FEATURES OF SYNTHESIS C₃S IN HIGH SULFUR CLINKER

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The results of thermodynamic analysis influence of clinker SO₃ on synthesis of C₃S, the technique of calculation of raw mix on formation in high sulfur clinker intermediate metastable phases are presented.

Key words: high sulfur clinker, alit C_3S .

The summary

In connection with growth of cost of the basic hydrocarbon fuel resources, manufacturers of Portland cement began to use the alternative energy sources containing significant amount of SO₃ for its manufacture. At presence in raw materials of significant amount SO₃, activity of Portland cement on the basis of such raw mix considerably decreases. It is caused by maintenance reduction in is high-sulfur clinker by alit C_3S . In present paper the reasons of suppression of formation alit in is high-sulfur clinker are considered and ways of increase of the maintenance to it alit at the expense of formation are offered at synthesis clinker intermediate meta-stable phases.

Introduction

Sulfur and it oxides in the form of sulfate and sulfide minerals can get to structure of a raw mix for manufacture of Portland cement clinker as together with the basic raw materials for clinker preparation - clay and lime stone, and with additives and fuel. The maintenance of sulfurous connections in additives of a man-caused origin of the metallurgical and heat power industry - slag, fuel, fly ash and fuel in the form of petro-coke is especially high. On data [1,2], in the conditions of oxidizing roasting sulfur forms with calcium oxides anhydrite of sulfate of calcium CaSO₄ which depending on roasting temperature forms sulfates of alkaline metals with alkaline components of a raw mix, with clinker's minerals sulphospurrite $2(C_2S)C\overline{S}$ and ye'elimit $C_4A_3\overline{S}$ and participates in a alkali - sulfur cycle of the furnace.

At maintenance SO_3 in clinker less than 2,0 % it makes positive impact on synthesis of Portland cement clinker as the sulfates of alkaline metals formed at its presence are effective fluxing agent, reducing temperature of occurrence of a liquid phase and it's viscosity, providing the accelerated synthesis clinker's minerals [3]. Positive role of SO_3 in clinker also is shown at use of a raw mix with the considerable maintenance of alkalis. With mole parity $SO_3/(Na_2O+K_2O)$ close to 1, removal of sulfates of alkali from a raw mix at the expense of ablation of sulfates of alkaline metals is provided at heating.

In excess of the specified limits clinker's SO_3 observed the negative effects associated with Portland cement clinker quality and with the technology of its production. According to [1,4], at maintenance increase in the raw mix SO_3 , quantity C_2S increases, C_3S decreases, and at the equal total maintenance of the given phases in clinker, their parity C_3S/C_2S decreases.

As alit C_3S is not only the most active mineral of clinker, defining durability of Portland cement clinker, but also it's most refractory phase, decrease in maintenance clinker's C_3S leads to decrease in its fire resistance and activity of Portland cement.

In the paper [5] established that the reason for the decline of C_3A in highsulfur clinker is isomorphic substitution of aluminum silicate phases of silicon and based on the research are invited to adjust the formula R.H. Bogue calculation of C_3A in high- sulfur clinker.

Reasons for the decline of C_3S in clinker with increasing content of clinker's SO_3 in references are absent.

Material and methods

The raw mixes calculated under modular characteristics on reception Portland cement clinker with modular characteristics LSF=0,92, SR=2,3 and AR=1,69 were exposed to roasting. For preparation Portland cement clinker the raw mix on a basis of lime stone, clay, quartz sand and natural gypsum was used. At preparation of a raw mix the case when gypsum gets to a raw mix together with raw components was modelled. Natural gypsum was entered into the raw mix prepared according to calculation in the ratio, a raw mix -95 %, gypsum -5 %, for achievement of maintenance SO₃ in a raw mix more than 2,0 %. The raw mix dried and homogenizing in a laboratory mill by a joint grinding of raw components and gypsum within 30 minutes. Homogenizing mix was pressed at 50 MIIa.

