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ANALYTICAL METHODS FOR THE DETERMINATION OF BORON IN  
REACTOR MATERIALS PROGRAMME

by

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Descriptors

BORON

QUANTITATIVE CHEMICAL ANALYSIS

SPECTROPHOTOMETRY

BORON COMPLEXES

CHEMICAL PREPARATION

CURCUMIN

CALIBRATION STANDARDS

ORGANIC BORON COMPOUNDS

DISTILLATION

HEAVY WATER

LEAVES

IRON

STEELS

COPPER ALLOYS

ALUMINIUM

ALUMINIUM ALLOYS

URANIUM

URANIUM DIOXIDE

THORIUM NITRATES

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INTRODUCTION

Boron, because of its high absorption cross-section for thermal neutrons, should essentially be absent in most of the materials used for nuclear applications. Strict purity control for this element in such materials is therefore necessary. This has led to the development of a large number of analytical methods for the determination of boron at ppm levels <sup>(1)</sup>.

The chemical methods described in literature are of two types; those which involve the separation of boron prior to its determination and those where the determination can be done directly.

1.1 1. Separation of boron:

This can be achieved by one of the following methods.

- a) separation by distillation as methyl borate ester <sup>(2)</sup>
- b) separation by extraction of tetrafluoroborates of tetraphenyl-arsonium <sup>(3)</sup>, methylene blue <sup>(4)</sup>.
- c) chromatographic separation using cation and anion exchangers <sup>(5, 6, 7)</sup>.
- d) separation by pyrolysis <sup>(8)</sup>.

Among these, the separation by distillation of boron as methyl borate is widely used.

### 1.2 Determination of boron:

Photometric methods are applied for the determination of microamounts of boron. Since boron has no chromophoric properties, only coloured reagents can be used for its photometric determination. In the last 20 years, a large number of highly sensitive and selective organic reagents were suggested for the determination of boron by photometric methods<sup>(9)</sup>. Among these, curcumin, which forms a red dye with boric acid, in acid medium is the most sensitive reagent for boron. Two boron-curcumin complexes are reported in literature.

a) Rosocyanin, the red boron-curcumin complex, of molar ratio 1:2 is formed in the absence of oxalic acid with excess amount of curcumin. The molar extinction coefficient (for 1 g atom of boron) is about 180,000 at the wavelength 555 nm. The sensitivity of the system is  $0.000051 \mu\text{g}/\text{cm}^2$  per 0.001 optical density. This is higher than that of any other organic reagent for boron.

b) Rubrocurcumin: This complex is formed in the presence of oxalic acid which considerably increases the rate of formation of the boron-curcumin complex. Unlike rosocyanin, this complex contains one molecule of curcumin and one molecule of oxalic acid per boron<sup>(10)</sup>. The molar extinction coefficient is almost half that of rosocyanin. A similar enhancement in the

rate of formation of the complex on addition of trichloro acetic acid is reported<sup>(11)</sup>.

The reaction between boric acid and curcumin occurs at a measurable rate only when the curcumin molecule is protonated in the presence of a strong acid. However, the curcumin in the protonated form is unstable and is rapidly decomposed by heating. The presence of water inhibits proton addition and thereby the formation of the complex. The protonated curcumin absorbs at the same wavelength as that of boron complex. This necessitates the deprotonation of the excess curcumin which is achieved by reducing the acidity of the complex after the protonation causing the excess protonated curcumine to revert back to the normal form. Based on these principles, Heys and Metcalfe<sup>(12)</sup> have developed a procedure, wherein the boron-curcumin complex is initially formed in a non-aqueous media of acetic acid and sulphuric acid. The boron-curcumin complex is readily produced in the boron-curcumin ratio of 1:3. The deprotonation of the excess curcumin is done by dilution with ethanol. This method obviates many precautions necessary to ensure formation of the coloured complex and hence it is widely applied, when boron is present in very small quantities. Modifications of the method of Hayes and Metcalfe have also been suggested.

The original method of Hayes and Metcalfe and the modifications are given in detail in Section I.

In Section II are given in detail, the methods followed in this laboratory for the determination of boron in different matrices. Part A of Section II deals with direct determination and Part B with those which involve separation of boron as methyl borate prior to its determination.

