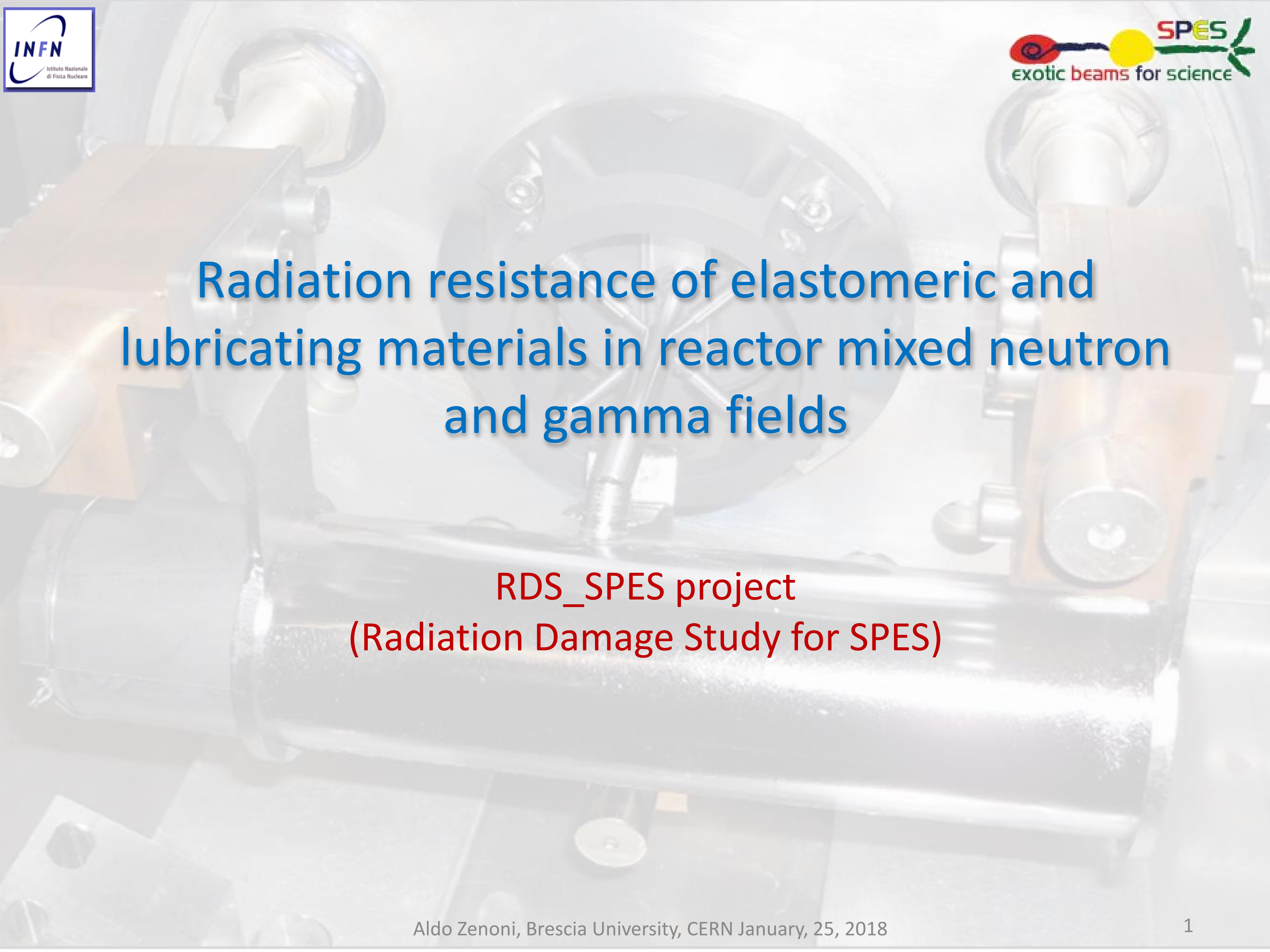
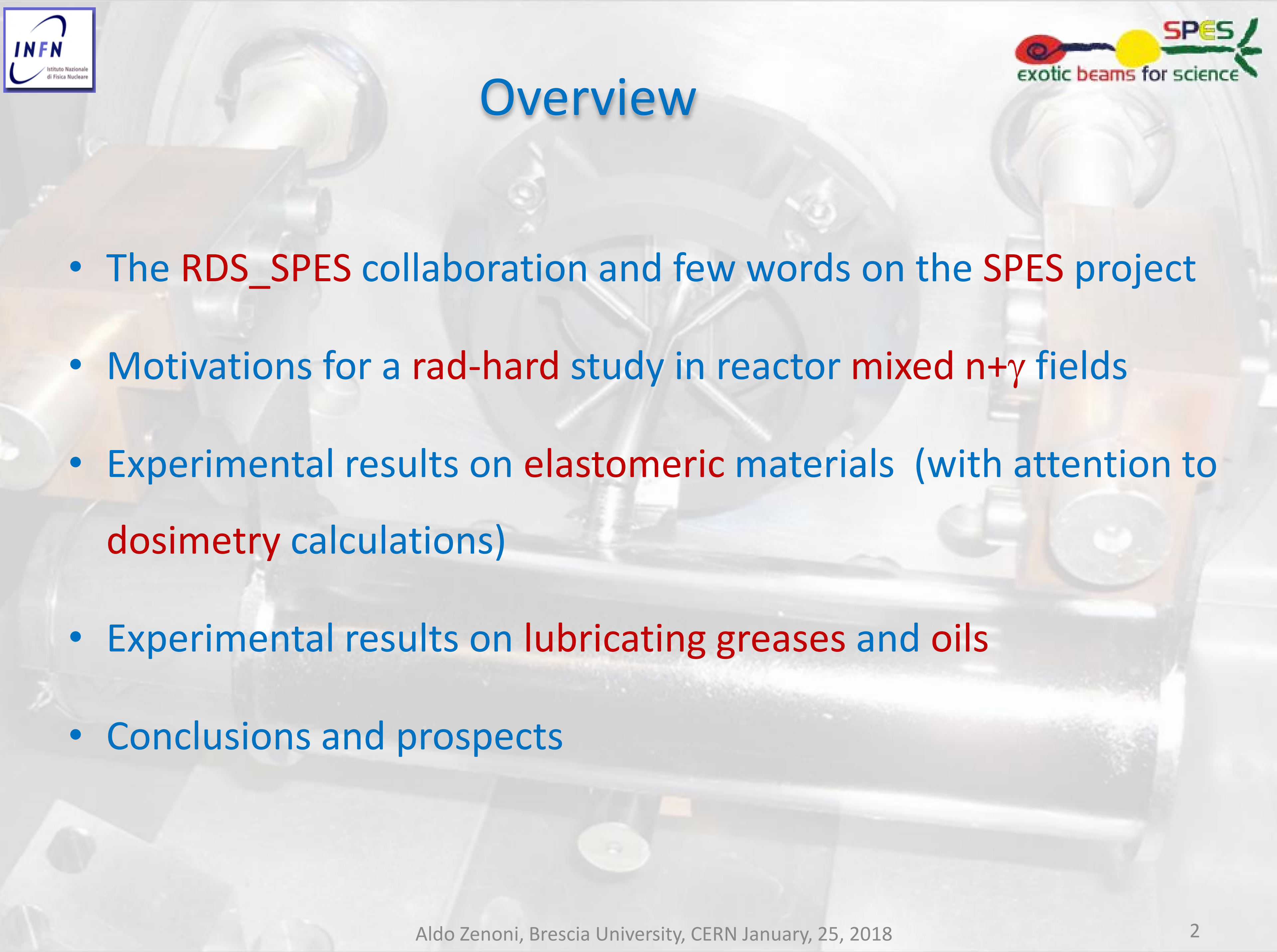


Radiation resistance of elastomeric and lubricating materials in reactor mixed neutron and gamma fields

RDS_SPES project
(Radiation Damage Study for SPES)



Overview

- The RDS_SPES collaboration and few words on the SPES project
- Motivations for a rad-hard study in reactor mixed n+γ fields
- Experimental results on elastomeric materials (with attention to dosimetry calculations)
- Experimental results on lubricating greases and oils
- Conclusions and prospects

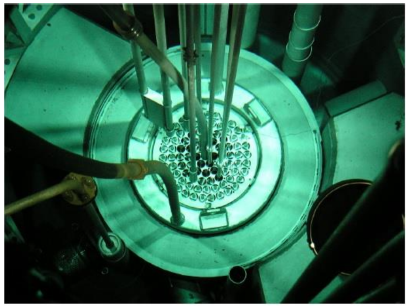
The partners of the RDS_SPES Project

A. The SPES Exotic Beam Production Group at LNL-INFN at Legnaro, Italy.

B. The L.E.N.A. Laboratory at Pavia University, Pavia, Italy. (Irradiations with n+γ mixed fields at TRIGA Mark II research reactor)

C. Physics and Materials Science Groups at Brescia University, Brescia, Italy. (Dosimetry calculations, experimental protocols, test of materials properties)

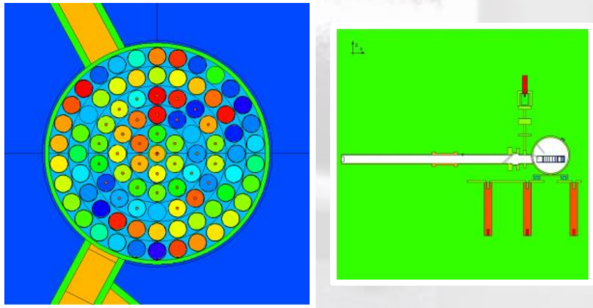
D. The ESS (European Spallation Source) ERIC project at Lund, Sweden.



Triga Mark II core at LENA

Materials scientists MaST Lab

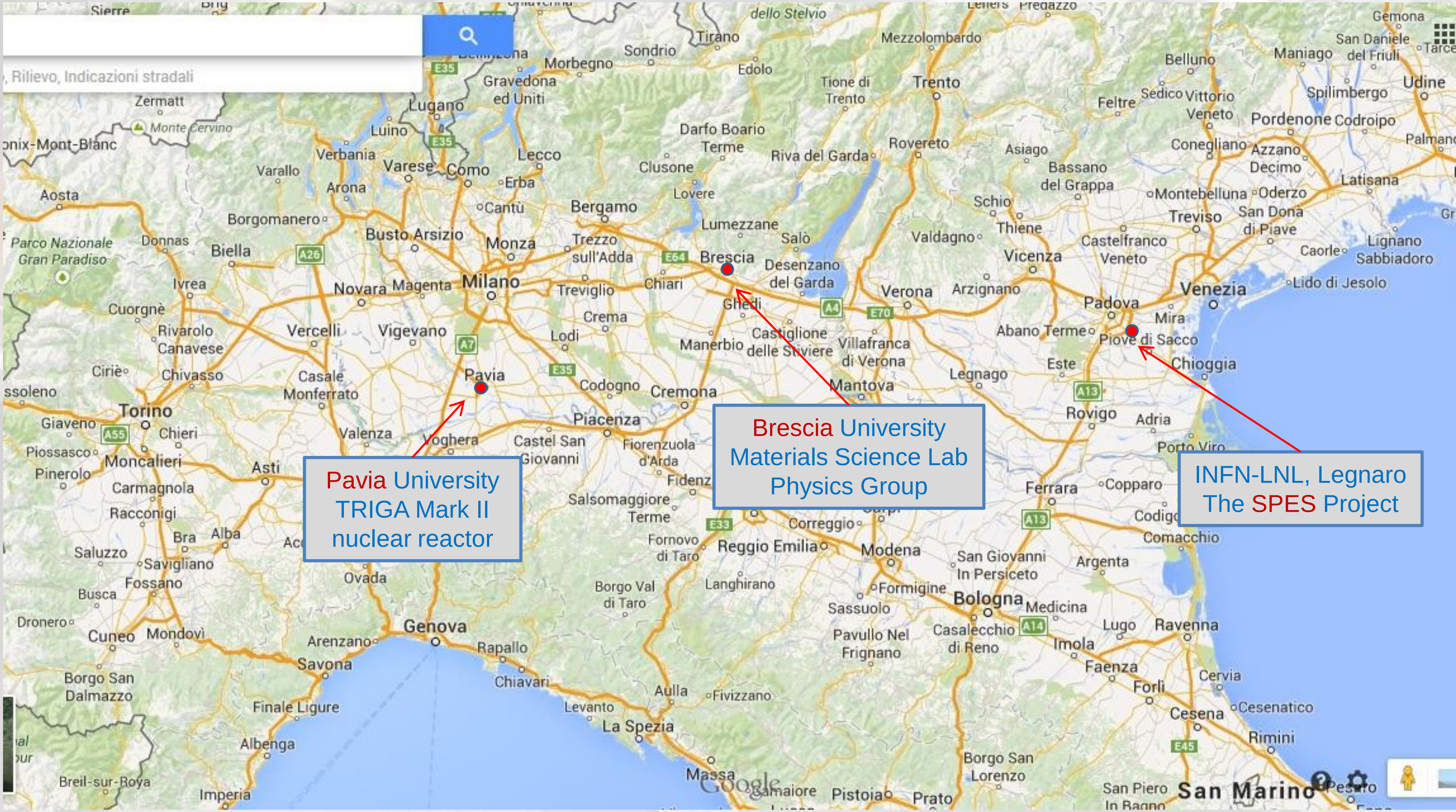
The Project is mainly supported by LNL-INFN, Italy; Brescia and Pavia Universities, Italy; ESS, Lund, Sweden.



MCNPX calculations

Partner locations (in Northern Italy)

exotic beams for science



Pavia University
TRIGA Mark II
nuclear reactor

Brescia University
Materials Science Lab
Physics Group

INFN-LNL, Legnaro
The **SPES** Project

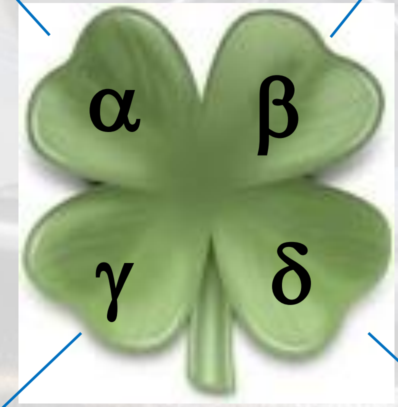
SPES: The future of LNL

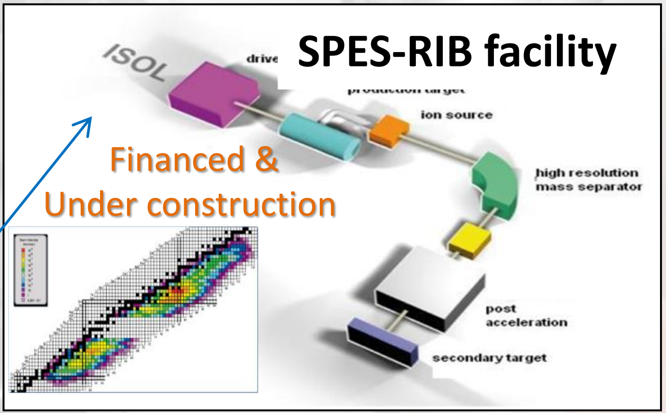
SPES is: 1) A second generation ISOL facility (for neutron-rich ion beams)
2) An interdisciplinary research center (for p,n applications)

SPES-DRIVER

Commissioned

Cyclotron installation & commissioning:
- E=70 MeV proton beam, I= 750 μA



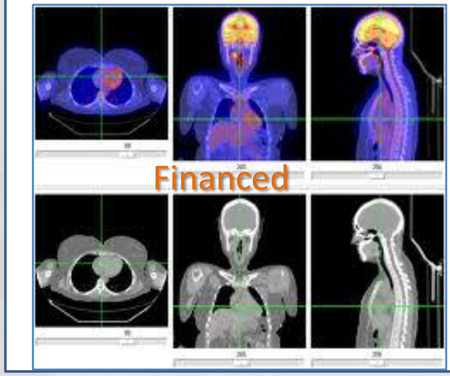


SPES-RIB facility

Financed & Under construction

Production & re-acceleration of **exotic beams,** from **p-induced Fission** on **UCx**

SPES for medicine



Financed

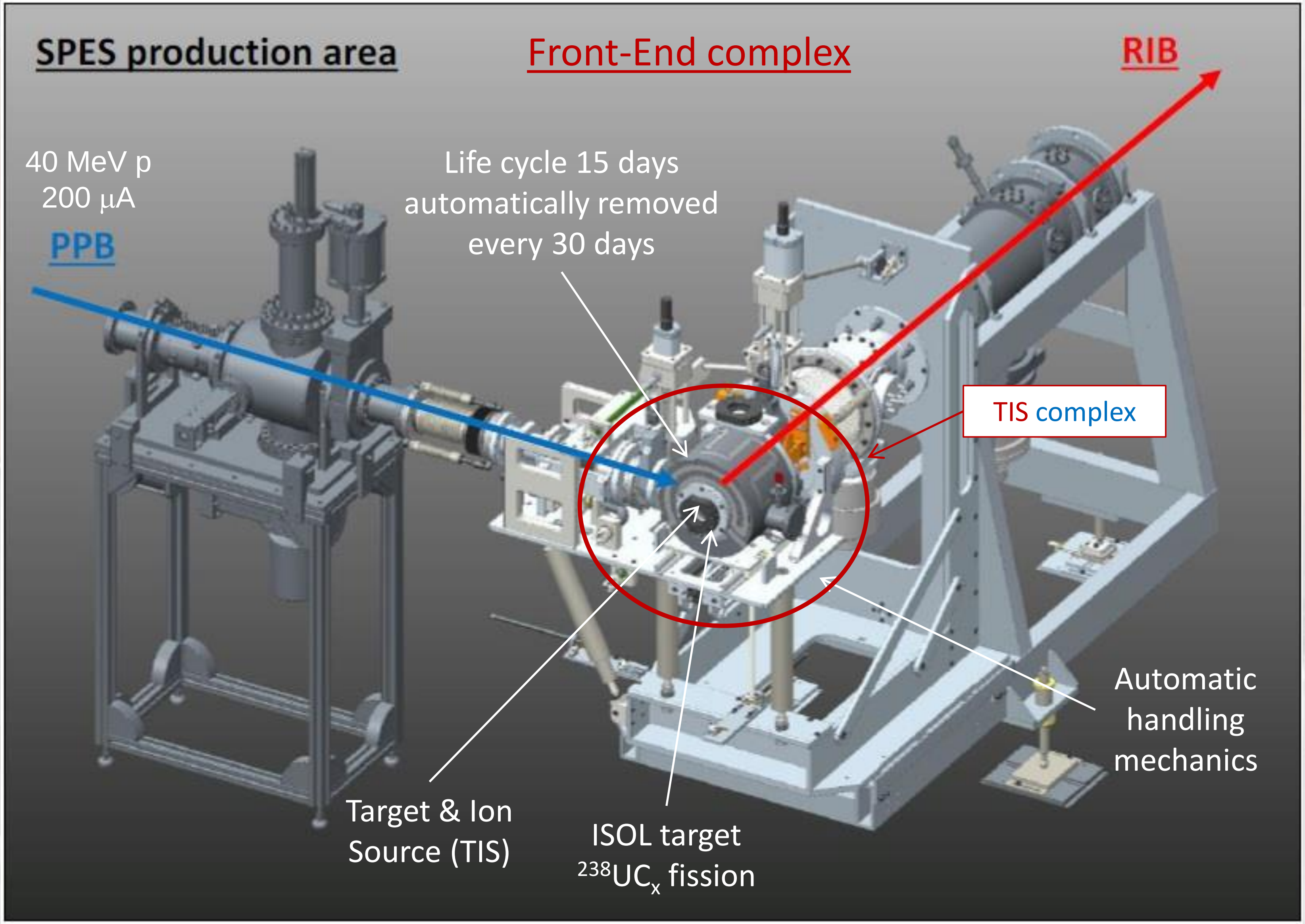
LARAMED & ISOLPHARM projects
Radisotopes for medical applications

SPES Neutron facility

Proposal

Accelerator based neutron source
(**Proton and Neutron Facility for Applied Physics**)

