Neutron Activation Analyses of Ultrapure Liquids in a Silicon-Made Irradiation Flask ◇

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Ultra-trace level analyses are often falsified by contamination of the samples due to storage and handling. In NAA quartz-made irradiation flasks have been used so far, whose purity is sufficient for many applications. To examine liquid scintillators at a purity-level required for the BOREXINO-experiment ($K < 10^{-14}$ g/g; $U < 10^{-16}$ g/g; $Th < 10^{-16}$ g/g) it is however necessary to avoid normal as well as fast neutron enhanced diffusion of impurities out of the wall into the sample. Besides after irradiation and removal of the sample, the flask has to be rinsed with aggressive acids to avoid loss of activated nuclei. These sometimes prefer to stick to the wall of the flask rather than to stay dissolved in the liquid sample. The importance of this effect depends on the chemical composition of the sample as well as on the element under consideration. Rinsing of the flask can wash out impurities from the flask what is another source of falsification of the analyse.

element	silicon	quartz
K	$< 2 \cdot 10^{-12}$	$4 \cdot 10^{-8}$
U	$< 3 \cdot 10^{-17}$	$< 3 \cdot 10^{-11}$
Th	$< 3 \cdot 10^{-16}$	$< 6 \cdot 10^{-12}$
Au	$< 2 \cdot 10^{-15}$	$7 \cdot 10^{-12}$
Ag	$1 \cdot 10^{-15}$	$1 \cdot 10^{-9}$
Co	$2 \cdot 10^{-15}$	$5 \cdot 10^{-9}$
Cr	$6 \cdot 10^{-13}$	$3 \cdot 10^{-8}$
Fe	$< 2 \cdot 10^{-13}$	$7 \cdot 10^{-7}$
W	$< 1 \cdot 10^{-14}$	$8 \cdot 10^{-11}$
Zn	$< 3 \cdot 10^{-15}$	$5 \cdot 10^{-8}$

Table 1: Comparison between impurities in FZ-silicon and synthetic quartz

The difficulties quoted above can be avoided by employing material as pure as possible for the fabrication of the irradiation flask. Electronic-grade silicon is one of the purest solids available at the moment but also a rather brittle material, that can be worked only with great difficulties. Nevertheless it has been possible for the first time to manufacture a 500 ml irradiation flask out of FZ-silicon whose purity level exceeds the one of synthetic quartz generally for several orders of magnitude (table 1).

As far as quartz-made irradiation flasks are concerned a drastic reduction of the irradiation time improves the sensitivity for potassium and the chemically similar sodium by nearly two orders of magnitude, what seems to be a hint to diffusion-like effects. First analyses with the silicon flask could not yet reproduce these data for every element:

A comparison of the different irradiations made so far shows variations of sodium, potassium, chromium and zinc by two orders of magnitude, that might be caused by difficulties during the sample preparation. Future analyses will show whether the clean room conditions are sufficient and whether the sensitivity for sodium is limited independently from potassium, for example by the fast neutron induced $^{28}Si(n,\alpha p)^{24}Na$ reaction.

The best values obtained so far are shown in table 2.

K	$< 3 \cdot 10^{-12}$
Na	$2 \cdot 10^{-12}$
Ar	$2 \cdot 10^{-7}$
As	$< 4 \cdot 10^{-14}$
Au	$5 \cdot 10^{-15}$
Br	$2 \cdot 10^{-12}$
Cr	$< 4 \cdot 10^{-12}$
La	$< 2 \cdot 10^{-14}$
Sb	$< 7 \cdot 10^{-14}$
W	$< 4 \cdot 10^{-14}$
Zn	$< 6 \cdot 10^{-12}$

Table 2: Impurities in subboiled water (activated in silicon-made irradiation flask)

◇ This work was supported by the "Sonderforschungsbereich 375-95 für Astro-Teilchenphysik" of the DFG and by the BMBF.