The pressed samples subjected to roasting at temperatures from 1100 to 1300 °C. Completely synthesised clinker crushed to the rest on a sieve 008 no more than 5 % (Blaine 4500) and by the chemical analysis defined the maintenance in them CaO_{free} and clinker's oxides [6]. At completely synthesised clinker chemical and x-ray ways defined phase structure. Qualitative x-ray phases analysis spent on device DRON-3. Quantitative x-ray phases analysis carried out on diffractometer STADI-P (STOE, Germany). Thermogravimetric researches spent on scanning calorimeter STA 449 F3 Jupiter (Netzsch-Geratebau GmbH) by the methods of DIN 51004:1994. The chemical compound of raw components and clinker is presented in Table 1.

Component	CaO	SiO ₂	Al_2O_3	Fe ₂ O ₃	SO ₃	MgO	P_2O_3	LOI	The
									sum
Lime stone	56,35	0,040	0,06	0,04	0,03	0,37	0,014	42,80	99,70
(CaCO ₃)									
Clay	1,21	50,70	19,90	11,80	0	1,40	0,17	14,81	99,90
Quartz	0,03	98,80	0,45	0,03	0	0	0	0,13	99,40
sand									
Natural	32,02	0,80	0,45	0,23	44,50	0	0	22,0	100,0
gypsum									
Clinker	67,17	22,19	6,08	3,59	0,035	0,85	0,067	0	99,99
without									
taking into									
account									
gypsum's									
oxides									
Clinker	65,25	21,12	5,80	3,42	2,26	0,80	0,064	1,1	99,99
with the									
account									
gypsum's									
oxides									

 Table 1: The Chemical compound of a raw mix, mas. %

Results

Results qualitative x-ray analysis of the investigated mixes are presented in Figure 1.



Figure 1: Results qualitative x-ray analysis of clinker with the additive 5 % of gypsum

The maintenance of free lime in high-sulfur clinker is presented in Table 2.

The material name	Maintenance of free lime in clinker, маз. %, at					
	temperature of roasting, °C					
	1100	1200	1300			
Raw mix	14,0	13,84	11,6			

Table 2: The maintenance of free lime in high-sulfur clinker

Results of tests testify, that introduction in a raw mix of gypsum of 5 % suppresses alito-formation and promotes formation in clinker a significant amount of free lime. Diffractional maxima, characteristic for free lime, are present in clinker, calcinated at temperature 1300 °C, and diffractional maxima, characteristic for C_3S , at this temperature are not fixed. At heating of all clinkers up to temperature 1350 °C they completely melting. The analysis of meltin's products, heated up to temperature of 1600 °C, testifies, that C_3S at such overheat is formed, but in clinker there are 6,2 % free lime.

Discussion

The reason of suppression of formation a C_3S in is high-sulfur clinker most likely is formation of free lime, which has a stable crystallization structure, is not

dissolved in a liquid phase, does not co-operate with C_2S and does not form C_3S . Absence heat-resistant C_3S in clinker leads to decrease in its fire resistance and fusion at temperatures, below usual temperatures of synthesis of Portland cement clinker.

The assumption of that superfluous free lime in clinker is formed owing to decomposition of the gypsum entered into a raw mix, it is erroneous. As show given tab. 1, maintenance free CaO in clinker, containing gypsum it is, less, than in clinker without gypsum, besides, a significant amount free lime to exhaust in is high-sulfur clinker it is formed at temperatures when gypsum does not decay yet (tab. 2).

For finding-out of the reasons of occurrence of a significant amount of free lime in high-sulfur clinker, the thermodynamic analysis of reactions of formation C_3A and C_4AF according to data of works has been carried out [7,8]. Results of the analysis are presented in Table 3.

№ п/п	Reaction	Gibbs energy ΔG , kJ/mol at temperature, 1					
		298	1023	1200	1400	1500	
1	$3CaO+Al_2O_3=3CaO\cdotAl_2O_3$	-17,0	-41,8	-47,0	-52,9	-55,7	
2	$4CaO+Al_2O_3+Fe_2O_3 = 4CaO \cdot Al_2O_3 \cdot Fe_2O_3$	-49,3	-64,9	-64,1	-60,83	-58,1	
3	$3CaO+3Al_2O_3+CaSO_4 =$ $3CaO\cdot3Al_2O_3\cdot CaSO_4$	-99,1	-445,1	-583,6	-758,9	-853,5	
4	$CaO \cdot Al_2O_3 + 2CaO = 3CaO \cdot Al_2O_3$	+33,7	+32,3	+31,7	+33,26	+34,4	
5	$CaO \cdot Al_2O_3 + CaO + 2CaO \cdot Fe_2O_3 = 4CaO \cdot Al_2O_3 \cdot Fe_2O_3$	+10,4	+39,7	+49,0	+60,3	+66,2	
6	$CaO \cdot Al_2O_3 + 2CaO + CaO \cdot Fe_2O_3 = 4CaO \cdot Al_2O_3 \cdot Fe_2O_3$	+42,45	+72,1	+79,77	+79.8	+83,7	

