ISOLDE seminar, 7.5.2014

The ERAWAST - initiative

How radioactive waste can be used for nuclear physics experiments

Dorothea Schumann, Rugard Dressler
Laboratory for Radiochemistry and Environmental Chemistry
Paul Scherrer Institute Villigen, Switzerland

Overview

- The ERAWAST project - history
- Sources for isotopes at PSI
- Prominent examples
  - ^{60}Fe from copper
    - source and radiochemical separation
    - applications
  - ^{44}Ti, ^{26}Al and ^{53}Mn from STIP samples
    - source and radiochemical separation
    - first experiment with a ^{44}Ti beam at ISOLDE
  - ^{7}Be from SINQ cooling water
    - source and radiochemical separation
- Proposal for the study of neutron capture cross sections of ^{53}Mn at n_TOF
- Potential proposal for the study of the $^{7}Be(n,\alpha)^{4}He$ and $^{7}Be(n,p)^{7}Li$ cross sections at n_TOF and SARAF

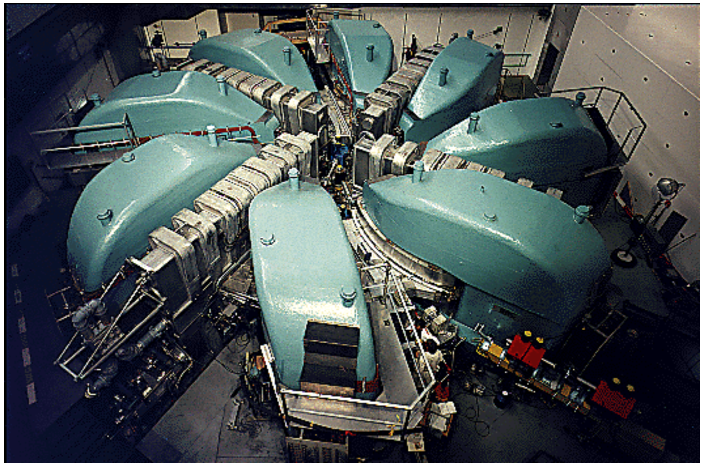
PSI accelerator facilities and the ERAWAST-project

Exotic Radionuclides from Accelerator WAste for Science and Technology

Objective:

Exploitation of accelerator waste for isolating rare exotic radionuclides

PSI cyclotron
590 MeV protons
2.4 mA beam current
High activiation of shieldings, targets, structure material



History:

- Radiochemical analytics of activated components for disposal
- Results showed high content of several rare isotopes
- Looking for potential users of these isotopes:
  I. ERAWAST workshop 2006 (PSI), funded by ESF
- Five-years working program
- II. ERAWAST workshop 2011 at PSI: first results and future program
- ~ 20 Partners (nuclear physics, astrophysics, AMS)

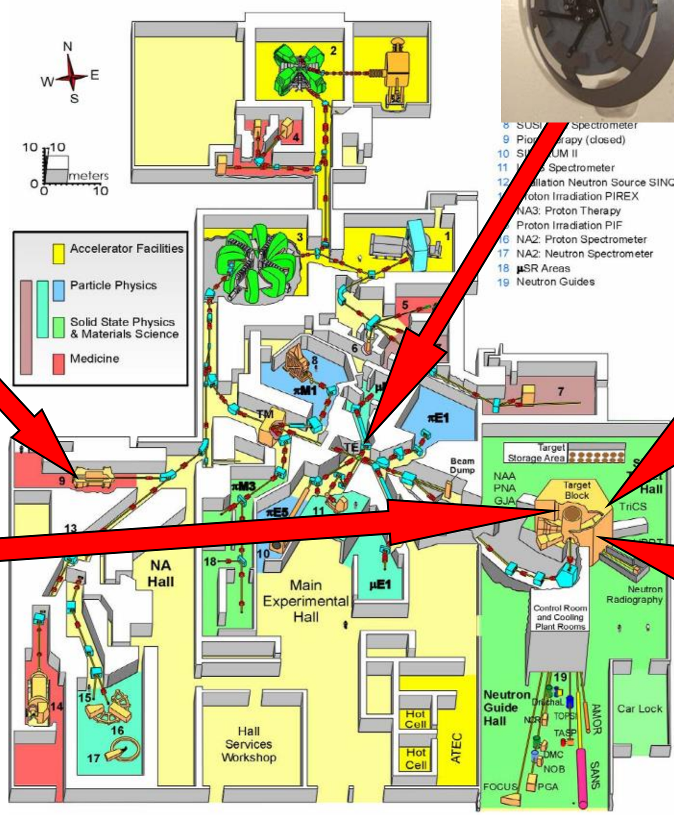
„Useful“ components of the PSI accelerator facilities

Copper beam dump

- ^{44}Ti, ^{53}Mn, ^{26}Al, ^{60}Fe, ^{59}Ni, ^{32}Si
- ^{60}Co – 5 GBq

Myon production station

- Operation 1-3 years
- Beam doses 4 – 11 Ah
- Source for ^{10}Be

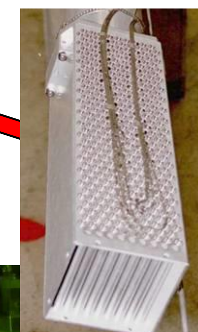


SINQ Target Irradiation Program-STIP

^{44}Ti, ^{53}Mn, ^{26}Al

SINQ cooling water

^{7}Be, ^{54}Mn, ^{22}Na, ^{88}Y



SINQ target

- ^{207}Bi, ^{172}Hf,
- ^{173}Lu, ^{194}Hg,
- ^{202}Pb, ^{125}Sb,
- ^{106}Ru, ^{44}Ti

Special irradiation positions with 590 MeV protons

V for ^{44}Ti production

Bi for ^{205}Pb production

Isotope production possibilities at PSI

Accelerator waste

Shielding, construction material, targets, beam dumps, cooling
intensely exposed by high-energetic protons and secondary particles
dismounted, cooled
ready or foreseen for disposal

Waste components:

Copper beam dump irradiated at the 590-MeV proton beam station at PSI, dismounted about 15 years ago

^{26}Al, ^{59}Ni, ^{53}Mn, **^{60}Fe**, ^{44}Ti

Proton-irradiated carbon from target E

^{10}Be, ^{7}Be ^{14}C, ^{3}H

Material from the SINQ facility

Lead targets

^{207}Bi, ^{182}Hf, rare earth elements (e.g. ^{146}Sm, several Dy isotopes) and lighter isotopes

STIP program (material research program)

Stainless steel for **^{44}Ti**, ^{26}Al, **^{53}Mn** production

SINQ cooling water

^{7}Be, long-lived isotopes from irradiated structure material (^{22}Na, ^{88}Y and many others)

Special irradiations

The SINQ facility offers the possibility to irradiate materials with 590 MeV protons at special positions. Tended experiments for isotope production can be offered

V for ^{44}Ti production
Bi for ^{205}Pb production

Irradiation with 71 MeV protons (injector 2) and up to 590 MeV neutrons (NAA, PNA)

Chemical separations with other material

^{60}Fe

Astrophysical background

Radiochemical separation

Determination of the half life

Determination of the neutron capture cross section at stellar energies

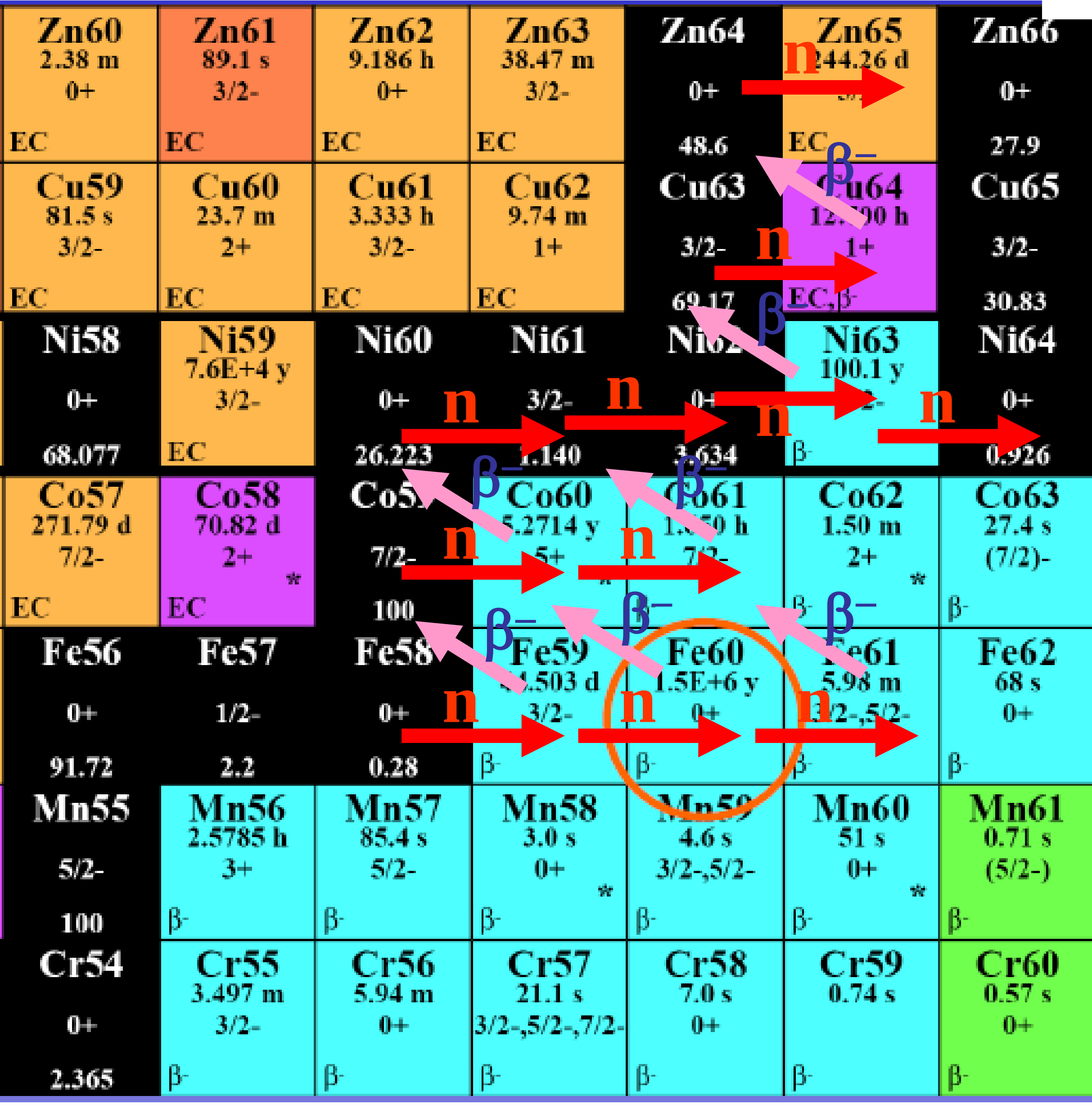
The astrophysical importance of ^{60}Fe

- represents an important chronometer for periods of several Million years ($t_{1/2}$ ~1.5 10^6 yr)

  ➡ formation of the solar system,
  nearby supernovae / AGBs
  γ-ray astronomy
- is produced in massive stars prior to the final supernova explosion (alternatively by AGB stars?)
- is in any case made by the s process via neutron capture reactions

Scientific aspects:

- Measured and calculated ratios of $^{60}Fe/^{26}Al$ are not in agreement
- Only one half-life measurement (Kutschera 1986; uncertainty 20%)
- Neutron capture cross sections unknown
- Nearly no alternative production route



s-process: neutron capture and following β^--decay

Necessary for evaluation

Cross sections for production

^{58}Fe(n,γ)^{59}Fe

^{59}Fe(n,γ)^{60}Fe

^{60}Fe(n,γ)^{61}Fe

Half-lives for decay

^{59}Fe ^{60}Co

^{61}Co ^{61}Fe

^{60}Fe

^{60}Fe sample material urgently needed!

