

“Environmental Guidelines for Compressed Biogas Plant (CBG)/Bio-CNG Plants”



(March, 2022)

**Central Pollution Control Board
(Ministry of Environment, Forest and Climate Change, Govt. of India)
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FOREWORD

Waste / Bio-mass sources like agricultural residue, cattle dung, sugarcane press mud, municipal solid waste and sewage treatment plant waste, etc. produce bio-gas through the process of anaerobic decomposition. The bio-gas is purified to remove hydrogen sulfide (H_2S), carbon dioxide (CO_2), water vapor and compressed as Compressed Bio Gas (CBG), which has methane (CH_4) content of more than 90%. The other waste streams viz. rotten potatoes from cold storage, rotten vegetables, dairy plants, chicken/ poultry litter, food waste, horticulture waste, forestry residues and industrial Effluent Treatment Plants (ETPs) treating organic waste can be used in the generation of biogas.

CBG has calorific value and other properties similar to CNG and hence can be utilized as green renewable automotive fuel. Thus it can replace CNG in automotive, industrial and commercial areas, given the abundance biomass availability within the country.

Presently, 32 million tonnes of CBG potential is estimated in the country. Though, of the total estimated potential, only 0.06% CBG is being produced currently on an annual basis.

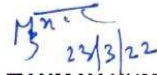
To promote CBG production and usage, Ministry of Petroleum and Natural Gas, Government of India, on October 01, 2018 announced setting up of 5,000 CBG plants by 2025 with an investment of about Rs. two lakh crore under the scheme of "Sustainable Alternative Towards Affordable Transportation (SATAT)". The scheme will open up a new window of green energy corridor in the transport sector and employment as well.

Central Pollution Control Board has categorized industrial sectors in various categories viz. Red, Orange, Green and White on the basis of pollution potential, which is represented as Pollution Index. CPCB has also recently categorized Compressed Bio-Gas (CBG) plants in light of the notifications issued by the Ministry of Agriculture and Farmers Welfare vide Gazette Notification No. 2051 dated 14.07.2020 and No. 1972 dated 01.06.2021 regarding inclusion of Fermented Organic Manure (FOM) and Liquid Fermented Organic Manure (LFOM) under Fertilizer (Inorganic, Organic or Mixed) (Control) Act, 1985.

The present report contains environmental best practices for CBG plants towards management of solid Waste, Solid Manure, Liquid Manure (Fermented Organic manure, Self-Monitoring system, Scrubber System and steps to be taken by Green & Orange category CBG/BIO-CNG plant to qualify as white category.

The contribution made by my colleagues Sh. P.K. Mishra, Additional Director, in preparing the report under the supervision of Dr. Prashant Gargava, Member Secretary is appreciated. Cooperation extended by NEERI, TERI, NSI and other collaborating agencies, are gratefully acknowledged.

Hope the report shall be of use to all concerned.


(TANMAY KUMAR)

March, 2022



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1. Background

India currently imports nearly 77% of its crude oil requirements and about 50% of natural gas requirement. The Government of India has set a target of reducing this import by at least 10% by 2022. Further, it has set a target of increasing the contribution of gas in India's energy mix from existing 6.5% to 15% by 2022 (global average is 23.5%)

Bio-degradable organic waste or biomass such as agricultural residue, cattle dung, sugarcane press mud, municipal solid waste and sewage treatment plant waste, etc. produce bio-gas through the process of anaerobic decomposition. The bio- gas is purified to remove hydrogen sulfide (H₂S), carbon dioxide (CO₂), water vapor and thereafter compressed as Compressed Bio Gas (CBG), which contains more than 90% methane (CH₄), a combustible gas and clean fuel. The other waste streams viz. rotten potatoes from cold storage, rotten vegetables, dairy plants, chicken/ poultry litter, food waste, horticulture waste, forestry residues and industrial Effluent Treatment Plants (ETPs) treating organic waste can be used in the generation of biogas.

CBG is a clean renewable fuel, that has calorific value and other properties similar to CNG, and hence it can replace CNG in automotive, industrial and commercial areas. Given the abundance availability of biodegradable organic waste within the country, production of CBG in a commercial scale is expected to have the following benefits:

- Import reduction of natural gas and crude.
- Utilization of agricultural residue, cattle dung and MSW for the production of CBG and thus to achieve reduction in emissions and pollution.
- A boost towards fulfillment of National commitments in achieving climate change goals.
- Providing a buffer against energy security concerns and crude/gas price fluctuations.
- Contribution towards Swatch Bharat Mission through responsible waste management
- Lowering pollution and carbon emission.
- Providing additional source of revenue to the farmers, rural employment and amelioration of the rural economy

Bio- CNG plant provides green solution to the stubble burning which plays a significant role in deteriorating of air quality. Present utilization of crop residue in the country is hardly 25%.

National Policy on Bio-Fuels 2018 (vide Gazette Notification No. 33004/99, dated 8.6.2018) emphasizes on promotion of advanced Bio-fuels including CBG. Ministry of Road Transport and Highways Vide Gazette Notification No. 395, dated 16th June 2015, permitted usage of bio-compressed natural gas (bio-CNG) for motor vehicles as an alternate to compressed natural gas (CNG).

According to estimate, country has potential to produce 32 million tonnes of CBG of which currently, only 0.06% of CBG is being produced. The research on biogas purification and its utilization as a vehicular fuel and power production is getting focused attention. To promote CBG production and usage, Ministry of Petroleum and Natural Gas, Government of India, on October 01, 2018 announced setting up of 5,000 CBG plants by 2025 with an investment of ₹ 1700 billion under the scheme of "Sustainable Alternative Towards Affordable Transportation (SATAT)". The scheme

will open up a new window of green energy corridor in the transport sector and employment as well. Oil and Gas PSUs, namely IOCL, HPCL, BPCL, GAIL and IGL are inviting Expression of Interest (EoI) for procurement of CBG from potential entrepreneurs. This initiative envisages expected annual production of 15 MMTCBF and 50 MMT bio manure.

2. Biogas

The Byproduct of Anaerobic digestion of organic material is commonly referred to as 'Biogas'. Biogas technology refers to the production of combustible gas (Biogas) and a value added fertilizer under controlled or uncontrolled conditions namely temperature, pH, C/N ratio etc.

3. Biogas generation process

The biogas generation process involves anaerobic digestion consisting primarily three stages, (i) Hydrolysis, (ii) Acid Formation, and (iii) Methane Fermentation.

The process is carried out by two sets of bacteria namely Acidogenic and Methanogenic Bacteria. Hydrolysis and Acid formation is generally combined and is called as 1st phase and Methane formation is considered as the 2nd phase.

1st phase - Hydrolysis and Acid formation.

In Hydrolysis process, the biomass (Protein, Fat and Carbohydrates) is broken down through the influence of Heat, Mechanical shredding, enzymatic treatment or a combination of all. The polymers are broken in to Monomers for ease of consumption of Methanogens.

Fats-----> Fatty Acids, Protein ----->Amino Acids, Carbohydrates -----> Sugars.

These products are fermented by the fermentation bacteria leading to the formation of the following products:

1. H₂, H₂O, CO₂, NH₄
2. Acetic acid (CH₃COOH)
3. Alcohol and low organic acids.

The pH of the material is lower in the process to around 5.5 to 6 and in some fast degrading materials such as corn starch or refined floor, it can drop to as low as 3.3. The pH has to be kept at around 6 for transitioning in to the second phase to avoid creating inhospitable conditions to the bacteria.

Acid reduction – The Alcohol and the low organic acids are fermented in to the following products through the action of Acetogenic Bacteria.

1. H₂O, CO₂, H₂
2. Acetic Acid

2nd Phase – Methane Production –

In this final phase, Acetic acid produced in the 1st phase is converted into CH₄ and CO₂ (combined called as Biogas) through the actions by Methanogenic bacteria. The residual waste is rich in Nitrogen and can usually be used as good organic manure. Depending on the raw material and its composition, other gases are also found in the Biogas such as Hydrogen Sulphide (H₂S) and water vapour.

4. Compressed Biogas (CBG)

Compressed Biogas is a common term used by the Ministry of Petroleum and Natural Gas. It can also be called as Bio-CNG. The standards and specifications are governed by IS 16087:2016 (**Annexure I**) of BIS.

IS 16087 : 2016 Standard		
S No.	Characteristic	Requirement
1	Methane percentage (CH ₄), minimum	90.0 %
2	Only Carbon Dioxide percentage (CO ₂),, maximum	4%
3	Carbon Dioxide (CO ₂)+ Nitrogen (N ₂)+ Oxygen (O ₂) percentage maximum	10%
4	Oxygen (O ₂) percentage maximum	0.5%
5	Total sulphur (including H ₂ S) mg/m ³ , maximum	20 mg/m ³
6	Moisture mg/m ³ , maximum	5 mg/m ³

The Standard also states the following:

1. CBG shall be free from liquids over the entire range of temperature and pressure encountered in storage and dispensing system.
2. The CBG shall be free from particulate matter such as dirt, dust, etc.
3. CBG delivered shall be odorized similar to a level found in local distribution (ref. IS 15319)

To achieve the desired quality stated in IS 16087:2016, The raw biogas needs to be cleaned /Scrubbed. There are many types of systems and processes available which have now matured to a certain level in India to confidently clean the Biogas to CBG standards to be used as vehicular fuel as per the Gazette Notification no. 395 dated 16th June 2015, Ministry of Road Transport and Highways, Government of India.

5. Scrubbing Technology

Composition of Biogas –

CH₄- 55-60%

CO₂ – 35 – 45%

H₂S- 0.1- 2%

Water vapour – saturated

Scrubbing depends on the end use of the biogas.

For H₂S scrubbing following are the comparative data:

Application	Scrubbing technology	Advantages	Disadvantages
Cooking	None	Low cost	Little hazardous with Sulphur di Oxide
			Small scale suitable
	Iron Oxide	Low CAPEX	Disposal of Fe ₂ SO ₄
Boiler	Iron Oxide	Low CAPEX and OPEX	Disposal of Fe ₂ SO ₄
	Water scrubber	Medium OPEX High Efficiency	High Capex Regeneration of water Disposal of H ₂ SO ₄
	Biological fixation	Medium OPEX Generation of elemental Sulphur	High Capex disposal of elemental Sulphur Disposal of H ₂ SO ₄ Disposal of sludge Low efficiency
Power generation	Iron Oxide	Low CAPEX and OPEX	Disposal of Fe ₂ SO ₄
	Water scrubber	High Capex Medium OPEX	Regeneration of water Disposal of H ₂ SO ₄
	NAOH chemical	High Efficiency	High Capex and OPEX Disposal of sulphates
CBG	Water scrubber	High Capex Medium OPEX	Regeneration of water Disposal of H ₂ SO ₄
	NAOH chemical	High Efficiency	High Capex and OPEX Disposal of sulphates
	PSA (Pressure Swing Absorption)	High Efficiency	High Capex and OPEX Disposal of zeolite media

For CO₂ removal the following technologies are suitable and are as governed by the economics of the project.

Parameter	PSA	Water Scrubber	Mono-ethylamine/ Mono-ethanolamine(MEA) System	Membrane Technology
Pre- H ₂ S removal required	Yes	No	Yes	Yes
Working pressure (bar)	4-7	4-7	No pressure	7-24
Methane loss	20-30%	5-10%	<0.1%	<0.5%
Methane content in upgraded gas	>96%	>97%	>99%	99%
Electricity consumption (kWh/m ³)	0.25	<0.25	<0.15	~0.23

6. Raw material/ Substrate for Biogas generation

The Biogas plant requires easily biodegradable organic matter / Biomass. The Substrates considered for Biogas plants are grossly categorized in the following categories.

1. Animal waste

Main source of animal waste are the following:

a. Dairy farms

India being one of the leading milk generating and processing country has one of the highest milking cattle population. They range from a single cattle in a house to a large mechanized dairy farm having thousands of cattle.

The Biogas plant is most suitable in any scale of the plant with variable end use of the generated biogas.

b. Poultry Farms

India has now growing poultry farms and there are clusters in some specific parts of the country where we have large concentration of poultry activity. They are located in parts of Telangana, Some parts of Maharashtra, Haryana, Tamilnadu and Chhatisgarh.

There are mainly two types of Poultry farms

1. Layer farm

2. Broiler farm

Layer farms are most suited for setting up of Biogas plant as medium to large poultries are now following cage batteries for keeping birds. This helps in collection of waste and ensures reduction in contamination. Many new large automatic layer farms are installing temperature controlled and belt extraction system where the litter/dropping are continuously removed to maintain hygiene. Such plant as most suited for large scale CBG or Electricity generation plants.

Broiler farms are abundant but are not very much suited as the birds are placed on hey/ sawdust or mud and the collection of waste is not practical.

The Manure generated through the biogas process is high in Nitrogen and is sought after by the farming community in both solid and liquid form.

c. Other animals such as Pigs, Horse, Camel etc.

In Indian society, piggeries and other animal husbandry is not very common and the economics and technology suitable for processing waste will be similar to cattle farming.

2. Plant waste/ Farm waste

Main source of animal waste are the following:

a. Paddy straw

The Paddy straw can be easily made available through mechanical harvesters and is easy to store in bales throughout the year.

The biggest challenge is the mechanical hydrolysis of paddy as it is coated with silica and is very abrasive to any moving parts in the process increasing the OPEX of the plant. The Manure produced through the process is rich in silica.

b, Corn/ Sweet sorghum

There are few varieties of Corn and sorghum specifically developed as fodder crop which has excellent biogas potential and can be used as an energy crop. The biggest challenge is

the completion with food security and high raw material rates making the biogas plant economically and socially unviable.

c. Napier grass

The grass is best suited as biogas plant energy crop as it can be easily grown on large areas and is easy to harvest. It is also found to be economically viable if the conditions to sell the products is easy.

3. **Process waste**

The process waste is generated through process industries processing organic material. They can be in form of High COD and BOD liquids and or solid organic waste.

4. **Municipal solid waste**

Due to urbanization, the increasing urban population has increased the amount of waste generated in urban Indian cities.

Out of the overall waste, around 45-55% waste is organic in nature. Out of this organic waste, only about 35-40% waste is suitable for Biogas.

Biggest challenge is the disposal of manure generated through the MSW plants as the raw material is not regulated, the quantity of heavy metals such as Mercury, Arsenic, lead might exceed the allowable parameters.

The Biomass such as Paper, Wood, Dried leaves, Wooden shavings are generally high in Lignin and Cellulose and these substances can theoretically be suitable for generation of biogas but practically looking at the technology and most importantly the economics is not suitable for the commercial biogas generation.