The RIB production area



SPES production area

Front-End complex

RIB

40 MeV p
200 μA

PPB

Life cycle 15 days
automatically removed
every 30 days

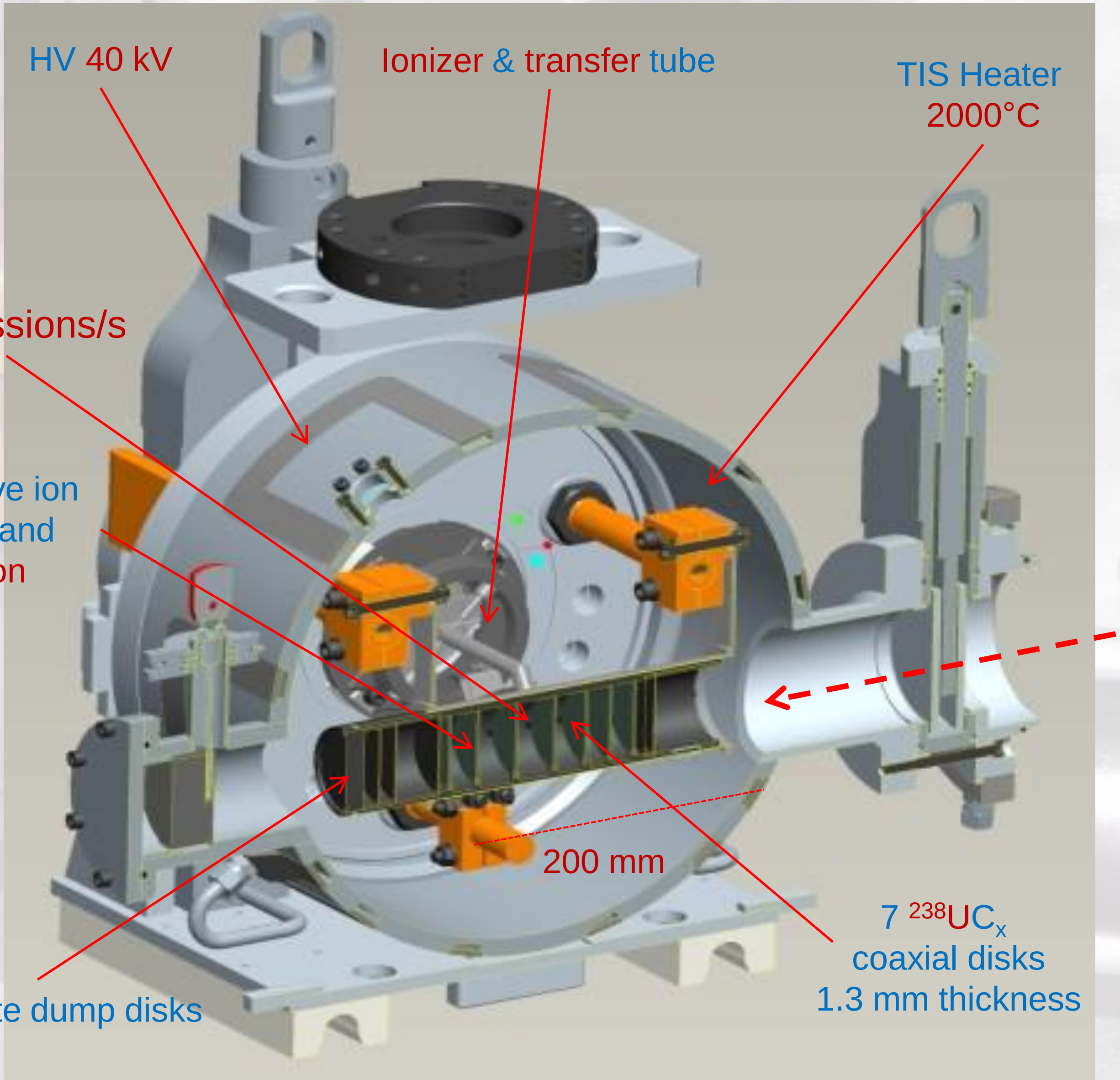
TIS complex

Automatic
handling
mechanics

Target & Ion
Source (TIS)

ISOL target
$^{238}UC_x$ fission

Target & Ion Source complex (TIS)



HV 40 kV

Ionizer & transfer tube

TIS Heater
2000°C

10^{13} fissions/s

Radioactive ion
effusion and
diffusion

200 mm

7 $^{238}UC_x$
coaxial disks
1.3 mm thickness

3 graphite dump disks

High thermo
mechanical stresses

15 day life cycle
5 year cooling

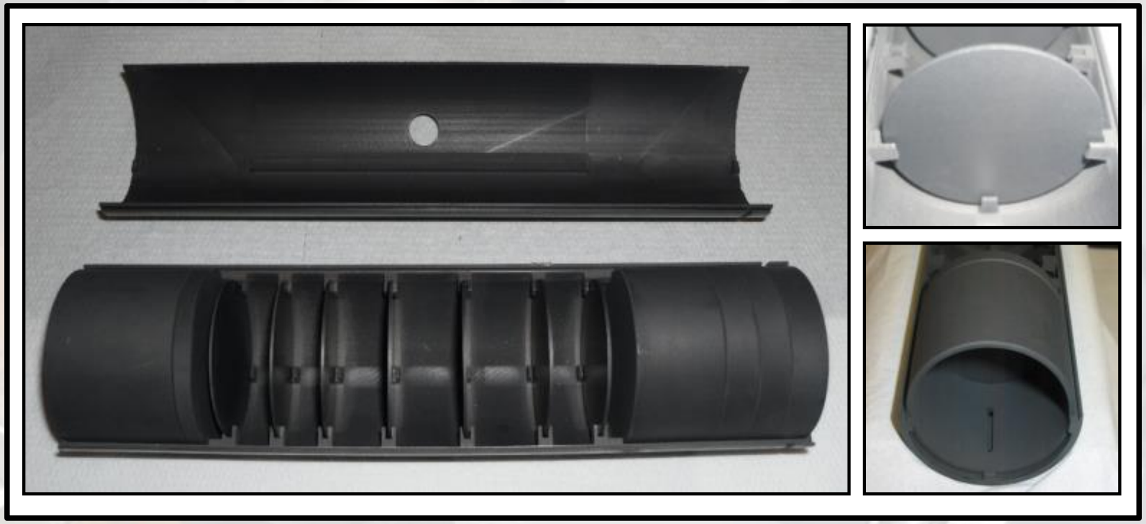
Highly radioactive
environment

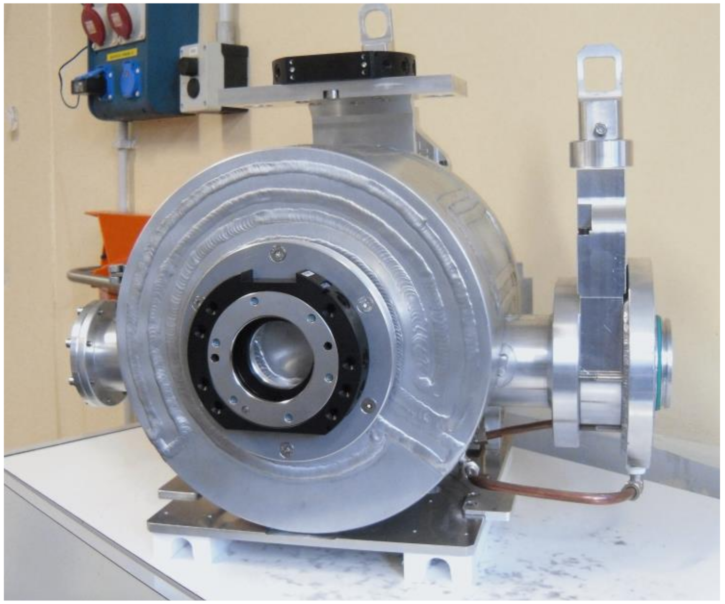
40 MeV protons
200 μA, 8 kW

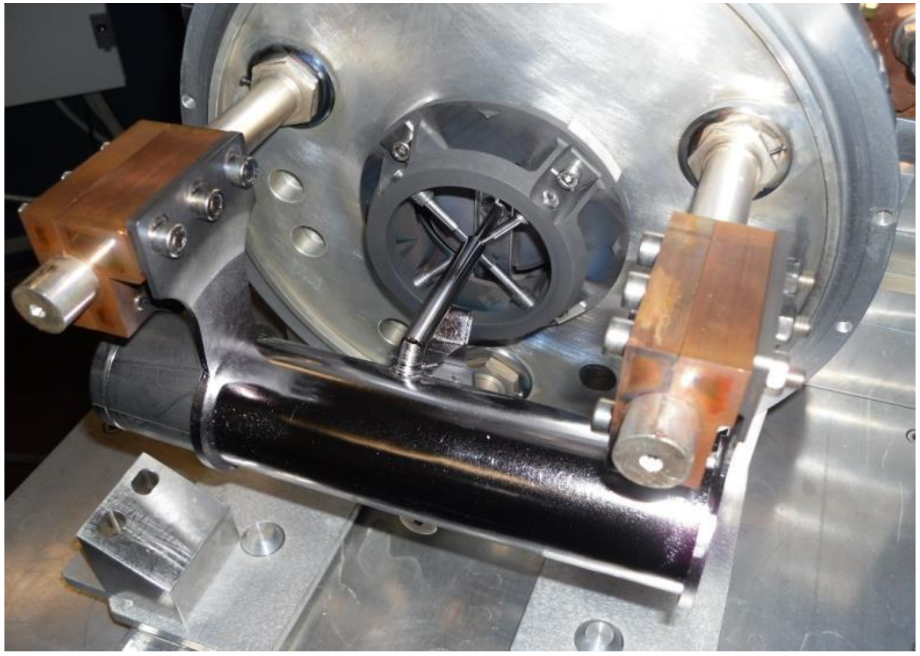
Intense fission neutron
and gamma fields
(10^{10} particle/cm^2/s)

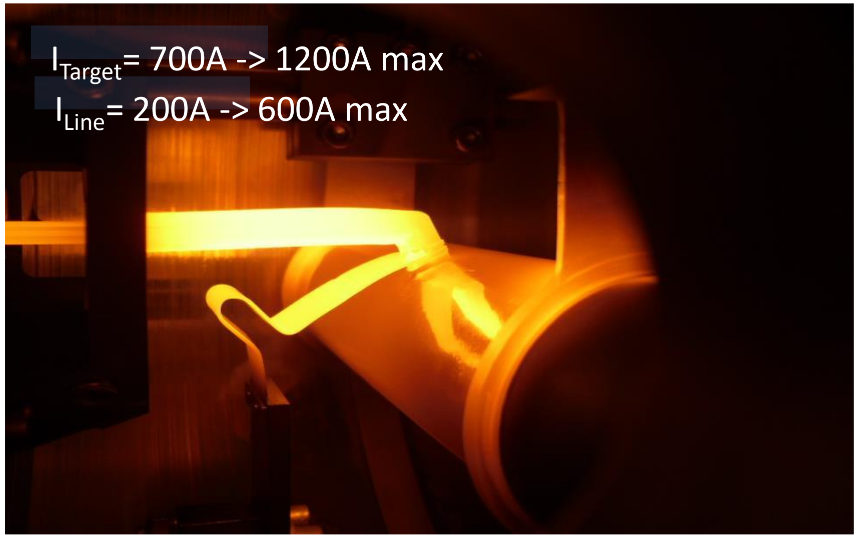
Only automatic
handling is possible

Target & Ion Source complex (TIS)









I_{Target}= 700A -> 1200A max

I_{Line}= 200A -> 600A max

Modification of material properties by radiation effects (radiation damage)

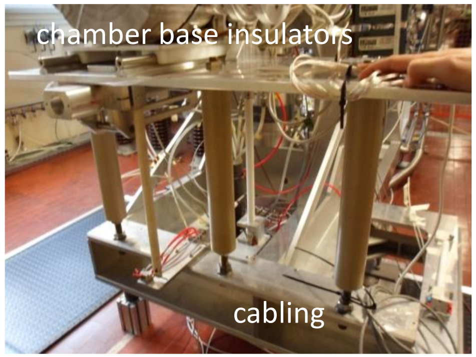
Some critical Front-End components (mainly polymeric materials)

1) Elastomeric joints (O-rings)
2) Lubricating oils and greases
3) Signal cable insulators
4) AC power wire jackets
5) Optical fiber core/cladding
6) Beam line insulators
7) Chamber base insulators
8) Chamber handling guides
9) Pneumatic motors
10) Electronic components

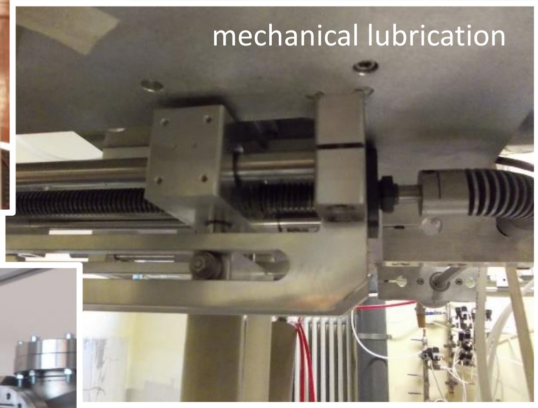
Component failure must be prevented

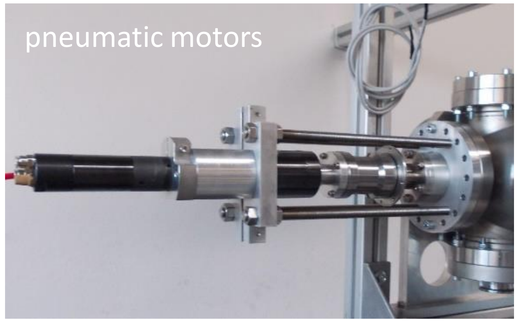

VAT valves O-rings


vacuum O-rings


chamber base insulators

cabling


mechanical lubrication

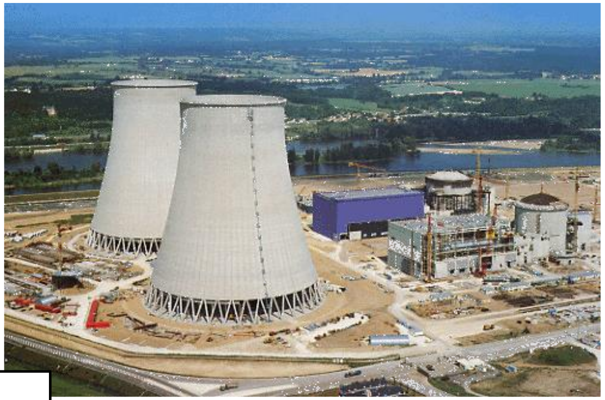

pneumatic motors

Operator interventions very problematic

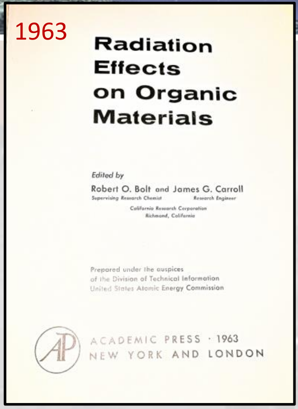
Damage of materials by radiation is a long-known technological problem

Space science

Nuclear reactor technology



Accelerator technology



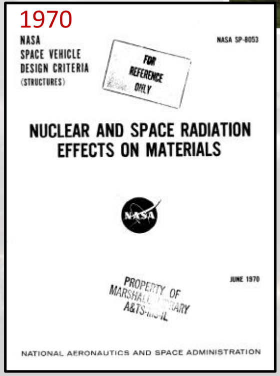
1963

Radiation
Effects
on Organic
Materials

Edited by
Robert O. Bolt and James G. Carroll
Supervising Research Chemist Research Engineer
California Research Corporation
Richmond, California

Prepared under the auspices
of the Division of Technical Information
United States Atomic Energy Commission

ACADEMIC PRESS · 1963
NEW YORK AND LONDON



1970

NASA
SPACE VEHICLE
DESIGN CRITERIA
(STRUCTURES)

NASA SP-8053

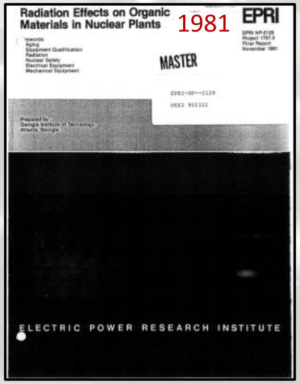
FOR REFERENCE ONLY

NUCLEAR AND SPACE RADIATION
EFFECTS ON MATERIALS

JUNE 1970

PROPERTY OF
MARSHALL LIBRARY
A&TS-MS-IL

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION



Radiation Effects on Organic
Materials in Nuclear Plants

1981

EPRI

EPRI NP-2129
Project 1707-3
Final Report
November 1981

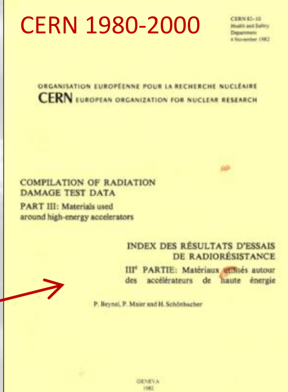
ywords:
Aging
Equipment Qualification
Radiation
Nuclear Safety
Electrical Equipment
Mechanical Equipment

MASTER

EPRI-NP--2129
DE82 901321

Prepared by
Georgia Institute of Technology
Atlanta, Georgia

ELECTRIC POWER RESEARCH INSTITUTE



CERN 1980-2000

CERN 82-10
Health and Safety
Department
4 November 1982

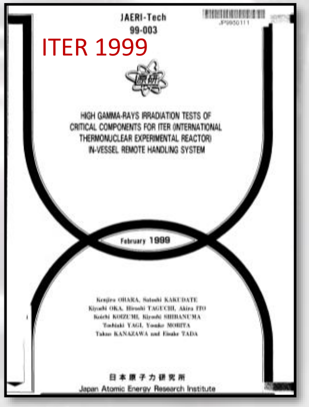
ORGANISATION EUROPÉENNE POUR LA RECHERCHE NUCLÉAIRE
CERN EUROPEAN ORGANIZATION FOR NUCLEAR RESEARCH

COMPILATION OF RADIATION
DAMAGE TEST DATA
PART III: Materials used
around high-energy accelerators

INDEX DES RÉSULTATS D'ESSAIS
DE RADIORÉSISTANCE
IIIe PARTIE: Matériaux utilisés autour
des accélérateurs de haute énergie

P. Beynel, P. Maier and H. Schönbacher

GENEVA
1982



JAERI-Tech
99-003

ITER 1999

HIGH GAMMA-RAYS IRRADIATION TESTS OF
CRITICAL COMPONENTS FOR ITER (INTERNATIONAL
THERMONUCLEAR EXPERIMENTAL REACTOR)
IN-VESSEL REMOTE HANDLING SYSTEM

February 1999

Kenjiro OBARA, Satoshi KAKUDATE
Kiyoshi OKA, Hiroshi TAGUCHI, Akira ITO
Koichi KOIZUMI, Kiyoshi SHIBANUMA
Toshiaki YAGI, Yosuke MORITA
Takao KANAZAWA and Eisuke TADA

日本原子力研究所
Japan Atomic Energy Research Institute

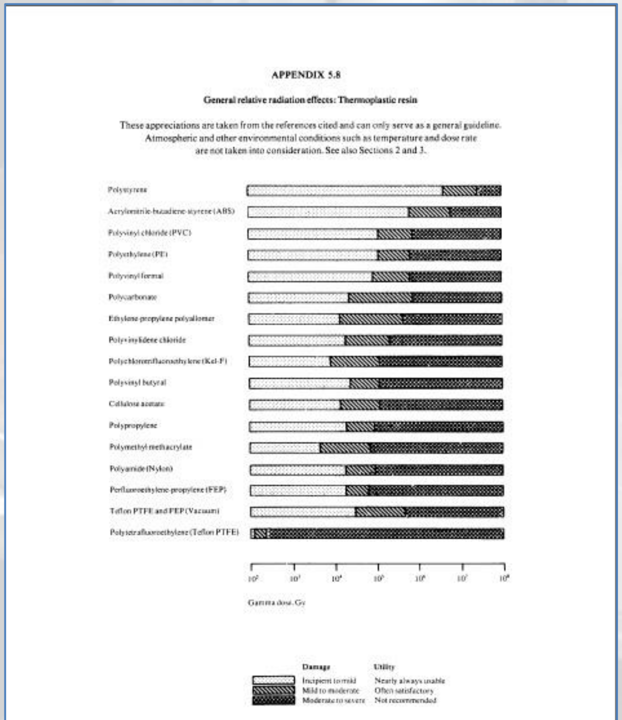
In accelerator technology and nuclear physics research, CERN Yellow Reports are the MAIN reference

+

information by producers, when available

Typical report information CERN 82-10 (1982)

(an exemplary job !)