Separation and preparation of ^{60}Fe

Source: copper beam dump

Dissolution of Cu chips (3 g) in 7 M HNO_3 (50MBq ^{60}Co)

Evaporation to dryness

Dissolution in 7 M HCl + 5 mg Co^{2+} as carrier

Extraction with methylisobutylketone

Aqueous phase:

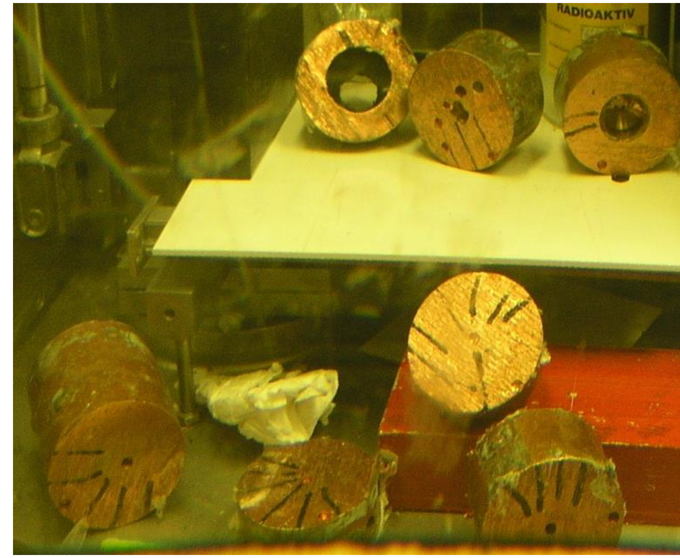
- Ni, Co, Cu,
- organic phase: Fe

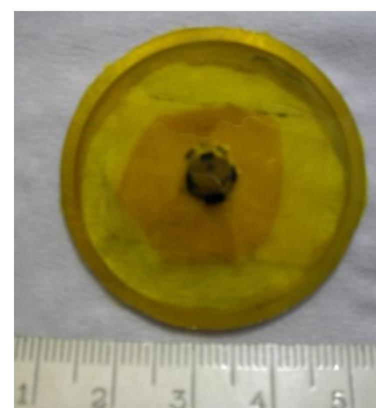
Back extraction with 0.1 M HCl, repetition of procedure

Result: $7.8 \cdot 10^{15}$ or 777 ng ^{60}Fe atoms, decontamination factor (Co) > 10^8 (0.3 Bq)

Evaporation of the final solution onto a graphite backing for the target

Solution for half life measurement





Re-determination of the half-life of ^{60}Fe

$$T_{1/2\,^{60}Fe} = \frac{N_{^{60}Fe}}{A_{^{60}Fe}} \ln 2$$

A: ingrowth of ^{60}Co; N: ICP-MS

^{60}Fe (1.5·10^6 y) $\xrightarrow{\beta^-}$ ^{60m}Co (10.5 min) $\xrightarrow{\gamma}$ ^{60}Co (5.3 y) $\xrightarrow{\gamma,\beta^-}$ ^{60}Ni (stable)

^{60}Fe: no γ radiation, low β-energy

⟶ very good chemical separation from Co necessary

- ICP-MS can measure isotope ratios for the iron isotopes
- ICP-MS in principle possible, but interference with ^{60}Ni
- Correction with other Ni-isotopes not possible, because no natural isotope ratios (production of stable isotopes via spallation in the beam dump)

⟶ addition of stable Fe carrier necessary

⟶ addition of stable Ni carrier necessary

$T_{1/2}$ = 2.62±0.04 · 10^6 years (1s)

$A_{(t=0)}\,^{60}Co$ = 0.207±0.006 Bq (starting with ~ 50 MBq in the Cu-chips)
$A\,^{60}Fe$ = 49.19±0.11 Bq
$m(Fe_{stable})$ = 2.6662±0.0009 mg
(M=55.9020±0.0033 g/mol because of non-natural abundance)
$I(N_{60Fe}/N_{Fe})$ = 2.0483±0.0035 · 10^{-4}

Rugel et.al. PRL 2009

KARLSRUHER NUKLIDKARTE

8. Auflage 2012

CHART OF THE NUCLIDES, 8th Edition 2012 / CARTE DES NUCLEIDES, 8ème Edition 2012
CARTA DE NUCLEIDOS, 8a Edición 2012 / ТАБЛИЦА НУКЛИДОВ, 8-е Издание 2012
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J. Magill[1], G. Pfennig[2], R. Dreher[1], Z. Sóti[2]
[1] Nucleonica GmbH, c/o European Commission, Hermann-von-Helmholtz-Platz 1, 76344 Eggenstein-Leopoldshafen, Germany, eMail: joseph.magill@nucleonica.com, http://www.nucleonica.com
[2] European Commission – Joint Research Centre – Institute for Transuranium Elements
P.O. Box 2340, 76125 Karlsruhe, Germany

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Fe 60
$2.62 \cdot 10^6$ a
β^- 0.2
m

G. Rugel, et al.:
Phys. Rev. Lett. **103** (2009) 072502

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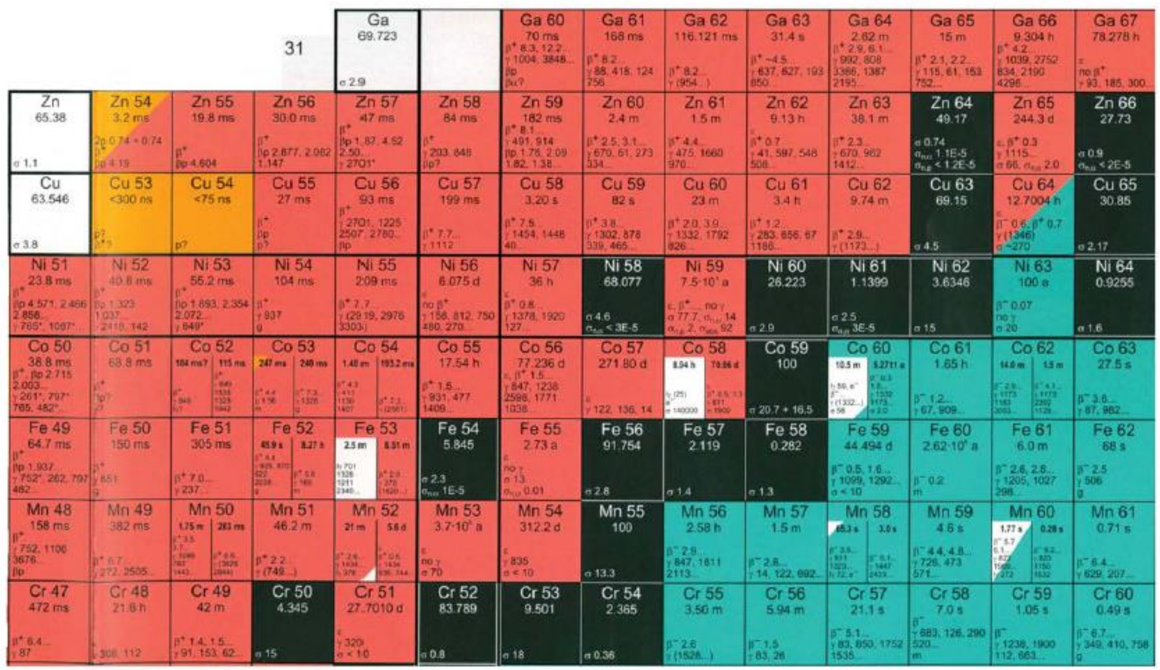
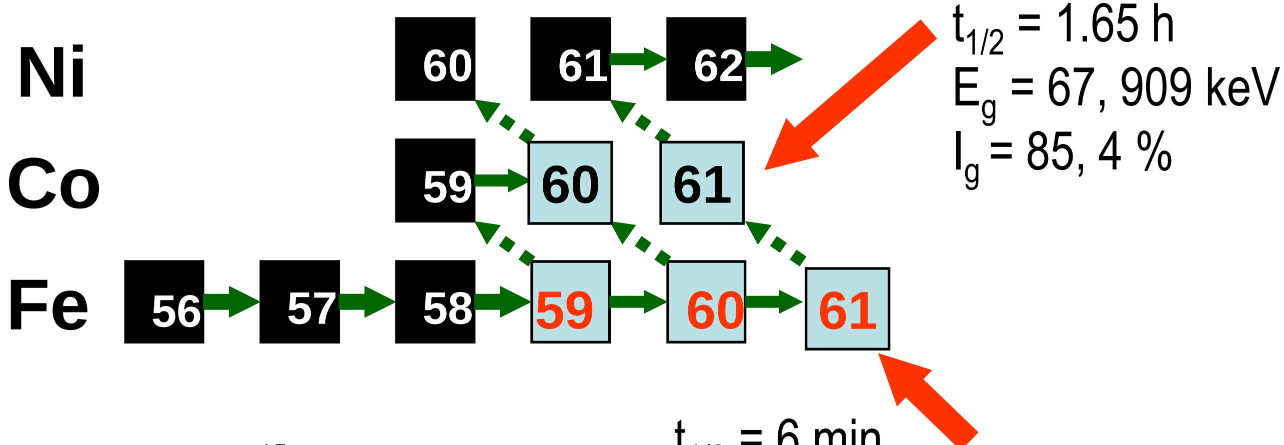


First determination of the neutron capture cross section

- **Measurement of the increase of the Co-daughter (determination of the number of ^{60}Fe-atoms)**
  ^{60}Fe (2.62·10^6 y) $\xrightarrow{\beta^-}$ ^{60m}Co (10.5 min) $\xrightarrow{\gamma}$ ^{60}Co (5.3 y) $\xrightarrow{\gamma,\beta^-}$ ^{60}Ni (stable)
  ^{60}Fe: no γ radiation, low β-energy
- **Measurement of the ^{61}Fe production (1027/1205 keV) → KIT Karlsruhe**



$t_{1/2}$ = 6 min
E_g = 298, 1027, 1205 keV
I_g = 22, 43, 44(5)%

Results

- total number of ^{60}Fe atoms = 7.8·10^{15} or 777ng (1.37 µg)
- total number of capture events = 118 (single) and 17 (coinc)
- time-integrated neutron flux = 1.7·10^{14}

Ueberseder et.al. PRL 2009

$^{60}Fe(n,\gamma)^{61}Fe$ cross section @ kT = 25 keV: **‹σ› = 5.8 ± 2.9$_{syst}$ ± 1.4$_{stat}$ mbarn**

^{44}Ti

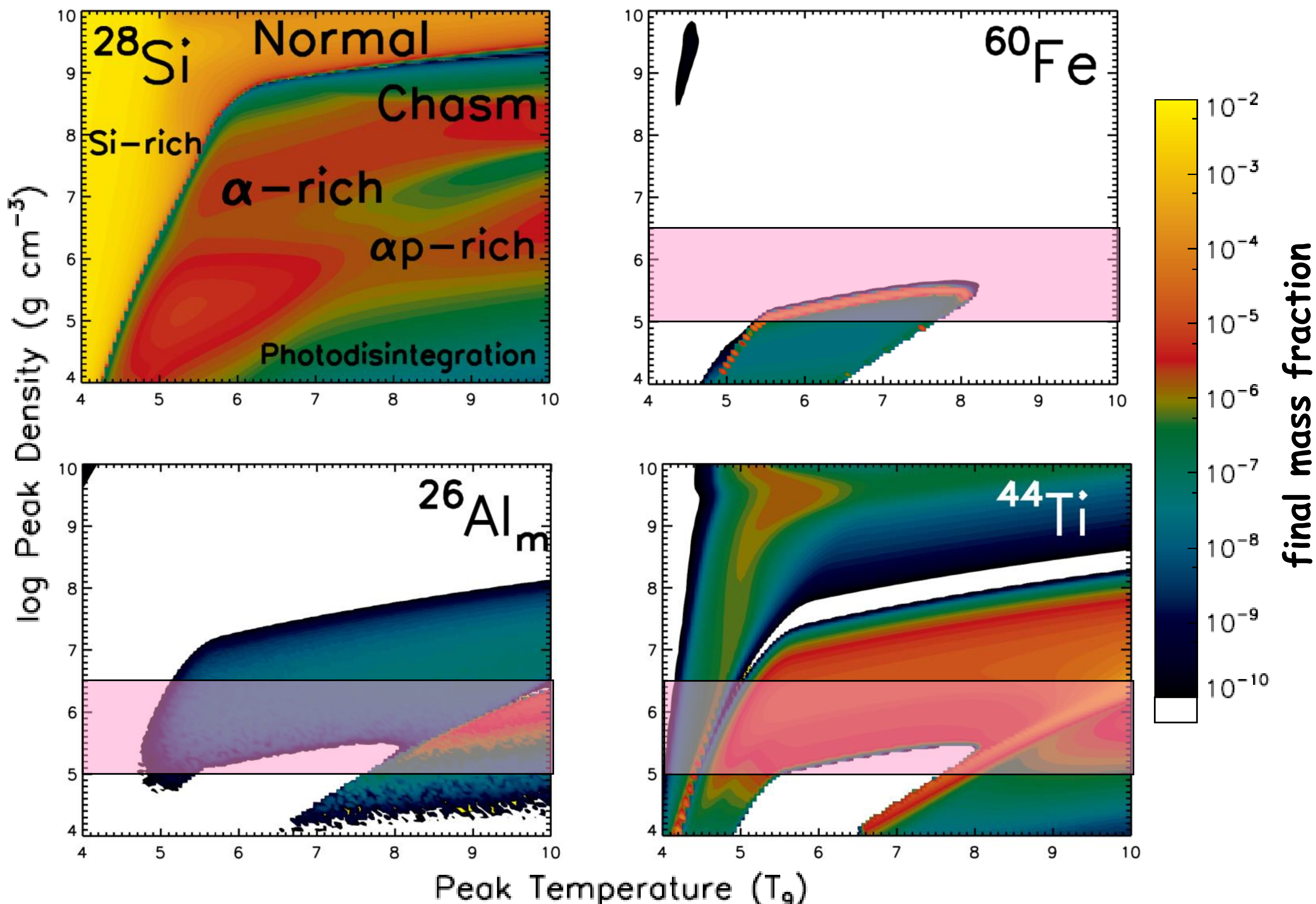
Astrophysical background

Radiochemical separation

Study of the ^{44}Ti(α,p)^{47}V reaction and implications for Core Collapse Supernovae

