Identity by LC–MS: the run

The calibration check, the identity solution, the acquisition, the averaging windows and every charge state read from the cluster, with the bench check before Not confirmed.

Not yet approved
No approval of this revision is on file. This form is a controlled record once approved; until then it is the proposed layout of the bench record its procedure names.

Worksheet

Number	WS-08
Revision	1, first issue
Governs	SOP-03 Identity by LC–MS, revision 1
Status	Not yet approved
Effective	Not yet in effect; takes effect when approved
Applies to	Test codes ID, IDP, FULL
Supersedes	Nothing; first issue
Fingerprint	50a9 b468 58c4 ee5c

Laboratory

Name	Cupel Laboratories
Address	2599 Speakman Dr Mississauga, ON L5K 1B1
Country	Canada
Not on file	Not yet published: legal entity, postal address, telephone and country
Web	cupel.ca
Email	help@cupel.ca

1 Header

Accession, or the set id before logging

Analyte, as labelled

Analyst (the “Working as” name)

Date of the work (UTC)

Reviewer: line-by-line check (initials, date)

Attached to the set as (caption “WS-08 page p”)

2 How this form is used

- This worksheet is the original record of every value measured away from the backend. It is filled in ink at the moment of measurement; each line carries the time of the measurement and the analyst’s initials.
- A correction is a single line through the value, the new value, initials, the date and the reason; nothing is overwritten or erased.
- The completed form is photographed or scanned the same day and attached to the set at /photo/<id>/<name> with the caption “WS-08 page p”, each file hashed into the set’s trail as a “photo” entry; the values are typed into the form from it, and the reviewer checks the typed set against these pages line by line before signing.
- Lines marked “model only” feed a key the backend’s form does not yet expose: the value is typed into the section’s notes until it does. Lines marked “worksheet only” stay on this paper and in the notes.
- A failed criterion stops the work at that line: the time and what was done are written here, and the event goes to the set the same day as a note, a stop or a deviation (SOP-08).

3 Record

Instrument and calibration

#	Field, and the result-set key it feeds	Unit, decimals	Criterion	Value	Time (UTC), initials
1	Instrument id; software; class ms.instrument.id ms.instrument.software ms.method.instrument_class	text			
2	Calibrant lot; calibration time; residual error at the m/z nearest the analyte worksheet only	ppm, 1 decimal	residual within 2 ppm for a 10 ppm identity criterion (SOP-03)		
3	Resolving power and the m/z it was measured at ms.method.resolving_power ms.method.resolving_power_at_mz	FWHM	at least 10 000 for a ppm criterion		
4	Source: ionisation; capillary voltage; scan range; source temperatures; divert window ms.method.ionisation ms.method.capillary_kv ms.method.scan_range.0 ms.method.scan_range.1	kV, 1 decimal	first 1.5 min of the run are diverted (SOP-03)		

Solution and acquisition

#	Field, and the result-set key it feeds	Unit, decimals	Criterion	Value	Time (UTC), initials
5	Identity solution: from the purity solution of P1, the purity solution 1 + 9 in mobile phase A; vial type; prepared worksheet only	hh:mm UTC	0.1 mg/mL nominal		
6	Data file; acquired (local, then UTC) ms.data_file ms.acquired_utc	hh:mm UTC			
7	Main-peak RT on the HPLC run; averaging window (from, to); background window ms.observed[].rt_min	min, 2 decimals	within 0.2 min of the HPLC main peak; from half height to half height of the main peak on the extracted-ion trace of its most abundant charge state (SOP-03)		

Charge states

#	Field, and the result-set key it feeds	Unit, decimals	Criterion	Value	Time (UTC), initials
8	Per state: z; m/z; relative intensity; peak read (monoisotopic, most abundant, average) ms.observed[].z ms.observed[].mz_obs ms.observed[].intensity ms.observed_peak	z; m/z; %, m/z to 4 decimals	within 10 ppm		
9	Non-identity ions seen (adducts) ms.notes	text			

Record, continued

#	Field, and the result-set key it feeds	Unit, decimals	Criterion	Value	Time (UTC), initials
10	Bench check before Not confirmed: calibrant error in criterion; charge states agree with the isotope spacing; ions within the RT window worksheet only	Y / N	If the calibration fails, the run is an instrument failure, not a sample result: it is repeated after recalibration and the first file is kept and named (SOP-03)	Y / N	
11	Vials consumed (0 from the HPLC preparation) ms.vials_consumed	count			
12	Deviations (text) audit trail: deviation entry	text			

Every solution container is labelled with the accession, the solution name (ID), the concentration, the date and time made, the analyst and the expiry.

4 Completion

Completed by (signature) and time (UTC)

Reviewed line by line by (initials) on (date)

5 Revision history

Revision	Change	Approval
1	First issue.	Not yet approved

The fingerprint in the control block is the SHA-256 of this document's text as composed, first 16 hex digits. A change in any controlled source changes it; a copy whose fingerprint differs from the approved one is not the approved text, and a change is approved as a new revision.

Cupel holds no ISO/IEC 17025 accreditation; results are not accredited results.

This document is published so that a chemist can judge how a result was produced and what it can and cannot show; it is not an accredited procedure and claims no conformity with any standard it cites.

The current revision of every laboratory document is the one published at cupel.ca/methods. A printed copy is uncontrolled: compare its fingerprint with the published one.

Current revisions

End of document