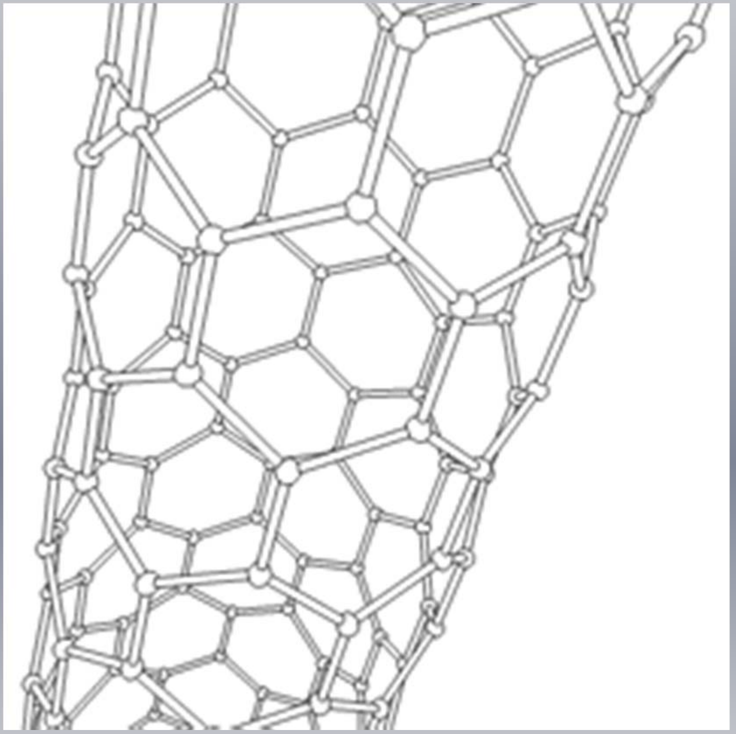
Carbon Nanotubes

Dirks, Carina.[1], Happel, S.[2], Jungclas, H.[1]

[1] Radiochemistry, Department of Chemistry, Philipps-University Marburg, Marburg, Germany
[2] TrisKem International, Bruz, France

Scope

- What are Carbon Nanotubes?
  - Properties
- Coating of Carbon Nanotubes
- Batch experiments
  - Selectivity
- Column experiments
  - Flow rate
- Summary
- Perspectives



http://en.wikipedia.org/wiki/File:Kohlenstoffnanoroehre_Animation.gif

Dimensions of Nanotubes

- **Nano** from Greek (nanos = dwarf)

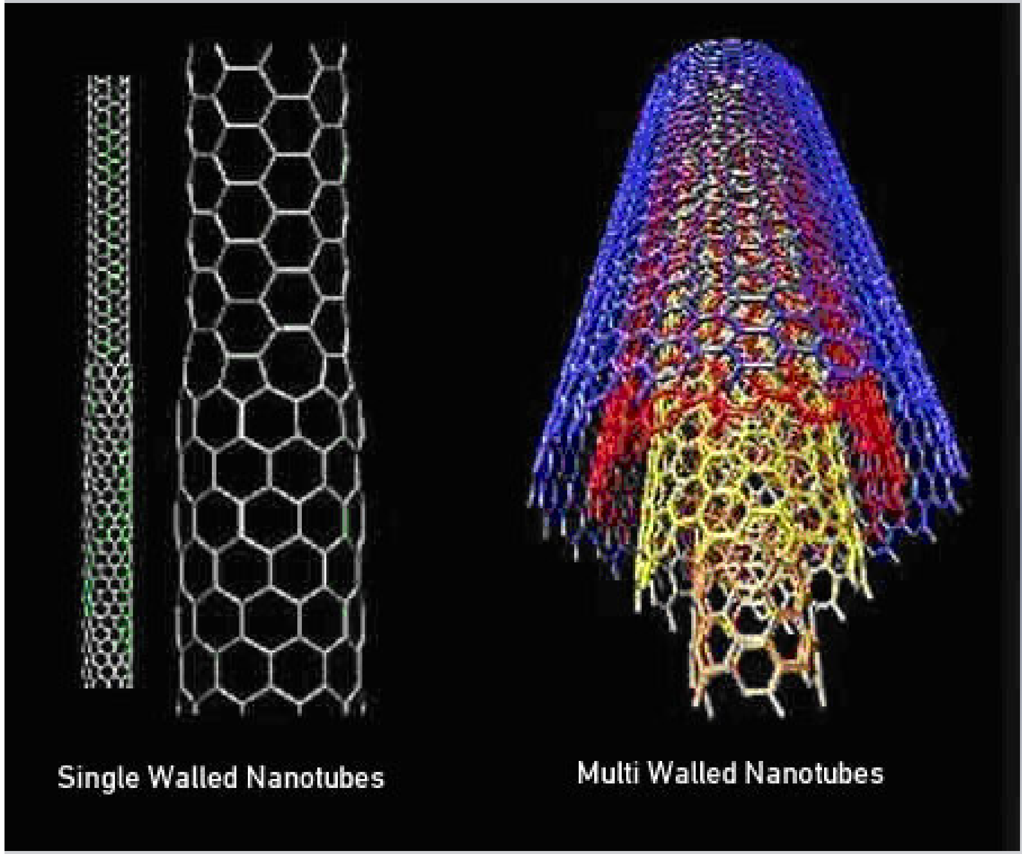
- 1 Nanometer = **1/1 000 000 mm** ≈ 3 Gold atoms

What are Carbon Nanotubes?

- Another C-modification besides graphene, diamant and fullerene
- sp^2 hybridisation
- hexagonal structure → rolled graphene
- Diameter between 0,4 and 100 nm
- Length up to 1 mm (record 20 cm [a])
- Variety of interesting properties
- High specific surface: elevated reactivity

[a] H.W. Zhu, C.L. Xu, D.H. Wu, B.Q. Wei, R. Vajtai und P.M. Ajayan,Science 2002, 296, 884.

