

Matteo Ferrari EN/STI/TCD



Radiation resistant lubricants Meeting • CERN • Oct 22st, 2019

NEUTRON AND GAMMA IRRADIATION TEST CAMPAIGNS ON GREASES AND OILS

Recent results

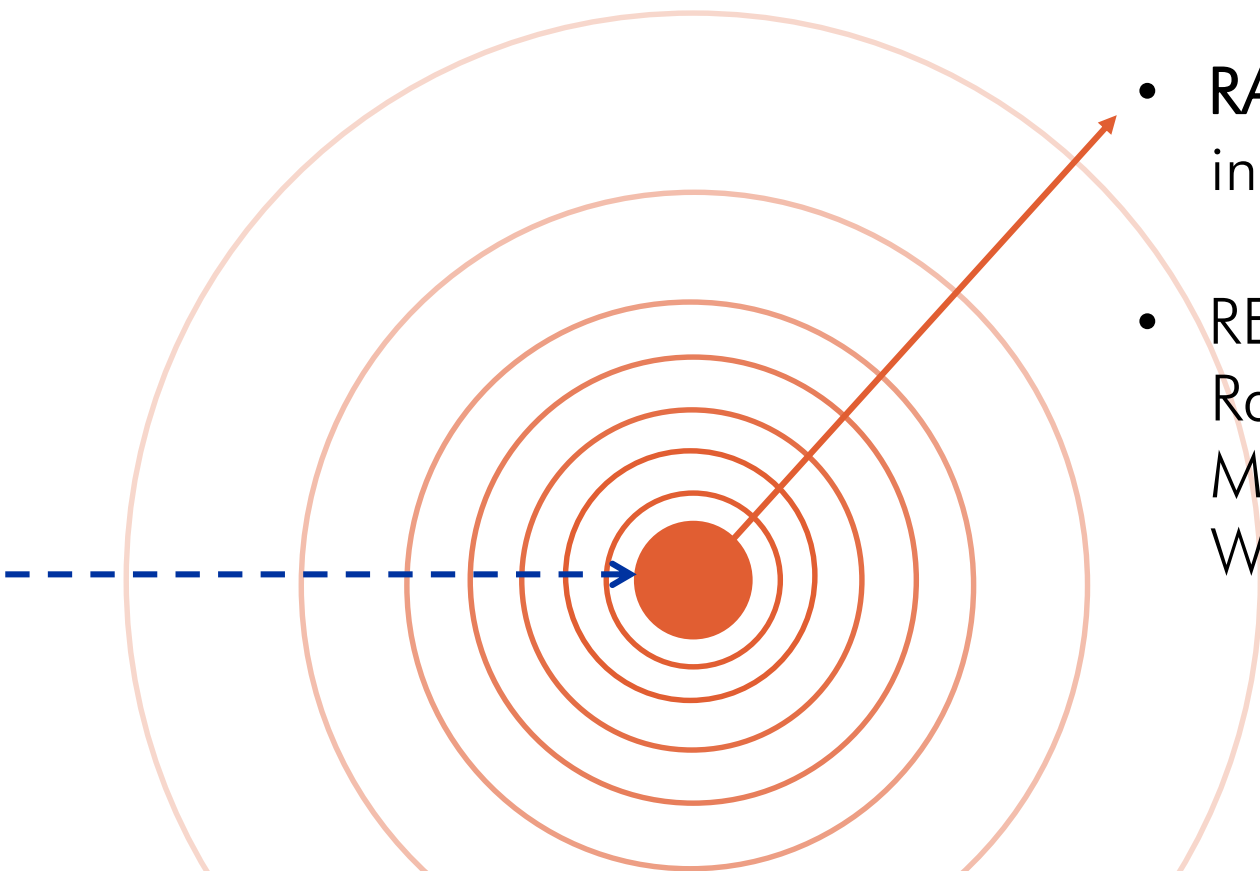


MORESCO
MORESCO Corporation

INTRODUCTION



HIGH-POWER FACILITIES

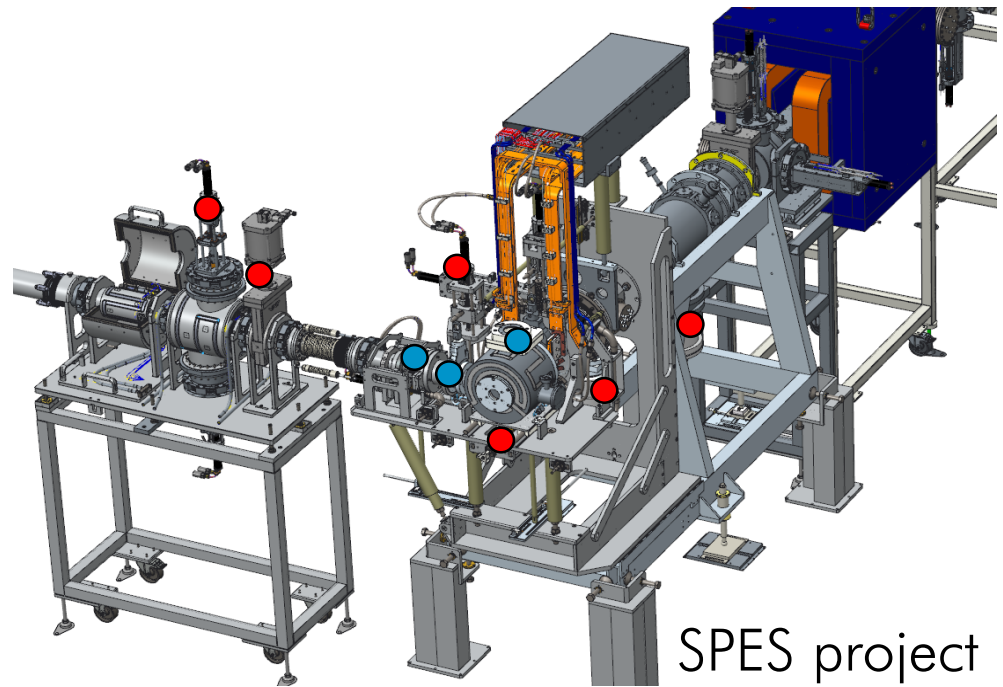
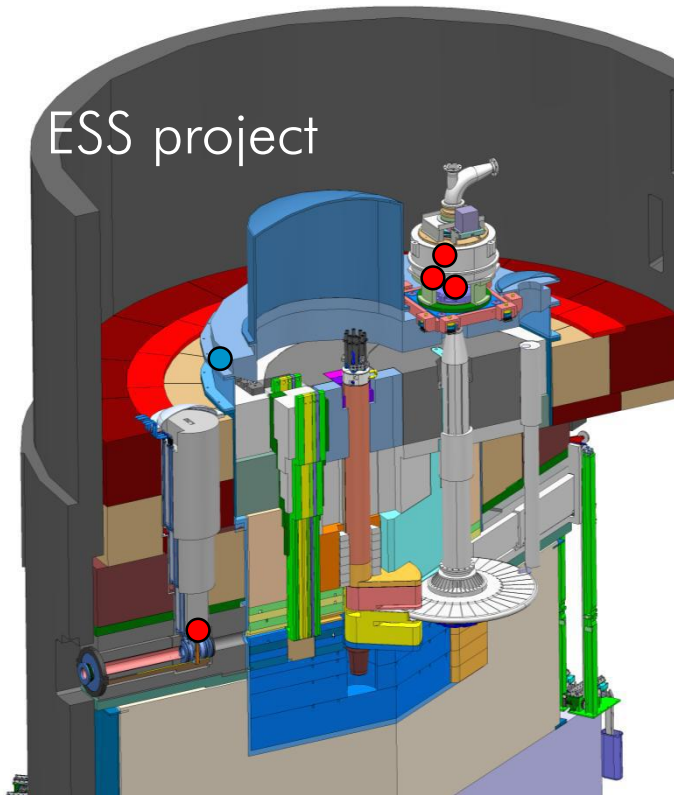


- RADIATION DAMAGE
in MIXED FIELDS
- RESIDUAL ACTIVATION
Radiation protection
Maintenance
Waste disposal

CRITICAL APPLICATIONS FOR POLYMERS

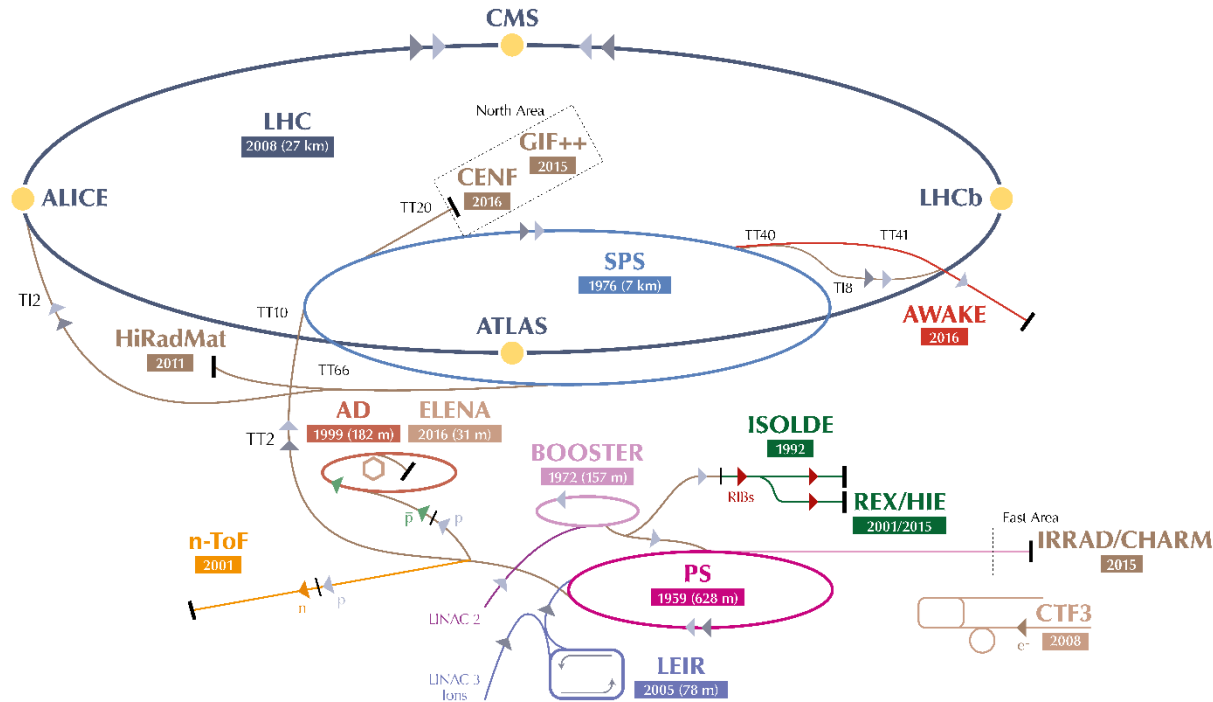
POLYMERS IN FACILITIES UNDER CONSTRUCTION

- O-rings
- Lubricants



NECESSARY BUT RADIATION-SENSITIVE

CERN ACCELERATOR COMPLEX



▶ p (protons) ▶ ions ▶ RIBs (Radioactive Ion Beams) ▶ n (neutrons) ▶ \bar{p} (antiprotons) ▶ e⁻ (electrons)

