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Determination of boron in uranium–aluminum–silicon alloy by spectrophotometry and estimation of expanded uncertainty in measurement

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ABSTRACT

Quantification of boron in diverse materials of relevance in nuclear technology is essential in view of its high thermal neutron absorption cross section. A simple and sensitive method has been developed for the determination of boron in uranium–aluminum–silicon alloy, based on leaching of boron with 6 M HCl and H_2O_2, its selective separation by solvent extraction with 2-ethyl hexane 1,3-diol and quantification by spectrophotometry using curcumin. The method has been evaluated by standard addition method and validated by inductively coupled plasma-atomic emission spectroscopy. Relative standard deviation and absolute detection limit of the method are 3.0% (at 1σ level) and 12 ng, respectively. All possible sources of uncertainties in the methodology have been individually assessed, following the International Organization for Standardization guidelines. The combined uncertainty is calculated employing uncertainty propagation formulae. The expanded uncertainty in the measurement at 95% confidence level (coverage factor 2) is 8.840%.

1. Introduction

For the chemical quality control of materials of relevance, analytical determinations play an important role. In addition to assessing precision and accuracy of the method, it is also desirable to have an estimate of total uncertainty in the methodology as this increases the level of confidence in measurand. This is particularly true in determination of critical trace constituent such as boron in nuclear materials, which has stringent specification of only a few $\mu g\, g^{-1}$ or less [1,2]. It may be mentioned that allowed maximum concentration of other metallic impurities in nuclear materials also depends on boron content since total boron equivalent content should be less than $4\,\mu g\, g^{-1}$ for thermal reactors and $7\,\mu g\, g^{-1}$ for fast reactors. Quantitative measurement of boron concentration with all its associated uncertainties is therefore a necessary requirement for appropriate reactivity calculations. Analytical chemists are therefore required to give not only the quantitative results but also a reasonable estimate of total uncertainty associated with the measurements. A method based on leaching of boron from uranium–aluminum–silicon alloy (U–Al–Si) material and its determination has been developed in our laboratory. We have considered this methodology as a typical example to illustrate the estimation of total uncertainty. It may be mentioned that U–Al–Si alloy is used in thermal reactors, test reactors and in nuclear submarines [3].

Sah and Brown, in their review article reported several methodologies to determine the boron content at various levels from diverse matrices [4]. Because of simplicity and high sensitivity, a method based on spectrophotometric determination of boron with curcumin as complexing agent [5–8] is widely used for routine analysis. Dyrssen et al. [9] dealt in detail the chemistry of complex formation between boron and curcumin. However, several other ions also form complexes with curcumin [10] and they behave like boron–curcumin complex resulting in interference in the determination of boron. To overcome the interference, boron has to be selectively separated from the matrix either by solvent extraction with 1,3/2,4 diols [11–13] or by isothermal distillation of borate-alkyl esters [14]. Solvent extraction with 2-ethyl hexane 1,3-diol (EHD) is preferable for routine analysis due to its simplicity, high extraction coefficient and ease of operation. Dyrssen and Uppstrom have studied in detail the extraction mechanism and kinetics of boron-2-ethyl hexane 1,3-diol (B-EHD) complex formation [12].

Puphal et al. [15] determined boron content, in percentage level, in uranium–aluminum alloy samples by spectrophotometry with carmanic acid after dissolving in HCl and H_2O_2. Hamner and Deaeth [16] determined boron in silicon bearing alloys by pyrohydrolysis separation of boron at 1473 K and its quantification employing inductively coupled plasma atomic emission spectroscopy (ICP-AES). Makishima et al. [17] and Orazio [18] determined boron in silicate rocks by inductively coupled plasma mass spectrometry (ICP-MS), after decomposition of the sample with HF in the presence of mannitol. Parashar et al. [19] determined boron in high purity silicon material by spectrophotometry with

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curcumin as a complexing agent after dissolving the sample in a mixture of concentrated HF, HNO_3 and 0.1% of mannitol. However, significant variation in the boron values was reported. Taddia et al. [20] determined boron content in silicon doped (max 56 $\mu g\,g^{-1}$) gallium arsenide. In their methodology, after dissolving the sample in aquaregia, boron was separated from the matrix employing 2-ethyl hexane 1,3-diol (EHD) in chloroform. After removing interfering species from separated organic phase by treating with dilute HCl and boron was determined by spectrophotometry with curcumin. The detection limit of the method is 0.6 $\mu g\,g^{-1}$ and relative standard deviation in the determination of boron (precision) is 20% at 95% confidence level.

