Behavior of the ^{222}Rn daughters on the copper surface during cleaning

M. Wojcik[a], G. Zuzel[b]

[a] *M. Smoluchowski Institute of Physics, Jagellonian University, 30-059 Krakow, Poland*

[b] *Max Planck Institute for Nuclear Physics, 69117 Heidelberg, Germany*

Outlook

Technique

- Testing cleaning procedures

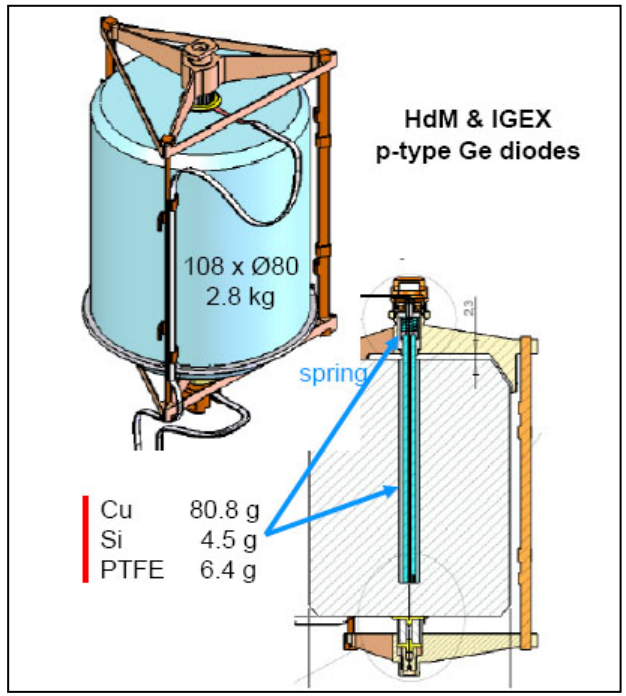
  - etching

  - electropolishing

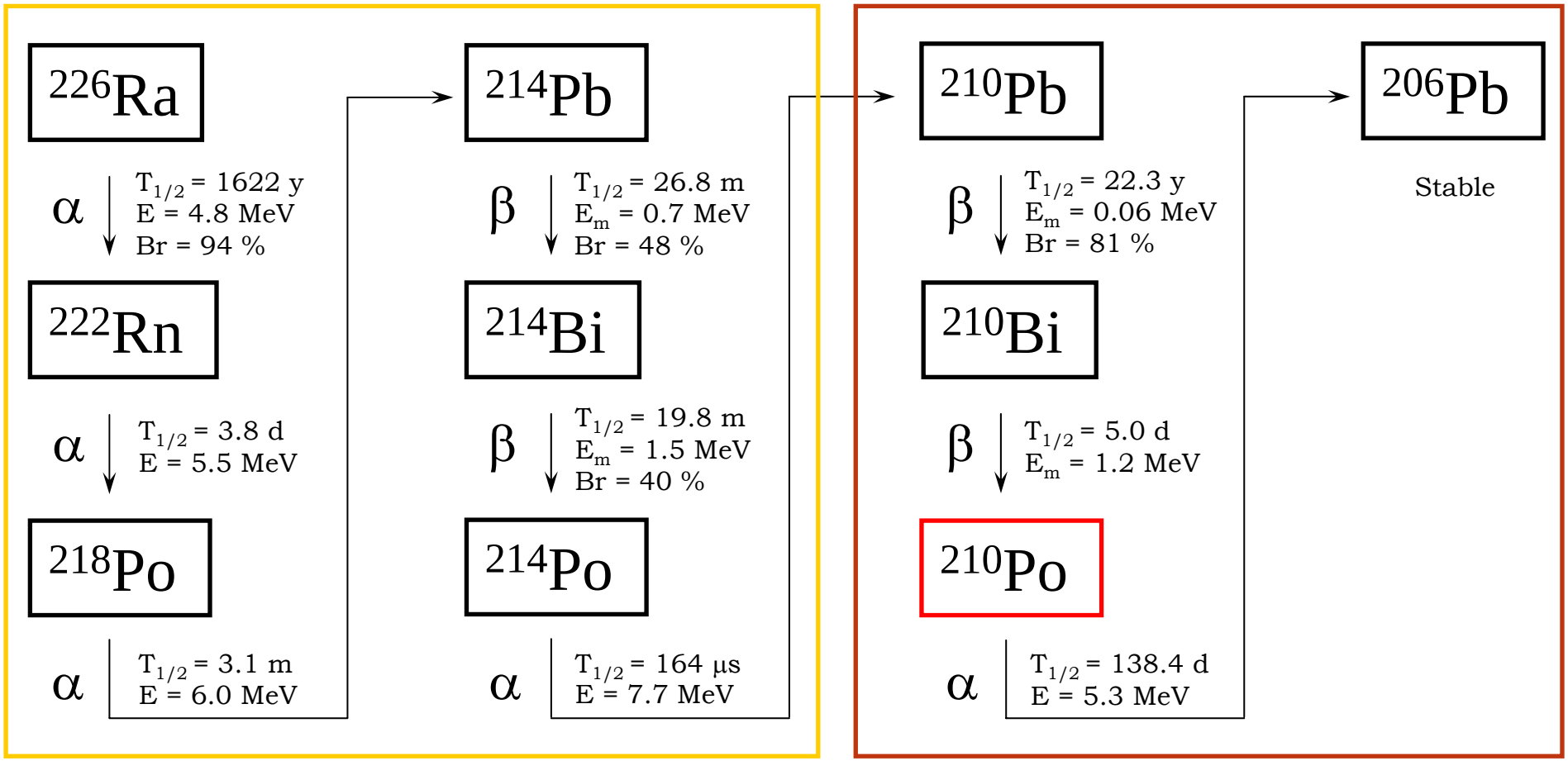
- Comparing etching with electropolishing
- Conclusions

