New Inorganic Boron Assessment in environmental Samples by Neutron Activation Analysis using CR-39 Solid State Nuclear Track Detectors.

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Abstract: A new trend in applying nuclear solid-state track detector CR-39 for neutron activation assessment of inorganic boron compounds in environmental samples was established. The nuclear reaction \(^{10}\text{B}(n, \alpha)^{7}\text{Li}\) was selected for this purpose by using gelatin film for a sample application. It possesses many advantages such as time saving, sensitivity, and reproducibility, and many related variables were studied. Linearity was found at the range of 1.0 - 12.5 ppm and at 10.0 -250 ppm with different slopes. There were no interfering nuclei among the naturally abundant elements and their isotopes, viz. Al, O, Na, Cl, P, Ca, Si, K, and Cu. It was applied to determine boron in simulated environments and industrial soil and water samples, with recovery within the range of 96-100 %, good precision and accuracy ± 1.5 - 3.0 %. The method sensitivity can be enhanced by using longer irradiation time, and few micrograms of boron can be analysed, and hence it is more suitable to be used in the activation analysis system of nuclear reactors.

Keywords: Nuclear solid state track detector; neutron activation analysis; boron compounds; \(^{10}\text{B}(n, \alpha)^{7}\text{Li}\); and gelatin film method.

1. Introduction:

Many nuclei react with neutron radiation through the nuclear reaction \((n, \alpha)\) and emit alpha particle (\(^{4}\text{He}^{2+}\)). It is possible to record the emitted alpha particle on a nuclear solid-state track detector (NSSTD) \([1-5]\). CR-39 is among such type of detector, composed of the polymer polyallyl diglycol carbonate (PADC), and it finds wide use to record alpha particles track \([2]\). The isotope \(^{10}\text{B}\) of boron is often employed in a vast nuclear application where the \((n, \alpha)\) reaction is of advantage, as well as of being a non-radioactive element and readily available \([3-5]\). Boron doped NSSTD, viz. orthocarbonarane, \(\text{B}_{10}\text{H}_{12}\text{C}_{2}\) were found convincing applications for neutron dosimetry \([6-8]\). Boron neutron capture therapy (BNCT) is widely used for radiation therapy mediated by the short-range (less than 10 \(\mu\)m) energetic alpha \([9]\). The size and characteristics of the alpha particle track depended upon the energy and direction of motion, in connection with NSSTD \([10-13]\).

Boron and its compounds can exist in gas, liquid and solid states, and are widely used for neutron detection and boron isotopic ratio determination \([14]\), or shielding and absorption through the \(^{10}\text{B}(n,\alpha)^{7}\text{Li}\) reaction \([15-17]\). Boron is an important element over a wide range of applications from meteorites to human tissue, there is a potential possibility to find the new analytical method for its determination by neutron activation analysis for environmental and industrial samples. This possibility is very more likely to be used in the presence of well-developed benchtop neutron generators as well as a nuclear solid-state track detector (NSSTD) \([18]\). It’s worth mentioning that Prompt Gamma-Ray Activation Analysis (PGRA) already finds wide applications for the extremely sensitive analysis of boron content in several mediums with simply modified techniques \([19-21]\). As far as we are aware,
there is no work reported in the literature for using the reaction (n,α) for activation analysis, a part from 
that of the very old attempt of Tsurut [22].

The aim of this study is to investigate the variables that facilitate the use of $^{10}$B nuclear reaction 
for the assessment of boron in many environmental and industrial matrices by combining neutron activation 
with NSSTD. Many traditional instrumental methods were suggested for the determination of boron by 
spectrophotometry with a specific reagent such as carmine, curcumin, and azomethine, in irrigation 
water and soil [23-25]. Boron was extracted from soil by many suggested methods, mostly depended on 
hot-water extraction [26-27]. Concentrations of boron in groundwater throughout the world were 
reported to be in range widely, from 0.3 to 100 ppm, and in plant tissue exceeding 2 ppm can be of 
harmful effect [28].