During the present studies, efforts to dissolve the uranium–aluminum–silicon sample with aquaregia were futile. This may be due to higher amounts of silicon ($\sim$36 $mg\,g^{-1}$) in the sample. No published literature is available, to our knowledge, for the determination of boron content in uranium–aluminum–silicon alloy samples. It is well known that HF interferes in the spectrophotometric determination of boron with curcumin since it will form strong complex with boron (BF_3 or BF_4^-) [6]. It is also observed in our earlier studies that HNO_3 above 1.0 M interferes in the determination of boron with curcumin [21]. Hence, present investigations have been aimed at developing a methodology employing spectrophotometry for the determination of boron in U–Al–Si alloy without using these interfering acids.

The fitness of the technique being developed, for the purpose of using it for routine analysis, has also been assessed by calculating precision and bias in the measurements, applicable linearity range, selectivity and detection limit of the method. Along with quantity of boron content in the sample, the combined or expanded uncertainty associated with the method employed has also been computed to increase the confidence on values of boron content. Uncertainty of measurement is defined [22] as ‘a parameter associated with the results of a measurement that characterizes the dispersion of the values that could reasonably be attributed to the measurand’. In view of its importance, International Organization for standardization (ISO) has given guidelines to compute expanded uncertainty with some examples [22,23]. It is desirable that all the analytical laboratories involve in analytical determinations follow these guidelines to arrive at a correct estimation of total uncertainty.

Two main approaches recommended by ISO [24,25] to evaluate the measurement uncertainty are ‘bottom–up’ and ‘top–down’ assessment procedures. Top–down approach is employed whenever a matrix-matched certified reference material with known concentration and uncertainty value is used for the validation of method. However, this approach does not give any information about the critical step associated in the analytical procedure that attributes as the major contributory in arriving at the uncertainty of the method. Since no matrix-matched certified reference material for boron is available, in the present studies, ‘bottom–up’ procedure, which uses each component of the quantification formula of measurand, was employed to evaluate the total uncertainty of measurement. These include sample preparation such as solid aliquoting from the sample, chemical treatment, absorbance measurement of test portion, evaluation of the measurement, quantification of the analyte along with associated uncertainty. This approach of reporting is different from the classical evaluation of the mean value of repetitive measurements and its standard deviation. Therefore, the sources of uncertainty in each step of measurement are identified and the size of each uncertainty is estimated. The next stage involves conversion of each uncertainty component (u_i) to relative standard uncertainty and calculation of the combined uncertainty (u_c) using uncertainty propagation formulae. Finally, the expanded uncertainty has been calculated using coverage factor of 2 at 95% confidence level.

The present paper describes in detail (i) the analytical methodology developed for determination of boron in U–Al–Si alloy by spectrophotometry and (ii) estimation of expanded uncertainty as per the ISO guidelines.

2. Experimental

2.1. Reagents

All the reagents used were of A.R. grade. High purity water obtained from Milli-Q Academic apparatus (Billerica, MA, USA) was used for all the dilutions/preparations of samples. Standard boric acid stock solution (96.6448 g) of 974.75 $\mu g\,g^{-1}$ was prepared by weight method by dissolving 0.5388 g of G.R. grade boric acid taken in polypropylene volumetric flask with water. The final working standard solution of 0.5957 $\mu g\,g^{-1}$ of boron was made by diluting above stock solution appropriately (118.082 and 11.257 $\mu g\,g^{-1}$ as intermediates). Quartz or polypropylene ware was used to avoid contamination of boron from glassware.

2.2. Instrument

Cary 500 Scan, Varian (Mulgrave, Victoria, Australia) UV–Vis-NIR double beam spectrophotometer, was used for absorbance measurements.