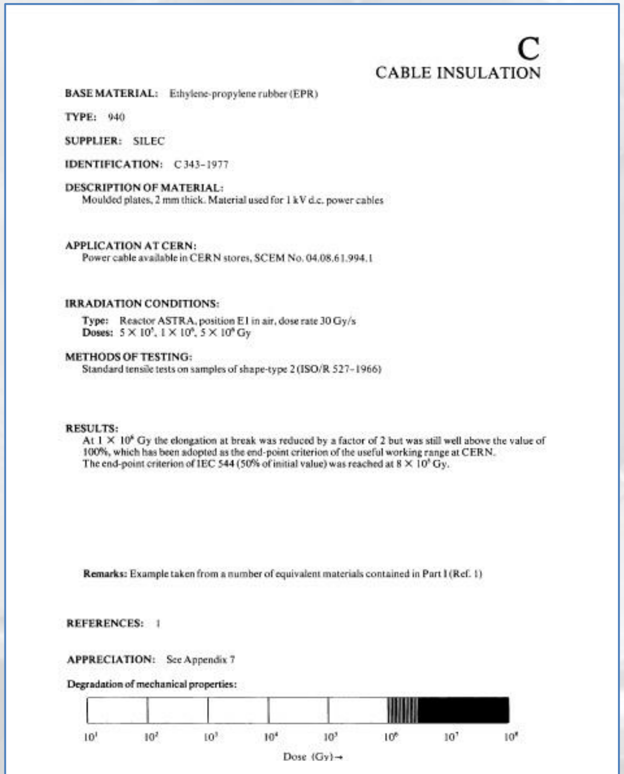
APPENDIX 5.8

General relative radiation effects: Thermoplastic resin

These appreciations are taken from the references cited and can only serve as a general guideline. Atmospheric and other environmental conditions such as temperature and dose rate are not taken into consideration. See also Sections 2 and 3.

	Damage	Utility
░	Incipient to mild	Nearly always usable
▨	Mild to moderate	Often satisfactory
▓	Moderate to severe	Not recommended

General information on materials: thermoplastic resins



C

CABLE INSULATION

BASE MATERIAL: Ethylene-propylene rubber (EPR)

TYPE: 940

SUPPLIER: SILEC

IDENTIFICATION: C 343-1977

DESCRIPTION OF MATERIAL:
Moulded plates, 2 mm thick. Material used for 1 kV d.c. power cables

APPLICATION AT CERN:
Power cable available in CERN stores, SCEM No. 04.08.61.994.1

IRRADIATION CONDITIONS:
Type: Reactor ASTRA, position E1 in air, dose rate 30 Gy/s
Doses: 5×10^5, 1×10^6, 5×10^6 Gy

METHODS OF TESTING:
Standard tensile tests on samples of shape-type 2 (ISO/R 527-1966)

RESULTS:
At 1×10^6 Gy the elongation at break was reduced by a factor of 2 but was still well above the value of 100%, which has been adopted as the end-point criterion of the useful working range at CERN.
The end-point criterion of IEC 544 (50% of initial value) was reached at 8×10^5 Gy.

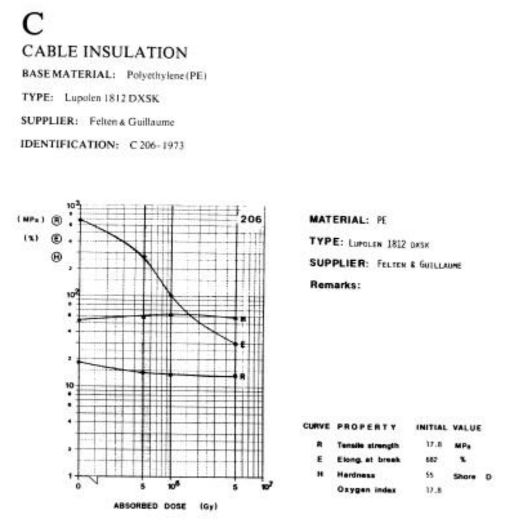
Remarks: Example taken from a number of equivalent materials contained in Part I (Ref. 1)

REFERENCES: 1

APPRECIATION: See Appendix 7

Degradation of mechanical properties:

Specific information: cable insulation by a supplier



C

CABLE INSULATION

BASE MATERIAL: Polyethylene (PE)

TYPE: Lupolen 1812 DXSK

SUPPLIER: Felten & Guillaume

IDENTIFICATION: C 206-1973

MATERIAL: PE

TYPE: Lupolen 1812 DXSK

SUPPLIER: Felten & Guillaume

Remarks:

CURVE	PROPERTY	INITIAL	VALUE
R	Tensile strength	17.8	MPa
E	Elong. at break	682	%
H	Hardness	55	Shore D
	Oxygen index	17.8	

Dose thresholds are specific to the item tested and to the end-point criteria applied

62

Typical irradiation sources used for organic materials:

a) 7 MW Astra reactor, Austria (Ebene 1 out of core position: 200 kGy/h; or switched off reactor))

b) ASTRA fuel elements (100-10 kGy/h), (to avoid activation)

c) ^{60}Co source (Dagneaux, France, 4.0 kGy/h) (to avoid activation)

d) CERN accelerators (10-100 Gy/h)

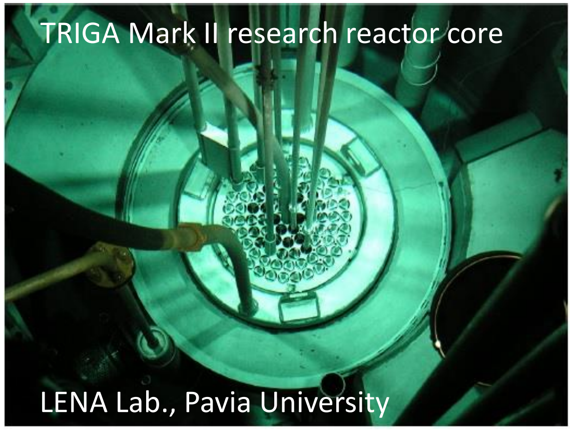
neutron dose
< 5%

Motivations for a new materials rad-hard study for SPES

- Rapid obsolescence of the data; new materials and suppliers available
- Rad-hard data are in general obtained in gamma fields only; very scarce data are available in mixed neutron and gamma fields. Is it really relevant?
- Large uncertainties in the rad-hard data provided by suppliers
- End use limits and dose thresholds are in general poorly defined

Market available products

→

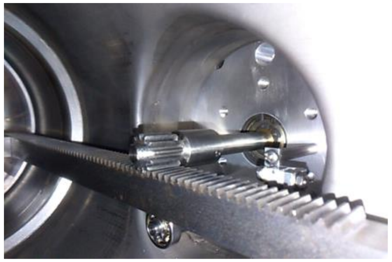


TRIGA Mark II research reactor core

LENA Lab., Pavia University

Nuclear reactor n + γ testing field

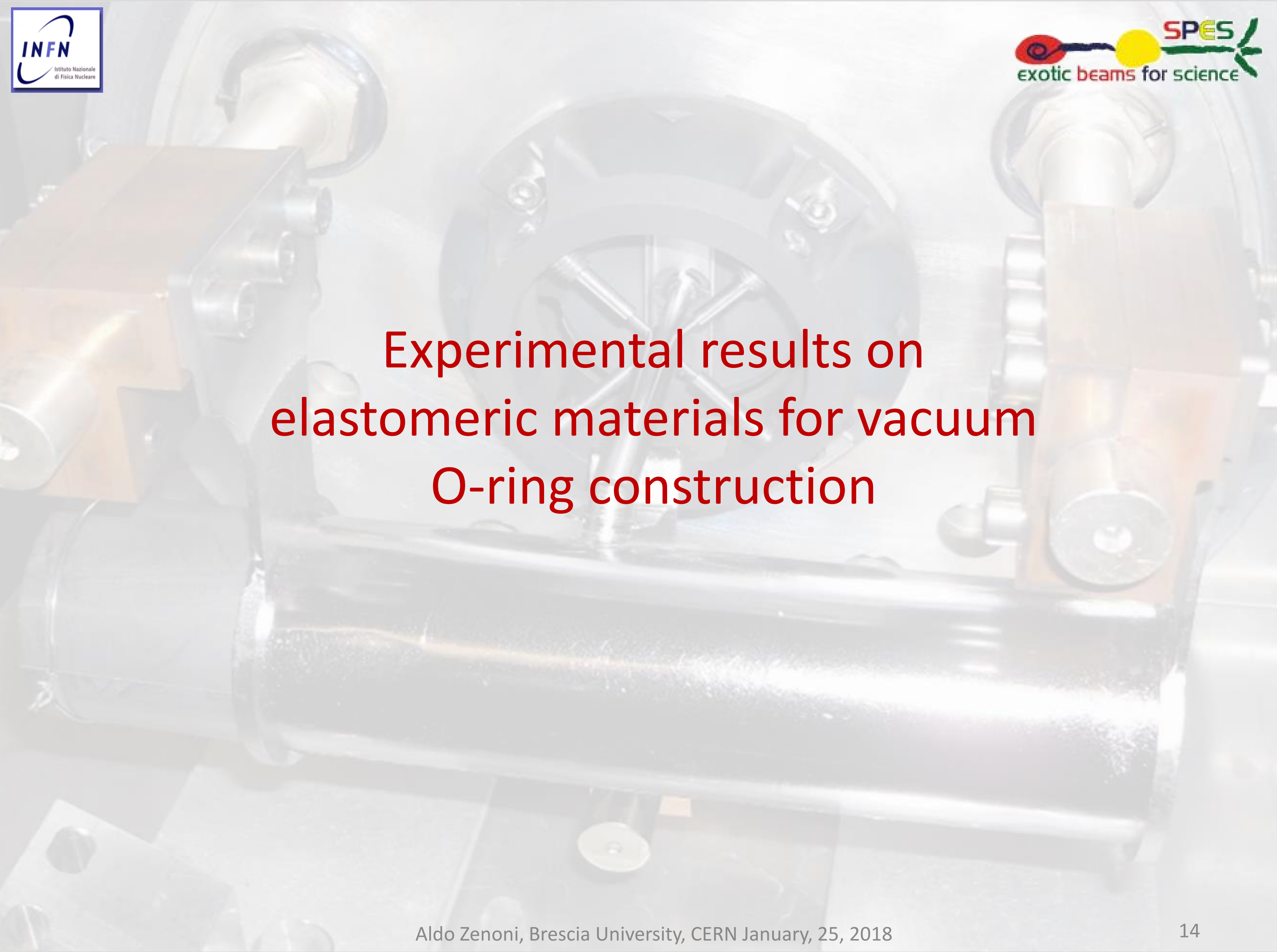
→



Specific applications

Ionizing radiation damage gamma fields vs fast neutron fields

- A generally accepted rule of thumb says that : “for equal energy absorbed, there will be equal radiation effect” (R.O. Bolt, 1963).
- In other words, it is assumed as a matter of fact that, for organic materials, the damage is to a large extent related to the absorbed dose irrespective of the type of radiation (CERN 82-10 (1982)).
- However, this is a not demonstrated fact, but only a first approximation. Gamma and neutron mechanisms for energy release are intrinsically different. Neutron energy release is more effective in the presence of hydrogen nuclei. Well known in biological applications.
- Radiation damage data with fast neutron fields are very scarce. It is easier and cheaper to irradiate polymers with gamma radiation rather than with neutron fields, which in addition generate material activation.
- Nevertheless, “there is a need to establish more exactly the relative effects of similar doses of fast neutrons and gammas in order to determine the validity of tests with gamma radiation in applications with fast neutron fields [fusion reactors]” (D.C. Phillips, AERE, R8923, Harwell (1978)).
- In the following, a comparison of radiation damage by gamma and neutron fields for lubricating greases and oils will be shown.



Experimental results on elastomeric materials for vacuum O-ring construction

Selected elastomeric materials

Selection criteria

- Good mechanical performance for vacuum and high temperatures
- EPDM is considered more radiation resistant than FPM
- FPM excellent vacuum behavior; it is more transparent to neutron fields (hydrogen content :1% vs 7-8%)
- Market availability and radiation stability indications by producers

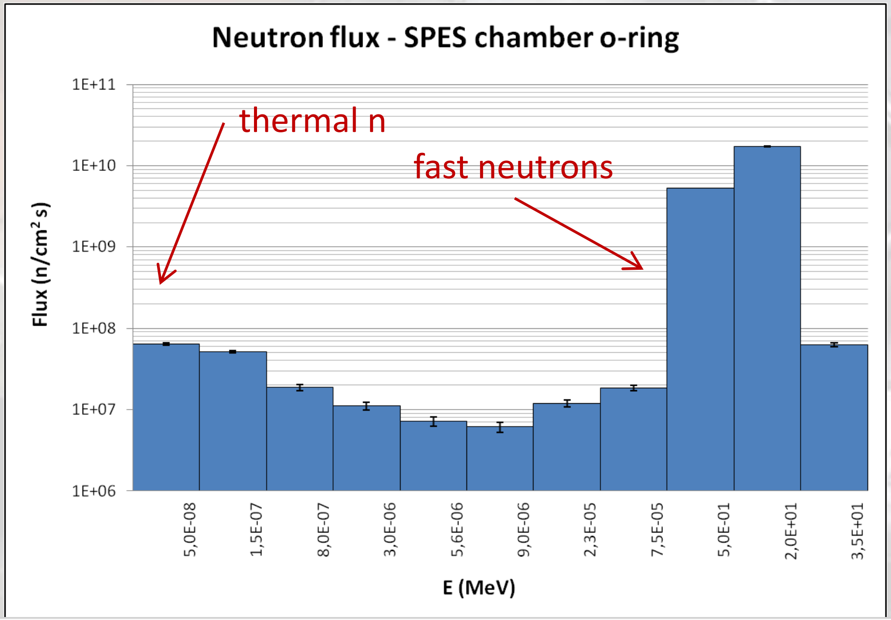
Product	Type/producer	Curing	Mechanical properties	Radiation Hardness	Quality	Notes
VITON	FPM/Generic		Good	Not declared	Standard	Low H content
EPDM1	EPDM/Generic	sulfur	Fair	Not declared	Cheap	LNL off-the-shelf
EPDM2	EPDM/ Dichtomatik (DE)	peroxide	Good	Not declared	High standard	Alternative standard
EPDM3	EPDM/ James Walker (UK)	peroxide	Very good	1.6 MGy dose ^{60}Co gammas	Certified material	Expensive

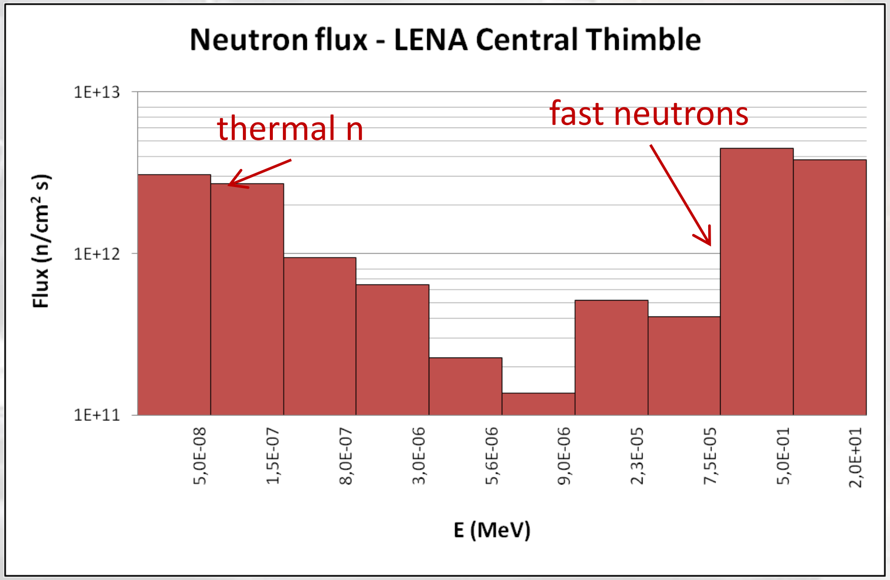
Modeling of the neutron and photon fields in the SPES-TIS and LENA Central Thimble (MCNPX)

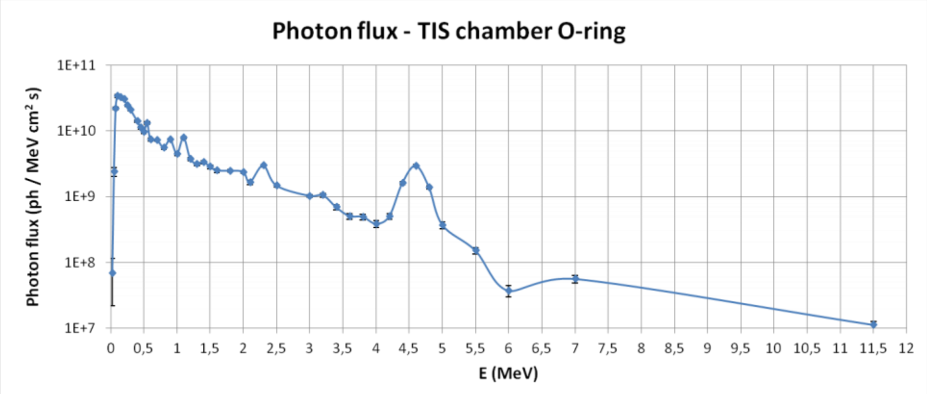
SPES Target & Ion Source

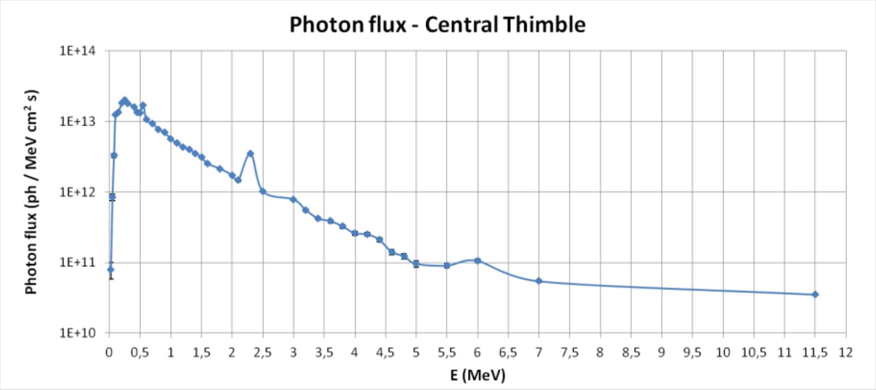
Central Thimble LENA

(0.0-0.05-0.15-0.8-3.0-5.6-9.0-23-75 eV -0.5-20-35 MeV)


