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Road vehicles — Test dust for filter evaluation —

Part 1:
Arizona test dust

Véhicules routiers — Poussière pour l'essai des filtres —

Partie 1: Poussière d'essai d'Arizona

Reference number
ISO 12103-1:1997(E)

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.

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.

International Standard ISO 12103-1 was prepared by Technical Committee ISO/TC 22, *Road vehicles*, Subcommittee SC 7, *Injection equipment and filters for use on road vehicles*.

ISO 12103 consists of the following parts, under the general title *Road vehicles — Test dust for filter evaluation*:

— *Part 1: Arizona test dust*

— *Part 2: Aluminium oxide test dust*

Annexes A and B of this part of ISO 12103 are for information only.

International Organization for Standardization
Case postale 56 • CH-1211 Genève 20 • Switzerland
Internet central@iso.ch
X.400 c=ch; a=400net; p=iso; o=isocs; s=central

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Introduction

This part of ISO 12103 specifies four grades of test dusts made from desert sand, which is composed of natural occurring compounds that motor vehicles are commonly subjected to. These test dusts are used to determine performance of filtration systems. Due to the abrasive characteristics of these materials, they have also been used in wear studies involving bearings, seals, fan blades, windshield wipers, etc.

This part of ISO 12103 specifies the particle size distribution of these four dusts by volume, as opposed to by number. The particle size distribution by number will be added to a revision of this part of ISO 12103.

Dusts complying with the volume distribution specified in this part of ISO 12103 are not appropriate for calibration of particle counters. For this purpose refer to ISO 4402, which is currently under review.

Road vehicles — Test dust for filter evaluation —

Part 1:
Arizona test dust

1 Scope

This part of ISO 12103 defines particle size distribution and chemical content limits involving four grades of test dust made from Arizona desert sand.

2 Test dust description

Test dusts according to this part of ISO 12103 are manufactured from Arizona desert sand. Arizona desert sand is a naturally occurring contaminant consisting primarily of silicon dioxide with smaller amounts of other compounds. It is collected from a select area of Arizona desert, jet-milled and classified to specific particle size.

NOTE — Arizona desert sand has also been referred to as Arizona road dust, Arizona test dust, Arizona silica, AC fine or coarse test dust, ACFTD or ACCTD, and SAE fine or coarse test dust (see annex A).

Arizona desert sand has a density of approximately 2 650 kg/m³. Bulk density of ISO-specified test dusts made from Arizona sand varies with particle size (see table 1).

Table 1 — Bulk density

Category	**Approximate bulk density** kg/m^3
ultrafine	500
fine	900
medium	1 025
coarse	1 200

3 Test dust designation

Arizona test dusts are available in four standard categories, designated as follows:

— ISO 12103-A1 for ultrafine test dust

— ISO 12103-A2 for fine test dust

— ISO 12103-A3 for medium test dust

— ISO 12103-A4 for coarse test dust

4 Particle size distribution

Particle size distribution is determined using a Coulter[1] Multisizer IIe™ particle size analyser. Table 2 specifies cumulative volume particle size limits for ISO-specified test dusts made from Arizona desert sand.

Table 2 — Particle size distribution

Size µm	Maximum volume fraction, %			
	A1 ultrafine	A2 fine	A3 medium	A4 coarse
1	1 to 3	2,5 to 3,5	1 to 2	0,6 to 1
2	9 to 13	10,5 to 12,5	4,0 to 5,5	2,2 to 3,7
3	21 to 27	18,5 to 22,0	7,5 to 9,5	4,2 to 6,0
4	36 to 44	25,5 to 29,5	10,5 to 13,0	6,2 to 8,2
5	56 to 64	31 to 36	15 to 19	8,0 to 10,5
7	83 to 88	41 to 46	28 to 33	12,0 to 14,5
10	97 to 100	50 to 54	40 to 45	17,0 to 22,0
20	100	70 to 74	65 to 69	32,0 to 36,0
40	—	88 to 91	84 to 88	57,0 to 61,0
80	—	99,5 to 100	99 to 100	87,5 to 89,5
120	—	100	100	97,0 to 98,0
180	—	—	—	99,5 to 100
200	—	—	—	100

5 Analysis equipment and operating procedure

5.1 Particle size analysis procedure

Analysis of ISO-specified Arizona test dusts shall be performed using a Coulter Multisizer IIe™ particle size analyser.

The Coulter Multisizer IIe™ measures particles individually by volume (mass) with a high degree of resolution. A dilute suspension of the sample to be analysed is made in Isoton II electrolyte solution complying with the Coulter manufacturer specification. The suspension is then stirred and drawn through a small aperture by means of a vacuum source.

An electrical current, passing through the aperture between two electrodes, enables the particles to be sensed by momentary changes in the electrical impedance as they pass through the aperture, since each particle displaces its own volume of electrolyte solution within the aperture. These changes in impedance are detected as a series of voltage pulses where the magnitude of each pulse is essentially proportional to the volume of the particle that produced it. These pulses are then amplified, counted and allocated to the correct size class. This principle allows thousands of individual particles to be measured every second, accurately, and in three dimensions.

