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# International Standard



# 4652

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INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

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## **Rubber compounding ingredients — Carbon black — Determination of specific surface area — Nitrogen adsorption methods**

*Ingrédients de mélange du caoutchouc — Noir de carbone — Détermination de la surface spécifique — Méthodes par adsorption d'azote*

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## Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 4652 was developed by Technical Committee ISO/TC 45, *Rubber and rubber products*, and was circulated to the member bodies in March 1980.

It has been approved by the member bodies of the following countries :

Austria	France	Romania
Belgium	Germany, F.R.	South Africa, Rep. of
Brazil	Hungary	Spain
Bulgaria	India	Sweden
Canada	Italy	Turkey
China	Korea, Rep. of	United Kingdom
Czechoslovakia	Mexico	USA
Denmark	Netherlands	USSR
Egypt, Arab Rep. of	Poland	

No member body expressed disapproval of the document.

# Rubber compounding ingredients — Carbon black — Determination of specific surface area — Nitrogen adsorption methods

## 1 Scope and field of application

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

## 2 Reference

ISO 1126, *Carbon black for use in the rubber industry — Determination of loss on heating.*

## 3 Method A using Ni-Count-1 apparatus

### 3.1 Principle

Degassing of a test portion, weighing and exposure to nitrogen in the presence of liquid nitrogen. Determination of the amount of nitrogen adsorbed onto the carbon black surface at equilibrium. From this value and the mass of the degassed test portion, calculation of the specific surface area.

### 3.2 Reagents

**3.2.1 Nitrogen**, in a cylinder, or other source of prepurified 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 to 140 kPa. If nitrogen from a cylinder is used, the cylinder shall be fitted with a two-stage regulator capable of controlling the outlet pressure in the specified range.

**3.2.2 Liquid nitrogen** (approximately 300 cm<sup>3</sup> are required for the determination).

### 3.3 Apparatus

**3.3.1 Ni-Count-1 specific surface area apparatus<sup>1)</sup>** (see figures 1 and 2), or an equivalent one-point adsorption apparatus.

**3.3.2 Heater and voltage control device**, capable of maintaining a temperature of  $300 \pm 10$  °C, for degassing the test portion.

[The heater (see figure 1) is furnished with the Ni-Count-1 apparatus.]

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

**3.3.4 Dewar flask**, of capacity approximately 265 cm<sup>3</sup> and height 145 mm.

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

**3.3.5 Nitrogen vapour pressure thermometer** (see figure 2).

(This constitutes a part of the Ni-Count-1 apparatus.)

**3.3.6 Sample tubes** (see figure 3).

The recommended volumes are given in table 1.

1) The Ni-Count-1 apparatus is available commercially. Details may be obtained from the Secretariat of ISO/TC 45 (BSI) or ISO Central Secretariat.

**3.3.7 Stopcock grease or polychlorotrifluoroethylene 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.**

**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  $23 \pm 2$  °C and  $50 \pm 5$  % relative humidity or  $27 \pm 2$  °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 testing room shall be free from fumes or vapours which could contaminate the reagents and 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 adjusted internal volume of 139,5 cm<sup>3</sup>. This internal volume includes all lines to the sample valve, and the volume in the bellows of the pressure gauge is 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) will yield accurate specific surface areas if the internal volume of the instrument has been accurately adjusted at the factory to 139,5 cm<sup>3</sup>. To confirm the volume, it is recommended that tests be made on a standard reference black<sup>1)</sup> having an agreed nitrogen surface area independently determined by a multipoint method.

**3.6.1.2** The Ni-Count-1 apparatus should be prepared as specified in the instructions furnished with the apparatus. This includes filling the nitrogen vapour pressure thermometer (3.3.5) with the prepurified 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 controlling the voltage to the heaters to maintain a temperature of  $300 \pm 10$  °C as measured with a thermometer in the heater well.

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

**3.6.1.3** The calibration and accuracy of the equipment should be checked by tests on standard reference blacks.<sup>1)</sup>

**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 mass of test portion. If the identity of the black is not known, carry out a preliminary test to determine the mass of the black which will give an adsorption pressure between 20,0 and 33,3 kPa (150 and 250 mmHg).

**3.6.2.2** Weigh, to the nearest 0,1 mg, a tuft of the glass wool (3.3.8) of 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 and glass wool tuft. Record the mass ( $m_1$ ).

**3.6.2.4** Roughly weigh the 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 (3.3.6), introduce the tuft 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 (3.3.7), taking care not to place 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  $300 \pm 10$  °C.

**3.6.2.8** Momentarily purge the test portion, several times during the evacuation, 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; then resume evacuation.

1) Standard reference blacks will form the subject of ISO 6809.

**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 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.

**3.6.2.14** Permit the adsorption to proceed until the pressure indicated by the large gauge becomes constant.

Observe and record the pressure to the nearest 0,1 kPa (1 mmHg). Ensure the liquid nitrogen surface is at the proper level on the tube stem. (If a variable stem correction is used in the calculations, measure and record the exposed stem length.)

**3.6.2.15** Lower the Dewar flask from the sample tube and place it around the sensing element of the nitrogen vapour pressure thermometer (3.3.5).

**3.6.2.16** After the gauge pressure of the nitrogen vapour pressure thermometer has become constant, observe the pressure and record its value to the nearest 0,1 kPa (1 mmHg).

**3.6.2.17** Allow the sample tube to warm to above the temperature of water vapour condensation on the tube. The warming process can be hastened by gentle heating.

**3.6.2.18** Add nitrogen gas to the reservoir and sample tube until the pressure gauge reads approximately 1,3 kPa (10 mmHg) above barometric pressure.

