QUALITY TOXICOLOGICAL SERVICES

OECD GLP CERTIFIED

REPORT

STUDY TITLE

Determination of Accelerated Storage Stability and

Corrosion Characteristics of

Chinchex Bed Bugs Insecticide

(Guidelines: CIPAC MT 46.3, OPPTS 830.6320)

Date : 04 November 2022

SPONSOR

Chinchex Limited,

12F, Wing Fat Loong Building,

136, Wai Yip Street, Kwun Tong,

Hong Kong, China

TEST FACILITY

INTOX PVT. LTD.

375, Urawade, Tal. Mulshi, Dist. Pune, Maharashtra, 412 115, INDIA.

Tel. : +91-20-66548700 Fax : +91-20-66548799 Email : info@intoxlab.com

www.intoxlab.com

Report No. : R/PCP4144/STB-AS/22 Number of Pages : 59

CONTENTS

STATEMENT OF GLP COMPLIANCE

The Study No. PCP4144, entitled "Determination of Accelerated Storage Stability and Corrosion Characteristics of **Chinchex Bed Bugs Insecticide**" (Guidelines: CIPAC MT 46.3, OPPTS 830.6320) was performed in compliance with the OECD Principles of Good Laboratory Practice (OECD, 1998).

No unforeseen circumstances were observed which might have affected the quality or integrity of the study.

This report represents a true and accurate record of the results obtained. I accept the responsibility for validity of the data, as well as the interpretation, analysis, documentation and reporting of the results.

The report contains 60 pages including contents, tables and certificates.

Devidas Waghade
Study Director

04-11-2022
Date

STATEMENT BY THE TEST FACILITY MANAGEMENT

The Study No. PCP4144, entitled "Determination of Accelerated Storage Stability and Corrosion Characteristics of **Chinchex Bed Bugs Insecticide**" (Guidelines: CIPAC MT 46.3, OPPTS 830.6320) was performed at INTOX PVT. LTD., Pune.

Management of the Test Facility had made available all the resources necessary for conduct of the study in compliance with the OECD Principles of GLP.

The Test Facility Management also hereby approves this report for issue :

Dr. Mukul Pore

04-11-2022

Date

QUALITY ASSURANCE STATEMENT

The Study No. PCP4144, entitled "Determination of Accelerated Storage Stability and Corrosion Characteristics of **Chinchex Bed Bugs Insecticide**" (Guidelines: CIPAC MT 46.3, OPPTS 830.6320) was subjected to periodic inspections by the Quality Assurance Unit in compliance with the OECD Principles of Good Laboratory Practice (OECD, 1998).

Dates of conduct of these inspections, the critical phases inspected and the dates on which findings of these inspections were reported to the Study Director and Test Facility Management have been presented below. The inspection did not lead to findings, which would have impaired this study in any way.

This report has been audited by the Quality Assurance Unit, INTOX PVT. LTD. It is an accurate account of the raw data generated and of the procedures followed.

Date of Inspection	Type of Inspection	Phase (s) of the Study Inspected	Reporting Dates to the Study Director	Reporting Dates to the Test Facility Management
12-09-2022	Study Plan Audit	Draft Study Plan	12-09-2022	12-09-2022
12-09-2022	Study Plan Audit	Final Study Plan	12-09-2022	12-09-2022
13-09-2022	Study Audit	Exposure to Storage Stability, Determination of Physical Appearance and pH (Before Storage)	13-09-2022	13-09-2022
14-09-2022	Study Audit	Determination of Acidity (Before Storage)	14-09-2022	14-09-2022
15-09-2022	Study Audit	Determination of Bulk Density (Pour Density and Dry Sieve Test) (Before Storage)	15-09-2022	15-09-2022
16-09-2022	Study Audit	Determination of Dry Sieve Test (Before Storage)	16-09-2022	16-09-2022
27-09-2022	Study Audit	Determination of Accelerated Storage Stability, Physical appearance and pH (After Storage)	27-09-2022	27-09-2022
28-09-2022	Study Audit	Determination of Acidity (After Storage)	28-09-2022	28-09-2022
29-09-2022	Study Audit	Determination of Bulk Density (Pour Density and Dry Sieve Test) (After Storage)	29-09-2022	29-09-2022
30-09-2022	Study Audit	Determination of Dry Sieve Test (After Storage)	30-09-2022	30-09-2022
03-10-2022	Study Audit	Method Validation and Quantification of Silicon (Before and After Storage)	03-10-2022	03-10-2022
29-10-2022	Report Audit	Draft Report	31-10-2022	29-10-2022
04-11-2022	Report Audit	Final Report	04-11-2022	04-11-2022

Sayali Nilakhe M.Sc.

Quality Assurance Unit : SNilakhe

Date : 04-11-2022

STUDY INFORMATION

Study No.	:	**PCP4144**
Report No.	:	**R/PCP4144/STB-AS/22**
Study Title	:	Determination of Accelerated Storage Stability and Corrosion Characteristics of **Chinchex Bed Bugs Insecticide** (Guidelines: CIPAC MT 46.3, OPPTS 830.6320)
Sponsor	:	**Chinchex Limited,** 12F, Wing Fat Loong Building, 136, Wai Yip Street, Kwun Tong, Hong Kong, China
Test Facility	:	**INTOX PVT. LTD.** 375, Urawade, Tal. Mulshi, Dist. Pune - 412 115, Maharashtra, India.

STUDY SCHEDULE

Study Initiation Date	:	12 September 2022
Experimental Starting Date	:	13 September 2022
Exposure to Accelerated storage stability test	:	13 September 2022 to 27 September 2022
Physical Appearance and pH (Before Storage)		13 September 2022
Acidity (Before Storage)	:	14 September 2022
Bulk Density (Before Storage)	:	15 September 2022
Dry Sieve Test (Before Storage)	:	16 September 2022
Physical Appearance and pH (After Storage)	:	27 September 2022
Acidity (After Storage)	:	28 September 2022
Bulk Density (After Storage)	:	29 September 2022
Dry Sieve Test (After Storage)	:	30 September 2022
Validation of Analytical Method and Quantification of Active Ingredient (Before and After Storage)	:	03 October 2022
Experimental Completion Date	:	03 October 2022

Study Director : **Devidas Waghade**

PRINCIPAL PERSONNEL PARTICIPATED IN THE STUDY

Name and Educational Qualification	Responsibility and Function
Devidas Waghade M.Sc.	**Study Director** Overall responsible for conduct of the study.
Vinod Mane Ph.D.	**Study Personnel** Assistance in conduct of the study.
Harshal Suryawanshi M.Sc.	**Study Personnel** Assistance in conduct of the study.
Mayur Pawar M.Sc.	**Study Personnel** Assistance in conduct of the study.
Umesh Gadade M.Sc.	**Study Personnel** Assistance in conduct of the study.

Address of all those listed above:
INTOX PVT. LTD.,
375, Urawade,
Tal. Mulshi,
Dist. Pune - 412 115,
Maharashtra, India
E Mail : info@intoxlab.com

LIST OF ABBREVIATIONS AND SYMBOLS

SR. NO.	ABBREVIATIONS / SYMBOLS	LONG FORM
1	°C	Degree Celsius
2	CAS	Chemical Abstracts Service
3	COA	Certificate of Analysis
4	Conc.	Concentration
5	Contd.	Continued
6	g	Gram
7	GLP	Good Laboratory Practice
8	LOD	Limit of Detection
9	LOQ	Limit of Quantification
10	mg	Milligram
11	min.	Minute
12	mL	Milliliter
13	µg	Micro gram
14	OECD	Organization for Economic Co-Operation and Development
15	OPPTS	Office of Prevention, Pesticides and Toxic Substances
16	Rep. No./ R	Replicate Number
17	%	Percent
18	%RSD	Percent Relative Standard Deviation
19	S	Sample
20	SD	Standard Deviation
21	TI	Test Item
22	v/v	Volume by Volume
23	w/w	Weight by Weight
24	w/v	Weight by Volume

1. SUMMARY

This study entitled "Determination of Accelerated Storage Stability and Corrosion Characteristics of **Chinchex Bed Bugs Insecticide**" was performed in compliance with the Study Plan No. **P/PCP4144/STB-AS/22** which incorporated the recommendations made in Guidelines: CIPAC MT 46.3, OPPTS 830.6320.

The study was conducted in two steps, Validation of Flame Atomic Absorption method for **Silicon** and detemination of Accelerated Storage Stability and Corrosion Characteristic of **Chinchex Bed Bugs insecticide**.

1.1 VALIDATION OF ANALYTICAL METHOD

The method of analysis was validated at Test Facility in this study for quantification of **Silicon** by determining System suitability, Linearity, Accuracy, Precision, Limit of Detection (LOD) and Limit of Quantification (LOQ).

Parameter		Acceptance Criterion	Active Ingredient
			Silicon
Method		-	Flame Atomic Absorption
Linearity		> 0.99	1.0000
System Suitability		%RSD ≤ 5%	< 5%
Accuracy	Level - 1	80 - 120%	98.00
	Level - 2		97.36
Precision		%RSD ≤ 20%	4.25
LOD (mg/L)		-	2.0040
LOQ (mg/L)		-	4.0080

The method of analysis was said to be validated as the validation parameters were found within the acceptance criteria. Method of analysis was found to be linear, accurate and precise for quantification of **Silicon.**

Based on the results of analytical method validations obtained in the study, it is concluded that the proposed methods of analysis is suitable for the analysis of **Silicon** in **Chinchex Bed Bugs Insecticide**.

1.2 DETERMINATION OF ACCELERATED STORAGE STABILITY AND CORROSION CHARACTERISTICS

The test item was subjected to accelerated storage at 54 ± 2 °C for 14 days in PE bottles. The test item was subjected to tests like active ingredient content, pH, acidity, Bulk Density, Dry Sieve and physical appearance before storage and at the end of accelerated storage period. The test item placed in contact with PE bottles (packaging materials) were observed for the compatibility of test item with it. The packaging materials were examined visually for any corrosion characteristics such as "Perforations", "Darkening", "Leaking or rust at the seam", "change in gross weights" due to storage.