^{44}Ti production conditions in super nova explosion

total proton to nucleon ratio Y_e=0.48



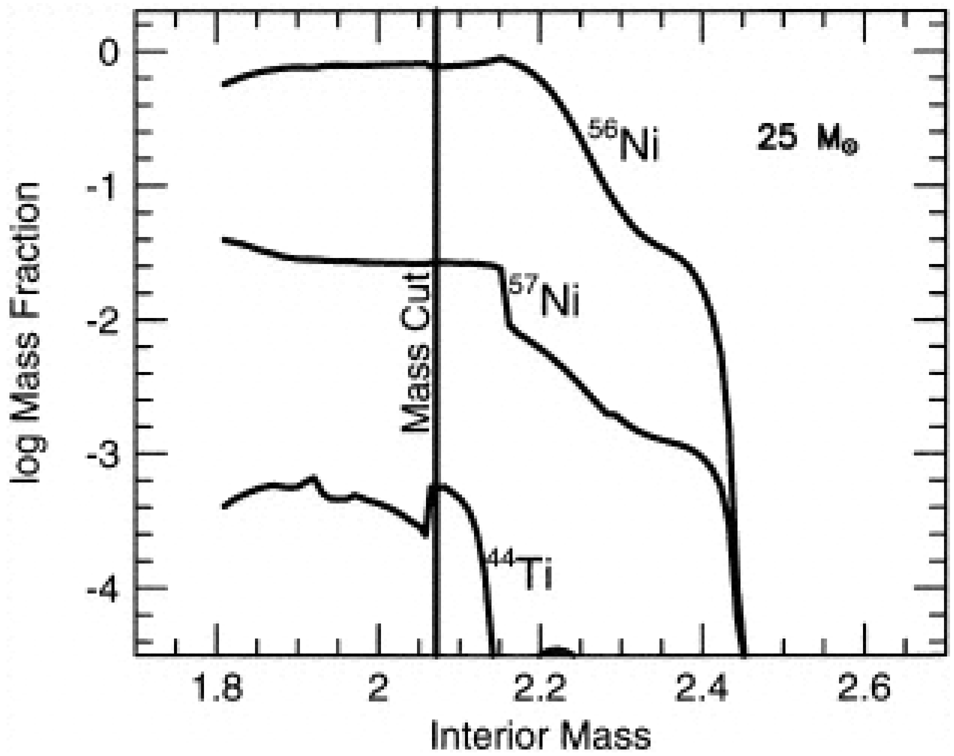
G. Magkotsios, et al.: Astrophys. J. 741 (2011) 78

Reactions and Mass cut of ^{44}Ti in super nova explosion

Order of Importance of Reactions Producing ^{44}Ti at $\eta = 0$[a]

Reaction	Slope
$^{44}Ti(\alpha, p)^{47}V$	−0.394
$\alpha(2\alpha, \gamma)^{12}C$	+0.386
$^{45}V(p, \gamma)^{46}Cr$	−0.361
$^{40}Ca(\alpha, \gamma)^{44}Ti$	+0.137
$^{57}Co(p, n)^{57}Ni$	+0.102
$^{36}Ar(\alpha, p)^{39}K$	+0.037
$^{44}Ti(\alpha, \gamma)^{48}Cr$	−0.024
$^{12}C(\alpha, \gamma)^{16}O$	−0.017
$^{57}Ni(p, \gamma)^{58}Cu$	+0.013
$^{58}Cu(p, \gamma)^{59}Zn$	+0.011
$^{36}Ar(\alpha, \gamma)^{40}Ca$	+0.008
$^{44}Ti(p, \gamma)^{45}V$	−0.005
$^{57}Co(p, \gamma)^{58}Ni$	+0.002
$^{57}Ni(n, \gamma)^{58}Cu$	+0.002
$^{54}Fe(\alpha, n)^{57}Ni$	+0.002
$^{40}Ca(\alpha, p)^{43}Sc$	−0.002

[a] Order of importance of reactions producing ^{44}Ti at $\eta = 0$ according to the slope of $X(^{44}Ti)$ near the standard reaction rates.



L.-S. The et al.: Astro.phys. J **504** (1998) 500

R. Diehl, F.X. Timmes: Publ. Astrosoc. Pacific **110** (1998) 637

SINQ Target Irradiation Program - STIP

- Different types of miniature specimens for assessing different mechanical properties such as tensile, fatigue, fracture properties and microstructural analyses

- Specimens were prepared by different participating laboratories based on their own request and then collected at PSI

Low-activation martensitic steels; the German version, Optifer, and the Swiss version, Optimax; mass%

Steel	Fe	Cr	Ni	Mo	Mn	Ti	V	W
Optifer	bal.	9.48	0.06	0.002	0.55		0.245	0.985
Optimax A	bal.	9.3	< 0.01	0.09	0.60	< 0.01	0.24	0.97
Optimax C	bal.	9.5	< 0.01	0.15	0.40	< 0.01	0.25	1.9

Samples for the separation

38 steel samples weighting from 1 to 2 g

^{54}Mn as a radioactive marker for the unknown amount of ^{53}Mn

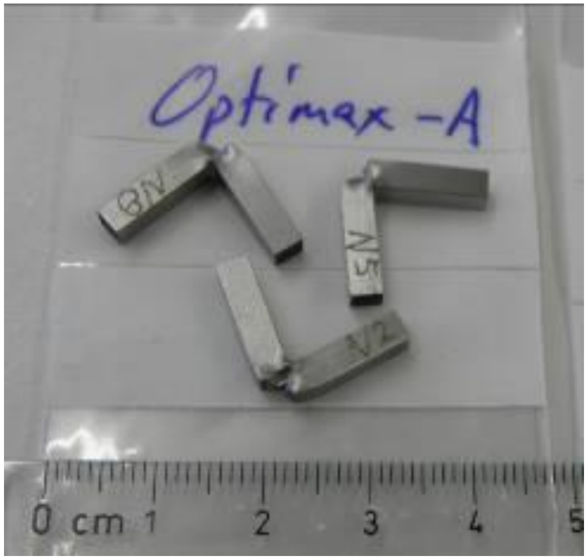
Steel samples and their amounts in MBq for the separation

Steel	Nr. of samples	^{44}Ti, MBq	^{54}Mn, MBq	^{60}Co, MBq
Optifer	9	95	23	17
Optimax A	9	47	13	10
Optimax C	8	74	19	29
Not identified	12	65	17	14
Total	38	**≈ 300**	**≈ 70**	**≈ 70**

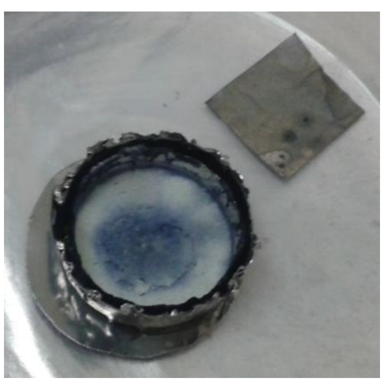
Separation of ^{44}Ti, ^{26}Al and ^{53}Mn from irradiated STIP-samples

Sample material:

60 g stainless steel from ***S***INQ ***T***arget ***I***rradiation ***P***rogram - STIP



Charpy-samples from STIP-I program



50 MBq $^{44}TiF_4$ in Mo-crucible

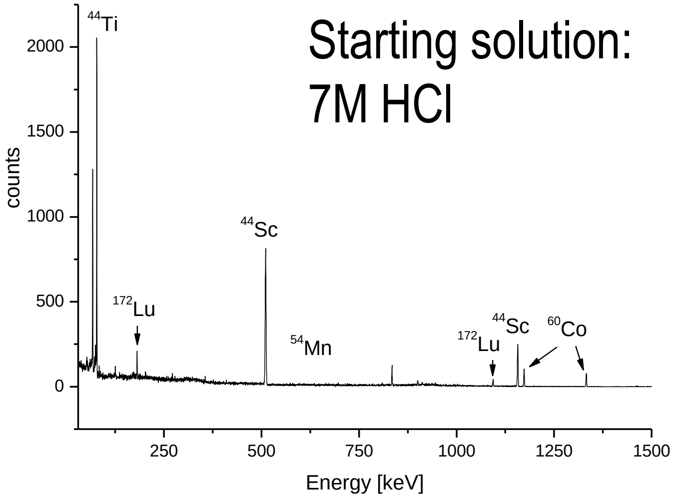
Chemical separation scheme:

- Stainless steel dissolved in 8M HCl+conc.HNO_3 — Fe, Cr, Ni, Mo, Mn, Co, Al, Ti
  - Extraction
    - Organic phase: Fe, Mo
    - Aqueous phase: Fe, Cr, Ni, Mo, Mn, Co, Al, Ti
      - Precipitation with NH_3
        - Filtrate: $[Ni(NH_3)_6]^{2+}$, MoO_4^{2-}, $[Co(NH_3)_6]^{2+}$
        - Precipitate: $Fe(OH)_3$, $Ti(OH)_4$, $Al(OH)_3$, $Cr(OH)_3$, $Mn(OH)_2$
          - Precipitation with $NaOH+H_2O_2$
            - Filtrate: **$[Al(OH)_4]^-$**, CrO_4^{2-} — Further separation and purification
            - Precipitate: $Fe(OH)_3$, MnO_2, $Ti(OH)_4$
              - Organic phase: Fe
              - Aqueous phase: **Mn, Ti** — Ion exchange chromatography for further separation and purification

Radionuclides available:

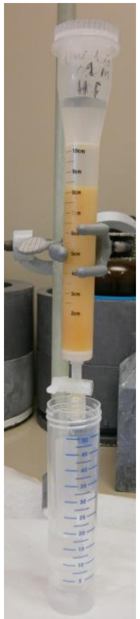
^{26}Al	300 Bq	≈ 10^{16} atoms
^{44}Ti	300 MBq	≈ $8 \cdot 10^{17}$ atoms
^{53}Mn	60 kBq	≈ 10^{19} atoms

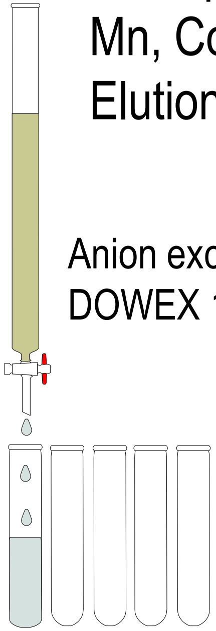
Separation of ^{44}Ti and ^{53}Mn



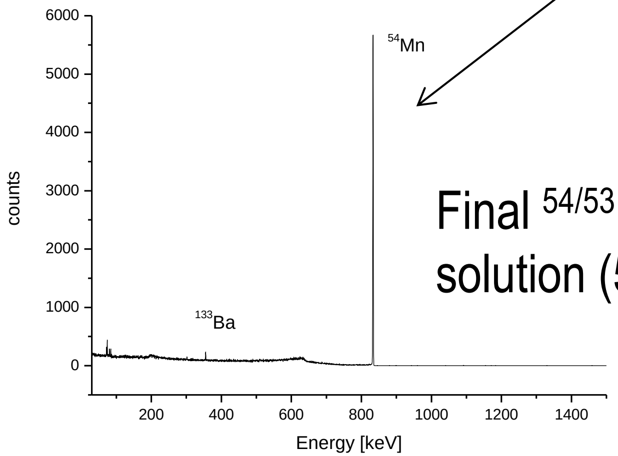
Starting solution:
7M HCl

Evaporation
Dissolution in 5M HF
Absorption on the column as TiF_6^{2-}
Mn, Co, Lu are stripped
Elution of Ti with 1M HNO_3

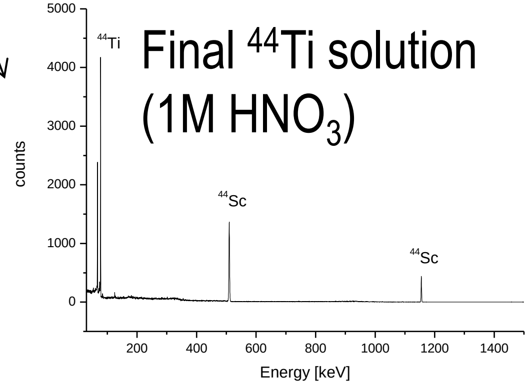




Anion exchanger
DOWEX 1x8



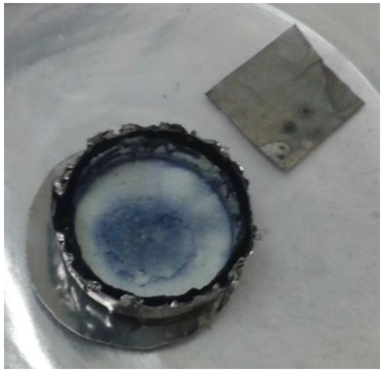
Final $^{54/53}Mn$ solution (5M HF)



