
**Rubber compounding ingredients —
Carbon black — Determination of specific
surface area by nitrogen adsorption
methods — Single-point procedures**

*Ingrédients de mélange du caoutchouc — Noir de carbone —
Détermination de la surface spécifique par méthodes par adsorption
d'azote — Modes opératoires à un point de mesure*

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 4652 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 3, *Raw materials (including latex) for use in the rubber industry*.

This second edition of ISO 4652 cancels and replaces ISO 4652-1:1994, which has been technically revised. The revision includes the following changes:

- The number of the standard has been changed from ISO 4652-1 to ISO 4652, since there were no other parts.
- The title has been modified accordingly.
- A statement has been added to the scope that the multipoint method specified in ISO 18852 is the preferred method.
- The normative references in Clause 2 have been updated.
- The Ni-Count-1 apparatus used in method A is no longer available from the manufacturer. However, it has been decided to keep method A for those still using this apparatus. A note has been added at the beginning of Clause 3 to explain this.

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Rubber compounding ingredients — Carbon black — Determination of specific surface area by nitrogen adsorption methods — Single-point procedures

WARNING — Persons using this International Standard should be familiar with normal laboratory practice. This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.

1 Scope

This International Standard specifies four methods for the determination of the specific surface area of types and grades of carbon black for use in the rubber industry:

- method A using the Ni-Count-1 apparatus (Clause 3);
- method B using the Areameter apparatus (Clause 4);
- method C using gas chromatography (Clause 5);
- method D using the Monosorb surface-area analyser (Clause 6).

Somewhat different results might be obtained from the four methods. The degassing procedure used differs from method to method, and it is important to investigate the possibility of correcting the results by using standard reference blacks.

The results might also differ from those obtained using the multipoint method specified in ISO 18852, which is the preferred method.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 1126, *Rubber compounding ingredients — Carbon black — Determination of loss on heating*

ISO 18852, *Rubber compounding ingredients — Determination of multipoint nitrogen surface area (NSA) and statistical thickness surface area (STSA)*

3 Method A, using Ni-Count-1 apparatus

NOTE Although the Ni-Count-1 apparatus is no longer being produced by the manufacturer, E.G. & G. Chandler Engineering, this method has been retained for the convenience of those who are still using this apparatus.

3.1 Principle

A test portion is degassed and weighed, then exposed to nitrogen at the temperature of liquid nitrogen. The amount of nitrogen adsorbed on to the carbon black surface at equilibrium is determined. From this value and the mass of the degassed test portion, the specific surface area is calculated.

3.2 Reagents

3.2.1 Nitrogen, in a cylinder, or another source of purified nitrogen of recognized analytical quality.

The nitrogen supply to the Ni-Count-1 apparatus (see 3.3.1) shall be controlled at a pressure of 70 kPa to 140 kPa. If nitrogen from a cylinder is used, the cylinder shall be fitted with a two-stage regulator capable of maintaining the outlet pressure within the specified range.

3.2.2 Liquid nitrogen (approximately 300 cm³ are required for the determination).

WARNING — Use caution. Gloves and safety glasses should be worn as the temperature of liquid nitrogen is –196 °C.

3.3 Apparatus

3.3.1 Ni-Count-1 specific surface area apparatus (see Figures 1 and 2), or an equivalent one-point adsorption apparatus.

NOTE The Ni-Count-1 apparatus is no longer produced (see the note at the beginning of this clause).

3.3.2 Heater and voltage-control device, capable of maintaining a temperature of 300 °C ± 10 °C, for degassing the test portion.

NOTE The heater (see Figure 1) is furnished with the Ni-Count-1 apparatus.

3.3.3 Vacuum pump, capable of producing an ultimate pressure of $(1,3 \times 10^{-2})$ Pa [(1×10^{-4}) mmHg].

3.3.4 Dewar flask, capacity approximately 265 cm³ and height 145 mm.

This is supplied with the Ni-Count-1 apparatus.

3.3.5 Nitrogen vapour pressure thermometer (see Figure 2).

This is supplied with the Ni-Count-1 apparatus.