The results of tests performed on the test item before storage and at the end of accelerated storage test were found to be as follows:

SUMMARIZED RESULTS OF ACCELERATED STORAGE STABILITY TEST

Sr. No.	Parameters		Results	
			Before storage	At the end of accelerated storage period
1	Active Ingredient Content (%)	Silicon Dioxide (SiO_2)	94.24	94.32
		Silicon (Si)	44.05	44.08
2	Appearance	Physical State	Powder	Powder
		Colour (Visual)	White	White
3	pH of 1% w/v aqueous solution (at 25.0 °C ± 1.0 °C)		6.70	6.65
4	Acidity (Calculated as H_2SO_4) (% m/m)		0.0054	0.0079
5	Dry Sieve Test		$R_x \geq 90\% = 93.22$	$R_x \geq 90\% = 91.40$
			$R_x \geq 10\% = 15.23$	$R_x \geq 10\% = 12.71$
6	Corrosion Characteristics		No "Perforations", "Darkening" and "Leaking or Rust at the seam" were observed on PE bottles due to contact with test item at accelerated storage temperature (54 ± 2 °C) after 14 days. No significant changes in gross weights of the PE bottles containing test item were found due to storage at accelerated temperature after 14 days.	

The various parameters tested in the study at the end of accelerated storage period (at 54 ± 2°C for 14 days) did not significantly differ from the results obtained before storage and were found within specification limits. The properties of formulation were not adversely affected by the storage at high temperature.

From the results obtained from the study, it is concluded that **Chinchex Bed Bugs Insecticide** was found to be **stable** at 54 ± 2 °C for 14 days storage.

2. INTRODUCTION

2.1 OBJECTIVE

The objective of this study was to determine the Accelerated Storage Stability and Corrosion Characteristics of **Chinchex Bed Bugs Insecticide** for 14 days at 54 ± 2 °C.

To ensure that the properties of formulations are not adversely affected by storage at high temperature, and to assess their long-term storage stability at more moderate temperature, with respect to content of active ingredient (and a possible consequent increase in relevant impurities formed by degradation of material) and certain physical properties.

2.2 REGULATORY REFERENCES

2.2.1 TEST GUIDELINE

The study was performed in compliance with the Study Plan No. **P/PCP4144/STB-AS/22** which incorporates the recommendations made in the test guidelines as listed below:

- CIPAC, Method MT 46.3 "Accelerated Storage Procedure". Volume J
- Product Properties Test Guidelines OPPTS 830.6320, "Corrosion characteristics", United States Environmental Protection Agency, Prevention, Pesticides and Toxic Substances (7101) EPA 712–C–96–028 August 1996.

2.2.2 GOOD LABORATORY PRACTICE

The study was conducted in compliance with the OECD Principles of Good Laboratory Practice as set forth in the OECD, 1998; Series on Principles of Good Laboratory Practice and Compliance monitoring Number 1, 'OECD Principles of Good Laboratory Practice' ENV/MC/CHEM(98)17 (as revised 1997).

2.3 STANDARD OPERATING PROCEDURES

All the procedures described in this study were performed in compliance with the 'Standard Operating Procedures' currently in force at INTOX PVT. LTD.

2.4 SAFETY PRECAUTIONS

Gloves, facemask and goggles were used in addition to protective body garments and footwear to ensure adequate personal health and safety and to avoid inhalation and skin contact with the test item.

3. MATERIALS AND METHODS

3.1 TEST AND REFERENCE ITEMS

3.1.1 TEST ITEM INFORMATION

*	Test Item Name	:	**Chinchex Bed Bugs Insecticide**
*	Batch Number	:	100113
*	Identity	:	Silicon Dioxide
	Molecular Structure	:	
*	Storage Conditions	:	Room Temperature (27 ± 9 °C)
*	Date of Manufacture	:	06-01-2022 (DD-MM-YYYY)
*	Date of Expiry	:	06-01-2032 (DD-MM-YYYY)
	Sponsor	:	**Chinchex Limited,** 12F, Wing Fat Loong Building, 136, Wai Yip Street, Kwun Tong, Hong Kong, China
	Manufactured and Supplied by	:	Sponsor
	Source of Information	:	*Sponsor

3.1.2 CHARACTERISATION OF THE TEST ITEM

Characterisation of test item was performed by the Sponsor. The Sponsor has provided an authorized 'Certificate of Analysis' identifying the test item with the batch number, manufacturing date, expiry date and other physical parameters. The Certificate of Analysis of test item provided by Sponsor is included in this report.

3.1.3 REFERENCE ITEM

SILICON AA STANDARD: 10000 µg/mL Si IN H_2O

Name	:	Silicon (Si)
Lot No.	:	0109476544
CAS No.	:	16919-19-0
Source and Supplied by	:	Agilent Technologies
Date of Release	:	15 September 2020
Date of Expiration	:	30 September 2023
Certified Concentration	:	10020 ± 50 µg/mL (w/v)
		9675 ± 44 µg/g (w/w)
Storage condition at INTOX	:	Room Temperature (27 ± 9 °C)

3.1.4 CHARACTERISATION OF THE REFERENCE ITEM

Characterisation of reference item was performed by the manufacturer, Agilent. The manufacturer has provided an authorized 'Certificate of Analysis' (COA) identifying the reference item with the lot number, date of release, date of expiration and other physical parameters. The 'Certificate of Analysis' is included in this report.

3.2 TEST SYSTEM

3.2.1 INSTRUMENTS AND MATERIALS

Following instruments and materials were used in this study:

Sr. No.	Name of the Instruments and Materials	Make
1	Weighing balance	Shimadzu and Contech
2	Oven	Thermolab and PSM Scientific
3	pH meter	Eutech
4	Water bath	Thermolab and Biotechnolab BTL
5	Stop watch	Sunsui
6	Desiccator	Tarson
7	Thermometer	JRM
8	Colour matching cabinet	B-Tex Engineering
9	Atomic Absorption Spectrophotometer (AAS)	Agilent
10	PE bottles	-
11	PMP (Polymethylpentene) Volumetric Flask	VITLAB
12	Micropipette	Fisher Scientific
13	Sieve Shaker	ACE
14	Tap Density Apparatus	Veego
15	General laboratory glassware	Borosil and Qualigens

3.2.2 CHEMICALS AND REAGENTS

Following chemicals and reagents were used in this study:

Sr. No.	Name of the Chemicals and Reagents	Make
1	Sodium Hydroxide	Merck
2	Phenolphthalein	Fisher Scientific
3	Potassium hydrogen phthalate	Merck
4	Analytical grade water	Millipore
5	Buffer solutions of pH 4.00, 7.00 and 9.20	Qualigens
6	Barium chloride	Fisher Scientific
7	Distilled Water	Inhouse
8	Sodium Hydroxide solution (0.02M)	Inhouse
9	Barium chloride solution (10% w/v)	Inhouse
10	Hydrofluoric acid	Qualigens

3.3 STUDY DESIGN

The study comprised of the following steps:

3.3.1 Validation of Analytical Method

Atomic Absorption Spectroscopy Method was employed for analysis of **Silicon**. The method was validated for determination of **Silicon** (active ingredient) in **Chinchex Bed Bugs Insecticide**. Certified analytical reference standard of **Silicon** was used for the validation of **Silicon**.

3.3.2 Performance of the Accelerated Storage Stability and Corrosion Characteristics Test

The test item was subjected to accelerated storage at 54 ± 2 °C for 14 days. The test item was analyzed for the following parameters before and after storage.

- Physical Appearance of test item (Physical state and Colour)
- Active ingredient content
- pH and Acidity/Alkalinity
- Dry Sieve Analysis
- Corrosion Characteristics

3.4 VALIDATION OF ANALYTICAL METHOD FOR SILICON

Validation, Identification and Quantification of **Silicon** was performed by using Atomic Absorption Spectrophotometer (AAS).

The **Silicon** (**Si**) in sample solution was determined by Flame Atomic Absorption (FAA) method. The sample containing **Silicon** was digested with concentrated Hydrofluoric acid. The digested sample solution was aspirated in Nitrous oxide/ Acetylene flame and detected at 251.6 nm using hollow cathode lamp.

The Flame Atomic Absorption (FAA) method of analysis was validated before use for the parameters viz., System suitability, Linearity, Accuracy, Precision, Limit of Detection (LOD) and Limit of Quantification (LOQ).

Following Spectrophotometric conditions were used for the identification and quantification of Silicon.

3.4.1 AAS SPECTROPHOTOMETRIC CONDITIONS:

Method	:	**Silicon** (Flame)
Sampling mode	:	Manual
Calibration Mode	:	Concentration
Measurement Mode	:	Integrate
Wavelength	:	251.6 nm
Slit width	:	0.2 nm
Lamp Current	:	10 mA
Background correction	:	On
Calibration Algorithm	:	New Rational
Flame Type	:	Nitrous oxide / Acetylene
Correlation Coefficient limit	:	0.99

3.4.2 LINEARITY AND SYSTEM SUITABILITY

The linearity of the method was determined by analyzing six concentrations of **Silicon** reference item. The six concentrations of the **Silicon** reference item were 10.02 mg/L, 20.02 mg/L, 30.06 mg/L, 40.08 mg/L, 50.10 mg/L and 60.12 mg/L.

The system suitability was determined from the six concentrations of the **Silicon** reference item (10.02 mg/L, 20.04 mg/L, 30.06 mg/L, 40.08 mg/L, 50.10 mg/L and 60.12 mg/L) analysed in linearity. In system suitability, the % relative standard deviation (% RSD) of absorbance of each solution analysed for **Silicon** was determined and reported.

