

ASSOCIATION FRANCAISE DE NORMALISATION

AFNOR – 11 av. F. de Pressensé – 93571 SAINT-DENIS LA PLAINE

REFERENCE POWDERS

For granulometric analysis and mass volume measurements
(equipment & method testing)

Powders prepared and distributed under the aegis of the Reference Materials Department of the National Bureau of Metrology, and supervised by the Competent Commission of the AFNOR.

Distributing agency :

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Certified ISO 9001 by AFAQ N° QUAL/1996/6027a

and certified ISO 14001 by AFAQ N° ENV/2002/18334a

I – IMPORTANT RECOMMENDATIONS :

Before using the powder, it is always necessary to pour into a 150 cm³ bottle and shake in order to re-homogenize it.

After use, the powder must never be re-used.

II – CHARACTERISTICS :

- Chemical composition : Alumine. Major impurities : $Na_2O + Fe_2O_3 + SiO_2 + CaO$ (less than 0,1 % for powder A and less than 0,3 % for powder B).
- Method of preparation : *electro-melted product obtained by crushing.*
- State of surface and shape of grains : *see figures below.*
- Inner porosity of grains : *négligible.*
- Specific volume mass in g/cm³ :

	<u>powder A</u>	<u>powder B</u>
By liquid pycnometry	3.95	3,79
By gas pycnometry	3.96	3.84



Powder A (G : 3000)



Powder B (G : 20 000)

Seen through a Scanning Electron Microscope

III – AREA MEASUREMENTS :

- By permeametry (cm ² /g) :	<u>powder A</u>	<u>powder B</u>
- By BET adsorption (cm ² /g) :	2 300	10 300
- Obligatory porosity :	5 000	31 000
	0,57	0,67

Statistic table

	Repeatability of measurements		Reproducibility of measurements	
	Permeability	Adsorption BET	Permeability	Adsorption BET
Powder A . standard deviation (cm ² /g) . Coefficient of variation (%)	12 0,55	X	100 4,7	1 200 23,5
Powder B . standard deviation (cm ² /g) . Coefficient of variation (%)	250 2,5		1 300 12,5	5 800 18,5

IV – PARTICULE SIZE ANALYSIS :

The average granulometric compositions (results of some 40 analyses per method) are given in the tables below :

Ø (µm)	POWDER A % < Ø						
	M	L	C	E	P	S	B
1,00		4,4					
1,20	1,8						
1,25	1,7						
1,50		4,7					
1,60	2,6			1,8			
2,00	3,0	6,0	1,4	2,6			
2,50	3,2		1,3	3,4	1,3		
3,00		6,2					
3,15	3,6		1,5	4,3	1,5		
4,00	4,5	6,3	1,5	6,3	1,7		
5,00	6		1,7	11,5	2,5		5,1
6,00		6,5					
6,30	10,7		2,7	26,2	3,9		6,9
8,00	19,7	7,1	10,2	47,8	11,3		13,4
10,00	43,9		44,7	68,1	37,2		28,7
12,00		44,5					
12,50	71,4		85,5	86,1	73,9	83,1	60,2
16,00	86,7	77,6	98,5	91,1	85,6	95,9	86,3
20,00	91,2		99,4	96,9	89,1	99,2	92,9
24,00		98,2					
25,00	93,8		99,6		94		93,8
31,50	96,2		99,9				
32,00		98,7					
40,00	98,3						
48,00		99,8					
50,00	98,3						
63,00	98,8						
64,00		99,9					

Median diametre - Laser Cilas : 12,7 µm

Ø (µm)	POWDER B % < Ø						
	M	L	C	E	P	S	B
1,00	16,6	14,5	13,8			9,1	
1,25	25		22,3			20,2	
1,50		22,9					
1,60	33		32,4	32,9	43,8	33,1	
2,00	44,3	46,5	47,2	42,8	48,4	46,1	42,7
2,50	54,4	71,2	61,1	52,7	53	58,6	52,9
3,00							
3,15	66,1		73,9	62,7	61,0	68,6	63,2
4,00	75,0	82,0	81,9	72,0	69,3	77,8	71,9
5,00	79,8		86,2	81,3	69,3	77,8	72,8
6,00		90,0					
6,30	83,1		87,7	86,2	73,2	82,6	77,7
8,00	87	93,6	88,8	91,3	76,5	87,2	83,1
10,00	90		91,7	94,4	80,3	91,2	88,7
12,00		95,9					
12,50	92,8		94,6	96,6	84	93,5	92,6
16,00	95,2	99,0	98,4	98	84,3	95,8	96
20,00	96,0		98,7	99,0	86,1		98,1
24,00		100,0					
25,00	97,3			99,4	90		
31,50	98,3						

Median diametre - Laser Cilas : 2,1 µm

- M Total of all methods together
- L Laser Cilas granulometry
- C Coultronic counter
- E Bacho elutriator
- P Andreasen Pipette
- S Coultronic Sédigraph
- B Martin scale

V – BIBLIOGRAPHY SUMMARY :

- NF X 11-601 - Sieving and granulometry – Determination of the area per unit of mass or volume of Powders by permeametry – Lea and Nurse method.
- NF X 11-602 - Determination of the area per unit of powders by various methods of air permeametry.
- NF X 11-621 - Determination of the area per unit mass of powders (specific surface) by means of gas Adsorption – B.E.T. method : volumetric measurement of the adsorption of nitrogen at low temperatures.
- NF X 11-622 - Determination of the area per unit mass of powders (specific surface) by means of gas adsorption. Various readings of the basic method.

Inter-laboratory test programmes organized by the AFNOR « Measurement of mass areas » Commission