2. Material and method:

2.1 Materials:
Boric acid, sodium tetraborate, sodium hydroxide, sodium chloride NaCl, potassium carbonate was 
obtained from BDH chemicals, UK as ANALAR grade. Hydrochloric acid, glycerin, sulphuric acid, 
was used without purification, gelatin, phosphorus acid, sodium silicate Na$_2$SiO$_3$, aluminium oxide, 
copper sulphate pentahydrate CuSO$_4$.5H$_2$O, sodium chloride NaCl, potassium carbonate was obtained 
as ANALAR grade and used without purification from Chemical Point, Germany.

2.2 Solid state nuclear track detector:
Nuclear solid-state track detectors (NSSTD) CR-39 used in this study were obtained from Moulding 
Ltd, UK with 500 μm thickness, and a density of 1.3 g/cm$^3$ [13]. The CR-39 detectors with samples 
were exposed at various times, 5.0 cm away from the neutron source, and space was occupied by wax 
to ensure the receiving of thermal neutrons. Sheets of CR-39 detectors were cut into pieces square 
with a dimension of 1.0 cm × 1.0 cm. Microscope description was performed by using Olympus 2BH.

2.3 Neutron source:
All samples were irradiated with neutron at ambient temperature with a $^{241}$Am-Be neutron source was 
supplied by the Radio - Chemical, Ltd., England, with a flux of $10^5$ neutrons.cm$^{-2}$.sec$^{-1}$. Thermal neutron 
bam was obtained by placing petroleum wax around the source, such a way the distance of samples from 
the source is 5.0 cm.

2.4 Solutions preparation:

2.4.1 Standard borax solution 290 ppm: Weigh accurately 2.6540 g of sodium tetraborate, 
Na$_2$B$_4$O$_7$.10H$_2$O using an analytical balance and dissolve it in 500 mL volumetric flask containing ~ 100 mL distilled, and complete the volume to the mark with distilled water. The concentration of this solution is 290 ppm of boron.

2.4.2 Preparation of working calibration boron solution: The successive dilution procedure was used 
to prepare concentration from 1- 250 ppm boron compound solutions from the standard borax solution 
of 290 ppm according to Table-1.

2.4.3 Gelatin solution: Gelatin (1.5 g) dissolved in 40 mL of hot distilled water, and used warm 
within the range 50-60ºC. It solidifies when reaching room temperature, as shown in Figure-1.

2.4.4 Sodium hydroxide solution (6.25 N): An amount of 12.5 g of sodium hydroxide pellets was 
carefully dissolved in 30 mL distilled water, and the solution was transferred to a 50.00 mL volumetric 
flask quantitatively. The volume was completed to the mark. This solution was used to treat the
irradiated SSNT detectors by immersing them in a hot solution at a specific temperature and time. The detectors were washed with distilled water after alkali treatment and dried before track counting.

Table 1: The successive dilution procedure used to prepare concentration from 1- 250 ppm boron compound solutions.

| #  | Volume of standard sodium tetraborate solution (290 ppm) | Final concentration (ppm) |
|----|--------------------------------------------------------|--------------------------|
| 1  | 86 mL of stock solution diluted to 100 mL              | 250                      |
| 2  | 69 mL of stock solution diluted to 100 mL              | 200                      |
| 3  | 52 mL of stock solution diluted to 100 mL              | 150                      |
| 4  | 34.5 mL of stock solution diluted to 100 mL            | 100.0                    |
| 5  | 17 mL of stock solution diluted to 100 mL              | 50.0                     |
| 6  | 8.5 mL of stock solution diluted to 100 mL             | 25.0                     |
| 7  | 3.5 mL of stock solution diluted to 100 mL             | 10.0                     |
| 8  | 10 mL of 50 ppm solution diluted to 100 mL             | 5.0                      |
| 9  | 10 mL of 10 ppm solution diluted to 100 mL             | 1.0                      |
| 10 | 0 mL of 10 ppm solution diluted to 100 mL              | 0.0                      |

Table 2: The effect of adding a similar concentration of a naturally abundant element in environmental samples to a similar concentration of boron or sodium tetraborate.

