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BASES OF THE CRYSTALLOPHYSICAL PURIFICATION OF INDIUM TRIIODIDE--ETC(U)

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FOREIGN TECHNOLOGY DIVISION



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OF INDIUM TRIIODIDE

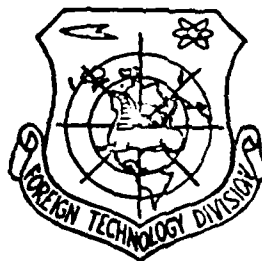
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Inorganic Chemistry

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| Block | Italic | Transliteration | Block | Italic | Transliteration |
|-------|------------|-----------------|-------|------------|-----------------|
| А а | <i>А а</i> | A, a | Р р | <i>Р р</i> | R, r |
| Б б | <i>Б б</i> | B, b | С с | <i>С с</i> | S, s |
| В в | <i>В в</i> | V, v | Т т | <i>Т т</i> | T, t |
| Г г | <i>Г г</i> | G, g | У у | <i>У у</i> | U, u |
| Д д | <i>Д д</i> | D, d | Ф ф | <i>Ф ф</i> | F, f |
| Е е | <i>Е е</i> | Ye, ye; E, e* | Х х | <i>Х х</i> | Kh, kh |
| Ж ж | <i>Ж ж</i> | Zh, zh | Ц ц | <i>Ц ц</i> | Ts, ts |
| З з | <i>З з</i> | Z, z | Ч ч | <i>Ч ч</i> | Ch, ch |
| И и | <i>И и</i> | I, i | Ш ш | <i>Ш ш</i> | Sh, sh |
| Й й | <i>Й й</i> | Y, y | Щ щ | <i>Щ щ</i> | Shch, shch |
| К к | <i>К к</i> | K, k | Ъ ъ | <i>Ъ ъ</i> | " |
| Л л | <i>Л л</i> | L, l | Ы ы | <i>Ы ы</i> | Y, y |
| М м | <i>М м</i> | M, m | Ь ь | <i>Ь ь</i> | ' |
| Н н | <i>Н н</i> | N, n | Э э | <i>Э э</i> | E, e |
| О о | <i>О о</i> | O, o | Ю ю | <i>Ю ю</i> | Yu, yu |
| П п | <i>П п</i> | P, p | Я я | <i>Я я</i> | Ya, ya |

*ye initially, after vowels, and after ь, ъ; e elsewhere.
When written as ë in Russian, transliterate as yë or ë.

RUSSIAN AND ENGLISH TRIGONOMETRIC FUNCTIONS

| Russian | English | Russian | English | Russian | English |
|---------|---------|---------|---------|----------|--------------------|
| sin | sin | sh | sinh | arc sh | sinh ⁻¹ |
| cos | cos | ch | cosh | arc ch | cosh ⁻¹ |
| tg | tan | th | tanh | arc th | tanh ⁻¹ |
| ctg | cot | cth | coth | arc cth | coth ⁻¹ |
| sec | sec | sch | sech | arc sch | sech ⁻¹ |
| cosec | csc | csch | csch | arc csch | csch ⁻¹ |

Russian English

rot curl
lg log

BASES OF THE CRYSTALLOPHYSICAL PURIFICATION OF
INDIUM TRIIODIDE

Speciality No. 02.070

Inorganic Chemistry

Author's abstract of a dissertation for competition of
the university degree of the Candidate of Chemical Sciences

Introduction

The contemporary development of science and technology has caused the intense application in industry of dispersed elements, which earlier served only as the object of laboratory investigations. Indium occupies a prominent place among them.

For a long time indium was limited, and the industrial production of it was insignificant. In connection with the appearance of a new branch of industry, the industry of semiconductors with the use of compounds of indium as semiconductor materials, interest in it has considerably grown. At the same time, requirements for the purity of a metal have increased significantly. In the examination of the different methods of the purification of indium, it is possible to see that the most promising is, probably, the method of purification of indium by its halogen compounds, in particular, by indium triiodide.

In the latter stages of the purification of indium and its compounds, used most frequently are the crystallophysical methods, zonal melting, directional crystallization, columnar crystallization, and

others, since these methods, in their effectiveness, considerably exceed the processes of purification which use the equilibrium liquid-liquid or liquid-vapor.

