

Titrette® and Digital Burette

(all models)

Testing Instructions (SOP)

May 2009

1. Introduction

The standard ISO DIS 8655 describes both the design and the testing of bottle-top burette. The following Testing Instructions describe how to apply the ISO standard in practice.

We recommend a testing every 3-12 months. This interval may be adjusted to individual requirements. For example, when working very frequently or when using aggressive media, the instrument should be tested more frequently.

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

For the regular examinations required by DIN EN ISO 9001, DIN EN ISO 10012, DIN EN ISO/IEC 17025 and the GLP Guidelines, BRAND additionally provides a calibration service (see page 9). Your instrument will be returned within a few days together with a test report. For more detailed information, please contact your labware supplier.



2. Preparation for testing and visual examination

2.1 Type and serial number

- Determine instrument type and nominal capacity. ⇒ Enter/ check in Test Record (1).
- Read Serial Number (embossed at the housing). ⇒ Enter number in Test Record (1).
- Read customer's identification, if present. ⇒ Enter identification in Test Record (1).

2.2 Minimal configuration Titrette® and Digital Burette

- Burette ⇒ Use only manufacturer's original parts.
- Titrating tube
- Filling tube
- Mounting tool for valves

2.3 Cleaning

- Rinse instrument with cleaning solution. Then rinse the instrument several times with distilled water. ⇒ Select suitable cleaning solution according to the medium which was used.
- Wipe off the exterior of the Titrette® and Digital Burette.

2.4 Visual examination (Damage, leakages)

- Housing ⇒ Enter fillings in Test Record (2).
- Titrating tube
- Filling tube
- Recirculation tube
- Display
- Controls
- Leakages

Possible faults and resulting measures:

Fault	Measures
Filling or titrating tube bent or damaged	Possible safety risk - therefore replace parts (see Operating Manual "Accessories").
Leakages	Possible safety risk - therefore replace parts or return instrument for repair.
Faulty controls, faulty display	Return instrument for repair.

2.5 Functional test

- Screw the Titrette® / Digital Burette on a bottle filled with distilled or deionised water.
- Priming (see Operating Manual). ⇒ A few bubbles up to 1 mm in the glass cylinder are permissible.
- Check controls ⇒ Hand wheels operating smoothly
⇒ Function of the keys On/Off, Pause and CLEAR.
- Result ⇒ Enter findings into Test Record (3)

If there is a malfunction of the instrument (e.g., piston difficult to move, sticking valves or leakage) please consult the "Troubleshooting" section of the operating manual.

3. Required equipment for testing

- Titrette® /Digital Burette
- **Bottle** (at least 500 ml) filled with distilled or deionised water (room temperature). ⇒ Match temperature of room, water and instrument.
- **Recipient** (e.g., Erlenmeyer flask, narrow-mouth) filled with some water. ⇒ Bottom of vessel should be covered.
- **Balance** with accuracy: ⇒ 0.001 g
- **Thermometer** with accuracy: ⇒ 0.2 °C
- Place the instrument into the testing room for at least 1 hour (unpacked!). ⇒ Allow instrument to adjust to room temperature.

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 standard is fulfilled. The calibration of the balance e.g. can be carried out either by DKD calibration or official certification of the balance, or by calibrating the balance with appropriate weights that are traced to the national standard. The calibration of the thermometer can also be carried out by DKD calibration or official certification, or by a comparison with thermometers that are traced to the national standard (under defined conditions).

4. Gravimetric test

1. Determine temperature of the water for testing. ⇒ Enter temperature into Test Record (4).
2. Fill the instrument to the upper stop („Fill“).
3. Dispense 5 drops into a separate vessel and wipe off the titrating tube.
4. Set display to zero („Clear“).
5. Place receiving vessel upon the balance. Tare the balance.
6. Place recipient under the titrating tube. Dispense the entire nominal volume without interruption (the titrating tube should not touch the inner wall of the recipient). ⇒ Dispense with both hands to facilitate smooth dispensing without interruption.
7. Wipe titrating tube at the receiving vessel.
8. Place receiving vessel upon the balance. Write down the value. ⇒ Enter weighing value into the Test Record (5).
9. Tare the balance again.
10. Repeat points 2 to 9 another ten times.
11. Then carry out ten more weighings each by dispensing 50% resp. 10% of the nominal capacity. ⇒ For partial volumes it is not necessary to refill each time before dispensing.

