

TECHNICAL REPORT

ISO
TR 6809

Third edition
1996-09-01

Rubber compounding ingredients — Carbon black — Standard reference blacks

*Ingrédients de mélange du caoutchouc — Noir de carbone —
Noirs de référence*



Reference number
ISO/TR 6809:1996(E)

Foreword

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The main task of technical committees is to prepare International Standards, but in exceptional circumstances a technical committee may propose the publication of a Technical Report of one of the following types:

- type 1, when the required support cannot be obtained for the publication of an International Standard, despite repeated efforts;
- type 2, when the subject is still under technical development or where for any other reason there is the future but not immediate possibility of an agreement on an International Standard;
- type 3, when a technical committee has collected data of a different kind from that which is normally published as an International Standard ("state of the art", for example).

Technical Reports of types 1 and 2 are subject to review within three years of publication, to decide whether they can be transformed into International Standards. Technical Reports of type 3 do not necessarily have to be reviewed until the data they provide are considered to be no longer valid or useful.

ISO/TR 6809, which is a Technical Report of type 3, 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 third edition cancels and replaces the second edition (ISO 6809:1989), which has been technically revised.

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Rubber compounding ingredients — Carbon black — Standard reference blacks

1. SCOPE

This Technical Report specifies the agreed values for the properties of Standard Reference Blacks (SRB) to be used in conjunction with methods of test for carbon black.

2. REFERENCES

ISO-1304-1985	Rubber compounding ingredients - Carbon black - Determination of iodine adsorption number - Titrimetric method.
ISO-4652-1-1994	Rubber compounding ingredients - Carbon black - Determination of specific surface area - Nitrogen adsorption methods. Part 1: Single-point procedure
ISO-4656-1-1992	Rubber compounding ingredients - Carbon black - Determination of dibutylphthalate absorption number - Part 1: method using absorptometer.
ISO-4656-2-1991	Rubber compounding ingredients - Carbon black - Determination of dibutylphthalate absorption number - Part 2: method using plastograph or plasticorder.
ISO-5435-1994	Rubber compounding ingredients - Carbon black - Determination of tinting strength.
ISO-6810-1995	Rubber compounding ingredients - Carbon black - Determination of surface area - CTAB adsorption methods.
ISO-6894-1991	Rubber compounding ingredients - Carbon black - Preparation of samples for determination of dibutylphthalate absorption number (compressed sample).
ASTM-D-4820-1993	Carbon black - Surface area by multipoint B.E.T. nitrogen adsorption.

Note: An ISO equivalent Standard in preparation in 1995.

3. AGREED VALUES

The agreed values for the properties of Standard Reference Blacks are given in the following tables 1, 2, 3, 4 and 5.

Table 1: agreed values of the Standard Reference Blacks certified in 1994 (SRB-5 series).

Table 2: agreed values of the Standard Reference Blacks certified in 1989 (SRB-4 series).

Table 3: agreed values of the Standard Reference Blacks certified in 1983 (SRB-3 series).

Table 4: agreed values of the Standard Reference Blacks certified in 1978 (SRB-2 series).

Table 5: agreed values of the Standard Reference Blacks certified in 1974 (SRB-1 series).

The tables 3, 4 and 5 are for historical documentation purposes only, as these Reference Materials are no more available commercially, for those who would still have some of them.

The SRB-4 were not exhausted totally, particularly the SRB-F4, by the end of 1994. This Standard will be supplied, till exhaustion, before switching to the SRB-F5.

For the sake of good order, the table 1 includes as well:

- the agreed values of the ITRB (Industry Tint Reference Black) used for calibration purposes in the test procedures for the determinations of the CTAB surface area and tinting strength of rubber grade carbon blacks.
- the accepted values of the current IRB (Industry Reference Black No. 6).