In application to the deep purification of the indium triiodide, the crystallization methods provide a minimal intensity of the introduction of contaminants, owing to the high effective concentration of the object of purification and, consequently, the minimal surface of contact with walls of the equipment, and also due to the fact that the substance is in an inert solid state.

The purpose of this work was to investigate the procedures and physicochemical basis for crystal purification of indium triiodide, which is required both for the obtaining of metal of a high degree of purity, and for the synthesis of a number of semiconductor compounds and the alloying of epitaxial layers of germanium and silicon. The dissertation gives results of the investigation of the phase-equilibrium diagrams of dual systems: indium triiodide and iodide of a doped component. It describes the apparatus used for purifying the indium triiodide by the method of directed crystallization and gives results of the experiments according to the behavior of certain trace contaminants with crystallization purification of the indium triiodide.

Literature review

In the first part of the literature review there are data which deal with the basic physicochemical properties of the indium triiodide revealed in the indium-iodide system. Contained in the second part of the review is detailed information about the liquid-crystal equilibrium in systems indium triiodide with iodides of other elements.

Examined in the last section are methods of the calculation and experimental determination of the distribution coefficients.

Experimental Part

I. Synthesis of parent substances

To study the interaction in systems of indium triiodide and iodide of the doped component, from elements in a vacuum we synthesized the diiodides of iron, cobalt, nickel, manganese, cadmium,

indium triiodides, arsenic, silicon tetraiodides, germanium, tin, and tellurium. In the obtaining of iodides of bivalent metals in excess (~20% of the stoichiometry), a metal is introduced, and in the remaining cases, iodine. Synthesis was conducted in evacuated quartz vials. The conditions of synthesis were selected based on the melting points of the final products. The obtained substances were additionally subjected to purification by means of distillation in a vacuum (with the exception of iodides of cobalt and tellurium). It was not possible to purify the cobalt diiodide by distillation, since at a temperature of 570°C its thermal decomposition was begun, and, therefore, they [?-parent substances] were freed from the excess in metal by liquation according to specific weight. In the obtaining of tellurium tetraiodide from an excess in iodine, they were freed by means of the washing of the latter by carbon tetrachloride.

II. Study of the interaction in systems $\text{InI}_3\text{-MeI}_x$ ($x = 2, 3, 4$)

Selected as the method of investigation by us was the method of differential-thermal analysis, which is the most effective in the study of diagrams of the condensed state and gives the most complete information about the interaction in the systems. The recording of curves of heating, and in certain cases cooling, were produced on pyrometers of the type FPK-59 and NTR-62m. The heating rate of the furnace was 5-8 deg/min. Used for the measurement of temperatures were Chromel-Copel and platinum and platinum-rhodium thermocouples, which were graduated according to the universally accepted reference points, using the appropriate reagents of the brand "KhCh." Suspensions of mixtures of components with a total weight of 2.0 ± 0.01 g were melted in sealed Stepanov vessels, carefully mixed by shaking, after which they were slowly cooled together with the furnace for an approach to an equilibrium state. In certain cases prolonged annealing was conducted at temperatures lower than temperatures of the phase conversions.

Furthermore, methods of X-ray and phase analysis and measurement of electrical conductivity have aroused interest.

1. Indium-triiodide - iron diiodide

This system belongs to the eutectic type with degenerated

eutectic from the side of the indium iodide. Observed at a temperature of 130°C is the formation of the compound of the composition $\text{InI}_3 \cdot 4\text{FeI}_2$ in the solid phase. The compound is stable, since after prolonged low-temperature annealing, observed on the thermograms are also the effects which correspond to the formation of the compound. The formation of the solid solution on the basis of iron diiodide is possible in the system.

2. Indium triiodide - cobalt diiodide

The system belongs to the eutectic type. The eutectic is melted at 180°C and corresponds to the content of ~ 6 mol.% of CoI_2 . Observed at 140°C is the formation of the compound in the solid phase of the composition $2\text{InI}_3 \cdot \text{CoI}_2$. The compound is metastable, since after the annealing of the alloys at 125°C for 200 hours, no effects corresponding to the formation of the compound were revealed on the thermograms. After the quenching of the specimens at 550°C and the repeated recording of the heating curves, the effects on the thermograms corresponding to the formation of the compound appeared again.

