	UGEE CHEMICALS Plant General SOP	SOP Standard Operating Procedure

VALIDATION OF MANUFACTURING & CONTROL PROCESSES

SOP #: UCL/IBDSITE/CD/Q/19.	Issuance Date:	As at Last Signature	Page 1 of 18
	Revision Date:	Maximum 2 Years from Effective Date	
	Effective date:	20 working days from issuance date.	

PURPOSE

- This SOP provides guidance to all business units for complying validation systems.

SCOPE

- This SOP applies to manufacturing, Utilities and control processes conducted in Ibadan plant but do not include validation of analytical testing methods.

RESPONSIBILITY

- **Department validation system owner** will develop a Department Validation Master Plan to track the status of required validation activities. This will be done by getting inputs from the departmental head on forecasted projects for the next fiscal.
- **QA organization(s)** will be involved in Validation planning, plan execution and analysis of validation plan results on a consultation and approval basis. Site QA leader will check the status of change control initiated before signing off validation close out report.
- **Department Managers** will prepare their department's project plans and share with all concerned and the department's validation system owner for the fiscal year. This will be done latest by December of every year, to enable finalizing of the plan (with alignment from all parties) for the New Year.
- **Validation Owner:** prepares validation protocol and sign-off prior to start of validation. He/she will also ensure close-out report is approved before product release. He also follows through on the change control raised for the validation (if applicable). He makes sure the change control is closed by completing the change control status column on the protocol before starting the actual validation and before presenting the close out report for the validation. Includes 4 eye checks done for Batch Production Records set points and centerlines in the validation reports for formulations.

POTENTIAL RISKS

- Not Applicable

PPE REQUIRED

SOP OWNER	QA APPROVAL	HS&E APPROVAL	AUTHORISATION
----------	----------NA----------	----------NA----------	----------
Anyamadu Ikechukwu			Alawode Olujide
Date: 13-09-2022	Date:	Date:	Site QA: Date: 13-09-2022

- Not Applicable

PROCEDURE

PRINCIPLES:

1 Process validation will assure that the process can meet all Formula Card, Product Specification and Process Standard requirements, not just those specified as product variables and attributes.

2 Validation, as defined in this SOP, recognizes that process capability may not be completely demonstrated until we experience the full range of possible sources of variation that occurs over time. Validation plans will assure that, when using representative materials, personnel and control strategies, the system/operation will produce repeatable results within specification and pre-defined success criteria stated in the approved protocol.

3 To demonstrate process uniformity and repeatability, the validation will require performance of operations, such as sampling, testing, monitoring, and documentation, not formally employed during routine production operations.

4 Unit operations developed on full scale test equipment/lines may be validated prior to line integration. The performance of such operations will be verified once installed on operational production lines. Validations of manufacturing processes and control and information systems in Ugee Plant will normally embody five basic elements - Installation Qualification (IQ), Operational Qualification (OQ) and Process Qualification (PQ), Initial Verification (IV), and Ongoing Verification (OV). However, a formal assessment should be done via the change control process to determine the extent of validation that needs to be done on a case by case issue.

PROCEDURE

5. The product and package specifications are determined by Client organization.
6. Starting material specifications and test methods are developed and suppliers capable of providing in-specification and representative materials from full-scale commercial equipment are qualified for use in the validation by Ugee QA Team.
7. The finished product testing methods are developed and validated by owning organization.
8. Equipment, process specifications and control strategies are developed by Ugee TSG and Process Team.
9. Personnel requirements are determined (e.g. numbers and skills) and representative teams formed and trained to operate the line during the process validation.
10. Any deviations from steps 5 to 9 will be identified and assessed as to potential risk of not allowing accurate demonstration of process capability. This will be documented and approved by the technical team and QA. Where necessary, the validation may continue with a plan in place to address deviations later in the process. This may include repeating the validation effort.

(Steps 5 to 10 are completed before validation of any processes for production of market products are begun in the plant).

