

## PREPARATION OF HIGHLY PURE POTASSIUM HYDROXIDE BY ZONE MELTING

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The method of preparing highly pure potassium hydroxide by zone melting of its monohydrate in a Teflon foil has been worked out. The effect of conditions, such as the rate of zone travel, width of molten zone, and number of zone passes on the distribution of impurities has been investigated. Zone refined  $\text{KOH}\cdot\text{H}_2\text{O}$  contained at most  $10^{-6}\%$  of heavy metals, at most  $10^{-5}\%$  of Fe, Ca, Mg, and less than  $10^{-4}\%$  of  $\text{PO}_4^{3-}$ ,  $\text{F}^-$ ,  $\text{Cl}^-$ ,  $\text{SO}_4^{2-}$ , and  $0.05\%$  of  $\text{CO}_3^{2-}$ .

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The preparation of highly pure hydroxides of alkali metals encounters many difficulties caused by their properties. Alkali hydroxides are little volatile, little soluble in nonpolar and highly soluble in polar solvents. These properties considerably limit selection of applicable purification methods and make difficult experimental carrying out the refining operations.

In the literature, several methods of preparing highly pure alkali hydroxides have been presented; none of them, however, is satisfactory. Recrystallization of alkali hydroxide from water<sup>1</sup> or ethanol<sup>2</sup> is fairly little effective. Chemical methods, as for example precipitation<sup>3-5</sup>, coprecipitation<sup>6</sup>, *etc.* can be to some extent used to remove certain contaminants from the solutions; however, contamination with another component cannot be, as a rule, avoided. A similar shortcoming can be seen also with the extraction methods<sup>3,7</sup>. Ion exchange<sup>8</sup>, preelectrolysis<sup>9</sup>, and dialysis<sup>10</sup>, which are able to remove some impurities from alkali hydroxides solutions, are not advantageous, because the alkali hydroxide solution comes into contact with a large surface area of the solid phase which, as a rule, is not inactive against it so that the solution in question is contaminated with other admixtures. Probably the highest purity can be attained by dissolution of very pure electrochemically prepared alkali amalgam<sup>9,11</sup>. This procedure, however, necessitates preparation of a highly pure alkali halogenide and can be applied only to the preparation of a small amount of dilute solutions.

We have found<sup>12</sup> that highly pure potassium as well as sodium hydroxides can be prepared by zone melting of their hydrates. This procedure makes use of the fact that both hydroxides form solid, congruently melting monohydrates of fairly low

melting points ( $\text{NaOH}\cdot\text{H}_2\text{O}$ , 68.9% NaOH, m.p. 66°C;  $\text{KOH}\cdot\text{H}_2\text{O}$ , 75.7% KOH, m.p. 146°C), which can be repeatedly melted in plastic alkali-resistant vessels. In this paper, results of the study on the preparation of very pure potassium hydroxide as well as effect of conditions on the course and effectiveness of the refining operation have been presented.

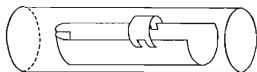
## EXPERIMENTAL

### Apparatus and Working Procedure

Zone refined hydrate  $\text{KOH}\cdot\text{H}_2\text{O}$  was placed in a glass tube lined with Teflon foil. The foil was welded or put together to form a boat. The Teflon foil applied perfectly prevents the zone refined hydroxide from contamination originating from the vessel walls, and moreover, it enables, after the zone refining had been finished, to take out very easily the charge by a mere moving it from the tube and by "unpacking" from the foil, which may be accomplished in the inert atmosphere of the dry-box. The most suitable technique of putting together the Teflon foil is presented in Fig. 1.

FIG. 1

Teflon Foil Put Together to Form a Boat



The alkali hydroxide monohydrate was prepared directly in the tube lined with Teflon foil, where the zone melting took place, involving mixing of solid reagent grade hydroxide with a stoichiometric amount of water, melting, and cooling down below the melting point. The Teflon foil was cleaned before use by boiling in hydrochloric acid, alkaline potassium permanganate solution, and finally, by a repeated washing in distilled water. Approximately two thirds of the Teflon boat were filled with molten hydrate.

The glass tubes were 1 m long, of inside diameter 50 mm; the charge itself was 60 cm long. The Teflon foil was 0.1–0.2 mm thick and 105 mm wide.

