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Mode d'emploi

Ubbelohde viscometer

Wichtige Hinweise: Die Gebrauchsanleitung vor der ersten Inbetriebnahme des Ubbelohde Viskosimeters und des Mikro- Ubbelohde Viskosimeters bitte sorgfältig lesen und beachten. Aus Sicherheitsgründen darf das Ubbelohde Viskosimeter und das Mikro- Ubbelohde Viskosimeter ausschließlich nur für die in dieser Gebrauchsanleitung beschriebenen Zwecke eingesetzt werden.

Bitte beachten Sie auch die Gebrauchsanleitungen für die anzuschließenden Geräte.

Alle in dieser Gebrauchsanleitung enthaltenen Angaben sind zum Zeitpunkt der Drucklegung gültige Daten. Es können jedoch von SCHOTT sowohl aus technischen und kaufmännischen Gründen, als auch aus der Notwendigkeit heraus, gesetzliche Bestimmungen der verschiedenen Länder zu berücksichtigen, Ergänzungen am Ubbelohde Viskosimeter und am Mikro- Ubbelohde Viskosimeter vorgenommen werden, ohne dass die beschriebenen Eigenschaften beeinflusst werden.

Important notes: Before initial operation of the Ubbelohde Viscometer and of the Micro- Ubbelohde Viscometer please read and observe carefully the operating instructions. For safety reasons the Ubbelohde Viscometer and the Micro- Ubbelohde Viscometer may only be used for the purposes described in these present operating instructions.

Please also observe the operating instructions for the units to be connected.

All specifications in this instruction manual are guidance values which are valid at the time of printing. However, for technical or commercial reasons or in the necessity to comply with the statuary stipulations of various countries, SCHOTT may perform additions to the Ubbelohde Viscometer and to the Micro- Ubbelohde Viscometer without changing the described properties.

Instructions importantes: Prière de lire et d'observer attentivement le mode d'emploi avant la première mise en marche du Viscosimètre selon Ubbelohde et du Micro Viscosimètre selon Ubbelohde. Pour des raisons de sécurité, le Viscosimètre selon Ubbelohde et le Micro Viscosimètre selon Ubbelohde pourront être utilisés exclusivement pour les usages décrits dans ce présent mode d'emploi.

Nous vous prions de respecter également les modes d'emploi pour les appareils à connecter.

Toutes les indications comprises dans ce mode d'emploi sont données à titre indicatif au moment de l'impression. Pour des raisons techniques et/ou commerciales ainsi qu'en raison des dispositions légales existantes dans les différents pays, SCHOTT se réserve le droit d'effectuer des suppléments concernant le Viscosimètre selon Ubbelohde et le Micro Viscosimètre selon Ubbelohde qui n'influencent pas les caractéristiques décrits.

Instrucciones importantes: Primeramente, lean y observen atentamente el manual de instrucciones antes de la primera puesta en marcha del Viscosímetro de Ubbelohde et del Viscosímetro de Micro- Ubbelohde. Por razones de seguridad, el Viscosímetro de Ubbelohde y el Viscosímetro de Micro- Ubbelohde debe ser empleado para los objetivos descritos en este manual de instrucciones.

Por favor, respeten las indicaciones descritas en los manuales de instrucciones de los equipos antes de conectarlos.

Todos los datos contenidos en este manual de instrucciones son datos orientativos que están en vigor en el momento de la impresión. Por motivos técnicos y/o comerciales, así como por la necesidad de respetar normas legales existentes en los diferentes países, SCHOTT puede efectuar modificaciones concernientes al Viscosímetro de Ubbelohde y al Viscosímetro de Micro- Ubbelohde sin cambiar las características descritas.

Viscometers within quality assurance systems

Recommendations for companies that have introduced a quality assurance system in accordance with the DIN/ISO 9000 ff resp. EN 29 000 ff standards. In this quality assurance system, an inspection of the measuring equipment is planned. The intervals and required accuracy can be defined by each company according to its own requirements. The standard DIN/ISO 10 012, Part 1 serves as a guideline in this matter. We recommend regular inspection of the viscometers in defined intervals.

