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FINAL REPORT

EFFECT OF CALCIUM NITRITE ON THE PROPERTIES OF CONCRETE USED IN BRIDGE DECKS

Celik Ozyildirim, Ph. D. Senior Research Scientist

(The opinions, findings, and conclusions expressed in this report are those of the author and not necessarily those of the sponsoring agencies.)

Virginia Transportation Research Council (A Cooperative Organization Sponsored Jointly by the Virginia Department of Transportation and the University of Virginia)

In Cooperation with the U.S. Department of Transportation Federal Highway Administration

Charlottesville, Virginia

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ABSTRACT

Chloride-induced corrosion of the reinforcing steel in bridge decks can impair its structural integrity and cause spalling, which reduces the ride quality of the deck. One system to prevent corrosion involves the use of corrosion inhibitors in freshly mixed concrete.

This study evaluates the properties of field concretes containing the inhibitor DCI (calcium nitrite) and assesses their field performance over a 3-year period. Comparative tests were made on a two-span bridge, one span of which had epoxy-coated bars with regular concrete and the other uncoated bars with concretes containing DCI.

The results indicate that the properties of the hardened concretes containing DCI were satisfactory and similar to those of the regular concretes. A conclusion on the effectiveness of DCI was not reached because of the limited time available; consequently, a follow-up evaluation is recommended. At present, the continued use of epoxy-coated reinforcing steel is recommended.

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FINAL REPORT

EFFECT OF CALCIUM NITRITE ON THE PROPERTIES OF CONCRETE USED IN BRIDGE DECKS

Celik Ozyildirim, Ph.D. Senior Research Scientist

INTRODUCTION

Corrosion of reinforcing bars in bridge decks can impair their structural integrity and can cause spalling, which reduces the ride quality of the deck. Chlorides penetrating into the concrete from deicing salts or the marine environment induce this corrosion.¹ To prevent or minimize corrosion of the reinforcing bars in decks, a number of protective systems have been used. These include the use of epoxy-coated bars, increased cover depth, two-stage construction with low permeability overlays, and concretes containing corrosion-inhibiting admixtures such as calcium nitrite.^{1,2,3} These systems vary in cost-effectiveness, and this depends somewhat on the conditions under which they are used.

Since the late 1970s, the Virginia Department of Transportation (VDOT) has routinely used epoxy-coated steel for reinforcing bars in bridge decks. This system primarily depends on preventing or greatly reducing the contact of the chloride ion, oxygen, and moisture with the steel. This prevents the occurrence of galvanic action that would otherwise result in corrosion of the reinforcement. The products of corrosion occupy a greater volume than the original steel with the consequent creation of disruptive forces in hardened concrete. Although the epoxy coatings protect the steel from corrosive elements, any breaks or flaws in the coatings that expose the steel render it vulnerable to corrosion. Bridge decks with uncoated bars are continuously exposed to the corrosion process. This is especially true when the concrete permits relatively rapid penetration of chloride, for example, when cracks are present or when high permeability results from the use of high water-cement ratios (w/c) or poor construction practices.

Uncoated steel can be protected against corrosion by using a corrosion inhibitor such as calcium nitrite.^{4,5} The inhibitor is added to freshly mixed concrete. It induces a reaction that results in a protective oxide layer around the steel. In effect, the chloride ions and the nitrite ions engage in competing actions, and corrosion does not occur when the amount of chloride is low compared to the amount of nitrite (low chloride-to-nitrite ratios). To achieve continuing protection with the inhibitor at minimum costs, the amount of calcium nitrite added to freshly mixed concrete varies with the amount of chlorides expected during the life of the structure. Calcium nitrite could be used either as the basic protective system or as added protection in conjunction with low permeability concrete and epoxy-coated reinforcing bars. Preliminary evaluation at the Virginia Transportation Research Council (VTRC) showed that in laboratory tests, some concretes containing calcium nitrite did not provide satisfactory resistance to deterioration resulting from cycles of freezing and thawing even though they had proper air-void systems by the usual criteria. The results varied depending on the source of the cement used, indicating possible differences in paste structure or chemical composition (but this has not been verified). However, the supplier of the calcium nitrite claims that field installations of concretes with the calcium nitrite admixture have been shown to have satisfactory resistance to deterioration resulting from freezing and thawing. If this claim can be substantiated, the use of concretes containing calcium nitrite as a protective system would likely provide a cost-effective system especially in severe conditions in which all the reinforcing bars are subject to exposure.

OBJECTIVE AND SCOPE

The objective of this study was to evaluate concretes containing calcium nitrite under field conditions. Their resistance to deterioration resulting from freezing and thawing was determined, and their effectiveness in preventing corrosion was evaluated and compared to that of epoxy-coated reinforcing bars. The study was limited to testing concretes placed on a single bridge.