An adapted horizontal zone melting apparatus EZK 2 (TOS, Rychnov n.n., ČSSR) was used for the zone refinement. The high frequency heating was replaced by small ring resistance furnaces ( $\approx 13 \Omega$ ) which were loosely put on the tube and guided by a fork. A perfect contact of the resistance coil, which heated the tube from below only, was achieved by pressing the furnaces against the tube by means of a spring. In this arrangement, the use of absolutely linear tubes was not necessary. The zone refining was carried out either with one furnace which passed through the whole charge, or by taking use of the intermittent motion of more furnaces which were 60 to 180 mm distant from one another. The width of the molten zone was controlled by electric current supply or by placing two furnaces close to each other. The rate of zone travel could vary from several tenths mm to several cm/h. The zone melting was carried out continuously in an atmosphere of dry and  $\text{CO}_2$ -free air.

After passing the required number of zones, the glass tube was taken out from the apparatus, opened in the dry-box, and the monohydrate charge was unpacked from the Teflon foil and by emitted heat energy divided into samples to be analyzed. To achieve this, two heating radiators

situated against one another were employed; they consisted of a spiral made of kanthal wire, 0.3 mm in diameter and of resistance 120  $\Omega$ , coiled up on a ceramic body, and heated by a current of 3–4 A. Each radiator was situated in a quartz tube for the charge not to be contaminated with the material dispersed from heaters. The efficiency was increased by using a cylindrical reflector made of polished platinum sheet having a 3 mm wide slot. When using two identical reflectors placed against one another and on each side about 3 cm distant from the charge, an arbitrarily great part of the charge could be separated by melting within 10 minutes. The samples were taken into Teflon beakers, weighed, dissolved in water, and neutralized. By adjusting to a suitable volume, the stock solutions were then prepared.

#### Analytical

In order to determine simultaneously iron, nickel, zinc, cadmium, manganese, silver, copper, and lead by spectrography, the alkali hydroxide solution was neutralized, the metals extracted by sodium diethyldithiocarbamate into chloroform and the chloroform extract was evaporated with powdered spectral carbon and the latter was filled into the carbon electrode.

In the study of the effect of conditions on the distribution of impurities along the ingot, when determination of small concentration changes involved could not be accomplished with high sensitivity spectrographically, other methods were employed. Silver, copper, and nickel were determined by atomic absorption photometry after a preliminary enrichment using extraction and reextraction<sup>13</sup>. Iron was determined photometrically with thiocyanate after coprecipitation with  $MnO_2$ <sup>14</sup>. The magnesium content was determined by atomic absorption photometry without a previous enrichment. The sodium content in potassium hydroxide and potassium content in sodium hydroxide were determined by flame photometry. The results were evaluated by means of the calibration curve constructed with use of the potassium hydroxide solution without sodium resp. sodium hydroxide solution without potassium, to which a known amount of chloride of the alkali metal to be determined was added.

Mercury was determined photometrically after extraction with diphenylthiocarbazone<sup>15</sup>. The contents of  $SO_4^{2-} + CO_3^{2-}$  and  $SO_2^{2-}$  were determined by nephelometry after reactions with barium salt in alkaline ( $SO_4^{2-} + CO_3^{2-}$ ) and acidic ( $SO_4^{2-}$ ) media. The  $Cl^-$  ion was determined nephelometrically as silver chloride, silicate and phosphate photometrically with use of molybdate after reduction with tin dichloride<sup>14</sup>. The fluorides were determined according to<sup>16</sup>.

All the reagents were carefully purified before use, most frequently by extraction. Redistilled water was made use of.

A Zeiss spectrograph Q 24 was employed for spectrographic emission measurements (d.c. arc of 10 A in cathode connection). For atomic absorption as well as flame photometry, an apparatus was used, assembled from a burner and atomizer of the Hilger and Watts apparatus H 1100, a prism monochromator SPM 2 (Zeiss Jena), as well as Soviet photomultiplier FEU 18 A. A high voltage source for the photomultiplier was built, according to the paper by Whisman and Ecclestone<sup>17</sup>, in our Institute's workshop. As strip chart recorder the apparatus EZ 2 (Laboratorní přístroje) was used. The tube valves with hollow cathode were of the Hilger and Watts production. For photometric and nephelometric determinations, Spekol apparatus (Zeiss Jena) was used.

\* This determination was made by J. A. Růžička, Research Institute of Stomatology, Prague.