Inspection of the viscometer constants:

1. Calibration using comparative measurements with reference measuring standards

Comparative measurements must be performed with a viscometer (reference measuring standard) which was tested at the PTB (Federal German Physical-Technical Institute) and provided with a constant. During this comparative measurement, the viscometer to be inspected and the PTB - tested viscometer were placed simultaneously in the same thermostat bath. The test liquid tested, the viscosity of which must not be known exactly, is filled into both viscometers, tempered and the flow-through time then measured.
The constants of the viscometers to be inspected are then calculated according to the following equation:

$$K = \frac{K_{Uref} \cdot t_{Uref}}{t}$$

K = constant of the tested viscometer
K_{Uref} = constant of the standard reference viscometer
t = flow time (HC) of the tested viscometer (corrected by Hagenbach-Couette)
t_{Uref} = flow time (HC) of the standard reference viscometer (corrected by Hagenbach-Couette)

Within the quality assurance system in accordance with DIN EN ISO 9000 ff, traceability of the measuring equipment to national measuring standards is demanded. This traceability can be achieved by inspecting the comparative viscometers (reference measuring standards) at regular intervals at the PTB. The time intervals are defined according to the specifications made in the quality assurance system of the user.

2. Calibration of the capillary viscometer with normal oils of the PTB

During this calibration, a normal oil from the PTB with known viscosity is used as a reference measuring standard. The measurement is performed by means of flow-through measurement of the PTB normal oil in the viscometer to be inspected in a temperature bath, the temperature of which must correspond precisely to the test temperature of the PTB. In this case, it is extremely important to make sure that the temperature is absolutely correct. In case of temperature variation, this will always result in a constant for the viscometer that deviates from the constant applied. A temperature variation of 0.01 K, for instance, will result in a measuring error of up to 0.01 %. The calibration of the deviating temperature into the viscometer constant is not permitted.

3. Inspection by SCHOTT-GERÄTE with quality certificates in accordance with DIN 55 350-18-4.2.2

The inspection at SCHOTT-GERÄTE is carried out by means of comparative measurements using viscometers as reference measuring standards that were tested at the PTB (corresponds to Item 1).

Information on the stability of viscometer constants

Each inspection (even with a certificate) can guarantee the technical measuring direction only for a limited period of time. The constants of viscometers made of the borosilicate glass DURAN®, however, can remain unchanged for long periods of time if the viscometers are kept away from altered influences. Especially extreme changes can be expected, for instance, during the use of liquids that attack glass or during glass-blowing repairs (even for apparently insignificant repairs).

Liquids whose components adhere to the glass wall also cause errors. In such cases, regular cleaning is required whereby the corrosive action cleaning agent on the glass must be eliminated.

For this reason, we recommend that the user should write up a special processing instructions for all important measurements and include them in his quality assurance manual in accordance with DIN EN ISO 9000 ff. In all cases the user is responsible for the correctness of his measuring and testing equipment and is not released from his responsibility for quality (cp. DIN 55 350, Part 18).

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Version 000514 E

Operating Instructions

Ubbelohde Viscometer

Micro-Ubbelohde Viscometer

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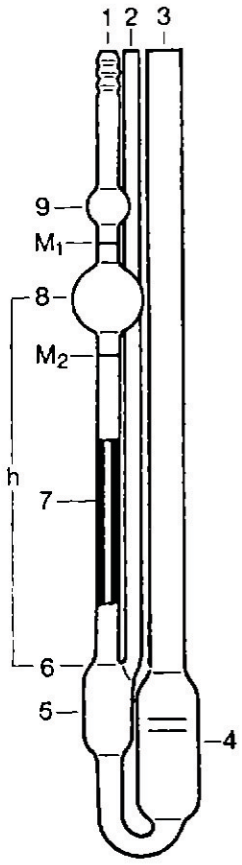


1 Kapillarrohr
Capillary tube
Tube avec capillaire
Tubo con capilar

2 Belüftungsrohr
Venting tube
Tube de ventilation
Tubo de ventilación

3 Befüllrohr
Filling tube
Tube de remplissage
Tubo de llenado

4 Vorratsgefäß
Reservoir
Réservoir
Reservorio

5 Niveaugefäß
Reference level vessel
Récipient de détente
Bola de nivel

6 Kugelkalotte
Dome-shaped top part
Calotte sphérique
Cúpula esférica

7 Kapillare
Capillary
Tube capillaire
Capilar

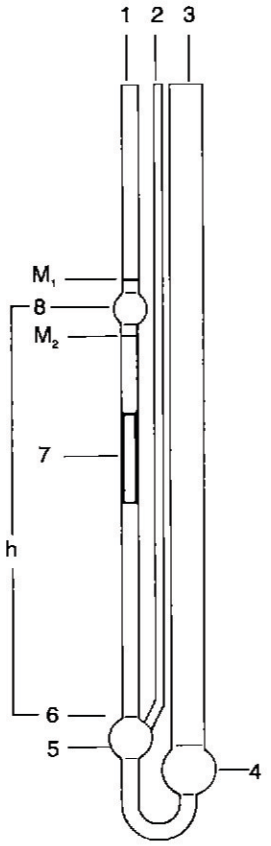
8 Messgefäß
Measuring sphere
Boule de mesure
Bola de medición