| #  | Boron (50 µL of 50 ppm) | Interfering element (50 µL of 50 ppm) | Alpha (Track.mm⁻²) | Boric acid | Sodium tetraborate |
|----|-------------------------|--------------------------------------|---------------------|------------|-------------------|
| 1  | B                       | -                                    | 1780                | 1670       |                   |
| 2  | B                       | Al                                   | 1753                | 1680       |                   |
| 3  | B                       | O                                    | 1768                | 1670       |                   |
| 4  | B                       | Na                                   | 1771                | 1687       |                   |
| 5  | B                       | Cl                                   | 1782                | 1672       |                   |
| 6  | B                       | P                                    | 1790                | 1690       |                   |
| 7  | B                       | Ca                                   | 1782                | 1675       |                   |
| 8  | B                       | Si                                   | 1769                | 1672       |                   |
| 9  | B                       | K                                    | 1785                | 1678       |                   |
| 10 | B                       | Cu                                   | 1790                | 1687       |                   |

Mean 1776.67 1679
Standard Deviation (s) 12.288 7.467
Relative average Change % -0.187 % 0.539 %

2.4.5 Calcium chloride hot water extraction for available B: 1.47 g of 0.01M CaCl₂ was dissolved in approximately 900 mL of distilled water. The solution volume was adjusted by using a 1000 mL volumetric flask and the volume was completed to the mark with distilled water.

2.5 Alpha track Observation by Gelatin film method:
The most common technique used to record the tracks of the emitted alpha particles is by developing the track pits within the SSNT detector by treatment with a strong hot alkali metal solution for the specified time. The track record was clearly observed with an optical microscope with magnification power of 400x. In this method, gelatin film is previously prepared by applying a gelatin solution (~ 100 µL), to SSNTD and leaving it to dry overnight. Boron samples can be added as a solution with volumes depend on their concentration, ~ 50 - 100 µL. Gelatin film-coated detectors were prepared by applying
a warm gelatin solution (temperature of 50 - 60°C) and left undisturbed overnight in a well-ventilated cabinet. Volumes of the above gelatin solution (100 µL) were carefully added to a set of detectors and left undisturbed overnight in a well-ventilated cabinet. Different volumes of the standard solution of 290 ppm of boric acid /or sodium tetraborate (0, 5, 10, 20, 30, 40, 50, 60, 70, 80, and 90 µL), was transferred by pipette to 100 mL volumetric flasks, then diluted with distilled water to the mark as shown in Table-1. The detectors were left overnight in a well-ventilated cabinet. The prepared detectors were irradiated with a thermal neutron beam for 24 hours, removed from the neutron source, and treated with 6.25 N sodium hydroxide solution at 70°C for 3 hours, as shown in Figures 2-6.

2.6 Studying variables in gelatin film method:
The following variables were studied, to find the convenient condition for obtaining good reproducibility for the method:

2.6.1 Effect of irradiation time: Samples of sodium tetraborate (100 mg) were prepared and irradiated at a different time; Pitting temperature was 70°C, the samples were irradiated with thermal neutrons at a different time: 1, 2, 3, 4, 5, 6, 24, 48, 72 hours, and the result was presented in Figure-2.

2.6.2 Effect of alkali temperature: Samples of 100 ppm of boric acid or sodium tetraborate were prepared by applying 100 µL onto the previously prepared gelatin film on SSNTD and irradiated with thermal neutrons for 24 hours. The detectors were treated with sodium hydroxide solution at a different time; 50, 55, 60, 65, 70, 75, 80, 85°C. The recorded track number were counted with the help of a microscope and the tracking number was plotted against temperature, and the result was presented in Figure-3.

2.6.3 Effect of alkali time treatment: A volume of 100 µL of solutions of 100 ppm of boric acid or sodium tetraborate was applied onto previously prepared gelatin film-coated SSNTD and irradiated at a different time; Pitting temperature was 70°C, the samples were irradiated at a different time; 0, 6, 24, 30, 48, 54, 72, 78, 96, 102 hours and the result were presented in Figure-4.

