

		FEA
Aerosol gaskets - Test for material identification and selection		641 - E Page 1
Gleichlautende Standards/Corresponding Standards/Standards Correspondants		
FEA 641 F	Jointes aérosols – Tests pour l'identification et la sélection du matériau	
FEA 641 D	Aerosoldichtungen – Tests zur Material Definition	

Foreword and Scope

All successful aerosol product work requires a good knowledge of the gasket materials and their interaction with the complete aerosol product.

1. Gasket materials are generally classified according to the chemical structure of their main polymer component. Internationally accepted nomenclature is defined in standard ISO 1629 and this is summarised on the final page of this standard together with common names and chemical formulae.
2. Gasket materials are based on polymers to which are added fillers and plasticizers to modify the physical properties and a crosslinking, or curing system, to give resistance to permanent set and swelling effects of aerosol products.

When the valve gasket is in contact with the complete aerosol product, it can be changed irrevocably in size and physicochemical properties due to the swelling effect on the polymer and the eventual extraction of plasticizers.

General methods of determination of swelling are described in ISO 1817 – Vulcanized Rubber – Determination of the effect of liquids - § 8.

For this reason, it is imperative for the laboratory practitioner to check such product influence changes as accurately as possible. In this way a correct choice of gasket may be achieved to give good valve integrity.

3. The interaction between gasket and aerosol product can also produce detrimental changes in the product, including precipitation, perfume degradation, colour change, and these aspects need to be expressed in the overall choice of gasket material.
4. Resistance to swelling in pure solvents gives a good guide to the polymer group of the gasket material. A test for material identification is given in this standard.

This FEA standard deals only with dimensional aspects of gasket materials under the following headings:

- The swell test – procedure and evaluation
- Gasket material identification
- Material nomenclature

Aerosol gaskets - Test for material identification and selection	FEA 641 - E Page 2
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Method

1) The swell test: procedure and evaluation

Use a dial gauge with a constant spring load around 50 g. against a measuring anvil surface of approximatively 50 mm².

Measure first the original thickness (**D**₁).

- a) This measurement can be realized on 3 separate gaskets with a precision of ± 0.01 mm and sum up the 3 single values found.
- b) This measurement can be also realized directly on a stack of 3 gaskets with a precision of ± 0.01 mm.

Using a separate suitable container for each gasket material, place the 3 gaskets in the test solution. Close the container and pressurise with the appropriate propellant (thus giving the complete aerosol formulation).

Replicate test packs will be required for each time interval – recommended time interval are 3 days, 7 days, 2 weeks, 4 weeks and 8 weeks. The test packs should be stored at $21 \pm 1^\circ\text{C}$.

At the end of each time interval, open the container with care, observing the safety procedures for opening pressurised packages. Remove the gaskets from the test solution and within 5 seconds, measure the thickness (**D**₂):

- a) This measurement can be realised on 3 separate gaskets with a precision of ± 0.01 mm and sum up the 3 single values found.
- b) This measurement can be also realised directly on a stack of 3 gaskets with a precision of ± 0.01 mm.

Using either method, it is essential that the measurement be made within 5 seconds, and that the gaskets are measured without any attempt to dry them.

This method is particularly adopted on stem gaskets. If this method is applied to cup gaskets, for example gaskets of 20 mm \varnothing it is recommended to make an identification of the place where the measurement has been made.

Note:

Do not immerse the gaskets in liquids which contain undissolved solids, or solids which may precipitate onto the surface of the gasket during cooling or depressurising, thus affecting measurements.

Aerosol gaskets - Test for material identification and selection	FEA 641 - E Page 3
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Calculation of percentage swell:

$$\frac{(3D_2 - 3D_1) \times 100}{3D_1} = \% \text{ swell}$$

The selection of a suitable gasket material for a given aerosol formulation requires that the swell of the gasket reach equilibrium point. The time taken to achieve equilibrium will vary with the type of gasket material and the composition of the aerosol formulation.

Most suitable formulation/gasket combinations will reach equilibrium within the period 3 days to 2 weeks. However some formulation/gasket combinations will require a longer period to equilibrate. Others may show some swell followed by shrinkage or continue to swell until the molecular of the base polymer is broken down (see graph Y).

Using the test described above, the most appropriate method of formulation/gasket combination selection is:

- Check swelling after 3 days immersion and record data. Alternatively, it may be more convenient to record the first data after 7 days.
- Check swelling after a total of 2 weeks immersion.

If the data at this point are not significantly different from the first results, equilibrium is confirmed (see graph X).

