Complex Processing tailings of the Balkhash Concentrating Factory

Viktor Mikhaylovich Shevko, Gulnara Ergeshovna Karataeva, Galimzhan Mendikaraevich Serzhanov, Alexandra Dmitrievna Badikova and Assiya Salidinovna Abzhanova

South Kazakhstan State University named after M.Auezov, Kazakhstan,160012, Shymkent, Tauke Khan avenue, 5

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At flotation concentration of copper ores the tailings are formed, which contain both nonferrous metals and a significant amount of silicon and iron. The nonferrous metals from the tailings can be taken by flotation or leaching; but the silicon and iron is used irrationally as a backfilling material in mined-out space. The work purpose was the development of a complex technology of processing the Balkhash concentrating factory's tailings (Kazakhstan) with production of a collective polymetallic concentrate and ferroalloys. The research has been fulfilled by a method of thermodynamic modelling using the software package HSC-5.1 (Outokumpy), a method of rototable experiment planning with regard to the chloride sublimation roasting of the Balkhash concentrating factory's (BCF) tailings (0.27-0.39 % of Cu; 0,54-2.03 % of Zn; 0.13-0.5 % of Pb; 12.35-22.73 % of Fe; 7.04-13.09 % of Ňàl; 0.3-1.48 % of Mgl; 6.2-11.71 % of Al₂O₃; 0.02-0.011 % of Mo; 0.04-0.07 % of As; 38.49-57.99 % of SiO₂; 0.41-0.58 % of Na; 0.61-0.74 % of K; 0.8-1.3 % of S) and electrosmelting cinders after the roasting. It is found, that copper is extracted from the flotation tails by chloride sublimation worse, than zinc and lead. For limitation of iron chloridosublimation it is necessary to carry out the process at 7-fold oxygen excess and 3-fold CaCl, one. High degree of nonferrous metals' chloridosublimation (80-89 % of Cu, 95-99 % of Zn, > 99 % of Pb) and low degree of iron chloridosublimation (1-1.23 % of Fe) is observed at 1140-1200°C during 39-60 min and the surplus of CaCl, in 3,2 times. Electrosmelting the cinders of the chloride sublimation roasting of the Balkhash concentrating factory's (BCF) flotation tailings in a mix with coke and quartzite allows to produce a ferroalloy with silicon content of 28-63 % (at extraction in the ferroalloy of 80-87.3 % of silicon). Ferrosilicon of a grade of FS50 is formed at the electrosmelting a charge containing 11 % of steel cuttings, 20 % of quartzite from the ore mass and 126-133 % of coke from a theoretically required quantity for the silicon and iron reduction. Ferrosilicon of a grade of FS45 is produced at electrosmelting the cinders in the presence of 20 % of quartzite, 6-11 % of steel cuttings and 100-106 % of coke.

Keyw ords: Concentration tailings, chloridosublimationà, cinder, electrosmelting, ferroalloy.

At flotation concentration of coppercontaining ores waste products are formed (to 98 % on 1 tonne of the ore (Samygin, Filipov, & Shehkirev ,2003; Morozov, Abdykirova, & Falei, 2015)). Now a quantity of flotation wastes only on the Balkhash and Dzhezkazgan concentrating factories makes nearly 1bn t. These wastes, occupying considerable ground areas (approximately 40 km² for the Balkhash factory), not only make worse environmental ecology, but also contain a significant amount of valuable metals. Kazakhstan produces 360-390 ths t of copper (Alshapov, 2004; State of Kazakhstan mineral raw material base (2015, February 18); World goods markets (2015, February 18)) and loses annually with the concentration tailings more 6-8

^{*} To whom all correspondence should be addressed.

ths t of copper, 36-48 ths t of zinc, 4-5 ths t of lead, to 500 ths t of silicon and 150 ths t of iron. Therefore the concentration tailings are potential raw materials not only for nonferrous metallurgy, but also for manufacture of iron-silicon alloys. In world practice sufficiently rich tails are processed by secondary flotation and leaching; it allows reextracting no more than 50-60 % of nonferrous metals. In the process, as a rule, a nonmetallic component does not earmark for manufacture of the products in demand (Turkebaev, & Sadykov, 1988; Glazunov, & Davydov, 1980; Snurnikov, 1986; Enne, Sycheva, Edakina, Bykov, etc., 1992; Rudnev, 2004; Vorobyev, & Iskushina, 1997; Jagin, Davydov, Rudnov, etc., 2001; Golik, 2010). In our opinion a perspective method of processing the Balkhash concentrating factory's tails is a combined chloride electrothermal way allowing to recover not only nonferrous metals, but also to produce ferroalloys of various grades (Rybnikova, Emelyanenko, & Gorbatova, 2009; Badikova, Shevko, & Karatayeva, 2015; Shevko, Badikova, Serzhanov, Uteyeva, & Karataeva, 2014).