3. Indium triiodide - nickel diiodide

The system belongs to the eutectic type with degenerated eutectics, which is adjacent to the ordinate of the low-melting component. At 140°C the formation of the compound in the solid phase of the composition $2\text{InI}_3 \cdot \text{NiI}_2$ is also observed. This compound, the same as for the compound $2\text{InI}_3 \cdot \text{CoI}_2$, is metastable.

4. Indium triiodide - manganese diiodide

The system belongs to the eutectic type. The eutectic is melted at 204°C and corresponds to the content of ~ 5 mol.% of MnI_2 . Also observed in the system at a temperature of 128°C is the formation of the compound in the solid phase with a maximum value of the effect for the composition corresponding to the content of 75 mol.% of InI_3 , and, consequently, the compound will correspond to the formula $3\text{InI}_3 \cdot \text{MnI}_2$. This compound is stable. In the metastable state, with an insufficient duration of the annealing, revealed in this system are the incongruent melting compounds of compositions $2\text{InI}_3 \cdot 3\text{MnI}_2$ and $\text{InI}_3 \cdot 3\text{MnI}_2$ with decomposition temperatures of 220° and 335°C , respectively.

The formation of the compounds in the solid phase in systems $\text{InI}_3 \cdot \text{MeI}_2$ was subjected to X-ray studies.

5. Indium triiodide - arsenic triiodide

The system is of the eutectic type. The eutectic of the composition of ~ 65 mol.% of AsI_3 is melted at a temperature of 130°C .

6. Indium triiodide - silicon tetraiodide

The system belongs to the eutectic type. The eutectic has the composition of ~ 85 mol.% SiI_4 and a melting point of 116°C .

7. Indium triiodide - germanium tetraiodide

The system is of the eutectic type. The eutectic of the composition ^{of} ~ 82 mol.% GeI_4 has a melting point of 131°C .

8. Indium triiodide - tin tetraiodide

The system also belongs to the eutectic type. The eutectic corresponds to the composition of ~ 75 mol.% SnI_4 and has a melting point of 132°C .

In systems of indium triiodide with arsenic triiodide and tetraiodide of silicon, germanium and tin, the formation of small regions of solid solutions on the basis of InI_3 is possible. The plotting of lines of liquidus of indium triiodide for these systems showed the good agreement with the ideal curve calculated according to the Schroeder equation. Thus there is basis to consider that the liquid phases in these systems approach the ideal.

9. Indium triiodide - tellurium tetraiodide

Two incongruent melting compound of compositions $\text{InI}_3 \cdot 2\text{TeI}_3 \cdot 3\text{TeI}_4$ are revealed in the equilibrium system. The compound $\text{InI}_3 \cdot 2\text{TeI}_4$ with the indium triiodide forms the eutectic melting at 122°C and corresponding to the content of ~ 33 mol.% of TeI_4 . The formation of a small region of the solid solution on the basis of indium triiodide is possible in the system. Revealed in the metastable state in the system is the compound of a composition of I:I being congruently melted at 165°C . The individuality of the revealed compounds was confirmed by results of the X-ray phase analysis.

10. Indium triiodide - cadmium diiodide

This system has already been studied, but the composition of the compound and its melting point have caused a certain doubt. Therefore, this system is investigated by us again.

Revealed in the system is one compound of the composition $2\text{InI}_3 \cdot \text{CdI}_2$ being congruently melted at 195°C . The compound forms the eutectic with indium triiodide, which is melted at 175°C and corresponds to the content of 80 mol.% InI_3 , and with cadmium diiodide, which is melted at a temperature of 185°C and corresponds to the content of 45 mol.% CdI_2 .

For confirmation of the data of the thermal analysis, the measurement of the electrical conductivity of certain alloys of this system in a temperature range of $200\text{--}250^\circ\text{C}$ was conducted. The measurement of electrical conductivity was conducted in a cell of the capillary type in an atmosphere of argon by means of a bridge of alternating current of the type R-568. A study showed that on the isotherms of electrical conductivity there is a break which corresponds to the composition of the compound $2\text{InI}_3 \cdot \text{CdI}_2$, which is indicative of the complex formation in the melt. Probably, the forming complex is a complex anion InI_4 , and the compound itself is a tetraiodoindate of cadmium - $\text{Cd}[\text{InI}_4]_2$. The absence of a break on the line of the dependence of the logarithm of specific electrical conductivity on the inverse temperature indicates that the compound is stable in the melt.