11 The department Master Validation Plan will be issued every year by the validation system owner. The plan will be issued by December of every year to enable allocation of resources for each project. **(See Attachment 3). Note: Change control should be done prior to validation of any process or equipment.**

The Master Plan will include at a minimum:

- Protocol Reference number
- Description of system
- Sub-System
- Change control number
- Type of validation (e.g. Prospective Validation)
- Method of validation (e.g. IQ, OQ, PQ, IV, or OV)
- Expected completion and actual completion date for Validation Protocol
- Expected completion and actual completion date for Validation Summary report
- Person responsible for validation
- Completion Status and on time completion status
- Comments
- Review date of Validation Status
- Total validation completion percentage and on time completion percentage
- Review date of validation master plan
- Training if required for the validation
- Procedure requirement if applicable

12 Products, process qualification must be done strictly by Prospective Validation that is products will be released after process qualification report is signed off. Products Disposition during Validation – Production disposition plan should be clearly stated in the Validation protocol

13 The validation owner will write and approve appropriate protocols to cover each phase of validation (IQ, OQ, PQ, IV, and OV) for each process/operation to be validated. Approval processes will include QA review and signature among other pre-aligned functions, see attachment 4. Follow **attachment 4** as a guide to developing an IQ/OQ validation protocol and **attachment 6** as a guide to developing a PQ protocol.

The detailed protocols will at a minimum, include sections describing:

- Names of people to approve the protocol
- purpose and scope of the validation being performed
- personnel responsibilities
- detailed plan for performing the qualification exercises
- any assumptions/boundaries relative to the system or process
- samples/data to be collected and rational for sampling plan
- testing to be performed/checks to be made
- tools/materials necessary for implementation

- data collection forms
- success criteria
- methods for evaluation of data
- Product disposition

14 Conduct each phase of validation in accordance with the approved validation protocol. Changes to protocols will be reviewed and approved by the persons responsible for initial approval to ensure modifications to the plan do not render the protocol inadequate by filling the form in attachment 8.

15 Analyze the data obtained against the pre-established success criteria in the protocol. Any discrepancies between data required/success criteria and that actually obtained will be evaluated and next steps agreed upon by protocol approvers. This may require another validation in order to adequately demonstrate that the process is capable of meeting product/package specifications.

16 Write the final validation report and assemble validation file which will include the documentation and data produced. The report and data file are to be reviewed by all personnel responsible for validation approval, follow **attachment 5** as a guide to developing a validation summary report. Validation report should also be written for validations that fail. 4 eye check done for Batch Production Records set points and centerlines is to be filed as part of the close out Report documents.

17 Compiled and retain in archival storage all protocols, data and final validation reports for the lifetime of the process documented or as defined in the Plant **SOP on Record Keeping** regarding archiving of records.

18 All digital control systems integrated into manufacturing equipment such as PLC, 2D cameras, OCS check-weigher, metal detector, Case Coder, Bag coder, Pad coder should be validated as part of the IQOQ or relevant equipment

19 Digital system validation protocols should include evaluation of the following;

- Hardware and software identity and functional capability
- Electronic signature compliance
- Data integrity
- Data Security
- Error detection capability
- Simulated failure modes
- Information transfer systems completeness and accuracy
- Load capability and data volume
- Data Back-up systems

20 During the course of validation, if any change has happened which is not fully in line with agreed protocol then this change will be assessed via Control Change Process (see attachment 10) which involves the following

- Change Rationale:
- Specific Deviation vs. Approved protocol:
- Impact on final product quality:
- Action taken to eliminate risk:

Approval of the change: (all those who approved the original validation)

21 Note: New equipment/process under validation would not be used for production until the validation report has been approved by all parties (i.e. all functions that approved the original protocol). If otherwise, this has to be approved by QA with input from relevant functions.

22. Product and process validation must be completed and documented prior to product release. This requirement applies to product released both for investigational and commercial purposes.

23 Product Validation – Product Validation verifies the design/performance of a new product to meet all applicable quality attributes.

24. Process Validation - Prior to shipment, process and process control validation reports must be signed off by all the functions that signed the protocol including QA. The report should document a pre-approved written protocol, supporting data, and results showing success criteria have been met.

25. Each page of the protocol and close-out report should be numbered.

DEFINITIONS

26. Validation - Establishing documented evidence that provides a high degree of assurance that a specific system will consistently produce product meeting its pre-determined specifications and quality attributes.
27. Validation Master Plan - A document outlining policies and procedures for a company and/or facility to follow in order to assure that all products and processes are adequately validated. Such plans normally identify all process/operations at the site that require validation and provide timelines for tracking their completion.
28. Process - The term includes all the key procedures (including those performed by personnel), equipment, materials and parameters used to produce and/or test product to assure its integrity and quality. It also includes other activities that are needed to establish and/or maintain proper environmental conditions for operations to occur. These include, but are not limited to, cleaning and sanitization of equipment and facilities, production/storage of process water, compressed air, steam or other gases, and the conditioning of room air supplied to processing areas. It also includes systems/activities for the collection, processing, storage, transmission and recovery of data associated with the above activities.
29. Control System - A group of hardware components and associated software designed and assembled to perform a specific function or group of functions. Examples are programmable Logic Controllers (PLC), Distributed Control Systems and hardwired systems.
30. Information System - A group of hardware components and associated software designed and assembled to perform a specific function or group of functions. Examples are personal computers and software, mid-range computer and programs (HP 3000 and HPI).
31. Installation Qualification (IQ) - Documented verification that all key aspects of the installation of equipment adhere to approved design intentions as defined in system specifications and that manufacturers recommendations are suitably considered.
32. Operational Qualification (OQ) - Documented verification that each unit or subsystem operates as intended throughout it's anticipated or designed operating range.
33. Process Qualification (PQ) - Documented evidence of the effectiveness and reproducibility of a process in producing a product to specification.

