

CCMX Competence Centre for Materials Science and Technology





Wir schaffen Wissen – heute für morgen

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Neutron Imaging Detectors



Outline

- A bit of history
- Present state-of-the-art detectors based on neutron sensitive scintillators and light-collection
- Scitnillators a bit detailed look into them
- Alternative detector options
- Outlook



A bit of history





Have you ever seen a neutron?

 1932 – Chadwick discovers the «HOLY» particle

 late 1938 – Kallmann & Kuhn take a first neutron image

[unpublished]



First neutron image to be found in literature



Dec 1944 – Otto Peter

Neutronen-Durchleuchtung

Von Otto Peter

Aus der Forschungsanstalt der Deutschen Reichspost, Berlin

(Z. Naturforschg. 1, 557-559 [1946]; aus Rottweil a. N. eingegangen am 21. Juni 1946)

Erstmalig werden Durchleuchtungsaufnahmen mit langsamen Neutronen gemacht. Zur photographischen Wirksamkeit braucht man einen "Strahlenwandler". Die Bilder zeigen infolge der größeren Strahlenquelle nicht die Schärfe der 7-Strahl-Durchleuchtungen, erlauben jedoch neue Anwendungen.

Zeitschrift fuer Naturforschung (1946) 1, 557-559 http://zfn.mpdl.mpg.de/data/1/ZfN-1946-1-0557.pdf



https://www.youtube.com/watch?v=WOVEy1tC7nk



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Definitions

Definitions defined by international standards

analog image

- an image produced by a continuously variable physical process (e. g. exposure of film)

autoradiograph

- an image of an object containing a radioelement obtained, on a recording medium, by means of its own radiation.

digital image

- an image composed of discrete pixels each of which is characterized by a digitally represented luminance level.

image processing

- a method whereby digital image data is transformed through mathematical function.

neutron radiography

- the process of producing a radiograph using neutrons as penetrating radiation.

neutron radiograph

- a permanent, visible image on a recording medium produced by neutrons passing through the material being tested.

neutron radiology

- the science and application of neutrons.

neutron tomography

- any neutron radiologic technique that provides an image of a selected plane in an object to the relative exclusion of structures that lie outside the plane of interest.

Other definitions

neutron imaging

- the term summarizes all advanced techniques using neutrons as a probe which are currently under development and not yet certified for industrial and commercial use.

From <u>www.isnr.de</u> (Intl Society for Neutron Radiography)



• Film + neutron-converter



A plate/layer converting neutron to a particle that blackens film (Gd, Dy, In, Ag)

- Standardized technique
 - ASTM Standard Practice for Thermal Neutron Radiography of Materials (ASTM-E-748)
 ASTM Standard Method for Determining Image Quality in Direct Thermal Neutron Radiographic Testing(CD II537)
- Application used nuclear fuel rods (www.inl.gov)



- No direct excitation possible (missing charge of the neutron)
- Conversion process needed (capture, nuclear collision)
- Prominent converter materials: Gd, B-10, Li-6, He-3, U-235

• Secondary particles or radiation initiate light emission, charge transfer or other measurable excitation



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^{3}He + ^{1}n \Rightarrow ^{3}H + ^{1}p + 0.77 MeV
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⁶Li + ¹n \Rightarrow ³H + ⁴He + 4.79 MeV

¹⁰B + ¹n \Rightarrow ⁷Li + ⁴He + 2.78 MeV (7%) \Rightarrow ⁷Li* + ⁴He + 2.30 MeV (93%)

¹⁵⁵Gd + ¹n \Rightarrow ¹⁵⁶Gd + γ (7.9 MeV) + conversion electrons ¹⁵⁷Gd + ¹n \Rightarrow ¹⁵⁸Gd + γ (8.5 MeV) + conversion electrons

²³⁵U, ²³⁹Pu + ¹n \Rightarrow fission products + 80 MeV



Neutron Imaging Devices – stand 2004





Neutron Imaging Devices – stand 2015





Parameters of CCD cameras

- Effective number of pixels (e.g. 2048 x 2048, even 16k x 16k exist)
- Pixel size can go down to few micrometres (4.0 µm)
- Chip size pixel size times effective number of pixels