Neutron flux - SPES chamber o-ring

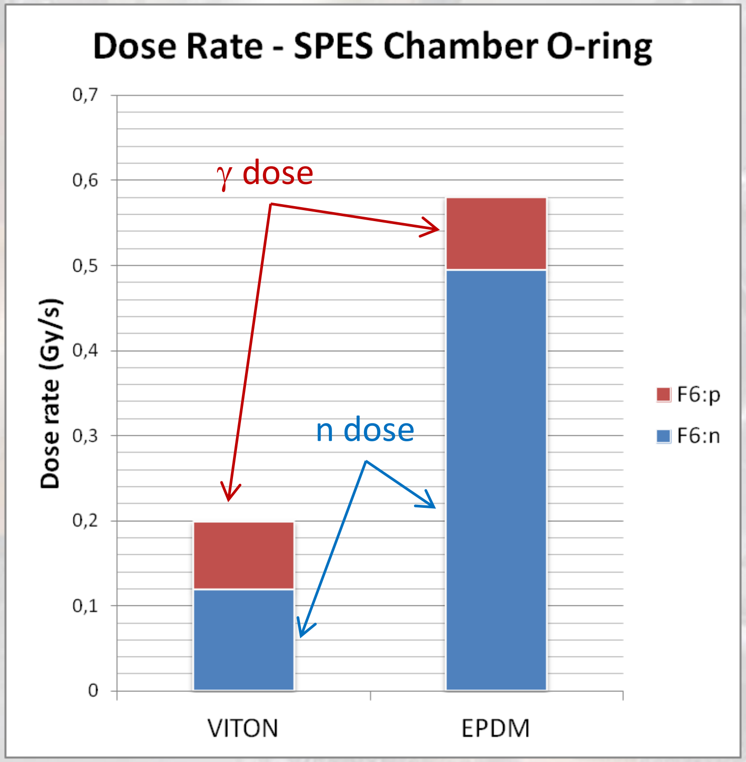

Neutron flux - LENA Central Thimble

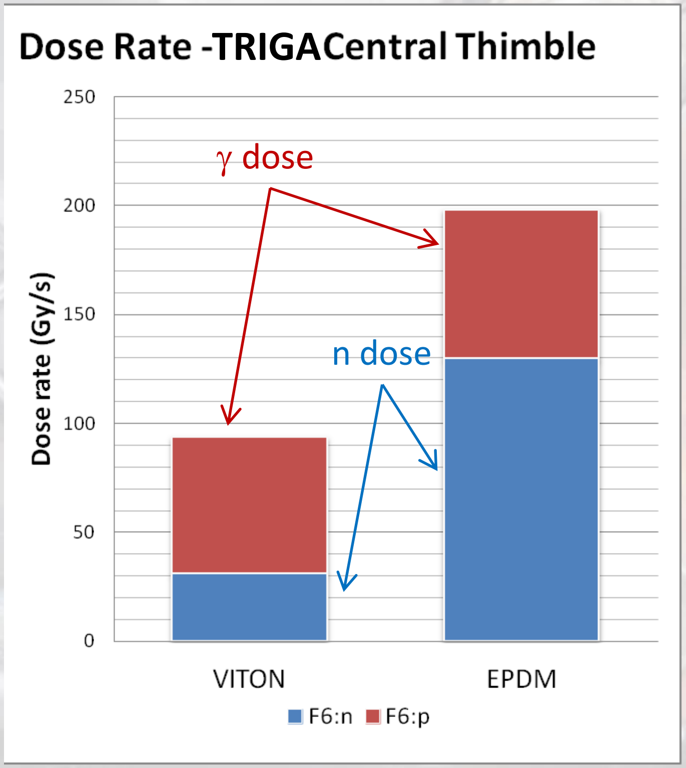

Photon flux - TIS chamber O-ring


Photon flux - Central Thimble

Dosimetry calculations (MCNPX)

FPM and EPDM typical compositions





FPM is more transparent to neutrons than EPDM

Most dose is delivered by fast neutrons

Doses n + γ on TIS integrated over 15 days

VITON	0.26 MGy
EPDM	0.75 MGy

Irradiation time to deliver 1 MGy by TRIGA Mark II reactor

VITON	2 h 58 min
EPDM	1 h 24 min

Materials properties tested as a function of absorbed dose

Mechanical and tensile properties

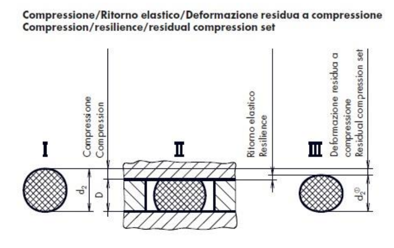
- Compression set (25% squeeze + ageing 24h at 100°C) (%)
- Elongation at break (%)
- Elastic Modulus (MPa)
- Tensile strength (MPa)
- Hardness (Shore, IRHD)

Physical and chemical properties

Density, FTIR analysis, DSC Calorimetry, CHNS composition, swelling test, DMTA

Correlate change in mechanical properties with microscopic modification

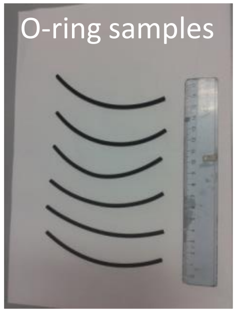
(ASTM D395-03)


Compressione/Ritorno elastico/Deformazione residua a compressione
Compression/resilience/residual compression set

Compression set

Universal dynamometer

(ASTM D1414)


O-ring samples


Calorimeter DSC

A sample of results: tensile properties EPDM2

3.0 h irr.
2.14 MGy

2.0 h irr.
1.43 MGy

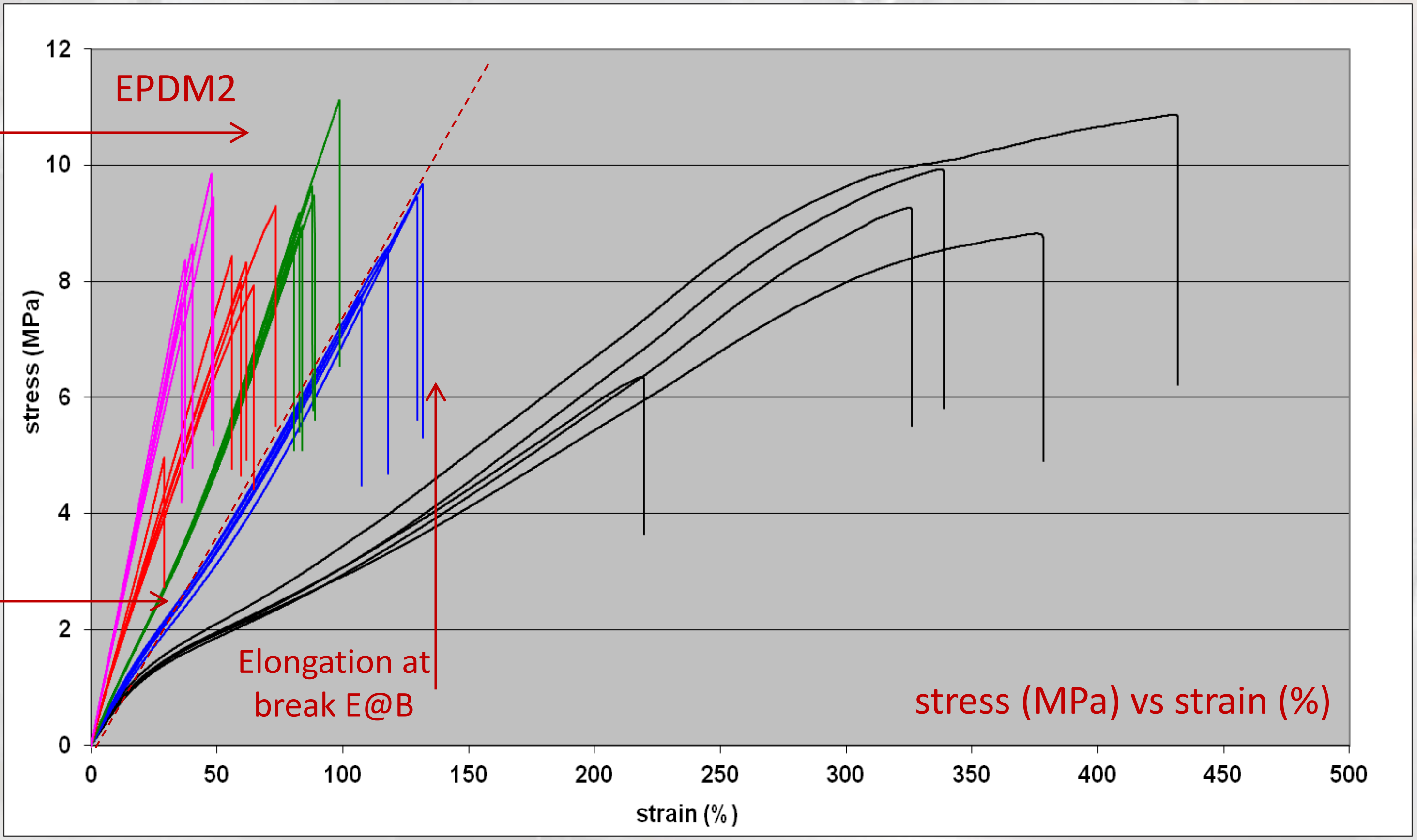
1.0 h irr.
0.71 MGy

0.5 h irr.
0.36 MGy

Non
irradiated

Tensile strength (TS)

Elastic modulus
(EM) slope



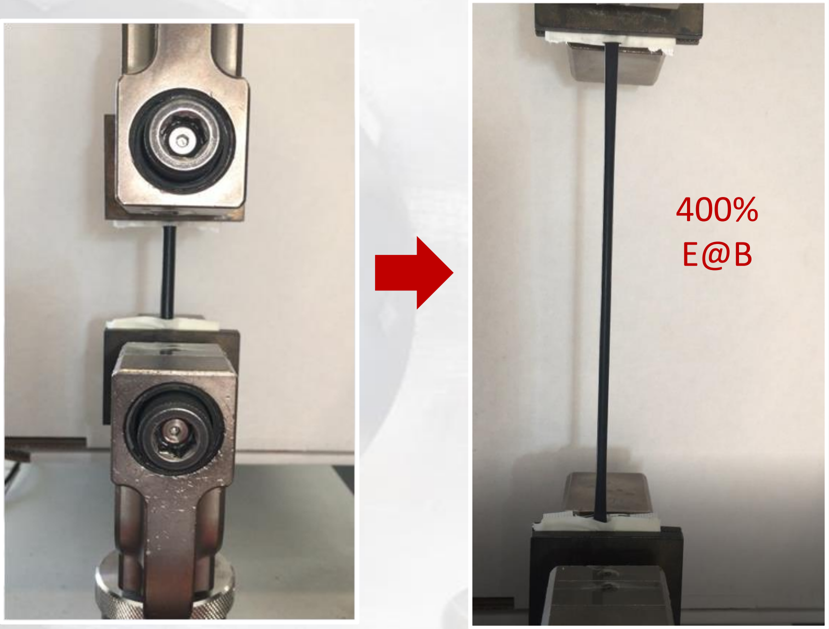
EPDM 2: 15 days in TIS	0.75 MGy

Good repeatability

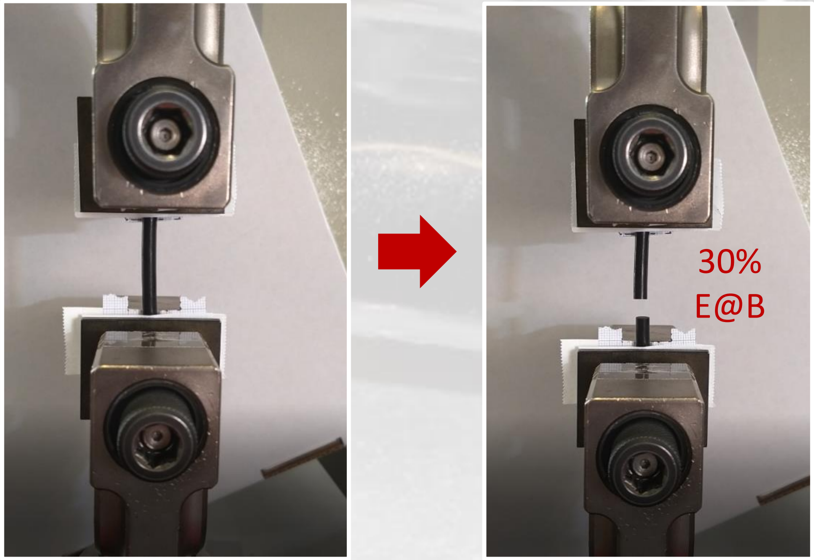
Material properties change considerably with irradiation

A sample of results: tensile properties EPDM2

Non Irradiated: 0.0 MGy
400% Elongation@break



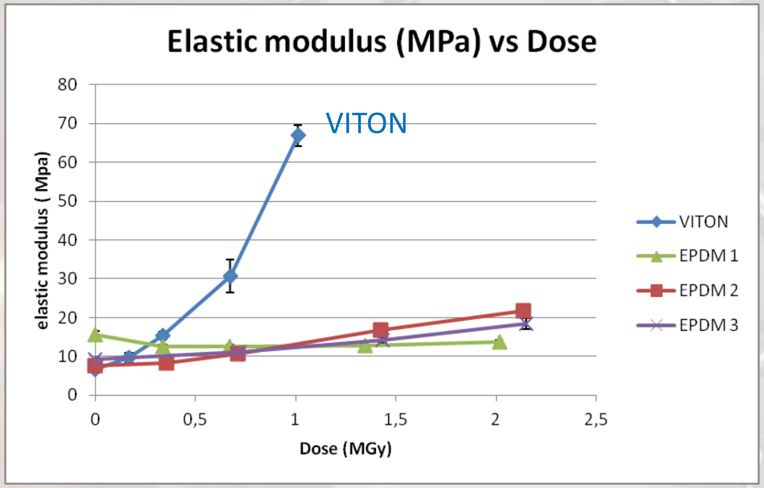
5 h irradiation: 3.55 MGy
30% Elongation@break

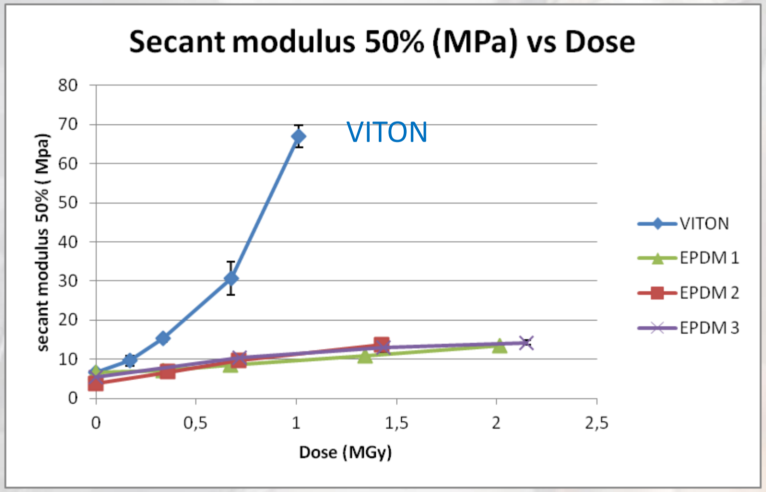


Huge radiation induced stiffness and embrittlement

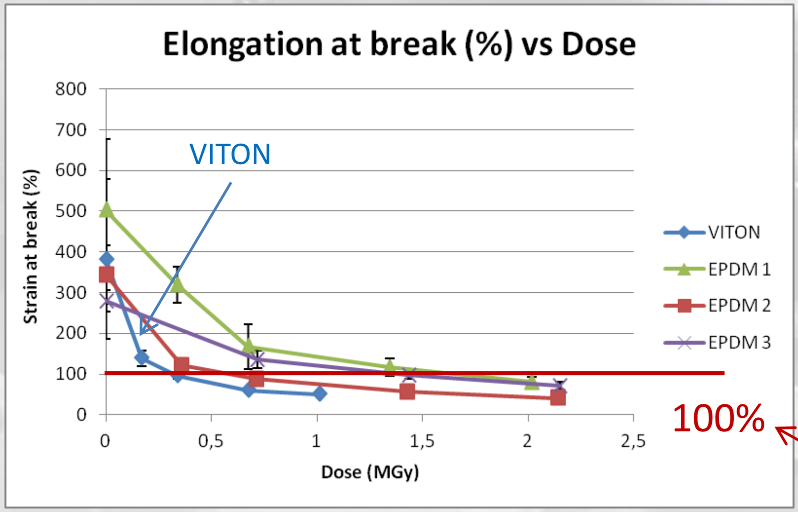
Comparison of tensile properties

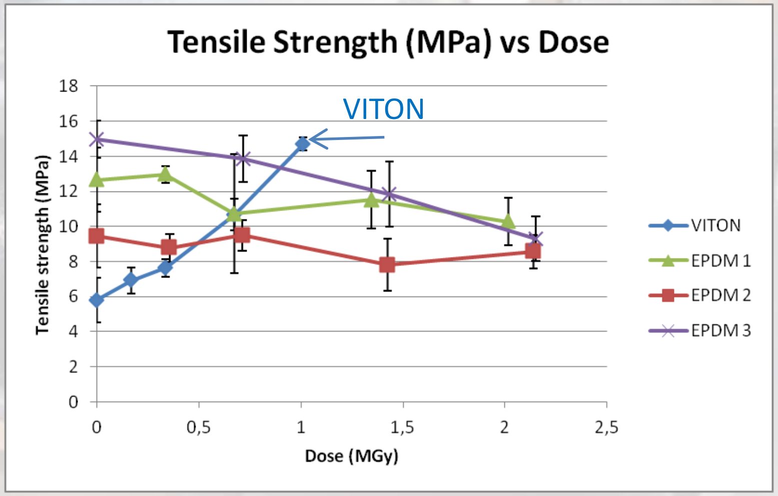
Modulus increase: stiffness, cross linking


Elastic modulus (MPa) vs Dose


Secant modulus 50% (MPa) vs Dose

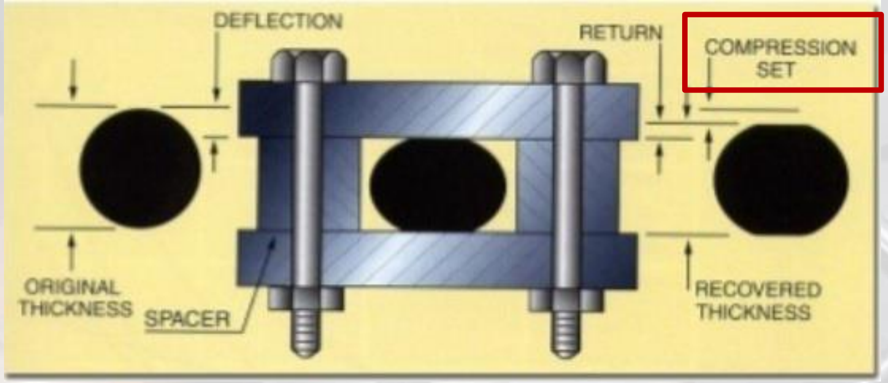
Ultimate properties: dramatic decrease of E@B


Elongation at break (%) vs Dose


Tensile Strength (MPa) vs Dose

Elastomer: E@B > 100%

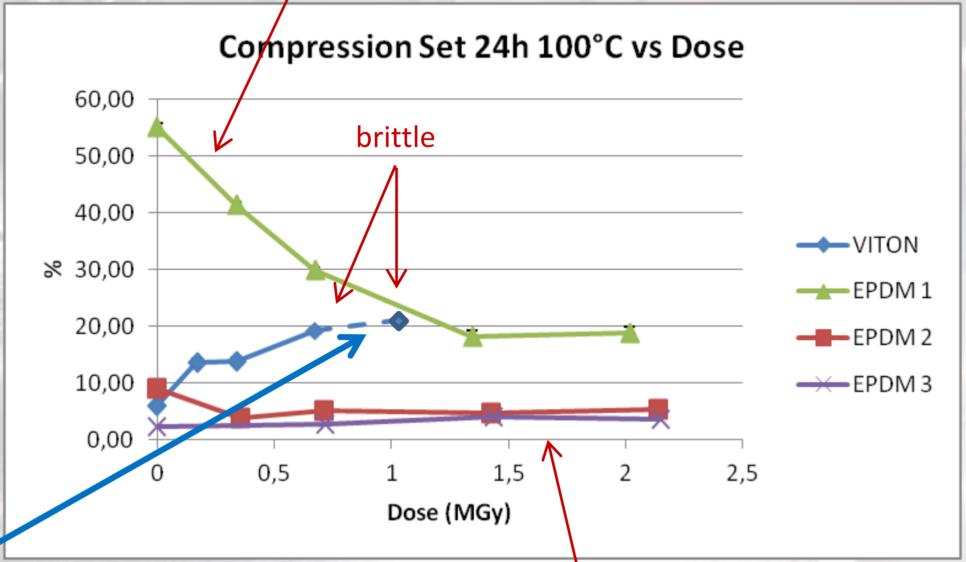
Comparison of functional response: compression set