LHC Large Hadron Collider SPS Super Proton Synchrotron PS Proton Synchrotron AD Antiproton Decelerator CTF3 Clic Test Facility
 AWAKE Advanced WAKEfield Experiment ISOLDE Isotope Separator OnLine REX/HIE Radioactive Experiment/High Intensity and Energy ISOLDE
 LEIR Low Energy Ion Ring LINAC LINear ACcelerator n-ToF Neutrons Time Of Flight HiRadMat High-Radiation to Materials
 CHARM Cern High energy AcceleRator Mixed field facility IRRAD proton IRRADiation facility GIF++ Gamma Irradiation Facility
 CENF CERN Neutrino platform

CERN Accelerator Complex © CERN, updated January 2017

MAIN CONCERNS

**BEST PRODUCTS
FOR SPECIFIC
APPLICATIONS**

Design

**UNDERSTANDING OF
RAD MECHANISMS**

Science



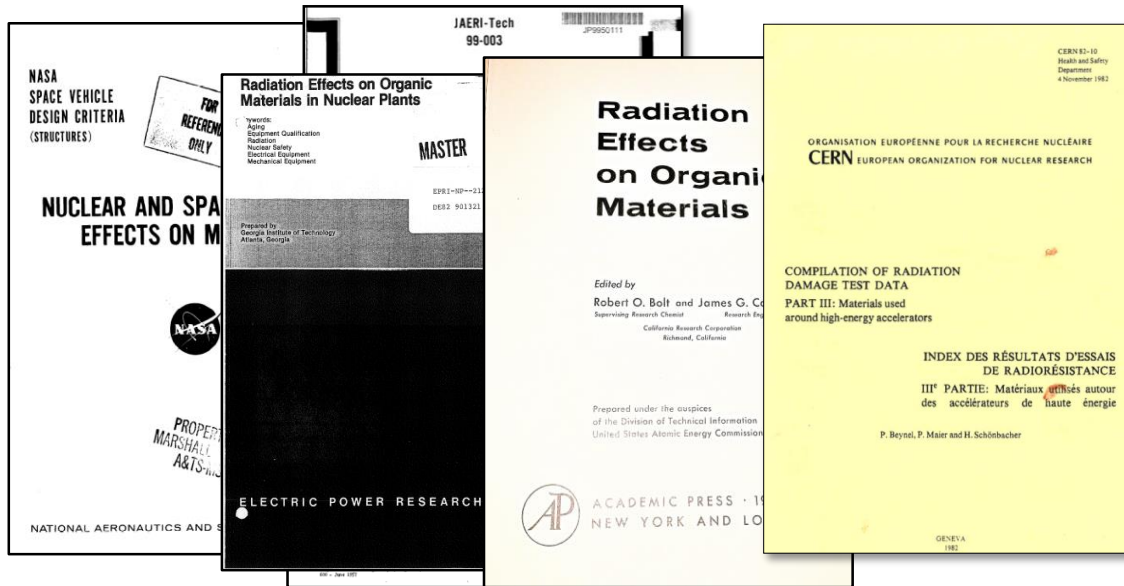
**PREDICITON
OF REAL-LIFE
DEGRADATION**

Maintenance
Prevent failure

**FOLLOW-UP ON
USED PRODUCTS**

Know-how

LIMITS OF THE LITERATURE



- Mostly gamma
- Old data, obsolete products
- Very general tests
- Assumptions
- Lack of understanding
- Lack of thresholds
- More challenging conditions

'Equal dose – equal damage' ?

FEW NEUTRON/MIXED FIELD DATA

SELECTION OF COMMERCIAL PRODUCTS



technical requirements

- Composition
- Temperature
- Atmosphere
- Extreme Pressure
- Compatibility
- Vacuum
- Mechanical features
- **Radiation tolerance**
- Product availability
- Price
- Producer reliability

OFTEN UNKNOWN
NO GENERAL VALIDITY

TECHNICAL SPECIFICATIONS AT CERN

Equipment ID	Asset number			
	Number of identical items	1/2	1/2	1/2
	Responsibility	Research/Service Dept.	Dept. Research/Engineering	Dept. Maintenance
	Location	16.01.010	16.01.010	16.01.010
	Type of device	16.01.010	16.01.010	16.01.010
	Manufacturer (Mfg)	16.01.010	16.01.010	16.01.010
	Reference (Ref)	16.01.010	16.01.010	16.01.010
	Frequency of use	16.01.010	16.01.010	16.01.010
	Level of operational condition	16.01.010	16.01.010	16.01.010
	Mechanical load	16.01.010	16.01.010	16.01.010
Operation details	Operational System	16.01.010	16.01.010	16.01.010
	Failure Impact	16.01.010	16.01.010	16.01.010
	Availability	16.01.010	16.01.010	16.01.010
	At which frequency	16.01.010	16.01.010	16.01.010
	Step 1	16.01.010	16.01.010	16.01.010
	Step 2	16.01.010	16.01.010	16.01.010
	Control	16.01.010	16.01.010	16.01.010
	Cost	16.01.010	16.01.010	16.01.010
	Can we participate in future?	16.01.010	16.01.010	16.01.010
	History	16.01.010	16.01.010	16.01.010
Equipment	Requirements	16.01.010	16.01.010	16.01.010
	At which frequency	16.01.010	16.01.010	16.01.010
	Step 1	16.01.010	16.01.010	16.01.010
	Step 2	16.01.010	16.01.010	16.01.010
	Control	16.01.010	16.01.010	16.01.010
	Cost	16.01.010	16.01.010	16.01.010
	Can we participate in future?	16.01.010	16.01.010	16.01.010
	History	16.01.010	16.01.010	16.01.010
	Requirements - vehicle assembly	16.01.010	16.01.010	16.01.010
	Can we participate in future?	16.01.010	16.01.010	16.01.010
Mechanics	Requirements - vehicle assembly	16.01.010	16.01.010	16.01.010
	Can we participate in future?	16.01.010	16.01.010	16.01.010
	History	16.01.010	16.01.010	16.01.010
	Requirements - vehicle assembly	16.01.010	16.01.010	16.01.010
	Can we participate in future?	16.01.010	16.01.010	16.01.010
	History	16.01.010	16.01.010	16.01.010
	Requirements - vehicle assembly	16.01.010	16.01.010	16.01.010
	Can we participate in future?	16.01.010	16.01.010	16.01.010
	History	16.01.010	16.01.010	16.01.010
	Requirements - vehicle assembly	16.01.010	16.01.010	16.01.010
Lubricant compatibility	Requirements - vehicle assembly	16.01.010	16.01.010	16.01.010
	Can we participate in future?	16.01.010	16.01.010	16.01.010
	History	16.01.010	16.01.010	16.01.010
	Requirements - vehicle assembly	16.01.010	16.01.010	16.01.010
	Can we participate in future?	16.01.010	16.01.010	16.01.010
	History	16.01.010	16.01.010	16.01.010
	Requirements - vehicle assembly	16.01.010	16.01.010	16.01.010
	Can we participate in future?	16.01.010	16.01.010	16.01.010
	History	16.01.010	16.01.010	16.01.010
	Requirements - vehicle assembly	16.01.010	16.01.010	16.01.010

