TN™FLEX : a new generation of fluorocarbon O-rings developed by COGEMA LOGISTICS with enhanced characteristics at low temperature (-40°C).

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ABSTRACT

Three main types of elastomers are used for the sealing of radioactive material transport casks with elastomeric gaskets : EPDM, fluorocarbons type Viton® (standard designation : FKM) and silicon rubbers. Each rubber has specific characteristics in terms of temperature range, permeability, coefficient of expansion… For the casks where high temperatures can be reached (200°C in continuous using), FKM gaskets are generally used. The problem is that this type of gasket does not guarantee the leaktightness at –40°C, which is a regulatory requirement. Two solutions are generally used : to specify a minimum heat load or a minimum ambient temperature. The direct consequence is that it is impossible to get B(U) approvals on the new concepts when FKM gaskets are used but only B(M) approvals, which generate significant additional justification costs (multiple submittals of Safety Analysis Reports, calculation of the minimum heat load or of the minimum ambient temperature…). Thus, it is important to develop gaskets with the same performance as FKM gaskets at high temperature but with enhanced performance at low temperature (and mainly, which guarantee the leaktightness at –40°C). COGEMA LOGISTICS has qualified a new generation of fluorocarbon O-rings (TN™FLEX gaskets) which can be used in continuous service on a –47°C/+200°C temperature range. TN™FLEX gaskets will be implemented on new casks designs.

1. PURPOSE

This paper presents a new type of fluorocarbon (FKM) gasket developed by COGEMA LOGISTICS with enhanced characteristics at low temperature (-40°C).

This report gives an overview of the characteristics of this new elastomer, called TN™FLEX, and the tests performed by COGEMA LOGISTICS for its qualification.

2. ISSUE

2.1. Types of elastomers currently used

Today, COGEMA LOGISTICS uses three types of elastomers for the manufacturing of the elastomeric gaskets fitted on its casks :

- fluorocarbon elastomers (FKM),
- EPDM elastomers,
- silicon elastomers (VMQ).

Each type of elastomer has specific characteristics :

	EPDM	FKM	Silicon
Maximum temperature in steady state	150°C ☆	200°C ☆☆☆	200°C ☆☆☆
Minimum temperature in steady state	-50°C ☆☆☆	-27°C ☆	-50°C ☆☆☆
Coefficient of volumetric expansion	515.10^{-6} K^{-1} ☆☆☆	650.10^{-6} K^{-1} ☆☆	830.10^{-6} K^{-1} ☆
Helium impermeability	☆☆☆	☆☆☆	☆
Air impermeability	☆☆	☆☆☆	☆
Compression set	☆☆	☆☆	☆
Gamma irradiation resistance	☆☆☆	☆☆	☆☆

☆ : medium / ☆☆ : good / ☆☆☆ : very good

2.2. Relevant properties

2.2.1. Temperature range

It is clear that the temperature range in continuous service (and also in accident conditions) for a gasket must be as large as possible.

A specific temperature must be noted for the applications in the field of the transportation of radioactive material : -40°C. Indeed, the 1996 edition of the IAEA “Regulations for the Safe Transportation of Radioactive Material” (paragraph 664 for type B(U) packages) specify that the leaktightness of a cask must be guaranteed even when the ambient temperature is equal to –40°C.

The problem is that the FKM gaskets can guarantee the leaktightness only for temperatures above (-20°C/-30°C). COGEMA LOGISTICS has demonstrated that the grades of FKM gaskets used on its casks can guarantee the leaktightness to –27°C. Consequently, to guarantee that the FKM gaskets temperature will never be below –27°C, two solutions are generally used : to specify a minimum heat load or to specify a minimum ambient temperature for the transport.

The direct consequence is that it is impossible to get type B(U) approvals on the designs when FKM gaskets are used but only type B(M) approvals, which generate significant additional justification costs (multiple applications, calculation of the minimum heat load or of the minimum ambient temperature…).

Thus, COGEMA LOGISTICS looks for gaskets which :

- perform as well as FKM gaskets at high temperature,
- guarantee the leaktightness at –40°C.

