VITLAB®
Volumetric instruments

Standard Operating Procedure (SOP)

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1. Introduction

The standard DIN EN ISO 4787 describes both the design and the testing of the volumetric instruments. The following testing instructions describe how to apply the ISO standard in practice.

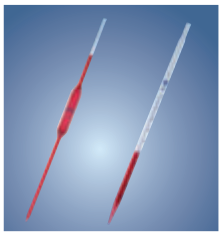
We recommend a recurring check every 1 - 3 years (more frequently depending on the application) because the accuracy may be affected e.g. by aggressive chemicals or by procedure and frequency of cleaning.

These instructions may be used as a basis for the supervision of testing devices according to DIN EN ISO 9001, DIN EN ISO 10012 and DIN EN ISO / IEC 17025.

Volumetric instruments by VITLAB


Volumemetric flasks


Graduated cylinders


Bulb and graduated pipettes


Burettes

Setting the meniscus

Concave meniscus

A downward curved meniscus is read at the lowest point of the liquid level. The lowest point must cover the top edge of the graduation.

Example: Aqueous solution and glass wall

Convex meniscus

The liquid volume with an upward meniscus is read at the highest point of the liquid level. The highest point of the meniscus should touch the upper edge of the graduation.

Example: Aqueous solution and plastic wall

2. Preparation for testing

Batch (Lot) or individual serial number, nominal volume and tolerances are directly printed on every VITLAB® volumetric instrument.

► The test starts with a clear identification of the volumetric instrument in the test record.

2.1 Instrument identification

Read batch number or individual serial number

► All class A volumetric instruments from VITLAB have a batch number. Instruments with an individual certificate can be ordered separately. Enter the number into the test record.

Identify type of instrument

► Enter identification in test record. Differentiation between instruments calibrated to contain ('In') like volumetric flasks and graduated cylinders and instruments calibrated to deliver ('Ex') such as bulb and graduated pipettes, burettes and titration apparatuses.

Read customers identification, if present

► Enter identification into test record.

2.2 Nominal volume and tolerances

Read nominal volume and, if appropriate, subdivision

► Enter into test record. In case of graduated instruments, specify the subdivision.
For example 20 : 0.1 ml

Read error limits (tolerances)

► Read inscriptions on the instrument and enter into test record.

2.3 Material

Identify material

► For example:
Plastic such as PFA, PMP, PP (volumetric flasks, graduated cylinders)
Borosilicate glass 3.3 (Burettes, titration apparatuses). Enter material in the test record.

3. Visual examination

3.1 Cleanliness

No media residues

Clean surface, free of grease

► To obtain the specified volumetric accuracy, the surface must be clean and free of grease. If drops adhere to the surface of the vessel or the meniscus does not form evenly, the instrument is contaminated and has to be cleaned. Rinse subsequently with tap water and finally with distilled or deionized water.

3.2 Inscriptions of volumetric instruments

Check for good readability

► All inscriptions must be clearly legible, e. g. DE-M marking, class A marking, nominal capacity, error limits, reference temperature, calibration ‚In' / ‚Ex', batch or serial number, and volumetric markings.

3.3 Damage

General Damage

► The surface must not show any significant damage such as scratches or breakages.

► Especially the opening in the tip of pipettes and burettes should not show any damage.

► Burette stopcocks must close tightly, smoothly and easily. Within 60 seconds, no drop should form at the tip.

4. Test instruments and environment

Test room

The calibration should be performed in a draught-free room with constant temperature and humidity.

Temperature

The instrument to be tested and the test liquid must be at equilibrium with the room temperature. For this, leave the volumetric instrument (without packaging) and the test liquid for at least 1 hour in the test room. Try to avoid temperature changes (e.g. from sun radiation). Then carry out a comparison of the temperature from device, liquid and room.

Test liquid

Distilled or deionized water (bottle filled with at least 500 ml) of a minimum quality 3 according to ISO 3969. The maximum difference of room and water temperature should be 0.5 °C.