The given article contains the research results of nonferrous metals' recovery from the Balkhash concentrating factory's flotation tails using chloride sublimation roasting and manufacture of ferroalloys from the roasting cinders.

METHOD

Investigated raw material

For the research performance we have used the BCF's tailings. Chemical content of the tailings (in accordance with the data of the production association "Balkhashstvetmet"): 0.27-0.39 % of Cu; 0.54-2.03 % of Zn; 0.13-0.5 % of Pb; 12.35-22.73 % of Fe; 7.04-13.09 % of CaO; 0.3-1.48 % of MgO; 6.2-11.71 % of Al₂O₂; 0.02-0.011 % of Mo; 0.04-0.07 % of As; 38.49-57.99 % of SiO₂; 0.41-0.58 % of Na; 0.61-0.74 % of K; 0.8-1.3 % of S. According to the results of roentgen-phase analysis fulfilled by means of a device D8 Advance (Bruker), the basic components of the tails are SiO₂, $NaCa_{2}Fe_{5}Si_{8}O_{22}(OH)_{2}, Ca_{3}Al_{0,84-0,88}Fe_{1,16}Si_{3}O_{12},$ $(K_{0,82}Na_{0,18})$ $(Fe_{0,03}Al_{1,92})(AlSi_3)O_{10}(OH)_{2-8}$ $Mg_{4,882}Fe_{0,22}Al_{1,881}Si_{2,96}O_{10}(OH)_{8-18}, CaSO_{4} \cdot 2H_{2}O,$ $K_{0.58}Na_{0.42}AlSi_{3}O_{8}$ and $Na(AlSi_{3}O_{8})$ (figure 1). Weight loss at the calcination at a temperature of 1200°C makes 5 %. Initial melting point is 1192°C, end melting point is 1289°C (figure 2).

These data have been got by a differential thermal method by means of a device of synchronous thermal analysis STA 449 F3 Jupiter. The samples were heated to 1300°C with heat rate of 15°C/min in a high-purity argon atmosphere. The total amount of inlet gas was kept up within 80 ml/min. Analysis of the results, obtained with the help of STA 449 F3 Jupiter, was made by means of the NETZSCH Proteus software.

Technical calcium chloride (96.5 % of CaCl₂) was used as a chlorinating agent.

Chemical composition of the raw materials used

coke: 4.9 % of SiO₂, 1.5 % of CaO, 0.41 %of MgO, 1.8 % of Al₂O₃, 2.2 % of Fe₂O₃, 0.8 % of S, 1.1 % of H₂O, 86 % of C, 1.3 % - other; steel cuttings: 98 % of Fe, 0.4 % of S, 1.6 % - other; quartzite: 97 % of SiO₂, 0.7 % of CaO, 0.3 % of MgO, 0.7 % of Al₂O₃, 0.8 % of Fe₂O₃, 0.2 % of H₂O, 0.3 % - other. **Technique of carrying out the research**

The thermodynamic modelling of nonferrous metals' chloridosublimation from the BCF's tailings has been performed with use of the software package ÍSC-5.1 (Outokumpy) based on a principle of the Gibb's energy minimum. For calculations we have used the subprogram "Equilibrium compositions", which allows on the basis of quantitative composition of substances to calculate equilibrium distribution degree of an element between all components of a system (Roine, 2002).