• Dark current noise – created from the thermal energy within silicon lattices comprising the CCD (as low as 0.01 e^{-1}/s) => That's why we cool the chip[©]

- Readout noise e.g. 1.6 e⁻ (either rms or median value)
- Total noise sqrt(readout noise² + dark noise²)
- Full well capacity largest charge a pixel can hold before saturation (e.g. 30'000 e⁻)
- Dynamic range ability of a camera to record simultaneously very low light signals alongside bright signals (Full well capacity/mean readout noise) e.g 18'000:1
- A/D converter 8 bit, 12 bit, 16 bit
- Temporal resolution CCD down to 10 µm regime up to 100s seconds
- Quantum efficiency incident photon to converted electron ratio
- Operating conditions, (temperature, triggers, interfaces)



Neutron or photon/electron statistics?

• It is important to realize what is the limiting factor of your CCD-scintillator set up?

Number of counts detected by one pixel

Vs.

Number of neutrons detected by the scintillator area corresponding to one pixel

> 1









CCD-detector MIDI (middle positions)





Micro-Tomography-Setup @ ICON





Neutron microscope – prototype 1.01



Trtik et al. Physics Procedia (2015) Neutron Microscope prototype 1.01 at BOA beamline





MICRO-setup vs Neutron Microscope

MICRO-setup



Neutron Microscope

Neutron Microscope Prototype 1.01

Neutron Microscope

Design and optimization of:

- Tailored magnifying optics
- High-resolution high-performance scintillators
- High-resolution high-sensitivity camera
- Tailored neutron optics for (2D samples)
- Neutron collimator on the basis of MCPs
- High-precision sample positioning stage
- Neutron-sensitive resolution test objects



Applications

Current frontiers of neutron imaging:

- Fuel Cell research
- Battery research and development
- Material failure and defect analysis
- Liquid transport in porous media
- Nuclear safety materials research

• Any other we are not aware of yet All enjoying complementarity of neutrons and X-rays





Design of final instrument

- to be installed on ICON and BOA
- user operation from 2016/17
- target resolution below 5 µm



Fonds national suisse Schweizerischer Nationalfonds Fondo nazionale svizzero Swiss National Science Foundation



- P. Trtik and E.H. Lehmann, NIM-A (2015)
- P. Trtik, et al., Physics Procedia (2015)

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Outlook



- High energy radiation creates free electrons and holes
- Holes convert to the top of the valence band and highly excited electrons convert (inelastic- / elastic scattering, etc.) to the bottom level of the conduction band
- The so-called thermalized electrons and holes can be captured by different traps (defects etc).).
- By recombination of the thermalized electrons and holes part of the energy is transferred to the emission center and lead to the observed luminescence.
- The fluorescence intensity of a phosphor is furthermore dependent from several factors:
 - Energy levels of the emitting ion in the crystal structure
 - Crystal structure (disorder, inhomogeneity, ...)
 - Purity of the phosphor (impurities)



Nikl, J o Luminescence (2006) review of X-ray scintillators

Slide courtesy of Walfort (RC TRITEC) et al., WCNR-10, (2014)



- Efficiency of the neutron capture (neutron stopping power)
- Light yield per neutron (ph/n)
- Emission spectrum
- Rise time, decay time and afterglow (persistance)
- Long-term stability of the above properties (degradation mainly due to radiation)
- Other relevant properties (stability in various environments, e.g. hygroscopicity)



- Efficiency of the neutron capture (neutron stopping power)
 - thickness (the thicker screen, the larger stopping power)
 - Cross-section (the larger the cross-section, the larger the stopping power)



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- Light yield per neutron (ph/n)
 - intrinsic number for the bulk material (e.g. for ZnS:Ag = 160'000 ph/n)
 - in reality, it is a function of thickness of the screen, microstructure of the scintillator screen, phosphor material properties (e.g emission spectrum, refractive index)





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- Efficiency of the neutron capture (neutron stopping power)
- Light yield per neutron (ph/n)
- Emission spectrum



THE EMISSION SPECTRUM IS CRUCIAL FOR (i) EFFICIENCY OF CCD (ii) DESIGN OF THE OPTICS



WAVELENGTH (Nanometers)





