MEGHMANI INDUSTRIES LTD.

CIN U29199GJ1993PLC019013

ATRAZINE, TECHNICAL – SPECIFICATION

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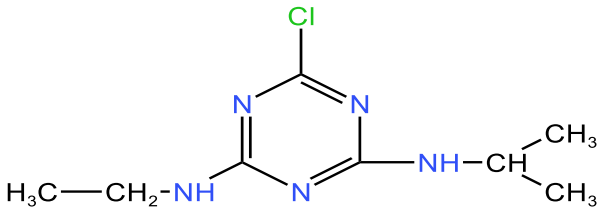
Unit: VII	Plot No. 20/G/2/A, Gallops Industrial Park, Vasna – Chacharwadi, Sarkhej – Bavla Road, Dist. Ahmedabad - 382220, Gujarat, India.

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standard on 23 March 1990, after the draft finalized by the Pesticide Sectional Committee had been approved by the Food and Agriculture Council.

Atrazine, technical is employed in the preparation if herbicidal formulations.

Atrazine is the accepted name by the International Organization for Standardization (ISO) for 2-Chloro-4-ethylamino-6-isopropyl amino-1,3,5-triazine. The Empirical, formula, the structural formula, and the molecular mass of atrazine are given below:

Empirical Formula.	Structural Formula	Molecular Mass
$C_8H_{14}ClN_5$		215.7

STRUCTURAL FORMULA

In the preparation of this standard, due consideration has been given to the provisions of the *Insecticide Act, 1968* and the Rules framed thereunder. However, this standard is subject to the restrictions imposed under these, wherever applicable.

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.

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1 SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for atrazine, technical.

2 REFERENCES

2.1 The Indian Standards listed in Annex A are necessary adjuncts to this standard.

3 REQUIREMENTS

3.1 Description

The material shall be in form of a white powder, free from extraneous impurities or added modifying agents.

3.2 The material shall comply with the requirements specified in Table 1.

4 PACKING

4.1 Material shall be packed in mild steel containers or double hessian jute bags (*see* IS 8115 : 1976) or DW tarpaulin laminated jute bags (*see* 8117: 1976) or HDPE woven Sacks [*see* IS 8069 (Part 1) : 1976]. The containers shall also comply with general requirements as specified in IS 8190 (Part 1): 1988.

5 MARKING

5.1 The Container shall bear legibly and indelibly the following information in addition to any other Information as required under the *Insecticide Act*, 1968 and the Rules framed thereunder:

a) Name of the materials;
b) Indication of the sources of manufacture;
c) Date of manufacture;
d) Date if expiry;
e) Batch number;
f) Net mass of contents;
g) Atrazine content, percent (*m/m)*; and
h) The minimum cautionary notice as required under the *Insecticides Act*, 1968 and Rules.

6 SAMPLING

6.1 Representative sample of the material shall be drawn as prescribed in IS 10946 : 1984.

7 TESTS

7.1 Tests shall be carried out by the methods referred to in col **4** and 5 of Table 1.

7.2 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (see IS 1070 : 1977) shall be employed in tests.

Table 1 Requirements for Atrazine, Technical

(*Clause 3.2*)

SI No.	Characteristics	Requirement	Method of Tests, Ref to	
			Annex of this Standard	Cl No. of IS 6940: 1982
(1)	(2)	(3)	(4)	(5)
1	Atrazine content, percent by mass, *Min*	97.0	B	-
2	Ionic Chloride content (as NaCl), percent by mass, *Max*	2.0	C	-
3	Moisture content, percent by mass, *Max*	1.0	-	4.1
4	Melting Point, °C	175-177	-	6
5	Particle Size, (Pass through 200 mesh), % w/w min.	98%	D	12

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LIST OF REFERRED INDIAN STANDARDS

IS No.	*Title*	*IS No.*	*Title*
1070 : 1977	Water for general laboratory use (*Second revision*)	8115 : 1976	Double hessian jute bags for pesticides
6940 : 1982	Methods of test for pesticides and their formulations (*First revision*)	8117 : 1976	DW tarpaulin/laminated jute bags for pesticides
8069 (Part 1) : 1981	High density polyethylene (HDPE) for packing pesticides: Part 1 woven sacks (*first revision*)	8190 (Part 1) : 1988	Requirements for packing of pesticides: Part 1 Solid pesticides (*Second revision*)
		10946 : 1984	Methods of sampling for technical grade pesticides

ANNEX B
[Table 1, Item (i)]
DETERMINATION OF ATRAZINE CONTENT

B-1 General

B-1.1 Atrazine content may be determined by either of two methods, namely, alkaline hydrolysis method (*see* **B-2)** or high performance liquid chromatography (HPLC) method (*see* **B-3**). In case of dispute, however; HPLC method shall be the referee method.