## RESULTS AND DISCUSSION

The course of the purification of potassium hydroxide monohydrate by zone melting may be followed even according to the change in the melt appearance. After passing the first zone, the melt is being stained apparently by oxygen compounds of heavy metals, produced by air oxidation. A repeated pass of the molten zone produces a clear colourless single crystal, at the beginning of the charge another part of the charge is formed by vitreous mass coloured yellowish and pink, which at the end attains grey-brown colour. The length of the single crystal increases with the number of passed zones and growth of the crystal is thus a reliable criterion of the course of the purifying operation. The change in the distribution of impurities is apparent even after several passes, but only if the rate of zone travel is sufficiently low. At the rate of 5 mm/h a colourless clear single crystal is produced at the beginning of the charge within the first pass already. At higher rate of zone travel, amounting to 7 mm/h the single crystal is not being formed before the second or third zone pass. At the rate of 30 mm/h the single crystal was not produced even after the hundredth zone

TABLE I

Change in the Impurity Amount of Potassium Hydroxide Monohydrate  $n$  35,  $v$  3 mm h<sup>-1</sup>,  $l$  30 mm

Impurity	Amount, %	
	original	after zone refining
Fe	$5 \cdot 10^{-4}$	$10^{-5} - 10^{-6}$
Ni	$4 \cdot 10^{-4}$	$1 \cdot 10^{-6}$
Cu	$3 \cdot 10^{-5}$	$1 \cdot 10^{-6}$
Ag	$2 \cdot 10^{-4}$	$< 10^{-6 a}$
Cd	$1 \cdot 10^{-5}$	$< 10^{-6 a}$
Zn	$5 \cdot 10^{-4}$	$10^{-6}$
Mn	$1 \cdot 10^{-5}$	$1 \cdot 10^{-6}$
Pb	$2 \cdot 10^{-4}$	$< 10^{-6 a}$
Hg	$1.6 \cdot 10^{-4}$	$< 10^{-5 a}$
Ca	$2 \cdot 10^{-4}$	$1 \cdot 10^{-5}$
Mg	$5 \cdot 10^{-4}$	$< 10^{-5 a}$
Na	0.63	$5 \cdot 10^{-4}$
CO <sub>3</sub> <sup>2-</sup>	1.5	0.05
PO <sub>4</sub> <sup>3-</sup>	$1 \cdot 10^{-3}$	$1 \cdot 10^{-5}$
F <sup>-</sup>	$5.8 \cdot 10^{-4}$	$< 10^{-4 a}$
Cl <sup>-</sup>	$2 \cdot 10^{-3}$	$1 \cdot 10^{-4}$
SO <sub>4</sub> <sup>2-</sup>	$1 \cdot 10^{-3}$	$< 10^{-4 a}$

<sup>a</sup> Below the limit of determination of the analytical method used.

pass. The course of purification very strongly depends on the KOH : H<sub>2</sub>O ratio. If the hydrate has no stoichiometric composition, monohydrate of an accurate composition is being formed at the beginning of the charge and excessive water or hydroxide are removed during the zone melting till the end of the charge is reached. In order to attain the purification effect many zone passes are required.

Analytical examination of the purification process showed that all the impurities studied are shifted in the direction of the molten zone travel. The amount of impurity in potassium hydroxide monohydrate before zone melting as well as that of the impurities in a clear single crystal (about 40% of the charge) after 35 zone passes are presented in Table I. An intermittent motion of five furnaces (length of the charge of 60 cm, zone width of 3 cm, zone travel rate of 3 mm/h) was used for the zone melting.

The finding is essential that the zone melting of potassium hydroxide monohydrate enables to decrease the amount of homogeneously dispersed cations and anions, as well as the amount of dispersed heavy metal hydroxides by at least one order

TABLE II

Effective Distribution Coefficients of Impurities in KOH.H<sub>2</sub>O, Found during Zone Refining under Different Conditions

*v* Rate of zone travel, *n* number of zone passes.

Impurity	<i>v</i> mm .h <sup>-1</sup>	<i>n</i>	<i>k</i>	
			experimental	calculated <sup>a</sup>
Fe	6.0	1	0.66	0.758
	4.0	1	0.60	0.549
	3.0	1	0.51	0.464
	2.0	8	0.40	0.379
	1.8	2	0.36	0.365
	1.8	1	0.37	0.365
	0	—	0.235 <sup>b</sup>	—
Ag	1.4	1	~0.5	—
Cu	1.4	1	~0.4	—
Ni	1.4	1	~0.5	—
Mg	1.4	1	~0.3	—
Na	6.0	1	~0.1 <sup>c</sup> ~0.5 <sup>d</sup>	—
	3.0	1	~0.03 <sup>c</sup> ~0.2 <sup>d</sup>	—
	1.8	1	~0.01 <sup>c</sup> ~0.1 <sup>d</sup>	—