Close the valve to the sample tube and remove the tube.

**3.6.2.19** Open the valve to the sample tube connection to equalize the nitrogen reservoir pressure with atmospheric pressure. Read the gauge to the nearest 0,1 kPa (1 mmHg) and record the pressure. Close the valve.

**3.6.2.20** As thoroughly as possible, wipe the vacuum grease from the sample tube ball and any moisture from the tube exterior.

Weigh the tube (containing the dry and degassed test portion, glass wool and filler rod) to the nearest 0,1 mg. Record the mass ( $m_2$ ).

**3.6.2.21** Add the value obtained in 3.6.2.16 to the barometric pressure to obtain the vapour pressure  $P_N$  for use in table 2 to obtain the liquid nitrogen temperature correction factor,  $B$ .

### 3.7 Expression of results

Calculate the specific surface area,  $S_m$ , in square metres per gram, from the formula

$$S_m = \frac{S}{m} \left[ 1 - F \left( V_t - V_{dt} - V_{gw} - \frac{m}{\rho} \right) \right] B$$

where

$S$  is the surface area at the equilibrium pressure, obtained from table 3;

$m$  is the mass, in grams, of the dry and degassed test portion ( $m_2 - m_1$ );

$F$  is the correlating factor, obtained from table 3;

$V_t$  is the volume, in cubic centimetres, of the sample tube with filler;

$V_{dt}$  is the volume, in cubic centimetres, in the sample tube stem with filler inserted, above the surface of the liquid nitrogen;

$V_{gw}$  is the volume, in cubic centimetres, of the glass wool tuft, calculated from its mass and the assumed density of 2,3 g/cm<sup>3</sup>;

$\rho$  is the density, in megagrams per cubic metre, of carbon black, assumed to be equal to 1,8 Mg/m<sup>3</sup>;

$B$  is the liquid nitrogen temperature correction factor, obtained from table 2.

Express the result to the nearest 0,1 m<sup>2</sup>/g.

### 3.8 Test report

The test report shall include the following information :

- a reference to this International Standard (indicating "method A");
- complete identification of the sample;
- the conditions of test;
- the mass of test portion used;
- the result and the method of expression used.