Why to investigate ^{222}Rn daughters?

- Equilibrium in the chain broken at the ^{210}Pb level
- ^{210}Pb may stay as a main residual contamination of the copper after cleaning (will appear after some years)
- ^{210}Po radio-chemistry not well understood
- Long-lived ^{222}Rn/^{210}Pb daughters implanted/deposited on the copper surface may contribute to the background in GERDA



Why to investigate ^{222}Rn daughters?



Technique

- Screening of ^{210}Po with an alpha spectrometer
  50 mm Si-detector, bcg ~ 5 α/d (1-10 MeV)
  sensitivity ~ 20 mBq/m^2 (50 mBq/kg, ^{210}Po - Ag)
- Screening of ^{210}Bi with a beta spectrometer
  2×50 mm Si(Li)-detectors, bcg ~ 0.14/0.26 cpm
  sensitivity ~ 6 Bq/kg (^{210}Bi – 0.012/0.023 cpm/(Bq/kg))
- Screening of ^{210}Pb (46.6 keV line) with a gamma spectrometer
  25 % - n-type HPGe detector with an active and a passive shield, sensitivity ~ 20 Bq/kg
- Only small samples can be handled – artificial contamination needed: copper discs loaded with ^{222}Rn daughters

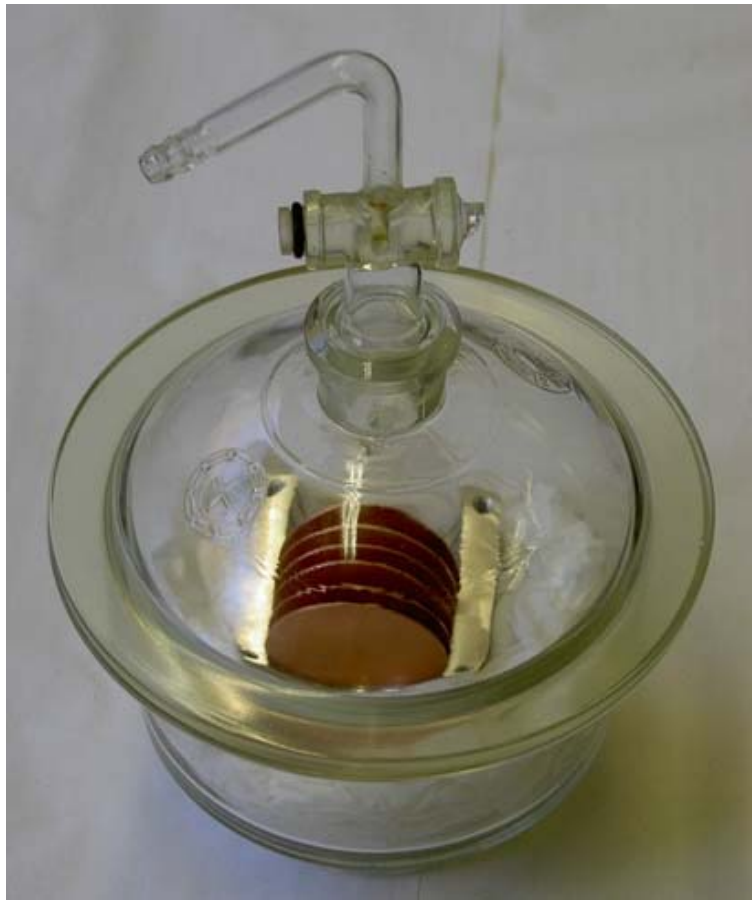
Technique

- LENS electrolytic copper used to fabricate sample discs (50 mm diameter, 1 mm thickness)
- Discs cleaned applying “Majorana procedure” (5 min in 1% H_2SO_4 + 3% H_2O_2; 5 min in 1% citric acid; rinsing with distilled water)
- Discs placed for 4 months in a strong ^{222}Rn source (1.4 MBq)

Technique

Discs before and after cleaning

Discs loaded with ^{222}Rn daughters



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Etching, disc No. 2

- „Majorana procedure“ tested:

  copper discs 5 min in 1% H_2SO_4 + 3% H_2O_2
  5 min in 1% citric acid
  rinsing with water

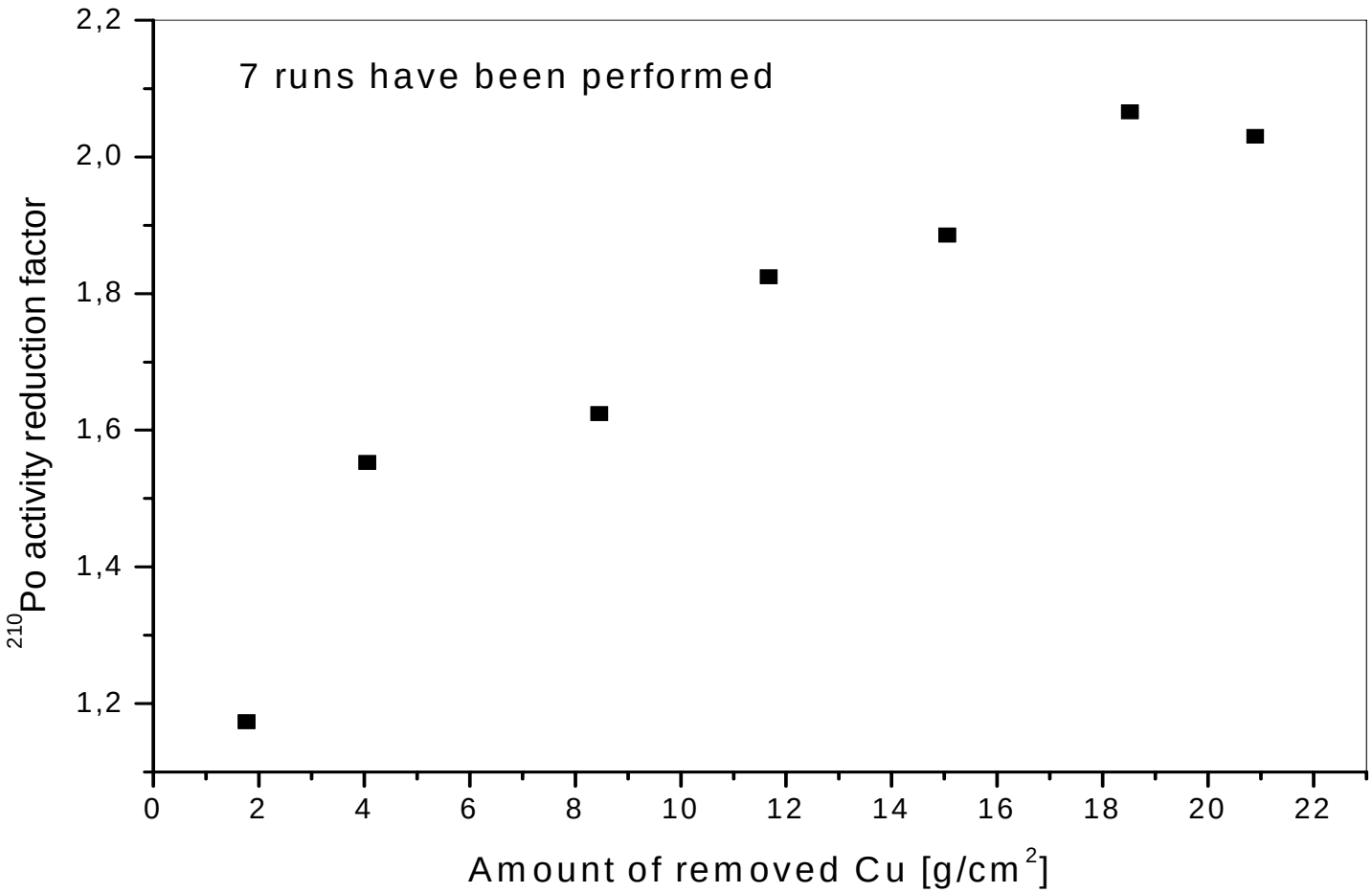
- Solution volume: each time 250 cm^3
- Temperature: 20 °C
- ^{210}Po measured on both disc sides
- Blank signal: (0.0042 ± 0.0005) cpm
- Disc loaded with ^{210}Po: side a: (2.97 ± 0.03) cpm
  side b: (2.64 ± 0.03) cpm