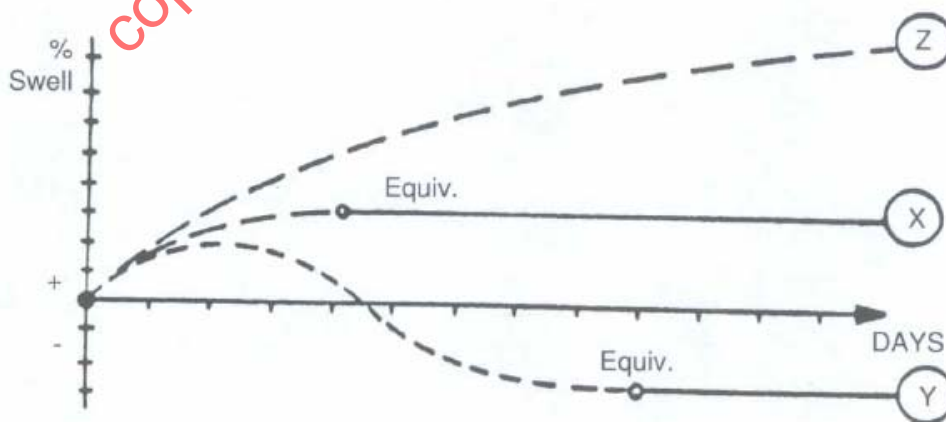
- If not, check swelling after a total of 4 weeks immersion.

If the data at this point are not significantly different from the 2 weeks data, equilibrium is confirmed (see graph X).

- If not, check swelling after a total of 8 weeks immersion.

If the data at this point are not significantly different from the 4 weeks data, equilibrium is confirmed (see graph X).

- If not it is unlikely that the formulation/gasket combination is satisfactory (see graph Z).



Aerosol gaskets - Test for material identification and selection	FEA 641 - E Page 4
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Note:

Valve designs from different manufacturers are designed to operate efficiently within carefully specified gasket swell ranges. The valve user should consult the manufacturer to obtain the recommended optimum swell characteristics for the selected valve.

2) Gasket material identification

The swell or shrink effects caused by the solvents used in aerosol formulations are partially related to the chemical structure of the gasket material. However, the additives present in the gasket material may cause variations in percentage swell even where a pure solvent is applied to gaskets with identical polymer bases.

The table below shows results obtained using all the available gasket material variations at the time of compilation.

The wide tolerances are a result of the additive variations used by the gasket manufacturers, but using a comparison of figures obtained after 3 days immersion in the pure solvents listed, it is possible to identify the material group of any aerosol gasket.

To perform the test for each gasket material, proceed as described in § 1, replacing the test solution and propellant by the 5 pure solvents mentioned hereunder.

In carrying out the test, note that the age of gasket and storage conditions may affect the result and should be kept constant if reliable comparisons are to be made.

Gasket material identification – Swell rate ranges for 3 days

Pure solvents	Nitrile	Chloroprene	Chlorobutyl	Fluorocarbon	EPDM	Polyuréthane
Hexane	1 ± 2	4 ± 2	40 ± 10	1 ± 2	30 ± 8	4 ± 2
Ethanol	1 ± 4	0 ± 2	1 ± 2	1 ± 2	2 ± 3	13 ± 8
Acetone	32 ± 14	7 ± 4	3 ± 3	56 ± 12	0 ± 3	35 ± 7
Methylenechloride	57 ± 22	35 ± 5	20 ± 5	5 ± 3	13 ± 5	50 ± 8
Perchloroethylene	9 ± 8	32 ± 4	45 ± 8	1 ± 2	37 ± 5	10 ± 4

This chart shows examples of swelling ratios. Obviously these ratios will have to be tightened for a more specified material.

Aerosol gaskets -
Test for material identification and selection

FEA

641 - E

Page 5

Classe Class	Denomination	Nom Commun Common Name	Formule Chimique Chemical Formula
BR	Butadiene rubber	Poly-butadiene	$(-CH_2-CH=CH-CH_2-)_n$
NBR	Nitrile-butadiene rubber	Nitrile	$[(CH_2-CH=CH-CH_2-)_m-CH_2-\underset{\text{CN}}{\text{CH}}]_n$
SBR	Styrene-butadiene rubber	Styrene-butadiene	$[(CH_2-CH=CH-CH_2-)_m-CH_2-\underset{\text{C}_6\text{H}_5}{\text{CH}}]_n$
EPDM	Ethylene-propylene with diene monomer	Ethylene-propylene-butadiene	$[CH_2-CH_2(CH_2-CH=CH-CH_2)_m-CH_2-\underset{\text{CH}_3}{\text{CH}}]_n$
IR	Isoprene rubber	Poly- isoprene	$(CH_2-\underset{\text{CH}_3}{\text{C}}=CH-CH_2)_n$
IIR	Isobutylene- isoprene rubber	Butyl	$[(CH_2-\underset{\text{CH}_3}{\text{C}}=CH-CH_2)_m-\underset{\text{CH}_3}{\overset{\text{CH}}{\text{C}}}-CH_3]_n$
CR	Chloroprene rubber	Polychloroprene	$(CH_2-\underset{\text{Cl}}{\text{C}}=CH-CH_2)_n$
CIIR	Chloro- isobutylene- isoprene	Chlorobutyl	$[(CH_2-\underset{\text{Cl}}{\text{C}}=CH-CH_2)_m-\underset{\text{CH}_3}{\overset{\text{CH}_3}{\text{C}}}-CH_3]_n$
FPM	Fluorocarbon rubber	Fluorocarbon	$[(CF_2-CH_2)_m-\underset{\text{CF}_3}{\text{CF}}-CF_2]_n$
PU	Polyester rubber	Polyurethane	$R \left[\begin{array}{c} \text{NH} - \text{C} - \text{O} \\ \\ \text{O} \end{array} \right] R'$

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