

ThermoFisher
S C I E N T I F I C

The world leader in serving science

Analysis of Advanced Materials with the ELEMENT GD

Joachim Hinrichs

Application Specialist GD-MS/ICP-MS

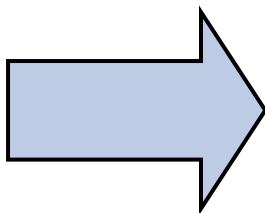
Thermo Scientific Bremen, Germany

joachim.hinrichs@thermofisher.com

Outline

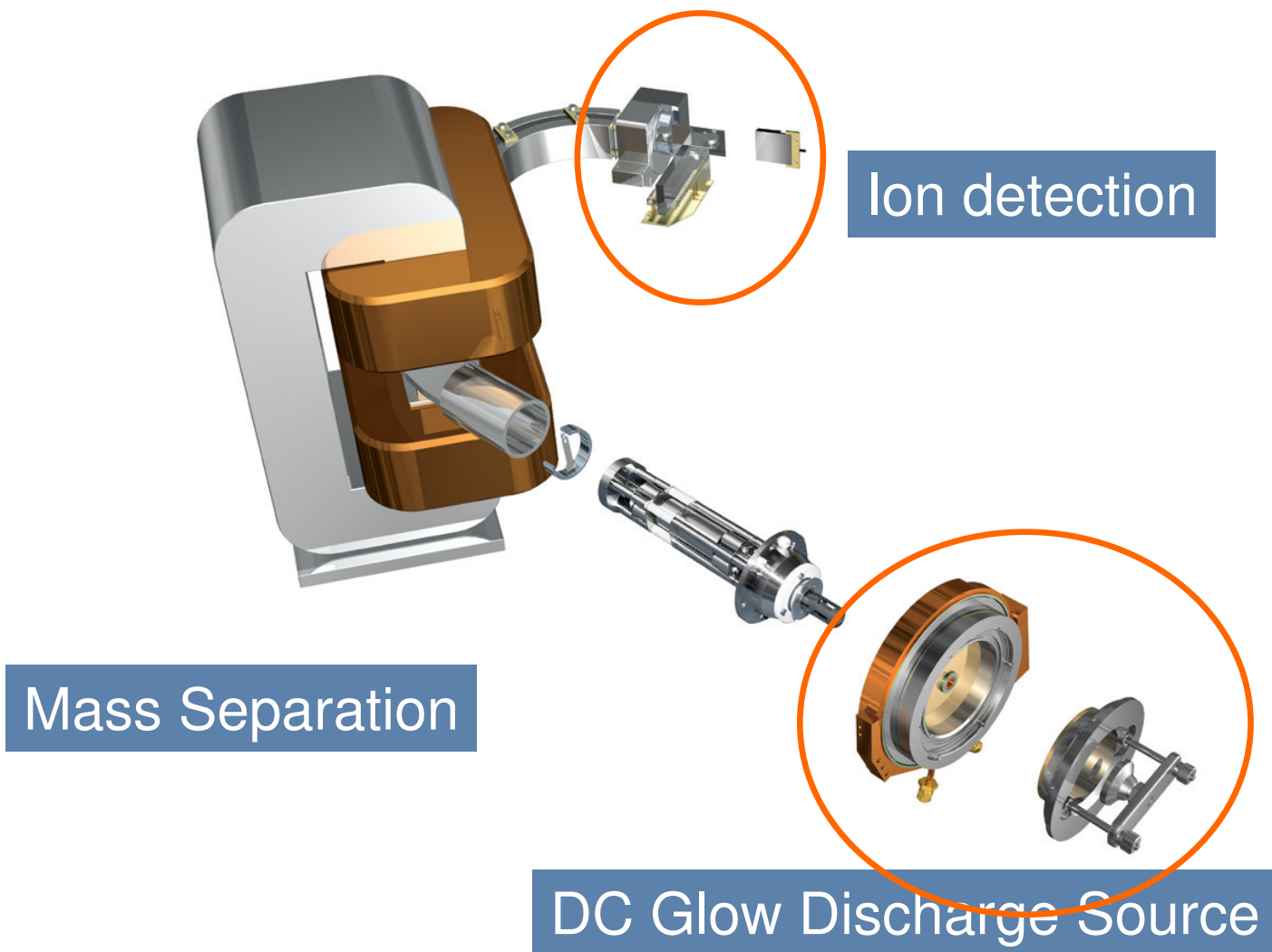
- What does an ELEMENT GD do
- Sales overview
- Application examples:
 - High purity metals
 - Solar cell silicon
 - Nickel super alloys
- ELEMENT GD News

What does the ELEMENT GD do?