7. **Products of Biogas Process**

Other value added products generated from the biogas plant are:

1. **Enriched manure**

After the anaerobic process, most of the Active carbon is converted to energy and Biogas. What remains is further enriched and blended with other inorganic ingredients such as Phosphates and Potash to make them PROM i.e Phosphate Rich Organic Manure. It has been Gazetted under Fertilizer control order (**Annexure II**).

2. **Liquid manure**

The digested slurry can be used as Liquid manure as per guidelines brought under the FCO (Annexure III).

3. **Liquid CO₂ and Dry Ice**

Biogas has around 35-40% CO₂. But it requires large CAPEX to set up the plant and the technological restrictions make it very OPEX intensive as well. Two known technologies are Cryogenic and Chemical Amine process.

4. **Waste heat recovery system**

When the Biogas is converted to electricity through an internal combustion Engine or a Gas turbine. The Out-put is Electricity, CO₂, and heat.

Above 100 kW capacity engines the heat can be recovered indirectly through exhaust gas and cooling circuit through waste heat recovery systems. They can internally be used to heat the biological process or provide process heat to other applications.

8. Categorization of CBG/Bio-CNG plants

Central Pollution Control Board has categorized industrial sectors in various categories viz. Red, Orange, Green and White on the basis of pollution potential, which is represented as Pollution Index, a function of quantity and quality of emissions (air pollutants), effluents (water pollutants), and types of hazardous wastes generated. Pollution Index is composite score (100 marks) in which 40 marks is assigned to air pollution, 40 marks to water pollution and 20 marks to hazardous waste.

CPCB revised the categorization of Compressed Bio-Gas (CBG) plants in light of the notifications issued by the Ministry of Agriculture and Farmers Welfare vide Gazette Notification No. 2051 dated 14.07.2020 and No. 1972 dated 01.06.2021 regarding inclusion of Fermented Organic Manure (FOM) and Liquid Fermented Organic Manure (LFOM) under Fertilizer (Inorganic, Organic or Mixed) (Control) Act, 1985.

As per revised categorization, CBG plants producing FOM & LFOM as by products in conformity with requirements of Gazette Notification No. 2051 dated 14.07.2020 & No. 1972 dated 01.06.2021, respectively, and utilizing entire FOM & LFOM as a fertilizer or manure on land and also not discharging any waste-water, are to be considered under White category, subject to verification by SPCB on case-to-case basis. The aforesaid criteria may be re-assessed based on ground conditions after a period of two years.

CBG plants producing FOM & LFOM as by products not conforming with requirements of Gazette Notification No. 2051 dated 14.07.2020 & No. 1972 dated 01.06.2021 are to be categorized based on the type of feed-stocks being used. CBG plants based on animal waste and crop residue as feedstock are categorized under green category. CBG plants based on Municipal Solid Waste (MSW) and process waste as feedstock are categorized under Orange Category.

Household bio-digesters/gobar-gas (cow-dung) plants based on biodegradable wastes, etc. with feed slurry to digesters having Volatile Organic Fraction more than 75 %, are to be considered under White' category. The details of categorization of 'Compressed Biogas (CBG)/Bio-CNG plants' and 'Household bio-digesters/gobar-gas (cow-dung) plants based on biodegradable wastes, etc.' are given in “**Annexure IV**”.

CBG plant currently placed under Green & Orange category can be considered as white category after ensuring recycle/reuse of entire liquid effluent generated by installing required additional treatment units based on effluent characteristics and producing FOLM & FOM as by products using digested biogas slurry & digested sludge in conformity with requirements of FCO as per Gazette Notification No. 2051 dated 14.07.2020 & No. 1972 dated 01.06.2021.

Green & Orange category CBG/BIO-CNG plant can take steps as given in “**Annexure V**” to qualify as white category.

9. Guidelines to be followed by CBG Plants

I Handling of Solids Waste / Input material

1. Biodegradable solid waste/input material should be stored in such a manner that leachates formed do not get mix with any natural water body or stream.
2. If solids have any fire hazard possibility due fire extinguishing system should be set up.
3. Reject/undigested Solids should be disposed in accordance with SWM Rules,2016 & its amendments and shall not be mixed with digested slurry for disposal.
4. Digested solid/sludge shall be used for producing FOM or Bio-compost. In case of Bio-compost, following shall be ensured:
 - a) The composting facilities of adequate size may be designed through expert institutions in the field.
 - b) The compost yard be provided with impermeable base with facility for collection of leachate and surface water run-off into lined drains leading to a leachate treatment and disposal facility.
 - c) The composting facility shall not be located within 300 m from the nearest dwelling and 100 m from any well or water course.
 - d) Necessary precaution shall be taken to minimise nuisance of odour, flies, rodents, bird menace and fire hazard.
 - e) The windrow area shall be provided with impermeable base. Such a base shall be made of concrete or compacted clay of 50 cm thick having permeability coefficient less than 10^{-7} cm/sec. The base shall be provided with 1 to 2 per cent slope and circled by lined drains for collection of leachate or surface run-off.
 - f) Leachate collection system shall be constructed. Leachate can be re-circulated in compost plant for moisture maintenance.
 - g) The end product compost shall meet the standards prescribed under Fertilizer Control Order notified from time to time.
 - h) The daily logbook record of the solid waste generated and bio-compost shall be maintained.
5. Groundwater monitoring
 - a) Location of piezometer- wells: Minimum at 2 places along the periphery of the bio-compost yard such that one is in the upstream of the Ground water flow direction and one in the downstream direction.
 - b) Hand pump: at least 30 meters depth, located within 500 meters to 1 Km from yards. Water quality of hand pump should be tested pre and post monsoon.
6. Approach Road to bio-compost yard: The entrance of the Bio-compost yard should be paved all-weather road for approach of vehicles.

7. Packaging and Labelling/Marking -The bio-compost or Bio manure shall be packed and labelled/marked properly.
8. Storage facilities for ready compost should be covered under shed having platform.
9. All other wastes such as iron filings, waste oils, transformer oils, batteries & other e-waste should be recycled using registered vendors.

II Solid Manure:

10. Solid manure should be stored in leachate-free way.
11. Solid manure can be used in farms with or without any value addition in conformity with requirements of Gazette Notification No. 2051 dated 14.07.2020 & No. 1972 dated 01.06.2021.
12. Solid manure may be converted to PROM (Phosphate Rich Organic Manure), Organic Potash fertilizer, organic silica fertilizer etc.

III Liquid Manure (Fermented Organic Manure):

13. Digested slurry while producing FOLM should be separated for solids as much as possible.
14. Use decanter, screw press, filter media or drying pit for separation of solids.
15. Liquid manure should be stored in leachate-free way.
16. Liquid manure can be used in farms with or without any value addition after confirming the stipulated quality requirements.
17. Liquid manure can be applied in farms after required treatment, depending on feed material and in conformity with requirements of Gazette Notification No. 2051 dated 14.07.2020 & No. 1972 dated 01.06.2021 and applicable effluent discharge standard. There shall be no adverse impact of application of digested biogas slurry on soil & environment.
18. Impermeable tank having total storage capacity of 15 days shall be provided for storing the liquid manure.
19. Any concurrent use of fertilizers (along with LOM) shall be done judiciously to avoid any superimposed effect.
20. In no case, the Liquid manure/effluent/leachate shall not be discharged into the drain/nallah/surface water bodies/channels/rivers.
21. The liquid concentrated nutrient rich fertilizer product after post processing shall be labelled and packed in containers for sale.
22. The daily logbook record of the Liquid manure produced by the unit shall be maintained.

IV Self-monitoring system

23. A flow-meter shall be installed at the wastewater/slurry generation/discharge line to quantify the slurry being generated for treatment. A PTZ camera shall be installed at storage tank and bio-compost area to monitor the bio-composting operation.

V Scrubber System:

24. CBG Plants use following types of scrubbers:

- a) Iron chelating based for H₂S removal.
 - Disposal of sulphur recovered from the process should be sold/disposed off in correct manner as per rule.
 - All waste streams coming from plant should be suitably treated & recycled/reused. In no case, effluent enters water body.
- b) PSA for CO₂ removal
 - Exhaust gas being released to atmosphere in safe manner.
 - Height of exhaust gas chimney to be raised to 3m above the roof level at least.
 - Iron filings, silica gel should be disposed of through certified vendors.
- c) Membrane for CO₂ removal
 - Membrane material disposal should be done through registered vendors only.
- d) Water scrubber for CO₂ and H₂S removal.
 - Effluent generated which is high in acids should be neutralized by using suitable method & then disposed off after meeting the notified effluent discharge norms.

VI Other Requirements

25. Ambient air quality monitoring shall be regularly carried out.
26. Odour nuisance at down-wind direction on the boundary of processing plant shall also be checked regularly & accordingly necessary steps for its control shall be taken.
27. The Gensets shall comply with the latest Indian emission norms for Gas based Engines.
28. The biogas plants may be integrated with Solar PVs to make these plants cost economically viable
29. The CBG plant shall install flow meter at all water abstraction points for measuring water consumption & maintain logbook for the same.

इंटरनेट

मानक

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Mazdoor Kisan Shakti Sangathan

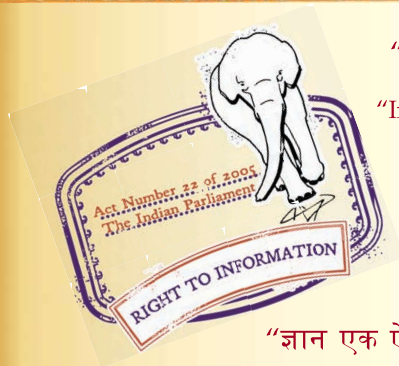
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“Step Out From the Old to the New”

IS 16087 (2013): Biogas (Biomethane) - Specification [PCD
3: Petroleum, Lubricants and their Related Products]



“ज्ञान से एक नये भारत का निर्माण”

Satyanarayan Gangaram Pitroda

“Invent a New India Using Knowledge”



“ज्ञान एक ऐसा खजाना है जो कभी चुराया नहीं जा सकता है”

Bharṭhari—Nītiśatakam

“Knowledge is such a treasure which cannot be stolen”

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IS 16087 : 2013

भारतीय मानक
बायोगैस (बायोमीथेन) — विशिष्टि

Indian Standard
BIOGAS (BIOMETHANE) — SPECIFICATION

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NEW DELHI 110002

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Price Group 1

Petroleum, Lubricants and Their Related Products Sectional Committee, PCD 3

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Petroleum, Lubricants and Their Related Products Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

The purpose of this standard is to provide general guidelines for the biogas (biomethane) composition, quality parameters and for biogas (biomethane) thermal application, applications in stationary engines, automotive applications and supply through piped network.

Biogas (biomethane) as a fuel, meeting the requirements of the compositional standards, should,

- a) provide the safe operation of the engine whether stationary or automotive and associated equipments;
- b) protect the fuel system from the detrimental effects of corrosion, poisoning and liquid or solid deposition; and
- c) not emit any pollutants or the greenhouse gases after combustion, beyond prescribed limit.

Biogas (biomethane) is primarily methane gas which is generated from an anaerobic digestion of organic wastes by micro organisms. It is a relatively simple and economical method to produce a fuel from waste. While technically biogas (biomethane) can be produced from any type of organic material, most times, biogas (biomethane) is produced from organic waste. This waste could comprise agricultural and crop waste, human waste and animal waste (cow dung for instance). Biogas (biomethane) is an environment friendly, clean, cheap and versatile fuel which can be used for various applications.

Biogas (biomethane) is a natural product produced from the biodegradable substrates like cattle dung, poultry waste, food waste, sewage waste etc. It has methane as the main component around 50-70 percent, CO₂ around 30-40 percent and H₂S and moisture in trace quantities. It varies in composition depending upon the substrates used. The raw biogas (biomethane) from the biogas (biomethane) digesters is properly processed and purified from the unwanted gases like CO₂, H₂S and moisture upto a certain required level.

While implementing this standard all the regulatory and statutory provisions shall be complied.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

*Indian Standard***BIOGAS (BIOMETHANE) — SPECIFICATION****1 SCOPE**

This standard prescribes the requirements and the methods of sampling and test for the biogas (biomethane) applications in stationary engines, automotive and thermal applications and supply through piped network.

2 REFERENCES

The following standards contain provisions which through reference in this text constitute the provisions of the standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standard indicated below:

<i>IS No./ International Standard</i>	<i>Title</i>
1070 : 1992	Reagent grade water (<i>third revision</i>)
7285 (Part 2) : 2004	Refillable seamless steel gas cylinders: Part 2 Quenched and tempered steel cylinders with tensile strength less than 1 100 MPa (112 kgf/mm ²)
15125 : 2002/ ISO 10715 : 1997	Natural gas — Sampling guidelines
15130 (Part 3) : 2002/ ISO 6974-3 : 2000	Natural gas — Determination of composition with defined uncertainty by gas chromatography: Part 3 Determination of hydrogen, helium, oxygen, nitrogen, carbon dioxide and hydrocarbons up to C ₈ using two packed columns
15319 : 2003/ ISO 13734 : 1998	Natural gas — Organic sulphur compounds used as odorants — Requirements and test methods
15320 : 2003/ ISO 15403 : 2000	Natural gas — Designation of the quality of natural gas for use as a compressed fuel for vehicles
15490 : 2004	Cylinders for on-board storage of compressed natural gas as a fuel for automotive vehicles
15641 (Part 2) : 2006/ ISO 10101-2 : 1993	Natural gas — Determination of water by Karl Fischer method: Part 2 Titration procedure

<i>IS No./ International Standard</i>	<i>Title</i>
ISO 6326-3 : 1989	Natural gas — Determination of sulphur compounds: Part 3 Determination of hydrogen sulphide, mercaptan sulphur and carbonyl sulphide sulphur by potentiometry
ISO 14532 : 2001	Natural gas — Vocabulary

3 TERMINOLOGY

For the purpose of this standard, the definitions given in ISO 14532 shall apply.

4 REQUIREMENTS

4.1 Biogas (biomethane) shall be free from liquids over the entire range of temperatures and pressures encountered in the storage and dispensing system, fuel containers, engine and fuel system and piped network.

4.2 The biogas (biomethane) fuel shall be free from particulate matter such as dust, dirt and mist.

4.3 Odour

Biogas (biomethane) delivered to any vehicle, stationary engine or piped network, shall be odorized similar to a level found in the local distribution (see IS 15319).

4.4 The biogas (biomethane) for automotive application and piped network shall also comply with the requirements given in Table 1 when tested in accordance with the methods given in col 4 of Table 1.

