Purity by RP-HPLC–UV: the run

The purity sequence from the mobile phases to the last injection: the solutions, the equilibration, the suitability read before the first sample, the integration and the hold-time evidence.

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-04
Revision	1, first issue
Governs	SOP-02 Purity by RP-HPLC–UV, revision 1
Status	Not yet approved
Effective	Not yet in effect; takes effect when approved
Applies to	Test codes PUR, IDP, FULL
Supersedes	Nothing; first issue
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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-04 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-04 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, column and mobile phases

#	Field, and the result-set key it feeds	Unit, decimals	Criterion	Value	Time (UTC), initials
1	Instrument id; software version hplc.instrument.id hplc.instrument.software	text			
2	Column id or lot; injections to date hplc.column.serial_or_lot	text	back-pressure at equilibration has risen by half (SOP-02)		
3	Mobile phase A: bottle number; prepared (date, time); lots (water, acid); expiry checked hplc.notes	text	Expiry: A 7 days, B 30 days (SOP-02)		
4	Mobile phase B: bottle number; prepared; lots (acetonitrile, acid); expiry checked hplc.notes	text			
5	Equilibration start; pressure; baseline drift; ready time worksheet only	bar; mAU/5 min, as displayed	for at least 15 min (about twenty column volumes); pressure is within 10% of the column's last recorded value; drifts less than 1 mAU over 5 min		
6	Autosampler temperature; column oven set and actual hplc.method.column_temp_c	°C	autosampler at 5 °C (SOP-02)		

Solutions

#	Field, and the result-set key it feeds	Unit, decimals	Criterion	Value	Time (UTC), initials
7	Suitability standard: name, lot, source; expiry checked hplc.system_suitability.standard.name hplc.system_suitability.standard.lot hplc.system_suitability.standard.source	text			
8	Suitability solution: from the WS-05 stock; concentration; prepared worksheet only	mg/mL nominal, as displayed	used within 24 h of preparation (SOP-02)		
9	Blank: the sample's matrix (medium 1 + 1 in mobile phase A); prepared worksheet only	hh:mm UTC			
10	Purity solution: stock source; stock concentration; dilution as made; vial type; prepared hplc.sample.source hplc.sample.stock_mg_ml_nominal hplc.sample.dilution_text hplc.sample.vial_type hplc.sample.prepared_utc	mg/mL nominal			
11	Injected concentration hplc.sample.injected_mg_ml_nominal	mg/mL nominal	1 mg/mL nominal, within 10% (the model warns beyond it)		

Record, continued

#	Field, and the result-set key it feeds	Unit, decimals	Criterion	Value	Time (UTC), initials
12	CDS clock against the laboratory UTC reference; offset typed at import worksheet only	+HH:MM, as displayed			

Sequence

Names carry the accession (or the set id before logging) and the role; every injection stays in the data file and is named here with its reason.

#	Field, and the result-set key it feeds	Unit, decimals	Criterion	Value	Time (UTC), initials
13	Position; name _BLK; data file; acquired (local, then UTC); volume worksheet only	µL, as displayed			
14	Position; name _STD1; data file; acquired; volume worksheet only	µL, as displayed			
15	Position; name _STD2; data file; acquired; volume worksheet only	µL, as displayed			
16	Position; name _STD3; data file; acquired; volume worksheet only	µL, as displayed			
17	Position; name _STD4; data file; acquired; volume worksheet only	µL, as displayed			
18	Position; name _STD5; data file; acquired; volume worksheet only	µL, as displayed			
19	Position; name _S1; data file; acquired; volume hplc.injections[].label hplc.injections[].data_file hplc.injections[].acquired_utc	µL, as displayed			
20	Position; name _S2; data file; acquired; volume hplc.injections[].label hplc.injections[].data_file hplc.injections[].acquired_utc	µL, as displayed			
21	Position; name _S3; data file; acquired; volume hplc.injections[].label hplc.injections[].data_file hplc.injections[].acquired_utc	µL, as displayed			

System suitability, read before the first sample injection

#	Field, and the result-set key it feeds	Unit, decimals	Criterion	Value	Time (UTC), initials
22	Blank peak at the main-peak RT (% of the standard's main area); interference hplc.system_suitability.blank_peak_area_pct hplc.system_suitability.blank_interference	%, as displayed	blank shows a peak at or above 0.05% of the standard's main area (SOP-02)		
23	Standard retention time hplc.system_suitability.standard_rt_min	min, 2 decimals			

Record, continued

#	Field, and the result-set key it feeds	Unit, decimals	Criterion	Value	Time (UTC), initials
24	Symmetry factor (As) hplc.system_suitability.tailing_factor	2 decimals	0.8–1.8 (USP <621>)		
25	Plate number (N); width basis hplc.system_suitability.plates hplc.system_suitability.width_basis	whole number	reported, no criterion		
26	Resolution (Rs); pair named with its RRT hplc.system_suitability.resolution_min hplc.system_suitability.resolution_pair	Rs, 1 decimal	at least 1.5; nearest impurity at or above 0.1% in the standard (SOP-02)		
27	Standard area RSD; n hplc.system_suitability.standard_area_rsd_pct hplc.system_suitability.n_standard_injections	%, 2 decimals	at most 2.0%, n = 5		
28	Pass or fail; action taken on a failure (text, time) worksheet only	text	In order, a fresh blank and one more standard injection; the needle wash, the seal and the standard vial's level; re-equilibration for 15 min; a fresh standard solution (SOP-02)		

Integration and results

#	Field, and the result-set key it feeds	Unit, decimals	Criterion	Value	Time (UTC), initials
29	Processing method name and version; manual events (injection, peak, reason) worksheet only	text	manual integration event is made only on an impurity, never on the main peak, is recorded in the data system's audit trail and listed on the worksheet with its reason (SOP-02)		
30	Integration start, from the blank; baseline treatment hplc.integration_start_min hplc.baseline_treatment	min, 1 decimal			
31	Per injection: main-peak RT; main area %; peaks at or above the reporting threshold (0.05%); largest impurity RRT (the paste is the record) hplc.injections[].peaks[].rt_min hplc.injections[].peaks[].area hplc.injections[].peaks[].role	min, 2 decimals			
32	Co-elution observed (shoulder, RRT); open point hplc.coeluting hplc.coeluting_pair hplc.open_point	text			
33	Impurity masses from the online LC–MS: RT, m/z, z hplc.impurity_ms[].rt_min hplc.impurity_ms[].mz_obs hplc.impurity_ms[].z	min; m/z; z, RT to 2 decimals, m/z to 4			

Record, continued

#	Field, and the result-set key it feeds	Unit, decimals	Criterion	Value	Time (UTC), initials
34	Time of the last injection (hold-time check against the preparation) worksheet only	hh:mm UTC	injected within 24 h of its preparation (SOP-02)		
35	Vials consumed hplc.vials_consumed	count			
36	Deviations (text) audit trail: deviation entry	text			

Every solution container is labelled with the accession, the solution name (MP-A, MP-B, BLK, STD, PUR), 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

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Current revisions

End of document