Mass per vial by external standard

Mass of peptide per vial against a reference standard of the named peptide, stated as free peptide or as the salt according to the standard's basis, and closed against the whole-vial fill by mass balance.

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-06	Name	Cupel Laboratories
Revision	1, first issue	Address	2599 Speakman Dr Mississauga, ON L5K 1B1
Status	Not yet approved		
Effective	Not yet in effect; takes effect when approved	Country	Canada
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Applies to	Test codes QNT, FULL		
Supersedes	Nothing; first issue	Web	cupel.ca
Fingerprint	1d02 ed55 9105 1c56	Email	help@cupel.ca

1 Purpose

To determine the mass of peptide in one vial as received, by reversed-phase HPLC with UV detection at 214 nm against an external reference standard of the named peptide, and to state what that mass is the mass of.

2 Scope

An add-on (code QNT, "Mass per vial") to IDP; the full panel includes it. Minimum sample, as the intake conditions state it: "three sealed vials as sold". It rides on identity and purity because it quantifies against a reference standard of the named peptide and is reported beside them.

3 Responsibilities

Role	Responsibility
Analyst	Weighs and reconstitutes the vials, prepares the calibration levels from the standard, runs them with the samples, records the standard's identity and content, and signs as analyst.
Reviewer	Checks the standard's content basis, the calibration, the range and the mass balance; signs the content as reviewed (SOP-07).

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

4 References and controlled sources

Standards and other documents

- USP <621> Chromatography: external-standard quantitation, and system suitability as SOP-02.
- JCGM 100:2008 (GUM) 6.2 and Annex G; ISO/IEC 17025:2017 7.6 and 7.8.6.
- SOP-02 (the chromatography and the suitability run), SOP-05 (the whole-vial gross fill and water), SOP-07, SOP-09.

References and controlled sources, continued

Controlled sources, read at every build

- lab/results.py: the calibration, the content correction, the basis labels, the counter-ion stoichiometry; lab/CONTRACT.md section 1 (qnt) and the mass-balance note.
- data.json method.rows, pricing and intake.

5 Safety

- Read the current safety data sheet of every reagent before first use and keep it where the work is done. Wear a laboratory coat, safety glasses and chemical-resistant gloves for every step below.
- Treat every sample as a potent, biologically active substance of unconfirmed identity until its report says otherwise: open and weigh lyophilised powder so that it is not inhaled, and keep it off bare skin.
- Trifluoroacetic acid (in mobile phases A and B) is corrosive in its neat form and its vapour is harmful: dispense the neat acid with eye and hand protection, with ventilation that keeps the vapour from the face.
- Acetonitrile is highly flammable and toxic by inhalation, ingestion and skin contact: keep it from ignition sources and keep reservoirs capped.
- The reference standard's weighing is the one step in the laboratory where a potent peptide powder is open on a pan: in the ventilated balance enclosure, double nitrile gloves, within 30 s of opening; no food, drink or contact-lens handling in the laboratory; a powder spill is wet-wiped (water, then 70% ethanol), never swept.
- The decrimper cuts and pinches; venting is done under the hood; a vial broken during the rinsing for W2 goes to the sharps container; a stock of Semaglutide, tirzepatide and retatrutide diluted into mobile phase A can foam, so cap before inverting.
- Collect solvent and reagent waste in closed, labelled containers for disposal as hazardous chemical waste; never pour it to drain.
- SAF-01, the laboratory safety document (the chemical hygiene plan, the waste plan and the exposure and spill response), is published beside this procedure and governs beyond the notes here: its hazard table, the protective equipment by task and the facts it marks as not yet supplied by the owner are the controlling text; the notes here are the bench reminders of it.

6 Equipment and materials

- The chromatograph, column and conditions of SOP-02; the instrument identifier, the column serial or lot, the data file and the acquisition time are recorded and required to issue. 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.
- A semi-micro balance (readability 0.01 mg, capacity at least 120 g, so one balance weighs the vials, the closures, the bare glass, the standard and up to 50 g of medium) with a draft shield, an anti-static ionizer and a vial adapter, in a room at 18–25 °C and 40–60% relative humidity, both recorded each day with the check weight near the vial load; the minimum weight printed on the balance (SOP-09; WS-02 (daily checks)).
- A calibrated thermometer reading to 0.5 °C for the medium; certified pyrogen-free low-binding polypropylene tubes (5, 15 and 50 mL) and low-binding autosampler vials; pyrogen-free serological pipettes; a crimper, a decrimper that removes the shell in one piece, forceps and venting needles. No volumetric glassware is needed: every volume is a mass.
- The vial-format table (SOP-09): the brimful capacity of each format received, measured once by weighing it brimful with water; it decides the in-vial or tube route below.