Test conditions: 100°C, 25% strain, 24 hours

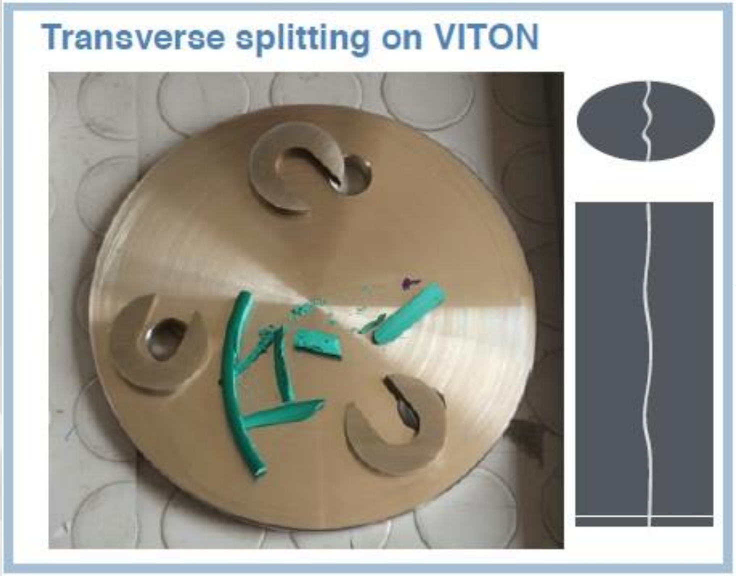
EPDM1: poor CS with large variations

brittle



EPDM2, EPDM3: stable at few %

Transverse splitting on VITON

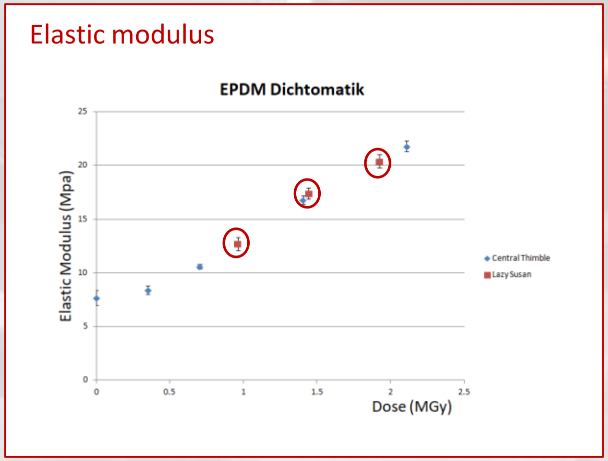


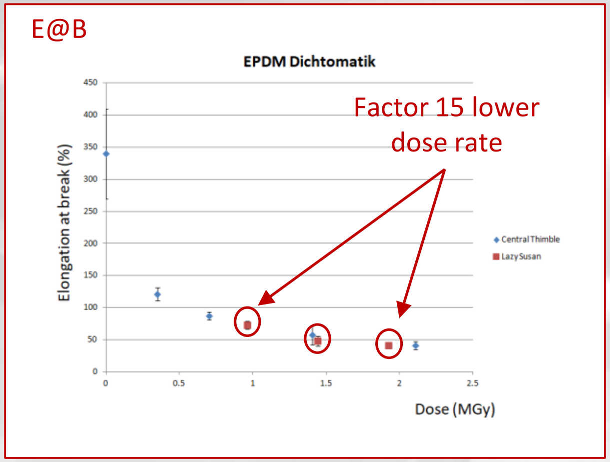
Conclusions on tested elastomers

- VITON (FPM) is more transparent than EPDM to fast neutron fields, but mechanical properties change a lot with dose. Dramatic increase of stiffness and brittleness under test irradiation conditions. (Not recommended).
- EPDM polymer based elastomers may have very different behaviors against irradiation, depending on the specific compound.
- EPDM1 (standard SPES) has fair mechanical properties that change considerably with irradiation. (Not recommended).
- EPDM2 and EPDM3 feature good mechanical properties that remain stable with absorbed dose (apart from E@B). Large price difference, apparently not justified by performance.
- Further elastomeric materials for O-ring construction to be tested ESS priority:
  1. Hayakawa 1111A, radiation resistant up to 2.0 MGy (gamma fields);
  2. Nichias EPDM-B, EP-176, radiation resistant up to 1.2 MGy (gamma fields) with excellent vacuum behavior.

Predictive capability of the performed tests on elastomers

- The predicting capability of accelerated aging tests in radiation environment is influenced by several factors: type of radiation fields, dose rate effects, presence and penetration of oxygen, temperature, sample geometry, synergistic effects.
- Reactor mixed fields have the same radiation components of the application, but order of magnitudes larger dose rate (300). Oxygen diffusion in the bulk material (DLO effect) may determine anaerobic or aerobic irradiation conditions, with different degradation mechanisms.
- Performed tests approximate anaerobic irradiation conditions, closer to conditions of the VAT valves O-rings irradiated in vacuum. For other vacuum O-rings, the results give a guideline of the behavior of the elastomers irradiated in the same conditions, on a comparative base.
- To test the effect of a different dose rate on material degradation, irradiations in a different reactor facility (Lazy Susan) has been performed , with a factor 15 less dose rate. Obtained results are encouraging.


Elastic modulus


E@B

Factor 15 lower dose rate


VAT valves O-rings

Experimental results on lubricating oils and greases

Lubricant typical composition and characteristic properties

Lubricant	Blend	Phase	Characteristic property
Oil	Base oil >75% Additives < 5%	Fluid	Dynamic Viscosity (cP=mPa s): *the ratio between the applied shear stress and the rate of shear of a liquid* (Viscometer)
Grease	Base oil > 75% Additives <5% Thickener 5%-25% sponge structure (metallic soap, fatty acids)	Semisolid two-phase systems	Consistency (mm/10): *resistance to deformation and flow; penetration of a cone of specified dimensions and mass under gravity within 5 s* (Penetrometer)

Polymeric materials 75% - 100% of the blend, petroleum based or synthetic

Additives <5% improve base oil performance

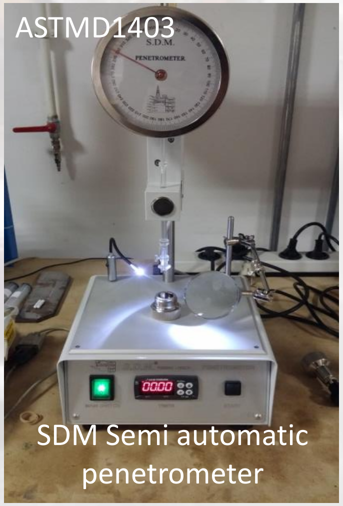
- Viscosity improvers
- Antiwear
- Foam inhibitors
- Corrosion inhibitors
- Thickeners
- Antioxydants
- Radiation Damage Inhibitors

Radiation sensitive or used up during irradiation

ASTMD445-06

Brookfield DV1 Viscometer

ASTMD1403



SDM Semi automatic penetrometer

Consistency and NLGI grade scale

Manipulator for worked consistency

Cone penetrating the worked grease during 5 s under his weight

The grease is a semi-fluid to solid two-phase system

NLGI number	ASTM worked (60 strokes) penetration at 25 °C *tenths of a millimetre*	Appearance
000	445-475	fluid
00	400-430	semi-fluid
0	355-385	very soft
1	310-340	soft
2	265-295	"normal" grease
3	220-250	firm
4	175-205	very firm
5	130-160	hard
6	85-115	very hard

Radiation damage

NLGI: National Lubricating Grease Institute

NLGI consistency numbers for worked penetration

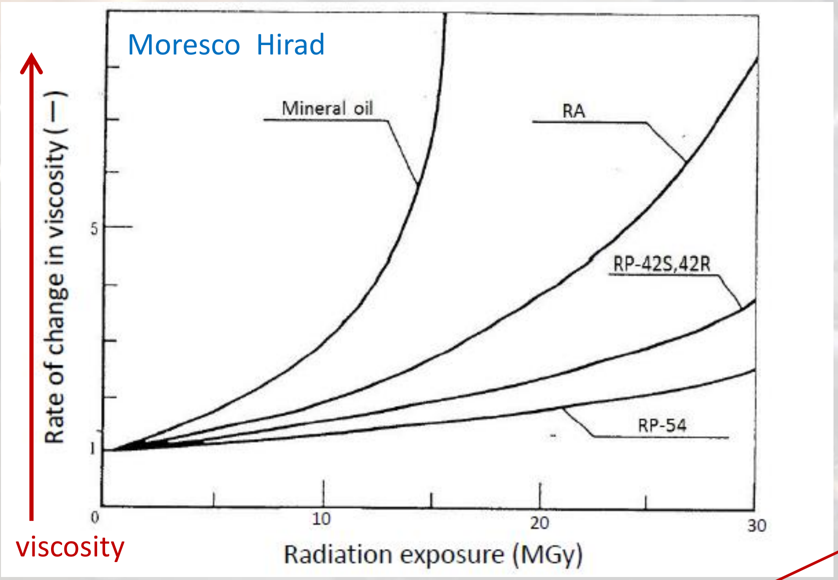
Expected effects of radiation on lubricants

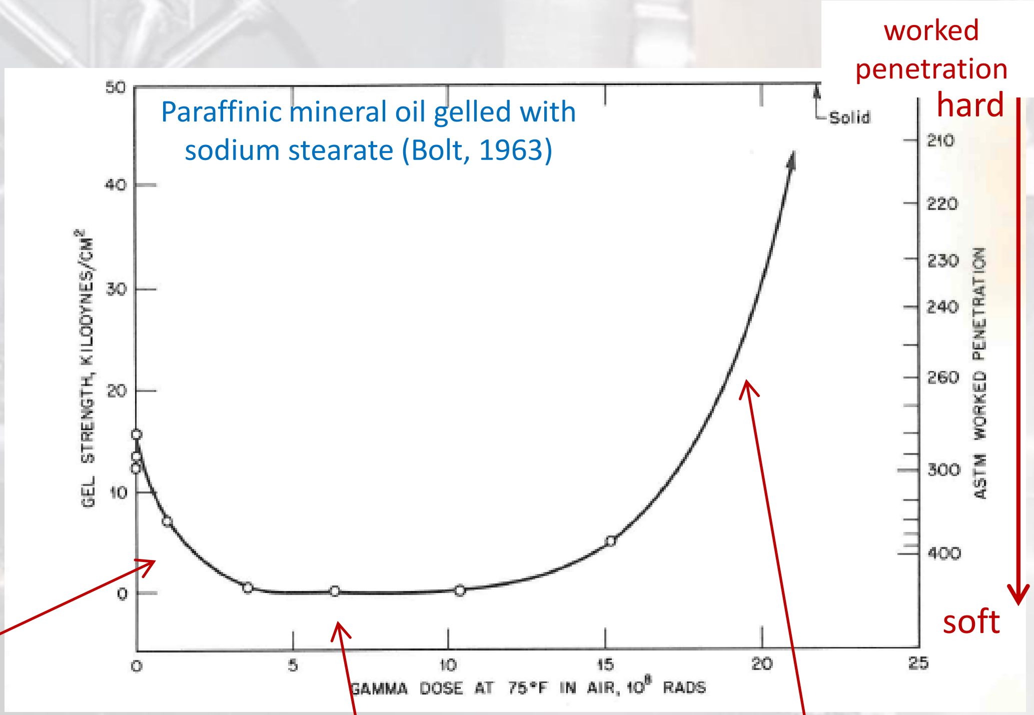
Lubricating oils

- Radiation stability mainly determined by base oil nature (aromatic content)
- Dominant effect: cross-linking
- Viscosity increase

Lubricating greases

- More complex situation
- Two main effects on consistency




Paraffinic mineral oil gelled with sodium stearate (Bolt, 1963)

Damage to the thickener grease softening

Dropping risk

Cross-linking of the base oil grease hardening

Selected greases to be tested

Product/Class	Producer	Base Polymer + Thickener	Consistency/ Viscosity (cSt)	Radiation Hardness	Quality
Grizzly Grease n.1	Lubcon (DE)	Mineral + Li/Ca	NLGI 0	1.2 MGy, gamma	Used at CERN
Turmopolgrease 2	Lubcon (DE)	Polyglicol + Li thickener	NLGI 2	Not declared	Interesting for ESS
AFB-LF	THK (J)	Mineral oil + Li thickener	NLGI 2	Not declared	Interesting for SPES (bearings)
Krytox^{TM} 240 AC	Dupont-Chemours (USA)	PFPE + PTFE, H free	NLGI 2	1.0 MGy, reactor mixed field	Fluorinated Hydrogen free
FAG Arcanol Load 220	Schaeffler (DE)	Mineral oil + mixed thickener	NLGI 1-2	Not declared	ESS priority
Kluberlub BE 41-542	Kluber (DE)	Mineral oil + Li thickener	NLGI 2	Not declared	ESS priority (EP)
Petamo GHY 133N	Kluber (DE)	Mineral oil + polyurea	NLGI 2	Not declared	ESS priority, standard SPES
RG-42R-1 (grease)	MORESCO (JP)	PPE + Polycarbonate	NLGI 1	15.0 MGy, gamma	High radiation hardness
RP-42R (base oil)	MORESCO (JP)	PPE	240 cSt at 40°C	15.0 MGy, gamma	base oil
Apiezon-M (thick oil)	M&I Materials (UK)	Hydrocarbon, halogen free	Molten grease 413 cSt at 50°C	1.0 MGy, electrons	Ultra high vacuum

Information on radiation resistance from producers with large uncertainties in testing procedures

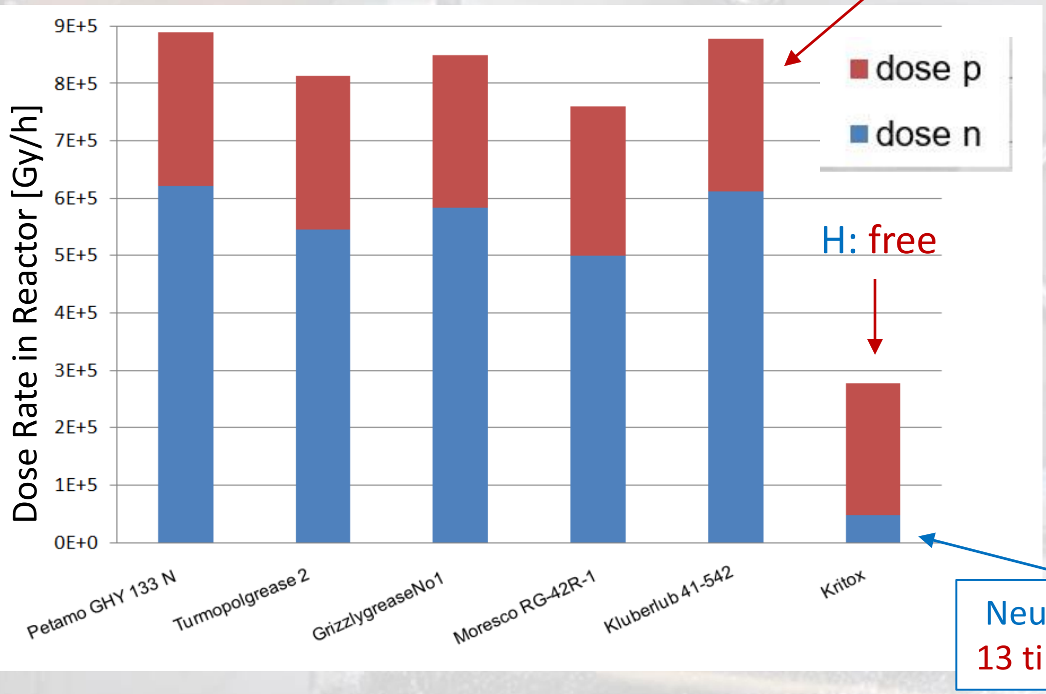
High radiation resistance

No H content , more transparent to neutrons

Dosimetry calculations (MCNPX)

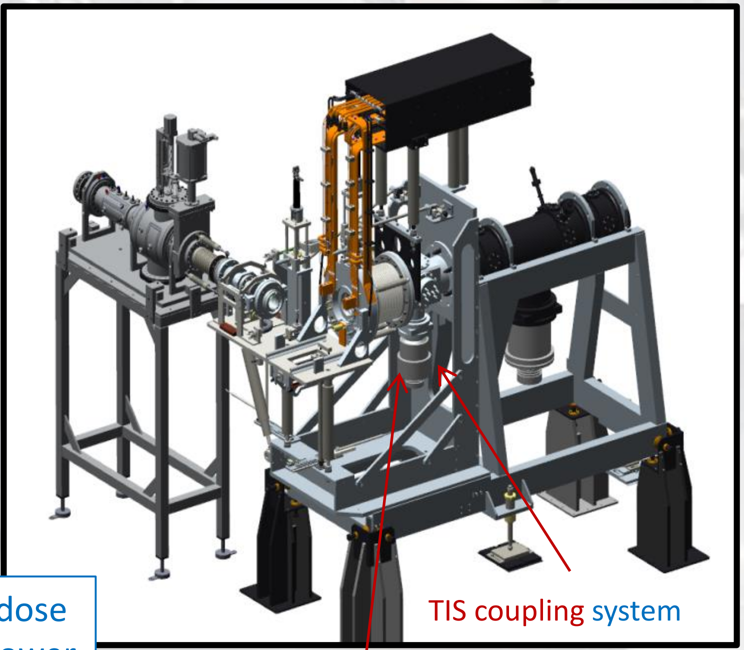
H: 9.68% - 12.43%

C, H, N content measured by CHNS analyses



H: free

Neutron dose
13 times lower



- Absorbed dose in mixed fields is strongly dependent on H content.
- The main mechanism of neutron dose absorption is scattering of fast neutron on H nuclei.
- Gamma dose is material independent.
- Material composition is crucial.

7 y integrated dose
70 cycles (15 days)

H: free
4.1 MGy

H: 12%
27 MGy

Preliminary tests performed/1

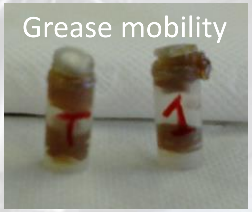
1) CHNS analyses to evaluate composition of materials: in particular Hydrogen content.