34. Initial Verification (IV) – Validation phase used to demonstrate total system quality capability over appropriate run duration, spanning several production cycles, where the majority of non-triggered sources of variation and cumulative effects are present.
35. Ongoing Verification – Assurance that process remains in a state of control and a state of validation during manufacturing process. Phase is used to demonstrate process, material, people and systems capability of validated systems under normal manufacturing conditions
36. Protocol - A written experimental plan describing how a qualification will be conducted and containing success criteria for evaluation of results. When executed, the protocol is intended to produce documented evidence that a system or sub-system does or does not perform as designed and intended,
37. Qualification - Actions of testing equipment or process to demonstrate that it works correctly and its operation leads to the expected results. The word is sometimes inappropriately used as synonymous with the broader aspect of validation.
38. Validation Report - The formal documentation of a completed process validation plan

End Of Procedure

SOP RELATED ATTACHMENTS

Attachment 1 – Qualification Sheet
Attachment 2 – Model Answer
Attachment 3 – Site Validation Master Schedule
Attachment 4 – IQOQ Protocol Example
Attachment 5 – IQOQ Report Example
Attachment 6 – Step Up Card

ATTACHMENT 3
VALIDATION MASTERPLAN

UGEE CHEMICALS IBADABPLANT	
DEPARTMENT NAME	
DEPT OWNER	

l Validation Compliance	#DIV/0!
l Validation Completion	#DIV/0!
FY Completion	#DIV/0!
On Time Completion	#DIV/0!
Review Date	

VALIDATION MASTER PLAN

Protocol Reference Number	System	Sub-System	Change Control Number	Type of Validation- IQ,OQ, PQ,IV,OV, IOOQCSV, Spreadsheet	Method of Validation- Concurence, Prospective	Expected Completion		Actual Completion		Responsible	Completion Status	On Time/Late	Comments/Reason for Date changes	Training required	SOP required/TS update	Production Status
						Validation Protocol	Validation Report	Validation Protocol	Validation Report							

SOP OWNER	QA APPROVAL	HS&E APPROVAL	AUTHORISATION
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Date: 13-09-2022	Date:	Date:	Date: Site QA: 13/09/2022

ATTACHMENT 4

Installation Qualification/Operational Qualification Protocol
The following format and sections are to be addressed in the development of an IQ/OQ protocol.

UGEE CHEMICALS - IBADAN PLANT
INSTALLATION / OPERATIONAL QUALIFICATION PROTOCOL
<Project or Equipment Name>

Protocol No.: XXX-YY/ZZZ
Change Control No.:
Change Control Completed: Yes/ No
Validation Owner's Name: .....

DEPARTMENT: **(Name of Dept. e.g. F&HC etc.)**

LOCATION: **<State Location of Equipment>**

SYSTEM[S]: **<Description of System to be validated>**

Protocol prepared by: <Protocol Originator> Date: ZZZZZ

Completion of the following signature block signifies the pre-approval of this protocol stating the type and number of validation trials required and the conceptual methodology by which the validation trials will be conducted and evaluated.

	NAME	SIGNATURE	DATE
Validation (F&HC)			
Safety			
Engineering			
Making Department (F&HC)			
Quality Assurance			

SOP OWNER	QA APPROVAL	HS&E APPROVAL	AUTHORISATION
---------- Anyamadu Ikechukwu Date: 13-09-2022	----------NA-------- Date:	----------NA---------- Date:	---------- Alawode Olujide Site QA: Date: 13/09/2022

TOPIC PAGE NO.

* The items on the protocol table of content are not limited to these but a function of the complexity of the system to be validated*

A. OBJECTIVE
State the objective(s) of the equipment qualification.

B. SYSTEM DESCRIPTION
Provide a description of the purpose and method of operation for the system being qualified.

C. TEST ASSUMPTIONS / BOUNDARIES
Provide a brief narrative to describe base assumptions associated with the qualification. Clearly state the boundaries of the system and the operating conditions, which the qualification will evaluate.