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- Rise time, decay time and afterglow (persistance)
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MUST BE STORED IN SEALED CONDITION



Neutron-sensitive scintillators

- Neutron scintillators convert neutrons to visible light
- There are two commonly used materials for neutron-senistive scintillatoin
- ⁶LiF/ZnS:(dopant e.g Ag)
- Gd₂O₂S:(dopant e.g. Tb)





⁶LiF/ZnS:(dopant e.g. Ag, Cu)

- Two (three) components scintillator
- neutron absorber (⁶LiF) ==
- light emitter
- (in/organic binder)

⁶Li + n ⇒ ³H (2.73 MeV) + ⁴He (2.05 MeV)

- Relatively low neutron capture efficiency
- Very high light output (for Ag doped material 160'000 ph/n)
- The range of ³H (2.73 MeV) ~10s μm
- The range of ${}^{4}\text{He}$ (2.05 MeV) ~ up to 10 μm









⁶LiF/ZnS:(dopant e.g. Ag, Cu)

- Light-output-optimized ratio of LiF to ZnS is 1:2
- The smaller thickness, the higher resolution
- The larger thickness, the higher light-output
- Self-shielding (saturation of light output) from about 400 µm





Walfort, Gruenzweig, Trtik, Hovind, Lehmann, WCNR-10 (2014)



⁶LiF/ZnS:(dopant e.g. Ag, Cu)

- Long term stability Double exponential decay
 - -Fast: oxidation of highly excited ZnS => passivation layer

-Slow: Degradation of ZnS crystal strucutre (sulphur vacancies)





Walfort, Gruenzweig, Trtik, Hovind, Lehmann, WCNR-10 (2014)

Brightness degradation in electroluminescent ZnS:Cu

Abstract

Nathaniel E. Brese^{a,*}, C. Lane Rohrer^b, Gregory S. Rohrer^b

Monte Carlo simulations using crystal chemical constraints have been used to elucidate the copper diffusion mechanisms in copper-doped zinc sulfide. Relaxation around the $ZnS|Cu_{2-x}S$ interface allows facile copper diffusion, particularly in the presence of substitutional oxygen or sulfur vacancies. Copper diffusion is responsible for the brightness degradation in this electroluminescent material. Reducing sulfur vacancies and substitutional oxygen species are outlined as ways to reduce the luminescence decay. © 1999 Elsevier Science B.V. All rights reserved.

Brese et al., Solid State Ionics (1999) 123, 19-24



ZnS light output degradation (neutron images)





ZnS decay times (afterglow)



Trtik, unpublished data (2014)



Gadolinium Oxysulfide Gd₂O₂S:(dopant)

- Gd is the element of the highest crosssection for neutrons (~50'000 barns)
- 99.99% Neutron capture of ^{nat}Gd => 18.43% ¹⁵⁵Gd + 81.56% ¹⁵⁷Gd
- Gd₂O₂S:Tb is rather stable substance
- Number of ways how to produce Gadox



 ${}^{155}\text{Gd} + {}^{1}\text{n} \Rightarrow {}^{156}\text{Gd}^* \Rightarrow {}^{156}\text{Gd} + \gamma (7.9 \text{ MeV}) + \text{conversion electrons}$ ${}^{157}\text{Gd} + {}^{1}\text{n} \Rightarrow {}^{158}\text{Gd}^* \Rightarrow {}^{158}\text{Gd} + \gamma (8.5 \text{ MeV}) + \text{conversion electrons}$

- For each captured neutron in Gd, there is at least 1 released gamma-ray
- The conversion electrons are produced only in about 76% of the cases of the neutron capture





^{.).} Abduschukurov, Appl. Math., 4 (2013) 27-33

- About 75% of conversion electrons from Gd157 are of sub-35 keV energy
- The travel range in Gadox is in single micrometres domain => OK for very high spatial resolution

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Getting the light out of Gd₂O₂S:Tb



b Gadox+AI+WG 10µm Gadox 12000 12000 WG = Waterglass = Na_2SiO_3 Counts 00001 10000 8000 8000 6000 6000 Gadox(G) G+WG G+AI G+AI+WG 10 20 30 40 Thickness, um Kardjilov, NIM-A 651 (2011) 95-99

