Equipment, calibration and maintenance

The instrument register, the equipment every result must name, qualification and calibration, the checks in every run, logbooks, and what happens when an instrument fails.

Not yet approved
No approval of this revision is on file. This revision states what the laboratory's results system enforces today; it takes effect when a named reviewer approves it.

Document		Laboratory	
Number	SOP-09	Name	Cupel Laboratories
Revision	1, first issue	Address	2599 Speakman Dr Mississauga, ON L5K 1B1
Status	Not yet approved	Country	Canada
Effective	Not yet in effect; takes effect when approved	Not on file	Not yet published: legal entity, postal address, telephone and country
Applies to	Every instrument behind a result	Web	cupel.ca
Supersedes	Nothing; first issue	Email	help@cupel.ca
Fingerprint	7ff4 3b93 d094 0914		

1 Purpose

To keep every instrument that produces a reported result identified, qualified, calibrated, maintained and fit for use, and to tie every result to the instrument, the column, the data file and the time that produced it.

2 Scope

Every instrument, device and piece of software whose output reaches a certificate, and the storage that holds samples and standards.

Instrument	Used for	Listed
UHPLC with UV or diode-array detection	Purity by RP-HPLC–UV (SOP-02); Mass per vial (SOP-06)	Not yet
LC–MS with electrospray source	Identity by LC–MS (SOP-03)	Not yet
Kinetic incubating microplate reader, 405 nm	Bacterial endotoxin (SOP-04)	Not yet
Coulometric Karl Fischer titrator	Water content (SOP-05)	Not yet
Analytical balance	Reconstitution, whole-vial masses, standards (SOP-02, SOP-05, SOP-06)	Not yet
Pipettes and volumetric glassware	Reconstitution and dilution (SOP-02, SOP-04, SOP-06)	Not yet
Refrigerator and freezer	Samples, retained vials, standards (SOP-01)	Not yet

3 Responsibilities

Role	Responsibility
Analyst	Checks an instrument before use, runs the checks each method requires, records the instrument, column, data file and time in the set, and takes an instrument out of service when it fails.
Reviewer	Confirms before signing that the record names the equipment and that its checks passed (SOP-07).
Management	Acquires, installs and qualifies each instrument, keeps its calibration current, and lists it once its serial plate can be photographed.

Named: Timothy Ian Benedicto, Medical Laboratory Technician; Roxanne Manlapaz, Laboratory Technician.

4 References and controlled sources

Standards and other documents

- USP <1058> Analytical Instrument Qualification: design, installation, operational and performance qualification.
- USP <41> Balances and USP <1251> Weighing on an Analytical Balance; ISO 8655, piston-operated volumetric apparatus.
- The methods' own checks: USP <621> (SOP-02, SOP-06), USP <85>; Ph. Eur. 2.6.14 Method D (SOP-04), USP <921> Method Ic; Ph. Eur. 2.5.32 (SOP-05).
- SOP-08 (an instrument found faulty after use).

Controlled sources, read at every build

- data.json instruments (the register below) and method.rows.
- lab/results.py: the checks of every run and the issue rules that tie a result to its equipment.
- lab/entry_server.py: the equipment block of each test section.
- make.py: the suites that verify the software.

5 The instrument register

No instrument is listed yet: make, model and serial number are published on the methods page and on each certificate once the instrument is installed and its serial plate can be photographed, and never for an instrument the laboratory does not own.

Each entry, once listed, carries: the laboratory's identifier (the one every result set names), make, model and serial number, location, software and version, the date of qualification, the calibration due date, and whether it is in service. The register is published on the methods page and the identifier on every certificate.

6 Every result names its equipment

The result must be traceable to the equipment that produced it. The instrument id is required to issue; the data file and the acquisition time are required to issue for every section that shows a figure (MS, LAL and quantitation; per injection for HPLC).

- For every test section: the instrument identifier, its make and model, and the software, recorded in the set; the identifier is required to issue.
- For HPLC and mass per vial: the column, with its serial number or lot.
- For every figure: the data file and the acquisition time (UTC); for HPLC, per injection. Water content shows no figure, so its data file is recorded and no acquisition time is required.
- The certificate prints all of it under Method parameters, so a result can be traced to the instrument and the run that produced it.