Table 3: The thermodynamic analysis of reactions of formation C_3A and C_4AF according to works [7,8]

According to the resulted data in is low-sulfur clinker synthesis C_3A and C_4AF thermodynamic is possible from simple minerals CaO, Al_2O_3 and Fe_2O_3 (reaction 1-2). In is high- sulfur clinker thermodynamic course of reaction of 3 formations ye'elimit $C_4A_3\overline{S}$ is more preferable. Concerning the formation mech-

anism ye'elimit in clinker at heating there are different opinions. According to work [8] synthesis ye'elimit, begins with formation of mayenite under scheme $C_{12}A_7 \rightarrow CA \rightarrow C_3A_3C\overline{S}$. According to data of our paper [9] in the pressed raw

mix proceeds under scheme $CA_2 \rightarrow CA \rightarrow (CA)_2 \rightarrow (CA)_3 \rightarrow C_3A_3C\overline{S}$ with formation at occurrence temperatures to exhaust monoaluminate of calcium CA and its presence at all temperature interval of roasting to temperature of 1300 °C. In the presence of monoaluminate of calcium formation C₃A and C₄AF thermodynamically is impossible (reactions 4-6). On the basis of the given researches authors [7] have drawn a conclusion, that in the presence of monoaluminate of calcium C₃A and C₄AF are formed from melt.

Hence, if calculation of a raw mix is conducted on formation in is highsulfur clinker minerals C_3A and C_4AF , and actually in such clinker before occurrence of a liquid phase are formed CA and CF, such process can lead to occurrence free lime under schemes

$$3CaO + Al_2O_3 \rightarrow CaO \cdot Al_2O_3 + 2CaO, \tag{1}$$

$$4\text{CaO} + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3 \rightarrow \text{CaO} \cdot \text{Al}_2\text{O}_3 + \text{CaO} \cdot \text{Fe}_2\text{O}_3 + 2\text{CaO}.$$
 (2)

Formed free lime in the course of heating has a stable crystallization structure, it is not dissolved in liquid phase and suppresses formation C_3S at the initial stage of its formation. Quantity free lime, formed in clinker at the expense of a difference of its maintenance in C_3A and CA it is insignificant, and makes only 3 %, but it is probable this quantity enough for formation suppression C_3S at the initial stage and creations of conditions for formation in clinker a significant amount free lime. As C_3S in clinker it is not formed, and it is formed C_2S , at the expense of a maintenance difference lime in the given minerals, the total maintenance free lime at temperatures of end of synthesis makes already about 11,0 %. At a considerable overheat melt to 1600 °C C_3S all the same it is formed, but free lime it is so a stable crystallization structure, that is not dissolved in melt even at such overheat and remains in quantity about 6,0 %. For prevention of formation free lime, suppressing formations C_3S in clinker presence, it is offered to settle an invoice the raw mix on synthesis in Portland cement clinker intermediate metastable phases existing in a solid phase, but breaking up at occurrence of a liquid phase. As at occurrence of a liquid phase thermodynamically is possible there is a formation C_3A and C_4AF phases, they can be formed from CA and CF only in the presence of free lime under schemes

$$CA+2CaO \rightarrow C_3A, \tag{3}$$

$$CA+CF+2CaO \rightarrow C_4AF. \tag{4}$$

In the conditions of temperatures of roasting of 1300 $^{\circ}$ C, in the absence of free lime its source may become the only transformation of C₃S and disintegration of calcium sulfate by schem:

$$C_3 S \rightarrow C_2 S + CaO, \tag{5}$$

$$2CaSO_4 \rightarrow 2CaO + 2SO_2 \uparrow + O_2 \uparrow.$$
(6)

If at structure designing Portland cement it is in advance known, that C_3S can decay, it is expedient to form in clinker its maximum quantity that it is possible to reach only at KSk=1.