EQUIPMENT

- Equipment ID
- Operation details
- Maintenance
- Existing components/follow up

ENVIRONMENT

- Radiation field/rate
- Atmosphere
- Temperature
- Materials

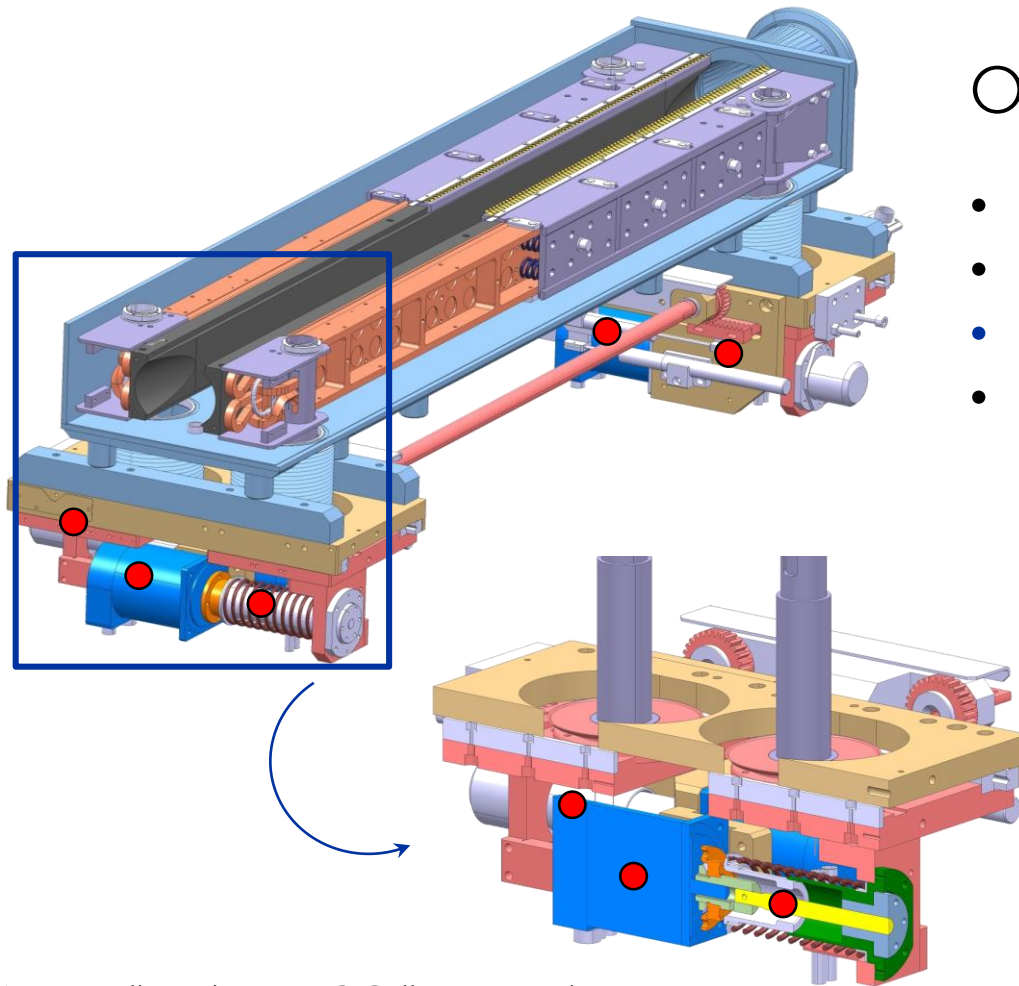
LUBRICANT

- Consistency/viscosity
- Requirements on composition
- Quantity needed
- Previous products?

NEW ONGOING STUDY

By Dominika Senajova

COLLIMATORS LUBRICATION



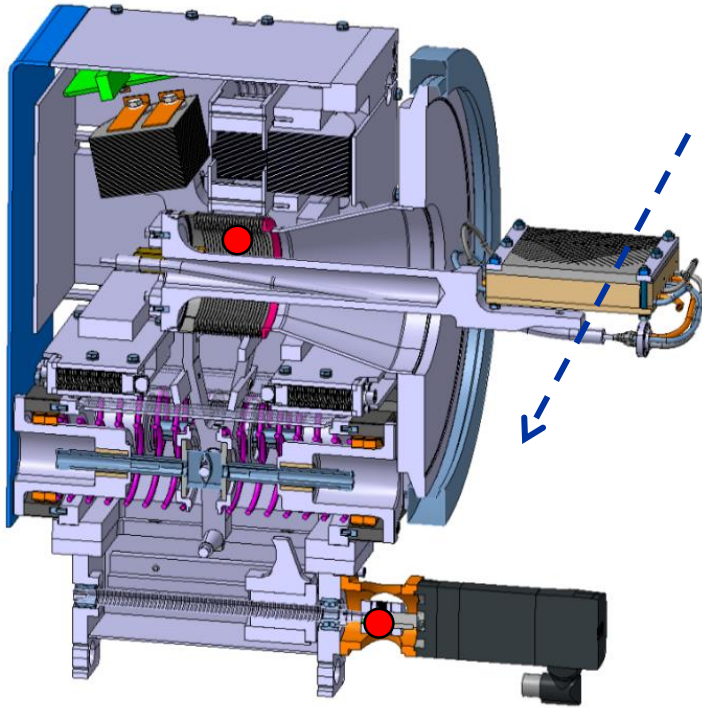
OPERATION CONDITONS

- ~130 collimators - LHC
- 4 roller screws per collimator
- ~10 MGy dose
- limited possible maintenance

**CRITICAL
DOSE LEVEL**

A. Bertarelli et al., HL-LHC Collimators and High Temperature Materials, HiLumi LHC Goes To Industry Workshop (2015)

PS DUMP LUBRICATION



F-X. Nuiry et al., Designing and prototyping of the CERN Proton Synchrotron Internal Dump in the Framework of the LHC Injectors Upgrade Project, 7th High Power Targetry Workshop (2018)

OPERATION CONDITONS

- High energy protons
- $\pm 6^\circ$ rotation
- 250 000 cycles/yr
- 1 MGy dose

CASE STUDIES

- Grease evolution
- Endpoint
- Monitoring
- Failure scenarios
- Maintenance
- Follow up

IRRADIATION AND TESTING METHODOLOGIES



RADIATION DAMAGE STUDIES: A TIMELINE



RDS Radiation Damage Study - polymers



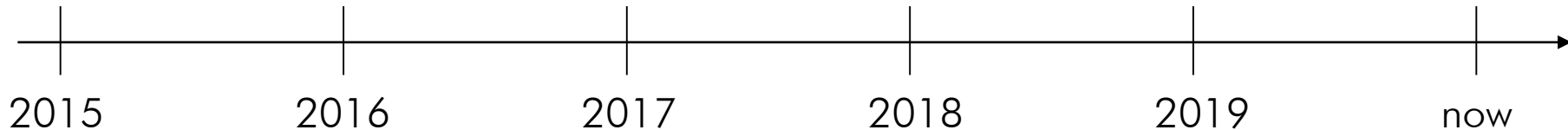
Phd - radiation damage on lubricants



case study - MORESCO grease/oil



R2M

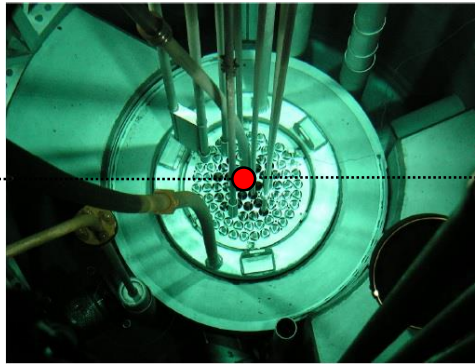


ELASTOMERIC O-RINGS

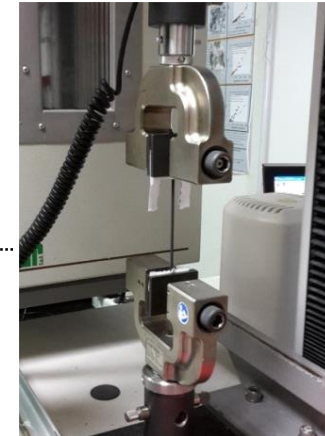
SAMPLES



IRRADIATION FACILITY



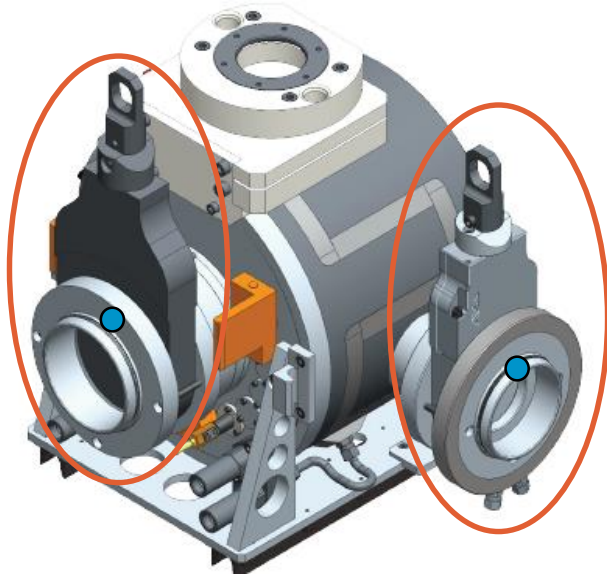
PIE



- A. Zenoni et al., **Radiation resistance of elastomeric O-rings in mixed neutron and gamma fields: Testing methodology and experimental results**, *Review of Scientific Instruments* 88, 113304 (2017) <https://doi.org/10.1063/1.5011035>
- M. Ferrari et al., **Degradation of EPDM and FPM elastomers irradiated at very high dose rates in mixed gamma and neutron fields**, *Polymer Engineering and Science* (2019) <https://doi.org/10.1002/pen.25249>



A REAL APPLICATION FOR AN EPDM O-RING

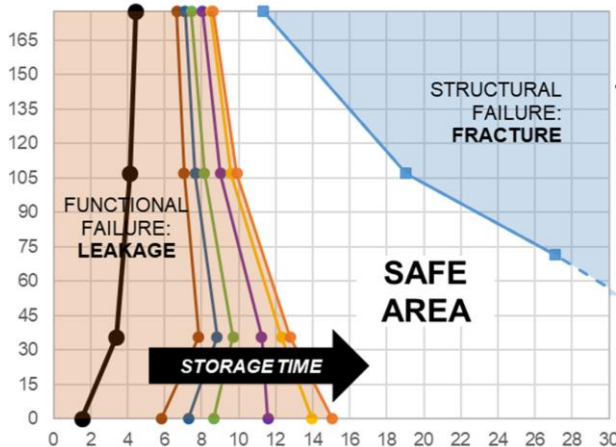


GATE VALVE - SPES facility

CONDITIONS

- Dynamic operation
- 0.7 MGy neutrons + gamma
- Vacuum
- About 85°C

SAFE USABILITY AREA



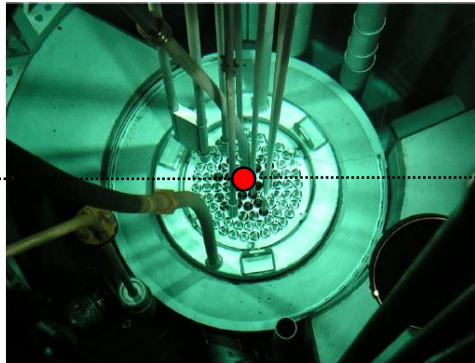
Battini, D. et al, *Experimental testing and numerical simulations for life prediction of gate valve O-rings exposed to mixed neutron and gamma fields*, *Materials and Design* vol. 156, 514-527 (2018)
<https://doi.org/10.1016/j.matdes.2018.07.020>