2.3. Procedure

About 200 mg of sample (U–Al–Si alloy granules) was taken in 20 mL quartz weighing bottle. Immediately after the addition of 1.0 mL of 6 M HCl solution, the weighing bottle was closed with leak tight quartz stopper (to avoid the loss of boron by evaporation) and kept in water bath for 5 min to bring it to room temperature (25 °C). To sustain the internal pressure due to release of gases, the lid and vessel were held tightly by means of metallic springs. Another portion of 3.0 mL of the acid solution (6 M HCl) was added and the bottle cooled to room temperature. Acid was added in two steps since single step addition of acid leads to vigorous reaction. After cooling to room temperature, 1.0 mL of 30% H_2O_2 was added to the contents, the bottle was stoppered and heated to $\sim$80 °C in water bath for 10 min to destroy the excess of H_2O_2 and to facilitate the dissolution of the sample to the maximum possible extent (about 90% of sample was dissolved) and also preventing loss of boron during heating (since boric acid is highly steam volatile). After cooling to room temperature, the stopper was removed and washed. The entire solution was quantitatively transferred to a quartz centrifuge tube using 1.0 mL of water. The solution was centrifuged at around 1300 rotation per minute (rpm) for 5 min to settle the undissolved material. The supernatant solution was separated and the undissolved material (about 20 mg, i.e. 10% of the sample) was washed with 1.0 mL of water, centrifuged and the supernatant liquid was added to the earlier separated solution.

To the separated and clear supernatant solution, 1.0 mL of 10% 2-ethyl hexane 1,3-diol (EHD) in $CHCl_3$ was added and stirred for 3 min with magnetic stirrer. The two phases were allowed to settle and the organic phase was separated by suction using polypropylene weight burette. This process was repeated for five times for quantitative separation of boron, i.e. boron was separated by batch extraction. From the entire organic phase thus collected, $CHCl_3$ was evaporated in a fume hood, either by heating on water bath ($\sim$80 °C) or natural evaporation at room temperature by keeping overnight. There will be no loss of boron during evaporation of $CHCl_3$ from the collected organic phase, since the boiling point of B-EHD complex (244 °C) [6] is much higher than that of $CHCl_3$

(78 °C). 1.0 mL of 0.375% curcumin in CH_3COOH and 0.5 mL of concentrated H_2SO_4 were then added to the residue left after evaporation of $CHCl_3$. After about 40 min, this mixture was transferred into a 10 mL standard quartz volumetric flask and diluted with *N*,*N*-dimethyl formamide (DMF), for deprotonation of protonated curcumin (since protonated curcumin and curcumin–boron complex absorb at same wavelength [7]). Blank solution was prepared in an identical manner by taking same amounts of all the reagents without the sample and following identical steps excepting centrifugation. The absorbance of the coloured complex of the sample was measured at 550 nm with reference to blank solution by using quartz cell having 1 cm path length.

3. Results and discussion

3.1. Quantitative extraction of boron

Detailed investigations on extraction of boron with 10% EHD in $CHCl_3$ from standard boric acid aliquot solutions showed that 80% of boron is extracted in each batch extraction and hence four batch extractions were adequate for quantitative recovery of boron. Hence, in all subsequent experiments, extraction was carried out for five times. Details about time required for quantitative complex formation between curcumin and boron under the experimental conditions; effect of different diluents viz. ethyl alcohol and DMF; interference studies with different cation and anions and effect of different acid media have been given in our earlier paper [21].

3.2. Calibration plot

By analyzing standard boric acid (0.5957 $\mu g\,g^{-1}$ B) aliquots employing above procedure, except centrifugation, calibration plot between absorbance and total amount of boron present in 10 mL solution, i.e. total amount of B present in aliquot was plotted. A linear regression equation attained as

$$A = 0.00131 \times w, \tag{1}$$

where A = absorbance; w = amount of boron present aliquot in ng.

The linear correlation coefficient is 0.9996 over the linear range of 80–730 ng with eight data pair points.

3.3. Precision and detection limit of the method

Ten independent standard boric acid aliquots were extracted and analyzed with relative standard deviation (R.S.D) of 3.0% (precision at 1σ level) and 98% mean of recovery was achieved. The calibration plot was checked for its reproducibility by analyzing standard boric acid aliquots at different time intervals and found that even after 6 months, same R.S.D., i.e. 3.0% was obtained. Ten independent blank solutions were prepared employing the procedure as mentioned for the sample and absorbance was measured with respect to water. The standard deviation (σ) in absorbance measurement was 0.00526. The absolute limit of detection [26] of this method as evaluated employing calibration plot (3σ/slope of calibration plot) was found to be 12 ng (0.06 $\mu g\,g^{-1}$ at sample size of 0.2000 g).