For calculation of factor of saturation in Russia used Kind V.A. formula [10]

$$KSk = \frac{CaO - (1,65Al_2O_3 + 0,35Fe_2O_3)}{2,8SiO_2};$$
(7)

At a conclusion of this formula are used molar parities CaO, Al_2O_3 , Fe_2O_3 and SiO₂ at formation in clinker the basic clinker minerals C₃S, C₂S, C₃A and C₄AF.

English analogue of the given formula is the formula for calculation Lime Saturation Factor[11]

$$LSF = \frac{CaO}{2,8SiO_{2}+1,2Al_{2}O_{3}+0,65Fe_{2}O_{3}}$$
(8)

Factors in the given formula are taken from phase diagram CaO-Al₂O₃-SiO₂ and CaO-Al₂O₃-SiO₂-Fe₂O₃ at optimum relationship oxides, providing absence free lime in clinker. If to use Kinda V.A's design procedure [10], that the formula (8) becomes full analogue of the formula (7)

$$LSF = \frac{CaO}{2,8SiO_2 + 1,65Al_2O_3 + 0,35Fe_2O_3}$$
(9)

As calculation on formation in high-sulfur clinker C_3A and C_4AF leads to formation in it of a significant amount of free lime, the given formulas have been corrected for formation in clinker metastable phases

$$KSk = \frac{CaO - 0.55Al_2O_3 - 0.35Fe_2O_3 - 0.7SO_3 - 1.18P_2O_5}{2.8SiO_2},$$
 (10)

$$LSF = \frac{CaO-1,18P_{2}O_{5}-0,7SO_{3}}{2,8SiO_{2}+0,55Al_{2}O_{3}+0,35Fe_{2}O_{3}}.$$
 (11)

The given formula for calculation of factor of saturation is received with the account, that at roasting of a raw mix in the presence of gypsum, in clinker minerals C_3S , C_2S , and metastable phases CA and CF are formed. Value 1,18 P_2O_5 is considered, if in quality mineralizer is used phosphogypsum. At synthesis ferrite of calcium C_2F use value 0,7Fe₂O₃.

For calculation of degree of saturation of metastable clinker's minerals by sulphate in the presence of gypsum used Atakuziev T.A.'s formula [12] with the account, that in clinker is possible sulfatization C_2S , CA, CF and C_2F . Taking into account the given updating, the formula of calculation of degree of saturation looks as follows

$$DS = \frac{SO_3 - 0.261Al_2O_3 - 0.167Fe_2O_3}{0.667SiO_2} .$$
(12)

In a case when it is synthesised C_2F , at Fe_2O_3 the factor 0,5 and when it is not supposed sulfatization of ferrite of calcium, values 0,167 Fe_2O_3 is used and 0,5 Fe_2O_3 into the formula are not entered. When synthesized C_2F , Fe_2O_3 is the coefficient 0,5, and when ferrites is not sulfatizized, the coefficients 0,167 and 0,5 in formula are not entered.

For check of possibility of synthesis C_3S in is high-sulfur clinker, calculation of a raw mix from the components which chemical compound is presented to table 1 on synthesis in products of roasting of silicate phase C_3S (KSk=1), intermediate metastable aluminate phase $C_4A_3\overline{S}$ and intermediate metastable ferrit phases CF, C_2F , 3(CF)C \overline{S} and $C_2FC\overline{S}$ was made. Settlement structures of raw mixes and a chemical compound clinkers, it is presented in Table 4.

The name of	Structure of a raw mix,			Chemical compound of clinker, маз. %				
clinker	маз. %							
	LS*	Clay	Gyp-	CaO	SiO ₂	Al_2O_3	Fe ₂ O ₃	SO ₃
			sum					
Clinker № 1	71,42	25,6	2,97	64,98	20,41	8,03	4,76	2,1
Clinker № 2	70,65	25,28	4,07	64,39	20,14	7,92	4,69	2,85
Clinker № 3	71,96	25,13	2,92	65,27	20,07	7,9	4,68	2,03
Clinker № 4	69,73	24,2	6,07	64,38	19,33	7,6	4,49	4,22

* - lime stone

Table 4: Structure of raw mixes and clinkers of synthesised cements

Modular characteristics for all clinkers have made KSk =1, DS=0, SR=1,6 and AR=1,69. The settlement intermediate phase clinker's structure is presented to Table. 5.