Classification of Nanotubes

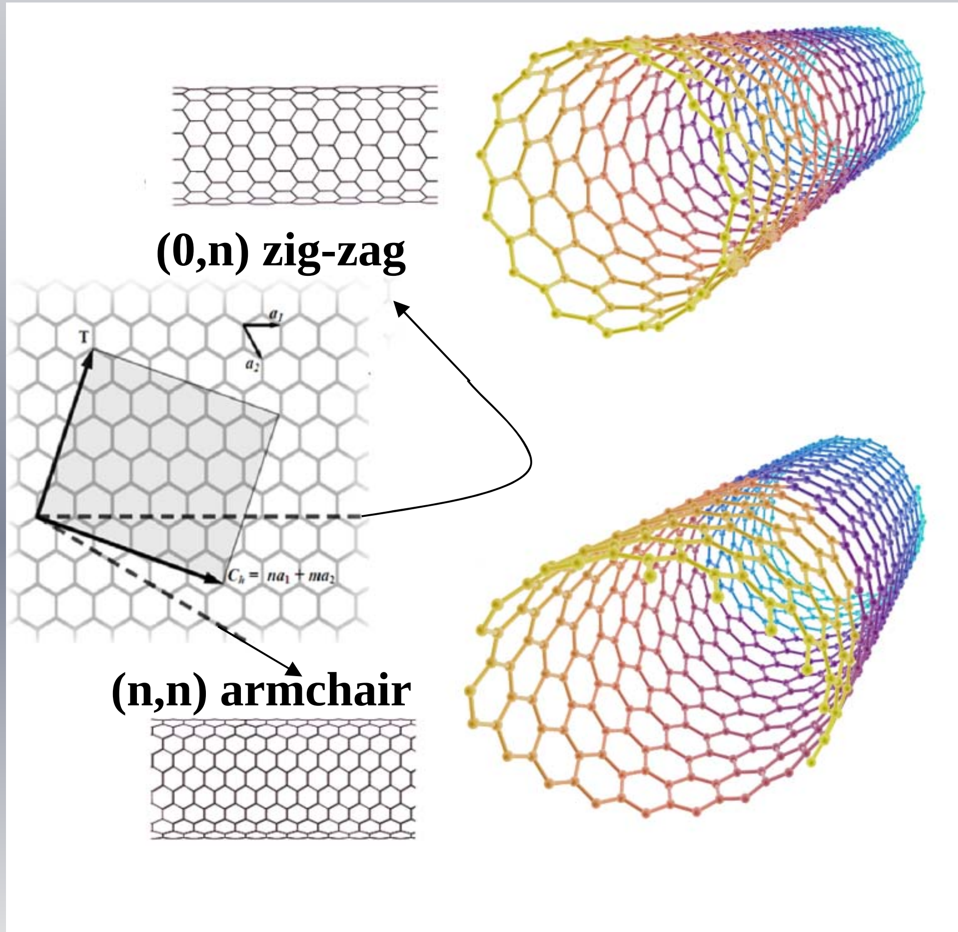


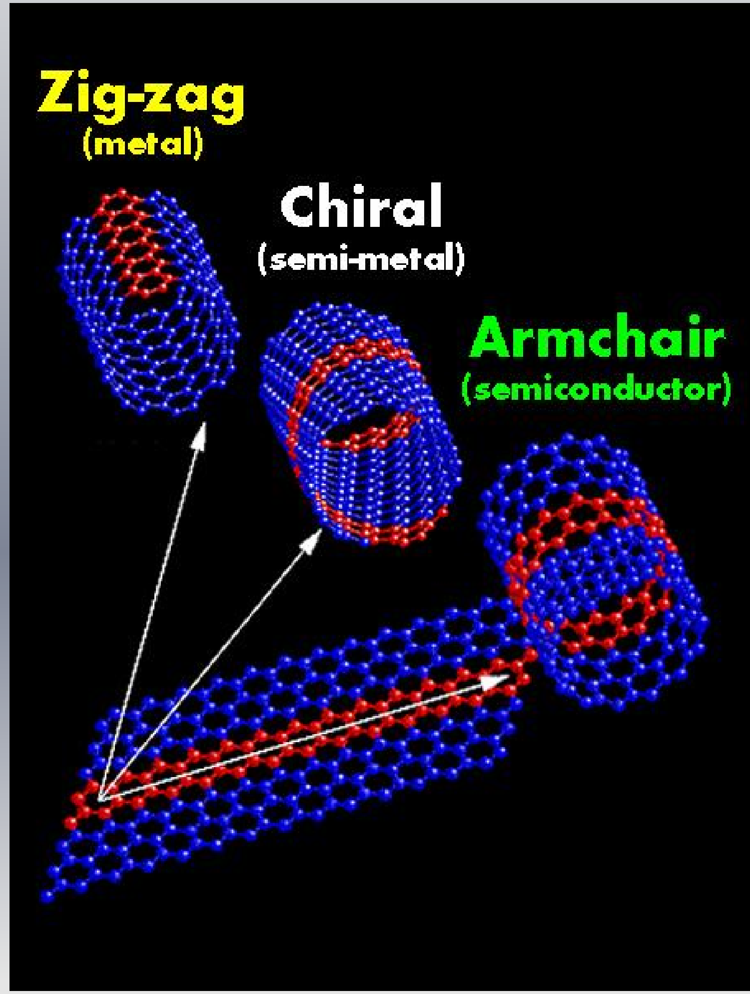
SWCNT Diameter	**Single-walled-carbon-nanotubes:** Rolled Graphene • **1 nm**
MWCNT Diameter	**Multi-walled-carbon-nanotubes:** Concentric SWNCTs • **5-80 nm**

http://www.tedpella.com/gold_html/Nanotubes.htm

http://coecs.ou.edu/Brian.P.Grady/nanotube.html

single-walled Nanotubes





http://www.thenanoage.com/carbon-nanotubes.htm

Properties of the carbon allotropes

Allotrope	Hardness	Tensile strength	Melting behaviour	Conductivity
Coal	+	+	+	No
Graphite	++	++	+++++	+++++
Diamant	+++++	unknown	+++	No
Buckyballs	+++++	++++	+	+
Carbon Nanotubes	++++++	+++++	+++++	++++++

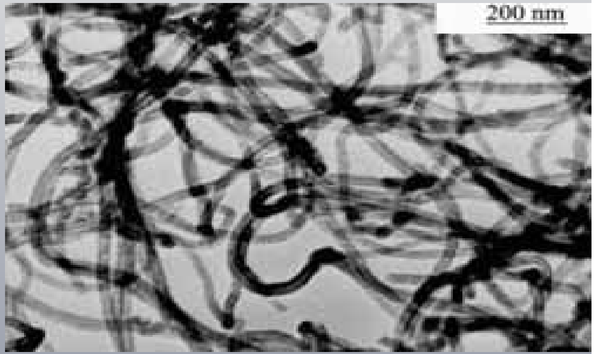
Carbon Nanotubes

- **Properties:**
  - Much higher tensile strength than steel, while lighter than steel
  - Conductivity similar to Cu
  - Thermal conductivity similar to diamant
  - Can be conductor or semi-conductor
  - **High chemical resistance**

- **For use in columns:**
  - MWCNT easier to handle and cheaper

Modified MWCNTs

- MWCNT used:
  - Industrial production (**C**hemical **V**apor **D**eposition)
    - Diameter: > 50 nm
    - Length 10-20 mm

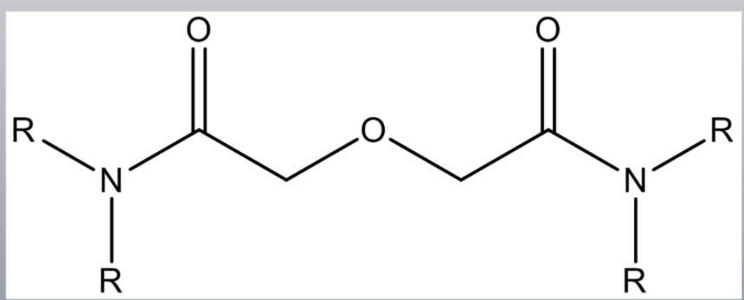


Transmission Electron Microscopy (TEM)

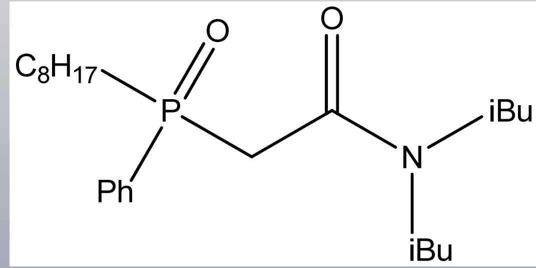
- Coating of MWCNT´s with different extractants
- Batch experiments with U-238 and Am-241 in comparison to DGA and TRU Resin
  - **TDNC vs. DGA**
  - **TTNC vs. TRU**