Preparation of Working Stock of Silicon Standard Solution

A volume of 5 mL of 10020 µg/mL solution Silicon was transferred to 50 mL volumetric flask and diluted up to mark with distilled water. This was 1002.00 µg/mL working stock of Silicon Standard Solution.

The solutions were prepared as per the following procedure:

Level	Volume of 1002.00 µg/mL Silicon (Si) standard solution added (mL)	Final Volume with distilled water (mL)	Concentration (mg/L)
0	0	100	Blank
1	1	100	10.02
2	2	100	20.04
3	3	100	30.06
4	4	100	40.08
5	5	100	50.10
6	6	100	60.12

3.4.3 PRECISION

The repeatability of the method was determined by analyzing five independent samples of similar concentration of **Silicon**.

The % RSD of **Silicon** content (% w/w) in sample of **Silicon** analysed was determined.

Preparation of solution of test item for determination of Silicon content:

A quantity of 5 ± 0.5 mg of test item was weighed and transferred to 50 mL polymethylpentene (PMP) volumetric flask. A volume of 2.5 mL of Hydrofluoric Acid was added to the flask and test item was allowed to dissolve. After dissolution volume was made up to mark with distilled water. This was 100 mg/L nominal concentration solution of test item.

Five samples were prepared as above. These solutions were analysed to determine the **Silicon** content in the test item.

The details of the test item weighed and concentration of test item solution are as given below:

Sample No.	Weight of the Test Item (g)	Volume of Hydrofluoric Acid added (mL)	Dilution Volume (mL)	Concentration of Test Item solution (mg/L)
P1	5.44	2.5	50	108.80
P2	5.07	2.5	50	101.40
P3	5.02	2.5	50	100.40
P4	5.08	2.5	50	101.60
P5	5.30	2.5	50	106.00

3.4.4 ACCURACY (% RECOVERY)

Accuracy of the method was determined by fortifying **Silicon** reference item in test item solutions. Accuracy was reported on the basis of % recovery at the fortified concentration (mg/L).

Preparation of Solutions for Accuracy:

A quantity of 25.16 mg of test item was weighed and transferred to 50 mL polymethylpentene (PMP) volumetric flask. A volume of 5 mL of Hydrofluoric Acid was added to the flask and test item was allowed to dissolve. After dissolution volume was made up to mark with distilled water. This was 503.20 mg/L nominal concentration solution of test item.

The solutions for the accuracy was prepared as given below:

Level	Volume of 503.20 mg/L test item solution added (mL)	Volume of 1002.00 mg/L Silicon standard solution added (mL)	Final Volume (mL)	Concentration of (mg/L)	
				Spiked Silicon Standard solution	Test Item
Un-spiked	5	0.0	50	-	50.32
1	5	0.5	50	10.02	50.32
2	5	1.0	50	20.04	50.32

The above solutions were analysed by aspirating them in atomic absorption spectrometer and the spiked concentration of **Silicon** in above analysed solutions was determined.

3.4.5 LIMIT OF DETECTION (LOD) AND LIMIT OF QUANTIFICATION (LOQ)

Solutions of lower concentrations (**Aluminium reference item**) than initial concentration of linearity were analysed successively. The concentration, at which analyte was detected, that concentration was taken as limit of detection (LOD) of the method. Based on response of LOD, the concentration of limit of quantification (LOQ) was decided. The solutions were prepared as per the following procedure:

Level	Volume of Silicon (Si) working standard solution (1002.00 mg/L, prepared for linearity test) added (mL)	Final Volume with distilled water (mL)	Concentration (mg/L)
1	0.04	20	2.0040
2	0.08	20	4.0080
3	0.12	20	6.0120
4	0.16	20	8.0160

3.5 PERFORMANCE OF THE ACCELERATED STORAGE STABILITY AND CORROSION CHARACTERISTICS TEST

3.5.1 TEST TEMPERATURE AND TEST PERIOD

The test was performed at 54 ± 2 °C for 14 days.

3.5.2 PACKAGING MATERIAL

PE bottles were used as packaging material. The information on packaging material was supplied by Sponsor.

3.5.3 EXPERIMENTAL PROCEDURE

The sample of test item was analysed prior to storage stability test for the parameters as mentioned in Section 3.3.2.

The test item was stored at accelerated storage in an oven at 54 ± 2 °C for 14 days. Eight PE bottles filled with test item provided by Sponsor were placed as such an oven at 54 ± 2 °C for accelerated storage period of 14 days. The gross weight of the PE bottles filled with test item was recorded.

An empty PE bottle (without test item) was kept along with above samples and weight was recorded.

The temperature of the oven was recorded once daily for up to 14 days. At the end of accelerated storage period, the PE bottles were removed from oven and were kept in desiccator to attain room temperature. Gross weights of packaging materials (empty and filled with test item) were recorded after storage period of 14 days. The test item was examined and tested for various parameters as mentioned in section 3.3.2.

The results obtained before storage were compared with results obtained after accelerated storage.

3.6 DETERMINATION OF ACTIVE INGREDIENT CONTENT IN THE TEST ITEM

Preparation of solution of test item for determination of Silicon content:

A quantity of 10 ± 0.5 mg of test item was weighed and transferred to 100 mL PMP volumetric flask. A volume of 5 mL of Hydrofluoric acid was added to the flask and test item was allowed to dissolve. After dissolution, volume was made up to mark with distilled water. This was 100 mg/L nominal concentration solution of test item.

Two samples of **Chinchex Bed Bugs Insecticide** were prepared as above to determine **Silicon** (% w/w) in the it.

The details of the test item weighed and concentration of test item solution are as given below:

Storage Period	Sample No.	Weight of the Test Item (g)	Volume of Hydrofluoric Acid added (mL)	Dilution Volume (mL)	Concentration of Test Item solution (mg/L)
Before Storage	S1	10.17	5	100	101.70
	S2	10.03	5	100	100.30
After Storage	R1	10.23	5	100	102.30
	R2	10.08	5	100	100.80

Results: The mean of two samples is taken as active ingredient content in the test item determined before and after storage.

3.7 DETERMINATION OF pH and ACIDITY OF THE TEST ITEM

3.7.1 Determination of pH of Test Item

Test Condition

The test was performed at 25.0 ± 1.0 °C.

pH Meter Calibration

The pH meter was calibrated with buffer solutions of pH 4.00, 7.00 and 9.20 at 25.0 ± 1.0 ºC before start of the test. The test temperature of the buffer solutions was maintained using water bath.

Preparation of sample for determination of pH (1 % w/v)

A quantity of 1 ± 0.1 g of the sample of **Chinchex Bed Bugs Insecticide** was weighed and transferred in 100 mL measuring cylinder containing about 50 mL of distilled water. The volume was made up to 100 mL mark with distilled water. It was shaken vigorously until the test item get completely mixed. The resultant mixture was then transferred to a beaker. The test temperature of the sample was maintained using a water bath.

The details of the amount of test item weighed are tabulated below.

Replicate No.	Weight of test item (g)	
	Before storage	At the end of accelerated storage
1	1.0860	1.0069
2	1.0413	1.0072
3	1.0088	1.0001

Determination of pH of suspension of test item

Once the samples achieved the test temperature of 25.0 ± 1.0 ºC, the pH of the solution was measured using the calibrated pH meter. The glass electrode and the temperature probe were dipped in the solution. For Replicate 1 of before storage, the pH value changed by more than 0.1 unit during equilibration time. Hence pH for Replicate 1 of before storage was recorded after 10 minutes. For all other replicates of before and after storage, the pH value after 1 minute did not change by more than 0.1 pH units hence, pH value after 1 minute was recorded.

3.7.2 Determination of Acidity test of Test Item

The acidity of **Chinchex Bed Bugs Insecticide** was determined by titration with standard alkali using electrometric end point determination. The titration was carried out in distilled water.

The test were performed in two parts:

1. Preparation and Standardization of reagents
2. Determination of acidity of test item

Preparation and standardization of reagent

The reagents were prepared and standardized before use. As pH of test item was less than 7, the acidity was determined by using 0.02 M sodium hydroxide solution.

Preparation of Barium Chloride Solution 10% w/v

A quantity of 1.0018 g of barium chloride was diluted to 10 mL with distilled water.

Preparation and Standardization of 0.02 M Sodium hydroxide

Preparation of 0.02 M Sodium hydroxide

A quantity of 0.4501 g of sodium hydroxide was weighed and dissolved in 250 mL volume of distilled water. 2 mL of 10% w/v barium chloride solution was added in above solution to precipitate carbonate present in it. The solution was shaken well and filtered through sintered fluoride funnel. It was transferred in 500 mL volumetric flask and diluted up to the mark to 500 mL with distilled water. This solution was stored in polyethylene bottle which protected by soda-lime guard tube.

Standardization of 0.02 M Sodium hydroxide

Potassium hydrogen phthalate was dried in oven at 105 °C for 2 hours and cooled in a desiccator. 0.10 ± 0.01 g (w_1) of potassium hydrogen phthalate was weighed and transferred to a conical flask from which the carbon dioxide was removed by a current of carbon dioxide-free air or nitrogen. Carbon dioxide free distilled water (about 50 mL) was added to the stoppered conical flask and swirled to dissolve the salt. A few drops of phenolphthalein indicator solution was added. The colourless salt solution was titrated with the 0.02 M sodium hydroxide solution to the first appearance of a permanent pink colour (t_1 mL). The solution was standardized immediately before use.

The details of the amount (w_1) of potassium hydrogen phthalate weighed and volume (t_1 mL) of 0.02 M sodium hydroxide solution required for titration are tabulated below.