Table 1 Requirements for Biogas (Biomethane)

Sl No.	Characteristic	Requirements	Method of Test, Ref to
(1)	(2)	(3)	(4)
i)	CH ₄ , percent, <i>Min</i>	90	IS 15130 (Part 3)
ii)	Moisture, mg/m ³ , <i>Max</i>	16	IS 15641 (Part 2)
iii)	H ₂ S, mg/m ³ , <i>Max</i>	30.3	ISO 6326-3
iv)	CO ₂ +N ₂ +O ₂ , percent, <i>Max (v/v)</i>	10	IS 15130 (Part 3)
v)	CO ₂ , percent, <i>Max (v/v)</i> (when intended for filling in cylinders)	4	IS 15130 (Part 3)
vi)	O ₂ , percent, <i>Max (v/v)</i>	0.5	IS 15130 (Part 3)

IS 16087 : 2013

This biogas (biomethane) may also be used for applications such as stationary engines or power generators.

5 SUPPLY OF BIOGAS (BIOMETHANE)

5.1 Biogas (biomethane) shall be stored and transported through cylinders conforming to IS 7285 (Part 2). For automotive use, it shall be filled in cylinders conforming to IS 15490.

5.2 It may be transported through piped network or injected into existing system of pipelines as per regulatory requirements.

6 SAMPLING

Proper sampling of biogas (biomethane) is extremely

important, if the tests are to be significant. Samples of biogas (biomethane) are examined by various test methods to determine physical and chemical characteristics. The test results are often used for custody transfer. It is, therefore, essential that the samples be representative of the product to be tested. The representative samples of biogas (biomethane) shall be drawn as prescribed under IS 15125.

7 QUALITY OF REAGENTS

Unless specified otherwise, pure chemicals and distilled water shall be used in tests (*see* IS 1070).

NOTE — Pure chemicals shall mean chemicals that do not contain impurities which affect the result of the analysis.

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This Indian Standard has been developed from Doc No.: PCD 3 (2573).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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MINISTRY OF AGRICULTURE
(Department of Agriculture and Co-operation)

ORDER

New Delhi, the 22nd June, 2012

S.O. 1420(E).—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 further to amend the Fertilizer (Control) Order, 1985, namely:-

1. (1) This Order may be called the Fertilizer Control (Amendment) Order, 2012.
(2) It shall come into force on the date of its publication in the Official Gazette.
 2. In Fertilizer (Control) Order, 1985,-
 - (A) in Schedule I, in Part A, under the heading "SPECIFICATIONS OF FERTILISERS",-
 - (i) in sub-heading "1 (d) N.P. COMPLEX FERTILISERS",-
 - (a) serial number "12. Nitrophosphate (23- 23- 0)" and the entries relating thereto, shall be omitted;
 - (b) after serial number 17 and the entries relating thereto, the following serial number and entries shall be inserted, namely:-
- "18. Nitrophosphate (24:24:0)
- | | |
|--|------|
| (i) Moisture per cent. by weight, maximum | 1.5 |
| (ii) Total nitrogen per cent. by weight, minimum | 24.0 |
| (iii) Nitrogen in ammonical form per cent. by weight, minimum | 13.5 |
| (iv) Nitrogen in nitrate form, per cent. by weight, maximum | 10.5 |
| (v) Neutral ammonium citrate soluble phosphate (as P_2O_5) per cent. by weight, minimum | 24.0 |
| (vi) Water soluble phosphate (as P_2O_5) per cent. by weight, minimum | 20.5 |
| (vii) Particle size: Not less than 90 per cent. of the material shall pass through 4mm IS sieve and be retained on 1mm IS sieve.
Not more than 5 per cent shall be below 1mm IS sieve." | |
- (ii) in sub-heading "1 (f) MICRONUTRIENTS", in serial number "8 Chelated Zinc as Zn-EDTA", for the words, "Appearance- free flowing crystalline/powder" the words "Appearance- free flowing crystalline or powder or tablet" shall be substituted
 - (iii) in sub-heading "1 (g) FORTIFIED FERTILISERS",-
 - (a) for serial number 3 and the entries relating thereto, the following serial number and entries shall be substituted, namely:-

"3. Zincated Phosphate (Suspension) – for seed treatment

37485/2022/IPC-III-HQ(i)	Total phosphate (as P_2O_5) per cent. by weight, minimum.	13.9
(ii)	Total zinc (as Zn) per cent. by weight, minimum.	17.6
(iii)	Neutral ammonium citrate soluble phosphate (as P_2O_5) per cent. by weight, minimum	2.8
(iv)	Lead (as Pb) per cent. by weight, maximum	0.003
(v)	pH	8+/-1";

- (b) after serial number 10 and the entries relating thereto, the following serial number and entries shall be inserted, namely:-

"11. DAP fortified with Zinc (18:46:0:0.5)

(i)	Moisture per cent. by weight, maximum.	2.5
(ii)	Total nitrogen per cent. by weight, minimum	18.0
(iii)	Ammonical nitrogen per cent. by weight, minimum	15.5
(iv)	Urea nitrogen percent. by weight, maximum	2.5
(v)	Neutral ammonium citrate soluble phosphate (as P_2O_5) per cent. by weight, minimum	46.0
(vi)	Water soluble phosphate (as P_2O_5) per cent. by weight, minimum	41.0
(vii)	Zinc (as Zn) per cent. by weight, minimum	0.5
(viii)	Particle size: Not less than 90 per cent of the material shall pass through 4mm IS sieve and be retained on 1mm IS sieve. Not more than 5 per cent shall be below 1mm IS sieve."	

- (iv) in sub-heading "1 (h) 100% WATER SOLUBLE COMPLEX FERTILISER", after serial number 16 and the entries relating thereto, the following serial number and entries shall be inserted, namely:-

"17 NPKZn (7.6 : 23.5 :7.6 :3.5)

(i)	Moisture per cent. by weight, maximum	0.5
(ii)	Total nitrogen per cent. by weight, minimum	7.6
(iii)	Nitrate nitrogen per cent. by weight, maximum	2.8
(iv)	Ammonical nitrogen per cent. by weight, minimum	5.0
(v)	Water soluble phosphate (as P_2O_5) per cent. by weight minimum	23.5
(vi)	Water Soluble Potash (K_2O) per cent. by weight, minimum	7.6
(vii)	Water Soluble Zinc (as Zn EDTA) per cent. by weight, minimum	3.5
(viii)	Sodium (as NaCl) per cent. by weight, on dry basis maximum	0.15
(ix)	Matter insoluble in water per cent. by weight, maximum";	0.5

- (B) in Schedule III, -

(i) in Part A, under the heading "SPECIFICATIONS OF BIOFERTILIZERS" after serial number 5 and the entries relating thereto, the following serial number and entries shall be inserted, namely:-

"6. Potassium Mobilizing Biofertilizers (KMB)

1.	Base	Carrier based in form of moist/dry powder or granules, or liquid based
2.	Viable cell count	CFU minimum 5×10^7 cells/g of powder, granules or carrier material on dry weight basis or 1×10^8 cell/ml of liquid
3.	Contamination	No contamination at 10^5 dilution
4.	pH	6.5 – 7.5 for carrier based in form of powder or granules and 5.0 – 7.5 for liquid based
5.	Particle size in case of carrier based moist powder	Powder material shall pass through 0.15 to 0.212 mm IS sieve
6.	Moisture per cent. by weight, maximum in case of powder based	30 – 40
7.	Efficiency character	Minimum 10 mm solubilization zone in prescribed media having at least 3mm thickness.

Type of carrier – The carrier material such as peat, lignite, peat soil, humus, talc or similar material favouring growth of microorganisms.

7. Zinc Solubilizing Biofertilizers (ZSB)

1.	Base	Carrier based in form of moist/dry powder or granules, or liquid based
2.	Viable cell count	CFU minimum 5×10^7 cells/g of powder, granules or carrier material on dry weight basis or 1×10^8 cell/ml of liquid
3.	Contamination	No contamination at 10^5 dilution
4.	pH	6.5 – 7.5 for carrier based in form of powder or granules and 5.0 – 7.5 for liquid based
5.	Particle size in case of carrier based moist powder	Powder material shall pass through 0.15 to 0.212 mm IS sieve
6.	Moisture content percent. by weight, maximum in case of carrier based	30 – 40
7.	Efficiency character	Minimum 10 mm solubilization zone in prescribed media having at least 3mm thickness."

(ii) in PART D, under the heading METHODS OF ANALYSIS OF BIOFERTILISERS", after serial number 1E and entries relating thereto, the following entries shall be inserted, namely:-

"IF. Method of analysis for Potash Solubilizing Biofertilizers (KSB)

1. Estimation of total viable count and contamination

1. Apparatus -

1.1 Pipettes graduated 1ml and 10 ml

1.2 Dilution bottles or flasks

1.3 Petri dishes clear, uniform, flat-bottomed

1.4 Hot -air oven

Capable of giving uniform and adequate temperature, equipped with a thermometer, calibrated to read upto 250°C and with vent suitably located to assure prompt and uniform heating.

1.5 Autoclave

1.6 Incubator

1.7 Hand tally or mechanical counting device

1.8 pH meter

2. Reagents

2.1 Medium

Use plating medium of the following composition for total viable count and contamination

Medium for analysis of total viable count and contamination

(Ingredients g/lit)

Manitol	15.0
Yeast extract	3.0
Peptone	2.0
Agar	18.5
Trace element solution	1 ml
Distilled Water	1000 ml

Trace element solution

(Ingredients g/lit)

Sodium molybdate	0.20
Boric acid	0.28
Manganese sulphate	0.23
Copper sulphate	0.01
Zinc sulphate	0.03
Distilled Water	1000 ml

Medium for studying zone of solubilization in KSB

(Ingredients g/lit)

Glucose	5.0
Magnesium sulphate	0.005
Ferric chloride	0.1
Calcium carbonate	2.0
Potassium mineral (mica powder)	2.0
Calcium phosphate	2.0
Distilled water	1000 ml

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2.2 Sterilizing and preparation procedure for plates

2.2.1 Sterilize the sampling and plating equipment with dry heat in a hot air oven at less than 160°C for not less than 2 hours;

2.2.2 Sterilize the media by autoclaving at 120°C for 20 min. To permit passage of steam into and from closed containers when auto claved, keep stoppers slightly loosened or plugged with cotton. Air from within the chamber of the sterilizer should be ejected allowing steam pressure to rise.

2.3 Preparation of plating medium and pouring

2.3.1 Prepare growth medium in accordance with the composition of the specific biofertiliser.

2.3.2 Melt the required amount of medium in boiling water or by exposure to flowing steam in partially closed container but avoid prolonged exposure to unnecessarily high temperature during and after melting. Melt enough medium which will be used within 3 h. Re-sterilisation of the medium may cause partial precipitation of ingredients.

2.3.3 When holding time is less than 30 min. promptly cool the molten medium to about 45°C, and store until used, in a water bath or incubator at 43 to 45°C. Introduce 12 to 15 ml of liquefied medium or appropriate quantity depending on size of the petri dish at 42 to 44°C into each plate. Gently lift the cover of the dish just enough to pour in the medium. Sterilise the lips of the medium containers by exposure to flame.

- (a) Immediately before pouring.
- (b) Periodically during pouring, and
- (c) When pouring is complete for each batch of plates, if portions of molten medium remain in containers and are to be used without subsequent sterilization for pouring additional plates. As each plate is poured thoroughly mix the medium with test portions in the petri dish.

2.3.4 By rotating and tilting the dish and without splashing the medium over edge, spread the medium evenly over the bottom of the plate. Provide conditions so that the medium solidifies with reasonable promptness (5-10 min) before removing the plates from level surface.

3. Preparation of Serial Dilution for Plate Counts:

3.1. Dispense 10 g of inoculants to 90 ml of sterile distilled demineralized water and shake for 10 min on a reciprocal shaker or homogenizer. Make serial dilutions upto 10^{10} . Take 1:0 ml or suitable aliquots of 10^6 to 10^9 dilutions using sterile pipettes and deliver to petri dishes containing set medium as given in 2.1 and spread it uniformly with a spreader or use droplet method. Invert the plates and promptly place them in the incubator.

3. Incubation of Plates:

4.1 Label the plates and incubate at $28 \pm 2^\circ\text{C}$ for 4 to 6 days.

4.2 Colony counting aids:

Count the colonies with the aid of magnifying lens under uniform and properly controlled, artificial illumination. Use a colony counter, equipped with a guide plate and rules in centimeter square. Record the total number of colonies with the hand tally. To distinguish colonies from dirt, specks and other foreign matter, examine doubtful objects carefully.

4.3 Count all plates but consider for the purpose of calculation plates showing more than 30 and less than 300 colonies per plate. Disregard colonies which absorb congo red and stand out as reddish colonies. *Fruturia aurentia* (KMB) stand out as white-opaque glistening and domed colonies. Count such colony numbers and calculate figures in terms of per litre, of carrier. Also check for freedom from contamination at 10^5 dilution.

4. Counting

Count the total number of colonies on the plates including colonies with solubilisation zone with the help of a colony counter.

5. Method for estimation of K solubilization zones

6.1 Take 10 g of KSB in 90 ml sterile distilled water

6.2 Make a tenfold dilution series up to 10^7 .

6.3 Take 1.0 ml aliquot of 10^5 to 10^7 dilutions using sterile pipettes and deliver to petri dishes containing K-solubilization zone media.

6.4 Spread it uniformly, Invert the plates and incubate for up to 2 weeks at $28 \pm 2^\circ\text{C}$.

6.5 Count the colonies showing solubilization zones and measure the diameter of solubilization zone. Calculate average zone of solubilization in mm.

1G. Method of analysis for Zinc Solubilizing Biofertilizers

2. Estimation of total viable count and contamination

1.Apparatus -

1.1 Pipettes graduated 1ml and 10 ml

1.2 Dilution bottles or flasks

1.3 Petri dishes clear, uniform, flat-bottomed

1.4 Hot -air oven

Capable of giving uniform and adequate temperature, equipped with a thermometer, calibrated to read upto 250°C and with vent suitably located to assure prompt and uniform heating.

1.5 Autoclave

1.6 Incubator

1.7 Hand tally or mechanical counting device

1.8 pH meter

2. Reagents

2.1 Medium

Use plating medium of the following composition for total viable count and contamination

Medium for analysis of Total Viable Count, Contamination and zone of solubilisation for Zn solubilizing biofertilizer

(Ingredients g/lit)

Glucose

10.0

Zinc oxide	1.0
Amm sulphate	0.5
Potassium chloride	0.2
Yeast extract	0.5
Ferrous sulphate	0.01
Manganese sulphate	0.01
Di Pot Hyd.phosphate	0.5
Distilled water	1000 ml

2.2 Sterilizing and preparation procedure for plates:

2.2.1 Sterilize the sampling and plating equipment with dry heat in a hot air oven at less than 160°C for not less than 2 hours;

2.2.2 Sterilize the media by autoclaving at 120°C for 20 min. To permit passage of steam into and from closed containers when auto claved, keep stoppers slightly loosened or plugged with cotton. Air from within the chamber of the sterilizer should be ejected allowing steam pressure to rise.