Extractants

DGA and TRU

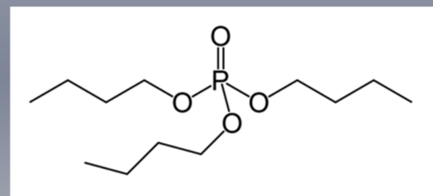


N,N,N’,N’-tetra-n-alkyl-3-oxopentandiamide
(R=C8)



CMPO: (Octyl(phenyl)-N,N-Diisobutyl Carbamoylmethyl Phosphinoxide

+



TBP: Tributylphosphate

TDNC

TTNC

General procedure - batch experiments

D_W (Weight distribution coefficient)

- Weigh 50 mg of the respective resin into a 2 mL Eppendorf cap
- Add 400 µL of the acid to be tested
- Close cap and shake for 30 min (preconditioning)
- Add 1mL of the sample solution
- (e.g.. 1 mL multi-element solution)
- Close cap and shake for 30 min (analyte extraction)
- Centrifuge
- Withdraw 1 mL of the supernatant, Analysis (ICP-MS)

- All D_W determined in triplicate

D_W

$$D_W = \frac{N_{A_0} - N_A}{N_A} \times \frac{V}{m_R}$$

- high D_w = Extraction
- low D_w = Elution

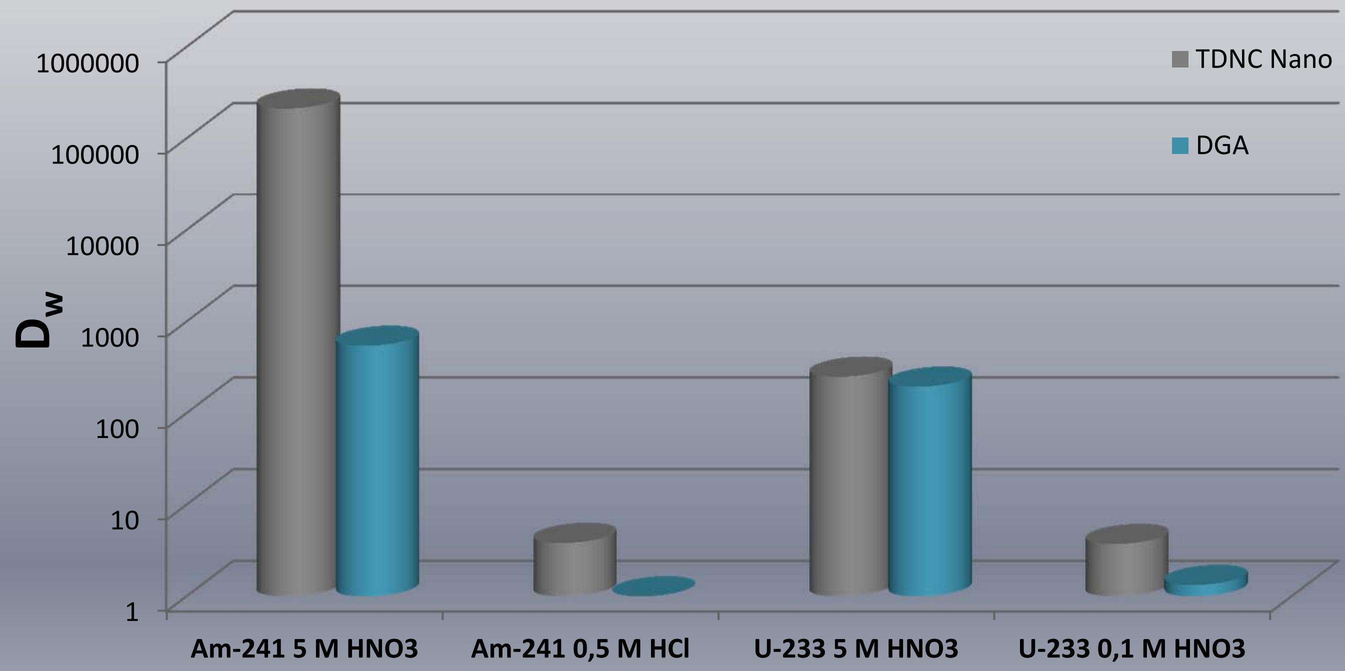
N_{Ao} = Net count rate A_o sample

N_A = Net count rate sample

V = Volume aq. phase (1,4 mL)

