

Digital Burette III

Testing Instructions (SOP)

July 2004

1. Introduction

The standard ISO DIS 8655 describes both the design and the testing of the piston operated burette Digital Burette III. The following Testing Instructions describe how to apply the ISO standard in practice.

We recommend a testing of the Digital Burette III 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 back of instrument). ⇒ Enter number in Test Record (1).
- ☞ Read customer's identification, if present. ⇒ Enter number in Test Record (1).
Purpose: Clear identification of each instrument.

2.2 Minimal configuration of the Digital Burette III

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

2.3 Cleaning

- ☞ Rinse instrument with cleaning solution. ⇒ Select suitable cleaning solution according to the medium
Following rinse the instrument again with distilled water. which was used.
- ☞ Wipe off the exterior of the Digital Burette III. ⇒ Connection glass cylinder – valve block.
⇒ Connection valve – titrating tube.

2.4 Visual examination for damage (Scratches, cracks, major mechanical damage)

- Titrating tube ⇒ Enter findings in Test Record (2).
- Filling tube
- Connection valve block – discharge valve
- Connection valve block – filling valve
- Connection valve block – glass cylinder
- Connection filling valve – filling tube
- Connection valve block – titrating tube
- Display
- Housing
- Switches

Possible faults and resulting measures:

Fault	Measures
Filling or titrating tube bent or damaged	☞ Safety risk – therefore replace part (see Operating Manual "Accessories").
Mechanically damaged connections	☞ Safety risk – therefore return instrument for repair.
Faulty operating or display elements	☞ Return instrument for repair.

2.5 Temperature adjustment

- ☞ Place the Digital Burette III into the testing room for at least 1 hour (unpacked!).
- ⇒ Allow instrument to adjust to room temperature.

2.6 Required equipment for testing

- **Bottle** (500 ml) filled with distilled or deionized water (room temperature). ⇒ Match temperature of room, water and instrument.
- **Recipient** (e.g. Erlenmeyer flask) filled with some water. ⇒ Bottom of vessel should be covered.
- **Balance** with accuracy: 0.001 g
- **Thermometer** with accuracy: 0.2 °C

Referring the test procedure to the national standard:

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 officially certified weights (accuracy F1). The calibration of the thermo-meter can also be carried out by DKD calibration or official certification, or by a comparison with officially certified thermometers (under defined conditions).

3. Functional test

3.1 Prepare test setup

- ☞ Mount titrating tube.
- ☞ Mount filling tube.
- ☞ Mount the Digital Burette III onto the bottle.
- ☞ Prime the Digital Burette III (see 3.2).

3.2 Priming the Digital Burette III

The Digital Burette III has to be **primed**.

- ⇒ See Operating Manual.
- ⇒ A few small bubbles in the glass cylinder are acceptable.
- ⇒ Enter findings into Test Record (3).

During priming, examine the following functions:

- ☞ Display in order? ⇒ "fill" - "titr".
- ☞ Switches and hand wheel operating smoothly? ⇒ See Operating Manual.

Possible faults and resulting measures:

Faulty	Possible causes	Measures
Instrument cannot be primed	<ul style="list-style-type: none"> ○ Filling valve leaking ○ Titrating tube leaking ○ Filling tube leaking 	<ul style="list-style-type: none"> ☞ Unscrew filling valve and check if seal is inserted. Mount filling valve firmly (use mounting tool). ☞ Unscrew titrating tube and check if seal is inserted. Mount titrating tube firmly. ☞ Push on firmly filling tube. If necessary cut off approx. 1 cm of tube at the upper end or replace filling tube.
Leaks	<ul style="list-style-type: none"> ○ Connection valve block - glass cylinder leaking ○ Piston seal damaged or leaking (liquid inside the glass cylinder above the seal) ○ Seal missing or damaged 	<ul style="list-style-type: none"> ☞ Return instrument for repair. ☞ Return instrument for repair. ☞ Insert or replace seal.
Operating or display elements damaged or stiff		<ul style="list-style-type: none"> ☞ Return instrument for repair.

4. Gravimetric test

1. Determine temperature of the water for testing. ⇒ Enter temperature into Test Record (4).
2. Fill the Digital Burette III to the upper stop ("Fill").
3. Switch to titrating mode ("Titr").
4. Compensate for backlash. ⇒ Dispense 5 drops into a separate vessel.
⇒ Any drop on the titrating tube? Wipe off before dispensing.
5. Set display to zero ("Clear").
6. Place recipient upon the balance. Tare the balance.
7. 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.
⇒ Any drop on the titrating tube? Wipe off before weighing.
8. Place recipient upon the balance. Write down the value.
9. Tare the balance again.
10. Repeat points 2 to 9 another five times. ⇒ Enter weighing values into the Test Record (5).
11. Then carry out five 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 the dispensed volume. In order to obtain the actual volume, an adjustment calculation 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 weight:**

$$\bar{x} = \frac{x_1 + x_2 + x_3 + x_4 + x_5}{5}$$

2. **Mean volume:**

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

⇒ For factor z, see Table 1.
⇒ Enter value into Test Record (6a).