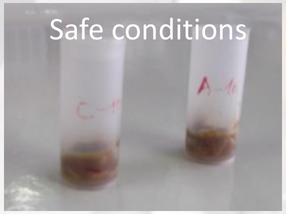
Product	C	H	N	Base oil
Grizzly Grease n.1	79.23%	11.63%	1.01%	Mineral
Krytox 143 AA	21.03%	0.0%	0.18	PFPE (fluorinated)

2) Gas development evolution under irradiation: mobility evaluation, design of irradiation set-up and protocol

Grizzly Grease n.1


Stopper removed


Grease mobility

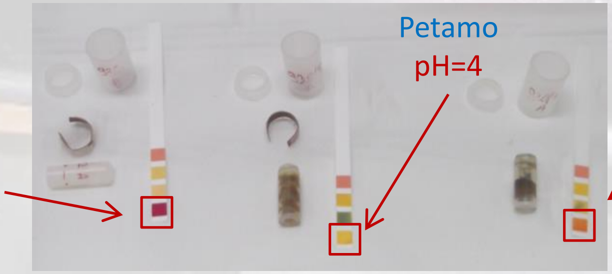

Safe conditions

3) Residual contact dose and NAA : activation, contamination and radioactive waste production

Safe handling after 10-15 days

Sample 100 mg (6 h irr)	3 day cooling	10 day cooling
Petamo GHY-133N	0.4 μSv/h	0.1 – 0.2 μSv/h
Krytox 283 AB	30 μSv/h (^{24}Na)	0.1 – 0.2 μSv/h

4) Acid production: halogen formation and ambient corrosion


Krytox pH<1
F content
critical

Petamo
pH=4

Grizzly Grease n.1
pH=7
neutral

Preliminary tests performed/2

Plastic syringe failure under gas development

Grizzly Grease n.1, 14 mL : 0.9 MGy

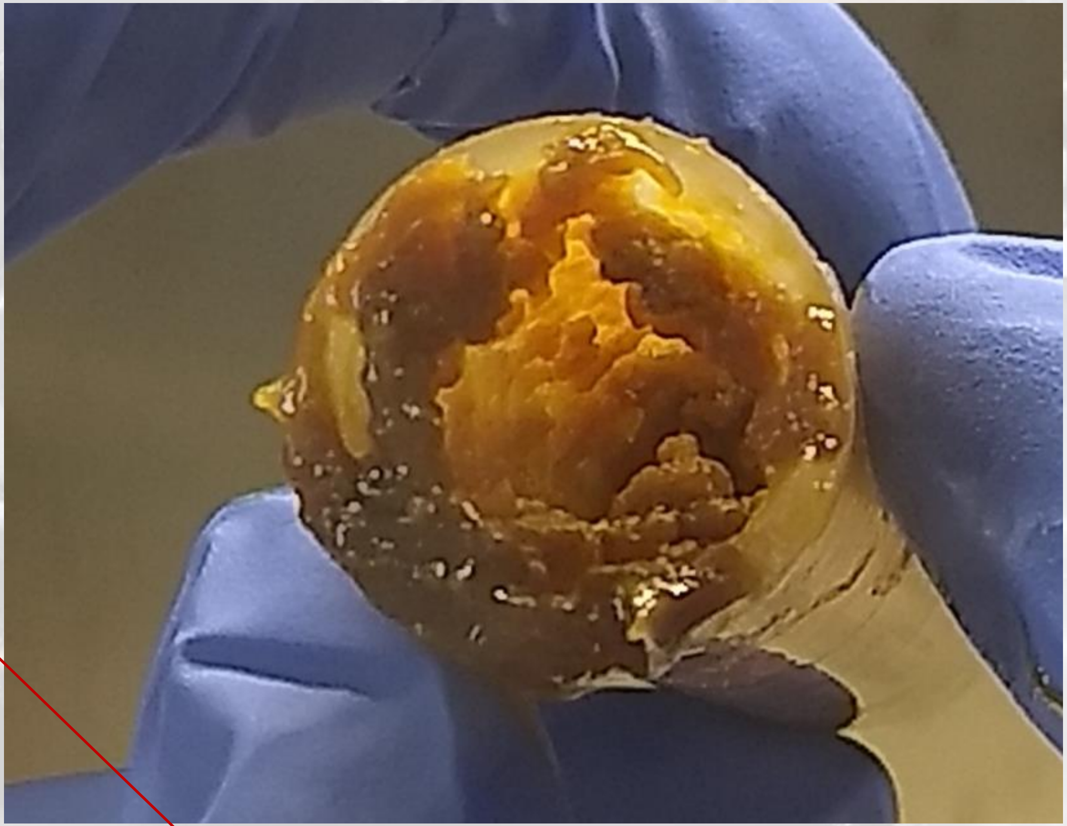
Final efficient and safe set-up

Ø = 27 mm
H = 140 mm

Perforated tapper
gas evolution

Welded tip

Gas development, grease mobility and overflow



20 mL plastic syringe not sealed
8 mL grease = one point

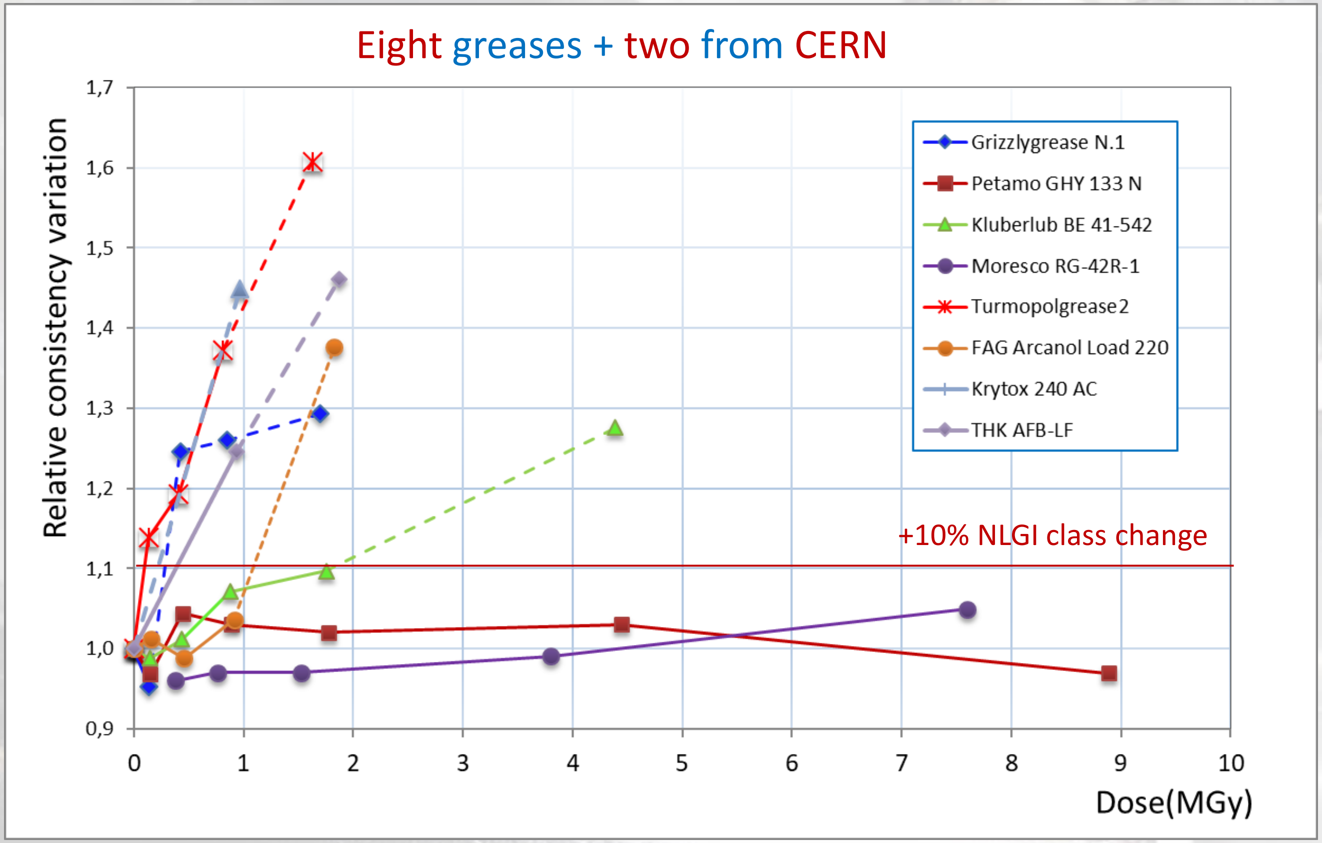
Experimental results on greases (overview)

Evolution of Consistency with dose

Irradiation times:
0 min, 10 min, 30 min,
1.0 h, 2.0 h, 5.0 h, 10 h

Dose range:
0.12 MGy – 8.9 MGy
(70% neutron dose)

Dose rate:
0.28 MGy/h – 0.94 MGy/h

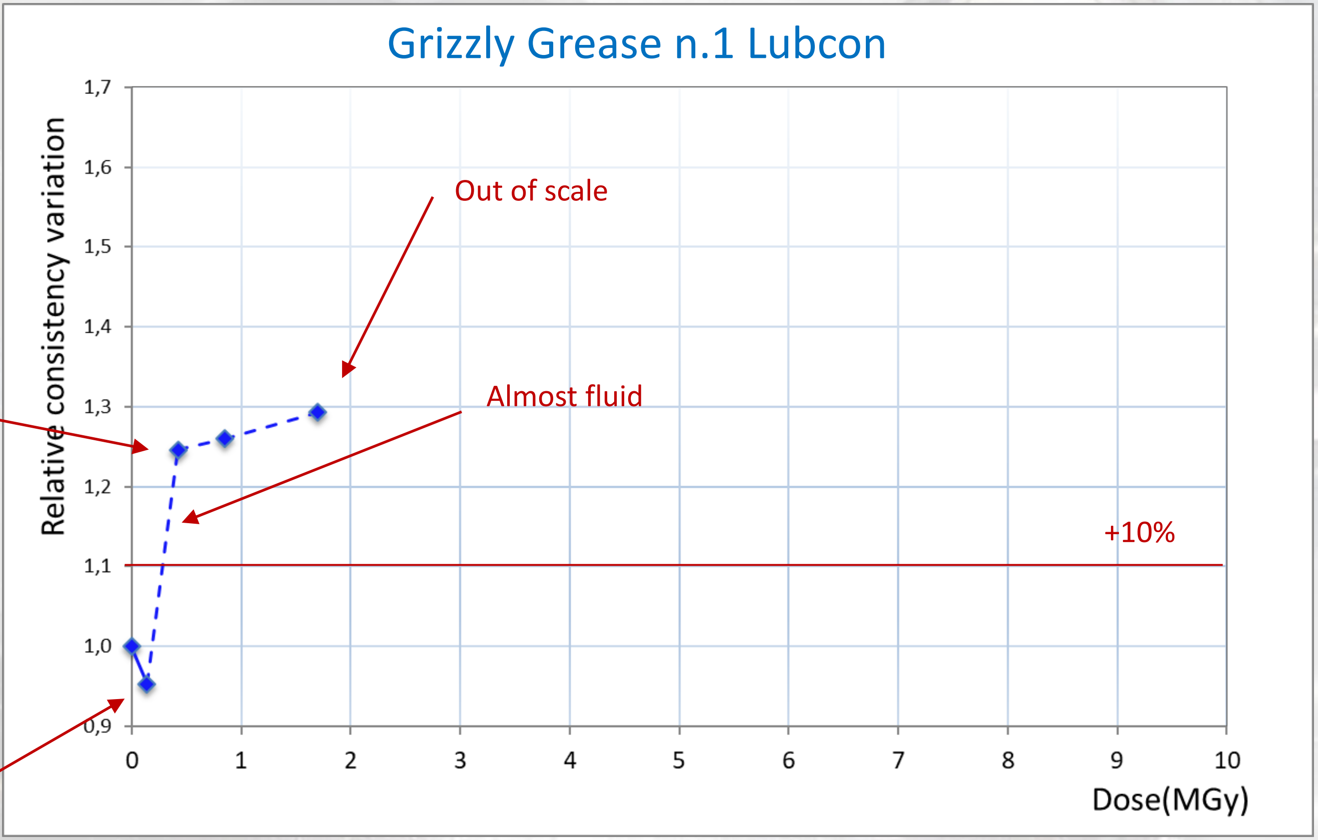


Overall results:

- Clear radiation induced modification: 10% Consistency variation = NGLI class change
- General increase of Consistency (grease softening, expected)
- Some rad-hard behavior at variance from gamma tests expectations
- In some cases very high radiation damage at low dose values: complete fluidization

Experimental results on greases/1

Evolution of Consistency with dose



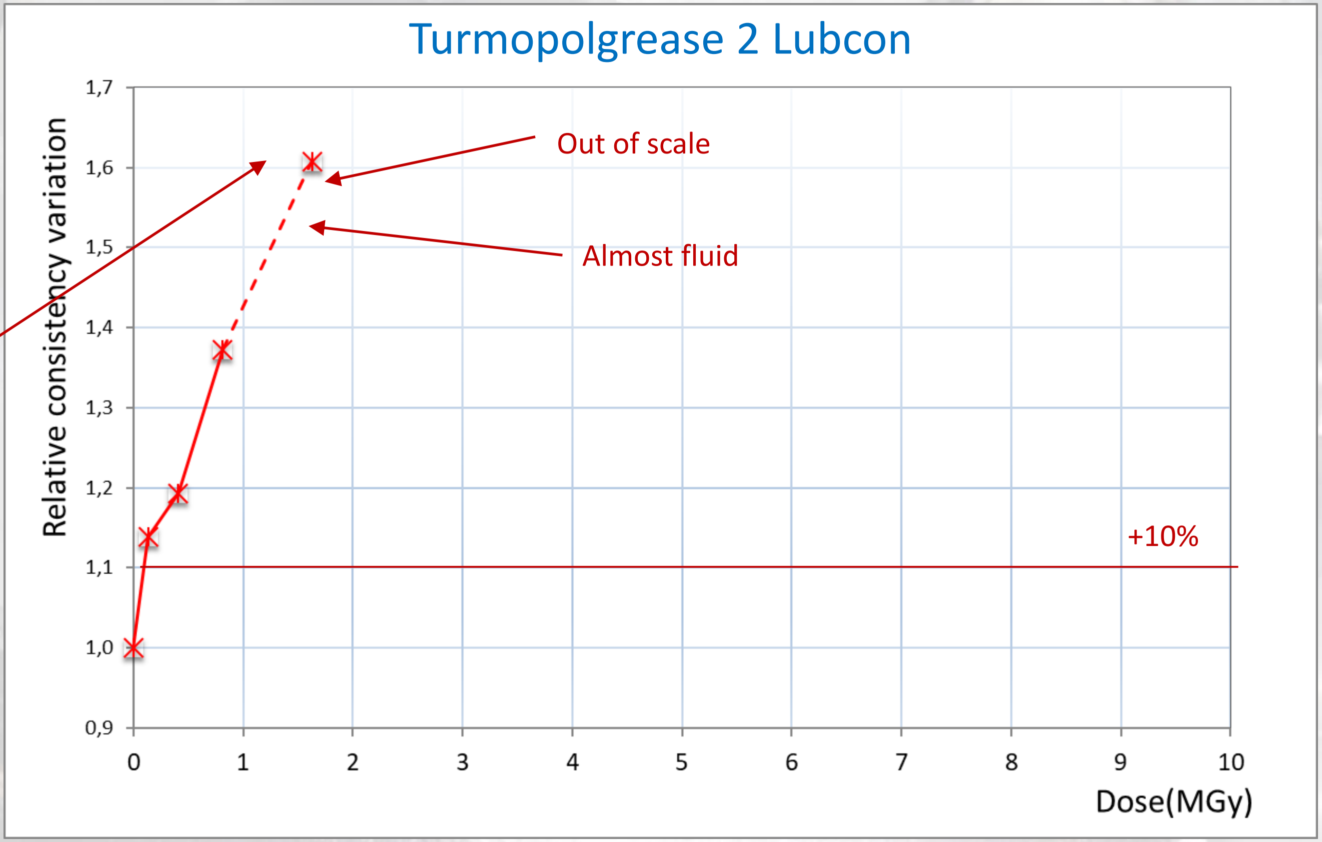
- standard at CERN
- Mineral oil + Li/Ca thickener: NLGI class 0
- Declared radiation resistance: up to 1.2 MGy (with gamma fields)
- Complete fluidization at 0.45 MGy
- Irradiation conditions (fields, dose rates) are crucial
- Definition of radiation resistance by producers to be questioned

Experimental results on greases/2

Evolution of Consistency with dose


1.63 MGy

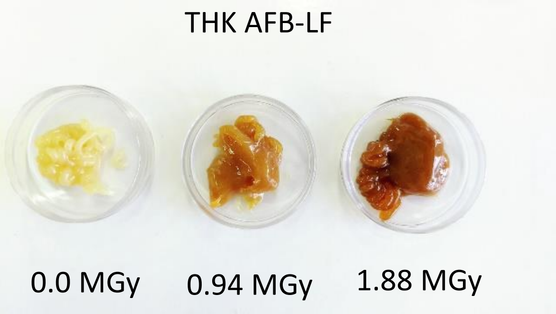
Turmopolgrease 2



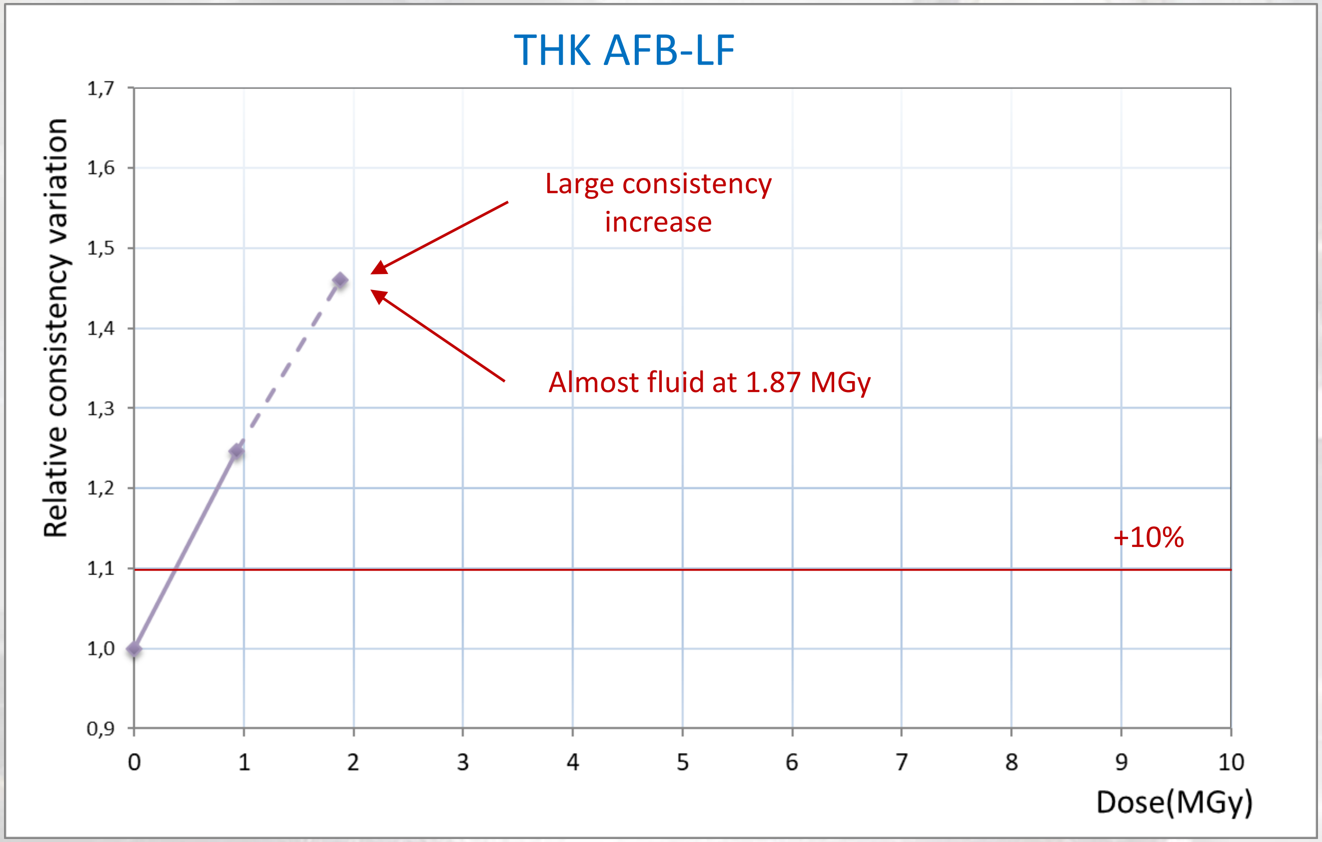
- Interesting for ESS
- Polyglicol + Li thickener: NLGI class 2
- Declared radiation resistance: Not declared
- Large Consistency increase up to about 1.0 MGy
- Almost fluid at 1.63 MGy

Experimental results on greases/3

Evolution of
Consistency with dose



Color change?
Expected effect of additives?