For the system $\text{InI}_3\text{--CdI}_2$ the calculation of the liquidus line was conducted by taking the interaction of the components in the melt into account. The theoretical curve practically coincided with the experimental curve, which indicates the correctness of the selected model of the interaction and confirms the formation of the compound $\text{Cd}[\text{InI}_4]_2$. One more, indirect, confirmation in favor of the formation in this system of the compound $\text{Cd}[\text{InI}_4]_2$ is the comparison of the electrical conductivity of the indium triiodide in a temperature range of $210\text{--}300^\circ\text{C}$ with the electrical conductivity of the cadmium tetraiodoindate.

According to results of the measurement of the electrical conductivity, these values of the activation energy of conductivity

were computed: ^{for} $\text{InI}_3 - E_a = 2.9$ kcal/mole and for $\text{Cd}[\text{InI}_4]_2 - E_a = 5.8$ kcal/mole.

An increase in the value of the activation energy of the conductivity indicates the complex formation in the melt.

Values of the specific electrical conductivity for the compound $\text{Cd}[\text{InI}_4]_2$ and temperature range of $200^\circ - 250^\circ\text{C}$ can be computed from the formula

$$\lg \kappa = - \frac{1234}{T} + 1.17.$$

For the indium triiodide the electrical conductivity is satisfactorily described by the equation

$$\lg \kappa = - \frac{656.5}{T} + 0.175 \quad (T = 210-300^\circ\text{C}).$$

The formation of the compound in the system $\text{InI}_3 - \text{CdI}_2$ was confirmed by X-ray analysis.

Thus in all the investigated systems, the addition of the second component to the indium triiodide, with the exception of FeI_2 and NiI_2 , lowers the melting point of the latter, from which it follows that the distribution coefficients of the corresponding impurities in the InI_3 will be less than unity, and, consequently, the crystallization methods of purification will be effective to a sufficient degree. Preliminary calculations of thermodynamic distribution coefficients according to the Hays-Chapman equation confirmed this conclusion (Table 1).

Table 1 Values of K_0 computed from phase-equilibrium diagrams according to the Hays-Chapman equation

| Component-impurity | CdI_2 | AsI_3 | SiI_4 | GeI_4 | SnI_4 | TeI_4 |
|--------------------|----------------|----------------|----------------|----------------|----------------|----------------|
| K_0 | 0.085 | 0.05 | 0.13 | 0.11 | 0.32 | 0.10 |

III. Study of the behavior of certain trace contaminants in the purification of indium triiodide by the method of directed crystallization

Experiments on the crystallization purification of indium

triiodide were conducted on a specially constructed apparatus. The crystallization rate was 3.9, 2.8, 2.25, 1.7, and 1.2 cm/h. The temperature of the melt was held constant and consisted of 250°C. Furthermore, mixing of the melt was accomplished at a rate of 5 [?-illegible] r/min.

The indium triiodide, with the impurity [illegible] into it, was filled into the container, which was a vial of quartz glass 10 mm in diameter and 220 mm in length. After the loading of the mixture, the vial was evacuated down to a residual pressure of 1.10^{-2} mm Hg and was [illegible]. The mixture was prepared by the method of successive dilution in such a way that the initial concentration of the impurity in the indium triiodide consisted of 1.10^{-2} , 1.10^{-3} and 1.10^{-4} wt.%, respectively.

Iron, arsenic and tellurium iodides were selected by us as the impurities. The selection of these impurities is conditioned, in the first place, by their effect on properties of the indium itself and, in the second place, by the probability of their presence in noticeable quantities in the parent metal.

The behavior of the impurities Fe, As and Te with directed crystallization of the indium triiodide were studied by the method of radioactive indicators [tracers], for which the appropriate radioisotopes were introduced into the InI_3 as the impurities.

