NANOPARTICLES ARE DIFFERENT

Synthesis, characterization and application of magnetic nanoparticles

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Acknowledgement

The organizers of this conference

- For the invitation
- The perfect organization
- Their extraordinary hospitality
- The marvelous weather

Outline of the talk

- **Why are nanoparticles interesting?**
- **X-ray absorption spectroscopy (XAS) with synchrotron radiation: some basics**
- **Characterizing nanoparticles (mainly Co) with XANES (and EXAFS)**
- **Biomedical applications of magnetic nanoparticles**

Why are nano-particles of interest/importance?

Nanoparticles

Clusters } **r = 1 – 10 (100) nm**

Colloids

- **“Mesoscopic systems” between atoms/ molecules and bulk solids**
- **Nanoparticles have very special properties (huge surfaces, quantum effects….!**
- **Hope and promise: The properties can be tailored by size, shape, “coating” etc. (????)**

Why are “nanoparticles” of interest/importance?

1. **“Huge” specific surface area (some Football-fields/g) →**

- High surface energy (Missing neighbors, catalysis…)
- Special surface properties (Bio- medical applications?...)

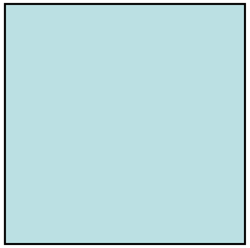
2. **Quantum effects:**

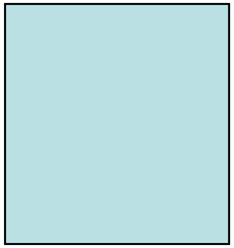
- ΔE = Energy-gap between valence - and conducting band is “size-dependent”
- Magnetic properties are “size-dependent”

3. **Properties can (in principle) be tailored” by modifying size, shape, composition, coating etc.**

Why are nanoparticles of special interest?

Cube → 10^{15} cubes



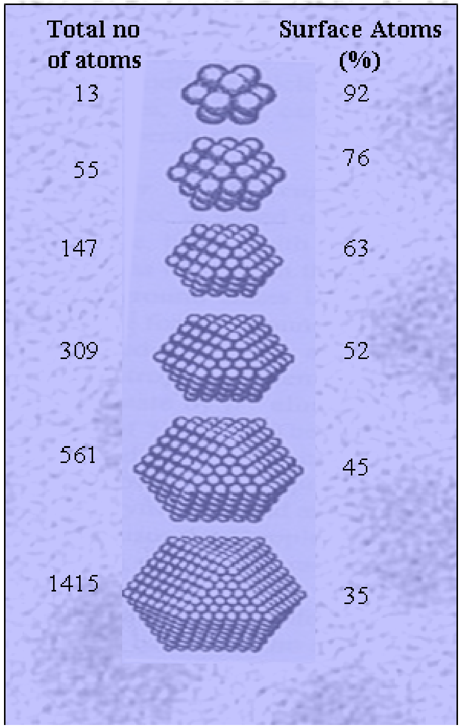


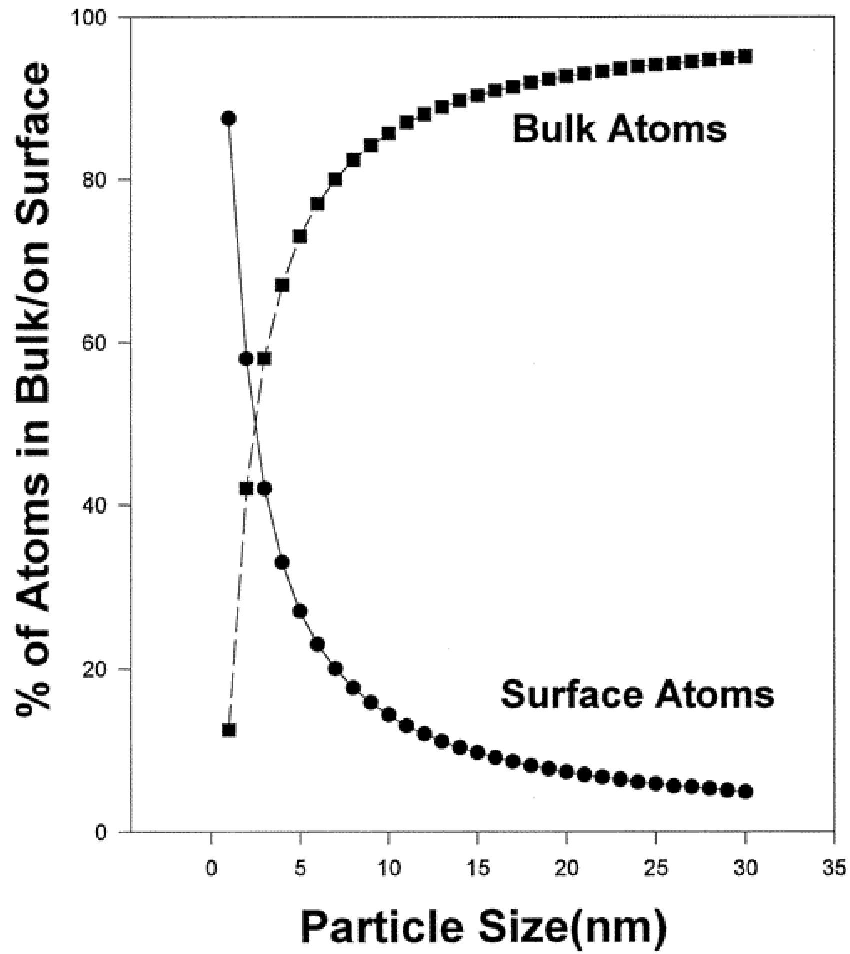
1cm → 100 nm

Surface-area: 6 cm^2 → 10^8 cm^2

(10^8 cm^2 = 100m x 100m is larger than a football-field!)

Properties of nanoparticles are determined by their surface:





- magnetic
- optical
- melting points
- specific heats
- surface reactivity
- catalytic

Why are "nanoparticles" of interest/importance?

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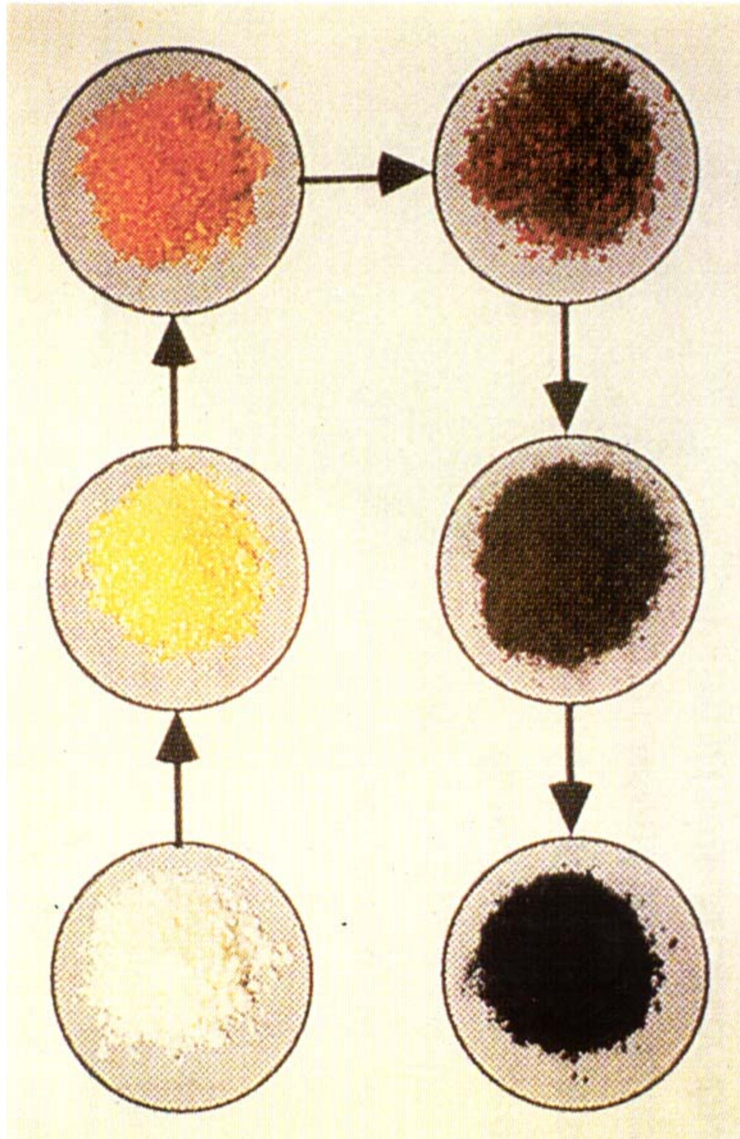
- Magnetic properties are "size-dependent"

3. **Properties can (in principle) be tailored" by modifying size, shape, composition, coating etc.**

Band gap as a function of particle size



Ø = 2 nm

Ø = 20 nm

Applications of nanoparticles in art

XVI century dish from Deruta with gold lustre decoration (Cu/Ag nanoparticles!) (Experiments at ESRF)

Why are "nanoparticles" of interest/importance?

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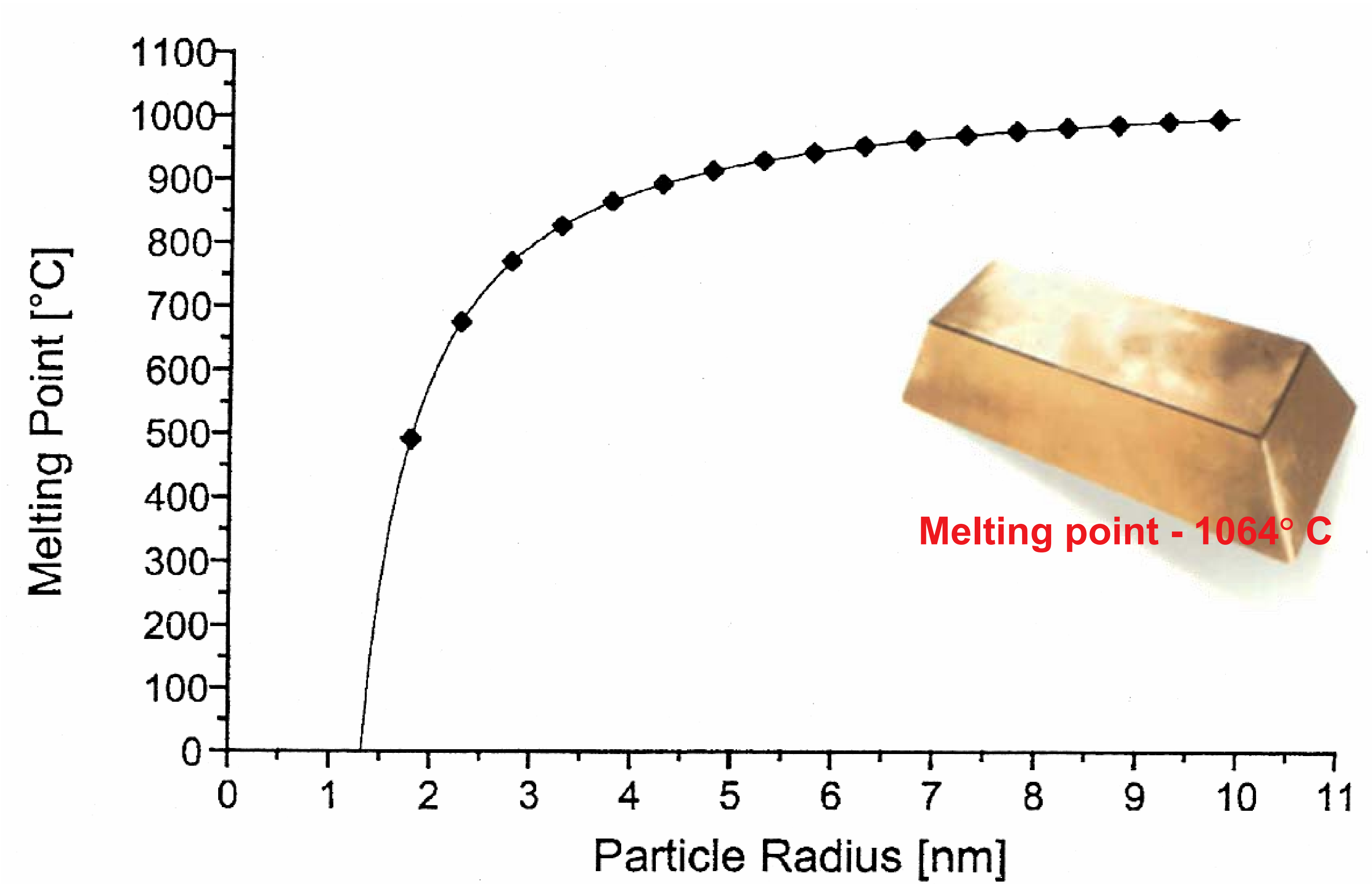
- High surface energy (Missing neighbors, catalysis…)
- Special surface properties (Bio- medical applications?...)

2. **Quantum effects:**

- ΔE = Energy-gap between valence - and conducting band is "size-dependent"
- Magnetic properties are "size-dependent"

3. **Properties can (in principle) be tailored" by modifying size, shape, composition, coating etc.**

Melting point of Au as fct. of particle size



Nanoresearch at CAMD

- Wet-chemical synthesis of (magnetic) nanoparticles

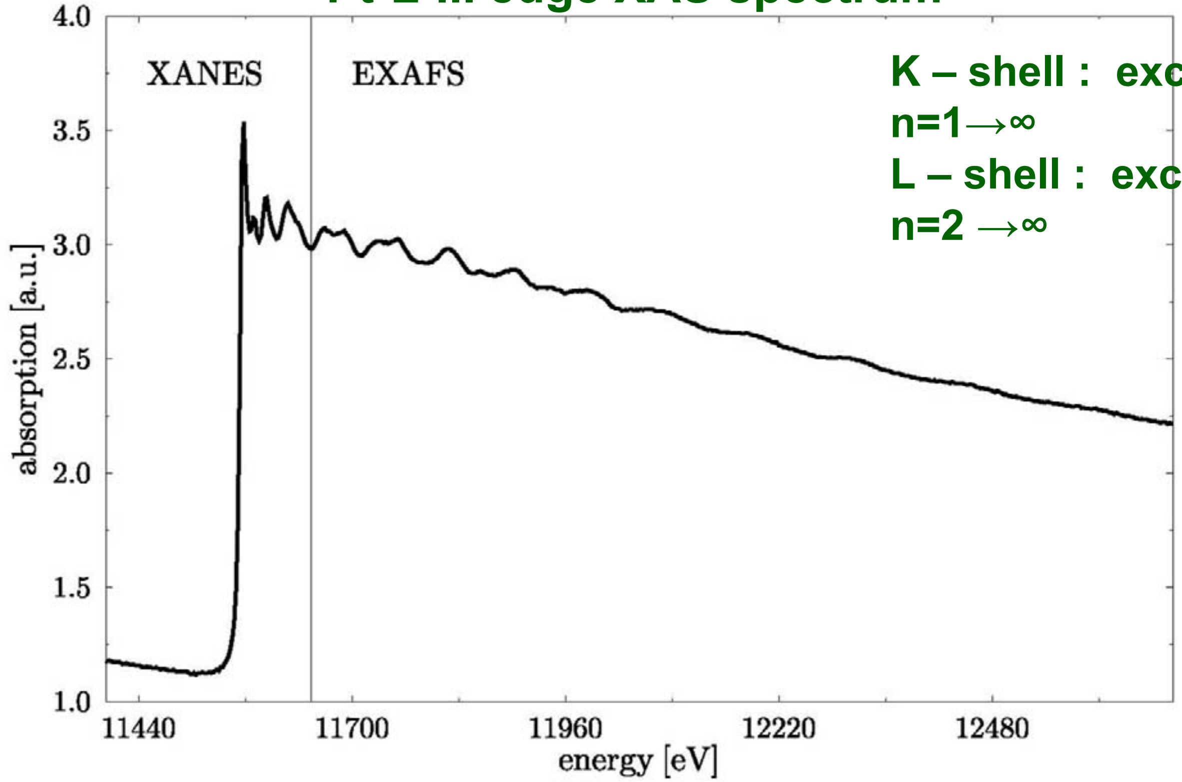
- Magnetic nanoparticles for drug delivery (Pennington Bio-medical Research Center)

- Sensor development for biological and chemical warfare agents based on GMR sensor + properly functionalized magnetic nanoparticles

- Microreactor for “continuous” synthesis of nanoparticles

Characterizing nanoparticles by X-ray absorption spectroscopy

Pt-L-III edge XAS spectrum



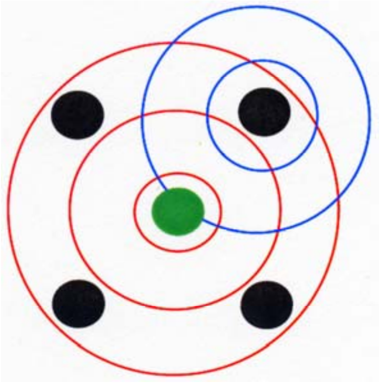
K – shell : excitation from n=1$\rightarrow\infty$

L – shell : excitation from n=2 $\rightarrow\infty$

XANES = X-ray absorption near edge structure

EXAFS = Extended X-ray absorption fine structure

The "EXAFS – theory"



- A) For a free atom: The wavefunction of the emitted electron is a spherical wave (k = 2π/λ = $[(4\pi m/h)E_{Kin}]^{1/2}$
- B) For a "bound" atom: There is an interference between the outgoing and the backscattered spherical wave
- Phase: ~ sin(2Rk) (R= distance "excited" atom – backscattering atom)
- Amplitude:~ N = number of backscattering atoms
  ~ Z = "type" of backscatterer

What can be learned from EXAFS spectra?

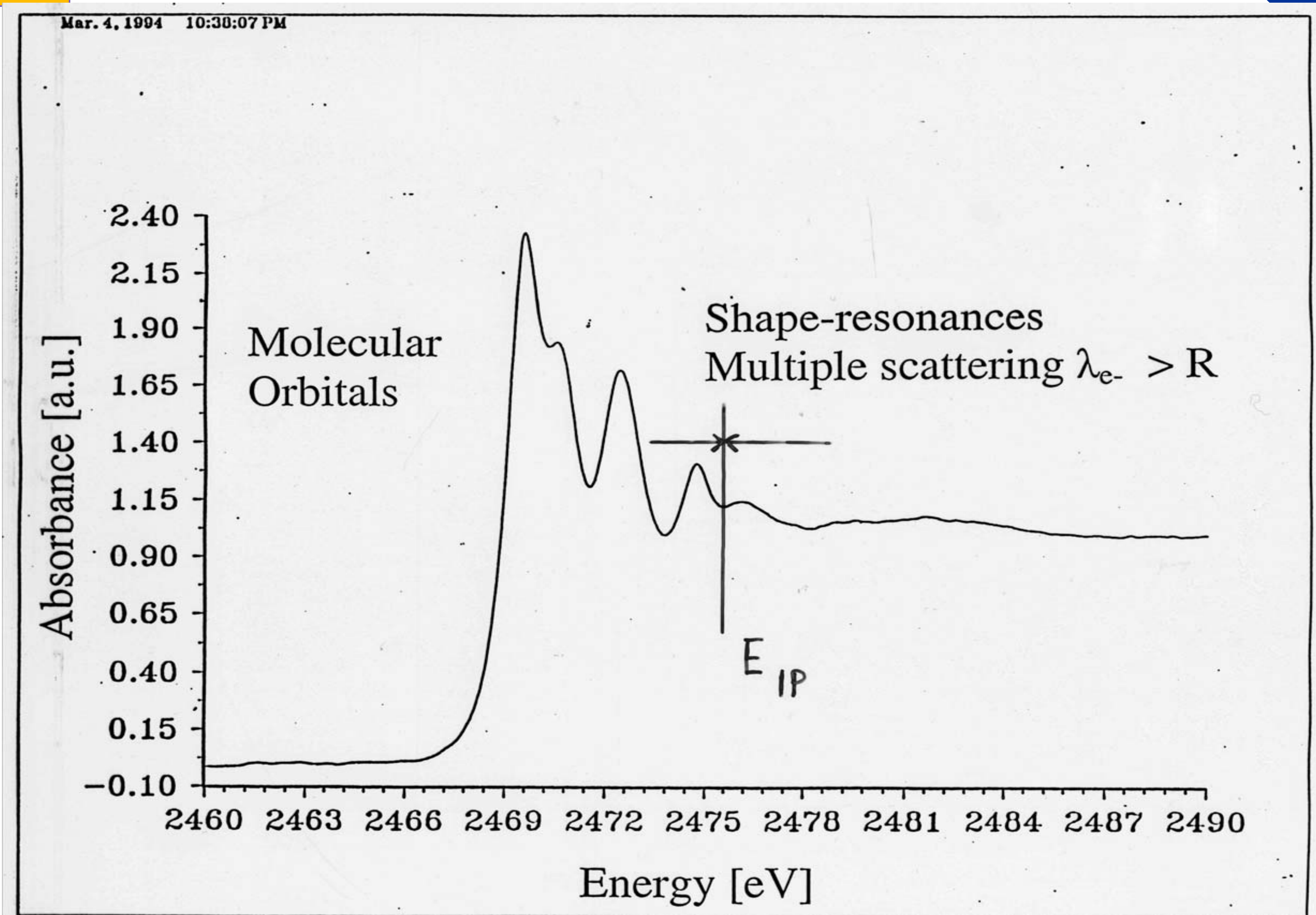
- Radial distances between the excited atom and the neighboring atoms in the “first” coordination shells (± 0,005Å → ± 0.01Å)

- Coordination number (± 25%)

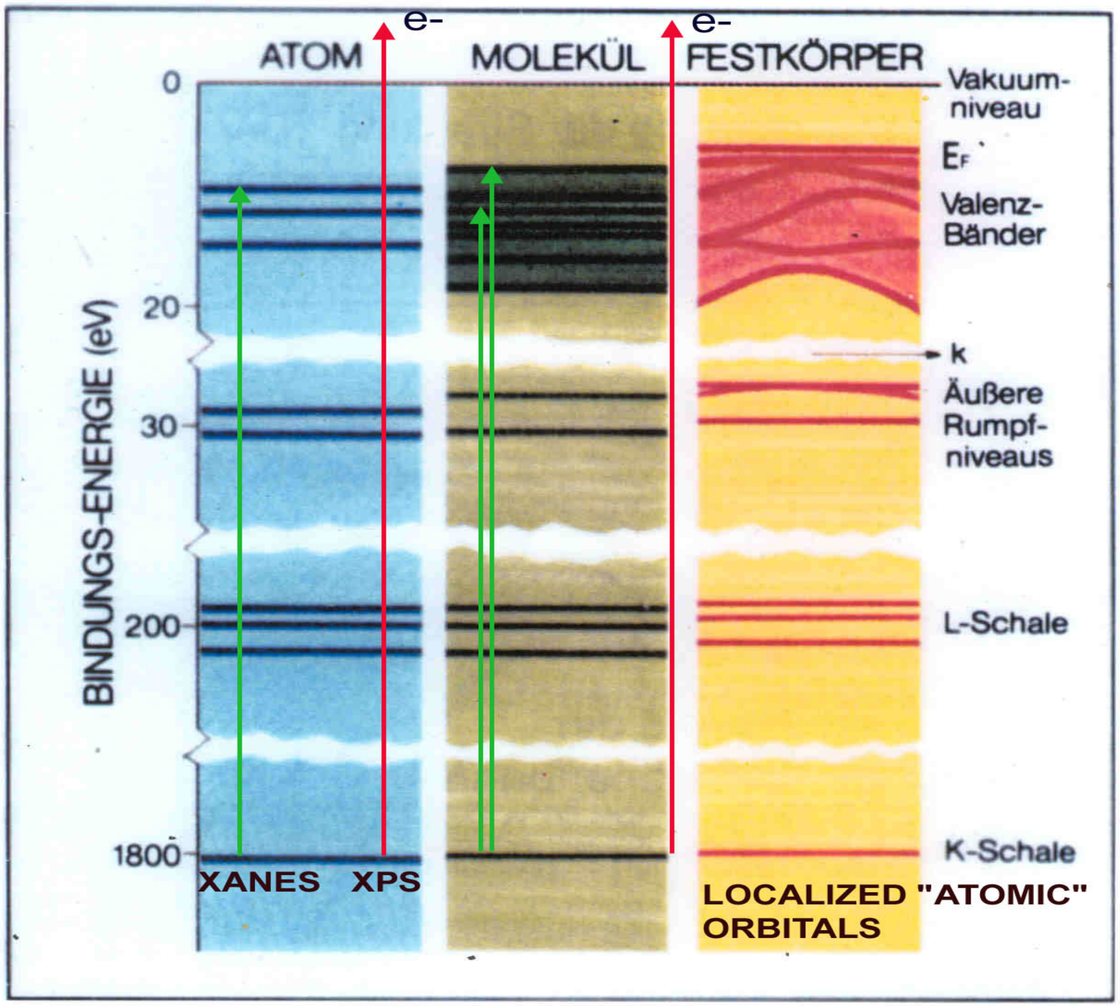
- “Type” of neighboring atoms Z (±5)

- “Information-depth” of EXAFS results is about 6 – 10 Å (Information about near range order!)