- The emission spectra of Gadox are dependent on the dopant content
- Refractive index of Gadox is about (n=2.3), many internal reflections within particles
- When matched with waterglass (n=1.52), the light is outcoupled more efficiently
- For natural Gadox, the self-shielding starts to be an issue at about 20 µm thickness

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"Long-term" stability of Gadox



- ICON, 80 mm aperture, and a focussing guide, i.e. about 2 x 10⁹ n cm⁻²s⁻¹ (e.g. after 6 hours 4.3 x 10¹³ n cm⁻²)
- About 2% decrease in the light output over 6 hours of exposure
- Only about 0.02% can be attributed to transformation of ¹⁵⁷⁽¹⁵⁵⁾Gd to ¹⁵⁸⁽¹⁵⁶⁾Gd

Available scintillators supplied by PSI and RC TRITEC





Base material	Emission	Dimension	Thickness	Comment
⁶ LiF / ZnS:Cu (ratio 1 / 2)	530 nm (green)	up to 400 x 400 mm	50 up to 400 μm	High light output and high resolution
⁶ LiF / ZnS:Ag (ratio 1 / 2)	450 nm (blue)	up to 400 x 400 mm	50 up to 400 μm	High light output and high resolution
Gd ₂ O ₂ S:Tb	447 / 549 nm (blue-green)	up to 100 x 150 mm	10 up to 40 μm	Very high resolution
Gd ₂ O ₂ S:Tb / ⁶ LiF	447 / 549 nm (blue-green)	up to 100 x 150 mm	10 up to 50 μm	Very high resolution with enhanced intensity

- Addition of ⁶LiF gives about 30-50% higher brightness with same spatial resolution³
- If interested in the purchase, please contact us without hesitation



	Gd-152	Gd-154	Gd-155	Gd-156	Gd-157	Gd-158	Gd-160	Gd-nat
Content [%]	0.20	2.16	14.72	20.41	15.64	24.86	22.02	
X-section [b]	735	85	61'100	1	259'000	2	1	49'700

- ¹⁵⁷Gd has the highest cross-section for cold and thermal neutrons of all stable isotopes
- The ratio between neutron absorption cross-sections of ¹⁵⁷Gd and ^{nat}Gd is approximately 5.

	Nuclear Instruments and Methods in Physics Research A 788 (2015) 67–70
Isotopically-enriche screens for the hig	ed gadolinium-157 oxysulfide scintillator (OcrossMark h-resolution neutron imaging
Pavel Trtik [*] , Eberhard H Neutron Imaging and Activation Group, L	I. Lehmann aboratory for Neutron Scattering and Imaging, Paul Scherrer Institut (PSI), CH-5232 Villigen, Switzerland
ARTICLE INFO	ABSTRACT
Article history: Received 23 February 2015 Received in revised form 26 March 2015 Accepted 26 March 2015 Available online 3 April 2015	We demonstrate the feasibility of the production of isotopically-enriched gadolinium oxysulfide scintillator screens for the high spatial-resolution neutron imaging. Approximately 10 g of $^{157}Gd_2O_2S$: Tb was produced in the form of fine powder (particle size approximately 2 μ m). The level of ^{157}Gd enrichment was above 88%. Approximately 2.5 μ m thick $^{157}Gd_2O_2S$:Tb scintillator screens were produced and tested for the absorption power and the light output. The results are compared to the reference
Keywords: Neutron imaging Isotopic enrichment Gadolinium oxysulfide Neutron microscope	3.6 for the absorption power and the light output, respectively. The potential of the scintillator screens based on ¹⁵⁷ Gd ₂ O ₂ S phosphor for the purpose of the (high-resolution) neutron imaging is discussed. © 2015 Elsevier B.V. All rights reserved.
Scintillator Gd-157	



 6 scintillator plates (2.5 µm thickness) tested in each category using the MICRO-setup



Gain Factors :	Absorption Theoretical	÷ 4.6
	Absorption Measured	÷ 3.8
	Light Output Measured	÷ 3.6