LUBRICANTS

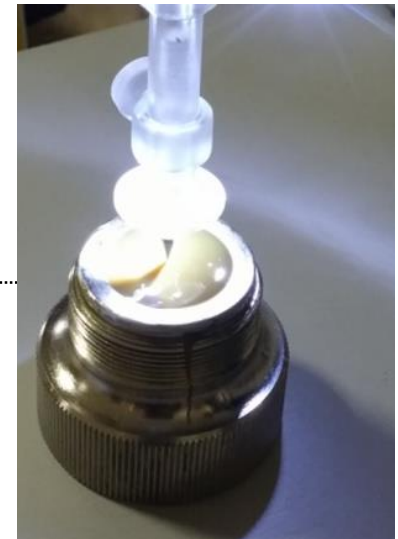
SAMPLES



IRRADIATION
FACILITY



PIE



M.Ferrari et al., **Experimental study of consistency degradation of different greases in mixed neutron and gamma radiation,**

Heliyon, Vol 5 Issue 9 e02489 (2019)

<https://doi.org/10.1016/j.heliyon.2019.e02489>

IRRADIATION CAMPAIGNS

- O-RINGS
4 products

2 experiments:



- O-RINGS
real application study

+ proton irr. at IRRAD

- O-RINGS
Dose rate effect

- GREASES
9 products

243 hours
83 samples
5 years

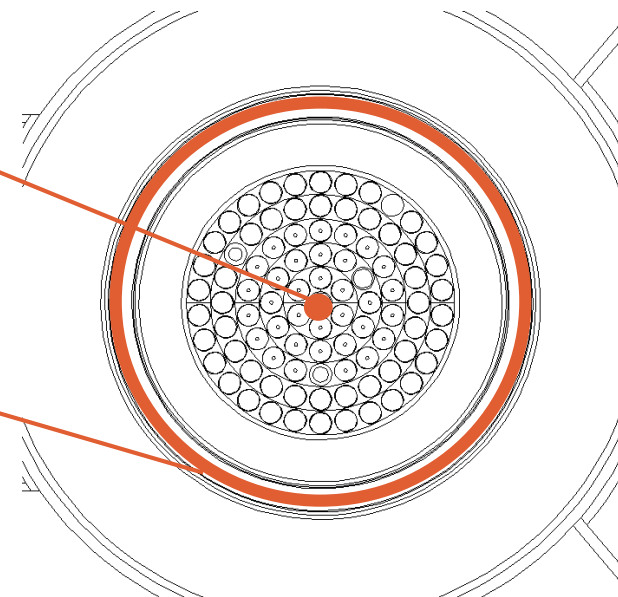
- LUBRICANT
1 oil



NEUTRON AND GAMMA REACTOR FACILITIES

In-core

Lateral



MIXED RAD FIELDS
HIGH FLEXIBILITY

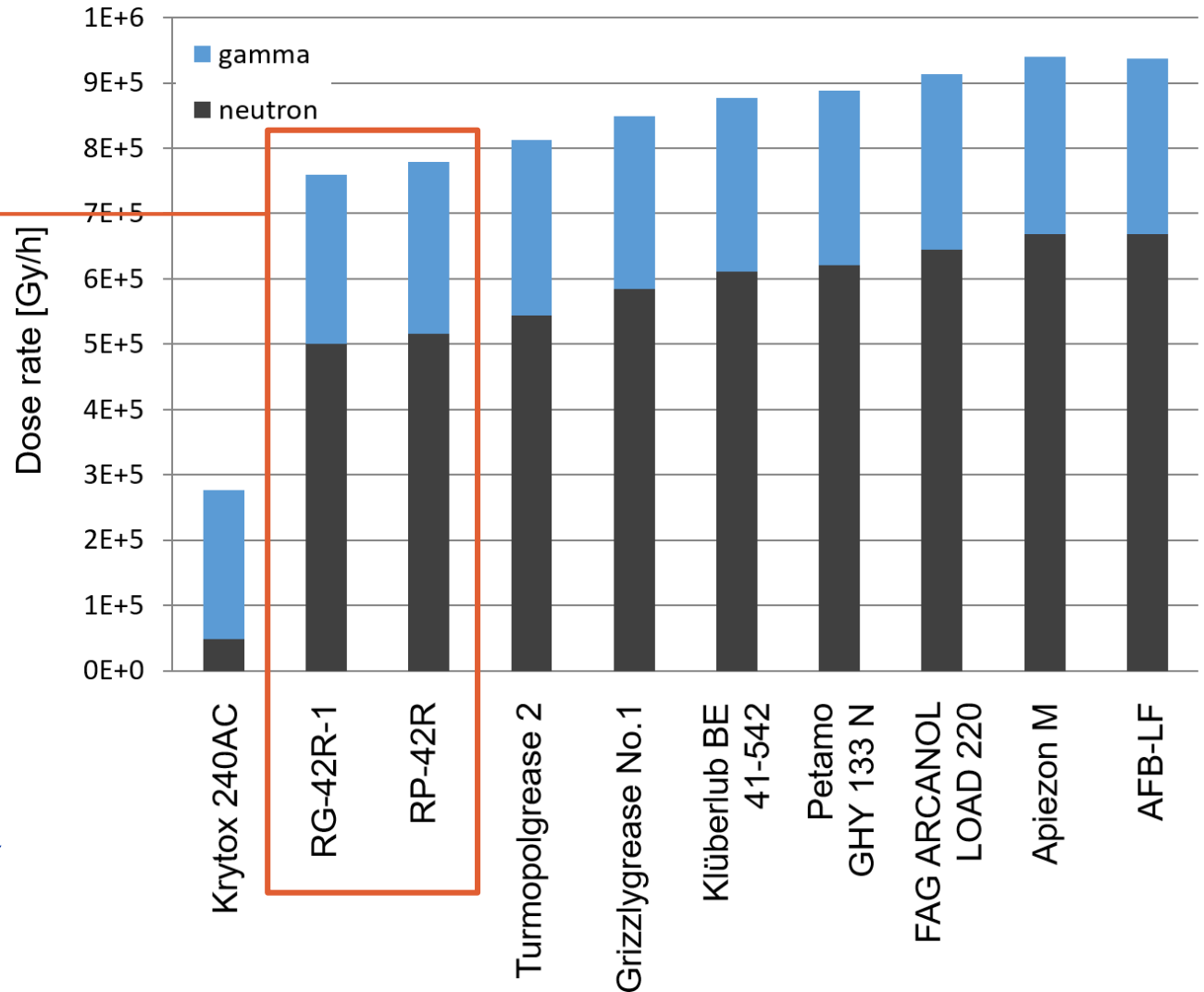
*Monte Carlo simulation
MCNPX reactor model*