m_R = amount of resin in g

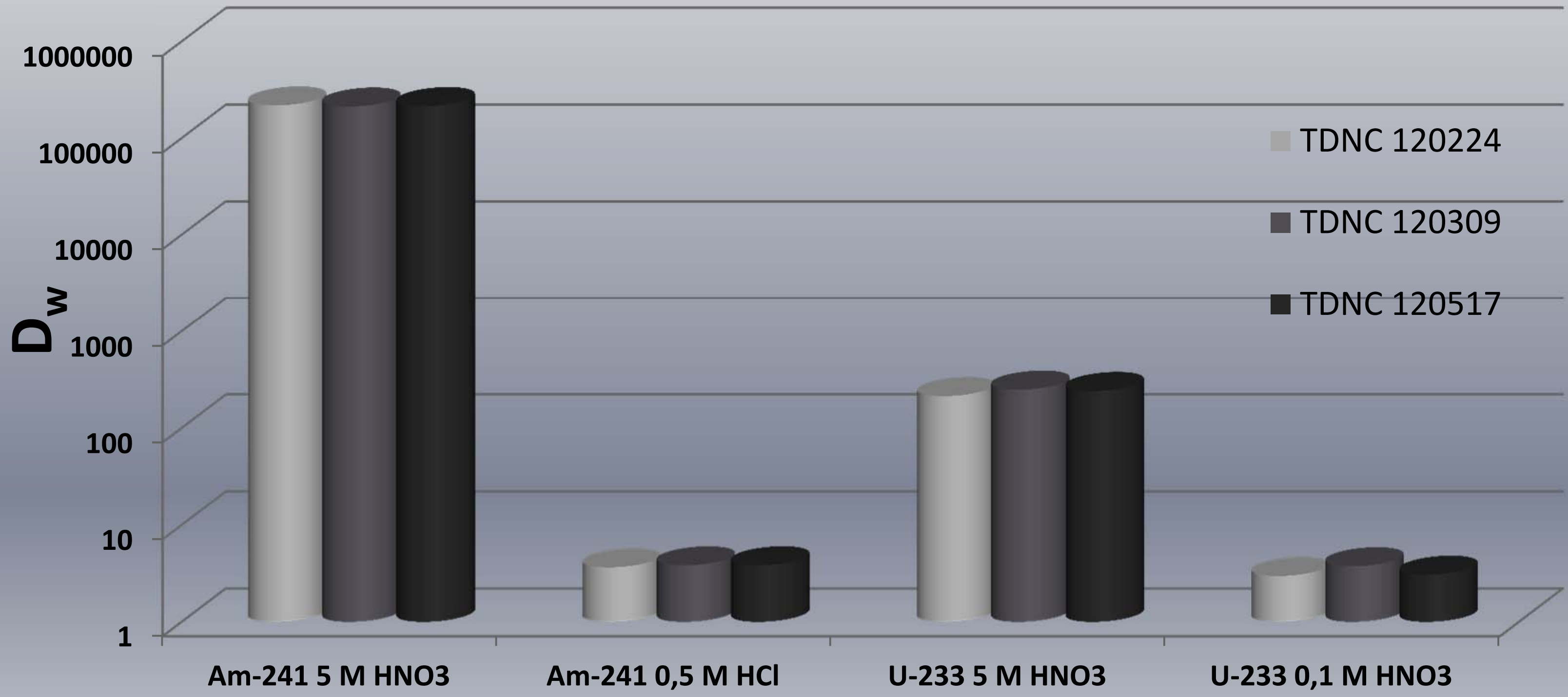
TDNC vs. DGA



Comparison coated MWCNT (TDNC) vs DGA Resin

Very high D_w Koeffizienten on TDNC especially for Am

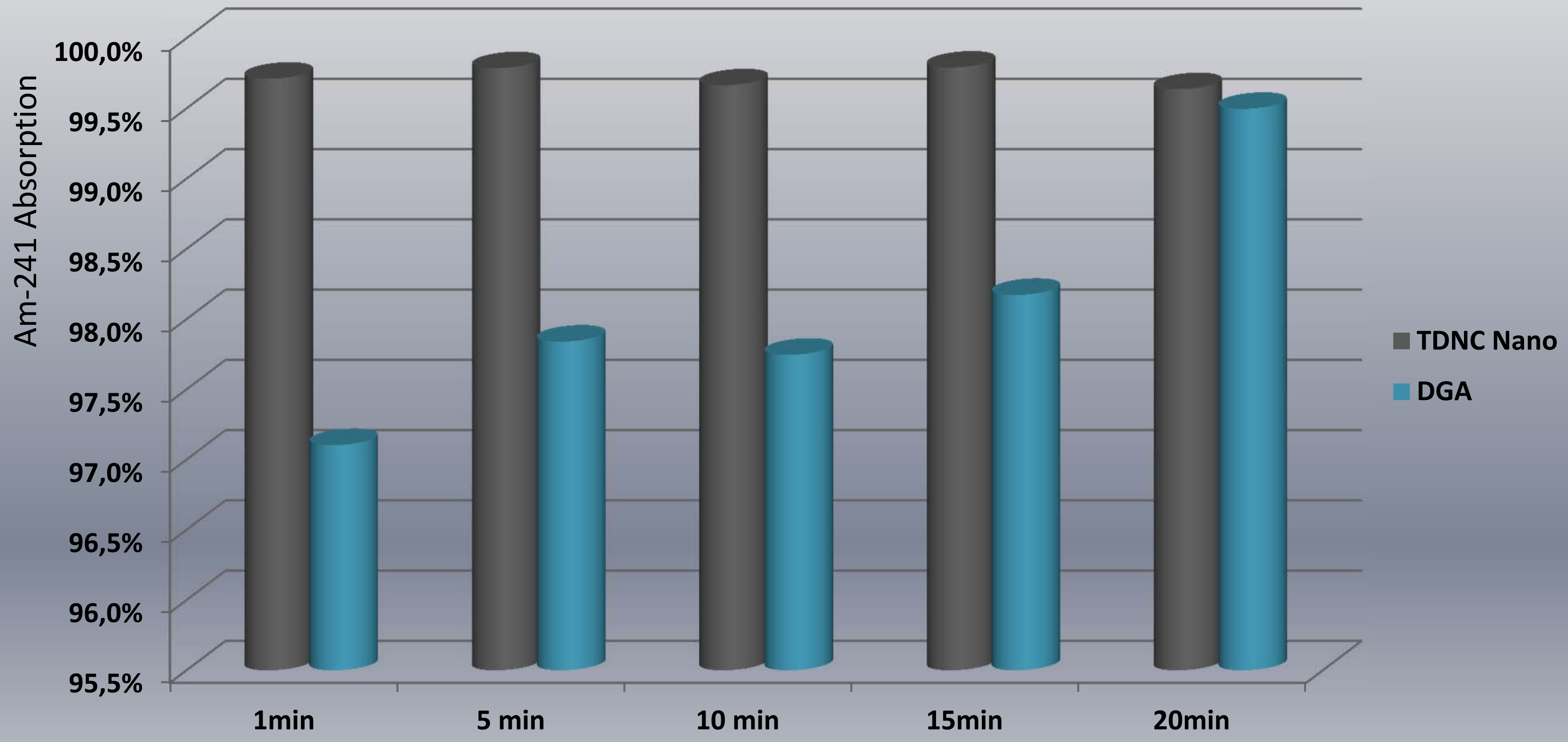
Reproducibility of TDNC coating



Comparison Am D_W for different coated MWCNT (TDNC) lots

- Coating reproducible

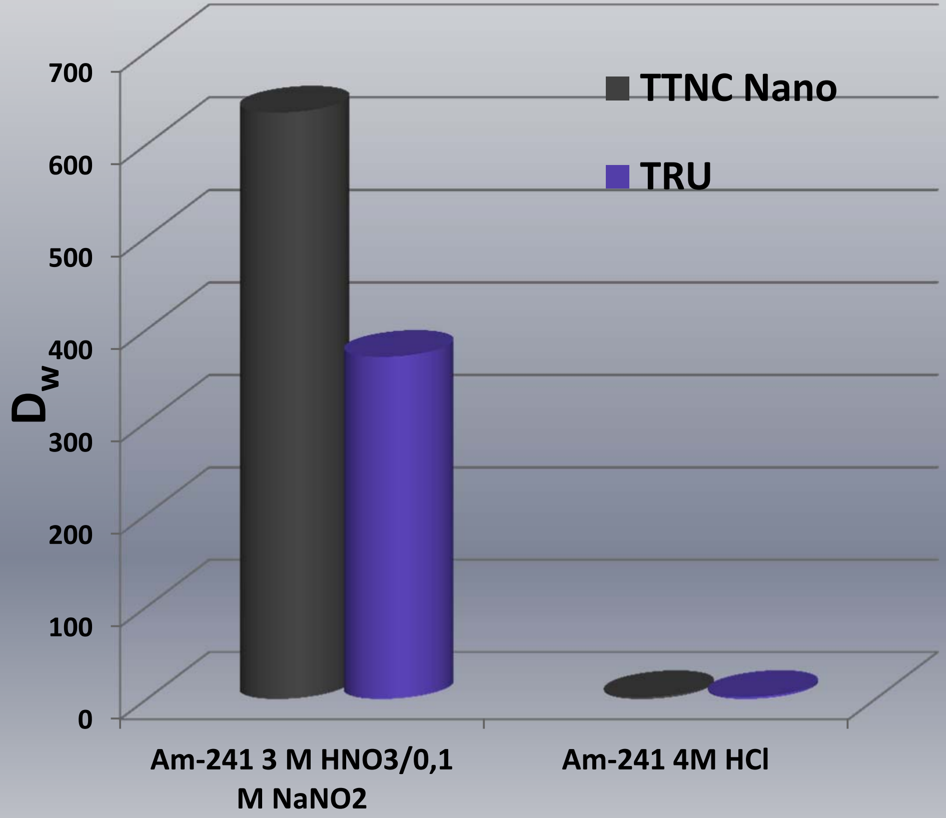
Kinetics: TDNC vs. DGA



Uptake kinetics coated MWCNT (TDNC) and DGA Resin

Very fast kinetics: eq. after < 1min

TTNC vs. TRU



Comparison Am D_W coated MWCNT (TTNC) and TRU Resin

High D_W