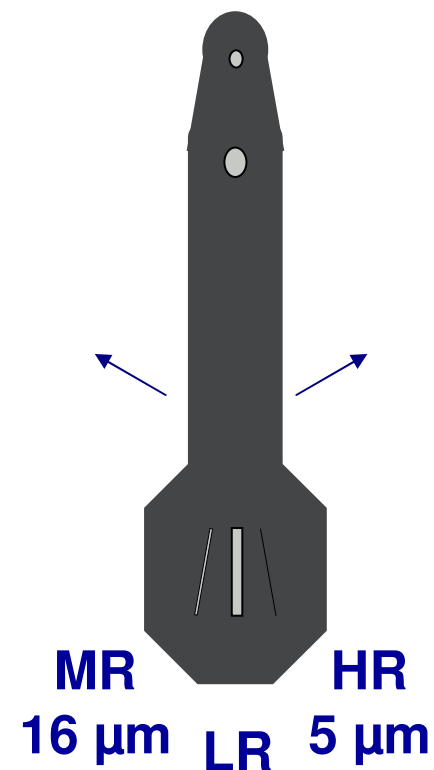
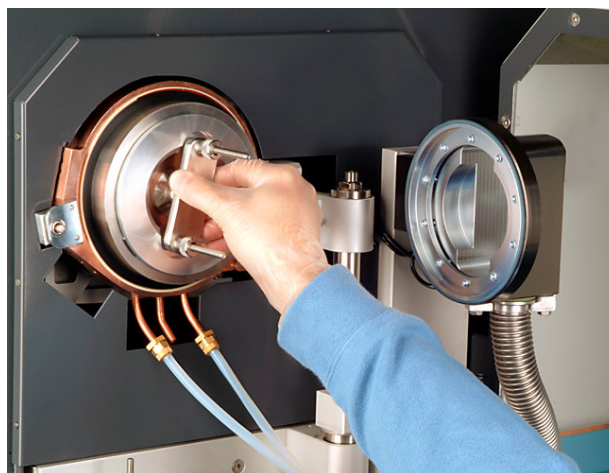
[ppb]				Sample #1	Sample #2
Isotope	Mass	Resolution	Calibration	Conc. [ppb]	Conc. [ppb]
Na23(LR)	23	LR	STD RSF	1.1	1.2
Mg24(MR)	24	MR	Cal	0.50	0.57
Al27(MR)	27	MR	Cal	0.67	7.79
Si28(MR)	28	MR	Cal	32	14
P31(MR)	31	MR	Cal	1.7	2.3
Cl35(MR)	35	MR	STD RSF	408	485
K39(HR)	39	HR	STD RSF	1.8	1.2
Ca44(MR)	44	MR	STD RSF	2.4	4.2
Sc45(MR)	45	MR	STD RSF	0.07	0.01
Ti48(MR)	48	MR	Cal	4.7	1.7
V51(MR)	51	MR	STD RSF	0.04	0.10
Cr52(MR)	52	MR	Cal	0.41	0.37
Mn55(MR)	55	MR	Cal	0.06	0.08
Fe56(MR)	56	MR	Cal	0.36	0.77
Ni58(MR)	58	MR	Cal	0.40	0.58
Co59(MR)	59	MR	Cal	0.10	0.07
Zn68(MR)	68	MR	Cal	3.8	5.2
Ga69(MR)	69	MR	STD RSF	0.43	0.40
Ge74(HR)	74	HR	STD RSF	3.4	3.5
As75(MR)	75	MR	Cal	1.9	0.9
Se77(MR)	77	MR	Cal	15	20
Br79(MR)	79	MR	STD RSF=1	2.8	2.4
Rb85(MR)	85	MR	STD RSF=1	0.06	0.07
Si88(LR)	88	LR	STD RSF	0.07	0.10
Y89(MR)	89	MR	STD RSF	0.06	0.06
Zr90(MR)	90	MR	Cal	0.18	0.20
Nb93(MR)	93	MR	STD RSF	0.02	0.07
Mo97(MR)	97	MR	STD RSF	0.71	0.64
Ru102(HR)	102	HR	STD RSF	0.52	0.81
Rh103(HR)	103	HR	STD RSF	9.4	7.4

ELEMENT GD

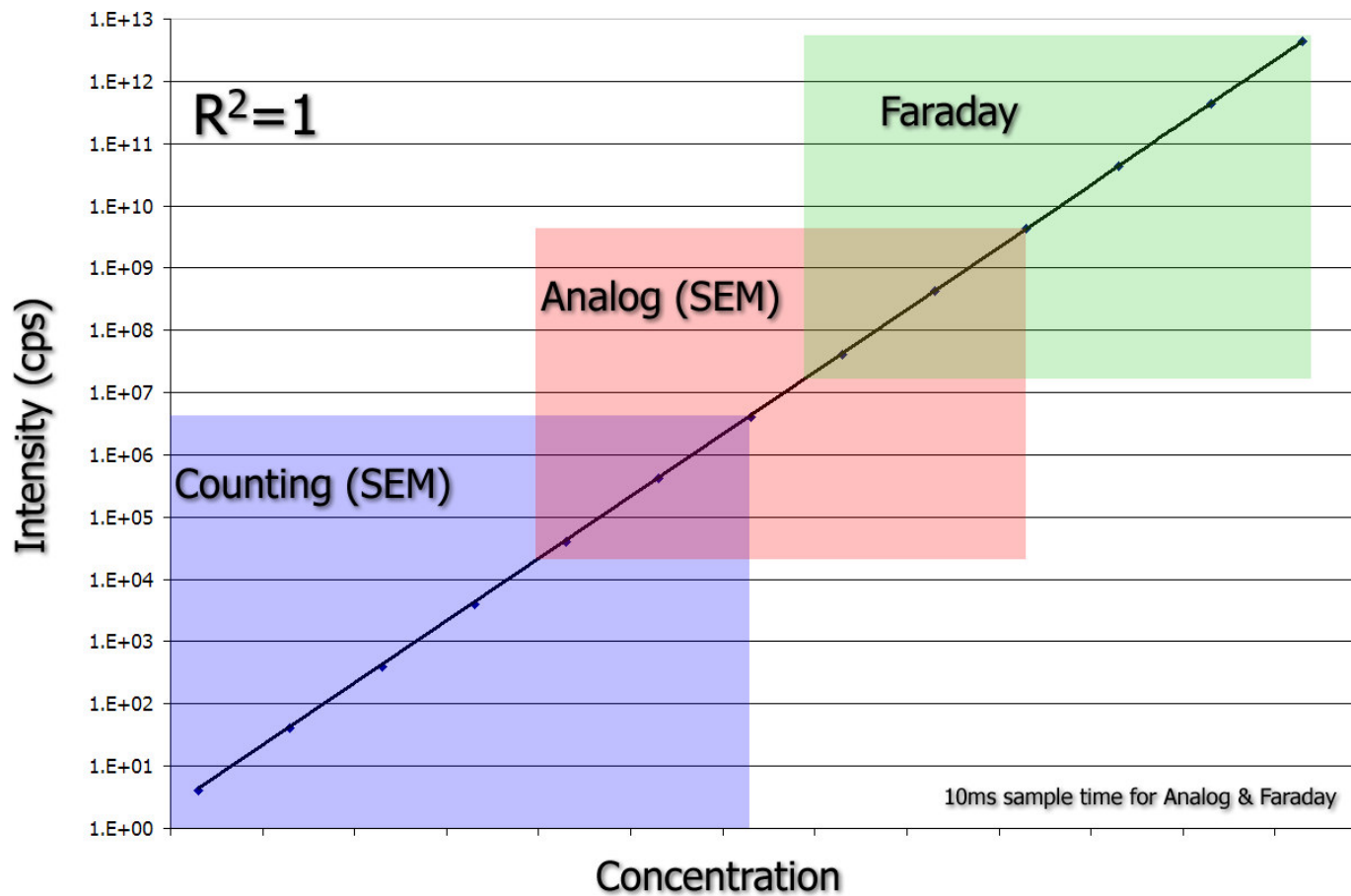


Key to fast GD-MS analysis

- Fast flow Grimm type source
- Easy sample change over
- Fast scanning high resolution mass spectrometer
- High mass stability
- Resolution switching
- Fast detection system
- Peltier cooling