9 Vorlaufkugel
Pre-run sphere
Boule d'entrée
Bola de entrada

M_1 Obere Ringmessmarke
Upper timing mark
Marque annulaire supérieure
Marca anular superior

M_2 Untere Ringmessmarke
Lower timing mark
Marque annulaire au-dessous
Marca anular inferior

Mikro-Ubbelohde Viskosimeter
Micro- Ubbelohde Viscometer
Micro Viscosimètre selon Ubbelohde
Viscosímetro de Micro-Ubbelohde



1 Description

The viscometer basically consists of the capillary tube (1), venting tube (2) and the filling tube (3), the capillary (7) with the measuring sphere (8), the pre-run sphere (9) (for Ubbelohde Viscometers) and the reference level vessel (5). Above and below the measuring sphere (8) are printed on timing marks M_1 and M_2. These marks not only define the flow-through volume of the sample, but also the mean hydrostatic head (h). The capillary (7) ends in the upper part of the reference level vessel (5). The sample runs down from the capillary (7) as a thin film on the inner surface of the reference level vessel (5) (suspended level bulb).

The same correction seconds (kinetic energy correction. HC = Hagenbach-Couette Korrektion) apply for Ubbelohde Viscometers of SCHOTT-GERÄTE with identical capillary diameters. No re-determination of the kinetic energy correction (HC) is necessary if SCHOTT-GERÄTE viscometers of identical capillary tube diameter are exchanged.

2 Preparation of sample

Low-viscosity samples should be passed through a SCHOTT glass filter (10 ... 100 µm) before measuring; high-viscosity samples through a strainer of 0.3 mm mesh (test strainer 0.2; DIN 4188). Samples whose setting point according to DIN 51 583 or pour point according to DIN 51 597 is not at least 30 °C below the test temperature, must be heated up to 50 °C before measuring.

3 Selection of capillary

The diameter of the capillary should be selected so that the uncertainty inherent in the kinetic energy correction (HC) does not exceed the error allowed for time measurement (see table). Therefore, for precision measurements, efflux times whose correction seconds are stated in parentheses should not be applied. A selection of a viscometer with a smaller capillary diameter is suggested.

4 Cleaning of viscometer

Before first use clean with 15 % H_2O_2 and 15 % HCl. Thereafter rinse viscometer with a suitable solvent. It must be completely dry and dust-free before it is put to use for either manual or automatic measuring.

In case of fully automatic program sequence with the ViscoSystem AVS 500, the viscometer is customarily rinsed with the successive sample where the number of rinsing operations can be selected.

5 Filling of viscometer

About 15 ml of the filtered sample, in case of Ubbelohde Micro Viscometers about 2.5 ml, are transferred through the filling tube (3) into the reservoir (4). Filling capacity is indicated by filling marks on reservoir (4).

6 Temperature conditioning of sample

After filling, hang viscometer with its stand (Ref. No. 053 92) into a glass-panelled thermal liquid/constant-temperature bath of SCHOTT-GERÄTE. To obtain the measuring accuracy of the viscometer, the constant-temperature bath should maintain the set temperature at a constant ± 0.01 °C **which is possible with SCHOTT-GERÄTE Constant-Temperature Immersion Circulators.** Differences in temperature of only 0.1 °C may cause an error of as much as 0.6 % in mineral oils. Measuring should take place only after an equilibration time of approx. 10 minutes and, in case of Ubbelohde Micro-Viscometers, of approx. 5 minutes.

7 Manual measuring operation

Apply vacuum to venting tube (2), closing venting tube (2) by a finger or rubber stopper. This will cause the successive filling of the reference level vessel (5), the capillary tube (1), the measuring sphere (8), and the pre-run sphere (9). In case of the Micro- Viscometer, follow the same procedure and fill to approx. 10 mm above the upper timing mark M_1.