3.4. Standardization of the method

The method has been evaluated by standard addition method. Standard boric acid aliquot was added (B_{added}) to the solid sample aliquot and then total boron (B_{total}) was determined as described in experimental section. Boron in the sample was calculated as

$$B_{sample} = B_{total} - B_{added}. \tag{2}$$

Table 1
Standardization of the method by standard addition method

Sample	Amount of sample (g)	Amount of standard boron added (ng) B_{added}	Total Amount of boron measured (ng) B_{total}	Amount of boron in sample (ng) $B_{sample} = B_{total} - B_{added}$	Concentration of boron in sample ($\mu g\,g^{-1}$)
U–Al–Si-01	0.2223	297.7	426.4	128.7	0.45
U–Al–Si-01	0.2170	0.0	236.2	236.2	0.45
U–Al–Si-02	0.1558	143.2	405.2	262.0	1.68
U–Al–Si-02	0.1789	0.0	304.5	304.5	1.70
U–Al–Si-03	0.1670	343.6	475.0	131.4	0.79
U–Al–Si-03	0.2281	0.0	182.1	182.1	0.80

Typical results on the analysis of boron for the method standardization are given in Table 1. Results obtained by both the methods, i.e. direct and standard addition methods, agreed well within ±3%.

3.5. Validation of the method

The method has been validated using an independent method by dissolving the sample by acid digestion in a mixture of HF and HNO_3 in the presence of mannitol and determining boron employing inductively coupled plasma-atomic emission spectroscopy (ICP-AES) [27]. The results obtained in the determination of boron from U–Al–Si alloy employing both the methods are given in Table 2. It can be seen that by applying statistical treatment of the data (*t* test), there is no significant difference (at 95% confidence interval) between the two methods. As mentioned in Section 1, since HF interferes in the quantitative extraction of boron with EHD, the quantitative dissolution of the sample in presence of HF was not employed in the present methodology.

3.6. Contamination of boron from reagents

The contamination of boron from various reagents (EHD, $CHCl_3$, HCl, H_2O_2, DMF, CH_3COOH, H_2SO_4 and high purity water) employed in the methodology could vary from batch to batch. Since same amounts of the reagents were taken from same batch for blank as well as for the sample, and the absorbance of the sample is measured against the blank, it is not necessary to determine boron contamination from these reagents separately as it would not affect the results.

3.7. Estimation of uncertainty

The concentration of boron in sample was determined as

$$\mathrm{B}\ (\mu\,g\,g^{-1}) = \frac{A}{S \cdot w \cdot 1000}, \tag{3}$$

where A = absorbance; S = slope of calibration plot (0.00131); w = weight of the sample in gram. Here 1000 is used to convert $ng\,g^{-1}$ to $\mu g\,g^{-1}$.

Since standard boric acid aliquots were used to construct the calibration plot, the uncertainty associated with preparation of standard boric acid has to be considered to evaluate the combined uncertainty in the methodology. The combined uncertainty in measurement is categorized into six parts. They are uncertainty in standard preparation (u_{std}), sample preparation (u_{samp}), absorbance measurement (u_{abs}), slope of calibration plot (u_{cal}), repeatability of measurements (u_{rep}) and recovery of measurement (u_{rec}). Since Eq. (2) involves only multiplication and division of independent quantities, the combined standard uncertainty u_c can be calculated according to the equation

Table 2
Validation of the method

Sample name	Present method[a] Concentration of boron ($\mu g\ g^{-1}$)	ICP-AES[b] Concentration of boron ($\mu g\ g^{-1}$)
U–Al–Si-04	0.39 ± 0.01	0.40 ± 0.10
U–Al–Si-05	1.05 ± 0.03	1.10 ± 0.20

[a] Mean of five determinations with standard deviation.
[b] Mean of five determinations with standard deviation.

$$u_c = C_m \times \sqrt{u_{\text{std}}^2 + u_{\text{sam}}^2 + u_{\text{abs}}^2 + u_{\text{cal}}^2 + u_{\text{rep}}^2 + u_{\text{rec}}^2}, \tag{4}$$

where C_m is concentration of measurand, provided each individual uncertainty component is expressed as relative standard deviation. In the present studies, an attempt has made to address the uncertainties arising due to all the components mentioned above. The sources of uncertainty evaluated are given below.