Particulars	Weight of potassium hydrogen phthalate (g)	Volume (t_1 mL) of 0.02 M sodium hydroxide solution
Before Storage	0.1010	24.6
After Storage	0.1020	24.3

Determination of Acidity of Test Item by Electrometric End Point

A quantity of 10.0 ± 0.001 g (w_2) of the test item was weighed and transferred to 250 mL beaker. 100 mL of distilled water was added in beaker and stirred with a glass rod for about 1 minute to homogenize the solution. The pH of the test item was recorded using calibrated pH meter (calibrated using 4.00, 7.00 and 9.20 pH buffer solutions). The solution was titrated electrometrically to pH 7.00 ± 0.05 at test temperature by using standardized 0.02 M sodium hydroxide solution depending upon pH of the test item. The volume of 0.02 M standardized sodium hydroxide solution (t_2 mL) required to achieve pH 7.00 ± 0.05 was noted. The test was carried out in duplicate and the mean of two values are reported as Acidity.

The details of the amount of test item weighed are tabulated below.

Replicate No.	Weight of test item (g)	
	Before Storage	After Storage
1	10.0008	10.0007
2	10.0005	10.0008

3.8 DETERMINATION OF BULK DENSITY AND DRY SIEVE TEST

3.8.1 Determination of Bulk Density

a) Objective

The objective of this study was to determine the Bulk Density of test item. This study provides information on the volume occupied by a known weight of test item.

The data may be used to assess the manner and extent that chemicals and components of mixtures are transported in the environment and locations where they may be deposited. This information is useful in determining the packing size suitable for the product.

b) Outline of Method

A known weight of a test item was placed in a glass measuring cylinder and its volume was measured (to determine the 'pour density'). The cylinder was then be raised and allowed to fall vertically through a distance of 25 mm on to a rubber pad (50 times) using a Dry Substance Jolting Voltmeter. The cylinder was rotated during tapping to minimize any possible separation of the test item during tapping. The volume was measured again (to determine the 'tap density').

c) Definition

The 'pour density' is the apparent density of a bed of material formed in a container of standard dimensions when a specified amount of the material is introduced without settling.
The 'tap density' is the density after the material is vibrated or tapped under standard conditions.

d) Procedure

Test item was poured smoothly into the 100 ml cylinder up to 90 % of its capacity. Test item was carefully levelled off without compacting it. Cylinder was fitted in its holder and was mounted on its port. Display shows the weight of test item. Unsettled volume of the test item (V_1 cc) was entered on display. All the test parameters were programmed correctly (50 taps) and run.

When tapping was completed, volume of the test item in the cylinder was observed and it was entered on display (V_2 cc). After the test over, display shows Bulk density and Tap density values. Above procedure was repeated again twice with the same test item. Mean value and the standard deviation of the three replicates were calculated and reported.

Details of test item weighed are mentioned below;

Particulars	Weight of Test Item (g)		
	Replicate -1	Replicate -2	Replicate -3
Before Storage	4.57	4.52	4.55
After Storage	4.63	4.68	4.58

e) Interpretation of Results

The density was reported as the mean value of the three replicates. The estimate of accuracy is given as Standard Deviation of the results.

3.8.2 Determination of Dry Sieve Test

a) Outline of the Method

Sieve analysis consists of the quantitative separation of test item into fraction of different particle size ranges.

b) Experimental Procedure

Sampling of the test item: Sufficient quantity of the test item was weighed and placed into a sufficiently large polyethylene bag by filling this at least one third. The contents in the polyethylene bag was mixed by turning closed bag end over end at least 10 times. This polyethylene bag containing test item was placed on flat surface and the test item was spread over large area as possible. (Note: The sample layer should be approximately 1 cm thick.) The required quantity (M g) of the test item was taken from minimum five positions from the test item layer.

The mass (M g) of the test item is calculated using following formula.

$$M = n \times D \times 20 \text{ g}$$

Where, n= number of sieves used, D = tap density (g/mL) of test item (calculated in this study).

The nest of the sieve was assembled in the correct order with the coarsest at the top and the finest at the bottom.

The weighed quantity of test item (M g) was transferred to the coarsest sieve and the lid was fitted to the nest of sieves.

The nest of the sieve was placed in the sieving machine and was run for a period of 5 minutes ± 1 minutes.

After 5 minutes (± 1.0 minute), the nest of sieves was removed from the machine and was allowed to stand for 2 minutes to allow the suspended dust to settle.

The lid was removed carefully and each sieve was inverted separately on sheet of butter paper.

The side of the sieves was tapped and the uppermost surface was brushed carefully.

The sieve was then inverted and any loose particles remaining inside the sieve was brushed out carefully.

The brushing was added to the bulk of test item taken from each sieve.

This procedure was followed for each sieve and the mass from each sieve was recorded.

The residue (r_x) on each sieve was calculated and the sum of residue (R_x) on all the x sieves were calculated. Details of test item weighed are mentioned below.

Particulars	Weight of Test Item (g)	
	Replicate -1	Replicate -2
Before Storage	5.0088	5.0026
After Storage	5.0858	5.0128

c) Interpretation of Results

The particle size distribution of the test item is specified by the range [x_1, x_2] of two sieves where $R_x \geq 90\%$ and $R_x \geq 10\%$.

3.9 DETERMINATION OF PHYSICAL APPEARANCE OF THE TEST ITEM

The test item was observed visually before and after completion of accelerated storage period for physical state and colour.

Determination of Physical State

Determination of physical state of the test item is based on visual inspection of the test item at 25.0 ± 1.0 °C.

A quantity of 2.0 ± 0.1 g of **Chinchex Bed Bugs Insecticide** was transferred to a glass container. The glass container was kept in the water bath till the temperature of the test item reached at 25.0 ± 1.0 °C.

The physical state of the test item was determined by visual observation at 25.0 ± 1.0 °C. Duplicate determinations was made using the same sample by two different Study Personnel.

Determination of Colour

A quantity of 2.0 ± 0.1 g of test item was transferred in a clean transparent test tube. The test item was visualized under colour matching cabinet with D-65 source. The visual description of the colour was reported qualitatively.

The test was performed in duplicate by two different study personnel using the same sample of test item and the result are reported.

The details of the amount of test item weighed are tabulated below.

Particulars	Weight of Test Item (g)	
	Physical State	Colour
Before Storage	2.0010	2.0010
After Storage	2.0013	2.0013

3.10 DETERMINATION OF CORROSION CHARACTERISTICS

The PE bottles placed in contact with test item was examined visually for "Perforations", "Darkening", "Leaking or rust at the seam".

The change in gross weight of the PE bottles was recorded.

3.11 CALCULATIONS

Following formulae were used for calculations.

1) Molarity of Sodium hydroxide (N) was calculated by using the following equation:

$$\text{c (Sodium hydroxide) moles / L (Molarity)} = \frac{4.898 \times w}{t}$$

2) The **Silicon** content was determined by using following formula:

Silicon Content (% w/w) =

$$\frac{\text{Analysed concentration of } \textbf{Silicon} \text{ by AAS (µg/L or mg/L)} \times 100}{\text{Actual Concentration of } \textbf{Chinchex Bed Bugs Insecticide} \text{ solution (µg/L)}}$$

3) The Silicon Dioxide Content (SiO2) (%w/w) was determined by using following formula:

Silicon Dioxide
(SiO_2) (%w/w) = % Content of the Silicon (Si) in **Chinchex Bed Bugs Insecticide** X F

4) The amount of **Silicon** recovered was calculated by using following formula:

Amount recovered (µg/L or mg/L) = Analyzed concentration of Silicon in spiked solution (µg/L or mg/L) - Analyzed concentration of Silicon in un-spiked solution of test item (µg/L or mg/L)

5) The recovery was calculated by using following formula:

$$\% \text{ Recovery} = \frac{\text{Amount recovered (µg/L or mg/L)}}{\text{Amount added (µg/L or mg/L)}} \times 100$$

6) The % m/m Acidity of test item was calculated by using the following equation

$$\text{Acidity calculated as } H_2SO_4 \text{ (\% m/m)} = \frac{4.904 \times t_2 \times c_1}{w_2}$$

Where,

t_2 = Volume of standardized 0.02 M sodium hydroxide (mL)

c_1 = Normality of 0.02 M sodium hydroxide (mol/L)

w_2 = Weight of test item (g)

3.12 INTERPRETATION OF RESULTS

The results obtained from the validation study parameters was evaluated on the basis of the acceptance criteria to decide the suitability of the method of analysis for **Silicon**. The method is said to be **validated**, as validation parameters are found within the acceptance criteria.

The results of test parameters as mentioned for Accelerated Storage Stability and Corrosion Characteristics are compared with their results before Accelerated Storage.

4. RESULTS

The study was conducted in two steps, Validation of Flame Atomic Absorption method for **Silicon** and detemination of Accelerated Storage Stability and Corrosion Characteristic of **Chinchex Bed Bugs insecticide**.

4.1 VALIDATION OF ANALYTICAL METHOD (Table 1)

The **Silicon** (**Si**) in sample solution was determined by Flame Atomic Absorption (FAA) method. The Flame Atomic Absorption (FAA) method of analysis was validated before use for the parameters viz., System suitability, Linearity, Accuracy, Precision, Limit of Detection (LOD) and Limit of Quantification (LOQ).

Validation, was performed using Atomic Absorption Spectrophotometer (AAS).

The summarized results of analytical method validation for **Silicon (Si)** are as given below:

Parameter		Acceptance Criterion	Results
Method		-	Flame Atomic Absorption
Linearity		**>0.99**	1.0000
System Suitability		**% RSD ≤ 5%**	< 5%
Accuracy (% Recovery)	**Level - 1**	**80-120%**	98.00
	Level - 2		97.36
Precision		**%RSD ≤ 20%**	4.25
LOD (mg/L)		-	2.0040
LOQ (mg/L)		-	4.0080

Based on the above results it is concluded that, the analytical method is **Validated** and **Suitable** for determination of **Silicon** (Si) in **Chinchex Bed Bugs insecticide**.

4.2 ACCELERATED STORAGE STABILITY TEST

The test item was subjected to accelerated storage at 54 ± 2°C for 14 days. The test item was subjected to following tests before storage and at the end of accelerated storage period.