## SECTION I

### 2. Hayes and Metcalfe method:

2.1 Principle: To an aqueous solution of boron restricted to 0.25 ml water, or to the mass dried with sodium hydroxide, curcumin in acetic acid is added. On acidification with sulphuric acid-acetic acid reagent, the curcumin is protonated which then reacts with boric acid. Unreacted curcumin is then deprotonated by dilution with ethyl alcohol.

2.2 Apparatus: Platinum dishes or small polythene beakers, polythene rod. The use of borosilicate glass shall be avoided.

2.3 Reagents: 1. Ethanol: Industrial grade, distilled over sodium hydroxide or potassium hydroxide.

2. Curcumin: (0.125%) in glacial acetic acid. Dissolve 0.125 g curcumin in 100 ml of glacial acetic acid with stirring, warm if necessary. Store in a plastic container.

3. Sulphuric acid-acetic acid reagent: Add 50 ml of sulphuric acid (Sp. gr. 1.84) to 50 ml previously cooled glacial acetic acid.

4. Sodium hydroxide solution (10% W/V): Dissolve 10 g sodium hydroxide pellets in 100 ml of distilled water in a boron-free glass beaker and store it in a plastic container.

5. Standard boron solution: (1 µg/ml): Dissolve 0.1430 g boric acid in distilled water and make upto 250 ml (i.e. 100 µg/ml). Dilute 10 ml of this solution to 1 litre with water (1 µg/ml).

#### 2.4 Calibration:

Transfer 0, 0.5, 1.0, 1.5 and 2.0 ml of boron standard (1 µg/ml) to platinum dishes containing 1.0 ml of sodium hydroxide. Evaporate gently to dryness on a water bath. Add 3 ml of curcumin reagent, warm the dish to dissolve the residue. Cool to room temperature and add 3.0 ml of sulphuric acid-acetic acid mixture. Stir the solution well with polythene rod and keep for 15 minutes. Dilute the mixture to 100 ml with ethanol. Filter and measure the absorbance of the filtrate at 555 nm (Table I).

#### 2.5 Method of Harrison and Cobb (13)

In this method, the filtration step as in Hayes and Metcalfe method is eliminated by diluting the boron-curcumin complex with acetone and water mixture (1:1). The final made up volume is 100 ml. The absorbances are shown in Table II.

#### 2.6 Grinstead and Snider Method (14)

The absorbances are further increased by using ammonium acetate-acetic acid buffer (15 ml) as diluent in place of ethanol. The volume of curcumin and sulphuric acid-acetic acid



reagent is maintained at 3.0 ml and the final volume at 22 ml. The absorbance values are given in Table III.

### 2.7 Method of Pakalns<sup>15</sup>:

In the method for direct determination of boron in various metals, developed by Hayes and Metcalfe, the following conditions are necessary.

- (a) The water content should be less than 0.25 ml.
- (b) Nitrate should be absent in the sample.

The above conditions have been eliminated by 'Pakalns'. In this procedure, the sample is dissolved in mixture of sulphuric acid and nitric acid. Hydrochloric acid is added if tin is present and nitrate is reduced to nitrite with formic acid. An aliquot of the solution (upto 2 ml containing 40 mg sample) is treated with acetic anhydride, curcumin followed by an acetone-water mixture and the absorbance of the boron-complex is measured (Table IV). The method is described in detail under 3.4.

### 2.8 Modification of the method of Hayes and Metcalfe:

The method of Hayes and Metcalfe is modified in the laboratory to increase the sensitivity.

Transfer aliquots containing 0-2  $\mu$ g of boron to platinum dishes containing 0.5 ml of sodium hydroxide (10%), evaporate to dryness on the water bath, add 3 ml of curcumin (0.125% in glacial acetic acid) and warm to dissolve the residue.

Add 1.5 ml of sulphuric-acetic acid mixture and keep for 15 mts. Dilute the mixture to 50 ml with ethanol. Filter and measure the absorbance of the filtrate at 555 nm (Table V).

In the following section, the determination of boron in different matrices are described. The precision obtained is also indicated.

## SECTION II - PART A

### 3.1 HEAVY WATER

Take a suitable aliquot of the sample so as to contain approx. 1.0  $\mu$ g boron; add 0.5 ml sodium hydroxide (10%), evaporate to dryness and follow the procedure as given in 2.8.

At 0.3 ppm boron in heavy water, the coefficient of variation is 6%.