Property	Unit	A-5 (1)	B-5 (1)	C-5 (1)	D-5 (1)	E-5 (1)	F-5 (1)	G (5)	ITRB (2)	IRB No. 6 (3)	Method of test
Iodine adsorption number	g/kg	151,7 (± 1.62)	79,1 (± 0.82)	121,1 (± 0.96)	28,7 (± 1.08)	35,1 (± 1.08)	38,5 (± 1.20)		-	80 (± 1)	ISO 1304
CTAB surface area	10 ³ m ² /kg	127,5 (± 2.08)	77,4 (± 2.56)	117,2 (± 1.36)	29,4 (± 1.06)	37,2 (± 1.30)	40,9 (± 1.18)		83	-	ISO 6810
Nitrogen surface area	10 ³ m ² /kg	141,5 (± 1.02)	74,3 (± 0.68)	122,0 (± 1.82)	27,5 (± 0.32)	35,3 (± 0.58)	39,1 (± 0.58)		-	-	ASTM D-4820
Dibutylphthalate absorption of compressed sample	10 ⁻³ m ² /kg	118,4 (± 1.04)	86,5 (± 1.24)	98,7 (± 1.02)	57,8 (± 1.16)	72,7 (± 1.26)	88,9 (± 1.36)		-	87,2 (± 1)	ISO 6894
Dibutylphthalate absorption (4) - mass used	10 ⁻³ m ² /kg g	136,7 (± 1.24) 20	99,9 (± 1.10) 20	113,7 (± 0.96) 20	64,1 (± 1.06) 25	89,4 (± 1.32) 20	129,5 (± 1.02) 20	40	-	100 (± 1) 20	ISO 4656-1 or ISO 4656-2
Tinting strength	% ITRB	123,1 (± 1.34)	98,3 (± 1.04)	117,5 (± 1.92)	49,5 (± 1.20)	55,3 (± 0.72)	58,0 (± 0.98)		100	99,2 ($\pm 1,5$)	ISO 5435
Type	-	N 135	N 330	N 220	N 762	N 660	N 683	N 990	N 330	N 330	-

Table 1 - Agreed values for properties of standard reference blacks dried at 125 °C

() Two standard deviations - 95 % confidence. "The limits of uncertainty" are shown to indicate ranges within which replicate interlaboratory testing has been able to fix the consensus standard values; they are not to be confused with or interpreted as the repeatability precision of the respective test methods.

(1) Samples of Standard Reference Blacks are available from:
Titan Specialties, P. O. Box 2316, Pampa, TX 79066, - 2316, USA

(2) ITRB is available from:
Titan Specialties, P. O. Box 2316, Pampa, TX 79066, - 2316, USA

(3) Industry reference black IRB No. 6 is available from:
J. M. Huber Customer Service Dept., P. O. Box 2831, Borger, TX 79008 - 2831, USA
and
Degussa AG, AC-KP-VK- Postfach 11 05 33, Weißfrauenstraße 9, D-60311 Frankfurt, Germany

(4) Data obtained after mechanical calibration of absorptometers with SRB-F4.

(5) In preparation early 1996

Property	Unit	A-4 (1)	B-4 (1)	C-4 (1)	D-4 (1)	E-4 (1)	F-4 (1)	Method of test
Iodine adsorption number	g/kg	82,7 ($\pm 0,57$)	79,3 ($\pm 0,65$)	121,1 ($\pm 1,01$)	26,5 ($\pm 1,03$)	36,0 ($\pm 0,38$)	37,7 ($\pm 0,70$)	ISO 1304
CTAB surface area	10 ³ m ² /kg	82,5 ($\pm 2,31$)	79,1 ($\pm 2,03$)	120,5 ($\pm 2,71$)	24,9 ($\pm 1,74$)	36,6 ($\pm 1,13$)	40,5 ($\pm 1,01$)	ISO 6810
Nitrogen surface area	10 ³ m ² /kg	77,3 ($\pm 0,53$)	74,4 ($\pm 1,06$)	124,2 ($\pm 1,09$)	24,1 ($\pm 0,25$)	34,6 ($\pm 0,54$)	38,5 ($\pm 0,41$)	ASTM D-4820
Dibutylphthalate absorption of compressed sample	10 ⁻⁵ m ³ /kg	68,3 ($\pm 1,30$)	86,6 ($\pm 1,35$)	108,9 ($\pm 1,09$)	58,9 ($\pm 1,11$)	75,7 ($\pm 1,19$)	88,0 ($\pm 1,33$)	ISO 6894
Dibutylphthalate absorption - mass used	10 ⁻⁵ m ³ /kg g	70,8 ($\pm 0,63$) 20	100,0 ($\pm 0,92$) 20	131,0 ($\pm 0,97$) 20	64,8 ($\pm 0,88$) 25	90,4 ($\pm 1,49$) 20	130,6 ($\pm 1,27$) 20	ISO 4656-1 or ISO 4656-2
Tinting strength	% ITRB	110,8 ($\pm 1,17$)	98,5 ($\pm 1,35$)	119,5 ($\pm 1,48$)	41,2 ($\pm 0,80$)	55,2 ($\pm 0,68$)	55,8 ($\pm 0,96$)	ISO 5435
Type	-	N 326	N 330	N 121	N 762	N 660	N 683	-
Table 2 - Agreed values for properties of standard reference blacks dried at 125 °C								

() Two standard deviations - 95 % confidence. "The limits of uncertainty" are shown to indicate ranges within which replicate interlaboratory testing has been able to fix the consensus standard values; they are not to be confused with or interpreted as the repeatability precision of the respective test methods.

