Titrette® and Digital Burette

(all models)

Testing Instructions (SOP)

April 2019

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. In the instructions in chapters 2–5, references are made to the respective fields or items in the test report on page 7, in order to simplify the collection of the relevant data. These references are shown in *italics*.

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.

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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 wich 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	Possibl safety risk - therefore replace parts (see Operating Manual "Accessories").
Leakages	Possibl 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 (according to ISO 3696, at least quality 3).
- 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 distiled or deionised water (according to ISO 3696, at least quality 3, room temperature). ⇒ Match temperature of water and room.
- **Recipient** (e.g., Erlenmeyer flask, narrow-mouth) filled with some water. ⇒ Bottom of vessel should be covered.
- **Thermometer** with a measuring error of maximum: ± 0.1 °C
- Place the instrument into the testing room for at least 1 hour (unpacked!). ⇒ Allow instrument to adjust to room temperature.
- **Balance,** recommended specifications according to ISO 8655-6:

Selected volume of appartatus under test V	Resulution mg	Repeatability and linearity mg	Standard uncertainty of measurement mg
10 ml	0.1	0.2	0.2
25 ml < V ≤ 50 ml	1	2	2

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 DAkkS calibration or official certification of the balance, or by calibrating the balance with appropiate 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 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:

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

⇒ 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 error limits, 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 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	■ Drop remaining on the titrating tube	⇒ Before weighing, wipe off any drop into the receiving vessel. Tare the balance.
	■ Dispensed too fast or unevenly	⇒ Repeat the test and dispense more slowly.
Volume too small	■ Dispensing with media which form deposits in the piston	⇒ Clean the instrument with a suitable agent according to the medium used.
	■ Filling valve/titrating tube leaking	⇒ Tighten the filling valve/titrating tube (see Operating Manual) or clean it. Replace if necessary.
Other causes	■ Instrument calibrated wrongly (<CAL> appears on the display)	⇒ Revert to original calibration (see Operating Manual).
	■ Jerky titration	⇒ Turn wheels evenly and without exerting any pressure.
	■ Temperature adjustment between instrument, room and water temperature not completed	⇒ Carry out temperature adjustment.

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 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).

		Titrette® bottle-top burette				Digital Burette (all models) bottle-top burette			
Volume ml	Partial volume, ml	A ≤± %	µl	CV ≤ %	µl	A ≤± %	µl	CV ≤ %	µl
10	10	0.10	10	0.05	5	0.3	30	0.1	10
	5	0.20	10	0.10	5	0.6	30	0.2	10
	1	1.00	10	0.50	5	3	30	1	10
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

Table 3:

Excerpt from DIN EN ISO 8655, part 3

Nominal volume	Error margins for systematic error		Error margins for random error	
ml	± % [A]	± µl	% [CV]	µl [s]
10	0.3	30	0.1	10
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.

Test Record for Volumetric Instruments

1. Instrument:

- ❑ Titrette®
- ❑ Digital Burette
- ❑ Dispensette®
- ❑ Transferpette®
- ❑ Transferpette®
- ❑ Transferpette® electronic
- ❑ Transferpettor
- ❑ _____

Type:

- ❑ fix
- ❑ analog
- ❑ digital

Nominal capacity: _____

Serial number: _____

Customers identification: _____

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.	At nominal volume in mg	At 50 % in mg	At 10 % in mg
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		At nominal volume	At 50 %	At 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**

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6. Declaration on the Absence of Health Hazards

To be sent together with the instruments or via Mail (if urgent by **Fax in advance**).

To
BRAND GMBH + CO KG
Otto-Schott-Straße 25
97877 Wertheim

Fax: 09342 808-91290

We are legally bound to give our staff a maximum of protection from health hazards caused by contaminated instruments. We therefore ask for your understanding that we cannot carry out any calibration/ repair unless this declaration is submitted completed and signed.

Media used:

❒ Acids ❒ Bases
❒ Solvents ❒ Serum, blood
❒ .....

Decontamination:

If there is a risk of contamination: What decontamination method was used?

❒ Autoclave ❒ Ethylene oxide
❒ Formaldehyde ❒ .....

Re: Instrument Consignment dated _____ / for Delivery Note no. _____

The Undersigned hereby declares:

- That the instruments have been carefully cleaned and decontaminated before shipment.
- That the instruments pose no danger through bacteriological, chemical, radiological or viral contamination.
- To be authorised to make declarations on behalf of the Institution represented.
- For calibrating service only: minor repairs of a value up to € 25,- + VAT will be carried out and invoiced without further queries (cross out if not applicable).

