) Use nitrogen. per cent by weight maximum	6.0	
(v) Neutral ammonium citrate soluble phosphat (as P ₂ O ₅)per cent by weight, minimum	28.0	
(vi) Water soluble phosphate (as P ₂ O ₅)per cent. by weight, minimum	23.8	
(vii) Water soluble potash (as K ₂ O) per cent. byweight, minimum	14.0	
(viii) Particle size - 90 per cent. of the material shall bepass through 4 mm IS sieve and be retained on 1 mm IS sieve. Notmore than 5 per cent shall be below 1 mm IS sieve.		
8. N.P.K.(19 - 19 - 19)	(i) Moisture per cent. by weight, maximum	1.5
(ii) Total nitrogen, per cent. by weight, minimum	19.0	
(iii) Ammoniacal nitrogen, per cent by weight, minimum	5.6	
(iv) Urea nitrogen, per cent by weight, maximum	13.4	
(v) Neutral ammonium citrate soluble phosphates (as P ₂ O ₅)per cent by weight, minimum	19.0	
(vi) Watersoluble phosphate (as P ₂ O ₅)per cent by weight, minimum	16.2	
(vii) Water soluble potash (as K ₂ O) per cent. by weight,minimum	19.0	
(viii) Particle size - 90 per cent. of the material shall bepass through 4 mm IS sieve and be retained on 1 mm IS sieve. Notmore than 5 per cent shall be below 1 mm IS sieve.		
	(i) Moisture per cent. by weight, maximum	1.5

9. [N.P.K. (17 - 17 - 17)] [Added by S.O. 673(E), dated 25th August, 1989 w.e.f. 25th August, 1989).]

(ii) Total nitrogen, per cent. by weight, minimum 17.0

(iii) Ammoniacal nitrogen per cent by weight, minimum 8.5

(iv) Nitrate nitrogen, per cent by weight, maximum 8.5

(v) Neutral ammonium citrate soluble phosphate (as P₂O₅)per cent by weight, minimum 17.0

(vi) Water soluble phosphate (as P₂O₅)per cent. by weight, minimum 13.6

(vii) Water soluble potash (as K₂O) per cent byweight, minimum 17.0

(viii) Particle size - 80 per cent. of the material shall bepass through 4 mm IS sieve and be retained on 1 mm IS sieve. Notmore than 20 per cent shall be below 1 mm IS sieve.]

10. [N.P.K. (20 - 10 - 10)] [Added by S.O. 835(E), dated 12th October, 1995.]

(i) Moisture per cent. by weight, maximum 1.5

(ii) Total nitrogen, per cent. by weight, maximum 20.0

(iii) Urea nitrogen per cent by weight, maximum 17.1

(iv) Ammoniacal nitrogen, per cent by weight, minimum 2.9

(v) Neutral ammonium citrate soluble phosphates (as P₂O₅)per cent by weight, minimum 10.0

(vi) Water soluble phosphate (as P₂O₅)per cent by weight, minimum 8.5

(vii) Water soluble potash (as K₂O) per cent. byweight minimum 10.0

(viii) Particle size - 90% of the material shall be pass through4 mm IS sieve and be retained on 1 mm IS sieve. Not more than 5%shall be below 1 mm IS sieve] ;

(h) [NK Fertilisers] [Added by S.O. 835(E), dated 12th October, 1995.]

1. Potassium Nitrate (13 - 0 - 45)	(i) Free flowing crystalline form	
(ii) Moisture per cent by weight, maximum	0.5	
(iii) Total nitrogen (all in Nitrate form), per cent byweight, minimum	13.0	
(iv) Water soluble Potash (as K ₂ O) per cent byweight, minimum	45.0	
(v) Sodium (as Na) (or dry basis) per cent by weight, maximum	1.0	
(vi) Total chloride (as Cl) (on dry basis) per cent by weight,maximum	1.5	
(vii) Matter soluble in water per cent by weight, maximum	0.05	
2. Potassium Nitrate	(i) Free flowing crystalline form	
(ii) Moisture per cent by weight, maximum	0.5	
(iii) Total Nitrogen (all in Nitrate form), per cent byweight, minimum	13.0	
(iv) Water soluble Potash (as k ₂ O) per cent by weight, minimum	45.0	
(v) Sodium (as Na) (on dry basis) per cent by weight, maximum	1.0	
(vi)Total Chloride (as Cl) (on dry basis) per cent by weight,maximum	1.5	
(vii)Matter insoluble in water per cent by weight, maximum	0.05	
(viii)Particle size—Not less than 95% material shallpass through 2.5 mm IS sieve and shall be retained on 1 mm ISsieve.		

1. (i) P.K. Fertilisers

1. Mono - potassium Phosphate (0 - 52 - 34)	(i) Moisture per cent by weight, maximum	0.5
(ii) Water soluble Phosphate (as P ₂ O ₅)per cent by weight, minimum	52.0	
(iii)Water soluble Potash (as K ₂ O) per cent byweight, minimum	34.0	
(iv) Sodium (as NaCl) per cent by weight (on dry basis,)maximum	0.025	
(v) Particle size - 90% of the material shall pass through 0.5mm IS sieve and he retained on 0.05 mm IS sieve."		

[1(j) Potassium, Magnesium Fertilisers]

[Inserted by S.O. 329 (E), dated 12th May,

1999 (W.e.f. 12th May, 1999)]:

Potassium Magnesium Sulphate	(i) Moisture, per cent by weight, maximum	0.5%
(ii) Potash content (as K ₂ O) per cent by weight, minimum	22.0%	
(iii) Magnesium as MgO, per cent by weight, minimum	18.0%	
(iv) Total Chlorides (as Cl), per cent by weight (on dry basis), maximum	2.5%	
(v) Sodium (as NaCl), per cent by weight (on dry basis) maximum	2.0%	
(vi) Particle size : 90 per cent of the material shall passthrough 3.35 mm IS sieve and shall be retained on 1 mm IS sieve.		

1. (k) Nitrogen, Calcium Fertilizers

Calcium Nitrate	(i) Total Nitrogen per cent by weight, minimum	15.5%
(ii) Ammoniacal Nitrogen per cent by weight, maximum	1.1%	
(iii) Nitrate Nitrogen as N per cent by weight, minimum	14.4%	
(iv) Water soluble Calcium as per cent by weight, minimum	18.8%	
(v) Water insolubles per cent by weight, maximum	1.5%	
(vi) Particle size : 90% of the material shall pass through 4.00 mm sieve and be retained on 1 mm IS sieve.		

Not more than 5% shall pass through 1 mm IS sieve.]

1. (f) Micronutrients

1. [Zinc Sulphate heptahydrate (ZnSO₄·7H₂O)]

[Substituted by S.O. 534(E), dated 20th July, 1992.] (i) Free flowing crystalline form

(ii) Matter insoluble in water, per cent. by weight, maximum	1.0
(iii) Zinc (as Zn), per cent. by weight, minimum	21.0
(iv) Lead (as Pb), per cent by weight, maximum	0.003
(v) Copper (as Cu), per cent by weight,	0.1

maximum		
(vi) Magnesium (as Mg), per cent. by weight, maximum	0.5	
(vii) pH	Not less than 4.0	
2. Manganese Sulphate	(i) Free flowing crystalline form	
(ii) Matter insoluble in water, per cent. by weight, maximum	1.2	
(iii) Manganese (as Zn), content per cent. by weight, minimum	30.5	
(iv) Lead (as Pb), per cent by weight, maximum	0.003	
(v) Copper (as Cu), per cent by weight, maximum	0.1	
(vi) Magnesium (as Mg), per cent. by weight, maximum	2.0	
(vii) pH	3.75 +0.25	
3. [Borax (Sodium Tetraborate) (ii) $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$]_ for soil application.]	(i) Content of Boron (as B) per cent. by weight, minimum	10.5
[Added by G.S.R. 1160(E), dated 12st October, 1986 (w.e.f. 21st October, 1986).]		
Matter insoluble in water per cent. by weight, maximum	1.0	
(iii) pH	9.0 - 9.5	
(iv) Lead (as Pb) per cent. by weight, maximum	0.003	
4. Solubor ($\text{Na}_2\text{B}_4\text{O}_7 \cdot 5\text{H}_2\text{O}$ + $\text{Na}_2\text{B}_{10}\text{O}_{16} \cdot 10\text{H}_2\text{O}$) for foliar Sulphate	(i) Content of Boron (as B) per cent. by weight, minimum	19.0
(ii) Matter insoluble in water per cent. by weight, maximum	1.0	
(iii) Lead (as Pb) per cent. by weight, maximum	0.003	
5. Copper Sulphate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$)	(i) Copper (as Cu) per cent. by weight, minimum	24.0
(ii) Matter insoluble in water per cent. by weight, maximum	1.0	
(iii) Soluble Iron & aluminium compounds (expressed as Fe), per cent. by weight, maximum	0.5	
(iv) Lead (as Pb) per cent. by weight, maximum	0.003	
(v) pH not less than	3.0	

6. Ferrous Sulphate ($\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$)	(i) Ferrous iron (as Fe), per cent. by weight, minimum	19.0
(ii) Free Acid (as H_2SO_4) per cent. by weight maximum	1.0	
(iii) Ferric Iron (as Fe), per cent by weight maximum	0.5	
(iv) Matter insoluble in water, per cent. by weight, maximum	1.0	
(v) pH not less than	3.5	
(vi) Lead (as Pb) per cent. by weight, maximum		
7. Ammonium Molybdate ($(\text{NH}_4)_6\text{Mo}_7\text{O}_{44} \cdot \text{H}_2\text{O}$)	(i) Molybdenum (as Mo) per cent. by weight, minimum	52.0
(ii) Matter insoluble in water, per cent. by weight, maximum	1.0	
(iii) Lead (as Pb), per cent. by weight, maximum	0.003	
8. Chelated Zinc as Zn - EDTA	(i) Appearance— Free flowing crystalline/powder	
(ii) Zinc content (Expressed as Zn), per cent. by weight, minimum in the form of Zn - EDTA	12.0	
(iii) Lead (as Pb), per cent. by weight maximum	0.003	
(iv) pH	6.0 - 6.5	
9. Chelated Iron as Fe - EDTA	(i) Appearance—Free flowing crystalline/powder	
	(ii) Iron content (expressed as Fe), per cent. by weight minimum in the form of Fe - EDTA	12.0
	(iii) Lead (as Pb) per cent by weight, maximum	0.003
	(iv) pH	5.5 - 6.5
10. [Zinc Sulphate Monohydrate ($\text{ZnSO}_4 \cdot \text{H}_2\text{O}$)] [Added by S.O. 261(E), dated 16th April, 1991 (w.e.f. 16th April 1991).]	(i) [Freeflowing powder form] [Substituted by S.O. 534(E), dated 20th July, 1992.]	
(ii) Matter insoluble in water, per cent. by weight, maximum	1.0	
(iii) Zinc (as Zn), per cent. by weight minimum	33.0	
(iv) Lead (as Pb), per cent. by weight	0.003	

maximum		
(v) Copper (as Cu), per cent, by weight maximum	0.1]	
(vi) Magnesium (as Mg), per cent. by weight maximum	0.5	
(vii) Iron (as Fe), per cent. by weight, maximum	0.5	
(viii) pH not less than	4.0]	
[1] [Added by S.O. 826(E), dated 9th November, 1992.]Magnesium Sulphate (i)	Free Flowing—crystalline form	
(ii) Matter insoluble in water, per cent. by weight, maximum	1.0	
(iii) Magnesium (as Mg), per cent. by weight, minimum	9.6	
(iv) Lead (as Pb), per cent. by weight, maximum		
(v) pH (5% solution)	5.0.8.0]	
1. [(g) Fortified Fertilisers.] [Added by S.O. 140(E), dated 12th February, 1990 (w.e.f. 12th February, 1990).]		
1. Boronated Single Super Phosphate (16% P ₂ O ₅)	(i) Moisture per cent. by weight, maximum	12.0
(ii) Free phosphate acid (as P ₂ O ₅) percent. by weight, maximum	4.0	
Powdered		
	(iii) Water soluble phosphate (as P ₂ O ₅) per cent. by weight, minimum	16.0
	(iv) Boron (as B) per cent. by weight, maximum	0.18
2. Zincated Urea	(i) Moisture per cent. by weight, maximum	1.0
(ii) Total nitrogen per cent. by weight (on dry basis), minimum	43.0	
(iii) Zinc (as Zn) per cent. by weight, minimum	2.0	
(iv) Biuret per cent. by weight, maximum	1.5	
(v) Particle size - 90 per cent of the material shall passthrough 2.8 mm IS sieve and not less than 80 per cent. by weightshall be retained on 1 mm IS sieve.]		

Part B – Tolerance limit in plant nutrient for various fertilizers

1. For fertilisers with definite compound like ammonium sulphate, urea, ammonium chloride, muriate of potash, sulphate of potash, super phosphate, decalcium phosphate, which contain more than 20 per cent plant nutrients	0.2		
For those which contain less than 20 per cent plant nutrients	0.1		
2. For calcium ammonium nitrate	0.3		for individual nutrients subject to the maximum of 2.5 per cent. for all combined nutrients.
3. [0.5 units each for N and P contents] [Substituted by S.O. 444(E), dated 2nd July, 1991 (w.e.f. 2nd July, 1991).]			
4. [] [Added by G.S.R. 508(E), dated 19th 1986 (w.e.f. 19th March, 1986).] For nitrophosphates, ammonium sulphate, nitrates, urea, ammonium phosphate, ammonium phosphatesulphate, bonemeal, granulated mixture, compound/complex fertilisers [Physical mixtures of fertilisers (N.P.K. mixtures.) [Substituted by S.O. 444(E), dated 2nd July, 1991 (w.e.f. 2nd July, 1991).] [mixtures of N.P.K. with micronutrients] [Added by S.O. 354(E), dated 3rd June, 1993].]			Tolerance varies with nutrient level infertiliser, subject to the maximum of 2 per cent. for all combined nutrients :—
		Nutrient level (%)	Tolerance level (unit)
		5 or less	0.5
		16 to 20	0.6
		21 or more	1.7.
5. [For borax, chelated Zinc - EDTA and chelated iron - EDTA] [Inserted by G.S.R. 1160(E), dated 21st October, 1986 (w.e.f. 21st October, 1986).]	0.1		
6. For solubor Copper Sulphate, Zinc Sulphate, Manganese Sulphate, and Ferrous sulphate.	0.2		
7. For ammonium molybdate	0.5		
8. [For Magnesium Sulphate] [Added by S.O. 926 (E), dated 9th November, 1992.]	0.1		
9. [For mixture of micronutrien fertilisers] [Added by S.O. 354(E), dated 3rd June, 1993]			Tolerance varies with combined nutrient level infertiliser.

Nutrient level (%)	Tolerance level (unit)
10 or less	0.1
11 to 20	0.2
21 or more	0.5]

II

[See Clause 28(1)(b) and 29]

Part A – Procedure for Drawal of Samples of Fertilizers

1. General requirements of sampling - In drawing samples, the following measures and precautions should be observed :

(a)samples shall not be taken at a place exposed to rain/sun;(b)the sampling instruments shall be clean and dry when used ;(c)the material being sampled, the sampling instrument and the bags of samples should be free from any adventitious contamination ;(d)to draw a representative sample, the contents of each bag selected for sampling should be mixed as thoroughly as possible by suitable means ;(e)the sample should be kept in suitable, clean dry and air tight glass or screwed hard polythene bottle of about 400 gms. capacity or in a thick gauged polythene bag. This should be put in a cloth bag which may be sealed with the inspector's seal after putting inside the detailed description as specified in Form 'J'. Identifiable details may also be put on the cloth bag like sample No/code No. or any other details which enables its identification ;(f)each sample bag should be sealed air tight after filling and marked with details of sample type and brand of fertiliser, name of dealer/manufacturer and the name of Inspector who has collected sample.

2. Sampling from bagged material : -

(i)Scale of sampling. - (a) Lot (for manufacturers). - All bags, in single consignment of the material of the sample grade and type drawn from a single batch of the manufacture shall constitute a lot. If a consignment is declared to consist of different batches of manufacture, all the bags of each batch shall constitute a separate lot. In the case of a consignment drawn from a continuous process, 2000 bags (or 100 tonnes) of the material shall constitute a lot.(b)Lot (for dealers). - The lot is an identifiable quantity of same grade and type of fertiliser stored at an identifiable place subject to a maximum limit of 100 tonnes. The lot shall be identified by the inspector based on visible appearance of bags, their packing and storage conditions. The stock of less than 100 tonnes with a dealer may also constitute one or more lots, if the material (fertilizer) of different sources and brand is available in such quantities.(c)(i)Selection of bags for sampling. - The number of bags to be chosen from a lot shall depend upon the size of the lot as given in the table below :

Lot Size (no. of bags)(N)

No. of bags to be selected for
sampling(N)

Less than 1010 - 100100 - 200200 - 400400 - 600600 - 800800 -
10001000 - 13001300 - 16001600 - 2000 12345678910

All the bags of a lot should be arranged in a systematic manner. Start counting from any bag randomly, go on counting as 1,2,3 up to r and so on, r being equal to the integral of N/n . Thus, every rth bag counted shall be withdrawn and all bags shall constitute the sample bags from where the sample is to be drawn for preparing a composite sample.(ii)Sampling from big godown/high stackings. - If the procedure given in Para 2(i)(c) is not possible to be adopted, the sample should be drawn from the randomly selected fertiliser bags from different layers, from top and from all open sides in a zig zag fashion.(iii)Sampling from small godowns. - All the fertilizer bags of the same grade and type of each manufacturer though received on different dates shall be segregated and properly stacked. All bags of grade and type of fertiliser manufactured by particular manufacturing unit may be considered as one lot based on their physical conditions and the sample shall be drawn as per procedure laid down in Paras 2(i) (c) and 4.(iv)Sampling from damaged stock. - (a) In case of torn or lumpy bags, damaged fertiliser bags or sweepings, the stock should be arranged according to identifiable lots. From each lot the number of bags shall be selected as per procedure (2) (i) (c) if the bags allow the use of Sampling probe conveniently, the samples should be drawn sampling probe.(b)In case it is not possible to use the sampling probe, the bags may be opened and fertiliser material mixed together uniformly by hammering the big lumps or putting pressure if required, and then samples drawn by using suitable sample device.

3. Sampling probe -

(i)An appropriate sampling instrument to be used by the Inspectors for collection of a representative sample is called Sampling probe. The probe may comprise of a slotted single tube with solid conetip made of stainless steel or brass. The length of the probe may be approximately 60 to 65 cms. and the diameter of the tube may be approximately 1.5 cms. and the slot width may be 1.2 to 1.3 cms. The probe may be used if the physical condition of the fertilisers and the packing material permits its use.(ii)In case of High Density Polyethylene Packings and also when the fertiliser material is not in free flowing condition, the use of sampling probe may not be possible, in such a case, selected bags for drawing samples may be opened and the fertilizers may be taken out of the bags and spread on a clean surface and samples drawn with the help of a suitable sampling device which may be made of stainless steel or brass cup.

4. Drawal of samples from bags -

(i)Drawal of sample and preparation of composites. Draw, with an appropriate sampling instrument, (sampling probe) small portions of the material from the selected bags as per procedure in Paras 2(i)(b), 2(11), 2(iii) and 2(iv) (a). The Sampling probe shall be inserted in the bag from one corner to another diagonally and when filled with fertiliser, the probe is withdrawn and fertiliser is emptied in a container/or on polythene sheet/or on a clean hard surface and made into one composite sample.(ii)If the bags do not permit the use of sampling probe empty the contents of the

bags on a level, clean and hard surface and drawn a composite sample by the process of quartering as described under Para 3(11) or 5.(iii)[In case of chelated micronutrients and mixtures of micronutrients, the three identical containers of the batch, grade, type and manufacturer, shall be selected which shall constitute the composite samples, provided it is not possible to draw a composite sample of the size given under Paragraph 4 - A (iii)] [Added by S.O. 354(E), dated 3rd June 1993.];["4A. Weight of one sample.] [Added by S.O. 354(E), dated 3rd June 1993] -One sample of fertiliser shall have the approximate weight, as specified below : -

(i) For straight micronutrient fertiliser	100 gms
(ii) For chelated micronutrient, fertiliser and mixture of orthe maximum packing size of similar quantity.	50 gms
(iii) For other fertilisers and mixtures of fertilisers	400 gms

5. Preparation of composite sample. -

If the composite sample collected from the different selected bags is larger than [required weight] [Substituted by S.O. 354(E), dated 3rd June 1993.] its size shall be reduced by method of quartering as detailed below : -Spread the composite sample on a level, clean, hard surface, flatten it out and divide it into four equal parts. Remove any diagonally opposite parts. Mix the two remaining parts together to form a cone, flatten out the cone and repeat the operation of quartering till a composite sample of [required weight] [Substituted by S.O. 354(E), dated 3rd June 1993.] is obtained.

6. Preparation of test sample and reference sample :

(i)The composite sample obtained above shall be spread out on a clean, hard surface and divided into three approximately equal portions [each of the weight as specified in paragraph 8] [Substituted by S.O. 354(E), dated 3rd June, 1993.]. Each of these samples shall constitute the test sample.(ii)Each test sample shall be immediately transferred to a suitable container as defined under Para 1(e). The slip with detailed description may be put inside the sample bag. Each bag shall also be properly labelled as mentioned in Para 1(f).(iii)Each test sample container shall then be sealed with the seals of the inspector. If possible, seal of the manufacturer/dealer or purchaser as the case may also be affixed.(iv)One sample so sealed shall be sent to the Incharge of the Laboratory notified by the State Government under Clause 29 or Central Fertiliser Quality Control and Training Institute, Faridabad for analysis and the second given to the manufacturer or dealer or the purchaser as the case may be. The third sample shall constitute the reference sample and shall be sent by the inspector to his next higher authority for keeping in safe custody for production in Court, if required.

7. Sampling from the bulk fertilisers in ships, bulk carriers and bulk containers :

(i) Sampling equipments : - (a) Sampling cup - The sampling cup can be fabricated from non - corrosive metal. Inside dimensions of cup mouth may be $3/4" \times 10"$ as per the diagram of the sampling cup given in Figure - A below : - Fig. A - Sampling Cup (b) Sampling probe for bulk fertilisers. - The sampling probe should be made of non - corrodable material such as stainless steel or brass. It may be a slotted double tube with solid cone tip having a length of about $4 1/2" - 5"$ and diameter of about $1 1/4" - 5"$ to $1 1/2"$. (c) Scope. - Samples from the hatch can also be collected by a suitable scoop made of stainless steel or brass. (ii) Sampling Procedure: - (a) Drawal of samples from the ship of bulk fertilisers - A. Sampling from conveyor belt. - When the material is unloaded from the ships and transported to temporary/storage godowns through conveyor belts. the sample can be drawn by passing the sampling cup through the entire stream of material as the material drops from a transfer belt or spout. The long slot in the top of sampling cup should be perpendicular to the falling stream. Pass the cup through the complete stream at a uniform speed, so that the cup will collect approximately equal amounts in each pass but will never over. A minimum of 10 equally timed and speed stream must be taken during the transfer operation. However, the stream samples are not applicable unless uniform continuous flow of fertiliser is maintained for more than 3 minutes while lot is being sampled. B. Sampling from hatch. - In case of bulk fertilisers, from each hatch, at least 5 - 6 composite samples should be drawn from different depths and different points during unloading operation. To make one composite sample, minimum 5 samples should be drawn from different points at a specific depth. All these samples should be mixed to make one composite sample. The depths may be 0 - 5 m., 10 - 15 m. and 20 - 25 m. depending upon the depth of hatch. The samples at a particular depth should be drawn after removal of material up to the required depth. The samples may be drawn with the help of sampling probe or scoop. (b) Drawal of samples from bulk carriers - trucks etc. - The Samples can be drawn as per vertical probing procedure of Association of official Analytical Chemists. The sampling probe should be about $4 1/2"$ to $5"$ length. Draw 10 vertical cuts from the following locations relative of the entire top of the conveyance. The 10 vertical cores are combined into a composite samples.

1 4 3
3 1 2 5
10 6 9

(b) Drawal of samples from bulk material in storage - A. The bulk storage Piles (level or falt) up to 100 tonnes could be sampled as per figure B. Take 10 cores to the maximum. possible depth of the probe from the position indicated in the figure B and all cores are composited. B. A one sided or sloped pile may be sampled at the points illustrated in Fig. C Withdraw one vertical core of material from locations 1 and 6 and two cores at locations 2,3,4 and 5. Composite all the probe samples and prepare the composite samples for analysis as per procedure laid down in Paras 1, 5 & 6. Fig. B - Sample points for Coned or Ridged pile. Fig. C - Sample points for $1/2$ on one side Pile

8. Method of sampling of anhydrous ammonia : -

(i) Scoop. - This method is for use in obtaining samples of anhydrous ammonia. The method is based on the assumption that the material to be sampled is as claimed and contains only a small amount of impurity primarily water. It is recommended that duplicate samples be taken from each tank or

vessel sampled.(ii)Apparatus - (a) Tubes for sampling tubes, heat resistant glass, conical centrifuge type 200 ml with lower 100 ml. graduated from 0.2 ml in 0.05 ml division, 2 - 4 ml. in 0.1 division 4 - 10 ml. in 0.5 ml. divisions and 10 - 100 ml. 1.0 ml divisions. Tube type shown in figure E.(b)Stoppers for tubes, rubber, with bent tube vent as in figure E.(c)Sample carrier, constructed of plywood or aluminium as in figure E.(d)Sampling line and connection assembly constructed as in figure D, with flexible steel sampling nose 48" long, 1/4" NPT coupling at each end and 1/8" insulated steel delivery tip at one end.(e)Sample tube adapter, constructed from rubber stopper and 6 mm. OD, glass or steel tubing as in figure E.(f)Protective equipment : Rubber or other non - porous gloves, offering complete protection to the hands and lower arms, full coverage goggles or approved gas mask.Sampling Line & ConnectionFig. E(iii)Reagents. - Charcoal, regent, 14 - 20 mesh.Note. - If the sample is expected to contain excessive amount of water (one per cent or more), one piece of the charcoal may be added to each before introduction of the sample.(iv)Procedure. - (a) Place two dry, clean sampling tubes in the sample carrier.(b)Connect the sampling line connection assembly to the unloading valve of the tank, vessel or line to be sampled.(c)Open the valves slowly and purge the sampling line connection assembly thoroughly by venting 3 to 4 litres of ammonia. Close the samples line globe valve.(d)Remove the vented stoppers from the tubes and insert the adapter end of the sampling line connection assembly.(e)Open the sample line valve and slowly fill the sampling tube to the 100 ml. mark, close the sample line valve.(f)Remove the sampling line adapter and insert the vented stopper in the sampling tube.(g)Repeat steps, d and e and f and fill the second tube.(h)Close tank discharge valve and remove the sampling line connection assembly.(i)Either know or note the vessel, container, or line pressure of material sampled.(j)Tag the collected samples for identification and submit to the laboratory for processing by following methods in this section.(v)Precaution. - (a) Liquid anhydrous ammonia causes severe burns on contact. It evaporates readily releasing the gas which may cause varying degrees of irritation of the skin and mucous membranes and may injure severely the respiratory mucosa with possible fatal outcome.(b)Avoid contacting liquid ammonia. In case of contact, immediately flush the affected parts with plenty of water for at least 15 minutes. Get medical attention at once in case of burns, especially to the eyes, nose and throat, or if the victim is unconscious.(c)Ammonia gas in concentrations of 6,000 to 10,000 ppm. (by volume) is lethal within a few minutes. Irritation of the eyes, respiratory tract and throat results from concentrations as low as 500 to 1,000 ppm., a concentration of 2,000 ppm produces conclusive coughing and may be fatal after a short exposure, i.e., less than half an hour. The maximum concentration tolerated by the skin for more than few seconds is 2 per cent (i.e. when suitable respiratory protection is worn). The maximum allowable concentration for 8 hour working exposure is 50 ppm. This is the least detectable order.(d)Obtain medical attention if exposure to the gas produce distress of any type.(e)Rubber or other non - porous gloves, offering complete protection to the hands and lower arms must be worn when sampling anhydrous ammonia. Full coverage goggles must also be worn to protect the eyes unless an approved gas mask is used. The gas mask need only to be used if sampling cannot be done without possible inhalation of the vapours.