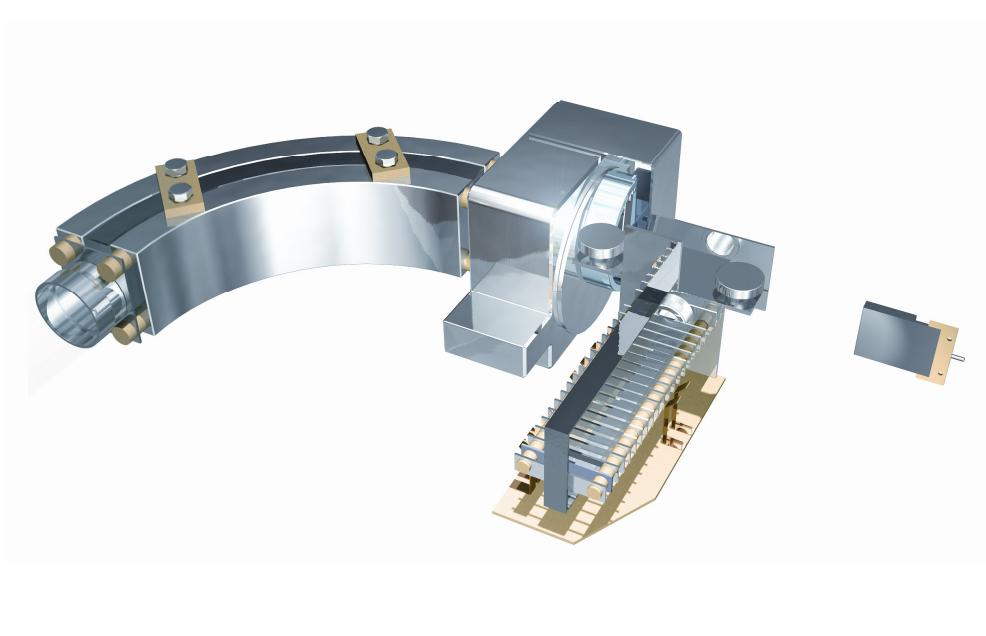


Dynamic Range ELEMENT GD



Why 12 orders of magnitude detection system ?

- GD-MS measures the total ion beam
- Matrix signal is used for normalization
- Semiquantitative results without any standards



How to calibrate ?

- **Basic:**

Measurement of analyte ions and simultaneous measurement of the matrix element. Calculation of the ion beam ratios (IBRs)

- **Semiquantitative:**

Standard RSF

- **Fully quantitative:**

a) calibrate with well characterized CRM's

b) BAM, Germany: Preparation of powder samples of pure metal matrix doped with graduated contents of trace elements down to the ppb-level ($\mu\text{g}/\text{kg}$).

Advantage: User definable calibration range, SI traceable

Application examples shown

- Copper
 - 5N to 6N quality
 - production control
 - high purity = high price
 - ppm/ppb levels of e.g. transition metals

- Silicon
 - solar cells
 - production control
 - blending control
 - ppb or below for PSE

- Nickel super alloys
 - Bi at 1 ppb level
 - Se at 10 ppb level
 - tough regulations (aerospace industry)
 - majors (% level) also useful

Others...

- sputter targets
 - 5N to 6N quality
 - production control
 - electronics industry
 - ppb levels PSE for wide range of matrices

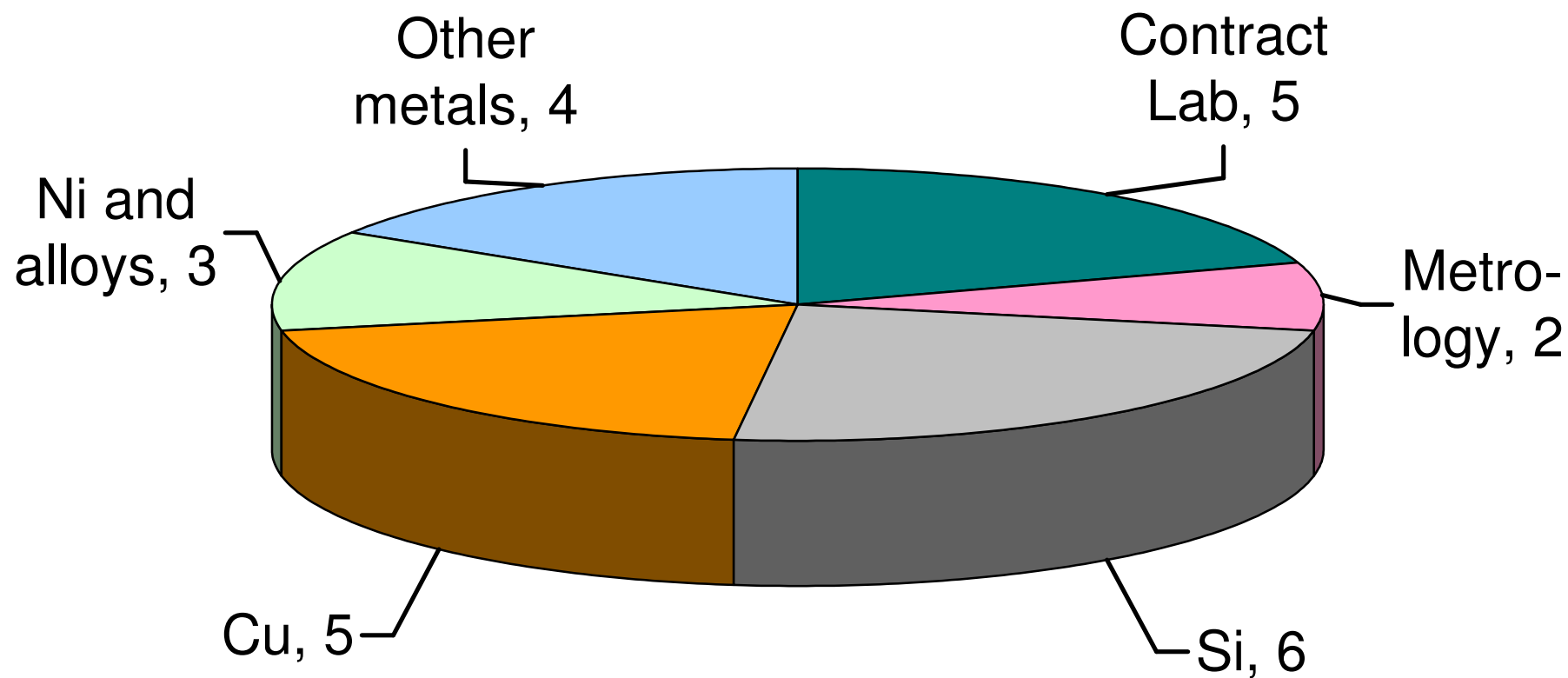
- non-conductors
 - mixing with graphite
 - mixing with 5N or better 6N metal