Company / Laboratory (Stamp)

Tel. / Fax / E-Mail

_____
Name

_____
Position

_____
Date, Signature

- In case of Return for Repair, please provide us with the following supplementary information:

Detected defect _____

Media which the instrument has been used with: _____

7. Calibration Service from BRAND

BRAND offers a full service including calibration and adjustment of Brand- and foreign instruments as well as maintenance and repair if necessary - only for BRAND- instruments. This saves money and adds the benefit of an independent review organisation for the calibration of the instruments. Further information and the order form for repair- and calibration service are found on www.brand.de.

7.1 Range of instruments covered

1. Piston-operated pipettes (single- and multichannel)
2. Bottletop dispensers
3. Piston burettes (bottle-top burettes)
4. Hand dispensers

7.2 Testing according to DIN EN ISO 8655

At BRAND, a team of qualified staff, working in temperature and humidity controlled rooms using the most modern balances and calibration software, calibrates Liquid Handling instruments, regardless of their make, according to DIN EN ISO 8655.

Instruments with adjustable volumes such as HandyStep® electronic, Transferpette®, Transferpette® *S,* Transferpette® electronic, Transferpette®S -8/-12, Transferpette®S -8/-12. Transferpettor or Dispensette®, Digital Burette or Titrette® are tested at nominal volume, and at 50%, 10% or 20% of nominal volume.

To document the results, a detailed test report is compiled which meets the requirement of different testing procedures.

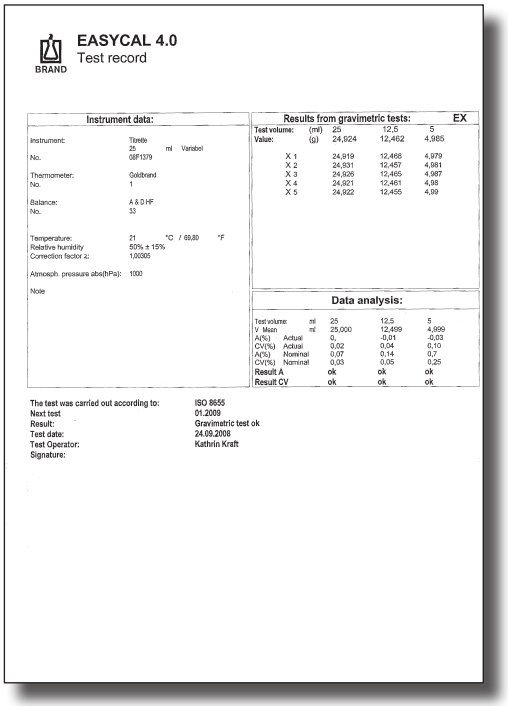
The BRAND Calibration Service offers:

1. Calibration of Liquid Handling instruments, regardless of their make
2. Detailed calibration certificate
3. Return of instrument within a few working days
4. Cost-efficient handling

8. EASYCAL™ Software – advanced calibration technology

8.1 For liquid handling instruments and glass or plastic volumetric instruments

EASYCAL™ simplifies the tedious task of calibrating liquid handling instruments and glass or plastic volumetric instruments to DIN EN ISO 9001, DIN EN ISO 10012, DIN EN ISO/IEC 17025 and GLP standards. The procedures are outlined step-by-step, and all calculations are performed automatically. Reports are generated to document the calibration. All you need is an analytical balance, a PC Windows® 98/2000, NT (SP6), XP, Vista, 7, printer (optional) and EASYCAL™ software.



EASYCAL 4.0
Test record

BRAND

Instrument data:

Instrument:	Titrette		
	25	ml	Variabel
No.	08F1379		
Thermometer:	Goldbrand		
No.	1		
Balance:	A & D HF		
No.	33		
Temperature:	21	°C / 69,80	°F
Relative humidity	50% ± 15%		
Correction factor z:	1,00305		
Atmosph. pressure abs(hPa):	1000		
Note			

Results from gravimetric tests: EX

Test volume:	(ml)	25	12,5	5
Value:	(g)	24,924	12,462	4,985
X 1		24,919	12,468	4,979
X 2		24,931	12,457	4,981
X 3		24,926	12,465	4,987
X 4		24,921	12,461	4,98
X 5		24,922	12,455	4,99

Data analysis:

Test volume:	ml	25	12,5	5
V. Mean	ml	25,000	12,499	4,999
A(%)	Actual	0,	-0,01	-0,03
CV(%)	Actual	0,02	0,04	0,10
A(%)	Nominal	0,07	0,14	0,7
CV(%)	Nominal	0,03	0,05	0,25
Result A		ok	ok	ok
Result CV		ok	ok	ok

The test was carried out according to:	ISO 8655
Next test	01.2009
Result:	Gravimetric test ok
Test date:	24.09.2008
Test Operator:	Kathrin Kraft
Signature:	

- Suitable for instruments from all manufacturers.
- Specifications of many instruments preloaded.
- Testing according to ISO 4787, ISO 8655, etc.

