



## BRACHI TESTING SERVICES

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<b>RAPPORTO DI PROVA N°:</b> Test Report N°:	005587/20
<b>DITTA PRESENTATRICE:</b> Company:	TESSILFIBRE SPA - VIA XXV APRILE, 33 - POGGIO A CAIANO (PO)
<b>CAMPIONE:</b> Sample:	Sample of textile fibers colour black named STYLE ACRYLYC TOW BLACK R 99000 DYED LOT 2070 COL BLACK.
<b>CAMPIONAMENTO:</b> Sampling:	Made by the customer
<b>DATA RICEVIMENTO:</b> Date of Receipt:	March 03, 2020
<b>DATA ESECUZIONE PROVE</b> Test execution date:	From the date of receipt to the date of issue
<b>OGGETTO:</b> Subject:	Various Analysis

### RISULTATI DI LABORATORIO

### TEST RESULTS



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**IL DIRETTORE**  
**P.I. Primo Brachi**

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<b>TEXTILES: METHODS FOR DETERMINATION OF CERTAIN AROMATIC AMINES DERIVED FROM AZO COLORANTS. PART 1: DETECTION OF THE USE OF CERTAIN AZO COLORANTS ACCESSIBLE WITH OR WITHOUT EXTRACTING THE FIBRES</b>				
Test method:	ISO 14362-1:2017			
Procedure:	Reductive cleavage of the azo group with sodium dithionite in a citrate buffered aqueous solution (pH 6,0; 70°C), spe purification, liquid-liquid extraction in Tertbutylmethylether phase. Gaschromatographic quantitative determination with mass detector GC/MSD.			
<b>NOTE:</b>				
A) The use of certain azo colorants (Table 1) cannot be reliably ascertained without additional information, e.g. the chemical structure of the colorants used.				
B) The product from which the sample was taken may have been coloured with colorants whose structures contain the amines but not azo bound.				
C) The product from which the sample was taken may have been coloured with an azo colorant whose structure does not contain preformed 4-methoxy-m-phenylenediamine but 2-amino-4-nitroanisole. In the course of the analytical procedure the azo colorant will release 2-amino-4-nitroanisole which in turn will form 4-methoxy-m-phenylenediamine.				
D) Care should be taken that detected aromatic amines originate from azo colorants and not for other materials such as polyurethane.				
SAMPLE:	black			
<b>RESULTS</b>				
AROMATIC AMINES	CAS	NOTE	DETECTIO N LIMIT (ppm)	RESULTS (ppm)
4 - Aminobiphenyl	92-67-1	A,B	1	N.D.
Benzidine	92-87-5	-	1	N.D.
4 - Chloro - o - Toluidine	95-69-2	-	1	N.D.
2 - Naphthylamine	91-59-8	A,B	1	N.D.
o - Aminoazotoluene (a)	97-56-3	-	1	N.D.
5 - Nitro-o-toluidine (a)	99-55-8	A,C	1	N.D.
p - Chloroaniline	106-47-8	-	1	N.D.
2,4 - Diaminoanisole	615-05-4	-	1	N.D.
4,4' - Diaminodiphenylmethane	101-77-9	-	1	N.D.
3,3' - Dichlorobenzidine	91-94-1	-	1	N.D.
3,3' - Dimethoxybenzidine	119-90-4	-	1	N.D.
3,3' - Dimethylbenzidine	119-93-7	-	1	N.D.
3,3' - Dimethyl-4,4' - Diaminodiphenylmethane	838-88-0	-	1	N.D.
p-Cresidine	120-71-8	-	1	N.D.
4,4' - Methylene-bis-(2-chloroaniline)	101-14-4	-	1	N.D.

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4,4' - Oxydianiline	101-80-4	-	1	N.D.
4,4' - Thiodianiline	139-65-1	C	1	N.D.
o - Toluidine	95-53-4	-	1	N.D.
2,4 - Toluenediamine	95-80-7	B,D	1	N.D.
2,4,5 - Trimethylaniline	137-17-7	-	1	N.D.
o - Anisidine	90-04-0	-	1	N.D.
4 - Aminoazobenzene (b)	60-09-3	-	1	N.D.
*2,4-xylidine	95-68-1	-	1	N.D.
*2,6-xylidine	87-62-7	-	1	N.D.
			DETECTED	NOT DETECTED
Aniline	62-53-3			X
1,4- Phenylendiamine	106-50-3			X
<i>Legend: ND= not detectable, lower than dection limit</i>				
<i>Note: (a) the CAS number 97-56-3 and 99-55-8 are further reduced to CAS number 95-53-4 and 95-80-7.</i>				
<i>(b) azo colorants that are able to form 4-aminoazobenzene, generate under the condition of this method aniline (CAS 62-53-3) and 1-4-phenylendiamine (CAS 106-50-3). Due to detection limits, only aniline may be detected. The presence of these colorants should be tested by EN 14362-3 (textiles).</i>				