- Other/Special metals
 - As, Ge
 - Fe, Al
 - graphite, SiC

- Thin films
 - tested on hard drives
 - metal on metal systems
 - depth resolution about 10nm/s – 10 elements, ppm level

ELEMENT GD sales by application

ELEMENT GD by application



Application examples - 1

- Application examples:

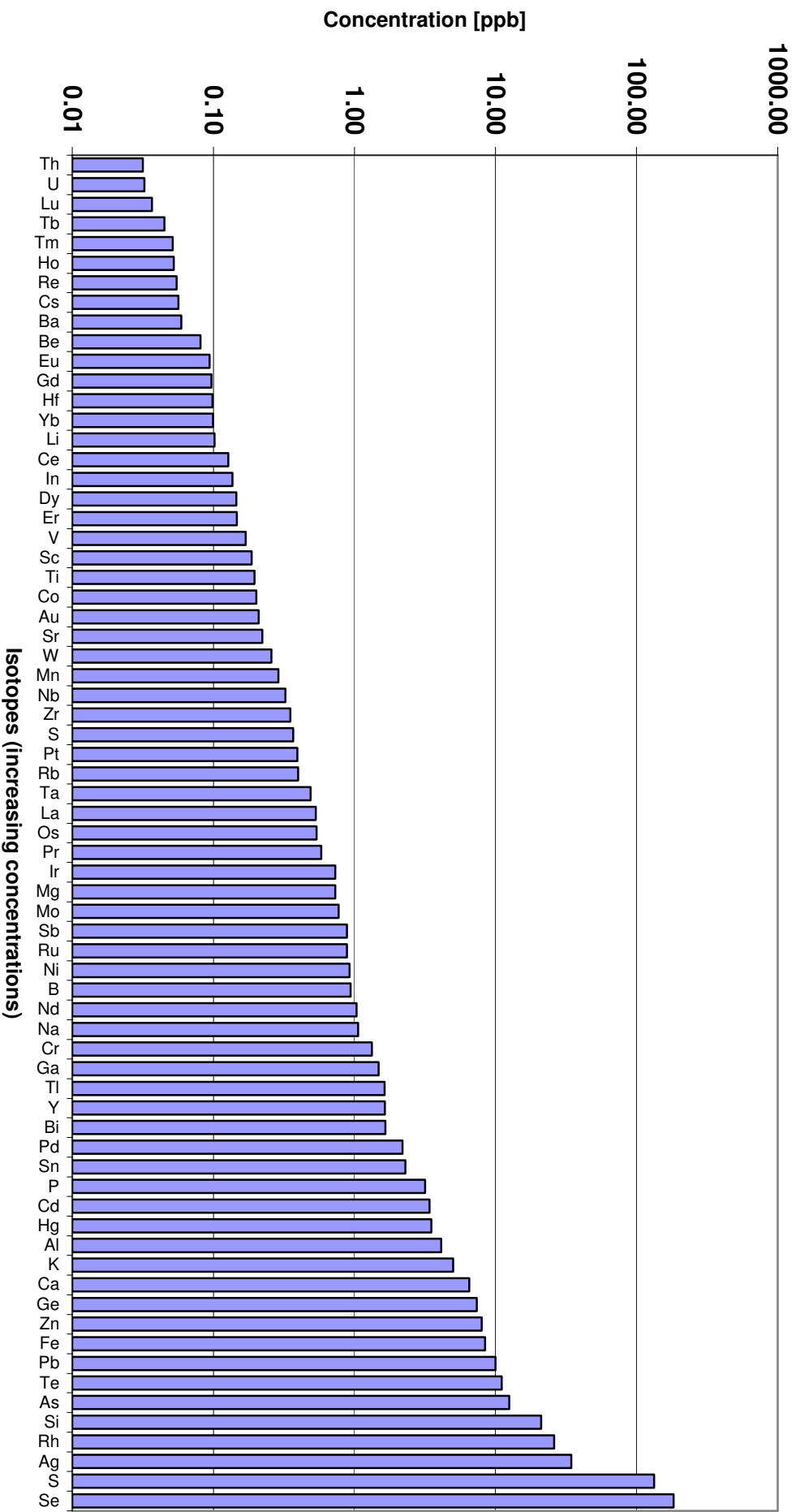
High purity metals

> 5N purity

routine determination of sub-ppm concentrations

Example: Copper

High purity copper (6N) analysis



Application examples - 2

- Application examples:

Solar cell silicon

Routine determination of sub-ppm and sub-ppb concentrations

Example: Repeat analysis of undoped Si sample

Drawback: lack of reference materials

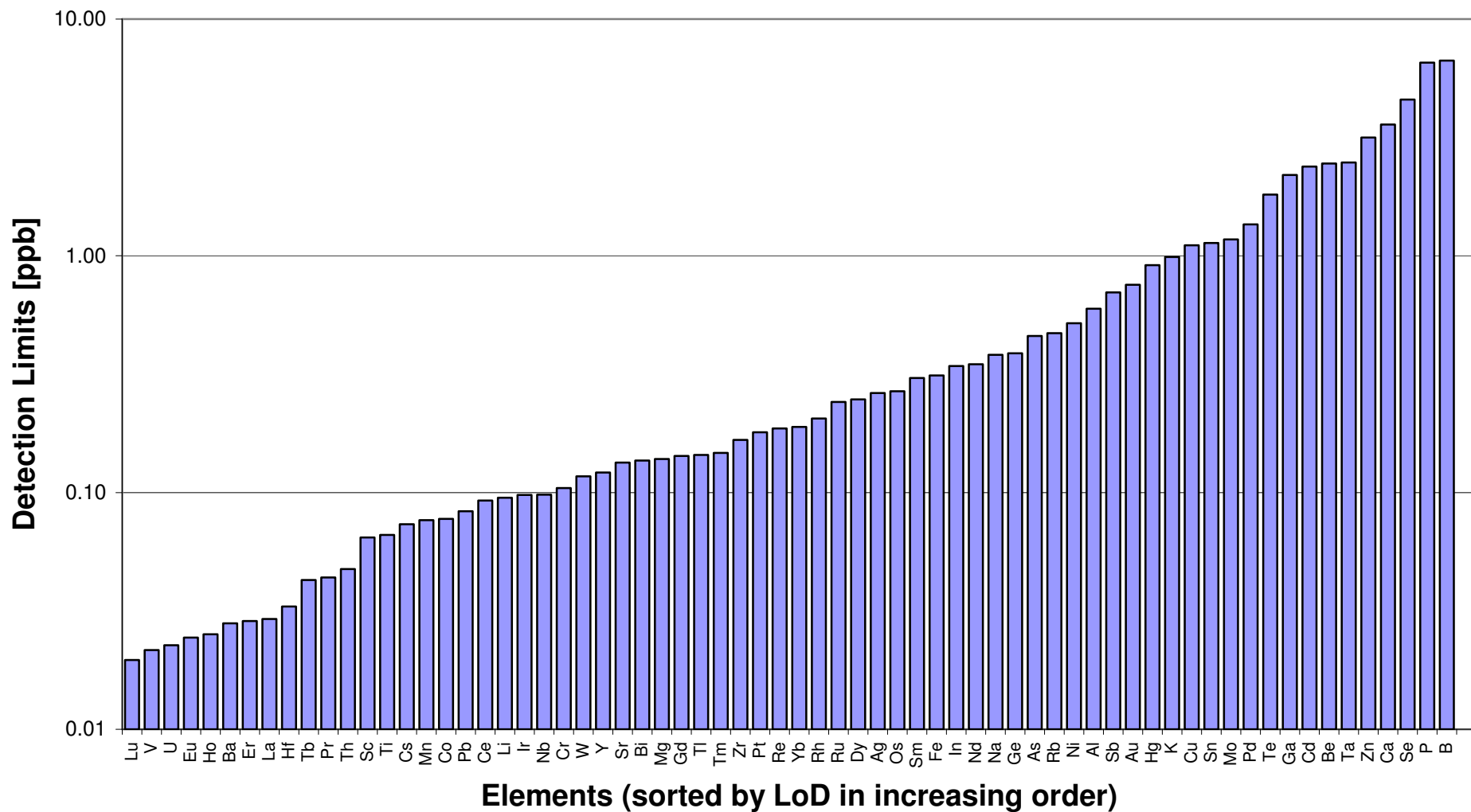
GD-MS Analysis of Solar Cell Silicon

Element	Mass	Resolution	Average concentration [ppb] n = 5 spots	LoD (3s) [ppb] n = 5 spots	Element	Mass	Resolution	Average concentration [ppb] n = 5 spots	LoD (3s) [ppb] n = 5 spots
Li	7	LR	< LoD	0.10	In	115	LR	2.0	0.3
Be	9	MR	< LoD	2.5	Sn	117	LR	1.2	1.1
B	11	MR	28.0	6.7	Sb	121	MR	1.3	0.7
Na	23	LR	0.6	0.4	Te	126	MR	1.9	1.8
Mg	24	MR	< LoD	0.14	Cs	133	LR	0.09	0.07
Al	27	MR	0.8	0.6	Ba	138	LR	< LoD	0.03
P	31	MR	16.0	6.5	La	139	LR	< LoD	0.03
K	39	HR	1.7	1.0	Ce	140	LR	0.10	0.09
Ca	44	MR	< LoD	3.6	Pr	141	LR	0.05	0.04
Sc	45	MR	< LoD	0.06	Nd	143	LR	< LoD	0.3
Ti	48	MR	< LoD	0.07	Sm	152	LR	0.4	0.3
V	51	MR	< LoD	0.02	Eu	153	LR	0.03	0.02
Cr	52	MR	< LoD	0.10	Gd	158	LR	< LoD	0.14
Mn	55	MR	< LoD	0.08	Tb	159	LR	< LoD	0.04
Fe	56	MR	0.4	0.3	Dy	164	LR	< LoD	0.25
Ni	58	MR	< LoD	0.5	Ho	165	LR	< LoD	0.03
Co	59	MR	< LoD	0.08	Er	166	LR	0.04	0.03
Cu	63	MR	1.8	1.1	Tm	169	MR	< LoD	0.15
Zn	64	MR	4.1	3.2	Yb	173	LR	< LoD	0.19
Ga	71	HR	< LoD	2.2	Lu	175	LR	< LoD	0.02
Ge	72	HR	1.6	0.4	Hf	178	LR	0.04	0.03
As	75	MR	0.5	0.5	Ta	181	LR	2.7	2.5
Se	77	MR	6.9	4.6	W	184	LR	< LoD	0.12
Rb	85	MR	< LoD	0.5	Re	187	LR	< LoD	0.19
Sr	88	MR	< LoD	0.13	Os	189	LR	< LoD	0.27
Y	89	MR	< LoD	0.12	Ir	193	LR	< LoD	0.10
Zr	90	MR	< LoD	0.17	Pt	195	LR	< LoD	0.18
Nb	93	MR	< LoD	0.10	Au	197	LR	< LoD	0.8
Mo	95	MR	< LoD	1.2	Hg	202	LR	< LoD	0.9
Ru	102	MR	< LoD	0.24	Tl	205	LR	< LoD	0.14
Rh	103	MR	< LoD	0.21	Pb	208	LR	0.09	0.08
Pd	105	MR	< LoD	1.4	Bi	209	LR	< LoD	0.14
Ag	107	MR	0.3	0.3	Th	232	LR	< LoD	0.048
Cd	111	MR	2.5	2.4	U	238	LR	< LoD	0.023

Total measurement time 10 min, plus 10 min Presputter time

GD-MS Analysis of Solar Cell Silicon

Detection limits (3s) in high purity Silicon sample (from 5 spots)