3. **Standard deviation:**

$$s = z \cdot \sqrt{\frac{(x_1 - \bar{x})^2 + (x_2 - \bar{x})^2 + (x_3 - \bar{x})^2 + (x_4 - \bar{x})^2 + (x_5 - \bar{x})^2}{4}}$$

⇒ 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 tolerance limits per Table 2 and 3, or define your individual tolerance limits.

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

Result:

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

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

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

If these measures are not successful, you may send the instrument to the manufacturer for calibration.

Possible 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 	<ul style="list-style-type: none"> ☞ Before weighing, wipe off any drop into the recipient, then tare the balance. ☞ Repeat the test and dispense more slowly.
Volume too small	<ul style="list-style-type: none"> ○ Dispensing with media which form deposits in the piston ○ Filling valve/titrating tube leaking ○ Backlash compensation not carried out 	<ul style="list-style-type: none"> ☞ Clean the instrument with a suitable agent according to the medium used. ☞ Tighten the filling valve/titrating tube (see Operating Manual) or clean it. Replace if necessary. ☞ Carry out backlash compensation (see point 4.4).
Other causes	<ul style="list-style-type: none"> ○ Instrument calibrated to different medium (<CAL> appears on the display) ○ Jerky movement of handwheels during titration ○ Temperature adjustment between instrument, room and water temperature not completed 	<ul style="list-style-type: none"> ☞ Revert to original calibration (see Operating Manual). ☞ Turn wheels evenly and without exerting any pressure. ☞ Carry out temperature adjustment.

Table 1:

Excerpt from DIN EN ISO 8655.
Table refers to 1013 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:

Error margins (EM) according to DIN EN ISO 8655.

Nominal volume ml	Error margins for systematic error		Error margins for random error	
	± % [A]	± µl	% [CV]	µl [s]
≤ 1	0.6	6	0.1	1
2	0.5	10	0.1	2
5	0.3	15	0.1	5
10	0.3	30	0.1	10
20	0.2	40	0.1	20
25	0.2	50	0.1	25
50	0.2	100	0.1	50
100	0.2	200	0.1	100

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.

Table 2:

Volume tolerances for the Digital Burette III:

The stated volume tolerances are final test values relative to the nominal capacity. These tolerances refer to new instruments under optimized testing conditions (qualified operators and standardized ambience conditions).

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 stated tolerances 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 tolerance 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.

Nominal volume ml	Accuracy* Value 6e		Coefficient of variation* Value 6f	
	≤ ± %	µl	≤ %	µl
50 (Nominal vol.)	0.2	100	0.1	50
25 (50%)	0.4	100	0.2	50
5 (10%)	2.0	100	1.0	50
25 (Nominal vol.)	0.2	50	0.1	25
12.5 (50%)	0.4	50	0.2	25
2.5 (10%)	2.0	50	1.0	25

* **Note:**
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:

- Digital Burette
- Dispensette®
- Transferpette®
- Transferpette®-8/-12
- Transferpette®
-

Type:

- fix
- variable
- digital
- EASY CALIBRATION

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.	Nominal volume	50 %	10 %
X ₁			
X ₂			
X ₃			
X ₄			
X ₅			

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:

.....

.....



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
Germany

Fax: +49 9342 808-354

We intend 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.

For consignment dd. / 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.
- That he/she is aware that shipment of contaminated instruments is a violation of law, and that he/she personally and the Institution represented may be held liable for any damages caused by contaminated instruments.
- 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 for Liquid Handling Instruments

BRAND offers full calibration service including instrument adjustment and repair if necessary. Instruments are normally returned within three working days. This saves money and adds the benefit of an independent review organisation for the calibration of the instruments. Calibrations can be arranged on an individual basis or as scheduled testings within the scope of a continual calibration agreement.

7.1 Instrument spectrum:

1. Piston-operated pipettes (single- and multichannel)
2. Bottletop dispensers
3. Digital bottletop burettes
4. Handdispensers

7.2 Testing according to DIN EN ISO 8655

At BRAND, a team of qualified personnel in temperature and humidity controlled rooms using the most modern balances and calibration software, calibrates liquid handling instruments, regardless of their make, according to ISO 8655 (resp. DIN 12 650).

Instruments with adjustable volumes such as Transferpette® or Dispensette® are tested at nominal volume, at 50%, 10% or 20% of the nominal volume.