7 Qualification and calibration

Requirements, and the status of each at this revision, read from the register.

Requirement	Status
Each instrument is installed, and its operation qualified against the manufacturer's specification and the method's needs, before it produces a reported result (USP <1058>).	Not performed: no instrument is installed.
The balance is calibrated with weights traceable to national standards, its minimum weight is established (USP <41>), and a check weight is weighed on each day of use.	No calibration on file.
Pipettes are calibrated gravimetrically (ISO 8655) at set intervals.	No calibration on file.

Qualification and calibration, continued

Thermometers and the storage units are checked against a calibrated reference, and storage temperatures are recorded.	No check or temperature record on file.
The mass spectrometer's mass calibration is run as its software prescribes before each sequence; resolving power is recorded with every run (SOP-03).	Recorded per run once installed.
Calibration intervals are set from the manufacturer's recommendation and the instrument's history.	Not yet set.

8 Checks in every run

What the result model checks on every run, with the criterion it applies; a missed check is printed on the certificate or refuses the result.

Instrument	Check	Criterion
UHPLC	Symmetry factor of the standard	0.8–1.8
UHPLC	Resolution of the named pair	At least 1.5
UHPLC	Area RSD of the standard injections	Not more than 2.0%
LC–MS	Criterion against the instrument class	At least 50 ppm on a unit-resolution class
LC–MS	Resolving power	A warning below 10 000 with a criterion under 50 ppm
Plate reader	Standard curve	|r| at or above 0.980
Plate reader	Positive product control	Recovery 50–200%
Titrator	Drift before titration	At or below 20 µg/min
Titrator	Water standard recovery	97.5–102.5%
UHPLC	Calibration for mass per vial	r^2 at or above 0.999

9 Maintenance, logbooks and faults

- Each instrument has a logbook: use, maintenance, faults, repairs, qualification and calibration, each dated and signed by name. No logbook exists yet, because no instrument is installed.
- Maintenance follows the manufacturer's schedule; the schedule is copied into the logbook when the instrument is installed.

Out of service

9.1 An instrument that fails a check, is damaged, is overdue for calibration or gives doubtful results is taken out of service at once and labelled so.

9.2 It produces no reported result until it is repaired and re-checked, and the re-check is recorded in its logbook.

9.3 The reports it produced since its last good check are reviewed under SOP-08. Every result set names its instrument identifier, so they can be listed.

9.4 Open a case in the corrective and preventive actions log (/capa) with the source "nonconforming work" and the instrument identifier as its reference; the repair, the re-check and the review of its reports are the case's action and verification.

10 Software

- The data system or acquisition software of each instrument is named, with its version, in every result set (the instrument's software field) and on the certificate.
- The results backend, the result model and the register are verified by their test suites, which python make.py check runs (register, test_analytes_reference, test_cds_import, test_e2e, test_emails, test_launch_facts, test_launch_preview, test_management_review, test_metrics, test_orders, test_qc_trend, test_report_api, test_results, test_safety, test_sop, test_tracking, test_validation, test_vendor_kit, test_worksheets, selftest, build, integrity, integrity_selftest, backup_selftest). A change to the model changes the fingerprint of every procedure that prints it.

Software, continued

– Every time the backend writes is the server's UTC clock, and acquisition times come from each instrument's data system; how those clocks are kept synchronised is not yet described.

Daily and interim checks

– Each instrument's daily check is recorded on WS-02 (daily checks) before the first measurement of the day: the balance's check weight near the vial load with the room's temperature and humidity, the ionizer and draft shield; the titrator's drift readings and standard recovery; the plate reader's set-point and displayed block temperature; the mass spectrometer's calibrant, residual error and resolving power; the chromatograph's pressure at equilibration; the refrigerator and freezer temperatures; the hood's flow monitor.

– The plate reader's block temperature is read against a calibrated thermometer monthly; each pipette used for a reported result has a monthly gravimetric check at its working volume (ten weighings of water; systematic error at most 0.8% and random at most 0.3%, or 1.0% and 0.5% at 100 µL) recorded on its card, and a failed check takes it out of service.

– The vial-format table is a controlled record of this procedure: the brimful capacity of each vial format received (weighed brimful with water once), its closure finish, whether it fits the oven vial whole, is re-crimped or is sampled as a weighed portion (SOP-05, SOP-06); the oven sampler's operational qualification (USP <1058>) covers every format in it. No format is on file yet.