3.3.6 Sample tubes (see Figure 3).

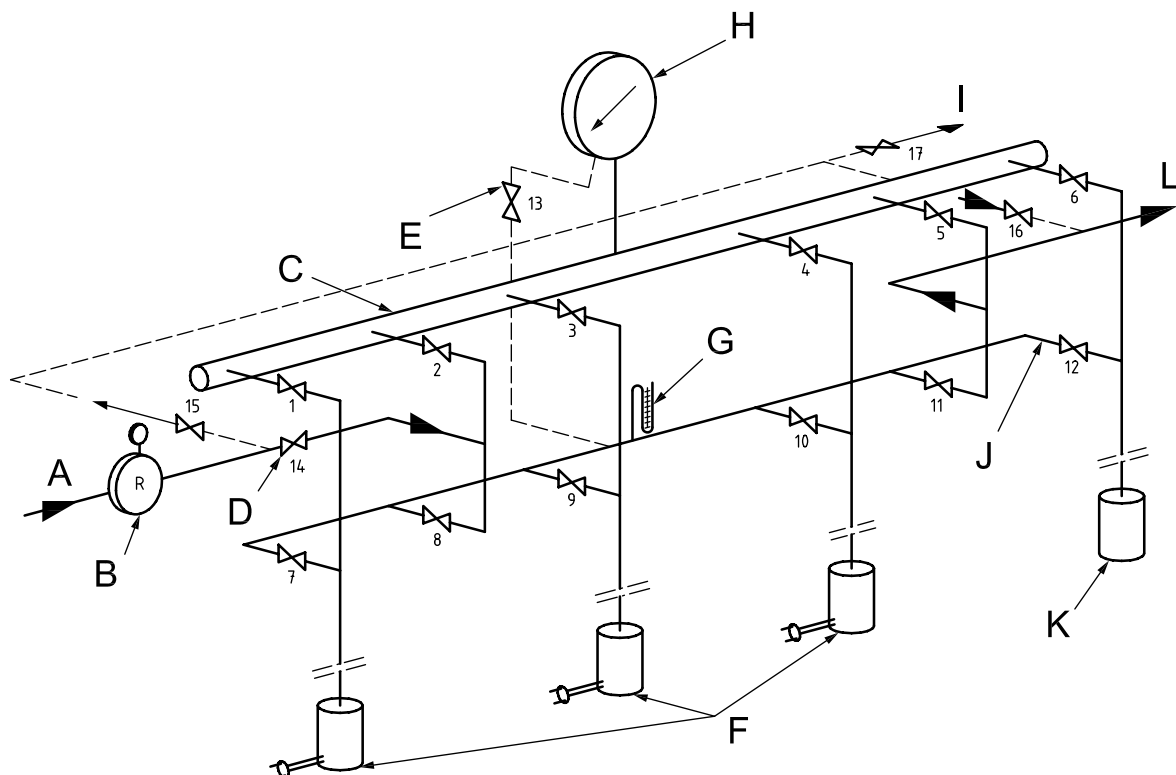
The recommended volumes are given in Table 1.

3.3.7 Stopcock grease or poly(chlorotrifluoroethylene) lubricant.

This is supplied with the Ni-Count-1 apparatus.