7 Reagents

Medium	LAL reagent water, or 10 mM Tris, pH 7.8, LAL grade for Semaglutide, tirzepatide and retatrutide whatever their salt form; the medium's lot, certificate and expiry are recorded, and its temperature at preparation to 0.5 °C.
Mobile phase A	Water, 0.1% trifluoroacetic acid; the diluent of every working solution, made as SOP-02 says.
Mobile phase B	Acetonitrile, 0.1% trifluoroacetic acid
Strong solvent	Acetonitrile and water 1 + 1 with 0.1% trifluoroacetic acid, for the fourth-rinse injection of Semaglutide, tirzepatide and retatrutide.

8 Reference standard

8.1 A reference standard of the named peptide, recorded with its name, supplier, catalogue number and lot, its certificate's reference, who assigned its content, the content's uncertainty and its expiry; stored as its certificate says, under its desiccant, the date first opened and the number of openings on the container.

8.2 Its content and the basis of that content, one of "net peptide content" or "HPLC purity", and how the content was assigned (the supplier's certificate with its date in words, or the laboratory's own assay). A standard carrying only an HPLC purity is corrected for purity only, and the mass per vial is then high by the water and counter-ion fraction of the standard; the model warns.

8.3 The content's uncertainty enters the budget one to one, by the route the content came by, which is recorded against the lot (REFERENCE_STANDARDS; the model reads the route from the record's words until it carries it as a field). Route C, a compendial standard used as the monograph prescribes: the pharmacopoeia states no uncertainty, so the laboratory verifies the content by mass balance and uses the standard uncertainty of that verification, or the default below. Route M, the laboratory's own assignment by mass balance (purity by SOP-02, water by SOP-05, the counter-ion by quantitative NMR, residual solvents and inorganic residue by an accredited provider), confirmed by quantitative NMR or isotope-dilution amino-acid analysis from a second provider: u is the quadrature sum of the characterisation, route-agreement, homogeneity and stability terms of the assignment record. Route S, a supplier-assigned content from an evaluated supplier (SOP-09): a certificate that states U with its k gives the term; one that states a bare tolerance is read as a rectangular half-width (divided by sqrt(3)); one that states nothing is given 3.0% relative with a warning; route H, a content that is only an HPLC purity, is given 5.0% and the mass is peptide as salt. The model carries the S and H figures and the U, k and tolerance readings (lab/results.py standard_content_u); the components of route M are combined in the assignment record and entered as one figure until the model lists them.

8.4 What the result is the mass of follows the basis: a "net peptide content" standard reports free peptide; a "HPLC purity" standard reports peptide as salt; an uncorrected standard reports as-is powder.

8.5 Weighing and the calibration stock (WS-05 (mass per vial, standard block)): let the container stand sealed at room temperature for at least 30 min; weigh at least 10 mg by difference into a tared low-binding tube on the semi-micro balance, within 30 s of opening, in the ventilated enclosure; record the mass, the balance id, the check-weight record and the time. Dissolve by mass in the sample's medium to 2 mg/mL (the medium's mass recorded): this is the calibration stock, and the same weighing serves the suitability solution of SOP-02. Levels by mass in mobile phase A, the stock added to the acid: 1 + 4, 3 + 17, 1 + 9, 1 + 19 and 1 + 39 of the stock give 0.4, 0.3, 0.2, 0.1, 0.05 mg/mL, with the fifth level (3 + 17) recommended so the top of the range is inside the curve; the masses on the worksheet; the model corrects each level for the content. Low-binding vials; 24 h at 2–8 °C or in the autosampler at 5 °C.