2.6.4 Effect of different boron concentration: A volume of 100 µL of varying concentration of sodium tetraborate solution (0, 0.1, 1.0, 10.0, 30.0, 40.0, 50.0, 60.0, 70.0, 80.0, and 90.0 ppm) carefully applied to a set of gelatin film-coated detectors. The solutions were left overnight and irradiated with thermal neutrons for 24 hours. After treatment with concentrated sodium hydroxide solution at 70°C for 3 hours, the tracking number was counted under a microscope, and the result was presented in Figure-5 and Figure-6.

2.7 Studying the interferences of light elements:
The interference effect of the naturally abundant elements usually presents in the environment was studied. It includes the addition of nine elements viz., oxygen as sulphuric acid, aluminium as aluminium oxide, sodium as sodium carbonate, chlorine as hydrochloric acid, phosphorus as phosphorus acid, copper as Copper sulphate, CuSO₄·5H₂O, calcium as calcium carbonate, silicon as sodium silicate Na₂SiO₃, potassium carbonate. A mixture of known concentration of boron compounds (50 µL + 50 ppm) with one of eight elements (50 µL + 50 ppm) was prepared and irradiated for 72 hours, then developed with strong alkali at etching temperature of 70°C for 3 hours, and the results were presented in Table-2.

2.8 Simulated soil samples with boron:
Samples from local Iraqi soils (10.00 g) were sieved to 500 µm and dried at 110°C for three hours. Volumes of 250 ppm standard boron solution; 10.0, 5.0, 2.0, 1.0, and 0.50 mL were added to each soil portion. Samples were mechanically shacked with reciprocal shaking for 30 minutes and then dried again at 110°C for 3 hours prior to extraction.
2.9 Extraction of boron from soil samples:
Each of the samples was transferred to 125 mL plastic flasks and treated with 25 mL of calcium chloride hot water extraction for available boron. Flasks were shaken on a hot water shaking bath for 30 minutes at 80°C. Filter into 125 mL plastic Erlenmeyer flasks, using Whitman No. 1 filter paper and refilter if necessary [26-28]. Wash the sediment thrice with 10 mL portions of calcium chloride hot water and combine the filtrate. Transfer the filtrates to a 50 mL volumetric flask and complete the volume to the mark.

2.10 Mounting boron sample solution onto CR-39 SSNT detector:
A volume of 100 mL of the above-prepared boron solutions was placed on the surface of 1.0 × 1.0 cm CR-39 SSNT detector coated with gelatin film, previously prepared by applying a gelatin solution (~100 µL), to SSNTD and leaving it to dry overnight.

Table-3: The content of boron measure for simulated environmental and industrial soil and waters as well as their recoveries.

| #  | Sample                  | B added (µg) | B obtained (µg) | RAD* % | Recovery % |
|----|-------------------------|--------------|-----------------|--------|------------|
| 1  | Standard boron solution 1 | 4.00         | 3.96            | + 3.7  | 99         |
| 2  | Standard boron solution 2 | 6.00         | 5.88            | + 2.8  | 98         |
| 3  | Standard boron solution 3 | 10.00        | 10.00           | + 2.6  | 100        |
| 4  | Standard boron solution 4 | 50.00        | 49.50           | + 2.4  | 99         |
| 5  | Standard boron solution 5 | 100.00       | 98.00           | + 2.1  | 98         |
| 6  | Standard boron solution 6 | 150.00       | 150.00          | + 1.7  | 100        |
| 7  | Standard boron solution 7 | 250.00       | 247.50          | + 1.5  | 99         |
| 8  | Soil 0 (10.0 g) Blank    | 0.00         | 0.00            | + 0.0  | 00         |
| 9  | Soil 1 (10.0 g)          | 2.50         | 2.38            | + 4.1  | 95         |
| 10 | Soil 2 (10.0 g)          | 5.00         | 4.80            | + 2.7  | 96         |
| 11 | Soil 3 (10.0 g)          | 10.00        | 9.80            | + 2.5  | 98         |
| 12 | Soil 4 (10.0 g)          | 25.00        | 24.25           | + 2.7  | 97         |
| 13 | Soil 5 (10.0 g)          | 50.00        | 48.50           | + 2.3  | 97         |

*RAD: Relative average deviation.