- Physical Appearance of Test Item (Physical state and Colour)
- Active Ingredient Content
- pH and Acidity
- Dry Sieve Analysis
- Corrosion Characteristics

The results of various parameters performed as mentioned above (before storage and at the end of accelerated storage) are as follows:

4.2.1 ACTIVE INGREDIENT CONTENT IN THE TEST ITEM (Table 2)

The results of active ingredient content in **Chinchex Bed Bugs Insecticide** before and after storage in two samples are presented in the following table:

ACTIVE INGREDIENT CONTENT				
Sample No.	**Silicon (Si) content (% w/w)**		**Silicon Dioxide (SiO_2) content (% w/w)**	
	Before Storage	**After Storage**	**Before Storage**	**After Storage**
1	43.69	43.89	93.47	93.91
2	44.41	44.28	95.01	94.73
Average	**44.05**	**44.08**	**94.24**	**94.32**

4.2.2 pH AND ACIDITY (Table 3)

The pH of test item before and after storage in three samples are presented in the following table:

Replicate No.	**pH**			
	Temperature (°C)	**Before Storage**	**Temperature (°C)**	**After Storage**
1	24.7	6.82	24.7	6.67
2	24.7	6.58	24.3	6.64
3	24.6	6.69	24.3	6.65
Average	-	**6.70**	-	**6.65**

The results of Acidity (% m/m) before and after storage in two samples are presented in the following table:

Sample No.	**Acidity (% m/m)**	
	Before Storage	**After Storage**
1	0.0050	0.0079
2	0.0059	0.0079
Average	**0.0054**	**0.0079**

4.2.3 DRY SIEVE ANALYSIS (Table 4)

a) Bulk Density

The bulk density of test of test item was performed in triplicate and results found are presented in below table.

Replicate No.	Before Storage		After Storage	
	Bulk Density	Tap Density	Bulk Density	Tap Density
1	0.050	0.056	0.051	0.056
2	0.050	0.056	0.052	0.056
3	0.050	0.056	0.050	0.057
Mean	**0.050**	**0.056**	**0.051**	**0.056**

b) Dry Sieve Test

The mass of test item required for dry sieve test was decided based on the tap density. Sieve analysis consists of the quantitative separation of powder into fraction of different particle size ranges. The results of % mass of Test Item Remains on Sieve (r_x) and Sum of % mass of Test Item (R_x) are presented in the following table:

Before Storage							
Sample No.	Weight of Test Item	Particulars	Sieve Size (µm)				
			150	250	355	500	850
1	5.0088	Test Item Remains on Sieve (g_x in g)	2.1792	0.5267	0.4535	0.6799	0.7630
		% mass of Test Item Remains on Sieve (r_x)	43.51	10.52	9.05	13.57	15.23
		% mass of Test Item (R_x)	91.88	48.38	37.86	28.81	15.23
Inference = $R_x \geq 90\%$ = **91.88**, $R_x \geq 10\%$ = **15.23**							
2	5.0026	Test Item Remains on Sieve (g_x g)	2.3501	0.5321	0.4853	0.6011	0.7618
		% mass of Test Item Remains on Sieve (r_x)	46.98	10.64	9.70	12.02	15.23
		% mass of Test Item (R_x)	94.56	47.58	36.94	27.24	15.23
Inference = $R_x \geq 90\%$ = **94.56**, $R_x \geq 10\%$ = **15.23**							
Average (R_x) = $R_x \geq 90\%$ =**93.22**, $R_x \geq 10\%$ =**15.23**							

After Storage							
Sample No.	Weight of Test Item	Particulars	Sieve Size (µm)				
			150	250	355	500	850
1	5.0858	Test Item Remains on Sieve (g_x in g)	0.8884	1.1747	0.8452	1.1038	0.6362
		% mass of Test Item Remains on Sieve (r_x)	17.47	23.10	16.62	21.70	12.51
		% mass of Test Item (R_x)	91.40	73.93	50.83	34.21	12.51
Inference = $R_x \geq 90\%$ = **91.40**, $R_x \geq 10\%$ =**12.51**							
2	5.0128	Test Item Remains on Sieve (g_x g)	0.8024	1.1008	0.8301	1.2018	0.6471
		% mass of Test Item Remains on Sieve (r_x)	16.01	21.96	16.56	23.97	12.91
		% mass of Test Item (R_x)	91.41	75.40	53.44	36.88	12.91
Inference = $R_x \geq 90\%$ = **91.41**, $R_x \geq 10\%$ = **12.91**							
Average (R_x) = $R_x \geq 90\%$ = **91.40**, $R_x \geq 10\%$ =**12.71**							

The results of dry sieve test of test item are found comparable before and after storage.

4.2.4 PHYSICAL APPEARANCE OF THE TEST ITEM (Table 5)

The test item was observed visually before and after completion of accelerated storage period for the determination of physical state and colour.

Parameters	Before Storage	After Storage
Physical state (25 ± 1°C)	Powder	Powder
Colour (Visual)	White	white

4.2.5 CORROSION CHARACTERISTICS (Table 6)

The PE bottles were kept in contact with the test item at accelerated storage temperature, 54 °C ± 2 °C up to 14 days. The PE bottles were removed after 14 days and examined physically and visually for "Perforations", "Darkening", "Rust at the seam".

No "Perforations", "Darkening", "Rust at the seam" were observed on PE bottles due to contact with test item at accelerated storage temperature (54 ± 2 °C) after 14 days. No significant changes in gross weights of the container containing test item were found due to storage at accelerated temperature after 14 days.

Hence, it is concluded that the packaging material (PE bottle) is **compatible** with test item.

5. CONCLUSION

From the results, it is concluded that **Chinchex Bed Bugs Insecticide** was **stable** at accelerated storage temperature (54 ± 2 °C) up to storage period of 14 days.

6. ARCHIVES

The following shall be retained in the Archives of INTOX PVT. LTD. for period starting from the date of submission of final report, as specified below.

Material	Study Plan	Raw Data	Draft Report	Final Report	Sample of Test Item
Period of Archiving	A period, whichever is less, among the period covering three GLP inspection / certification cycles undergone by the Test Facility, or Nine years after completion of the study.				Till expiry date of Test Item

7. QUALITY ASSURANCE UNIT REVIEW

The Quality Assurance Unit has conducted inspections at various phases of the study and of certain repetitive operations, as per the Principles of Good Laboratory Practice, (OECD, 1998). The dates on which the findings of these inspections are reported to the Study Director and to the Test Facility Management have been specified in this report.

This report has been reviewed by Quality Assurance Unit comparing individual findings against raw data and comparing the statement and results presented in the report with individual data presented in the tables of the report.

8. DEVIATION

Sr. No.	As in Study Plan	Deviation
1	**Study Plan Page No. 10 of 27** **2.3 VALIDATION OF ANALYTICAL METHOD FOR SILICON** The Silicon (Si) in sample solution will be determined by Flame Atomic Absorption (FAA) method. The sample containing Silicon will be digested with concentrated 5% w/v Nitric acid.	**Report Page No. 16 of 59** **3.4VALIDATION OF ANALYTICAL METHOD FOR SILICON** The **Silicon** (**Si**) in sample solution was determined by Flame Atomic Absorption (FAA) method. The sample containing **Silicon** was digested with concentrated Hydrofluoric acid.
Justification: Typographical error.		
Impact on the Study: The Study Director declares that, this deviation from the approved study plan does not have any impact on the outcome of the study or the interpretation of the results.		

9. REFERENCES

- Product Properties Test Guidelines OPPTS No. 830.6303 "Physical State", United States Environmental Protection Agency, Prevention, Pesticides and Toxic Substances (7101) EPA 712–C–96–020 August 1996.
- Product Properties Test Guidelines, OPPTS 830.6302 "Color", United States Environmental Protection Agency, Prevention, Pesticides and Toxic Substances (7101) EPA 712–C–96–019 August 1996.
- OECD Guidelines for Testing of Chemicals No. 122, 'Determination of pH, Acidity and Alkalinity.'
- CIPAC Method MT 170. Dry sieve analysis of water dispersible granules, Handbook F.
- CIPAC Method MT 186 Guideline for determination of pour density, Volume K.
- EEC A.3 Guideline for determination of Relative Density.
- CIPAC Method MT 46.4, Accelerated Storage Procedures.
- EUROPEAN COMMISSION Directorate General Health and Consumer Protection SANCO/3030/99 rev.4 11/07/00 Technical Material and Preparations: Guidance for generating and reporting methods of analysis in support of pre- and post-registration data requirements for Annex II (part A, Section 4) and Annex III (part A, Section 5) of Directive 91/414. Working document.
- Manual on development and use of FAO and WHO specifications for pesticides (March 2006 revision of first edition and November 2010, 2nd revision of first edition).

10. TABLES

Note: All calculations are performed using Validated Excel Worksheet.

TABLE – 1

ANALYTICAL METHOD VALIDATION FOR SILICON (Si)

A) Linearity and System Suitability: Silicon

Nominal Concentration (m/L)	Actual Concentration (mg/L)	*Mean Absorbance	*Calculated Concentration (mg/L)	*% RSD
10	10.02	0.0211	10.29	1.4
20	20.04	0.0388	19.36	0.6
30	30.06	0.0597	30.40	0.9
40	40.08	0.0784	40.30	0.2
50	50.10	0.0968	49.90	0.7
60	60.12	0.1173	60.13	0.8

***Note:** Mean Absorbance, Calculated Concentration and %RSD were calculated by the SpectraAA software.

Curve Fit	:	New Rational
Characteristic Concentration	:	2.09 mg/L
Correlation Coefficient (r)	:	1.0000

TABLE – 1 (Contd.)