8.2 Data Entry

- Connect PC and balance (optional), then start the EASYCAL™ software.
- 100 common balances are preprogrammed for ease of installation.

8.3 Documentation – clearly arranged

The calibration certificate contains all important test data on one page.

9. DAkkS-Calibration Service for Volumetric Instruments at BRAND

9.1 DAkkS – Deutsche Akkreditierungsstelle GmbH and DKD

ilac-MRA DAkkS Deutsche Akkreditierungsstelle D-K-18572-01-00

The German Calibration Service (DKD) was founded in 1977 as a joint task of state and economy and constitutes the link between the measuring equipment in industrial and research laboratories, testing institutions and authorities and the national standards of the PTB (the German Institute of Physics and Metrology). It effectively supplements the existing verification system which serves above all the purposes of consumer protection. Based on the legal requirements the DKD Accreditation was successively transformed to the DAkkS Accreditation (Deutsche Akkreditierungsstelle GmbH), starting from 2010. BRAND has been accredited by the DAkkS since Apr. 23, 2013, with the certificate number D-K-18572-01-00.


Deutsche Akkreditierungsstelle GmbH

Akkreditierung


BRAND GMBH + CO KG

Deutsche Akkreditierungsstelle GmbH

Deutschen Kalibrierdienst

9.2 DAkkS-Calibration Certificate and Calibration Symbol

The DAkkS-Calibration Certificate documents officially on a high level the traceability of measuring results to national and international standards and to international SI-units, as required by standards as DIN EN ISO 9001 and DIN EN ISO/IEC 17025 for monitoring of measuring devices.

DAkkS-Calibration Certificates are issued when calibrations of an accredited laboratory are requested, when high level calibrations are necessary, when national and international standards are demanded and when reference instruments have to be calibrated.

9.3 DAkkS – A member in the International Accreditation Network

DAkkS is a member of the **International Laboratory Accreditation Cooperation (ILAC),** the highest level international institution for laboratory calibration, and is a signatory to the MRA – Mutual Recognition Agreements.

The accreditation bodies that are signatories to the ILAC mutual recognition agreements (MRAs) recognize their mutual equivalence, and the equivalence of the calibration certificates issued by those same signatories. Likewise, signatories are obliged generally to promote and recommend recognition of the calibration certificates of other signatories (excluding factory calibration certificates).

The DAkkS is a member of the EA (European Co-operation for Accreditation), which again is a member of the ILAC (International Laboratory Accreditation Cooperation). A multilateral agreement assures obligatory recognition of the DAkkS calibration certificate in a variety of countries.

9.4 DAkkS-Calibration Laboratory at BRAND

In 1998 a calibration laboratory for volumetric instruments at BRAND has been accredited by the German Calibration Service according to DIN EN ISO/ IEC 17 025. Our calibration laboratory is therefore authorized to issue DAkkS-Calibration Certificates (in several languages) for the volumetric instruments listed below. Furthermore we offer adjustment and – for BRAND Liquid Handling instruments – repair and maintenance.

For ordering information on DAkkS-Calibration Certificates for volumetric instruments please consult our General Catalog.

9.5 Volumetric instruments for which BRAND issues DAkkS Calibration Certificates

BRAND calibrates the following volumetric instruments (new or already in use and regardless of their make):

- **Piston-operated pipettes,** from 0.1 µl to 10 ml
- **Multichannel piston-operated pipettes,** from 0.1 µl to 300 µl
- **Piston-operated burettes,** from 5 µl to 200 ml
- **Dispensers, Dilutors,** from 5 µl to 200 ml
- **Volumetric instruments of glass,** calibrated to contain (TC, In) from 1 µl to 10000 ml
- **Volumetric instruments of glass,** calibrated to deliver (TD, Ex) from 100 µl to 100 ml
- **Volumetric instruments of plastic,** calibrated to contain (TC, In) from 1 ml to 2000 ml
- **Volumetric instruments of plastic,** calibrated to deliver (TD, Ex) from 1 ml to 100 ml
- **Density bottles of glass,** from 1 cm^3 to 100 cm^3

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