3. Results and Discussion:
The tracks of alpha particles on the CR-39 SSNT detector was developed by using a strong alkali solution, viz. 6.25 N NaOH solution for specified time and temperature. Gelatin was used as the film that sticks to the surface of SSNTD in order to hold the boron compound samples applied as a solution, as shown in Figure-1. This method is supposed to offer an easy way to handle samples, where the gelatin solution was spread over the SSNTD and left to dry forming the uniform film. The tendency of gelatin to absorb the aqueous solution inside its body is the driving force to bring boron compound to be in touch with the detector surface homogeneously. By following the procedure described in Section 2.4 of the experimental part. The track density was found to be directly proportional to the irradiation time good way, as shown in Figure-2. The uniformity of the sodium tetraborate signal seemed to be good, probably due to its good water solubility.

By following the procedure described in Section 2.4 of the experimental part. The track density was found to be directly proportional to the irradiation time in a much better way than that in the disc method. As shown in Figure 2. The uniformity of the sodium tetraborate signal seemed to be good, probably due to its good water solubility.

The effect of alkali temperature was also studied by applying 100 µL onto the previously prepared gelatin film on SSNTD and irradiated with thermal neutrons for 24 hours. The detectors were treated with sodium hydroxide solution at different temperature (50, 55, 60, 65, 70, 75, 80, 85°C). The recorded
track number were counted and plotted as shown in Figure-3 with the help of a microscope and the tracking number was plotted against temperature. The plot reveals a slight increase in the recorded track density for $^{10}$B within the range from 55 – 85 °C by a value of 20 %, and 2 % within the range 60-65 °C. The later temperature range was used along with the following experiments.

**Figure-1**: Gelatin solution applied onto the CR-39 SSNT detector, was distributed in a way to give the homogenous film.

**Figure-2**: The effect of irradiation time on the alpha particle recorded CR-39 SSNT detectors applied with the 100 μL gelatin film, then with the sample containing 100 μL 100 ppm.
Figure 3: The effect of etching temperature of the alpha particle recorded on CR-39 SSNT detectors coated with gelatin film, and applied with 100 µL 100 ppm of boron. The detectors were treated with 6.25 N NaOH at a different etching temperature in the range 55 – 85°C.

Figure 4: The alpha particle recorded for different 100 mg disc of boron compound irradiated with a thermal neutron beam for 24 hours, and varying etching time in hours with a solution of 6.25 N NaOH.

To prepare a standard calibration curve for boron in the range 1 - 250 ppm, gelatin film-coated detectors were prepared by carefully applying gelatin solution (100 µL) (at a temperature of 50 - 60°C) on a set of CR-39 detectors (10-20), and left undisturbed overnight in a well-ventilated cabinet. Different volumes of the standard solution of 290 ppm of sodium tetraborate (0, 5, 10, 20, 30, 40, 50, 60, 70, 80, and 90 µL), were transferred by pipette to 100 mL volumetric flasks, then diluted with distilled water to the mark as shown in Table-1.
The detectors were left overnight in a well-ventilated cabinet to dry and then, were irradiated with a thermal neutron beam for 24 hours. Removed from the neutron source, and treated with 6.25 N sodium hydroxide solution at 70°C for 3 hours. The value of track densities was plotted against concentrations, a plot with the linear region was obtained at a concentration range 10 - 250 ppm, as shown in Figure-4. For the determination of the linearity between the concentrations below of 10 ppm, another calibration curve was prepared, as shown in Figure-5. The relation was not found to be perfectly linear in the same mode as that of the higher range (10 – 250 ppm). It is more likely to be a semi-linear relation, with some convex curvature, a little hump with a small tendency to the form of a pseudo plateau. The reason for this difference can be attributed to the increased interaction with the film component, viz. gelatin molecules. The amount of boron $^{10}$B atoms present (natural abundance of 19.9 %) at such low concentration will be depleted at a prolonged irradiation time, provided at a concentration lower than 0.5 µg. At such concentration, the domination of gelatin molecule concentration compared to that of $^{10}$B compounds is much larger than the previous range of 1.0 - 10.0 ppm.

