

Interlaboratory Round-Robin Design for Vat-Photopolymer Materials

Specimen state, protocol transfer and reproducibility statistics

Juan Seguro

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Correspondence: info@3Dresyns.com · www.3Dresyns.com

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Abstract

An interlaboratory study can estimate how a material or method behaves when transferred beyond one laboratory, but it can also confound resin state, machine, operator, post-processing and measurement. Vat-photopolymer systems are unusually sensitive to this problem because the test article is created through a coupled chain of liquid-material handling, optical exposure, layerwise manufacture, washing, drying, secondary cure, conditioning and measurement. A nominally shared resin and CAD file do not establish a common measurand.

This review develops a round-robin architecture for vat-photopolymer materials. It separates method-precision studies from manufacturing-capability comparisons, defines specimen-state controls, compares centralised and distributed operations, and connects protocol transfer to repeatability and reproducibility statistics. The design uses a pilot phase, locked data definitions, process-control records, blind duplicates and hierarchical analysis. Direct evidence from an interlaboratory working-curve study demonstrates how large dispersion can arise when procedure, light engine and measurement are insufficiently harmonised. The central conclusion is that a useful round robin must be designed around a declared decision and variance structure. More laboratories, specimens or measurements do not repair an ambiguous protocol, and a reproducibility standard deviation is not a universal material property.

Keywords: interlaboratory study; round robin; vat photopolymerisation; reproducibility; repeatability; protocol transfer; specimen state; variance components; pilot study; qualification

1. Significance and scope

Single-laboratory precision can conceal systematic differences in radiometry, printer firmware, environmental control, washing, post-cure, conditioning and data reduction. An interlaboratory study deliberately exposes some of those differences. Its value depends on whether the exposed variation matches the intended use.

A study may seek the precision of a measurement method, the transferability of a manufacturing procedure, equivalence among suppliers, or the range of finished-part performance. These are different questions. The controlled and intentionally variable factors must be selected accordingly.

1.1 Corpus boundary

ER-209 owns design and interpretation of interlaboratory round-robin studies for vat-photopolymer materials. ER-005 owns reproducibility limits of working-curve characterisation in vat photopolymerisation. ER-203 owns radiometric and actinometric traceability for photopolymer processing. ER-210 owns reference artefacts and benchmark geometries for additive-manufacturing qualification. ER-344 owns machine-capability studies for additive manufacturing. ER-297 owns calibration management for photopolymer production and testing. ER-209 uses outputs from those subjects but does not replace their method-specific qualification.

2. Define the study question and measurand

The protocol should state one primary decision, such as estimating reproducibility of a test method, comparing execution of a fixed manufacturing plan, or assessing finished-part variability across qualified suppliers. The measurand must be defined with unit, specimen state, calculation and reporting precision.

A round robin that asks several laboratories to print tensile bars and report strength contains at least two processes: manufacture and mechanical testing. If both vary locally, the final dispersion cannot be assigned uniquely to either. The design should centralise one process, nest it explicitly, or accept that the result represents the complete distributed chain.

3. Round-robin classes

Three useful classes are:

- 1 **Measurement-method precision:** common stable specimens are circulated or distributed; laboratories apply the same measurement method.
- 2 **Manufacturing-protocol transfer:** common feedstock and digital definition are supplied; laboratories manufacture specimens under a fixed plan; testing is preferably centralised.
- 3 **End-to-end supplier reproducibility:** each participant executes its normal qualified workflow; the reported dispersion represents delivered-part performance.

Combining these classes without an explicit factorial or nested design produces an uninterpretable variance estimate. NIST guidance for additive-manufacturing round robins treats the manufacturing plan and complete process chain as central design objects [1].

4. Specimen state is part of the measurand

For vat-photopolymer materials, record resin identity and lot; storage and mixing; open-vat age; printer and light engine; wavelength and radiometric state; build orientation and location; support strategy; wash solvent, time and agitation; drying; post-cure spectrum, radiant exposure and temperature; time since post-cure; and conditioning environment.

The protocol should define the instant at which a specimen becomes eligible for measurement. Shipping can add time, temperature, humidity, light and mechanical exposure. If properties evolve, a central calendar date does not create a common age. Use elapsed time from a defined state transition and specify permitted windows.

5. Common material and commutability

Distributing aliquots from one homogenised lot reduces formulation variation but does not prove that the aliquots remain equivalent. The coordinator should define mixing, fill level, container, headspace, light protection, temperature, transport and receipt checks. Retain witness material for stability assessment.

A reference material or specimen must be commutable: it should respond to the tested method in a way that represents routine materials. An unusually transparent, rigid or stable resin may make transfer look better than it is. Where one material cannot span the intended domain, use predeclared levels rather than treating materials as interchangeable replicates.

6. Protocol transfer and the manufacturing plan

The protocol has two layers. A normative layer states what must be achieved and recorded. A local implementation layer records how each laboratory's equipment realises those requirements. Machine-specific settings should not be forced into nominal equality when their physical outputs differ.

The manufacturing plan should cover digital-file version, slicing, orientation, build position, machine condition, material handling, exposure, separation, washing, drying, post-cure, conditioning, identification, packaging and deviations. Moylan and co-workers emphasised that AM round-robin plans must extend beyond machine-processing parameters and that a pilot exposes ambiguity before the full study [1].

7. Pilot study and protocol freeze

A pilot should include at least the coordinator and representative laboratories with different equipment or experience. Its purpose is to test instructions, file compatibility, sample logistics, measurement resolution, data fields and the ability to execute controls. Pilot data should not be silently pooled with the main study after the procedure changes.