### 3.2 Plant leaves (Orchard leaves):

Take the sample in a platinum dish containing 5 ml of carbonate fusion mixture solution (3%). Evaporate to dryness on water bath. Char on wire gauze and ignite over Mecker burner to remove the organic matter.

Add 3 ml curcumin reagent and 1.5 ml of sulphuric acid-acetic acid mixture and follow the procedure as given in 2.8.

At 33 ppm boron level the coefficient of variation is 5%. (As per the certification of NBS, this standard 1571, is reported to contain Boron  $33 \pm 3$  ppm).

### 3.3 Iron and steels:

A method developed by Tolk, Tap and Linerak<sup>(16)</sup> is used for the direct determination of boron in the range of 20-200 ppm in iron and low alloy steels. The absorbance of the boron-curcumin complex formed in the sample solution is compared directly with that of another aliquot of the same sample in which boron is made inactive before curcumin is added using sodium fluoride solution. The acid required to develop the coloured complex is partially neutralised with ammonium acetate and partially by dilution.

#### 3.3.1 Reagents:

1. Acetate buffer: Dissolve 225 g ammonium acetate in 300 ml glacial acetic acid and make up to 1000 ml volume.

2. Fluoride reagent (4% W/V): Dissolve 4.0 g sodium fluoride A.R. in 100 ml distilled water.

3.3.2 Procedure: Dissolve 0.25 g of sample in 10 ml of sulphuric acid (1:3) by gentle heating in a long necked quartz vessel. If dissolution is incomplete, filter off the residue, wash with water and transfer the filter pad to a platinum crucible; add 1.0 ml of 30% sodium carbonate solution. Dry, ignite very carefully at low temperature and fuse for 15 minutes. Cool the fused mass, dissolve it in the filtrate from the initial dissolution. Transfer the solution to 25.0 ml standard flask and dilute to mark with water.

Pipette out two 0.25 ml portions of the solution into two 50 ml polythene bottles. To one, add 3-4 drops of fluoride reagent. Mix by swirling and allow to stand for 1 hour at room temperature. Add 3 ml sulphuric acid-acetic acid mixture to both portions, wait at least for 5 minutes. Add 3.0 ml of curcumin to both and keep at 25°C for 2 hrs. in a thermostat. Add 15 ml of acetate buffer, keep the bottles in thermostat for 10 minutes. Read the absorbance at 555 nm. The absorbance of the solution without fluoride reagent measured against the one with the fluoride reagent is the absorbance of the boron complex. At 25 ppm of boron in stainless steel, the coefficient of variation is 6%.

3.4 Copper and copper base alloys: The method suggested by Pakalns is followed for the determination of boron.

3.4.1 Apparatus:

1. Boron-free glassware.
2. All silica round bottomed flask, 250 ml capacity with silica air condenser (height 12 cm.).

3.4.2 Reagents:

1. Curcumin: Dissolve 0.125 g of curcumin in 100 ml of acetic acid (glacial).
2. Acid mixture: Add 50 ml of sulphuric acid (Sp. Gr. 1.84) to 50 ml of previously cooled glacial acetic acid with stirring and cooling.

3. Dilute sulphuric acids: Prepare 20%, 25% and 50% acids (v/v).

4. Acetone-water solvent (1:1): Mix equal volumes of distilled water and acetone A.R.

5. Acetic anhydride AnalaR grade.

6. Nitric acid and hydrochloric acid AnalaR grade.

7. Formic acid (98% - 100%) AnalaR grade.

3.4.3 Dissolution of sample:

3.4.3.1 Copper: Transfer 1.5 g sample to a 250 ml silica flask. Add 15 ml water, 6 ml sulphuric acid (50%) and 5.5 ml of nitric acid. Insert the air condenser, dissolve the sample by gentle heating in an isomantle. Cool, add 34 ml of sulphuric acid (50%) from a burette and 3 ml of formic acid. Heat gently to initiate the reduction. After the reaction has subsided, add 2 ml of formic acid followed by an additional 2 ml of formic acid after 1 minute. Remove the air condenser, boil the solution for 1 minute to expel the last traces of nitrate. Cool, transfer to a 100 ml standard flask, and make up with water.