S-K-XANES: COS in the gasphase

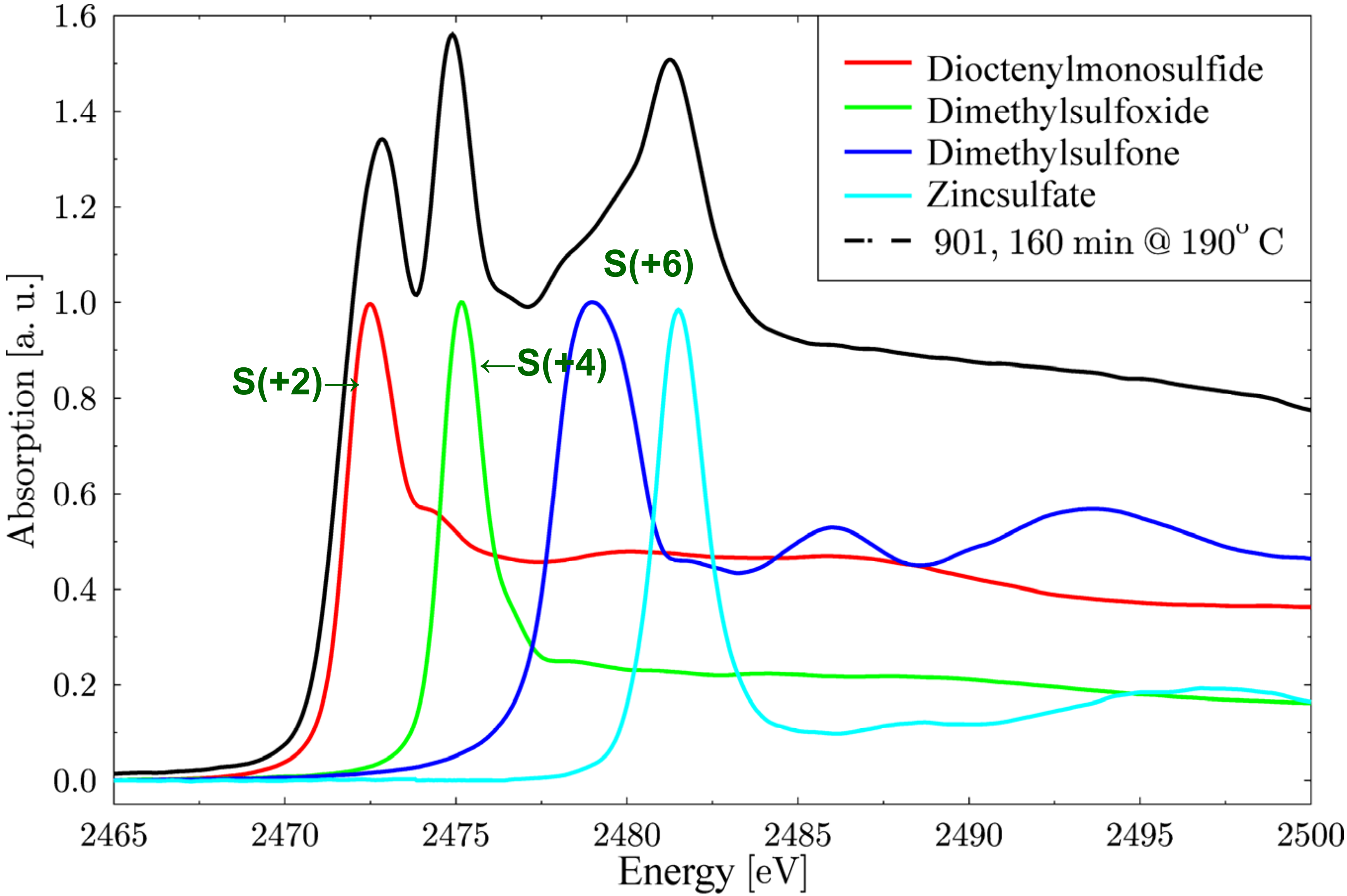


Physical basis of XANES spectroscopy

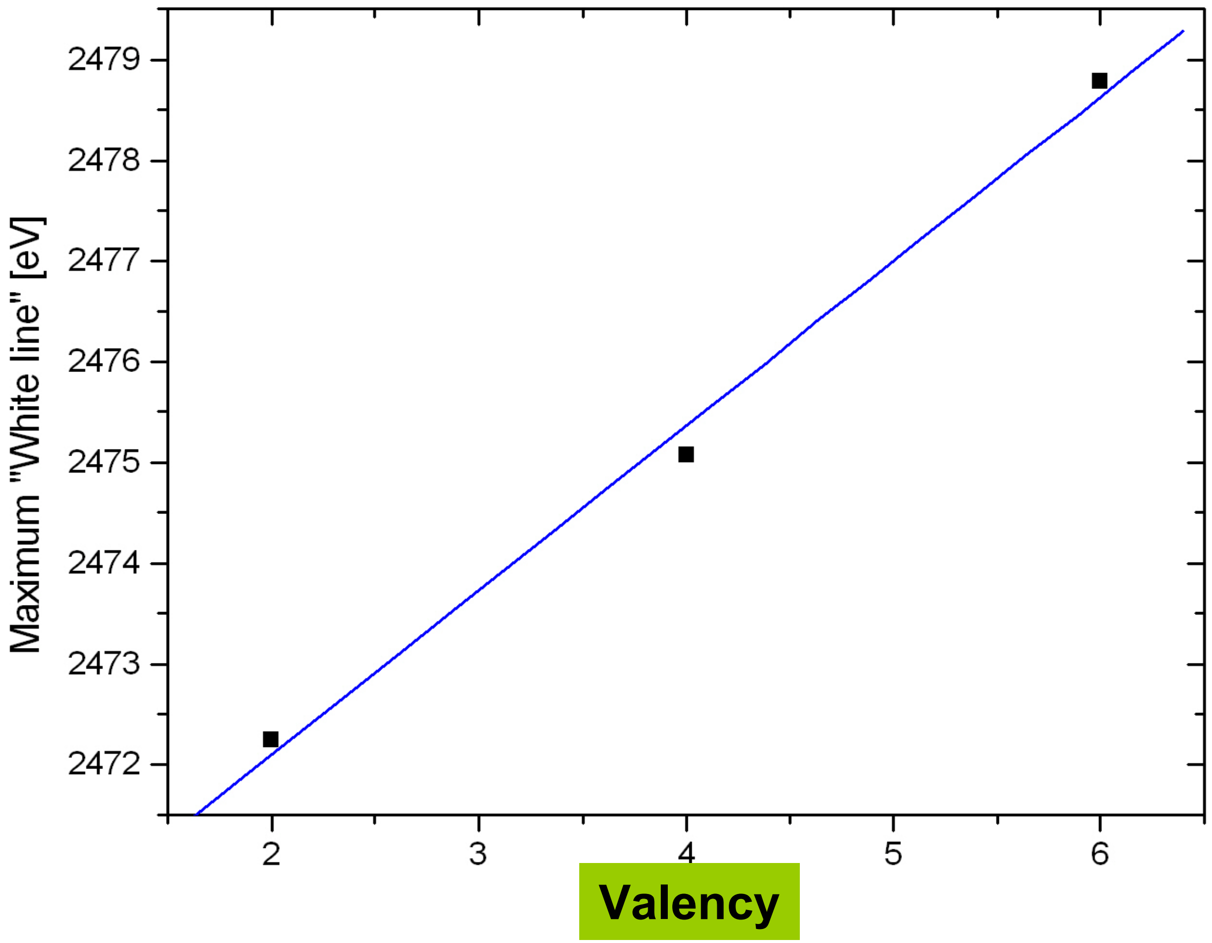


- **XPS** determines the shift of core shell levels (Chemical shift)
- **XANES** determines the difference between the shifts of the core levels and "empty" MOs and "empty bands" respectively

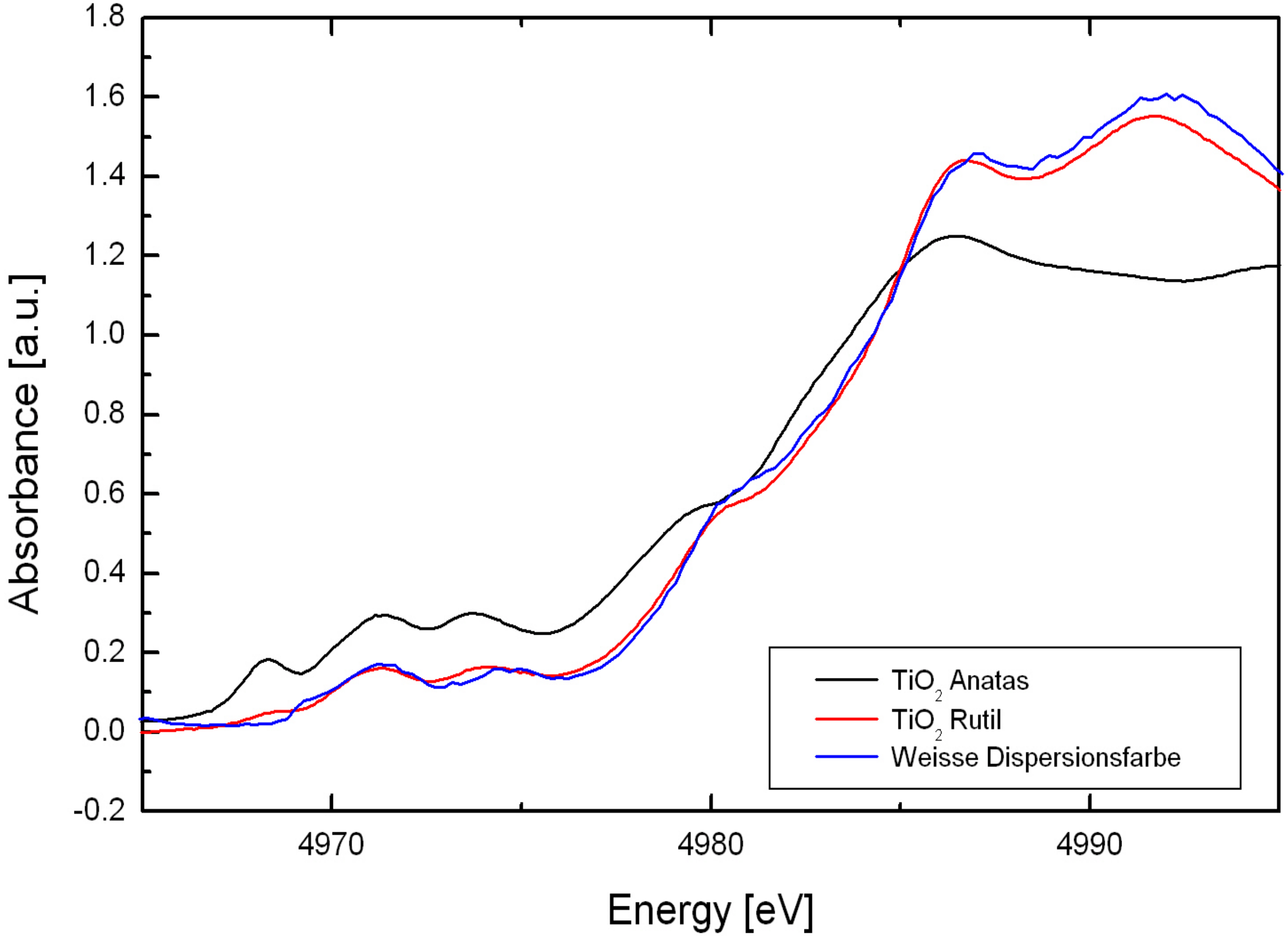
Influence of "valency" on S-K-XANES spectra (I)



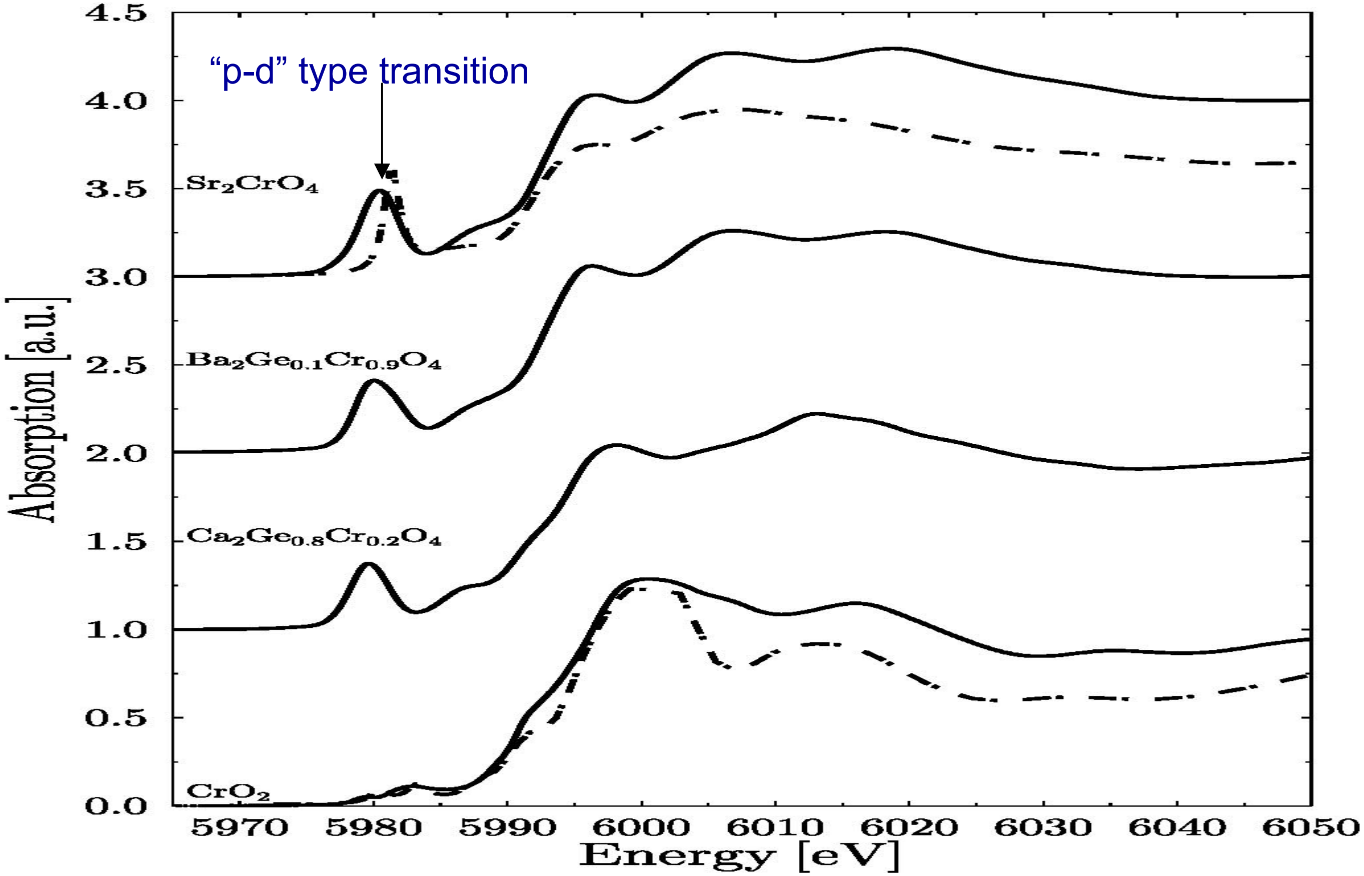
Influence of "valency" on XANES spectra (II)



Influence of the local geometry on XANES spectra (I)



Influence of the local geometry on XANES spectra (II)



- A. Pantelouris et al. Chem. Phys. 2004

What can be learned from XANES spectra?

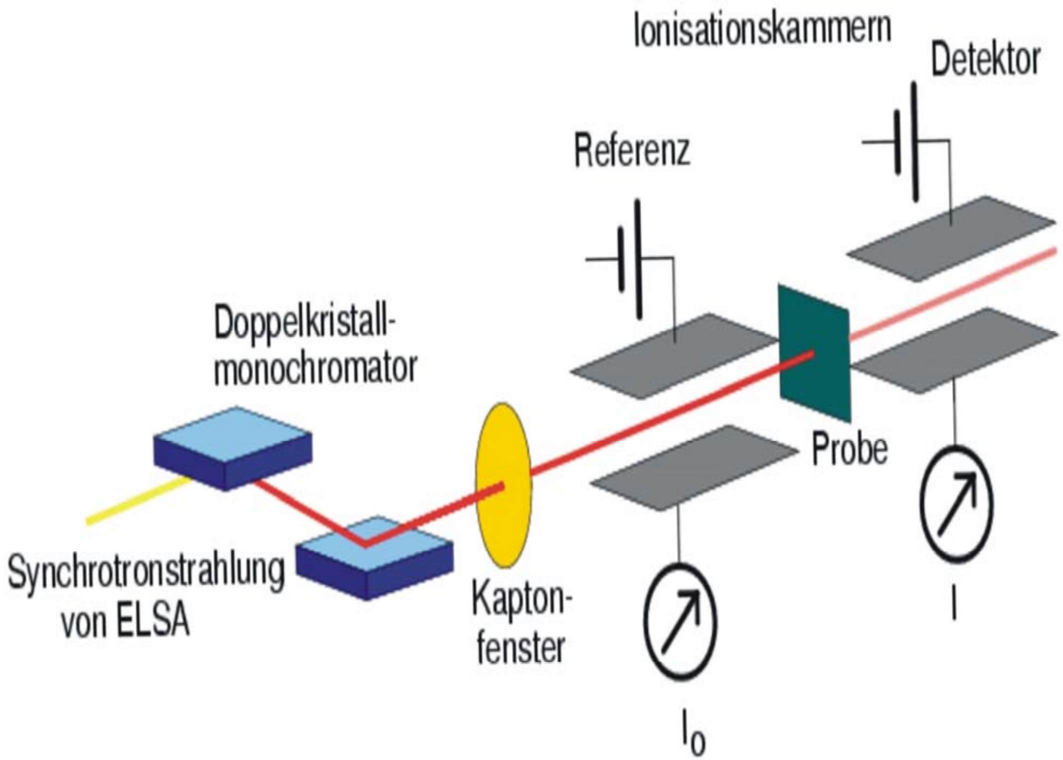
- Valency of the excited atom
- Symmetry of unoccupied electronic levels
- Elektronegativity of neighboring atoms
- “Band structure”
- (with FEFF 8 – calculations: DOS, Charge transfer etc.)

Advantages of XAS:

- No long range order is required in the sample
- The local surrounding of each type of atom can be investigated separately
- The investigation does not destroy the sample
- Due to the penetration strength of X-rays, measurements (at least in transmission and fluorescence mode) do not require a good vacuum and in many cases “real” in situ measurements are possible.

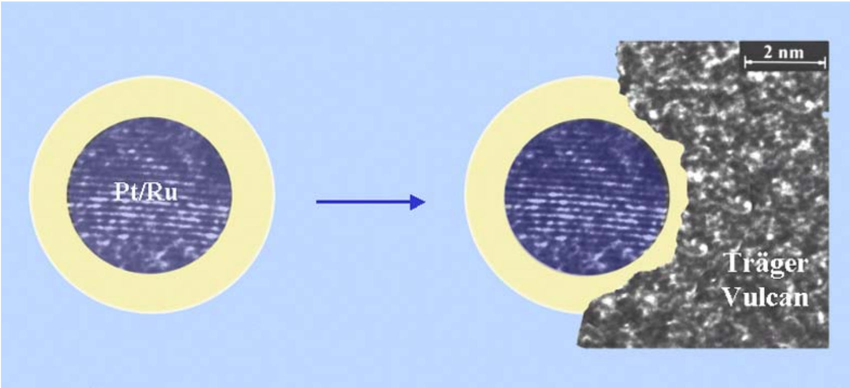
XAS: experimental set-up

XAS measures the energy dependence of the x-ray absorption coefficient *μ(E)* at and above the absorption edge of a selected element.



Scheme of experiment in transmission mode.

The structure of the investigated nanoparticles



- Metallic nanoparticles with organic protective layer
- (Prof. Bönnemann, CAMD (Dr. Kumar), Prof. Gedanken (Bar-Ilan University)

- Magnetic nanoparticles with metallic protective layer
- (Prof. Bönnemann, Prof. O'Connor, AMRI, UNO, CAMD (Dr. Kumar), LSU/Chem. Engineering)

Challa2.wmv

The electronic structure of metallic nanoparticles (2002/2003?)

Example of a XANES analysis

Problem:

The properties of nanoparticles are determined by their size/shape

→ goal: tailoring properties for special applications!

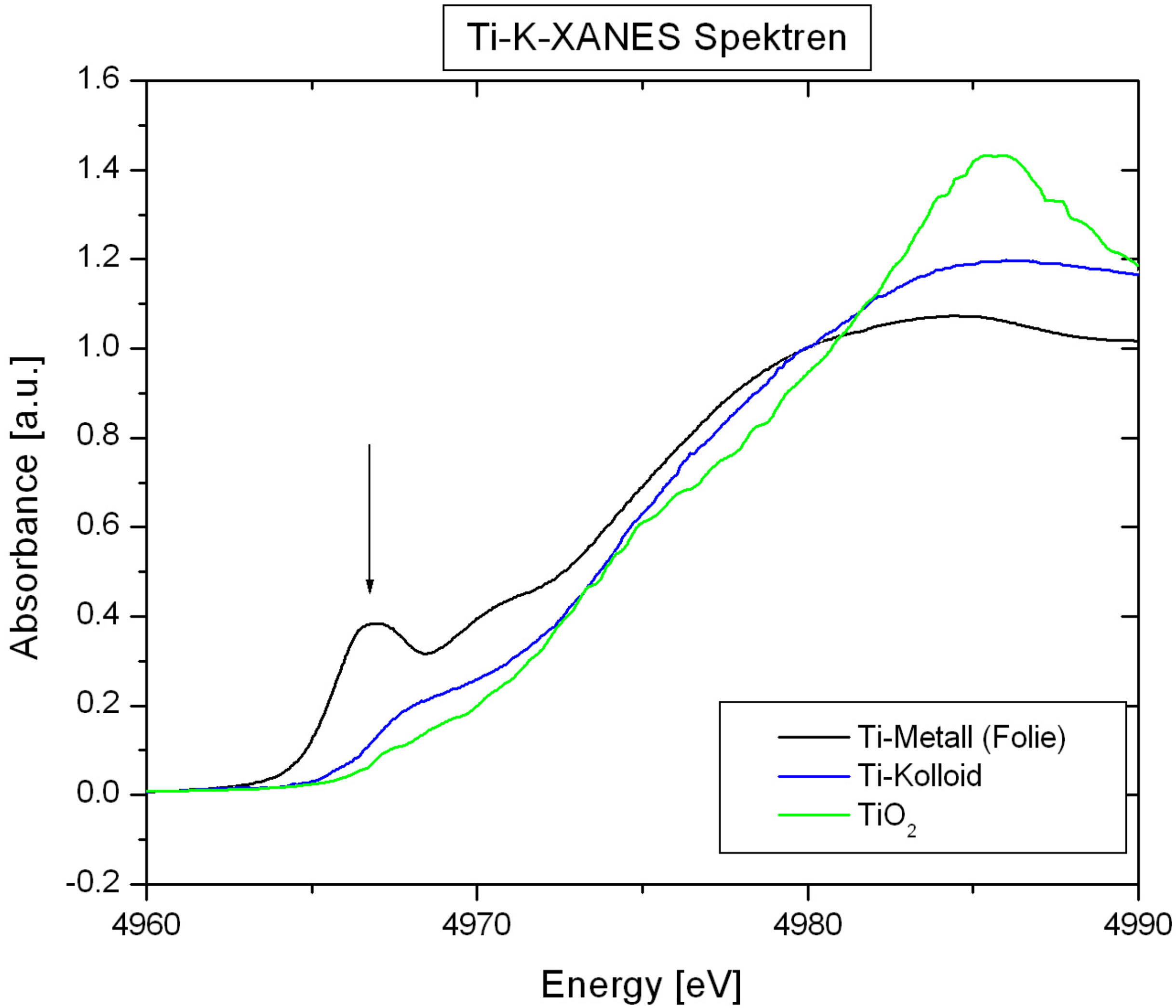
Task:

- Determining the valency of the metal
- Investigation: Electronic structure as a function of size
- Investigation: Interaction “Metal - core” – protective layer