Reference standard, continued

8.6 Aliquots and whole-aliquot use: a lot bought in bulk is divided once, in a dry enclosure (a nitrogen-flushed glove bag or dry box below 10% relative humidity, the humidity recorded), into single-use aliquots of about 10–12 mg in tared amber vials (low-adsorption for Semaglutide, tirzepatide and retatrutide), each aliquot's net mass to 0.01 mg being its identity, capped under the dry gas, labelled "use whole; do not weigh" and stored desiccated at −20 °C. At use the whole aliquot is dissolved in a known mass of medium, so no powder is weighed outside the enclosure, and the calibration stock is the aliquot's mass times the content over the volume. A lot that cannot be divided (too little material, or a compendial standard whose instructions forbid it) is weighed from its container as the step above says. The model carries one weighing term (0.5%) for either until it reads the aliquot's own mass (REFERENCE_STANDARDS M3).

8.7 Storage, re-qualification and expiry: −20 °C, in the dark, desiccated, in the freezer on the equipment register with its temperature log (SOP-09); a container stands closed in a desiccator at room temperature for at least 30 min before it is opened, and an opened aliquot not used is discarded and logged. The initial expiry is 12 months from the assignment date (6 months for a lot of 95–97% purity or a sequence that deamidates quickly), extended only after re-qualification (purity, water, identity and one cross-check); a lot ends early when its purity has fallen by more than 0.5% area from assignment, a new impurity reaches 0.1% area, or its water has risen by more than 0.5% w/w. At a lot change two lots overlap for at least three content runs: the ratio of their responses per mg of free peptide must agree with the ratio of their contents within the combined U, and certificates issued across the change say which lot they used.

8.8 What the certificate says about the standard follows the route and never claims more than the chain supports: a compendial standard makes the result traceable to the pharmacopoeial standard, not to the SI; an in-house assigned standard is an in-house material, not a certified reference material, whose purity term is method-defined; a supplier-assigned content makes the result traceable to the supplier's assignment and to nothing more authoritative; an HPLC-purity standard to the supplier's purity figure. Not one of them says certified, CRM, USP grade or traceable to the SI for the whole result. Today the certificate prints the standard's name, supplier, catalogue number, lot, content and basis with how the content was assigned, in the record's own words; the sentence by route is composed once the model carries the route (REFERENCE_STANDARDS M9).

9 Sample preparation

The preparation, as the methods page states it:

> Mass per vial: three vials, each vented, weighed and dissolved whole, by mass, in LAL reagent water (10 mM Tris of LAL grade for semaglutide, tirzepatide and retatrutide, the Tris stock added to mobile phase A with mixing) to 2.0 mg/mL nominal, the volume being the nominal fill in mg divided by 2.0 (a 5 mg fill in 2.50 mL), then the stock 1:10 in mobile phase A, 0.20 mg/mL; the masses and volumes are recorded and printed on every certificate.
>
> The methods page, "Sample preparation" (data.json method.rows)

9.1 Vials: numbered on WS-01 (receipt); one unopened vial per preparation, labelled P1, P2 and P3 after the vial numbers. P1 supplies the endotoxin aliquot, the purity solution and the identity solution, and the certificate says so; every tube and autosampler vial is labelled with the accession, the preparation and the solution (STOCK, PUR, ID, CONT, LAL).

9.2 Venting before the first weighing (the sealed vial's headspace gas and pressure as the vendor stoppered it, 0.1–3 mg on a 2 mL vial): remove the crimp, lift the stopper for about a minute under the hood, reseat, equilibrate 10 min, then weigh; record it (vented, vent method). The stopper is held in forceps and never laid down, and closes the vial again during mixing.

Sample preparation, continued

9.3 Three weighings on the semi-micro balance, each with its time: W0, the vented vial with its stopper; Wc, the stopper (and the shell, weighed separately or not at all) within 2 min of decapping; W2, the bare glass after the analysis, rinsed three times with water and once with methanol, dried 1 h at 105 °C and equilibrated open in the balance room for at least 2 h. G = W0 – Wc – W2, the gross fill of the vial dissolved (u(G) 0.04 mg for a lifted stopper, a design estimate until measured). Powder on the stopper is rinsed into the vial with part of the medium, noted, and G flagged; a decrimper that cuts the shell leaves G not determined.

9.4 The stock: the whole vial dissolved by mass, to 2.0 mg/mL nominal in LAL reagent water (10 mM Tris of LAL grade for semaglutide, tirzepatide and retatrutide). V (mL) = nominal fill (mg) / 2; the medium is added to a target mass m_w = V × rho_w(T), read on the balance, and the stock volume follows from the masses and the density, V = (m_w × 1.00106 + G / 1000) / (rho_w(T) + 0.0006) (the air-buoyancy correction of the water mass and the density increment of a 2 mg/mL peptide salt, a design estimate); rho_w from the model's table (15 °C 0.9991 g/mL; 20 °C 0.9982 g/mL; 25 °C 0.99705 g/mL; 30 °C 0.99565 g/mL).