Part B – Methods of Analysis of Fertilizers

1. Preparation of sample for analysis in the laboratory :

(i) Procedure. - (a) Reduce gross sample to quantity sufficient for analysis or grand not 250 gms. of reduced sample without previous sieving. (b) For fertiliser materials and moist fertiliser mixtures, that form a paste on putting pressure, grind in porcelain pestle and mortar to pass sieve with 1 mm. circular opening or No. 20 standard sieve. (c) For dry mixtures that tend to segregate, grind in a porcelain pestle and mortar to pass No. 40 standard sieve. (d) Grind as rapidly as possible to avoid loss or gain of moisture during operation. (e) Mix thoroughly and store in tightly stoppered bottles.

2. Determination of moisture : -

(Not applicable to samples that yield volatile substances other than water at drying temperature). (i) Procedure. - (a) Weight to the nearest mg. about 2 gms. of the prepared sample in a weighed, clean, dry squat form weighing bottle. (b) Heat in an oven for about 5 hours at 99 - 101°C to constant weight. Cool in a desiccator and weight. (c) In case of sodium nitrate, ammonium sulphate and potassium salts heat to constant weight at 129 - 131°C. (d) Report percentage loss in weight as moisture at temperature used. Calculations

Free moisture percent by weight = $100 \frac{(B - C)}{B - A}$

A = Weight of the bottle. B = Weight of the bottle plus material before drying. C = Weight of the bottle plus material after drying. (Reference "Methods of Analysis", AOAC, 1965) (ii) Moisture in ammonium Chloride. - (a) Weight accurately about 5 gms. of prepared sample in a weighed shallow porcelain dish and dry for 24 hours in a vacuum desiccator over sulphuric acid and re - weight. (b) Preserve the dried material for subsequent tests. Calculations Moisture per cent by weight = $100 \times \frac{W_1}{W_3}$ W₁ = Loss in weight in gm. in drying, and W₃ = Weight in gm. of the papered sample taken for test. (iii) Samples. - Like urea, diammonium phosphate and ammonium nitrate which yield volatile substances other than water at drying temperature, the Karl Fischer method given below is used for the determination of moisture. (iv) Reagents. - (a) Iodine solution. - Added 125 gm. of iodine to a mixture of 650 ml. of methanol and 200 ml. of pyridine contained in flask, and immediately close the flask tightly. (b) Sulphur dioxide solution. - Pass dry sulphur dioxide into 100 ml. of pyridine contained in a 250 ml. graduated cylinder and cooled in an ice bath, until the volume reaches 200 ml. (c) Fischer reagent. - Slowly add iodine solution to cooled sulphur dioxide solution, stopper immediately and shake well until the iodine is dissolved. Transfer the solution to an automatic pipette, protected from absorption of moisture by a drying agent and allow to stand for 24 hours before standardising. The reagent deteriorates continuously and it should be standardized within one hour before use. (d) Standard water solutions. - Measure exactly 2 ml. of water into a thoroughly dry 1 litre volumetric flask, dilute to volume with methanol. Retain sufficient quantity of the same methanol for the blank determination. Keep the solution in tightly closed containers. (v) Procedure. - (a) Determination of end point in Karl Fischer titration. - In many cases the end point can be detected by the change of colour from a light brownish yellow to amber. But when the end point is not clearly defined, the electrometric method for determining the end point should be adopted. Adjust the potentiometer so that when a small excess (0.02 ml) of the reagent is present, a current of 50 to 150 microampers is recorded : The solution should be continuously and vigorously stirred. At the beginning of the titration, a current of only a few microampers will flow.

After each addition of reagent, the pointer of the micro-meter deflected but rapidly returns to the original position. At the end point a deflection is obtained which endures for a longer period.(b)Standardization of the fischer reagent. - Pipette exactly 10 ml. of methanol into a dry titration flask and titrate with the Fischer reagent to the end point (V₁). Pipette exactly 10 ml. of the standard water solution into the flask and titrate to the end point (V₂). (c)Titration of the material. - Transfer 25 ml. of methanol to the titration flask and titrate to the end point (V₃) with the Fischer reagent. Do not record the volume consumed. Quickly transfer to the titrated liquid an accurately weighed quantity of the material containing 10 to 50 mg. of water, stir vigorously and titrate to the end point. Calculation

$$\text{Moisture per cent by weight} = 0.1 \times W(V_3 - 2.5V_1) \frac{(V_2 - V_1) A}{\text{Weight in mg. of water contained in 10 ml. of standard water solution}}$$

where, W = Weight in mg. of water contained in 10 ml. of standard water solution V₃ = Total volume in ml. of the reagent used in titration in (c). V₁ = Volume in ml. of reagent used in titration of methanol in (b), and V₂ = Total volume in ml. of the reagent used in titration in (b). A = Weight in gm. of the material taken for the test in (c). (Reference IS. Specification for urea, technical and pure. IS. 1781 - 1961)

3. Determination of nitrogen :

Methods of determination of total nitrogen, ammoniacal nitrogen, nitrate nitrogen and urea nitrogen have been described separately in this section. These methods can be adopted both for straight and mixed fertilisers. Scope of each method with various combinations have also been described with each method. The relevant method of analysis which have been described are as given below : (a) Total Nitrogen - for nitrate free samples. (b) Total Nitrogen - for nitrate containing samples. (c) Total Nitrogen - for materials with high CL : NO₂ ratio and to materials containing only water soluble nitrogen. (d) Determination of ammoniacal nitrogen. (e) Determination of ammoniacal and nitrate nitrogen. (f) Determination of nitrate nitrogen. (g) Determination of water insoluble nitrogen. (h) Determination of urea nitrogen. (i) Detection of nitrate. - For adopting a specific method as described above, it is necessary to detect the presence of nitrates in the sample before a particular method is adopted. The procedure for detection of nitrates is given below : (a) Mix. 5 gm. sample with 25 ml. hot water and filter. (b) To one volume of this solution add 2 volumes of sulphuric acid, free from HNO₃ and oxides of N, and let it cool. (c) Add a few drops of concentrated FeSO₄ solution in such a manner that fluids do not mix. (d) If the nitrates are present junction shows at first purple, afterward brown, or if only minute quantity is present. reddish colour. (e) To another portion of solution add 1 ml. 1 per cent NaNO₃ and test as before to determine whether enough H₂SO₄ was added in the first test. (Reference "Methods of Analysis" AOAC, 1965) (ii) Reagents for determination of total nitrogen (a) Sulphuric acid - 93 - 98 per cent H₂SO₄, N free. (b) Copper sulphate - CuSO₄·5H₂O reagent grade, N free. (c) Potassium sulphate (or anhydrous sodium sulphate) - reagent grade. (d) Salicylic acid - reagent grade, N free. (e) Sulphide or thiousulphate solution - Dissolve 40 gm. commercial K₂S in 1 litre distilled water. (Solution - of 40 gm. Na₂S or 80 gm. Na₂S or 80 gm. Na₂S₂O₃·5H₂O in 1 litre may be used.) (f) Sodium hydroxide - Pellets or solution, nitrate free. For solution dissolve approximately 450 gms. solid NaOH in distilled water and dilute to 1 litre (Sp. gr. of solution should be 1.36 or higher). (g) Zinc granule - reagent grade. (h) Zinc dust - Impalpable powder. (i) Methyl red indicator - Dissolve 1 gm. methyl red in 200 ml. alcohol. (j) Hydrochloric or sulphuric acid standard solution. - 0.5 N or 0.1N when amount of N is small. (k) Sodium hydroxide

standard solution - 0.1N (or other specified concentration)(1)Standardize each standard solution with primary standard and check one against another.(2)Test reagents before using, by blank determination with 2 gms. sugar which ensures partial reduction of any nitrates present.Caution : Use freshly opened sulphuric acid or add dry P_2O_5 to avoid hydrolysis of nitrites and cyanates. Ratio of salt to acid (wt : Vol) should be approximately 1 : 1 : at end of digestion for proper temperature control. Digestion may be incomplete at lower ratio, N may be lost at higher ratio.(iii)Apparatus :(a)For digestion - Use Kjeldahl flask of hard moderately thick, well annealed glass with total capacity approximately 500 - 800 ml. Conduct digestion over heating device, adjusted to bring 250 ml. water at $25^\circ C$ to cooling boil in approximately for 5 minutes. Add 3 - 4 rolling chips to prevent superheating.(b)For distillation - Use Kjeldahl or other suitable flask of 500 - 800 ml. capacity filled with rubber stopper through which passes lower end of efficient scrubber bulb or trap to prevent mechanical carryover of NaOH during distillation. Connect upper end of bulb tube to condenser tube by rubber tubing. Trap outlet of condenser in such a way as to ensure complete absorption of ammonia distilled over into acid in receiver.(iv)Total nitrogen (in nitrate free samples) :(a)Procedure -(1)Place weighed sample (0.7 - 2.2 gm.) in digestion flask.(2)Add 0.7 gm. copper sulphate, 15 gm. powdered K_2SO_4 or anhydrous Na_2SO_4 and 25 ml. H_2SO_4 .(3)If sample more than 2.2 gm. is used, increase sulphuric acid by 10 ml. for each gm. sample.(4)Place flask in inclined position and heat gently until frothing ceases (if necessary add small amount of paraffin to reduce frothing).(5)Boil briskly until solution clears and then for at least 30 minutes longer (2 hours for sample containing organic material)(6)Cool add approximately 200 ml. distilled water, cool below $25^\circ C$.(7)Add to the flask a layer of sodium hydroxide (25 gm. solid reagent or enough solution to make contents strongly alkaline) without agitation.(8)Immediately connect flask to distilling bulk or condenser and with tip of condenser immerse in standard acid in receiver.(9)Rotate flask to mix contents thoroughly, then heat until all ammonia has distilled (at least 150 ml. distillate)(10)Titrate excess standard acid in distillate with standard sodium hydroxide solution, using methyl red as an indicator.(11)Correct for blank determination on reagents.Calculations :

$$\text{Per cent Nitrogen} = (A_{Na} - B_{Nb}) \times 0.01401 \times 100 \frac{W}{\quad}$$

A = ml. of standard acid used. B = ml. of standard NaOH used. Na = Normality of standard acid. Nb = Normality of standard NaOH. weight of the sample taken in grams. (Reference "Methods of Analysis", A.O.A.C., 1965).(v)Total Nitrogen (for nitrate containing samples) :(Not applicable to samples containing high concentrations of nitrate nitrogen and chlorides)(a)Procedure -(1)Place weighed sample (0.7 - 2.2 gm.) in digestion flask.(2)Add 40 ml. H_2SO_4 containing 2 grams salicylic acid. Shake until thoroughly mixed and let stand, with occasional shaking, 30 minutes or more.(3)Then add (i) 5 grams $Na_2S_2O_3 \cdot 5H_2O$ or (ii) 2 grams zinc dust (as impalpable powder not granulated zinc or filings).(4)Shake the flask and let it stand for five minutes, then heat over low flame until frothing ceases.(5)Turn off heat, add 0.7 grams copper sulphate, 15 gm. powdered K_2SO_4 (or anhydrous Na_2SO_4), and boil briskly until solution clears, then at least 30 minutes longer (2 hours for samples containing organic material). Proceed further as in 6 - 11 of 3(iv). Calculations :

$$\text{Per cent Nitrogen} = (A_{Na} - B_{Nb}) \times 0.01401 \times 100 \frac{W}{\quad}$$

A = ml of standard acid used. B = ml. of standard NaOH used. Na = Normality of standard acid. Nb = Normality of standard NaOH. weight of the sample taken in grams. (Reference "Methods of

Analysis", A.O.A.C., 1965).(vi)Total nitrogen (for materials with high CI : NO₃ ratio and to materials containing only water soluble nitrogen).(a)Reagents :Reduced iron powder, electrically reduced, N.F. (National Formulary). For other reagents see 3(ii).(b)Procedure -Mixed Fertilisers :(1)Place 0.5 - 2.0 gm. sample in Kjeldahl flask and add 2.5 grams reduced Fe (5 gms. is enough for 0.185 grams. NO₃). (2)Add approximately 25 ml. distilled water, rotating flask at angle to wash down the sample.(3)Let it stand for 15 minutes with occasional agitation, to ensure complete solution of all soluble salts.(4)While rotating flask add 25 ml. cold H₂SO₄ (1 + 1) and let it stand until visible reaction ceases (use hood or vented digestion unit).(5)Add boiling chip and boil 15 - 20 minutes, but do not take to dryness. (For samples containing organic matter, use 50 ml. cold H₂SO₄ (1 + 1), boil for 15-20 minutes, add 0.7 gm. HgO add heat again for 30 minutes). Cool and proceed further as in (6) - (11) of 3(iv).Calculations :

Per cent Nitrogen = $(A_{Na} - B_{Nb}) \times 0.01401 \times 100 \frac{W}{A}$
 A = ml. of standard acid used. B = ml. of standard NaOH used. Na = Normality of standard acid. Nb = Normality of standard NaOH. weight of the sample taken in grams. (Reference "Methods of Analysis", A.O.A.C., 1965).(vii)Determination of ammoniacal nitrogen (Distillation Method).(This method is for the determination of nitrogen present or available in the samples as ammonium ion. This method assumes that Urea is absent from the sample. Not applicable to MgNH₄PO₄ and Fe NH₄PO₄).(a)Procedure -(1)Place 0.7 - 3.5 grams; according to NH₃ content of the sample, in distillation flask with approximately 300 ml. water and 2 grams of freshly ignited carbonate free MgO or NaOH solution.(2)Connect the flask to condenser by Kjeldahl connecting bulb.(3)Distill 100 ml. liquid into measured quantity of standard acid and titrate with standard NaOH solution, using methyl red as an indicator.Calculations :

Per cent Nitrogen = $(A_{Na} - B_{Nb}) \times 0.01401 \times 100 \frac{W}{A}$
 A = ml. of standard acid used. B = ml. of standard NaOH. Na = Normality of acid. Nb = Normality of NaOH. W = weight of the sample taken in gms. (Reference - Recommended analytical methods of the National Plant Food Institute, Washington, D.C., 1961).(viii)Determination of ammoniacal and nitrate nitrogen (Devarda Method)(This method is for the determination of total nitrogen when only nitrate or mixture of nitrate and ammoniacal is present. This method assumes the absence of Urea, Calcium Cyanamide and organic matter from the sample).(a)Procedure -(1)Place 0.35 - 0.5 gms. samples in 600 - 700 ml. flask and add 300 ml. water, 3 grams Devarda Alloy, and 5 ml. NaOH solution (42 per cent by weight pouring later down side of flask so that it does not mix at once with contents).(2)Allow the flask to stand for 15 minutes.(3)By means of Division (J. Ind. Eng. Chem. 11,465 (1919) or other suitable scrubbing bulb that will prevent passing over of any spray connect with condenser, tip of which always extends beneath surface of standard acid in receiving flask.(4)Mix contents of distilling flask by rotating.(5)Heat slowly at first, and then at a rate to yield 250ml. distillate in 1 hour.(6)Collect distillate in measured quantity of standard acid and titrate with standard NaOH solution, using methyl red as an indicator.(7)In analysis of nitrate salts dissolve 3.5 or 5.0 grams in water, to 250 ml. and use 25 ml. aliquot.Calculations :

Per cent Nitrogen = $(A_{Na} - B_{Nb}) \times 0.01401 \times 100 \frac{W}{A}$
 A = ml. of standard acid used. B = ml. of standard NaOH used. Na = Normality of standard acid. Nb = Normality of standard NaOH. weight of the sample taken in grams. (Reference "Methods of Analysis", A.O.A.C., 1965).(ix)Determination of nitrate nitrogen(Applicable in presence of calcium cyanamide and urea in the mixture).(a)Procedure -(1)Determine total nitrogen as in 3(v).(2)Determine water insoluble N as in 3(x) but use 2.5 grams sample. Dilute to 250

ml.(3)Determine ammoniacal N in 50 ml. filtrate as in 3(vii).(4)Place another 50 ml. portion filtrate in 500 ml. Kjeldahl flask and 2 grams. $\text{Fe SO}_4 \cdot 7\text{H}_2\text{O}$ and 20 ml. H_2SO_4 . (If total N is 5 per cent use 5 gm. $\text{Fe SO}_4 \cdot 7\text{H}_2\text{O}$). Digest over hot flame until all water is evaporated and white fumes appear and continue digestion at least ten minutes to drive off nitrate N. If severe bumping occurs, add 10 - 15 glass beads. Add 0.65 grams Hg or 0.7 grams HgO and digest until all organic matter is oxidized cool, dilute add the K_2S solution and complete determination as 3(iv). Before distillation add a pinch of mixture of Zinc dust and granular "20 mesh", Zinc to each flask to prevent bumping. Calculations : Total N (a) - Water insoluble N(b) - Water soluble N. Water Sol N - N obtained in (d) Nitrate N.(Reference Methods of Analysis A.O.A.C. 1965).(x)Determination of water insoluble nitrogen(a)Procedure -(1)Place 1 or 1.4 grams sample in 50 ml. beaker, wet with alcohol.(2)Add 20 ml. water and let it stand for 15 minutes stirring occasionally.(3)Transfer supernatant liquid to 11 cm. Whatman No. 42 paper in 60° long stem funnel 2.5" diameter and wash residue 4 to 5 times by decanting with water at room temperature (20 - 25°C).(4)Finally, transfer all residue to filter and complete washing until filtrate measures 250 ml.(5)Determine N as in 3(iv).(xi)Determination of Urea nitrogen(This method is for the determination of urea content of any mixed fertilisers).(a)Reagents -(1)Neutral urease solution - shake 1 gm. jack bone meal with 100 ml, water for 5 minutes. Transfer 10 ml. solution to 250 ml. (Erlenmeyer flask, dilute with 50 ml. water and add 4 drops methyl purple indicator. Titrate with 0.1 N HCL to reddish purple, then back titrate to green with 0.1 N NaOH. From difference in ml., calculate amount of 0.1 N HCL required to neutralize remainder of solution (usually approximately 2.5 ml. per 100 ml.) add this amount of acid and shake well.(b)Procedure -(1)Weight 10 ± 0.01 gm. sample and transfer to 15 cm. Whatman No. 12 fluted filter paper.(2)Leach with approximately 300 ml. water into 500 ml. volumetric flask.(3)Add 75 - 100 ml. saturated barium hydroxide solution to precipitate phosphates.(4)Let it settle and test for complete precipitation with few drops of saturated barium hydroxide solution.(5)Add 20 ml. 10 per cent sodium carbonate solution to precipitate excess barium and any soluble calcium salts.(6)Let it settle and test for complete precipitation.(7)Dilute to volume, mix and filter through 15 cm. Whatman No. 12 fluted paper.(8)Transfer 50 ml. aliquot (equivalent to 1 gm. sample) to 200 or 250 ml. Erlenmeyer flask and add 1 to 2 drops methyl 1 purple indicator.(9)Acidify solution with 2NHC 1 and add 2 to 3 dropse excess.(10)Neutralize solution with 0.1 N NaOH to first change in colour indicator.(11)Add 20 ml. neutral urease solution, close flask with rubber stopper and let it stand for 1 hour at 20 - 25°C.(12)Cool the flask in ice water slurry and titrate at once with 0.1 N HCl to full purple colour, then add approximately 5 ml. excess.(13)Record total volume added, back titrate excess HCl with 0.1 N NaOH to neutral end point. Calculations: Percentage urea = $(\text{ml. 0.1 N HCl} - \text{ml. 0.1 N NaOH}) \times 0.3003 \text{ wt. of sample}$ (Reference "Methods of Analysis" A.O.A.C., 1965)(xii)Determination of biuret(a)Reagents :(1)Alkaline tartarate solution - Dissolve 40 gm. NaOH in 50 ml. water cool, add 50 gm. of $\text{NaKC}_4\text{H}_4\text{O}_6 \cdot \text{H}_2\text{O}$ and dilute to 1 litre. Let it stand for one day before use.(2)Copper sulphate solution - Dissolve 15 gm. $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ in CO_2 free water and dilute to 1 litre.(3)Biuret standard solution - 1 mg./ml. Dissolve 100 mg. reagent grade biuret in CO_2 free water and dilute to 100 ml.(4)Ion exchange resin. - Fill 50 ml. burette with 30 cm. column of Amberlite IR 120 (H) resin on glass wool plug. Regenerate column after each use by passing 100 ml. H_2SO_4 (1 + 9) or HCl (1+4) through the column approximately 5 ml. per minute and then washing with water until pH of effluent is 6:0.(b)Preparation of standard curve :(1)Transfer series of aliquots, 2 - 50 ml. of standard biuret solution to 100 ml. volumetric flask.(2)Adjust volume to approximately 50 ml. with CO_2 free water. Add one drop of methyl red and neutralize with 0.1 N

H₂SO₄ to pink colour.(3)Add with swirling 20 ml. alkaline tartarate solution and then 20 ml. CuSO₄ solution.(4)Dilute to volume shake for 10 seconds and place in water bath for 15 minutes at 30±5°C.(5)Also prepare reagent blank.(6)Determine absorbance of each solution against blank at 555 mw (instrument with 500 - 750 mw filter is also satisfactory) with 2 - 4 cm. cell, and plot standard curve.(c)Procedure -A. In urea(1)Stir continuously 2 - 5 gm. sample in 100 ml. approximately 50°C water for 30 minutes.(2)Filter and wash into 250 ml. volumetric flask and dilute to volume.(3)Transfer 25 ml. aliquot to 100 ml. volumetric flask and proceed as given under preparation of standard curve 3(xii).B. In mixed fertilisers -(1)Stir continuously 10 - 20 gm. sample in 150 ml. approximately (50°C) hot water for 30 minutes.(2)Filter and wash into 250 ml. volumetric flask and dilute to volume.(3)Transfer 25 ml. aliquot to column 3(xii) (a) (4) and adjust flow to 4 - 5 ml./ minute.(4)Receive eluate in 100 ml. beaker.(5)When liquid level falls to top of resin bed, wash with two 25 ml. portion water.(6)To eluate and washings add two drops of methyl red and then 0.1 N NaOH to yellow colour.(7)Add 0.1 NH₂SO₄ until solution just turns pink.(8)Transfer to 100 ml. volumetric flask and dilute to volume with CO₂ free water.(9)Transfer 50 ml. aliquot to 100 ml. volumetric flask and proceed as in preparation of standard curve given above.Calculations :From standard curve determine concentration of biuret in final dilution, then per cent ofbiuret = $Cx 100 \frac{\quad}{W}$ Where C = Concentration in mg/ml. of biuret in final dilution obtained from standard curve.W = Concentration of original sample in final dilution expressed as mg/ml. (Reference "methods of Analysis "A.O.A.C., 1965)(xiii)Determination of free acidity in ammonium sulphate (as H₂SO₄)(a)Reagents :(1)Standard sodium hydroxide solution - 0.02 N.(2)Methyl red indicator - Dissolve 0.15 gm. of water soluble methyl red in 500 ml. water :(3)Methyl red - Methyl blue mixed indicator solution - prepared by mixing equal volumes of 0.2 per cent solution in rectified spirit of methyl red and 0.1 per cent solution in rectified spirit of methylene blue.(b)Procedure -(1)Dissolve about 20 gm. of prepared sample, accurately weighed in about 50 ml. cold natural water.(2)Filter and make up the volume to about 200 ml.(3)Titrate with standard sodium hydroxide solution, using one or two drops of methyl red as indicator.(4)If satisfactory end point with methyl red is not obtained, methylene red - methylene blue mixed indicator may be used.(5)Use preferably a micro biuret for this titration.The filtering medium shall be neutral and shall not contain any alkaline material which would neutralize free acid.Calculations :

$$\text{Free acidity as H}_2\text{SO}_4 \text{ per cent by weight} = \frac{4.904}{AN} W$$