3.3.8 Fine glass wool.

3.3.9 Analytical balance, accurate to 0,1 mg.

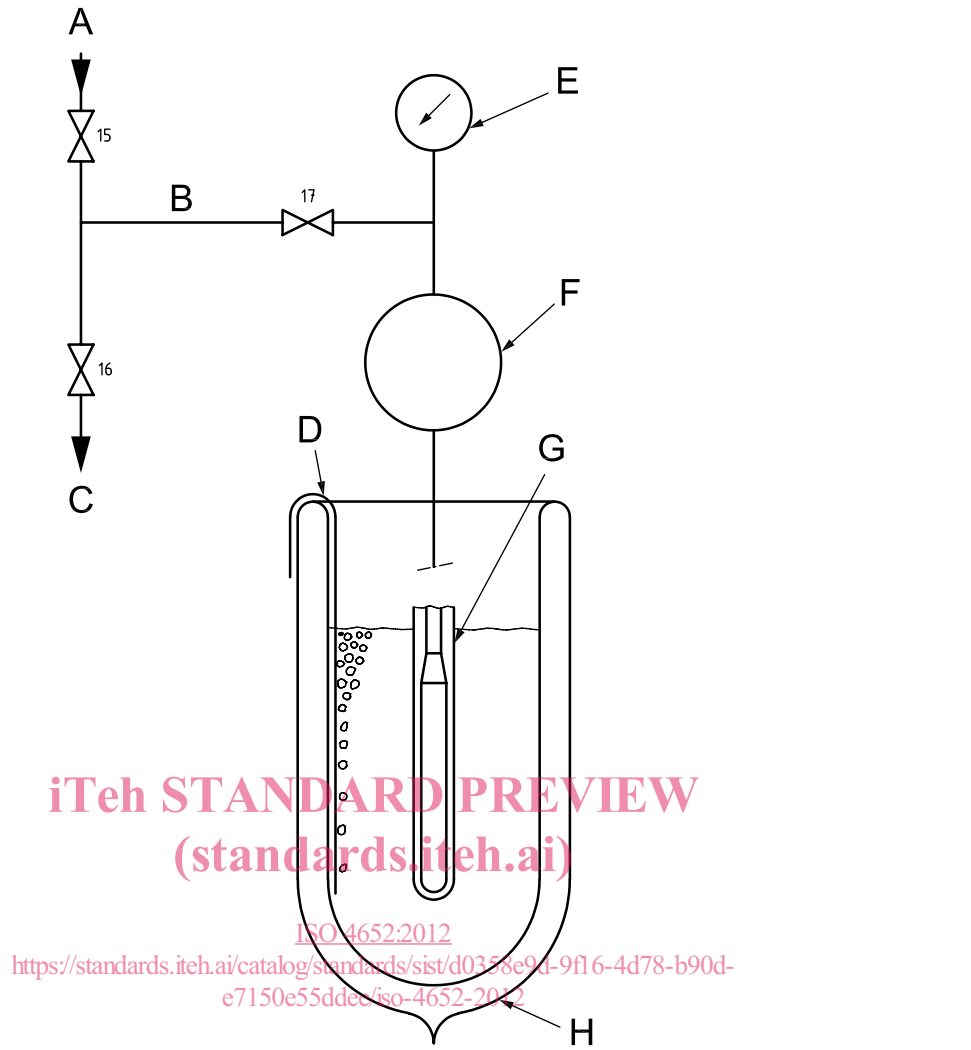


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Key

- | | | | |
|---|--|---|---|
| A | nitrogen gas | G | vacuum indicator |
| B | flow regulator | H | precision pressure gauge, 0 kPa to 103 kPa (0 mmHg to 775 mmHg) |
| C | calibrated-volume chamber and manifold | I | to nitrogen vapour pressure thermometer |
| D | flow-control valve | J | vacuum manifold |
| E | line and valve for evacuating case of pressure gauge | K | Dewar flask with liquid nitrogen |
| F | electric heaters (see 3.3.2) | L | to vacuum pump |