3.4.3.2 Brass and bronze: Transfer 2.0 g of sample to 250 ml silica flask. Add 40 ml of sulphuric acid (20%), 10 ml of hydrochloric acid and 7 ml of nitric acid. Continue as per procedure 3.4.3.1 except for the addition of 20 ml sulphuric acid (50%) after the dissolution of the sample.

3.4.4 Spectrophotometric procedure:

Transfer 2 ml aliquot of the sample solution to a 100 ml dry, boron-free conical flask or polythene beaker. Add 10 ml glacial acetic acid, cool below 20°C. Add 5 ml acetic anhydride and wait until the reaction takes place (flask or beaker becomes warm). Add further 3.5 ml of acetic anhydride, cool to room temperature. Add 4 ml of acid mixture, mix and add 3 ml of curcumin reagent. Mix the contents and leave for 1 hour. Add acetone-water solvent. Shake to dissolve the salts, transfer to 100 ml volumetric flask, dilute to the mark with the acetone-water mixture. Measure the absorbance at 555nm. Compare with corresponding standards a or b.

3.4.5 Calibration of standards:

Dissolve 0.1429 g of boric acid in water, make upto 250 ml with water (1 ml = 100  $\mu$ g). Dilute 50 ml of the above solution to 250 ml with sulphuric acid (25% V/V) (1 ml = 20  $\mu$ g B).

Prepare two sets of standards a and b. One in 20% sulphuric acid and other in 18% sulphuric acid and 10% hydrochloric acid. 2 ml equivalents containing different concentrations as 2 ml = 0.8  $\mu$ g B, 2 ml = 2.0  $\mu$ g B, 2 ml = 4.0  $\mu$ g B and 2 ml = 6.0  $\mu$ g B, etc. are prepared.

Transfer 2 ml aliquots of standards to polythene beakers and continue as per procedure 3.4.4. The absorbances are

shown in Table IV. The coefficient of variation is 8% at 40 ppm of B in copper alloys.

### 3.5 Aluminium metal and aluminium alloys:

Pakalns<sup>(17)</sup> has established a method for direct determination of boron in aluminium alloys, similar to that of copper and copper alloys.

#### 3.5.1 Reagents and apparatus:

Same as given in 3.4.2 and 3.4.1.

#### 3.5.2 Dissolution of sample:

Transfer 1 g of sample to a 250 ml quartz flask, add 20 ml of sulphuric acid (20%), 5 ml conc. nitric acid and 2 ml conc. hydrochloric acid. Insert the air condenser, heat the sample in an electric mantle. Do not allow the reaction to take place rapidly. Add another 8 ml of conc. hydrochloric acid and 28 ml of sulphuric acid (50%). Heat the sample gently until complete dissolution. Cool, add 3 ml of formic acid and heat to initiate the reduction. Add 2 ml of formic acid followed by additional 2 ml of it after 1 minute.

Remove the condenser and boil the solution for 1-2 minutes to expel the last traces of nitric oxide. Cool, filter the solution through 7 cm low ash coarse paper (Whatman 541) with small amount of paper pulp into a 100 ml. Standard flask, wash the paper with hot water. Cover the residue with 1 g sodium carbonate and transfer to a platinum crucible. Dry and char the paper at a low heat. Heat until clear melt is obtained. Cool to

room temperature, dissolve the melt in part of the filtrate collected in the 100 ml flask. Transfer the solution to the 100 ml standard flask, cool and dilute to the mark.

3.5.3 Spectrophotometric procedure: Transfer 2 ml aliquot of the sample solution, filter if necessary through a fast paper to dry polythene beaker (250 ml capacity), add 10 ml glacial acetic acid and follow the procedure given in 3.4.4.

The procedure gave a coefficient of variation of 7% in the determination at 44 ppm of B.

## PART B

### SEPARATION OF BORON BY DISTILLATION AS METHYL BORATE

At very low content of boron, when it cannot be determined directly, as in case of uranium metal, uranium oxide and thorium nitrate, separation of boron becomes necessary.

On distillation with methanol from acid media the borate forms an ester with boiling point  $65^{\circ}\text{C}$ . The distilled ester is absorbed by hydroxide solution and after removal of the methanol, boron is determined.

#### 4.1 Apparatus:

As shown in Figure 1 (1) All silica flask 250 ml capacity. (2) Condenser (3) Receiving flask.