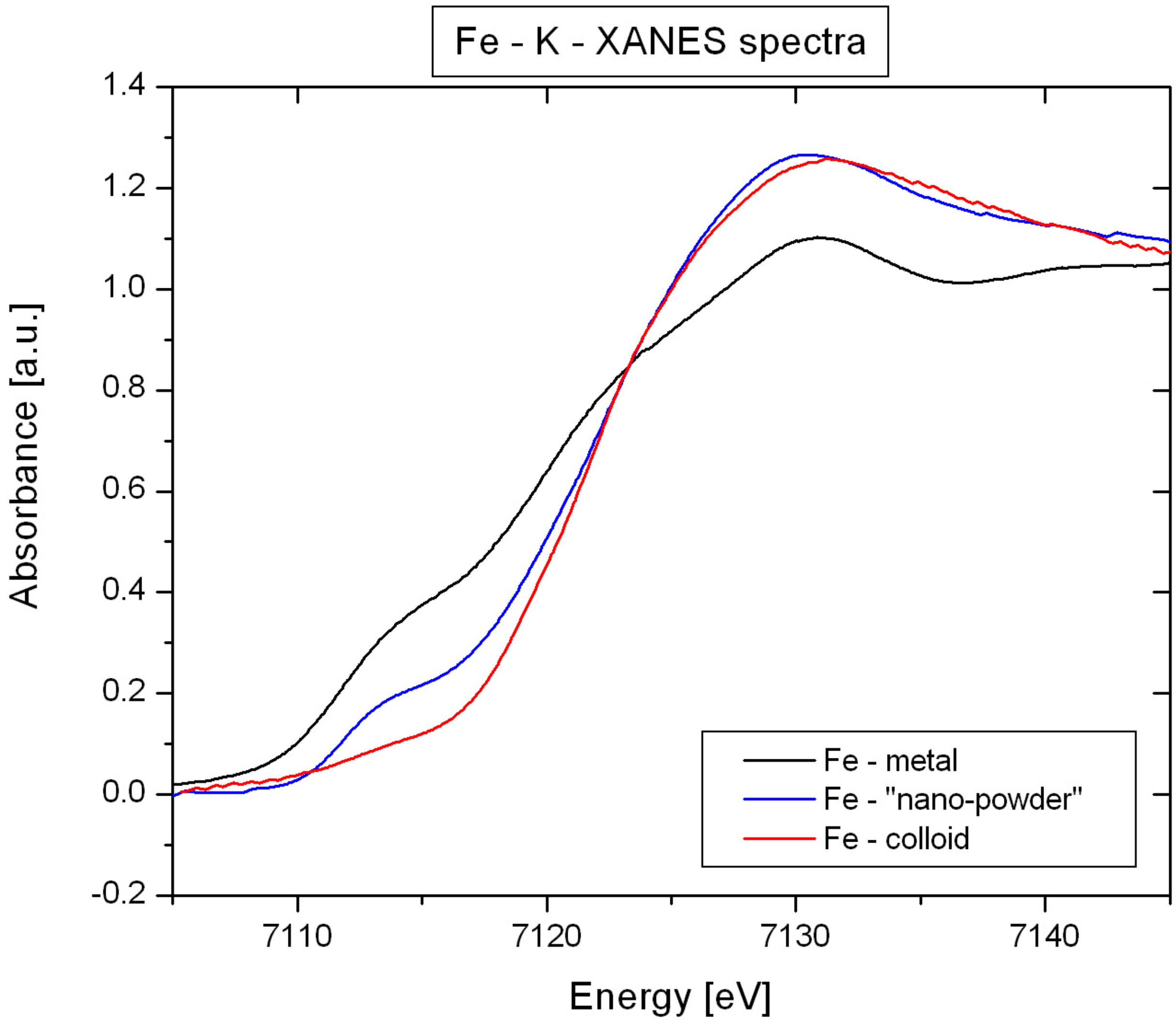
Investigated systems :

Ti, Fe, Mn, Pt, Rh, Ru, Cr, Au, Co

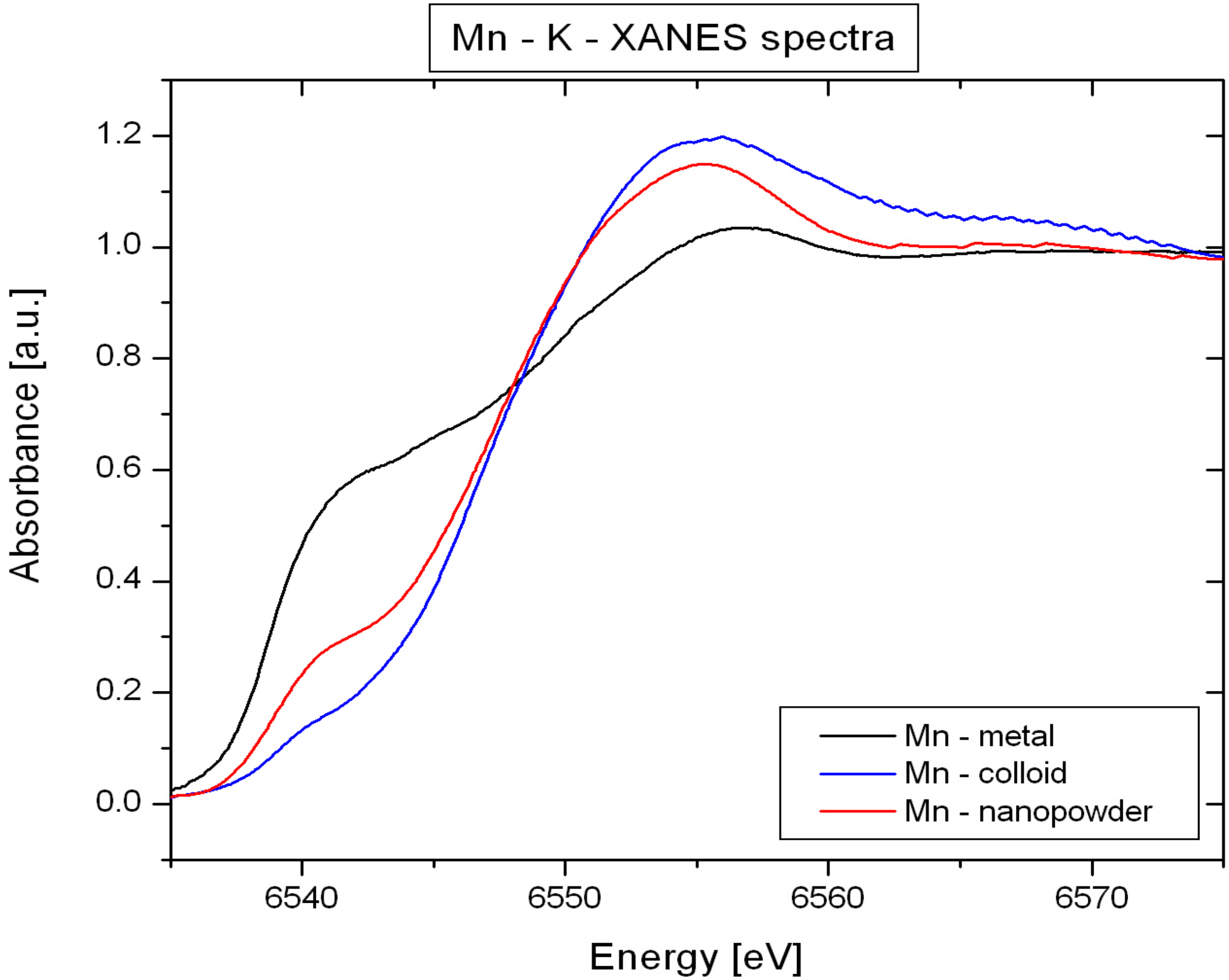
Size dependence of electronic properties (I)



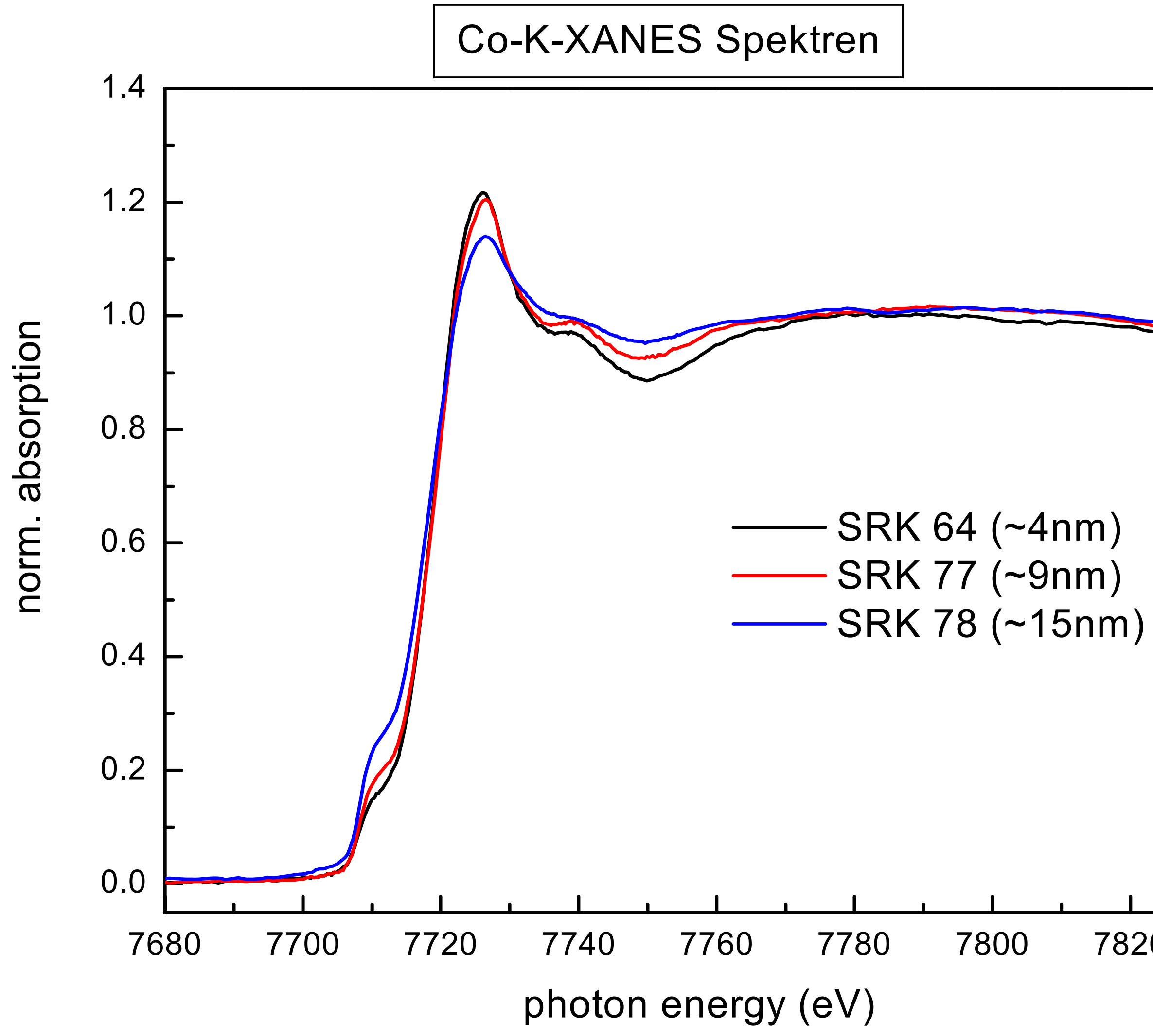
Size dependence of electronic properties (II)



Size dependence of electronic properties (III)

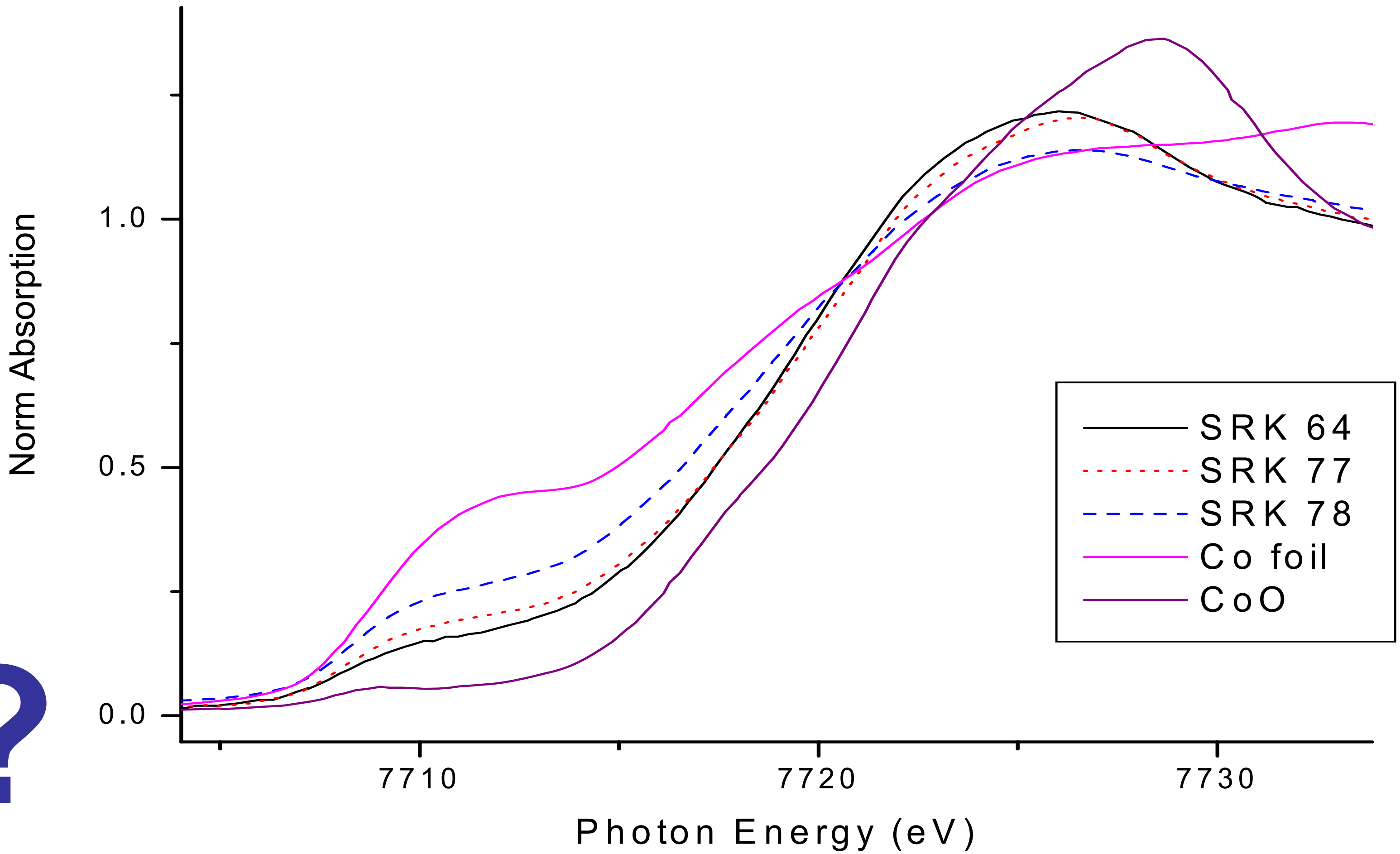


Size dependence of electronic and geometric properties



?

Size dependence of electronic and geometric properties



?

Interaction with surfactant??????

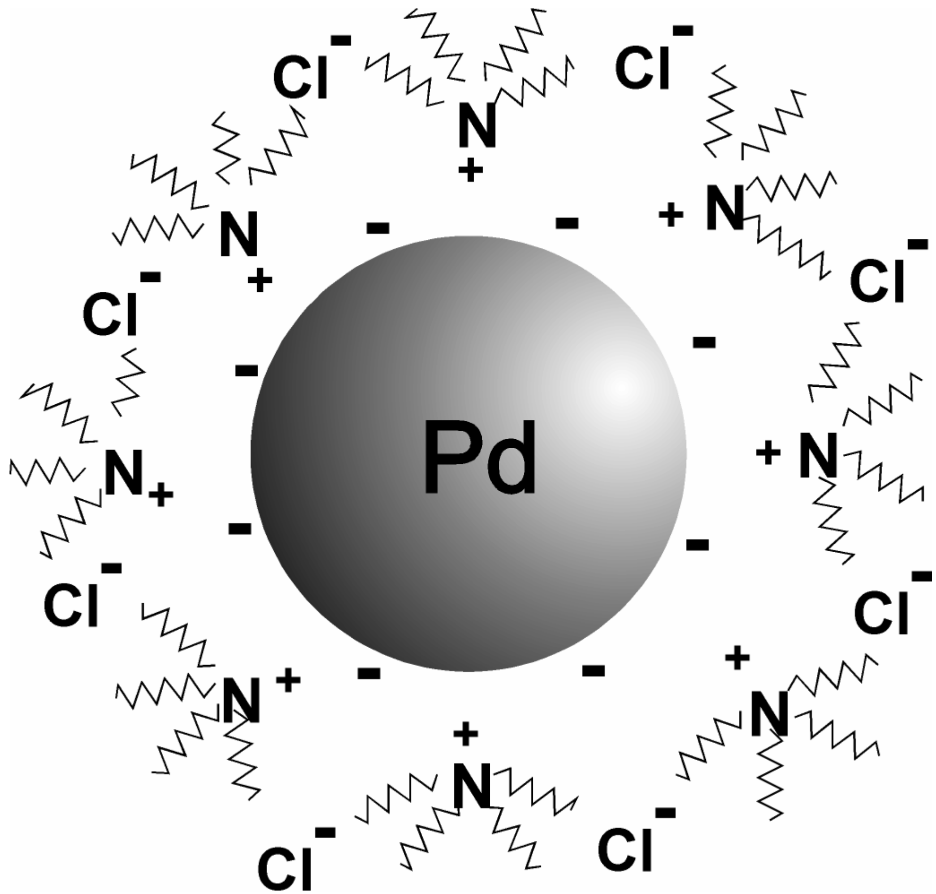
Interaction between nanoparticles and their protective coating

Example of an EXAFS/XANES analysis

Problem:

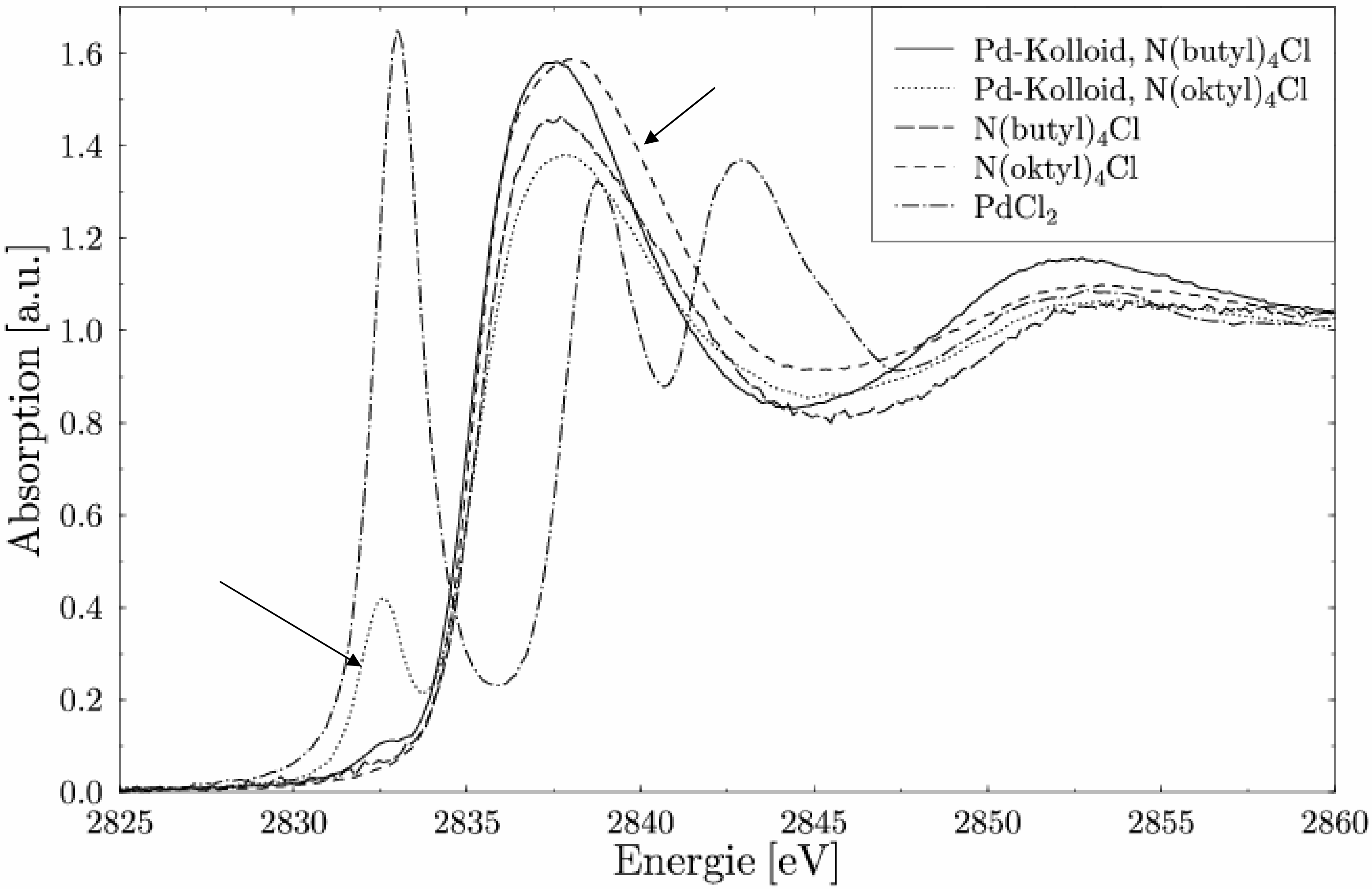
- The “type” of chemical bond between the metal core and the protective coating is not known
- Chemical bond should modify the electronic structure of the metal core