Etching, disc No. 2

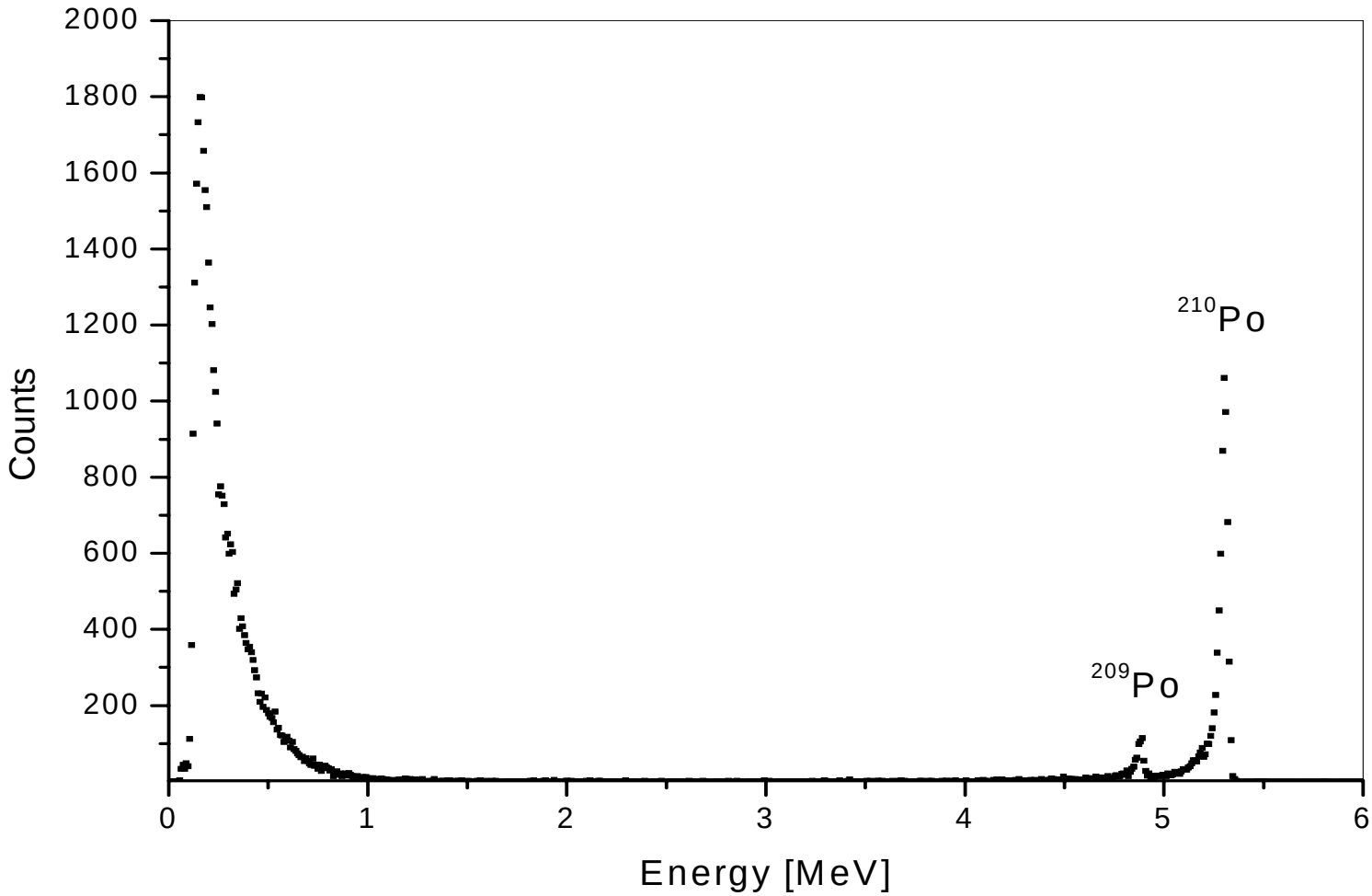
Loaded disc, side a: (2.97 ± 0.03) cpm
side b: (2.64 ± 0.03) cpm