To document the calibration a detailed test report is generated which fully meets the requirements of different testing procedures.

The BRAND Calibration Service offers:

1. Calibration of liquid handling instruments regardless of their make
2. Cost-efficient handling
3. Return of instrument within few working days
4. Detailed test report

7.3 Calibration Agreement

For regular calibration of liquid handling instruments at our facilities, BRAND offers an Agreement for Calibration Service which covers the following topics:

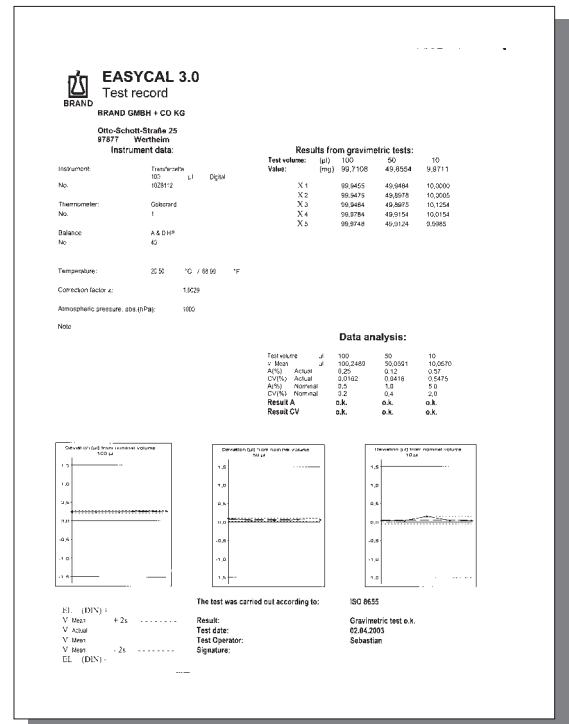
- Pick-up and return of instruments
- Number and type of instruments to be calibrated
- Monitoring of confirmation intervals (you are automatically reminded when calibration is due).

8. EASYCAL™ 3.0 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® 95/98/2000 or Windows® NT, printer (optional) and EASYCAL™ 3.0 software.

- 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™ 3.0 software.
- ☞ 40 common balances are preprogrammed for ease of installation.

8.3 Documentation – clearly arranged

The test record contains all important test data on one page, including a graphic representation of test results.

9. DKD-Calibration from BRAND for Volumetric Instruments

9.1 DKD – German Calibration Service

DKD
DKD-K-20701

The 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.



9.2 DKD-Calibration Certificate and DKD-Calibration Symbol

The DKD-Calibration Certificate with the DKD-Calibration Symbol 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, DIN EN ISO 10012, DIN EN ISO/IEC 17025 and DIN EN 45000 for monitoring of measuring devices.

The major difference between traditional works calibration services and PTB-accredited calibration laboratories is the accurate determination of uncertainty of each measurement, guaranteed by the accredited laboratory and supervised by the PTB.

DKD-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 Internationally recognized

The DKD is member of the European Cooperation for Accreditation (EA). A multilateral agreement assures obligatory recognition of the DKD Calibration Certificate in a variety of countries. In addition, a „Mutual Recognition Arrangement“ (MRA) has been signed in Washington, DC, on 2 November 2000. This new arrangement, which involves 36 member bodies from 28 economies represented at the General Assembly of the International Laboratory Accreditation Cooperation (ILAC, www.ilac.org), has enabled accredited laboratories, as such accredited by PTB in Germany, to achieve a form of international recognition, and allowed test data accompanying exported goods to be more readily accepted on overseas markets. This is a major step towards reducing or eliminating the need for re-testing of the goods by the importing country.

9.4 DKD-K-20701

In 1998 a calibration laboratory for volumetric instruments at BRAND has been accredited by PTB according to DIN EN 45001, registration number DKD-K-20701. Our calibration laboratory is therefore authorized to issue DKD Calibration Certificates for the volumetric instruments listed below. Calibration is available for new instruments directly from the factory as well as for those already in use – regardless of their make. Furthermore we offer adjustment and – for BRAND-products – repair. For ordering information on DKD Calibration Certificates for volumetric instruments please consult our General Catalog 600.

9.5 Volumetric instruments for which you can obtain DKD Calibration Certificates from BRAND

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**, adjusted to contain (TC, In) from 1 µl to 10000 ml
- **Volumetric instruments of glass**, adjusted to deliver (TD, Ex) from 100 µl to 100 ml
- **Volumetric instruments of plastic**, adjusted to contain (TC, In) from 1 ml to 2000 ml
- **Volumetric instruments of plastic**, adjusted to deliver (TD, Ex) from 1 ml to 100 ml
- **Density bottles of glass**, from 1 cm³ to 100 cm³

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