A = Volume of ml. of standard NaOH solution.N = Normality of standard NaOH solution.W = Weight in gm. of prepared sample taken for the test.(Reference - Specifications for ammonium sulphate, fertilizer grade IS : 826 - 1967)(xiv)Determination of Arsenic in Ammonium Sulphate (A₂O₃)(a)Reagents :(1)Lead acetate solution - Prepare 10 per cent solution of lead acetate with sufficient acetic acid added to clear the solution.(2)Dry lead acetate paper - Cut filter paper (Whatman No. 1 or equivalent) into strips 70 x 50 mm and keep them permanently suspended in lead acetate solution in a glass stoppered bottle. Before use, take out the strips and dry them in an atmosphere free from hydrogen sulphide.(3)Mercuric Bromide solution - Dissolve 5 gm. of mercuric bromide in 100 ml. rectified spirit.(4)Sensitized Mercuric Bromide paper strips - cut filter paper (Whatman No. 1 or equivalent) into strips 120 x 2.5 mm. Keep the strips permanently suspended in dark in a glass stoppered cylinder or amber bottle having mercuric bromide solution. Before use take out a strip, press it between sheets of filter paper and dry it in an atmosphere free from hydrogen sulphide.(5)Dilute sulphuric acid - approximately 5 N.(6)Concentrated hydrochloric

acid.(7)Potassium iodide solution - 15 per cent.(8)Stannous chloride solution - Dissolve 80 gm. of stannous chloride in 100 ml. water containing 5 ml. of concentrated hydrochloric acid. If the solution is turbid add a few more ml. of hydrochloric acid and boil until clear solution is obtained. Add some metallic tin to the solution to prevent oxidation.(9)Zinc - It is recommended that zinc rods prepared as described below should be used for routine work, however, pellets described may be used.(b)Preparation of zinc rods -(1)Take a clean and dry hard glass test tube of 10 mm. internal diameter and 20 cm. length.(2)Heat the test tube over a flame of Bunsen or blow pipe burner and add slowly arsenic free granulated zinc in small portions (1 to 2 gm. at a time) the next portion being added after the first one has completely melted.(3)Continue heating and adding zinc until the melt is about 10 cm. high.(4)Heat the clean melt for half an hour and then cool to room temperature.(5)Break the tube to obtain the rod of zinc.(6)Cut rod into pieces 20 mm. long.(7)Coat the plane ends of the pieces with a paste of magnesium carbonate and gum arabic solution and dry.(8)Coat the pieces all over with 1.5 mm. thick layer of paraffin wax.(9)When required for use, scrap off the wax from the plane ends with a knife. protecting wax colour round the rods.(10)Remove the past from the plane ends by soaking in water and activate the exposed surface by dipping in a solution containing one part of stannous chloride solution and seven parts of concentrated hydrochloric acid.(c)Preparation of zinc pellets -Treat zinc shots passing through IS sieve 570 (aperture 5600 microns) and retained on IS sieve 280 (aperture 2818 microns) with concentrated hydrochloric acid until the surface of zinc becomes clean and dull. Weight and keep under water, preventing contamination with dust.(d)Standard sodium hydroxide solution - approximately 20 per cent.(e)Standard arsenic trioxide solution.(1)Dissolve 1.0 gm. of resublimed arsenic trioxide (As_2O_3) in 25 ml. sodium hydroxide solution and neutralize with dilute sulphuric acid.(2)Dilute with freshly distilled water containing 10 ml. of concentrated sulphuric acid per litre and make up the volume to 1 litre.(3)Again dilute 10 ml. of this solution to 1 litre with water containing sulphuric acid and finally dilute 100 ml. of this solution to 1 litre with water containing sulphuric acid. 'One ml. of this solution contains 0.001 mg. of arsenic trioxide (As_2O_3). The dilute solution shall be prepared freshly when required.(f)Procedure -(1)Dissolve 1.0 gm. of the prepared sample in 20 ml. water.(2)Place dry lead acetate paper in the lower portion of the tube B (in figure 6) and glass wool moistened with lead acetate solution in its proper portion.(3)Place the sensitized strips of mercuric bromide paper in tube A and connect the tubes together with a rubber stopper.(4)Introduce the solution of the material into the bottle C (120 ml.) and then add 10 ml. of dilute sulphuric acid. Add 0.5 ml. of stannous chloride solution, 5 ml. potassium iodide solution and make up the volume with water to about 50 ml.(5)Mix the contents and drop about 10 gm. of zinc. Immediately fit in position the rubber stopper carrying the tube B.(6)Place the bottle in a warm place at about 40°C.(7)At the end of two hours remove the test strip by means of tweezers.(8)Carrying out the test prescribed above using a volume of standard arsenic trioxide solution containing 0.1 mg. of arsenic trioxide, in place of the solution of the material and compare the strain produced with the material with that produced with arsenic trioxide solution.(9)The limit prescribed in the material specification shall be taken as not having been exceeded if the length of the strain as well as the intensity of its colour produced in the test with the material is not greater than those produced with arsenic trioxide solution.(Reference "Modified Gutzet method of test for arsenic" IS - 2088 - 1962)(xv)Determination of calcium nitrate (for calcium ammonium nitrate and nitrophosphates)(a)Reagents :(1)N = Amyl Alcohol(2)Dilute hydrochloric acid - approximately 4 N :(3)Standard calcium solution - weight 1.0 gm. of calcium carbonate dried at $120 \pm 5^\circ C$ and dissolve

in the minimum quantity of dilute hydrochloric acid. Dilute the solution to 1 litre in a graduated flask. (4) Ammonium chloride - hydroxide buffer solution. Dissolve 67.5 gm. ammonium chloride in a mixture of 570 ml. of ammonium hydroxide (sp. gr. 0.92) and 250 ml. water. Also dissolve separately a mixture of 0.931 gm. of disodium ethylene diamine tetra - acetate dihydrate and 0.616 gm. of magnesium sulphate ($\text{Mg SO}_4 \cdot 7\text{H}_2\text{O}$) in about 50 ml. of water. Mix the two solutions and dilute to 1 litre. (5) Standard disodium ethylene diamine tetra - acetate (EDTA) solution - weigh 3.72 gm. of disodium ethylene diamine tetra - acetate dihydrate in water, and dilute in a graduated flask to 1 litre. The solution shall be standardized frequently against standard calcium solution following the procedure given below. (6) Eriochrome Black T Indicator solution - Dissolve 0.1 gm in 20 ml. of rectified spirit. The solution shall be used for not more than a week. (b) Procedure - (1) Grind quickly about 5 gm of the material, accurately weighed, with about 50 ml. of amyl alcohol in a pestle and mortar and transfer the contents to a conical flask. (2) Wash the pestle and mortar with a few ml. of amyl alcohol and add the washings to the flask. (3) Shake the contents of the flask manually or in a mechanical shaker for about half an hour and then filter. (4) Transfer the filtrate to a separating funnel and extract calcium nitrate completely with water in five to six installments. (5) A few drops of dilute hydrochloric acid may be added during the extraction with water to avoid formation of an emulsion of amyl alcohol with water. (6) Concentrate the water extract at low temperature to nearly half its volume. (7) Transfer the concentrated solution to a conical flask, add 5 ml. of ammonium chloride - ammonium hydroxide buffer solution, 5 drops of eriochrome black T indicator solution and titrate against standard EDTA solution to a pure blue end point. Calculations :

Calcium nitrate per cent by weight = $8.2 \text{ NV} \frac{W}{V}$

where N = Normality of standard EDTA solution. V = Volume in ml. of standard EDTA solution used in the titration, and W = Weight in gm. of the material taken for test. (Reference : IS specification of CAN IS : 2409 - 1963) (xvi) Determination of Chlorides other than ammonium chloride (a) Reagents

(1) Standard silver nitrate solution - 0.1 N. (2) Concentrated nitric acid - conforming to IS : 264 - 1950. (3) Ferrous ammonium sulphate solution saturated in water and stabilized by addition of 50 ml. nitric acid. (4) Standard Ammonium Thiocyanate solution - 0.1 N. (b) Procedure - (1) Dissolve about 0.2 gm. of the prepared sample, previously dried as in procedure for determination of moisture and accurately weighed, in about 40 ml. water. (2) Add exactly 50 ml. of standard silver nitrate solution and 5 ml. of concentrated nitric acid. (3) Add 0.5 ml. of nitrobenzene and make up the volume of the mixture to 100 ml. with water. (4) Take exactly 50 ml. of the solution and add 2 ml. of ferric ammonium sulphate solution. (5) Titrate the excess of silver nitrate in this portion with standard ammonium thiocyanate solution. (6) Carry out a blank test following the procedure given as above but without using the material. Calculations :

Total Chlorides (as Cl) per cent by weight (on dry basis) = $\frac{7.07 (V_1 - V_2) N}{W} \times 100$

where V_1 = Volume in ml. of standard ammonium thiocyanate used in the blank determination. V_2 = Volume in ml. of standard ammonium thiocyanate solution used in the test with the material. N = Normality of standard ammonium thiocyanate solution. W = Weight in gm. of the derived prepared sample taken for the test. Express the ammoniacal nitric content per cent by weight of the material determined earlier in terms of Cl as follows : Chloride equivalent of the ammoniacal nitric content, per cent by weight = $2.531 \times A$ where A is the ammoniacal nitric content determined earlier. Balanced chloride equivalent to sodium chloride (NaCl) per cent by weight = $1.648 \times (X - Y)$ = Balanced chloride other than ammonium chloride. (Reference IS specification for ammonium

chloride fertiliser grade (Revised) IS : 1114 - 1964 calculations notified).

4. Determination of phosphates -

Methods of determination of total phosphates, water soluble phosphates, citrate soluble phosphates, citrate insoluble phosphates and citric acid soluble phosphates have been described separately in this section. These methods are applicable to straight as well as mixed phosphatic fertilisers.

(i) Preparation of solution of the samples (Separate methods of preparing solutions of the samples have been described according to the nature of the sample).

(a) Reagent : Magnesium nitrate solution - Dissolve 950 gm. P - free $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ in water and dilute to 1 litre.

(b) Procedure - (1) Treat 1 gm. sample by (A), (B), (C), (D) or (E), method depending on the nature of the sample. (2) Cool solution transfer to 200 to 250 ml. volumetric flask, dilute to volume, mix and filter through dry filter.

A. (Suitable for materials containing small quantities of organic matter). Dissolve in 30 ml. HNO_3 and 3 - 5 ml. HCl and boil until organic matter is destroyed.

B. (Suitable for fertilisers containing much Fe or Al phosphate and basic slag). Dissolve in 15 - 30 ml. HCl and 3 - 10 ml. HNO_3 .

C. (Suitable for organic material like cotton seed meal alone or in mixture). Evaporate with 5 ml. of the $\text{Mg}(\text{NO}_3)_2$ solution, ignite and dissolve in HCl .

D. (Generally applicable to materials or mixtures containing large quantities of organic matter). Boil with 20 - 30 ml. H_2SO_4 in 200 ml. flask adding 2 - 4 gm. of NaNO_3 or KNO_3 at beginning of digestion and small quantity after solution is nearly colourless, or adding the nitric acid in small portions from time to time. When solution is colourless, cool add 150 ml. water boil for few minutes. Before adding NaNO_3 or KNO_3 let mixture digest, at gentle heat if necessary, until violence of reaction is over.

E. (Suitable for all fertiliser). Boil gently for 30 - 45 minutes with 20 - 30 ml. HNO_3 in a suitable flask (Preferably a Kjeldahl for samples containing large quantities of organic matter to oxidize all easily oxidizable matter). Cool and add 10 - 20 ml. of 70-72 per cent perchloric acid. Boil very gently until solution is colourless or nearly so and white dense fumes appear in flask. Do not boil to dryness at any time. (Danger). (With samples containing large quantities organic matter temperature should be raised to fuming point approximately 170°C , cover a period of 1 hour at least). Cool slightly, add 50 ml. water and boil for few minutes.

(ii) Gravimetric Quinoline, Molybdate method for determination of total phosphorus

(a) Reagents : (1) Citric molybdic acid reagent - Dissolve 54 gms. 100 per cent molybdic anhydride (MgO_3) and 12 gms. NaOH with stirring in 400 ml. hot water and cool. Dissolve 60 gms. citric acid in mixture of 140 ml. HCl and 300 ml. water and cool. Gradually add molybdic solution to citric acid solution with stirring. Cool, filter and dilute to 1 litre. (Solution may be green or blue, colour depends on exposure to light). If necessary add 0.5 per cent KBrO_3 solution drop by drop until green colour becomes pale. Store in dark in polyethylene bottle.

(2) Quinoline solution - Dissolve 50 ml. synthetic quinoline with stirring in mixture of 60 ml. HCl and 300 ml. water. Cool dilute to 1 litre, and filter. Store in polyethylene bottle.

(3) Quimociac reagent - Dissolve 70 gms. of sodium molybdate dihydrate in 150 ml. water. Dissolve 60 gms. citric acid in mixture of 85 ml. HNO_3 and 150 ml. water and cool. Gradually add molybdate solution to citric acid - nitric acid mixture with stirring. Dissolve 5 ml. synthetic quinoline in mixture of 35 ml. HNO_3 and 100 ml. water. Gradually add this solution to molybdate citric - nitric acid solution mix and let it stand for 24 hours. Filter, add, 28 ml. acetone, dilute to 1 litre with water and mix well. Store in polyethylene bottle.

(b) Procedure - (1) Treat 1 gm. sample as prescribed in 4(i) and dilute it to 200 ml. (2) Pipette into 500 ml. Erlenmeyer flask, aliquot containing not more than 25 mg. P_2O_5 dilute to

approximately 100 ml. with water. Proceed with one of the following methods. A. Add 30 ml. citric molybdic acid reagent and boil gently for 3 minutes. (Solution must be precipitate free at this stage). Remove from heat and swirl carefully. Immediately add from burette 10 ml. quinoline solution with continuous swirling (Add first 3 - 4 ml. dropwise and remainder in steady stream) or B. Add 50 ml. quimociac reagent, cover with watch glass place on hot plate in well ventilated hood, and boil for a minute. After treatment with A or B cool to room temperature, swirl carefully 3 - 4 times during cooling, filter into gooch with glass fibre filter paper previously dried at 250°C and weighed. and wash five times with 25 ml. portion of water. Dry crucible and contents for 30 minutes at 250°C. Cool in desicator to constant weight as $(C_9H_7N)_3 H_3PO_4 \cdot 12MoO_3$. Subtract weight reagent blank. Multiply by 0.03207 to obtain weight of P_2O_5 . Report as per cent P_2O_5 .

(iii) Determination of water soluble phosphorus (a) Procedure - (1) Place 1 gm. sample on 9 cms. filter paper and wash with small portion of water until filtrate measure approximately 250 ml. (2) Let each portion pass through filter before adding more and use suction if washing would not otherwise be complete within 1 hour. (3) If the filtrate is turbid, add 1 - 2 ml. HNO_3 dilute to 250 ml. and mix. (4) Pipette into 500 ml. Erlenmeyer flask aliquot more than 25 mg. P_2O_5 . (5) Dilute if necessary to 50 ml. (6) Add 10 ml. HNO_3 (1 plus 1) and boil gently for 10 minutes. Cool and dilute to 100 ml. and proceed as 4(ii) (b) (2) (B). (Reference "Methods of Analysis" A.O.A.C., 1965).

(iv) Citrate insoluble phosphorus (a) Reagents : (1) Ammonium citrate solution - should have specific gravity of 1.09 at 20°C and pH of 7.0 as determined electrometrically. Dissolve 370 gms. crystalline citric acid in 1.5 litre distilled water and nearly neutralized by adding 345 ml. NH_4OH (28 - 29% NH_3). If concentration of ammonia is less than 28 per cent add correspondingly large volume, and dissolve citric acid in correspondingly smaller volume of water, cool and check pH. Adjust with NH_4OH (1 plus 7) or citric acid solution to pH - 7. Dilute solution if necessary to specific gravity of 1.09 at 20°C. (Volume will be approximately 2 litres). Keep in tightly stopped bottles and check pH from time to time. If pH has changed from 7.0, readjust. (2) Other reagents and solutions as in 4(i) and 4(ii).

(b) Procedure (Acidulated samples and mixed fertilisers) - (1) After removing water soluble P_2O_5 in 4(iii) transfer filter and residue within 1 hour to 200 or 250 ml. flask containing 100 ml. ammonium acetate solution previously heated to 65°C. (2) Close flask tightly with smooth rubber stopper. (3) Shake vigorously until paper is reduced to pulp and relieve pressure by removing stopper momentarily. (4) Continuously agitate contents of stopper flask in apparatus equipped to hold contents of flask at exactly 65°C. (Action of apparatus should be such that dispersion of sample in citrate solution is continually maintained and entire inner surface of flask and stopper is continually bathed with solution). (5) Exactly 1 hour after adding filter and residue, remove flask from apparatus, and immediately filter contents by suction as rapidly as possible through whatman No. 5 paper or equivalent, using buchner or ordinary funnel with platinum or other cone. (6) Wash with distilled water and at 65°C until volume of filtrate is approximately 350 ml. allowing time for through draining before adding more water. (7) If material is one that will yield cloudy filtrate, wash with 2 per cent NH_4NO_3 solution. (8) Determine P_2O_5 in citrate insoluble residue by one of the following methods : A. Dry paper and contents, transfer to crucible, ignite until all organic matter is destroyed and digest with 10 - 15 ml. HCl until all phosphate dissolve, or B. Treat wet filter and contents as in method prescribed in 4(i)(b) (2) (A) (C) (D) or (E). - Dilute solution to 250 ml. or other suitable volume, mix well filter through dry paper and determine P_2O_5 as in 4(ii). (Reference "Methods of Analysis", A.O.A.C., 1965).

(v) Citrate soluble phosphorus Subtract [* * *] [Omitted by S.O. 957(E), dated 24th December, 1990.] and citrate insoluble P_2O_5 from total P_2O_5 to obtain citrate soluble P_2O_5 - (Reference "Methods of Analysis"

A.O.A.C., 1965). (vi) Citric acid soluble P_2O_5 in fertilisers other than basic slag (a) Reagents : (1) Concentrated hydrochloric acid. (2) Concentrated citric acid. (3) Calcium carbonate finely ground. (4) 5 N Sodium hydroxide solution. (5) Dilute hydrochloric acid - Dilute 240 ml. of concentrated hydrochloric acid with water to 1 litre. (6) Citric molybdic acid solution - Stir 54 gms. of molybdic anhydride (MoO_3) with 200 ml. of water, add 11 gms. of sodium hydroxide and stir the mixture whilst heating to boiling point until the molybdic anhydride dissolves. Dissolve 60 gms. of citric acid in about 250 to 300 ml. of water and add 140 ml. of concentrated hydrochloric acid. Pour the molybdate solution into the acid solution, which is stirred throughout the addition. Then cool and if necessary filter the solution through paper pulp pad. Dilute the solution to 1 litre. If the solution is slightly green or blue in colour add dropwise a dilute (0.5 to 1.0 per cent) solution of potassium bromate until the colour is discharged. This reagent should be kept in dark. (7) Quinoline solution - Measure 60 ml. of concentrated hydrochloric acid and 300 to 400 ml. of water into 1 litre beaker and warm to 70° - 80°C. Pour 50 ml. of quinoline in a thin stream into the dilute acid while stirring. When quinoline has dissolved, cool the solution dilute to 1 litre, and if necessary filter through a paper pulp filter. (8) Sodium hydroxide solution - 0.5 N carbonate free. (9) Indicator solution - Mix three volumes of thymol blue solution and two volumes of phenolphthalein solution prepared as follows : A. Thymol blue solution - Dissolve 250 mgs. thymol blue in 5.5 ml. of 0.1 N sodium hydroxide solution and 125 ml. of industrial methylated spirit. Dilute with water to 250 ml. B. Phenolphthalein solution - Dissolve 250 mgs. phenolphthalein in 150 ml. of industrial methylated spirit and dilute with water to 250 ml. (10) Hydrochloric acid - 0.5 N (11) Sodium hydroxide solution - 0.5 N. (12) Hydrochloric acid - 0.1 N. (13) Surface active agent - 0.5 per cent. solution of sodium dedecyl benzene sulphonate suitable. (b) Preparation of solution - (1) Weigh to the nearest mg. about 5 mgs. of the sample and transfer to a stoppered bottle of about 1 litre capacity. (2) Dissolve 10 gms. of pure crystallized citric acid (monohydrate) in water. dilute to 500 ml. and adjust the temperature to 20°C. (3) Add the solution to the sample in the bottle, shaking so as to avoid the possibility of caking. (4) Shake the bottle continuously for 30 minutes. (5) Pour the whole of the liquid at once on a large medium - fine - filter and collect the filtrate. If the filtrate is not clear, pass it again through the same filter. (c) Procedure - (1) Transfer a volume of the solution prepared according to procedure given above containing less than 70 mg. of phosphoric acid and preferably about 50 mgs. to a 500 ml. stoppered conical flask marked at 150 ml. (2) Dilute the solution with water to 100 ml. (3) If the sample does not contain calcium add 100 to 200 mg. of calcium carbonate. (4) Then add 5 N sodium hydroxide solution dropwise until a faint permanent turbidity or precipitate is formed. (5) Dissolve the precipitate by the dropwise addition of dilute hydrochloric acid, but avoid an excess. (6) Dilute to 150 ml. and add 50 ml. of the citric molybdic acid reagent, and heat the solution to incipient abolition, maintain it at this temperature for 3 minutes and then bring it to the boiling point. (7) From burette slowly add 25 ml of the quinoline solution with constant swirling throughout the first few ml. being added dropwise, the rest in slow stream. (8) Keep the solution gently boiling during the addition. (9) Immerse the flask in boiling water for 5 minutes, then cool it to 15°C, in running water. (10) Filter with suction the contents of the flask on a paper pad, and wash the flask precipitate and filter such successive small washes of cold water until they are free from acid. (11) Transfer the filter pad and precipitate to the original flask, rinse the funnel with water and collect the rinsing in the flask. (12) If necessary, wipe the funnel with small piece of damp filter paper to ensure complete removal of the precipitate, and place and paper in the flask. (13) Add water to a total of about but not exceeding 100 ml. Stopper the flask and shake it vigorously until the

pulp and precipitate are completely dispersed.(14)Remove the stopper and wash in with water, returning the washing of the flask.(15)Add a measured volume of 0.5 N sodium hydroxide solution sufficient to dissolve the precipitate and leave a few ml. in excess.(16)Shake the flask vigorously until all the precipitate dissolves. (To facilitate the dispersal of the precipitate, after the addition of 0.5 N sodium hydroxide solution, a few drops of the surface active agent may be added if necessary).(17)Add 0.5 - 0.1 ml. of the indicator solution, and titrate the excess of sodium hydroxide with 0.5 N hydrochloric acid, until the indicator changes from violet to green - blue, and then very sharply to yellow at the end point.(18)Deduct the number of ml. of 0.5 N hydrochloric acid used from the number of ml. 0.5 N sodium hydroxide, to ascertain the volume of 0.5 N sodium hydroxide equivalent to the phosphoric acid.(19)Carry out a blank determination on all the reagents, omitting only the sample, and using 0.1 N standard alkali and acid instead of 0.5 N for the titration. Calculate the blank in terms of 0.5 N alkali and subtract in from the original result.(20)Calculate the amount of phosphoric acid in the portion taken for analysis from the factor 1.0 ml. of 0.5 N sodium hydroxide = 1.366 mgs. P₂O₅. (Reference Statutory Instruments 1960. No. 1165, Agriculture, the Fertiliser and Feeding Stuffs Regulations, 1960).(vii)Free phosphoric acid as P₂O₅(a)Reagents : (1)Acetone - conforming to IS : 170 - 1950 specification for acetone.(2)Standard sodium hydroxide solution - 0.1 N(3)Bromocresol green indicator solution - dissolve 0.1 gm. of bromocresol green in 100 ml. of rectified spirit conforming to IS : 323 - 1959 specification for rectified spirit (Revised).(b)Procedure -(1)Weigh accurately about 2.5 mgs. of the prepared sample and transfer to a soxhlet extract or(2)Add about 100 ml. of acetone and extract for three hours.(3)Cool and distil off the acetone as far as possible.(4)Take up the residue with water and make up the volume to 250 ml.(5)Pipette out exactly 100 ml. of this solution and titrate with standard sodium hydroxide solution, using bromocresol green as indicator until colour just changes from yellow to blue.Calculations :

Free phosphoric acid (as P₂O₅) per cent by weight = $17.75 \times V \times N \times \frac{W}{100}$
 Where V = volume in ml. of standard sodium hydroxide solution used. N = normality of standard sodium hydroxide solution, and W = weight in gm. of sample taken for the test. (Reference - IS Specification for superphosphate (Revised) (IS : 294 - 1962).