Figure 1 — Ni-Count-1 apparatus with provision for four test portions

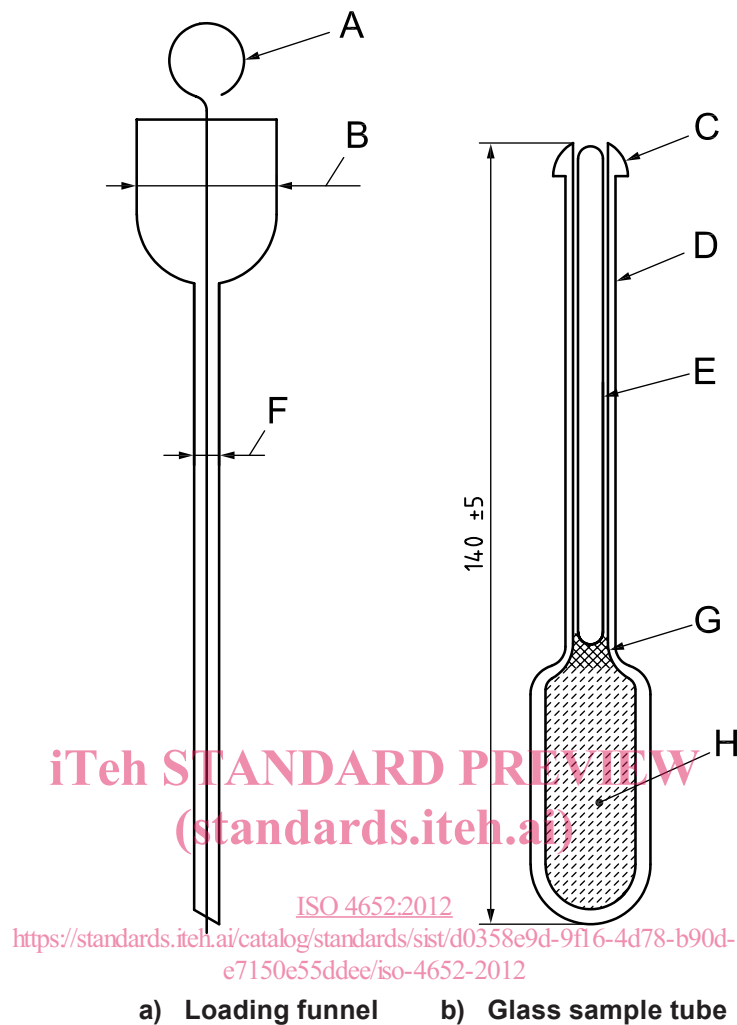


- Key**
- | | |
|---|--|
| A pure nitrogen | E precision pressure gauge, 0 kPa to 25 kPa (0 mmHg to 186 mmHg) |
| B filling connection | F volume chamber |
| C to vacuum pump | G sensing element of thermometer |
| D copper wire looped over edge of Dewar flask | H Dewar flask containing liquid nitrogen |

NOTE The system is filled with pure nitrogen to a pressure of 13 kPa to 20 kPa (100 mmHg to 150 mmHg).

Figure 2 — Nitrogen vapour pressure thermometer

Dimensions in millimetres

**Key**

- | | | | |
|---|--|---|-----------------------|
| A | stainless-steel wire | F | outside diameter 5 mm |
| B | outside diameter 25 mm | G | glass-wool plug |
| C | 12/5 ball joint | H | test portion |
| D | tubing, outside diameter 8 mm, inside diameter 6 mm | | |
| E | filler rod or tubing, outside diameter 5 mm, sealed at both ends | | |

Figure 3 — Loading funnel and glass sample tube**3.4 Preparation of the sample**

Pellets of carbon black need not be crushed. Unagitated, unpelletized carbon black may be densified if desired.

3.5 Test conditions

The test should preferably be carried out in ambient conditions of either $23\text{ °C} \pm 2\text{ °C}$ and $(50 \pm 5)\%$ relative humidity or $27\text{ °C} \pm 2\text{ °C}$ and $(65 \pm 5)\%$ relative humidity.

The reagents and the apparatus shall be maintained at temperature equilibrium in the same room for at least 24 h before being used.

The test room shall be free from fumes or vapours which could contaminate the reagents or apparatus and thus affect the results.

3.6 Procedure

3.6.1 Preparation and calibration of apparatus

3.6.1.1 The all-metal Ni-Count-1 apparatus has an internal volume adjusted to 139,5 cm³. This internal volume includes all lines of the pressure gauge adjusted so that the gauge indicates 66,7 kPa (500 mmHg) at a room temperature of 27 °C. The tables of surface area versus pressure (furnished with the Ni-Count-1 apparatus) yield accurate specific surface areas if the internal volume of the instrument has been accurately adjusted at the factory to 139,5 cm³. To confirm the volume, it is recommended that tests be made on a standard reference black having an agreed nitrogen surface area independently determined by the multipoint method specified in ISO 18852.

3.6.1.2 The Ni-Count-1 apparatus shall be prepared as specified in the instructions furnished with the apparatus. This includes filling the nitrogen vapour pressure thermometer (3.3.5) with purified nitrogen gas (3.2.1), evacuating the case of the large pressure gauge and closing the case valve, flushing the reservoir and vacuum manifolds several times with nitrogen until air is eliminated and adjusting the voltage to the heaters to maintain a temperature of 300 °C ± 10 °C as measured with a thermometer in the heater well.

If air is admitted to the reservoir at any time, the purging shall be repeated.

3.6.1.3 The calibration and accuracy of the equipment shall be checked by tests on standard reference blacks.

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3.6.2 Determination

3.6.2.1 Using the data in Table 1 as a guide, select the proper sample tube and take the appropriate test-portion mass. If the grade of the carbon black is not known, carry out a preliminary test to determine the mass of the black which gives an adsorption pressure of between 20,0 kPa and 33,3 kPa (150 mmHg and 250 mmHg).