### DETERMINATION OF SOLUBLE HEXAVALENT CHROMIUM

Process :

Test Method

Process

: BS 6810: 1987 SEZ. 4 E 8.5

: Extraction with sodium tetraborate solution at 40°C for 30 min., reaction with dyphenylcarbazide (DPC) and spectrophotometric determination at 540 nm.

Results:

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	DETECTION LIMIT (mg/kg)	QUATIFICATION LIMIT (mg/kg)	TEST 1 (mg/kg)	TEST 2 (mg/kg)	AVERAGE (mg/kg)
Chromium VI (CrVI)	1	3	N.D.	N.D.	---

*Legend: N.D. not detectable (lower then detection limit)*

*Note: determination of volatile substances is carry out only if the sample is positive.*

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## \* DETERMINATION OF HEAVY METALS TOTAL CONTENT

Process:

Test method: Internal Method: Microwave acid digestion followed by ICP-OES determination based on CPSC-CH-E1002-08.03 -2012

Results:

Heavy Metal	DETECTION LIMIT	RESULTS (mg/kg)
Lead	0,1	N.D.
Cadmium	0,1	N.D.
Mercury	0,1	N.D.

N.D. = Not Detectable

## DETERMINATION OF PHTHALATES (CPSC)

Process:

Test Method : EN ISO 14389:2014  
Method : Dissolution with tetrahydrofurane (THF), precipitation of PVC residue with hexane and gaschromatographic analysis with mass detector (GC-MS)

Correct mass (mc): --

Total mass (mt) : 0,350 g

Not detectable : </= LOD

Testing date : 12/03/2020

Results:

PHTHALATES	CAS-Nr	LOD (mg/kg)	LOQ (mg/kg)	Results (mg/kg)	Results %
Dibutyl phthalate (DBP)	84-74-2	3	10	N.D.	
Benzyl butyl phthalate (BBP)	85-68-7	3	10	N.D.	
Di (2 ethylexyl) phthalate (DEHP)	117-81-7	3	10	N.D.	
Sum of three (DBP+BBP+DEHP)		--	--	N.D.	
Di iso buthyl phthalate (DIBP)	84-69-5	3	10	N.D.	
Din-n-octyl phthalate (DNOP)	117-84-0	5	15	N.D.	
Di iso nonyl phthalate (DINP)	28553-12-0, 68515-48-0	15	50	N.D.	
Di iso decyl phthalate (DIDP)	26761-40-0 68515-49-1	15	50	N.D.	
Sum of three (DnOP+Didp+DINP)		--	--	N.D.	
Di-C6-8-branched alkylphthalates (DIHP)	71888-89-6	15	50	N.D.	
Di-pentylphthalate (n-,iso-or mixed) (DPP)	131-18-0, 605-50-5, 776297-69-9	3	10	N.D.	
Di-(2-methoxyethyl)-phthalate (DMEP)	117-82-8	15	50	N.D.	
Di n-hexyl phthalate (DNHP)	84-75-3	3	10	--	

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Legend: n.d.: not detectable

<b>DETERMINATION OF ETHOXYLATED ALKYLPHENOLS (APEOs)</b>			
Test Method	ISO 18254-1: 2016		
Method	Extraction with methanol for 60 min at 70°C in ultrasonic bath, filtration and liquid chromatography determination with mass detector (LC/MS/MS)		
<b>Results:</b>			
Compounds	CAS	Results mg/kg	
Nonylphenol monoethoxylates (NPEO 1)	104-35-8	N.D.	
Nonylphenol diethoxylates (NPEO 2)	20427-84-3	N.D.	
Nonylphenol polyethoxylates (NPEO 3-18)	68412-54-4, 9016-45-9, 127087-87-0, 37205-87-1, 26027-38-3	N.D.	
Octylphenol monoethoxylates (OPEO 1)	51437-89-9, 2315-67-5	N.D.	
Octylphenol diethoxylates (OPEO 2)	51437-90-2, 2315-61-9	N.D.	
Octylphenol polyethoxylates (OPEO 3-16)	9002-93-1, 68987-90-6, 9036-19-5	3,7	
Detection limit (LOD): 0,3 mg/kg			
Quantification limit (LOQ): 0,4 mg/kg			
NOTE: ND: not detectable, lower than detection limit			