5. Determination of potassium :

Determination of potassium in all kinds of fertilisers is given in this section. Two alternative methods have been described. Any of these two methods may be used depending upon the availability of reagent, and suitability of the method.(i)Perchloric acid method(This method depends on the insolubility of potassium perchlorate and the solubility of sodium perchlorate in alcohol and is applicable in presence of alkali metals chlorides and nitrates. Sulphates and ammonium salts must be absent on account of the low solubility of sodium sulphate of ammonium perchlorate in alcohol. Phosphates must be removed. Method are given for the elimination of the effect of interfering substances).(a)Reagents : (1)Concentrated hydrochloric acid.(2)Barium chloride solution. Dissolve 100 gms. of barium chloride in water ; filter, the solution and dilute to 1 litre.(3)Dilute hydrochloric acid - Dilute 250 ml. of concentrated hydrochloric acid with water to one litre.(4)Calcium oxide - finely ground.(5)Ammonium hydroxide - - sp. gr. 0.88(6)Ammonium carbonate solution - saturated aqueous solution.(7)Ammonium oxalate solution - saturated aqueous

solution.(8)20 per cent perchloric acid solution.(9)Alcohol - industrial methylated spirit 95 - 96 per cent V/V.(10)Wash solution - add potassium perchlorate to alcohol and shake until saturated solution is obtained. Keep the solution over solid potassium perchlorate and filter immediately before use.(b)Potassium salts free from sulphates and other interfering substances. -(1)Dissolve in water a portion of the sample weighed to the nearest mg. equivalent to potassium content to 1.5 to 2.0 gms. of potash.(2)Cool the solution to 20°C, dilute to 500 ml. in a volumetric flask, mix well and filter through a dry filter.(3)Determine the potash in 50 ml. of solution by precipitating with perchloric acid as described under procedure.(c)Potassium salts with sulphates or other interfering substances. -(The following method is given by eliminating the interference caused by presence of sulphate. If salts contain phosphates, iron, manganese or substances other than sulphate that interfere with the determination of potash, the method described for mixed fertiliser should be used).(1)Weigh to the nearest mg. a portion of the sample equivalent in potassium content to 1.5 to 2.0 gms. of potash, into a 50 ml. beaker. Add about 300 ml. of water and 20 ml. concentrated hydrochloric acid and heat the solution to boiling.(2)To the boiling solution cautiously add, drop by drop barium chloride solution in an amount slightly in excess of that previously determined as necessary to ensure the complete precipitation of sulphate.(3)Cool the liquid to 20°C, transfer to a 500 ml. volumetric flask, dilute to 500 ml. mix and filter through a dry filter.(4)Take 50 ml. of the filtrate and evaporate to dryness in a basin, moisten the residue with concentrated hydrochloric acid.(5)Again evaporate to dryness, dissolve the residue with 5 - 10 ml. dilute hydrochloric acid and filter, if necessary. Determine the potash in solution by the method described under procedure.(d)Potassium in mixed fertilizers. -(1)Weigh to nearest centigram about 10 gm. of the sample and if organic matter is present, gently incinerate at temperature not exceeding 500°C.(2)Transfer the weighed portion of the sample or the incinerated residue to a 500 ml. beaker with a little water, and 10 ml. concentrated hydrochloric acid and then warm for ten minutes.(3)Dilute with water to about 300 ml. and bring gradually to boiling point.(4)Add 10 gms. of calcium oxide made into a paste with water.(5)Bring contents again gently to the boiling point, and keep so heated for about half an hour with frequent stirring.(6)Cool to 20°C, transfer to a 500 ml. volumetric flask, dilute to 500 ml. and after thoroughly shaking filter through a dry filter paper.(7)Transfer 250 ml. of the filtrate to another 500 ml. volumetric flask, make just acid with hydrochloric acid and heat to boiling point.(8)To the boiling solution cautiously add drop by drop, barium chloride solution until there is no further precipitation of barium sulphate.(9)Render the contents of the flask alkaline with ammonium hydroxide solution and precipitate the calcium and any excess of barium by adding ammonium carbonate solution until no further visible precipitation occurs followed by the addition of about 1 ml. of ammonium oxalate solution.(10)Cool to 20°C, dilute with water to 500 ml. and after thoroughly shaking, filter through a dry filter paper.(11)Measure 100 ml. of the filtrate and evaporate to dryness in a basin. Expel the ammonium salts from the residue by gently heating the basic over a low flame, being careful to keep the temperature below that of faint redness.(12)Cool the residue, moisten with concentrated hydrochloric acid and again evaporate to dryness.(13)Take up the residue with water the filter if necessary.(14)Determine the potash in the solution by precipitation with perchloric acids described under procedure.(e)Procedure -(1)Transfer the solution obtained as described about into a basin and add about 7 ml. of perchloric acid solution.(2)Place the basin on a hot plate or sand bath and evaporate the contents until the white fumes are copiously evolved.(3)Cool and dissolve the precipitate in a little hot water.(4)Add about 1 ml. of perchloric acid solution and again concentrate

to the fuming stage.(5)Thoroughly cool the residue in the basic and stir in 20 ml. of alcohol.(6)Allow the precipitate to cool and settle, then pour the clear liquid through a dry filter paper, draining the precipitate in the basin and stir in 20 ml. of alcohol.(7)Allow the precipitate to cool and settle, then pour the clear liquid through a dry filter paper, drain the precipitate in the basin as completely as possible.(8)Redissolve the precipitate on the paper and that remaining in the basin with hot water.(9)Add 2 ml. of perchloric acid solution to the combined solution and evaporate the whole down to the fuming stage.(10)Cool the residue in the basin and thoroughly stir the contents with 20 ml. of alcohol.(11)Allow the precipitate to cool and settle and pour the clear liquid through a weighed gooch or sintered glass crucible, draining the precipitate as completely as possible from the liquid before adding 5 ml. of wash solution.(12)Wash the precipitate by decantation with several similar small portions of the wash solution, pouring the washing through the crucible.(13)Transfer the precipitate to the crucible and wash it well with the wash solution until free from acid.(14)Dry the precipitate at 100°C and weigh.(15)Regard the precipitate as potassium perchlorate ($KClO_4$), and calculate its equivalent as potash (K_2O) by multiplying its weight by 0.34.(Reference - Statutory Instruments 1960, No. 1165. Agriculture. The Fertiliser and Feeding Stuffs Regulation, 1960).

(ii)Sodium tetraphenyl boron methodThis method is applicable to both mixed and straight potassium fertilisers.

(a)Reagents : (1)Sodium hydroxide solution - 20 per cent. Dissolve 20 gms. NaOH in 100 distilled water. (2)Formaldehyde solution - 37 per cent. (3)Sodium tetraphenyl boron (STPB) solution approximately 1.2 per cent. Dissolve 12 gms. sodium tetraphenyl boron in approximately 800 ml. water. Add 20 - 25 ml. $Al(OH)_3$, stir for 5 minutes, and filter (Whatman No. 42 paper or equivalent) into 1 litre volumetric flask. Rinse breaker sparingly with water and add to filter. Collect entire filtrate, add 2 ml. 20 per cent NaOH solution, dilute to volume with water, and mix. Let it stand for 48 hours and standardize. Adjust to that 1 ml. STPB - 1 per cent K_2O . Store at room temperature. (4)Quaternary ammonium chloride solution - approximately 0.625 per cent Dilute 50 ml. of 12.8 per cent Zephiran chloride to 1 litre with water, mix and standardize. Cetyltrimethyl ammonium bromide may be substituted for Zephiran chloride. If other concentrated is used, adjust volume. (5)Clayton yellow - 0.04 per cent. Dissolve 40 mgs. in 100 ml. water. (b)Standardization of solution. - (1)Zephiran chloride - To 1.0 ml. STPB solution in 125 ml. Erlenmeyer flask, add 20 - 25 ml. water, 1 ml. 20 per cent NaOH 2.5 ml. HCHO 1.5 ml. 4 per cent $(NH_4)_2 C_2O_4$ and 6 - 8 drops of indicator (5) above. (2)Titrate to pink end point with Zephiran chloride solution, using 10 ml. semimicro burette. Adjust Zephiran chloride solution so that 2.0 ml. - 1.0 ml. STPB solution. (3)Sodium tetraphenyl boron solution - Dissolve 2.5 gms. of KH_2PO_4 in water in 250 ml. volumetric flask, add 50 ml. 4 per cent $(NH_4)_2 C_2O_4$ solution, dilute to volume with water and mix. (It is not necessary to bring to boil). Transfer 15 ml. aliquote. (51.92 mgs. K_2O 3.10 mgs. K) to 100 ml. volumetric flask, add 2 ml. 20 per cent NaOH, 5 ml. HCHO and 43 ml. STPB reagent dilute volume with water, mix thoroughly, let stand 5 - 10 minute and pass through dry filter. Transfer 50 ml. aliquot of filtrate to 125 ml. Erlenmeyer flask, add 6 - 8 drops of indicator (5) above and titrate excess reagent with Zephiran solution. Calculate titration values as follows : $F = 34.61 / (43 \text{ ml.} = \text{ml. Zephiran}) = \%K_2O / \text{ml. STPB reagent}$. Factor applies to all fertilizers if 2.5 gms. sample is diluted to 250 ml. and 15 ml. aliquot is taken for analysis. If results are to be expressed as K rather than K_2O . substitute 28.73 for 34.61 in calculating the value of F. (c)Preparation of solution. - (1)Mixed fertilisersA. Place 2.5 gms. sample or factor weight 2.430 gms. in 250 ml. volumetric flask. B. Add 125 ml. water and 50 ml. saturated $(NH_4)_2 C_2O_4$ solution. Add 1 ml. of diglycol stearate solution if needed to prevent foaming. C. Boil for 30 minutes, add slight excess of NH_4OH and after

cooling dilute to 250 ml. Mix and pass through dry filter. (2) Potassium salts (potassium chloride sulphate, potassium magnesium sulphate, and kainite). A. Dissolve 2.5 gms. or factor weight 2.430 gms. and dilute to X 250 ml. without adding NH_4OH and $(\text{NH}_4)_2\text{C}_2\text{O}_4$. B. When interfering substances such as NH_3Ca , Al, etc., are present proceed as in (1) above. (d) Procedure - (1) Transfer 1.5 ml. aliquot of sample solution to 100 ml. volumetric flask and add 2 ml. 20 per cent NaOH and 5 ml. HCHO . (2) Add 1 ml. standard STPB solution for each 1 per cent K_2O expected in sample plus additional 8 ml. excess to ensure complete precipitation. (3) Dilute to volume with water, mix thoroughly, let it stand for 5 - 10 minutes and pass it through dry filter (Whatman 12 or equivalent). (4) Transfer 50 ml. filtrate to 125 ml. Erlenmeyer flask, add 6 - 8 drops of indicator (5) and titrate excess reagent with standard Zephiran solution. per cent K_2O in the sample = $(\text{ml. STPB added} - \text{ml. Zephiran XF}) \text{ where } F = \text{per cent } \text{K}_2\text{O} / \text{ml. STPB} = \text{reagent}$. (Reference "Methods of Analysis", AOAC, 1985).

6. Method of analysis of anhydrous ammonia -

(i) Estimation of Water and Ammonia (a) Scoop : - This method is for the determination of water at 20°C (or room temperature), and usually ammonia by difference of any essentially anhydrous ammonia liquid product. Normally, the water will be low (less than 0.5 per cent) or sample of synthetic ammonia. (b) Apparatus : (1) Ammonia sampling tubes. (2) Fume hood with exhaust dust. (3) Sample tube holder rack in hood fabricated as convenient to hold number of tubes desired. (4) Gloves protective, elbow length. (c) Reagents : Charcoal, reagent, 14 - 20 mesh. (d) Procedure, - (1) Remove the sample tubes from the sample carrier and place in the provided sample tube holders in the fume hood. (2) Allow the ammonia sample to boil and to evaporate (approximately 30 minutes) spontaneously in the fume hood. Because of the low temperature of the boiling ammonia, atmospheric moisture freezes on the sample container. Exercise care to see that one of this ice contaminates the sample. (3) When the boiling of the sample ceases, remove the tube from the holder. Wipe the outer surface of the tube dry and read and record the volume of liquid residue in the tube. (4) From the volume recorded, calculate the weight per cent water in the sample. Ignore the small piece of charcoal, if used. Calculation :

$$\text{per cent water} = \frac{A \times F \times 0.890 \times 0.684}{V \times 0.682} \times 100$$

where A = volume of residue in tube (ml.) V = Volume of sample taken (ml.) F = Evaporation factor, taken from Table I corresponding to noted sample pressure when sampling was obtained. 0.890 = density of residue (gm./ml.) 0.684 = weight fraction of water in residue. 0.682 = density of sample (gm./ml.) (e) Precaution. - The evaporation in the fume hood should be conducted with the window lowered to protect personnel from possible spray. Whenever it is necessary to handle the sample tubes, protective gloves should be worn. Note. - Determination of residue on evaporation will not usually be necessary for normal ammonia sample. If sample is clear and water residue is clear and colourless, the residue may be considered to be nil. However, should it become necessary to determine the dissolved solids and suspended solids in anhydrous ammonia, proceed as follows : (f) Sampling. - Thoroughly clean and dry the sample tubes, weigh (W_1) each selected tube to the nearest milligram with a similar tube as counterpoise. Obtain the samples as desired earlier. (g) Apparatus. - (1) Analytical balance. (2) Ammonia sampling tubes. (3) Rubber tubing, 1/4 OD length desired. (h) Reagents : Air dry, carbon dioxide - free. (i) Procedure. - (1) Start test following

elimination of the water content of the sample as given earlier.(2)Connect one end of a rubber hose to a source of dry, carbon dioxide - free air. Insert the other end of the hose into the sample tube and gently force air through the sample tube until all liquid has been evaporated and no ammonia gas remains in the tube.(3)Wipe a counterpoise and the sample tube containing the residue to substantially the same extent to remove moisture which may have condensed on their outer surfaces.(4)Weigh the sample tube and residue to nearest milligram.(5)Calculate the weight per cent residue of the sample.Calculation :

$$\text{Per cent residue} = (W_2 - W_1) \times F \times 100 \times \frac{V}{0.682}$$

Where W_1 = weight (gm.) of tube (before sampling). W_2 = weight (gm.) of tube and residue. V = volume of sample taken (ml.) F = Evaporation factor, taken from Table 1 corresponding to noted sample pressure when sample was obtained. 0.682 density of sample (gm./ml.) (j) Precaution. -A check of completeness of evaporation may be made by visual inspection and by carefully smelling the air in the tube making sure that no carbon dioxide or moisture from the breath enters the sample tube. (k) Per cent ammonia content (by difference) -In general, the ammonia content of the sample will : per cent ammonia = 100 - (Per cent water - per cent residue, if determined). (l) Precaution. -The evaporation in the fume hood should be conducted with the window lowered to protect personnel from possible spray. Whenever it is necessary to handle the sample tubes, protective gloves should be worn. (ii) Determination of oil content (a) Apparatus : (1) 500 ml. conical flask calibrated to hold 300 ml. (about 200 gms.) of sample and with a bung fitted with glass through which the exit gas can be led away to a safe place. Ensure that the glass tube and exit line are free from constructions. (2) Platinum dish 75 mm. in diameter. (b) Reagent : The reagent used shall be of a recognised analytical reagent quality like petroleum spirit with a boiling range of 40°C to 60°C. (c) Procedure. - (1) Quickly running the sample (as per the sample procedure) up to the calibration mark, insert the bung. (2) Immerse the flask in a continuous stream of cold water and allow the ammonia to evaporate slowly. (3) When the evaporation is complete, remove the bung and gently blow out the last traces of ammonia with a small jet of filtered air free from carbon dioxide. (4) Dry the outside of the flask. (5) Place the flask in an oven at 105±5°C for 15 minutes to remove the moisture blowing out the last traces with a gentle stream of filtered air at the end of this period ; allow to cool. (6) Add to the flask approximately 100 ml. of the petroleum spirit and swirl to dissolve the oil. (7) Filter the solution through a small filter paper (a Whatman No. 31 paper is suitable) direct into the platinum dish previously traced to the nearest 0.1 mg. (8) Repeat the operation with two more successive 10 ml. portions of petroleum spirit, filtering through the same paper as before into the same dish. (9) Evaporate the combined extracts to dryness on a water bath in a fume cup board, taking care to avoid naked lights and finally dry in an oven at 105±5°C for 30 minutes. (10) Allow the dish to cool thoroughly in a desiccator and weight against to the nearest 0.1 mg. (11) At the same time, can, out a blank determination on the petroleum spirit and filter paper. (12) Make an appropriate correction in the calculation. Calculation :

$$\text{Oil content, parts per million by weight} = (W_1 - W_2) - W_3 \times \frac{0.68 V_2}{V_1} \times 10^6 F$$

Where W_1 = weight in grams of platinum dish and residue. W_2 = weight in grams of dish alone. W_3 = weight in grams of oil in blank determination. V_2 = volume in ml. of sample taken. F = evaporation factor taken from Table corresponding to noted sample pressure when sample was obtained. Table Evaporation factor for different vessels or line pressures.

Vessel or Line Pressure (Psig.)

Evaporation Factor (F)

1	2
0	1.000
10	0.963
20	0.940
30	0.920
40	0.900
50	0.885
60	0.870
70	0.860
80	0.850
90	0.840
100	0.830
110	0.821
120	0.813
130	0.805
140	0.797
150	0.789
160	0.782
170	0.776
180	0.770
190	0.764
200	0.758

Note. - When a sample of liquid ammonia is transferred to an open flask/tube from a container in which the pressure is higher than the atmospheric pressure that sample quickly reaches the thermodynamics equilibrium with its new environment. Some of the liquid ammonia will evaporate and since the ammonia so lost as vapour contains non - volatile constituents present in the original product, the concentration of these constituents will increase in the liquid sample taken. This may be significant factor in subsequent analysis and fortunately it is possible to calculate from the known thermodynamic properties of ammonia an appropriate correction. This correction is usually known in this context at the evaporation factor of flask factor and is simply that fraction by weight of the original liquid ammonia which remains as liquid in the sample Multiplication of the determined quantity of a non - volatile constituents (oil, water dissolved or suspended solids, etc.) by the evaporation factor gives a result which will be closer to the true figure. The Table above presents the evaporation factor for different vessel or line pressure. When a sample is transferred (example by gravity) from a container of a cylinder or the sampler at the same pressure, there should be no loss by evaporation and in this case it is not necessary to use evaporation factor :

7. Method of analysis of zinc sulphate, [Both Heptahydrate and Monohydrate] [Substituted by S.O. 534(E), dated 20th July, 1992.]

(i) Quality of reagents Pure chemicals and distilled water shall be used in tests. Note. - "Pure chemicals" shall mean chemicals that do not contain impurities which effect the results of analysis.

(ii) Determination of matter insoluble in water (a) Reagents : Dilute Sulphuric Acid - 10 percent. (b) Procedure. - Dissolve 25.0 gms. of the material in 125 ml. of water and add 1 ml. of dilute sulphuric acid. Heat the solution to boiling, filter through a weighed and prepared Gooch crucible or sintered glass crucible (G.No. 4) and wash the residue thoroughly with hot water. Dry the crucible at $110^{\circ} \pm 5^{\circ}$ to constant mass. Calculation: Matter insoluble in water, per cent. by mass weight = $\frac{4A}{W}$ Where, A = weight in gm. of the residue

(iii) Determination of Zinc in Zinc Sulphate by modified EDTA titration method [Substituted by G.S.R. 1160(E), dated 12st October, 1986 (w.e.f. 12st, October, 1986).] - (a) Reagents : (1) EDTA Solution. - Dissolve 3.72 gms. of Di - sodium Ethylene Diamine Tetraacetate dihydrate in distilled water and make up the volume to 1 litre. (2) Standard Zinc metal solution. - Weight about 1.0 gm. of zinc metal and record the weight accurately. Express this as W. Add HCl (1:1) @ 20 ml. per gm. of zinc metal. Keep it for few hours and allow it to dissolve completely. Make up the volume of the solution to exactly 100 ml. (3) Ammonium Hydroxide (20 per cent) (m/m/). (4) Ammonium Chloride - AR grade salt. (5) [Sodium Cyanide] [To be used with extreme care.] - AR/GR grade salt. (6) Eriochrome Black (T) indicator mixture. Mix thoroughly 1 gm. of Eriochrome Black (T) indicator with 100 gm. of AR Grade sodium chloride. (7) Formaldehyde - Acetic Acid solution (4 per cent.). - Dissolve 100 ml. of Formaldehyde (37 - 40 per cent) in about 100 ml. of distilled water. Add 40 ml. glacial Acetic acid and make volume to 1 litre with distilled water. (8) Hydroxylamine Hydrochloride . - AR Grade solid salt. Note. - Distilled water means glass distilled water. (b) Procedure. - (1) Standardization of EDTA solution : (A) Take 10 ml. of Zinc metal solution (standard). (B) Add about 0.1 gm. of ammonium chloride and 30 ml. of ammonium hydroxide solution (20 per cent). (C) Dilute it by adding about 30 ml. distilled water. (D) Add a pinch of eriochrome black (T) indicator mixture. It will give red colour. (E) Titrate it with EDTA solution to obtain clear blue end point. Note the volume of EDTA used as V_1 ml. (2) Estimated of Zinc in sample : (A) Weigh accurately 1.0 gm. of given zinc sulphate sample and dissolve it in 100 ml. of distilled water in a volumetric flask. (B) Take 10 ml. of aliquote in beaker. Add 0.1 gm. of hydroxylamine hydrochloride and 0.1 gm of ammonium chloride. (C) Cautiously add small quantity of sodium cyanide. White precipitate will appear. Continue adding sodium cyanide till white precipitate disappears while swirling the beaker with hand. Add about 0.5 gm. excess of sodium cyanide. (D) Dilute it by adding about 30 ml. of ammonium hydroxide (20 per cent.) and add about 30 ml. of distilled water. (E) Add a pinch of eriochrome black (T) indicator mixture. It will give red colour. (F) Titrate with EDTA solution till there is a sharp change to violet colour. Note the volume of EDTA used as V_2 (ml.). (G) Add 20 ml. of Formaldehyde - acetic acid solution into above titrate solution and mix well. Red colour will reappear. (H) Titrate it with EDTA solution to get blue end point without red tinge. Note the volume of EDTA used in second titration as V_3 ml. Calculation : $\text{Zinc \%} = \frac{10}{V_1} \times V_3 \times \frac{W}{100}$ = Weight in gm. of piece of Zinc metal taken for preparation of standard zinc solution. V_1 = Volume of EDTA solution (in ml.) used for 10 ml. of Zinc metal solution. V_3 = Volume of EDTA solution (in ml.) used for second titration.]

(iv) Calorimetric Method for Determining of Copper (a) Outline of the method - Sodium diethyldithio carbonate reacts with slightly acidic or ammoniacal solution of copper in low concentration to produce a brown colloidal

suspension of the cupric diethyl - dithiocarbonate. The suspension can be used with extreme care. be extracted with an organic solvent and the colour extracted determined spectrophotometrically. (b) Apparatus : Photometer - Any suitable photoelectric calorimeter. (c) Reagents : (1) Ammonium Citrate Solution - 40 per cent (m/v) (2) Sodium Diethyldithiocarbamate Solution - 0.1 per cent (m/v). (3) Standard Copper Solution - Clean the surface of a copper wire with sand paper. Dissolve 100 gms of pure copper in dilute nitric acid. Heat the solution nearly to dryness to drive off the acid. Add about 10 ml. of water and heat again nearly to dryness. Take up the residue in water, add 25 ml. of 1N sulphuric acid and dilute to 1000 ml. One millilitre of this solution contains 0.1 mg. of copper (as Cu). Alternatively, dissolve 3.928 gms. of cupric sulphate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$) in sufficient water containing 1 or 2 ml. of concentrated Sulphuric acid and dilute to 1000 ml. One millilitre of the solution contains 1.0 mg. of copper (as Cu). (4) Ammonium Hydroxide - 18 N. (5) Carbon Tetrachloride. (d) Preparation of sample. - (1) Weigh 5 gms. of sample accurately, dissolve in distilled water and add 1 ml. of sulphuric acid. Filter the solution and make up to 250 ml. with water in a volumetric flask. Take 50 ml. of the above solution in a beaker, heat, pass hydrogen sulphide gas or add sodium sulphide solution and ensure complete precipitation. Filter hot and keep the filtrate for subsequent analysis. (2) Boil the residue with dilute nitric acid and filter, if necessary. To the filtered solution add sulphuric acid, evaporate, dilute and filter. Keep the residue for determination of lead. (e) Procedure of determination of copper - Determine copper in the filtrate from (d) (2) above by diethyldithiocarbonate method as detailed below. - Transfer an aliquot containing not more than 0.65 mg. of copper to a 100 ml. separating funnel. Add 2 ml. of ammonium citrate solution and adjust the pH to 8.5 with ammonium hydroxide. The presence of ammonium ions helps to disintegrate iron, if present and to prevent rapid fading of developed colour. Add 10 ml. of sodium diethyldithiocarbamate solution and develop the colour. Extract the coloured complex with 5 - 10 ml. of carbon tetrachloride. Separate the carbon tetrachloride layer from aqueous layer and centrifuge for 5 minutes to separate the water droplets. Transfer the carbon tetrachloride solution to a photometric cell and measure its absorbance at the wavelength of maximum absorption 440 mμ relative to reagent blank. Calculate the corrected absorbance by subtracting the reading obtained for the solution containing - no copper. Transfer to a series of 100 ml. separating funnels aliquots of standard copper solution corresponding to 0, 0.1, 0.2, 0.5, 1.0, 2.0, 2.5 gms. of copper and proceed exactly as prescribed above. Plot a graph of corrected, absorbance of solutions against their copper contents. Note. - If Bismuth is suspected it has to be separated or otherwise accounted for. Calculation. - Calculate the corrected absorbance of subtracting the value obtained for the blank from that obtained for the test solution and read from the calibration curve the corresponding mass of copper.

Copper (as Cu). per cent by mass = $\frac{M_1}{M_2} \times 100$

Where M_1 = mass in gm. of copper as determined in the given aliquot of the test solution, and M_2 = mass in gm. of the material present in the aliquot of the test solution. (v) Determination of lead by colorimetric method using dithizone. (a) Apparatus : Nessler Cylinders - 50 ml. capacity. (b) Reagents : (1) Standard Lead Solution. - Dissolve 0.40 gm. of lead nitrate [$\text{Pb}(\text{NO}_3)_2$] in water containing 2 or 3 ml. of concentrated nitric acid and make up the volume to 1000 ml. with water. Transfer 10 ml. of this solution to a volumetric flask, add 2 or 3 ml. of concentrated nitric acid and dilute with water to 1000 ml. one millilitre of this solution contains 2.5 mgs. of lead (as Pb). The diluted solution shall be freshly prepared. (2) Dilute Ammonium Hydroxide. - 1N, approximately. (3) Reagent A - Dissolve 25

gms. of triammonium citrate or 22 gms of citric acid and 4 gms. of hydroxylamine hydrochloride in about 200 ml. of water. Add dilute ammonium hydroxide to bring the pH to 8.5 Dilute the solution to 500 ml. Purify this solution by extracting with 15 ml. portions of 0.01 per cent dithizone solution until the final colour of the dithizone extract is green. Wash the aqueous remainder portion three times with 25 ml. portions of chloroform and finally with 25 ml. portion of carbon tetrachloride.

(4) Dithizone (Dephenyl thiocarbazone or phenylazothiomofomic Acid) Solution - Dissolve 0.01 gm. of dithizone in 100 ml. of carbon tetrachloride, shaking intermittently for 1 hour. Allow to stand overnight and shake once again before using. This shall be kept in a cool and dark place. This gives a 0.01 per cent solution. Filter, if necessary. Dilute 10 ml. of this solution to 100 ml. with carbon tetrachloride in a 100 ml. volumetric flask. This shall be prepared fresh before determination. This gives a solution of 0.001 per cent.

Note 1. - Carbon tetrachloride used should be further purified. One litre of carbon tetrachloride is extracted with two portions of 25 ml. dilute ammonium hydroxide and then kept over 100 gms. to activated carbon. Before use, it is decanted and distilled at about 80°C over a little fresh lime.