Now suction is discontinued and the venting tube (2) opened again. This causes the liquid column to separate at the lower end of the capillary (7) and to form the suspended level at the dome-shaped top part (6). What is measured in the time interval (flow time t) it takes the leading edge of the meniscus of the sample to descend from the upper edge of the upper timing mark M_1 to the upper edge of the lower timing mark M_2.

Note: When measuring highly viscous samples with capillary No. IV, Ivc, IVa and V, it is advisable, after opening of venting tube (2), to keep capillary tube (1) closed long enough for reference level vessel (5) to run empty and the suspended level to form. If, during measurement, venting tube (2) should be clogged by a drop of liquid, the measuring operation must be repeated. In the event of renewed clogging, the vertical axis of the capillary may be slightly modified.

8 Automated measuring operation

The automated viscosity-measuring instruments by SCHOTT-GERÄTE (see AVS brochure) replace the above described manual operation of viscosity measurement. Subjective measuring errors are eliminated, and the efflux times measured appear as a 6-digit print-out with sample identification. According to the type and number of samples to be measured, an optimum measuring device may be assembled which can be expanded to an automatic sampler. Rinsing and filling of Ubbelohde Viscometers and Ubbelohde Micro-Viscometers and measuring of sample with subsequent changing of sample is performed automatically. The timing marks required for manual measuring are replaced by light barriers.

All Ubbelohde Viscometers and Ubbelohde Micro-Viscometers of SCHOTT-GERÄTE can be used in the AVS automatic viscosity-measuring instruments by SCHOTT-GERÄTE.

9 Example of manual measuring operation

Required accuracy of measurement for test temperatures 10 ... 30 °C:	**[%]**	**± 1**	**± 0.5**	**± 0.1**
Accuracy of thermostat	[°C]	± 0.1	± 0.05	± 0.01
Accuracy of reading thermometer	[°C]	± 0.05	± 0.03	± 0.005
Equilibration time	[min]	15	15	15
Allowable deviation from vertical suspension of viscometer	[°]	4.5	3.3	1.5
Permissable parallax in viewing viscometer marking	[°]	10	5	1
Minimum flow time[1)] in capillary I in capillary II in capillary III	[s]	 250 100 100	 350 135 100	 650 240 100
Maximum permissable time measurement error	[%]	± 0.33	± 0.17	± 0.03
Number of single measurements required	[%]	4	4	4
Allowable deviations between measurements	[%]	± 1	± 0.5	± 0.1

The accuracy obtained with the AVS automated measuring system is greater since certain parameters such as errors in reading, clock errors, etc. are eliminated.

[1)] Chosen so that the uncertainty inherent in the kinetic energy correction (HC) does not exceed the error allowed for time measurement.

10 Calculation of viscosity

The number of seconds stated for the various capillaries in the tables of the kinetic energy correction (HC) are subtracted from the determined efflux time. Intermediate values may be interpolated.

For absolute measurements, the corrected flow time multiplied by the viscometer constant K gives the kinematic viscosity [mm^2/s] *) directly.

$$M = K\,(t - y)$$

The viscometer constant K is mentioned in the enclosed production certificate.

11 Example of calculation

Ubbelohde Viscometers Ref. No. 501 10

Capillary I			
Constant		= 0.01000	
Flow time (averaged)		= 180.00	[s]
Kinetic energy correction (HC) for 180.00 s	y	= 0.30	[s]
Kinematic viscosity	m	= K (t − y)	
		= 0.01 • (180.00 − 0.30)	
		= 1.797	[mm^2/s]*
*) previously centistokes [cSt];	1 [cSt]	= 1	[mm^2/s]

12 Measurements and device constants

Ubbelohde Viscometers ISO 3105 / DIN 51 562 / Part 1 / BS 188 / NFT 60-100
Ref. No. 501 ... 530 ... 532 ..