ANALYTICAL METHOD VALIDATION FOR SILICON (Si)

B) Accuracy

Level	Actual Conc. of Test Item (mg/L)	Conc. of Silicon Standard Solution added (mg/L)	*Mean Absorbance	*Analysed Conc. by AAS (mg/L)	Amount Recovered (mg/L)	% Recovery
Un-spiked	50.32	-	0.0480	24.21	-	-
1	50.32	10.02	0.0666	34.03	9.82	**98.00**
2	50.32	20.04	0.0849	43.72	19.51	**97.36**

***Note:** Mean Absorbance and Analysed Concentration are calculated by the SpectraAA software.

Typical Calculation: For Level 1

A) The amount of Silicon recovered was calculated by using following formula:

Amount recovered (mg/L) = Analysed concentration of **Silicon** in spiked solution (mg/L) - Analysed concentration of **Silicon** in un-spiked solution of test item (mg/L)

Amount recovered (mg/L) = 34.03 - 24.21 = **9.82 mg/L**

B) The %recovery was calculated by using following formula:

$$\% \text{ Recovery} = \frac{\text{Amount recovered (mg/L)}}{\text{Amount added (mg/L)}} \times 100 = \frac{9.82}{10.02} \times 100 = \mathbf{98.00\%}$$

TABLE – 1 (Contd.)

ANALYTICAL METHOD VALIDATION FOR SILICON (Si)

C) Limit of Detection (LOD) and Limit of Quantification (LOQ)

Concentration of Silicon Standard (mg/L)	*Mean Absorbance	* % RSD of Mean Absorbance
2.0040	0.0064	2.8
4.0080	0.0109	3.6
6.0120	0.0134	3.9
8.0160	0.0182	1.5

Result: *Based on Response (Mean Absorbance) and % RSD calculated by AAS.

Limit of Detection (LOD)	:	2.0040 mg/L
Limit of Quantification (LOQ)	:	4.0080 mg/L

D) Precision (Repeatability)

Sample No.	Weight of Test Item (g)	Conc. of Solution of Test Item (mg/L)	*Mean Absorbance	*Calculated Conc. of Silicon (mg/L)	% Content of Silicon (Si)	% Content of Silicon Dioxide (SiO_2)
P1	5.44	108.80	0.0865	44.55	40.95	87.61
P2	5.07	101.40	0.0879	45.29	44.66	95.56
P3	5.02	100.40	0.0879	45.26	45.08	96.45
P4	5.08	101.60	0.0883	45.48	44.76	95.78
P5	5.30	106.00	0.0868	44.71	42.18	90.25
Average			**0.0875**	**45.06**	**43.53**	**93.15**
SD			**0.001**	**0.404**	**1.851**	**3.961**
%RSD			**0.89**	**0.90**	**4.25**	**4.25**

***Note:** Mean Absorbance and Calculated Concentration were calculated by the SpectraAA software

Typical Calculation for Sample No. P1:

1) The Silicon (Si) content (%w/w) was determined by using following formula:

$$= \frac{\text{Analysed concentration of } \textbf{Silicon} \text{ by AAS (mg/L)} \times 100}{\text{Actual Concentration of } \textbf{Chinchex Bed Bugs Insecticide} \text{ solution (mg/L)}}$$

$$= \frac{44.55 \times 100}{108.80} = \textbf{40.95\% w/w}$$

2) The Silicon Dioxide Content (SiO_2) (%w/w) was determined by using following formula:

Silicon Dioxide (SiO_2) (%w/w) = % content of the Silicon (Si) in **Chinchex Bed Bugs Insecticide** X F

= 40.95 X 2.14 = **87.63% w/w**

Where,

$$\text{F (Factor)} = \frac{\text{Molar weight of } SiO_2}{\text{Molar weight of Si}} = \frac{68.08}{28.09} = 2.14$$

TABLE – 2

ACTIVE INGREDIENT CONTENT IN TEST ITEM

Sample No.	Weight of Test Item (g)	Conc. of Solution of Test Item (mg/L)	*Mean Absorbance	Analysed Conc. of Silicon by AAS (mg/L)	% Content of Silicon (Si)	% Content of Silicon Dioxide (SiO_2)
Before Storage						
1	10.17	101.70	0.0863	44.43	43.69	93.47
2	10.03	100.30	0.0865	44.54	44.41	95.01
Average	-	-	**0.0864**	**44.49**	**44.05**	**94.24**
After Storage						
1	10.23	102.30	0.0872	44.90	43.89	93.91
2	10.08	100.80	0.0867	44.63	44.28	94.73
Average	-	-	**0.0870**	**44.77**	**44.08**	**94.32**

***Note:** Mean Absorbance and Calculated Concentration were calculated by the SpectraAA software.

Typical Calculation for Before Storage Sample No. 1:

1) The Silicon content (% w/w) was determined by using following formula:

$$= \frac{\text{Analysed concentration of } \textbf{Silicon} \text{ by AAS (mg/L)} \times 100}{\text{Actual Concentration of } \textbf{Chinchex Bed Bugs Insecticide} \text{ solution (mg/L)}}$$

$$= \frac{44.43 \times 100}{101.70} = \textbf{43.69\%}$$

2) The Silicon Dioxide Content (SiO_2) (%w/w) was determined by using following formula:

Silicon Dioxide (SiO_2) (%w/w) = % content of the Silicon (Si) in **Chinchex Bed Bugs Insecticide** X F

= 43.69 X 2.14 = **93.47% w/w**

Where,

$$\text{F (Factor)} = \frac{\text{Molar weight of } SiO_2}{\text{Molar weight of Si}} = \frac{68.08}{28.09} = 2.14$$

TABLE – 3

pH AND ACIDITY

RESULTS OF pH OF TEST ITEM

(BEFORE STORAGE)

a) CALIBRATION OF pH METER

pH of the Buffer used	Before Calibration		After Calibration	
	Temperature of the Buffer Solution in °C	pH Observed	Temperature of the Buffer Solution in °C	Corrected pH
4.00	25.5	4.01	25.6	4.02
7.00	25.5	7.00	24.5	7.02
9.20	25.5	9.18	25.6	9.18
Remark = Passed the calibration check				

The pH meter passed the calibration test as the pH of buffer solutions were found within ± 0.05 units.

b) pH of 1% w/v AQUEOUS SOLUTION OF CHINCHEX BED BUGS INSECTICIDE

Preparation of Aqueous Solution of the Test Item:

Replicate No.	1	2	3
Weight of Test Item (g)	1.0860	1.0413	1.0088
Total Volume made with Distilled Water (mL)	100	100	100

c) RESULTS OF pH OF TEST ITEM:

Replicate No.	Initial Temperature (°C)	Initial pH	Temperature After 1 minute (°C)	pH After 1 minute	Temperature After 10 minutes (°C)	pH After 10 minutes	Final pH
1	25.8	8.29	25.5	7.40	24.7	6.82	6.82
2	24.7	6.63	24.7	6.58	-	-	6.58
3	24.7	6.65	24.6	6.69	-	-	6.69
Mean	-	-	-	-	-	-	**6.70**
± SD	-	-	-	-	-	-	**0.12**

TABLE – 3 (Contd.)

pH AND ACIDITY

RESULTS OF pH OF TEST ITEM
(AFTER STORAGE)

a) CALIBRATION OF pH METER

pH of the Buffer Used	Before Calibration		After Calibration	
	Temperature of the Buffer Solution in °C	pH Observed	Temperature of the Buffer Solution in °C	Corrected pH
4.00	24.9	4.01	24.8	4.05
7.00	24.9	7.00	25.1	7.04
9.20	24.9	9.18	24.9	9.21
Remark = Passed the calibration check				

The pH meter passed the calibration test as the pH of buffer solutions were found within ± 0.05 units.

b) pH of 1% w/v AQUEOUS SOLUTION OF CHINCHEX BED BUGS INSECTICIDE

Preparation of Aqueous Solution of the Test Item:

Replicate No.	1	2	3
Weight of Test Item (g)	1.0069	1.0072	1.0001
Total Volume made with Distilled Water (mL)	100	100	100

c) RESULTS OF pH OF TEST ITEM:

Replicate No.	Initial Temperature (°C)	Initial pH	Temperature After 1 minute (°C)	pH After 1 minute	Final pH
1	25.5	6.68	24.7	6.67	6.67
2	24.4	6.61	24.3	6.64	6.64
3	24.3	6.65	24.3	6.65	6.65
Mean	-	-	-	-	**6.65**
± SD	-	-	-	-	**0.02**

TABLE – 3 (Contd.)

pH AND ACIDITY

RESULTS OF ACIDITY OF TEST ITEM (BEFORE STORAGE)

CALIBRATION OF pH METER

pH of the Buffer Used	Before Calibration		After Calibration	
	Temperature of the Buffer Solution (°C)	pH Observed	Temperature of the Buffer Solution (°C)	Corrected pH
4.00	24.5	4.01	24.2	4.01
7.00	24.5	7.00	24.7	7.02
9.20	24.5	9.18	25.0	9.19
Remark = Passed the Calibration Check				

The pH meter passed the calibration test as the pH of buffer solutions were found within ± 0.05 units.

ACIDITY OF TEST ITEM

Sample No.	1	2
Weight of the test item used (g)	10.0008	10.0005
Volume of distilled water added (mL)	100	100
Initial pH of the solution of test item	6.77	6.78
Volume of 0.020 M standardized sodium hydroxide solution required to achieve pH 7.00 ± 0.05 (mL)	0.5	0.6
pH value obtained after addition of 0.020 M standardized sodium hydroxide solution	7.01	7.02
Acidity (% m/m)	0.0050	0.0059
Mean Acidity (% m/m)	**0.0054**	

Typical Calculation for Sample No. 1:

Acidity calculated as H_2SO_4 (% m/m) = $\frac{4.904 \times t_2 \times c_1}{w_2} = \frac{4.904 \times 0.5 \times 0.020}{10.0008} =$ **0.0050% m/m**

Where,

t_2 = Volume of standardized 0.020 M sodium hydroxide (mL)

c_1 = Normality of 0.020 M sodium hydroxide (mol/L)

w_2 = Weight of test item (g)

TABLE – 3 (Contd.)

pH AND ACIDITY

RESULTS OF ACIDITY OF TEST ITEM (AFTER STORAGE)

CALIBRATION OF pH METER

pH of the Buffer Used	Before Calibration		After Calibration	
	Temperature of the Buffer Solution (°C)	pH Observed	Temperature of the Buffer Solution (°C)	Corrected pH
4.00	24.4	4.01	25.5	4.04
7.00	24.4	7.00	25.6	7.00
9.20	24.4	9.18	25.5	9.19
Remark = Passed the Calibration Check				

The pH meter passed the calibration test as the pH of buffer solutions were found within ± 0.05 units.