Note 2 - Sometimes dithizone solid and its 0.01 per cent solution deteriorate on storage. The 0.01 per cent solution should, therefore, be tested before further dilution, by shaking 2 ml. of the solution with 5 ml. of 1 per cent ammonium hydroxide. If the organic layer is only faintly yellow under these conditions, the solution may be used. If it is deeply coloured it shall be discarded and fresh solution be prepared. The solution as well as the reagent should be stored in a refrigerator and exposure to sunlight should be avoided during analytical work. To increase the stability of 0.01 per cent solution, it should be covered with a thin aqueous layer saturated with sulphur dioxide.

(5) Thymol Blue Indicator Solution. - 1 per cent (m/v) solution in rectified spirit.

(c) Procedure. - (1) Take several aliquots of standard lead solution into a series of separating funnels and 5 ml. of the water and 10 ml. of the Reagent A. Then add 2 drops thymol blue indicator and bring the pH of the solution to 8.5 by addition of dilute ammonium hydroxide. Add 5 ml. of the dithizone solution and shake well for about 10 seconds. Drain the organic layers into stoppered Nessler cylinders. (2) Dissolve the residue obtained in (iv) (d) (2) above, in dilute nitric acid and dilute with water. Take a suitable aliquot of the solution and transfer to a separating funnel. Develop the colour as prescribed above. Drain the organic layer into a stoppered Nessler cylinder, add 10 ml. of dilute ammonium hydroxide, and shake for about 10 seconds. Compare the colour developed with those of the standard solutions. Note the volume of the standard with which the colour of test solution matches.

Note. - If the colour of the test solution is intermediate between two standard solutions, then the experiment is repeated by taking more number of standard solutions in that range and exact colour matching is arrived at.

Calculation :

Heavy metals as (PB), per cent by mass = $100 \times V \times f \times \frac{M}{1000}$

where V = Volume in ml. of standard lead solution matching with the test solution. f = mass in gm. of heavy metals (as Pb) equivalent to 1ml of standard lead solution, and M = mass in gm. of the material in the aliquot taken for the test.

(vi) Determination of magnesium in the sample material

(a) Reagents : (1) Eriochrome Black T Indicators. - Dissolve 0.1 gm. of eriochrome black T in 25 ml. or methyl alcohol. (2) Ammonium Hydroxide Ammonium Chloride Buffer Solution - Mix. 350 ml. of ammonium hydroxide (20 per cent w/w) with 54 gms. of ammonium chloride. Dilute with water and make up the volume to 1000 ml. (The PH of the solution should not be more than 10). (3) Standard Magnesium Solution. - 0.01 M Weigh 2.4640 gm. of magnesium sulphate ($\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$) and dissolve it in water. Make up the volume to one litre. (4) Ethylenediamine Tetra Acetate (EDTA) Solution. - Dissolve 3.72 gms. of disodium ethylenediamine tetra acetate dihydrate

in water and make up the volume to one litre. (b) Procedure. - Standardization of EDTA Solution. - Take 10 ml. of standard magnesium solution in a conical flask. Add 20 ml. of water, one millilitre of eriochrome black T indicator and 25 ml. of ammonium hydroxide ammonium chloride buffer solution. Heat at 40°C to 50°C and then titrate with EDTA solution, maintaining the temperature between 40°C and 50°C until the colour changes from wine red to distinct blue. IOM

Molarity of EDTA solution = $10M_1 \frac{V_1}{V_2}$

where M_1 = molarity of standard magnesium solution, and V_1 = Volume in ml. to EDTA solution used for titration. (c) Determination of Magnesium in the sample material - Take the filtrate from (iv) (d) (1) (after removal of copper) add a few drops of concentrated nitric acid, boil and cool and then add solid ammonium chloride (about 2 grams). boil and cool add ammonium - hydroxide till the strong smell of ammonia comes and filter the precipitate through sintered crucible, take filtrate and add dilute sulphuric acid till the solution is acidic (test with methyl red), heat the solution to boil and add excess of di - ammonium - hydrogen phosphate with continuous stirring. Add 10 percent ammonia solution with continuous stirring till the solution is just alkaline (test with methyl red) while precipitate of zinc ammonium phosphate will be formed the optimum pH for precipitation is 6 - 7, allow it to stand for 3 - 4 hours. Filter through Whatman filter paper No. 40. Collect the filtrate in a volumetric flask. Make up the volume (say 100 ml.) Take a suitable aliquot (say 10 ml.) for the determination of magnesium. Add 20 ml. of water, one ml. of eriochrome black - T indicator and 20 ml of ammonia hydroxide ammonia chloride buffer solution. Heat to 40° to 50°C and titrate with standard EDTA solution maintaining the temperature between 40°C and 50°C until the colour changes from wine red to distinct blue.

1 ml. of 0.01 M - EDTA = 0.2432 mg. of "Mg" $Mg = X \times 0.2432 \frac{V_1}{V_2}$

where (vii) Determination of pH Dissolve 5 gm. of the material in freshly boiled and cooled water.

Dilute to [100 ml.] [Substituted by S.O. 534(E), dated 20th July, 1992.] and mix. Determine the pH value of the solution with a pH meter. (viii) [Determination of Iron in the sample material (Spectro - Photometric Method)] [Inserted by S.O. 534(E), dated 20th July, 1992.]. (This method is applicable for Iron contents up to 1mg. in the final aliquot) (a) Outlines of the method - In test solution, iron is determined absorptions metrically as Ferrous Iron form red complex with Ortho -

phenanthroline. (b) Apparatus. - Photo - electric colorimeter. (c) Reagents. - (1) Bromophenol - blue indicator solution - 0.4 per cent solution in 95 per cent ethanol. (2) Sodium Citrate solution - 25 per cent. (3) Hydroquinone solution - 1 per cent in a Acetic acid buffer of pH 4.5 obtained by mixing 65 ml. of 0.1 M Acetic acid with 35 ml. of 0.1 M Sodium Acetate solution. (4) Ortho - phenanthroline solution - 0.25 per cent in 25 per cent ethanol. (5) Dilute Hydrochloric acid - 1:3 (VN). (6) Standard Iron solution - Dissolve 0.7002 gm. of Ammonium Ferrous Sulphate (AR) in distilled water containing 2 ml. of perchloric acid and dilute to 100 ml. with water. One ml. of this solution contains 1 mg. of Iron (d) Preparation of sample solution. - Boil 1 gm. sample in 20 ml. of Hydrochloric Acid (1:1) for 30 minutes and filter through Whatman No. 40 filter paper in 100 ml. volumetric flask. Follow with repeated extracted with boiling dilute Hydrochloric acid (1:5) decanting the solution each time through the same filter paper. Dilute the filtrate to 100 ml. (e) Procedure - (1) Take 5 ml. of the sample solution, add bromophenol blue indicator solution and titrate with Sodium Citrate solution until yellow colour changes to blue. (2) Take another 5 ml. of sample solution in a 25 ml. volumetric flask. Add 1 ml. of Hydroquinone solution, 3 ml. of Orthophenanthroline solution and an amount of Sodium Citrate solution equivalent to the above titration in step (1), Dilute with distilled water to 25 ml. Allow the solution to stand for one hour. (3) Measure the absorbance of test solution

at 510 nm using 1 cm. cell and also blank with water.(4)Place 4, 8, 12, 16 and 20 ml. of standard Iron solution in a series of 100 ml. volumetric flasks. To each add 40 ml. of diluted Hydrochloric acid and dilute to 100 ml. with water.(5)Using ml. aliquote proceed as for sample solution.(6)Measure the absorbance of the solution and plot a graph relating to absorbance to number of mgs. of Iron.Calculation :Per cent Fe = $2x/W$ where, X= Concentration (in mg.) of Iron in final sample solution aliquot as determined from the graph.W= Weight in gm. material taken for the preparation of sample solution.]

8. Alternate method of analysis of zinc - sulphate, [Both Heptahydrate and Monohydrate] [Substituted by S.O. 534(E) dated 20th July, 1992.]

(i)Quantity of reagents(1)Unless specified otherwise, pure chemicals and glass distilled or demineralised water shall be used in tests.Note. - "Pure chemicals" means chemicals that do not contain impurities which affect the results of analysis."Demineralised water" means the water obtained after passing distilled water through a cation and a anion exchange resins or a combined cationanion exchange resin.(ii)Determination of Zinc(a)Reagents :(1)Standard Zinc solution. - Weigh 0.4398 gm. of zinc sulphate ($ZnSO_4 \cdot 7H_2O$ - A.R. grade) on a clear watch glass and transfer it to one litre flask through the funnel giving several washings to watch glass and funnel with glass distilled or demineralised water. Add one ml. of 10 per cent sulphuric acid (A.R. Grade) and make the volume upto the mark. Stopper the flask and shake the solution well. This is 100 ppm Zinc solution hereinafter called Standard A. This solution should be stored in a clean bottle for further use. Dilute 10 ml. of 100 ppm solution of zinc (Standard A) to 100 ml. to get 10 ppm standard zinc solution designated as Standard B.(2)Glass distilled or mineralised water of pH 2.5 ± 0.5 . - Dilute 1 ml. of 10 per cent sulphuric acid to one litre with glass distilled or mineralised water and adjust the pH to 2.5. with a pH meter using H_2O_4 or NaOH. This solution is called acidified water and 5 to 10 litre of this solution should be prepared at a time.(3)Preparation of working standards. - Pipette the following volume of Standard B in 50 ml. numbered volumetric flask and make the volume with acidified water.

Flask No.	Volume of Standard B taken (ml.)	Concentration of zinc after making volume to 50ml. (ppm)
1	0.0	0.0
2	1.0	0.2
3	2.0	0.4
4	3.0	0.6
5	4.0	0.8
6	5.0	1.0
7	6.0	1.4
8	9.03	1.8
9	10.0	2.0

Stopper the flasks and shake them well. Prepare the standard in duplicate. The same acidified water should be used for preparing the solution of unknown fertilizer samples. Fresh standards should be

prepared every time when a fresh lot of acidified water is prepared. (b) Procedure. - (1) Preparation of Zinc Sulphate fertiliser samples. - Weigh 0.25 gm. of the material on a clean watch glass and transfer it to one litre volumetric flask through the funnel giving repeated washings with glass distilled water and dissolve the material by shaking well. Then make the volume up to mark with glass distilled water and shake well. (2) Take 5ml. of the prepared solution in 250 ml. volumetric flask and make the volume with acidified water. Shake the solution well and filter through Whatman No. 42 filter paper in dry clean flasks. The flasks should be rinsed with a 10 to 15 ml. of the filtrate and then continue filtration. (3) Flaming the solutions. - Flame the standards and the filtered samples on atomic absorption spectrophotometer at a wavelength of 213.8 nm (Zn line of the instrument). Calculations : Prepare a standard curve of known concentration of zinc solution by plotting the absorbance values on Y - axis against their respective zinc concentration on X - axis. Calculate the percentage zinc in zinc fertilizer by multiplying zinc concentration value calculated from standard curve by 20. Example:

Weight of fertiliser sample	= 0.25 gm.
Volume made	= 1000 ml.
Further dilution	= 5.0 times
Reading of the samples from Atomic Absorption	= Y
Corresponding, concentration value of zinc from standard curve against Y absorbance	= X ppm
Percentage Zinc in the fertilizer	= 20(X)

(d) Precautions (1) Weighing must be done on an electric balance. (2) All the glass apparatus to be used should be of corning make and washed with dilute hydrochloric acid (1:4) and washed thoroughly with distilled and then with demineralised water. (3) The pipette should be rinsed with the same solution to be measured. (4) The outside of the pipette should be wiped with filter paper after taking out from the solution to be measured. (5) After using the pipette, place it on a clean dry filter paper in order to prevent contamination. (6) To start filtration, only a few drops should be added first in order to the one filter paper and then continue further filtration. (iii) Determination of magnesium (a) Reagents : (1) Strontium chloride. - Dissolve 7.5 gms. of strontium chloride ($\text{SrCl}_2 \cdot 6\text{H}_2\text{O}$) in one litre of glass distilled water. (2) Standard Magnesium solution. - Weigh 0.507 gm. of magnesium sulphate ($\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$) on a clean watch glass and transfer it to one litre, flask through the funnel giving several washings to watch glass and the funnel with glass distilled or demineralised water. This is 50 ppm Mg. solution Dilute 10 ml. of 50 ppm solution of Mg to 10 ml. to get 5 ppm standard Mg. solution. (3) Preparation of working standards. - Pipette the following volume of 5 ppm standard Mg. solution in 50 ml. numbered volumetric flasks. Add 10 ml. of strontium chloride solution to each flask and make up the volume to 50 ml.

Flask No.	Volume of 5 ppm Mg. solution taken (ml.)	Volume of strontium chloride added (ml.)	Concentration of magnesium after making the volume to 50 ml. (ppm)
1	0.0	10.0	0.0
2	2.0	10.0	0.2
3	4.0	10.0	0.4
4	6.0	10.0	0.6
5	8.0	10.0	0.8

6 10.0 10.0 1.0

Stopper the flask and shake them well. Prepare fresh standards every fortnight.(b)Procedure.

-(1)Pipette 20 ml. of the solution which was prepared for the determination of zinc by dissolving 0.25 gm. of the fertilizer sample in one litre flask (Step A - 2.2.1). Add 10 ml. of strontium chloride. Make up the volume to 50 ml.(2)Flame the standards and the samples on atomic absorption spectrophotometer at a wavelength of 285.5 mu (Mg. line of the instrument).Calculations :Prepare a standard curve of known concentrations of Mg. solutions by plotting the absorbance value on Y - axis against their respective concentration values on X - axis. Percentage magnesium in the zinc fertilizer will correspond to the concentration values calculated from the standard curve.Example :

Weight of the fertiliser	=0.25gm.
Volume make	=1000 ml.
Further dilution	= 2.5 time
Reading of the sample from atomic absorption spectrophotometer	=Y
Corresponding, concentration of Mg. from standard curveagainst Y absorbance	= X ppm
Percentage magnesium in the fertiliser	=X

(iv)Determination of copper(a)Reagents :(1)Standard Copper Solution. - Weigh 0.1965 gm. of copper sulphate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$) on a clean watch glass and transfer it to one litre flask through the funnel giving several washings to watch glass and the funnel with glass distilled water. Add one ml. of 10 per cent sulphuric acid and made up the volume up to the mark. Stopper the flask and shake the solution well. This is 50 ppm Cu solution and should be stored in a clean bottle for further use. Dilute 10 ml. of 50 ppm solution of copper to 100 ml.to get 5 ppm standard copper solution.(2)Glass distilled or mineralised acidified water of pH 2.5 ± 0.5 (same as given in 8(ii) (a)(2)).(3)Preparation of working standards. - Pipette the following volume of 5 ppm standard copper solution in 50 ml. numbered volumetric flask and make the volume with acidified water.

Flask No.	Value of 5 ppm standard Cu solution taken (ml.)	Concentration of copper after making volume to 50ml. (ppm)
1	0.0	0.0
2	2.0	0.2
3	4.0	0.4
4	6.0	0.6
5	8.0	0.8
6	10.0	1.0

Stopper the flasks and shake them well. Prepare fresh standards every fortnight.(b)Procedure.

-(1)The solution with was prepared for the determination of zinc by dissolving 0.25 gm. of the fertilizer sample in one litre flask Step 8(ii)(b)(1) should be used for the determination of copper.(2)Flame the standards and the samples on an atomic absorption spectro - photometer at the wavelength of 324.8 mu (Cu line of the instrument).Calculation:Prepare a standard curve of known concentrations of copper solutions by plotting the absorbance values on Y - axis against their respective concentration values on X - axis. Calculate the percentage copper in the zinc fertilizer by multiplying the copper concentration value calculated from the standard curve by 0.4.Example :

Weight of the fertilizer sample	=0.25gm.
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Volume made	=1000 ml
Reading of the samples from atomic absorptionspectrophotometer	=Y
Corresponding, concentration of copper from standard curveagainst Y	= X ppm
absorbance percentage copper in the fertilizer	=0.4X

(v) Determination of lead (a) Reagents : (1) Standard Lead Solutions. - Weigh 0.1965 gm. of lead nitrate $[Pb(NO_3)_2]$ on a clean watch glass and transfer it to one litre flask through the funnel giving several washings to watch glass and funnel with glass distilled or demineralised water. Add 10 ml. of concentrated distilled nitric water. Add 10 ml. of concentrated distilled nitric acid and make the volume up to the mark. Stopper the flask and shake the solution well. This is 100 ppm lead solution and should be stored in a clean bottle for the further use. Dilute 10 ml. of 100 ppm solution of lead to 100 ml. with 1 per cent nitric acid solution to get 10 ppm standard lead solution. (2) per cent nitric acid solution. - Dilute 10 ml. of concentrated distilled nitric acid to one litre with glass distilled water. (3) 20 per cent zinc sulphate solution. - Weigh 20 gms. of zinc sulphate $(ZnSO_4 \cdot 7H_2O)$ and dilute to 100 ml. with 1 per cent nitric acid solution. (4) Preparation of working standards. - Pipette the following volume of 10 ppm standard lead solution in ml. numbered volumetric flask. Add 5 ml. of 20 per cent zinc sulphate solution to each flask and make the volume with 1 per cent nitric acid solution.

Flask No.	Volume of 10 ppm lead solution taken (ml.)	Volume of 20% zinc sulphate solution added (ml.)	Concentration of lead after making the volume to 50 ml. (ppm)
1	0.0	5.0	0.0
2	2.0	5.0	0.4
3	4.0	5.0	0.8
4	6.0	5.0	1.2
5	8.0	5.0	1.6
6	10.0	5.0	2.0

Stopper the flasks and shake them well. (b) Procedure. - (1) Preparation of zinc sulphate fertiliser samples. - Weigh 1 gm. of the material on a clean watch glass and transfer to 50 ml. volumetric flask through the funnel giving washings with 1 per cent nitric acid solution. Dissolve the material and make the volume with 1 per cent nitric acid solution. Samples should be prepared in duplicate. (2) Flaming the solutions. - Flame the standards and samples on atomic absorption spectrophotometer at a wavelength of 217 μ (Lead line of the instrument.) Calculations : Prepare a standard curve of known concentrations of lead solution by plotting the absorbance values on Y - axis against their respective lead concentration on X - axis. calculate the percentage lead in zinc fertilizer by multiplying lead concentration value calculated from standard curve by 0.005. (vi) Determination of pH (a) Procedure : Dissolve 5 gm. of the material in freshly boiled water. Dilute to [100 ml] [Substituted by S.O. 534(E) 20th July, 1992.] and mix. Determine the pH value of the solution with pH meter. (vii) Determination of matter insoluble in water (a) Procedure : Dissolve 25.0 gms. of the material in 125 ml. of water. Filter through a weighed and prepared Gooch crucible or sintered glass crucible (G. NO. 4) and wash the residue thoroughly with water. Dry the crucible at $110^\circ \pm 8^\circ$ to constant mass. Calculations : Matter insoluble in water per cent by weight = $4 - A$. Where, A = Weight in gm. of the residue. (viii) [Determination of Iron (Atomic Absorption

Spectrophotometric method)] [Inserted by S.O. 534(E) 20th July, 1992.](a)Reagents. -(1)Standard Iron Solution (1000 ppm.) - - Weigh accurately 1 gm. pure Iron wire and put it in approximately 30 ml. of 6N HCl in a beaker and boil. Transfer it to one litre volumetric flask through the funnel giving several washings to the beaker and funnel with glass distilled water. Make the volume up to the mark. Stopper the flask and shake the solution well. This is 1000 ppm Iron solution.(2)Glass distilled or demineralised water of pH 2.5 ± 5.0 .(3)Preparation of working standards. - Pipette 10 ml. Iron stock solution in 100 ml. volumetric flask and dilute to volume. This is 100 ppm Iron solution. Pipette the following volumes of 100 ppm Iron solution in 50 ml. volumetric flask and make the volume with acidified water.

Flask No.	Volume of 100 ppm standard Iron solution	Concentration of Iron after making volume 50 ml.(ppm)
1	0.0	0.0
2	1.0	2.0
3	2.0	4.0
4	3.0	6.0
5	4.0	8.0
6	5.0	10.0
7	6.0	12.0
8	7.0	14.0
9	8.0	16.0
10	9.0	18.0
11	10.0	20.0

Stopper the flask and shake the solution well.(b)Procedure. -(1)Preparation of fertiliser sample solution. - Weigh exactly 1 gm. of the material on a clean watch glass and transfer it to a one volumetric flask through the funnel giving repeated washing with acidified water and dissolve the material by shaking well. Make the volume up to the mark with acidified water and shake well and filter through. Whatman No. 42 filter paper in dry clean flask. The flask should be rinsed with a 10 - 15 ml. of filtrate and then continue filtration.(2)Flaming the solution. - Flame the standards and the filtered sample on Atomic Absorption Spectrophotometer at a wavelength of 248.3 nm. using clean air acetylene flame.Calculations : Prepare a standard curve of known concentrations of Iron solution by plotting the absorbance value of Y - axis against the respective iron concentration on X - axis: Determine the concentration of Iron in the sample solution from the graph.Total Iron (as Fe) per cent = $X/10$ Where X= Concentration of Iron (in ppm) obtained from the standard curve.Note. - In case a sample has been analyzed by both the methods. viz. indicated under the heading '7' and '8' the result obtained by the method indicated under the heading '8' shall prevail.

9. Method of analysis of manganese sulphate, agricultural grade -

(i)Quality of ReagentsUnless specified otherwise, pure chemicals and glass distilled or demineralised water shall be used in tests.Note. - "Pure chemicals" means chemicals that do not contain impurities which affect the results of analysis. "Demineralised water" means the water obtained after passing distilled water through a cation and an anion exchange resins or a combined

cation anion exchange resin.(ii)Determination of Manganese(a)Reagents :(1)Standard Manganese Solution. - Weigh 3.0763 gms. of manganese sulphate ($\text{MnSO}_4 \cdot \text{H}_2\text{O}$) - A.R. grade) on a clean watch glass and transfer it to one litre flask through the funnel giving several washings to watch glass and funnel with acidified water and make the volume up to the mark. This solution will be 100 ppm Mn. A secondary dilution of 5 ml. to 100 ml. with acidified water gives 1 50 ppm working standard.(2)Glass distilled or demineralised water of pH 2.5 ± 0.2 - Dilute 1 ml. of 10 per cent sulphuric acid to one litre with glass distilled or demineralised water and adjust the pH to 2.5 with a pH meter using 10% H_2SO_4 of NaOH. This solution is called acidified water.(3)Preparation of working standard. - Pipette the following volume of working standard solution in 50 ml. numbered volumetric flasks and make the volume with acidified water.

Flask No.	Volume of working standard taken (ml.)	Concentration of manganese after making volume to 50 ml. (ppm)
1	0.0	0.0
2	0.5	5.0
3	1.0	10.0
4	1.5	15.0
5	2.0	20.0
6	2.5	25.0
7	3.0	30.0
8	3.5	35.0
9	4.0	40.0

Stopper the flasks and shake them well. Prepare the standard in duplicate. The same acidified water should be used for preparing the solution of unknown fertiliser samples. Fresh standards should be prepared every time when a fresh lot of acidified water is prepared.(b)Procedure. -(1)Preparation of manganese sulphate fertiliser samples. - Weight exactly 0.4 gm. of the material on the clean watch glass and transfer it to a one litre volumetric flask through the funnel giving repeated washings with acidified water and dissolve the material by shaking well. Make the volume up to the mark with acidified water and shake well.(2)Take 5 ml. of the prepared solution in 250 ml. volumetric flask and make the volume with acidified water. Shake the solution well and filter through What man No. 42 filter paper in dry clean flasks. The flasks should be rinsed with a 10 to 15 ml. of the filtered and then continue filtration.(3)Flaming the solution. - Flame the standards and the filtered samples on atomic absorption spectrophotometer at a wavelength of 279.5 mμ (Mn line of the instrument).Calculation :Prepare a standard curve to known concentration of manganese solution by plotting the absorbance value on Y - axis against their respective manganese concentrations on X - axis. Calculate the percentage manganese in manganese fertiliser by multiplying manganese concentration value calculated from the standard curve by 12.5.(b)Precaution :(1)Weighing must be done accurately with a precision balance.(2)All the glass apparatus to be used should be conning/pyrex make and washed with dilute hydrochloric acid (1:4) and washed thoroughly with distilled and then with demineralised water.(3)The Pipette should be rinsed with the same solution to be measured.(4)The outside of the pipette should be wiped with filter paper after pipetting the solution to be measured.(5)After using the pipette place it on a clear dry filter paper, in order to

prevent contamination.(6)To start filtration, only a few drops should be added first in order to wet the filter paper and then continue further filtration.