Balance

Recommended specifications, see table:

Selected volume* of the device to be tested V	Resolution of the balance display mg	Standard uncertainty (Repeatability) mg	Linearity mg
100 µl < V ≤ 10 ml	0.1	0.2	0.2
10 ml < V ≤ 1,000 ml	1	1	2
1,000 ml < V ≤ 2,000 ml	10	10	20
V > 2,000 ml	100	100	200

*For practical purposes, the nominal volume may be used to choose the balance.

Thermometer

Use only thermometers with a maximum measurement error of 0.1 °C.

Barometer

For testing the atmospheric pressure with an accuracy of 1 kPa.

Recipient vessel

Vessel (e.g. Erlenmeyer flask) filled with water, so that the bottom of the vessel is covered with liquid.

Pipette helper

For example, VITLAB maneus®

Test setup

For the testing of pipettes and burettes calibrated ‚Ex' (to deliver), a support for vertical mounting of the instrument is required.

Stopwatch

To keep track of the waiting time. With an accuracy of ± 1 second.

Lint-free tissue

For wiping.

Traceability of test results to national standards

Through the use of calibrated testing devices (balance and thermometer), the requirement of DIN EN ISO 9001, DIN EN ISO 10012 and DIN EN ISO / IEC 17025 to refer the test to the national standards is fulfilled. The calibration of the balance e.g. can be carried out either by DAkkS calibration or official certification of the balance, or by calibrating the balance with the appropriate weights that are traced to the national standard. The calibration of the thermometer can also be carried out by DAkkS calibration or official certification, or by a comparison to thermometers that are traced to the national standard (under defined conditions).

5. Gravimetric testing

5.1 Volumetric instruments calibrated to deliver ‚Ex'

Volumetric flasks and graduated cylinders (PFA, PMP, PP)

1. Determine temperature of the test liquid.
   ▶ Enter temperature in test record.
2. Determine empty weight of the dry volumetric instrument (W1).
   ▶ Enter weight in test record.
3. Fill the instrument with the test liquid to approx. 5 mm above the ring mark.
   ▶ The vessel surface above the meniscus must not be wetted. If necessary, wipe dry with a tissue.
4. Adjust the meniscus precisely to the ring mark by removing liquid with a pipette.
   ▶ If the meniscus is concave, the lowest point must be aligned with the upper edge of the ring mark. If the meniscus is convex, the highest point must be aligned with the upper edge of the ring mark. Read without parallax, i.e. your eyes have to be at the same level.
5. Determine weight of the filled instrument (W2).
   ▶ Enter weight in test record.

5.2 Volumetric instruments calibrated to deliver ‚Ex'

Bulb and graduated pipettes

1. Determine temperature of the test liquid.
   ► Enter temperature in test record.
2. Determine weight of the weighing vessel (W1).
   ► Enter weight in test record.
3. Clamp the pipette vertically in a stand.
4. Fill the pipette with a pipette helper to approx. 4 mm above the mark of the nominal volume.
5. Dry the outside of the pipette tip with a tissue.
6. Adjust the meniscus precisely by releasing liquid.

   Attention: At this point, do not pipette into the weighing vessel.

   ► If the meniscus is concave, the lowest point must be aligned with the upper edge of the ring mark. If the meniscus is convex, the highest point must be aligned with the upper edge of the ring mark. Read without parallax, i.e. your eye have to be at the same level.
   ► If a drop still adheres to the tip, wipe it off with a tissue.

7. Now release the liquid into the weighing vessel. The tip should touch the inclined vessel wall.
   ► As soon as the meniscus has come to a stop inside the pipette tip, the waiting time begins.
   ► If a drop adheres to the tip, wipe it off on the inside of the weighing vessel.
8. Determine the weight of the filled weighing vessel.
   ► Enter weight (W2) in test record.