Application examples - 3

- Application examples:

Nickel super alloys

Routine determination of ppm and ppb concentrations in mixed matrix

Example: Repeat analysis of Ni alloy

Especially important: Se at double digit ppb level, Bi at single digit ppb level

GD-MS Analysis of Ni-Alloys

Nickel alloy	calib	unit	Average one spot, four	Stdev one spot, four	RSD one spot, four	RSD four spots
Majors:						
C	BAS 345	[%]	0.059	0.0002	0.3%	5.4%
Al	BAS 345	[%]	0.53	0.013	2.5%	1.9%
Si	Standard RSF	[%]	0.14	0.002	1.5%	4.3%
Ti	BAS 345	[%]	1.01	0.005	0.5%	4.7%
V	BAS 345	[%]	0.023	0.0001	0.2%	2.9%
Cr	BAS 345	[%]	18.4	0.07	0.4%	0.2%
Fe	Standard RSF	[%]	15.7	0.08	0.5%	1.2%
Co	BAS 345	[%]	0.11	0.0004	0.3%	0.7%
Nb	Standard RSF	[%]	1.91	0.02	1.1%	4.6%
Mo	BAS 345	[%]	3.30	0.01	0.3%	2.2%
Traces:						
B	BAS 345	[ppm]	35	0.5	1.4%	8.1%
P	Standard RSF	[ppm]	53	0.5	0.9%	4.8%
S	Standard RSF	[ppm]	5.0	0.3	6.7%	9.1%
Mn	Standard RSF	[ppm]	219	1	0.4%	1.3%
Se	BAS 346A	[ppm]	0.016	0.006	20%	16%
Zr	BAS 345	[ppm]	57	0.4	0.7%	4.6%
Ag	BAS 346A	[ppm]	0.032	0.003	9.2%	28%
Sn	BAS 345, BAS346A	[ppm]	7.1	0.1	0.8%	3.6%
Te	BAS 346A	[ppm]	0.011	0.001	4.9%	14%
W	Standard RSF	[ppm]	163	1	0.9%	3.6%
Pb	BAS 345, BAS346A	[ppm]	0.055	0.006	10%	13%
Bi	BAS 346A	[ppm]	0.002	0.0003	16%	26%

Bold = calibrated; normal font = semiquant (STD RSF)

Benefits

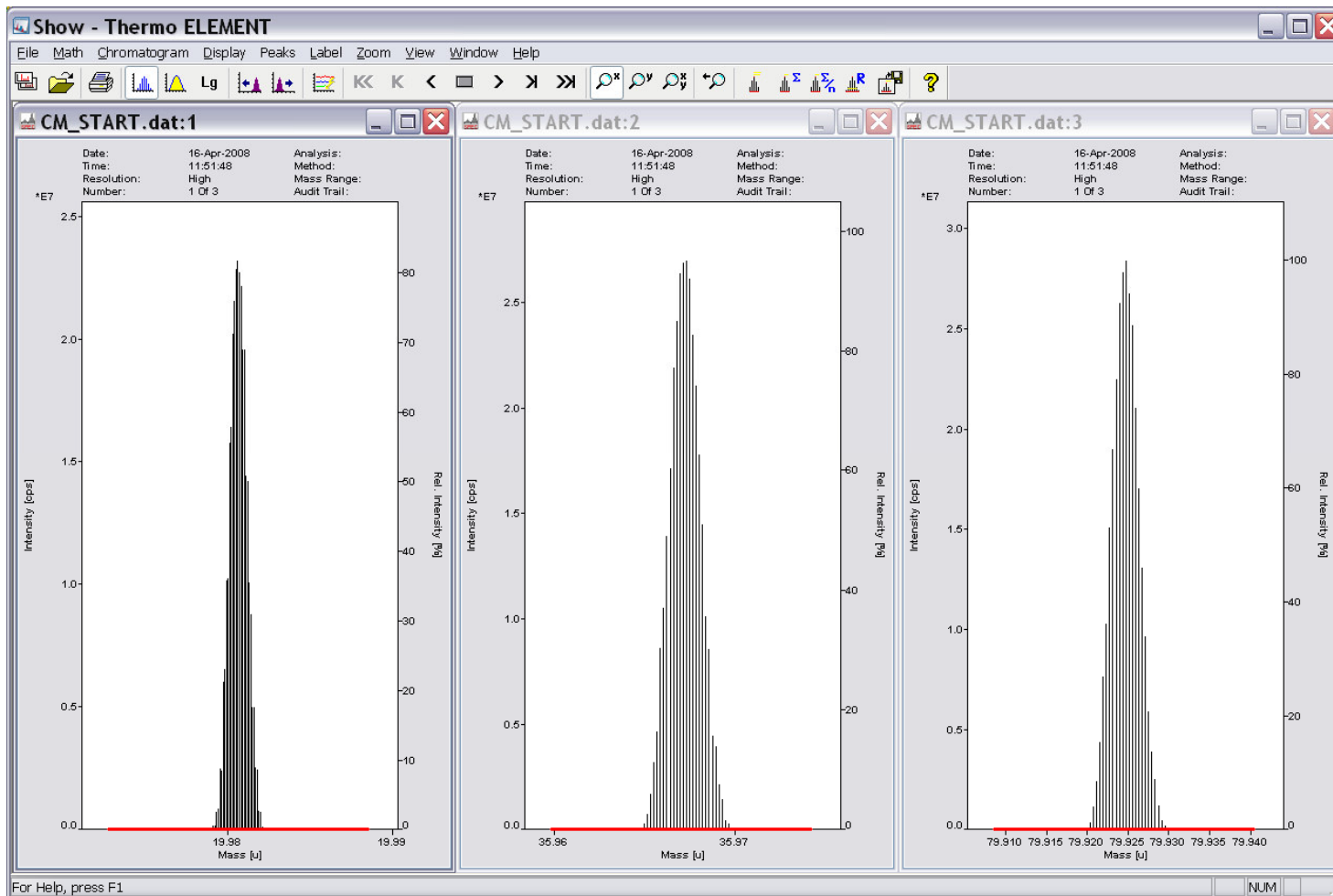
- <ppm range. But not necessarily only ppb level, also reliable problem solver for certain elements/tasks
- perform quality control for highly valuable materials at the ppb level within several minutes
- No need for digestive methods (good for PGE, Ta etc)
- Ultra-trace analysis without clean room – although clean bench for sample prep and storage is of advantage
- Full coverage of PSE
- Even semiquant data for unknown/uncalibrated matrices are usually accurate to $\pm 30\%$ (but there are exceptions)
- Easy calibration since MS is linear up to the % level
- Make the lab prepared for the future – not just meet the current specs, easily make (and analyse) more sophisticated products