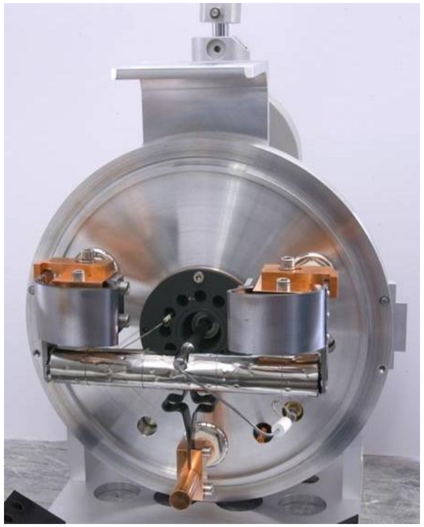
Final ^{44}Ti solution (1M HNO_3)

PAUL SCHERRER INSTITUT

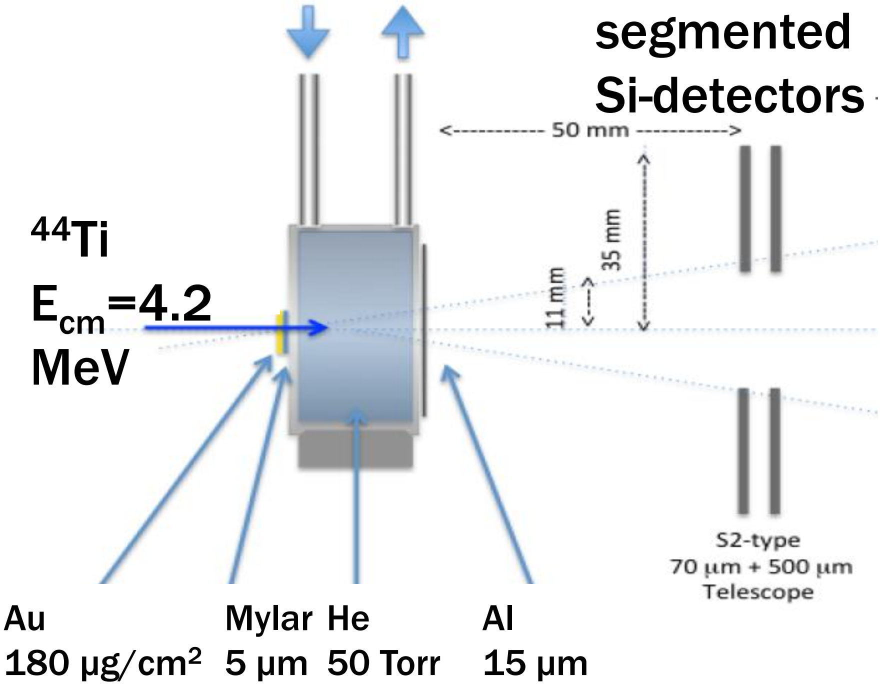
$^{44}Ti(\alpha,p)^{47}V$ at CERN ISOLDE Dec.2012

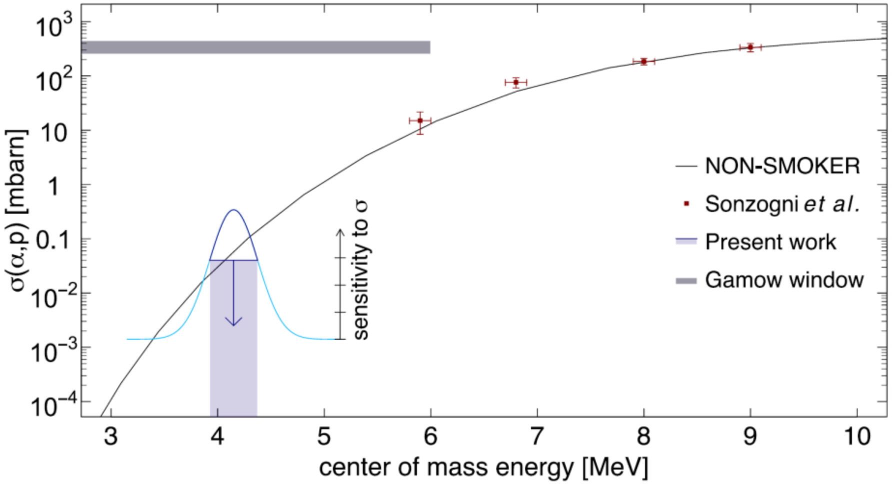


Final sample



ISOLDE oven





^{44}Ti delivered: 50 MBq = 5×10^{18} atoms
ion source: modified Mk 5 Febiad
TiF_3 extracted and post accelerated
10^5 pps ^{44}Ti with out any stable Ti

Margerin et.al. PLB 2014

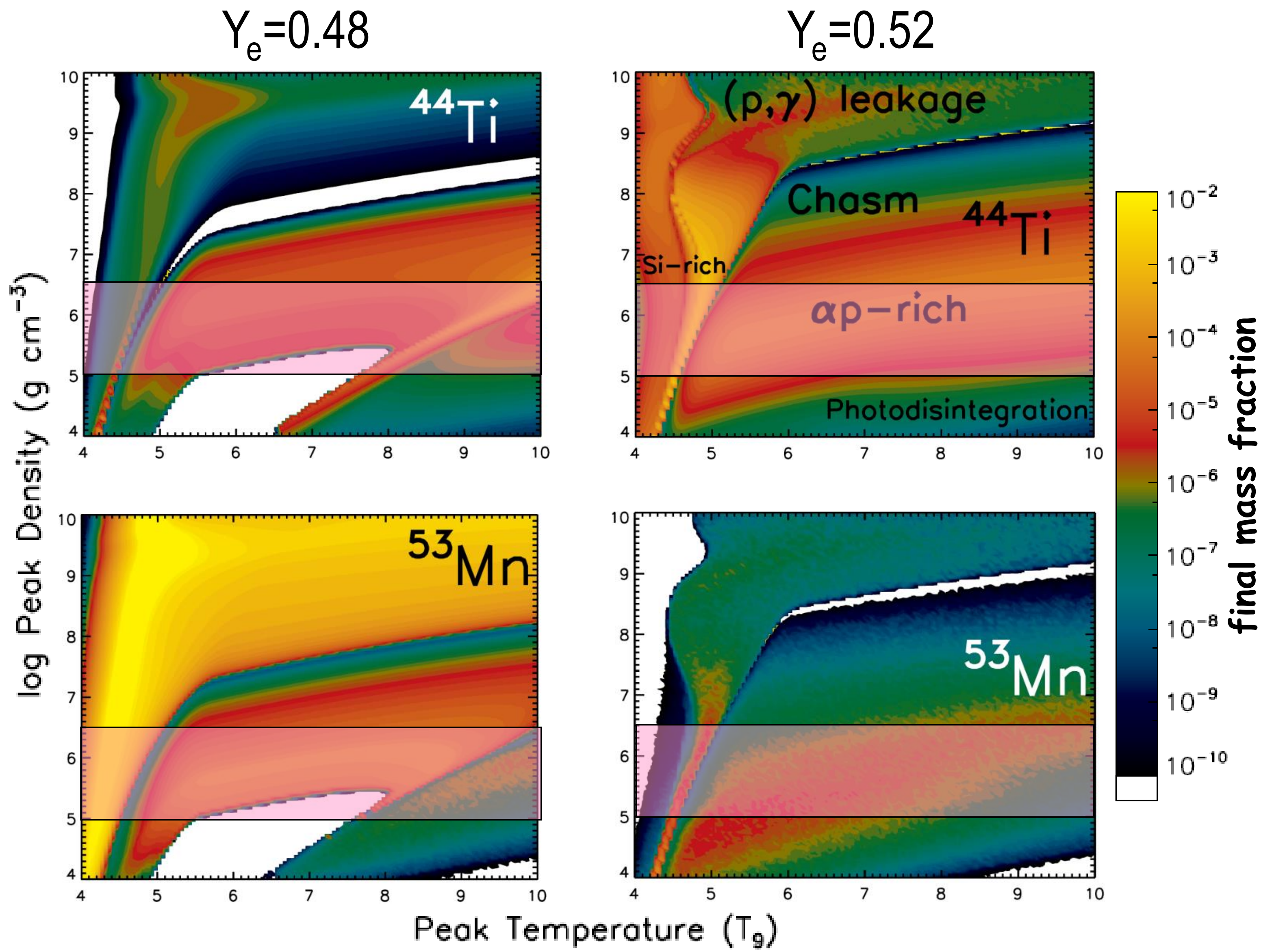
^{53}Mn

Astrophysical background

Radiochemical separation

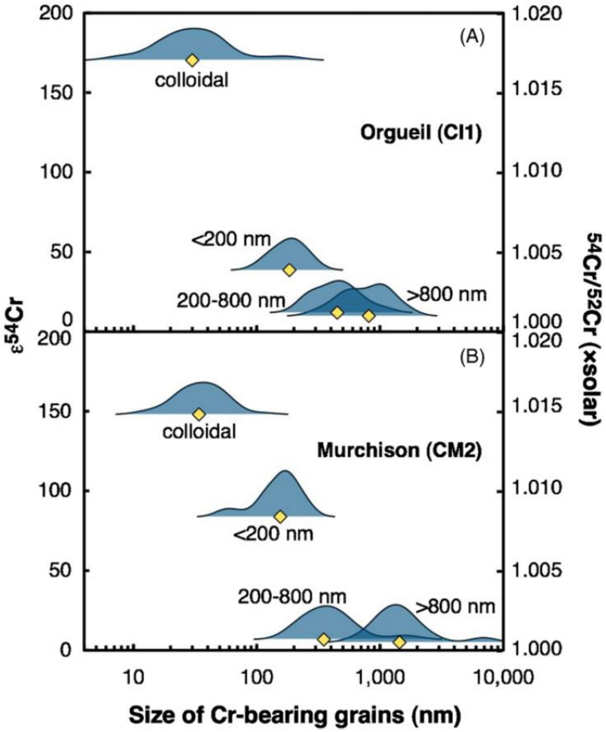
Planned experiments

^{53}Mn production conditions in super nova explosion

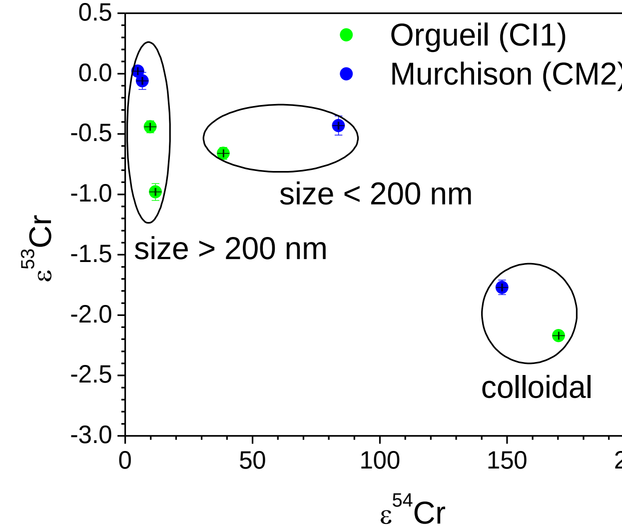


G. Magkotsios, et al.: Astrophys. J. 741 (2011) 78

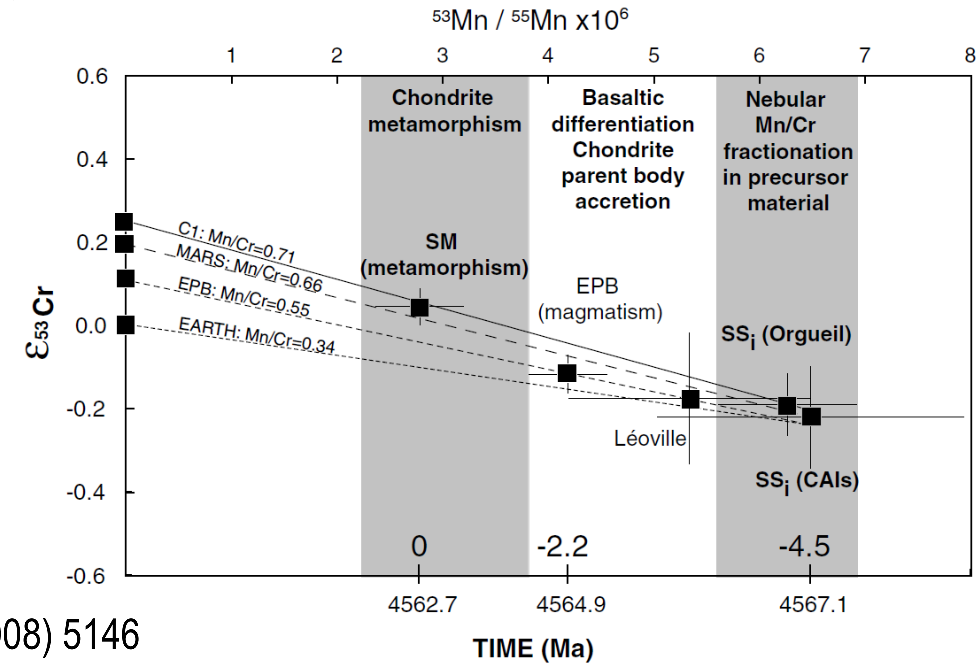
^{53}Mn neutron capture in meteoritic samples