B-2 ALKALINE HYDROLYSIS METHOD

B-2.1 Principle

In this method the chlorine atom of atrazine is converted to ionic chloride. The total ionic chloride thus formed is estimated titrimetrically by Volhard's method by black titrating the excess silver nitrate added with standard potassium thiocyanate solution. Ionic chloride present initially is estimated and appropriate correction is made. The determined ionic chloride content after multiplying by appropriate conversion factor gives atrazine content.

B-2.2 Reagents

B-2.2.1 *Ethanol*

B-2.2.2 Alcoholic Potassium Hydroxide Solution-1 N, prepared by dissolving 56 g of pure potassium hydroxide in one liter of anhydrous ethanol.

B-2.2.3 *Nitric Acid* – 1 : 1

B-2.2.4 *Silver Nitrate Solution* – 0.1 N.

B-2.2.5 *Standard Potassium Thiocyanate Solution*- 0.1 N

B-2.2.6 *Phenolphthalein Indicator Solution* – one percent in 96 percent ethanol.

B-2.2.7 *ferric Alum Indicator Solution*

B-2.3 Procedure

B-2.3.1 Weigh accurately about 0.5 g of the sample into a 250-ml Erlenmeyer flask with ground glass joint. Add 50 ml of alcoholic potassium hydroxide solution and reflux gently for 30 minutes. Cool, rinse the condenser with 50 ml of distilled water. Neutralize with nitric acid using phenolphthalein, add 10 ml in excess. Add a known excess of silver nitrate solution accurately with pipette and titrate the excess against standard potassium thiocyanate solution using ferric alum indicator.

B-2.3.2 Carry out blank determination with exactly same weight of the material and titrating against standard potassium thiocyanate solution to make correction for ionic chloride.

B-1.4 Calculation

Atrazine Content,

$$\text{percent by mass} = \frac{21.57 \times (V_1 - V_2) \times N}{M}$$

where,
V_1 = Volume in ml of standard potassium thiocyanate solution consumed for the sample taken for analysis;
V_2 = Volume in ml of standard potassium thiocyanate solution consumed for blank determination;
M = Mass in g of sample taken for analysis; and
N = Normality of standard potassium thiocyanate solution

B-3 HIGHPERFORMANCE LIQUID CHROMATOGRAPHY METHOD

B-3.1 Principle

A high performance liquid chromatograph unit with an ultra-violet (UV) detector is used for the determination. Using solution containing known amounts of the standard atrazine and internal standard, the response factor (Rf) for atrazine in the internal standard is arrived at. A solution containing known mass of the atrazine sample and internal standard is injected subsequently. The percentage of atrazine in the sample is then computed by the standard relationship.

B-3.2 Apparatus

B-3.2.1 *High Performance Liquide Chromatograph (HPLC)*

Equipped with a printer-plotter-cum-integrator and ultra-violet (UV) detector. The suggestive HPLC operating conditions are likely to change with change in the HPLC equipment employed, provide standardization is done.

Column	C8/C18, Stainless steel, 25 cm length and 4.6 mm ID
Detector	UV (at 254 nm)
Solvent System	Methyl alcohol
Solvent flow rate	1 ml/min
Pressure	34.5 kPa
Chart speed	0.5 cm/min
Sample size	20 µl

B-3.3 Reagents

B-3.3.1 *Internal Standard* – Diphenyl oxide, AR grade.

B-3.3.2 *Methyl Alcohol* – Spectroscopic grade.

B-3.3.3 *Standard Atrazine* – of known purity.

B-3.4 Preparation of Solution and Calibration

B-3.4.1 *Preparation of Internal Standard* (Is) *Solution*

Weigh accurately 1.0 g of diphenyl oxide (Is) into a 100 ml volumetric flask and dilute up to the volume mark using methyl alcohol. This solution will contain 10 mg/ml of diphenyl oxide.

B-3.4.2 *Preparation of Standard Atrazine Solution*

Weigh accurately 0.1g of standard atrazine of known purity into a 100-ml volumetric flask. Add 10-ml *Is* (**B-3.4.1**) solution and dilute upto the mark with methyl alcohol. This solution will contain 1000 µg/g of atrazine and diphenyl oxide.