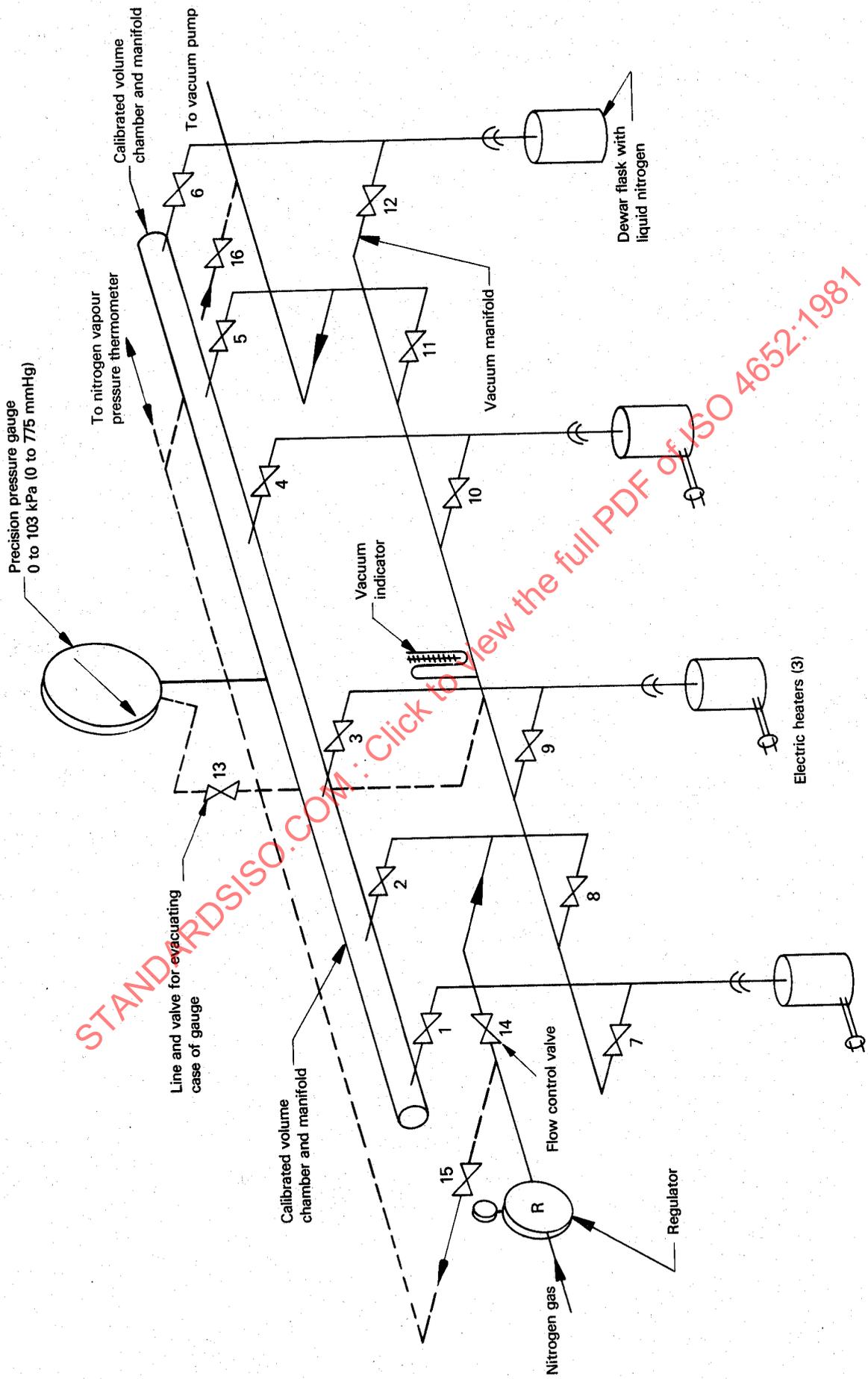
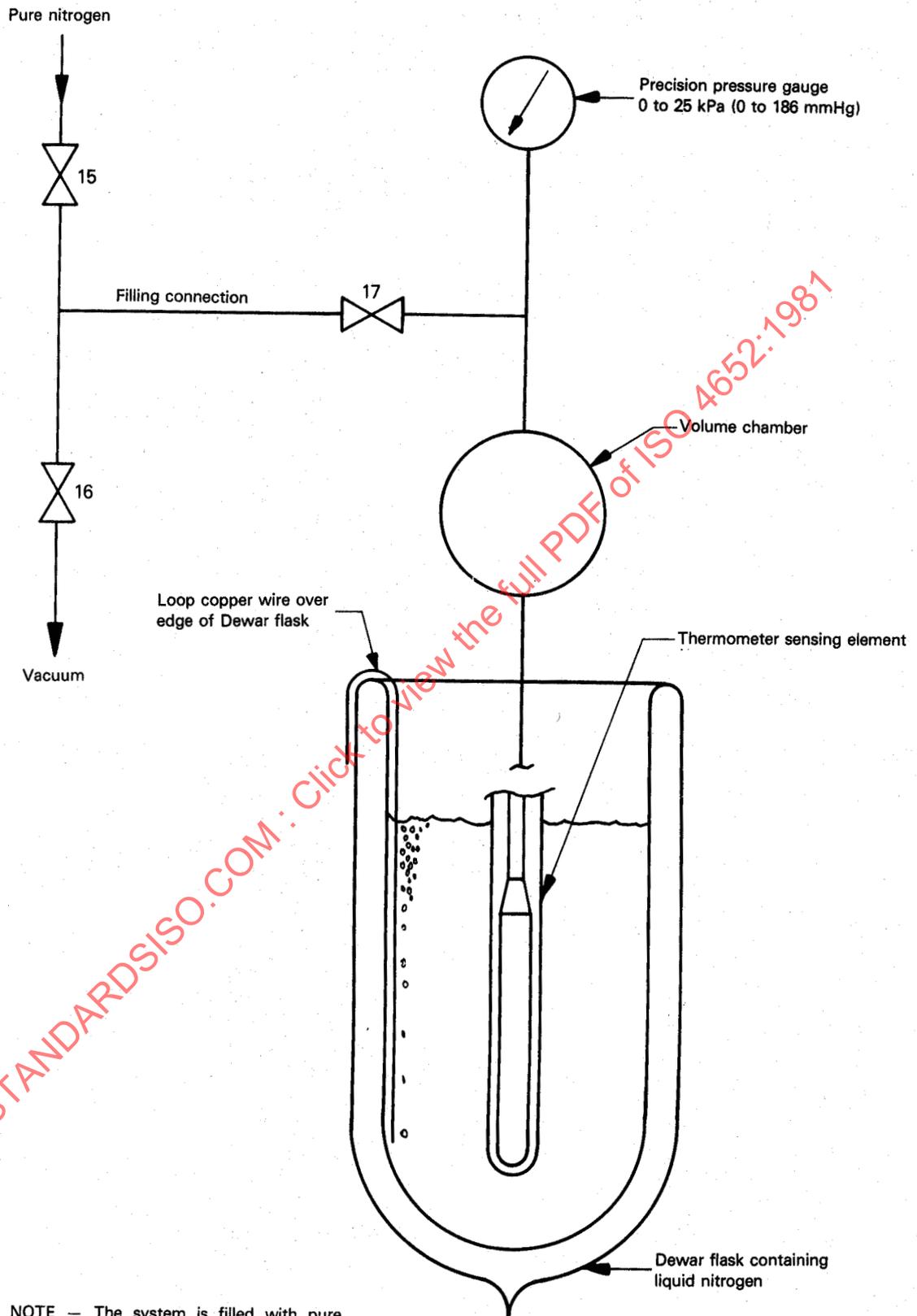
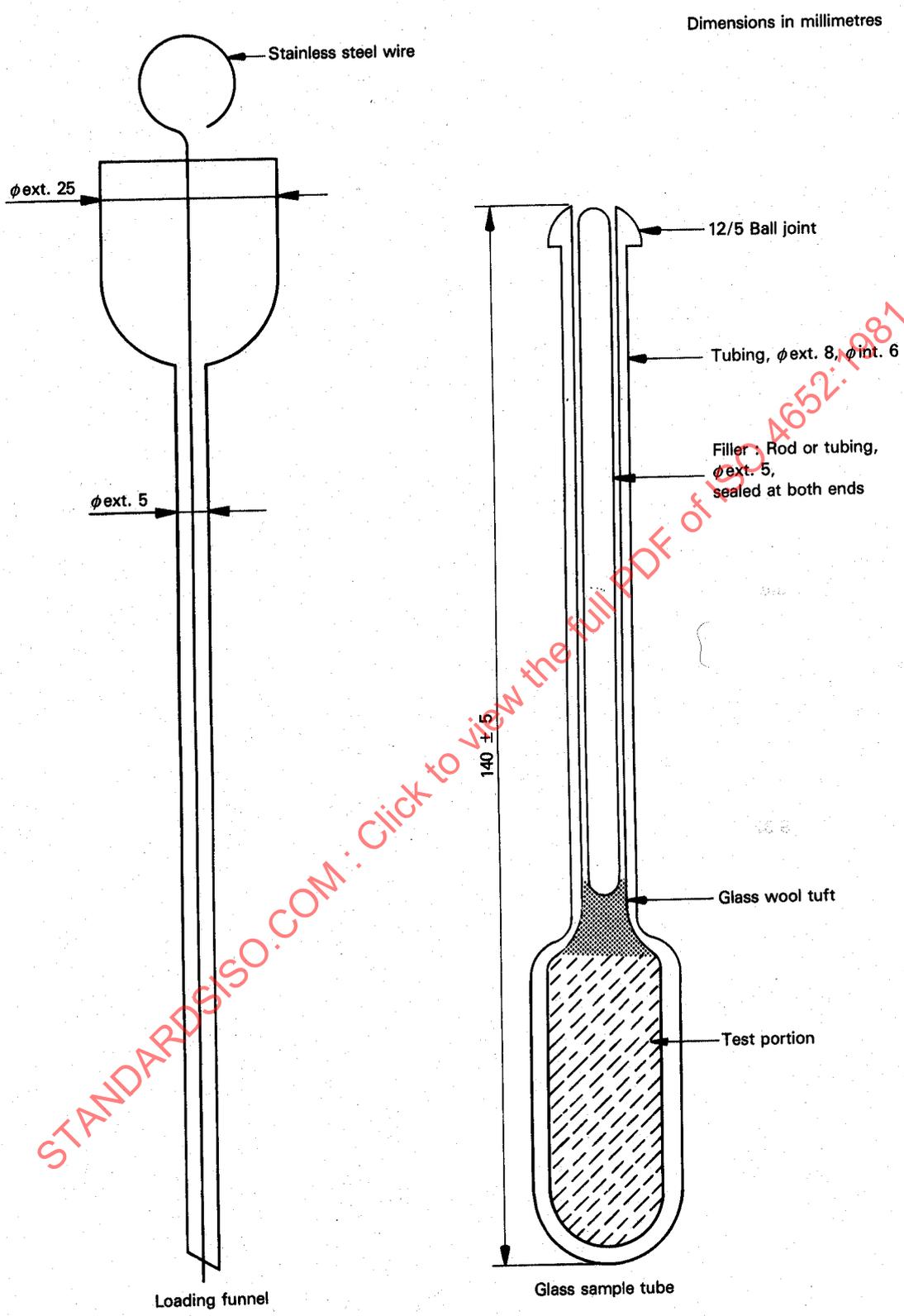