Determination of optimum technological parameters of the nonferrous metals' chloridosublimation from the flotation wastes has been carried out by a method of experiment planning using rototable plans of the second order with the subsequent construction of volume and plane images of independent variables' influence on optimization parameters (chloride sublimation degree of a metal $- \dot{a}_{chl Me}$) (Ahnazarova, & Kafarov, 1978). Chloride sublimation of Cu, Pb, and Zn from the tails has been fulfilled in an air flow in a vertical furnace with carborundum heaters. Before the experiment carrying out the BCF's tailings have been pelletized in a bowl granulator (diameter -0.4m, height of an edge -0.08 m). A calcium chloride solution is applied as a binder. The pellet granules in diameter 0.8-1.0 cm were dried at 230-240°C during 30 minutes. After the drying the granules in

where $\rm G_{charge}$ and $\rm G_{cinder}$ – a charge mass and a cinder mass accordingly, g;

 $G_{Me(charge)}$ and $C_{Me(cinder)}$ – a metal content in a charge and in a cinder, g.

The metals' content in the tails and in the cinder was determined by means of a device AAS-1 (Germany). Electrosmelting the cinder has been carried out in an arc electric furnace with controlled output (from 0 to 10 of kilovolt-ampere). The cinder with coke and steel cuttings was melted during 55-60 minutes in a graphite crucible, which is 10 cm high and its internal diameter is 7 cm. Diameter of a graphite electrode is 3 cm. The charge weight is 250-280 g. Extraction degree of silicon and iron in an alloy is calculated according to the formula:

$$_{\text{chl}Me} \quad \frac{G_{\text{charge}} \quad C_{Me \text{ charge}} \quad G_{\text{cinder},C_{Me(\text{cinder})}}}{G_{\text{charge}} \quad C_{Me(\text{charge})}} \quad 100 \qquad \dots(1)$$

97

96

95

200

400

600

diameter of 1 cm have static durability of 8-12 kg/

granule. The dried granules were placed in a

container (a basket) made from an alloy X20H80

(melting point is 1400°C) and were loaded into a vertical resistance furnace. Air was fed in the

bottom part of the furnace by means of a

compressor "Matens" (Italy) through a regulating

cock and a rheometer. The temperature in the furnace was regulated by a platinum-platinum-rhodium

thermocouple. The chlorides formed were caught

in a gas cooler and a bag filter. The weight of pellet

charge loaded (G_{charge}) was 8-10 g. The metals'

chloridosublimation degree has been calculated

under the formula:

$${}^{Me \text{ alloy}} \quad \frac{G_{alloy}C_{Me \text{ alloy}}}{G_{cinder}} \times 100 \qquad ...(2)$$

0

-5

-20

oDTA "10-3/

0.05 -10

-0.10 -15

1200

-0.15

-0.20

-0.25

-0.30



800 /*C Fig. 2. Derivatogramme of the BCF's tailings

1000

where

 G_{cinder} and G_{alloy} – a cinder mass and an alloy mass accordingly, g;

 $C_{Me (cinder)}$ and $C_{Me (alloy)}$ - a metal content in a cinder and in an alloy accordingly, %.

The silicon content in the alloy was determined by a bottle method using kerosene.

RESULTS AND DISCUSSION

The information about chloridosublimation of copper, zinc and lead from the BCF's tails in the presence of calcium chloride is represented in Figure 3.

It follows from the figure 3 that at the 3,2fold excess of CàCl, for the reactions:

$$CuS + CaCl_2 + O_2 = CuCl_2 + CaO; \qquad ...(3)$$

$$ZnS + CaCl_2 + O_2 = ZnCl_2 + CaO; \qquad ...(4)$$

$$PbS + CaCl_2 + O_2 = PbCl_2 + CaO, \qquad \dots (5)$$

increase of the oxygen amount to 7-fold

excess somewhat reduces α_{chl} Cu (from 93 to 88 %), slightly influences on α_{chl} Zn (90-92 %) and Pb (> 99 %), however in this process undesirable chloridosublimation of iron (as FeCl₂ and FeCl₃) decreases from 10.4 to 2.4 % at 1200°C.