9.5 In the vial when V is at most 0.6 of the format's brimful capacity (SOP-09's format table); otherwise 0.8–1.0 mL of medium in the vial, transferred with a pyrogen-free serological pipette into a tared certified pyrogen-free low-binding polypropylene tube, the vial rinsed three times with at least 0.3 of the first portion, wetting the whole interior and the neck each time, each rinse transferred, and the tube made up to m_w on the balance. The tube's lot and certificate, the first portion and the rinses go on WS-03 (dissolution by mass).

9.6 Mixing: gentle inversion or rolling for 10 min at 20–25 °C, the vial closed with its own stopper; no vortexing or sonication for Semaglutide, tirzepatide and retatrutide (foaming, surface denaturation); at most 1 min of sonication at or below 25 °C for the others when a cake does not disperse, recorded.

9.7 Clarity before any aliquot: the stock against black and white backgrounds under the Ph. Eur. 2.9.20 viewing conditions, "clear and free of visible particles" or the description (opalescent, particles, gel, foam; the colour of a copper complex is recorded, not "colourless") and the time to dissolve; recorded on the worksheet and in the set (the first vial's observation is a required field). A stock that is not clear is not used: the vial is reported as not dissolved in the method's medium, and the preparation is repeated as VP-05's dissolution study directs.

9.8 Order of operations, within 15 min of dissolution and before any mobile phase A is opened at the bench: clarity, the endotoxin aliquot (SOP-04), the purity solution (SOP-02), the content solution, the identity solution (SOP-03); every dilution of a Tris stock the Tris stock added to the pre-weighed mobile phase A with immediate mixing, never the reverse. The stock is then held capped at 2–8 °C and discarded after the sequence has been checked (hold time 24 h until VR-05 measures longer); the times go on the worksheet.

9.9 Content solution: weigh 1.800 g of mobile phase A into a low-binding vial, add 0.200 g of the stock, cap and invert at once; D_m = (m_s + m_d) / m_s, a factor of 10 by mass (equal to the volume factor within 0.05%), 0.2 mg/mL nominal, the middle of the calibration; both masses on WS-05 (mass per vial).

9.10 Fourth-rinse injection for Semaglutide, tirzepatide and retatrutide, in every run: after the stock is withdrawn, the vial (and the tube) is rinsed with 1.0 mL of the strong solvent and the rinse injected under this method; peptide found at most 0.2% of the content, else the content is a lower bound and an open point.

9.11 Independent preparations, one vial each, are recorded as "preparations"; repeat injections of one preparation as "injections", and the certificate says which. Under this design the replicates are preparations; with the minimum sample of one vial they are injections. Vials consumed default to the number of preparations.

In a full panel the method as written consumes 5 vials (mass per vial 3; water content 2; HPLC from the mass per vial preparation; LC–MS from the HPLC preparation; endotoxin from the mass per vial preparation) (results.vials_required). This test takes 3 vials of its own there, and its preparation serves the HPLC run, the LC–MS run and the endotoxin run. Because the endotoxin aliquot (SOP-04) is drawn from these solutions, they are made in LAL reagent water and the aliquot is taken first, before any dilution in mobile phase A.

10 Calibration and system suitability

Check	Criterion	Basis
Calibration levels	At least three, at least two distinct concentrations, each level's nominal concentration corrected for the standard's content	lab/results.py (validate refuses fewer)
Linearity	r^2 at or above 0.999	lab/results.py
Sample response	Every sample area between the lowest and highest level areas; otherwise dilute or add a level	lab/results.py
Back-calculated recovery	Reported per level against the corrected level	USP <621>
System suitability	As SOP-02, on the same run	USP <621>

11 Procedure

11.1 Prepare the calibration levels from the standard (Reference standard); record each nominal concentration (mg/mL) and its peak area. Sequence names carry the accession and the role (_L1 to _L5, _P1 to _P3, _RINSE1).