#### 4.2 Reagents:

1. Methanol: Industrial grade methanol kept overnight with calcium oxide and distilled over solid sodium hydroxide or



potassium hydroxide.

2. Double-distilled methanol: The methanol obtained in (1) is again distilled over alkali with few drops of glycerol, in a complete quartz apparatus, at  $64^{\circ}\text{C} - 65^{\circ}\text{C}$ .

3. Purified acid: The acid used in this procedure is purified by removal of boron as methyl borate by repeated addition of double distilled methanol and heating on a water bath.

4. Sodium hydroxide 10% (W/V): 10 g of sodium hydroxide pellets are dissolved in 100 ml distilled water and stored in a polythene bottle.

5. Distilled water: Distilled water is collected directly from the water still through the metal outlet in a polythene container.

#### 4.3 Procedure:

A weighed quantity of material (5 g) to be analysed is taken in the flask and suitable acid is added to dissolve it, through the thistle-cup arrangement. The flask is heated in an isomantle, if necessary. When the solution is complete, about 120 ml double distilled methanol is added and the flask is heated. The temperature is maintained at  $64^{\circ}\text{C} - 65^{\circ}\text{C}$ . The methyl borate vapours are condensed and collected in a flask containing 2 ml sodium hydroxide (10%); a drop of glycerol and phenol-phthalein indicator (2 drops). About 100 ml distillate is collected.

The distillate is evaporated on water bath to a small volume and transferred to a platinum dish and evaporated to

dryness. Further procedure followed is as given in 2.8.

In the above procedure, excess alkali in the distillate is kept, which helps in the formation of stable form of sodium borate. In its absence the formation of unstable metaborate will take place<sup>(18)</sup>.

In the case of the following samples, preliminary separation of boron by distillation is carried out before the determination of boron.

4.4.1 Uranium metal: The sample is washed with ether and acetone. The surface cleaning is done by pickling the sample with nitric acid (1:1) followed by washing and drying.

Take a suitable quantity of the sample so as to contain 1.0  $\mu$ g B, dissolve in hydrochloric acid and add few drops of hydrogen peroxide to get a clear solution. Add about 120 ml double distilled methanol and distill.

4.4.2 Di-uranates: Transfer a quantity of the sample equivalent to 1.0  $\mu$ g boron to the flask. Add about 5 ml of hydrochloric acid; add about 120 ml double distilled methanol and distill.

4.4.3 Uranium oxide ( $U_3O_8$ ): Transfer about 5.0 g of the sample weighed correctly to the nearest 10 mg, to the distillation flask. Add hydrochloric acid till the sample dissolves (about 10 ml). Add hydrogen peroxide till the solution becomes clear. Add about 120 ml double distilled methanol and distill.

4.4.4 Thorium nitrate: Take about 5.0 g of the sample weighed exactly to 10 mg in a distillation flask. Dissolve the sample by adding the hydrochloric acid and heating if necessary. Add about 120 ml of double distilled methanol and distill.

The coefficient of variation is 15% at 1 ppm of B for samples mentioned under 4.4.

5. Boron losses and contamination:

In all the methods described, due care should be taken in preventing losses of boron due to volatilisation and contamination from the laboratory atmosphere. For the preparation and preservation of solutions, platinum, polyethylene or low boron glass (Corning U.S. 7280) should only be used. Reagents as water, hydrochloric acid, nitric acid should be redistilled from a quartz distillation unit.

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All the absorbances given in the following tables are measured against blank.

TABLE I  
HAYES AND METCALFE METHOD

Wavelength - 555 nm		Volume - 100 ml (ethanol)
Boron standard ( $\mu\text{g}$ )	Absorbance	
0.5	0.080	
1.0	0.158	
1.5	0.238	
2.0	0.325	

TABLE II  
HARRISON AND COBB METHOD

Wavelength - 555 nm		Volume - 100 ml (Acetone + water) 1+ 1
Boron standard ( $\mu\text{g}$ )	Absorbance	
0.5	0.082	
1.0	0.170	
1.5	0.255	
2.0	0.330	

TABLE III  
GRINSTEAD AND SNIDER METHOD

Wavelength - 555 nm                      Volume - 22 ml (Acetate buffer)