Such gaskets interest COGEMA LOGISTICS in the following cases :

- cases where the maximum temperature (in normal or accident conditions) is too high to use EPDM gaskets and requires FKM gaskets (which do induce specific requirements at low temperature (-40°C) : minimum heat load,…),
- cases where EPDM gaskets are used but where the maximum temperatures in normal and/or accident conditions are close to the maximum temperature at which this type of gaskets can be used and where it is useful to anticipate an increase of the heat load (using FKM gaskets being not the ideal solution, as explained previously).

2.2.2. Coefficient of volumetric expansion

If the coefficient of volumetric expansion of an elastomeric gasket is too high, it can be difficult to find dimensions for the gasket dove-tail groove which guarantee to maintain the gasket in place, to compress it enough and to limit the groove filling coefficient to a value below 100%.

A gasket with too high a coefficient of volumetric expansion can induce extrusion problems (if the groove filling coefficient is too high). Therefore, it is important to use gaskets with coefficients of volumetric expansion as low as possible to limit the groove filling coefficients.

2.2.3. Permeability

The permeability of an elastomer is an important characteristic. Indeed, a gasket with a high permeability can complicate a helium leak test because the test gas diffuses too quickly through the gasket. In this case, it can be difficult to know if the leak measured is a «true » leak (bypass leakage through the seal/fixture interface) or a high permeation leak (« leak » by permeation through the gasket). A high permeability can also be an issue when gaseous elements must be contained.

For example, in spite of a wide temperature range (-50°C to 200°C in continuous service), silicon gaskets are not used on a lot of COGEMA LOGISTICS casks because of too high a permeability (which upsets the leak test if the leak criteria is very low).

2.2.4. Compression set

The compression set characterises the elastic recovery of an elastomeric gasket when it has been compressed and heated. Thus, the compression set expresses the capacity of a gasket to be used at high temperature. It must be as low as possible.

2.3. Characteristics expected for COGEMA LOGISTICS gaskets

For its applications, COGEMA LOGISTICS looks for elastomeric gaskets which have the following characteristics :

- a wide temperature range,
- a low coefficient of volumetric expansion,
- a high impermeability,
- a low compression set,
- a good mechanical resistance (tear…),
- an easy manufacturing (casting,…),
- a low price.

Each type of gasket (EPDM, standard FKM, silicon) presents its own pros and cons :

- the EPDM gaskets have a low coefficient of volumetric expansion and can be used at very low temperatures (below –40°C) but have a limited capacity for the applications at high temperature,
- the standard FKM gaskets have very good characteristics at high temperature but they cannot be used at –40°C and have a high coefficient of volumetric expansion,
- the silicon gaskets temperature range covers the temperature range of the EPDM and FKM gaskets but their permeability and their coefficient of volumetric expansion are high and they have poor mechanical characteristics (compression set…).

The TN^{TM}FLEX is a new answer which fully complies with the above mentioned specification.

3. TN^{TM}FLEX CHARACTERISTICS AT LOW TEMPERATURE

The characteristics at low temperature of the FKM elastomer TN^{TM}FLEX are significantly improved as regards standard FKM elastomers, as shown in the table below :

	Standard FKM	TN^{TM}FLEX
Glass transition temperature (Tg)	-15 to –25°C	-40°C
TR10 [1]	-15 to –25°C	-40°C
Brittle point [2]	-20 to – 30°C	-60°C

These different characteristics indicate below which temperature an elastomer starts to loose its elasticity. Consequently, they also give an idea of the low temperature where an elastomeric gasket looses its leaktightness. Indeed, an elastomeric gasket can not be leaktight when its elasticity is too weak.

The leakage appears in general at a temperature between the glass transition temperature and the Brittle Point. Thus, we can expect that a leak will appear on the gaskets manufactured with the elastomer TN^{TM}FLEX between –40°C and –60°C.

4. LEAK TESTS AT LOW TEMPERATURE

COGEMA LOGISTICS has done some tests on TN^{TM}FLEX gaskets at low temperature to determine their failure temperature. These tests are detailed below.