3.7.1. Uncertainty in standard boric acid preparation

3.7.1.1. Purity of ortho boric acid. In the suppliers certificate it is given as 99.5 ± 0.5% pure. The quoted uncertainty is taken as rectangular (uniform) distribution, so the relative standard uncertainty u_1 is equal to $\frac{0.5\times100}{\sqrt{3}\times99.5} = 0.290\%$.

3.7.1.2. Mass determination. As quoted by the manufacturer of the balance, the uncertainty in mass determination is 0.2 mg over the entire range. Since 538.8 mg of the ortho boric acid is taken for preparation of standard boric acid solution, the relative uncertainty involved in weighing the standard, u_2 is $\frac{0.2\times100}{538.8} = 0.037\%$.

3.7.1.3. Molecular weight. The uncertainty in molecular weight of boric acid is arrived at considering the individual element atomic masses and their quoted uncertainties as given by IUPAC [28]. Rectangular distribution has been applied to quoted uncertainties. The molecular weight of boric acid (61.83302) is calculated using atomic weights of respective elements. The combined uncertainty in the molecular weight of boric acid is calculated employing uncertainty propagation formula and it is 0.004051. The relative combined uncertainty in molecular weight of boric acid, u_3 is $\frac{0.0040516\times100}{61.83302} = 0.007\%$.

3.7.1.4. Atomic weight of boron. Since standard solutions are prepared as μg of boron per gram of solution, the uncertainty in atomic weight of boron has to be considered. The quoted uncertainty in atomic weight of boron by IUPAC [28] is 0.007 and after applying rectangular distribution it becomes to 0.00404. Therefore the relative uncertainty in atomic weight of boron, $u_{4,}$ is $\frac{0.00404\times100}{10.811} = 0.037\%$.

3.7.1.5. Dilution. The working standard, employed in all experiments, is prepared by diluting the stock solution appropriately (through two intermediate dilutions) on weight basis. By considering the uncertainty in mass determination by balance as 0.0002 g, the relative combined uncertainty due to dilution, $u_{5,}$ is 0.009%.

The relative combined uncertainty in standard preparation is 0.295%.

3.7.2. Uncertainty in sample preparation

3.7.2.1. Mass of the sample. Approximately 200 mg of solid sample aliquot is taken for each analysis. The relative uncertainty in measurement of sample mass, u_6, is $\frac{0.2\times100}{200} = 0.100\%$.

3.7.2.2. Final dilution of sample. The uncertainty stated by the manufacturer for 10 mL quartz volumetric flask is 0.1 mL at 95% confidence level. Hence the uncertainty at 1σ level, u_7, is 0.500%.

The relative combined uncertainty in sample preparation is 0.510%.

3.7.3. Uncertainty in absorbance measurement

3.7.3.1. Blank absorbance. To determine the standard uncertainty in blank absorbance, 10 independent blank solutions were prepared and absorbance was measured with respect to water. The mean value of blank absorbance is found to be 0.24658 with standard deviation (σ_{blank}) of 0.00526. This uncertainty will take care of minor variations (uncertainties) arising due to variation of mass addition of various reagents like HCl, H_2O_2, H_2SO_4, curcumin, DMF and small variations in absorbance due to drift in wavelength.

3.7.3.2. Absorbance of sample. To arrive at this uncertainty, absorbance of a particular sample with respect to water solution was measured 10 times and found a mean absorbance of 0.62154 with standard deviation (σ_{sam}) of 0.00035.

Therefore, standard uncertainty in measurement of absorbance is $\sqrt{\sigma_{\text{blank}}^2 + \sigma_{\text{sample}}^2} = 0.005$. Hence the relative standard uncertainty in measurement of absorbance of sample is $\frac{0.005\times100}{0.62154-0.24658} = 1.406\%$.

The uncertainty arising in the absorbance measurement due to the interference of other elements is neglected since boron is selectively extracted [21] from solution.