TTNC higher D_w - for Am than TRU

Problems of working with Nanotubes

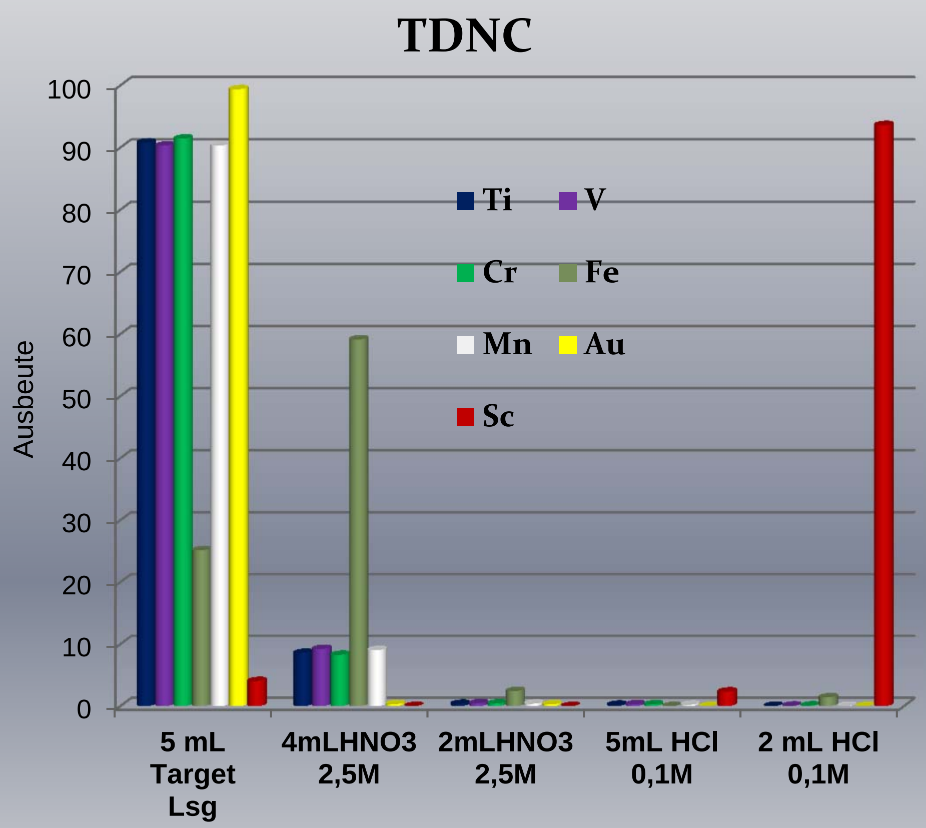
- Transfer from batch experiments to column geometry very difficult
- Low flow-rates
- Finding suitable filter / pore sizes

- Some solutions:
  - Special packing technique
  - Adequate poresize identified
  - Flowrates between 1-5 mL possible (using vacuum)

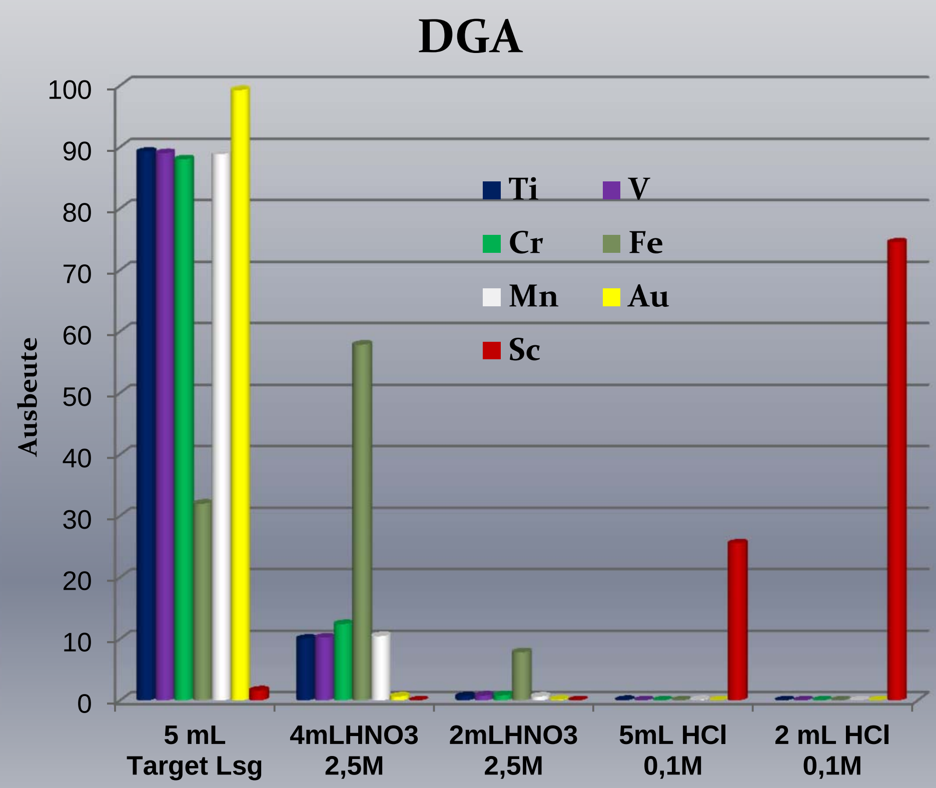
Conclusions I

- ✓ TDNC and TTNC show very promising D_W in batch experiments
- ✓ Very rapid kinetics
- ✓ Column experiments possbile
- ✓ Suitable flow-rates achievable

