

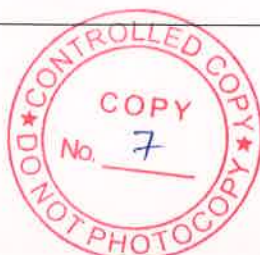
Annexure-1  
Out of specification ✓  
Laboratory investigation form

OOS Investigation Report No.: <span style="color: blue;">00S/2016-001.</span>		Date of OOS occurrence: <span style="color: blue;">04/07/2016</span>	
<b>A] OOS REPORTING</b> (To be completed by original analyst)			
Product Name	<span style="color: blue;">MATRIX #6</span>		
Test Name	<span style="color: blue;">Ammonia Content</span>	Batch No.	<span style="color: blue;">B8216886</span>
Summary of OOS Test Results (state result and specification)			
<p><span style="color: blue;">- Lab analyst analysed the sample of bulk for ammonia content &amp; found the results below norms.</span></p> <p style="text-align: center;"><span style="color: blue;">8.740 &amp; 8.835</span></p> <p style="text-align: center;"><span style="color: blue;">Specification (9.03 - 9.98)</span></p>			
Analyst Name	<span style="color: blue;">Kuldeep Deshmukh</span>	Signature & Date	<span style="color: blue;">KAD</span> <span style="color: blue;">04/07/16</span>
<b>B] LABORATORY INVESTIGATION</b>			
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).		<span style="color: blue;">B8210924</span>
1.2	Initial results of Reference sample:		<span style="color: blue;">9.320</span>
1.3	Results of reference sample after retesting in duplicate: (Repeatability NMT 1.3%)		
	Result-1	Result-2	Average of 1 & 2
	<span style="color: blue;">9.237</span>	<span style="color: blue;">9.350</span>	<span style="color: blue;">9.294</span>
	Repeatability		<span style="color: blue;">1.22%</span>
	Formula (Max-Min) X 100		
	Min		
1.4	Difference between initial and re-analysis results of reference sample (Reproducibility NMT 1.5%)		
	Initial Results	re-analysis results	Reproducibility
	<span style="color: blue;">9.320</span>	<span style="color: blue;">9.294</span>	<span style="color: blue;">0.28%</span>
	Formula (Max-Min) X 100		
	Min		



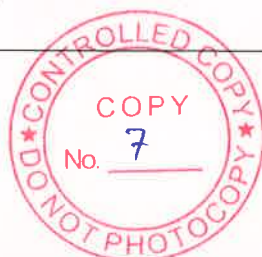
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1.5	Conclusion: <i>Cross verification with reference sample found reproducible results. So there is no lab error identified.</i>		
	Guiding rules: If reference sample re analysis result does not confirm initial results: Lab error confirmed. Identify the lab error.		
	If reference sample re analysis result confirm initial results: Proceed for resampling and reanalysis.		
2.0	<b>Identification of lab error.</b> <b>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.</b>		
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 for 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.		



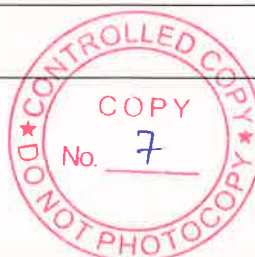
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2.14	Use of appropriate grade of chemicals and reagents within the validity period.				
2.15	Water used is same as specified in the method.				
2.16	Correct normality / molarity of volumetric solutions used?				
2.17	Solution used	Valid up to date	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 $29.5 \pm 1$ UD at 20degree.				
<b>3.0</b>	<b>Sample and standard preparation</b>				
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.)				
<b>4.0</b>	<b>Chromatography</b>				
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?				
<b>5.0</b>	<b>Any other finding</b>				
	NA				
<b>6.0</b>	<b>Was similar OOS reported earlier for same product?</b> <b>If yes share the identified cause, corrective and preventive actions taken at that time.</b>				
	NA				



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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.		
8.2	Correction In Documents		
8.3	Any other (If any)		
	Retest result-1	Retest Result-2	
	NA		
	Average: (Repeatability NMT1.3)		
	Repeatability :		
	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 NMT1.3)		
	Repeatability : 1.2%		
	QC analyst (sign and date)	Lab manager (sign and date)	
	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	Retest Result-2
	Average: : Repeatability :	(Repeatability NMT1.3)
11.5	Final remarks of Quality control head: <i>check for Manufacturing error. Find out the exact root cause and do accordingly.</i>	
11.6	OOS Valid/Invalid	
12.	Proceed for manufacturing investigation : Yes/No	
	Sign/date ( Head Quality control)	

*Results after addition of 1.2 kg Anhydrous - 9.606.  
FC result: - 9.78.*