2.3 Preparation of plating medium and pouring

2.3.1 Prepare growth medium in accordance with the composition of the specific Biofertiliser.

2.3.2 Melt the required amount of medium in boiling water or by exposure to flowing steam in partially closed container but avoid prolonged exposure to unnecessarily high temperature during and after melting. Melt enough medium which will be used within 3 hours. Re-sterilization of the medium may cause partial precipitation of ingredients.

2.3.3 When holding time is less than 30 min. promptly cool the molten medium to about 45°C, and store until used, in a water bath or incubator at 43 to 45°C. Introduce 12 to 15 ml of liquefied medium or appropriate quantity depending on size of the petri dish at 42 to 44°C into each plate. Gently lift the cover of the dish just enough to pour in the medium. Sterilise the lips of the medium containers by exposure to flame.

a. Immediately before pouring.

b. Periodically during pouring, and

c. When pouring is complete for each batch of plates, if portions of molten medium remain in containers and are to be used without subsequent sterilization for pouring additional plates. As each plate is poured thoroughly mix the medium with test portions in the petri dish.

2.3.4 By rotating and tilting the dish and without splashing the medium over edge, spread the medium evenly over the bottom of the plate. Provide conditions so that the medium solidifies with reasonable promptness (5-10 min) before removing the plates from level surface.

3. Preparation of Serial Dilution for Plate Counts:

3.1 Dispense 10 g of inoculants to 90 ml of sterile distilled de-mineralized water and shake for 10 min on a reciprocal shaker or homogenizer. Make serial dilutions upto 10^{10} . Take 1.0 ml or suitable aliquots of 10^6 to 10^9 dilutions using sterile pipettes and

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deliver to petri dishes containing set medium as given in 2.1 and spread it uniformly with a spreader or used droplet method. Invert the plates and promptly place them in the incubator.

4. Incubation of Plates:

- 4.1 Label the plates and incubate at $28 \pm 2^\circ\text{C}$ for 4 to 6 days.
- 4.2 Colony counting aids:

Count the colonies with the aid of magnifying lens under uniform and properly controlled, artificial illumination. Use a colony counter, equipped with a guide plate and rules in centimeter square. Record the total number of colonies with the hand tally.

- 4.3 Count all plates but consider for the purpose of calculation plates showing more than 30 and less than 300 colonies per plate. Disregard colonies which absorb congo red and stand out as reddish colonies. Zinc solubilising biofertilisers stand out as white, translucent, glistening and elevated colonies. Count such colony numbers and calculate figures in terms of per litre, of carrier. Also check for freedom from contamination at 10^5 dilution.

5. Counting

Count the total number of colonies on the plates including colonies with solubilization zone with the help of a colony counter.

6. Method for estimation of Zinc solubilisation zones

- 6.1 Take 10 g of ZSB in 90 ml sterile distilled water
- 6.2 Make a tenfold dilution series up to 10^7 .
- 6.3 1.0 ml aliquot of 10^5 to 10^7 dilutions using sterile pipettes and deliver to petri dishes containing Zinc - solubilization zone media.
- 6.4 Spread it uniformly, Invert the plates and incubate for up to 2 weeks at $28 \pm 2^\circ\text{C}$.
- 6.5 Count the colonies showing solubilization zones and measure the diameter of solubilization zone. Calculate average zone of solubilization in mm.

- (D) In Schedule IV, in PART A, under the heading "SPECIFICATIONS OF ORGANIC FERTILISERS", after serial number 2 and the entries relating thereto, the following serial number and entries shall be inserted, namely: -

"3. Phosphate rich Organic manure (PROM)

(i)	Moisture per cent. by weight, maximum	15.0-25.0
(ii)	Particle size- Minimum 90% material should Pass through 4.0 mm IS sieve	
(iii)	Bulk density (g/cm^3)	1.646
(iv)	Total organic carbon per cent. by weight, minimum	7.87
(v)	Total nitrogen (as N) per cent. by weight, minimum	0.42
(vi)	Total phosphates (as P_2O_5) per cent. by weight, minimum	10.42
(vii)	Total potash (as K_2O) per cent. by weight, minimum	-

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(viii)	C: N ratio	18.73:1
(ix)	pH (1:5 solution) maximum	6.72
(x)	Conductivity (as dSm^{-1}) not more than	8.27
(xi)	Heavy metal content (as mg/kg), maximum	
	Arsenic (as As_2O_3)	10.0
	Cadmium (as Cd)	5.0
	Chromium (as Cr)	50.0
	Copper (as Cu)	300.0
	Mercury (as Hg)	0.15
	Nickel (as Ni)	50.0
	Lead (as Pb)	100.0
	Zinc (as Zn)	1000.0

[F. No. 2-1/2012-Fert.Law]

NARENDRA BHOOSHAN, Jt. Secy.

Note :—The principal order was published in the Gazette of India, Extraordinary, Part II, section 3, sub-section (i) vide number G.S.R. No. 758(E) dated 25th September, 1985 and was subsequently amended vide S.O. No. 2203 dated 22nd September, 2011



भारत का राजपत्र The Gazette of India

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असाधारण
EXTRAORDINARY

भाग II—खण्ड 3—उप-खण्ड (ii)
PART II—Section 3—Sub-section (ii)

प्राधिकार से प्रकाशित
PUBLISHED BY AUTHORITY

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No. 1972]

NEW DELHI, TUESDAY, JUNE 1, 2021/JYAISTHA 11, 1943

कृषि और किसान कल्याण मंत्रालय

(कृषि, सहकारिता और किसान कल्याण विभाग)

आदेश

नई दिल्ली, 31 मई 2021

का.आ. 2126(अ).— केन्द्रीय सरकार, आवश्यक वस्तु अधिनियम, 1955 (1955 का 10) की धारा 3 द्वारा प्रदत्त शक्तियों का प्रयोग करते हुए, उर्वरक (अकार्बनिक, कार्बनिक या मिश्रित) (नियंत्रण) आदेश, 1985 का और संशोधन करने के लिए निम्नलिखित आदेश बनाती है, अर्थात्:-

1. (1) इस आदेश का संक्षिप्त नाम उर्वरक (अकार्बनिक, कार्बनिक या मिश्रित) (नियंत्रण) तीसरा संशोधन आदेश, 2021 है।

(2) ये राजपत्र में प्रकाशन की तारीख को प्रवृत्त होंगा।

2. उर्वरक (अकार्बनिक, कार्बनिक या मिश्रित) (नियंत्रण) आदेश, 1985 (जिसे इसमें इसके पश्चात् उक्त आदेश कहा गया है) की अनुसूची 1 के भाग क में शीर्ष "उर्वरक की विशिष्टताएं" के अधीन,-

(i) "1(ग) स्ट्रेट पोटेशियम उर्वरक" उपशीर्ष के अधीन क्रम संख्यांक 6 और उससे संबंधित प्रविष्टियों के पश्चात् निम्नलिखित क्रम संख्यांक और प्रविष्टियां अंतःस्थापित की जाएंगी, अर्थात्:-

"7 पोटेशियम मैग्निशियम सल्फेट (दानेदार)"

(i)	भार के आधार पर आर्द्रता का प्रतिशत, अधिकतम	0.5
(ii)	भार के आधार पर एमजीओ के रूप में मैग्निशियम का प्रतिशत, न्यूनतम	10.0

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(iii)	भार के आधार पर के ₂ ओ के रूप में पोटाश का प्रतिशत, न्यूनतम	30.0
(iv)	भार के आधार पर एस के रूप में सल्फेट सल्फर का प्रतिशत, न्यूनतम	17.0
(v)	भार के आधार पर कुल क्लोराइड का प्रतिशत, न्यूनतम	2.5
(vi)	सामग्री के कण का आकार ऐसा होगा कि सामग्री का न्यूनतम 90 प्रतिशत 5 एमएम और 2 एमएम भारतीय मानक छलनी के बीच रह जाएगा।	

(ii) उपशीर्ष “1(छ). सूक्ष्म पोषकतत्व” के अधीन क्रम संख्यांक 23 और उससे संबंधित प्रविष्टियों के पश्चात् निम्नलिखित क्रम संख्यांक और प्रविष्टियां अंतःस्थापित की जाएगी, अर्थात्:--

“24. मैग्निशियम हाइड्रोक्साइड और जिंक फास्फेट

(i)	भार के आधार पर एमजी के रूप में मैग्निशियम का प्रतिशत, न्यूनतम	24.0
(ii)	भार के आधार पर जेडएन के रूप में जिंक का प्रतिशत, न्यूनतम	10.0
(iii)	पीएच (50 ग्रा/ली)	8.5+/-1
(iv)	भार के आधार पर पी ₂ ओ ₅ के रूप में उपलब्ध फास्फोरस का प्रतिशत, न्यूनतम	2.5”

(iii) उपशीर्ष “1(झ). 100 प्रतिशत जल में घुलनशील संश्लिष्ट उर्वरक” के अधीन पोटेशियम नाइट्रेट (प्रिल्ड)(मृदा उपयोजन) से संबंधित क्र.सं. 7 के मद (vi) में अंक “0.5” के स्थान पर “1.5” अंक रखे जाएंगे।

(iv) उपशीर्ष “1(ज). फायदाप्रद अवयव उर्वरक” के अधीन क्रम संख्यांक (2) और उससे संबंधित प्रविष्टियों के पश्चात् निम्नलिखित क्रम संख्यांक और प्रविष्टियां अंतःस्थापित की जाएगी, अर्थात्:--

“3 सोडियम सिलिकेट (द्रव)

(i)	भार के आधार पर एसआईओ ₂ के रूप में सिलिकॉन का प्रतिशत, न्यूनतम	23.8
(ii)	भार के आधार पर एनए के रूप में सोडियम का प्रतिशत, न्यूनतम	6.0
(iii)	विनिर्दिष्ट गुरुत्व	1.3”

(v) उपशीर्ष “1(ट). द्रव उर्वरक” के अधीन क्रम संख्यांक 5 और उससे संबंधित प्रविष्टियों के पश्चात् निम्नलिखित क्रम संख्यांक और प्रविष्टियां अंतःस्थापित की जाएगी, अर्थात्:--

“6. एनके 6:0:18 कैल्शियम, मैग्निशियम और बोरॉन के साथ फोर्टीफाईड (सस्पेंशन)

(i)	भार के आधार पर नाइट्रोजन का प्रतिशत, न्यूनतम	6.0
(ii)	भार के आधार पर एन के रूप में नाइट्रेट नाइट्रोजन का प्रतिशत, न्यूनतम	5.8
(iii)	भार के आधार पर के ₂ ओ के रूप में जल में घुलनशील पोटाश का प्रतिशत, न्यूनतम	18.0
(iv)	भार के आधार पर जल में घुलनशील कैल्शियम (सीएओ के रूप में) का प्रतिशत, न्यूनतम	5.0
(v)	भार के आधार पर जल में घुलनशील मैग्निशियम (एमजीओ के रूप में) का प्रतिशत, न्यूनतम	2.0
(vi)	बी के रूप में बोरॉन	0.5-0.8
(vii)	20 डिग्री सेंटीग्रेड पर पीएच (1 प्रतिशत विलयन)	8 से 9

7. 11:11:8 एनकेपी जिंक और बोरॉन के साथ फोर्टीफाईड (सस्पेंशन)

(i)	भार के आधार पर नाइट्रोजन का प्रतिशत, न्यूनतम	11.0
(ii)	भार के आधार पर यूरिया नाइट्रोजन का प्रतिशत	7.2

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(iii)	भार के आधार पर अमोनिकल नाइट्रोजन का प्रतिशत	3.0
(iv)	भार के आधार पर जल में घुलनशील फास्फोरस (पी ₂ ओ ₂ के रूप में) का प्रतिशत, न्यूनतम	11.0
(v)	भार के आधार पर जल में घुलनशील पोटेशियम (के ₂ ओ के रूप में) का प्रतिशत, न्यूनतम	8.0
(vi)	जैडएन-ईडीटीए के रूप में भार के आधार पर जैडएन के रूप में जिंक का प्रतिशत	0.7
(vii)	बी के रूप में बोरॉन	0.5-0.7
(viii)	20 डिग्री सेंटीग्रेड पर पीएच (1 प्रतिशत विलयन)	7.0-8.0

8. मैग्निशियम के साथ फोटोफाइट कैल्शियम नाइट्रेट (सस्पेंशन)

क्रम सं.		मान
(i)	भार के आधार पर कुल नाइट्रोजन का प्रतिशत, न्यूनतम	10.0
(ii)	भार के आधार पर नाइट्रेट नाइट्रोजन का प्रतिशत, न्यूनतम	8.5
(iii)	भार के आधार पर जल में घुलनशील सीएओ के रूप में कैल्शियम का प्रतिशत, न्यूनतम	15.0
(iv)	भार के आधार पर एमजीओ के रूप में जल में घुलनशील मैग्निशियम का प्रतिशत, न्यूनतम	2.0
(v)	भार के आधार पर कुल (सीएल के रूप में) क्लोराइड का प्रतिशत	2.5
(vi)	20 डिग्री सेंटीग्रेड पर पीएच (1 प्रतिशत विलयन)	8.0-9.0

3. उक्त आदेश की अनुसूची 2 के भाग ख की क्रम संख्यांक 26 में,-

(1) मद (i) के स्थान पर निम्नलिखित रखा जाएगा, अर्थात् :-

“(i) कुल जस्ते का अवधारण”

क्षेत्र: जिंक आक्साइड सस्पेंशन सांद्रित से जस्ते (जैडएन), आर्सेनिक (एएस), सीसा (पीबी) और कैडमियम (सीडी) का कुल अर्क।

मूल : बायलिंग एक्वा रेजिन के साथ नमूने से प्राप्त जिंक और भारी धातु अशुद्धियां।

(क) नमूना तैयार करना :

उर्वरक के नमूने निकालने के लिए प्रक्रिया के ब्यौरे अनुसूची 2 के भाग क की क्रम संख्या 9 पर दिए गए हैं (हाइड्रोस अमोनिया से भिन्न तरल उर्वरक के नमूने लेने की पद्धति) तथापि नमूने लेने से पहले और विश्लेषण के लिए अशेषभाजक निकालने से पहले तत्वों को समुचित रूप से हिलाने और मिलाने के महत्व पर विशेष जोर दिया जाता है। चूंकि सस्पेंशन फॉर्मूलेशन अधुलनशील सामग्री समय के साथ नीचे बैठ जाती है, तत्वों को मिलाने के दौरान आद्यान की समुचित गहराई के साथ प्रतिनिधितात्मक संमिश्रण सुनिश्चित किया जाएगा।

(ख) अभिकर्मक :

अवधारित किए जाने वाले तत्वों का नगण्य सांद्रण सुनिश्चित करने के लिए सभी अभिकर्मक विश्लेषक श्रेणी के होने चाहिए।

(1) ग्लास द्वि आसुत जल (सूक्ष्म पोषकतत्वों से मुक्त)

(2) हाइड्रोक्लोरिक अम्ल 37 प्रतिशत एचसीएल (एचसीएल)=12 एमओएल/ली, पी=1.18 ग्रा./मि.लि.)