Individual apertures are capable of measuring particles ranging between 2 % and 60 % of the aperture diameter. Therefore it is necessary to use multiple apertures to measure broad band particle size ranges found in fine, medium, and coarse test dusts according to this part of ISO 12103.

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1) Coulter is a trade name. This information is given for the convenience of the users of this part of ISO 12103 and does not constitute an endorsement by ISO of this company or its products.

Multiple aperture procedure involves removal of particles larger than aperture capacity by wet sieving prior to analysis. Data generated from individual apertures is mathematically correlated to determine full range particle size distributions.

Table 3 — Aperture/sieve correlation size

Dust grade	**Aperture procedure**	**Aperture size** µm	**Sieve size** µm
ultrafine	single	30	none
fine	multiple	30	20
		100	45
		280	none
medium	multiple	30	20
		100	45
		280	none
coarse	multiple	30	20
		100	45
		200	75
		400	none

The Coulter Multisizer IIe™ instrument offers many set-up options that are selected prior to sample analysis.

Set-up mode options shall read as follows:

— channels: 256;

— edit: off;

— coincidence corr: off.

Sample data accumulation screen options should be set as follows:

— *x*-axis: diameter (logarithmic scale);

— *y*-axis: volume difference.

Blank subtraction option should be used in conjunction with 30 µm aperture tube analysis. Blank subtraction involves running a background count of particulates in the electrolyte prior to sample analysis. The background count is then subtracted from the sample data. The majority of background counts occur in the first 20 channels of data. Multiple aperture reduction of data eliminates use of the first 20 channels of data in all apertures, except the smallest or 30 µm size. Therefore, it is not necessary to use blank subtract with larger apertures.

Typical sample preparation is as follows:

a) Place one or two drops of suitable dispersant into a clean 20 ml vial.

b) Add 1 ml of water to dispersant.

c) Add 5 mg to 10 mg sample of test dust. Insure that sample removed is representative of lot of test dust.

d) Mix by gently moving the vial in a circular motion. Be careful not to create bubbles.

e) Add approximately 10 ml deionized water.

f) Place sample vial and contents in an ultrasonic bath for 10 s. Ultrasonic bath should be approximately 80 W power.

g) If the sample prepared is to be used with 280 µm or 400 µm aperture tubes, proceed to step j).

h) Pour the sample into a thoroughly cleaned 76 mm diameter test sieve of appropriate mesh size to remove oversized particles. Rinse sample vial using a small amount of deionized water so that rinse solution drains into test sieve.

i) Pour sample that passed through the test sieve into a clean vial. Rinse test sieve pan with deionized water to remove sample residue and include in sample vial.

j) Using a pipette, mix sample and liquid in vial so that all particles are suspended in solution. Withdraw a portion of prepared sample with pipette and place into stirred electrolyte solution. Add sample until concentration index reaches 5 % to 10 %. Stirring speed shall be high enough to keep largest particles in suspension.

k) Start accumulation process. It is necessary to accumulate a minimum of 1 200 000 particles per aperture to obtain repeatable multi-aperture data. Larger aperture analysis requires repeated stopping of the accumulation process to refill electrolyte solution, add additional sample, and restart accumulation.

All of the prepared sample shall be consumed or counted when using 280 µm or 400 µm apertures. This is necessary because it is virtually impossible to remove a representative portion of the prepared sample if it contains particles above 75 µm size.

l) When accumulation of data is complete using all apertures, proceed to "multi-tube overlap" or multi-aperture data reduction. In order to obtain repeatable results, it is necessary to consider as many channels as possible in the overlap process. This process is easier to accomplish using Coulter Multisizer Accu Comp™ software versions issued later than version 1.10.

Aperture overlap in the analysis of coarse test dust normally occurs as follows:

Aperture size	**Approximate overlap**
µm	µm
between 400 and 200	20
between 200 and 100	10
between 100 and 30	3,5

Aperture overlap in the analysis of A3 medium and A2 fine test dusts normally occurs as follows:

Aperture size	**Approximate overlap**
µm	µm
between 280 and 100	12
between 100 and 30	3,5

The intention of listing sample handling and analysis procedures is to reduce variability in results which may occur due to procedure differences between laboratories.

Coulter Multisizer IIe™ operations and application manuals designate other principles of operation involving sample analysis which must be followed.

5.2 Instrument calibration

The instrument shall be calibrated using traceable latex spheres of certified diameters[2].

6 Chemical composition

6.1 Typical chemical content of ISO-specified Arizona test dusts

See table 4.

Table 4 — Chemical content

Chemical	Mass fraction %
SiO_2	68 to 76
Al_2O_3	10 to 15
Fe_2O_3	2 to 5
Na_2O	2 to 4
CaO	2 to 5
MgO	1 to 2
TiO_2	0,5 to 1
K_2O	2 to 5
Loss on ignition (1 050 °C): 2 % to 5 %.	