^{53}Fe (e^-, ν_e)^{53}Mn
^{52}Fe (e^-, ν_e)^{52}Mn
^{60}Zn (e^-, ν_e)^{50}Cu
^{52}Mn (p, n)^{52}Fe
^{42}Ti (e^-, ν_e)^{42}Sc
^{46}Cr (e^-, ν_e)^{46}V
^{53}Fe (p, γ)^{54}Co
^{53}Ni (e^-, ν_e)^{53}Co
^{52}Fe (p, γ)^{53}Co
^{53}Mn (n, γ)^{54}Mn



Mn 53 3.7·10^6 a ε no γ σ 70	Mn 54 312.2 d ε γ 835 σ < 10	Mn 55 100 σ 13.3
Cr 52 83.789 σ 0.8	Cr 53 9.501 σ 18	Cr 54 2.365 σ 0.36



A. Trinquier, et al.: Geochim. Cosmochim. Acta **72** (2008) 5146

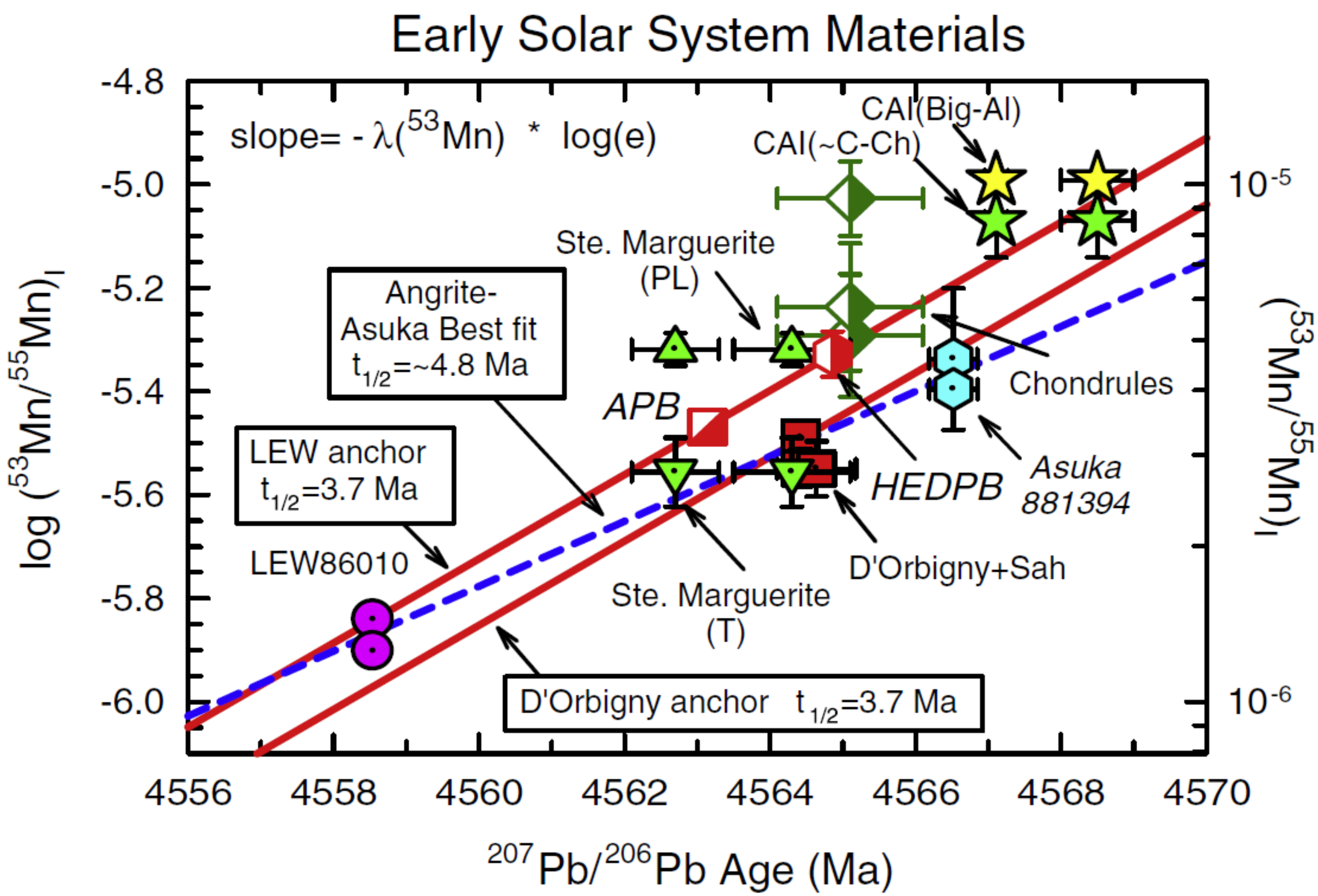
^{53}Mn half life

Author (Year)	Method	$t_{1/2}$ [Ma]
Wilkinson, et al. (1955)	Compared nuclear reaction yield with ^{54}Mn	$140 \cdot 10^{-6}$
Sheline, et al. (1957)	Calculated nuclear reaction yield	~2
Kaye, et al. (1965)	Spallation yield of meteorites	1.9 ± 0.5
Hohlfelder (1969)	Mass spectrometry (MS) of meteoric samples	10.8 ± 4.5
Matsuda, et al. (1971)	MS of 730 MeV proton activation products	2.9 ± 1.2
Honda, et al. (1971)	MS of artificial and meteoritic samples	3.7 ± 0.37
Wölfle, et al. (1972)	Neutron activation of meteoritic samples	3.9 ± 0.6
Heimann, et al. (1974)	Decay of meteoritic ^{53}Mn	3.85 ± 0.4

Limited amount of sample material: 2.5×10^{11} to 1.3×10^{13} atoms of ^{53}Mn extracted from meteorites ⟶ relatively high uncertainty

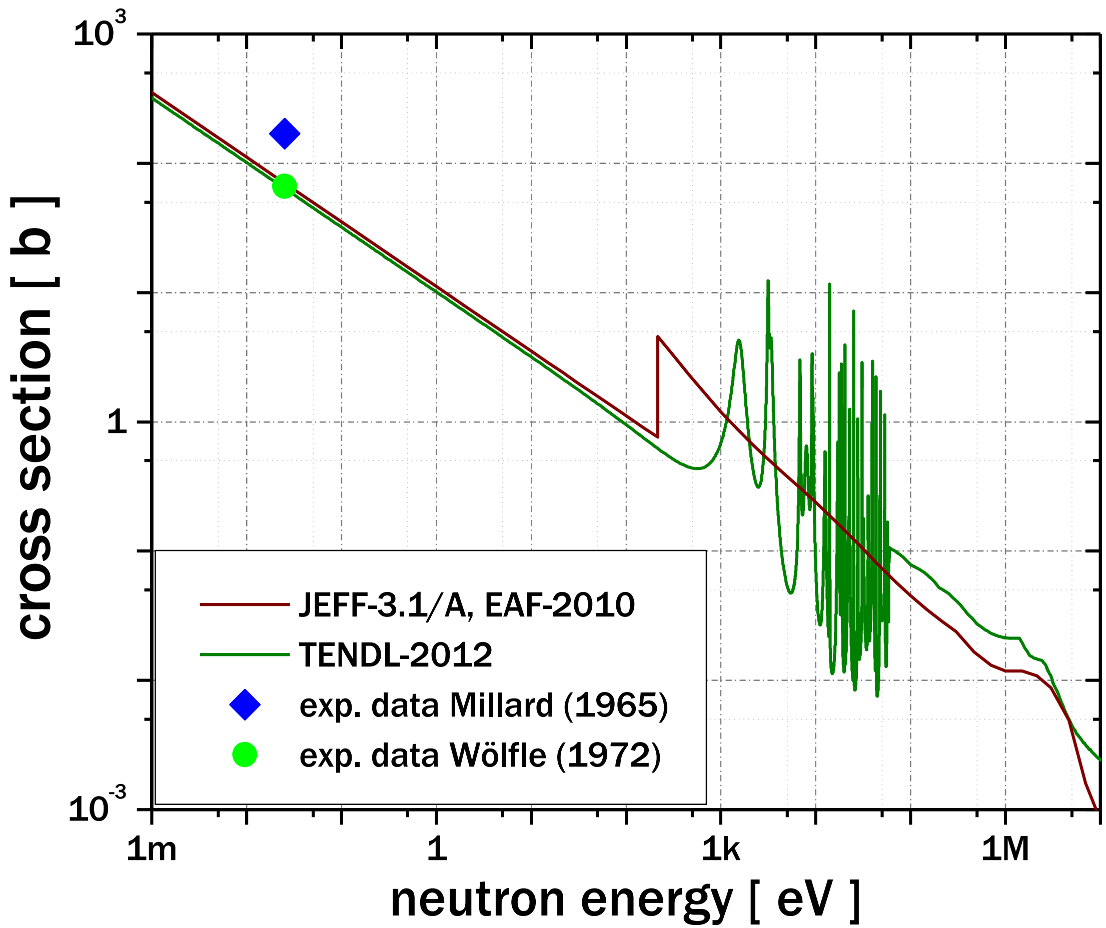
Improvements:
Sample material: more than 10^4 more atoms! Improved uncertainty!
High-sophisticated measurement technique (LSC; ICP-MS)

Dating Problem $^{53}Mn/^{55}Mn$ vs. $^{207}Pb/^{206}Pb$



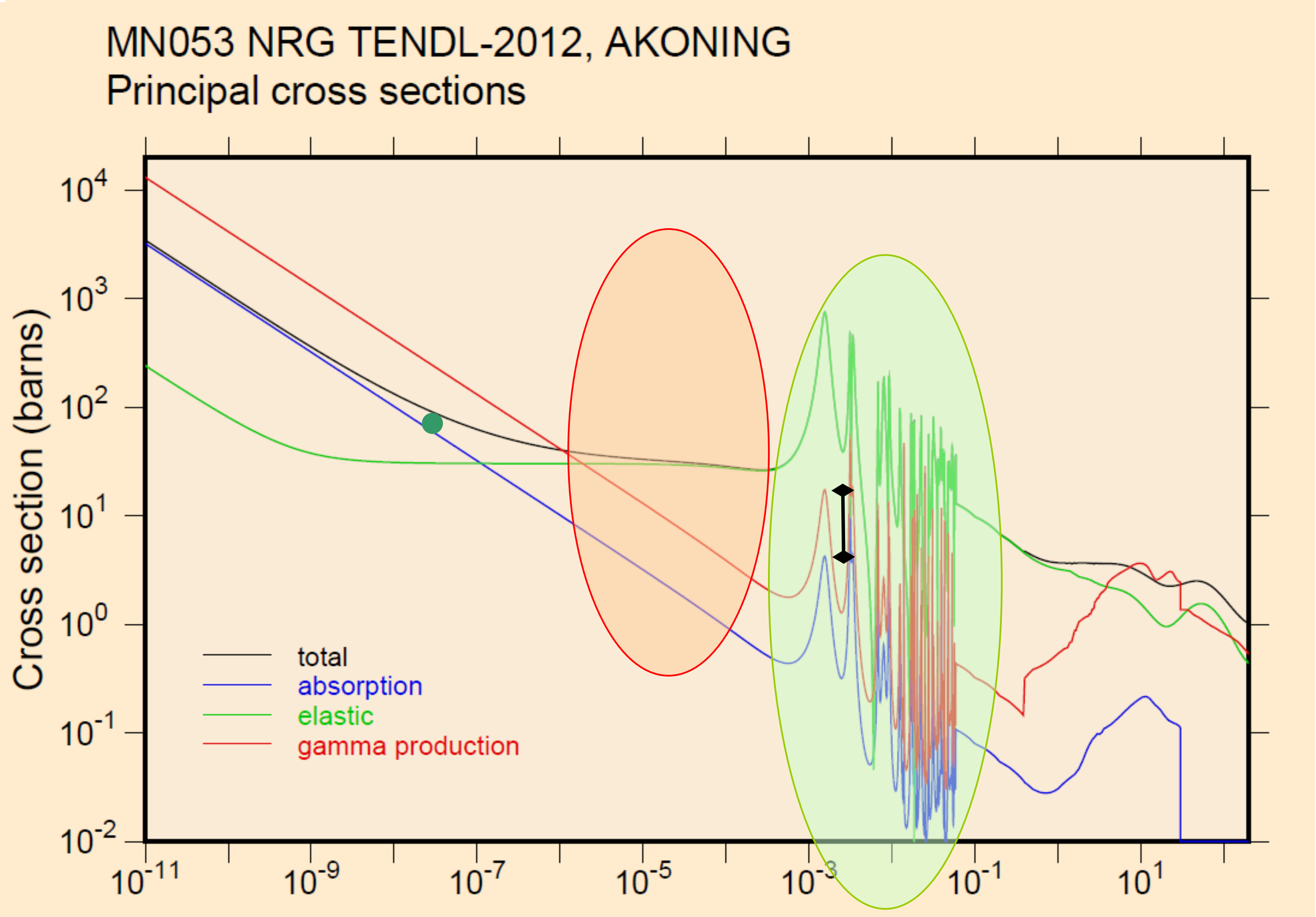
L.E. Nyquist, et al.: Geochim. Cosmochim. Acta **73** (2009) 5115