After pilot correction, freeze the protocol, data dictionary, analysis plan and allowed deviations. Late changes require version control and an impact decision. A laboratory that cannot execute a critical step should be excluded or assigned to a declared alternative stratum rather than given an undocumented exception.

8. Replication, randomisation and blinding

Replicates should cover the variance components required by the decision. Multiple measurements on one specimen estimate measurement repeatability, not build-to-build or laboratory-to-laboratory variation. Multiple specimens in one build do not necessarily represent independent builds.

Randomise specimen positions and test order where feasible. Use blind duplicates to detect data or identification problems. Laboratories should not see other participants' results before submitting locked data. The coordinator should predefine whether technical failures are repeated and how replacement specimens are labelled.

9. Statistical model

For a balanced design with laboratory i , build j , specimen k and repeated measurement l , a nested random-effects model can be written as:

$$y_{ijkl} = \mu + L_i + B_j(i) + S_{k(ij)} + \varepsilon_{l(ijk)}$$

with variance:

$$\sigma^2 = \sigma_L^2 + \sigma_B^2 + \sigma_S^2 + \sigma_r^2$$

The repeatability component σ_r describes variation under repeatability conditions. A reproducibility standard deviation may combine laboratory and repeatability components, but its exact definition must match the selected standard and design. ASTM E691 and ISO 5725-2 provide established frameworks for interlaboratory precision experiments [2,3]. Their controlled texts should be applied directly when conformity to those standards is claimed.

10. Balanced design, missing data and heterogeneity

A balanced uniform-level design is efficient and supports standard precision calculations. Vat-photopolymer studies may become unbalanced through failed builds, shipping damage or incompatible equipment. The analysis plan should state whether mixed-effects estimation will be used and how incomplete cells are handled.

Variance can change across property level, specimen geometry or wavelength. Transformations or level-specific estimates may be necessary. A single pooled coefficient of variation can hide that the method is stable in one range and unusable in another.

11. Outliers and protocol deviations

An extreme result may reveal a real transfer failure, not a disposable statistical outlier. First verify identity, units, transcription, calibration, raw data and compliance. Classify deviations before examining their effect. Report analyses with and without excluded results when an exclusion materially changes the conclusion.

The direct interlaboratory study of vat-photopolymer working curves by Kolibaba and co-workers used one resin batch across 24 participants and still found very large dispersion in fitted parameters, attributed to procedural variation, light engines, model uncertainty and measurement capability [4]. The lesson is not a transferable numerical factor. It is that common material alone does not create a common method.

11.1 Measurement capability and data resolution

The measurement system must resolve the expected specimen variation. Report instrument model, calibration status, resolution, analysis software and operator. If thickness is reported to a coarse increment while fitted parameters are sensitive to thickness, additional specimens cannot recover the lost information.

Centralised testing reduces one source of between-laboratory variability but also changes the claim: the study then estimates distributed manufacture with common measurement. Distributed testing estimates the whole chain unless a separate measurement study quantifies laboratory effects.

12. Interpretation matrix

Observation	Supported interpretation	Unsupported leap
Within-laboratory variation is small	The local repeated workflow is precise under the study conditions	Results are comparable between laboratories
Between-laboratory variance dominates	Transfer or laboratory-specific factors contribute materially	The printer model alone causes the difference
A common analysis script reduces dispersion	Data reduction was one contributor	Manufacturing is now reproducible
One laboratory is extreme	Its result requires technical and procedural investigation	The result may be deleted automatically
A reproducibility estimate is obtained	Precision is quantified for the stated method, levels and population	A universal material-property uncertainty is known

13. Minimum reporting set

Report objective and decision rule; study class; measurand; participating-laboratory selection; material and aliquot controls; digital files and hashes; protocol version; pilot findings; equipment and software; manufacture, post-processing and conditioning; randomisation and blinding; replicate hierarchy; raw-data schema; deviations and missing data; statistical model and assumptions; repeatability and reproducibility estimates with uncertainty; outlier treatment; sensitivity analyses; and the population and property range to which conclusions apply. ASTM F2971 provides an official reporting framework for test specimens prepared by additive manufacturing [5].

14. Knowledge boundary

The available direct multi-laboratory evidence for vat photopolymerisation is concentrated on working-curve characterisation and does not yet establish reproducibility distributions for the full range of mechanical, dimensional, optical or ageing measurements. The literature also does not isolate every contribution from material state, light engine, manufacture, post-processing and measurement across a common broad design. Consequently, the variance architecture in this review is a study-design framework, not a claim that component magnitudes or rankings are already known for other VPP measurands.

15. Limitations

A round robin is a top-down experiment. It can demonstrate transfer variability without uniquely identifying every cause. Small numbers of laboratories produce uncertain between-laboratory estimates. Volunteers may not represent production sites. Shared material can degrade during distribution, and rapidly changing hardware or software can make a frozen protocol obsolete. Reproducibility established for one resin, geometry, property level and post-cure state must not be generalised without evidence.

16. Conclusions

Interlaboratory evidence is valuable only when the study design matches the intended decision. For vat-photopolymer materials, specimen state and the complete manufacturing plan are part of the measurand. A pilot, frozen protocol, process-control record, deliberate replication and hierarchical analysis prevent avoidable ambiguity. Repeatability, build variation, laboratory effects and measurement error should remain visible. The final precision statement belongs to the declared procedure and population, not to the resin name alone.

References

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