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Note

Detection of boron compounds on chromatograms by (n, α) nuclear reaction with thermal neutrons

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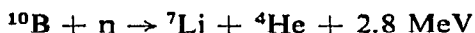
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Natural boron contains *ca.* 20% of the ^{10}B isotope which, in contrast to most elements, exhibits a relatively high probability of the process of thermal neutron capture followed by α -particle emission. The α -particles, created in the (n, α) nuclear reaction



have an isotropic angular distribution and energies of 1.79 MeV (7%) and 1.48 MeV (93%). Detection of this α -radiation was formerly used for the evaluation of the boron content in different materials^{1,2}. The short range of α -particles in the sample materials, however, permits only the particles emitted from the surface to be detected. A homogeneous distribution of the boron compound through the whole depth of the sample is therefore a necessary condition for the accurate determination of the boron content in thick layers. If this condition obtains, a surface concentration mapping of boron content is possible.

This paper deals with an application of the above method in TLC, representing the first general method for the detection of boron compounds and the quantitative determination of boron content in separated spots. Silufol sheets (Kavalier, Votice, Czechoslovakia), *i.e.*, pre-coated silica gel starch-bound layers (25 μm) on aluminium foil (100 μm), were found to be the best material for the separation of all types of boron compounds³⁻⁶.

METHODOLOGY

The thermal neutron beam passes through an aluminium sheet, a silica gel layer and the α -particle detector, which is located very close to the surface of the adsorbent. In the areas that contain adsorbed boron compounds, the capture of thermal neutrons produces α -radiation, the intensity of which can be quantitatively measured with an α -particle detector. The chromatogram is moved stepwise through the beam of neutrons, each step being combined with a measurement of the number

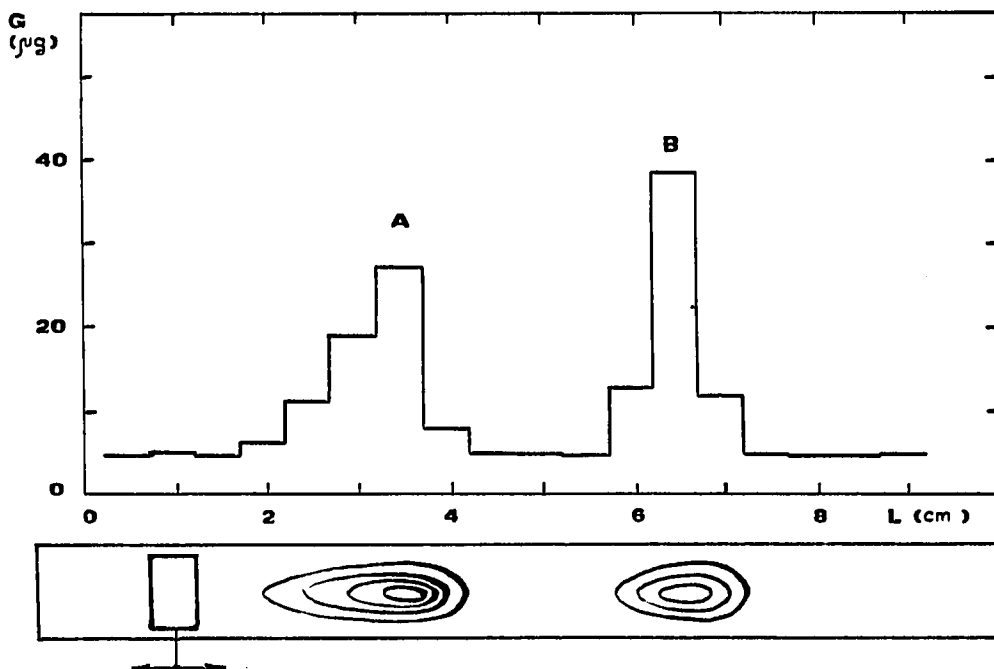


Fig. 1. Detection and quantitative mapping of spots in a separated mixture of $C_5H_5-Co-C_2B_9H_{11}$ (A) and $8,8'-CH_3S(C_2B_9H_{10})_2Co$ (B) using the $^{10}B(n,\alpha)$ reaction on Silufol.

of α -particles emitted from the given area. The comparison of these values with a calibration line (obtained by determining the intensity of α -radiation of samples with known concentrations of boron) permits the quantitative evaluation of the boron content in the particular cross-sections of the chromatogram (Fig. 1).