TRIGA Mark II
RESEARCH REACTOR
Pavia, Italy

Irradiation and testing methodologies

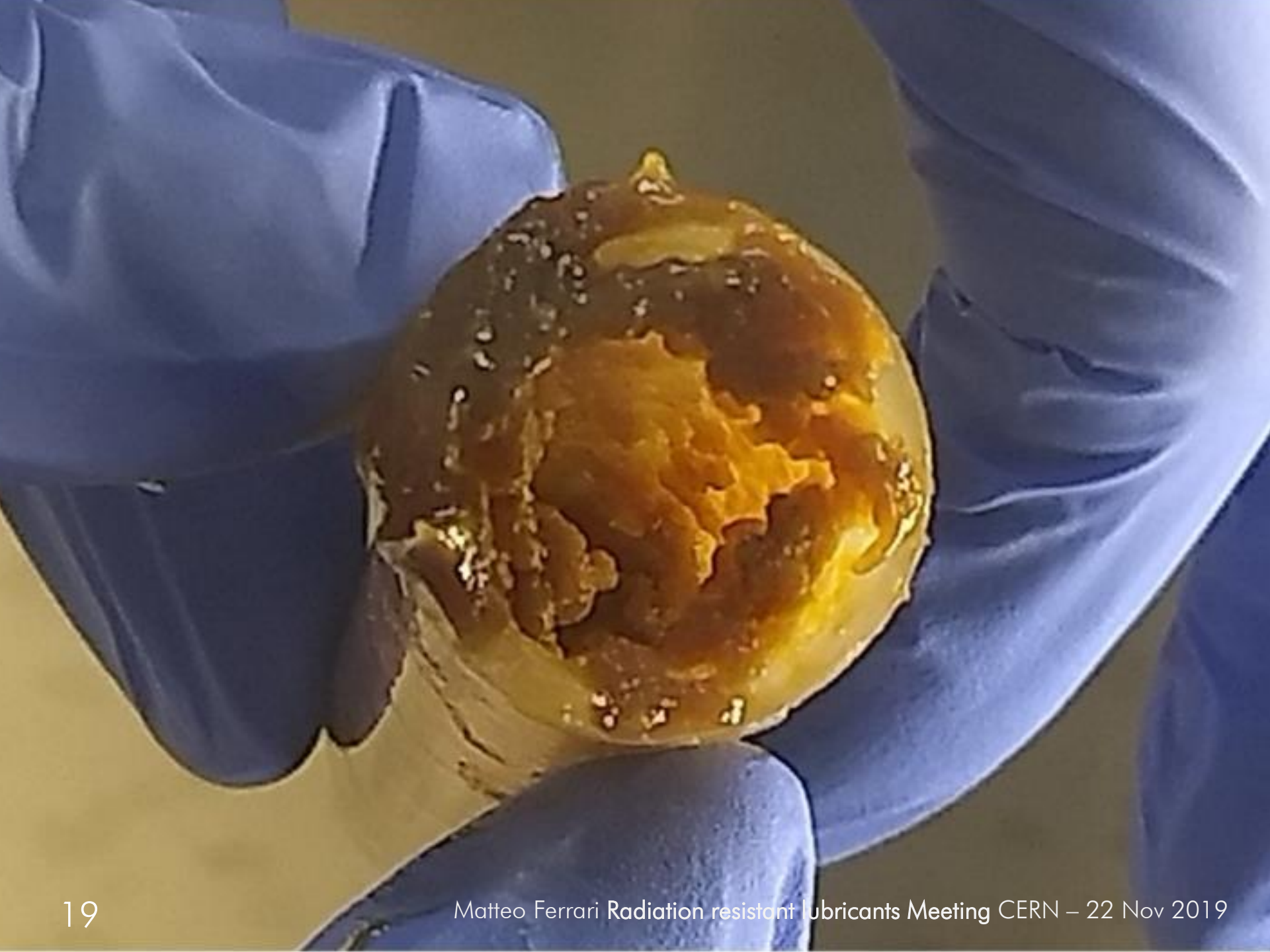
DOSIMETRY CALCULATIONS



COMPLEX
DOSIMETRY

RADIATION STUDY ON GREASES





GREASE CONSISTENCY

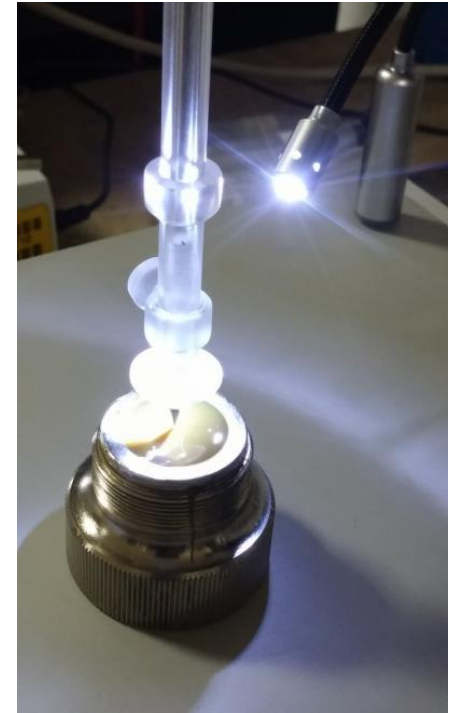
MULTI-PHASE:

- Oil
- Thickener
- Additives

CONSISTENCY:

ASTM D217-02

Semi-fluid



*'Grease is an exceptionally **complex** product incorporating a **high degree of technology** in chemistry, physics, rheology, tribology and the environmental sciences.'*

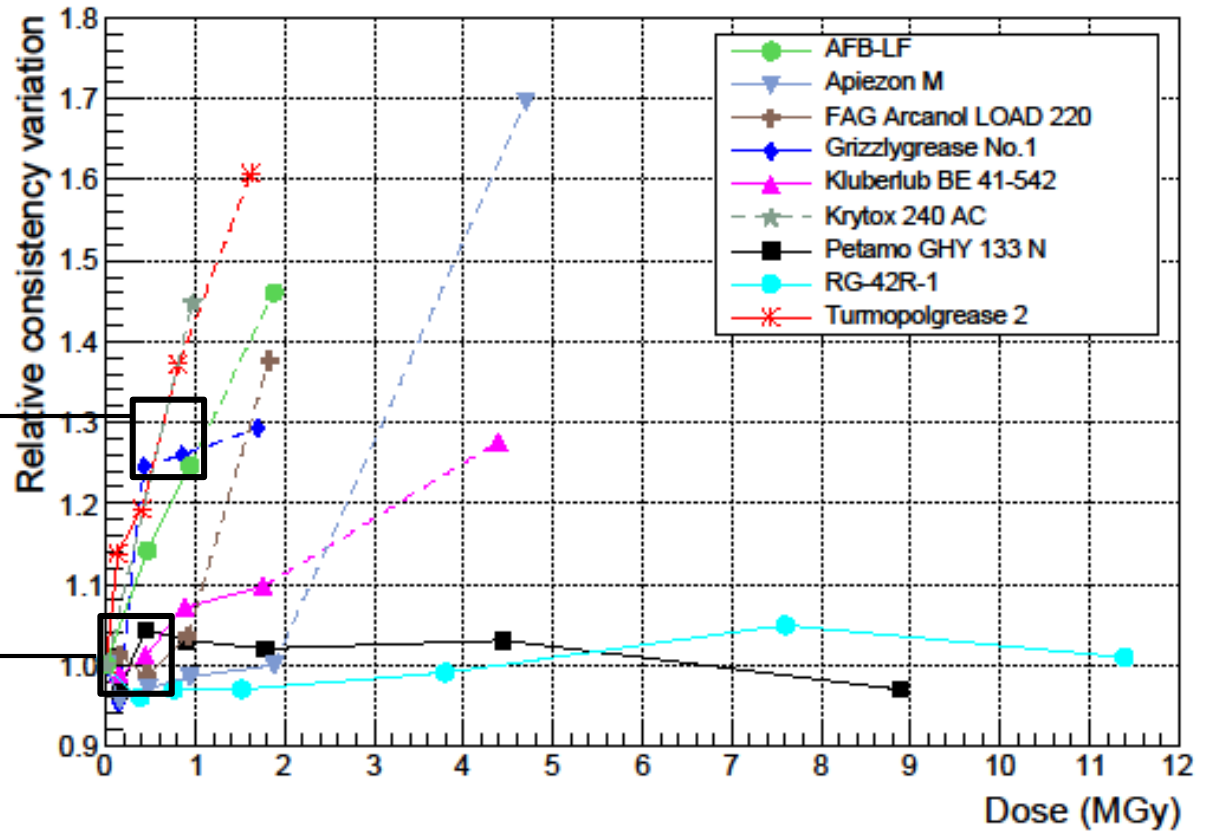
R. M. Mortier et al., Chemistry and Technology of Lubricants, 3rd edn., Springer (2010).

CONSISTENCY TEST: GRIZZLYGREASE No.1

0.45 MGy



0.15 MGy



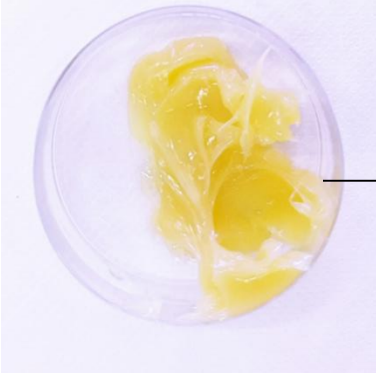
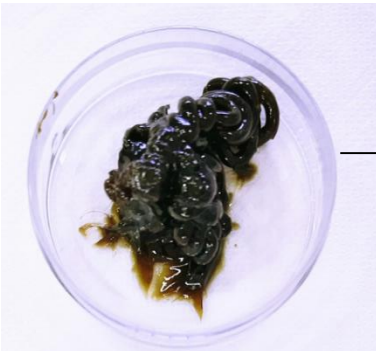
RADIATION-INDUCED FLUIDIZATION