Boron standard ( $\mu\text{g}$ )	Absorbance
0.5	0.305
1.0	0.615
1.5	0.880

TABLE IV  
PAKALNS METHOD

Wavelength - 555 nm                      Volume - 100 ml (Acetone + water) 1 + 1

Boron standard ( $\mu\text{g}$ )	Absorbance
0.8	0.120
2.0	0.298
4.0	0.579
6.0	0.880

TABLE V  
HAYES AND METCALFE METHOD (MODIFIED)

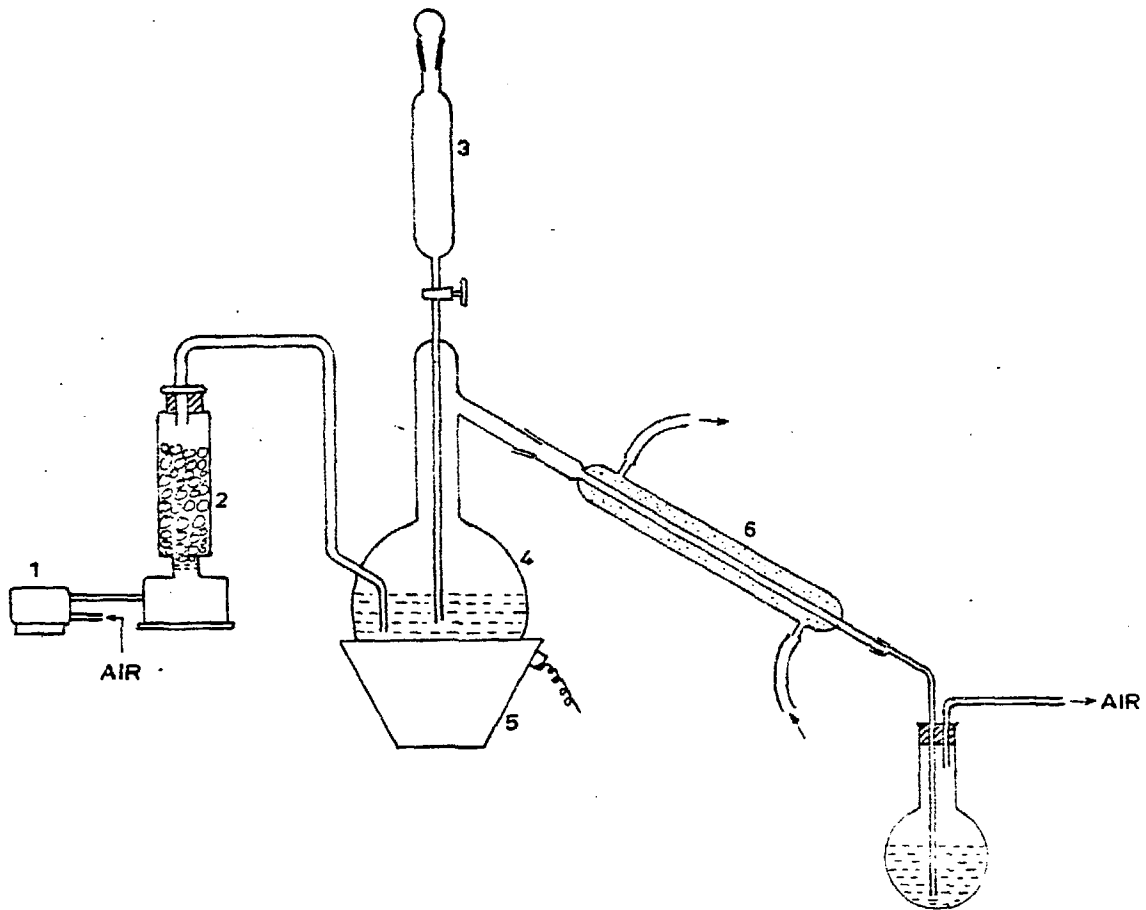
Wavelength - 555 nm                      Volume - 50 ml (ethanol)

0.5	0.156
1.0	0.307
1.5	0.458
2.0	0.640

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- |             |             |                  |                  |
|-------------|-------------|------------------|------------------|
| 1 AIR PUMP  | 2 CaO TOWER | 3 THISTLE FUNNEL | 4 REACTION FLASK |
| 5 ISCMANTLE | 6 CONDENSER | 7 RECEIVER FLASK |                  |

FIG.1 AN ALL QUARTZ DISTILLATION SYSTEM FOR BORON