Annexure-1
Out of specification
Laboratory investigation form

OOS Investigation Report No.: OOS/2016-001.	Date of OOS occurrence: 04/07/2016

A] OOS REPORTING *(To be completed by original analyst)*

Product Name	MATRIX #6		
Test Name	Ammonia Content	Batch No.	B8216886

Summary of OOS Test Results *(state result and specification)*

- Lab analyst analyse the sample of bulk for ammonia content & found the results below norms.
  8.740 & 8.835
  Specification (9.03 - 9.98)

Analyst Name	Kuldeep Deshmukh	Signature &Date	~~KAD~~ 04/07/16

B] LABORATORY INVESTIGATION

Sr. No.	**Cross verification with reference sample (Previous approved FG batch) :** Not applicable for viscosity/density test				
1.1	Reference sample batch No: (Take about 1 month old batch wherever possible).			B8210924	
1.2	Initial results of Reference sample:			9.320	
1.3	Results of reference sample after retesting in duplicate: (Repeatability NMT **1.3%**)				
	Result-1	Result-2	Average of 1 &2	Repeatability	Formula (Max-Min) X 100 / Min
	9.237	9.350	9.294	1.22%	
1.4	Difference between initial and re-analysis results of reference sample (Reproducibility NMT **1.5%**)				
	Initial Results	re-analysis results	Reproducibility		Formula (Max-Min) X 100 / Min
	9.320	9.294	0.28%		

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1.5	Conclusion: Cross verification with reference sample found reproducible results. So there is no lab error identified.		
	Guiding rules: If reference sample re analysis result does not confirm initial results: Lab error confirmed. Identify the lab error.		
	If reference sample re analysis result confirm initial results: Proceed for resampling and reanalysis.		
2.0	**Identification of lab error.** **Check against each point. This list is only guiding. Investigation team can explore the other areas also. Additional pages can be used to describe the findings.**		
Sr. No.	Check Parameters	Observations (Yes/No/NA)	Comments
2.1	Any error in calculation?		
2.2	Any abnormality observed during testing?		
2.3	Was the method discussed with the analyst?		
2.4	Correct analytical method used?		
2.5	Analyst was trained to perform the test?		
2.6	Correct glassware used tor dilutions?		
2.7	Glassware was properly cleaned?		
2.8	Instrument used are qualified?		
	Instruments used within calibration validity period		
	Instruments Used (Name & Id) / Calibration Due		
2.9	Instrument setup & operation as per standard operating procedures.		
2.10	Correct electrode is used.		
2.11	Solution inside electrode is correct.		
2.12	Blank reading is similar as earlier.		
2.13	Any unusual trend in autotitrator graph.		

N.A.

N.A.

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2.14	Use of appropriate grade of chemicals and reagents within the validity period.				
2.15	Water used is same as specified in the method.				
2.16	Correct normality / molarity of volumetric solutions used?				
2.17	Solution used	Valid up to date	Strength		
2.19	Leakage observed in case of Buchi apparatus.				
2.20	The pipe of buchi apparatus is properly dipped in collecting solution.				
2.21	In case of viscometer, check viscosity of water using spindle M1. It should be 29.5±1 UD at 20degree.				
3.0	**Sample and standard preparation**				
3.1	Sample and standard preparation is done as per the test method,				
3.2	Is any weighing error identified?				
3.3	Is the sample properly shaken/sonicated/warmed as per test method?				
3.4	Any noticeable difference noticed between sample and standard preparation?				
3.5	Are the sample and standard stored under same environment before testing?				
3.6	Any error in transcription? e.g correct value of placebo is used.)				
4.0	**Chromatography**				
4.1	Correct column used?				
4.2	Any leakage noticed from column?				
4.3	Correct instrument parameters are used? Like flow rate, injection volume, column oven temperature , wavelength etc.				
4.4	Mobile phase preparation is done as per standard method?				
4.5	System suitability criteria met during testing?				
5.0	**Any other finding**				
6.0	**Was similar OOS reported earlier for same product?** **If yes share the identified cause, corrective and preventive actions taken at that time.**				

N.A.

N.A

N.A

N.A

N.A

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7.0	**Laboratory error identified** YES/NO		
	If yes describe the error NA		
	QC analyst (Sign and Date)	Lab Manager (Sign and Date)	
8.0	Actions to be followed (In case lab error is identified)	Yes/No	Comments
8.1	Retesting of same sample by the original analyst in duplicate.	NA	
8.2	Correction In Documents		
8.3	Any other (If any)		
	Retest result-1 NA	Retest Result-2 NA	
	Average: Repeatability :	**(Repeatability NMT1.3)**	
	Conclusion OOS Valid/ Invalid ✓		
	Analyst (sign & date)	Lab manager (sign and date)	
9.0	**Resampling** Resampling Done for same.		
10.0	**Results of retest**		
	1. 8.961	2. 8.852	
	Average: 8.906 Repeatability : 1.2%	**(Repeatability NMT1.3)**	
	QC analyst (sign and date) 04/07/16	Lab manager(sign and date) 04/07/16	
	Conclusion OOS Valid/ ~~Invalid~~ ✓		
11.0	**In case No lab and sampling error is identified,** Hypothesis/ Simulation testing like testing on alternate instrument and any other testing. (Additional pages can be used. The decision to perform Hypothesis/simulation testing depends upon the confidence level gained during lab error investigation)		
11.4	**Conclusion of simulation testing:** NA		
	Signature of lab manager: **Date:**		

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	If reason identified Retest result-1 N A	Retest Result-2
	Average: : **(Repeatability NMT1.3)** Repeatability :	
11.5	**Final remarks of Quality control head:** check for Manufacturing error. Find out the exact root cause and do accordingly.	
11.6	**OOS Valid/~~Invalid~~**	
12.	**Proceed for manufacturing investigation : ~~Yes~~/No** **Sign/date (Head Quality control)**	

Results after addition of 1.3 kg Ammonia – 9.606.

FG result:- 9.78.