11.2 Inject the sample preparations; record each area.

11.3 A level is never removed. r^2 below 0.999: re-inject the levels once; still failing, prepare the levels again from the stock; a third failure is an instrument or standard problem (SOP-08). Every injection stays in the data file and is named on the worksheet.

11.4 Record the standard's identity, content and uncertainty, each preparation's weighings, medium mass, temperature and dilution factor, the replicate kind and the vials consumed.

11.5 The model corrects the levels for content, fits the line, checks r^2 and the range, back-calculates every level, computes each preparation's volume and content, the fill uniformity over the gross fills and, when SOP-05 was run on whole vials, closes the mass balance per preparation.

12 Calculations

$C_{corrected} = C_{nominal} \times content / 100$

$A = slope \times C + intercept$ (least squares over the corrected levels; r^2 reported to five decimals)

V_i (mL) = $(m_w \times 1.00106 + G_i / 1000) / (rho_w(T) + 0.0006)$ (each preparation's own stock volume)

content per vial (mg) = $(A_i - intercept) / slope \times D_{m,i} \times V_i$ (per preparation; D_m the dilution factor by mass)

% of nominal fill = $100 \times content / nominal\ fill$

fill uniformity: mean, SD, RSD, lowest and highest of the gross fills G of every vial weighed (the content vials and the water vials)

RSD (%) = $100 \times s$ / mean over the preparations

Mass balance (with whole-vial Karl Fischer)

gross fill = content + water + impurities + counter-ion + unassigned

impurities (mg) = content × impurity area / main-peak area

counter-ion maximum (mg) = content / M_{avg} × basic sites × M_{anion}

unassigned (mg) = gross fill − water − content − impurities − counter-ion maximum (a lower bound: the counter-ion is taken at its maximum)

– Basic sites are the protonatable sites a monovalent counter-ion can occupy (the N-terminal amine plus K, R, H), stated or derived from a one-letter sequence; the anion masses on file, in g/mol, are trifluoroacetate (tfa) 113.02; acetate 59.04; hydrochloride (chloride) 35.45.

Calculations, continued

- Mass balance per vial: free peptide + water + impurities (content × impurity area / main area) + counter-ion (at most basic sites × anion mass per mole of peptide) + unassigned = the gross fill of the vials dissolved for content (the water vials' when the content vials were not weighed); with the counter-ion at its maximum the unassigned remainder is a lower bound; it is flagged above the remainder's expanded uncertainty (k = 2, the content, the gross fill, the water, the impurities and the counter-ion maximum in quadrature), below which an excipient or residual solvent is invisible to the balance (stoichiometry; the peptide's average molar mass from its formula and the standard atomic weights; JCGM 100:2008 5.1 for the remainder (METHOD_REVIEW S10); results.calculation_references qnt.mass_balance).
- The unassigned remainder is significant, and a warning, when it exceeds the combined uncertainty of the remainder itself (k = 2; the content's term dominates it, with the gross fill's and the water's inside it), so that the balance is not flagged inside its own uncertainty; a content above the gross fill less water makes the balance impossible and is a warning to check the standard's content and the fill. An excipient or residual solvent below that amount is invisible to the balance, and the certificate says so.
- A cation salt (an arginate) is bounded by the acidic sites, not the basic ones, and no anion mass on file applies to it: the balance then reports counter-ion and impurities together, by difference, and says why.
- The certificate prints the balance per preparation in one line each: gross fill, free peptide, water, impurities, counter-ion at most, unassigned; and the fill-uniformity line over every vial weighed.

What the balance cannot assign, as the laboratory publishes it:

> The counter-ion content (trifluoroacetate or acetate) is bounded by stoichiometry on the certificate, not measured; residual solvents, bulking excipients such as mannitol, elemental impurities and, for GHK-Cu, copper are not determined. Anything of that kind in the powder appears in the mass balance as "unassigned" and nowhere else.
>
> The limits page, "What we do not measure"

13 Measurement uncertainty and decision rule

Expanded uncertainty is reported with every quantitative result as U with k = 2. The standard error of the replicate mean is combined in quadrature with a method floor stated as an expanded value; for this method it is ±2% relative, and the floor is a provisional pre-validation constant, the laboratory's prior estimate, until the method's validation has measured intermediate precision.