TDNC vs. DGA simulated Ti target

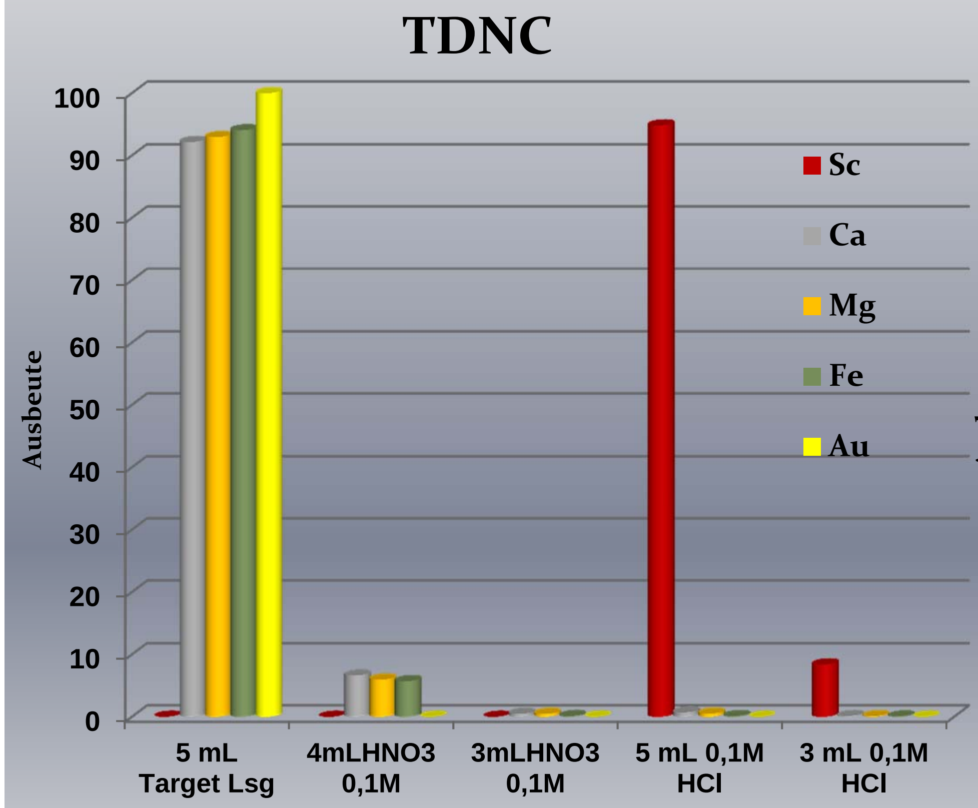


Elution study, simulated Ti target, coated MWCNT (TDNC)

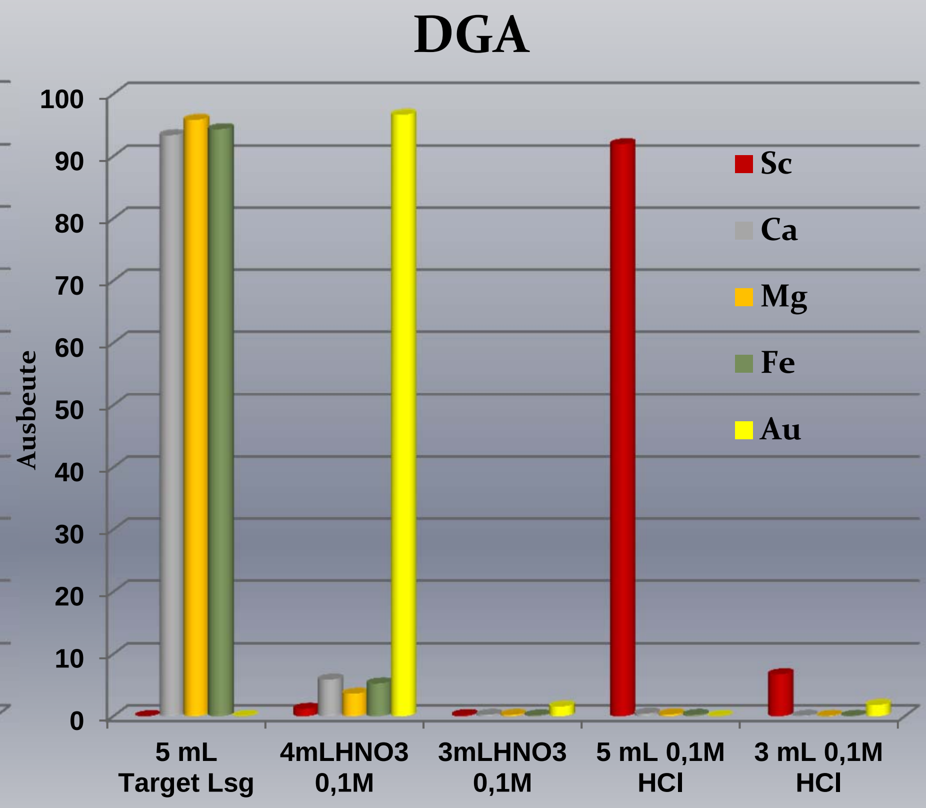


Elution study, simulated Ti target, DGA resin

TDNC vs. DGA simulated Ca target

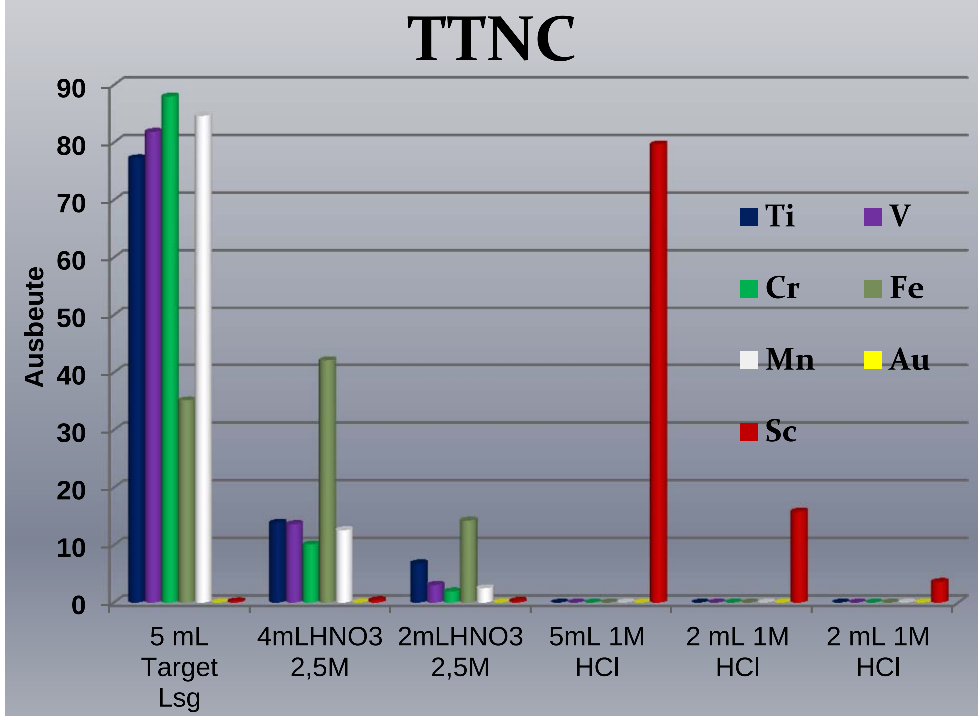


Elution study, simulated Ca target, coated MWCNT (TDNC)