N(alkyl)$_4$Cl-stabilised Pd-colloids

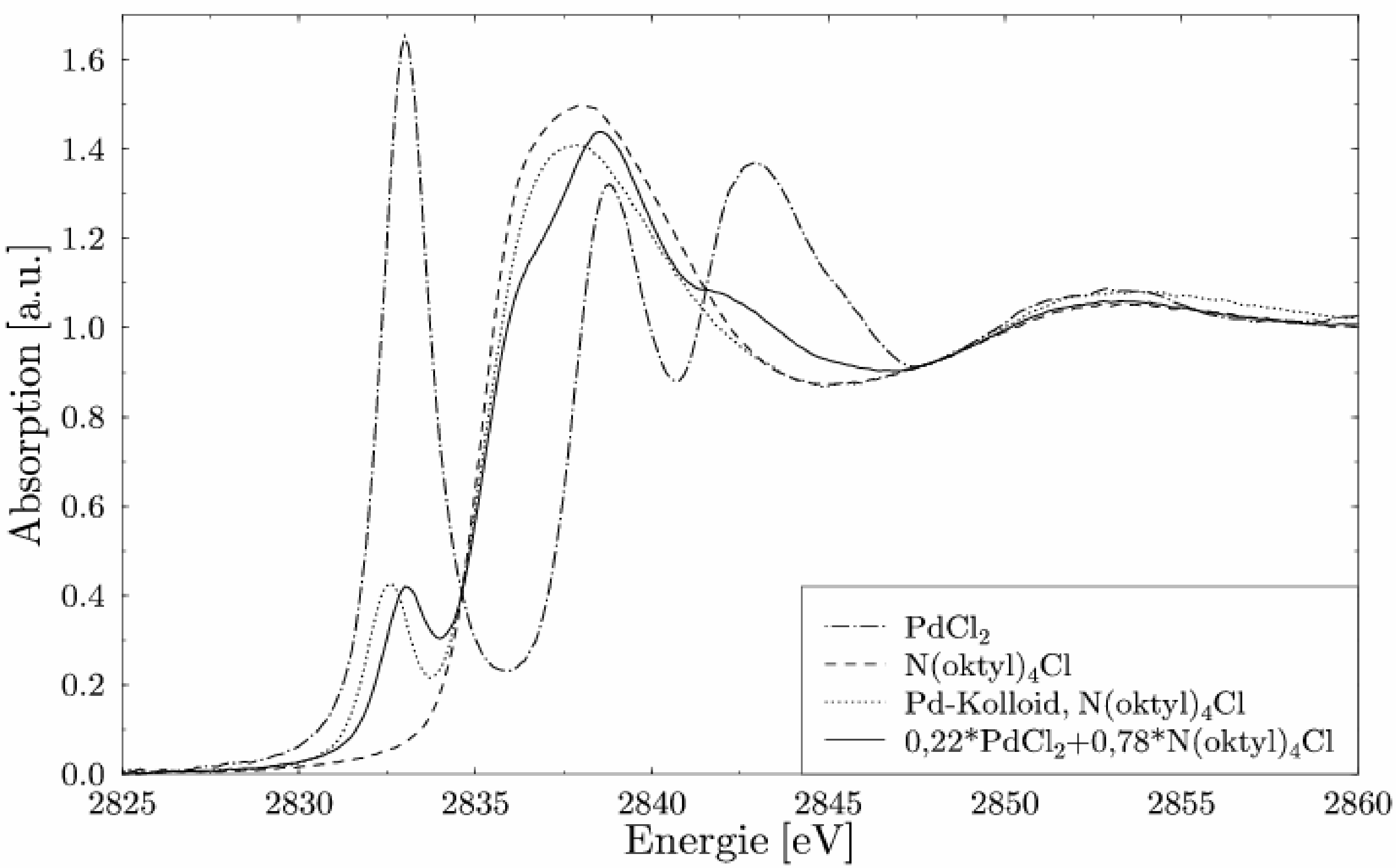


S. Bucher, PhD.-Thesis, Bonn 2002
S. Bucher et al. Surface Science, 497, 321, 2002

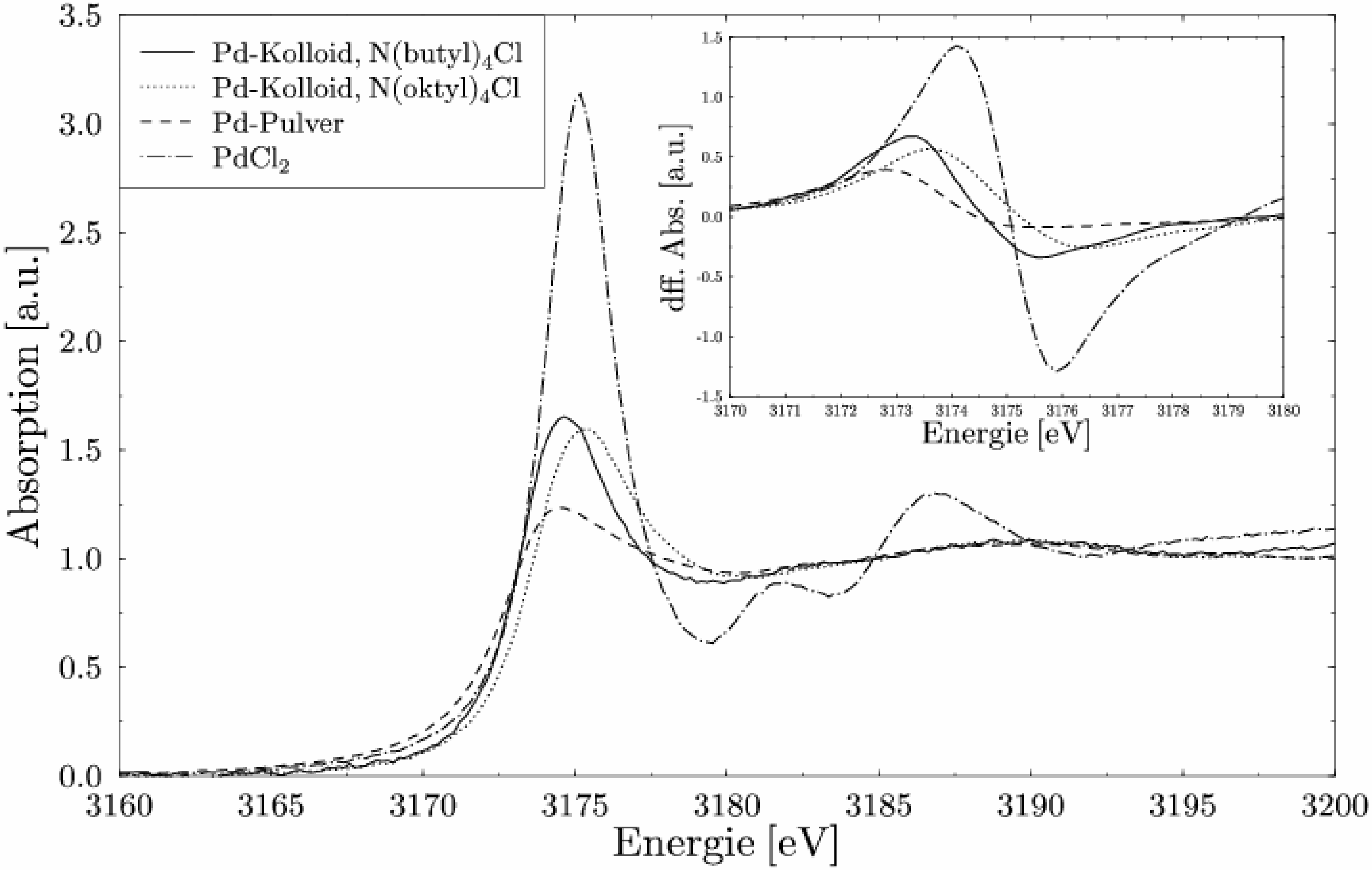
Cl-K-XANES spectra



Comparison: "simulated" and measured Cl-K-XANES spectra

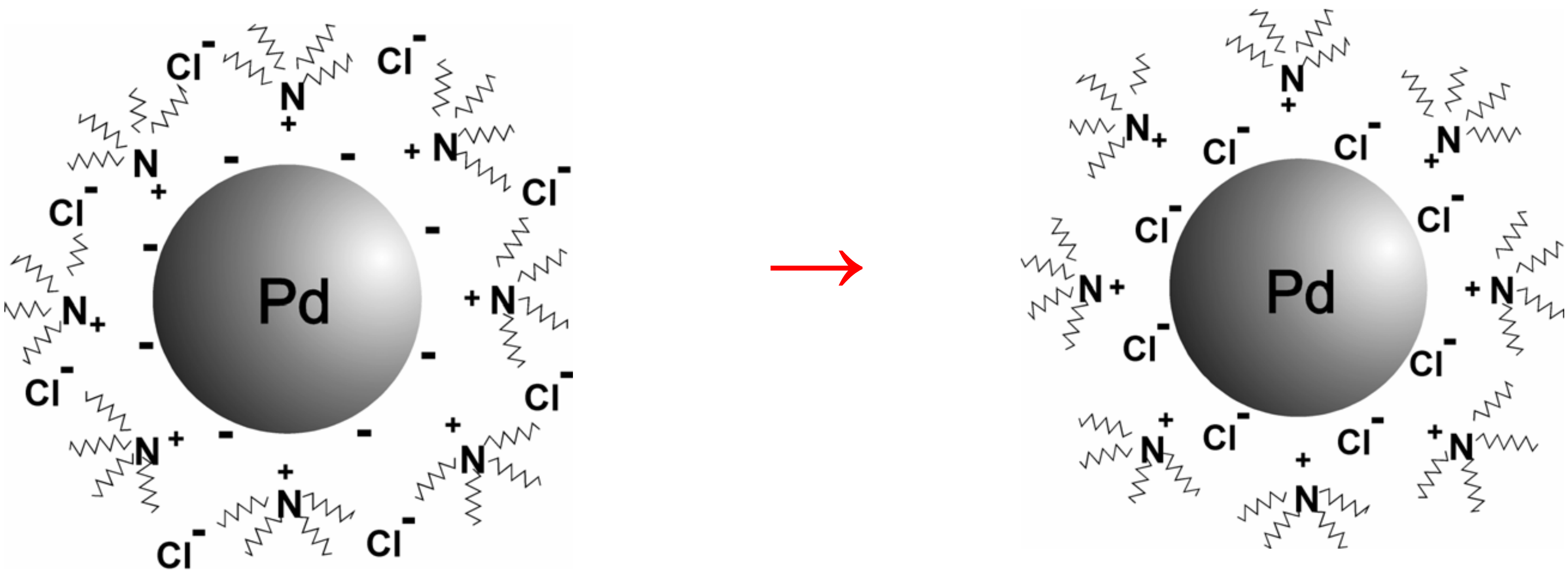


Pd-L-III XANES of NR4Cl stabilized Pd-colloids



N(alkyl)$_4$Cl-stabilised Pd-colloids:

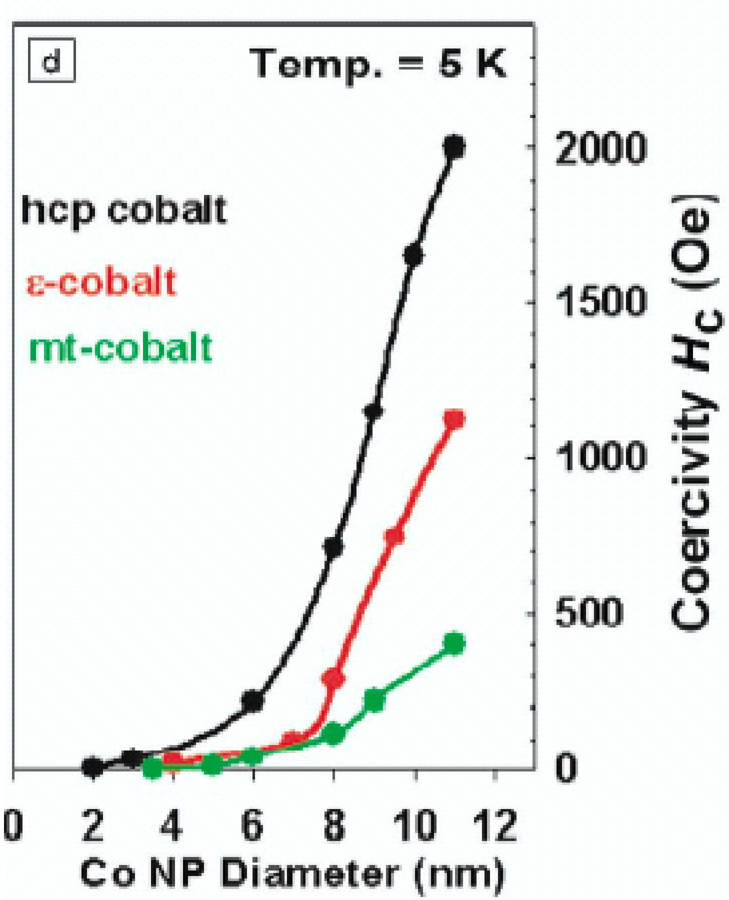
The “right” model



Co-nanoparticles:geometric, electronic and magnetic properties

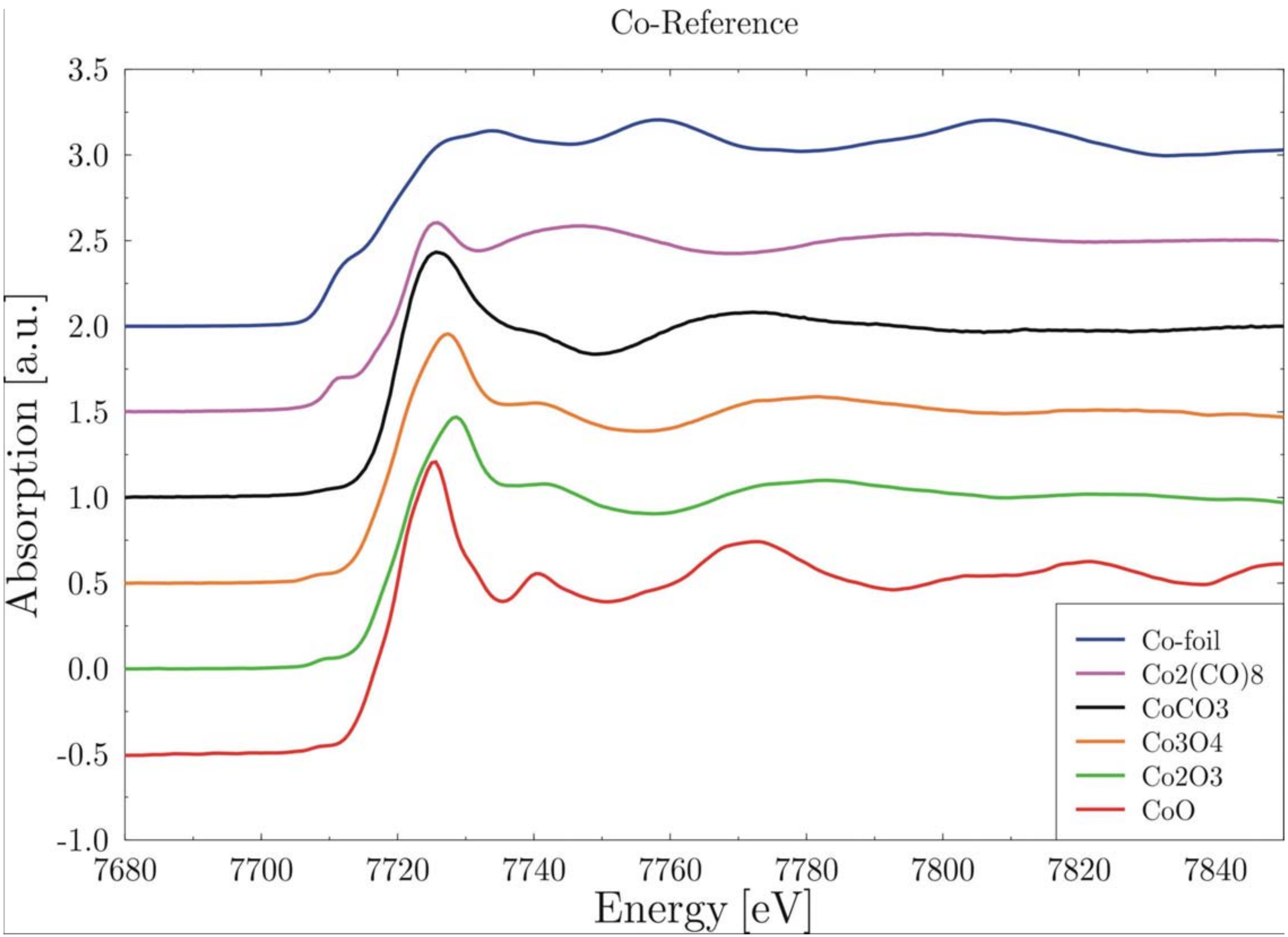
Why are Co-nanoparticles of special interest?

•3 crystallographic phases with different magnetic properties (hcp, fcc, ε)
•Very high magnetic moment
•Industrial applications (magnetic storage technology, ferro-fluidics)

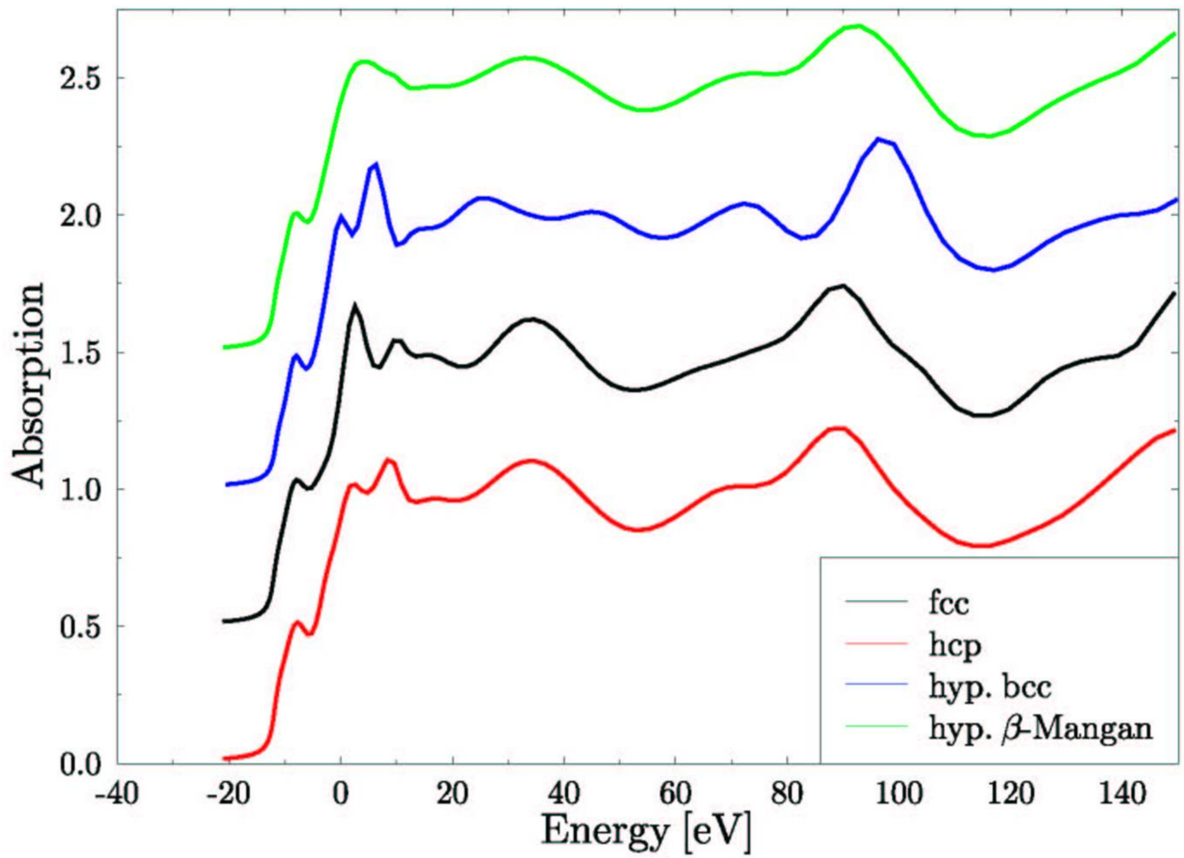


C.B. Murray, S. Sun, H. Doyle, T. Betley, MRS Bulletin, December 2001, 985

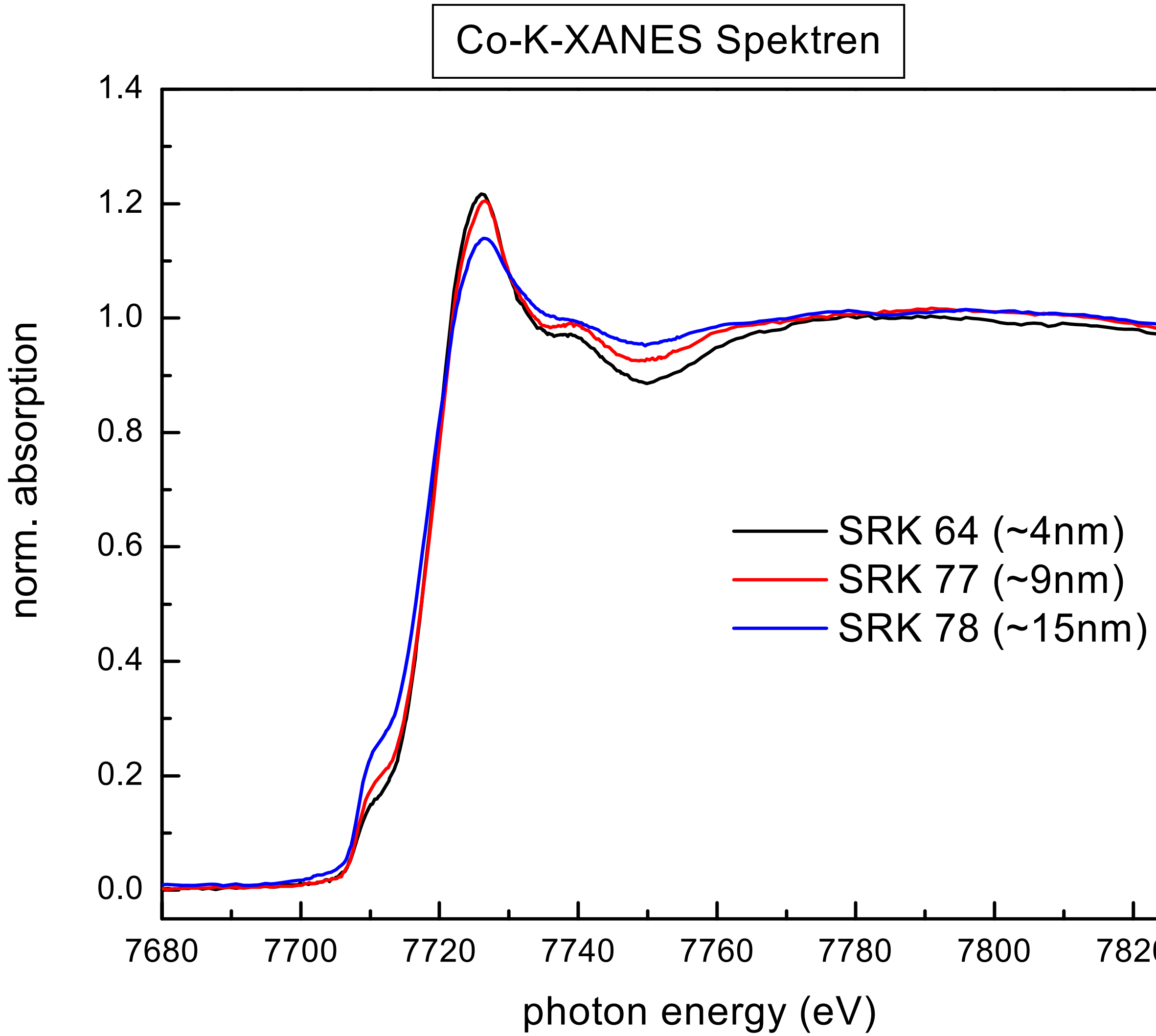
Co-K-XANES: reference spectra



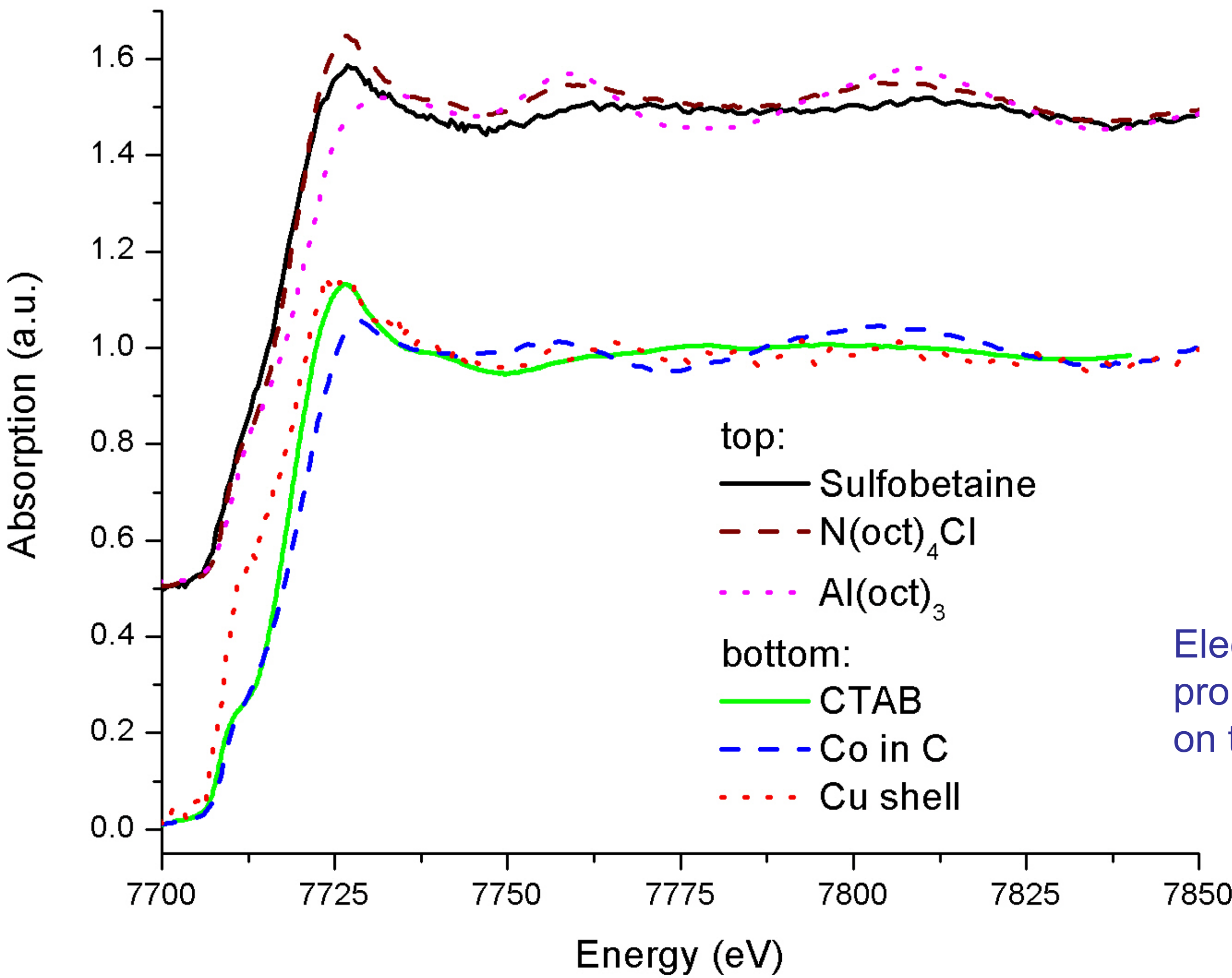
Co-K-XANES: FEFF 8 calculations for various crystal structures



Size dependence of electronic and geometric properties

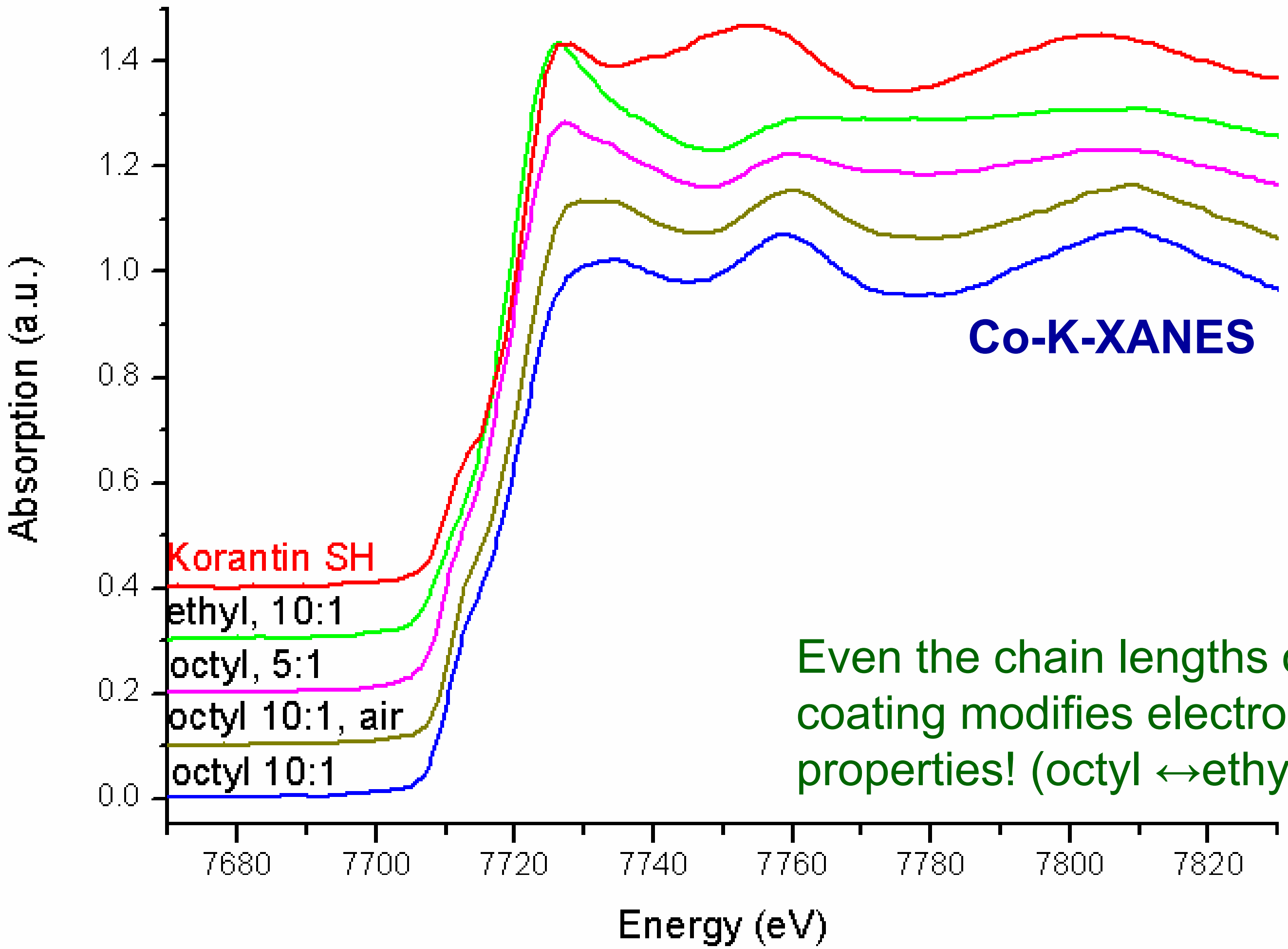


Electronic and geometric properties as a fct. of “coating”(I)



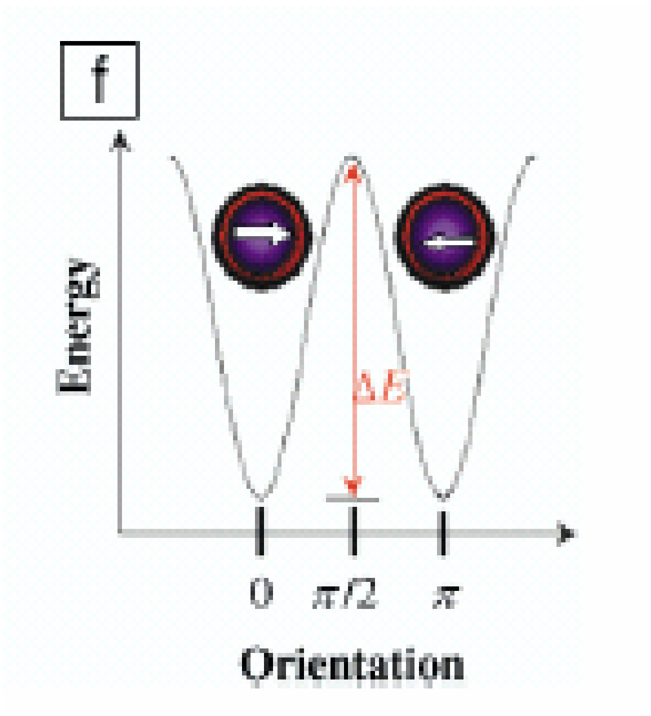
Electronic (and geometric) properties depend strongly on the respective coating!!