Neutron capture cross-section ^{53}Mn



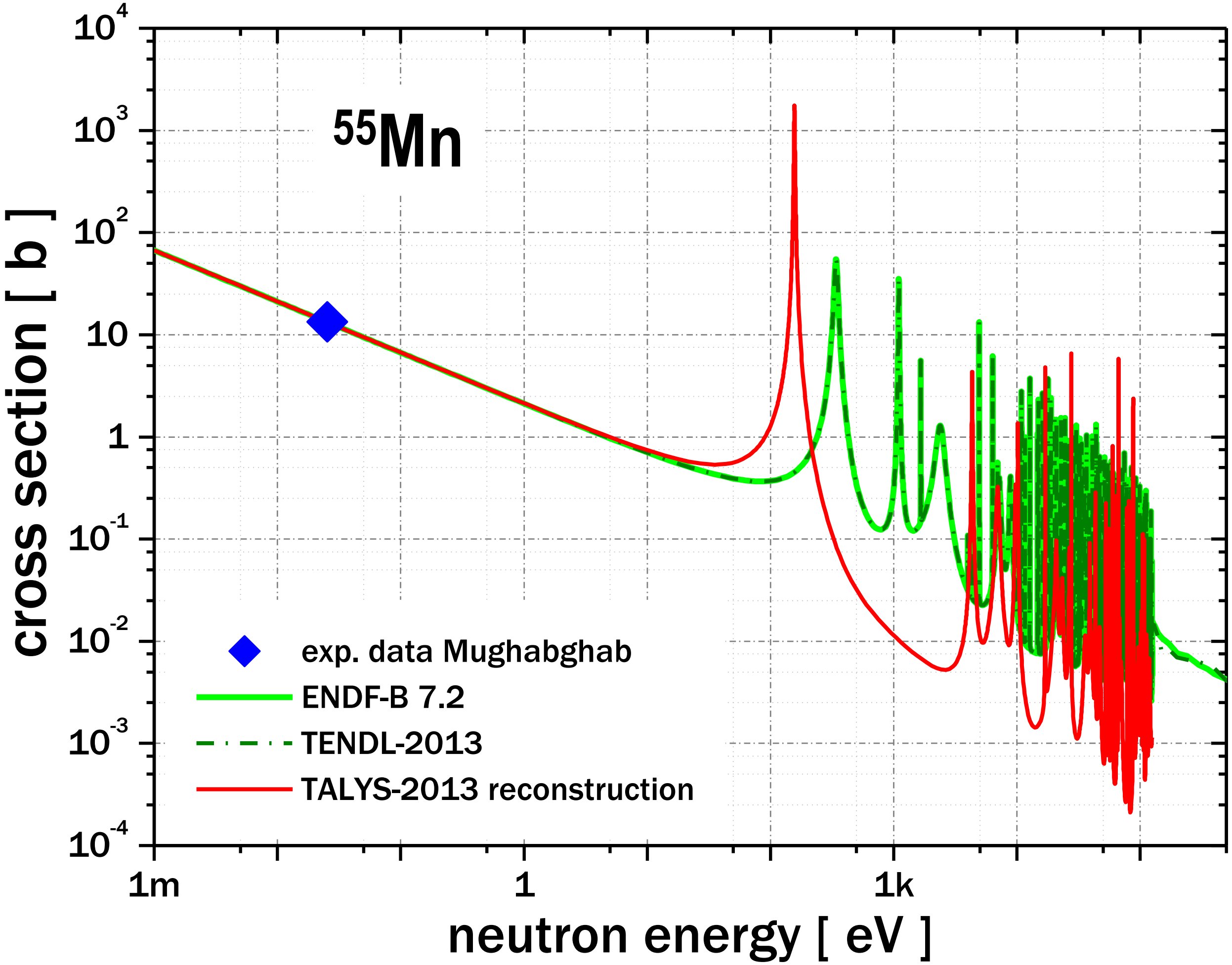
JEFF-3.1/A = R. A. Forrest, J. Kopecky, J.-Ch. Sublet (2003) UKAEA FUS 486
EAF-2010 = J.-Ch. Sublet, et al. (2010) CCFE-R (10) 05
TENDL-2012 = A.J. Koning, D. Rochman (2012) Nucl. Data Sheets 113, 2841

Changes in TALYS predictions



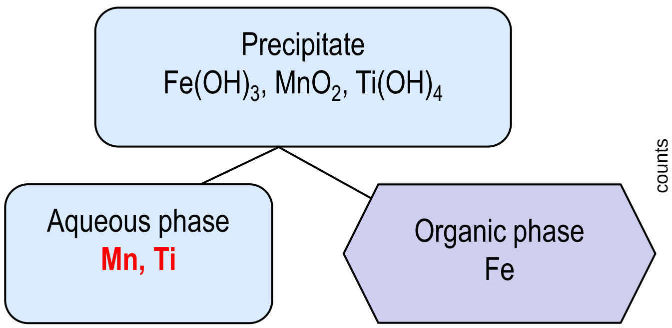
taken from www.talys.eu

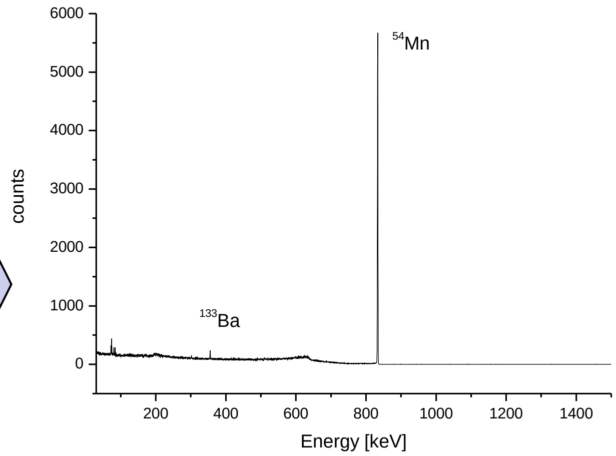
TALYS forecast using thermal cross section only



S.F.Mughaghab: Atlas of Neutron Resonances Resonance Parameters and Thermal Cross Sections Z=1-100 (2006)

Chemical separation and purification of ^{53}Mn





Precipitation of Cr with Urotropin (pH 5.5):
Mn stays in solution
precipitation of $Cr(OH)_3$

content of STIP-samples **3×10^{19} atoms ^{53}Mn**

1.8×10^{21} atoms V	6.6×10^{22} atoms Cr
3.3×10^{21} atoms ^{55}Mn	5.7×10^{23} atoms Fe

chemical yield: 70%

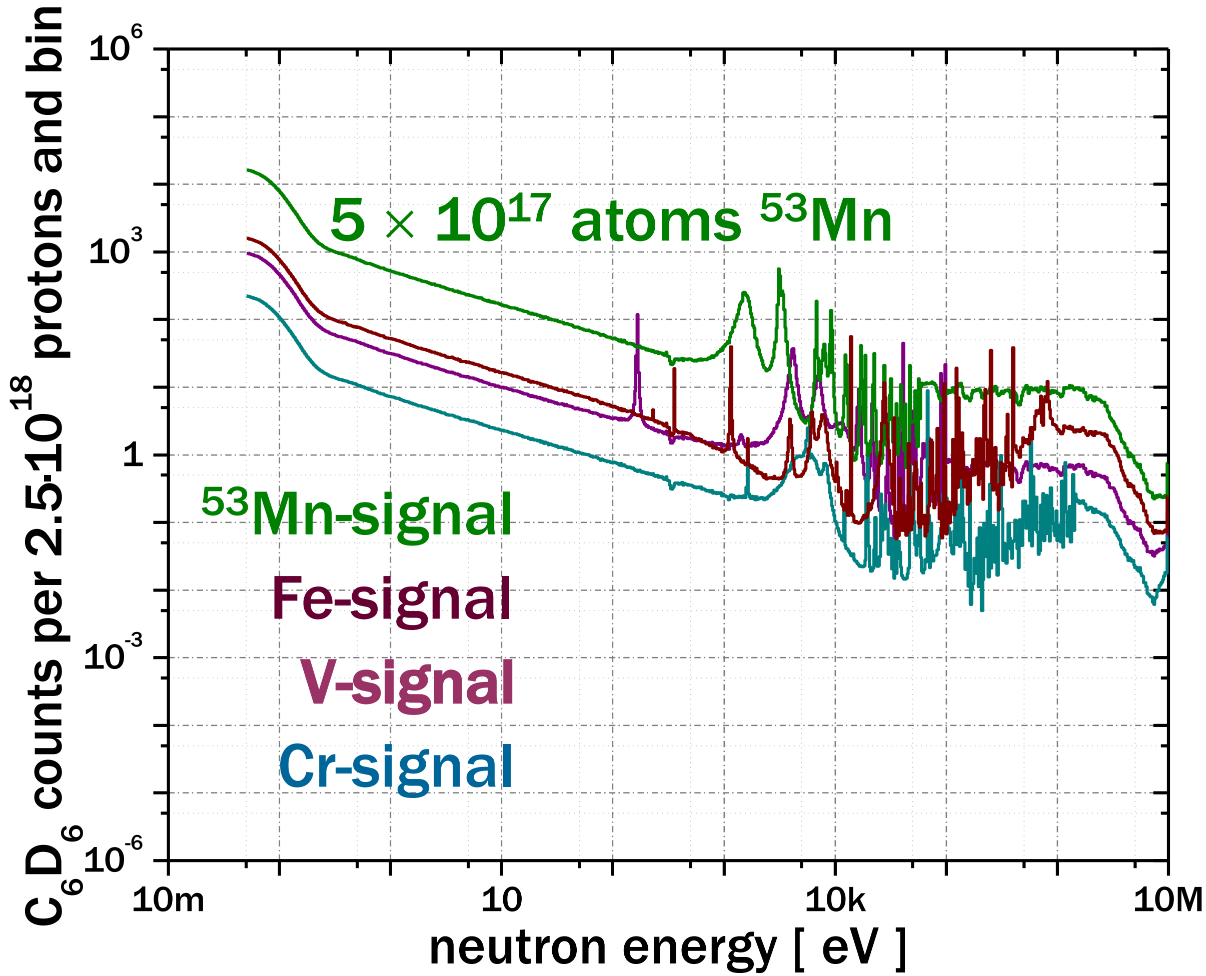
suppression of other elements 10^{-4}

stock solution: **2×10^{19} atoms ^{53}Mn**

1.8×10^{19} atoms V	6.6×10^{18} atoms Cr
2.3×10^{21} atoms ^{55}Mn	5.7×10^{19} atoms Fe

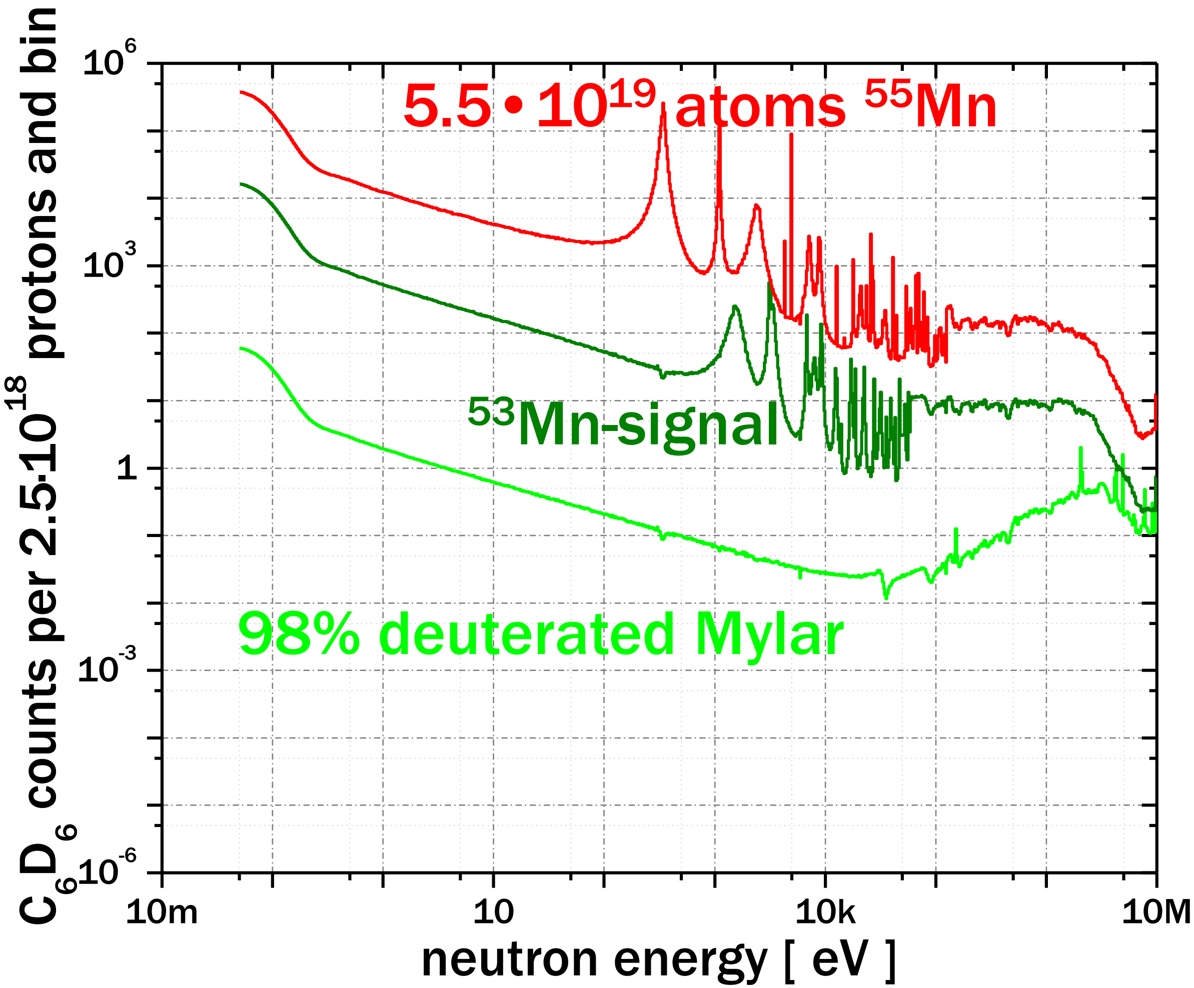
Expectations of signal contributions

Contribution of other elements



Expectations of signal contributions

Contribution of ^{55}Mn and backing material



ISOLDE mass separator

FEBIAD output:	100 μA
(single charged Mn)	6.2×10^{14} part. per sec
suppression Δmass = 1 :	$> 10^3$
suppression Δmass > 1 :	$> 10^4$
ionisation yield ISOLDE:	2.5% Mn
separation time:	5 hours per 10^{17} atoms ^{53}Mn