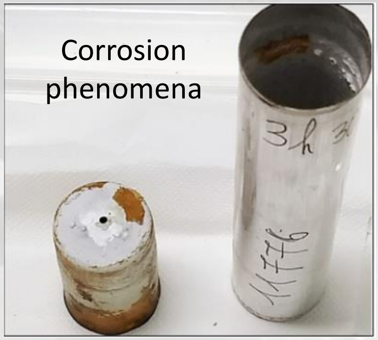
Large consistency increase

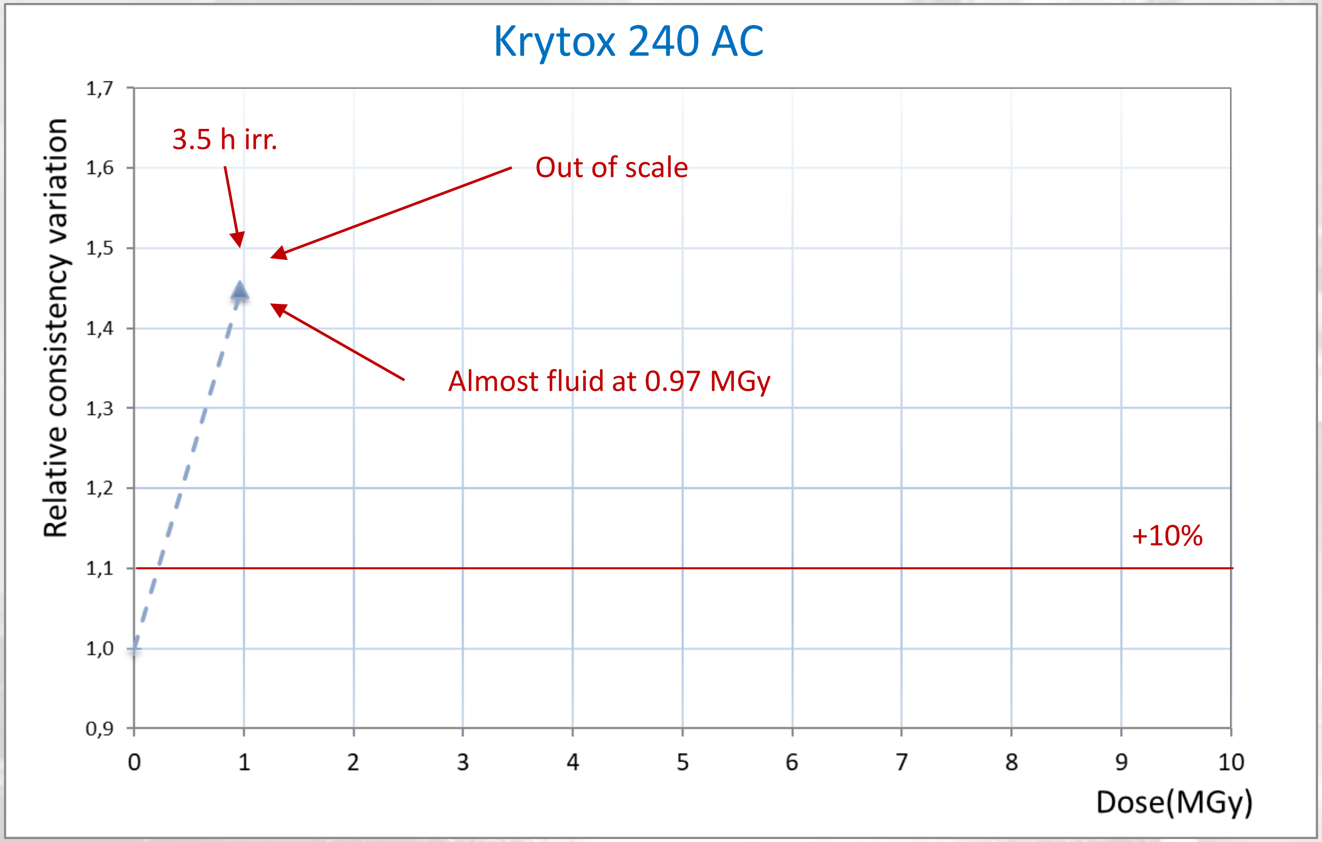
Almost fluid at 1.87 MGy

- Interesting for SPES (bearings)
- Mineral oil + Li thickener: NLGI class 2
- Declared radiation resistance: Not declared
- Large Consistency increase up to about 1.0 MGy
- Almost fluid at 1.87 MGy
- SPES TIS coupling system: 27 MGy dose in 7 years

Experimental results on greases/4

Evolution of Consistency with dose





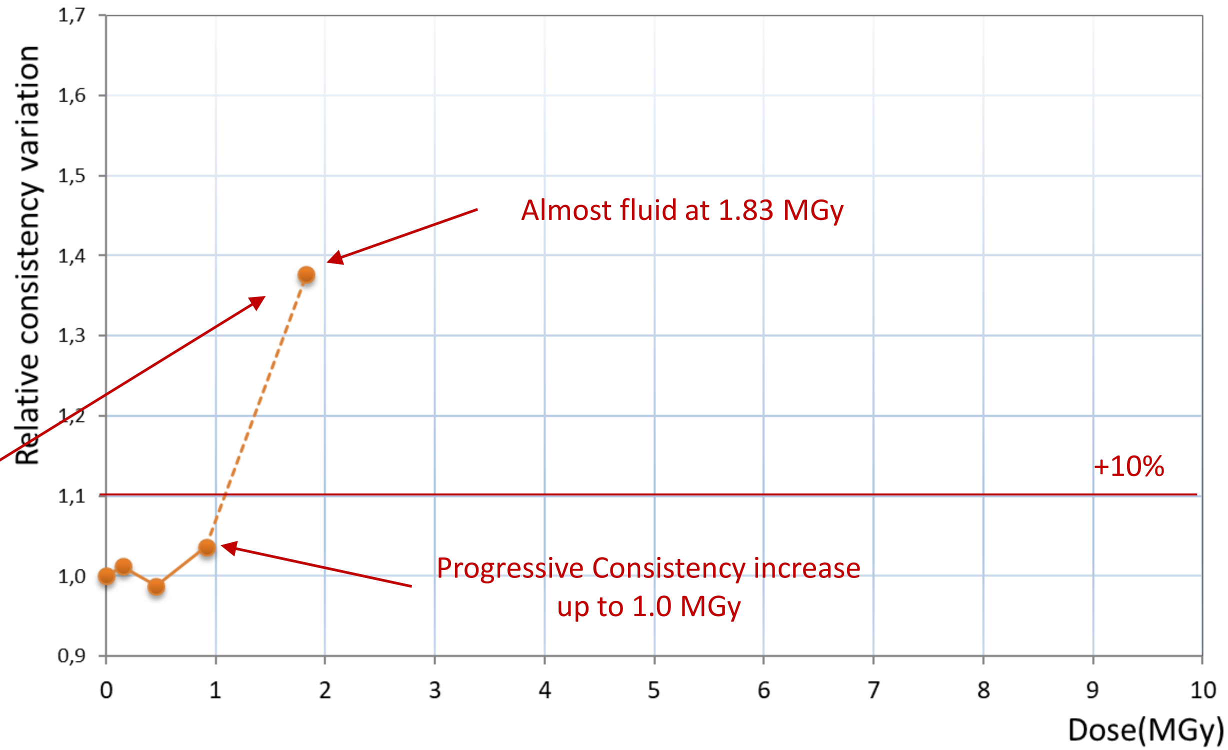
- Aero spatial applications (very expensive!)
- Fluorinated material (very low H content), NLGI class 2
- Almost fluid at 0.97 MGy
- Declared radiation resistance: 0.2 MGy – 1.0 MGy
- Evident corrosion effects, acid gas production

Experimental results on greases/5

Evolution of
Consistency with dose

FAG Arcanol Load 220

Schaeffler FAG Arcanol Load 220



Almost fluid at 1.83 MGy

+10%

Progressive Consistency increase
up to 1.0 MGy

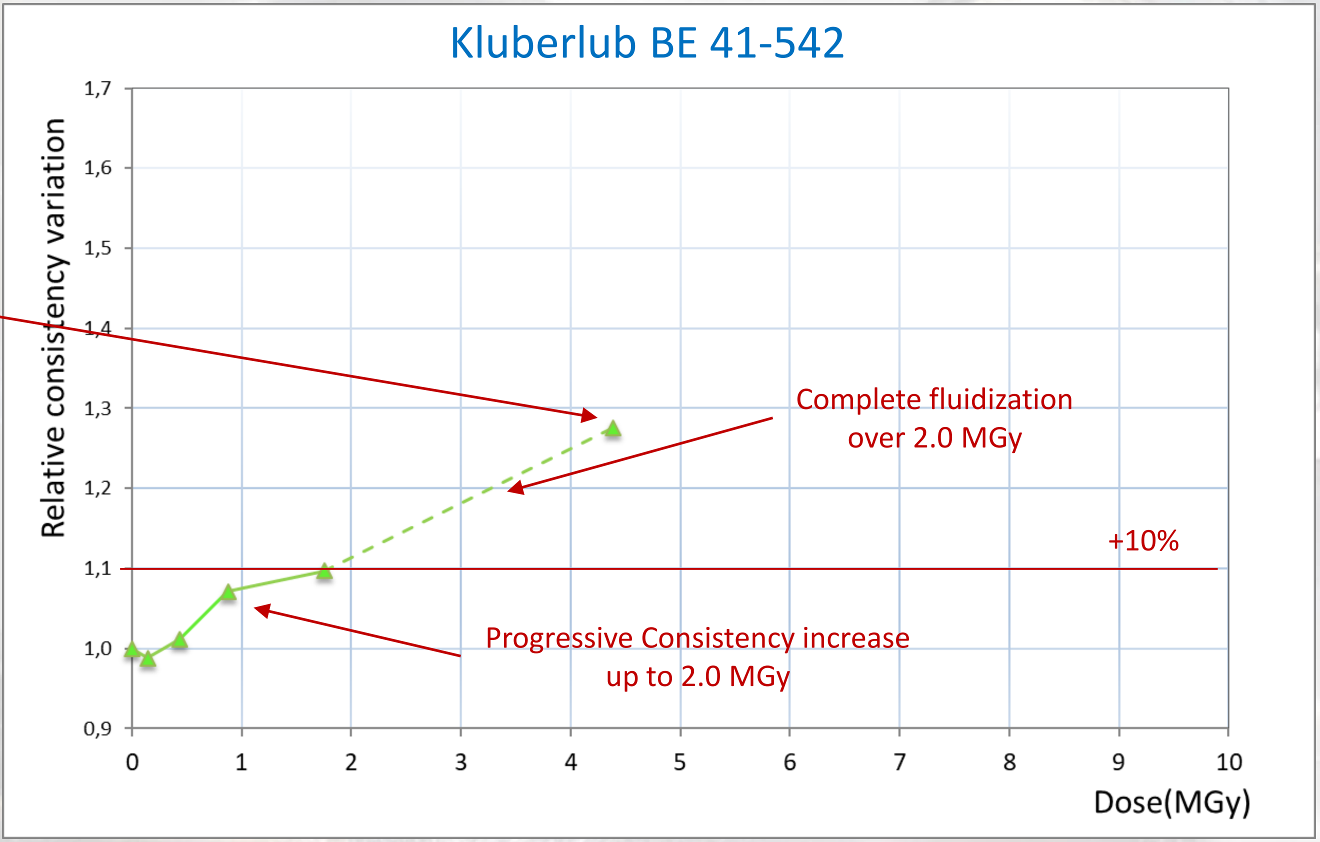
- Priority for ESS
- Mineral oil + mixed thickener: NLGI class 1-2
- High Load Grease
- Declared radiation resistance: Not declared
- Progressive Consistency increase up to about 1.0 MGy
- Almost fluid at 1.83 MGy

Experimental results on greases/6

Evolution of Consistency with dose

Kluberlub BE 41-542

Kluberlub BE 41-542



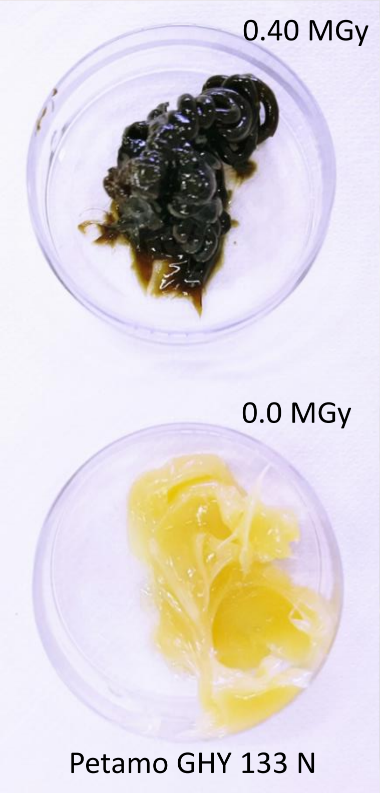
Complete fluidization over 2.0 MGy

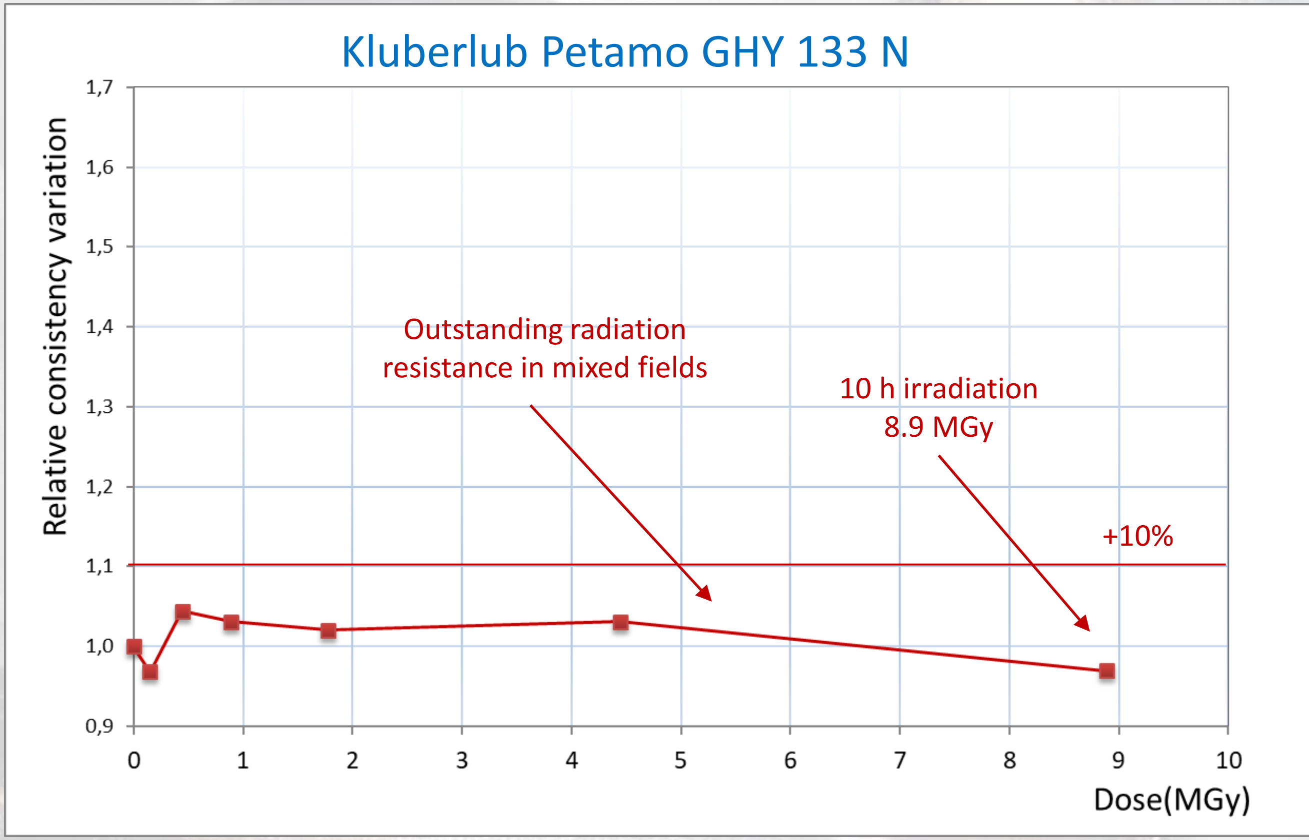
Progressive Consistency increase up to 2.0 MGy

- Priority for ESS
- Mineral oil + Li thickener: NLGI class 2
- Extreme Pressure (EP)
- Declared radiation resistance: Not declared
- Progressive Consistency increase up to about 2.0 MGy
- Complete fluidization over 2.0 MGy

Experimental results on greases/7

Evolution of Consistency with dose



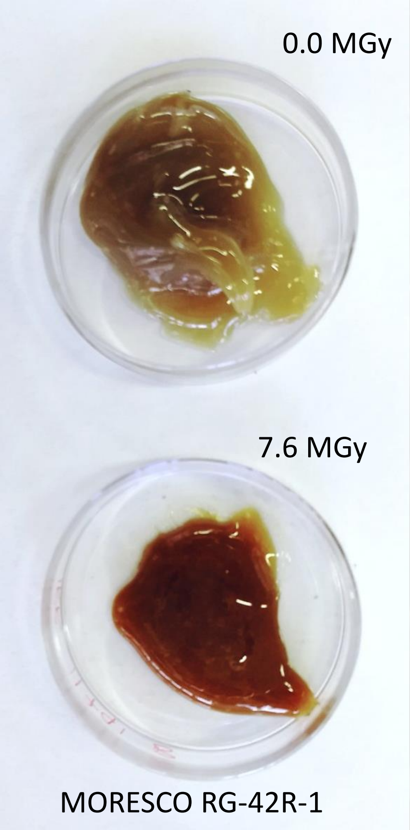


Outstanding darkening
Expected effect of additives?