Electronic and geometric properties as a fct. of "coating"(II)



Even the chain lengths of the coating modifies electronic (geometric) properties! (octyl ↔ethyl)

Co: magnetic properties as a fct. of particle size and coating



Magnetic properties of nanoparticles

Competition between:

Short range exchange interaction → Parallel alignment of nearby spins

Long range dipolar couplings of spins → Antiparallel alignment of distant spins

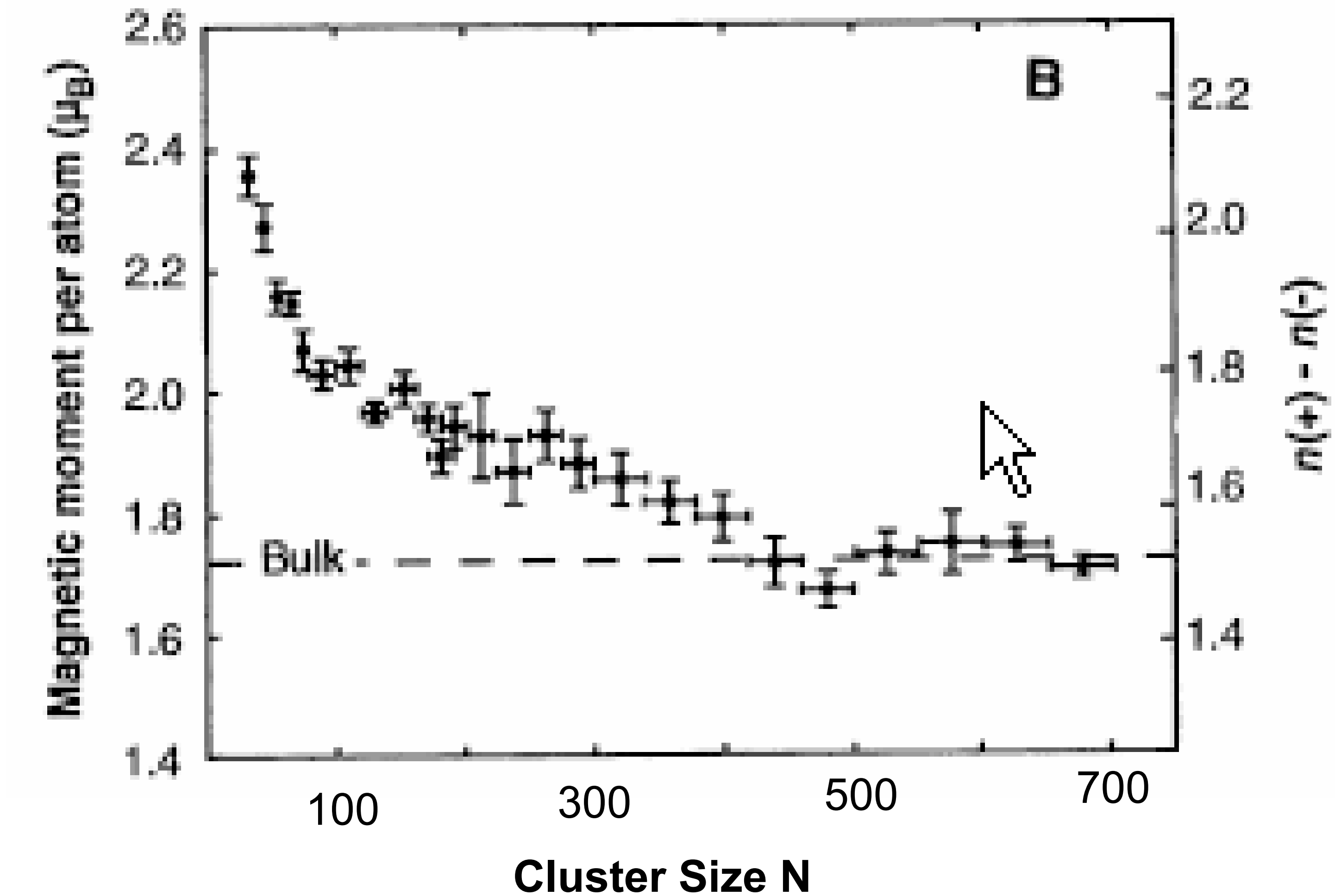
For small particles: no long range coupling → "single domain" magnets

ΔE for "spontaneous reorientation"

ΔE ~ V (particle volume) K (anisotropy constant)

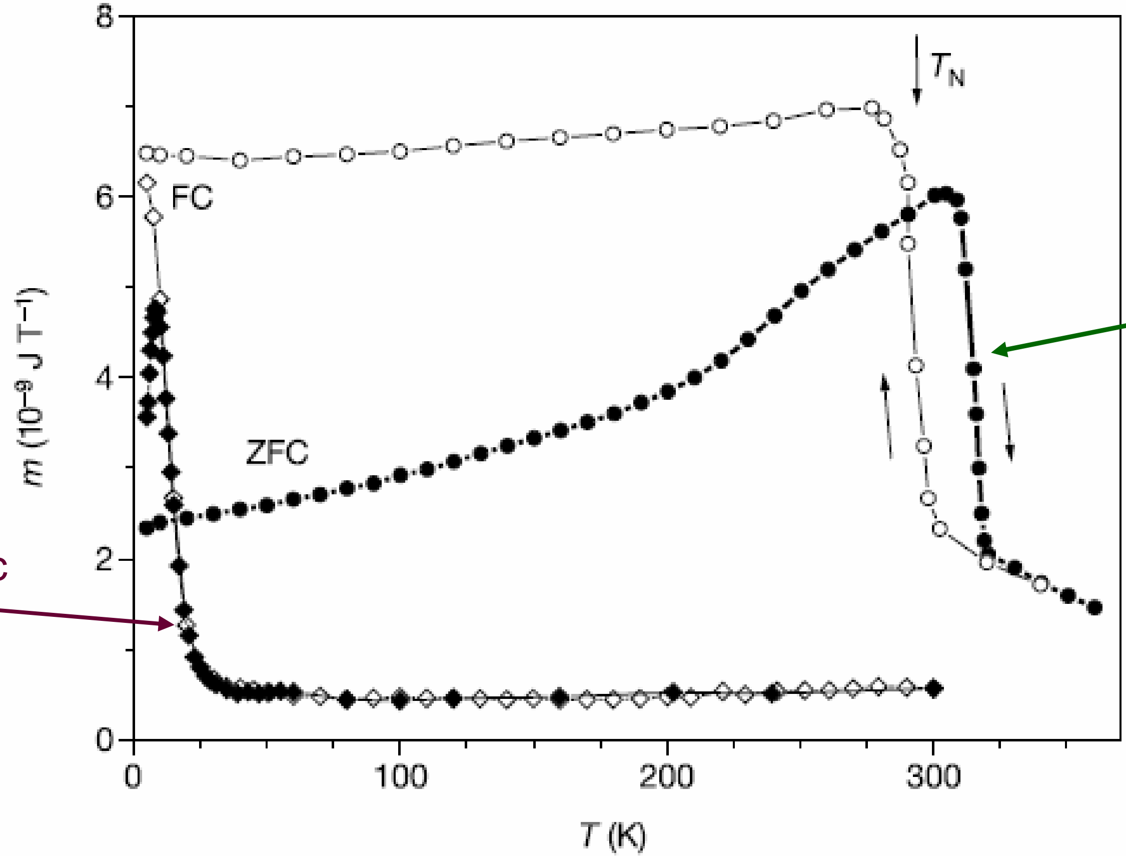
ΔE ~ kT → particle is "superparamagnetic"

Magnetic moments of "naked" Co -nanoparticles



I.M.L. Billas, A. Châtelain, W.A. de Heer, Science <u>265</u>, 1683 (1994)

Co: magnetic properties as a fct. of particle size and coating



In an antiferromagnetic matrix CoO

In a paramagnetic matrix Al2O3

4nm Co_{core} CoO $_{shell}$ nanoparticles

V. Skumryev, S. Stoyanov, Y. Zhang, H. Hadjipanayis, D. Givord, J. Nogués, Nature, 423, 850 (2003)

Co: magnetic properties as a fct. of particle size and coating

Co-nanoparticles are superparamagnetic

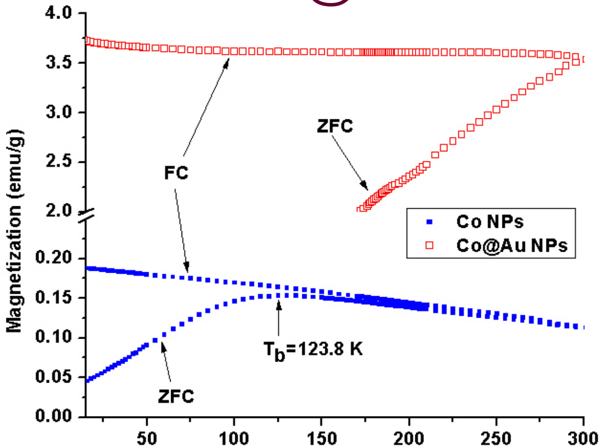
Cobalt „nano"-powder in the presence of a static magnetic field

SIZE [NM]	BLOCKING TEMPERATURE [K]	COATING
5, 9, 12	≈ 70, 304, 656	"no-coating" theory
2.5	12	Oleic acid
3	28	Oleic acid
3	60	Tetraalkylammonium-chlorid
3	10	Au
≈ 3	> 300	Au
≈ 3	235	Cu
≈ 3	28	SB-12
3, 6, 8, 11	≈ 10, 40, 100, 250	Oleic acid/ trioctylphosphine
4	≈ 600	polyethylene
4	10	C or Al2O3
4	≈ 290	CoO
4.4	30	Didodecyldimethyl ammoniumBromide
5	200	CoO
5	Above 300	Polydimehylphenyloxide

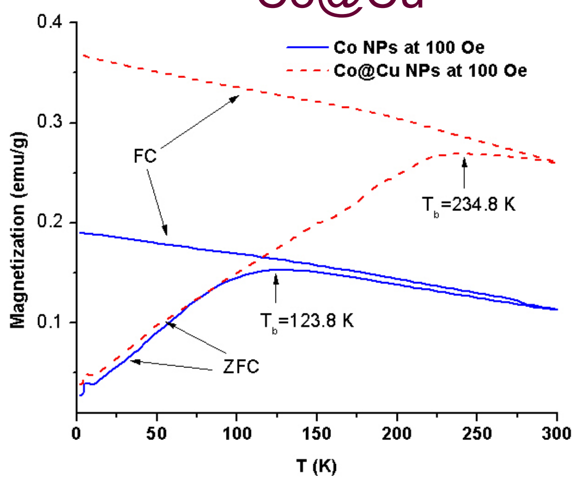
Values from "reliable" publications

Co: magnetic properties as a fct. of particle size and coating

Co@Au



Co@Cu



Material	T (K)	M (emu/g)	H (Oe)	Susceptibility (emu/mole)	Magnetic moment (μB)
Co-Au NPs (3- 4nm)	300	3.53	100	0.00087	2.83
	10	0.27	100	0.00038	0.19
Co-Cu NPs (3-4 nm)	300	0.26	100	0.0011	1.63
	10	0.05	100	0.0002	0.13
Fe_3O_4 (SRK-89) (~ 10 nm)	300	77.5	20000	1.67*(E-5)	0.20
	10	87.5	20000	1.89*(E-5)	0.04
Fe_2O_3 (SRK-88) (~ 10 nm)	300	67.5	20000	2.1*(E-5)	0.23
	10	72.5	20000	2.27*(E-5)	0.04
MicroFe_3O_4 (SRK93) (~2.8 μm)	300	10.0	20000	2.16*E(-6)	0.07
	10	12.5	20000	2.69*(E-6)	0.01
Co NPs (~ 3 nm)	300	0.11	100	0.00022	0.73
	10	0.04	100	8.12 (E-5)	0.08

Magnetic moment bulk: ~ 1.7 μ_B

Conclusions:

1. We have never investigated a bi-metallic nanoparticles that was a real statistical alloy!

2. There is a strong interaction between the coating and the metallic core of (small) nanoparticles

3. The coating determines the size (shape) of the particles (1 bottle of champagne for 2, 4, 6, 8 nm Co or Fe particles with identical coating!) (Wet chemical synthesis)

4. The type of coating determines (very strongly) the magnetic properties (blocking temperature, magnetic moment etc.) of nanoparticles

5. The thickness of the coating (Co@Au...)determines the magnetic properties of nanoparticles → new opportunity to tailor magnetic properties!?

Development of Nanoparticles for Early Detection and Treatment of Cancers and Metastases

Collaboration CAMD-Pennington Bio-medical Research Center (C. Leuschner)

Here still iron-oxide particles (biocompatibility etc.); Co would be much better

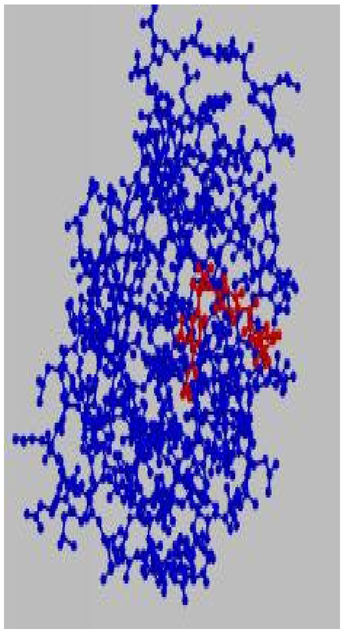
Background Information:

The 5 year survival rate for metastatic cancers has not improved since 1973 – 600,000 cancer deaths occurring in the US per year

This is partially due to the:

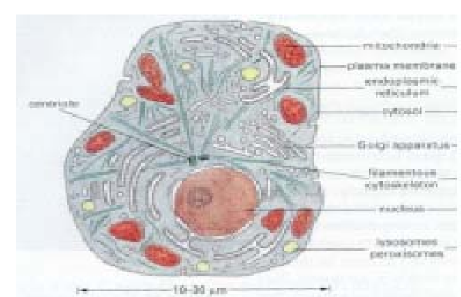
- Lack of highly sensitive detection tools to the sub-millimeter level – cell clusters
- Lack of chemotherapeutics which specifically select for cancerous cells leaving healthy tissues unharmed, causing severe systemic side effects.
- Development of multi-drug-resistance in advanced disease

Lytic peptides and conjugates in cancer treatment

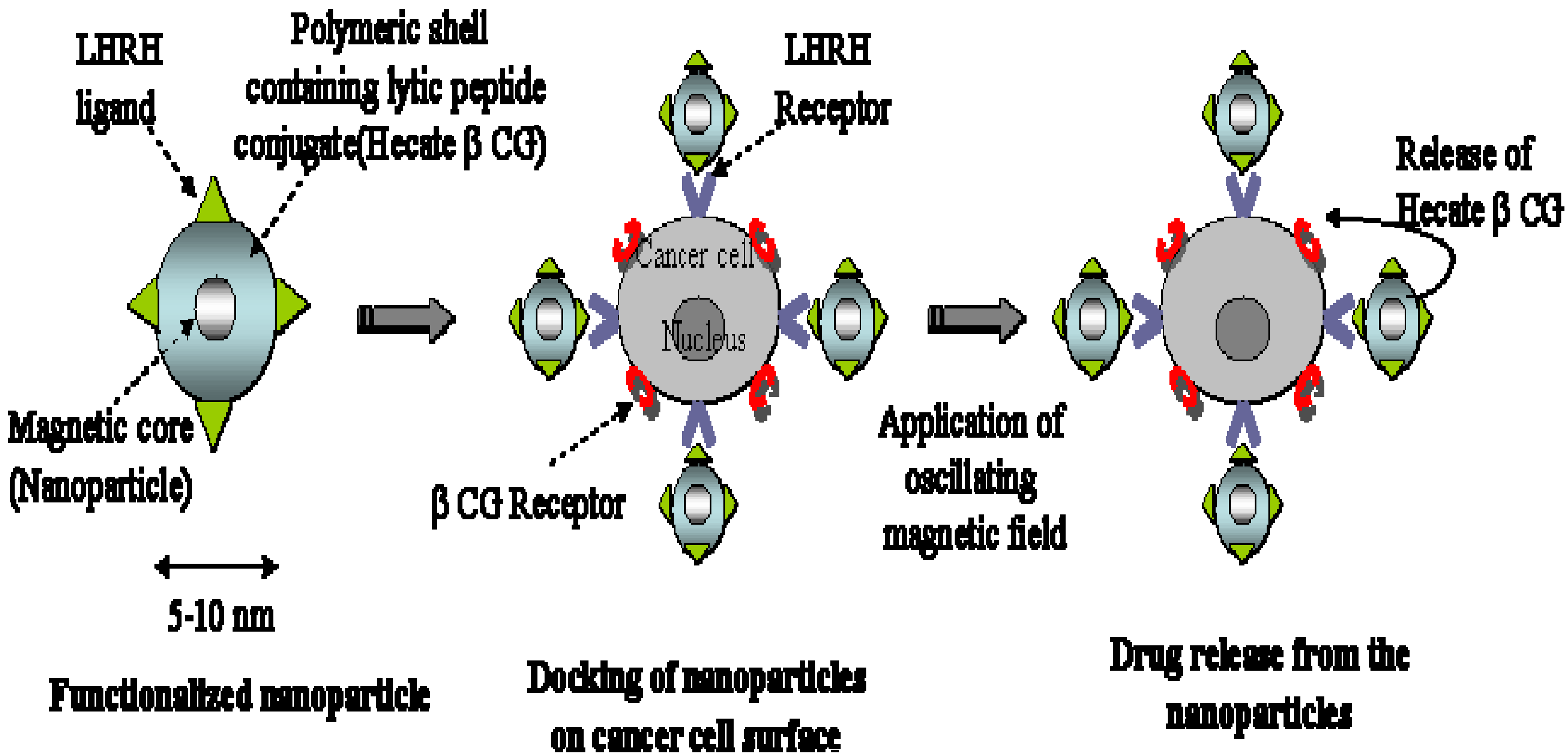


- Lytic peptides are membrane-disrupting peptides
- Lytic peptides are potent against "hormone controlled" cancer cells (prostate, breast, ovarian..)
- These cancers cells express receptors for
  βCG = chorionic gonadotropin and
  LHRH = luteinizing hormone releasing hormone
- Thus, lytic peptide conjugates (Hecate-βCG) are "site" specific at the tumor
- However, side effects – Gonads (Reproductive organs) have the same receptors!

W. Haensel, C. Leuschner, Pennington Research Center

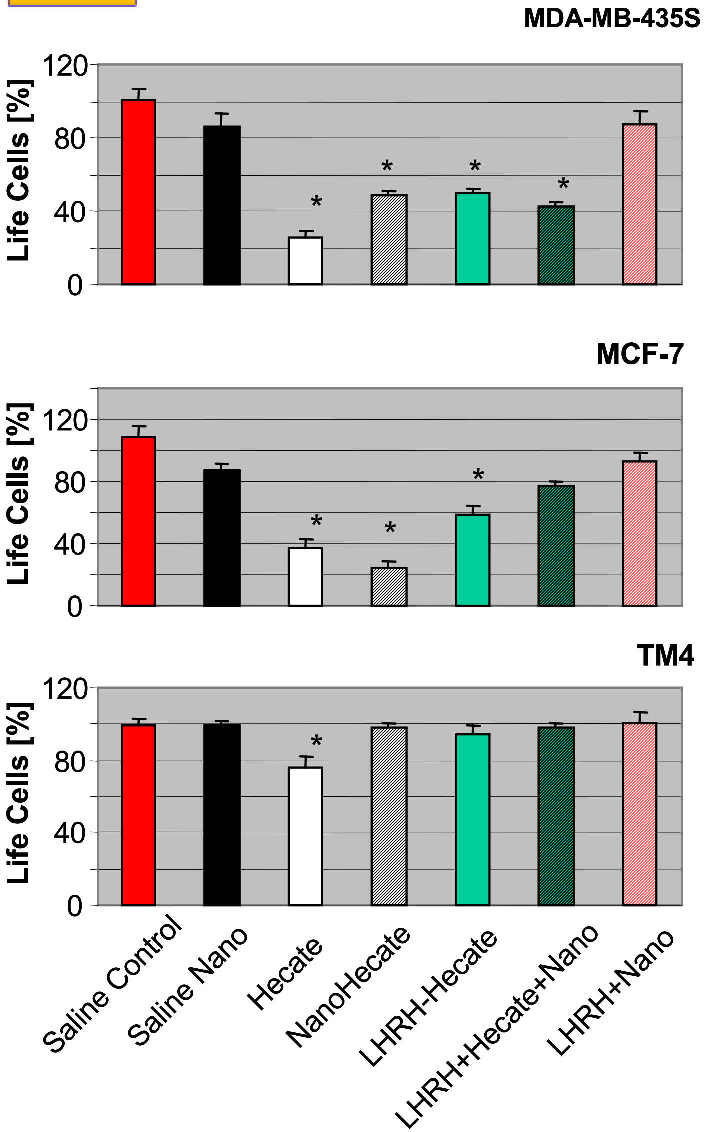


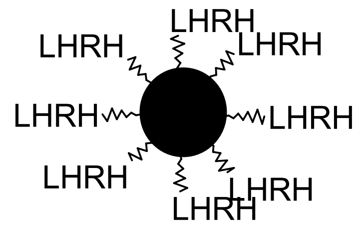
Drug delivery: Lytic peptides and conjugates in cancer treatment