The activity was recorded by a standard scaler of 1-2 [illegible] or by a [illegible] counter SI-25 or by means of a gamma spectrometer with a semiconductor germanium-lithium pickup and 4000-channel analyzer. Identical conditions were observed in the measurement, and this made it possible to compute the concentrations in terms of a ratio of activities. The error of the measurements did not exceed $\pm 10\%$.

From results of the directed crystallization, the computation of values of effective distribution coefficients of the impurities was conducted. Used to do this was the basic equation which describes the impurity distribution with the normal directed crystallization in logarithmic form:

$$\lg C = \lg C_0 + (K-1) \lg (1-g)$$

Values of the effective distribution coefficients were determined by the graphic method according to the tangent of the angle of slope of the straight line to the axis of the abscissa.

Investigations on the crystallization purification of InI_3 were conducted in two directions: the behavior of each impurity separately and the effect of the joint presence of the impurities on the effectiveness of purification of the indium triiodide were studied.

The investigation of the behavior of the trace contaminants in the indium triiodide in its crystallization purification showed that all the controllable impurities, including the iron diiodide, are forced back into the end of the specimen and, consequently, possess distribution coefficients less than unity. From results of the experiments we calculated the effective distribution coefficients, the values of which indicate that by the conducted directed crystallization of the indium triiodide it is possible to achieve considerable purification of it from the impurities of iron, arsenic and tellurium.

Conducted according to the method of Barton-Prim-Slichter was the extrapolation of the rate [velocity] dependence of the effective distribution coefficients to the zero value, and determined are the equilibrium distribution coefficients (Table 2), which confirmed the possibility of the successful purification of InI_3 from impurities of iron, arsenic and tellurium by the crystallization methods.

Table 2 Values of K_{eff} of impurities FeI_2 , AsI_3 and TeI_4 in the indium triiodide at different rates of crystallization. At the zero rate values of K are obtained by extrapolation.

| Примеси 1) | $K_{\text{эф}}$ при скорости, см/час 2) | | | | | K_0 |
|----------------|---|-------|------|------|-------|-------|
| | 3,90 | 2,80 | 2,25 | 1,70 | 1,20 | |
| FeI_2 | 0,61 | 0,495 | 0,5 | 0,48 | 0,45 | 0,37 |
| AsI_3 | - | - | 0,21 | 0,16 | 0,135 | 0,07 |
| TeI_4 | 0,40 | 0,32 | 0,25 | 0,20 | 0,175 | 0,095 |

Key: 1) Impurities; 2) K_{eff} , at a rate, cm/h.

In the study of the effect of the initial concentration on the effectiveness of purification of the indium triiodide, it has been established that the distribution coefficient is practically unchanged

with a lowering of the initial concentration of AsI_3 and increases somewhat with a decrease in the initial concentration of the tellurium tetraiodide. For FeI_2 it was not possible to establish the regularities in the change of the value of K_{eff} at different initial concentrations, since its content in the purifiable indium triiodide [illegible - two words] of the limit of sensitivity [illegible - two words]. [Illegible - two words] are given in Table 3.

Table 3 Effective distribution coefficients with different initial concentrations of trace contaminants in indium triiodide.

| Исходная концентрация, вес. % 1) | Коеффициент распределения 2) | |
|----------------------------------|------------------------------|---------|
| | AsI_3 | TeI_4 |
| $1 \cdot 10^{-2}$ | 0,195 | 0,175 |
| $1 \cdot 10^{-3}$ | 0,14 | 0,20 |
| $1 \cdot 10^{-4}$ | 0,14 | 0,25 |

Key: 1) Initial concentration, wt.%; 2) Distribution coefficient.

The majority of the available literature data touches upon the study of the behavior of the individual impurity in a purifiable component. In actual conditions the purification from any one impurity is hardly possible, since in the material to be purified, as a rule, other impurities are always present; and the behavior of these impurities is not considered, and, consequently, the effects of the interaction between them are not considered. Therefore, for the evaluation of the nature of the interaction of the impurities between each other, we studied by the DTA [Differential Thermal Analysis] method the interaction in systems FeI_2-AsI_3 , FeI_2-TeI_4 and AsI_3-TeI_4 . The obtained results showed that all the systems belong to the eutectic type, and in the system FeI_2-AsI_3 there is a wide region of separation (up to 70 mol.% AsI_3). This made it possible to draw the conclusion that with the combined presence these impurities will behave as individual components.