10. Alternative method of analysis of manganese sulphate agricultural grade. -

(i)Quality of ReagentsUnless specified otherwise, pure chemicals and glass distilled or demineralized water shall be used in tests.Note. - "Pure Chemicals" means chemicals that to not contain impurities which affect the results of analysis."Demineralised water" means the water obtained after passing distilled water through a cation and anion exchange resins or a combined cation - anion exchange resin.(ii)Determination of manganese(a)Reagents :(1)Hydrogen peroxide (H_2O_2) - 30 per cent.(2)Nitric acid (HNO_3) - Concentrated.(3)Orthophosphoric acid (H_3PO_4 - From steel industry) - 85 per cent.(4)Potassium periodate (KIO_4)(5)Glass distilled or mineralised water of pH 2.5 ± 0.5 Dilute 1 ml. of 10 per cent H_2SO_4 to one litre with glass distilled or demineralised water and adjust the pH to 2.5 with a pH meter using 10% H_2SO_4 or NaOH.(6)Standard Manganese solution. - Weigh 3.0763 gms. of manganese sulphate ($MnSO_4 \cdot H_2O$ - A.R. grade) on a clear watch glass and transfer it to one litre flask through the funnel giving several washings to the watch glass and the funnel with acidified water and make the volume up to the mark. This solution will be 1000 ppm Mn. A secondary dilution of 5 ml. to 100 ml. with acidified water gives a 50 ppm working standard.(7)Preparation of working standards. - Pipette 0.0, 0.5, 1.0, 1.5, 2.0, 2.5, 3.0, 3.5, 4.5 and 5.0 ml. of standard solution of manganese in a series of 11 different 100 ml. beakers. Evaporate the solutions to dryness at low temperature on a hot plate or steam bath. Add 5 ml. of HNO_3 and 2 ml. of 30% H_2O_2 Cover the beakers with watch glass and digest the contents on a steam bath or hot plate for 30 minutes. Remove the glass cover and evaporate the contents to dryness. Cool the beakers, add 2 ml. of HNO_3 5ml. of phosphoric acid and heat till boiling. Again cool and mix the contents with 10 ml. of demineralised water. Add 0.3 of KIO_4 and heat the solutions on water bath until pink colour appears. Add about 20 ml. water and heat for about 40 minutes or until there is no more increase in colour intensity. Cool the beakers to room temperature, transfer the solutions to 50 ml. volumetric flasks. Make the volume with deionised water and shake thoroughly. The concentration of manganese ion will be as follows :

Flask No.	Volume of working standard taken (ml.)	Concentration of manganese after making volume to 50 ml. (ppm)
1	0.0	0.0
2	0.5	0.5
3	1.0	1.0
4	1.5	1.5
5	2.0	2.0
6	2.5	2.5
7	3.0	3.0
8	3.5	3.5
9	4.0	4.0

10	4.5	4.5
11	5.0	5.0

Measure the light transmittance at 540 mμ (green filter). Make the standard curve by plotting per cent transmittance against the concentration of manganese. (b) Procedure. - (1) Analysis of manganese sulphate fertiliser sample. - Weigh exactly 0.500 gm. of the material on a clean watch glass and transfer it to a one liter volumetric flask through the funnel giving repeated washings with acidified water and dissolve the material by shaking well. Make the volume up to the mark with acidified water and shake well. Pipette one ml. solution in a 100 ml. pyrex/ corning beaker and proceed further as described above for preparation of standard curve and make the volume to 50 ml. The standard and the test samples should be prepared and processed for estimation at the same time. Calculation : The percentage of manganese in the manganese sulphate fertiliser sample is calculated by multiplying manganese concentration value calculated from standard curve by 10. [11.

Method of analysis of borax (sodium tetraborate decahydrate fertiliser)] [Sub - headings 11 to 16 inserted by G.S.R 1160(E), dated 12th October 1986 (w.e.f. 21st October, 1986).] (i) Quality of reagents Unless specified otherwise, pure chemicals & glass distilled water shall be used in tests. Note : Pure chemicals means chemicals that do not contain impurities which affects the results of analysis. (ii) Determination of boron : (A) Atomic Absorption spectrophotometer method : (a) Reagents : (1) Standard Boron Solution. - Weigh 44.095 gms. of sodium tetraborate decahydrate ($\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$) - A.R. grade on clean watch glass and transfer it to one litre volumetric flask through the funnel giving several washings to watch glass and funnel with glass distilled water. Make the volume up to the mark. Stopper the flask and shake the solution well. This is 5000 ppm boron solution hereinafter called standard A. This solution should be stored in clean bottle for further use. (2) Preparation of working standard. - Pipette the following volume of standard A in 50 ml. numbered volumetric flasks and make the volume with water.

Flask No.	Volume of standard A taken (ml.)	Concentration of boron after making volume to 50ml. (ppm)
1.	0.0	0.0
2.	4.0	400
3.	6.0	600
4.	8.0	800
5.	10.0	1000
6.	12.0	1200
7.	14.0	1400
8.	16.0	1600

(b) Procedure. - (1) Preparation of sample solution. - Weigh 2.500 gms. of the material on a clean watch glass and transfer it quantitatively in a 250 ml. volumetric flask through the funnel giving repeated washing with water. Make up the volume stopper the flask and shake well. (2) Flaming the solution. - Flame the standards and the sample solution on atomic absorption spectrophotometer at a wavelength of 249.8 nm using nitrous oxide-acetylene flame. Calculations : Prepare a standard curve of known concentrations of boron solutions by plotting the absorbance value on Y - axis against their respective boron concentration on X - axis. Calculate the percentage boron in the given sample by multiplying boron concentration value obtained from standard curve by 0.01. Example

:Weight of the fertiliser sample = 2.50 gm. Volume made = 250 ml. Dilution factor $250/2.5 = 100$. Reading of sample from atomic absorption = Y. Corresponding value of boron from standard curve against Y absorbance = X ppm. Percentage of boron in the sample = $0.01X$. (B) Titrametric method : (a) Outline of the method : Borax is determined by first converting it to boric acid with hydrochloric acid and then titrating against Sodium hydroxide solution after complexing boric acid with mannitol or sorbitol. (b) Reagents : (1) Standard hydrochloric acid - 0.5 N. (2) Standard sodium hydroxide - 1.0 N. (3) Methyl red indicator - Dissolve 0.1 gm. of the material in 60 ml. of rectified spirit and dilute with water to 100 ml. (4) Phenolphthalein indicator - Dissolve 1 gm. of the material in 100 ml. of rectified spirit. (5) Mannitol or sorbitol. (c) Procedure : (1) Dissolve 3.0 gm. of the material accurately weighed in 60 ml. of water and titrate with hydrochloric acid, using methyl red solution as indicator. (2) Boil and cool the solution. (3) Add 20 gm. of mannitol or sorbitol, and titrate with sodium hydroxide, using phenolphthalein solution as indicator. Calculation : Boron content per cent by Weight = $1.0819 \frac{V}{W} \frac{N}{V}$ where V = Volume in ml. of standard sodium hydroxide used. N = Normality of sodium hydroxide, and W = Weight in gm. of the material taken for the test. (Ref - ISI - 110 - 1980) (iii) Determination of matter insoluble in water : Same as method No. 8(viii) (a); (iv) Determination of pH : Dissolve 3.8 gm. of material in water and make it to 100 ml. Measure the pH value of the solution with the help of suitable pH meter, using glass electrodes. (v) Determination of lead : Same as method No. (v) Substitute Zinc sulphate by borax.

12. Method of analysis of copper sulphate fertiliser ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$)

(i) Quality of reagents : Unless specified otherwise pure chemicals and glass distilled or demineralised water shall be used in tests. Note. - "Pure chemicals" means chemicals which do not contain impurities which affect the result of analysis. Demineralised water, means the water obtained after passing distilled water through a cation and anion exchange resin or a combined cation anion exchange resin. (ii) Determination of copper : (A) Atomic absorption Spectrophotometer method (a) Reagents : As specified in 8(iv)(a), (1), (2) and (3). (b) Procedure. - (1) Weigh 0.25 gm. of the material on a clean watch glass and transfer it to one litre volumetric flask through the funnel giving repeated washing with glass distilled water. Add one ml. of 10 per cent sulphuric acid and make up the volume. (2) Take 5 ml. of the prepared solution in 500 ml. volumetric flask and make up the volume. Shake the solution well and filter through Whatman No. 42 filter paper in dry clean flask. The flask should be rinsed with a 10 to 15 ml. of the filtrate and then continue filtration. (3) Flaming the solution : Flame the standards and the filtered samples on an atomic absorption spectrophotometer at a wavelength of 324.8 nm. using air - acetylene flame. Calculation : Prepared a standard curve of known concentrations of Copper solution by plotting the absorbance value on Y - axis against their respective concentration values on X - axis : Per cent, copper in sample = $10 \frac{x}{w}$ where W = Weight in gm. of the material taken for test. X = Concentration of copper (in ppm) as determined from the graph. (B) By Iodometric Titration method : (a) Outline of the method : Copper is determined with the addition of potassium iodide and titrating the liberated iodine against standard sodium thio - sulphate solution. (b) Reagents : (1) Sodium Carbonate - A.R. Grade. (2) Potassium Iodide - A.R. - Crystals. (3) Acetic Acid - A.R. Grade. (4) Standard Sodium Thiosulphate solution. - 0.1 N. (5) Starch indicator solution. - Triturate 5 gm. of starch and 0.01 gm. of mercuric iodide with 30 ml. of cold water and slowly pour it with stirring into one litre of boiling water. Boil for three minutes. Allow to cool and decant off the supernatant clear liquid. (6) Potassium

thiocyanate crystals.(c)Procedure. -(1)Dissolve about 1 gm. of the test sample (accurately weighed), in 50 ml. of water.(2)Add a pinch of sodium carbonate till a slight turbidity appears. Then add 5 ml. of acetic acid, 3 gms. of potassium iodide and titrate the liberated iodine with sodium thiosulphate solution, using starch as an indicator, until only a faint blue colour remains.(3)Add about 2 gms. of potassium thiocyanate, shake and continue the titration until the blue colour disappears.Calculation:

Copper per cent mass = $6.35 \frac{V.N.}{M}$

where V = Volume in ml. of standard sodium thiosulphate solution. N = Normality of standard thiosulphate solution, and M = Mass in gm. of the sample taken for the test.[Ref. IS - 261(1982)].(iii)Determination of lead (Pb) - Same as method No. 8(v). Substitute zinc sulphate by copper sulphate.(iv)Determination of soluble Iron and Aluminium compounds (expressed as Fe) : (a)Outline of the method - iron and aluminum are determined gravimetrically by precipitation ammonium hydroxide.(b)Reagents :

1. Concentrated nitric acid - A R grade.

2. Ammonium Chloride - A R grade.

3. Dilute Ammonium Hydroxide - approximately 15 per cent NH₃(m/v).

4. Dilute hydrochloric acid - 33 per cent (m/v).

(c)Procedure : (1)Take 10 gms of the test sample and add 25 ml. of water, 2 ml. of nitric acid and 5 gms. of ammonium chloride.(2)Make the solution alkaline by adding ammonium hydroxide solution.(3)Keep it on a water bath until the precipitate has flocculated, keeping the solution alkaline by the addition of more ammonium hydroxide, if necessary.(4)Filter and wash the residue with dilute ammonium hydroxide.(5)Dissolve the residue in hot dilute hydrochloric acid.(6)Make the solution again alkaline by adding ammonium hydroxide and allow the precipitate to settle.(7)Filter and wash residue with water.(8)Dry the residue, ignite and weigh till a constant mass is obtained.Calculation :

Soluble iron and aluminum compounds (as Fe) percent by mass = $70 \frac{M_1}{M_2}$

where M₁ = Mass in gm. of the residue obtained, and M₂ = Mass in gm. of the sample taken for the test. (Ref. : IS : 261 = 1982)(v)Determination of matter insoluble in water : (a)Reagents

:(1)Concentrated sulphuric acid. - A R Grade(b)Procedure. -Weigh accurately about 10 gms. of the test sample and dissolve in 100 ml. of water, Add 3 ml. of sulphuric acid and stir thoroughly at room temperature. Filter through a tared filter paper or tared Gooch or Sintered glass crucible (G. No. 4). Wash the residue with water till it is free from acid. Dry the filter paper or crucible in an oven maintained at a temperature of 105 to 110°C till constant mass is obtained.Calculation :

Insoluble matter, per cent by mass = $\frac{M_1}{M_2} \times 100$

where M₁ = Mass in gm. of the residue obtained for the test. M₂ = Mass in gm. of the material taken for the test.(vi)Determination of pH:(a)Procedure :Dissolve 5 gm. of the test sample in water and

make up the volume to 100 ml. Determine the pH with glass electrodes using a suitable pH meter.

13. Method of analysis of ferrous sulphate (heptahydrate) $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$

(i) Quality of reagents: Unless specified otherwise, pure chemicals and glass distilled or demineralised water shall be used in tests. Note. - "Pure Chemicals" means chemicals that do not contain impurities which affect the results of analysis. Demineralised water means the water obtained after passing distilled water through a cation and an anion exchange resin or a combined cation - anion exchange resin. (ii) Determination of ferrous iron : (a) Outline of the method - Ferrous iron content is determined by titration with standard potassium permanganate solution. (b) Reagents :

1. Dilute Sulphuric acid - 4 N approximately.

2. Standard Potassium permanganate solution. - 0.1 N.

3. Orthophosphoric acid.

(c) Procedure.

1. Weigh accurately about 5 gm. of the prepared sample and dissolve it in water.

2. Add 1 to 2 ml. of dilute sulphuric acid and make up the solution to 250 ml. in a volumetric flask.

3. Pipette out exactly 50 ml. of the solution in a flask.

4. Add 10 ml. of dilute sulphuric acid and 2 ml. of Orthophosphoric acid and titrate with standard potassium permanganate solution.

Calculation :

Ferrous iron, per cent by weight = $27.92 \frac{V}{W} \times N$

Where V = Volume of standard potassium permanganate used. N = Normality of standard potassium permanganate solution. W = Weight in gm. of the material taken for the test. (Ref. : IS : 262 -

"1982) (iii) Determination of total iron (A) Titrametric method using potassium dichromate

:(a) Outline of the method : Total iron is determined by reducing ferric iron with stannous chloride and then titrating the total ferrous iron with standard potassium dichromate solution. (b) Reagents

:(1) Concentrated hydrochloric acid. (2) Stannous chloride solution. - Dissolve 6.0 gms. of stannous chloride crystals ($\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$) in 60 ml. of concentrated hydrochloric acid and dilute with water to 100 ml. Keep this solution in stoppered bottle. (3) Mercuric chloride solution. -

saturated.(4)Concentrated sulphuric acid.(5)Diphenylamine indicator - Dissolve 0.1 gms. of Diphenylamine in 100 ml. of concentrated sulphuric acid.(6)Standard potassium dichromate solution. - 1.0 N.(c)Procedure: -(1)Weigh accurately about 5 gm. of the prepared sample and dissolve it in water.(2)Add 1 to 2 ml. of the dilute sulphuric acid and make up the volume to 250 ml. in volumetric flask.(3)Take 50 ml. aliquot of this solution and add 100 ml. of concentrated hydrochloric acid and heat to boiling.(4)Reduce the iron by adding stannous chloride solution until the yellow colour of the ferric chloride disappears. Do not add more than 2 to 3 drops of stannous chloride in excess.(5)Cool the solution, add rapidly mercuric chloride solution and stir so that a white precipitate of mercurous chloride forms slowly, indicating that a slight excess of stannous chloride is present.(6)Allow to stand for 5 to 10 minutes, dilute to 150 to 200 ml. and add 5 ml. of phosphoric acid and 5 drops of diphenylamine indicator. Titrate with standard potassium dichromate solution until the colour of the solution changes to a deep blue which does not fade on stirring.Calculation :

Total iron, per cent by weight = $27.92 \frac{V}{W} \times N$

Where, V = Volume in ml. of potassium dichromate used in titration. N = Normality of standard potassium dichromate solution, and W = Weight in gm. of the material present in the aliquot.(Ref. : IS 262 - 1967)(B)Atomic Absorption Spectrophotometer Method:(a)Reagents :(1)Standard Iron Solution. - (1000 ppm) - Weigh accurately 1 gm. pure iron wire and put it in approximately 30 ml. 6N HCl, in a beaker and boil. Transfer it to one litre volumetric flask through the funnel giving several washings to the beaker and funnel with glass distilled water. Make the volume up to the mark. Stopper the flask and shake the solution well. This is 100 ppm iron solution.(2)Glass distilled or mineralised water of pH 2.5 ± 0.5 .(3)Preparation of working standards. - Pipette 10 ml. iron stock solution in 100 ml. volumetric flask and dilute to volume. This is 100 ppm iron solution. Pipette the following volumes of 100 ppm iron solution in 50 ml. numbered volumetric flask and make the volume with acidified water.

Flask No.	Volume of 100 ppm standard iron solution taken(ml.)	Concentration of iron after making volume to 50ml. (ppm)
1	2	3
1.	0.0	0.0
2.	1.0	2.0
3.	2.0	4.0
4.	3.0	6.0
5.	4.0	8.0
6.	5.0	10.0
7.	6.0	12.0
8.	7.0	14.0
9.	8.0	16.0
10.	9.0	18.0
11.	10.0	20.0

Stopper the flask and shake the solution well.(b)Procedure.(1)Preparation of ferrous sulphate fertiliser sample: Weigh exactly 1.000 gm. of the material on a clean watch glass and transfer it to a one litre volumetric flask through the funnel giving repeated washing with acidified water and

dissolve the material by shaking well. Make the volume up to the mark with acidified water and shake well.(2)Take 5 ml. of the prepared solution in 100 ml. volumetric flask and make up the volume with acidified water. Shake the solution well and filter through Whatman No. 42 filter paper in dry clean flasks. The flasks should be rinsed with a 10 to 15 ml. of the filtrate and then continue filtration.(3)Flaming the solution. - Flame the standards and the filtered sample on atomic absorption spectrophotometer at a wavelength of 248.3 nm using clean air acetylene flame.Calculation :Prepare a standard curve of known concentrations of iron solutions by plotting the absorbance value on Y - axis against their respective iron concentrations on X - axis. Determine the concentration of iron in the sample solution from the graph.Total iron = $2X/W$ Where, X= Concentration of Fe (in ppm) obtained from the standard curve. W = Weight in gm. of the material taken for the test.(iv)Determination of ferric iron :Subtract the value of ferrous iron per cent from total iron per cent to obtain the Ferric iron per cent in the sample.(Ref. IS : 262 - 1967)(v)Determination of pH :Dissolve 5.0 gms. of the sample in 100 ml. of freshly boiled and cooled water and determine the pH by means of a pH meter using glass electrode.(vi)Determination of matter insoluble in water:(a)Procedure :Dissolve 25.0 gms. of the material in 125 ml. of water. Filter through a weighed and prepared Gooch crucible or sintered glass crucible (G.No. 4) and wash the residue thoroughly with water. Dry the crucible at $100^{\circ} \pm 81^{\circ}$ to constant mass.Calculation:Matter insoluble in water per cent by weight = $4A$. Where A = Weight in gm. of the residue.Note. - In case a sample has been analysed by both the methods, viz. indicated under the heading '7' and '8' the result obtained by the method indicated under the heading '8' shall prevail.(vii)Determination of lead :(a)Reagents :(1)Standard Lead Solutions. - Weight 0.1599 gm. of lead nitrate $[Pb(NO_3)_2]$ on a clean watch glass and transfer it to one litre flask through the funnel giving several washings to watch glass and funnel with glass distilled or demineralised water. Add 10 ml. of concentrated distilled nitric water Add 10 ml. of concentrated distilled nitric acid and make the volume up to the mark. Stopper the flask and shake the solution well. This is 10 ppm lead solution and should be stored in a clean bottle for further use. Dilute 10 ml. of 100 ppm solution of lead to 100 ml. with 1 per cent nitric acid solution to get 100 ppm standard lead solution.(2)1 per cent nitric acid solution. - Dilute 10 ml. of concentrated distilled nitric acid to one litre with glass distilled water.(3)20 per cent zinc sulphate solution. - Weigh 20 gms of zinc sulphate $(ZnSO_4 \cdot 7H_2O)$ and dilute to 100 ml. with 1 per cent nitric acid solution.(4)Preparation of working standard. - Pipette the following volume of 10 ppm standard lead solution in ml. numbered volumetric flask. Add 5 ml. of 20 per cent zinc sulphate solution to each flask and make the volume with 1 per cent nitric acid solution.

Flask No.	Volume of 10 ppm lead solution taken (ml.)	Volume of 20% zinc sulphate solution added (ml.)	Concentration of lead after making the volume to 50 ml. (ppm) ¹
1	0.0	5.0	0.0
2	2.0	5.0	0.4
3	4.0	5.0	0.8
4	6.0	5.0	1.2
5	8.0	5.0	1.6
6	10.0	5.0	2.0

Stopper the flask and shake them well.(b)Procedure. -(1)Preparation of zinc sulphate fertiliser samples. - Weigh 1 gm. of the material on a clean watch glass and transfer to 50 ml. volumetric flask

through the funnel giving washings with 1 per cent nitric acid solution. Dissolve the material and make the volume with 1 per cent nitric acid solution. Samples should be prepared in duplicate.(2)Flaming the solution. - Flame the standards and the samples on atomic absorption spectrophotometer at a wavelength of 217 mu (Lead line of the instrument).Calculations: Prepare a standard curve of known concentrations of lead solution by plotting the absorbance values on Y - axis against their respective lead concentration on X - axis. Calculate the percentage lead in zinc fertilizer by multiplying lead concentration value calculated from standard curve by 0.005.Substitute zinc Sulphate by ferrous sulphate.

14. Method of analysis of ammonium molybdate $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24}\cdot 4\text{H}_2\text{O}$

(i)Quality of reagents :Unless specified otherwise, pure chemicals and glass distilled or demineralised water shall be used in tests :Note. - "Pure Chemicals" means chemicals that do not contain impurities which affect the result of analysis. "Demi neralised Water" means the water obtained after passing distilled water through a cation and anion exchanges resins or a combined cationanion exchange resin.(ii)Determination of molybdenum:(A)Atomic Absorption spectrophotometer method :(a)Reagents :(1)Potassium Sulphate. - Dissolve 25 gm. of AR Potassium sulphate in 1 litre of distilled water.(2)Standard molybdenum solution. - Weigh 1.5 gm. of molybdenum trioxide (MoO_3) and transfer it to one litre volumetric flask through the funnel giving several washings to beaker and funnel with glass distilled. Make up the volume up to the mark. This is 1000 ppm standard molybdenum solution.(3)Preparation of working standards. - Pipette the following volume of 1000 ppm standard Mo solution in 100 ml. numbered volumetric flasks. Add 20 ml. of potassium sulphate solution to each flask and make up the volume to 100 ml. Stopper the flask and shake them well.

Flask No.	Volume of 1000 ppm Mo Solution taken (ml.)	Volume of Potassium sulphate added (ml.)	Concentration of molybdenum after making the volume to 100 ml.(ppm)
1.	0.0	20.0	0
2.	2.0	20.0	20
3.	3.0	20.0	30
4.	4.0	20.0	40
5.	5.0	20.0	50
6.	6.0	20.0	60

(b)Procedure. -(1)Preparation of Ammonium Molybdate fertiliser sample : Weigh exactly 0.1 gm. of the material on the clean watch glass and transfer it to a 100 ml. volumetric flask through funnel giving repeated washing and dissolve the material by shaking well. Make the volume upto the mark.(2)Take 10 ml. of the prepared solution in a 100 ml. volumetric flask, add 20 ml. of potassium sulphate solution and make the volume upto the mark.(3)Flaming the solution : Flame the standards and the filtered samples on an atomic absorption spectrophotometer at a wavelength of 313.3 nm using nitrous oxideacetylene flame.Calculation:Prepare a standard curve of known concentration of molybdenum solution by plotting the absorbance value on Y - axis against their respective molybdenum concentration on X - axis.per cent of Mo = $\frac{X}{10W}$ Where X =Concentration of molybdenum (ppm) as obtained from the calibration curve. W= Weigh in gm. of the material

taken for the test. (B) Calorimetric Method for Determination of Molybdenum : (a) Outline of Method : Molybdenum (vi) in acid solution when treated with stannous chloride (best in the presence of a little ferrous ion) is converted largely into molybdenum (v). This forms a complex with thiocyanate ion, probably largely $\text{Mo}(\text{SCN})_5$, which is red in colour. The latter may be extracted with solvents possessing donor oxygen atoms (3 - Methylbutanol is preferred). The colour depends upon the acid concentration (optimum concentration 1M) and the concentration of thiocyanate ion (K 1.0 per cent but colour intensity is constant in the range 2 - 10 per cent); it is little influenced, by excess of stannous chloride. The molybdenum complex has maximum absorption at 465 nm. (b) Apparatus :

1. Photometer. - Any suitable photoelectric colorimeter

(c) Reagents :

1. Standard molybdenum solution. - (0.001 per cent molybdenum) - Weigh 0.184 of AR Ammonium molybdate $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}$ on a clean watch glass and transfer it to one litre volumetric flask through the funnel giving several washings to watch glass and funnel with glass distilled water. Make the volume upto the mark. Pipette out 10 ml. of this solution into 100 ml. volumetric flask and make the volume upto the mark, This gives 0.001 per cent Mo solution.

2. Ferrous Ammonium Sulphate. - Dissolve 10 gm. of AR ferrous ammonium sulphate salt in 100 ml. of every dilute H_2SO_4 .

3. Stannous chloride solution. - Dissolve 10 gm. of AR stannous chloride dihydrate in 100 ml. of 1 M - hydrochloric acid.

4. Potassium thiocyanate solution - Dissolve 10 gm. of AR potassium thiocyanate salt in 100 ml. of distilled water.

5. Iso - amly alcohol.

(d) Preparation of Sample : Weigh 0.1 gm. of sample on a clean watch glass and transfer it to one litre volumetric flask, through the funnel giving several 'washings to watch glass and funnel with glass distilled water. Make the volume up to the mark. Pipette out 5 ml. of this solution into a 100 ml. of volumetric flask and make up the volume up to the mark. (e) Procedure. - (1) Place 1.0, 2.0, 3.0, 4.0 and 5.0 ml. of the 0.001 per cent molybdenum solution (containing 0.01 mg., 0.02 mg., 0.03 mg., 0.04 mg. and 0.05 mg. molybdenum) severally in 50 ml. capacity separating funnels and diluting each with an equal volume of water. (2) Add to each funnel 12.0 ml. of conc. Ha. 1.0 ml. of ammonium ferrous sulphate and 3.0 ml. of the potassium thiocyanate solution. (3) Shake gently and then introduce 3.0 ml. of the stannous chloride solution. (4) Add water to bring the total volume in each

separating funnel to 25 ml. and mix.(5)Pipette 10 ml. of redistilled 3 - methylbutanol (iso - amyl alcohol) into each funnel and shake individually for 30 seconds.(6)Allow the phases to separate and carefully run out the lower aqueous layer.(7)Remove the glass stopper and pour the alcoholic extract through small plug of purified glass wool in a small funnel and collect the organic extract in a 1.0 cm. absorption cell.(8)Measure the absorbance at 465 nm in spectrophotometer against a 3 - methylbutanol blank.(9)Plot absorbance against concentration of standard molybdenum solutions and draw and draw the calibration curve.(10)Take 10 ml. of the sample solution and determine to absorbance of it by subjecting it to the same treatment as the standard solutions using calibration curve, determine the corresponding concentration of molybdenum in the sample solution.Calculation :% Mo = $2000X / Y.W$. Where Y= Volume of sample solution taken for the test.W= Weight in gm. of the material taken for the preparation of the sample solution. (Ref. : Vogel's Text Book of Quantitative Analysis).(iii)Determination of matter insoluble in water : (a)Procedure :Dissolve 25.0 gm. of the material in 125 ml. of water. Filter through a weighed and prepared Gooch crucible of sintered glass crucible (G.No. 4) and wash the residue thoroughly with water. Dry the crucible at 110°C to constant mass.Calculation :Matter insoluble in water percent by weight= $4A$ Where A =Weight in gm. of the residue.(iv)Determination of lead : (a)Reagents : (1)Standard Lead Solutions. - Weigh 0.1599 gm. of lead nitrate ($Pb(NO_3)_2$) on a clean watch glass transfer it to one litre flask through the funnel giving several washings to watch glass and funnel with glass distilled or demineralised water. Add 10 ml. of concentrated distilled nitric water. Add 10 ml. of concentrated distilled nitric acid and make the volume up to the mark. Stopper the flask and shake the solution well. This is 100 ppm lead solution and should be stored in a clean bottle for further use. Dilute 10 ml. of 100 ppm solution of lead to 100 ml. 1 per cent nitric acid solution to get 10 ppm standard lead solution.(2)1 per cent nitric acid solution. - Dilute 10 ml. of concentrated distilled nitric acid to one litre with glass distilled water.(3)20 per cent zinc sulphate solution. - Weigh 20 gms. of zinc sulphate ($ZnSO_4 \cdot 7H_2O$) and dilute to 100 ml. with 1 per cent nitric acid solution.(4)Preparation of working standards. - Pipette the following volume of 10 ppm standard lead solution in ml. numbered volumetric flask. Add 5 ml. of 20 per cent zinc solution to each flask and make the volume with 1 per cent nitric acid solution.