ELEMENT GD news

- **Software update**
- **Hardware**
- **Others...**

ELEMENT GD news

- **Software update including....**
 - AutoLockMass at 3 points instead of only one



ELEMENT GD news

- **Software update including....**
 - Merge calibrated RSF with Standard RSF

Sample name :
Matrix :
Operator :

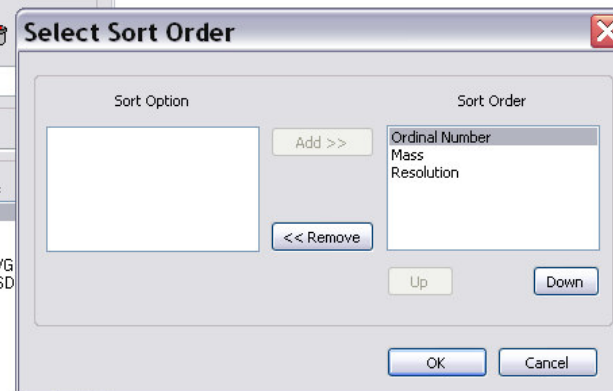
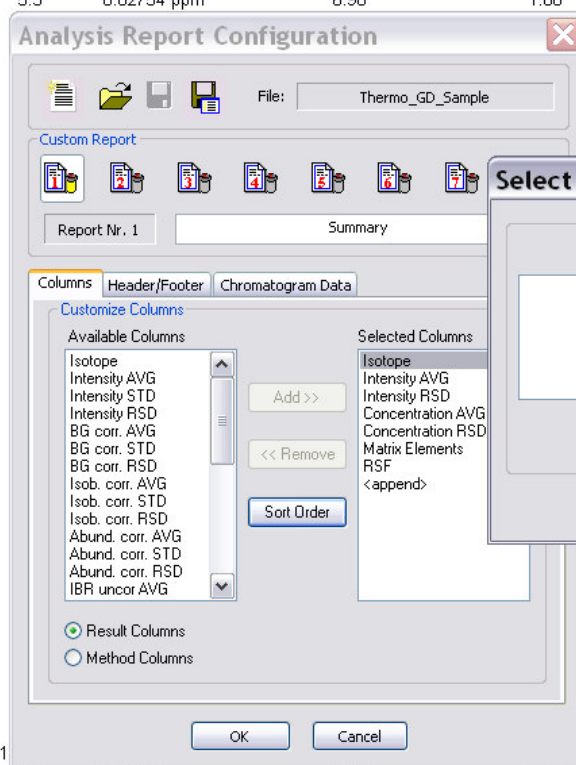
Isotope	Intensity AVG [cps]	Intensity RSD [%]	Concentration AVG	Concentration RSD [%]	Matrix Elements	Error
Be9(MR)	4.2	100.0	0.000 ppm	100.1		R
B11(MR)	160.9	73.2	0.019 ppm	73.2		R
C12(MR)	54771.0	39.8	7.464 ppm	39.7		R
Mg24(MR)	11.7	24.7	0.000 ppm	24.5		R
Al27(MR)	1.18E+007	0.2	269.079 ppm	0.3		C
Si28(MR)	3.48E+006	0.5	207.905 ppm	0.6		C
P31(MR)	56349.2	1.3	6.684 ppm	1.3		C
S32(MR)	113628.3	1.3	5.975 ppm	1.4		R
Ti48(MR)	1738.6	4.4	0.014 ppm	4.3		R
Cr52(MR)	4813.7	2.9	0.107 ppm	2.7		R
Mn55(MR)	1.04E+007	1.1	219.598 ppm	1.0		C
Fe56(MR)	1.71E+008	0.7	2062.704 ppm	0.5		C
Co59(MR)	1.45E+007	1.1	193.422 ppm	1.1		C
Ni60(MR)	1.24E+007	1.2	1048.260 ppm	1.3		C
Cu63(MR)	1.03E+010	0.5	581825.501 ppm	0.4		C
Cu63(HR)	3.43E+009	2.4	623713.148 ppm	1.1		C
Zn68(MR)	1.45E+009	0.6	381821.406 ppm	0.6		C
Zn68(HR)	4.98E+008	1.0	374410.967 ppm	1.8		C
As75(MR)	2.68E+006	0.9	228.814 ppm	0.8		C
As75(HR)	230.7	20.8	3.209 ppm	21.6		C
Se77(MR)	463.3	8.4	0.334 ppm	8.4		R
Se77(HR)	26.0	7.7	0.058 ppm	7.7		R
Zr90(MR)	73.3	32.2	0.001 ppm	32.4		R
Ag107(MR)	1.83E+006	1.0	200.133 ppm	1.2		C
Ag109(MR)	1.42E+006	1.0	168.098 ppm	0.9		C
Cd111(MR)	141275.5	3.9	86.910 ppm	3.6		C
Sn119(MR)	6.75E+006	0.7	2076.625 ppm	0.9		C
Sn119(HR)	1.88E+006	2.7	1872.630 ppm	4.1		C
Sb121(MR)	1.48E+006	0.6	120.388 ppm	0.8		C
Te128(MR)	275231.6	0.6	46.437 ppm	0.8		C
Te130(MR)	334391.3	0.9	52.896 ppm	1.1		C
Hg202(MR)	8.7	48.0	0.002 ppm	48.2		R
Pb208(MR)	9.86E+008	1.0	29521.916 ppm	1.0		C
Bi209(MR)	1.76E+006	0.3	69.348 ppm	0.2		C

Errorcodes: S=Amplifier Skipped D=Intensity Defocused O=Overflow C=RSF Calib. R=Std RSF Calib.