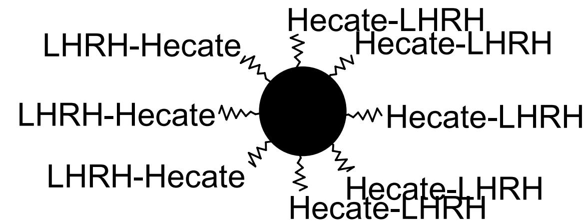


Drug delivery.mov

"Targeted" Destruction of Breast Cancer Cells in Vitro

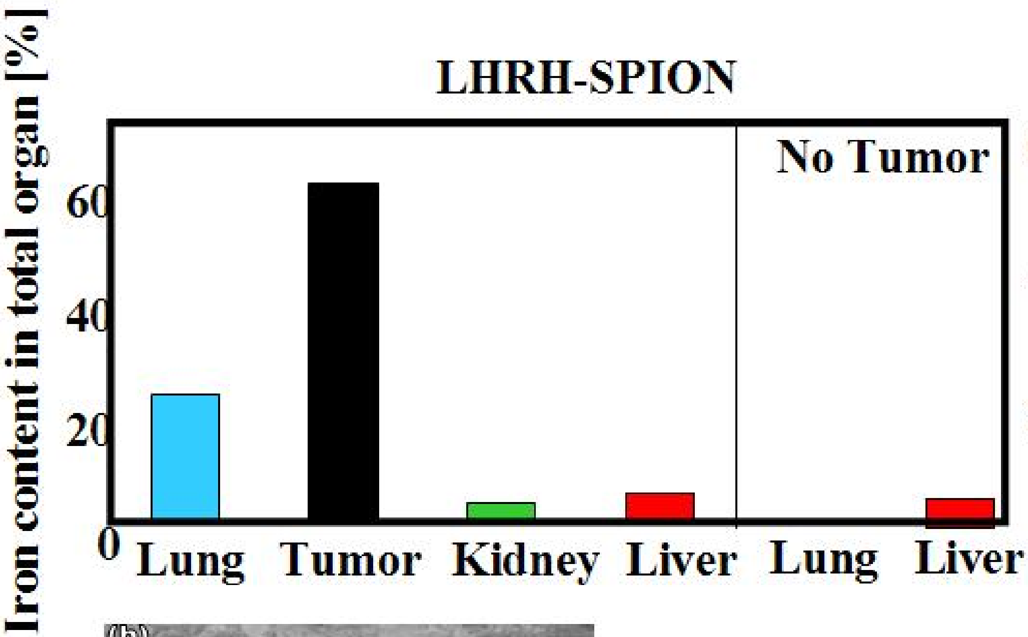


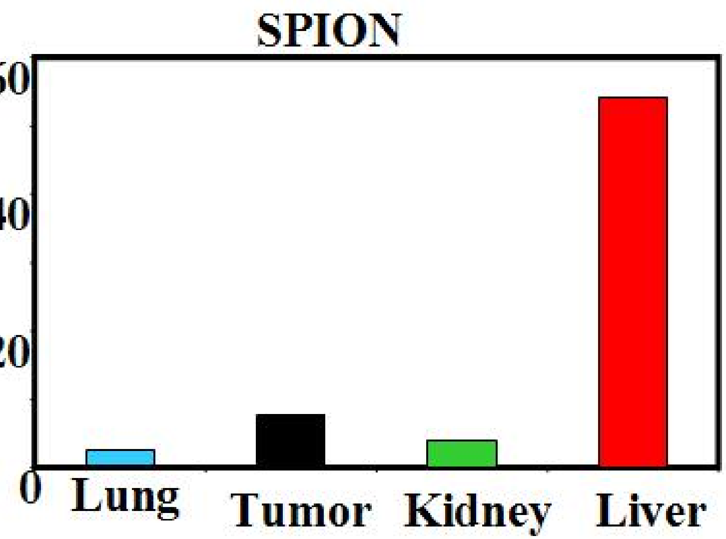


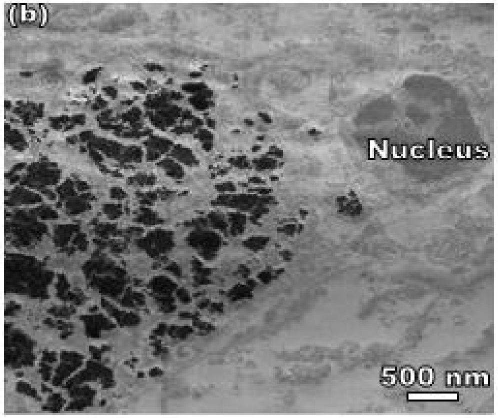


- **The non-cancerous TM4 cell line responds only to Hecate with cytolysis**
- **Lytic peptides are up to 50 times more effective in killing cancer cells than normal cells**
- **Hecate, Nano-Hecate, LHRH-Hecate and Nano-LHRH-Hecate are toxic at 10μ mol.**

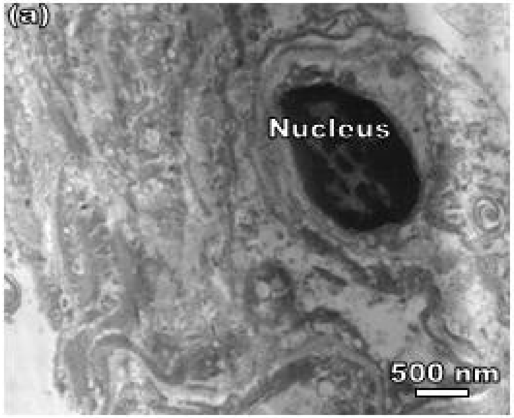
Relative Iron Distribution in Mice after injection of SPION and LHRH-SPION







TEM - Lungs



Iron content of organ subtracted

Tumors: MDA-MB-435S.luc, 100 % = 5 mg Fe/ mouse

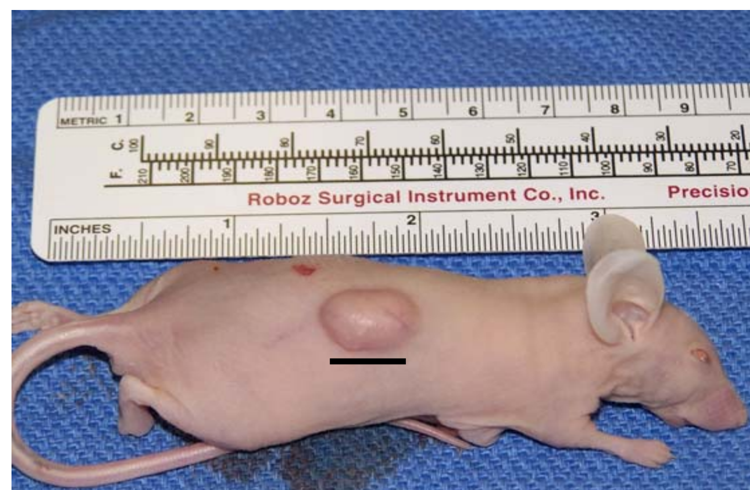
Result no. 2: LHRH-SPION-Hecate kills cancer cells

Saline

LHRH-SPION

LHRH-SPION-Hecate

Hecate-SPION



Summary of Results

LHRH-SPION-Hecate is highly specific in destroying

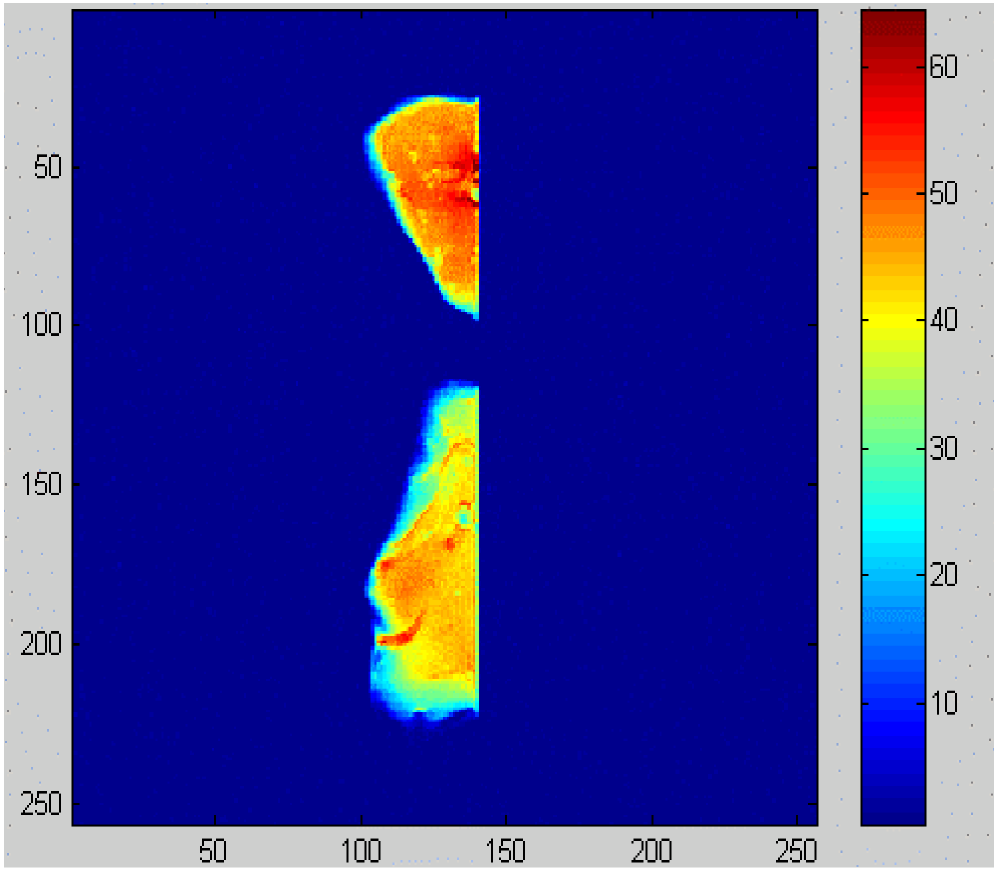
- primary tumors from breast cancer xenografts
- lymph node metastases
- lung metastases
- bone metastases

Iron from LHRH-SPION-Hecate is retained in treated tissues

No side-effects

- Body weights unchanged in treated mice
- Gonadal weight unchanged in treated mice
- Liver and kidney function normal in treated mice
- Platelets, erythrocytes and leukocytes normal in treated mice

MR Images from Breast Cancer Xenografts after Injection of LHRH-SPION and SPION - Resolution 200 micron

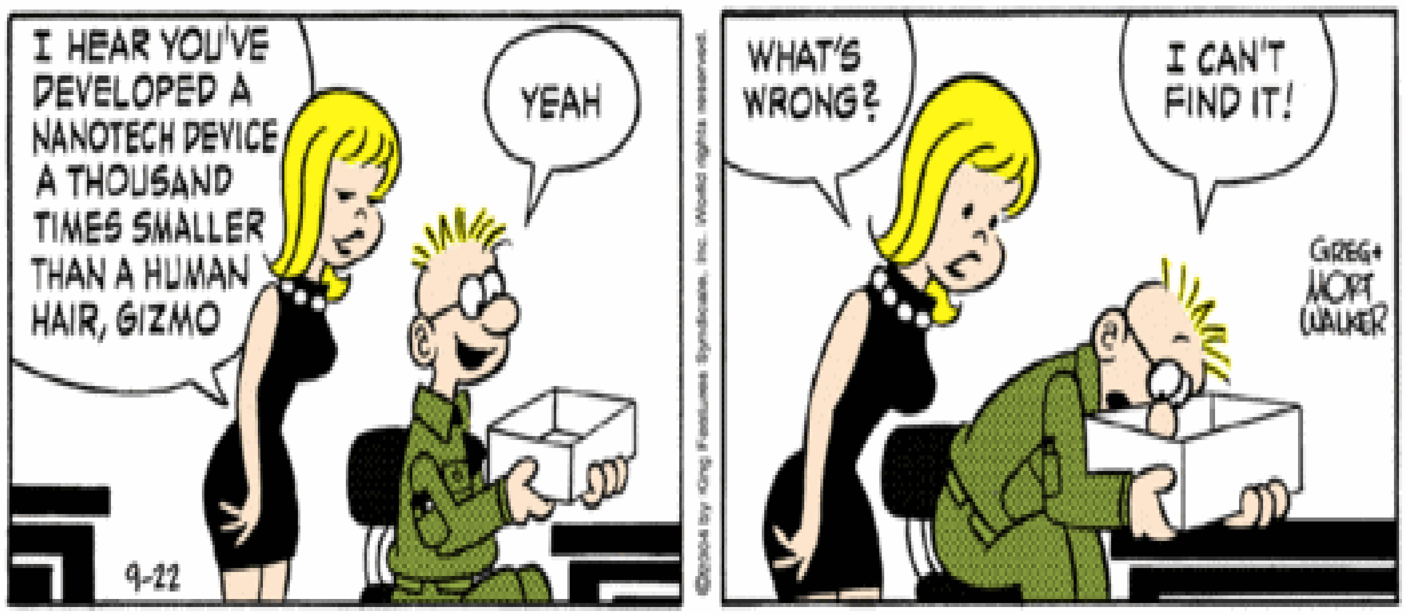


MR imaging resolution can be improved to micrometer range by increasing the concentration of a contrast agent in the target tissue

Direct labeling of cancer cells in vivo

Zhou JK, Meng J, Thieraux C, Leuschner C,Kumar C, Hormes J Soboyejo WO:
LHRH-Functionalized Magnetite Nanoparticles for Breast Cancer Detection and Treatment,
American Academy for Nanomedicine, Baltimore MD, August 2005.
Shannon et al 2004, Magnetic Res Imag 22, 1407-1412

Nanotechnology is still full of surprises



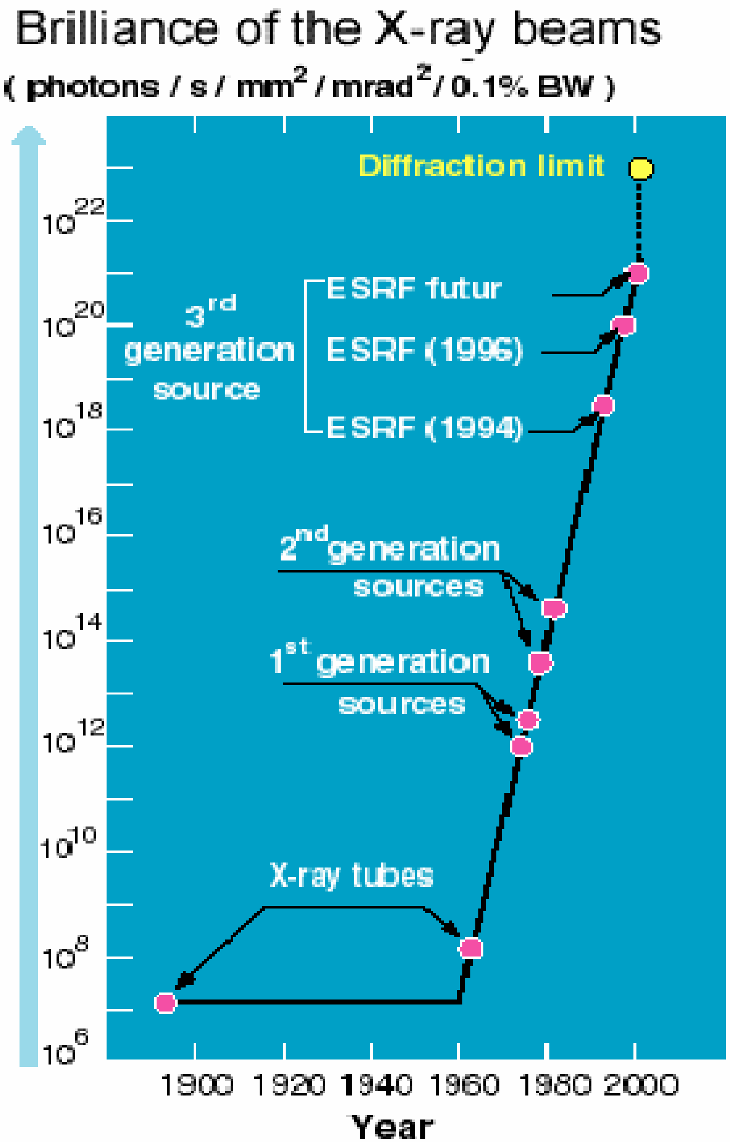
by Mort Walker

Thank you very much for your attention

Questions are welcome????

A Synchrotron Radiation Facility for Pakistan!?

The Brilliance of X-ray Sources



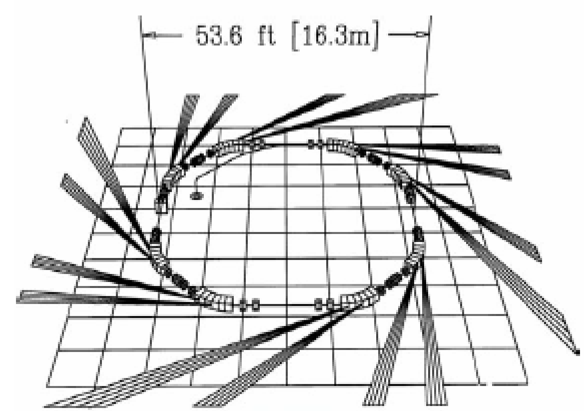
For SR: Brilliance is determined by a combination of emittance of the machine, electron current, layout of insertion devices, and other factors

The emittance of an accelerator is determined by the dimension of the electron beam x its divergence

Emittance ~ φ^3 (φ = deflection angle per bending magnet)
Emittance ~ E^2 (E = Energy of the electrons)

CAMD storage ring: 2nd generation ring + wiggler

CAMD Storage Ring



Lattice	Chasman-Green
Circumference	55.2 meters
Radius of dipole magnets	2.928 m
Energy	1.3/1.5 GeV
Characteristic energy	1.7/2.6 keV
Emittance	320 nm rad
Current	300 mA/150 mA
Lifetime	> 8 hours

"Boundary conditions" for the machine design I:

- Full costs of the machine (including four insertion devices + beam lines) less than 100 M\$. ("size").

- Machine parameters "as good as possible", i.e. emittance, current, lifetime etc. should be at least comparable to the ALS and SPEAR III (Optics).

- Radiation from the bending magnets should have good intensity at the Se-K-edge for protein crystallography experiments (Energy).

"Boundary conditions" for the machine design II:

- The machine specifications should allow the use of undulator radiation for energies up about 5 keV with "conventional" devices and up to 13 keV (again Se-K-edge) with superconducting mini-undulators .

- The design should include all developments from existing 3rd generation SR facilities (i.e. full energy injection, topping up, mini-beta sections etc.)

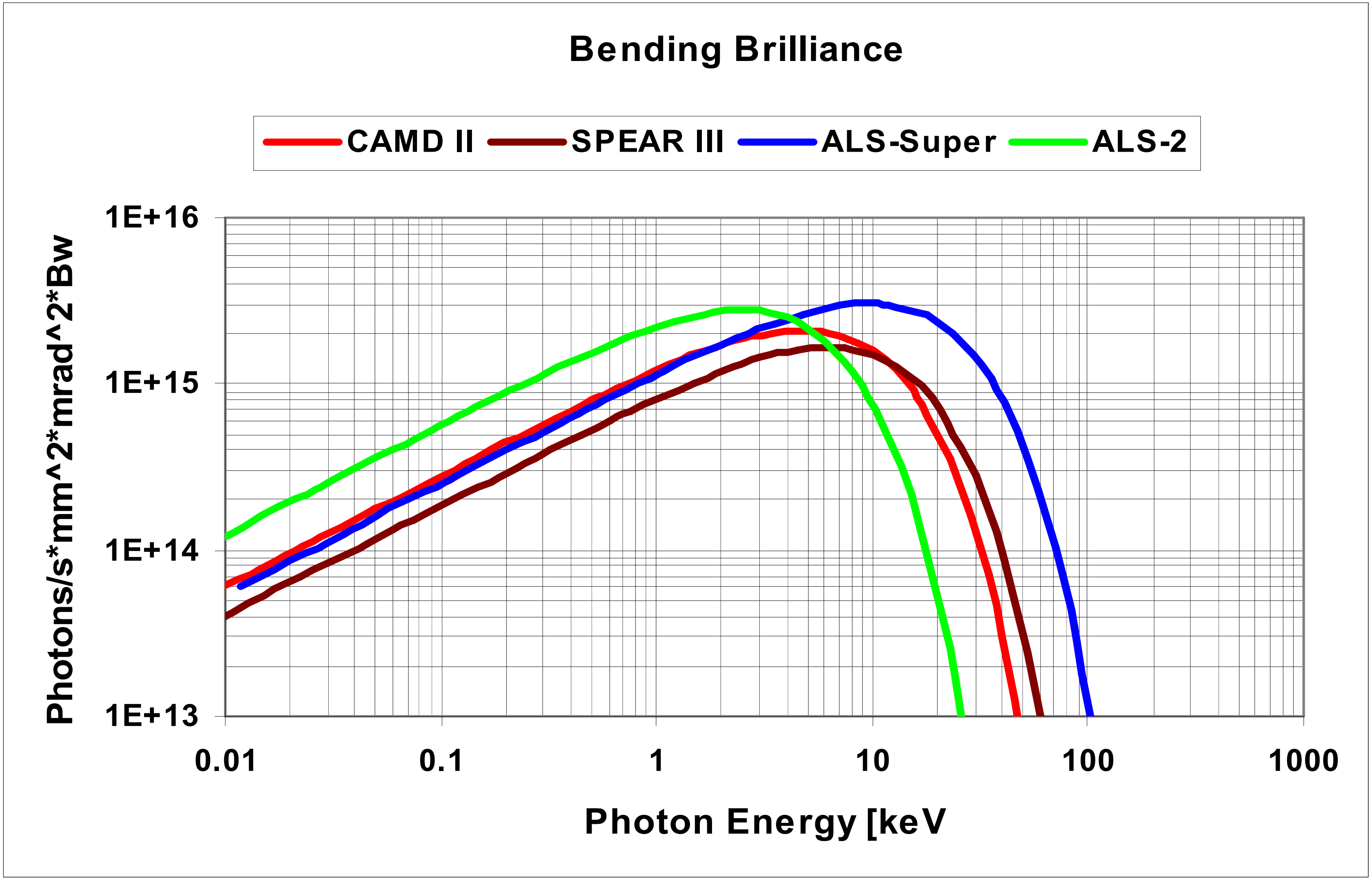
Medium energy 3rd generation SR facilities

Source	Energy (GeV)	Emittance nmrad	Ins. Length (m)	Angle (rad)	Circumf. (m)	Percent. (%)	Norm Emitt **)	Factor Brilliance	Factor A
SESAME III	2.5	24.4	49.456	0.3927	124.8	39.628	64.5	66.6	9.54
ANKA	2.5	88	31.34	0.3927	110.8	28.285	232.5	3.7	0.52
ALS	1.9	5.6	81	0.1745	196.8	41.159	291.9	1312.5	0.48
BESSY II	1.9	6.4	89	0.1963	240	37.083	234.4	905.4	0.68
ELETTRA	2	7	74.78	0.2618	258	28.984	97.5	591.5	3.05
INDUS II	2	44	36.48	0.3927	172	21.209	181.6	11.0	0.64
MAX II	1.5	9	31.4	0.3142	90	34.889	129.0	430.7	2.10
SLS	2.4	5	63	0.244	288	21.875	59.8	875.0	6.13
NSLS	2.5	44	36	0.3927	170	21.176	116.2	10.9	1.57
SOLEIL	2.75	3.72	159.6	0.1963	354	45.085	65.0	3257.9	10.66
Boom I	3	12	64.92	0.2618	180	36.067	74.3	250.5	6.53
Boom II	3	6.88	76.72	0.2244	216	35.519	67.7	750.4	7.76
CLS	2.9	18.2	62.4	0.2618	170.4	36.620	120.6	110.6	2.52
DIAMOND	3	2.74	218.2	0.1309	561.6	38.853	135.7	5175.2	2.11
LSU-TBA-II	2.5	4.46	87.6	0.244	256	34.219	49.1	1720.3	14.18
LSU-Boom(0.25)	2.5	3.29	87.6	0.19635	240.6	36.409	69.5	3363.7	7.53
LSU-QBA	2.5	3.99	139.2	0.2269	280	49.714	54.6	3122.7	16.65
LSU-SESAME	2.5	13.6	63.58	0.31416	154.8	41.072	70.2	222.1	8.34
LSU-CAMD II	2.5	8.3	48	0.31416	154.8	31.008	42.8	450.1	16.90