Flask No.	Volume of 10 ppm Lead solution taking (ml.)	Volume of 20% zinc sulphate solution added (ml.)	Concentration of lead after making the volume to 50 ml.(ppm).
1	0.0	5.0	0.0
2	2.0	5.0	0.4
3	4.0	5.0	0.8
4	6.0	5.0	1.2
5	8.0	5.0	1.6
6	10.0	5.0	2.0

Stopper the flask and shake them well;.(b)Procedure. - (1)Preparation of zinc sulphate fertiliser samples. - Weigh 1 gm. of the material on a clean watch glass and transfer to 50ml. volumetric flask through the funnel giving washings with 1 per cent nitric acid solution. Dissolve the material and make the volume with 1 per cent nitric acid solution. Samples should be prepared in duplicate.(2)Flaming the solution. - Flame the standards and the samples on atomic absorption

spectrophotometer at a wavelength of 217 mμ (Lead line of the instrument.)(3)Calculations. - Prepared and standard curve of known concentrations of lead solution by plotting the absorbance values on Y - axis against their respective lead concentration on X - axis. Calculate the percentage lead in zinc fertilizer by multiplying lead concentration value calculated from standard curve by 0.005. Substitute zinc sulphate by ammonium molybdate.

15. Method of analysis of chelated zinc (As Zn - EDTA)

(i)Reagents :(a)EDTA Solution. - (0.05M) - Dissolve 18.612 gm. of disodium ethylene diamine tetraacetate dehydrate (EDTA) in distilled water and make up the volume to 1 litre.(b)Standard Zinc solution (1000 ppm) : Weight accurately 1.0 gm. of zinc metal in a beaker. Add 20 ml. HCL (1:1). Keep it for few hours and allow it to dissolve completely. Transfer the solution to 1 litre volumetric flask. Make the volume up to the mark.(c)Concentrated Ammonia solution (sp. gr. 0.88).(d)Ammonium Nitrate. - AR grade salt.(e)Buffer solution (pH - 10) - Dissolve 8.0 gm. AR grade ammonium nitrate in 65 ml. of water and add 35 ml. of concentrated ammonia solution (sp. gr. - 0.88)(f)Eriochrome black (T) indicator mixture - Mix thoroughly 1 gm. of eriochrome black (T) indicator with 100 gms. of AR grade potassium nitrate.(g)Hydroxylamine Hydrochloride - AR grade.(h)Potassium cyanide - AR grade (to be used with extreme care) 15 per cent. aq.solution.(i)Manganese sulphate solution.Dissolve 11.15 gm. of AR grade manganese sulphate in 1 litre of distilled water.(j)Sodium Fluoride AR grade.(ii)Preparation of sample solution :Weigh accurately 1.0 gm. of the sample and transfer it to 100 ml. volumetric flask. Make up the volume with distilled water. Keep it overnight.(iii)Procedure :(a)Standardization of EDTA solution.(1)Take 10 ml. of zinc solution (standard).(2)Dilute it by adding 30 ml. distilled water.(3)Add 10 ml. of buffer solution and 30 - 40 mg. of indicator mixture.(4)Titrate with EDTA solution till clear blue end point is obtained. Note the volume of EDTA used as V₁ ml.(b)Standardisation of manganese sulphate solution.(1)Take 25 ml. of manganese sulphate solution.(2)Dilute it by adding 100 ml. distilled water.(3)Add 0.25 gm. of hydroxylamine hydrochloride and 10 ml. of buffer solution.(4)Add 30 - 40 mg. of indicator mixture.(5)Titrate with EDTA solution till clear blue end point is obtained. Note the volume of EDTA used as V₂ ml.(c)Determination of EDTA content of Zn - EDTA fertiliser.(1)Take 10 ml. of sample solution.(2)Dilute it by adding 100 ml. of distilled water.(3)Add 0.25 gm. of hydroxylamine hydrochloride.(4)Add 10 ml. of buffer solution and 30 - 40 mg. of indicator mixture.(5)Warm to 40° and titrate with standard EDTA solution (Preferably stirring magnetically) to clear blue end point. Note the volume of EDTA used as V₃ ml.(6)After the end point add 2.5 gms. of sodium fluoride and stir for one minute.(7)Titrate the solution with standard manganese sulphate solution, slowly, till a permanent red colour is obtained. Note the volume of manganese sulphate added as V₄ ml.(8)Stir for 1 minute.(9)Titrate the excess of manganese ions with EDTA solution until the colour changes to pure blue. Note the volume of EDTA used as V₅ ml.(10)After the second end point (step 9) add 4 - 5 ml. of 15 per cent aqueous potassium cyanide solution.(11)Titrate it with manganese sulphate solution till colour changes sharply from blue to red. Note the volume of manganese sulphate solution added as V₆ mlCalculation:Molarity of standard zinc. Molarity of EDTA solution(M₁)= solution x volume of standard zinc solution taken.

Volume of EDTA used (V₁)

Molarity of standard manganese sulphate solution

M1V2

Volume of standard manganese sulphate taken. Nos. of millimoles of EDTA used in titrating Zn + other metals (A) = M_1V_3 No. of millimoles of EDTA liberated by Sodium fluoride (B) = M_2V_4 - M_1V_5 Hence Nos. of millimoles of EDTA used for titrating zinc (C) = A - B But Nos. of millimoles of EDTA liberated by KCN (D) = M_2V_6 Hence No. of millimoles of EDTA contained by Zn - EDTA sample = D - C EDTA percent = $372.24 (D - C)$ Per cent of magnesium in the sample = $24.31 \times B$ Per cent of free zinc = $65.38 \times C$ Per cent Zinc chelated with EDTA = $65.38 (D - C)$ (Ref. Vogel's Text Book of Quantitative Inorganic Analysis).

16. Method of analysis of chelated iron (as Fe - EDTA) :

(i) Quality of reagents : Unless specified otherwise, pure chemicals and glass distilled or demineralised water shall be used in tests. Note. - "Pure chemicals" means chemicals that do not contain impurities which affect the results of analysis. "Demineralised Water" means the water obtained after passing distilled water through a cation and an anion exchange resins or a combined cation - anion exchange resin. (ii) Determination of chelated iron: (a) Reagents : (1) Sodium hydroxide solution. - 0.5N. Dissolve 20 gm. NaOH in water & dilute to 1 litre. (2) Disodium EDTA solution. (3) Iron standard solution (A) Stock solution. - (1000 ppm). Dissolve. 1.000 gm. pure Fe wire in approximately 30 ml. 6N HCL with boiling. Dilute to 1 liter in a volumetric flask with distilled water. (B) Intermediate solution. - (100 ppm) Pipette 10 ml. iron stock solution and 10 ml. Na_2H_2EDTA solution in 100 ml. volumetric flask and dilute to volume. (C) Working solution. - Pipette the following volumes of 100 ppm. intermediate solution in 50 ml. numbered volumetric flask and make the volume with 0.5N HCL.

Flask No.	Volume of 100 ppm. standard iron solution taken (ml.)	Concentration of iron after making volume to 50 ml. (ppm)
1.	1.0	2.0
2.	2.0	4.0
3.	3.0	6.0
4.	4.0	8.0
5.	5.0	10.0
6.	6.0	12.0
7.	7.0	14.0
8.	8.0	16.0
9.	9.0	18.0
10.	10.0	20.0

(b) Apparatus - Atomic Absorption Spectrophotometer - with air/aceilylene flame. (c) Procedure. (1) Preparation of sample solution : (A) Weigh sample containing approximately 40 mg. Fe into 200 ml. tall form beaker. (B) Wet with 2 - 3 drops of alcohol and dissolve in 100 ml. of water. (C) Add 4 drops of 30 per cent H_2O_2 mix and adjusts pH of solution to 8.5 with 0.5N NaoH. If pH drifts above 8.8 discard solution and repeat analysis. (D) Transfer solution to 200 ml. volumetric flask, dilute to volume with water and mix (E) Filter solution through quantitative paper. (F) Pipette

10 ml. filtrate into 200 ml. volumetric flask and dilute to volume with 0.5 N HCl.(2)Flaming the solution :Flame the standards and the sample solution on atomic absorption spectrophotometer and a wavelength of 248.3nm using air acetylene flame. In same manner determine Fe blank on all reagents used.Calculation :Prepare a standard curve of known concentration of Fe solution by plotting the absorbance value on Y axis against their respective Fe concentration on X axis.

Chelated Iron (ppm Fe in sample ppm - Fe in blank

% = $\frac{\text{ppm Fe in sample} - \text{Fe in blank}}{0.4} \times 100$ g sample

(Ref.: Method of analysis AOAC, 1984)(iii)Determination of pH:(a)Procedure :Dissolve 5 gm. of the material in freshly boiled water. Dilute to 25 ml. and mix. Determine the pH value of the solution with pH meter.[17. Determination of Sodium in Potassium Chloride and Potassium Sulphate by Atomic Absorption Spectrophotometric Method] [Inserted by S.O. 337(E) dated 29th May, 1995.]

-(i)Quantity of Reagents. - Unless specified otherwise, pure chemicals and glass distilled or demineralised water shall be used in tests.(ii)Reagents :(1)Ammonium Oxalate Solution. - Dissolve 40g of Ammonium Oxalate in 1 litre of distilled water.(2)Standard Sodium Solution. - (1000 ppm) - Weigh accurately 2.5421g of dried NaCl on a clean watch glass and transfer it to one litre volumetric flask. Make up the volume up to the mark. Stopper the flask and shake the solution well. This is 1000 pm. sodium solution.(3)Preparation of working standard. - Pipette the following volumes of 1000 ppm. standard sodium solution in 100 ml. volumetric flask and make up the volume with glass distilled water.

Flask No.	Volume of 1000 ppm standard sodium solution taken (ml.)	Concentration of sodium after making volume to 100 ml. (ppm).
1	0.0	0.0
2.	2.0	20.0
3.	4.0	40.0
4.	6.0	60.0
5.	10.0	100.0
6.	12.0	120.0
7.	14.0	140.0
8.	16.0	160.0
9.	18.0	180.0
10.	20.0	200.0

Stopper the flask shake the solution well.(iii)Procedure :(1)Preparation of sample solution. - Weigh exactly 2.5 grams sample on a clean watch glass and transfer it to 250 ml. volumetric flask through the funnel giving repeat washings with glass distilled water (about 125 ml. of water should be used). Add 50 ml. of Ammonium Oxalate solution. Boil for 30 minutes, cool dilute to volume mix and pass through dry filter paper (Whatman No. 1 or equivalent).(2)Flaming the solution. - Flame the standards and the filtered sample on Atomic Absorption Spectro - photometer at a wavelength of 330.3 nm using clean air acetylene flame.Calculation :Prepare a standard of known concentrations of Sodium solution by plotting the absorbance value on Y - axis against their respective sodium concentration on X - axis. Determine the concentration of sodium in the sample solution from the graph.Sodium (Na) as Sodium Chloride % = 0.0254 X.Where X is the concentration of Sodium in ppm obtained from the standard curve.(Ref.: AOAC , 1984)]

18. Determination of Total Chlorides in Potassium Sulphate by Silver Nitrate Volumetric Method :

(i) Reagents : (1) Standard Silver Nitrate (AgNO_3) solution - 0.1N. (2) Potassium Chromate (K_2CrO_4) Indicator solution. - 5 per cent. (ii) Procedure : (1) Weigh accurately about 10 grams of the prepared sample and transfer into 500 ml. beaker. (2) Add about 250 ml. of water and warm gently for about 20 minutes. (3) Cool and dilute the solution to 500 ml. in a volumetric flask. (4) Allow to stand to let any insoluble matter settle. (5) Transfer with pipette 100 ml. of the clear solution into a conical flask. (6) Titrate with standard AgNO_3 solution (0.1N) using 1 ml. of Potassium Chromate Indicator solution. Calculation :

Total Chlorides (as Cl), per cent by Weight (ondry basis). = $17.73 \text{ N.V.} \frac{W}{V}$

Where N = Normality of standard AgNO_3 solution. V = Volume of standard AgNO_3 used. W = Weight in gram of the material taken for test. [19. Method of analysis of magnesium sulphate] [Inserted by S.O. 826(E) dated 9th November, 1992.]: (i) Quality of Reagents : Unless specified otherwise pure chemicals and glass distilled or demi neralised water shall be used in tests. (ii) Determination of Magnesium : (A) Atomic Absorption Spectrophotometric method. (a) Reagents : (1) Hydrochloric Acid 0.5 N (2) Magnesium Standard Solution : - Dissolve 1.013 gm Magnesium Sulphate ($\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$) in 0.5N Hydrochloric acid solution and dilute to 100 ml. with this acid in a volumetric flask. This is 1000 ppm magnesium stock solution. (3) Strontium Chloride solution : - Dissolve 15 gm Strontium Chloride ($\text{SrCl}_2 \cdot 6\text{H}_2\text{O}$) in 0.5 N Hydrochloric acid and dilute to 100 ml. with the same solvent. (b) Preparation of working standards : (1) Pipette 1 ml. of 1000 ppm Magnesium stock solution to 100 ml. capacity volumetric flask and make up the volume with 0.5 N Hydrochloric acid. This is 10 ppm Magnesium Solution. (2) Pipette the following volume of 10 ppm Magnesium solution in 100 ml. numbered volumetric flask. Add 10 ml. of Strontium Chloride solution to each flask and make up the volume to 100 ml. with 0.5 N Hydrochloric acid.

Flask No.	Volume of 10 ppm. Mg. solution taken (ml.)	Volume of strength chloride added (ml.)	Concentration of Mg (ppm) after making the volume to 100 ml.
1.	0.0	10	0.0
2.	2.0	10	0.2
3.	4.0	10	0.4
4.	6.0	10	0.6
5.	8.0	10	0.8
6.	10.0	10	1.0

(c) Procedure (1) Preparation of sample solution. - (A) Weigh 1 gm of the sample and place in a 500 ml. volumetric flask. Add about 300 ml. water and boil for 30 minutes. Allow to cool, dilute to the mark with water, mix and filter. (B) Pipette 5 ml. of the filtrate (A) into a 100 ml. volumetric flask, make up to the mark with water and mix. (C) Transfer by pipette 5 ml. of the diluted filtrate (B) into 1(X) ml. volumetric flask and make up to the mark with 0.5 N.H. 1. (2) Blank solution. - Prepare a blank solution from which only the sample has been omitted. (3) Flaming of solution. - Flame the standard and sample solutions on Atomic Absorption Spectrophotometer at wavelength of 285.2 nm using Air Acetylene flame. Calculation: Plot the calibration curve using the mean absorbances on Y -

axis and the corresponding concentrations of Magnesium (ppm) on X - axis. Determine the concentration of Magnesium in the sample by reference to the calibration curve. Magnesium per cent - $20X$ Where X - concentration of Magnesium (in ppm) obtained from the standard curve. (B) Titrametric method (EDTA) Titrations (Applicable to the samples which do not contain phosphate as impurity). (a) Reagents : (1) Buffer solution (pH - 10.0) - Dissolve 67.5 gm ammonium chloride in 200 ml distilled water add 570 ml ammonia solution and dilute to 1 litre. (2) Potassium hydroxide - Potassium cyanide solution - Dissolve 280 gm potassium hydroxide and 66 gm potassium cyanide in 1 litre of distilled water. (3) Potassium cyanide solution (2%) - Dissolve 2 gm potassium cyanide in 100 ml. of distilled water. (4) Eriochrome Black T indicator solution - Dissolve 0.2 gm. of indicator in 50 ml of methyl alcohol containing 2 gm of hydroxylamine hydrochloride. (5) Calcium standard solution (1 mg/ml) - Dissolve 2.4973 gm calcium carbonate, primary standard grade, previously dried for 2 hours at 285° in HCl (1+10). Dilute to 1 litre with distilled water. (6) Calcein indicator mixture - Grind together 1 gm calcein indicator 2', 7' - bis [bis (Carboxymethyl) amino] methyl - (fluorescein, sodium derivate sodium salt.) 10 gm charcoal and 100 gm potassium chloride. (7) Disodium dihydrogen ethylene diamine tetra acetic acid standard solution (0.4%) - Dissolve 4 gm NH_2H_2 - EDTA in 1 litre of distilled water. (8) Triethanolamine (1 + 1) (9) Potassium ferrocyanide solution (4%) - Dissolve 4 gm potassium ferrocyanide in 100 ml of distilled water. (b) Standardization of Calcium solution : (1) Pipette 10 ml. calcium standard solution into 250 ml erlenmeyer flask. (2) Add 100 ml. of distilled water, 10 ml. KOH - KCN solution, 2 drops of - triethanolamine solution, 5 drops of potassium ferrocyanide solution and 15 ± 1 mg of calcein indicator. (3) Immediately place the flask on a magnetic stirrer in front of daylight fluorescent light with white background. (4) While stirring, titrate with EDTA solution to disappearance of all fluorescent green and until solution remains pink, titrate more than 3 aliquots. From average, calculate calcium titer value. Calcium Titer - Volume of calcium standard (mg/ml) Volume EDTA solution used (ml.) From calcium titer, calculate magnesium titer value as follows : Magnesium titer - Calcium titer $\times 0.6064$ (c) Preparation of sample solution. (1) Weigh 1 gm magnesium sulphate fertiliser sample into 250 ml volumetric flask. (2) Add 200 ml of distilled water and boil for 30 minutes. (3) Cool dilute to volume with water and mix. (d) Procedure : (1) Titration for Ca + Mg (A) Pipette 25 ml of aliquot in 250 ml erlenmeyer flask. (B) Dilute with 100 ml. of distilled water. (C) Add 5 ml of buffer solution (pH 10), 2 ml potassium cyanide solution, 2 drops of triethanolamine solution, 5 drops of potassium ferrocyanide solution, and 8 drops of eriochrome black T indicator solution. (D) Titrate immediately with EDTA solution, stirring and lighting as in standardisation. Colour changes are wine, red, purple, dark blue, to clear blue end point, becoming green if over titrated. Note the volume of EDTA used as V_1 - ml. (2) Titration of Calcium : (A) Pipette 25 ml of aliquot in 250 ml erlenmeyer flask. (B) Dilute with 100 ml. of water. (C) Add 10 ml COH - KCN solution, 2 drops of triethanolamine solution, 5 drops of potassium ferrocyanide solution and 15 ± 1 mg of calcein indicator. (D) Titrate immediately with EDTA solution as in standardization. Note the volume the EDTA used as V_2 - ml. Calculation: Magnesium per cent = $(V_1 - V_2) \times \text{Mg Titer EDTA}$ (Reference : AOAC, 14th edition, 1984). Ministry of Human Resource Development (Department of Women and Child) [20. Determination of particle size in different fertilizers] [Added by S.O. 354(E) dated 3rd June, 1993.]: (i) Apparatus : (A) Indian Standard sieves of 20 cms diameter and 5 cms in width with lid and bottom pan of required sizes. (B) Sieve shaker or vibrator with automatic timer and variable cycle control (C) Balance top loading with suitable capacity and sensitivity of 0.1 gm. (D) Weighing pan, round suitable for holding sample and approximately 23 cms. in

diameter.(E)Brush either soft, fine wire or stiff bristle.(ii)Procedure : (A)Take the sieve of requires sizes and stack them in progressive order. The biggest sieve sizes should be on the top and the smallest in the bottom. Place the empty pan at the bottom.(B)Put 200 gms. of ungrinded sample on the top sieve and place lid on the top of stack.(C)Position and secure sieve stack in shaking apparatus.(D)Weighing pan, round suitable for holding matic timer is used, set timer for 5 minutes. If vibrator is used, also set vibrator control at 3000 cycle per minutes.Note. - If mechanical sieve shaker is not available, use hand sieving Conduct sieving by appropriate lateral and vertical motions accompanied by jarring action. Continue until no appreciable change is noted in sieve fractions.(E)After completion of shaking time, transfer material from each sieve to weighing pan with brush and weight to ± 0.1 gm.(F)Record weight from each sieve (sum of weights from each sieve should agree closely with original sample weight.)Calculations :

Per cent weight on sieve = $\frac{\text{Weight on sieve(gm.)}}{\text{Total weight of sample(gm.)}}$

Reference. - Method IV - A TH - 1982).][21. Method of Analysis of Calcium Nitrate] [Inserted by S.O. 329 (E), dated 12th May, 1999 (w.e.f. 12th may,1999).](i)Determination of total nitrogenBy the method as specified in 3(viii)(ii)Determination of Ammoniacal NitrogenBy the method as specified in 3 (vii)(iii)Determination of nitrateBy the method as specified in 3(viii) & (vii)(iv)Determination of water insoluble matterBy the method as specified in 8(vii)(v)Determination of water soluble calciumA. Titrametric Method (EDTA) Titrations(a)Reagents(i)Buffer solution (pH - 12.0) - Dissolve 225 gm. KOH (AR) in 250 ml water. Add 15 ml Triethanolamine and make volume to one litre after cooling the solution.(ii)Pattan and Reeder's indicator (HHSNNA) - Dissolve 0.2 gm indicator in 100 ml. methanol.(iii)Calcium Standard Solution (mg/m1) - - Dissolve 2.4973g calcium carbonate primary standard grade, previously dried for 2 hours at 285°C, in HCl (1+10). Dilute to 1 litre with distilled water.(iv)Disodium dihydrogent ethylene diamine tetra acetic standard solution (0.01M) - Dissolve 3.7224 gm Na₂H₂ - EDTA (AR) previously dried at 105 to 110°C for 1 hr. in distilled water and make volume to one litre.(b)Standardisation of Calcium Solution(i)Pipette 10 ml. calcium standard solution into 250 ml. Erlenmayer Flask.(ii)Add 10 - 15 ml. distilled water and 20 ml. Buffer solution.(iii)Add few drops of indicator's solution. A red rose colour will appear.(iv)Titrate with EDTA till a blue colour is obtained. Note the volume of EDTA used as V₁ ml.

Calcium Titer (mg/m1) = $\frac{\text{Volume of Calcium standard solution(ml)}}{\text{solutionused (ml) (V}_1\text{)}}$ Volume of EDTA

(c)Preparation of sample solution(i)Weigh 2.5g calcium nitrate fertiliser sample into 250 Volumetric Flask.(ii)Make up the volume with distilled water. Shake well.(d)Procedure(i)Pipette 5 ml. of aliquot in 250 ml. Erlenmayer Flask.(ii)Add 10 - 15 ml. distilled water & 20 ml Buffer solution.(iii)Add few drops of indicator's solution. A red rose colour will appear.(iv)Titrate with EDTA till a blue colour is obtained. Note the volume of EDTA used as V₂ ml.CalculationCalcium per cent = $\frac{V_2 \times \text{Calcium Titer}}{2B}$. Atomic Absorption Spectrophotometric Method(1)Reagent. - Unless specified otherwise pure chemicals and glass distilled or demineralised water shall be used in test.(a)Hydrochloric Acid = 3N(b)Preparation of stock solution : -(i)Calcium stock solution. - Dissolve 2.498 gram CaCO₃ in a minimum amount of 3 N Hcl. Dilute to 1liter (1000 ppm Ca). Dilute 10 ml to 100 ml. This is 100 ppm stock solution.(C)Preparation of working standard: -(ii)Lanthanum stock solution. - 50g La/L. Dissolve 58.65 gram La₂O₃ in 250 ml. HCl, adding acid slowly. Dilute to 1 liter.Add 0, 5, 10, 15 and 20 ml. of Ca stock solution in a 100 ml volumetric flask. Add 20 ml. La stock solution and make up

the volume to 100 ml with distilled water. This will correspond to 0, 5, 10, 15 and 20 Mg Ca/ml containing 1% La.

Flask No.	Volume of Ca taken from stock solution	Volume of La added in vol. flask	Concentration of Ca in mg/ml (ppm)
1.	0 ml	20 ml	0
2.	5 ml	20 ml	5
3.	10 ml	20 ml	10
4.	15 ml	20 ml	15
5.	20 ml	20 ml	20

(2) Procedure. - Weigh 1.00 gram well grind sample in a 100 ml. volumetric flask. Add 25 ml water and shake to dissolve. Then add 20 ml Lanthanum solution in the flask and make volume to 100 ml with distilled water. Shake well and allow to settle. Filter, if required, before it is

aspirated. (3) Flaming of the solution. - Flame the standard and sample solution on AAS at wave length of 422.7 nm using Air - Acetylene flame. (4) Calculation. - Plot the calibration curve using the mean absorbance on Y axis and the corresponding concentration of Ca (ug/ml) at X axis.