Figure 1 — Ni-Count-1 apparatus with provision for four samples



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

Figure 2 — Nitrogen vapour pressure thermometer



NOTE — Mark the tube and filler identically.

Figure 3 — Loading funnel and glass sample tube

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

Carbon black group	Specific surface area m <sup>2</sup> /g	Sample tube volume		Mass of test portion g
		Pressed cm <sup>3</sup>	Pelleted cm <sup>3</sup>	
N 900 (formerly MT)	20 to 6	15 to 30	10 to 25	10 to 15
N 600 (formerly GPF)	30	22	11	5,3
N 500 (formerly FEF)	44	18	9	3,6
N 300 — S 300 (formerly HAF)	80	10	5,5	2,0
N 200 — S 200 (formerly ISAF)	110	7,5	4,5	1,5
N 100 (formerly SAF)	140	5,0	3,5	1,1

Table 2 — Liquid nitrogen temperature correction factor

NOTE — The liquid nitrogen temperature correction factor, *B*, is derived from the formula

$$1 + 0,0573 \left[ \frac{(P_N - 98,7)}{13,3} \right]$$

where

*P<sub>N</sub>* 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 the surface area *S* at the equilibrium pressure (see table 3).

If the pressure is expressed in millimetres of mercury, the above formula becomes

$$1 + 0,0573 \left[ \frac{(P_N - 740)}{100} \right]$$

Nitrogen vapour pressure <i>P<sub>N</sub></i>		Correction factor <i>B</i>	Nitrogen vapour pressure <i>P<sub>N</sub></i>		Correction factor <i>B</i>	Nitrogen vapour pressure <i>P<sub>N</sub></i>		Correction factor <i>B</i>	Nitrogen vapour pressure <i>P<sub>N</sub></i>		Correction factor <i>B</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