The name of	C ₃ S	$C_4A_3\overline{S}$	CF	$3(CF)C\overline{S}$	C_2F	$C_2FC\overline{S}$	The
clinker							sum
Clinker № 1	76,23	16,94	6,81	-	-	-	99,98
Clinker № 2	75,18	16,68	-	8,15	-	-	100,01
Clinker № 3	74,9	16,65	-	-	8,43	-	99,98
Clinker № 4	73,75	15,92	_	_	-	10,43	100,1

Table 5: Settlement intermediate phase structure of clinkers

The raw mixes prepared according to calculation subjected to homogenization in a laboratory mill by a joint grinding within 30 minutes. Homogenization mixes pressed at 50 MIIa. Roasting of all mixes was spent at temperatures 1150, 1200, 1250 and 1300 $^{\circ}$ C. Roasting products investigated x-ray

methods. Results qualitative x-ray the analysis clinker N_2 1 calcinated at temperatures 1150,1200, 1250 and 1300 $^{\circ}C$ are presented in Figure 2.



Figure 2: Results qualitative x-ray analysis of clinker № 1, calcinated at temperatures from 1150 to 1300 °C

Change of intensity diffractional maxima of the basic phases clinker N_{2} 1 depending on roasting temperature, is presented in Figure 3.



Figure 3: Intensity change diffractional maxima of the basic phases of clinker № 1 depending on roasting temperature

Data qualitative x-ray testify, that in clinker, calculated on synthesis in the presence of gypsum of intermediate metastable phases, free lime at temperature of roasting of 1200 °C and above, no find. Diffractional maximum with d=1,76 Å, characteristic for C₃S, to temperature of 1250 °C increases, and since temperature of 1250 °C, decreases. Diffractional maximum with d=2,28 Å, characteristic for C₂S, on the contrary, to temperature of 1250 °C decreases, and above this temperature – increases, that testifies to part transformation C₃S in C₂S. With increase in temperature of roasting intensity diffractional maximum with d=1,91 Å increases, characteristic for brownmillerite C₄AF. Intermediate metastable phase C₄A₃ \overline{S} (d=3,72 Å), with increase in temperature of roasting breaks up, to what reduction of its intensity of the basic diffractional a maximum after temperature of 1150 °C testifies.

Similar results are received at roasting of the raw mixes calculated on reception in clinkers as intermediate metastable feffit $3(CF)C\overline{S}$, C_2F , $C_2FC\overline{S}$ and aluminate $C_4A_3\overline{S}$ phases.

For definition of actual structure of clinkers, synthesised at temperature 1300 °C, has been spent quantitative x-ray the analysis. Results quantitative x-ray the analysis for clinker \mathbb{N} 1 are presented on Figure 4.



Figure 4: Results of quantitative x-ray the analysis for clinker № 1

The name of mineral	The maintenance, маз. %, in						
	clinker 1	clinker 2	clinker 3	clinker 4			
Alite C ₃ S	77,8	70,8	72,2	46,1			
Brownmillerite C ₄ AF	15,3	19,6	14,6	14,2			
Belite C_2S	6,9	9,6	13,2	39,7			

In Table 6 presents summary results of quantitative x-ray the analysis synthesised clinkers.

Table 6: Summary results quantitative x-ray synthesised clinkers

Results quantitative x-ray the analysis testify, that in clinkers on the basis of the corrected raw mix really there is a significant amount of alit (except clinker N_{2} 4). In synthesised clinkers presence of C₃A is not fixed, and absolutely are absent sulfated phases. X-ray ways of identification of phase structure badly define light oxides, in particular sulphates. Definition of the maintenance of sulphates in synthesised clinkers a weight method has shown, that in them contains from 0,5 to 2,75 Mas. % SO₃ that means, that from 40 to 75 Mas. % of the sulphates entered into a raw mix disappears, and remained in clinker sulphates, possibly, are connected in sulfated phases or are in alit in the form of a solid solution.

Results qualitative, quantitative x-ray and data of the chemical analysis synthesised clinkers have allowed to establish, that at synthesis of cements with intermediate metastable phases at a finishing stage difficult physical and chemical processes proceed.