4.1. Test assembly

The test assembly consists of (see [3]) :

- two identical test rigs in which the gaskets to be tested are fitted,
- a set of electrovalves controlled by a computer to perform the leak tests.

The two test rigs are made up of two flat flanges (no grooves) separated by a spacer to adjust the compression rate (for these tests, the gasket nominal compression rates were 30%). The gaskets tested had the following nominal dimensions : 190 x 10 mm.

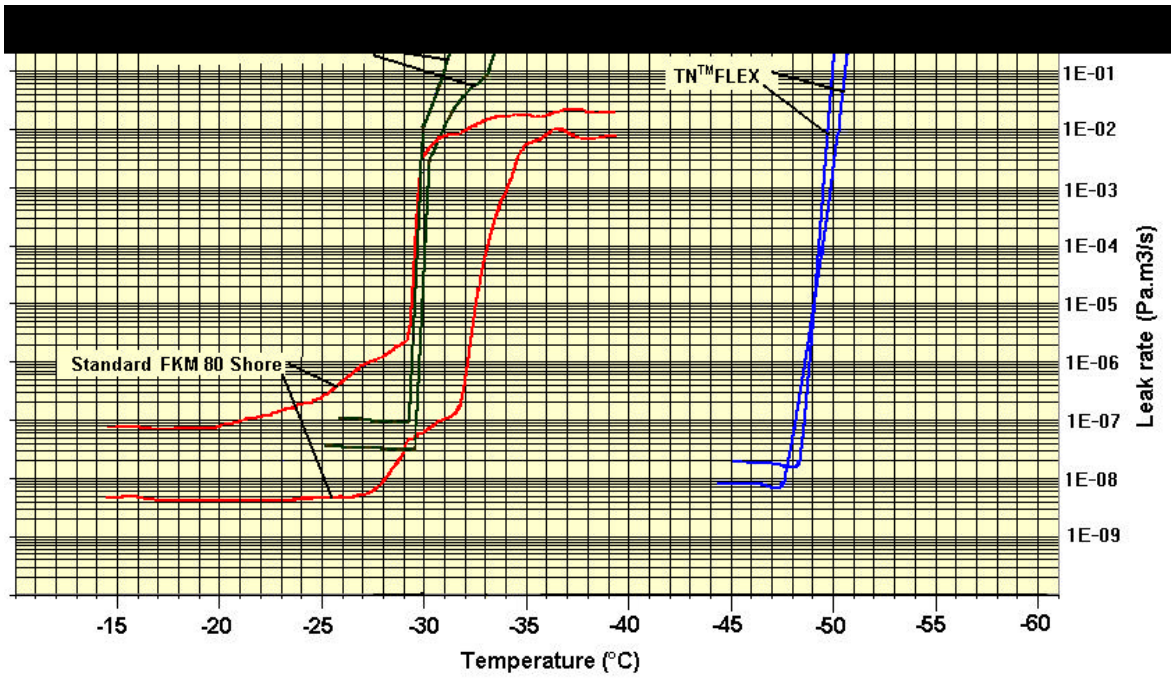
The electrovalves allow connecting the interspace or the center of one of the two test rigs to the following devices : a spectrometer, a vaccum pump, a helium bottle, a compressed air bottle.

4.2. Temperature profile

To determine the low failure temperature of the TN^{TM}FLEX gaskets tested, both test rigs are set in the same oven and submitted to a progressive decreasing temperature by two hour stages spread out every 1°C. A leak test is performed at the end of each stage of two hours on one rig and then on the other one.

4.3. Test results

These tests have shown that, as with standard FKM gaskets, the leakage at low temperature appears suddenly. The figure below shows the leak rate versus the temperature for the TN^{TM}FLEX gaskets (and a comparison with standard FKM gaskets) :



The replication for the two TN^{TM}FLEX gaskets tests is very good, **<u>the leakage occurs at a temperature below –47°C,</u>** which is a significant improvement in comparison with standard FKM grades used by COGEMA LOGISTICS which can guarantee the leaktightness only to –27°C.

