

Nitrogen determination in boron carbide by IPAA and gas fusion analysis

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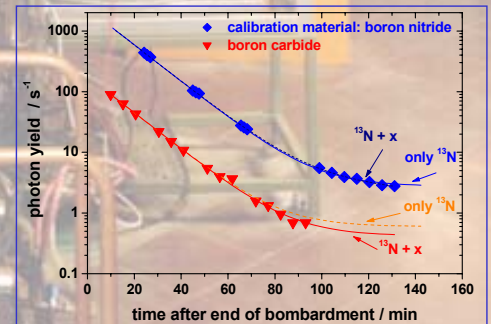
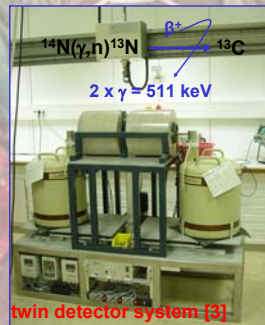
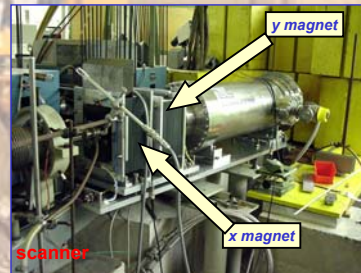
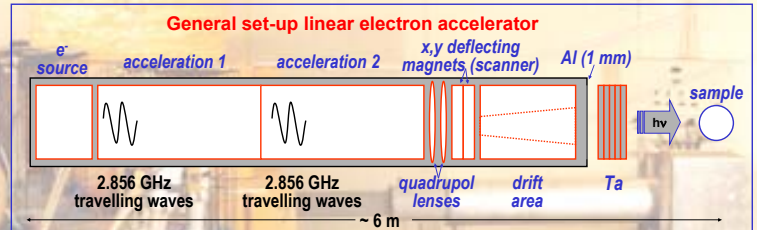
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Motivation

- 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 $^{14}\text{N}(\gamma, n)^{13}\text{N}$

Instrumental photon activation analysis (IPAA)

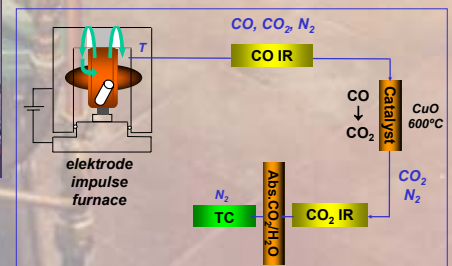
- linear electron accelerator
 - maximum e⁻ energy: 35 MeV, but here: below C (matrix, PE) threshold!
 - water-cooled Ta target >>> bremsstrahlung
- pneumatic rabbit system (Al rabbits)
- e⁻ beam position
 - computer controlled magnet system (horizontal/vertical) [2]
 - >>> very low lateral activating 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)	$^{12}\text{C}(\gamma, n)^{11}\text{C}$	18.74	20.38
N (analyt)	$^{14}\text{N}(\gamma, n)^{13}\text{N}$	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 N₂ and other gases formed directly or after oxidation by heated CuO catalysts (e.g. CO₂, H₂O)
- scrubbers remove all interfering gases
- remaining N₂ detected by thermal conductivity detector
- traceable calibration by analysing stoichiometric KNO₃ 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 KNO₃, Ni capsules, high temperature carbon crucibles, analysis power ~2600°C)
- 6 sub-samples analysed on 2 different days



Results & Discussion

- 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

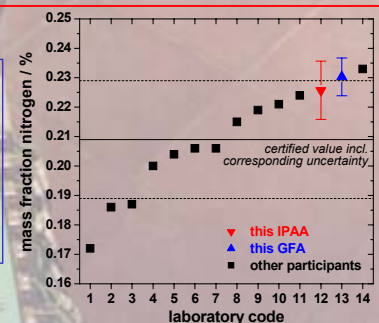
Individual GFA results

[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

boron carbide	boron nitride	nitrogen mass fraction / %				
		no correction	with geometry correction only	with air & geometry & air correction	with geometry, air & blank correction	
1177.84	74.8230	0.264	0.265	0.235	0.236	0.227
1101.83	73.3476	0.265	0.265	0.227	0.229	0.226
1120.86	64.1246	0.224	0.226	0.251	0.263	0.244
1093.81	65.5035	0.267	0.268	0.223	0.224	0.216
1177.84	74.8230	0.261	0.263	0.232	0.234	0.225
1101.83	73.3476	0.265	0.267	0.231	0.233	0.223
mean & standard deviation		0.265 ± 0.002	0.267 ± 0.002	0.233 ± 0.010	0.235 ± 0.010	0.228 ± 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

- [1] Zertifikat BAM-S003, http://www.bam.de/pdf/service/referenzmaterialien/zertifikate/special_materials/bam_s003report.pdf
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- [4] R. Matschat, A. Dette, *Final certification report on BAM S-003 Silicon carbide powder* (2004).
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