<sup>a</sup> Calculated using the following values:  $k_0 = 0.235$ ;  $\delta = 1 \cdot 10^{-1}$  cm;  $D = 8 \cdot 10^{-7}$  cm<sup>2</sup> s<sup>-1</sup>;

<sup>b</sup> extrapolated; <sup>c</sup> calculated for the beginning of the charge;  $x = 0$ ; <sup>d</sup> calculated for the middle part of the charge;  $x = 1/2$  L.

of magnitude, and in some cases, by three to four orders. For the found values of the distribution coefficients (Table II), a deeper purification than by 4–5 orders cannot be expected even theoretically during zone melting leading to the limit distribution. Since the alkaline hydroxide, both solid and liquid, came into contact with Teflon within the zone refining, the fluoride amount in repeated experiments was followed at the beginning and at the end of the charge. The pure part of the charge appeared to contain always a smaller quantity of fluoride than the starting substance, but the end of the charge is so contaminated with the fluoride that Teflon must be assumed not to be quite resistant to the melt of alkaline hydroxide monohydrate, and that the released fluoride traces are quickly transported towards the end of the charge.

In further experiments, effect of working conditions on the efficiency of the purification process has been established. The distribution after one pass of the molten zone through the charge is according to Pfann<sup>18</sup> given by

$$c_{x,1} = c_0[l - (l - k_0)e^{-k_0x/l}], \quad (1)$$

where  $c_{x,1}$  is the concentration of the considered impurity at distance  $x$  from the beginning of the ingot,  $c_0$  is the original impurity concentration,  $l$  denotes width of the molten zone,  $k_0$  is the equilibrium distribution coefficient defined as  $k_0 = c_s/c_l$  ( $c_s$  and  $c_l$  are equilibrium concentrations of the approximate impurity in the solid and liquid phases). The equilibrium distribution coefficient is associated with the effective distribution coefficient which is applied by the relation<sup>19</sup>

$$k = \frac{k_0}{k_0 + (l - k_0)e^{-f\delta/D}} \quad (2)$$

under nonequilibrium conditions of the zone refining. Here,  $\delta$  is the thickness of the phase interface,  $D$  the diffusion coefficient of the appropriate impurity in the melt,  $f$  denotes the rate of crystallization, which is given by the rate of zone travel  $v$  under conditions of the zone refining. The impurity for which  $k_0 > 1$  accumulates at the beginning of the charge, and that for which  $k_0 < 1$ , this being a more frequent case, is accumulated at the end of the charge. It follows from relation (1) that the separation is more favourable, the more different is  $k_0$  from unity. In real cases, *i.e.* for  $f\delta/D > 0$ , provided that  $k_0 > 1$  and  $k_0 < 1$  holds, the effective coefficient values are  $k_0 < k < 1$  and  $k_0 > k > 1$ , respectively;  $k$  is then always closer to unity than  $k_0$  and the distribution is less favourable than under equilibrium conditions. We studied, therefore, the effect of the rate of zone travel on the values of effective distribution coefficients, as well as effect of the rate of zone travel and zone width on the distribution of impurities along the charge. The distribution was established by analyzing the charge which was refined by one furnace under various conditions.

In Fig. 2a,b,c, the distribution of iron after the pass of one zone at the rate of zone travel of 1.8, 3, and 6 mm h<sup>-1</sup> along the charge having length of 4 cm and width

of the molten zone of 3 and 18 cm, is graphically presented. The values of the effective distribution coefficients, given in Table II were calculated as ratios of the iron concentration in original monohydrate and in the monohydrate after the pass of the first zone at  $x = 0$ . The value of the equilibrium distribution coefficient  $k_0$  for Fe was achieved by extrapolation for  $v = 0$ . The effective distribution coefficient value rises rapidly with the increasing rate of zone travel and at the rate of 10 mm/h its value is so close to unity that an analytically detectable change in the iron

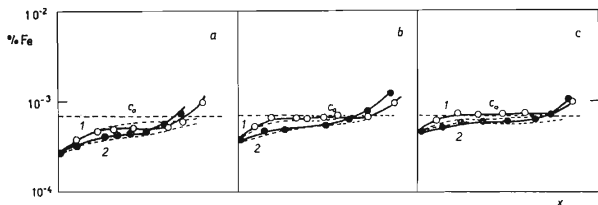


FIG. 2

Effect of Zone Width  $l$  on the Distribution of Fe in  $\text{KOH} \cdot \text{H}_2\text{O}$  after One Zone Pass ( $n = 1$ )

$x$  distance from the beginning of the charge. 1 / 3 cm. 2 / 18 cm. Rate of zone travel ( $\text{mm h}^{-1}$ ) 1a 1.8; 1b 3; 1c 6. Full curves are constructed on the basis of experiments, dotted curves are calculated for  $k$  taken from Table II.