(1) Samples of Standard Reference Blacks are available from:

Titan Specialties, P. O. Box 2316, Pampa, TX 79066 - 2316, USA

Property	Unit	A-3	B-3 (IRB-5)	C-3	D-3	Method of test
Iodine adsorption number	g/kg	22,5 ± 1	79,4 ± 1	111,4 ± 1	32,0 ± 1	ISO 1304
CTAB surface area	10 ³ m ² /kg	24,2 ± 1	79,9 ± 1	113,0 ± 1	40,6 ± 1	ISO 6810
Nitrogen surface area	10 ³ m ² /kg	23,6 ± 1	79,5 ± 1	119,8 ± 1	37,9 ± 1	ISO 4652
Dibutylphthalate absorption of compressed sample	10 ⁻⁵ m ³ /kg	58,9 ± 1	89,2 ± 1	102,0 ± 1	89,0 ± 1	ISO 6894
Dibutylphthalate absorption - mass used	10 ⁻⁵ m ³ /kg g	67,0 ± 1 25	101,2 ± 1 20	119,8 ± 1 20	129,8 ± 1 20	ISO 4656-1 or ISO 4656-2
Tinting strength	% ITRB	110,8 (± 1,17)	98,5 (± 1,35)	119,5 (± 1,48)	41,2 (± 0,80)	ISO 5435
Type	-	N 762	N 330	N 234	N 683	-

Table 3 - Agreed values for properties of standard reference blacks dried at 125 °C

The SRB-3 are no more available.

Property	Unit	A-2	B-2 (IRB-4)	C-2	D-2	E-2	F-2	Method of test
Iodine adsorption number	g/kg	80,3 ± 1	80,5 ± 1	116,5 ± 1	36,1 ± 1	-	-	ISO 1304
CTAB surface area	10 ³ m ² /kg	-	-	-	-	-	-	ISO 6810
Nitrogen surface area	10 ³ m ² /kg	85,0 ± 1	80,5 ± 1	122,3 ± 1	38,7 ± 1	-	-	ISO 4652
Dibutylphthalate absorption of compressed sample	10 ⁻⁵ m ³ /kg	66,6 ± 1	86,6 ± 1	103,2 ± 1	88,6 ± 1	76,7 ± 1	59,8 ± 1	ISO 6894
Dibutylphthalate absorption - mass used	10 ⁻⁵ m ³ /kg g	71,0 ± 1 20	97,5 ± 1 20	122,7 ± 1 20	129,5 ± 1 20	90,3 ± 1 20	65,1 ± 1 25	ISO 4656-1 or ISO 4656-2
Tinting strength (1)	% ITRB	113,5 ± 1,8	105,9 ± 0,9	117,5 ± 1,5	56,0 ± 1,5	-	-	ISO 5435
Type	-	N 326	N 330	N 234	N 683	N 660	N 762	-

Table 4 - Agreed values for properties of standard reference blacks dried at 125 °C

(1) Measured with NBS zinc oxide.

The SRB-2 are no more available.

Property	Unit	A-1	B-1 (IRB-4)	C-1	Method of test
Iodine adsorption number	g/kg	27,9 ± 1	81,0 ± 1	120,4 ± 1	ISO 1304
CTAB surface area	10 ³ m ² /kg	-	-	-	ISO 6810
Nitrogen surface area	10 ³ m ² /kg	30,7 ± 1	79,8 ± 1	140,9 ± 1	ISO 4652
Dibutylphthalate absorption of compressed sample	10 ⁻⁵ m ³ /kg	64,0 ± 1	86,4 ± 1	99,8 ± 1	ISO 6894
Dibutylphthalate absorption mass used	10 ⁻⁵ m ³ /kg g	72,4 ± 1 25	96,6 ± 1 20	122,6 ± 1 20	ISO 4656-1 or ISO 4656-2
Tinting strength (1)	% ITRB	56,5 ± 1,8	106,4 ± 1,3	128,5 ± 1,6	ISO 5435
Type	-	N 774	N 330	N 234	-
Table 5 - Agreed values for properties of standard reference blacks dried at 125 °C					

(1) Measured with NBS zinc oxide.

The SRB-1 are no more available.