Nitrogen determination in boron carbide by IPAA and gas fusion analysis

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Motivation

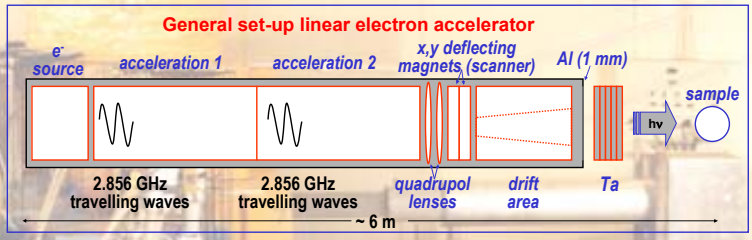
- Boron carbide widely used as industrial material
  - extreme hardness
  - neutron absorber
- round-robin exercise >>> certification of new reference material (ERM-ED102) [1]

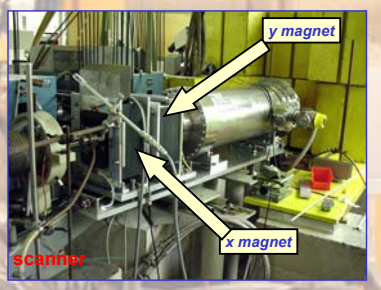
>>> nitrogen in boron carbide by
- inert gas fusion analysis (GFA)
- instrumental photon activation analysis (IPAA) using $^{14}N(\gamma,n)^{13}N$

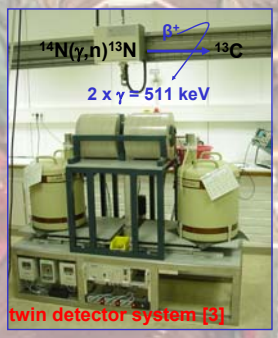
Instrumental photon activation analysis (IPAA)

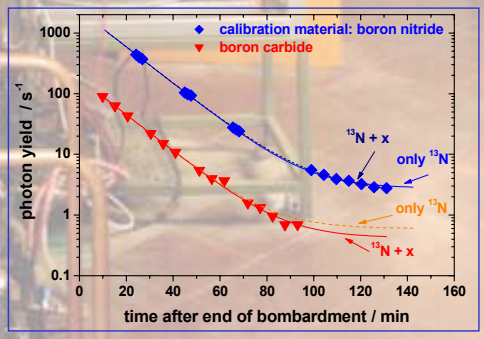
- linear electron accelerator
  - maximum e^- energy: 35 MeV, but here: **below C (matrix, PE) threshold!**
  - water-cooled Ta target >>> bremsstrahlung
- pneumatic rabbit system (Al rabbits)
- e^- beam position
  computer controlled magnet system (horizontal/vertical) [2]
  >>> very low lateral activating flux gradient
- sample preparation
  - ~1 g boron carbide powder in PE-LD container (8 mm Ø, 32 mm length), then wrapped in Al foil
  - ~60 mg of stoichiometric calibration material boron nitride (tubes of 2.7 mm Ø, 8 mm length; cti GmbH Idstein), wrapped in Al foil
- irradiation (20 min)
  - rabbit rotated in front of beam
  - beam itself wiggled 20 mm in y-direction
  - cooling: 3-5 min (Al & O) >>> unwrapped & put into glass tubes
- counting
  - sample in vertical/upright position between two HPGE detectors [3]
  - counting times 300 s (sample) 60 s (calibration material)
    >>> equal absolute counting events
- 4 sub-samples analysed on 4 different days, analyses repeated for 2 of these sub-samples >>> six data points
- container, filled with boron carbide during irradiation (to avoid recoil contamination from inside air) but emptied before counting, was also analysed >>> blank correction
- data evaluation: extra precautions, i.e. self-absorption & background (air!) correction, deconvolution of complex decay curves
- IPAA advantages vs. GFA
  - non-destructive >>> no risk of incomplete gas release
  - higher sample masses (1 g vs. 40 mg) >>> smaller risk of inhomogeneity

target	nuclear reaction	threshold energy [MeV]	$t_{1/2}$ [min]
C (matrix)	$^{12}C(\gamma,n)^{11}C$	18.74	20.38
N (analyt)	$^{14}N(\gamma,n)^{13}N$	10.56	9.96



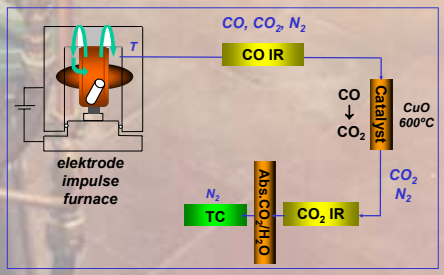






Inert gas fusion analysis (GFA)

- sample in graphite crucible, heated by impulse furnace
- sample fused under reductive conditions to release all gases
- He gas stream carries N_2 and other gases formed directly or after oxidation by heated CuO catalysts (e.g. CO_2, H_2O)
- scrubbers remove all interfering gases
- remaining N_2 detected by thermal conductivity detector
- traceable calibration by analysing stoichiometric KNO_3 under same conditions
- based on earlier work with SiC [4,5], adaptation of special measurement conditions to boron carbide (35-40 mg boron carbide, 0.6-0.9 mg KNO_3, Ni capsules, high temperature carbon crucibles, analysis power ~2600°C)
- 6 sub-samples analysed on 2 different days



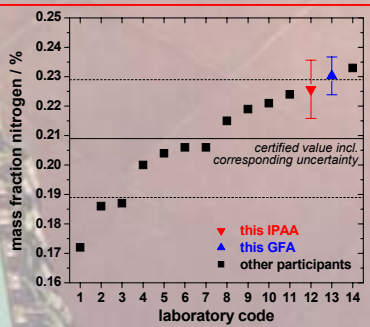
Results & Discussion [6]

- 2nd (GFA) & 3rd highest (IPAA) of rather untypical (non-S-shape) distribution of data of 14 round-robin participants
- our IPAA value is the only one not produced by GFA
  >>> the only one not influenced by a possible incomplete release of nitrogen from high-melting (2470°C) boron carbide, i.e. non-destructive
  >>> of utmost importance for the certification process

Individual GFA results
[mass fraction in %]
0.2294
0.2209
0.2386
0.2293
0.2272
0.2364
>>>
mean value:
0.2303 ± 0.0064 (1 s)

Individual IPAA results

mass / mg		nitrogen mass fraction / %				
boron carbide	boron nitride	no correction	with geometry correction only	with air correction only	with geometry & air correction	with geometry, air & blank correction
1177.84	74.8230	0.264	0.266	0.235	0.236	**0.227**
1101.83	73.3475	0.261	0.263	0.227	0.229	**0.220**
1120.86	64.1246	0.264	0.266	0.251	0.253	**0.244**
1093.81	65.5035	0.257	0.259	0.223	0.224	**0.215**
1177.84	74.8230	0.261	0.263	0.232	0.234	**0.225**
1101.83	73.3475	0.266	0.267	0.231	0.233	**0.223**
mean & standard deviation		0.265 ± 0.009	0.267 ± 0.009	0.233 ± 0.010	0.235 ± 0.010	**0.226 ± 0.010**



Take-home-messages

- It seems of utmost importance to find participants taking part into forthcoming round-robin exercise leading to the certification of a new reference material which are able to analyse light elements by other techniques than GFA: "old-fashioned" nuclear or wet chemistry ones.
- For the trueness of future reference materials and the quality of industrial products which may rely on these reference materials, scientific research should be continued in these techniques.

References

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