5. Evaluation of gravimetric test results

The values obtained by weighing during the gravimetric test are only the mass values of dispensed volume without correction of air buoyancy. In order to obtain the actual volume, an adjustment calculation to account for water density and air buoyancy must

be carried out. To facilitate your calculations and evaluations, we recommend the use of the Windows-compatible calibration software EASYCAL™ from BRAND.

The following calculations must be carried out:

1. Mean weighing values:

(Example for ten weighing values)

$$\bar{x} = \frac{x_1 + x_2 + x_3 + x_4 + x_5 + x_6 + x_7 + x_8 + x_9 + x_{10}}{10}$$

2. Mean volume:

$$\bar{V} = \bar{x} \cdot Z$$

⇒ For factor Z, see Table 1.

⇒ Enter value into Test Record (6a).

3. Standard deviation volume:

$$s = Z \cdot \sqrt{\frac{(x_1 - \bar{x})^2 + (x_2 - \bar{x})^2 + (x_3 - \bar{x})^2 + (x_4 - \bar{x})^2 + \dots + (x_{10} - \bar{x})^2}{9}}$$

⇒ For factor Z, see Table 1

⇒ Enter value into Test Record (6b)

4. Accuracy:

$$A [\%] = \frac{\bar{V} - V_{\text{nominal value}}}{V_{\text{nominal value}}} \cdot 100$$

⇒ Enter value into Test Record (6c)

5. Coefficient of variation:

$$CV [\%] = \frac{s \cdot 100}{\bar{V}}$$

⇒ Enter value into Test Record (6d)

Comparison actual/nominal values:

- Use the error limits per Table 2 and 3, or define your own error limits.

⇒ Enter values into Test Record (6e, f)

Result:

If calculated values A [%] and CV [%] are smaller than or equal to the tolerance limits of 0.3 %, the instrument is in good working order.

If the calculated values are **larger** than the error limits:

- Verify if the above instructions have been carefully followed step by step.
- Observe the suggestions under "Troubleshooting" in the Operating Manual.
- Calibrate the Titrette® and Digital Burette according to the instructions in the Operating Manual.

If these measures are not successful, we offer you the possibility to have your instruments calibrated by the BRAND Calibration Service (see page 9).

Possible volume faults and resulting measures:

Fault	Possible causes	Measures
Volume too large	<ul style="list-style-type: none"> ■ Drop remaining on the titrating tube ■ Dispensed too fast or unevenly 	<p>⇒ Before weighing, wipe off any drop into the receiving vessel. Tare the balance.</p> <p>⇒ Repeat the test and dispense more slowly.</p>
Volume too small	<ul style="list-style-type: none"> ■ Dispensing with media which form deposits in the piston ■ Filling valve/titrating tube leaking 	<p>⇒ Clean the instrument with a suitable agent according to the medium used.</p> <p>⇒ Tighten the filling valve/titrating tube (see Operating Manual) or clean it. Replace if necessary.</p>
Other causes	<ul style="list-style-type: none"> ■ Instrument calibrated wrongly (<CAL> appears on the display) ■ Jerky titration ■ Temperature adjustment between instrument, room and water temperature not completed 	<p>⇒ Revert to original calibration (see Operating Manual).</p> <p>⇒ Turn wheels evenly and without exerting any pressure.</p> <p>⇒ Carry out temperature adjustment.</p>

Table 1:

Excerpt from DIN EN ISO 8655

Table refers to 1013 hPa

The validity range 980 hPa to 1040 hPa

Temperature °C	Factor Z ml/g	Temperature °C	Factor Z ml/g
15	1.0020	23	1.0035
15.5	1.0020	23.5	1.0036
16	1.0021	24	1.0038
16.5	1.0022	24.5	1.0039
17	1.0023	25	1.0040
17.5	1.0024	25.5	1.0041
18	1.0025	26	1.0043
18.5	1.0026	26.5	1.0044
19	1.0027	27	1.0045
19.5	1.0028	27.5	1.0047
20	1.0029	28	1.0048
20.5	1.0030	28.5	1.0050
21	1.0031	29	1.0051
21.5	1.0032	29.5	1.0052
22	1.0033	30	1.0054
22.5	1.0034		

Table 3:

Excerpt from DIN EN ISO 8655, part 3

Nominal volume ml	Error margins for systematic error		Error margins for random error	
	± % [R]	± µl	% [VK]	µl [s]
25	0.2	50	0.1	25
50	0.2	100	0.1	50

Test:

10 single measurements per test volume according to DIN EN ISO 8655. For definition of A, CV and s see sample calculation chapter 5. For checking of partial volumes the values for A [%] and CV [%] which are related to the nominal volume have to be converted.

Table 2:

Error limits for Titrette® and Digital Burette:

The stated error limits refer to new instruments under optimized testing conditions (qualified operators and standardized ambience conditions).

Volume ml	Partial volume, ml	Titrette® bottle-top burette				Digital Burette (all models) bottle-top burette			
		R ≤± %	µl	VK ≤ %	µl	R ≤± %	µl	VK ≤ %	µl
25	25	0.07	18	0.025	6	0.2	50	0.1	25
	12.5	0.14	18	0.05	6	0.4	50	0.2	25
	2.5	0.70	18	0.25	6	2	50	1	25
50	50	0.06	30	0.02	10	0.2	100	0.1	50
	25	0.12	30	0.04	10	0.4	100	0.2	50
	5	0.60	30	0.20	10	2	100	1	50

For calibration, the error limits to be observed by the operator must be individually defined by the user. For this purpose, the following methods can be applied:

- If required by the application and if the optimized conditions for measuring are present, the error limits in Table 2 can also be expected in the case of used volumetric instruments in good working order.
- In analogy to the German regulations for official testing, it is also admissible to apply the limits which are typical for practice. These practice limits correspond to double the limits for official testing. In this case, the values found in Table 2 should be **doubled**.
- The user may also define his own individual error limits corresponding to his particular application, and apply these tolerances for the calibration of his instrument.

The above procedures fulfil the requirements of DIN EN ISO 9001, DIN EN ISO 10012 and DIN EN ISO/IEC 17025

Test Record for Volumetric Instruments

1. Instrument: <input type="checkbox"/> Titrette® <input type="checkbox"/> Digital Burette <input type="checkbox"/> Dispensette® <input type="checkbox"/> Transferpette® <input type="checkbox"/> Transferpette® <input type="checkbox"/> Transferpette® electronic <input type="checkbox"/> Transferpettor <input type="checkbox"/> _____	Type: <input type="checkbox"/> fix <input type="checkbox"/> analog <input type="checkbox"/> digital Nominal capacity: _____ Serial number: _____ Customers identification: _____
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2. Damage: None
 Type of damage: _____
 Damage repaired

3. Functional defects: None
 Type of functional defect: _____
 Functional defect repaired

4. Water temperature: _____ °C
Balance: _____
Thermometer: _____

5. Results of gravimetric test

Weighing No.	Nominal volume	50 %	10 %
x ₁			
x ₂			
x ₃			
x ₄			
x ₅			
x ₆			
x ₇			
x ₈			
x ₉			
x ₁₀			

6. Evaluation of gravimetric test

Procedure	Nominal volume	50 %	10 %
a	\bar{V}		
b	s		
c	A [%] Found		
d	CV [%] Found		
e	A [%] Nominal		
f	CV [%] Nominal		
g	result		

The testing was carried out according to DIN EN ISO 8655.

Date

Signature