Table 3 — Values of  $F$  and  $S$  for observed equilibrium pressures

Equilibrium pressure	Corre- lating factor $F$		Surface area $S$	Equilibrium pressure		Corre- lating factor $F$	Surface area $S$	Equilibrium pressure		Corre- lating factor $F$	Surface area $S$	Equilibrium pressure		Corre- lating factor $F$	Surface area $S$
	mmHg	kPa		mmHg	kPa			mmHg	kPa			mmHg	kPa		
100	13,3	0,006 99	255,7	140	18,6	0,010 94	213,8	180	23,9	0,015 98	176,3	220	29,3	0,022 63	143,3
101	13,4	0,007 08	254,6	141	18,7	0,011 05	212,8	181	24,1	0,016 12	175,4	221	29,4	0,022 82	142,5
102	13,5	0,007 17	253,5	142	18,9	0,011 17	211,8	182	24,2	0,016 26	174,5	222	29,5	0,023 02	141,7
103	13,7	0,007 26	252,4	143	19,0	0,011 28	210,8	183	24,3	0,016 41	173,7	223	29,7	0,023 22	141,0
104	13,8	0,007 35	251,3	144	19,1	0,011 39	209,8	184	24,5	0,016 56	172,8	224	29,8	0,023 42	140,2
105	13,9	0,007 44	250,2	145	19,3	0,011 51	208,8	185	24,6	0,016 70	171,9	225	29,9	0,023 62	139,5
106	14,1	0,007 53	249,1	146	19,4	0,011 62	207,9	186	24,7	0,016 85	171,0	226	30,1	0,023 82	138,7
107	14,2	0,007 62	248,0	147	19,5	0,011 73	206,9	187	24,9	0,017 00	170,2	227	30,2	0,024 02	138,0
108	14,3	0,007 71	247,0	148	19,7	0,011 85	205,9	188	25,0	0,017 15	169,3	228	30,3	0,024 23	137,2
109	14,5	0,007 81	245,9	149	19,8	0,011 97	204,9	189	25,1	0,017 31	168,5	229	30,5	0,024 44	136,5
110	14,6	0,007 90	244,8	150	19,9	0,012 08	204,0	190	25,3	0,017 46	167,6	230	30,6	0,024 65	135,7
111	14,7	0,007 99	243,7	151	20,1	0,012 20	203,0	191	25,4	0,017 61	166,8	231	30,7	0,024 86	135,0
112	14,9	0,008 09	241,7	152	20,2	0,012 32	202,0	192	25,5	0,017 77	165,9	232	30,9	0,025 07	134,2
113	15,0	0,008 18	241,6	153	20,3	0,012 44	201,1	193	25,7	0,017 93	165,1	233	31,0	0,025 29	133,5
114	15,1	0,008 28	240,5	154	20,5	0,012 56	200,1	194	25,8	0,018 08	164,2	234	31,1	0,025 50	132,8
115	15,3	0,008 37	239,5	155	20,6	0,012 68	199,2	195	25,9	0,018 24	163,4	235	31,3	0,025 72	132,1
116	15,4	0,008 47	238,4	156	20,7	0,012 80	198,2	196	26,1	0,018 40	162,5	236	31,4	0,025 94	131,3
117	15,5	0,008 56	237,3	157	20,9	0,012 93	197,3	197	26,2	0,018 56	161,7	237	31,5	0,026 16	130,6
118	15,7	0,008 66	236,3	158	21,0	0,013 05	196,3	198	26,3	0,018 73	160,9	238	31,7	0,026 39	129,9
119	15,8	0,008 76	235,2	159	21,1	0,013 17	195,4	199	26,5	0,018 89	160,0	239	31,8	0,026 61	129,2
120	15,9	0,008 86	234,2	160	21,3	0,013 30	194,5	200	26,6	0,019 05	159,2	240	31,9	0,026 84	128,4
121	16,1	0,008 96	233,1	161	21,4	0,013 42	193,5	201	26,7	0,019 22	158,4	241	32,1	0,027 07	127,7
122	16,2	0,009 06	232,1	162	21,5	0,013 55	192,6	202	26,9	0,019 39	157,6	242	32,2	0,027 31	127,0
123	16,3	0,009 16	231,0	163	21,7	0,013 68	191,7	203	27,0	0,019 56	156,8	243	32,3	0,027 54	126,3
124	16,5	0,009 26	230,0	164	21,8	0,013 81	190,7	204	27,1	0,019 73	155,9	244	32,5	0,027 78	125,6
125	16,6	0,009 36	229,0	165	21,9	0,013 93	189,8	205	27,3	0,019 90	155,1	245	32,6	0,028 02	124,9
126	16,7	0,009 46	227,9	166	22,1	0,014 06	188,9	206	27,4	0,020 07	154,3	246	32,7	0,028 26	124,2
127	16,9	0,009 56	226,9	167	22,2	0,014 19	188,0	207	27,5	0,020 24	153,5	247	32,9	0,028 50	123,5
128	17,0	0,009 66	225,9	168	22,3	0,014 33	187,1	208	27,7	0,020 42	152,7	248	33,0	0,028 74	122,8
129	17,1	0,009 77	224,9	169	22,5	0,014 46	186,1	209	27,8	0,020 59	151,9	249	33,1	0,028 99	122,1
130	17,3	0,009 87	223,8	170	22,6	0,014 59	185,2	210	27,9	0,020 77	151,1	250	33,3	0,029 24	121,5
131	17,4	0,009 97	222,8	171	22,7	0,014 73	184,3	211	28,1	0,020 95	150,3	251	33,4	0,029 50	120,8
132	17,5	0,010 08	221,8	172	22,9	0,014 86	183,4	212	28,2	0,021 13	149,5	252	33,5	0,029 75	120,1
133	17,7	0,010 19	220,8	173	23,0	0,015 00	182,5	213	28,3	0,021 31	148,7	253	33,7	0,030 01	119,4
134	17,8	0,010 29	219,8	174	23,1	0,015 13	181,6	214	28,5	0,021 50	147,9	254	33,8	0,030 27	118,7
135	17,9	0,010 40	218,8	175	23,3	0,015 27	180,7	215	28,6	0,021 68	147,2	255	33,9	0,030 53	118,1
136	18,1	0,010 51	217,8	176	23,4	0,015 41	179,8	216	28,7	0,021 87	146,4	256	34,1	0,030 79	117,4
137	18,2	0,010 61	216,8	177	23,5	0,015 55	178,9	217	28,9	0,022 06	145,6	257	34,2	0,031 06	116,7
138	18,3	0,010 72	215,8	178	23,7	0,015 69	178,1	218	29,0	0,022 25	144,8	258	34,3	0,031 33	116,1
139	18,5	0,010 83	214,8	179	23,8	0,015 83	177,2	219	29,1	0,022 44	144,0	259	34,5	0,031 60	115,4

## 4 Method B using an Areameter apparatus

### 4.1 Principle

Filling two laboratory flasks of equal volume with nitrogen — one of the flasks containing the prepared test portion, the other empty — under atmospheric pressure at room temperature. Immersion of both flasks in liquid nitrogen.