Table 1 — Recommended sample-tube volumes and test-portion masses for common grades of pressed and pelleted carbon blacks

Carbon black series	Specific surface area m ² /g	Sample-tube volume		Mass of test portion g
		Pressed cm ³	Pelleted cm ³	
N100	140	5,0	3,5	1,1
N200 — S200	110	7,5	4,5	1,5
N300 — S300	80	10	5,5	2,0
N500	44	18	9	3,6
N600	30	22	11	5,3
N900	20 to 6	15 to 30	10 to 25	10 to 15

3.6.2.2 Weigh, to the nearest 0,1 mg, a plug of glass wool (3.3.8) of a suitable size to support the filler tube in the sample-tube stem. Record the mass.

3.6.2.3 Weigh, to the nearest 0,1 mg, a clean, dry sample tube (3.3.6) with its filler tube and glass-wool plug. Record the mass (*m*₁).

3.6.2.4 Roughly weigh the predried test portion. (This is the non-degassed mass and is not used in the calculation.)

3.6.2.5 Place the test portion in the sample tube, introduce the plug of glass wool and push in the filler rod to its proper position.

3.6.2.6 Sparingly lubricate the ball joint of the sample tube with the high-vacuum grease or lubricant (3.3.7), taking care not to place grease or lubricant inside the stem. Fit the sample-tube ball into the mating metal receptacle on the Ni-Count-1 apparatus and retain the sample tube in place with the metal spring clip.

3.6.2.7 Start the evacuation of the sample tube through the vacuum manifold and raise the heater around the tube to degas the test portion at $300\text{ °C} \pm 10\text{ °C}$ for a minimum of 15 min.

3.6.2.8 During the evacuation, purge the test portion several times with nitrogen gas. To do this, close the valve to the vacuum pump and momentarily open the valve from the nitrogen supply to the vacuum manifold and set the rate by the flow-control valve; then resume evacuation.

3.6.2.9 Close the vacuum valve and observe the leak detector to determine whether gases are still evolving from the test portion. If the test portion is properly degassed, the leak indicator should not show a change of pressure greater than 0,1 kPa (1 mmHg) over 5 min.

3.6.2.10 Isolate the degassed test portion from the vacuum manifold by closing the valve. Remove the heater.

3.6.2.11 If the pressure in the purged nitrogen reservoir is above 65,7 kPa (493 mmHg) at 23 °C [or above 66,7 kPa (500 mmHg) at 27 °C], evacuate to a lower pressure. Complete evacuation is not necessary unless air has been permitted to enter.

Fill the purged reservoir gauge and manifold with nitrogen gas to a pressure of 65,7 kPa (493 mmHg) if the temperature is 23 °C, or to 66,7 kPa (500 mmHg) if the temperature is 27 °C. For each degree respectively above or below the indicated temperatures, add or subtract 0,222 kPa (1,67 mmHg) from the specified pressures.