(3) नाइट्रिक अम्ल 65 प्रतिशत एचएनओ₃ {सी(एचएनओ₃)=14.3 एमओएल/ली, पी=1.4 ग्रा./मि.लि.}}

(ग) उपकरण :

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अभिक्रिया बरतन और प्रतिवाह संग्राही के साथ तापीय तापन उपचारण के लिए उपकरण।

बरतन उपयोग किए गए एक्वा रेजिया के आयतन का कम से कम पांच गुणा होना चाहिए।

यदि प्रतिवाह संग्राही के साथ अभिक्रिया बरतन उपलब्ध न हो, उस प्रयोजन के लिए वाँच ग्लास से ढके हुए एलैनमेयर फ्लास्क या उच्च बीकर प्रयुक्त किए जा सकते हैं।

यदि छानना आवश्यक है तो राख रहित फिल्टर पेपर अपेक्षित है।

(घ) प्रक्रियां:

- (1) नमूने का एक ग्राम भार (1 ± 0.001 ग्रा) तौले और अभिक्रिया बरतन में मात्रात्मक रूप से अंतरित करे।
- (2) लगभग 0.5 से 1.0 मि.लि. आसुत जल के साथ नमूने को भिगोये।
- (3) तत्वों को अच्छे से मिलाए और झाग कम करने के लिए 7 मि.लि. एचएनओ₃ अभिकर्मक के साथ 21 मि.लि एचसीएल दोनों को बूंद बूंद मिलाए।
- (4) अभिक्रिया बरतन के साथ संवाहक को जोड़ें और बुदबुदाहट बंद होने तक मिश्रण को प्रयोगशाला में कमरे के तापमान पर छोड़ दें।
- (5) तापन युक्ति को चालू करे और उतार स्थिति के लिए अभिक्रिया मिश्रण का तापमान धीरे धीरे बढ़ाए। इसे 2 घण्टे तक बनाये रखे।
- (6) यह सुनिश्चित करे कि संवाहन क्षेत्र संवाहक की ऊँचाई के आधे से कम रहे।
- (7) उतार के 2 घण्टे पश्चात्, उसे ठण्डा होने दें और 10 मि.लि. आसुत जल के संवाहक को धोकर साफ करे।
- (8) पदार्थ को 500 मि.लि. आयतन वाले फ्लास्क में अंतरित करे और चिन्ह तक पानी के साथ तनु करे। परीक्षण विलयन नमूने से 500 गुना तनु हो।
- (9) परीक्षण विलयन को यदि आवश्यक हो छानना (जिंक ऑक्साइड संपेशन सांद्रण के लिए अपेक्षित नहीं होगा)। यदि छान लिया जाए, विश्लेषण के लिए प्रथम 20 मि.लि. (लगभग) भाग अलग निकाल दें।
- (10) नमूने के समान प्रक्रिया का अनुसरण करते हुए निरंक परीक्षण विलयन तैयार करना। इसे अभिक्रमक के माध्यम से संभाव्य किसी संदूषण की दशा में विश्लेषण के पार्श्व शोधन के लिए उपयोग में लिया जाएगा।
- (11) मापन तुरंत किया जा सकेगा या पंद्रह दिन तक कसकर बंद किए गए प्लास्टिक के पात्र में भंडारित किया जा सकेगा।

टिप्पण:- अभिक्रिया पात्र में आक्टोनल की एक बूंद मारक कर्मक के रूप में प्रयुक्त की जा सकती है।

(ङ) निष्कर्षित पदार्थ में जिंक का अवधारण :

जिंक के विश्लेषण के लिए प्रक्रिया पद्धति संख्या 7(iii) (ख) (2) चरण (आ) या पद्धति संख्या 8(ii) (ख) चरण (2) में विनिर्दिष्ट प्रक्रिया के अनुसार होगी।

टिप्पण:- संगणना को मूल नमूने के तनुकरण के परिमाण को ध्यान में रखते हुए तदनुसार समायोजित किए जाने की आवश्यकता है।

(2) मद (ii) में “8((v) में विनिर्दिष्ट” शब्दों, अंक और कोष्टकों के स्थान पर “मद (i) के उपमद (घ) में यथा विनिर्दिष्ट एक्वारेजिया के माध्यम से निष्कर्षण के पश्चात् 8(v) में विनिर्दिष्ट” शब्द, अंक और कोष्टक रखे जाएंगे ;

(3) प्रविष्टि (iii) के पश्चात् निम्नलिखित प्रविष्टियां अंतःस्थापित की जाएंगी, अर्थात्:-

“(iv) आर्सेनिक के अवधारण के लिए :

मद (i) के उपमद (घ) में यथा विनिर्दिष्ट एक्वा रेजिया के माध्यम से निष्कर्षण के पश्चात् 8(ix) में विनिर्दिष्ट पद्धति द्वारा।

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(v) कैडमियम के अवधारण के लिए :

मद (i) के उपमद (घ) में यथा विनिर्दिष्ट एक्वा रेजिया के माध्यम से निष्कर्षण के पश्चात् 8(x) में विनिर्दिष्ट पद्धति द्वारा।”

(4) क्रम संख्यांक 29 और उससे संबंधित प्रविष्टियों के पश्चात् निम्नलिखित क्रम संख्यांक और प्रविष्टियां अंतःस्थापित की जाएंगी, अर्थात्:-

“30. सोडियम सिलिकेट के विश्लेषण की पद्धति :

(i) सिलिकोन का अवधारण :

(क) रसायन और कांचपात्र :-

(1) सांद्रित एचसीएल

(2) सांद्रित एचएनओ₃

(3) गर्म प्लेट

(4) घड़िया प्लेटेनियम

(5) मफल भट्टी (एक हजार डिग्री सेंटीग्रेड तापमान क्षमता वाली)

(6) शोषित्र और अन्य नित्यक्रम प्रयोगशाला कांचपात्र।”

(ख) प्रक्रिया :

(1) टैफलॉन या कॉर्निंग बीकर में तैयार किए गए नमूने का एक ग्राम लें, उसमें 2-3 मि.ली. एचसीएल विलयन (1:1) और 2-3 मि.ली. एचएनओ₃ विलयन (1:1) मिलाएं।

(2) विलयन को एक गर्म प्लेट में उसके अर्द्ध ठोस होने तक संग्रहित करें, उसे ठंडा करें और उसमें दुबारा 3-4 मि.ली. एचएनओ₃ विलयन मिलाएं। उसे गर्म प्लेट पर पूरी तरह से सुखाएं।

(3) 2-3 मि.ली. एचसीएल विलयन (1:1) मिलाएं और उसे पीला धुआं खत्म होने तक उबालें। उसे फिल्टर पेपर संख्या 1 से छान लें, 10 मि.ली. एचसीएल विलयन (1:1) के साथ एक बार धो लें और पीला रंग गायब होने तक प्रत्येक 10 मि.ली. भाग को गर्म पानी से 2-3 बार धोएं।

(4) तुलाई-पूर्व घड़िया प्लेटेनियम के अवशिष्ट के साथ फिल्टर पेपर डाले और उसे मफल भट्टी में 250 डिग्री मापमान पर डेढ़ घंटे तक सुखाएं और अंतिम रूप से अवशिष्ट को 950 डिग्री तापमान पर 30 डिग्री के लिए प्रज्वलित करें।

(5) शोषित्र में घड़िया को ठंडा करें और पुनः तुलाई करें और संगणित करें।

संगणना:-

एसआईओ₂ के रूप में एसआई का प्रतिशत = घड़िया का अंतिम भार – खाली घड़िया का भार X 100/नमूने का भार

(i) सोडियम का अवधारण

क्रम संख्यांक 17 में यथा विनिर्दिष्ट पद्धति द्वारा

(ii) विनिर्दिष्ट गुरुत्व का अवधारण

क्रम संख्यांक 17 में यथा विनिर्दिष्ट पद्धति द्वारा

31. नेनो नाइट्रोजन के विश्लेषण की पद्धति :

(i) नाइट्रोजन का अवधारण – कुल केजलदाल नाइट्रोजन

(क) प्रक्रिया :-

(1) तरल नेनो उर्वरक नमूना: केजलदाल फ्लास्क में नमूने की समभाग मात्रा (10 मि.ली.) लें।

(2) ठोस नेनो उर्वरक नमूने की दशा में: केजलदाल फ्लास्क में चूर्णित नेनो उर्वरक नमूने का एक ग्राम डालें।

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- (3) 15 ग्राम के₂एसओ₄ या 12 ग्राम एन-हाइड्रोस एनए₂एसओ₄, 0.4 ग्राम एन-हाइड्रोस सीयूएसओ₄ या 0.6 ग्राम सीयूएसओ 4.5 एच₂ओ और लगभग 0.8 ग्राम एलूनडम कणिकाएं मिलाएं।
- (4) यदि पर्याप्त संवातन उपलब्ध हो, जल एच₂एसओ₄ + एच₂ओ (1+1, वी/वी) या 20 मि.ली. सांद्रित सल्फ्यूरिक अम्ल के साथ 37 मि.ली. तनु सल्फ्यूरिक अम्ल मिलाएं
- (5) परीक्षण द्रव्यमान वाले पर्याप्त भाग संक्षिप्त रूप से क्रमशः 30 से 5 प्रतिशत नाइट्रोजन के साथ उर्वरक के लिए 0.1000 से 2.800 ग्राम मिलाएं। 10 मि. ली. पानी से भीतरी दीवार धोएं।
- (6) फ्लास्क को पहले से गर्म केजलदाल ब्लॉक उपचारक (400 डिग्री सेंटीग्रेड) में डालें और परीक्षण भाग का 75 मिनट के लिए उपचारण करें।
- (7) फ्लास्क को गर्म ब्लॉक से हटाएं और ठंडा करें (अभिक्रिया मिश्रण कमरे के तापमान के आसपास होना चाहिए)। 20-30 मि.ली. पानी के साथ आंतरिक दीवार साफ करें।
- (8) परीक्षण भाग में कुल प्रत्याशित नाइट्रोजन को ट्रेप करने के लिए 30 मि.ली. 0.25 एन मानकीकृत सल्फ्यूरिक अम्ल मिलाकर आसुत ग्राही फ्लास्क तैयार करें।
- (9) मिथाइल बैंगनी संकेतक की 2-3 बूंदें मिलाएं और तनुकरण इकाई की निर्गम नली पर यह सुनिश्चित करते हुए कि तनुकरण निर्गम नली का सिरा मानकीकृत अम्ल विलयन में निमज्जित हो जाए, ग्राही प्रतिष्ठापित करें।
- (10) तनुकरण इकाई पर उपचारण नली प्रतिष्ठापित करें। भाप बनाना प्रारंभ करें और फ्लास्क में लगभग 80 मि.ली. (30-35 प्रतिशत) सोडियम हाइड्रोक्सिल धीरे-धीरे छोड़ दें।
- (11) भाप तनुकरण इकाई को तब तक जारी रखें जब तक कि लगभग 250 मि.ली. या उससे अधिक भाप संवहन ग्राही फ्लास्क में एकत्रित न हो जाए। इसमें सामान्य रूप से 6-8 मिनट लगना अपेक्षित है।
- (12) यदि रंग बदल कर हरा हो जाता है, तो पुनः बैंगनी रंग लाने के लिए 0.25 एन एच₂एसओ₄ मिलाएं और मिलाए गए अम्ल की मात्रा अभिलिखित करें।
- (13) 0.25 एन मानक एनएओएच के साथ धूसर संतुलांक (पीएच 5.7) का अनुमापन करें। आसुत का रंग, परीक्षण भाग में नाइट्रोजन की कुल मात्रा पर निर्भर करता है, जो ग्राही फ्लास्क में ट्रेड अमोनिया की मात्रा का कृत्य है।
- (14) हरा रंग उपदर्शित करता है कि ट्रेप में अम्ल, अमोनिया द्वारा निष्प्रभावी कर दिया गया था। इस बिंदु पर, धूसर संतुलांक पाने के लिए मानकीकृत एच₂एसओ₄ की अतिरिक्त ज्ञात मात्रा मिलाएं।
- (15) मानकीकृत अम्ल का कुल आयतन (मि.ली.) प्रारंभ ग्राही फ्लास्क में मिलाए गए अम्ल की कुल मात्रा और आसवन के पश्चात् धूसर संतुलांक पर पहुंचने के लिए मिलाए गई अम्ल की मात्रा के बराबर होगा। नीला या बैंगनी रंग उपदर्शित करता है कि ग्राही फ्लास्क में अभी अम्ल है और एनएओएच के साथ वापस अनुमापन अपेक्षित है।
- (16) मानकीकृत अम्ल का कुल आयतन, धूसर संतुलांक तक पहुंचने के लिए आसवन के पश्चात् डाले गए क्षार की मात्रा घटाने के पश्चात् ग्राही फ्लास्क में अम्ल की मात्रा के बराबर होगा।

(ख) परिकलन:-

कुल नाइट्रोजन के भार प्रतिशत का निम्नानुसार परिकलन किया जाएगा :-

कुल एन का प्रतिशत = (कुल मानक अम्ल मि.ली. X मानक अम्ल का एन) / (कुल मानक क्षार मि.ली. के X मानक क्षार का एन) X 1.4008/नमूने का भार, ग्रा.