METHODS AND MATERIALS

The bridge tested is on Rte. 60 over the Willis River in Cumberland County, 1.9 miles east of the Buckingham County line (Lynchburg District). It has two 70-ft spans, and the deck has a minimum thickness of 8.5 in. One span was used as the control; it contains epoxy-coated bars. Concrete with no inhibitor was placed on this span on May 7, 1988. The other has uncoated bars, and an experimental concrete containing a calcium nitrite corrosion inhibitor marketed under the trade name DCI was placed on this span on May 11, 1988.

Control and experimental concretes were prepared using Type II cement. The chemical and the physical properties of the cement are shown in Table 1. The mixture proportions are given in Table 2. The w/c of the control concretes was 0.44 and that of the experimental concretes was 0.43. The coarse aggregate was crushed granite gneiss with a maximum aggregate size of 1 in. The fine aggregate was siliceous sand. A commercially available air-entraining admixture and a water-reducing admixture were used in all the concretes. The experimental concrete differed from the conventional control concrete in two ways: (1) 3.5 gal/yd³ of DCI containing 30 percent by weight calcium nitrite was added, and (2) the amount of air specified was 7.0 ± 1.5 percent rather than 6.5 ± 1.5 percent as in the control. The increase was recommended by the manufacturer of the inhibitor to compensate for any loss of air in the concrete containing calcium nitrite. Both the control and the

CHEMICAL AND	PHYSICAL ANALYSIS O	F THE CEMENT

Chemical	%	Physical	m²/kg	
SiO ₂	21.1	Fineness, Blaine	364	
$A1_{2}O_{3}$	4.4			
Fe_2O_3	2.7			
CaO	63.2			
MgO	3.5			
SO_3	2.8			
Na ₂ O equiv.	0.56			
Ignition loss	0.84			
C_3S	56.0			
C ₃ A	7.0			

Table 2

MIXTURE PROPORTIONS IN LB/FT³

Ingredient	Control	DCI Concrete
Cement	682	682
w/c ^a	0.44	0.43
Coarse aggregate	1,764	1,764
Fine aggregate	1,105	1,105
DCI (gal/yd ³)		3.5

^a Water from the DCI is included.

experimental concretes were placed using conventional equipment and procedures. Curing was accomplished by applying a curing compound.

Two batches from the control concrete and two from the experimental concrete were sampled. Tests on the freshly mixed concrete were conducted for air content (ASTM C231) and slump (ASTM C143) for each batch on both the control and the experimental concretes. Specimens were prepared for tests at the hardened stage for strength, resistance to deterioration from freezing and thawing, dimensional stability, resistance to chloride ion penetration, and the effectiveness of the corrosion inhibitor. The specimen size, the test method, and the age at which the tests took place are summarized in Table 3. The results of laboratory tests for chloride permeability were compared with the results of tests of concretes containing

		Specimens	,	Age (days) Tested
Test	No.	Size (in)	Test Method	
Compressive strength	6	4 x 8	AASHTO T22	7, 28
Flexural steength	3	3 x 3 x 11 1/4	ASTM C78	28
Rapid permeability	2	4 x 2	AASHTO T277	28
Chloride content	2	12 x 12 x 3	AASHTO T259	2.5 yrs
Freeze-thaw	6	3 x 4 x 16	ASTM C666 ^a	a
Drying shrinkage	2	3 x 3 x 11 1/4	ASTM C157	b
Petrography	1	4 x 8	ASTM C457	28
Inhibiting properties	2	4.5 x 6 x 11	ASTM G109	с

NUMBER OF SPECIMENS AND TESTS FROM EACH BATCH

^a Some cured 2 weeks moist and 1 week dry and tested in 2% NaCl (modified ASTM C666), others cured 2 weeks moist and tested in water only (standard ASTM C666).

^bLength changes were determined at 4, 7, 14, and 28 days; and after 8, 16, and 32 weeks of drying at 50% RH subsequent to 28 days of curing.

^c Proposed ASTM test for evaluation of corrosion inhibitors in reinforced concrete.

slag or silica fume, which are known to have lower permeabilities than conventional concretes. Evaluations of the control concrete deck and the adjacent experimental concrete deck were made after the placement and then after the first and third winters.

RESULTS AND DISCUSSION

Freshly Mixed Concretes

The results of tests on air content and slump are summarized in Table 4 along with the w/c. The air content ranged from 5.6 percent to 7.5 percent and the slumps from 3.5 in to 5 in.