3.7.4. Uncertainty in the slope of calibration plot

Calibration plot was plotted using Origin 6.0 software developed by Origin Lab Massachusetts, USA. This program was used to determine the slope of calibration plot. The slope of calibration plot is 0.00131 with standard error of 0.00001086. Hence u_{10}, relative uncertainty in slope of calibration plot is $\frac{0.00001086\times100}{0.00131} = 0.824\%$.

3.7.5. Recovery of B in measurement

Since no matrix-matched certified reference material for boron is available to evaluate the uncertainty in the recovery of measurement, boron was determined in 10 independent aliquots of standard boric acid solution. The mean of recovery of 98% was observed. Hence the standard relative uncertainty of recovery, u_{11} = 2.0%.

3.7.6. Repeatability of the measurement

Boron was determined in six independent solid sample aliquots taken from same lot and found that mean of boron concentration is 1.13 with 3.540% as relative standard deviation at 1σ level. (Table 3). The standard relative uncertainty for repeatability is therefore given as u_{12} = 3.540%.

3.7.7. Calculation of expanded uncertainty

Numerical values of variables, uncertainties with their classifications and their conversion to the relative standard uncertainties are summarized in Table 4. Combined uncertainty (relative) is calculated employing error propagation formula and it is found to be

Table 3
Determination of boron in U–Al–Si alloy samples (replicates n = 6)

S. no.	Amount of sample (g)	Amount of boron measured (ng)	Concentration of boron ($\mu g\ g^{-1}$)
1	0.1863	220.3	1.18
2	0.1889	208.8	1.11
3	0.2185	257.2	1.18
4	0.1974	220.2	1.12
5	0.2194	244.1	1.11
6	0.2437	267.2	1.10

Table 4
Uncertainty components of the boron determination in U–Al–Si alloy sample with the relative standard deviations

S. no.	Source of uncertainty	Type of uncertainty	Value of variable	Standard uncertainty (1σ)	% Relative standard uncertainty (1σ)	% Each category uncertainty (1σ)
Preparation of standard (u_{std})						0.295
u_1	Purity of Standard Boric acid	B	99.5	0.28868	0.290	
u_2	Weight determination of standard (mg)	B	538.8	0.2	0.037	
u_3	Molecular weight of boric acid	B	61.83302	0.0040516	0.007	
u_4	Atomic weight of boron	B	10.811	0.00404	0.037	
u_5	Dilution of stock solution to working standard solution	B	1	0.0000937	0.009	
Preparation of sample (u_{sam})						0.510
u_6	Weight determination of sample (mg)	B	200	0.2	0.100	
u_7	Volume of final solution	B	10	0.05	0.500	
Measurement of absorbance (u_{abs})						1.406
u_8	Variation in blank absorbance	A	0.24568	0.00526	–	
u_9	Absorbance of sample	A	0.37496	0.00035	–	
Slope of calibration plot (u_{cal})						0.824
u_{10}	Slope of calibration pot	B	0.00131	0.0000108	0.824	
Recovery (u_{rec})						2.000
u_{11}	Recovery	A	100	2.000	2.000	
Repeatability (u_{rep})						3.540
u_{12}	Repeatability	A	1.13	0.04	3.540	
Total relative combined uncertainty (1σ level)						4.420

4.420%. The expanded uncertainty at 95% confidence level is 8.840% (coverage factor 2). The boron concentration for a typical sample was 1.13 μg g^{-1} and the associated expanded uncertainty amounted to 0.10 μg g^{-1} at 95% confidence level. The main sources of uncertainty in the measurement are repeatability, recovery and absorbance measurement.

4. Conclusion

Boron concentration has been determined in U–Ai–Si alloy samples without using hydrofluoric acid and nitric acid. Boron content has been determined at levels less than 0.5 μg g^{-1}, in 200 mg sample (quantification limit of the method is 0.2 μg g^{-1}). Relative standard deviation of the method at 1σ level is 3.0%. The expanded uncertainty in measurement at 95% confidence level is 8.840%. The estimates of the expanded uncertainty give confidence and thus demonstrate the fitness of the methodology developed. The present method could be employed for the determination of boron in silicon related nuclear reactor materials like silicon–aluminum–nickel alloy (SILUMIN) and uranium–silicon alloy.

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