   Note: In case of pipettes graduated for partial delivery, release the water until approx. 10 mm above the lowest mark, while the pipette tip touches the inclined wall of the weighing vessel. After the waiting time, adjust the meniscus precisely to the ring mark.

Burettes and titration apparatus

1. Determine temperature of the test liquid.
   ▶ Enter temperature in test record.

2. Determine weight of the weighing vessel (W1).
   ▶ Enter weight in test record.

3. Clamp burette vertically in a stand.

4. Fill the burette to approx. 5 mm above the zero mark and release some liquid to aerate the burette stopcock. Release the liquid no further than the nominal volume.
   ▶ After the first filling, a small air bubble may be inside the burette stopcock. To remove the bubble, hold the burette inclined and gently tap with a finger at the location of the bubble.

5. Fill the burette to approx. 5 mm above the zero mark.
   ▶ The vessel wall above the zero mark shall not be wetted. If necessary, wipe dry with a tissue.

6. Adjust precisely to the zero mark by releasing liquid.

   Attention: At this point, do not release liquid into the weighing vessel.

   ▶ If the meniscus is concave, the lowest point must be aligned with the upper edge of the ring mark. If the meniscus is convex, the highest point must be aligned with the upper edge of the ring mark. Read without parallax, i.e. your eye must be at the same level.
   ▶ If testing a burette with Schellbach stripes, the point where the two arrowheads touch, must be aligned with the zero mark.

7. Release the test liquid into the weighing vessel to approx. 5 mm above the lowest graduation mark.
   ▶ The burette tip shall not touch the vessel wall.

8. After 30 seconds waiting time (use stopwatch) adjust the meniscus precisely to the graduation of the nominal volume.
   ▶ Wipe the burette tip off the inner vessel wall.

9. Determine the weight of the filled weighing vessel (W2).
   ▶ Enter weight in test record.

6. Evaluation

Generally, for all volumetric instruments calibrated to contain ('In'), one test should be sufficient. For volumetric instruments calibrated to deliver ('Ex'), it is advisable to use the mean value resulting from 3 measurements to be on the safe side. The scatter of the individual results should be not greater than ¼ of the admissable error limit (tolerance) of the measuring instrument.

Example: The error limit of a 50 ml burette is ± 0.10 ml. Therefore, the scatter of the individual results should be ± 0.025 ml. If the scatter is greater, the testing procedure should be revised and the test should be repeated.

The standard DIN EN ISO 4787 describes the gravimetric testing of volumetric instruments and provides the following general equation for calculations:

$$V_{20} = (W_2 - W_1)\left(\frac{1}{\rho_W - \rho_L}\right)\left(1 - \frac{\rho_L}{\rho_G}\right)\left(1 - \gamma\,(t - 20\,°C)\right)$$

Since this equation is very complicated and requires a multitude of tables, we are providing a simplified calculation using the factor z:

$$V_{20} = (W_2 - W_1) \cdot z$$

The parameters are:
- V_{20} [ml]: Volume of the test instrument at 20 °C
- W_1 [g]: Weight of the empty volumetric instrument / resp. weight of the weighing vessel before delivery
- W_2 [g]: Weight of the filled volumetric instrument / resp. weight of the weighing vessel after delivery
- z [ml/g]: Factor of the combined parameters (see tables)

To further simplify the list of test instruments, we recommend to use DE-M marked volumetric instruments with batch or individual serial number. For certified volumetric instruments, the initial testing can be omitted because the test results are recorded in the certificate.

Factor „z"

The factor z takes the following parameters into account:

Density of the calibration weight of the balance (ρ_G):

- 8 g/ml (see specifications provided by the balance manufacturer)

Air density (ρ_L) in relation to atmospheric pressure, temperature and relative air humidity 40 - 90 %:

- For all volumetric instruments (except volumetric flasks > 250 ml) the influence of atmospheric pressure in relation to the stated error limits is relatively small. Therefore, the factor z should generally be read from the table „Medium atmospheric pressure range"
- For volumetric flasks > 250 ml, select the appropriate table for lower, medium or upper atmospheric pressure range, according to the present conditions. To determine the appropriate table, measure the atmospheric pressure or inquire at your local meterological station.
- The atmospheric pressure related at sea-level has to be converted into local level.