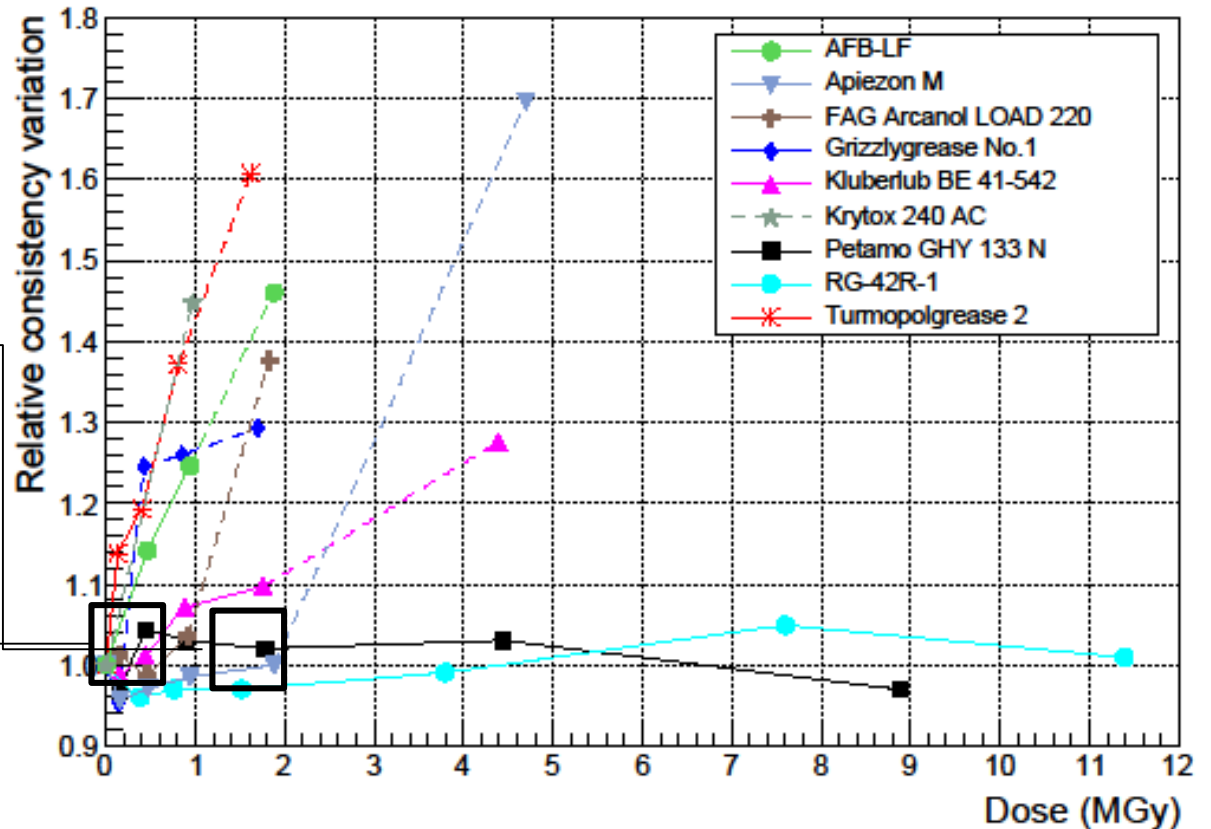
**DRIPPING
GREASE**

CONSISTENCY TEST: PETAMO GHY 133 N

0.40 MGy



0 MGy

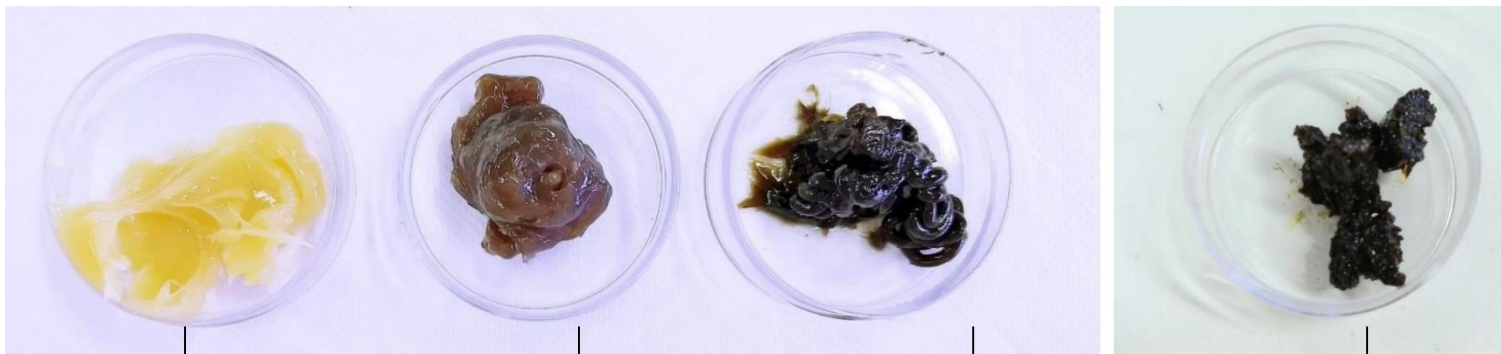


COLOR CHANGE BUT STABLE

DIFFERENT BEHAVIOURS AT HIGH DOSES



RG-42R-1



Petamo
GHY 133N

NON IRR

1 MGy

5 MGy

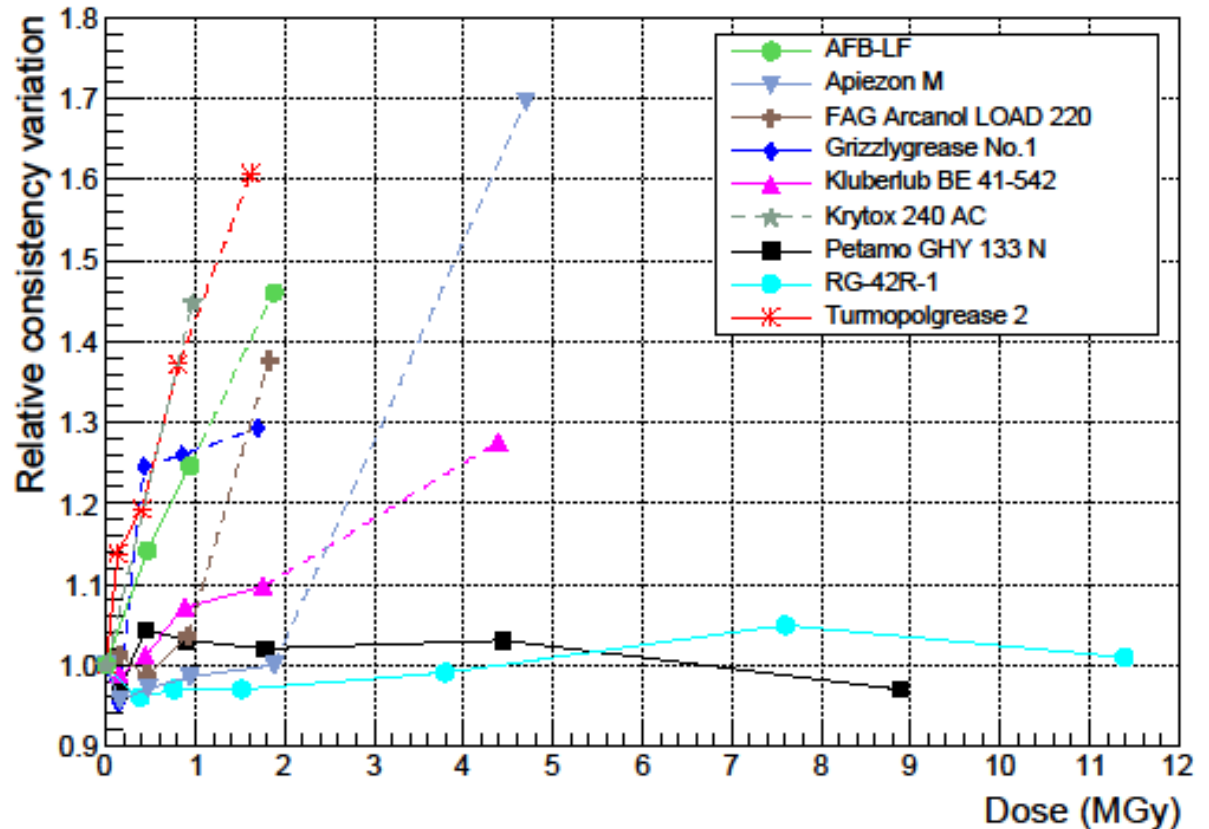
10 MGy

SOFTENING VS HARDENING

USABILITY ENDPOINTS FOR GREASES (1)

FLUID

Structural
endpoint



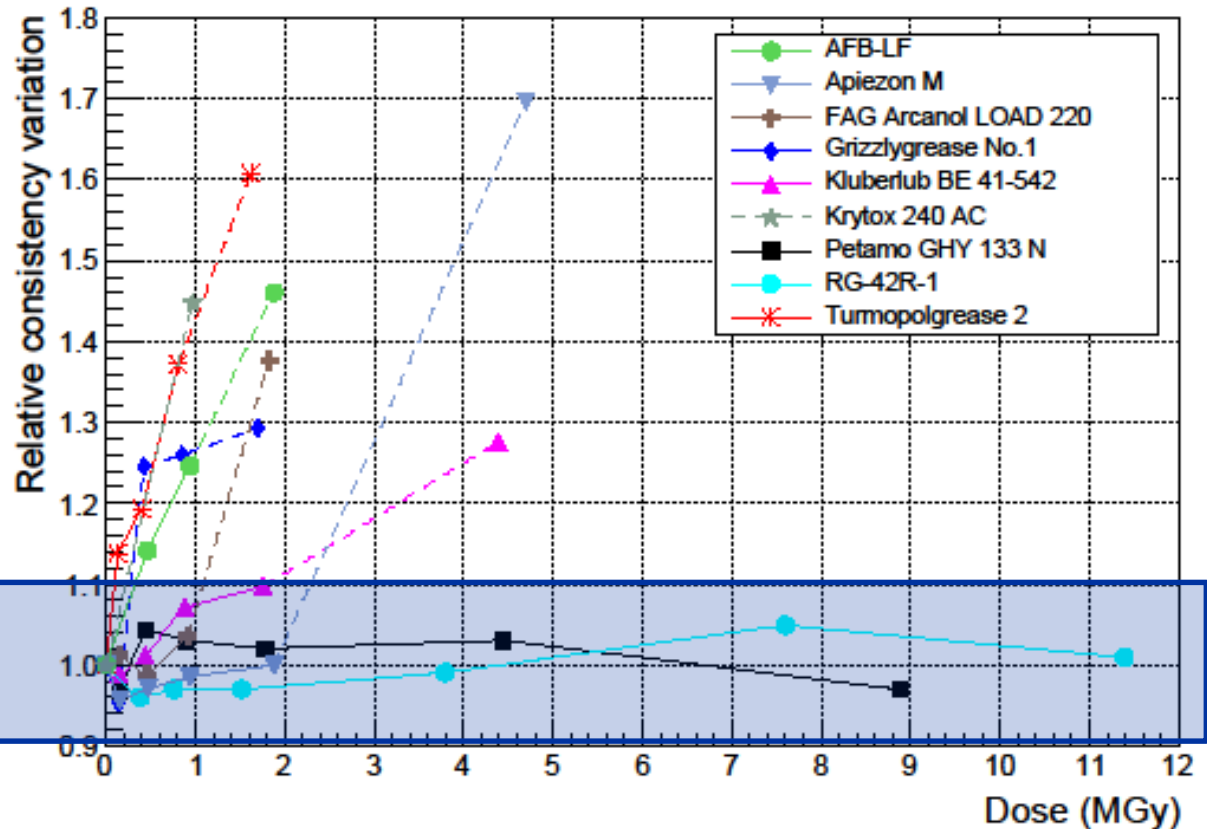
SEVERE MATERIAL MODIFICATION

USABILITY ENDPOINTS FOR GREASES (2)

CONSIST.
CLASS

Functional
endpoint

STABLE <10%



DOSE THRESHOLDS FOR USERS

RP-42R OIL: A CASE STUDY



A RESEARCH COLLABORATION

ESS project

Lund, Sweden

UniBs DIMI

Brescia, Italy

MORESCO

Kobe, Japan



OIL CHARACTERIZATION

PERFORMED TESTS:

- Kinetic viscosity
- Total acid number
- NMR spectra
- GPC gel permeation
- GC gas chromatography
- FT-IR spectroscopy



Kinetic viscosity



GC



Acid
number



MORESCO RP-42R: VISCOSITY



NON IRR



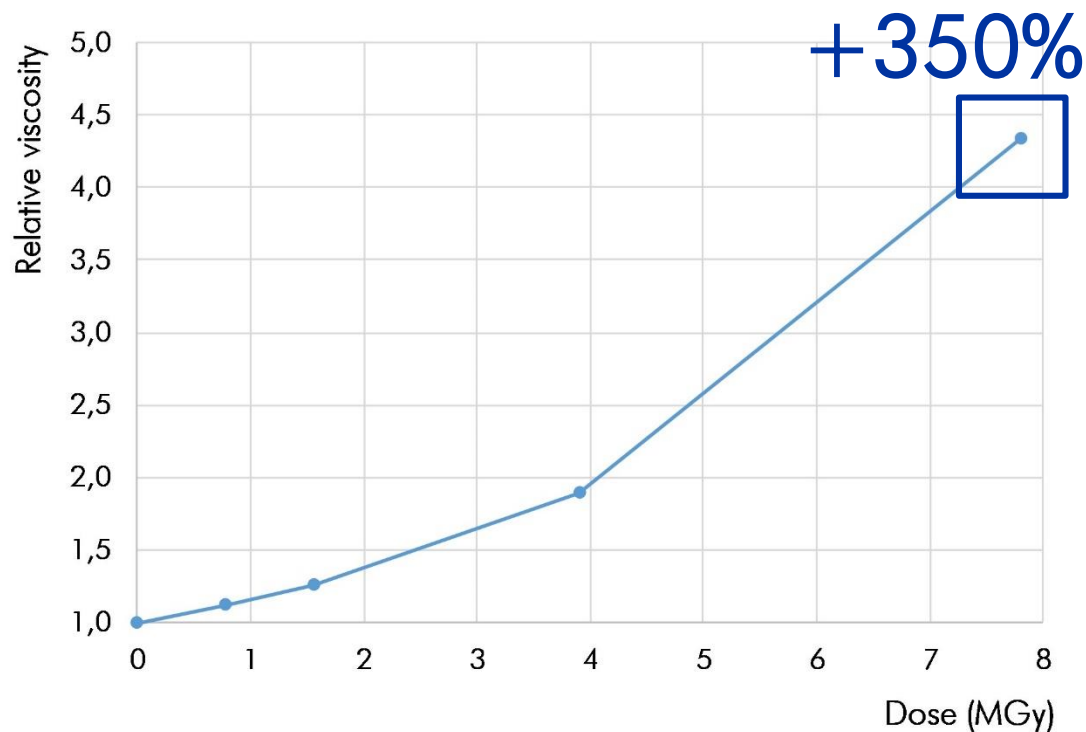
0.78 MGy



1.55 MGy



7.78 MGy

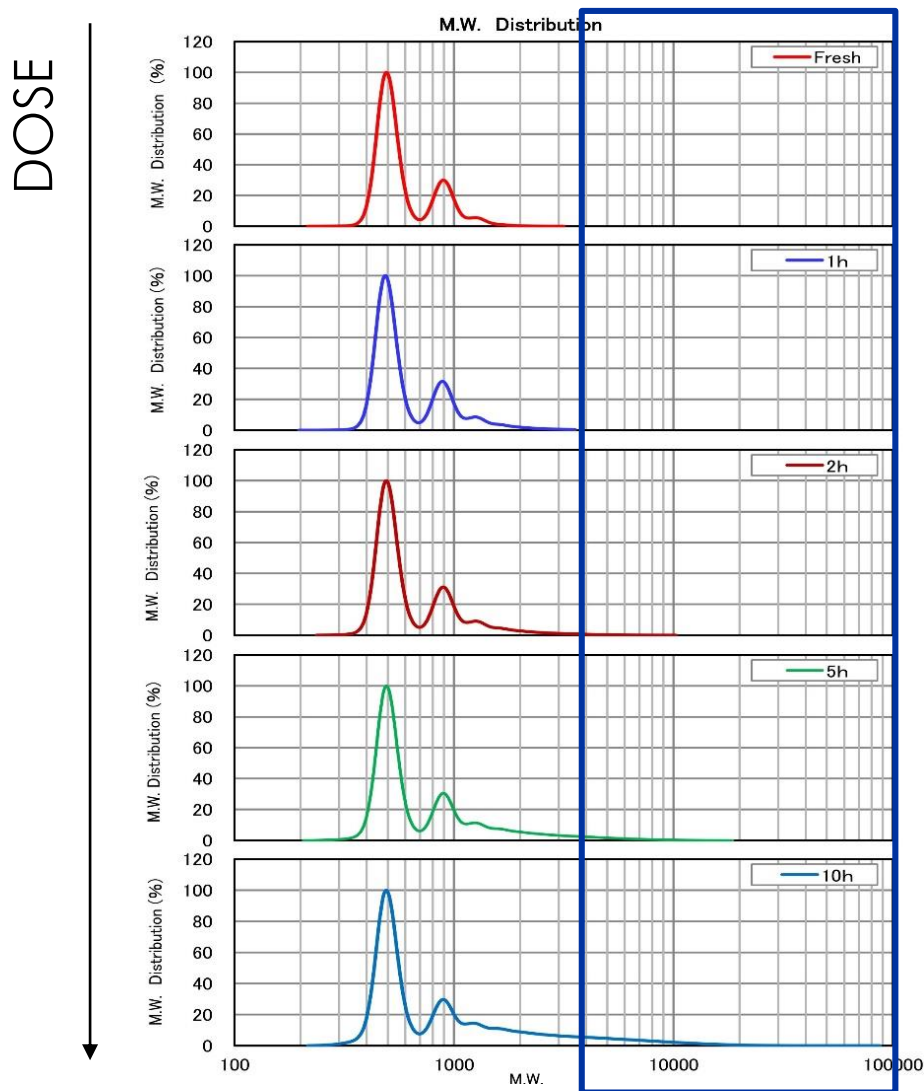


**REMARKABLE
MODIFICATIONS**

RP-42R-1 grease: stable



GEL PERMEATION FRACTION ANALYSIS

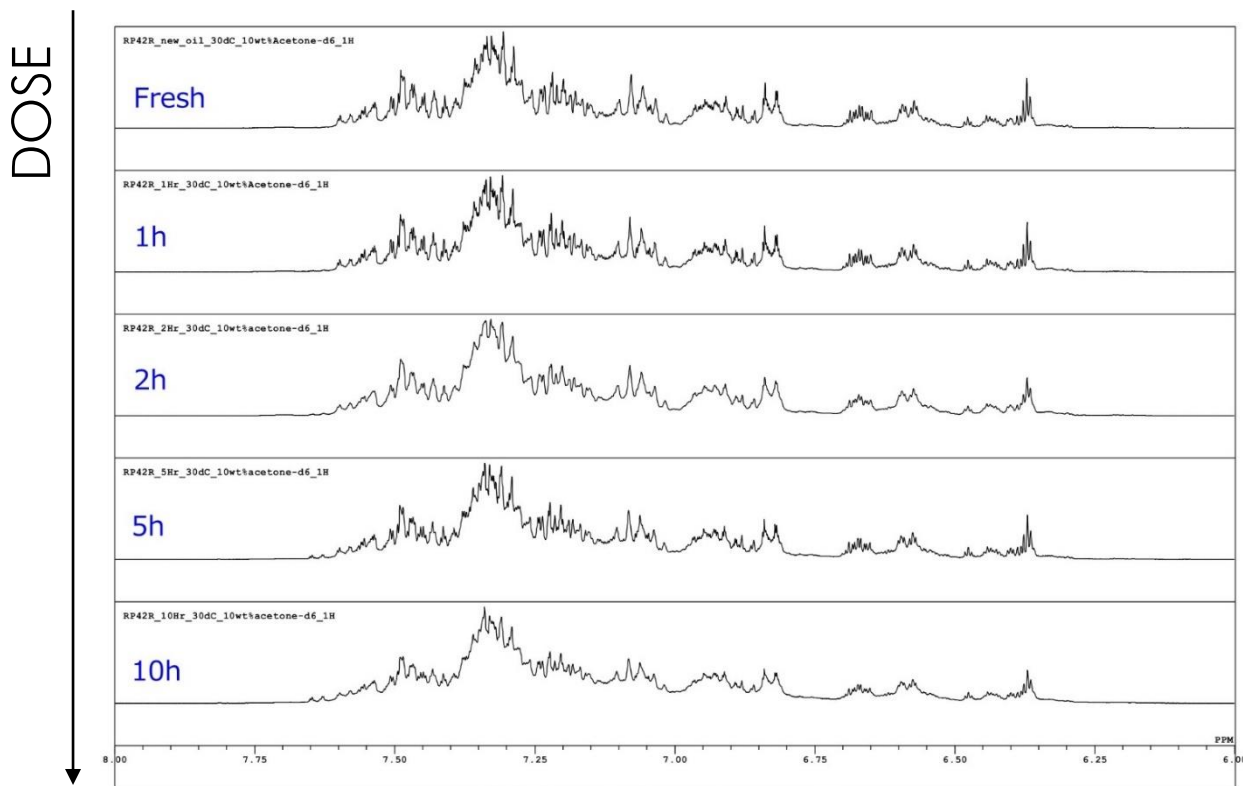


Molecular chains >2500

0% nonIRR
20% at 8 MGy

RAD-INDUCED POLYMERIZATION

NMR ANALYSIS

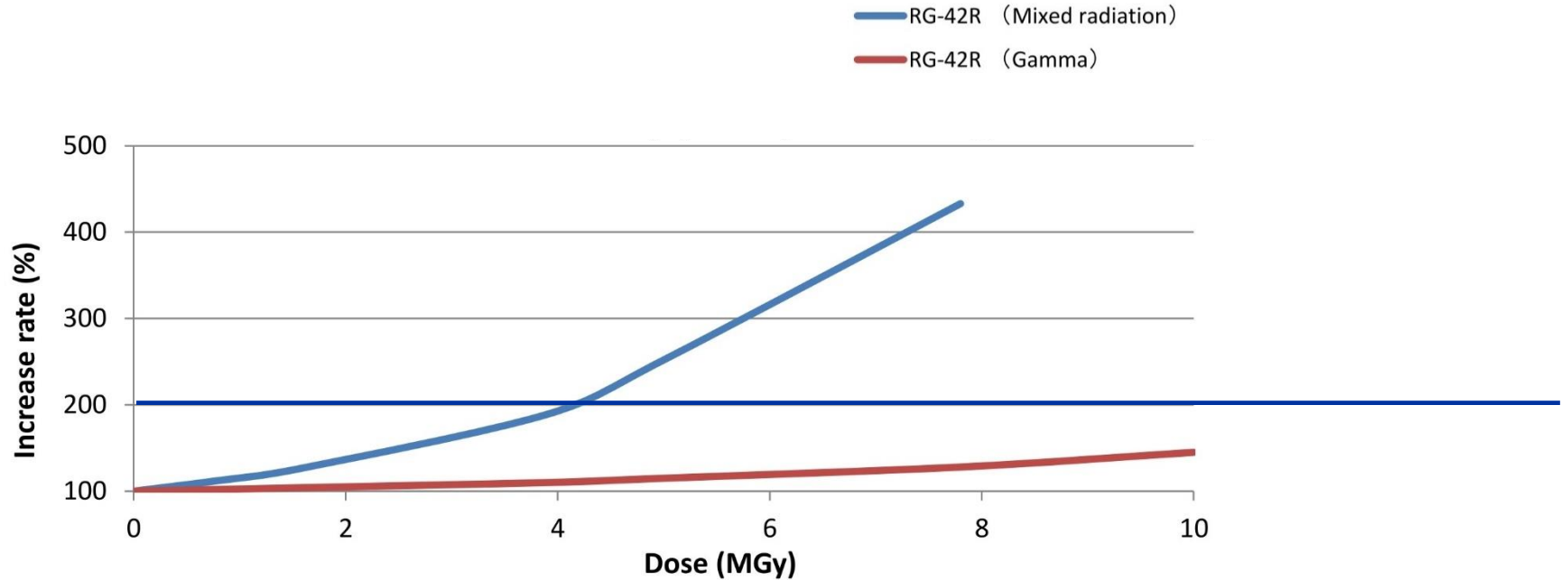


CHEMICALLY STABLE

ONGOING STUDIES

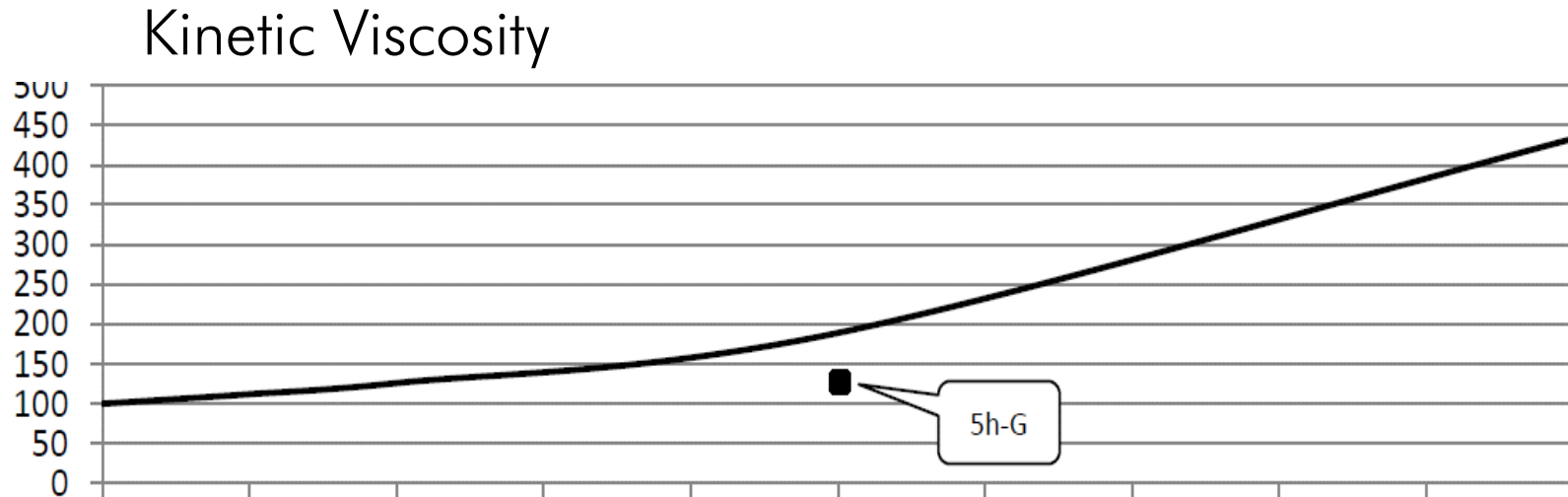


GAMMA VS MIXED FIELD: VISCOSITY



DIFFERENT EFFECT AT EQUAL DOSE

OIL EXTRACTED FROM THE GREASE



**BULK OIL vs OIL IN GREASE:
DIFFERENT EFFECTS**

CONCLUSIONS AND FURTHER DEVELOPMENTS



TAKE HOME MESSAGE