(ii) भौतिक कण आकार {पारेषण इलेक्ट्रान सूक्ष्मदर्शी (टीईएम) विश्लेषण के अनुसार} :

(क) उपस्कर और उपकरण :

(1) पारेषण इलेक्ट्रान सूक्ष्मदर्शी

(2) नमूना ग्रीड

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(3) मोचनी, पेट्रीडिश, इथेनॉल और वियायनीकृत जल।

(ख) प्रक्रिया:

- (1) टीईएम नमूना ग्रिड कार्बन आवरण वाली फिल्म जो जस्ते की जाली या समतुल्य द्वारा समर्थित इलेक्ट्रान पारदर्शी हो, उपयुक्त होगी।
- (2) नमूने तैयार करने के लिए उपयोग में लिए गए कांचपात्र और उपकरण, फिल्टर से साफ किए गए, पानी द्वारा धातु रहित किए गए और शुष्क भंडारित होने चाहिए।
- (3) कणों का प्रसार और रचना करने के लिए उपयोग किए जाने वाले में उपकरण चूड़ीदार ढक्कन के साथ छोटी कांच की शीशी, एक दस मि.मी. उचाई का टैफ्लॉन स्तंभ, जो शीशी में अतःस्थापित किया जा सकेगा, एक पेट्री डिश और 40 गुणा 40 चौड़ाई का एक टैफ्लॉन खंड सम्मिलित होंगे।
- (4) ग्रिड को मोचनी से पकड़ा जाएगा, ग्रिड को पूर्ण रूप से इथेनॉल में डूबोकर सांफ किया जाएगा। ग्रिड से फिल्टर पेपर का प्रयोग करते हुए, तरल के आधिक्य को हटाया जाएगा। शुष्क ग्रिड को साफ टैफ्लॉन खंड में रखा जाएगा।
- (5) ग्रिड में नैनो कण विलयन की 10 μ ली. मात्रा रखी जाएगी।
- (6) ग्रिड को पेट्री डिश ढक्कन से ढका जाएगा और ग्रिड को शुष्क होने के लिए कमरे के तापमान पर आम तौर पर, विलायक के प्रकार पर निर्भर करते हुए 5 से 30 मिनट तक छोड़ दिया जाएगा।
- (7) ग्रिड को टीईएम नमूना धारक पर अंतरित करें। नियत आवर्धन पर प्रचालित अच्छी तरह से संरेखित और स्थिर टीईएम का प्रयोग करते हुए ग्रिड के व्यापक रूप से पृथक्कित न्यूनतम दो क्षेत्रों से न्यूनतम 200 नैनो कण प्रति नमूना प्रति ग्रिड स्ववायर प्रतिबिम्बित करने के लिए पर्याप्त सूक्ष्म ग्राफ अभिलिखित करें जो यह सुनिश्चित करते समय कि प्रत्येक व्यष्टि नैनो कण प्रतिबिम्ब पिक्सल की वृहद संख्या के साथ अभिलिखित किया जाए, दृष्टि क्षेत्र के सूक्ष्म ग्राफ क्षेत्र के भीतर दृश्य होने के लिए वृहत् संख्या में नैनो कणों को अनुज्ञात करता है।

(ग) पूर्वविधानियां :

- (1) पारेषण इलेक्ट्रान सूक्ष्मदर्शी ग्रिड (विशिष्टतया पतला फिल्म का पर्दा) बहुत भंगुर होता है और अच्छी मोचनी के साथ उसके किनारों द्वारा पकड़ा जाना चाहिए जिससे झिल्ली को क्षति या उसमें दरार न हो।
- (2) धूल संदूषण की संभावना को कम करने के लिए पर्यावरण परिवेश में पारेषण इलेक्ट्रान सूक्ष्मदर्शी ग्रिड का प्रसार न्यूनतम होना चाहिए। ग्रिड धूल-मुक्त या शुष्क कमरों में उपयुक्त बक्सों में भंडारित की जानी चाहिए।
- (3) सही कण आकार परिणाम प्राप्त करने के लिए एक अच्छी तरह से संरेखित पारेषण इलेक्ट्रान सूक्ष्मदर्शी का होना आवश्यक है।
- (4) नमूने के व्यापक रूप से पृथक्कित कम से कम दो क्षेत्रों से (जो भिन्न-भिन्न ग्रिड चौरस या झिल्ली क्षेत्र हैं) न्यूनतम 200 पृथक कण मापे जाने चाहिए। दिए गए प्रतिबिम्ब में बाहरी कचरे (उदाहरणार्थ साफ करने और सुखाने की प्रक्रिया से धूल के कण या अपशिष्ट) से बचना चाहिए।
- (5) पारेषण इलेक्ट्रान सूक्ष्मदर्शी माप से प्राप्त परिणामी कण आकार उनके अनुरूप नहीं होंगे जो अन्य प्रौद्योगिकियों (उदाहरणार्थ गतिशील प्रकाश प्रकीर्ण) से प्राप्त किए गए हैं। यह प्रत्येक मामले में अवधारित भार अनुपात में भिन्नता साथ ही साथ भौतिक गुणधर्म में भिन्नता जो वास्तविक रूप से मापी गई है (उदाहरणार्थ प्रक्षिप्त क्षेत्र बनाम द्रवगति के प्रसार क्षेत्र) से टुकड़ों में रह जाते हैं (उदाहरणार्थ पारेषण इलेक्ट्रान सूक्ष्मदर्शी के लिए संख्या बनाम गतिशील प्रकाश प्रकीर्ण के लिए तीव्रता)।

(iii) द्रव्यगति कण आकार (गतिशील प्रकाश प्रकीर्ण विश्लेषण के अनुसार) :**(क) उपस्कर और उपकरण :**

- (1) गतिशील प्रकाश प्रकीर्ण उपस्कर
- (2) आकार की नमूना कुठाली

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(3) विआयनीकृत जल

(ख) प्रक्रियाएं

- (1) तरल नेनो- उर्वरक नमूने के लिए, एक मिनट के लिए दस मि.ली. ध्वनीकृत किया जाएगा और विश्लेषण के लिए उपयोग में लिया जाएगा। ठोस नेनो उर्वरक की दशा में, उर्वरक:आसुत जल (1:10 अनुपात) प्रलंबन तैयार किया जाएगा, एक मिनट ध्वनिकृत किया जाएगा।
- (2) मशीन को स्थापित करने के लिए ज्ञात मानक नमूने (जो कोई एक नेनो पार्टिकल जैसे कि एजी, एयू या टीआईओ₂ है) का उपयोग किया जाएगा।
- (3) कुठाली में नमूना रखा जाएगा।
- (4) विलायक के साथ पूर्व प्रक्षालन (फिल्टर के आकार और फिल्टर धारक या कार्टेज के निष्क्रिय आयतन पर निर्भर करते हुए कम से कम 1 मि.ली.)
- (5) नमूने में सूई लगाने के पश्चात् और फिल्टर में सूई निविष्ट करने के पश्चात्, पहली चार बूंदें बह जाने देंगे। अगली चार बूंदें कुठाली के पूर्व प्रक्षालन के लिए उपयोग की जाएगी और फैंक दी जाएगी। शेष बचे हुए का नमूने के मापन के लिए उपयोग किया जाएगा।
- (6) आपके विशिष्ट उपकरण के विन्यास के लिए लेजर बीम की प्रवेश ऊंचाई से लगभग 2 मि.ली. ऊपर तक तरल स्तर सुनिश्चित करने के लिए आवश्यक न्यूनतम मात्रा का उपयोग करते हुए कुठाली में नमूना डाला जाएगा।
- (7) कुठाली में नमूना डालते समय इस बात का ध्यान रखा जाएगा कि आप खाली हाथ से उसके किरिचगोले को न छुएं। यदि आवश्यक हो तो लेंस कागज से क्वार्टेज या कांच की कुठाली को बाहर से पोंछें। धूल संदूषण और विलायक वाष्पीकरण को रोकने के लिए कुठाली को ढकें।
- (8) कुठाली का निरीक्षण यह सुनिश्चित करने के लिए करें कि वायु के बुलबुले प्रकाशीय किरिचगोला क्षेत्र में न हों।
- (9) नमूने को गतिशील प्रकाश प्रकीर्ण मशीन में डालें।
- (10) प्रति नमूने तीन से दस स्वतंत्र माप लें।

(ग) पूर्वावधानियां:

- (1) ठोस चरण नेनो नाइट्रोजन कणों का आकार नापने के लिए एक ग्राम नेनो कणों का नमूना बीस मि.ली. पानी में मिलाया जाना चाहिए तत्पश्चात् गतिशील प्रकाश प्रकीर्ण द्वारा आकार मापने से पहले एक मिनट के लिए अति ध्वनिकृत किया जाना चाहिए।
- (2) मापन कुठाली को छाने हुए विलवणीकृत पानी से साफ किया जाना चाहिए और शुष्क भंडारित किया जाना चाहिए।
- (3) छिद्र आकार का विकल्प परीक्षण कणों के अधिकतम विन्यास पर और उनकी फिल्टर झिल्ली से चिपकने की प्रवृत्ति पर निर्भर करता है। μ प्रलंबित माध्यम (जैसे कि विलायक, डिसप्रसेंट, विलयन) को नमूना तैयार करने से पहले 0.1 या 0.2 का प्रयोग करते हुए छान लेना चाहिए।
- (4) प्ररूपिक आरंभिक नमूना सांद्रण 1 मि.ग्रा./मि.ली. होगा।
- (5) क्वार्टेज या समतुल्य प्रकाशीय-क्वालिटी किरिचगोलों के साथ कुठाली का प्रयोग करें।
- (6) कुठाली का छाने हुए विलायक के साथ कम से कम तीन बार प्रक्षालन करें।

(iv) जेटा पोटेंशियल विश्लेषण :**(क) उपस्कर और उपकरण**

- (1) गतिशील प्रकाश प्रकीर्ण (डीएलएस)
- (2) जेटा की नमूना कुठाली

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(3) विआयनीकृत जल

(ख) प्रक्रियाएं:

- (1) तरल नेनो- उर्वरक नमूने के लिए दस मि.ली. एक मिनट के लिए ध्वनीकृत किया जाएगा और विश्लेषण के लिए उपयोग में लिया जाएगा और ठोस नेनो उर्वरक की दशा में, उर्वरक:आसुत जल (1:10 अनुपात) प्रलंबन तैयार किया जाएगा, एक मिनट के लिए ध्वनीकृत किया जाएगा।
- (2) मशीन को स्थापित करने के लिए ज्ञात मानक नमूने (जो कोई एक नेनो कण जैसे कि एजी, एयू या टीआईओ₂ है) का उपयोग किया जाएगा।
- (3) कुठाली में नमूना रखा जाएगा।
- (4) विलायक के साथ पूर्व प्रक्षालन किया जाएगा (फिल्टर के आकार और फिल्टर धारक या कार्टेज के निष्क्रिय आयतन पर निर्भर करते हुए कम से कम 1 मि.ली.)
- (5) नमूने में सूई लगाने के पश्चात् और फिल्टर में सूई निविष्ट करने के पश्चात्, पहली चार बूंदें बह जाने देंगे। अगली चार बूंदें कुठाली के पूर्व प्रक्षालन के लिए उपयोग की जाएगी और फैंक दी जाएगी। शेष बचे हुए का नमूने के मापन के लिए उपयोग किया जाएगा।
- (6) आपके विशिष्ट उपकरण विन्यास के लिए लेजर बीम की प्रवेश ऊंचाई से लगभग 2 मि.ली. ऊपर तक तरल स्तर सुनिश्चित करने के लिए न्यूनतम आवश्यक मात्रा का उपयोग करते हुए कुठाली में नमूना डाला जाएगा।
- (7) कुठाली में नमूना डालते समय इस बात का ध्यान रखा जाएगा कि आप खाली हाथ से उसके किरिचगोले को न छुएं। यदि आवश्यक हो तो लेंस कागज से क्वार्टेज या कांच की कुठाली को बाहर से पोंछें।
- (8) धूल संदूषण और विलायक वाष्पीकरण को रोकने के लिए कुठाली को ढकें।
- (9) कुठाली का निरीक्षण यह सुनिश्चित करने के लिए करें कि वायु के बुलबुले प्रकाश किरिचगोला क्षेत्र में न हों।
- (10) नमूने को गतिशील प्रकाश प्रकीर्ण मशीन में डालें।
- (11) प्रति नमूने तीन से दस स्वतंत्र माप लें।

(ग) पूर्वावधानियां:

- (1) मापन कुठाली को छाने हुए विलवणीकृत जल से साफ किया जाना चाहिए और शुष्क भंडारित किया जाना चाहिए। छिद्र आकार का विकल्प परीक्षण कणों के अधिकतम विन्यास पर और उनकी फिल्टर झिल्ली से चिपकने की प्रवृत्ति पर निर्भर करता है। μ प्रलंबित माध्यम (जैसे कि विलायक, डिसप्रसेंट, विलयन) को नमूना तैयार करने से पहले 0.1 या 0.2 का प्रयोग करते हुए छान लेना चाहिए।
- (2) प्ररूपिक आरंभिक नमूना सांद्रण 1 मि.ग्रा./मि.ली. होगा।
- (3) क्वार्टेज या समतुल्य प्रकाशीय-क्वालिटी किरिचगोले के साथ कुठाली का प्रयोग करें।
- (4) कुठाली का छाने हुए विलयन के साथ कम से कम तीन मिनट प्रक्षालन करें।

(v) श्यानता माप :

(क) उपस्कर और उपकरण

- (1) विलवणीकृत जल
- (2) श्यानता मापी
- (3) मापने का बेलन
- (4) बोतल अनुकूलक

(ख) प्रक्रिया:

- (1) 1 सीपीएस के रूप में मशीन को स्थापित करने के लिए आसुत जल के साथ श्यानता का अंशांकन करेगा।
- (2) श्यानतामापी जोड़ेगा।

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- (3) उस दशा में प्रदान किए गए प्रकुंच का उपयोग करते हुए आधार से लंब स्तंभ को जोड़ना।
- (4) लंब स्तंभ से श्यानता मापी को जोड़ना।
- (5) श्यानता मापी को शक्ति केबल से जोड़ना।
- (6) श्यानता मापी और अंशांकन को चालू करना।
- (7) चालू करना-अपेक्षित धूरी का चयन करना और जोड़ना।
- (8) लंब स्तंभ पर के पेच का प्रयोग करते हुए श्यानता मापी को उच्चतम स्तर तक उठाना।
- (9) धूरी के अधीन बीकर में तरल डालना।
- (10) श्यानता मापी को नीचे करना जब तक कि धूरी, धूरी चिन्ह के साथ निमग्न हो जाए।
- (11) श्यानता मापी का प्रचालन:-उपकरण के सॉफ्टवेयर पर निर्भर करता है। विशिष्ट उपकरण के लिए विनिर्माता के प्रचालन अनुदेशों का विश्लेषक द्वारा पालन किया जाना चाहिए। कमरे के ताप पर 1 सीपीएस मापने के लिए आसुत जल का प्रयोग करते हुए श्यानता मापी को स्थापित करें।