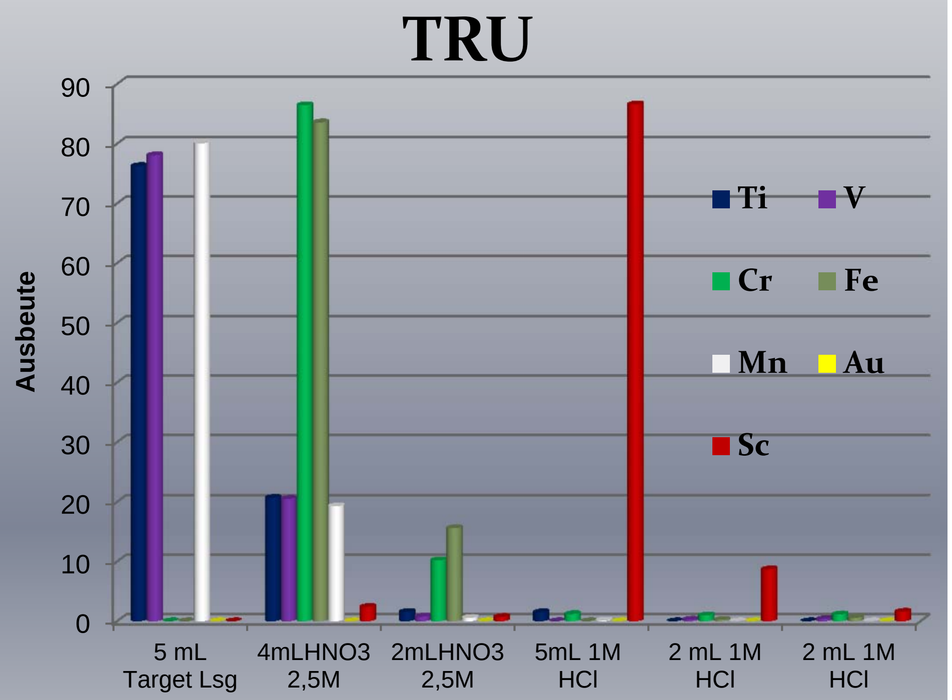


Elution study, simulated Ca target, DGA resin

TTNC vs. TRU simulated Ti target

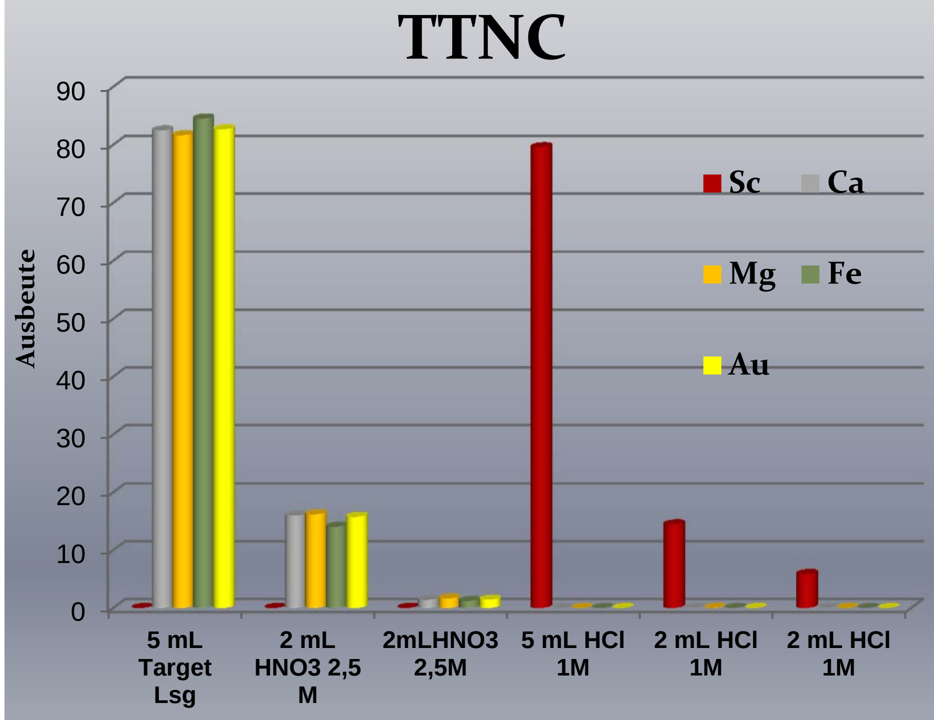


Elution study, simulated Ti target, coated MWCNT (TTNC)

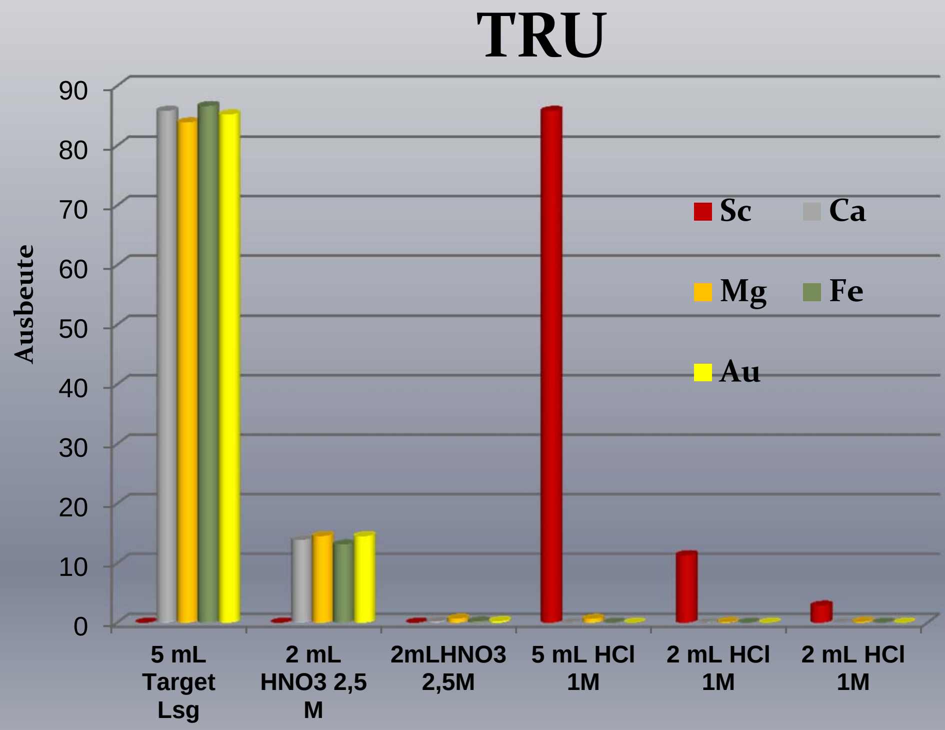


Elution study, simulated Ti target, TRU resin

TTNC vs. TRU simulated Ca target



Elution study, simulated Ca target, coated MWCNT (TTNC)