Determined the concentration of calcium in the sample by reference to the calibration curve. % Ca = $\frac{\text{Concentration (ug/ml)} \times 10^{-2}}{\text{Concentration (ug/ml)}}$

22. Method of Analysis of Potassium Magnesium Sulphate

Determination of water soluble K₂O By the method as specified in 5(i) Determination of Magnesium By the method as specified in 19(B) and 19(A) Determination of Total Chlorides By the method as specified in 18 Determination of Sodium By the method as specified in 17 Determination of particle size By the method as specified in 20. Forms Emblem Form A [See Clause 8] Form of application to obtain dealer's (wholesale or retail [or industrial] [Inserted by S.O. 795 (E), dated 22nd November, 1991.] certificate of registration To The Registering Authority - ["/Controller (If the application for industrial dealer's certificate of Registration)"] [Added by S.O. 795(E), dated 22nd November, 1991.] Place : State of

1. Full name of address of the applicant :

(a) Name of the concern, and postal address : (b) Place of business (Please give exact address) : (i) for sale (ii) for storage

2. Is it a proprietary/partnership/limited company/Hindu Undivided Family concern ? Give the name (s) and address (es) of proprietor/partners/manager/ Karta :

3. In what capacity is this application filed.

(i) Proprietor (ii) Partner (iii) Manager (iv) Karta

4. Whether the application is for wholesale or retail [or industrial] [Inserted by S.O. 795 (E), dated 22nd November, 1991.] dealership?

5. Have you ever had a fertiliser dealership registration certificate in the past ? If so, give the following details :

(i)Registration number(ii)Place for which granted(iii)Whether wholesale or retail [or industrial] [Inserted by S.O. 795 (E), dated 22nd November, 1991] dealership(iv)Date of grant of registration certificate(v)Whether the registration certificate is still valid?(vi)If not, when expired ?(vii)Reasons for non - renewal(viii)If suspended/cancelled and if so, when(ix)Quantity of fertilizers, handled during last year(x)Names of products handled.(xi)Names of source of supply of fertilizers.

6. Was the applicant ever convicted under the Essential Commodities Act, 1955, or any Order issued thereunder, including the Fertiliser (Control) Order, 1957, during the last three years preceding the date the application ? If so give details.

7. Give the details of the fertilizers to be handled :

Sl. No. Name of Fertiliser Source of supply

8. Please attach certificate (s) of source from the supplier (s) Indicated under Column 3 of Sl. No. 7.

9. I have deposited the registration fee of Rs. vide Chall No dated in Treasury/Bank

[or I enclose the Demand Draft No dated for Rs drawn on Bank, in favour of payable at towards registration fee.(Please strike out whichever is not applicable)] [Added by S.O. 795 (E) dated 22nd November, 1991.]

10. Declaration. -

(a)I/We declare that the information given above is true to the best of my/our knowledge and belief, and no part thereof is false.(b)I/We have carefully read the terms and conditions of the Certificate of Registration given in Form "B" appended to the Fertiliser (Control) Order, 1985, and agree to abide by them.(c)[I/We declare that I/We do not possess a certificate of registration for industrial dealer and that I/We shall not sell fertilizers for industrial use. (Applicable in case a person intends to obtain a wholesale dealer or retail dealer certificate of registration, excepting a State Government, a manufacturer or a pool handling agency). [Added by S.O. 795(E) dated 22nd November,

1991.](d)I/We declare that I/We do not possess a certificate of registration for wholesale dealer or retail dealer and that I/We shall not sell fertilisers for agricultural use. (Applicable in case a person intends to obtain a industrial dealer certificate of registration, excepting a State Government a manufacturer or a pool handling agency).]Signature of the Applicant (s)Dated :Place :Note. - (1) Where the business of selling fertiliser is intended to be carried on as more than one place, a separate application should be made for registration in respect of each such place.(2)Where a person intends to carry on the business of selling fertilizers both in retail and wholesale separate applications for retail and wholesale business should be made.(3)Where a person represents or intends to represent more than one State Government, Commodity Board, manufacturer on Wholesale dealer, separate certificate of source from each such source should be enclosed.For use in office of Registering Authority. [controller] [Added by S.O. 795(E) dated 22nd, November, 1991.]Date of receipt :Name and designation of officer receiving the application.EmblemForm B[See Clause 9]Book.NoRegistration NoDate of IssueValid up toCertificate of registration to carry on the business of selling fertilisers as a wholesale/ retail dealer [Industrial Dealer] [Added by S.O. 795(E) dated 22nd November, 1991.] in the State of is hereby granted certificate of registration to carry on the business of selling fertilisers in retail/wholesale [for industrial use] [Inserted by S.O. 795(E), dated 22nd November, 1991.] at the place specified below in the State of subject to the terms and conditions specified below and to the provisions of the Fertiliser (Control) Order, 1985.Description of the place and type of business

Name and style by which the business is carried on	Location of sale depot	Location of godowns attached to sale depot	Type of fertiliser	Source of supply
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Date:[Controller/] [Added by S.O. 795(E) dated 22nd November, 1991.] Registering Authority State ofSeals :Terms and Conditions of certificate of registration :

- 1. This certificate of registration shall be displayed in a prominent and conspicuous place in a part of the business premises open to the public.**
- 2. The holder of the certificate shall comply with the provisions of the Fertilisers (Control) Order, 1985 and the notification issued thereunder for the time being in force.**
- 3. The certificate of registration shall come into force immediately and be valid up to unless previously cancelled or suspended.**
- 4. The holder of the certificate shall from time to time report to the Registering Authority any change in the premises of sale depot and godown attached to sale depot.**

5. The [The wholesaler dealer/retail dealer.] [Substituted by S.O. 795(E) dated 22nd, November, 1991.] shall submit a report to the Registering Authority, with a copy to the Block Development officer or such other officer as the State Government may notify, in whose jurisdiction the place of business is situated, by the 5th of every month, showing the opening stock, receipts, sales and closing stocks of fertilisers in the preceding month. He shall also submit in time such other returns as may be prescribed by the Registering Authority.

6. [The industrial dealer shall submit a report to the Central Government by the 15th of April for the preceding year, showing the opening stocks as on 1st April of the reporting year, source - wise receipts during the year, sale and closing stocks of fertiliser along with the source - wise purchase/sale price.] [Added by S.O. 795(E) dated 22nd November, 1991]

7. The wholesale or the retail dealer, except where such a dealer is a State Government a manufacturer or a pool handling agency, shall not sell fertilisers for industrial use and, as the case may be, in industrial dealer for agricultural use.

Note. - (1) The original is meant for the holder of the certificate which will be delivered against his proper and adequate acknowledgement. The original certificate of registration shall be torn off at the place performed while all duplicate shall be kept intact bound in the registration book by the the Registering Authority.(2)Where the business of selling fertilisers is intended to be carried on at more than one place, as separate registration certificate should be obtained in respect of each such place.(3)Where a person intends to carry on the business of selling fertilisers both in retail and wholesale, [and, as the case may be, a State Government, a manufacturer or a pool handling agency also for industrial use,] [Inserted by S.O. 795(E) dated 22nd November, 1991.] separate registration certificate should be obtained for retail and wholesale business [and for sale for industrial use] [Added by S.O. 795(E) dated 22nd November, 1991.].EmblemForm C[See Clause 11]Application for renewal of the certificate of registration to carry on the business of selling fertilisers in retail/wholesale [for industrial use] [Added by S.O. 795 (E) dated 22nd November, 1991.]ToThe Registering Authority [/Controller (If the application for industrial dealer's Certificates of Registration)] [Added by S.O. 795(E), dated 22nd November, 1991.]Place :State :I/We hereby apply for renewal of the certificate of registration to carry on the business of selling fertilisers in retail/wholesale [for industrial use] [Added by S.O. by 795(E), dated 22nd November, 1991.] under the name and style of The certificate of registration desired to be renewed was granted by Registering Authority for the (mention place and State) and allotted Registration Certificate No. on the day of 19

2. (i) I/We hereby declare that the situation of my/our premises where fertilisers are (a) stored and (b) sold is as stated below :

(a)

(b)

Premises where fertiliser are stored Premises where fertiliser are sold

(ii) I/We hereby declare that the fertiliser(s) in which I/We am/are carrying on the business of selling and the name(s) of manufacturer(s), Commodity Board(s), State Government (s) and wholesale dealer whom I/We represent are as stated below :

(a)

(b)

Name (s) of fertiliser (s) Name (s) of manufacturer (s), Commodity Board(s), Wholesale dealer (s)

3. I/We enclose a certificate of source from the manufacturer (s), Commodity Board (s), State Government (s), wholesale dealer (s) whom I/We represent or intend to represent and from whom fertiliser (s) will be obtained by me/us.

4. [I have deposited the renewal fee of Rs. vide Challan No Dated in Treasury/Bank or I enclose the Demand Draft No. dated for Rs. drawn on Bank, in favour of payable at towards renewal fee. (Please strike out whichever not applicable).] [Added by S.O. 795(E), dated 22nd November, 1991.]

Full name and address of the applicant(s) (in block letters) Signature of applicant(s) Date : Place : Certified that the certificate of registration bearing number granted on for the period from to carry on the business of selling fertilisers in retail/wholesale [or industrial use] [Inserted by S.O. 795(E) dated 22nd November, 1991.] at the premises situated at is hereby renewed till the unless previously suspended or cancelled under the provisions of the Fertiliser (Control) Order, 1985.

(a) Location of sale depot	(b) Location of godowns attached to sale depot. where fertiliser will be stored	(c) Name (s) of fertiliser(s) for which certificate of registration is renewed (d)	(d) Name (s) manufacture (s) Commodity Board(s) State Government (s) wholesale dealer (s) whom the dealer represents.
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[Controlled] [Added by S.O. 795(E) , dated 22nd November, 1991] Registering Authority Place: State Date : Renewal Number : Seal : Emblem Form D [See Cls. 14(2)(a) & 18(1)] Form of application to obtain a certificate of manufacture of physical / granulated [* *] [Omitted by S.O. 354(E), dated 3rd June, 1993.] / renewal. To The Registering Authority, Place State of

- 1. . Full name and address of the applicant :**
- 2. Does the applicant possess the qualification prescribed by the State Government under sub - clause (1) of Clause 14 of the Fertiliser (Control) Order, 1985:**
- 3. Is the applicant a new comer / (Say "Yes" or "No") :**
- 4. Situation of the applicant's premises where physical/granulated mixture will be prepared :**
- 5. Full particulars regarding chemical analysis of the physical/granulated [* * *] [Omitted by S.O. 354(E), dated 3rd June, 1993] for which the certificate is required and the raw materials used in making the mixture.**
- 6. Full particulars of any other certificate of manufacture, if any, issued by any other Registering Authority :**
- 7. How long has the applicant been carrying the business of preparing physical/ granulated [* * * *] [Omitted by S.O. 354(E), dated 3rd June, 1993.].**
- 8. Quantities of each physical/granulated [* * * *] [Omitted by S.O. 354(E), dated 3rd June, 1993.] (in tonnes) in my/our possession on the date of the application and held at different addresses noted against each :**
- 9. (i) If the applicant has been carrying on the business of preparing physical/ granulated [* * * *] [Omitted by S.O. 354(E), dated 3rd June, 1993.] give all particulars of such mixtures handled, the period and the place (s) at which the mixing of fertilisers was done :**
- (ii)Also give the quantities of physical/granulated fertiliser mixtures handled during the past calender year :**
- 10. If the application is for renewal indicate briefly why the original certificate could not be acted on within the period of its validity :**
- 11. I have deposited the prescribed registration certificate fee/renewal fee :**

Declaration :(a)I/We declare that the information given above in true and correct to the best of

my/our knowledge and belief, and no part thereof is false.(b)I/We have carefully read the terms and conditions of the certificate of manufacture given in Form F appended to the Fertiliser (Control) Order, 1985 and agree to abide by them.(c)I/We declare that the physical/granulated mixture for which certificate of manufacture is applied for shall be prepared by me/us or by a person having such qualifications as may be prescribed by the State Government from time to time or by any other person under my/our direction. supervision and control or under the direction, supervision and control of person having the said qualifications.(d)[I/We declare that the requisite laboratory facility specified by the controller. under this Order this possessed by me/us] [Added by S.O. 261(E), dated 16th April, 1991 (w.e.f. 16th, April, 1991).].Signature of the applicant(s)Name and address of applicant in block letters :Date :Place :EmblemForm E[See Clause 14(2) (B)]Form of application to obtain a certificate of manufacture for special mixture of fertilisersToThe Registering AuthorityPlaceState

1. Full name and address of the applicant :

2. Does the applicant possess the qualification prescribed by the State Government under sub - clause (1) of Clause 14 of the Fertiliser (Control) Order, 1985 :

3. Name and address of the person requiring the special mixture of fertilisers :

4. Particulars of certificate (s) of manufacture already obtained from the same Registering Authority :

5. Situation of the applicant's premises where fertilisers are/will be mixed :

6. Full particulars regarding chemical analysis of the special mixture of fertilisers required to be manufactured and the materials used in making the special mixture :

7. I am enclosing_ an attested copy of the requisition made by the purchaser of the special mixture of fertilisers.

8. I have deposited the prescribed registration certificate fee:

Declaration :(a)I/We declare that the information given above is true and correct to the best of my/our knowledge and belief, and no part thereof is false.(b)I/We have carefully read the terms and condition of the certificate of manufacture given in Form "G" appended to the Fertiliser (Control) Order, 1985 and agree to abide by them.(c)I/We declare that the special mixture for which certificate of manufacture is applied for, shall be prepared by me/us or by a person having such

qualification as may be prescribed by the State Government from time to time or by any other person under my/our direction, supervision and control or under the direction, supervision and control of a person having the said qualifications.(d)[I/We declare that the requisite laboratory facility specified by the controller, under this Order is possessed by me/us] [Added by S.O. 261(E), dated 16th April, 1991 (w.e.f. 16th, April, 1991).].Signature of the applicant (s)Name and address of applicant (s) in block letters :Date :Place :EmblemForm F[See Clauses 15(2) & 18(2)]Book NoCertificate NoDate of issueValid up toCertificate for Manufacture in Respect of Physical/Granulated [* * *] [Omitted by S.O. 354(E), dated 3rd June, 1993.]...... is/are hereby by given the certificate for manufacture of the physical/granulated [* * *] [Omitted by S.O. 354(E), dated 3rd June, 1993.] specified below subject to the terms and conditions of this certificate and to the provisions of the Fertiliser (Control) Order, 1985.Full particulars of the [* * *] [Omitted by S.O. 354(E), dated 3rd June, 1993.]Full address of the premises where the [* * *] [Omitted by S.O. 354(E), dated 3rd June, 1993.] will be made Registering AuthorityStateDate :Seal :Renewed up toRegistering AuthorityStateDate :Seal :Terms and conditions of this certificate :

- 1. The holder of this certificate shall display the original thereof in a conspicuous place open to the public in part of the principal's premises in which the business of making the physical/granulated [* * *] [Omitted by S.O. 354(E), dated 3rd June, 1993.] is carried on and also a copy of such certificate in similar manner in every other premises in which that business is carried on. The required number of copies of the certificate shall be obtained on payment of the fees thereof.**
- 2. The holder of this certificate shall not keep in the premises in which he carries on the business of making physical/granulated [* * *] [Omitted by S.O. 354(E), dated 3rd June, 1993.] any [* * *] [Omitted by S.O. 354(E), dated 3rd June, 1993.] in respect of which a certificate of registration has not been obtained under the Fertiliser (Control) Order, 1985.**
- 3. The holder of this certificate shall comply with the provisions of the Fertiliser (Control) Order, 1985 and the notification, order and directions, issued thereunder for the time being in force.**
- 4. The holder of the certificate shall report forthwith to the Registering Authority any change in the premises specified in the certificate or any new premises in which he carried on the business of making physical/granulated mixture and shall produce before the authority the original certificate and copies thereof so that necessary corrections may be made therein by that**

authority.

5. The holder of this certificate shall ensure that the physical/granulated mixture in respect of which a certificate of manufacture has been obtained is prepared by him or by a person having such qualifications, as may be prescribed by the State Government, from time to time or by any other person under the directions, supervision and control of the holder or the person having the said qualifications.

6. The certificate and copies thereof, if any, will be machine numbered and delivered against the signature of the holder thereof or his agent on the carbon copy of the certificate which will be kept intact bound in the 'Certificate Book' by each Registering Authority.

EmblemForm G[See Clause 15(2)]Book NoCertificate NoDate of issueCertificate for manufacture in respect of special mixture of fertilisers..... is hereby given this certificate for the preparation of tonnes of special mixture of fertiliser specified below subject to the terms and conditions of the certificate, and to the provisions of the Fertiliser (Control) Order, 1985.Full particulars of the special mixture of fertilisers

2. The certificate is valid up to

Full address of the premises where the special mixture of fertilisers will be made.Registering Authority :StateDate :Seal :Terms and conditions of this certificate :

1. The holder of this certificate shall display the original thereof in a conspicuous place open to the public in part of the principal's premises in which the business of making the special mixture of fertilisers is carried on and also a copy of such certificate in similar manner, in every other premises in which that business is carried on. The required number of copies of the certificate shall be obtained on payment of the fees therefor.

2. The holder of this certificate shall not keep in the premises in which he carries on the business of making special mixture of fertilisers, any mixture of fertilisers in respect of which a certificate of manufacture has not been obtained under the Fertiliser (Control) Order, 1985.

- 3. The holder of this certificate shall comply with the provisions of the Fertiliser (Control) Order, 1985 and the notification, orders and directions, issued thereunder for the time being in force.**
- 4. The holder of the certificate shall report forthwith to the Registering Authority any change in the premises specified in the certificate or any new premises in which he carries on the business of making the special mixture and shall produce before the authority the original certificate and copies thereof so that necessary corrections may be made therein by that authority.**
- 5. The holder of this certificate shall ensure that the special mixture in respect of which a certificate of registration has been obtained is prepared by him or by a person having such qualifications as may be prescribed by the State Government, from time to time or by any other person under the direction supervision and control of the holder or the person having the said qualifications.**

Note. - The certificate and copies thereof, if any, will be machine numbered and delivered against the signature of the holder thereof or his agent on the carbon copy of the certificate which will be kept intact bound in the "Certificate Book" by each Registering Authority. Emblem Form H [See Clause 23(1)(b)] Application for grant of Permission to Sell Non - Standard Fertilisers To The Registering Authority, State of

- 1. Full name and address of the applicant :**
- 2. The name of fertilisers in which the applicant has been authorised to carry on business :**
- 3. Number and date of registration certificate granted in favour of the applicant :**
- 4. The particulars of non - standard fertilisers which are intended to be sold :**

(i) Name of the fertiliser : (ii) The source from which the fertilisers were originally purchased and the period during which they have been held by the applicant : (iii) Brief reasons leading to the material having become non - standard : (iv) The chemical specifications as analysed by an authorised chemical laboratory : (v) Name and appearance of the non - standard fertilisers : Note. - Chemical analysis to be indicated shall be one which has been certified by an authorised chemical laboratory on the basis of the samples drawn, sealed and despatched in the presence of the local Inspector of Fertilisers or anybody authorised to inspect the fertilisers. I enclose a copy of the certificate of

registration for dealing in fertilisers already granted to me and I have deposited the prescribed fee for permission to sell non - standard fertilisers. I certify that I will comply with the requirement of sub - clause (a) of Clause 23 of the Fertiliser (Control) Order, 1985, which requires that each container of non - standard fertilisers is to be conspicuously superscribed with the words 'Non - standard' and also with the sign 'X', both in red colour. Signature of the applicant Name and address of the applicant in block letters Date : Place : Emblem Form I [See Clause 23(1)(b)] Book No Date of issue Valid up to Authorisation for the sale of non - standard fertilisers M/s is/are hereby permitted to sell the non - standard fertilisers described below subject to the terms and conditions noted hereunder : Particulars of non - standard fertilisers :

1. Name of the fertilisers :

2. Detailed chemical specifications including the nutrient content.

3. Nature and appearance of the non - standard fertiliser.

4. Place of location of the non - standard fertiliser.

Terms and conditions :

1. The seller of the non - standard fertiliser described above shall comply with the provisions of sub - clause (a) of Clause 23 of the Fertiliser (Control) Order, 1985.

2. The non - standard fertilisers described above shall be sold at a price not exceeding per metric tonne or per bag of kgs.

3. The non - standard fertiliser described above shall be sold before the expiry of the validity period mentioned above or before the expiry of the extended period, if any

Registration Authority State of Date : Seal : Emblem Form J [See Clause 28(1)(b)] Form indicating particulars of fertiliser sampled

1. Name & Address of dealer/manufacturer

2. Date of sampling

3. Details of markings on bags from where sample has been taken

(i) Type & grade of fertiliser with brand (ii) Name of manufacturers
..... (iii) Registration No (iv) Lot No. & date of manufacture
..... (v) Composition

4. Date of receipt of the stock by the dealer/manufacturer/pool handling agency

5. Serial No./Code No. of sample

6. Stock position of the lot

7. Physical condition of fertiliser

8. Whether samples drawn from open bags or stitched bags

9. Name & Address of Fertiliser Inspector drawing sample

Signature & Metallic Seal impression of Fertiliser Inspector
Receipt of the Dealer
Certified that the sample of fertiliser has been drawn in accordance with the procedure laid down in the Fertiliser (Control) Order, 1985, from the stock in my possession, and I have signed the test samples at the time of wax sealing. I have also received one test sample out of the three test samples prepared.
Signature & Seal of Fertiliser Inspector
Signature of dealer/manufacturer/Pool handling agency with address
Emblem Form K [See Clause 30(1)]
Memorandum to accompany fertiliser sample for

analysis
No. From To, Incharge Fertiliser Quality Control Laboratory
The fertiliser samples as per details given below are sent for analysis
:(1) Name of fertiliser grade and brand (2) Date of sampling (3) No. of samples taken (4) Serial No. of sample (5) Physical condition of fertiliser (6) The analysis report may please be forwarded to Place : Date :
Signature of Fertiliser Inspector
Emblem Form L [See Clause 30(2)]
1. Analysis report of fertiliser sample No Government of (Name of the Laboratory) Date : To, The Fertiliser Inspector The analysis report of the fertiliser sample forwarded vide your reference No dated is as per details below :

1. Name of fertiliser, grade and brand

2. Date of sampling

3. Serial No. of sample as indicated by the Inspector

4. Code No. of sample as indicated by the Inspector

5. Date of receipt of the sample in the laboratory

6. Laboratory sample No

7. Date of analysis of sample

8. Chemical analysis of fertiliser (on fresh weight basis except in the case of Urea on dry weight basis)

Sl. No.	Specification as per F.C.O	Composition as per analysis	Variation	Permissible tolerance limit
1	2	3	4	5

(i) Moisture(ii) Total N(iii)NH₄N(iv)
NH₄+NO₃H(v)Total P₂O₅(vii) Nitrate
AmmoniumCitrate soluble P₂O₅(ix) Water
solubleP₂O₅(x) Water soluble
K₂O(xi)Particle size(xii) Others

Remarks. - The samples is/is not according to specifications. Signature of the Incharge Fertiliser
Quality Control LaboratoryCopy to :Director of

Agriculture.....[Emblem] [Form 'M' and 'N' added by S.O.
261(E), dated 16th April, 1991(w.e.f. 16th April, 1991).]Form M[See Clause 5]Form for Cash/Credit
Memorandum to be issued by the Dealer/Manufacturer/Pool Handling Agency to the Purchaser of
FertilizersST/CST NoDateSl. No

.....Certificate of Registration No

.....Name and Address of Firm

.....Name and Address of purchaser

.....

Sl. No.	Name of fertiliser and brand name	Number of of bags	Batch number (wherever application)	Quantity	Rate	Amount
1	2	3	4	5	6	
1					Total	
2.					ST/CST	
X					@	
XX					G. Total	

Signature of purchaser Signature of Dealer, etc. [Emblem] [Form 'M' and 'N' added by S.O. 261(E), dated 16th April, 1991 (w.e.f. 16th April, 1991).] Form N [See Clause 35(1)(a)] Form for Stock Register to be Maintained by the Dealer/Manufacturer/Pool Handling Agency Page No. :

Date	Opening Balance	Receipts During the day	Total	Sales during the day	Closing Balance	Remarks	Signature of the dealer
1	2	3	4	5	6	7	8
						1. Mention source of supply with Bill No. and date 2. Sl. No. of first and last cash/Credit memo issued	

Note. - The pages of the stock register should be numbered. A certificate should be recorded by the inspecting authority confirming the pages in the register. [Form O] [Added by S.O. 354(E), dated 3rd June, 1993.] [See Clauses 8 and 11] No Date of Issue Certificate of source for carrying on the business of selling fertilisers in wholesale/ retail for industrial use.

1. Particulars of the concern issuing the certificate of source :

(a) Name and full address : (b) Status : (i) State Government. (ii) Manufacturer. (iii) Pool handling agency. (iv) Wholesale dealer. (c) If manufacturer of mixture of fertilisers, the details of certificate of manufacturer of mixture of fertilisers possessed : (i) Number. (ii) Date of issue. (iii) Date of expiry. (iv) Grades of mixtures of fertilisers allowed to be manufactured. (v) Authority by whom issued. (d) Details of certificate of registration : (i) Number (ii) Date of issue (iii) Date of expiry (iv) Authority by whom issued.

2. Particulars of the person to whom the certificate of source is being issued.

(a) Name and full address : (b) Status : (i) Wholesale dealer (ii) Retail dealer. (iii) Industrial dealer. (c) If holds a valid certificate of registration, the details thereof : (i) Number, (ii) Date of issue. (iii) Date of expiry (iv) Authority by whom issued. (d) Purpose of obtaining the certificate of source : (i) For obtaining a fresh certificate of registration. (ii) For renewal of the certificate of registration.

3. Details of fertiliser(s) to be supplied :

Sl.No. Name of fertilisers Trade mark/Brand name

(1) (2) (3)

1.2.3.

4. Declaration. - Declared that the fertilisers mentioned above will be supplied conforming to the standards laid down under the Fertiliser (Control) Order, 1985, and, as the case may be, grades/formulations (of mixtures of fertilisers) notified by the Central/State Government and packed and marked in

container as provided under Clause 21 of the Fertiliser Control) Order, 1985.

Signature with stamp of the Authorised officer[***] [The 'Note', omitted by S.O. 459(E), dated 22nd May, 1995.]