At this temperature, the test portion adsorbs nitrogen, whereby a pressure difference is created between the flask containing the test portion and the reference flask.

Measurement of the pressure difference by means of a pressure differential gauge. Calculation of the specific surface area from the measured pressure difference, the feed pressure and the mass of the test portion.

### 4.2 Reagents

**4.2.1 Nitrogen**, in a cylinder, or other source of prepurified nitrogen, of recognized analytical quality.

**4.2.2 Liquid nitrogen.**

### 4.3 Apparatus

**4.3.1 Adsorption apparatus**<sup>1)</sup> (see figure 4), comprising a reference flask (G) and a sample flask (H) mounted with gas-tight connections.

These connections are provided with one valve each (A and B) by means of which the flasks can be connected with the atmosphere. The gas to be adsorbed is fed into each flask through the connection pieces.

**4.3.1.1** The flasks are made of glass which is resistant to sudden changes of temperature and have a volume of approximately 100 cm<sup>3</sup>. The volume difference between the two flasks shall not exceed 0,1 %.

The flask necks are made of calibrated precision glass tubes with an inside diameter of  $5 \pm 0,02$  mm. This ensures that several flasks can be used as sample or reference flasks without needing to adjust the equalizing volume for every combination.

**4.3.1.2** A U-tube is mounted between the two flasks, and the legs of the pressure gauge are connected by capillary tubes to the two adsorption flasks. By means of valve D, the two adsorption vessels can be either separated from one another or connected to one another via their capillary tubes. Using valve E, both liquid legs of the differential pressure gauge can be separated or joined together. The measuring fluid is dibutylphthalate.

The legs of the differential pressure gauge are made of calibrated precision glass tubing with an inside diameter of  $5 \pm 0,02$  mm. Therefore, any change in volume during the gas adsorption can be sufficiently accurately calculated. The feed capillary to the sample flask, owing to its short length, is considered as an equalizing volume which is adjusted during the preparation of the equipment (see the annex, clause A.2).

The gas is allowed into the equipment through valve C. If valves A, B and D are open, the gas flows through both flasks. If valves A and D are closed, the reference flask is shut off and only the sample flask is rinsed with the gas.

During measurement, only part of the volume enclosed by valves A, B and C is cooled to the measuring temperature by the liquid nitrogen. The remaining volume, at room temperature, may be only 10 % of the total volume. The connections to the adsorption flasks are therefore capillaries which almost completely fill out the necks of the flasks. In this manner, the volume of gas at room temperature is kept to a minimum.

NOTE — Procedures for commissioning new equipment and for control purposes are given in the annex.

**4.3.2 Regulating thermostat**<sup>1)</sup>, permitting maintenance of the adsorption flasks (with the test portions) at a constant temperature, and either rinsing with dry nitrogen or evacuation.

**4.3.3 Analytical balance**, accurate to 0,1 mg.

**4.3.4 Drying oven**, capable of being controlled at  $105 \pm 2$  °C.

**4.3.5 Cold bath**, containing the liquid nitrogen (4.2.2).

1) This apparatus is available commercially. Details may be obtained from the Secretariat of ISO/TC 45 (BSI) or ISO Central Secretariat.

#### 4.4 Preparation of the sample

The maximum indication (400 mm) on the differential pressure gauge corresponds to a surface area of approximately 50 m<sup>2</sup>. The mass of the test portion must therefore be adjusted so that  $\Delta h$  on the differential pressure gauge is as great as possible and at least 50 mm. If the approximate specific surface area is not known, preliminary tests with various masses of test portion shall be performed to establish the most suitable mass of test portion.

NOTE — As a guide, the following table gives masses of test portion according to specific surface area.

Specific surface area, m <sup>2</sup> /g	Mass of test portion, g
20	0,6 to 0,8
30	0,4 to 0,6
40	0,3 to 0,5
80	0,2 to 0,3
120	0,15 to 0,2
140	0,1 to 0,15
> 200	< 0,1

Introduce the test portion into the adsorption flask (see 4.3.1), taking care that no material adheres to the walls or the neck. Dry the samples in accordance with ISO 1126.

Before the determination, remove as much as possible of the matter that has already been adsorbed from the surface of the test portion. Carry out desorption by rinsing in a flow of nitrogen. The desorption temperature should be as high as possible and should be selected so that the desorption time is not long.

NOTE — For rubber grade furnace blacks, a desorption time of 30 min at 170 to 190 °C is sufficient.

After desorption, cool the test portion to room temperature under a flow of nitrogen and close and store the flasks until required for the determination.

Determine the mass of the test portion, after desorption, by weighing, to the nearest 0,1 mg, the adsorption flask containing the test portion and deducting the mass of the empty flask.

#### 4.5 Procedure

**4.5.1** Connect the adsorption flask containing the prepared test portion to the nitrogen source and open valves B and C. Allow nitrogen to flow into the flasks.

**4.5.2** Open valves D, A and E and place the flasks in a water bath controlled at  $23 \pm 2$  °C.

**4.5.3** After 5 to 10 min, determine the pressure difference in the flasks by closing valves A, B, C and D. If a pressure difference exists, reopen the valves in the order D, C, B and A and continue rinsing with nitrogen. Close valves A, B, C and D when the pressure is equal.