At temperatures of roasting to 1200 $^{\circ}$ C, in the conditions of absence of a liquid phase, synthesis proceeds with formation of intermediate metastable minerals and their quantities, defined settlement by. As soon as at the temperature close to temperature of 1200 $^{\circ}$ C, in system there is a liquid phase, thermodynamically it becomes more preferable formation through liquid phase C₄AF, and C₃A which start to be formed in the liquid environment, involving in reaction already generated in conditions of solid-phase synthesis intermediate metastable aluminate and ferrit minerals, causing their decomposition. Lime, necessary for synthesis of C₄AF, and C₃A from metastable low lime aluminates and calcium ferrites, arrives from partial transformation alit in belit and also gypsum decomposition under schemes 5 and 6. After partial transformation of alit in belit, actual KSk (LSF) synthesised clinkers 1-4 it is equal accordingly 0,965; 0,949; 0,935 and 0,823. The sulphate liberated at decomposition of intermediate phases, either disappears, or enters reaction with alit with formation of a solid solution. Thus, remained in clinkers sulphate can be connected residual $C_4A_3\overline{S}$ or silicate phases.

Absence in clinkers C_3A in is high-sulfur clinker is possibly connected with isomorphic replacement Si in silicate phases by the Al, that will be coordinated with data of paper [4]. Owing to aluminium occurrence in silicate phases, the maintenance of alit also raises.

On the basis of received at temperature 1300 °C clinkers, 4 cements by a joint grinding clinker with natural gypsum have been prepared. As in synthesised clinkers sulphates already contain, the quantity of natural gypsum at a grinding was added from calculation, that maintenance SO_3 in Portland cement would not exceed admissible standard documents of quantity - 4,0 %. Activity of cement was defined on cubes with a size 2x2x2 the sm, prepared of the cement test of normal density.

	Material		SO ₃ ,	S,	R ₀₀₈ ,	ND,	Terms	of ce-
—	struct	ure	%	m²/kg	%	%	ment	ation,
Type of							hour-r	ninute
clinkers	alinkar	aun					bagin	and
	CHIKEI	дур-					begin	enu
		sum						
Clinker № 1	96	4	3,96	348	12,7	25,2	2-50	4-15
Clinker № 2	97,5	2,5	4,0	368	12,0	29,3	2-15	3-15
Clinker № 3	96	4	3,86	353	7,8	25,0	2-35	4-05
Clinker № 4	100	0	4,22	344	10,7	30,1	2-00	3-05

Results of definition of physico-mechanical properties of cements are presented in Table 7 and Table 8.

*S – a specific surface; R_{008} – the rest on a sieve No 008; W/C – a water-cement parity; ND – normal density

Table 7: Results of tests of Portland cement*

Type of clinkers	Strength at compression, MPa, through, days								
	2 7 14 28								
Clinker № 1	21,8	31,1	42,5	67,3					
Clinker № 2	22,5	33,2	44,7	70,0					
Clinker № 3	25,2	37,3	46,4	72,5					
Clinker № 4	30,8	46,3	54,1	73,7					

Table 8: Results of tests of Portland cement

Conclusions

The reason of suppression of alito-formation in is high-sulfur clinkers is formation at heating clinker a significant amount of free lime.

The thermodynamic analysis of reactions of formation of clinker's phases in the presence of calcium sulphate has shown, that in such conditions synthesis aluminate phases proceeds on mono aluminate to the scenario.

At presence Mono aluminate of calcium formation in a firm phase of C_3A and C_4AF thermodynamically is impossible, hence formation CA instead of C_3A leads to formation of a significant amount of free lime, suppressing alito-formation. As instead of alit in clinker it is formed belit, superfluous quantity of lime is formation, equal to a difference of its maintenance in the given minerals, it is allocated in a free kind. The total maintenance free lime at the synthesis closing stages makes about 11 %.

Updating of structure of a raw mix allows to generate in is high-sulfur clinker a significant amount of alit. Calculation should be made on formation in is highsulfur clinker the intermediate metastable low-lime alluminate and ferrit minerals.

In the following part of paper results of experimental researches on synthesis alit in the high-sulfur clinker, containing to 15 % SO₃ through intermediate meta-stable low-lime alluminate, ferrit and silicate clinker's minerals will be presented.

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