Ref. No.	Capillary No.	Capillary $\varnothing_i$ (mm)	Constant K (approx.)	Measuring range mm^2/s (cSt) (approx.)		
... 00	0	0.36	0.001	0.2	to	1.2
... 03	0c	0.46	0.003	0.5	to	3
... 01	0a	0.53	0.005	0.8	to	5
... 10	I	0.63	0.01	1.2	to	10
... 13	Ic	0.84	0.03	3	to	30
... 11	Ia	0.95	0.05	5	to	50
... 20	II	1.13	0.1	10	to	100
... 23	IIc	1.50	0.3	30	to	300
... 21	IIa	1.69	0.5	50	to	500
... 30	III	2.01	1	100	to	1000
... 33	IIIc	2.65	3	300	to	3000
... 31	IIIa	3.00	5	500	to	5000
... 40	IV	3.60	10	1000	to	10000
... 43	IVc	4.70	30	3000	to	30000
... 41	IVa	5.34	50		above	10000
... 50	V	6.40	100		above	10000

*) previously centistokes [cSt]; 1 [cSt] = 1 [mm^2/s]

13 Table of the kinetic energy correction (HC)

Ubbelohde Viscometers ISO 3105 / DIN 51 562 / Part 1 / BS 188 / NFT 60-100
Ref. No. 501 ... 530 ... 532 ..

Correction seconds[1]:

Flow time [s]	Capillary no. 0	0c	0a	I	Ic	Ia	II
40	– [2]	– [2]	– [2]	(1.03)	0.45	0.15	
50	– [2]	– [2]	– [2]	(3.96)	0.66	0.29	0.10
60	– [2]	– [2]	– [2]	(2.75)	0.46	0.20	0.07
70	– [2]	– [2]	– [2]	(2.02)	0.34	0.15	0.05
80	– [2]	– [2]	(4.78) [2]	(1.55)	0.26	0.11	0.04
90	– [2]	– [2]	(3.78) [2]	1.22	0.20	0.09	0.03
100	– [2]	(7.07) [2]	(3.06) [2]	0.99	0.17	0.07	0.02
110	– [2]	(5.84) [2]	(2.53)	0.82	0.14	0.06	0.02
120	– [2]	(4.91) [2]	2.13	0.69	0.12	0.05	0.02
130	– [2]	(4.18) [2]	1.81	0.59	0.10	0.04	0.01
140	– [2]	(3.61) [2]	1.56	0.51	0.08	0.04	0.01
150	– [2]	(3.14) [2]	1.36	0.44	0.07	0.03	0.01
160	– [2]	2.76	1.20	0.39	0.06	0.03	0.01
170	– [2]	2.45	1.06	0.34	0.06	0.02	0.01
180	– [2]	2.18	0.94	0.30	0.05	0.02	0.01
190	– [2]	1.96	0.85	0.28	0.05	0.02	0.01
200	(10.33) [2]	1.77	0.77	0.25	0.04	0.02	0.01
225	(8.20)	1.40	0.60	0.20	0.03	0.01	0.01
250	(6.64)	1.13	0.49	0.16	0.03	0.01	< 0.01
275	(5.47)	0.93	0.40	0.13	0.02	0.01	< 0.01
300	4.61	0.79	0.34	0.11	0.02	0.01	< 0.01
325	3.90	0.66	0.29	0.09	0.02	0.01	
350	3.39	0.58	0.25	0.08	0.01	0.01	
375	2.95	0.50	0.22	0.07	0.01	0.01	
400	2.59	0.44	0.19	0.06	0.01	< 0.01	
425	2.30	0.39	0.17	0.05	0.01	< 0.01	
450	2.05	0.35	0.15	0.05	0.01	< 0.01	
475	1.84	0.31	0.13	0.04	0.01		
500	1.66	0.28	0.12	0.04	0.01		
550	1.37	0.23	0.10	0.03	0.01		
500	1.15	0.20	0.09	0.03	0.01		
650	0.98	0.17	0.07	0.03	< 0.01		
700	0.85	0.14	0.06	0.02	< 0.01		
750	0.74	0.13	0.05	0.02	< 0.01		
800	0.65	0.11	0.05	0.01			
850	0.57	0.10	0.04	0.01			
900	0.51	0.09	0.04	0.01			
950	0.46	0.08	0.03	0.01			
1000	0.42	0.07	0.03	0.01			

[1] The correction seconds stated are related to the respective theoretical constant.
[2] For precision measurements, **these** flow times should not be applied.
A selection of a viscometer with a smaller capillary diameter is suggested.

14 Measurements and device constants

Ubbelohde Viscometers ISO 3105 / ASTM D 2515
Ref. No. 525 ... 526 ..