Table 1 contains the information about influence of independent factors (temperature, T, °C; duration of the process, τ , minute; CaCl₂ amount, XA, % from theoretically possible for the reactions 1-3). On the basis of these data using a program Microsoft Excel (Inkov, 2006) we have found adequate regression equations $f(T, \tau, XA)$. The regression equations' significance was defined in accordance with the Student's test, and adequacy of the equations – in accordance with the Fisher's test. The following equations were obtained:

$\alpha_{chl}Cu = -600.25 + 0.88 \cdot T + 1.75 \cdot t +$	0.048XA - 3.10
$^{4}\cdot \overset{\circ}{O}^{2}$ - $6\cdot 10^{-3}\cdot \tau^{2}$ - $4\cdot 10^{-4}XA$ - $8\cdot 10^{-4}\cdot$	$T \cdot \tau + 0.1 \cdot 10^{-3} \cdot T$
\cdot XA+0.1 \cdot 10 ⁻² \cdot τ \cdot XA;	(6)

 $\begin{array}{l} \alpha_{chl} Zn = -121.44 + 0.13 \cdot T + 3 \cdot \tau - 0.097 XA - 9 \cdot 10^{-3} \cdot \tau^2 \\ -0.6 \cdot 10^4 \, .XA^2 - 2 \cdot 10^{-3} \cdot \dot{O} \cdot \tau + 0.14 \cdot 10^{-3} \cdot T \cdot XA + 1.01 \\ \cdot 10^{-3} \cdot \tau \cdot XA; & \dots(7) \end{array}$

Experiment	Code kind			Natural kind			α _{chl} Cu, %	$\alpha_{chl}Zn, \%$	α_{chl} Fe, %
number	\mathbf{X}_{1}	X_2	X_3	T,°C	τ, \min	CaCl ₂ , %			
1	1	1	1	1159.6	51.9	275.4	78.0	89.5	1.04
2	-1	1	1	1040.6	51.9	275.4	53.5	80.3	0.50
3	1	-1	1	1159.6	28.1	275.4	61.6	82.0	0.79
4	-1	-1	1	1040.6	28.1	275.4	35.0	65.4	0.31
5	1	1	-1	1159.6	51.9	144.6	67.8	76.2	0.80
6	-1	1	-1	1040.6	51.9	144.6	45.5	67.3	0.36
7	1	-1	-1	1159.6	28.1	144.6	56.0	70.0	0.58
8	-1	-1	-1	1040.6	28.1	144.6	31.0	57.7	0.13
9	1.68	0	0	1200	40	210	74.5	87.4	0.89
10	-1.68	0	0	1000	40	210	32.6	66.1	0.21
11	0	1.68	0	1100	60	210	66.3	80.5	0.68
12	0	-1.68	0	1100	20	210	42.0	65.5	0.31
13	0	0	1.68	1100	40	320	61.0	83.2	0.80
14	0	0	-1.68	1100	40	100	42.3	68.6	0.36
15	0	0	0	1100	40	210	57.5	76.0	0.55
16	0	0	0	1100	40	210	55.7	75.1	0.61
17	0	0	0	1100	40	210	56.9	74.4	0.51
18	0	0	0	1100	40	210	58.0	76.4	0.57
19	0	0	0	1100	40	210	57.8	76.8	0.59
20	0	0	0	1100	40	210	56.2	75.8	0.60

Table 1. The experiment planning matrix and the results of Cu, Zn and Fe chloride sublimation from the tailings of the Balkhash concentrating factory

 $\begin{aligned} \alpha_{chl} Fe = -1.912 + 54 \cdot 10^{-4} \cdot T + 91 \cdot 10^{-3} \cdot \tau - 418 \cdot 10^{-3} \cdot XA \\ + 9 \cdot \tau^2 - 114 \cdot 10^{-4} \cdot t^2 - 3 \cdot 10^{-5} \cdot XA^2 + 8 \cdot 10^{-6} \cdot \tau + 4 \cdot 10^{-5} \\ \cdot T \cdot XA - 16 \cdot 10^{-6} \cdot \tau \cdot XA. \end{aligned}$

Similar researches have been performed for the chloride sublimation of lead (in a temperature interval of 800-1000°C). A regression equation $\alpha_{cbl}Pb = f(T, \tau, XA)$ looks like:

 $\begin{array}{l} \alpha_{chl} Pb = -1014.1 + 2.06 \cdot T + 1.79 \cdot \tau + 0.31 X A - 1.01 \cdot 10^{-3} \cdot \tau^2 - 1.99 \cdot 10^{-2} \cdot \tau^2 - 9 \cdot 10^{-5} \cdot X A^2 + 46 \cdot 10^{-4} \cdot X A \cdot \tau - 24 \\ \cdot 10^{-4} \cdot T \cdot X A. \end{array}$