B-3.4.3 *Calibration*

Introduce 20 µl of the solution (**B-3.4.2**) to the HPL unit and from the chromatogram, calculate response factor as follow:

$$\text{Response factor } (Rf) = \frac{A_1}{A_2} \times \frac{m_1}{m_2}$$

Where

A_1 = peak area of the atrazine;
m_1 = concentration in µg of diphenyl oxide;
A_2 = peak area of the diphenyl oxide; and
m_2 = concentration in µg of atrazine;

B-3.5 Estimation

B-3.5.1 Weigh 0.5 g of atrazine sample into a 100-ml volumetric flask and make up the volume with methyl alcohol. Add with a pipette 10- ml of the solution into a 50-ml volumetric flask. Then add with pipette 5-ml of '*Is*' solution I the same flask. Mix well. Make up The Volume with methanol.

B-3.5.2 Introduce 20 µl of the solution to the HPLC unit and, from chromatogram, calculate atrazine content in the sample.

B-3.6 Calculation

Atrazine Content,

$$\text{percent by mass} = \frac{A_3 \times m_3 \times N \times 100}{A_4 \times m_4 \times Rf}$$

where

A_3 = peak area of the atrazine sample taken for the test;

m_3 = mass in g of '*Is*' (**B-3.4.1**) added;

P = percent purity of the standard atrazine;

A_4 = peak area of the '*Is*';

m_4 = mass in g of the sample taken for the test; and

Rf = response factor (**B-3.4.3**)

ANNEX C
DETERMINATION OF IONIC CHLORIDE CONTENT

C-1 Reagents

C-1.1 Silver Nitrate Solution – 0.1 N

C-1.2 Ferric Alum – Indicator.

C-1.3 Standard Potassium Thiocyanate Solution – 0.1 N.

C-1.4 Nitric Acid Solution – 10 percent (*v/v*).

C-2 Procedure

C-2.1 Weigh accurately about 1.0 g of the sample in a 250-ml Erlenmeyer flask add 50 ml water and 10 ml dilute nitric acid. Then add 25 ml silver nitrate with a pipette and add 2 ml of the ferric alum indicator solution. Titrate against standard potassium thiocyanate solution till faint red colour appears. Carry out a blank determination.

C-3 Calculation

Ionic chloride (as NaCl) ,

$$\text{percent by mass} = \frac{(V_1 - V_2) \times 5.846 \times N}{M}$$

where

V_2 = Volume in ml of standard potassium thiocyanate solution used in the titration for sample;

V_1 = Volume in ml of standard potassium thiocyanate solution used for blank determination;

N = Normality of standard potassium thiocyanate solution used; and

M = Mass in g of the sample taken for test.

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ANNEX D

DETERMINATION OF PARTICLE SIZE

D-1 Reagents

D-1.1 Distilled Water / DM water

D-2 Procedure

1) Switch on the instrument and allow it to stabilize for 30 minutes. 2) Start the Mastersizer software on the Computer. 3) Wash the Sample dispersing unit with distilled water /de-ionized water 3 to 4 time; The laser intensity value should be between 70 to 90 %. Apply Ultra sonication if Required for cleaning. 4) in the dry condition the laser obscuration value should be 0%. 5) Fill the sample dispersing unit with distilled water, and set conditions as mentioned Below for sample measurement, carry out blank measurement first.

Dispersant name : No Dispersant in dispersing unit
Refractive index of Dispersant: 1.33
Refractive index of Product: 1.60
Analysis Model: General Purpose
Particle Shape: Irregular
Sensitivity: Normal
Stirring Speed /Pump Speed: 3000 RPM
Obscuration (%): 10 -15
Result Unit: Volume
Sonication time: 1 min. with 100 % Power
Weighted avg Residue: < or = 2 %
Absorption: 0.01

6) Take Approx. About 2 grams of sample in clean 50 or 100 ml Beaker and wet the sample by slowly Adding 18 ml Water while stirring with spatula, sonicate for 2 minutes. Add this wetted sample very Slowly in the sample Dispersing unit of the instrument using a Dropper. After the required obscuration value is obtained, sonicate the dispersion for 1 minute. 7) Select the Appropriate file from the software. Set All essential parameters manually as per the manufacturer 's standard operating procedure Requirements. (see conditions mentioned above). 8) Report the median diameter d (0.5) Values as the median Particle Size of Sample. The Software should be set to report an Average Value of 3 readings. Other parameter as Specified should be taken from Particle Size distribution.