EXPERIMENTAL

The horizontal beam of collimated neutrons (*ca.* 10^6 neutrons/cm²·sec) from the VVR-S nuclear reactor (Řež, near Prague) impinged perpendicularly on the chromatogram strip, which was fixed in a moveable holder (Fig. 2). On the opposite side of the sheet, very close to the surface of the silica gel (0.5 mm), a silicon semiconductor surface barrier detector was located, detecting the α -particles from the boron (n,α) reaction within the surface layer of the chromatogram. The silicon detector was provided with a 5×10 mm window, which was adequate for scanning the path of the chromatographed mixture. The α -particle spectrometer, comprising a charge-sensitive pre-amplifier, linear amplifier and multichannel analyser, allowed the energy spectrum to be recorded (Fig. 3). In practical applications of the method, the multichannel analyser may be replaced with a scaler with a discriminator. A stepping motor moved the thin-layer chromatogram by 5 mm in one step, each area being scanned for 5 min. The rate of emission of α -particles in the interval of 190–340 channels, corrected for the background of the Silufol sheet, is proportional to the amount of boron on the surface of a window area of 5×10 mm.

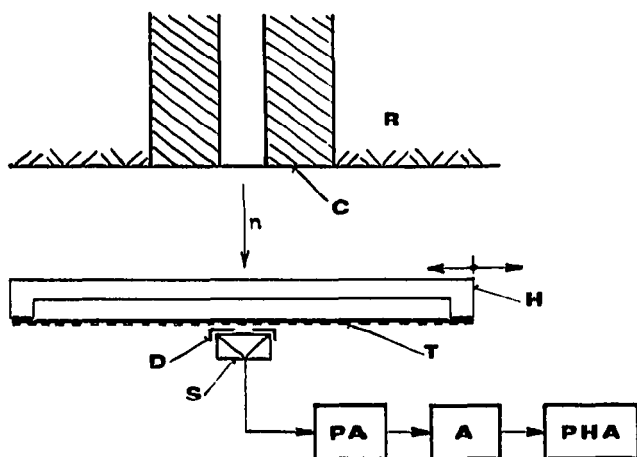


Fig. 2. Experimental arrangement (plan view). R = nuclear reactor shielding; C = collimator of neutron beam; H = traversable holder of chromatogram; T = Silufol sheet; S = silicon semiconductor detector; PA = charge pre-amplifier; A = linear amplifier; PHA = multichannel amplitude analyzer; D = aluminium window, 5×10 mm.

A series of chromatograms was investigated with the aim of determining the depth homogeneity of the Silufol sheets. Identical amounts of the compound $C_5H_5-Co-C_2B_9H_{11}$ were developed to different R_F values, so that the spot areas and hence the surface density of the boron compound differed by a factor of about four. The (n, α) measurements indicated that the boron content of all the spots was constant to

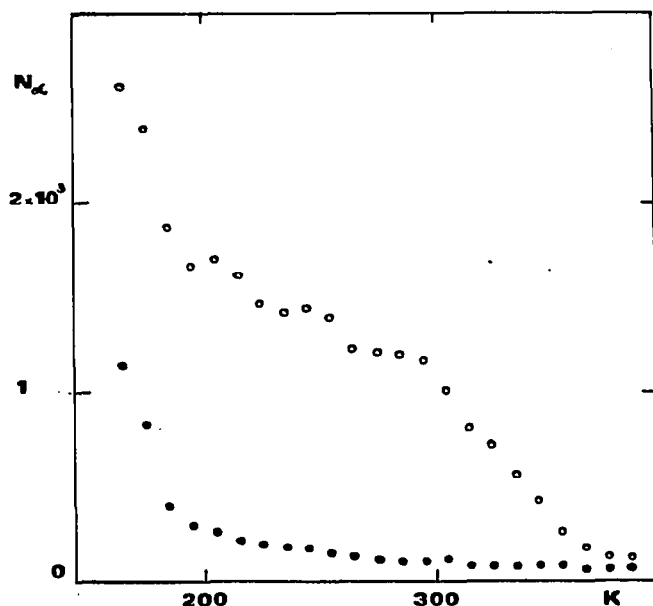


Fig. 3. α -Particle energy spectrum from the $^{10}B(n, \alpha)$ reaction with thermal neutrons, irradiation time 5 min. O, $C_5H_5-Co-C_2B_9H_{11}$; ●, background of Silufol.