5. COEFFICIENT OF VOLUMETRIC EXPANSION

The coefficient of volumetric expansion of several TN™FLEX samples has been measured.

These tests have shown that **the TN™FLEX coefficient of volumetric expansion is comparable with that of EPDM elastomers.**

This coefficient of volumetric expansion is low for a fluorocarbon elastomer. It is due to the compounding of the TN™FLEX elastomer grade.

This low coefficient of volumetric expansion is useful for limiting the groove filling coefficients.

6. GAMMA IRRADIATION RESISTANCE

6.1. Tests description

Some tests have been performed under gamma irradiation on the TN™FLEX elastomer and, for comparison, on EPDM (80 Shore) and standard FKM (70 Shore) elastomers.

For each elastomer grade, mechanical tests are done on elastomer sheets before and after irradiation to study its effect on the material properties. The influence of the total dose is studied, three doses are studied : 0 kGy (no irradiation), 1 kGy et 10 kGy. These last two doses are much higher than the doses received by the gaskets fitted on a cask.

6.2. Tests results

For every dose studied (0, 1 et 10 kGy), several mechanical characteristics have been studied : hardness, modulus at 50/100/150/200% of strain, ultimate tensile strength, strain at break.

These tests have shown that the behaviour under gamma irradiation of the TN™FLEX elastomer is very similar to the behaviour of the other elastomers commonly used by COGEMA LOGISTICS (EPDM, standard FKM and silicon elastomers) : almost no evolution of the mechanical characteristics.

The gamma irradiation, up to doses of 10 kGy (10^6 rad), does not affect the properties of the TN™FLEX elastomer.

7. AIR AND HELIUM PERMEABILITY

7.1. Principle of the permeability measurements

The permeability measures have been performed in accordance with standard [4]. These measures involve measuring a gas flow through a diaphragm with a 0,7 mm thickness and a 65 mm diameter. This diaphragm leans on a porous rigid surface. A one bar pressure drop is applied between the two sides of the diaphragm.

7.2. Tests description

The permeability of the TN™FLEX elastomer has been determined for two gases :

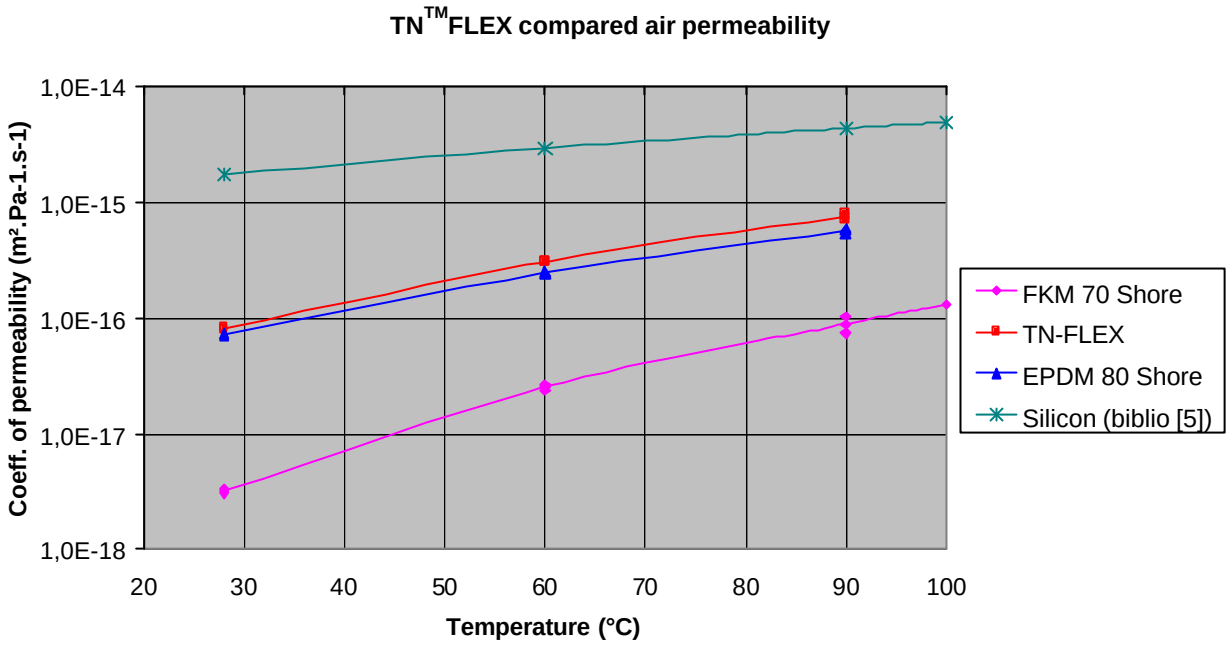
- helium : to know if helium leak tests on TN™FLEX gaskets are possible (see paragraph 2.2.3),
- air : to evaluate the capacity of TN™FLEX gaskets to limit the permeation of radioactive gases through their matrix.