Ref. No.	Capillary No.	Capillary $\varnothing_i$ (mm)	Constant K (approx.)	Measuring range mm^2/s (cSt) (approx.)		
... 00	0	0.24	0.001	0.3	to	1
... 03	0c	0.36	0.003	0.6	to	3
... 01	0b	0.46	0.005	1	to	5
... 10	I	0.58	0.01	2	to	10
... 13	Ic	0.78	0.03	6	to	30
... 20	II	1.03	0.1	20	to	100
... 23	IIc	1.36	0.3	60	to	300
... 30	III	1.83	1	200	to	1000
... 33	IIIc	2.43	3	600	to	3000
... 40	IV	3.27	10	2000	to	10000
... 43	IVc	4.32	30	6000	to	30000

* previously centistokes [cSt]; 1 [cSt] = 1 [mm^2/s]

15 Table of the kinetic energy correction (HC)

Ubbelohde Viscometers ISO 3105 / ASTM D 2515
Ref. No. 525 ... 526 ..

Correction seconds[1]:

Flow time [s]	Capillary No. 0	0b	0c	I	Ic
50	– [2]	(5.06) [2]	(6.69) [2]	(2.45) [2]	0.41
75	– [2]	2.25	2.98	1.09	0.18
100	(3.69) [2]	1.26	1.67	0.61	0.10
125	2.36	0.81	1.07	0.39	0.07
150	1.64	0.56	0.74	0.27	0.05
175	1.21	0.41	0.55	0.20	0.03
200	0.92	0.32	0.42	0.15	0.03
225	0.73	0.25	0.33	0.12	0.02
250	0.59	0.20	0.27	0.10	0.02
275	0.49	0.17	0.22	0.08	0.02
300	0.41	0.14	0.19	0.07	0.01
325	0.35	0.12	0.16	0.06	0.01
350	0.30	0.10	0.14	0.05	0.01
375	0.26	0.09	0.12	0.04	0.01
400	0.23	0.08	0.11	0.04	0.01
425	0.20	0.07	0.09	0.03	0.01
450	0.18	0.06	0.08	0.03	< 0.01
475	0.16	0.06	0.07	0.03	< 0.01
500	0.15	0.05	0.06	0.02	< 0.01

[1] The correction seconds stated are related to the respective theoretical constant.
[2] For precision measurements, **these** flow times should not be applied.
A selection of a viscometer with a smaller capillary diameter is suggested.

16 Measurements and device constants

Micro-Ubbelohde Viscometers DIN 51 562. Part 2
Ref. No. 536 ... 537 ... 538 ..

Ref. No.	Capillary No.	Capillary $\varnothing_i$ (mm)	Constant K (approx.)	Measuring range mm^2/s (cSt) (approx.)		
... 10	I	0.40	0.01	0.4	to	6
... 13	Ic	0.53	0.03	1.2	to	18
... 20	II	0.70	0.1	4	to	60
... 23	IIc	0.95	0.3	12	to	180
... 30	III	1.26	1	40	to	800

* previously centistokes [cSt]; 1 [cSt] = 1 [mm^2/s]

17 Table of the kinetic energy correction (HC)

Micro-Ubbelohde Viscometers DIN 51 562. Part 2
Ref. No. 536 ... 537 ... 538 ..

Correction seconds[1]:

Flow time [s]	Capillary No.	
	I	Ic
30	0.46	0.08
40	0.26	0.04
50	0.17	0.03
60	0.12	0.02
70	0.09	0.01
80	0.07	0.01
90	0.05	0.01
100	0.04	0.01

[1]) The correction seconds stated are related to the respective theoretical constant.

Typ / Type / Type / Tipo:

Serien Nr. / Serial no. / No. de série / N° de serie:

Bescheinigung des Herstellers

Wir bestätigen, dass das oben genannte Gerät gemäß DIN EN ISO 9001, Absatz 4.10.4 "Endprüfung" geprüft wurde und dass die festgelegte Qualitätsanforderung an das Produkt erfüllt wird.

Supplier's Certificate

We certify that the equipment EN ISO 9001, part 4.10.4 "Final inspection and testing" and that the specified requirements for the product are met.

Certificat du fournisseur

Nous certifions que le produit a été vérifié selon EN ISO 9001, partie 4.10.4 "Contrôles et essais finals" et que les exigences spécifiées pour le produit sont respectées.

Certificado del fabricante

Nosotros certificamos que el equipo verifica la producción conforme a EN ISO 9001, parte 4.10.4 "Inspección y control final" y que las especificaciones requeridas para el equipo son respetadas y cumplidas.