final sample: 5×10^{17} atoms ^{53}Mn

8.7×10^4 atoms V	2.4×10^{12} atoms Cr
5.8×10^{15} atoms ^{55}Mn	4.9×10^9 atoms Fe

total separation time: 25 hours

First joint experiment ISOLDE - n_TOF

^{7}Be

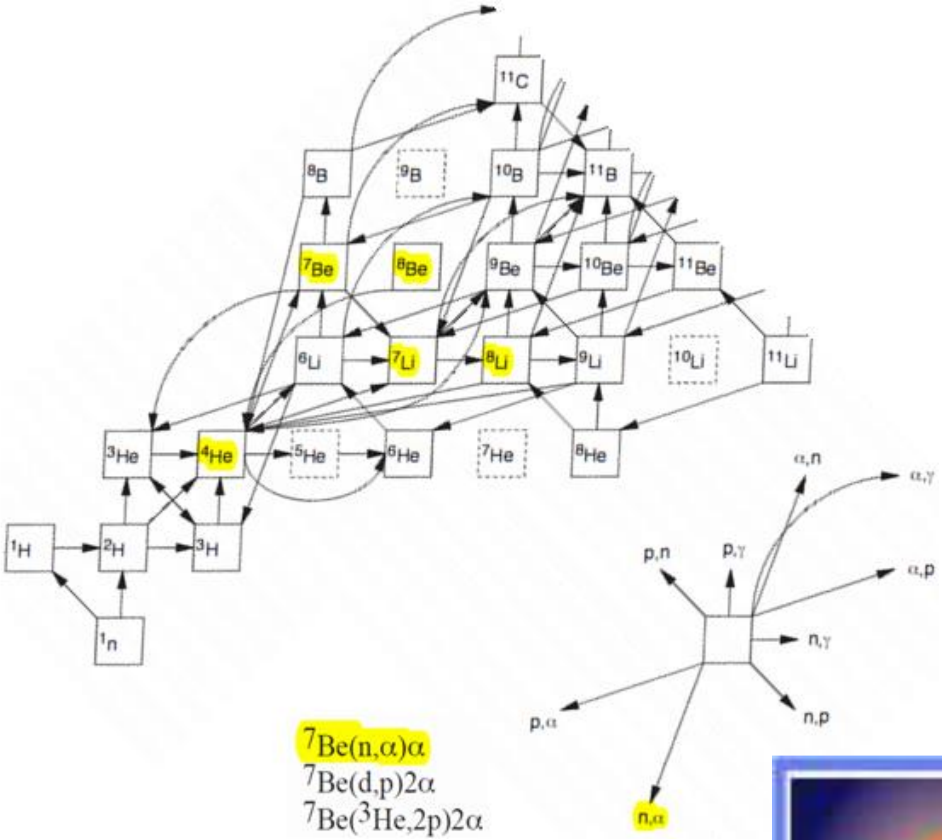
Astrophysical background

Radiochemical separation

Planned experiments

^{7}Be(n,x)y Reactions and the problem of primordial ^{7}Li

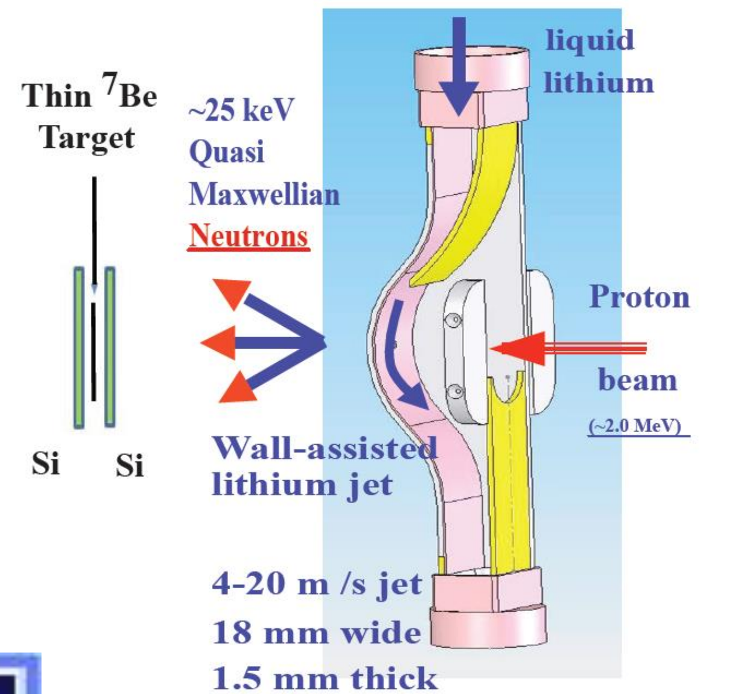
Connecticut University, PSI, CERN, SOREQ



^{7}Be(n,p)^{7}Li and ^{7}Be(n,α)^{4}He at n_TOF

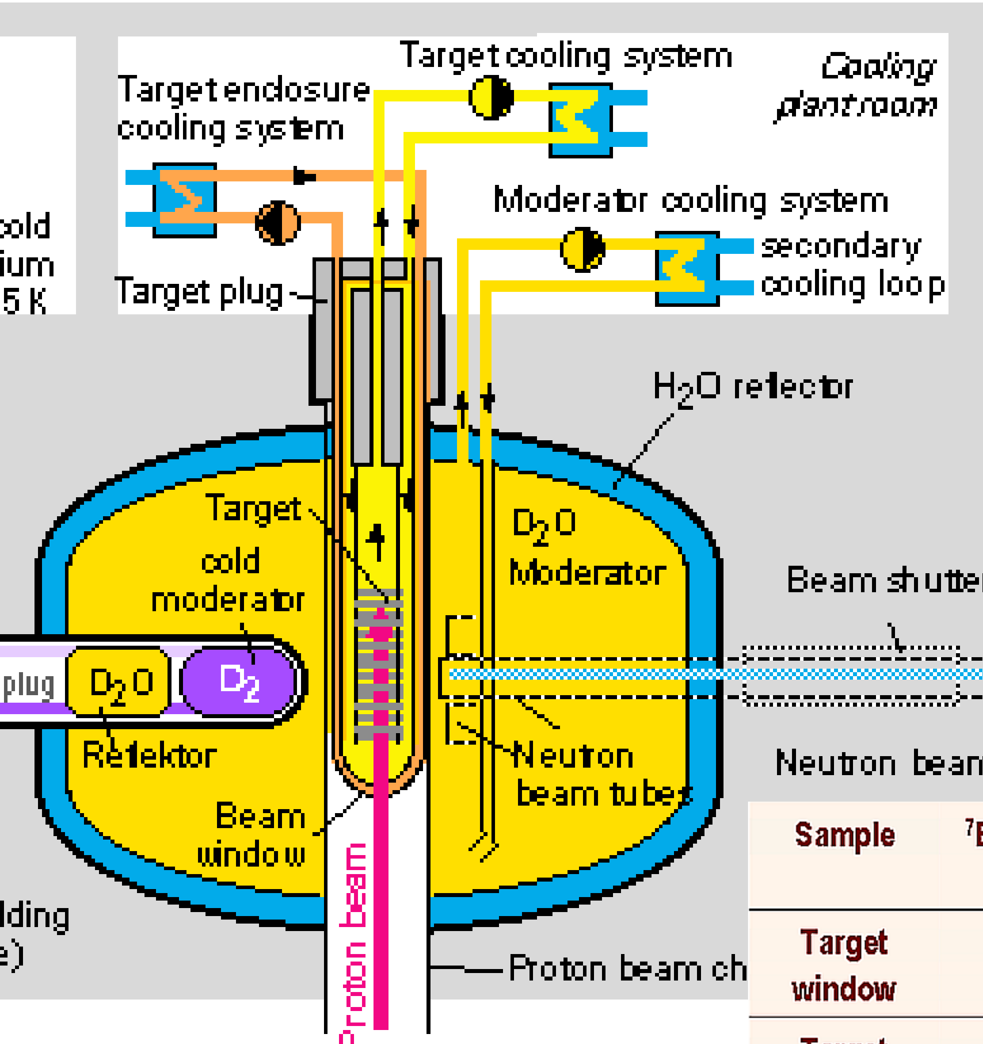
The LiLiT at the SARAF facility in the Soreq Nuclear Center, Israel

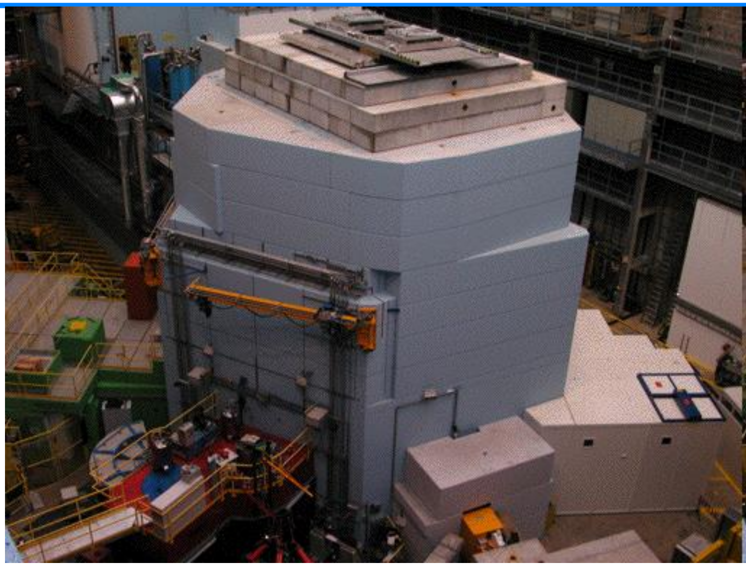
For the Study of the ^{7}Be(n,α)α Reaction



Clean and Thin ^{7}Be Target
~10^{17} ^{7}Be/cm^2(~5 GBq 0.4x0.4 cm^2)
~200 GBq ^{7}Be
Implantation at CERN-ISOLDE

^{7}Be separation from SINQ cooling water





^{7}Be (477.6 keV, 53.3 days) produced by spallation reactions on ^{16}O in the cooling water (D_2O) of SINQ
No by-products - high specific activity of ^{7}Be

Sample	^{7}Be, Bq/g	^{3}H, MBq/g	^{22}Na, Bq/g	^{110m}Ag, Bq/g	^{88}Y, Bq/g
Target window	480	24	0.4	-	-
Target	73	25	-	-	0.2
Moderator	535	21	-	1.2	-

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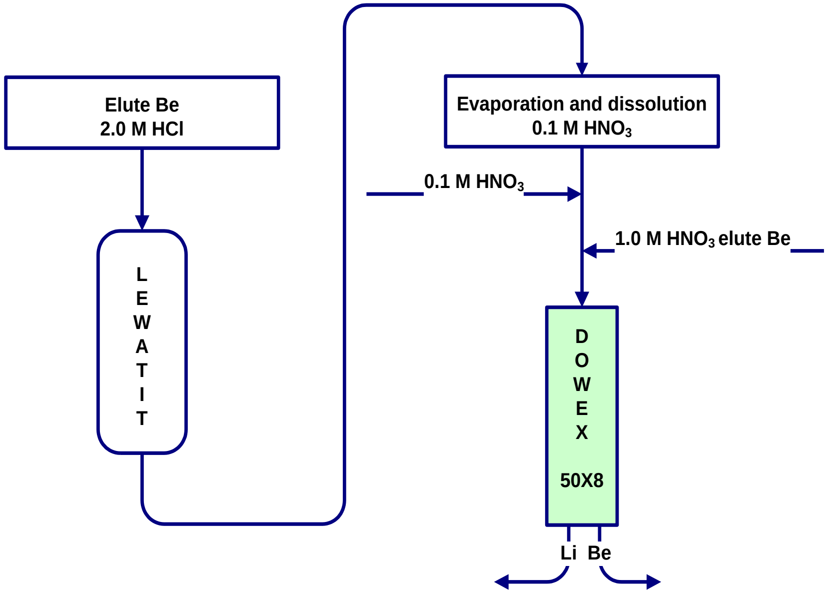
Radiochemical separation