Run No.	Disc side	^{210}Po activity [cpm]	^{210}Po reduction factor R	Amount of removed Cu	Remarks
1	a	2.36 ± 0.03	1.3	(1.77 ± 0.02) mg/cm^2 1.98 µm	Acid mixed during etching
	b	2.16 ± 0.02	1.2		
2	a	1.83 ± 0.04	1.3	(2.29 ± 0.02) mg/cm^2 2.56 µm	Acid mixed during etching
	b	1.79 ± 0.03	1.2		
3	a	1.84 ± 0.03	0.99	(4.40 ± 0.02) mg/cm^2 4.91 µm	Acid mixed during etching
	b	1.62 ± 0.03	1.1		
4	a	1.65 ± 0.03	1.1	(3.21 ± 0.02) mg/cm^2 3.58 µm	Acid mixed during etching
	b	1.43 ± 0.02	1.1		
5	a	1.62 ± 0.03	1.0	(3.38 ± 0.02) mg/cm^2 3.77 µm	Acid mixed during etching
	b	1.35 ± 0.02	1.1		
6	a	1.47 ± 0.02	1.1	(3.47 ± 0.02) mg/cm^2 3.87 µm	Acid mixed during etching
	b	1.25 ± 0.03	1.1		
7	a	1.50 ± 0.02	0.98	(2.37 ± 0.02) mg/cm^2 2.64 µm	Acid mixed during etching ^{209}Po added (1.42 Bq)
	b	1.26 ± 0.03	0.99		

Etching, disc No. 2



Etching (with ^{209}Po), disc No. 2



Etching, disc No. 1

Results for ^{210}Pb, ^{210}Bi and ^{210}Po:

Isotope	Original activity [cpm]	Activity after cleaning [cpm]	Reduction factor R	Amount of removed Cu	Remarks
^{210}Pb	1.49 ± 0.04	< 0.022	> 68	3.91 mg/cm^2 4.4 µm	Only side a was investigated
^{210}Bi	31.17 ± 0.71	0.77 ± 0.02	40.5		Only side a was investigated
^{210}Po	2.55 ± 0.01	2.06 ± 0.01	1.2		Only side a was investigated

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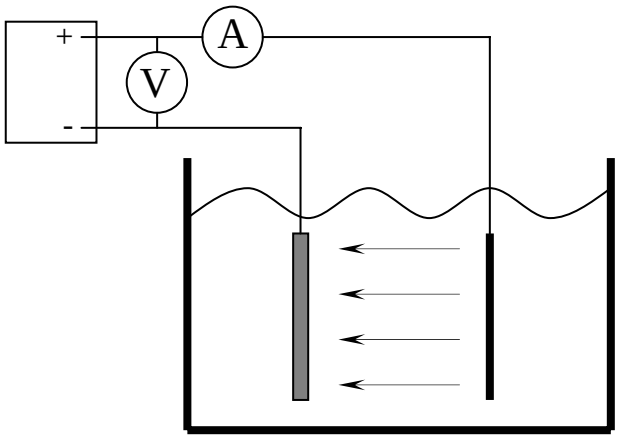
Electropolishing, disc No. 4