On the basis of the received equations using the program Mathcad (Ochkov, 2007) a 3D image of a response surface of α_{chlMe} and its horizontal sections has been constructed for XA = 32 % (smaller amount of CaCl₂ does not provide high α_{chl} Me). It follows from the figure 4, that 85-90 % of α_{chl} Cu can be reached in the area *fnm* (320 %, 1174-1200°C, 51.6-60 min), 95-99 % of α_{chl} Zn – in the area *zyx* (320 %, 1160-1200°C, 39-60 min), 90-98 % of α_{chl} Pb – in the area *uko* (320 %, 920-1000°C, 41-56 min). As α_{chl} Cu < α_{chl} Zn < α_{chl} Pb, that the

Experiment		Code kind		Natural kind				C _{si} , %
number	\mathbf{X}_{1}	X_2	X ₃	С, %	S, %	Q, %		
1	1	1	1	139.9	12.7	15.95	81.4	51.0
2	-1	1	1	110.1	12.7	15.95	68.3	44.0
3	1	-1	1	139.9	3.23	15.95	74.9	57.4
4	-1	-1	1	110.1	3.23	15.95	60.0	51.0
5	1	1	-1	139.9	12.7	4.05	67.7	44.4
6	-1	1	-1	110.1	12.7	4.05	53.0	35.9
7	1	-1	-1	139.9	3.23	4.05	52.0	52.2
8	-1	-1	-1	110.1	3.23	4.05	42.4	46.3
9	1.68	0	0	150	8	10	75.0	52.0
10	-1.68	0	0	100	8	10	49.6	42.0
11	0	1.68	0	125	16	10	73.3	49.0
12	0	-1.68	0	125	0	10	55.2	58.3
13	0	0	1.68	125	8	20	76.3	53.0
14	0	0	-1.68	125	8	0	53.0	37.6
15	0	0	0	125	8	10	66.4	48.0
16	0	0	0	125	8	10	65.0	47.4
17	0	0	0	125	8	10	67.3	47.0
18	0	0	0	125	8	10	64.8	48.1
19	0	0	0	125	8	10	65.7	46.0
20	0	0	0	125	8	10	67.0	46.3

Table 2. The experiment planning matrix and the results of electrosmelting the cinder of chloride sublimation roasting the BCF's tailings

chloridosublimation of copper from the BCF's tailings is a constraint.

At the chloride sublimation roasting of the tails the chloride sublimation of iron is undesirable. At the development of iron chloridosublimation the nonferrous metals' sublimates are polluted by iron chlorides. Therefore it is necessary to define conditions at which α_{chl} Me (for the present instance, copper) will has a maximum value, and α_{chl} Fe – a minimum one. Figure 5 represents the joint information about α_{chl} Cu and α_{chl} Fe from which follows that high (80-89 %) α_{chl} Cu and low (1-1.25 %) α_{chl} Fe are observed at 1140-1200°C during 39-60 minutes at 320 % of CaCl₂ (the area *ltz*). For this case a_{chl} Zn makes 95-99 % and α_{chl} Pb is > 99.0 %.

Chemical composition of the cinder formed after the roasting: 48.3 % of SiO₂, 25.4 % of Fe₂O₃, 12.6 % of CaO, 1.1 % of MgO, 0.9 % of Na₂O + K₂O, 11.0 % of Al₂O₃, 0.8 % - other.

The experiment matrix of electrosmelting the cinder formed after the tailings' roasting is represented in the table 2. Optimization parameters in this experiment series are: silicon extraction



I-without excess of O₂, II-2.5-fold excess of O₂, III- 5-fold excess of O₂, IV- 7-fold excess of O₂

Fig. 3. Influence of temperature and oxygen amount on chlorido sublimation degree of copper, zinc, lead and iron from the BCF's tailings



I - copper, II - zinc, III - lead

Numbers of lines – α_{chl} Me, %

Fig. 4. Horizontal sections of a response surface – α_{chl} Me



Numbers on lines – α_{chl} Fe (–), α_{chl} Cu (—)