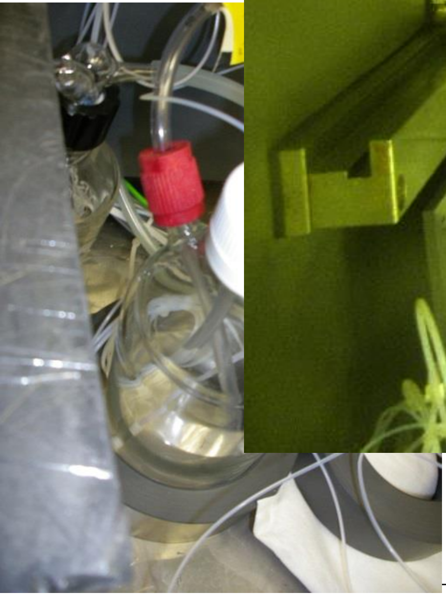
1. Elution from the LEWATIT ion exchanger with 2M HCl
2. Purification of the ^{7}Be fraction (^{22}Na, ^{88}Y, ^{54}Mn, ^{10}B, ^{7}Li)

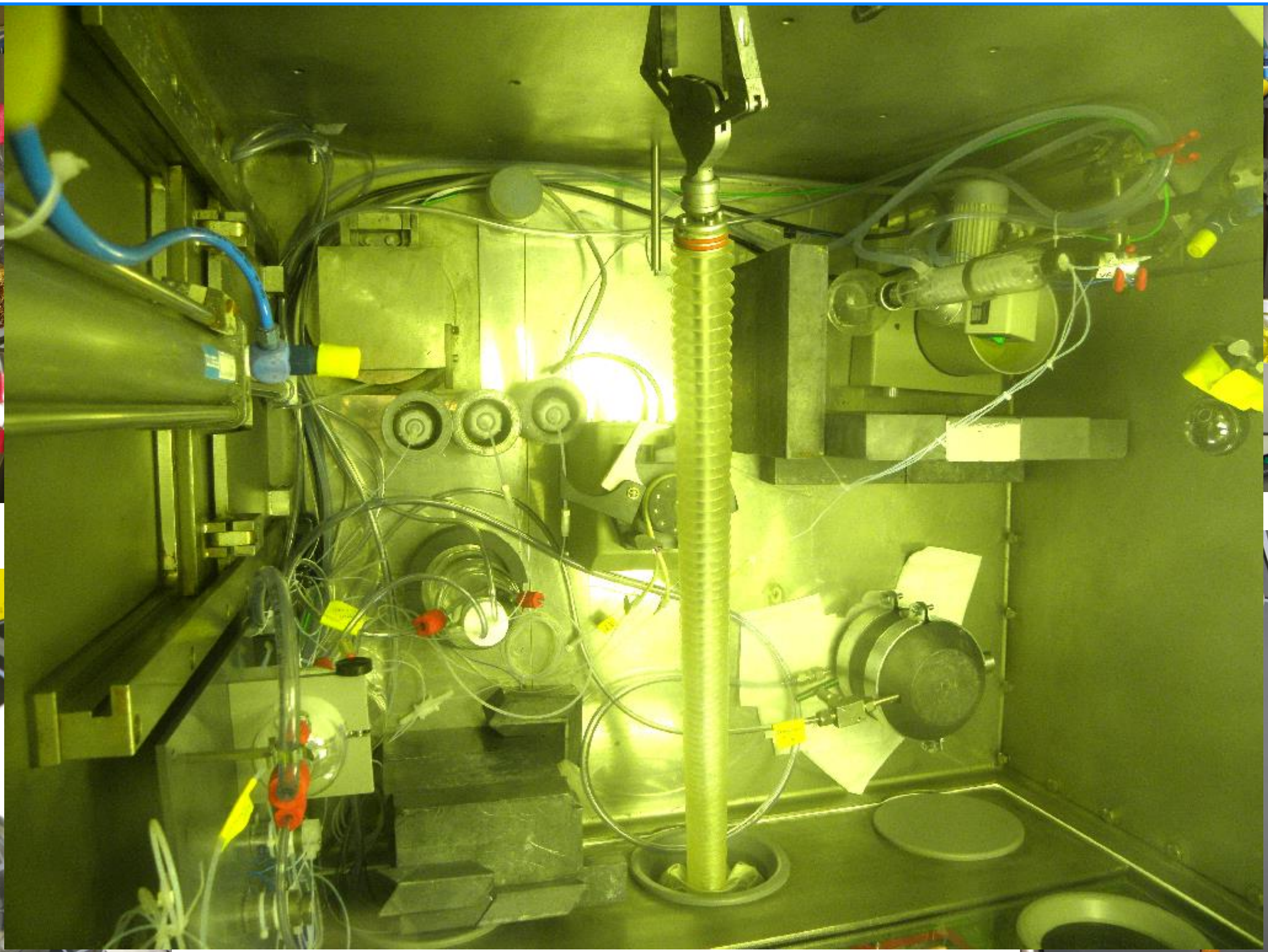


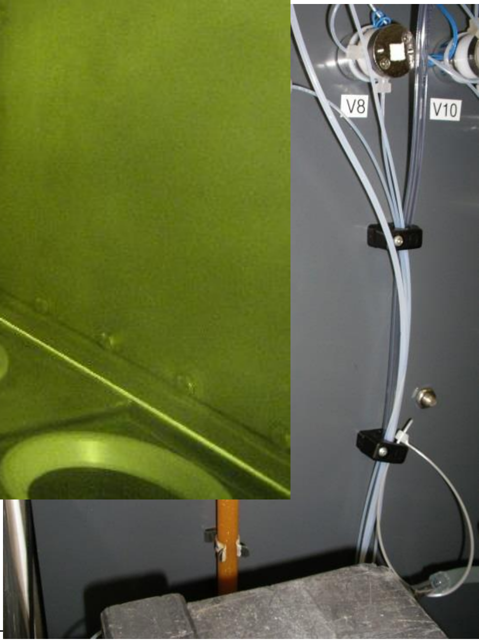
Procedure:

- Evaporation of the HCl solution
- Dissolution in 0.1M HNO_3
- Loading onto the column, washing with 0.1M HNO_3 – elution of ^{10}B (BO_3^-)
- Elution of ^{22}Na and ^{7}Li with 0.1M HNO_3
- Elution of ^{7}Be
- Elution of ^{88}Y, ^{54}Mn and others

Separation Scheme for the ^{7}Be separation







~ 200 GBq ^{7}Be can be processed in one batch

Contains comparable amounts of $^{9/10}Be$

Planned experiments with ^{7}Be at n_TOF EAR2

Proposal for the ISOLDE and Neutron Time-of-Flight Committee

Measurement of ^{7}Be(n,α)^{4}He and ^{7}Be(n,p)^{7}Li cross sections

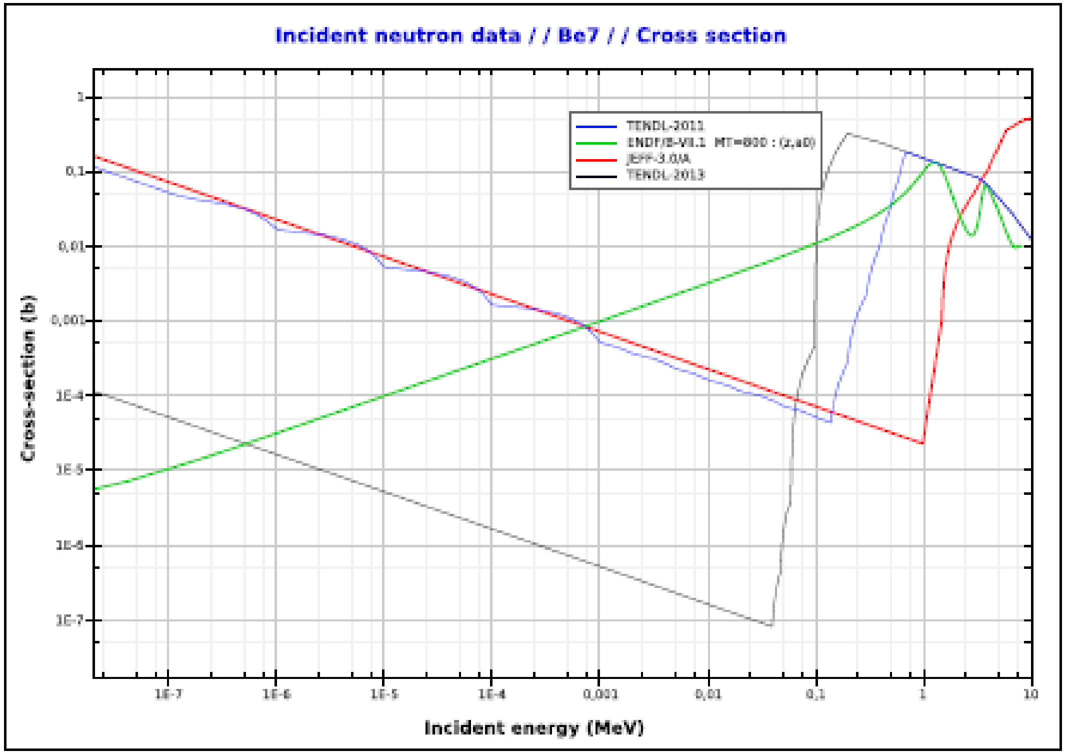


Figure: Comparison of the evaluated cross section of the ^{7}Be(n,α)^{4}He reaction in various libraries, from thermal neutron energy to 1 MeV

^{7}Be(n,α)^{4}He:

Very low cross sections, high amounts of ^{7}Be necessary (~ 1-10 μg, 10^{17}-10^{18} atoms, 10-100 GBq))

Impurities of $^{9/10}$Be have only slight influence

Background suppression possible

^{7}Be(n,p)^{7}Li:

Higher cross sections, less amounts needed (~ 0.1 μg - 10^{16} atoms, 1 GBq)

Impurities of $^{9/10}$Be have more influence

Background suppression more difficult due to lower energy of the proton

Mass separation necessary!

Second joint experiment ISOLDE - n_TOF

Previous ^{7}Be target preparation at ISOLDE

^{7}Be(p,γ)^{8}B: importance for the physics of the Sun and the evaluation of the solar neutrino flux

First target:
Online production
1GeV protons on graphite
extraction of $1.8\cdot10^{10}$/s ^{7}Be atoms at 60 keV
implanted in Cu ($5\cdot10^{15}$ ^{7}Be atoms)

Second target:
Irradiated graphite from target M (PSI)
$1.17\cdot10^{16}$ ^{7}Be atoms

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Precision Measurement of the ^{7}Be$(p,\gamma)^{8}$B Cross Section with an Implanted ^{7}Be Target

L. T. Baby,[1] C. Bordeanu,[1,*] G. Goldring,[1] M. Hass,[1] L. Weissman,[2] V. N. Fedoseyev,[3] U. Köster,[3] Y. Nir-El,[4] G. Haquin,[4] H.W. Gäggeler,[5] R. Weinreich,[5] and ISOLDE Collaboration
[1]*Department of Particle Physics, Weizmann Institute of Science, Rehovot, Israel*
[2]*NSCL, Michigan State University, East Lansing, Michigan 48824*
[3]*ISOLDE, CERN, Geneva, Switzerland*
[4]*Soreq Research Centre, Yavne, Israel*
[5]*Paul Scherrer Institute, Villigen, Switzerland*
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A new measurement of the ^{7}Be(p,γ)^{8}B cross-section with an implanted ^{7}Be target

M. Hass [a,1], C. Broude [a], V. Fedoseev [b], G. Goldring [a], G. Huber [c], J. Lettry [d], V. Mishin [b], H.J. Ravn [d], V. Sebastian [c], L. Weissman [e], ISOLDE Collaboration [d]
[a] *The Weizmann Institute of Science, Rehovot, Israel*
[b] *Institute of Spectroscopy, Troitzk, Russia*
[c] *Johannes Gutenberg University, Institute of Physics, Mainz, Germany*
[d] *ISOLDE, CERN, CH-1211 Geneva, Switzerland*
[e] *Instituut voor Kern-en Stralingsfysica, KU Leuven, Leuven, Belgium*

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Summary and outlook

ERAWAST provides rare exotic isotopes in sufficient amounts for several nuclear physics experiments

- ^{60}Fe half life and neutron capture cross section
- ^{44}Ti $^{44}Ti(\alpha,p)^{47}V$ reaction
- ^{10}Be from graphite targets; for p-induced reactions (Denmark), neutron capture (FRANZ)
- ^{63}Ni separation of the decay product ^{63}Cu; target for n_TOF
- ^{207}Bi calibration source for PTB
- and many others

Two proposals for joint experiments: ISOLDE – n_TOF

- Neutron capture cross section of ^{53}Mn at n_TOF
- Measurement of $^{7}Be(n,\alpha)^{4}He$ and $^{7}Be(n,p)^{7}Li$ cross sections