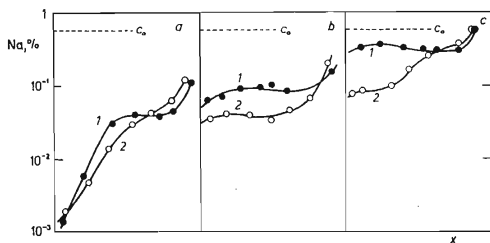


FIG. 3

Effect of Zone Width  $l$  on the Distribution of Na in  $\text{KOH} \cdot \text{H}_2\text{O}$  after One Zone Pass ( $n = 1$ )

$c_0$  original concentration of Na 1 / 3 cm. 2 / 18 cm. Rate of zone travel ( $\text{mm} \cdot \text{h}^{-1}$ ) 2a 1.8; 2b 3; 2c 6.

concentration along the charge is not attained even after 3 passes. Relation (2) agrees with the established dependence of the effective distribution coefficient upon the rate of zone travel, if we substitute the value reported by Pfann as the usual one ( $\delta = 1 \cdot 10^{-1}$  cm) and for the diffusion coefficient use the value  $D = 8 \cdot 10^{-7}$  cm<sup>2</sup> s<sup>-1</sup>.

The fact that the latter value is by one to two orders of magnitude lower than is that reported for the diffusion of ions in the melt<sup>20</sup>, is obviously associated with the fact that iron is in the melt present as dispersed colloidal hydroxide and not in the form of simple ions. The form of distribution and effect of conditions on the values of effective distribution coefficients, similar to those found when studying the distribution of iron, were established even during the study of the distribution of magnesium, copper, manganese, silver, and nickel. Values of the effective distribution coefficients of the metals in question are summarized in Table II. For Ag, Cu, Mn, Mg, Ni, and Cu, however, only approximate distribution coefficient values have been determined, for these metals are either contained in the original KOH in small amounts, or their determination is of such a low sensitivity that their contents at the beginning of the charge are decreased below the limit of determinability during zone melting and only comparison of a further part of the whole distribution curve is possible.

The effect of the rate of zone travel and zone width on the distribution of sodium is given in Fig. 3. The effective distribution coefficient  $k$  is very favourable; with the increasing rate of zone travel, however, its value rapidly approaches unity (Table II). Unfavourable effect of a higher rate of zone travel on the distribution may be seen from the finding that after two zone passes at the rate of 1.4 mm/h, an approximately equal distribution is being attained as is that achieved at the rate of 7 mm/h only after 30 zone passes. In Fig. 3 effect of the zone width on the distribution of sodium is likewise obvious. At the slow rate of zone travel the effect of a change in the zone width is small, at higher rates the extension of the zone affects the distribution in some-

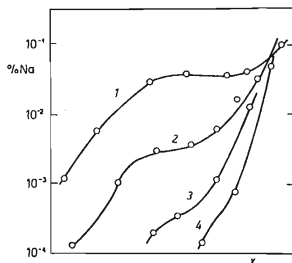


FIG. 4

Effect of the Number of Zone Passes  $n$  on the Distribution of Na in KOH · H<sub>2</sub>O

$v$  1.8 mm · h<sup>-1</sup>;  $l$  3 cm. 1  $n$  1; 2  $n$  2; 3  $n$  3; 4  $n$  4.