INSTALLATION QUALIFICATION (IQ)

Installation Qualification involves establishing documented evidence that process equipment and ancillary systems equipment are constructed and installed to meet design intent. IQ does not typically include operation of the equipment. IQ may only need to be performed once after initial installation. Thereafter, IQ may not be required for each different process validation. All or parts of IQ may need to be repeated following relocation of equipment or major repairs, renovations or modifications to the equipment.

D. EQUIPMENT LIST & DESCRIPTION
List all major equipment and provide a brief description of the function/construction of each.

E. ENGINEERING SPECIFICATIONS

1. Drawing List - Provide a list of the approved drawings which are pertinent to the system including flow diagrams, equipment layout, P&ID's, etc.
2. Instrument List - List all key instruments that are associated with the system, using manufacturer's name and description. Including equipment numbers from the drawings will be helpful. Instruments can be broken into two categories - critical, and non-critical. If critical instruments (i.e., instruments requiring calibration) are listed in the Calibration section, they need not be listed in this section.
3. Utility Requirements - Discuss here utility requirements including compressed air, treated water, potable water, steam, etc. Hookups to these systems should be verified as part of the test plans.

F. INSTALLATION QUALIFICATION TEST PLAN WITH SUCCESS CRITERIA

Detail here the test plan to verify that the system is constructed and installed according to the design. The test plan should provide sufficient instruction to assure that the task is carried out correctly. Where other systems or procedures are relied upon to provide instruction on how to do a task (lubrication SOP, calibration SOP, PSI Procedure, yellow lining, green tagging, etc.) that document or procedure must be attached to the protocol. SOP's may be referenced with SOP # and Revision # instead of attaching.

G. PREVENTIVE MAINTENANCE PROCEDURES

Detail here the plans for developing preventive maintenance procedures for all new equipment along with plans for adding this equipment to any existing maintenance tracking systems. Specify which lubricants/sealants to use in applications where the potential for product contact exists.

Develop a list of instruments that require calibration and discuss plans for initial (pre-startup) calibration. Also, discuss plans for assuring that ongoing calibrations are carried out.

I. INITIAL CLEANING PROCEDURES

Detail what cleaning must be performed to prepare the equipment for initial production. Initial cleaning must remove any residual oil, grease, welding residue, metal shavings, etc. that accumulate during the equipment manufacturing and facility construction. Completion of initial cleaning steps must be clearly documented and included in the IQ/OQ report or referenced appropriately. This section does not apply for qualification or requalification of existing equipment.

OPERATIONAL QUALIFICATION (OQ)

Operational Qualification involves establishing documented evidence that process equipment and ancillary systems equipment operate as intended throughout anticipated operational ranges. This activity requires evaluation of equipment under dynamic operational conditions, which may include no-load or loaded conditions at centerline conditions and may also include operation outside of the normal operating range envisioned for any particular production process. OQ may not need to be repeated for each new product/process, if each new product/process is conducted within originally qualified operational ranges. Major modifications or repairs or use of the equipment in processes where operational parameters are outside those originally investigated will require re-qualification.

J. CRITICAL PARAMETERS

List operating parameters that are judged to be critical to the process as a result of pilot plant work, vendor information, or technical judgment. Also include any parameters that are critical to proper functioning of the process or equipment. The critical parameters listed here should be verified via test plans (see Section "N").

K. TEST INSTRUMENTS

Detail which test instruments will be used to execute the verifications in the test plan. Reference the instruments by name and number and either provide proof of calibration or state that the instrument is calibrated and reference the location of the calibration records.

L. OPERATIONAL QUALIFICATION TEST PLAN WITH SUCCESS CRITERIA

Detail here the test plan to verify that the system operates according to the design. The test plan should provide sufficient instruction to assure that the task is carried out correctly. The test plan must specify acceptance criteria for each critical parameter.

M. STANDARD OPERATING PROCEDURES FOR PROCESS

Verify that appropriate standard operating procedures for this operation are developed, clearly written, and approved through appropriate channels

N. ONGOING CLEANING PROCEDURES

Similar to the SOP verification, verify that appropriate standard operating procedures for cleaning are developed, clearly written, and approved through appropriate channels. In addition to this, a separate validation of the effectiveness of the cleaning must be carried out following the cleaning and sanitization validation SOP.