Density of water (ρ_W) in relation to temperature

Cubic expansion coefficient (γ) of the volumetric instrument in relation to its material:

- Boro 3.3: $\gamma = 9.9 \times 10^{-6}\ °C^{-1}$
- Polypropylene: $\gamma = 450 \times 10^{-6}\ °C^{-1}$
  (Manufacturer's information, average value of $\gamma = 300 \times 10^{6}\ °C^{1}$ to $\gamma = 600 \times 10^{6}\ °C^{1}$)
- Polymethylpentene: $\gamma = 351 \times 10^{-6}\ °C^{-1}$ (Manufacturer's information: Mitsui)
- PFA: $\gamma = 330 \times 10^{-6}\ °C^{-1}$

7.1 Factor z [ml/g] for Borosilicate glass 3.3 and PFA

Temperature °C	Lower air pressure range 980 to 1,000 hPa		Medium air pressure range 1,000 bis 1,020 hPa		Upper air pressure range 1,020 to 1,040 hPa	
	Boro 3.3	PFA	Boro 3.3	PFA	Boro 3.3	PFA
15	1.00200	1.00360	1.00202	1.00362	1.00204	1.00365
15.5	1.00207	1.00351	1.00209	1.00353	1.00211	1.00356
16	1.00214	1.00343	1.00216	1.00345	1.00218	1.00347
16.5	1.00222	1.00334	1.00224	1.00336	1.00226	1.00338
17	1.00230	1.00326	1.00232	1.00328	1.00234	1.00330
17.5	1.00238	1.00318	1.00240	1.00320	1.00242	1.00322
18	1.00246	1.00311	1.00248	1.00313	1.00251	1.00315
18.5	1.00255	1.00303	1.00257	1.00305	1.00260	1.00308
19	1.00264	1.00296	1.00266	1.00298	1.00268	1.00301
19.5	1.00274	1.00290	1.00276	1.00292	1.00278	1.00294
20	1.00283	1.00283	1.00285	1.00285	1.00287	1.00287
20.5	1.00293	1.00277	1.00295	1.00279	1.00297	1.00281
21	1.00303	1.00271	1.00305	1.00273	1.00307	1.00275
21.5	1.00313	1.00265	1.00316	1.00267	1.00318	1.00269
22	1.00321	1.00260	1.00323	1.00262	1.00325	1.00264
22.5	1.00335	1.00255	1.00337	1.00257	1.00339	1.00259
23	1.00346	1.00250	1.00348	1.00252	1.00350	1.00254
23.5	1.00358	1.00245	1.00360	1.00247	1.00362	1.00249
24	1.00369	1.00240	1.00371	1.00243	1.00373	1.00245
24.5	1.00381	1.00236	1.00383	1.00238	1.00385	1.00240
25	1.00393	1.00232	1.00395	1.00234	1.00397	1.00234
25.5	1.00405	1.00229	1.00408		1.00410	1.00233
26	1.00418	1.00225	1.00420		1.00422	1.00229
26.5	1.00431	1.00222	1.00433		1.00435	1.00226
27	1.00444	1.00219	1.00446		1.00448	1.00223
27.5	1.00457	1.00216	1.00459		1.00461	1.00220
28	1.00471	1.00213	1.00473		1.00475	1.00218
28.5	1.00485	1.00211	1.00487		1.00489	1.00215
29	1.00499	1.00209	1.00501		1.00503	1.00213
29.5	1.00513	1.00207	1.00515		1.00517	1.00211
30	1.00527	1.00205	1.00529		1.00531	1.00210