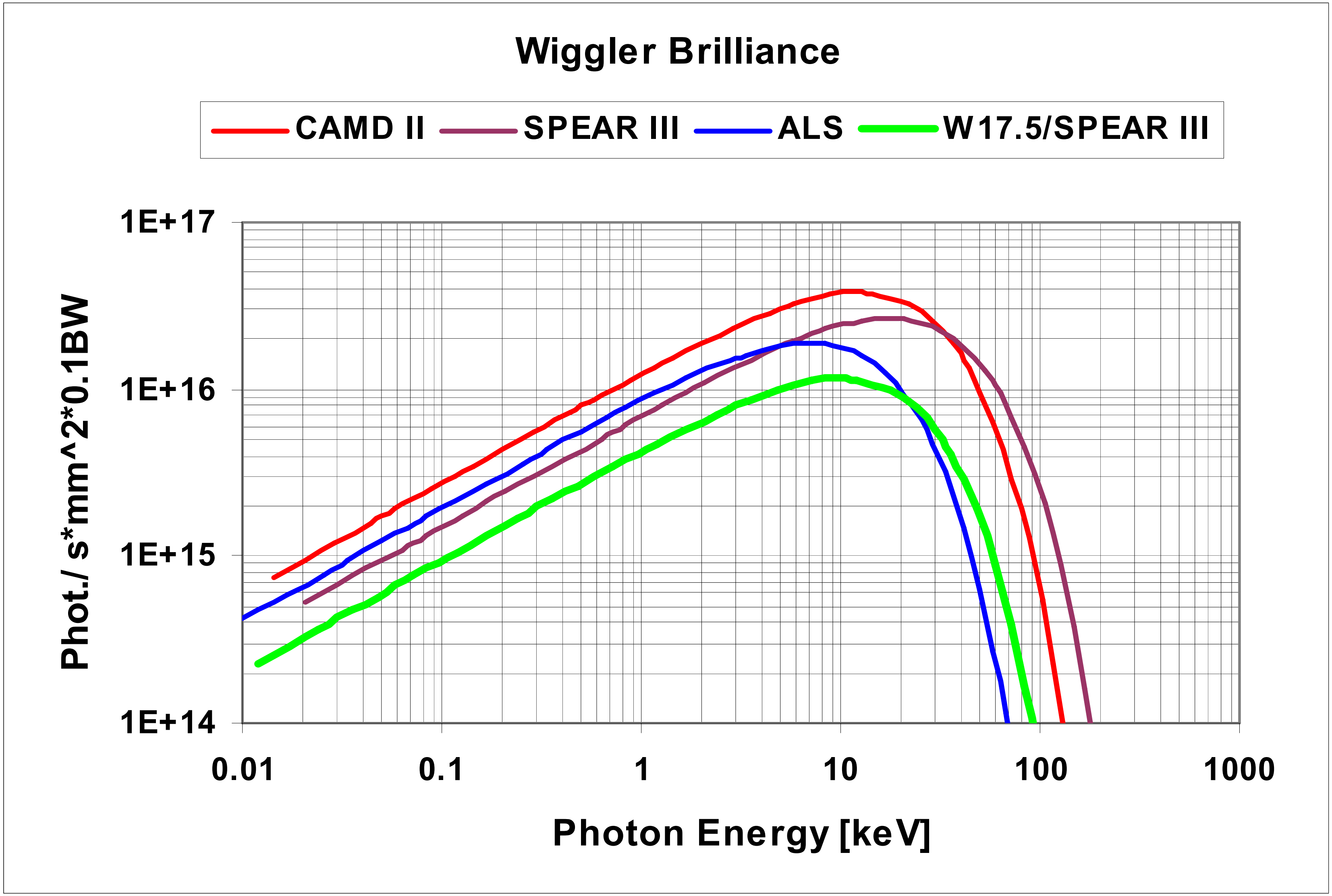
Parameters of the proposed machine

- ---
- Particle momentum, cp................ = 2.500 GeV
- Gamma .... = 4892.36801
- Beam current......................... = 400.000 mA
- Ring circumference,C................. = 154.79237 m
- Energy loss/turn .... = 580.131 keV
- Tot. radiation power .... = 232.0 kW
- Horiz.damping time .... = 3.145 msec
- Vert.damping time .... = 4.444 msec
- Synchrotron damping time .... = 2.801 msec
- Betatron tunes,Q_x................... = 10.81304
- Q_y .... = 3.73497
- Natural chromaticity,xi_xo .... = -19.43581
- xi_yo .... = -15.01676
- Corrected chromaticity,xi_x .... = 0.00000
- xi_y .... = 0.00000
- ---
- Horiz.beam emittance .... = 8.355 nm
- Vert.beam emittance .... = 83.548 pm
- Coupling .... = 1.00000 %
- Rel. energy spread .... = 0.09861 %
- Momentum comp. Fact., alpha_c . = 0.003308
- ---

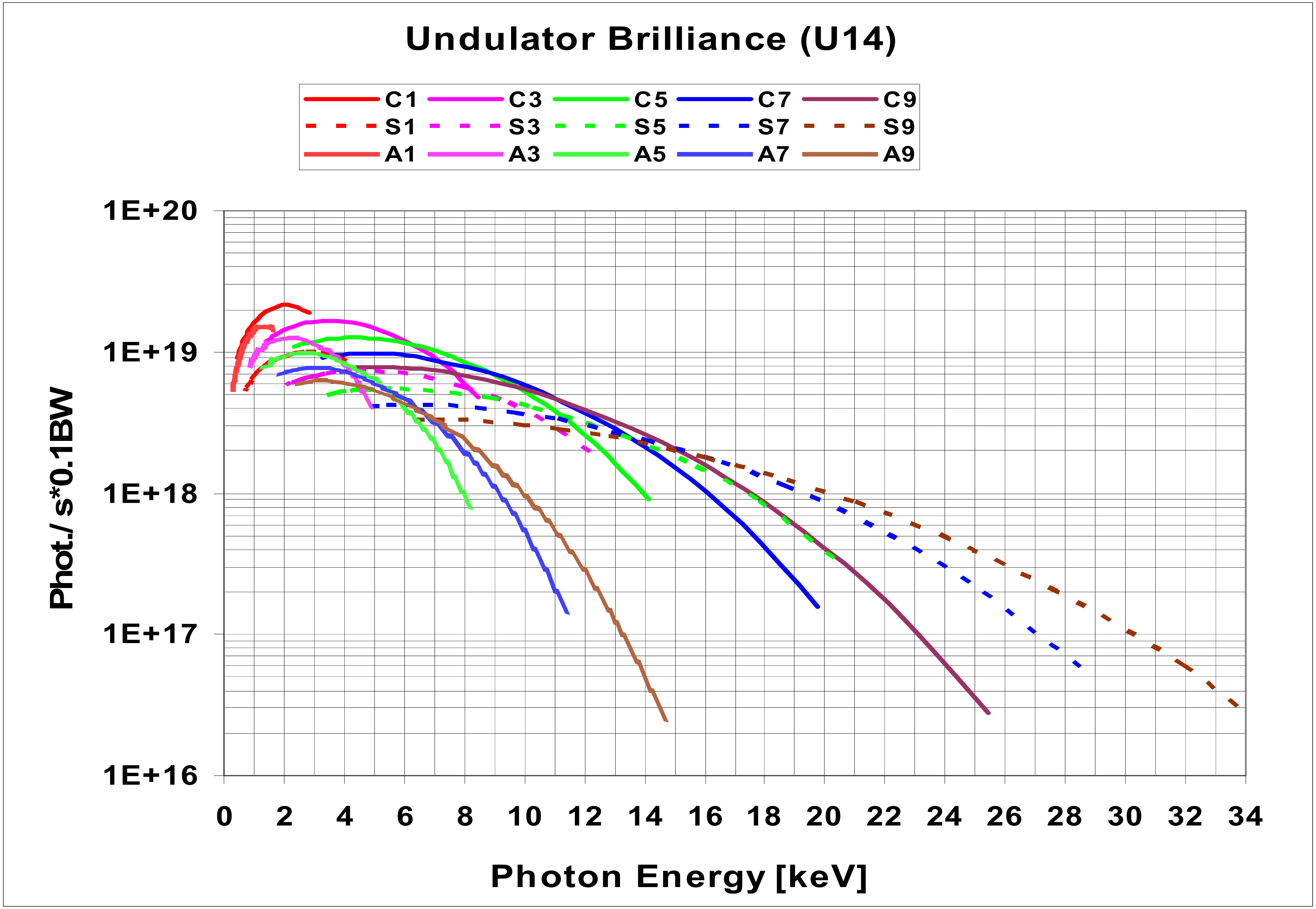
Brilliance from bending magnets



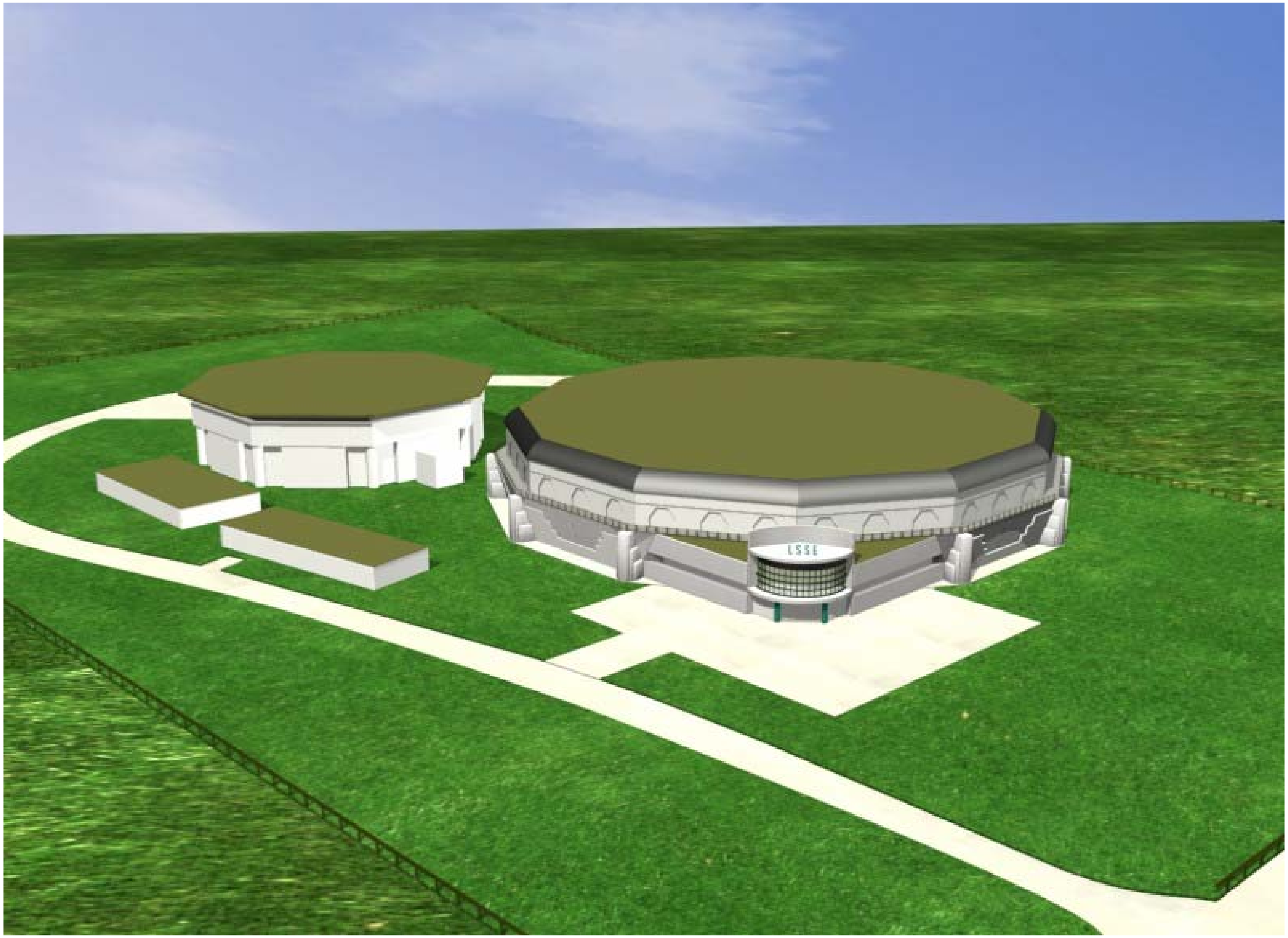
Brilliance from wiggler



Brilliance from undulator

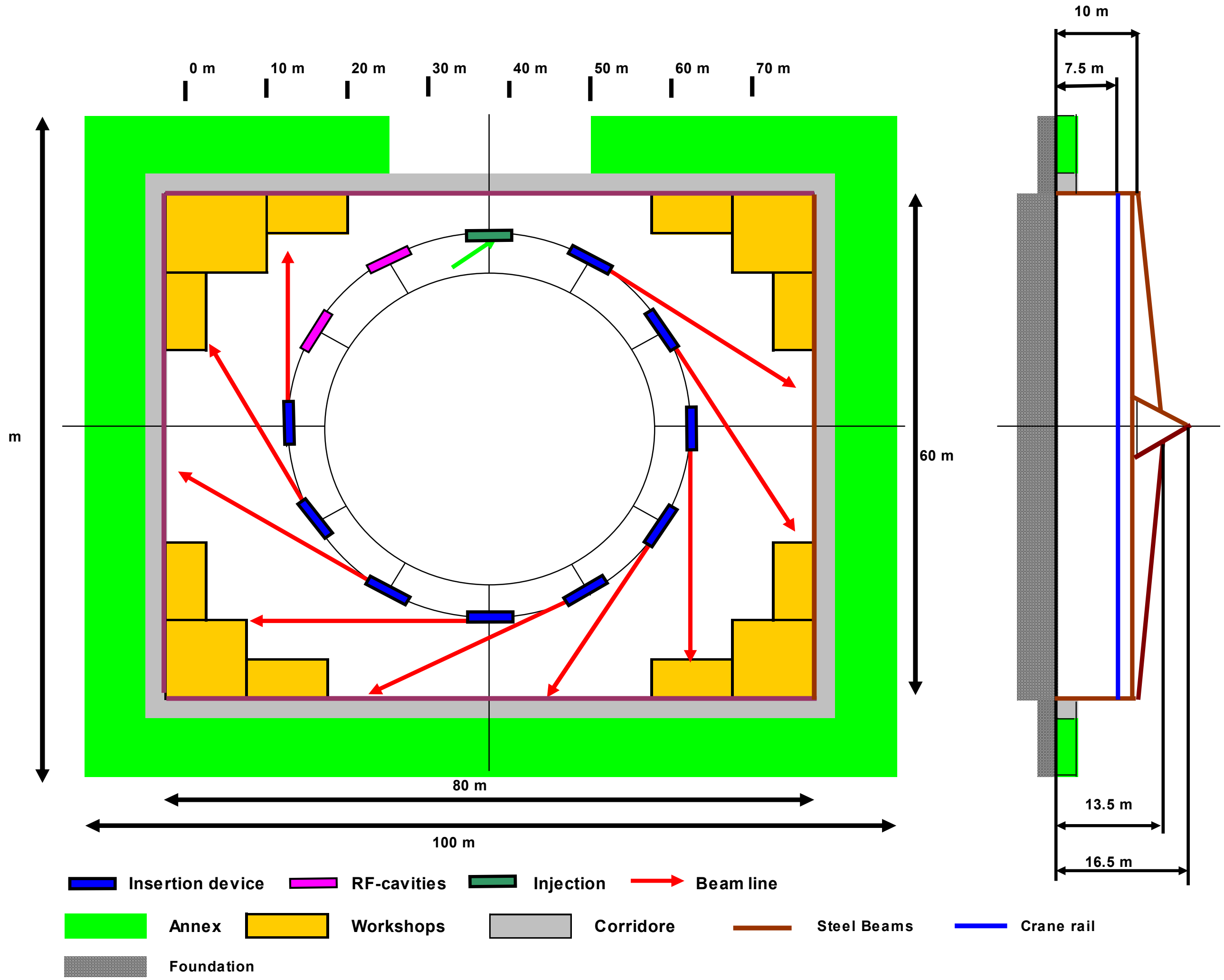


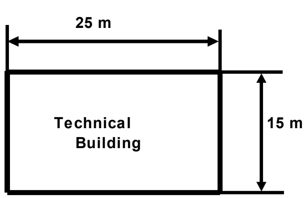
CAMD II: The building



CAMD II: the building

Layout of the building





On the roof of the Technical Building
are three "Chiller Units" with a weight
of 7.5 ton's each

The height of the building is 3.5 m

Cost estimate for the proposed machine

• Machine	34.4 M$	40.3 M$
• 4 beamlines	16.0 M$	16.0 M$
• Buildings	24.7 M$	24.7 M$
• Personnel	11.3 M$	18.3 M$
• Administrative cost	3.0 M$	3.0 M$
• Total	89.4 M$	102.3 M$

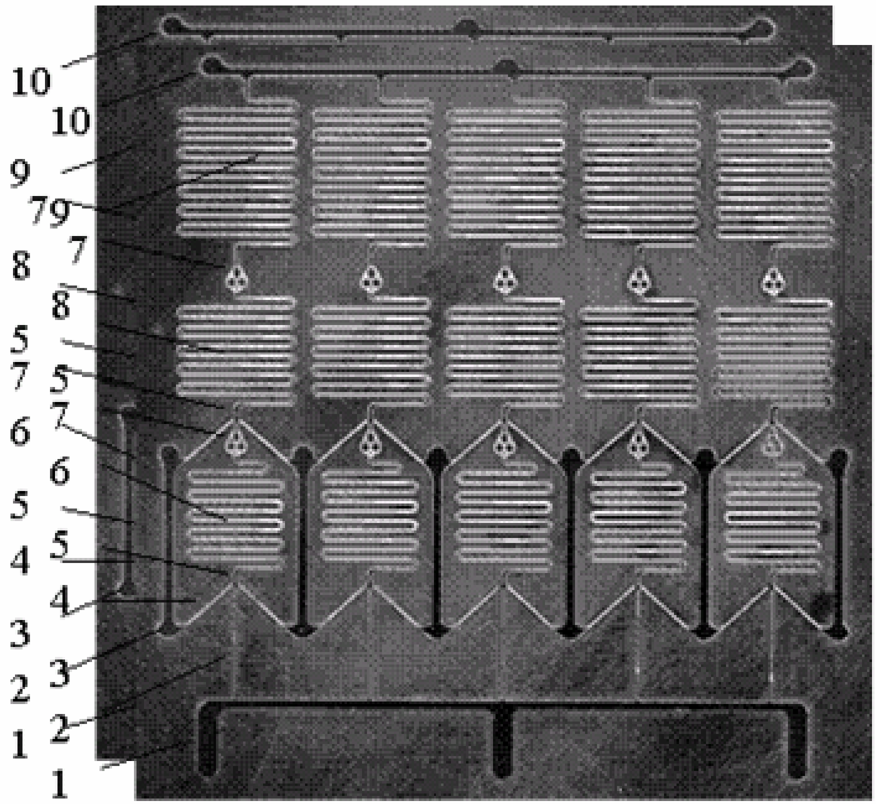
The next steps:

- Evaluation of the machine proposal (Has taken place (October 16/17))
- The Scientific Case (Till February/ March) (Louisiana, Texas, Alabama, Florida, Mississippi, Georgia)
- Presentation at “Upper Administration” + DoE/NSF (asap)

The Scientific Case:

- Medical/Bio-medical applications
- Environmental research
- Surface & interfaces (UV/VUV)
- Material Sciences (Nano-technology)
- Chemical applications (Polymers, catalysis..)

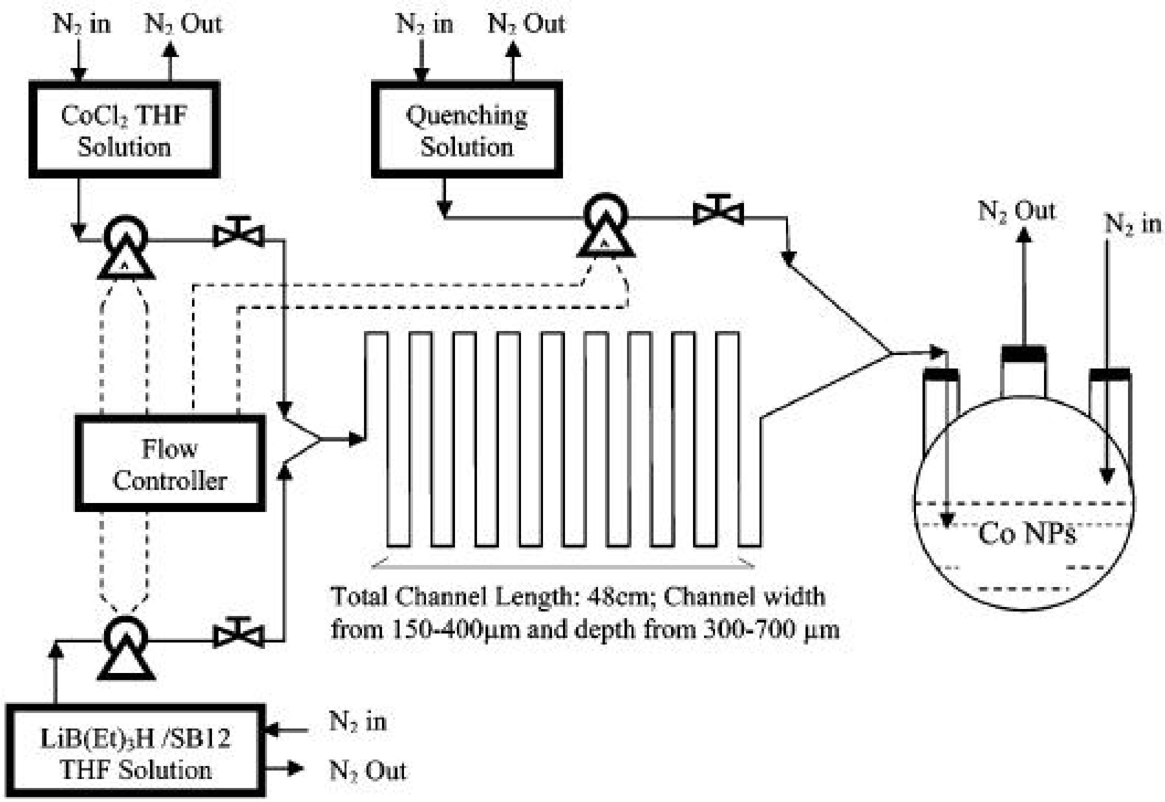
A polymeric microreactor for synthesis of nanoparticles

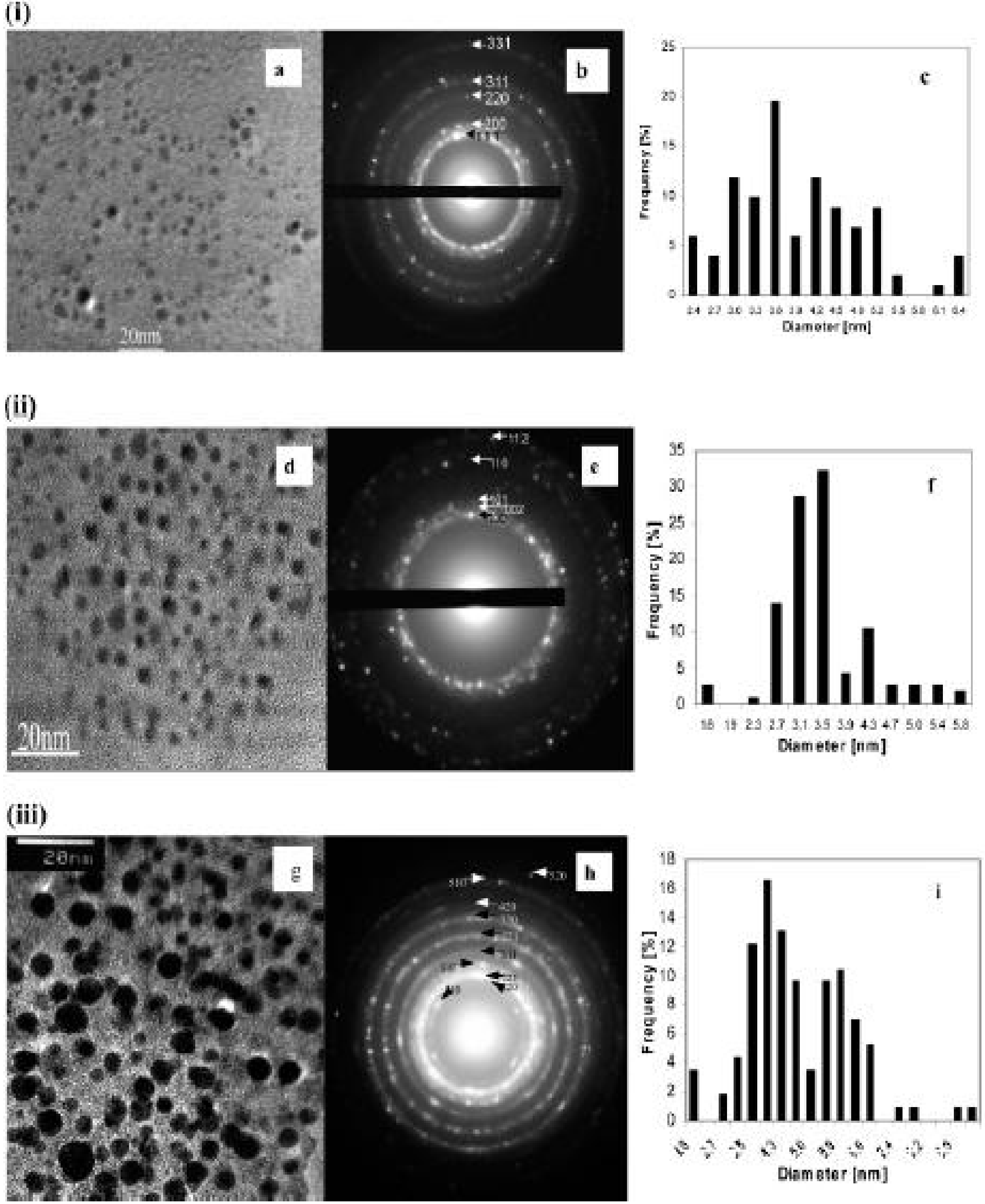


Challa1.wmv

Advantages: continuous and better controlled synthesis!