**4.5.4** When pressure equilibrium is attained, close valve E and stop the nitrogen flow by closing the valves.

**4.5.5** Wipe off the water drops from the flasks and immerse the flasks in the liquid nitrogen bath (4.3.5) to the lower mark of the neck and open valve E very slowly.

**4.5.6** After the resulting pressure differential has stabilized, read the difference in liquid heights of the U-tube arms to the nearest 0,5 mm.

**4.5.7** Close valve E. Replace the cold bath (4.3.5) with a water bath controlled at approximately 40 °C. After a few minutes, start the nitrogen flow and open the valves D, C, B, A and E in that order.

**4.5.8** When the connections have reached room temperature, close valves A, D and E and disconnect the sample flask.

#### 4.6 Expression of results

##### 4.6.1 Method of calculation

Calculate the specific surface area,  $S_m$ , in square metres per gram, from the formula

$$S_m = 1,187 \times 10^{-7} \left[ \frac{(1,044 \times 10^5) - p}{m} \right] \times \left[ \left( 13,6458 + (6,65 \times 10^{-5} p) \right) \cdot \Delta h + \frac{1}{\rho Q} \left( \frac{p}{77,6} - \frac{p_B}{295} \right) \right]$$

where

$$p = \frac{105,55 p_B}{393,11 + (0,0049 \Delta h)} - 10,2 \Delta h$$

and

$p$  is the equilibrium pressure, in pascals;

$m$  is the mass, in grams, of the test portion;

$\Delta h$  is the difference in liquid heights, in millimetres, in the U-tube arms;

$p_B$  is the atmospheric pressure, in pascals;

$\rho$  is the density, in megagrams per cubic metre, of the test sample, assumed to be equal to 1,8 Mg/m<sup>3</sup>.

NOTE — For specific surface areas greater than 1 m<sup>2</sup>/g, the term  $\frac{1}{\rho Q} \left( \frac{p}{77,6} - \frac{p_B}{295} \right)$  may be ignored.

Express the result to the nearest 0,1 m<sup>2</sup>/g.

**4.6.2 Calculation using a nomogram**

The calculation may be simplified by the use of a nomogram (see figure 5).

Draw a straight line connecting the measured value of  $\Delta h$  on the vertical  $\Delta h$  scale with the measured value of  $p_B$  on the vertical  $p_B$  scale. Record the value at the point of intersection of the line and scale  $A$ .

Draw a straight line connecting the measured value of  $\Delta h$  on the vertical  $\Delta h$  scale with the measured value of  $p_B$  on the inclined reduced  $p_B$  scale. Record the value at the point of intersection of the line and scale  $B$ .

The specific surface area, in square metres per gram, is given by the formula

$$S_m = \frac{A \Delta h}{m} + \frac{B}{\rho p}$$

where

$A$  and  $B$  are values derived from the nomogram as described above;

$\Delta h$ ,  $m$ ,  $\rho$  and  $p$  have the same meanings as in 4.6.1.

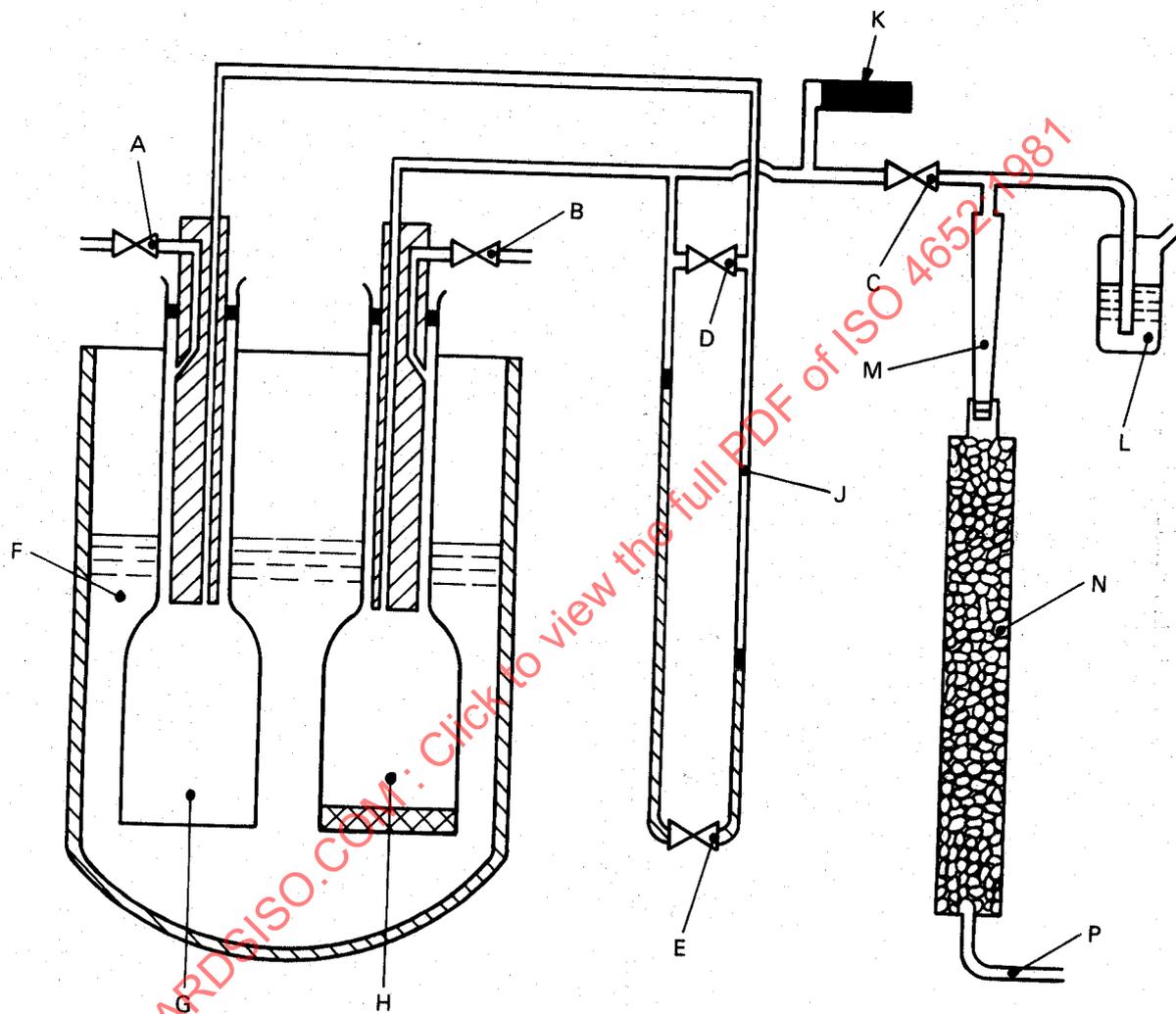
Express the result to the nearest 0,1 m<sup>2</sup>/g.