As the permeability increases with the temperature, the permeability has been measured at three temperatures : +28°C, +50°C, +100°C.

For comparison, the permeability has also been measured on two other elastomer grades commonly used by COGEMA LOGISTICS (EPDM 80 Shore and FKM 70 Shore).

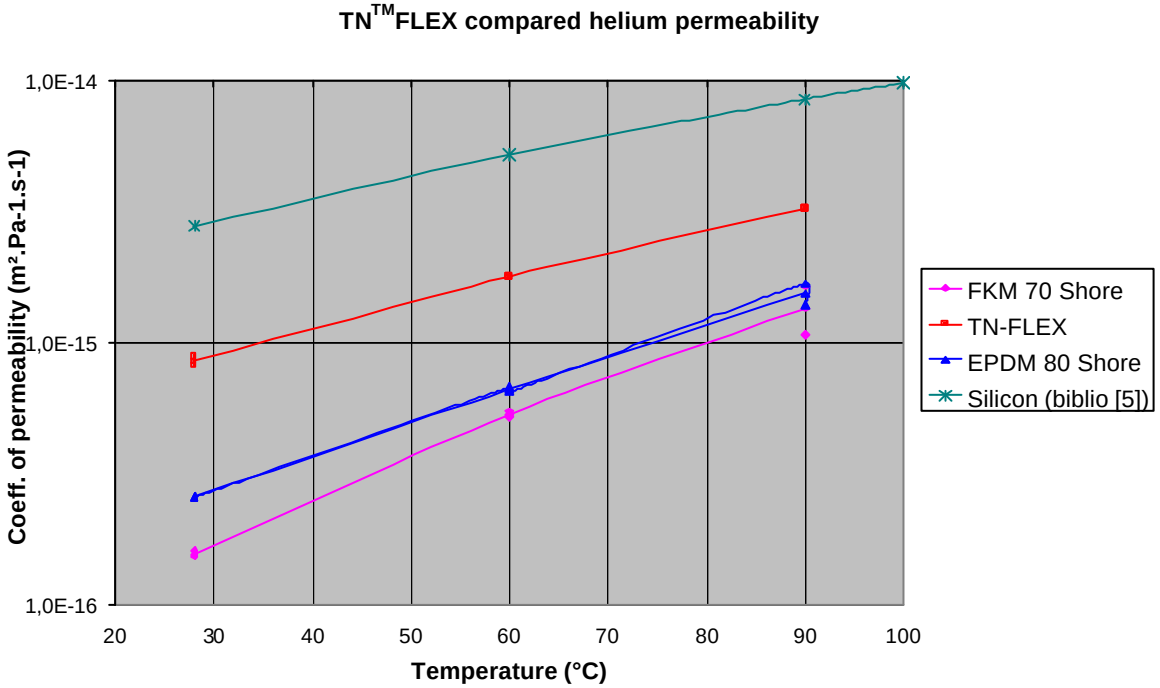
7.3. Test results

- **Air permeability test results :**



This figure shows that the TN ™FLEX elastomer has the same air permeability as EPDM.