(ग) पूर्वावधानियां:

- (1) पठन से पहले 30-60 सेकंड के लिए प्रतीक्षा करें।
- (2) 60%-80% बल आघूर्ण की सीमा में अच्छे परिणाम आते हैं।
- (3) परिणाम, बीकर और तरल के आयतन पर निर्भर करते हैं इसलिए तुलनात्मक माप के लिए समान बीकर का प्रयोग करें। तरल नेनो-उर्वरक की श्यानता एन सीधे रूप से मापी जाती है।
- (4) ठोस नेनो उर्वरक के लिए, उर्वरक: जल विन्यास आसुत जल के साथ 1:10 अनुपात में होना चाहिए।

(vi) पीएच का माप :

(क) उपकरण :

पीएच मीटर, निर्वात पंप, बीकर, नल्लीका, कांच की डंडी, चाइना डिश, स्पैचुला, आदि।

(ख) अभिकर्मक

पीएच 4.0, 7.0 और 9.2 का बफर विलयन : पानी में संबंधित पीएच की एक बफर टेबलेट घोली जाएगी और आयतन 100 मि.ली. किया जाएगा।

प्रक्रिया:

- (1) तरल नमूने का 10 मि.ली. लेंगे, उसे एकरूप करेंगे और पीएच का माप लेंगे। ठोस/पाउडर नमूनों के लिए (1 ग्रा. नमूना/10 मि.ली. जल) एक मिनट के लिए नमूने का एकरूपकरण या अल्ट्रासोनिक आलोड़न करेंगे और पीएच माप नमूने के स्थिर हो जाने के पश्चात् लिया जाएगा।
- (2) पीएच मीटर को कमरे के ताप पर रखा जाएगा और पीएच 4.0, 7.0 और 9.2 के विभिन्न बफर विलयनों में इलेक्ट्रोडों को डुबो कर अंशांकन किया जाएगा।
- (3) एकरूपकृत किए हुए नमूनों को बीकर में भरा जाएगा और उसमें इलेक्ट्रोडों को डुबोया जाएगा और पीएच की रीडिंग लेंगे।
- (4) प्रत्येक अवधारण के पश्चात् इलेक्ट्रोडों को आसुत जल से धो देना चाहिए और साधारण फिल्टर कागज से साफ कर देना चाहिए।

(घ) पूर्वावधानियां :

- (1) उचित एकरूपकरण/ध्वनिकरण किया जाना चाहिए।

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(2) पीएच मीटर का कांच और संदर्भ इलेक्ट्रोड हमेशा पानी में डूबी रहनी चाहिए।

(3) बफर विलयन विशुद्ध रूप से तैयार किया और कांच के आधान में अच्छी तरह भंडारित किया जाना चाहिए।

(4) यह वांछनीय है कि कुछ दिनों के पश्चात् नया बफर विलयन तैयार किया जाना चाहिए। पीएच पठन में उतार-चढ़ाव से बचने के लिए पीएच मीटर को स्टेबलाइजर पर जोर देना चाहिए। पीएच के सही अवधारण के लिए पीएच मीटर की घुण्डी को कमरे के ताप पर समायोजित करना चाहिए।

4. अनुसूची 4 के भाग क में क्रम संख्यांक 9 और उससे संबंधित प्रविष्टियों के पश्चात् निम्नलिखित क्रम संख्यांक और प्रविष्टियां अंतःस्थापित की जाएंगी, अर्थात्:-

“10. तरल किण्वित जैविक खाद :

क्र.सं.	प्राचल	विशिष्टताएं
(i)	भार द्वारा आर्द्रता प्रतिशत	90-97
(ii)	भार द्वारा कुल जैविक कार्बन का प्रतिशत, न्यूनतम	14(शुष्क आधार पर)
(iii)	कुल एन, पी ₂ ओ ₂ और के ₂ ओ पोषक तत्व, न्यूनतम	1.2(शुष्क आधार पर)
(iv)	सी:एन	<20
(v)	पीएच	6.5-8.0
(vi)	से अनधिक चालकता (डी एसएम-1 के रूप में)	4
(vii)	भारी धातु तत्व मि.ग्रा./कि.ग्रा.	
	आर्सेनिक (एस ₂ ओ ₃ के रूप में)	10 (शुष्क आधार पर)
	कैडमियम (सीडी के रूप में)	5 (शुष्क आधार पर)
	क्रोमियम (सीआर के रूप में)	50 (शुष्क आधार पर)
	कॉपर (सीयू के रूप में)	300 (शुष्क आधार पर)
	एचजी के रूप में पारा	0.15 (शुष्क आधार पर)
	पीबी के रूप में सीसा	50 (शुष्क आधार पर)
	जैड एन के रूप में जस्ता	1000(शुष्क आधार पर)

[फा.सं. 2-6/2020 उर्व. विधि]

नीरजा अड्डिम, संयुक्त सचिव

टिप्पण: मूल आदेश भारत के राजपत्र में सा.का.नि. सं. 758(अ), तारीख 25 सितंबर, 1985 द्वारा प्रकाशित किया गया था और उसका अंतिम संशोधन का.आ. 884(अ), तारीख 24 फरवरी, 2021 द्वारा किया गया।

MINISTRY OF AGRICULTURE AND FARMERS WELFARE

(Department of Agriculture, Cooperation and Farmers Welfare)

ORDER

New Delhi, the 31st May, 2021

S.O. 2126(E).—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 further to amend the Fertiliser (Inorganic, Organic or Mixed) (Control) Order, 1985, namely:-

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1. (1) This Order may be called the Fertiliser (Inorganic, Organic or Mixed) (Control) Third Amendment Order, 2021.

(2) It shall come into force on the date of its publication in the Official Gazette.

2. In the Fertiliser (Inorganic, Organic or Mixed) (Control) Order, 1985 (hereinafter referred to as the said Order), in Schedule –I, in Part-A, under the heading “SPECIFICATIONS OF FERTILIZERS”,-

(i) under the sub-heading “1(c) **STRAIGHT POTASSIUM FERTILIZERS**”, after serial number 6 and the entries relating thereto, the following serial number and entries shall be inserted namely:-

“7. Potassium Magnesium Sulphate (granular)

(i)	Moisture per cent. by weight, maximum	0.5
(ii)	Magnesium as MgO per cent. by weight, minimum	10.0
(iii)	Potash as K ₂ O per cent. by weight, minimum	30.0
(iv)	Sulphate Sulphur as S per cent. by weight, minimum	17.0
(v)	Total Chlorides per cent. by weight, maximum	2.5
(vi)	Particle Size 90% of the material shall be retained between 5 mm IS sieve and on 2 mm IS sieve”	

(ii) under sub-heading 1(g) relating to “**MICRONUTRIENTS**”, after serial number 23 and entries related thereto, the following serial number and entries shall be inserted, namely:-

“24. Magnesium Hydroxide and Zinc Phosphate

(i)	Magnesium as Mg per cent. by weight minimum	24.0
(ii)	Zinc as Zn per cent. by weight minimum	10.0
(iii)	pH (50 g/L)	8.5+/-1
(iv)	Available Phosphorus as P ₂ O ₅ , per cent. by weight minimum	2.5.”

(ii) under sub- heading “1 (i) 100% Water Soluble Complex Fertilisers”, in serial number 7 relating to Potassium Nitrate (prilled) (soil application), in item (vi) for figure “0.5”, the figure “1.5” shall be substituted;

(iii) under the sub-heading “1(j) Beneficial Element Fertilisers”, after serial number (2) and the entries relating thereto, the following serial number and entries shall be inserted namely,-

“3 Sodium Silicate (liquid)

i.	Silicon as SiO ₂ per cent. by weight minimum	23.8
ii.	Sodium as Na per cent. by weight minimum	6.0
iii.	Specific gravity	1.3.”

(iv) under sub-heading “1 (k) LIQUID FERTILISER”, after serial number 5 and the entries relating thereto, the following serial number and entries shall be inserted namely:-

“6. NK 6:0:18 Fortified with Calcium, Magnesium & Boron (suspension)

(i)	Total nitrogen per cent. by weight, minimum	6.0
(ii)	Nitrate Nitrogen as N per cent by weight minimum	5.8
(iii)	Water Soluble potassium as K ₂ O per cent by	18.0

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	weight minimum	
(iv)	Water soluble Calcium (as CaO), per cent by weight, minimum	5.0
(v)	Water soluble Magnesium (as MgO), per cent by weight, minimum	2.0
(vi)	Boron as B	0.5-0.8
(vii)	pH (1 % Solution) at 200 C	8 to 9

7. NPK 11: 11: 8 Fortified with Zinc & Boron (suspension)

(i)	Total nitrogen per cent. by weight minimum	11.0
(ii)	urea nitrogen, per cent by weight, minimum	7.2
(iii)	Ammonical nitrogen, per cent. by weight maximum	3.0
(iv)	Water soluble phosphorus (as P ₂ O ₅), per cent. By weight minimum	11.0
(iii)	Water Soluble potassium (as K ₂ O) per cent. By weight minimum	8.0
(iv)	Zinc as Zn percent by weight minimum in the form of Zn-EDTA	0.7
(vi)	Boron as B	0.5-0.7
(vii)	pH (1 % Solution) at 200 C	7.0-8.0

8. Calcium Nitrate Fortified with Magnesium (suspension)

(i)	Total nitrogen per cent. by weight minimum	10.0
(ii)	Nitrate Nitrogen per cent by weight, minimum	8.5
(iii)	Water soluble calcium as CaO per cent. by weight minimum	15.0
(iv)	Water Soluble magnesium as MgO per cent,by weight minimum	2.0
(v)	Total chloride as Cl per cent. by weight maximum	2.5
(vi)	pH (1 % Solution) at 200 C	8.0-9.0"

3. In the said order, in schedule II, in part- B, in serial number 26,-

(i) for item (i), the following entry shall be substituted, namely,-

“ (i) Determination of total zinc

Scope: Total extraction of Zinc (Zn), Arsenic (As), Lead (Pb) and Cadmium (Cd) from Zinc Oxide Suspension Concentrate.

Principle: Zinc and heavy metal impurities are extracted from the sample with boiling Aqua Regia.

(a) Sample preparation:

The details of the procedure for drawl of samples of fertilizers have been provided in Schedule II Part A, Serial No. 9 (Method for sampling of liquid fertilizers (other than anhydrous ammonia), however importance of proper shaking and mixing of contents before withdrawal of sample and before drawing aliquot for analysis is particularly emphasized. Since in suspension formulation

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insoluble materials might settle down over time, thorough mixing of contents would ensure representative composition along the entire depth of the container.

(b) Reagents:

All the reagents should be of analytical grade to ensure negligible concentration of the elements to be determined.

- (1) Glass double distilled water (free from micronutrients)
- (2) Hydrochloric acid 37% HCl (ρ = 1.18 g/ml)
- (3) Nitric acid 65% HNO₃ { $c(\text{HNO}_3) = 14.3 \text{ mol/l}$, $\rho = 1.4 \text{ g/ml}$ }

(c) Apparatus:

Apparatus for thermal heating digestion – with reaction vessel and reflux condenser.

The vessel should be at least 5 times the volume of the aqua regia used.

In case reaction vessel with reflux condenser is not available, Erlenmeyer flask or high beakers covered with watch glass can be used for the purpose.

Ash free filter paper is required if filtration is necessary.

(d) Procedure:

- (1) Weight one gram ($1 \pm 0.001 \text{ g}$) of the sample and transfer quantitatively to the reaction vessel
- (2) Moisten the sample with about 0.5 to 1.0 ml distilled water
- (3) Mix the contents well and 21 ml of HCL) followed by 7 ml of HNO₃ (reagent both drop wise to reduce foaming.
- (4) Connect the condenser to the reaction vessel and let the mixture stand at laboratory room temperature until effervescence ceases.
- (5) Turn on the heating device and slowly raise the temperature of the reaction mixture to reflux condition. Maintain for 2 hours.
- (6) Ensure that the condensation zone is lower than half of the height of the condenser.
- (7) After 2 hours of reflux, allow to cool and rinse the condenser with 10 ml of distilled water.
- (8) Transfer the contents quantitatively into a 500 ml volumetric flask and dilute to the mark with water. The test solution corresponds to a 500 times dilution of the sample.
- (9) Test solution can be filtered, if necessary (should not be required for Zinc Oxide Suspension concentrate). If filtered, discard the first 20 ml (approx) portion for analysis
- (10) Prepare a blank test solution following the same procedure as the sample. This is to be used for background correction of analysis, in case of any possible contamination through reagents.
- (11) Measurement can be carried out immediately, or can be stored in tightly closed plastic vessels for up to 15 day.

Note:- Addition of one drop of octanol to the reaction vessel can be used as an antifoaming agent.

(e) Determination of Zinc in the extracted material:

Process for analysis of Zinc shall be as per the procedure specified at Method no. 7 (iii) (b) (2) step (B) onwards or method No. 8 (ii) (b) step (2) onward.

Note :- Calculation need to be adjusted accordingly, in view of extent of dilution of original sample.

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(2) In item (ii) for the words, figures and brackets “specified in 8 (v)” the words, figures and brackets “specified in 8(v) after extraction thorough aqua regia as specified in sub-item (d) of item (i)” shall be inserted;

(3) After entry (iii) the following entries shall be inserted, namely,-

“(iv) For determination of Arsenic

By the method specified as 8 (ix) after extraction thorough aqua regia as specified in sub-item (d) of item (i).

(v) For determination through Cadmium

By the method specified as 8 (x) after extraction thorough aqua regia as specified in sub-item (d) of item (i).”

(4) after serial number 29, and entries relating the following serial numbers and entries shall be inserted, namely,-

“30. Method of analysis of Sodium Silicate

(i) Determination of Silicon

(a) Chemicals and Glasswares :

(1) Conc. HCl

(2) Conc. HNO₃

(3) Hot Plate

(4) Platinum Crucible

(5) Muffle Furnace (Temp. capacity by 10000C)

(6) Desiccators and other routine laboratory glasswares

(b) Procedure :

(1) Take 1 gm of prepared sample in Teflon or corning beaker, add 2-3 ml HCl solution (1:1) and 2-3 ml HNO₃ solution (1:1).

(2) Digest the solution on a hot plate till it becomes semi-solid, cool it and again add 3-4 ml HNO₃ solution. Dry it completely on hot plate.

(3) Add 2-3 ml HCl solution (1:1) and boil it till yellow fumes cease. Filter it with Filter Paper No. 1, wash with 10 ml HCl solution (1:1) one time and 2-3 washing with hot water of 10 ml portion each till yellow colour disappear.