ACIDITY OF TEST ITEM

Sample No.	1	2
Weight of the test item used (g)	10.0007	10.0008
Volume of distilled water added (mL)	100	100
Initial pH of the solution of test item	6.66	6.68
Volume of 0.020 M standardized sodium hydroxide solution required to achieve pH 7.00 ± 0.05 (mL)	0.8	0.8
pH value obtained after addition of 0.020 M standardized sodium hydroxide solution	7.04	7.01
Acidity (% m/m)	0.0079	0.0079
Mean Acidity (% m/m)	**0.0079**	

TABLE - 4

BULK DENSITY AND DRY SIEVE TEST

A) RESULTS OF BULK DENSITY

Replicate No.	Weight of Test Item (W g)	Volume of Test Item in Measuring Cylinder before Tapping (V_1 mL)	Volume of Test Item in Measuring Cylinder after Tapping (V_2 mL)	Bulk Density (D_p) (g/mL)	Tap Density (D_T) (g/mL)
BEFORE STORAGE					
1	4.57	90	81	0.050	0.056
2	4.52	90	80	0.050	0.056
3	4.55	90	82	0.050	0.055
Mean	-	-	-	**0.050**	**0.056**
± SD	-	-	-	**0.000**	**0.001**
AFTER STORAGE					
1	4.63	90	82	0.051	0.056
2	4.68	90	83	0.052	0.056
3	4.58	90	80	0.050	0.057
Mean	-	-	-	**0.051**	**0.056**
± SD	-	-	-	**0.001**	**0.001**

Typical Calculation, After Storage, for Replicate No.1:

$$\text{Bulk Density} = \frac{W}{V_1} = \frac{4.63}{90} = \mathbf{0.051\ g/mL}$$

$$\text{Tap Density} = \frac{W}{V_2} = \frac{4.63}{82} = \mathbf{0.056\ g/mL}$$

TABLE – 4 (Contd.)

BULK DENSITY AND DRY SIEVE TEST

B) RESULTS OF DRY SIEVE TEST

(BEFORE STORAGE)

Sample No.	Weight of Test Item (g)	Particulars	Sieve Size (µm)				
			150	250	355	500	850
1	5.0088	Test Item Remains on Sieve (g_x in g)	2.1792	0.5267	0.4535	0.6799	0.7630
		% mass of Test Item Remains on Sieve (r_x)	43.51	10.52	9.05	13.57	15.23
		% mass of Test Item (R_x)	91.88	48.38	37.86	28.81	15.23
Sum of % mass of Test Item (R_x) = $R_x \geq 90\%$ = **91.88**, $R_x \geq 10\%$ = **15.23**							
2	5.0026	Test Item Remains on Sieve (g_x g)	2.3501	0.5321	0.4853	0.6011	0.7618
		% mass of Test Item Remains on Sieve (r_x)	46.98	10.64	9.70	12.02	15.23
		% mass of Test Item (R_x)	94.56	47.58	36.94	27.24	15.23
Sum of % mass of Test Item (R_x) = $R_x \geq 90\%$ = **94.56**, $R_x \geq 10\%$ = **15.23**							
Mean		$R_x \geq 90\%$ = **93.22**		$R_x \geq 10\%$ = **15.23**			

Typical Calculations for Sample No. 1:

The % quantity (r_x) of the test item on sieve *x* is calculated:

$$r_x = \frac{g_x}{m} \times 100 \text{ \% m/m} = \frac{2.1792}{5.0088} \times 100 = \mathbf{43.51\%}$$

The % quantity (R_x) of the test item on sieve is calculated: (For Sieve size 150 µm)

R_x= [(% mass of test item remains on sieve 150 µm) + (% mass of test item on all remaining sieves)]

R_x= [43.51 + (10.52 + 9.05 + 13.57 + 15.23)] = **91.88%**

TABLE – 4 (Contd.)

BULK DENSITY AND DRY SIEVE TEST

C) RESULTS OF DRY SIEVE TEST

(AFTER STORAGE)

Sample No.	Weight of Test Item (g)	Particulars	Sieve Size (µm)				
			150	250	355	500	850
1	5.0858	Test Item Remains on Sieve (g_x in g)	0.8884	1.1747	0.8452	1.1038	0.6362
		% mass of Test Item Remains on Sieve (r_x)	17.47	23.10	16.62	21.70	12.51
		% mass of Test Item (R_x)	91.40	73.93	50.83	34.21	12.51
Sum of % mass of Test Item (R_x) = $R_x \geq 90\%$ = **91.40**, $R_x \geq 10\%$ = **12.51**							
2	5.0128	Test Item Remains on Sieve (g_x g)	0.8024	1.1008	0.8301	1.2018	0.6471
		% mass of Test Item Remains on Sieve (r_x)	16.01	21.96	16.56	23.97	12.91
		% mass of Test Item (R_x)	91.41	75.40	53.44	36.88	12.91
Sum of % mass of Test Item (R_x) = $R_x \geq 90\%$ = **91.41**, $R_x \geq 10\%$ = **12.91**							
Mean		$R_x \geq 90\%$ = **91.40**			$R_x \geq 10\%$ = **12.71**		

TABLE – 5

PHYSICAL APPEARANCE

A) PHYSICAL STATE

Replicate No.	Before Storage		After Storage	
	Temperature (°C)	Visual Observation	Temperature (°C)	Visual Observation
1	25.2	Powder	25.3	Powder
2	25.2	Powder	25.2	Powder

B) COLOUR

Replicate No.	Visual Observation	
	Before Storage	After Storage
1	White	White
2	White	White

TABLE – 6

% CHANGE IN GROSS WEIGHT

Sample ID	Gross Weight of Packaging Material + Test Item (kg)		Difference in Weight (kg)	% Change in Gross Weight (% Loss)	Observations
	Initial	Final			
R	1.170	1.167	-0.003	-0.256	No "Perforations", "Darkening", "Leaking or Rust at the seam" were observed and also no significant changes in gross weights of the container containing test item were found.
Control	0.089	0.089	0.000	0.000	

Typical Calculation for Sample ID R:

Difference in Weight (kg) = (Final Gross Weight - Initial Gross Weight)

= (1.167 - 1.170) = **- 0.003 kg**

$$\% \text{ Change in Weight} = \frac{(\text{Final Gross Weight} - \text{Initial Gross Weight}) \times 100}{\text{Initial Gross Weight}}$$

$$= \frac{(1.167 - 1.170) \times 100}{1.170} = \textbf{- 0.256\%}$$

TABLE – 7

TEMPERATURE RECORD OF ACCELERATED STORAGE

Day	Date (DD-MM-YYYY)	Temperature (°C)
0	13-09-2022	54
1	14-09-2022	54
2	15-09-2022	54
3	16-09-2022	54
4	17-09-2022	54
5	18-09-2022	54
6	19-09-2022	53
7	20-09-2022	54
8	21-09-2022	54
9	22-09-2022	54
10	23-09-2022	53
11	24-09-2022	54
12	25-09-2022	54
13	26-09-2022	54
14	27-09-2022	54

TABLE – 8

SUMMARIZED RESULTS OF ACCELERATED STORAGE STABILITY TEST

Sr. No.	Parameters		Results	
			Before storage	At the end of accelerated storage period
1	Active Ingredient Content (%)	Silicon Dioxide (SiO_2)	94.24	94.32
		Silicon (Si)	44.05	44.08
2	Appearance	Physical State	Powder	Powder
		Colour (Visual)	White	White
3	pH of 1% w/v aqueous solution (at 25.0 °C ± 1.0 °C)		6.70	6.65
4	Acidity (Calculated as H_2SO_4) (% m/m)		0.0054	0.0079
5	Dry Sieve Test		$R_x \geq 90\% = 93.22$	$R_x \geq 90\% = 91.40$
			$R_x \geq 10\% = 15.23$	$R_x \geq 10\% = 12.71$
6	Corrosion Characteristics		No "Perforations", "Darkening" and "Leaking or Rust at the seam" were observed on PE bottles due to contact with test item at accelerated storage temperature (54 ± 2 °C) after 14 days. No significant changes in gross weights of the PE bottles containing test item were found due to storage at accelerated temperature after 14 days.	

R/PCP4144/STB-AS/22

CERTIFICATE OF ANALYSIS OF TEST ITEM SUPPLIED BY THE SPONSOR

FORM 320
ISSUE DATE: 10.05.2022

CERTIFICATE OF ANALYSIS

Certificate Number	003	Supplier	Chinchex Limited
Product Code	3614657400013	Manufacture's Batch No.	100113
Product Name	Chinchex Bed Bugs Insecticide	Sydco's Lab.Batch No	
Date of Manufacture	06-Jan-2022	Expiry Date	06-Jan-2032

The Understigned hereby certifies the following data to be true specification of the obtained results of tests and assays.