Micro-reactor set-up for the synthesis of Co nanoparticles



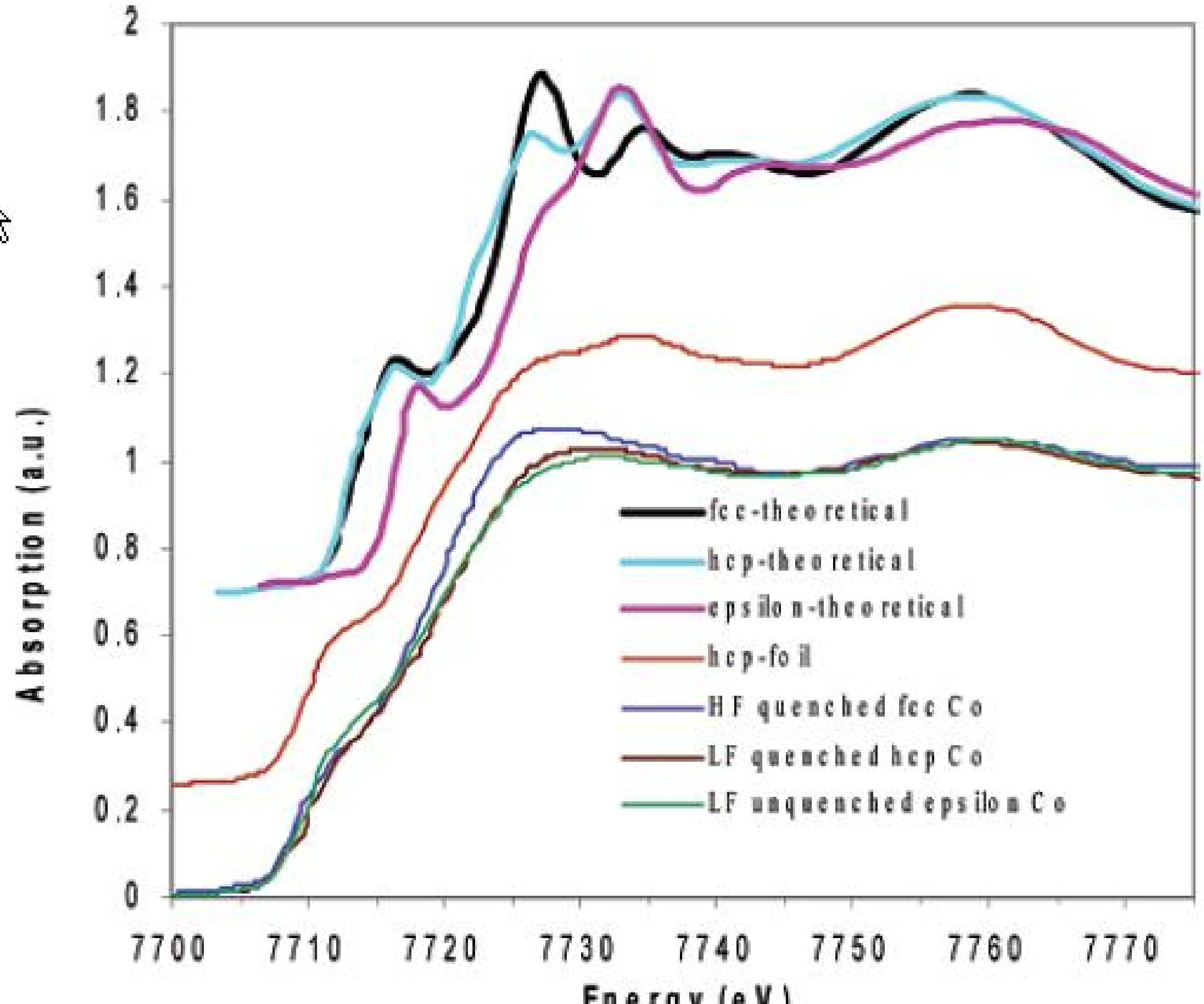


High flow rate
Fast quench

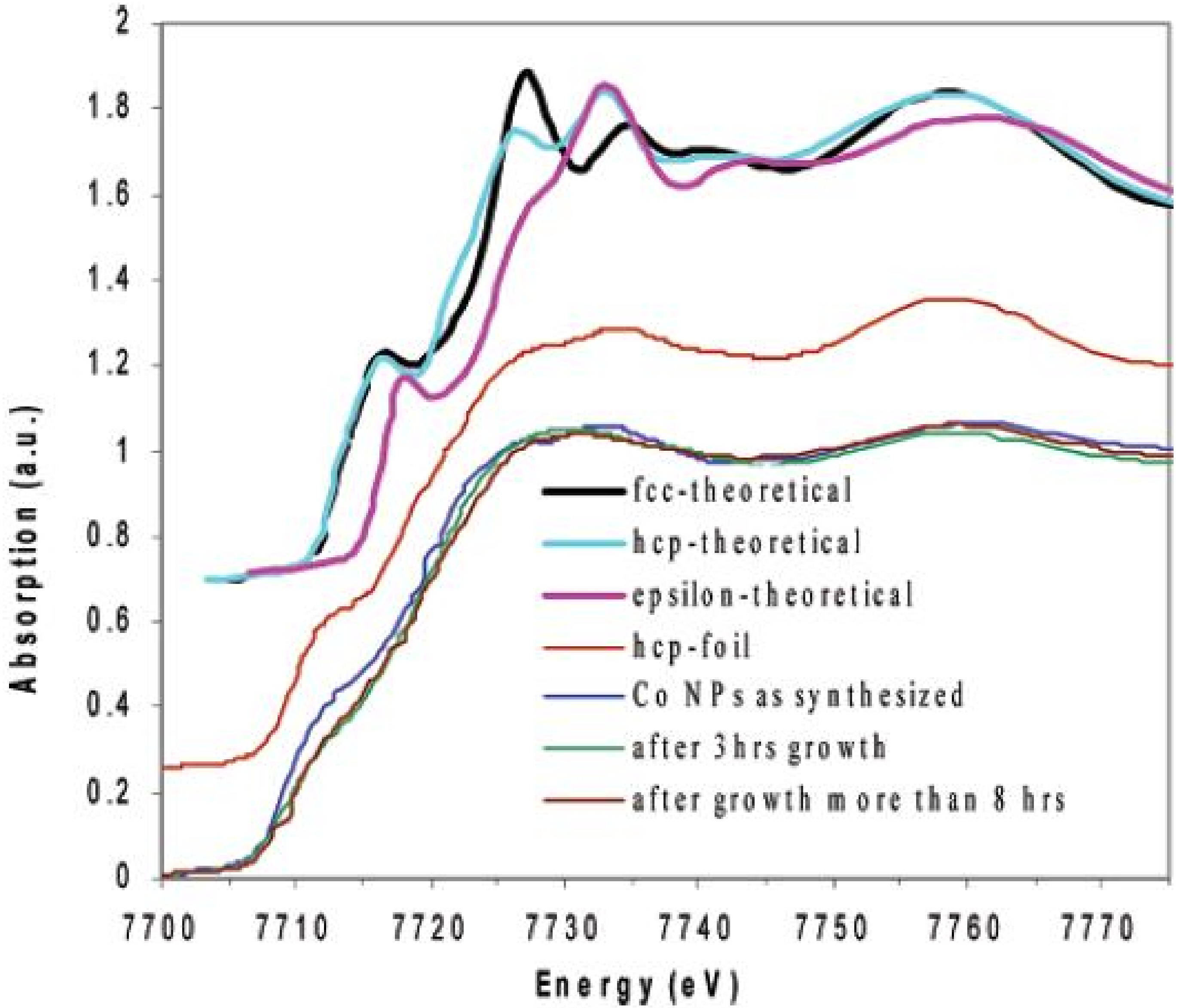
Low flow rate
Fast quench

Low flow rate
Late quench

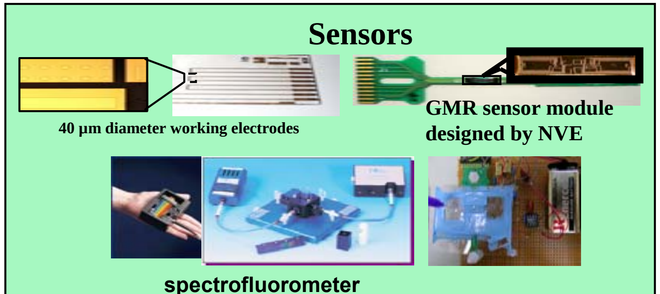
Micro-reactor synthesized Co nanoparticles

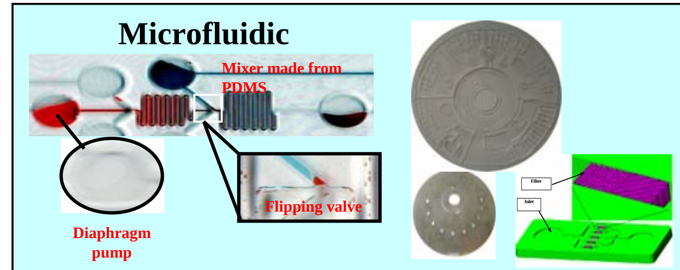


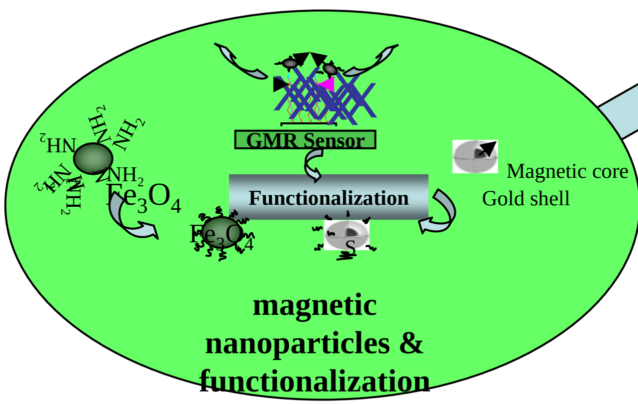
Micro-reactor synthesized Co nanoparticles

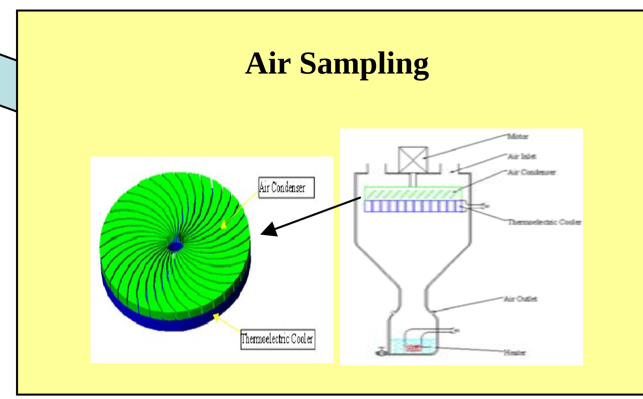


Sensor development based on GMR effect + functionalized magnetic nanoparticles



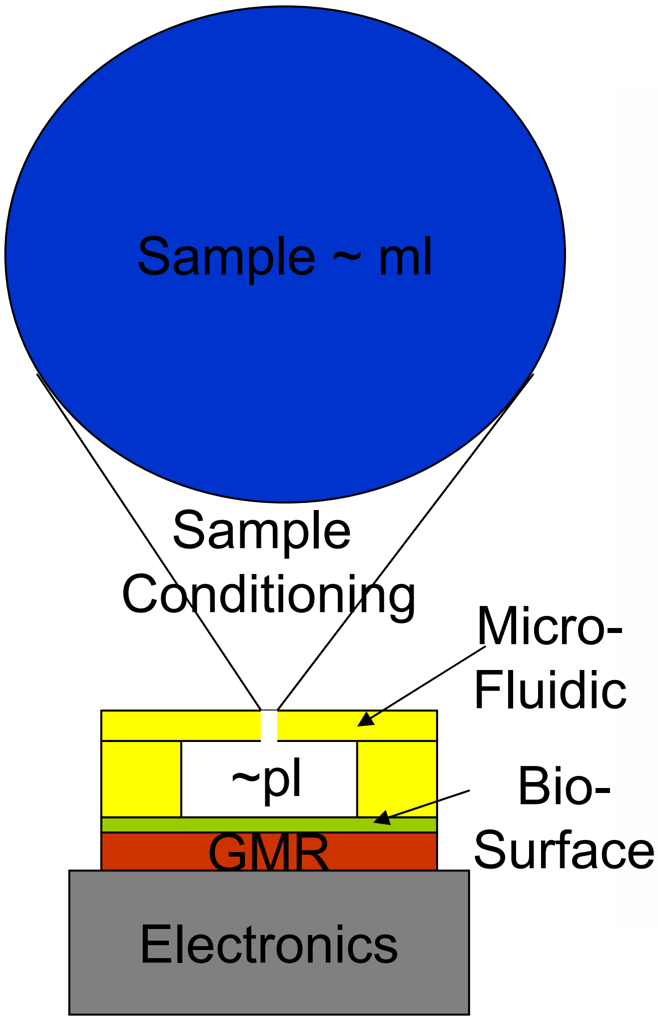


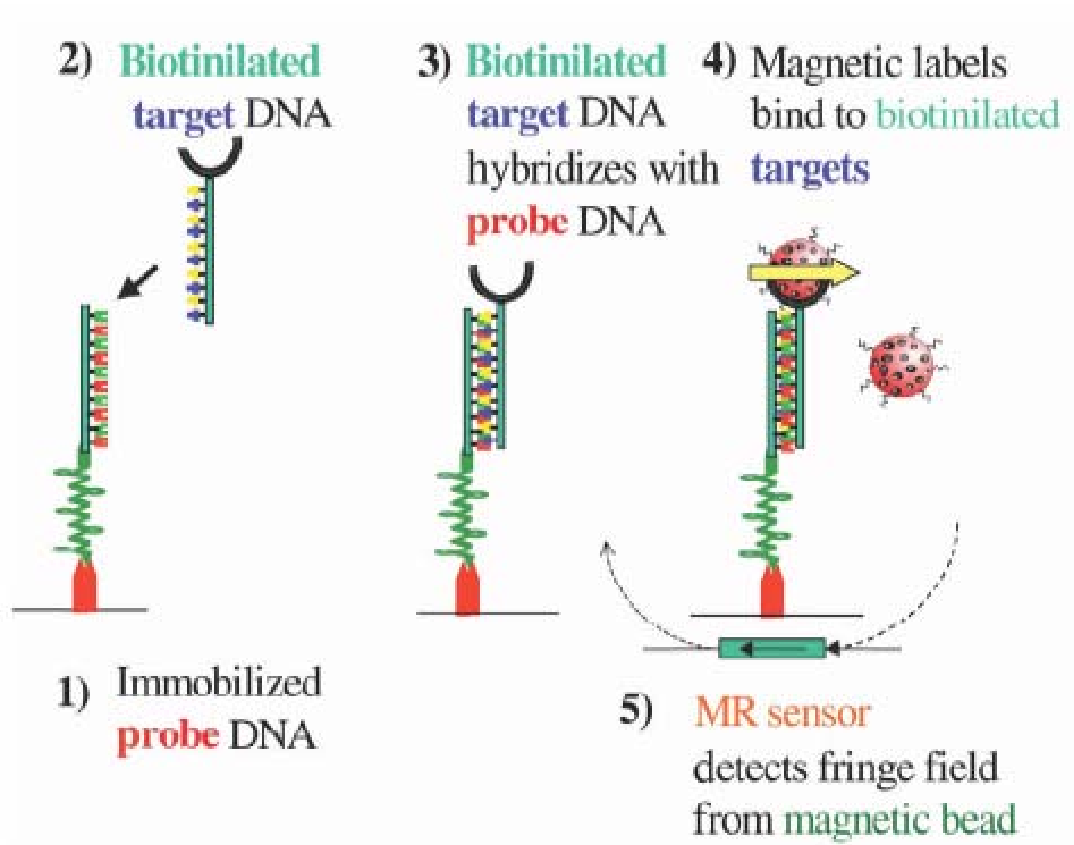




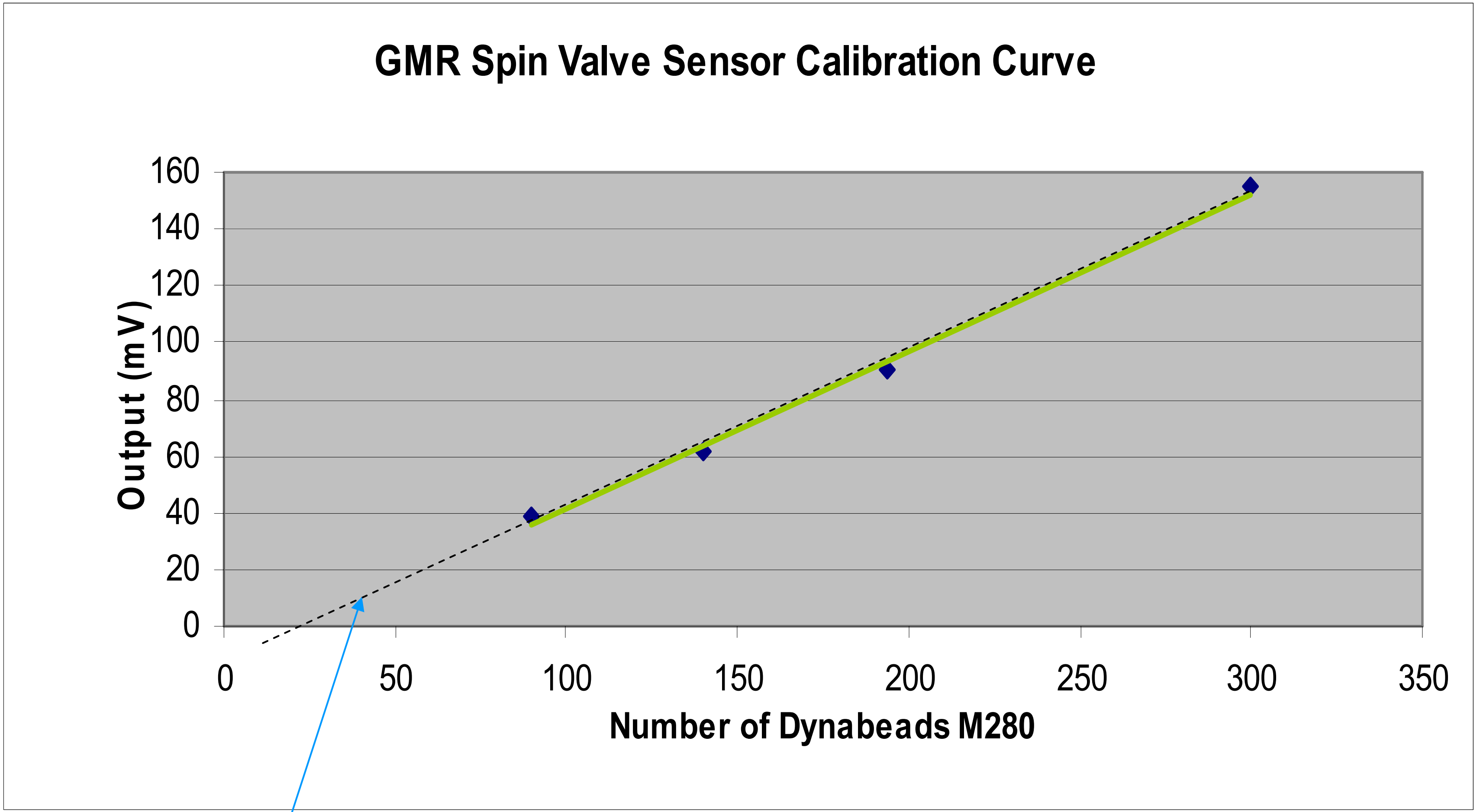
Miniaturized sensor system for bio-chemical warfare agents detection

Detecting "bio-molecules" with a GMR sensor using bio-recognition





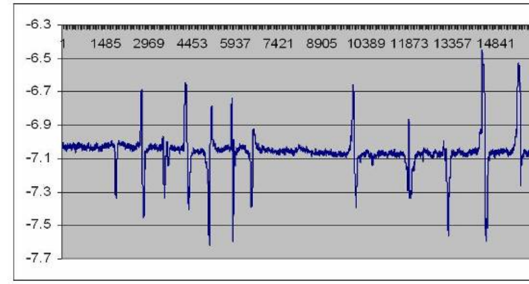
100×100μm² Sensor Output vs Number of Beads

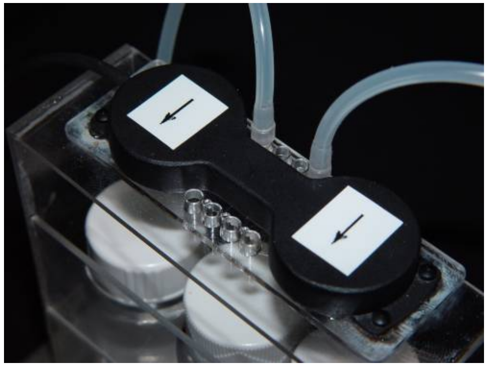


Detection limit ≈ 40 M280, ~2.5% Coverage with 8mV noise
12V bias, 2V field, full bridge, 200um step

GMR Sensor System

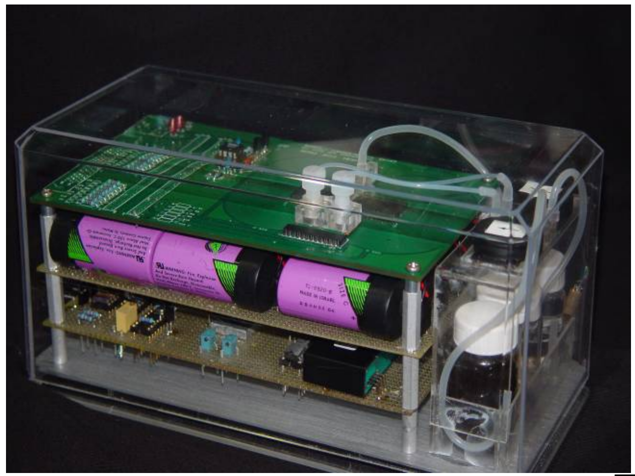
Sensor signal

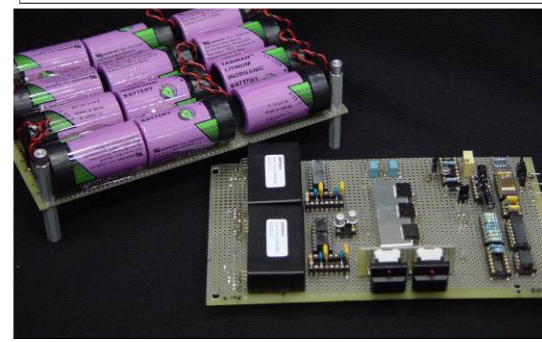




Fluidic handling

Integrated System

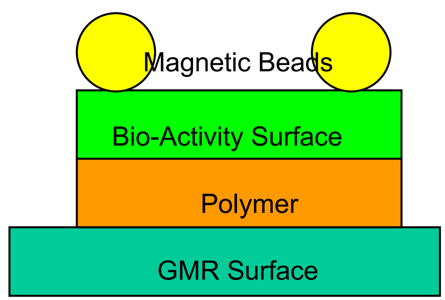




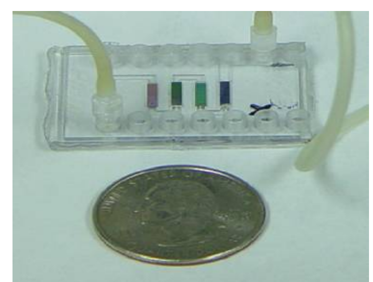
Electronic control and utilities

Microfluidic design

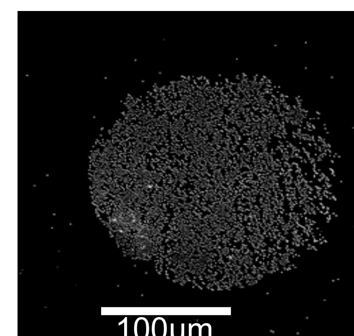
GMR Diving Board



Bio - Surface

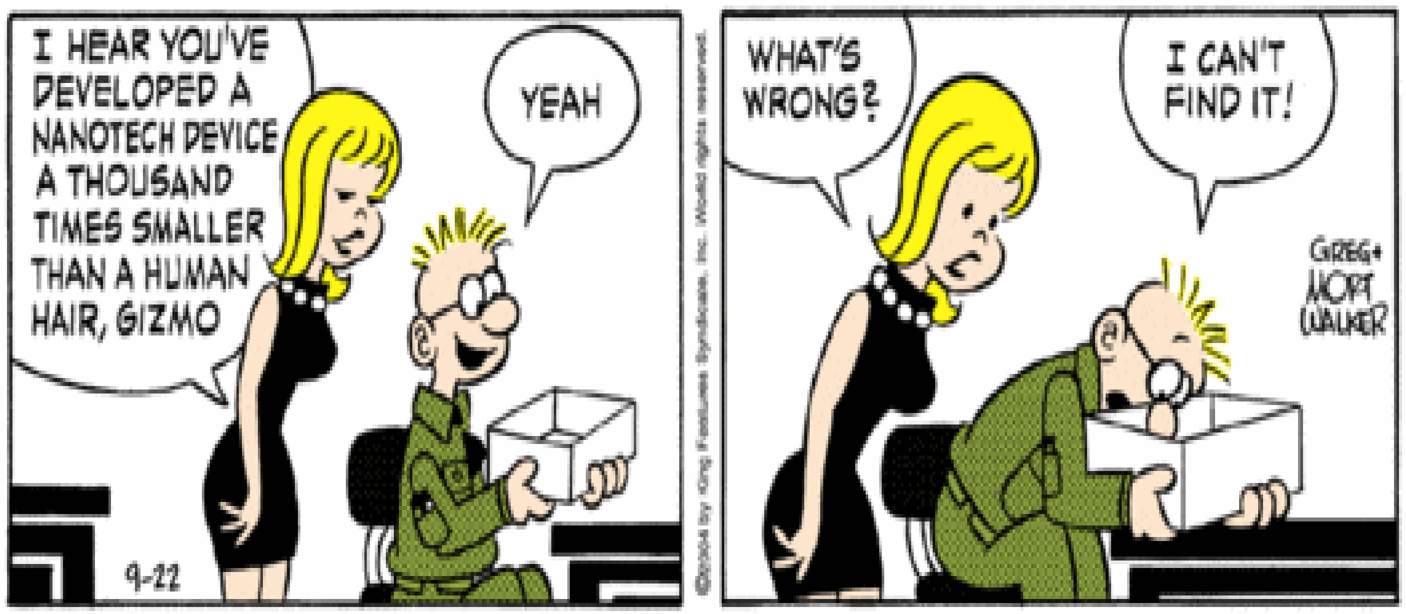


Lab-Ware



Bead Binding

Nanotechnology is still full of surprises



by Mort Walker

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