**Figure 5:** The alpha particle recorded for different CR-39 SSNT detectors coated with different concentration of boron compounds (10 -250 ppm). The detectors were irradiated for 24 hours, then developed with alkali at different etching temperature.

**Figure 6:** The alpha particle recorded for different CR-39 SSNT detectors coated with different concentration of boron compounds (1.0 - 12.5 ppm). The detectors were irradiated for 24 hours, then developed with alkali at different etching temperature.
The effect of the naturally abundant elements usually present in the environment, on boron signal were examined. The results were presented in Table-2, which indicates that only a decrease of -0.187 % in the boric acid signal, while an increase of 0.539 % in the sodium tetraborate signal. Both deviations are very small compared to many analytical methods and can be considered insignificant. In another meaning, these elements contribute no significant change in reading the alpha track originated from the nuclear reaction of $^{10}$B with neutron radiation in such conditions.

From the present experiments, it is possible to suggest three modes of thermal neutron interaction with $^{10}$B in boron compounds in three different modes according to the concentration and irradiation time, as illustrated in Figure-7. At low concentration; the number of the boron $^{10}$B atoms producing alpha particle, is dominated by the molecules of the solid diluent molecules, viz. the gelatin molecules with a weight of 3.75 mg per 100 µL, or 0.0589 mg.mm$^{-2}$ (calculated on the basis of a circle diameter of 9 mm). On applying 100 µL of boron compound solution on gelatin film, it penetrates through the film to the detector surface. This amount of boron is good enough to show the effect on the detector after thermal development with a strong alkali. On increasing the concentration, the number of boron atoms will be increased, resulting in a sharp increase of the alpha particle. In the second mode, the interaction between alpha particle with detector the number of $^{10}$B atoms (higher than 2.0 ppm) will be in a way dominating the number of gelatin molecules. This increase in the tracking number of this mode is less than that of the previous mode, and the depletion of the $^{10}$B atoms starts to appear in a small magnitude due to the long irradiation time. The third mode is a result of an equilibrium between the organic diluent effect and the apparent depletion effect. However, the effects of this procedure can be used for the determination of boron in the different environmental and industrial sample with good specificity and accuracy. To prepare simulated environmental samples, local Iraqi soils were sieved and dried, then different volumes of standard boron solution were added to them, then mechanically shacked prior to extraction. Extraction was performed by following hot calcium chloride solution [26-30]. Boron samples were applied to gelatin film coated on CR-39 SSNT detectors, dried and then irradiated with thermal neutrons for 60 hours. The result of the alpha particle tracks produced by each solution was recorded and the concentrations obtained from the calibration curve, as well as the original concentration, and the recovery was presented in Table-3.

![Figure 7](image-url): The recorded alpha particle tracks from $^{10}$B in boron compounds upon interaction with thermal neutrons in thin gelatin film, showing three areas of interaction with CR-39 SSNT detectors.

4. Conclusions:
The nuclear reaction $^{10}$B(n, α)$^7$Li was selected for activation analysis of boron compounds in environmental samples by coupling with very simple instrumentation and procedures by using gelatin film on the nuclear solid-state track detector CR-39 for neutron. The method poses many advantages for
being time-saving, sensitive, reproducible, and good linearity relationship with concentration. Many related variables were studied, and the best were selected and found to have two ranges. The method presents an accurate determination of boron in the range of 1-250 ppm. There were no interfering nuclei among the naturally abundant elements and their isotopes, viz. Al, O, Na, Cl, P, Ca, Si, K, and Cu. The method can be applied to measure boron in simulated environments and industrial soil and water samples, with recovery within the range of 96-100 %, good precision and accuracy ± 1.5 - 3.0 %. The method sensitivity can be enhanced by using longer irradiation time, and a few micrograms µg of boron can be analysed, and hence it can be used in the activation analysis system of a nuclear reactor. Future work can expand their use of samples related to industrial production, such as fossil fuels, mining, food, agricultural products, and waste management and recycling.

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