- Both disc sides investigated separately for ^{210}Po
- Electrolyte: 85 % H_3PO_4 + 5 % 1-butanol ($C_4H_{10}O$)
- Only one cathode (Cu disc)
- Several runs performed, each time using a new cathode and fresh solution



U = 1.8 V

I = 150 – 10 mA

Electropolishing, disc No. 4

Loaded disc side a: (9.52 ± 0.06) cpm
side b: (1.78 ± 0.04) cpm

Results:

Run No.	Disc side	^{210}Po activity [cpm]	^{210}Po reduction factor R	Amount of removed Cu*	Remarks
1	a	0.50 ± 0.03	19	17 mg/cm^2	Polished for 35 min Total charge: 70 mAh
	b	1.38 ± 0.03	1.3		
2	a	0.062 ± 0.003	8		Polished for 35 min Total charge: 70 mAh
	b	0.74 ± 0.01	1.9		
3	a	0.024 ± 0.002	2.6		Polished for 35 min Total charge: 70 mAh
	b	0.017 ± 0.002	44		

- disc side facing the cathode

*) measured after all runs

After all	a	0.024 ± 0.002	397	≤ 12.6 μm	Polished for 70 min
	b	0.017 ± 0.002	105	≤ 6.3 μm	Polished for 35 min

Electropolishing, disc No. 3

- Both sides investigated separately for ^{210}Po
- Electrolyte: 85 % H_3PO_4 + 5 % 1-butanol ($C_4H_{10}O$)
- Only one cathode (Cu disc)
- One run performed, disc was turned around several times
- Total polishing time: 3 h

Results:

Disc side	Original ^{210}Po activity [cpm]	^{210}Po activity after pol. [cpm]	^{210}Po reduction factor R	Amount of removed Cu	Remarks
a	2.18 ± 0.02	0.011 ± 0.001	198	20 mg/cm^2 22.3 µm	Facing the cathode 3 times, each time for 30 min
b	2.45 ± 0.03	0.014 ± 0.001	175		Facing the cathode 3 times, each time for 30 min