O. RESPONSIBILITIES

In this section, list the individuals by names who are responsible for conducting each element of the Equipment Qualification

P. PRODUCT DISPOSITION

Indicate here what happens to any finished product produced during the qualification.

Installation Qualification/Operational Qualification Report
The following sections are to be addressed in the development of an IQ/OQ Report.

INSTALLATION QUALIFICATION/OPERATIONAL QUALIFICATION REPORT

<<Project or Equipment Name>>
Validation Protocol Number: XXX-YY/ZZZ
Change Control No:
Protocol prepared by: <Protocol Originator> Date: ZZZZZ

A CONCLUSIONS

State here the conclusion (system qualified or system not qualified) and provide the reasoning for the conclusion.

Example:

The _____ system is qualified. Based on the attached completed protocol and this report, the process equipment and associated critical process equipment and associated critical subsystems have been shown to be installed and operating within design specifications.

B. DISCUSSION

1. Compliance within the Installation/Operational Qualification Protocol
   Make reference to the protocol and indicate whether it was followed completely or not. Clearly explain and document any deviations from the protocol and their impact on the capability of the system.
2. Results versus Success Criteria
   Provide a brief summary of the results vs. the success criteria. The intent is to provide a brief summary of all the raw data to support the conclusions. This can be done in paragraph format or tables. Proper consideration should be given on how to best provide the summary since this information will be referred to in the future as part of change-control. In addition, this section should be used to discuss any result(s) that may not be obvious in the test plan(s).

   Example:

   All protocol test plans were completed and all other protocol requirements fulfilled as illustrated by Attachment "XYZ" pages 1-12. All of the success criteria were met with the exception that the Pot turbine mixers showed a high degree of vibration during the Operational Qualification. Since these mixers are not required for the current formulations, they have been removed from the tank and the penetration has been sealed.
3. Attach the completed IQ/OQ test plans with documented signoffs. This section may also be used to discuss any results that may not be obvious in the test plans. Where appropriate, use of summary tables to compare results to acceptance criteria is recommended in addition to the raw data. The following completed protocol sections must be attached or discussed in detail here

SOP OWNER	QA APPROVAL	HS&E APPROVAL	AUTHORISATION
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Date: 13-09-2022	Date:	Date:	Site QA: Date: 13/09/2022

- Installation Qualification Test Plan with Success Criteria
- Operational Qualification Test Plan with Success Criteria
- Standard Operating Procedures for Process
- Ongoing Cleaning Procedures
- Calibrations
- Initial Cleaning Procedures
- Test Instruments Used to Conduct Tests
- Preventive Maintenance Procedures

C. RECOMMENDATIONS
Use this section to recommend acceptance of equipment or system as qualified and/or discuss any follow-up items from the IQ/OQ.

D. SIGNATURES
Completion of the following signature block signifies that the appropriate parties have reviewed the data generated during the validation testing and agree that the intent of the protocol and the standards reflected in the acceptance criteria have been met.

* Signatories indicated above are not definite, at a minimum QA, Validation Owner and Operating department manager needs to approve a protocol.

STEP UP CARD FOR VALIDATION OF MANUFACTURING & CONTROL PROCESSES

Trainee: _____ Skill Owner_____

Role: _____ Qualifier: _____

S/N	Skill Block/Skill		Knowledge/Task/Skill	Target Profic.	Self Evaluation Date	Self Evaluation	First Evaluation Final Evaluation Date	First Evaluation Final Evaluation	Date	Evaluation
1	Shelf Quality Measures	1	Understand the purpose of the SOP	3		1 2 3 4 5		1 2 3 4 5		1 2 3 4 5
		2	Can explain the content of the validation masterplan	3		1 2 3 4 5		1 2 3 4 5		1 2 3 4 5
		3	Can explain the start-up work process plan	3		1 2 3 4 5		1 2 3 4 5		1 2 3 4 5
		4	knows how to prepare a standard validation protocol	3		1 2 3 4 5		1 2 3 4 5		1 2 3 4 5
		5	Knows how to prepare a standard close-out report	3		1 2 3 4 5		1 2 3 4 5		1 2 3 4 5
		6	Can explain the diffrence beween the different types of validation and when each is needed	3		1 2 3 4 5		1 2 3 4 5		1 2 3 4 5

Signature of Trainee _____ Signature of Qualifier _____

Date of Qualification _____ Date of Qualification _____

SOP OWNER	QA APPROVAL	HS&E APPROVAL	AUTHORISATION
Anyamadu Ikechukwu	----------NA----------	----------NA----------	Alawode Olujide
Date: 13-09-2022	Date:	Date:	Site QA: Date: 13/09/2022