- Large sensitivity range: 0.1 MGy -10 MGy
- Irradiation and testing methodologies
- Dose thresholds are not universal
- Scarce knowledge of radiation damage mechanisms
- Ingredients and final product: different resistance
- Prediction of damage in real application: a challenge

**EXPERIMENTAL TEST OF PRODUCTS:
MANDATORY FOR FACILITIES**

FURTHER RESEARCH TOPICS

- Different rad fields
- Dose rate effect
- Oxygen effect
- Grease/bulk oil/extracted oil
- Radiation mechanisms: thickener vs oil
- Thresholds determination
- Specific case studies
- New products

TO BE INVESTIGATED

THANK YOU FOR YOUR ATTENTION!



*Special thanks to Hayashi San
(MORESCO)*



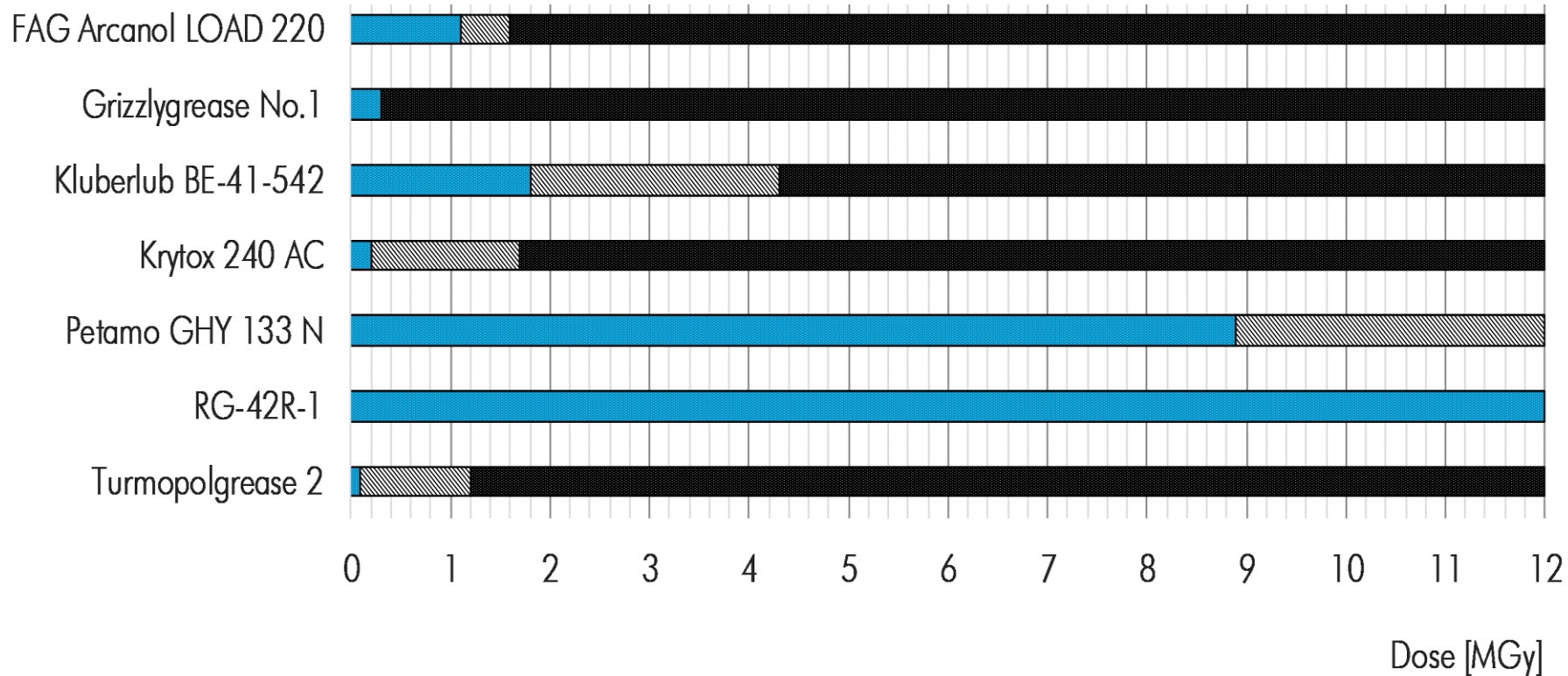
R2M

Radiation to Materials

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THRESHOLDS OF RADIATION DAMAGE: GREASES



M.Ferrari et al., Heliyon, Vol 5 Issue 9 e02489 (2019)