Fig. 5. Combination of α_{chl} Cu and α_{chl} Fe from the BCF's tailings

degree in an alloy (α_{si}) and silicon content in an alloy (C_{si}). Independent factors are: coke amount, % from all the silicon, which is theoretically required for the iron reduction (C, %), steel cuttings amount, % from weight of the cinder (S, %), quartzite amount, % from weight of the cinder (Q). On the basis of the data of the table 2 the following regression equations have been found:

 $\begin{array}{l} C_{Si} = -600.24 + 0.879 C + 1.75 S + 0.048 \cdot Q - 3 \cdot 10^4 \cdot C^2 - \\ 6 \cdot 10^3 \cdot S^2 - 4 \cdot 10^4 \cdot Q^2 - 8 \cdot 10^4 \cdot C \cdot S + 1.01 \cdot 10^3 \cdot S \cdot Q \dots (11) \end{array}$

Fig. 6 contains the information on influence of the independent factors on C_{si} . It follows from the given figure, that the increase of

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I - without quartzite, II - 10 % of quartzite, III - 20 % of quartzite Number on lines - α_{si} %





I - a volume image of a response surface

II– horizontal sections of a response surface with isolines of equal degree C_{si} , %.

Numbers on lines - α_{si} (–), α_{si} (—)

Fig. 7. Effect of coke and steel cuttings amounts on α_{s_i} and C_{s_i} at 20 % of quartzite

steel cuttings amount in the charge leads to the reduction of C_{si} , and the increase of coke amount – to the growth of C_{si} . And the increase of quartzite amount in the charge leads to the growth of silicon content in the alloy formed. Depending on concentrations of the charge's components at the electrosmelting the silicon content in the alloy changes from 28 to 63 %. In compliance with (State Standard 1415-93, 1993) now it is supposed that the ferrosilicon corresponding a grade of FS45 is a ferroalloy with silicon content from 41 % to 47 %, FS50 – from 47 to 52 %, and FS65 – from 63 to 68 % of silicon. At the processing the cinder without quartzite it is possible to obtain ferrosilicon FS45 (the area *abcde* in the figure 6 (I)) and ferrosilicon

FS50 (the area *edon* in the figure 6 (II)). At the increase of quartzite amount the areas of FS45 and FS50 production are displaced towards the increase of steel cuttings consumption and reduction of coke consumption (figures 6 (I) and 6 (II)). However, proceeding from the figure 6 (III) it is impossible to produce ferrosilicon FS65 from the cinder.

It follows from the figure 7, that at $\alpha_{si} = 80-83\%$ the ferrosilicon FS50 is formed in the area *zyz* (11% of steel cuttings, 126-133% of coke, 20% of quartzite). The ferrosilicon FS45 can be obtain from the cinder in the area *tfo* (20% of quartzite, 6-11% of steel cuttings, 100-106% of coke), however in this case α_{si} is low (62-67%).

CONCLUSIONS

- a) In the equilibrium state high chloride sublimation degrees of copper (88 %), zinc (92 %) and lead (> 99 %) from the Balkhash concentrating factory's tailings at low degree of iron chloride sublimation (\leq 2.4 %) are observed at the following conditions: 1200°C, 3.2-fold CaCl₂ excess and 7-fold oxygen excess.
- b) At chloride sublimation roasting the granulated tails in air stream $\alpha_{chl}Cu < \alpha_{chl}Zn$ $< \alpha_{chl}Pb; \alpha_{chl}Cu$ is 80-89 %, $\alpha_{chl}Zn$ is 95-99 %, $\dot{\alpha}_{chl}Pb$ is more than 99.0 % and $\alpha_{chl}Fe$ makes 1-1.23 % at the following conditions: 1140-1200°C, duration of the process – 39-60 min and 3.2-fold CaCl₂ excess.
- c) Electrosmelting the cinders formed after the chloride sublimation roasting of the Balkhash concentrating factory's tailings in a mixture with coke and quartzite allows to produce a ferroalloy containing 28-63 % of silicon; at the extraction of 80-87.3 % of silicon in the alloy the ferrosilicon FS50 can be obtained at 11 % of steel cuttings, 20 % of quartzite (from the ore weight) and 126-133 % of coke (from the theoretical amount required for the silicon and iron reduction); the ferrosilicon FS45 is formed at the electrosmelting the cinder in the presence of 20 % of quartzite, 6-11 % of steel cuttings and 100-106 % of coke.

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