- **Helium permeability test results :**



This figure shows that the TN™FLEX helium permeability is three times higher than that of EPDM and three times lower than for silicon. The different leak tests performed by COGEMA LOGISTICS have shown that this permeability is low enough to perform helium leak tests without being upset by the permeation.

7.4. Conclusion of the permeability tests

These tests have shown that :

- the TN™FLEX air permeability is the same as EPDM elastomers,
- the TN™FLEX helium permeability is low enough to perform helium leak tests.

8. COMPRESSION SET

It is interesting to compare the TN™FLEX compression set with the compression set of the elastomers commonly used by COGEMA LOGISTICS :

	Conditions	Compression set
EPDM	70 hours at 150°C	20% max
FKM	70 hours at 200°C	25% max
Silicon	70 hours at 150°C	40% max
TN™FLEX	70 hours at 200°C	25% max

The TN™FLEX elastomer compression set is very similar to those of EPDM and standard FKM elastomers. Thus, we can say that TN™FLEX gaskets do have the same elastic recovery as EPDM and standard FKM gaskets.

The fact that the TN™FLEX compression set after 70 hours at 200°C is similar to the compression set of standard FKM elastomers shows that TN™FLEX heat resistance (which is also a FKM) is comparable with standard FKM (200°C in steady state).

9. HEAT-AGEING TESTS

Heat ageing tests are currently performed by COGEMA LOGISTICS on the TN™FLEX elastomer in comparison to a standard FKM elastomer. These tests involve doing heat-ageing tests at the following temperatures : 200°C, 230°C, 250°C.

At the date of writing this paper, these tests are under way. COGEMA LOGISTICS expects that the results obtained for the TN™FLEX elastomer and the standard FKM elastomer will be close.

10. CONCLUSION

The TN™FLEX gaskets, a new type of fluorocarbon gaskets developed and qualified by COGEMA LOGISTICS, present very interesting characteristics for COGEMA LOGISTICS packages :

- a –47°C/200°C temperature range in steady state,
- a low coefficient of volumetric expansion,
- a good impermeability,
- a low compression set,
- a good gamma irradiation resistance.

	EPDM	**FKM**	**Silicon**	**TN™FLEX**
Maximum temperature in steady state	150°C ☆	200°C ☆☆☆	200°C ☆☆☆	200°C ☆☆☆
Minimum temperature in steady state	-50°C ☆☆☆	-27°C ☆	-50°C ☆☆☆	-47°C ☆☆☆
Coefficient of volumetric expansion	☆☆☆	☆☆	☆	☆☆☆
Helium impermeability	☆☆☆	☆☆☆	☆	☆☆
Air impermeability	☆☆	☆☆☆	☆	☆☆
Compression set	☆☆	☆☆	☆	☆☆
Gamma irradiation resistance	☆☆☆	☆☆	☆☆	☆☆

The TN™FLEX gaskets will allow to improve the performance of COGEMA LOGISTICS casks and to simplify the thermal justifications in the Safety Analysis Reports. Indeed, it will be no more necessary to specify a minimum heat load or a minimum ambient temperature as it is the case when standard FKM gaskets are used and if a a type B(U) approval is needed. Consequently, they will constitute a significant advantage for a wide range of applications.

11. REFERENCES

[1] Standard ISO 2921:1997 : “Rubber, vulcanized – Determination of low-temperature characteristics – Temperature-retraction procedure (TR test)”.

[2] Standard ISO 812:1991 : “Rubber, vulcanized – Determination of low-temperature of brittleness”.

[3] PATRAM 2004 (Berlin) : “A general approach for quantifying the heat-ageing of seals” (Régis ANDRE, Pierre MALESYS (COGEMA LOGISTICS)).

[4] ISO 2782 standard (May 15th, 1995) : ”Rubber, vulcanized or thermoplasic – Determination of permeability to gases”.

[5] Publication VUOTO, vol. XX n°2 (April 1990) : « Gas permeation through common elastomer sealing materials » (BAM : H.P. WEISE, K.H. ECKER, Th. WOLK, H. KOWALESKY).