Test	Results	Specifications (BP)
Appearance	White Powder	Conforms
Identity	Silicon Dioxide	Conforms
Carbonates		Less than Ph8.6
Solubility		Conforms
Ammonium		Lest than 20ppm
Calcium		Lest than 100ppm
Arsenic		Lest than 2ppm
Heav Metals		Lest than 2ppm
Iron		Lest than 20ppm
Sulphates		Lest than 150ppm
Chlorides		Lest than 150ppm
Appearance of Solution	Powder	Conforms
Micro Status	Nano	Conforms

10.05.22
DATE OF RELEASE

LABORATORY MANAGER

COMPANY ADDRESS:	**CONTACTS:**
12F, Wing Fat Loong Building 136, Wai Yip street Kwun Tong. Hong Kong China	Agurtzane Pombo 852 64807024 a.pombo@chinchex.com

CERTIFICATE OF ANALYSIS OF REFERENCE ITEM

Agilent

CERTIFICATE OF ANALYSIS

Agilent Product Name: Silicon Standard: 10000 µg/mL Si in H_2O

Agilent Part No: 5190-8450

Lot No: 0109476544

Product Specifications

Analyte	Starting Material	CAS #	Matrix	Certified Concentration
Si	$(NH_4)_2SiF_6$	16919-19-0	H_2O	10,020 ± 50 µg/mL (w/v)
				9675 ± 44 µg/g (w/w)

Intended Use: This solution is intended for use as a certified reference material or calibration standard for inductively coupled plasma optical emission spectroscopy (ICP-OES), inductively coupled plasma mass spectrometry (ICP-MS), atomic absorption spectroscopy (flame AAS or GFAAS), microwave plasma atomic emission spectroscopy (MP-AES), x-ray fluorescence spectroscopy (XRF), and other techniques for elemental analysis.

Certification & Traceability: This CRM was manufactured under a quality management system that is accredited to **ISO 17034, ISO/IEC 17025,** and registered to **ISO 9001.** This CRM was prepared to a nominal concentration of 10000 µg/mL by gravimetric methods using 99.999% ammonium hexafluorosilicate [$(NH_4)_2SiF_6$] dissolved and diluted with ASTM Type I Water. The balances used in the preparation of this CRM are calibrated regularly with traceability to NIST. All volumetric dilutions are performed in Class A calibrated glassware. The certified concentration and uncertainty were determined using the "High Performance ICP-OES" protocol developed by NIST and both the certified concentration and uncertainty values are traceable to NIST SRM 3150, lot #130912. The uncertainty associated with the certified concentration represents the expanded uncertainty at the 95% confidence level using a coverage factor of k=2.

Uncertified Values: Agilent ICP-MS was used to determine trace metal concentrations for this product (nd = not determined).

Trace Concentrations (µg/L)

Ag	<5	Co	<10	Ge	<5	Lu	<2	P	<1000	Sb	48	Te	<10
Al	<20	Cs	<5	Hf	<2	Mg	<50	Pb	<10	Sc	107	Ti	<20
As	<20	Cr	<5	Hg	<5	Mn	<10	Pd	<5	Se	<20	Tl	<5
Au	<5	Cu	<10	Ho	<2	Mo	<5	Pr	<2	Si	MAJOR	Tm	<2
B	<50	Dy	<2	In	nd	Na	<250	Pt	<5	Sm	<2	V	<10
Ba	28	Er	<2	Ir	<2	Nb	<5	Rb	<5	Sn	23	W	<5
Bi	<2	Eu	<2	K	<250	Nd	<2	Re	<2	Sr	<10	Y	<5
Ca	<250	Fe	<100	La	<5	Ni	<20	Rh	<5	Ta	13	Yb	<2
Cd	<5	Ga	<5	Li	<20	Os	<5	Ru	<5	Tb	<5	Zn	<20
Ce	<2	Gd	<2										

Instructions for Use: Agilent recommends that the solution be thoroughly mixed by repeated shaking or swirling of the bottle immediately prior to use. To achieve the highest accuracy the analyst should: (1) use only pre-cleaned containers and transferware, (2) avoid pipetting directly from the CRM's original container, (3) use a minimum sub-sample size of 500 µL, (4) make dilutions using calibrated balances or certified volumetric class A flasks and pipettes, (5) dilute to volume using the same matrix as the original CRM, and (6) never pour used product back into the original container. The solution should be kept tightly capped. Store at controlled room temperature per USP 35 (10.30.60). Do not freeze, heat, or expose to direct sunlight. Minimize exposure to moisture or high humidity.

CERTIFICATE OF ANALYSIS OF REFERENCE ITEM (Contd.)

Agilent

Period of Validity: Agilent ensures the accuracy of this solution until the expiration date shown below, provided the instructions for use are followed. During the period of validity, the purchaser will be notified if this product is recalled due to any significant changes in the stability of the solution.

Sample lot approver:

Date of release: 15 September 2020
Date of expiration: 30 September 2023

Chuck Goudreau, Certifying Officer

CERTIFICATE OF ANALYSIS OF REFERENCE ITEM (Contd.)

Agilent

Hazard Information: Refer to the Safety Data Sheet (SDS), which can be obtained at www.agilent.com/chem/sds.

Homogeneity: This solution was determined to be homogeneous by procedures consistent with the requirements of ISO 17034 and ISO Guide 35. Replicate samples of the finished solution were analyzed to confirm its homogeneity, in accordance with QSP 6-13 Assessment of Homogeneity and Stability. To ensure homogeneity, users should not take a smaller sub-sample than specified in the Instructions for Use, as doing so will invalidate the certified values and uncertainties.

Further Information: Please contact Agilent for further information about this CRM.

Quality Certifications: This CRM was prepared under a quality management system that is:

- Registered to ISO 9001 – Quality Management Systems – Requirements (TUV NORD Cert. Reg. No. 44 100 16560231)
- Accredited to ISO 17034– General Requirements for the Competence of Reference Material Producers (A2LA Cert. No. 2848.02)
    - ISO 17034 references additional requirements specified in ISO Guide 31 and ISO Guide 35.
- Accredited to ISO/IEC 17025 – General Requirements for the Competence of Testing and Calibration Laboratories (A2LA Cert. No. 2848.01)
- LGC Standards, 276 Abby Road, Manchester, NH (03103)

National Good Laboratory Practice (GLP) Compliance Monitoring Authority (NGCMA)
Department of Science and Technology
GOVERNMENT OF INDIA

Certificate of GLP Compliance

This is to certify that

Intox Private Limited
375, Urawade, Tal – Mulshi
Pune-412115, Maharashtra (India)

is a GLP certified test facility in compliance with the NGCMA's Document No. GLP-101 "Terms & Conditions of NGCMA for obtaining and maintaining GLP certification by a test facility" and OECD Principles of GLP.

The test facility conducts the below-mentioned tests/ studies:

- **Physical-chemical Testing (Including Five Batch Analysis)**
- **Toxicity Studies**
- **Mutagenicity Studies**
- **Environment Toxicity Studies on Aquatic and Terrestrial Organisms**
- **Studies on Behavior in Water, Soil and Air; Bioaccumulation**
- **Residue Studies**
- **Studies on Effects on Mesocosms and Natural Ecosystems**
- **Analytical and Clinical Chemistry Testing**
- **Others**

The specific areas of expertise, test items and test systems are listed in the annexure overleaf.

Validity: March 15, 2022 – March 14, 2025

Certificate No. : GLP/C-181/2022
Issue Date : 18-06-2022

(Dr. Ekta Kapoor)
Head, NGCMA

National GLP Compliance Monitoring Authority (NGCMA)

Annexure to Certificate of GLP Compliance No. GLP/C-181/2022

Area(s) of Expertise:

- **Physical-chemical Testing (Including Five Batch Analysis)**
- **Toxicity Studies**
  - Acute Toxicity
  - Developmental and Reproductive Toxicity
  - Eye Irritation/ Serious Eye Damage (*in vivo*)
  - Guinea Pig Maximization
  - Immunogenicity
  - Inhalation Toxicity
  - Local Lymph Node Assay (LLNA)
  - Local Tolerance
  - Neurotoxicity
  - Pyrogen Test
  - Repeated Dose Toxicity
  - Skin Irritation/ Corrosion (*in vitro*)
  - Skin Irritation/ Corrosion (*in vivo*)
  - Skin Sensitization (*in vivo*)
- **Mutagenicity Studies**
  - Bacterial Reverse Mutation (AMES) Test
  - Cell Gene Mutation Test
  - Chromosomal Aberration Test (*in vitro*)
  - Chromosomal Aberration Test (*in vivo*)
  - Cytotoxicity (*in vitro*)
  - Micronucleus Test (*in vitro*)
  - Micronucleus Test (*in vivo*)
  - Mouse Lymphoma Assay (MLA)
  - MTT Assay
- **Environment Toxicity Studies on Aquatic and Terrestrial Organisms**
- **Studies on Behavior in Water, Soil and Air; Bioaccumulation**
- **Residue Studies**
- **Studies on Effects on Mesocosms and Natural Ecosystems**
- **Analytical and Clinical Chemistry Testing**
- **Others**
  - ADME Studies
  - Bioanalysis
  - Bioassays (*in vitro*)
  - Bioassays (*in vivo*)
  - Biocompatibility Studies
  - Drug Metabolism & Pharmacokinetic (DMPK)
  - Efficacy Studies
  - Hemocompatibility Studies
  - Maximum Tolerated Dose (MTD) Studies
  - Method Development
  - Method Validation
  - Safety Pharmacology
  - Toxicokinetic Studies

Test Item(s): Agrochemicals, Feed Additives, Food Additives, Industrial Chemicals, Medical Devices (*Applicable only for Bio-compatibility, not applicable for Batch Release parameters required as per MDR, 2017*), Pharmaceuticals (Human) and Pharmaceuticals (Veterinary)

Test System(s): Algae, Cell line, Chicken, Daphnia, Earthworm, Fresh Water Fish, Guinea Pig, Honey bee, Japanese Quail, Larva, Mouse, Pigeon, Rabbit, Rat and *Salmonella typhimurium*

Ekta Kapoor

(Dr. Ekta Kapoor)
Head, NGCMA