what more favourable way. The distribution of iron (Fig. 2a,b,c) as well as that of magnesium, copper, manganese, nickel, and silver in  $\text{KOH}\cdot\text{H}_2\text{O}$ , after the pass of one molten zone through the charge, may be expressed by the Pfann equation (1), the found value of the effective distribution coefficient being used; the shape of the distribution of sodium (Fig. 3a,b,c), however, cannot be expressed by this equation and is quite different. It is of interest that removal of sodium from  $\text{KOH}\cdot\text{H}_2\text{O}$  is very effective at the beginning of the charge provided that the zone is passing slowly, but that it deteriorates considerably with the zone progress. The effect of the zone width and rate of zone travel on the distribution is likewise greater than would correspond to relations (1) and (2). This may be explained in the following way: the Pfann's theory assumes<sup>18</sup> that the distribution coefficient remains constant during the whole zone refining and is independent of the impurity concentration in the melt. This is really fulfilled in some cases, see for example, stability of the value of the effective distribution coefficient of iron for the rates  $v = 1.8$  and  $v = 2 \text{ mm h}^{-1}$ , and for  $n = 1$ ,  $n = 2$ , and  $n = 8$ . In the first and further passes a partially, and for  $n = 8$ , a considerably purified charge is taken for the start; in spite of this, the  $k$  value does not change more distinctly. It may be shown<sup>21</sup> that in case of the non-constant distribution coefficient, the effect of the value of the distribution coefficient and of the zone width on the impurities distribution is more pronounced. The same effect was also observed in experiments following the distribution of sodium in zone melted charges of  $\text{KOH}\cdot\text{H}_2\text{O}$ . The fact that the second zone pass, which starts from a partially purified charge, is at the beginning of the charge more effective than would correspond to the Pfann theory (Fig. 4), gives evidence of the dependence of the distribution coefficient change on the concentration. Also the difference in the distribution coefficients for the beginning and middle part of the charge, even when approximately established values of the coefficients are concerned, reminds of the fact that the effective distribution coefficient  $k < 1$  rises with the increasing concentration of sodium in the  $\text{KOH}\cdot\text{H}_2\text{O}$  melt. This effect is also assumed by Matz<sup>22</sup> in discussion of behaviour of some systems during zone melting. Because of the possibility of dehydrating the monohydrate, the effect of the molten zone temperature could not be studied in our case.

The effect of conditions on the zone melting process of sodium hydroxide monohydrate has been studied similarly, though in less detail. Even in this case, it has been established that the impurities, both ions and metal hydroxides, move in the direction of zone travel. Mutual ratio of effective distribution coefficients for individual impurities little differs from the values found during the zone refining of potassium hydroxide; however, they are higher at comparable rates of zone travel.

Details concerning the possibilities of practical utilization of the zone melting for the preparation of highly pure sodium and potassium hydroxides are referred in paper<sup>11</sup>.

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## REFERENCES

1. Pentland N., Bockris J. O. M., Sheldon E.: *J. Electrochem. Soc.* *104*, 182 (1957).
2. Kuczerpa A.: *Anal. Chem.* *39*, 1197 (1967).
3. Duke F. R., Haas T. W.: *J. Phys. Chem.* *63*, 304 (1961).
4. D'Ans J., Mattner J.: *Angew. Chem.* *64*, 448 (1952).
5. Shanlay F. S., Greenspan F. P.: *Ind. Eng. Chem.* *39*, 153 (1947).
6. Lister M. W., Petterson R. C.: *Can. J. Chem.* *40*, 729 (1962).
7. Koubek E.: *J. Am. Chem. Soc.* *85*, 2263 (1963).
8. Powell J. E., Hiller M. A.: *J. Chem. Educ.* *34*, 330 (1957).
9. Vepřek-Šiška J., Ettl V., Regner A.: *This Journal* *31*, 1237 (1966).
10. Slough W.: US-Pat. No 3013905 (1961).
11. Vepřek-Šiška J., Eckschlager K., Ettl V.: *Chem. průmysl* *20*, 312 (1970).
12. Vepřek-Šiška J., Eckschlager K., Ettl V.: *Czechoslov. Pat. No 131809*, January 27th, 1969; *British Pat. No 1,175,476*, October 24th (1969).
13. Eckschlager K.: *This Journal* *34*, 1321 (1969).
14. Sandell E. B.: *Colorimetric Determination of Metals*, 3rd Ed. Interscience, New York 1959.
15. Koch G. O., Koch-Dedic G. A.: *Handbuch der Spurenanalyse*, p. 593. Springer, Berlin 1964.
16. Růžička J. A.: *This Journal* *30*, 2117 (1965).
17. Whisman M., Eccleston B. H.: *Anal. Chem.* *27*, 1861 (1965).
18. Pfann W. G.: *Zone Melting*, 2nd Ed., p. 29. Wiley, New York 1966.
19. Burton J. A., Prim R. C., Schlichter W. P.: *J. Chem. Phys.* *21*, 1987 (1953).
20. Janz G. J.: *Molten Salts*. Handbook, p. 349. Academic Press, New York—London 1967.
21. Eckschlager K., Ettl V., Stopka P., Kodejš Z.: *This Journal*, in press.
22. Matz G.: *Chem.-Ingr. Techn.* *36*, 381 (1964).

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