ELEMENT GD news

- **Software update including....**
 - User definable sorting option in RESULTS

Isotope	Intensity AVG [cps]	Intensity RSD [%]	Concentration AVG ppm	Concentration RSD [%]	Matrix Elements	RSF
B11(MR)	883.78	7.3	0.26949 ppm	5.73		6.49
F19(MR)	730.81	5.3	0.02754 ppm	6.90		1.00
Mg24(MR)	743.31					
Mg25(MR)	93.06					
Mg26(MR)	122.22					
Al27(MR)	186474.11					
Si28(MR)	9245.83					
Si29(MR)	448.61					
Si30(MR)	352.78					
Cl35(MR)	343494.89					
Ca44(MR)	1011.58					
Ti47(MR)	1904.28					
Ti48(MR)	20872.36					
Ti48(HR)	5800.58					
V51(MR)	33993.44					
V51(HR)	229.17					
Cr52(MR)	54319.56					
Cr52(HR)	15243.08					
Cr53(MR)	6198.33					
Mn55(MR)	6237.39					
Fe56(MR)	3.95E+006					
Fe57(MR)	103865.39					
Co59(MR)	3835.72					
Ni60(MR)	7344.36					
Ni61(MR)	283.33					
Cu63(MR)	711421.33					
Cu65(MR)	325299.67					
Zn66(MR)	50765.39					
Zn68(MR)	34868.58					
As75(MR)	640.44					
Se76(HR)	12.50					
Se77(MR)	982.11					
Se77(HR)	1.39					
Se78(HR)	8596.75					
Se82(MR)	8.33	173.2	0.01309 ppm	173.21		3.77
Zr90(MR)	174848.17	1.9	7.16651 ppm	0.29		0.56



ELEMENT GD news

- **Software update including....**
 - Sample details now individually in Reports

Analysis Details

Sample Name : Testsample
 Matrix : Ruthenium
 Operator : Joachim

Isotope	Intensity AVG [cps]	Intensity RSD [%]	Concentration AVG
B11(MR)	883.78	7.3	0.26949 pprr
F19(MR)	730.81	5.3	0.02754 pprr
Mg24(MR)	743.31	17.2	0.05342 pprr
Mg25(MR)	93.06	56.0	0.05263 pprr
Mg26(MR)	122.22	18.8	0.06309 pprr
Al27(MR)	186474.11	3.5	8.91648 pprr
Si28(MR)	9245.83	14.1	1.14609 pprr
Si29(MR)	448.61	15.8	1.09801 pprr
Si30(MR)	352.78	22.9	1.30057 pprr
Cl35(MR)	343494.89	4.6	17.08320 pprr
Ca44(MR)	1011.58	10.9	0.82114 pprr
Ti47(MR)	1904.28	8.5	0.40276 pprr
Ti48(MR)	20872.36	8.2	0.43636 pprr
Ti48(HR)	5800.58	8.9	0.46863 pprr
V51(MR)	33993.44	4.1	0.69281 pprr
V51(HR)	229.17	14.2	0.01803 pprr
Cr52(MR)	54319.56	11.2	3.12202 pprr
Cr52(HR)	15243.08	10.1	3.38581 pprr
Cr53(MR)	6198.33	8.7	3.14290 pprr
Mn55(MR)	6237.39	5.6	0.23716 pprr
Fe56(MR)	3.95E+006	2.5	162.33857 pprr
Fe57(MR)	103865.39	0.8	177.80495 pprr
Co59(MR)	3835.72	10.0	0.15012 pprr
Ni60(MR)	7344.36	5.6	1.59950 pprr
Ni61(MR)	283.33	17.0	1.42595 pprr
Cu63(MR)	711421.33	1.0	94.51251 pprr
Cu65(MR)	325299.67	1.4	96.95400 pprr
Zn66(MR)	50765.39	1.6	26.24622 pprr
Zn68(MR)	34868.58	1.8	26.75254 pprr
As75(MR)	640.44	11.2	0.12366 pprr
Se76(HR)	12.50	33.3	0.07593 pprr

Analysis Report Configuration

File: Thermo_GD_Sample

Custom Report: Report Nr. 1 Summary

Columns: Header/Footer Chromatogram Data

Header Definitions

- Printed ...
- Global Acquisition Parameters Analysis Details [Details...](#)
- Global Evaluation Parameters Tune Parameters

Footer Definitions

- Page a of b

ASCII Export

- Remove Unit(s) Separate Flags (<, *)
- Attach RSF Table

OK Cancel

Details

Analysis Details

- Sample Name Matrix Lot Number
- Order Number Laboratory Operator
- Comment 1 Comment 2

Global Details

- Instrument Vendor Name Instrument Model
- Software Version File Version

OK Cancel

ELEMENT GD news

- **Software update including....**
 - Merge calibrated RSF with Standard RSF
 - Fast presputter
 - Automatic switching off source after analysis for all samples

Report: Thermo_GD_Sample

PCL Script: lgdoff

Standard:

Evaluation Parameters

Quantification Type: Quant. (Merge RSF)

Sample Amount: g

Spike Amount: mg

Stochiometric Factor:

Additional

Pre-Sputter Time: 120 s

Fast Pre-Sputter Time: 60 s

Fast Pre-Sputter Tune Parameters: Ru_pellet_presputter

Discharge Voltage [V]

Discharge Current [mA]

Discharge Gas [mL/min]

Peltier Temp. [°C]

Source Vacuum [mbar]

Sample Vacuum [mbar]

Fore Vacuum [mbar]

High Vacuum [mbar]

Button

HV

Peltier Cool

GD HV

Slider

Peltier Temp. [°C]

ELEMENT GD news

- **Software update including....**
 - Concentration calculation in Chromatogram view (+export)
 - Easier handling of Faraday calibration
 - Bug fixing & minor changes (~30 items)

ELEMENT GD news - hardware

- Plug-in extraction lens – first deliveries started
- Plug-in cone (data were collected for Ni alloy)
- Matrix kits – define new from scratch. E.g. standard (disposable steel caps, tubes, cones + graphite extraction lens) and high purity (re-usable graphite caps, tubes, cones + extraction lens). Ta parts might now be available in sufficient purity, but at high cost. Proposal for necessity and price target welcome

ELEMENT GD news - misc

- SEMI Si method: procedure started
- ELEMENT GD mailing list: already installed and active – will email details to all users within the next days.

Operated by independent organisation: Rob Franks, University of Santa Cruz, CA, USA.

Rob is running the ELEMENT ICP-MS user-list since >10a.

Knowledge exchange amongst users.

PLEASE JOIN THE LIST