$u_{rep} = s / \sqrt{n}$

$u_{floor} = \text{floor} / k$

$u_c = \sqrt{u_{rep}^2 + u_{floor}^2}$

$U = k\, u_c,\ k = 2$

$nu_{eff} = u_c^4 / (u_{rep}^4 / (n - 1))$ (Welch–Satterthwaite, GUM G.4.1)

The model reports the effective degrees of freedom and the t factor they imply (GUM Annex G, Table G.2) and warns when nu_eff is below 10, where k = 2 gives less than 95% coverage. A single determination carries the floor alone.

Decision rule (ISO/IEC 17025:2017 7.8.6; ILAC-G8:09/2019): the conclusion is reached by simple acceptance (guard band w = 0) on the value rounded to its reported precision; the statement beside it says whether the conclusion still holds with the expanded uncertainty (k = 2) included, the guard band w = U. Where a limit is stated more coarsely than the result, comparing at the reported precision is stricter than USP General Notices 7.20 and Ph. Eur. General Notices 1.2, which round to the limit's own digits.

The result model carries this rule, with its sources, as results.calculation_references stats.decision_rule; the certificate's method page prints it.

Point comparisons with a limit are made on the value rounded to its reported precision: content to 2 decimals in mg, 1 decimal as percent of the nominal fill.

Measurement uncertainty and decision rule, continued

The budget's terms, relative (lab/results.py; the floor's status for this method is "provisional"): first the reference standard's assigned content, which enters one to one (its certificate's U, or 3.0% relative by default; for a standard the laboratory assigned itself, the characterisation, route-agreement, homogeneity and stability components of the assignment record, entered as one figure until the model lists them); the standard's weighing and its uptake on the pan (0.5%); the calibration's prediction at the sample level, from each run's own curve; the preparation (stock by mass, dilution by mass, density, buoyancy, temperature, 0.1% in all); the repeatability of the three preparations, which holds the vial-to-vial fill; and the between-run term the floor stands in for until VR-05 measures s_run. Once a run's own calibration is inside the measured s_run, the calibration term is dropped for the validated state, and the certificate prints the rule it applied.

The standard's term is shared by every lot measured against the same standard lot: two of this laboratory's results on one standard lot compare with the repeatability and between-run terms alone, not with the full U, and the certificate says so for a sender tracking lot-to-lot consistency.

References: JCGM 100:2008 (GUM) 6.2 and Annex G; ISO/IEC 17025:2017 7.6 and 7.8.6.

14 Acceptance and reporting

- Against a supplied specification in mg or as a percent of nominal: Conforms or Does not conform. Against a mass on the sender's label: Meets claim, Below claim or Does not meet claim. Without either: "None supplied; for information".
- The printed mass is the "nominal fill", never the "label claim"; the result row reads "Mass per vial, <basis>" and the basis label is printed beside every mg.
- The certificate prints the calibration curve with the sample response marked, the levels with corrected concentration, area, back-calculation and recovery, each replicate's area and content, the mean and RSD, the standard's full identity and the mass balance.

15 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.
- A standard assigned on HPLC purity only overstates the free peptide; the certificate says the mass is "peptide as salt".
- A counter-ion not on file, or basic sites not stated, leaves the counter-ion maximum uncomputed; the balance then reports counter-ion and impurities together, by difference.
- The backend's form does not yet take the three weighings, the venting, the medium's mass and temperature, the clarity record, the dilution factor by mass, the balance or the standard's uncertainty, certificate and expiry; they are on WS-03 (dissolution by mass) and WS-05 (mass per vial) and in the set's notes until it does.
- The vial-format table and the oven's format (SOP-09) are owner facts not yet on file; the in-vial rule cannot be applied to a format whose brimful capacity has not been measured.

16 Records

- The result set lab/results/<set>.json, its audit trail and its emails; the register row; the certificate and record.json as issued (SOP-07).
- WS-03 (dissolution by mass) and WS-05 (mass per vial): the vial numbers, the venting and the three weighings with their times, the medium and its mass and temperature, the route and the rinses, the mixing, the clarity record, the order and times of the aliquots, the standard's weighing and levels, the content solutions' masses (SOP-07, Records and retention).

Records, continued

- The levels, the areas, each preparation's weighings and the standard's identity, content and uncertainty, in the result set.
- The supplier's certificate for the standard; the data file named on the certificate with its acquisition time.

17 Revision history

Revision	Change	Approval
1	First issue.	Not yet approved

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Current revisions

End of document