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Spatial resolution using very thin Gadox







Alternative scintillators:

Ce-doped Li glass

Cs₂LiYCl₆(Ce) - CLYC

 $^{6}\text{LiGd}(\text{BO}_{3})_{3}$

GGG:Eu+ (Gadolinium Galium Garnet)

GAGG:Eu+ (Gadolinium Aluminium Galium Garnet)

Gdl₃:Ce

Lil(Eu) – Lithium Iodide

LISe (Lithium Indium Diselenide)

LNI (Lithium Sodium Iodide), LCI (Lithium Calcium Iodide)

Outlook – microstructured scintillators

- Well-known concept from X-ray imaging (e.g. Rutishauser, APL, 2011)
- Decoupling of the detector resolution from the scintillator thickness





• MC simulations of light output from microstructured neutron-sensitive scintillators



E. Cazalas, M. Morgano, C. David, E. Lehmann, P. Trtik, in preparation (2015)





Source: P. Boillat, Electrochemistry, PSI



"Tilted Scintillator adapter"





ANISOTROPIC PIXEL SIZE AND SPATIAL RESOLUTION OF IMAGES!!





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Gaseous detectors

3 He + 1 n \Rightarrow 3 H + 1 p + 0.77 MeV





Corner of electrode structure inside a 20cm×20cm detector, showing upper cathode wires, anode wires,





^{To} X_{-axis} C_{entroid} Finding Electronics</sub> B. Yu et al. / Nuclear Instruments and Methods in Physics Research A 513 (2003) 362–366

Rear view of a detector showing signal feedthroughs and preamplifiers, as well as the gas purification system.



Gaseous detectors

Inert gases (³He, ¹⁰BF₃)

Wire chambers

Large area available (~50x50 cm²)

High counting rate capability (up to 10⁸ n cm⁻² s⁻¹)

Very good temporal resolution

Moderate spatial resolution (0.5-1 mm FWHM)

Need to replace gas every 3-5 years (becomes very expensive)

(³He is becoming limited in availability, therefore change to ${}^{10}B_4C$)

High pressure (20 bar) – mechanical construction





Imaging plates

- Photostimulated luminescence by a phosphor excited by neutrons
- Imaging plates read by scanner
- Spatial resolution below 100 µm
- Thickness in single-cm regime
- Useful for only for "single-shot" images in constrained conditions





Images courtesy of Knittel (1998)



Neutron detection by color centre formation in LiF





- ⁽⁶⁾LiF crystals behind samples exposed
- •⁶Li + n ⇒ ³H (2.73 MeV) + ⁴He (2.05 MeV)
- The range of ³H (2.73 MeV) ~33 μm
- The range of ${}^{4}\text{He}$ (2.05 MeV) ~ 6 μm
- The color centres (F) are created by excitation of valence electron to the conduction band with energy 14 eV
- LiF crystal then transferred to a laser confocal microscope
- Moderate exposure times needed

Matsubayashi, Faenov, et al., NIM-A (2010) 622, 637-641 🔹 🚺

• Indirect method, spatial res. - 5.4 µm



Flat Panel Detectors

- Amorphous Si-panel
- Layer of X-ray/neutron sensitive scintillator
- Very radiation resistant©

	X-Ray		
Gtb02S			
a-Si TFE)	<u></u>	
		a-S _i TFT	
Gla	ss Substrate	•	833

- FPD with about 50 um pixel size are commercially available
- Temporal resolution up to 10 Hz.
- Thickness in single-cm regime
- See presentation by M. Raventos

Hybrid detector based on PILATUS/EIGER



Fig. 5. Layout of the PILATUS pixel detector, which is sensitive for neutrons by a 5 µm thick Gd-157 layer on the top.

- PILATUS / EIGER no dark current, no readout noise CMOS detector
- Single photon counting regime (used for coherent diffraction X-ray imaging)
- Absorber converts neutrons into electrons
- Pixel sizes (120 and 75 um, respectively)
- Limitations on the FOV

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• Spatial resolution limited by the charge spread in the absorber/ cross talk between the pixels



Fig. 7. Radiography image of a sprinkler nozzle made with different imaging systems, PILATUS (left), imaging plate (right).