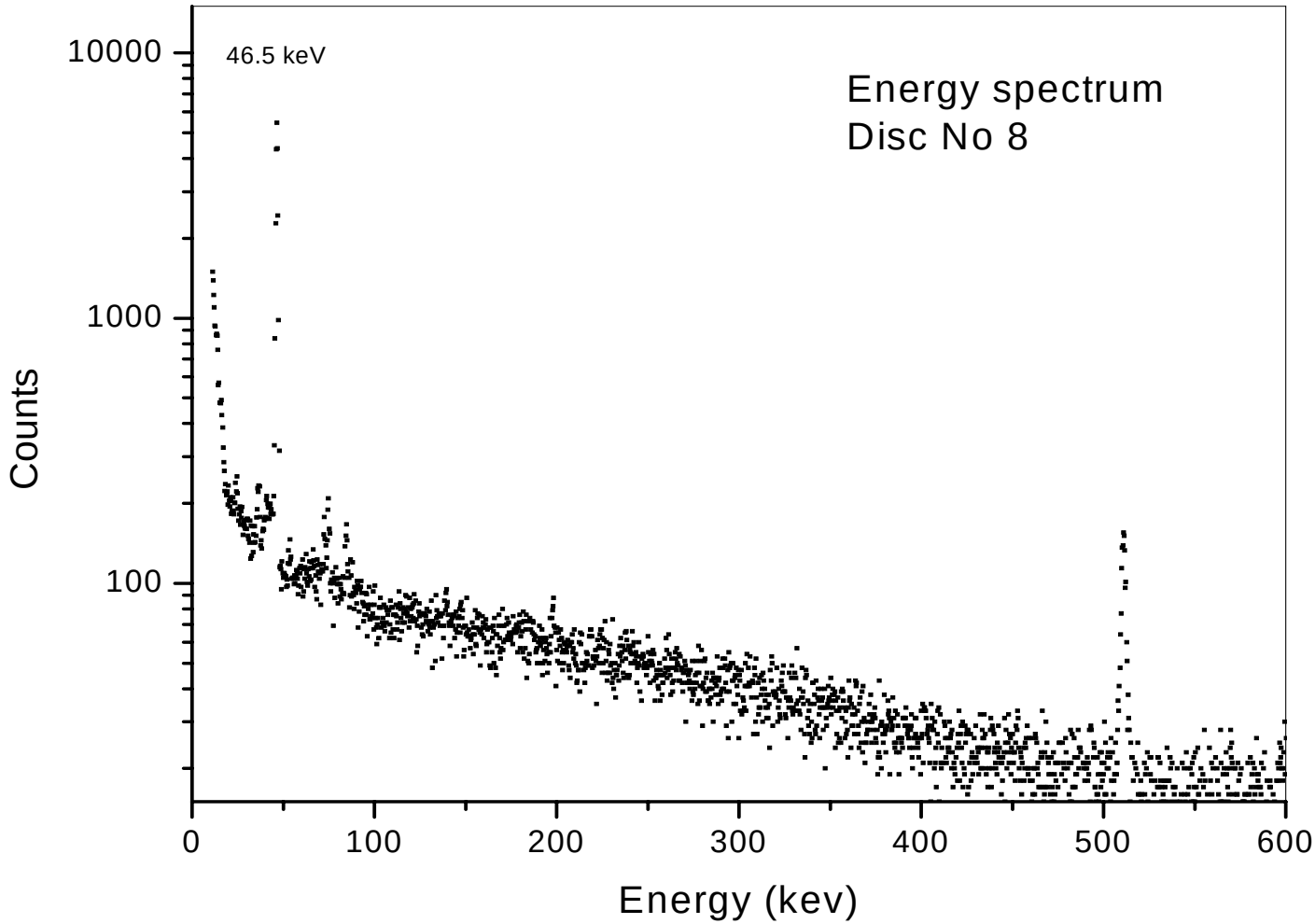
Electropolishing, disc No. 8

- Both sides investigated separately for ^{210}Po, ^{210}Bi and ^{210}Pb
- Electrolyte: 85 % H_3PO_4 + 5 % 1-butanol ($C_4H_{10}O$)
- Only one cathode (Cu disc)
- One run performed, disc was turned around every 5 min.
- Total polishing time: 1 h

Results:

Disc side	^{210}Po [cpm] before/after	^{210}Bi [cpm] before/after	^{210}Pb [cpm] before/after	Amount of removed Cu	Remarks
a				4.5 mg/cm^2 5.2 µm	Facing the cathode 6 times, each time for 5 min
b	5.31 ± 0.12 0.18 ± 0.01	36.55 ± 0.47 0.15 ± 0.01	2.08 ± 0.02 0.002 ± 0.002		Facing the cathode 6 times, each time for 5 min
	R = 30	R = 244	R = 1040		

^{210}Pb energy spectrum, disc No. 8



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Comparing etching with electropolishing

- Amount of removed material:
  - after 7 “Majorana” runs (30 min): 20.9 mg/cm^2
  - after one polishing run (35 min): 5.7 mg/cm^2

- Amount of removed ^{210}Po activity:
  - after 7 “Majorana” runs (35 min, 20.9 mg/cm^2): R_{av} = 2
  - after polishing (1 h, 4.5 mg/cm^2): R_{av} = 30
  - after long-polishing run (3 h, 20 mg/cm^2): R_{av} = 187

- Amount of removed ^{210}Pb and ^{210}Bi activity:
  - one “Majorana” run (5 min, 3 mg/cm^2): R_{Bi} = 40, R_{Pb}> 68
  - electropolishing (1 h, 4.5 mg/cm^2): R_{Bi} = 240, R_{Pb}= 1000

Conclusions

- Etching and electropolishing remove up to 20 mg/cm^2 Cu (depending on the exposure time)
- ^{210}Po deposited on- or close to the copper discs surface (relatively narrow α-peaks)
- Etching does not remove ^{210}Po from the copper – re-deposition (see test with ^{209}Po)
- Long electropolishing reduces ^{210}Po activity by a factor of ~200 – much more effective than etching
- Etching removes most of ^{210}Pb and ^{210}Bi (> 98 %)
- Electropolishing removes ^{210}Pb and ^{210}Bi more effective than etching (99.5 % ^{210}Bi and > 99.9 % ^{210}Pb removed)