3.6.2.12 Open the valve from the nitrogen reservoir to the sample tube by rotating it through three complete turns.

3.6.2.13 Place the Dewar flask (3.3.4) filled with liquid nitrogen (3.2.2) around the sample tube.

Table 2 — Liquid-nitrogen temperature-correction factor

Nitrogen vapour pressure		Correction factor <i>B</i>	Nitrogen vapour pressure		Correction factor <i>B</i>	Nitrogen vapour pressure		Correction factor <i>B</i>	Nitrogen vapour pressure		Correction factor <i>B</i>
<i>p_N</i>			<i>p_N</i>			<i>p_N</i>			<i>p_N</i>		
mmHg	kPa		mmHg	kPa		mmHg	kPa		mmHg	kPa	
660	88,0	0,954 16	695	92,7	0,974 22	730	97,3	0,994 27	765	102,0	1,014 32
661	88,1	0,954 73	696	92,8	0,974 79	731	97,5	0,994 84	766	102,1	1,014 90
662	88,3	0,955 31	697	92,9	0,975 36	732	97,6	0,995 42	767	102,3	1,015 47
663	88,4	0,955 88	698	93,1	0,975 93	733	97,7	0,995 99	768	102,4	1,016 04
664	88,5	0,956 45	699	93,2	0,976 51	734	97,9	0,996 56	769	102,5	1,016 62
665	88,7	0,957 03	700	93,3	0,977 08	735	98,0	0,997 14	770	102,7	1,017 19
666	88,8	0,957 60	701	93,5	0,977 65	736	98,1	0,997 71	771	102,8	1,017 76
667	88,9	0,958 17	702	93,6	0,978 23	737	98,3	0,998 28	772	102,9	1,018 34
668	89,1	0,958 74	703	93,7	0,978 80	738	98,4	0,998 85	773	103,1	1,018 91
669	89,2	0,959 32	704	93,9	0,979 37	739	98,5	0,999 43	774	103,2	1,019 48
670	89,3	0,959 89	705	94,0	0,979 95	740	98,7	1,000 00	775	103,3	1,020 05
671	89,5	0,960 46	706	94,1	0,980 52	741	98,8	1,000 57	776	103,5	1,020 63
672	89,6	0,961 04	707	94,3	0,981 09	742	98,9	1,001 15	777	103,6	1,021 20
673	89,7	0,961 61	708	94,4	0,981 66	743	99,1	1,001 72	778	103,7	1,021 77
674	89,9	0,962 18	709	94,5	0,982 24	744	99,2	1,002 29	779	103,9	1,022 35
675	90,0	0,962 76	710	94,7	0,982 81	745	99,3	1,002 86	780	104,0	1,022 92
676	90,1	0,963 33	711	94,8	0,983 38	746	99,5	1,003 44	781	104,1	1,023 49
677	90,3	0,963 90	712	94,9	0,983 96	747	99,6	1,004 01	782	104,3	1,024 07
678	90,4	0,964 47	713	95,1	0,984 53	748	99,7	1,004 58	783	104,4	1,024 64
679	90,5	0,965 05	714	95,2	0,985 10	749	99,9	1,005 16	784	104,5	1,025 21
680	90,7	0,965 62	715	95,3	0,985 68	750	100,0	1,005 73	785	104,7	1,025 78
681	90,8	0,966 19	716	95,5	0,986 25	751	100,1	1,006 30	786	104,8	1,026 36
682	90,9	0,966 77	717	95,6	0,986 82	752	100,3	1,006 88	787	104,9	1,026 93
683	91,1	0,967 34	718	95,7	0,987 39	753	100,4	1,007 45	788	105,1	1,027 50
684	91,2	0,967 91	719	95,9	0,987 97	754	100,5	1,008 02	789	105,2	1,028 08
685	91,3	0,968 49	720	96,0	0,988 54	755	100,7	1,008 59	790	105,3	1,028 65
686	91,5	0,969 06	721	96,1	0,989 11	756	100,8	1,009 17	791	105,5	1,029 22
687	91,6	0,969 63	722	96,3	0,989 69	757	100,9	1,009 74	792	105,6	1,029 80
688	91,7	0,970 20	723	96,4	0,990 26	758	101,1	1,010 31	793	105,7	1,030 37
689	91,9	0,970 78	724	96,5	0,990 83	759	101,2	1,010 89	794	105,9	1,030 94
690	92,0	0,971 35	725	96,7	0,991 41	760	101,3	1,011 46	795	106,0	1,031 51
691	92,1	0,971 92	726	96,8	0,991 98	761	101,5	1,012 03	796	106,1	1,032 09
692	92,3	0,972 50	727	96,9	0,992 55	762	101,6	1,012 61	797	106,3	1,032 66
693	92,4	0,973 07	728	97,1	0,993 12	763	101,7	1,013 18	798	106,4	1,033 23
694	92,5	0,973 64	729	97,2	0,993 70	764	101,9	1,013 75	799	106,5	1,033 81

NOTE The liquid-nitrogen temperature-correction factor *B* is derived from the formula $1 + 0,057\ 3 \times [(p_N - 98,7)/13,3]$,

where

p_N is the vapour pressure, in kilopascals, of pure nitrogen at the nitrogen adsorption temperature, measured by means of the nitrogen vapour pressure thermometer (3.3.5);

98,7 is the barometric pressure, in kilopascals, during calibration of the apparatus to determine area *S* at the equilibrium pressure (see Table 3).

If the pressure is expressed in millimetres of mercury, the above formula becomes $1 + 0,057\ 3 \times [(p_N - 740)/100]$.