11 Columns, standards and reagents

– Each column is identified by its serial number or lot in every run that uses it (C18, 2.1 × 100 mm, 1.7 µm), and has a log sheet: date installed, injections, the pressure at equilibration of every run, the storage solvent, and the reason it was retired.

– Reference standards, the control standard endotoxin, the water standard, the lysate, the medium and the anolyte are recorded with their lot (and, for standards, their content or potency, its source and its uncertainty) in every result set that uses them, and printed on the certificate.

– Each is stored as its supplier's certificate says and is not used past its expiry date; every worksheet carries an "expiry checked" tick beside each lot until the backend's form takes the expiry fields the model carries.

– Suppliers of standards, the control standard endotoxin, the lysate and the medium are evaluated once before first purchase and at each new lot, on a record of this procedure cited against the lot (REFERENCE_STANDARDS 4.1): the supplier's legal identity and whether it manufactures or resells; its certification or accreditation (ISO 9001, ISO 17034 or ISO/IEC 17025 with its scope), verified on the certification body's own register and never on the supplier's page; the lot certificate's content (identity, purity with its conditions, net peptide content with its method, water, the counter-ion named with its share, residual solvents, and U with its k); the agreement of the certificate with the laboratory's own identity, purity and water results (a disagreement in purity above 1% area or in water above 1% w/w downgrades the supplier); agreement between two lots or two suppliers where the laboratory has it; the supply facts without any price; and the exclusion of a supplier whose public pages make claims about the laboratory's results or who asks that results be shared or shaped (QM-01). A supplier with no certificate for the specific lot, or whose certificate carries no method for the content, is not approved.

– The approval states are approved (a certificate with its method and its U; the laboratory's tests agree), conditional (a certificate with its method and no U; the model's default uncertainty applies to every result on the lot) and not approved. A provider of a subcontracted analysis (quantitative NMR, amino-acid analysis, residual solvents, elemental or ion analysis) is evaluated the same way with its ISO/IEC 17025 accreditation, the scope line that names the test, the reference materials its method rests on, and a written confidentiality undertaking; a term of an uncertainty budget is taken only from a certificate or a provider's report that states U with its k. The approved-supplier list is a quality record of this procedure, reviewed at the management review (QM-00). No supplier or provider is on file yet (data.json standards.items is empty).

Columns, standards and reagents, continued

- Storage of standards and reagents: a reference standard at −20 °C in the dark, desiccated, in a freezer on the equipment register with its temperature log; an excursion above −10 °C for more than 24 h, a freezer failure or a lot's re-qualification due date passed is recorded against the lot and re-opens its assignment (SOP-06, Reference standard); the desiccator's indicator state and the relative humidity of the dry enclosure used for aliquoting are recorded on the worksheet each time they are used.

12 Deviations and open points

- A departure from this procedure, planned or found afterwards, is nonconforming work and is handled under SOP-08. One that bears on a result is stated on the certificate, in the open point or the notes of the section concerned, never left out.
- No instrument is listed yet: make, model and serial number are published on the methods page and on each certificate once the instrument is installed and its serial plate can be photographed, and never for an instrument the laboratory does not own.
- No qualification, calibration, logbook or maintenance record exists, and no calibration interval is set.
- The backend does not record expiry dates of standards and reagents, or storage temperatures; until it does, they are checked by hand before use.
- No validation report exists for the backend and the result model as a computerised system; their test suites are the evidence on file.

13 Records

- WS-02 (daily checks): the record of each day's checks before the first measurement (the balance and its check weight, the room, the pipettes, the storage units, the hood, each instrument's daily check), a record per day rather than per set, filed by date (SOP-07, Records and retention).
- The instrument register in data.json, published on the methods page.
- Faults and their corrective actions, in the corrective and preventive actions log (lab/records/capa.jsonl).
- The equipment block of every result set (instrument, column, data file, acquisition time), printed on every certificate.
- Logbooks, qualification and calibration certificates, once they exist, kept for the life of the instrument and never discarded while a report it produced is in the register.

14 Revision history

Revision	Change	Approval
1	First issue.	Not yet approved

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Current revisions

End of document