(4) Transfer the filter paper alongwith residue in pre-weighed platinum crucible, dry for one and half hour in muffle furnace at 250°C temperature and finally ignite the residue at 950°C temperature for 30 minutes.

(5) Cool the crucible in desiccator and re-weigh and calculate

Calculation :

$$\% \text{ Si as SiO}_2 = \frac{\text{Final weight of crucible} - \text{empty weight of crucible}}{\text{Weight of sample}} \times 100$$

Weight of sample

(i) Determination of sodium

By the method as specified in serial number 17

(ii) Determination of Specific gravity

By the method as specified in serial number 21

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31. Method of analysis of nano Nitrogen

(1) Determination of Nitrogen – Total Kjeldahl Nitrogen

(a) Procedure:

- (1) Liquid Nano Fertilizer sample : Take aliquot quantity (10 ml) of sample in the Kjeldahl flask.
- (2) In case of solid Nano Fertilizer Sample : Take one gram of powdered nano-fertilizer sample is transferred to the Kjeldahl flask
- (3) Add 15 g K_2SO_4 or 12 g anhydrous Na_2SO_4 , 0.4g anhydrous $CuSO_4$, or 0.6g $CuSO_4.5H_2O$, and approximately 0.8g alundum granules.
- (4) Add 37 ml diluted Sulfuric acid with water $H_2SO_4+H_2O(1+1,v/v)$ or 20 ml concentrated Sulfuric acid, if adequate ventilation is available.
- (5) Add sufficient test portion mass, precisely 0.1000 to 2.800 g for fertilizers with 30 to 5% nitrogen, respectively. Rinse the inner wall with about 10ml water.
- (6) Transfer the flask to a preheated (400°C) Kjeldahl block digester and digest test portions for 75 minutes.
- (7) Remove the flask from the heating block and upon cooling (the reaction mixed must be near room temperature). Wash the inner wall with 20-30 ml water and mix.
- (8) Prepare the distillate receiving flask (300ml Erlenmeyer flask) by adding 30ml of 0.25 N standardized Sulfuric acid to trap the expected total Nitrogen in the test portion.
- (9) Add 2-3 drops of Methyl purple indicator and install the receiver on the outlet tube of the distillation unit, being sure that the distillate outlet tube end is totally immersed in the standardized acid solution.
- (10) Install the digestion tube on the distillation unit. Initiate steam generation and slowly dispense about 80ml (30 -35%) Sodium Hydroxide into the flask.
- (11) Continue steam distillation until about 250 ml or more of steam condensate has been collected in the receiving flask. This usually requires about 6-8 minutes.
- (12) If color changes to green, add more 0.25 N H_2SO_4 to bring the color back to purple and record the amount of acid added.
- (13) Titrate to a grey end point (pH5.7) with 0.25 N Standard NaOH. The color of the distillate depends upon the amount of total nitrogen in the test portion, which is a function of the amount of ammonia trapped in the receiver flask.
- (14) A green color indicates that the acid in the trap was neutralized by the Ammonia. At this point, add an additional known amount of standardized H_2SO_4 to get to the grey end point.
- (15) The net volume (in ml) of standardized acid would be equal to the total amount of acid initially added to the receiving flask plus the amount of the acid added, after distillation, to reach the grey end point. A blue or Purple colour indicates that there is still acid in the receiving flask, and back titration with NaOH is required.
- (16) The net volume standardized acid would be equal to the amount of acid in the receiving flask minus the amount of base added, after distillation, to reach to the grey end point.

(b) Calculations:

Weight percent total nitrogen is calculated as follows:

$$\text{Total N \%} = \frac{(\text{net mL std acid} \times \text{N of std acid}) - (\text{net mL std base} \times \text{N of std base}) \times 1.4008}{\text{Sample weight, g}}$$

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(ii) Physical Particle Size (as per Transmission Electron Microscope (TEM) Analysis)**(a) Equipment and Apparatus:**

- (1) Transmission Electron Microscope
- (2) Sample grid
- (3) Tweezers, Petri dish, Ethanol and Deionized water

(b) Procedure:

- (1) TEM sample grids carbon coated film that is electron transparent supported by copper mesh or equivalent are suitable.
- (2) Glassware and apparatus used for sample preparation should be cleaned with filtered, demineralized water and stored dry.
- (3) The apparatus used for dispersion and deposition of particles consists of a small glass vial with a screw-on cap, a teflon pillar about 10 mm high that may be inserted into the vial, a petri dish, and a teflon block about 40 mm by 40 mm square.
- (4) Hold the grid with tweezers, dip rinse the grid thoroughly with ethanol. Wick the excess liquid off the grid using filter paper. Place the dried grid onto the clean Teflon block.
- (5) Place a 10 μ L drop of the nanoparticle solution onto the grid.
- (6) Cover the grid with a petri dish lid and let stand at room temperature for getting the grid dry, typically from 5 to 30 minutes, depending on solvent type.
- (7) Transfer the grid on TEM sample holder. Record enough micrographs to image a minimum of 200 nanoparticles per sample per grid square from a minimum of 2 widely separated regions of the grid using a well-aligned and stable TEM, operated at a fixed magnification that allows a large number of nanoparticles to be visible within the micrograph field of view, while ensuring that each individual nanoparticle is recorded with a large number of image pixels.

(c) Precautions:

- (1) Transmission Electron Microscope grids (especially thin film membranes) are very fragile and must be held by their edges with fine tweezers so as not to damage or crack the membrane.
- (2) Exposure of Transmission Electron Microscope grids to the ambient environment should be minimized to reduce the likelihood of dust contamination. Grids should be stored in suitable boxes in dust-free or desiccating cabinets.
- (3) A well-aligned Transmission Electron Microscope is essential to obtain accurate particle size results.
- (4) A minimum of 200 discrete particles should be measured from each of at least two widely separated regions of the sample (that is, different grid squares or membrane regions). Foreign debris in a given image (e.g., dust particles or residues from the rinsing and drying process) should be avoided.
- (5) Particle size results obtained from Transmission Electron Microscope measurements may not coincide with those obtained from other techniques (e.g., dynamic light scattering). This is due in part to differences in the weighted averages determined in each case (e.g., number for Transmission Electron Microscope versus intensity for dynamic light scattering), as well as differences in the physical property that is actually measured (e.g., projected area versus hydrodynamic diffusion area).

(iii) Hydrodynamic particle size (as per Dynamic Light Scattering (DLS) Analysis)**(a) Equipment and Apparatus:**

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(1) Dynamic Light Scattering Equipment

(2) Sample Cuvettes of size

(3) Deionized water

(b) Procedure:

- (1) For liquid nano-fertilizer sample, 10 ml is sonicated for one minute and used for the analysis. In case for solid nano-fertilizer, fertilizer : distilled water (1:10 ratio) suspension is prepared, sonicated for one minute
- (2) Known standard samples (either one of the nano particles such as Ag, Au or TiO₂) are used to set the machine
- (3) Load sample into the Cuvette
- (4) Pre-rinse filter with solvent (at least 1 ml, depending on filter size and dead volume of filter holder or cartridge).
- (5) After loading syringe with sample and inserting syringe filter, allow the first 4 drops to go to waste. Use the next 4 drops to pre-rinse the cuvette, and discard. The remainder can be used for the sample measurement.
- (6) Load sample into cuvette using minimum amount necessary to ensure liquid level is at least 2 mm above the entrance height of the laser beam for your particular instrument configuration.
- (7) Take care not to touch the cuvette windows with your bare hands while loading. Wipe outside of quartz or glass cuvette with lens paper if needed. Cap the cuvette to prevent dust contamination and solvent evaporation.
- (8) Inspect the cuvette to ensure that air bubbles are not clinging to the optical window area.
- (9) Load the sample into Dynamic Light Scattering machine.
- (10) Perform 3 to 10 independent measurements per sample.

(c) Precautions:

- (1) To measure the size of solid phase nano nitrogen particles, 1 gram of nanoparticles sample should be suspended in 20 ml water followed by 1 minute ultra-sonication before the size measurement carried out by the Dynamic Light Scattering.
- (2) Measurement cuvettes should be cleaned with filtered demineralized water and stored dry.
- (3) The choice of pore size depends on the maximum dimension of the test particles and their tendency to adhere to the filter membrane. μ Suspended medium (such as solvent, dispersant, solution) should be filtered prior to sample preparation using a 0.1 or 0.2 μ .
- (4) A typical starting sample concentration is 1 mg/ml.
- (5) Use cuvette with quartz or equivalent optical-quality windows.
- (6) Pre-rinse cuvette with filtered solvent at least 3 times.

(iv) Zeta Potential Analysis

(a) Equipment and Apparatus:

- (1) Dynamic Light Scattering (DLS).
- (2) Sample Cuvettes of zeta.
- (3) Deionized water.

(b) Procedure:

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- (1) For liquid nano-fertilizer sample, 10 ml is sonicated for one minute and used for the analysis and in case for solid nano-fertilizer, fertilizer : distilled water (1:10 ratio) suspension is prepared, sonicated for one minute.
- (2) Known standard samples (either one of the nano particles such as Ag, Au or TiO₂) are used to set the machine.
- (3) Loading Sample into the Cuvette.
- (4) Pre-rinse filter with solvent (at least 1 ml, depending on filter size and dead volume of filter holder or cartridge).
- (5) After loading syringe with sample and inserting syringe filter, allow the first 4 drops to go waste. Use the next 4 drops to pre-rinse the cuvette, and discard. The remainder can be used for the sample measurement.
- (6) Load sample into cuvette using minimum amount necessary to ensure liquid level is at least 2 mm above the entrance height of the laser beam for your particular instrument configuration.
- (7) Take care not to touch the cuvette windows with your bare hands while loading. Wipe outside of quartz or glass cuvette with lens paper if needed.
- (8) Cap the cuvette to prevent dust contamination and solvent evaporation.
- (9) Inspect the cuvette to ensure that air bubbles are not clinging to the optical window area.
- (10) Load the sample into Dynamic Light Scattering machine.
- (11) Perform 3 to 10 independent measurements per sample.

(c) Precautions:

- (1) Measurement cuvettes should be cleaned with filtered demineralized water and stored dry. The choice of pore size depends on the maximum dimension of the test particles and their tendency to adhere to the filter membrane. μ suspending medium (such as solvent, dispersant, solution) should be filtered prior to sample preparation using a 0.1 or 0.2
- (2) A typical starting sample concentration is 1 mg/ml.
- (3) Use cuvette with quartz or equivalent optical-quality windows.
- (4) Pre-rinse cuvette with filtered solvent at least 3 times.

(v) Viscosity Measurement:**(a) Equipment and Apparatus:**

- (1) Demineralised water.
- (2) Viscometer.
- (3) Measuring cylinder.
- (4) Bottle Adapter.

(b) Procedure:

- (1) Calibrate the viscometer with distilled water to set the machine as 1 cps.
- (2) Viscometer Assembly.
- (3) Attach vertical pole to the base using the wrench provided in the case.
- (4) Attach the viscometer to the vertical pole.
- (5) Connect power cable to the viscometer.
- (6) Turn on the viscometer and calibrate.

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- (7) Starting up – Select and attach required spindle.
- (8) Raise the viscometer to the highest level using a screw on the vertical pole.
- (9) Place beaker with liquid under the spindle.
- (10) Lower the viscometer until the spindle is submerged to the spindle's mark.
- (11) Viscometer Operation:- This depend on the software of the instrument make. The analyst should follow the manufacturer's operating instructions for a particular instrument. Set the viscometer using distilled water to measure 1cps at room temperature.

(c) Precautions:

- (1) Wait for 30-60 sec before reading.
- (2) Good results are in range 60%-80% of torque.
- (3) Results depend on beaker and volume of liquid so use the same beakers for comparison measurements. Viscosity of a Liquid nano-fertilizer N is measured directly.
- (4) For solid nano-fertilizer, fertilizer: water suspension should be in 1:10 ratio with distilled water.

(vi) Measurement of pH

(a) Apparatus:

pH meter, vacuum pump, beaker, pipette, glass rod, china dish, spatula etc.

(b) Reagents:

Buffer solutions of pH 4.0, 7.0 and 9.2: One buffer tablet of the respective pH is dissolved in water and the volume is made to 100 ml.

(c) Procedure:

- (1) Take 10 ml of liquid sample, homogenise it and take the pH measurement. For solid / powder samples (1 g dry sample / 10 ml water) homogenisation or ultrasonic agitation of the sample for 1 minute followed by pH measurement should be taken after settling of the samples
- (2) pH meter is set at room temperature and calibrated by immersing the electrodes in different buffer solutions of pH 4.0, 7.0 and 9.2.
- (3) Take the beaker of homogenised samples and dip the electrodes into it and note the pH reading.
- (4) After each determination the electrodes must be washed with distilled water and wiped out by ordinary filter paper.

(d) Precautions:

- (1) Proper homogenisation / sonication must be done.
- (2) The glass and reference electrode of pH meter should always remain dipped in water.
- (3) Buffer solutions should be prepared accurately and stored well in glass container.
- (4) It is desirable to prepare fresh buffer solutions after few days. Connect the pH meter to the stabilizer to avoid the fluctuations in pH readings. Adjust the temperature knob of pH meter at room temperature for correct pH determination".

4. In Schedule IV, Part-A, after serial number 9 and the entries relating thereto, the following serial numbers and entries shall be inserted namely,-

"10. Liquid Fermented Organic manure

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S.No.	Parameters	Specifications
(i)	Moisture, per cent. by weight	90-97
(ii)	Total organic Carbon per cent. by weight minimum	14 (on dry basis)
(iii)	Total N, P ₂ O ₅ and K ₂ O nutrient minimum	1.2 (on dry basis)
(iv)	C:N	<20
(v)	pH	6.5-8.0
(vi)	Conductivity (as dsm ⁻¹) not more than	4
(vii)	Heavy Metal content mg/kg	
	Arsenic As (As ₂ O ₃)	10 (on dry basis)
	Cadmium (as Cd)	5 (on dry basis)
	Chromium (as Cr)	50(on dry basis)
	Copper (as Cu)	300(on dry basis)
	Mercury as Hg	0.15(on dry basis)
	Lead as Pb	50 (on dry basis)
	Zinc as Zn	1000 (on dry basis)

[F.No. 2-6/2020 Fert.Law]

NEERAJA ADDIDAM, Jt. Secy.

Note: The principal Order was published in the Gazette of India vide number G.S.R. 758(E) dated the 25th September, 1985 and last amended vide number S.O 884(E) dated 24th February, 2021.