E.H. Lehmann et al. / Nuclear Instruments and Methods in Physics Research A 531 (2004) 228-237



Hybrid based on PILATUS/EIGER detector







E.H. Lehmann et al. / Nuclear Instruments and Methods in Physics Research A 531 (2004) 228-237



Neutron counting MCP detectors







- Based on (MCP) multichannel plate
- Timepix detector (256 x 256 pixels, 55 μm pixel size)
- Each neutron detected: X, Y, Time
- Detection efficiency up to 50%
- Operated in vacuum

A. S. Tremsin, et al., *IEEE Trans. Nucl. Sci.* **52**, 1739 (2005) O.H.W. Siegmund et al., *Nucl. Instr. Meth. A* **576**, 178 (2007) A.S. Tremsin, et al., *Nucl. Instr. Meth. A* **592**, *p.374* (2008). A.S. Tremsin, et al., *Nucl. Instr. Meth. A* (2011)



Neutron counting MCP detectors



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- Detection of photons, ions, neutrons, alphas, high energy electrons, atoms.
- Detection efficiency for neutrons > 50% for thermal and cold neutrons, <1% for epithermal neutrons.
- Up to ~25000 simultaneous events can be detected.
- Active area 28x28 mm² (2x2 Timepix chips).
- Fast parallel readout (x32) allowing ~1200 frames per second with ~320 ms readout time
- Event centroiding (~12 µm resolution, at ~5x10⁶ events/s) or 55 µm resolution at >5x10⁸ events/s.
- Time resolution can be ~20 ns at ~2.5x10⁷ events/s rates with 55 µm resolution.
- Timing within frames TOF(energy) or dynamic processes can be studied. Wide energy range or most phases measured in one experiment.

TimePix hybrid detector



Fig. 2. TimePix device with 300µm thick Silicon sensor chip covered by a ⁶LiF converter with illustration of a neutron conversion in the converter layer.



Fig. 4. Cluster of pixels (left) caused by a single 5.5 MeV alpha particle. The detector was operated in TOT mode. The response of each pixel was translated to energy (see Fig. 1). The cluster is perfectly gaussian shaped (right).

Area: 14x14 mm² Limited

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High spatial resolution:

3.8 µm (low count rate)

Timing resolution: 10 ns





J. Jakubek, et al., "Energy (TOF) and Position Sensitive Detection of Ultra Cold Neutrons with Micrometric Resolution Using the TimePix Pixel Detector ", 2008 IEEE Nuclear Science Symposium Conference Record N42-1



WidePix hybrid detector



WIDE PIX4x5 10x10

- 1280x1024 pixels (6.5 Megapixel)
- 55 um pixel size
- 10 frames per sec



J. Jakubek et al., Czech Technical University



Future outlook

- Present limit of the scinitillator-and-camera neutron imaging is at about 5 µm
- Physical limit by intrinsic properties of the scintillator screen

New approaches:

• ILL beamport with foreseen flux 6 x 10⁹ n cm⁻² s⁻¹

• NIST neutron microscope based on neutron achromatic Wolter optics (*Liu, Hussey et al., APL, 2013*). Current resolution of this microscope 75 µm.

+ one crazy notion on the top:

How precisely are we able to detect inertial mass?

7 Zg (zeptograms) = 3000 Daltons = 7 x 10^{-21} g

Yang et al. 2006 Zeptogram-scale Nanomechanical Mass Sensing

¹³⁵Xe (2'000'000b)+ ¹n \Rightarrow ¹³⁶Xe (stable); ¹³⁵Xe instable t_{1/2}=9.2 hours \Rightarrow ¹³⁵Cs



- Neutron Detectors reviewed
- The scintillators are at the heart of the standard CCD/sCMOS-camera detectors
- There are still number of ways for the future development of higher-performance neutron-sensitive (microstructured) scintillators.

Thanks for several slides to: Eberhard Lehmann (PSI), Bernhard Walfort (RC TRITEC), Manuel Morgano (PSI), Anton Tremsin (UCBerkeley), Jan Jakubek (ADVACAM)