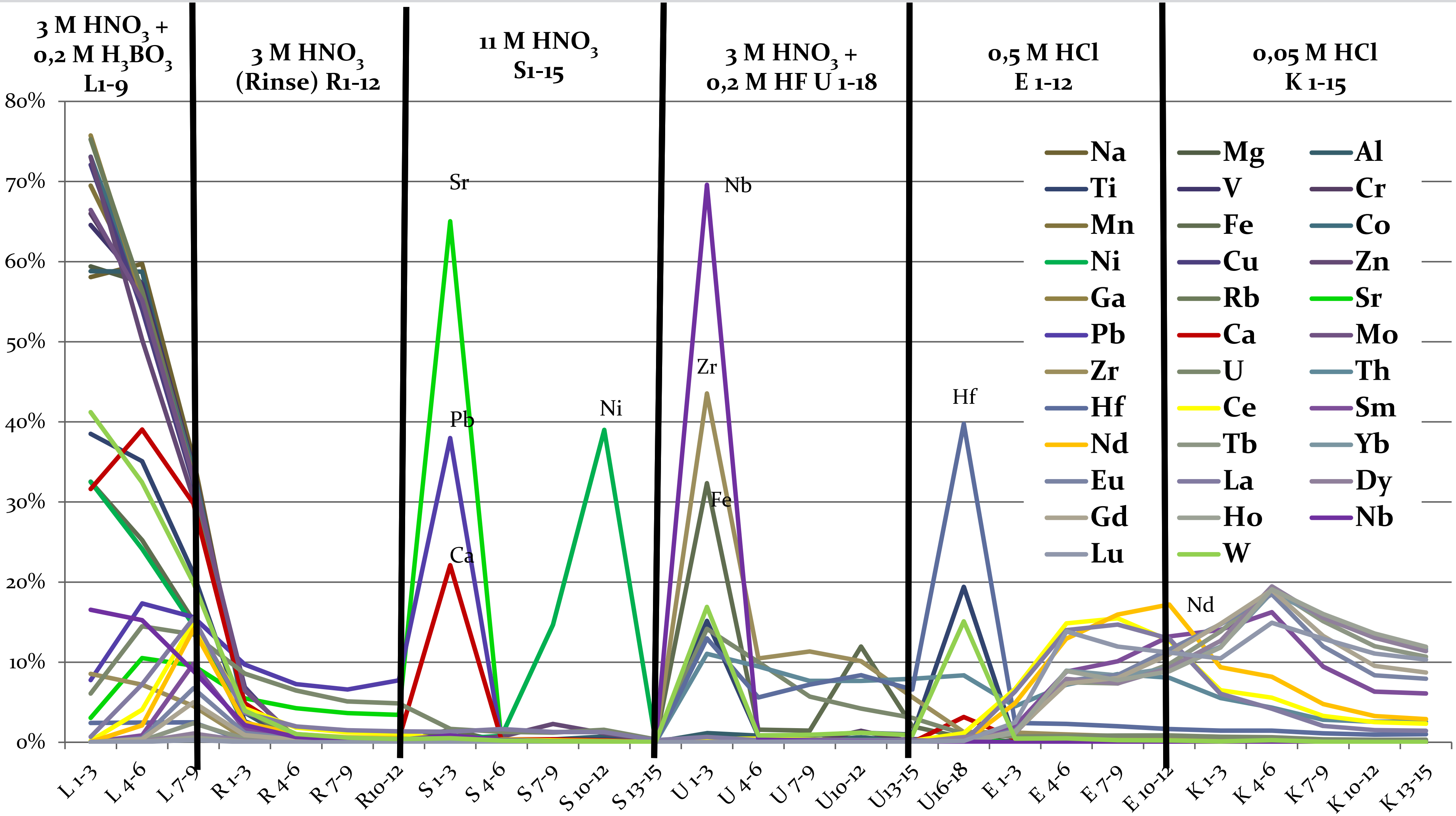


Elution study, simulated Ca target, TRU resin

Conclusions II

- Developed separation methods for Scandium (DGA and TRU) directly applicable to coated MWCNT
- Very good separation performance in column tests
- Adequate flow rates

Complex separation on TDNC



Perspectives

- Optimisation:
  - Flow rate
  - Extraktant / Nanotube ratio
  - Column volume
- Improvement / variation of column material

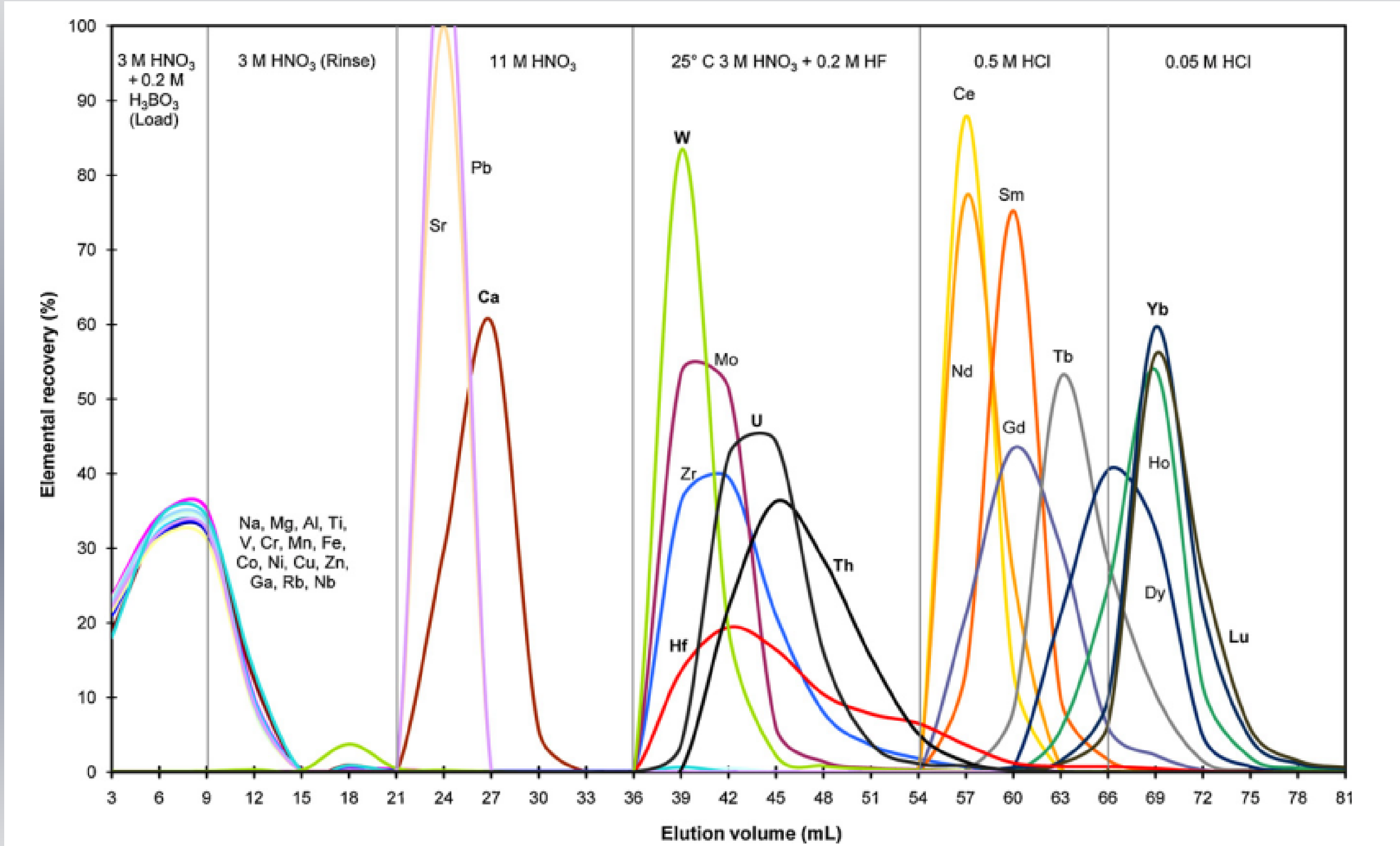
Thank you very much

- Prof. Jungclas, Kernchemie Marburg
- TrisKem International

Optimized method

- Vacuum supported flow (1 – 3 mL/min)
  - TDNC resin (400 mg)
    - Load from 5 mL 2,5 M HNO_3 (Ti) or 0,1M HNO_3 (Ca)
    - Rinse with 4 mL and 3 mL 2,5 M HNO_3
    - Load and rinse contain ~100% Ti bzw. Ca
    - Sc elution in 5 and 3 mL 0,1M HCl
  - TTNC resin (400 mg)
    - Load from 5 mL 2,5 M HNO_3
    - Rinse with 2 x 2 mL 2,5 M HNO_3
    - Load and rinse contain ~100% Ti bzw. Ca
    - Sc elution in 5 and 2 mL 1 M HCl
  - Sc Yield > 98%
- Separation time: 10 min

TODGA



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