<b>DETERMINATION OF ALKYLPHENOLS (APs)</b>			
Test Method	Internal method MI E025:2008 rev 01		
Method	Extraction with methanol for 60 min at 70°C in ultrasonic bath filtration and gaschromatography determination with mass detector (GC/MS/MS)		
<b>Results:</b>			
Compounds	CAS	RESULTS mg/kg	
Nonylphenols (NP)	84852-15-3, 25154-52-3, 104-40-5, 11066-49-2, 90481-04-2	N.D.	
Octylphenols (OP)	140-66-9, 1806-26-4, 27193-28-8	N.D.	
Detection limit (LOD) : 0,2 mg/kg			
Quantification limit (LOQ) : 0,4 mg/kg			
NOTE: ND not detectable, lower than detection limit			

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## \*DETERMINATION OF PERFLUORINATED COMPOUND

Process:

Test Method : Based on CEN/TS 15968: 2010  
solvent extraction, LC-MS-MS/ GC-MSMS analysis

Results:

	CAS N.	LOD µg/l	RESULT µg/kg
Perfluorbutansulfonic acid (PFBS)	29420-49-3/ 375-73-5	0,4/1	n.d.
Perfluorohexane sulfonic acid (PFHxS)	3871-99-6/ 355-46-4	0,4/1	n.d.
Perfluoro 1-heptanesulphonic acid (PFHpS)	375-92-8/ 60270-55-5	0,4/1	n.d.
Perfluorobutanoic acid (PFBA)	375-22-4	0,4/1	n.d.
Perfluoropentanoic acid (PFPeA)	2706-90-3	0,4/1	n.d.
Perfluoro n-hexanoic acid (PFHxA)	307-24-4	0,4/1	n.d.
Perfluoro n-heptanoic acid (PFHpA)	375-85-9	0,4/1	n.d.
7H-Perfluoroheptanoic acid (HPFHpA)	1546-95-8	0,4/1	n.d.
Perfluorooctane sulfonic acid (PFOS)	56773-72-3/ 1763-23-1	0,4/1	n.d.
Perfluorooctane-sulfonamide (PFOSA)	754-91-6	0,4/1	n.d.
Perfluoro n-octanoic acid (PFOA)	335-67-1/ 3825-26-1	0,01	n.d.
Perfluoro n-nonanoic acid (PFNA)	375-95-1	0,1	n.d.
Perfluoro n-decanoic acid (PFDA)	335-76-2	0,4/1	n.d.
Perfluoroundecanoic acid (PFUnA)	2058-94-8/ 4234-23-5	0,4/1	n.d.
2H,2H,3H,3H- Perfluoroundecanoic acid (4HPFUnA)	34598-33-9	0,4/1	n.d.
Perfluorododecanoic acid (PFDoA)	307-55-1	0,4/1	n.d.
Perfluorotridecanoic acid (PFTrDA)	72629-94-8	0,4/1	n.d.
Perfluorotetradecanoic acid (PFTeDA)	376-06-7	0,4/1	n.d.
Perfluoro-3,7-dimethyloctanoic acid (PF-3,7-DMOA)	172155-07-6	0,4/1	n.d.
1H, 1H,2H,2H,Perfluorooctanesulfonic acid (H4PFOS 6-2)	27619-97-2	0,4/1	n.d.
2- (N- methylperfluoro-1-octonesulfonamido) - ethanol(N-MeFOSE)	24448-09-7	0,4/1	n.d.
2- (N- Ethylperfluoro-1-octonesulfonamido) - ethanol(N-EtFOSE)	16191-99-2	0,4/1	n.d.
N-methylperfluoro-1-octanesulfonamide (N-MeFOSA)	31506-32-8	0,4/1	n.d.
N-Ethylperfluoro-1-octanesulfonamide (N-EtFOSA)	4151-50-2	0,4/1	n.d.
2H,2H- perfluorodecaneacid ( 2H-PFDeA)	--	0,4/1	n.d.
21H,1H,2H,2H-Perfluorooctanesulphonic acid (1H,1H,2H,2H- PFOS)	27854-31-5	0,4/1	

Legend: n.d.:not detectable (lower than detection limit LOD)

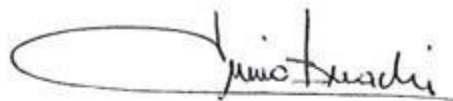
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Prato, 19 marzo 2020



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Direttore



FINE DEL RAPPORTO DI PROVA  
END OF TEST REPORT



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