Water content: the run

The titrator's day from the drift to the last replicate: the reagent, the blanks, the water standard at the start and the end, and each whole vial weighed, crimped and titrated.

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-06
Revision	1, first issue
Governs	SOP-05 Water content by coulometric Karl Fischer, revision 1
Status	Not yet approved
Effective	Not yet in effect; takes effect when approved
Applies to	Test codes H2O, FULL
Supersedes	Nothing; first issue
Fingerprint	d2b1 4c42 5d3a 7db9

Laboratory	
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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-06 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-06 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

Titrator, oven and reagent

#	Field, and the result-set key it feeds	Unit, decimals	Criterion	Value	Time (UTC), initials
1	Titrator id; software; method or program name kf.instrument.id kf.instrument.software	text			
2	Oven temperature set; extraction time or drift stop; carrier gas and flow kf.oven_temp_c kf.extraction_time_s kf.carrier_flow	°C; s; mL/min			
3	Anolyte (and catholyte): name, lot, expiry checked; changed (date, time) kf.reagent	text			
4	Drift readings, three a minute apart; stable; action kf.drift_ug_min kf.drift_utc kf.drift_corrected	µg/min, 1 decimal	at most 10 µg/min (oven), 20 (open cell); less than 2 µg/min over three consecutive readings a minute apart (SOP-05); Not reached within 30 min: septum and cell seals checked, cell dried, anolyte changed; the cell is not used until stable (SOP-05)		

Blanks and the water standard

#	Field, and the result-set key it feeds	Unit, decimals	Criterion	Value	Time (UTC), initials
5	Oven blanks, empty vials through the same program: gross µg and time of each; drift-compensated; mean; SD; n kf.blank_vial_ug kf.blank_sd_ug kf.blank_n kf.blank_utc	µg, 1 decimal	mean at most 25 µg, SD at most 3 µg, n at least 2		
6	Methanol technique only: solvent lot and volume; syringe masses; blank µg at the start and the end of the series; subtracted kf.solvent kf.blank_ug kf.blank_subtracted	µg, as displayed			
7	Water standard: name, supplier, lot, certified value, certificate, uncertainty, expiry checked kf.standard_check.name kf.standard_check.supplier kf.standard_check.lot kf.standard_check.certificate_ref kf.standard_check.u_rel_pct kf.standard_check.expiry_utc	% relative, standard, 1 decimal			
8	Standard taken: syringe or vial full mass, empty mass, mass taken worksheet only	mg, as displayed			

Record, continued

#	Field, and the result-set key it feeds	Unit, decimals	Criterion	Value	Time (UTC), initials
9	Certified µg; found µg; recovery; pass; time kf.standard_check.certified_ug kf.standard_check.found_ug kf.standard_check.measured_utc	µg, 1 decimal	recovery 97.5–102.5%		
10	Standard repeated at the end of the day: found; recovery worksheet only	µg; %, as displayed	recovery 97.5–102.5%		

Replicate: W1

#	Field, and the result-set key it feeds	Unit, decimals	Criterion	Value	Time (UTC), initials
11	Vial number (WS-01); format fits the oven vial: whole, re-crimped or weighed portion worksheet only	text	A format that fits the oven vial goes in whole (SOP-05)		
12	Vented: method; time kf.replicates[].vented kf.replicates[].vent_method	text	for at least 10 min in the hood, withdraw the needle and equilibrate 10 min (SOP-05)		
13	W0, the vented sealed vial kf.replicates[].w0_mg kf.replicates[].w0_utc	mg, 2 decimals	gross fill within 25% of the nominal fill		
14	Wc, the closure, dry kf.replicates[].wc_mg kf.replicates[].wc_utc	mg, 2 decimals	within 2 min of decapping (SOP-05)		
15	Stopper pulled to crimped: within the limit; oven-vial crimp number worksheet only	Y / N	crimp within 15 s (SOP-05)	Y / N	
16	Gross µg titrated; drift × time subtracted; net µg; extraction or titration time; end criterion met kf.replicates[].water_ug kf.replicates[].titration_s kf.replicates[].measured_utc kf.titration_end_criterion	µg, 1 decimal			
17	W2, the bare glass: rinsed, dried, equilibrated; weighed kf.replicates[].w2_mg kf.replicates[].w2_utc	mg, 2 decimals	dried 1 h at 105 °C and equilibrated open in the balance room for at least 2 h (SOP-05)		
18	G = W0 − Wc − W2 (the model derives it) kf.replicates[].mass_mg	mg, 2 decimals			

Replicate: W2

#	Field, and the result-set key it feeds	Unit, decimals	Criterion	Value	Time (UTC), initials
19	Vial number (WS-01); format fits the oven vial: whole, re-crimped or weighed portion worksheet only	text	A format that fits the oven vial goes in whole (SOP-05)		
20	Vented: method; time kf.replicates[].vented kf.replicates[].vent_method	text	for at least 10 min in the hood, withdraw the needle and equilibrate 10 min (SOP-05)		
21	W0, the vented sealed vial kf.replicates[].w0_mg kf.replicates[].w0_utc	mg, 2 decimals	gross fill within 25% of the nominal fill		

Record, continued

#	Field, and the result-set key it feeds	Unit, decimals	Criterion	Value	Time (UTC), initials
22	Wc, the closure, dry kf.replicates[].wc_mg kf.replicates[].wc_utc	mg, 2 decimals	within 2 min of decapping (SOP-05)		
23	Stopper pulled to crimped: within the limit; oven-vial crimp number worksheet only	Y / N	crimp within 15 s (SOP-05)	Y / N	
24	Gross µg titrated; drift × time subtracted; net µg; extraction or titration time; end criterion met kf.replicates[].water_ug kf.replicates[].titration_s kf.replicates[].measured_utc kf.titration_end_criterion	µg, 1 decimal			
25	W2, the bare glass: rinsed, dried, equilibrated; weighed kf.replicates[].w2_mg kf.replicates[].w2_utc	mg, 2 decimals	dried 1 h at 105 °C and equilibrated open in the balance room for at least 2 h (SOP-05)		
26	G = W0 – Wc – W2 (the model derives it) kf.replicates[].mass_mg	mg, 2 decimals			

The day's end and the pair

#	Field, and the result-set key it feeds	Unit, decimals	Criterion	Value	Time (UTC), initials
27	Drift after the last sample worksheet only	µg/min, as displayed	At or below twice the drift before the first (SOP-05)		
28	Data file; weighing statement and mode; balance id and check kf.data_file kf.weighing kf.weighing_mode kf.balance.id kf.balance.check_record	text			
29	Pair difference against r; third vial run kf.replicates[].water_ug	Y / N	r = as SOP-05 states; a pair beyond r is not averaged	Y / N	
30	Whole vial per replicate; vials consumed kf.whole_vial kf.vials_consumed	count			
31	Deviations (text) audit trail: deviation entry	text			

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