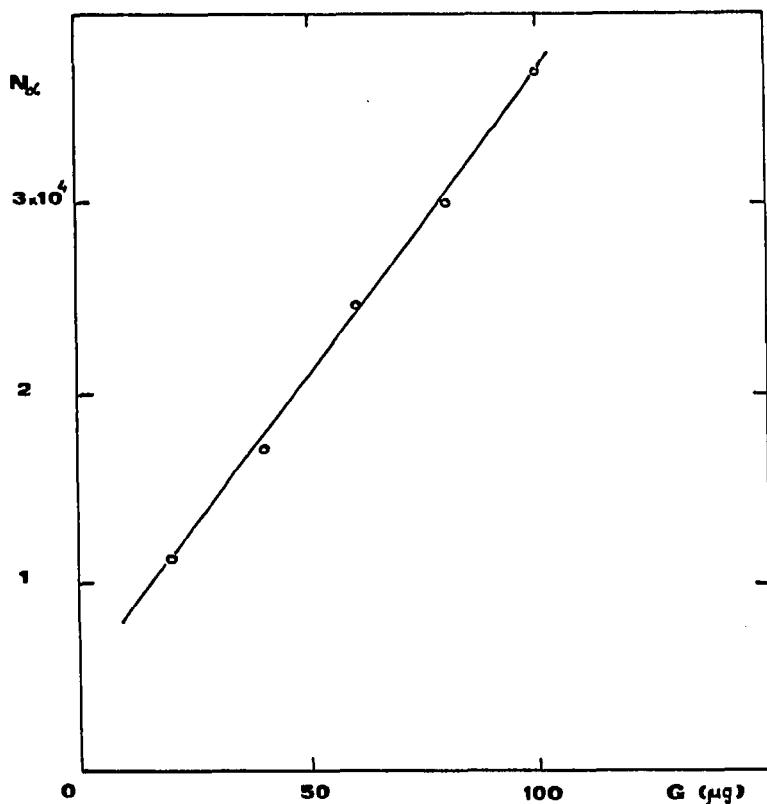


Fig. 4. Dependence of the α -particle counting rate from the ^{10}B (n, α) reaction on the boron content in $\text{C}_5\text{H}_5\text{-Co-C}_2\text{B}_9\text{H}_{11}$.

within $\pm 6\%$, which shows that the depth homogeneity of the Silufol sheets is satisfactory. For quantitative evaluation of the boron content of the investigated compounds, a calibration line (Fig. 4) was obtained by plotting the α -particle counts against the known boron concentrations in selected spots of the $\text{C}_5\text{H}_5\text{-Co-C}_2\text{B}_9\text{H}_{11}$ compound.

SCOPE AND LIMITATIONS

The method described permits the selective and simple determination of the boron content in the TLC spots in the range 2–100 $\mu\text{g}/\text{cm}^2$. The accuracy of the quantitative determination of boron content is influenced by several factors, the most important of which are the statistical error in the number of α -particle counts, the error of the calibration and the error resulting from the non-homogeneity of the Silufol surface. The statistical error may be reduced by prolonging the scanning time. The depth non-homogeneity of the Silufol sheet is responsible for an error of about 6%. In order to reduce the error of calibration, we employed a set of boron standards of different weights. Nevertheless, even in this case the error is likely to be about 5%. This calibration method allows a systematic error to be determined. This is also demon-

strated in Fig. 4, where the calibration line does not pass through the origin. The overall accuracy of the method is expected to be about 10%.

Of other isotopes, only ${}^7\text{Li}$ exhibits a similar type of (n,α) nuclear reaction. The α -particles from the ${}^7\text{Li}$ (n,α) reaction cannot be separated from the α -particle energy spectrum obtained with the experimental arrangement described. Fortunately, water-stable lithium compounds exist mainly in the form of salts which are not suitable for TLC separation.

The possibility of placing the samples in a vacuum chamber was investigated, which permits a more suitable geometrical arrangement with the silicon detector outside the neutron beam. Such an arrangement permits a considerably more intensive neutron beam to be used and lowers the background. The experiments showed, however, that the results obtained from a series of measurements on one sample are not reproducible. In particular, it was observed that the α -particle counting rate from the Silufol sheet increased with the time during which the sample was kept in a vacuum. This effect is believed to be due to evaporation of water from the Silufol surface.

In conclusion, it is suggested that the method described based on the (n,α) reaction is likely to become an efficient analytical procedure for the determination of boron.

ACKNOWLEDGEMENT

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