7.2 Factor z c[ml/g] for PMP and PP

Temperature °C	Lower air pressure range 980 to 1,000 hPa		Medium air pressure range 1,000 bis 1,020 hPa		Upper air pressure range 1,020 bis 1,040 hPa	
	PMP	PP	PMP	PP	PMP	PP
15	1.00371	1.00420	1.00373	1.00423	1.00375	1.00425
15.5	1.00361	1.00406	1.00363	1.00408	1.00365	1.00410
16	1.00351	1.00391	1.00353	1.00393	1.00355	1.00395
16.5	1.00342	1.00376	1.00344	1.00379	1.00346	1.00381
17	1.00332	1.00362	1.00334	1.00364	1.00337	1.00366
17.5	1.00324	1.00348	1.00326	1.00351	1.00328	1.00353
18	1.00315	1.00335	1.00317	1.00337	1.00319	1.00339
18.5	1.00307	1.00322	1.00309	1.00324	1.00311	1.00326
19	1.00298	1.00308	1.00301	1.00310	1.00303	1.00313
19.5	1.00291	1.00296	1.00293	1.00298	1.00295	1.00300
20	1.00283	1.00283	1.00285	1.00285	1.00287	1.00287
20.5	1.00276	1.00271	1.00278	1.00273	1.00280	1.00275
21	1.00269	1.00259	1.00271	1.00261	1.00273	1.00263
21.5	1.00262	1.00247	1.00264	1.00249	1.00266	1.00251
22	1.00256	1.00232	1.00255	1.00235	1.00257	1.00237
22.5	1.00250	1.00225	1.00252	1.00227	1.00254	1.00229
23	1.00243	1.00214	1.00245	1.00216	1.00247	1.00218
23.5	1.00238	1.00203	1.00240	1.00205	1.00242	1.00207
24	1.00232	1.00192	1.00234	1.00194	1.00236	1.00196
24.5	1.00227	1.00182	1.00229	1.00184	1.00231	1.00186
25	1.00222	1.00172	1.00224	1.00174	1.00226	1.00176
25.5	1.00217	1.00162			1.00221	1.00167
26	1.00212	1.00153			1.00216	1.00157
26.5	1.00208	1.00144			1.00212	1.00148
27	1.00204	1.00134			1.00208	1.00139
27.5	1.00200	1.00126			1.00204	1.00130
28	1.00196	1.00117			1.00201	1.00121
28.5	1.00193	1.00109			1.00198	1.00113
29	1.00190	1.00100			1.00194	1.00104
29.5	1.00187	1.00093			1.00191	1.00097
30	1.00184	1.00085			1.00188	1.00089

Test record for volumetric instruments

1. Volumetric instrument, Class A / AS, DE-M marked

Serial Number: _____

Lot-No.: _____

Calibration: ❑ ‚In' ❑ ‚Ex'

Type of instrument: _____

Nominal capacity : Subdivision: _____

Error limits: _____

Material
- ❑ PFA
- ❑ PMP (Polymethypentene)
- ❑ PP (Polypropylene)
- ❑ Borosilicate glass 3.3
- ❑ _____

Customer specific marking: _____

2. Damage: ❑ none
❑ Type of damage
.....
.....
.....

3. Test conditions:

Test temperature: ..... °C
Balance: .....
Thermometer: .....

Air pressure range: ❑ lower ❑ medium ❑ upper
Device No.: .....
Device No.: .....

4. Calculation: $V_{20} = (W_2 - W_1) \cdot z$

5. Evaluation:

Weighing values	Weighing value W_2 [g]	Weighing value W_1 [g]	Factor z [ml/g]	Volume V_{20} [ml]
x_1				
x_2				
x_3				
			Mean value:	

❑ **Test passed (within error limits)**

❑ **Test not passed (not within error limits)**

VITLAB®

_____ _____
Date Signature (Tester)