**4.7 Test report**

The test report shall include the following information :

- a) a reference to this International Standard (indicating "method B");
- b) complete identification of the sample;
- c) the conditions of test;
- d) the mass of test portion used;
- e) the result and the method of expression used.

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A to E Valves

F Cold bath

G Reference flask

H Sample flask

J Pressure differential gauge

K Equalizing volume

L Safety valve

M Flow meter

N Drying tower

P Nitrogen inlet

Figure 4 — Areameter adsorption apparatus

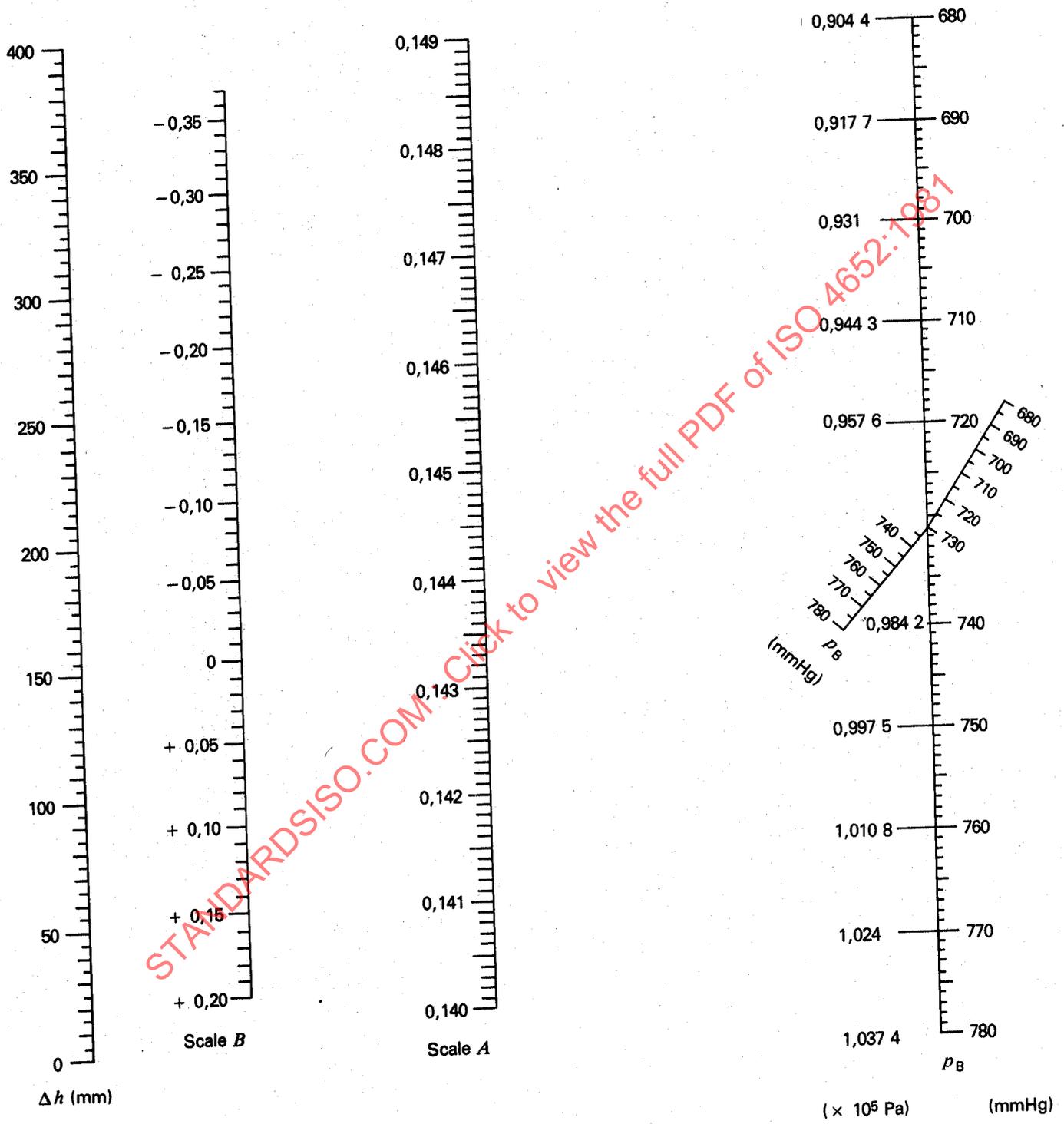


Figure 5 — Nomogram