Purification of the indium triiodide with the combined presence of the impurities was also conducted by the method of directed crystallization. The crystallization rate was 1.2 cm/h, and the initial concentration of the impurity was $1 \cdot 10^{-2}$ wt.%. Values of the

effective distribution coefficients were computed according to the obtained data. There was not revealed a considerable difference in values of K_{eff} for the impurities of iron, arsenic and tellurium with their combined presence in the indium triiodide in comparison with values of K_{eff} for each impurity separately.

Values of the equilibrium distribution coefficients, computed from the phase-equilibrium diagram according to the Hays-Chapman equation and based upon experiments on the purification of InI_3 by the method of directed crystallization according to the Barton-Prim-Slichter equation, have a good convergence. The addition of microquantities of FeI_2 to the indium triiodide lowers the melting point of the latter, since the value of the effective distribution coefficient of the iron diiodide is less than one. By considering the nature of the interaction in the system $\text{InI}_3\text{-NiI}_2$, it is possible to assume that the nickel iodide with crystallization of the InI_3 will be forced back into the end of the specimen.

Values of the equilibrium distribution coefficients, obtained by us as a result of the investigation of systems from indium triiodide with iodides of cadmium, silicon, germanium, and tin, indicate that the crystallization methods of purification will be effective with removal of these impurities.

IV. Conclusions

1. For the purpose of the physicochemical basis of crystallographic purification by the method of the differential thermal analysis, a study is made of the interaction of indium triiodide with iodides of iron, cobalt, nickel, manganese, cadmium, arsenic, silicon, germanium, tin, and tellurium, and phase-equilibrium diagrams of the appropriate dual systems are plotted. It is shown that with the diiodides of iron, cobalt, nickel, manganese, arsenic triiodide, and tetraiodides of silicon, germanium and tin, systems of the eutectic type are formed. Indium triiodide with AsI_3 , SiI_4 , GeI_4 , and SnI_4 forms systems close to the ideal.

2. In systems with FeI_2 , CoI_2 , NiI_2 , and MnI_2 , the formation of compounds in the solid phase is revealed for the first time. Compounds $2\text{InI}_3 \cdot 3\text{MnI}_2$ and $\text{InI}_3 \cdot 3\text{MnI}_2$ are revealed from data of DTA

in the system $\text{InI}_3\text{-MnI}_2$ in a metastable state.

3. Revealed in systems with CdI_2 and TeI_4 are compounds of compositions $\text{Cd}[\text{InI}_4]$, $\text{InI}_3\text{-TeI}_4$ (metastable), $\text{InI}_3 \cdot 2\text{TeI}_4$, and $\text{InI}_3 \cdot \text{TeI}_4$.

The formation of the compounds is confirmed by data of X-ray phase analysis.

4. Measurements of the specific electrical conductivity of indium triiodide and certain alloys of the system $\text{InI}_3\text{-CdI}_2$ are given.

Equations for the calculation of values of the specific electrical conductivity of indium triiodide and compound $\text{Cd}[\text{InI}_4]_2$ at different temperatures are given, and the activation energies of the conductivity are calculated.

5. Determined from data of DTA according to the Hays-Chapman formula are the thermodynamic distribution coefficients [illegible - two words] of impurity elements in indium triiodide. Values of the distribution coefficients proved to be less than one.

6. An apparatus is constructed for the purification of indium triiodide and other similar objects by the method of directed crystallization.

7. The behavior of the trace contaminants of iron, arsenic and tellurium with the directed crystallization of the indium triiodide is studied, and the effective distribution coefficients at different rates of crystallization and at different initial concentrations are determined. It is shown that all these impurities have a distribution coefficient of less than one and with crystallization purification of InI_3 are forced back into the end of the specimen.

8. With the combined presence the impurities FeI_2 , AsI_3 and TeI_4 in indium triiodide behave as individual components, which agrees with the assumption made on the basis of the study of the interaction of the impurities with each other.

9. Values of the equilibrium distribution coefficients, computed from the phase-equilibrium diagram according to the Hays-Chapman formula and on the basis of experiments on the purification of indium triiodide by the method of directed crystallization according to the

Barton-Prim-Slichter equation, have a good convergence.

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