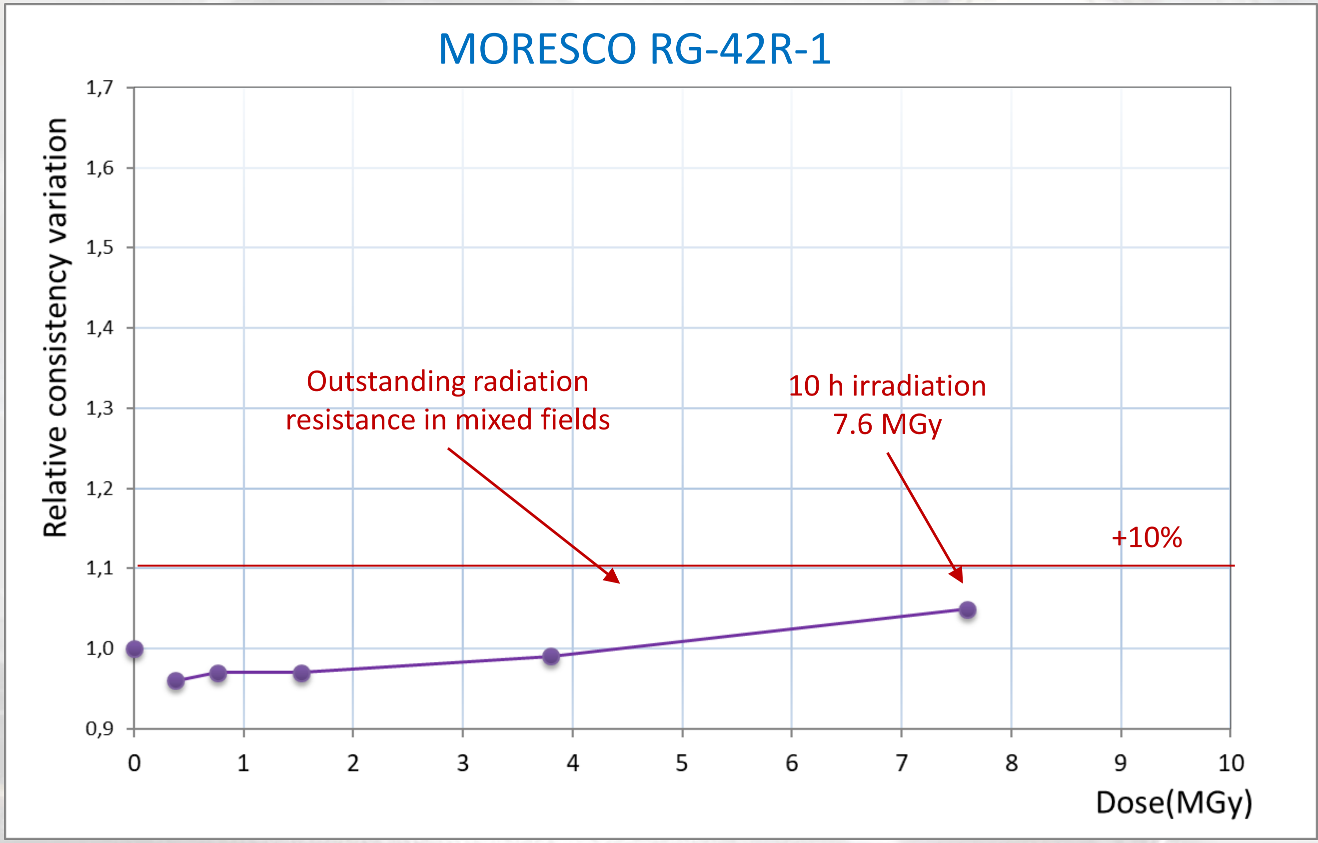
- Priority for ESS
- Mineral oil + Polyurea: NLGI class 2
- Declared radiation resistance: Not declared
- Outstanding radiation resistance in mixed fields up to 8.9 MGy

Experimental results on greases/8

Evolution of
Consistency with dose



Slight color change



Outstanding radiation resistance in mixed fields

10 h irradiation 7.6 MGy

- Interesting for ESS
- Polyphenylether + polycarbonate thickener: NLGI class 1
- Declared radiation resistance: referred to the base oil one (15 MGy) measured in gamma fields. Grease not tested.
- Outstanding radiation resistance in mixed fields up to 7.6 MGy

Experimental results on MORESCO oil/1

Evolution of base oil viscosity with dose

0.0 MGy

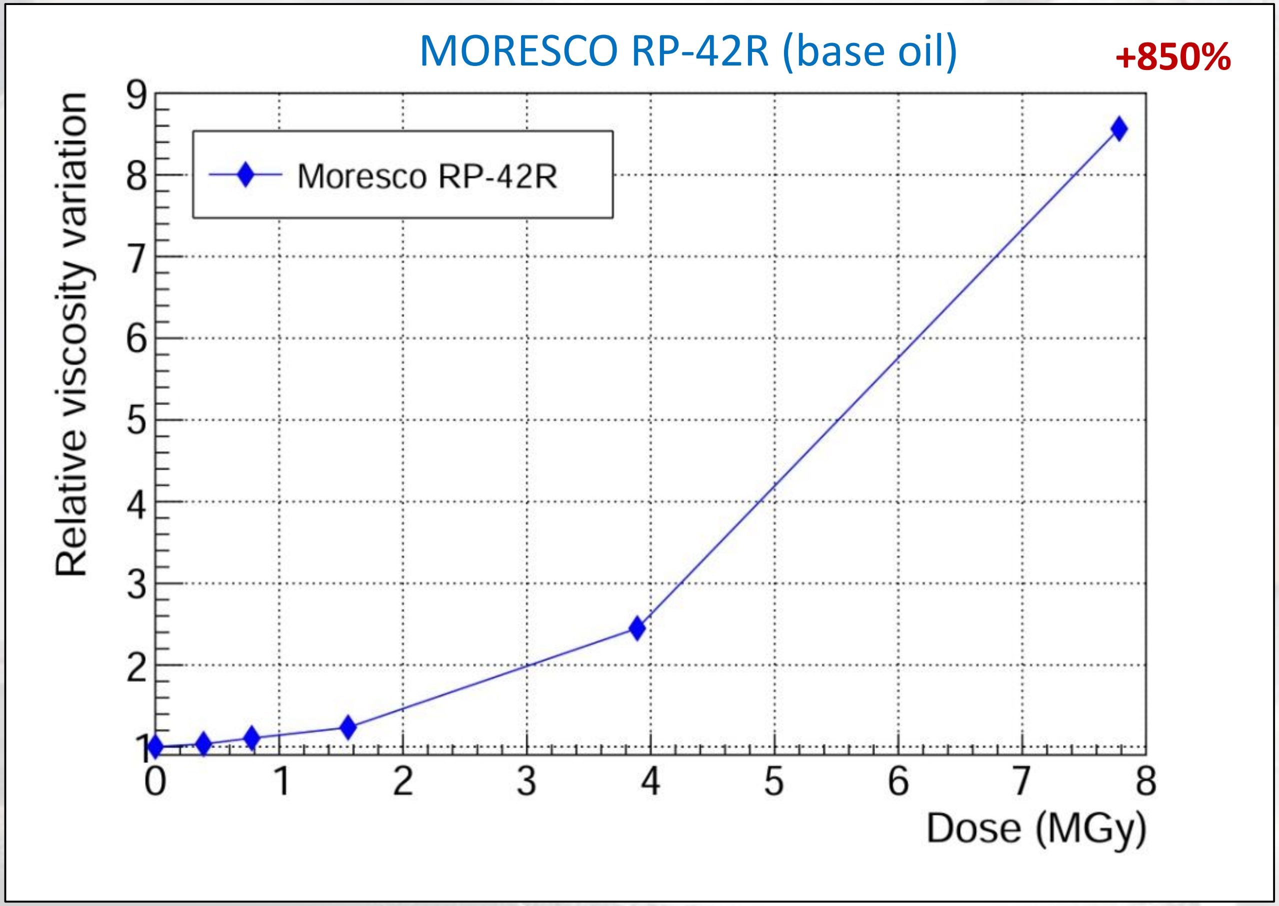
0.78 MGy

1.56 MGy

7.78 MGy

MORESCO RP-42R

Progressive color change



MORESCO RP-42R (base oil)

+850%

Intriguing results

- Very large viscosity increase of the base oil
- Declared radiation resistance of the base oil: up to 15 MGy in gamma fields
- Declared oil (and grease) end-point: 100% viscosity increase
- Is grease stability directly associated to oil stability? The role of the thickener.

Experimental results on MORESCO oil/2

Evolution of base oil viscosity with dose

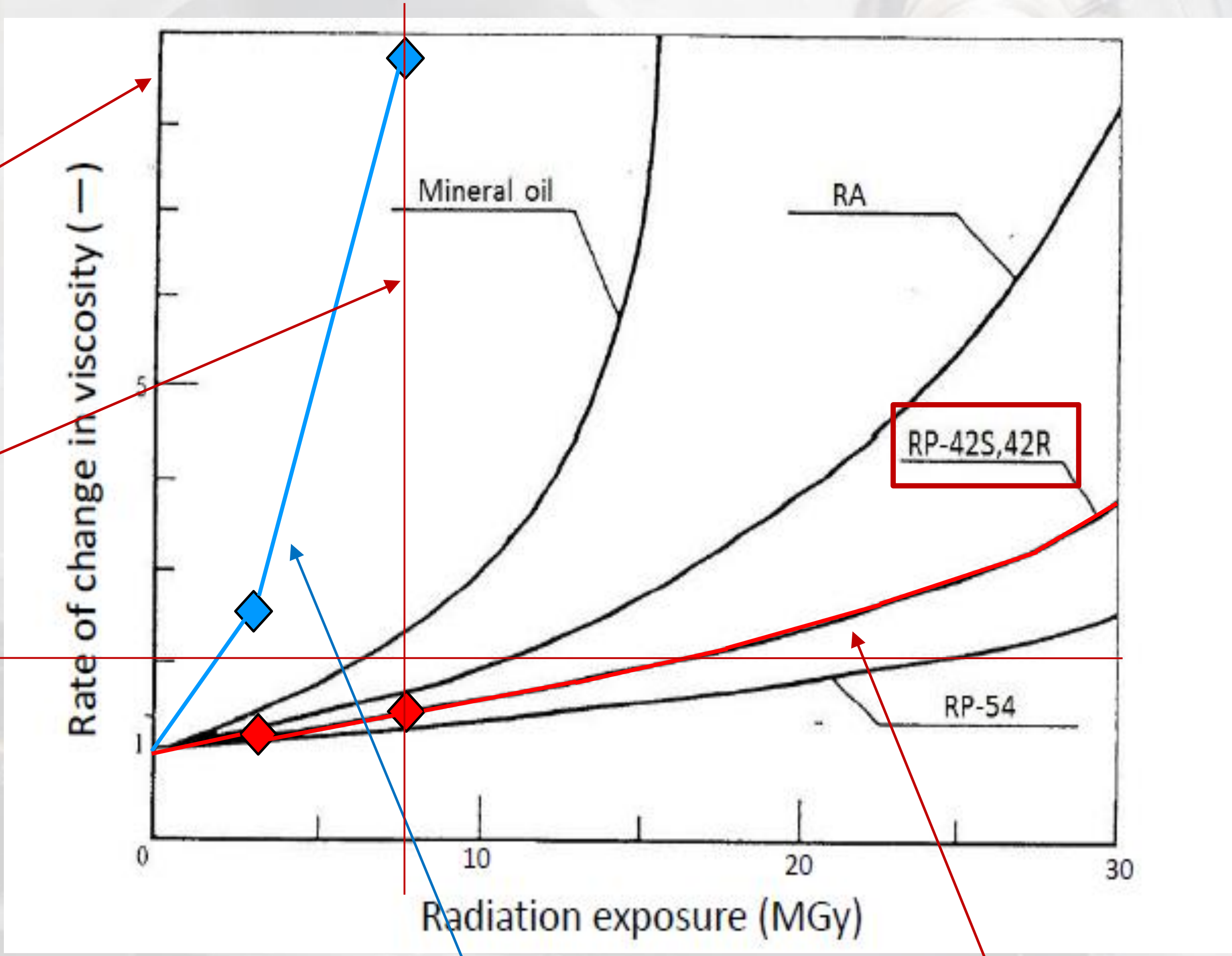
MORESCO data sheet

Intriguing results

Much larger oil damage in mixed fields

Producer oil end-point 100% viscosity increase

Different dose thresholds:
Gamma fields: 15 MGy
Mixed fields: < 3 MGy



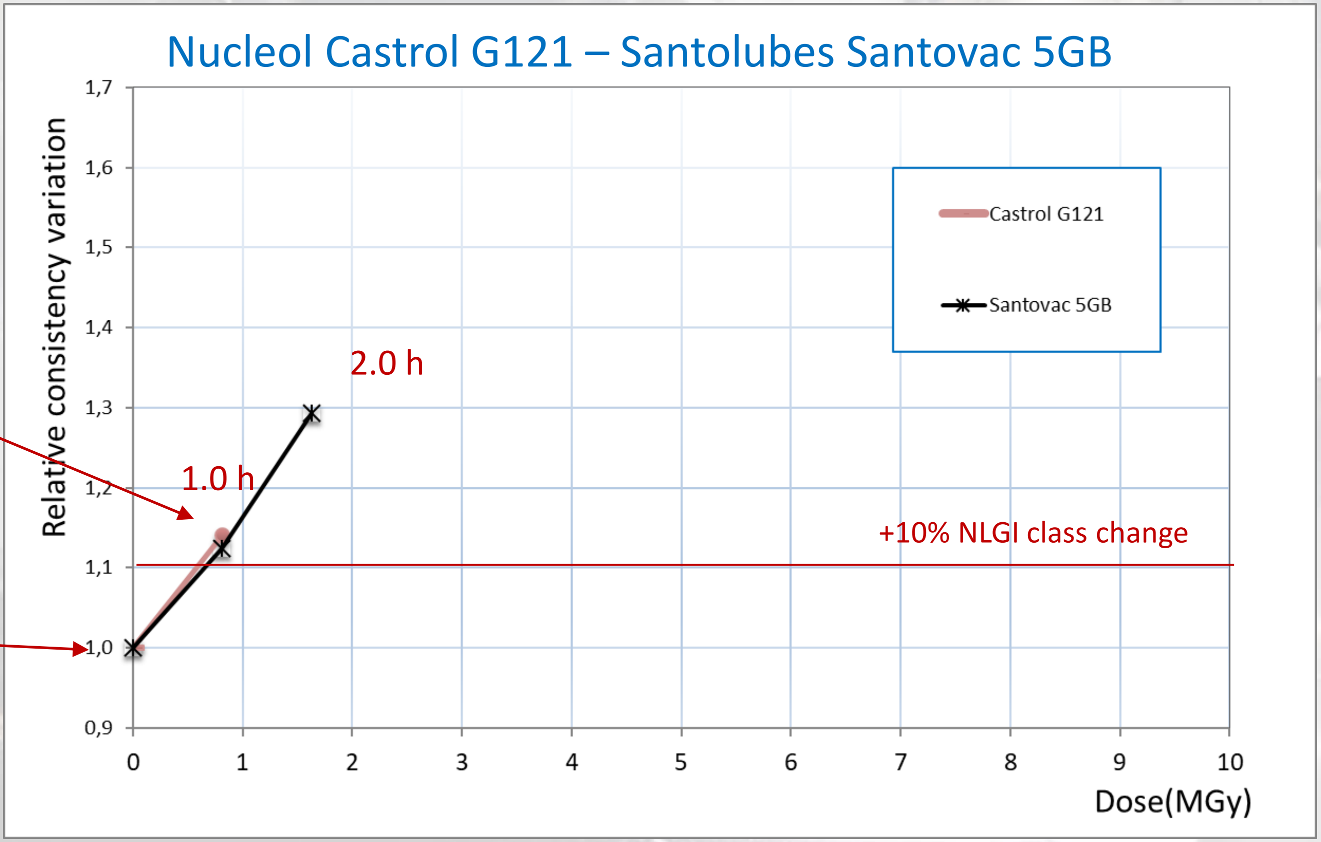
Neutron dose 66%
Gamma dose 34%

Gamma dose

Experimental results on greases (CERN products: S. Marzari)

Evolution of Consistency with dose

Santolubes Santovac 5GB



Grease darkening

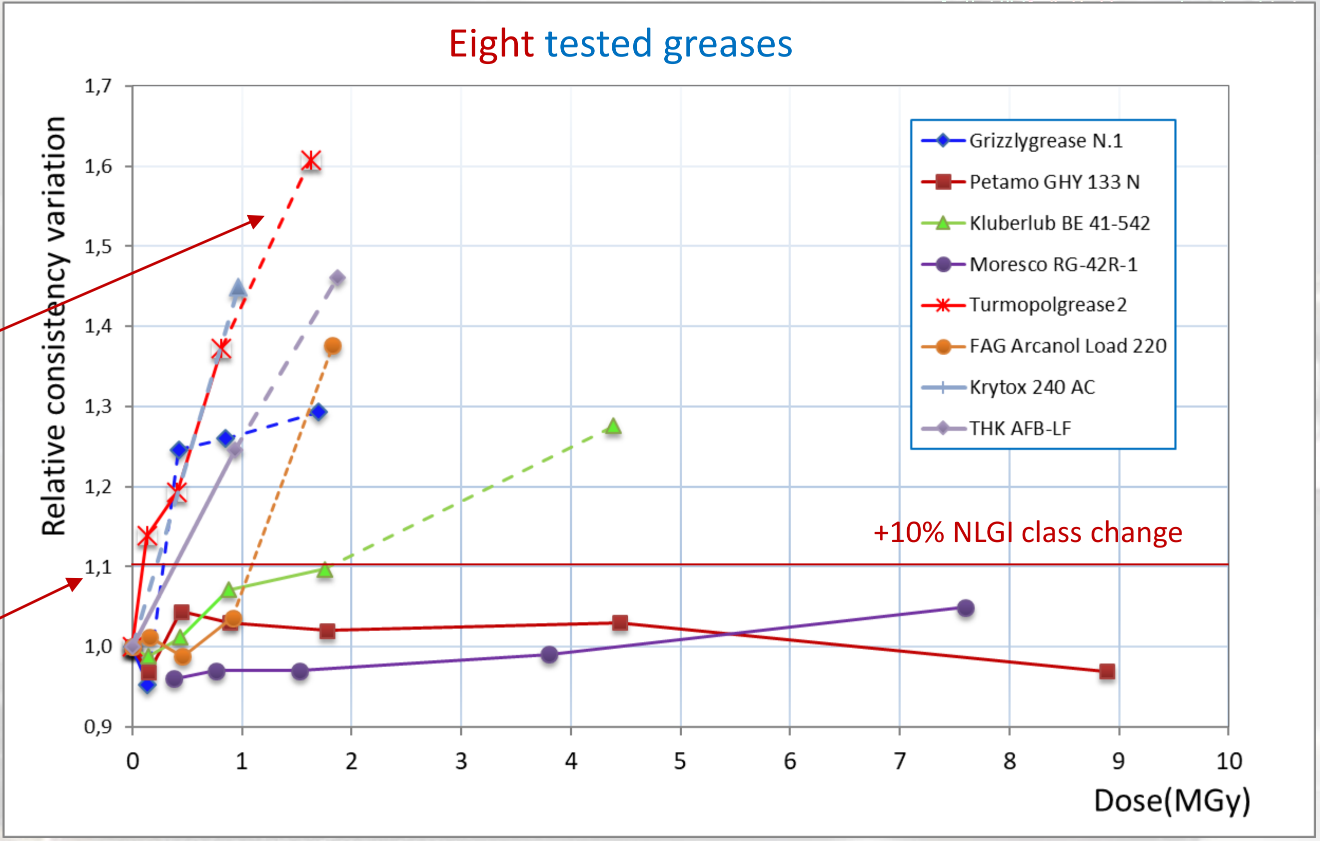
- Only few points, very little quantities provided
- Declared radiation resistance: generic declaration.
- Nucleol Castrol G121 changes NLGI class from 1 to 0 at about 0.8 MGy
- Santolube Santovac 5GB changes class from 3 to 2 at about 0.8 MGy

Summary of user end-points for greases

Evolution of Consistency with dose

Fluidization

NLGI class change



Better performance

Grease	NLGI class change	Fluidization	Radiation resistance declaration
Petamo	Stable up to 8.9 MGy	No fluid point up to 8.9 MGy	No declarations
MORESCO	Stable up to 7.6 MGy	No fluid point up to 7.6 MGy	Up to 15 MGy (base oil)
Kluberlub BE 41-542	> 1.75 MGy	> 1.75 MGy	No declarations
FAG Schaeffler Arcanol Load 200	> 0.91 MGy	> 0.81 MGy	No declarations
Krytox 240 AC	about 0.5 MGy	about 0.97 MGy	Rad resistant (0.2 -1.0 MGy)
THK AFB-LF	> 0.47 MGy	About 1.88 MGy	No declarations
Grizzly Grease n. 1 Lubcon	> 0.42 MGy	> 0.42 MGy	Up to 1.2 MGy
Turmopolgrease 2	about > 0.13 MGy	> 0.81 MGy	No declarations

Conclusions on lubricating greases

- A protocol for irradiation and testing of lubricating oils and greases in reactor mixed fields has been successfully developed
- Products declared as radiation resistant in gamma fields experience unexpected degradation in mixed fields.
- Irradiation parameters play a relevant role in assessing radiation damage. Radiation damage definition cannot be based only on total absorbed dose with pure gamma field.
- According to the literature, the grease radiation stability should be predominantly determined by the base oil chemical composition.
- The radiation induced fluidization of many products in mixed fields suggests that the thickener is the most sensitive grease component.
- As for elastomers, predictive capability of the obtained results may depend on many factors: radiation fields, dose rate, oxygen penetration, temperature, synergetic effects. Anyway obtained results may provide a guideline on a comparative base.

Overall conclusions and prospects

- Protocols for irradiation and testing of polymeric materials in reactor mixed fields have been successfully developed and validated.
- Experimental data on elastomers for O-ring construction and lubricating products useful for the construction of the SPES facility and the ESS project have been obtained.
- Intriguing evidence for a difference in radiation damage effects by pure gamma and mixed neutrons and gamma fields have been obtained.
- The generally adopted definition of radiation resistance for organic materials should be reconsidered.
- Apart for the significance for the target applications, the present work is providing original samples of data on radiation damage of common organic materials in conditions different from the ones usually employed.
- Next groups of materials to be considered in the study are signal cable insulators, power wire jackets (a lot of data from CERN reports) and optical fibers.