6.2 Chemical analysis methodology

Samples are to be dried in an oven at 105 °C. Aliquots — 1 g of the dried samples — are to be weighed into tared platinum crucibles and ashed at 1 050 °C. Loss on ignition is to be calculated from the weight loss. The ignited samples are then analysed for silica (SiO_2) by volatilization using hydrofluoric acid. The residues remaining after the silica analyses are fused with lithium metaborate ($LiBO_2$). The resulting melts are dissolved in dilute hydrochloric acid then transferred to volumetric flasks and diluted to volume using deionized water. These solutions are analysed for the remaining metals by inductively coupled plasma spectrometry (ICP). The ICP calibration standards contain matching acid and $LiBO_2$ concentrations. Metals content determined by ICP are to be calculated as the oxides reported in the table.

7 Handling and preparation

7.1 Preparation prior to use

Care should be taken in preparation of test dusts because stratification or agglomeration of particles may occur during shipping and handling. Stratification of particles involves formation of layers of particles of differing sizes within a vessel containing test dust. Stratification is caused by natural migration of particles propelled by vibration during handling or storage. Although no documented scientific studies have been conducted relative to stratification of Arizona test dust particles, it is possible that coarse particles migrate to the top and fine particles concentrate near the bottom of storage containers. Therefore, it is recommended test dusts be mixed or reblended immediately preceding use.

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2) Suitable latex spheres, for calibration purposes, are commercially available. Details can be obtained from the Secretariat of ISO/TC 22 or the ISO Central Secretariat.

Test dusts may retain or absorb moisture during the manufacturing process or in storage. Moisture content of test dusts is not considered significant relative to many types of testing involving these materials. However, sensitive or closely controlled test programs may require that test dust be dried in an oven prior to use.

7.2 Health hazards in handling test dusts

7.2.1 Physical and safety data

7.2.1.1	Melting point	1 723 °C
7.2.1.2	Density	2 650 kg/m^3
7.2.1.3	Bulk density	500 kg/m^3 to 1 200 kg/m^3, depending on grade.
7.2.1.4	Solubility in water	Insoluble.
7.2.1.5	Hazardous reactions	Test dust is stable.
7.2.1.6	Transport restrictions	Unrestricted.

7.2.2 Protective measures for storage and handling

7.2.2.1	Industrial hygiene	Hazardous condition occurs if respiratory system is exposed to airborne particulates. A respirator or dust mask that is approved by national regulations is recommended.
7.2.2.2	Fire and explosion	Not flammable.
7.2.2.3	Disposal	Dispose of in accordance with local regulations. Landfills may be acceptable for discarded material.

7.2.3 Measures in case of accident or fire

7.2.3.1	Spillage or leakage	Vacuum or gather spilled material in a manner which does not create airborne dust.
7.2.3.2	Fire	No reaction with water or foam fire fighting measures.

7.2.4 Health aspects

7.2.4.1	Toxicity	Moderately toxic as an acute irritating dust. The prolonged inhalation of dusts may result in the development of a disabling pulmonary fibrosis known as silicosis.
7.2.4.2	Abrasiveness	Protective goggles should be worn to protect eyes. If eye contact occurs, flush eyes with plenty of water.
7.2.4.3	First aid	Eyes: rinse immediately with water. If irritation occurs, seek medical advice. Skin: wash with soap and water. If irritation develops, seek medical advice.

Annex A
(informative)

History

During the 1930's, air intake cleaning devices became widely used by engine manufacturers for use in reducing particulates ingested by internal combustion engines. By 1940, the SAE Journal indicated a need for a standardized test contaminant to evaluate performance of air cleaner systems. The 1943 edition of the SAE Handbook recommends collecting test dust by placing canvas cloth on the ground behind tractors or implements in the Salt River Valley, Arizona, USA.

During following years, a more controlled method of producing large volumes of test dust was required. As a result, the AC Spark Plug company, division of General Motors corporation, devised a method of manufacturing test dust by ball milling Arizona desert sand. Two grades of test dust were produced to the particle size specification given in table A.1 and analysed for particle size using a Roller™ analyser.

Table A.1 — Particle size distribution

Size µm	Mass fraction, %	
	fine	coarse
0 to 5	39 ± 2	12 ± 2
5 to 10	18 ± 3	12 ± 3
10 to 20	16 ± 3	14 ± 3
20 to 40	18 ± 3	23 ± 3
40 to 80	9 ± 3	30 ± 3
80 to 200	—	9 ± 3

In 1979 AC Spark Plug began using a Leeds and Northrup Microtrac™ particle size analyser, in place of the Roller™ analyser, to determine particle size distributions of AC fine and coarse test dusts. AC indicated that considerable correlation testing between the L & N Microtrac™ and Roller™ analysers was performed and in 1982 requested that SAE subcommittees revise dust identification tables as follows:

> The particle size distribution by volume, as measured with an L & N Microtrac™ analyser, shall be in accordance with table A.2.

Table A.2 — Particle size distribution by volume

Size µm	Maximum volume fraction, %	
	fine	coarse
5,5	38 ± 3	13 ± 3
11	54 ± 3	24 ± 3
22	71 ± 3	37 ± 3
44	89 ± 3	56 ± 3
88	97 ± 3	84 ± 3
176	100	100