Hardened Concretes

Strength

Compressive strengths were determined in accordance with AASHTO T22 using 4- by 8-in cylinders and neoprene pads in steel end caps were used in lieu of

CHARACTERISTICS OF THE FRESHLY MIXED CONCRETE

Batch	w/c	Air Content (%)	Slump (in)
Control 1	0.44	5.6	3.5
Control 2	0.44	6.2	3.5
DCI 1	0.43	7.5	5.0
DCI 2	0.43	7.5	4.5

Table 5

	Compressive Strength (psi)		Flexural	Chloride	
Batch	7 day	28 day	Strength (psi) 28 day	(coulombs)	Absorption
Control 1	4,260	4,800	680	5,300 (High)	5.3
Control 2	4,090	5,260	755	5,210 (High)	5.3
DCI 1	5,650	7,170	730	7,590 (High)	5.4
DCI 2	5,910	7,180	720	7,530 (High)	5.2

STRENGTH AND CHLORIDE PERMEABILITY DATA

^a Specimens moist cured for 14 days, tested at 28 days.

capping. The flexural strengths were conducted in accordance with ASTM C78 using 3- by 3- by 11 1/4-in prisms. The test results given in Table 5 indicate satisfactory compressive and flexural strengths. The required minimum 28-day design strength of 4,500 psi at 28 days was exceeded in all cases. The concretes containing DCI developed higher compressive strengths than the controls. However, increases in flexural strengths were not evident.

Permeability and Absorption

The permeability of concretes to chloride ions were determined using both the rapid permeability test (AASHTO T277) and the 90-day ponding test (AASHTO T259). In the rapid permeability test, the charge passing through the specimen in a 6-hour period is determined and expressed in coulombs. These values are related to the chloride permeability. The cylindrical samples were moist cured for 2 weeks and then air dried until vacuum saturated and tested at 28 days. The results (given in Table 5) indicate that all the concretes had a high chloride permeability. The concretes with DCI had higher permeability values (but in the same high range)

Material		Depth			
	1/4 - 3/4 in	3/4 in - 1 1/4 in	1 1/2 - 2 in		
Control 1	21.4	10.5	2.5		
Control 2	18.2	10.2	1.2		
DCI 1	18.9	11.5	2.7		
DCI 2	16.3	10.4	0.8		
Slag 1	23.9	1.4	0.4		
Slag 2	9.3	0.8	0.0		
Silica fume 1	11.4	0.7	0.1		
Silica fume 2	10.8	1.6	0.0		

CHLORIDE CONTENTS IN LB/YD³ AFTER 2.5 YEARS

than the controls; this is attributed to the presence of calcium nitrite. Because of the ionic nature of this compound, the applicability of the rapid permeability test to concretes containing calcium nitrite is questionable.

In the ponding test, slabs were ponded with 3 percent NaCl for 2.5 years rather than the standard 90 days to provide sufficient time for chloride penetration. At the same time, a variety of concretes containing slag or silica fume were also subjected to ponding for comparison. These concretes were included because they are expected to exhibit low or very low chloride permeability. The slag concretes had half of the portland cement replaced with slag and had a water-cementitious ratio (w/c) of 0.39. The silica fume concrete had 7 percent silica fume by mass of portland cement added in addition to the portland cement at a w/c of 0.39. The chloride contents were determined at three depths, 1/4 to 3/4 in, 3/4 to 1 1/4 in, and $1 \frac{1}{2}$ to 2 in and the results are summarized in Table 6. All concretes has a high chloride content at 1/4 to 3/4 in depth. At this depth, high variability is expected because of the proximity to the textured salted concrete surface. As expected, chloride contents decreased at increasing depth. Both the control and the experimental concrete with DCI had high chloride contents, which exceeded the threshold value⁶ of 1.3 lb/vd³ at all depths except in two cases at the 1 1/2- to 2-in depth. The concretes with slag or silica fume had very low chloride contents at the 1 1/2 to 2-in depth in all cases. The values were also below or just slightly above the threshold value at the 3/4- to 1 1/4-in depth. At this depth, the chloride contents of the control or the experimental concretes were at least six times greater than that of the slag or silica fume concretes. Thus, it is apparent that the concretes in this bridge deck do not have as high a resistance to chloride ion penetration as would be provided with slag or silica fume concretes with low w/c. Epoxy coating or the calcium nitrite are expected to provide the necessary resistance against corrosion. The water absorption of concretes was determined in accordance with ASTM C642 using the boiling

procedure. The results given in Table 5 indicate that values are comparable for all batches, and all are above 5 percent, indicating high absorption.

Resistance to Freezing and Thawing

The resistance of concretes to damage from cycles of freezing and thawing was determined using ASTM C 666 Procedure A in which the beams are moist cured for 2 weeks and tested by freezing and thawing in water. In addition, specimens were tested with modifications to the ASTM procedure. In the modified procedure, a 2 percent NaCl solution was used as the test water and the curing period was extended by 1 additional week in a dry condition. These modifications are expected to simulate the actual field conditions more closely than the standard test. The results summarized in Table 7 indicated that all the specimens tested had a high resistance to damage from cycles of freezing and thawing and no significant difference between concretes containing DCI and the controls was apparent. The durability factors of specimens tested in water with two weeks of moist curing were less than the others, however the lowest value was still in the high 80's.

Batch WL (%) ^c		In Water ^a		In Salt ^b		
	DF	SR	WL (%)	DF	SR	
Control 1	0.7	90	1.2	0.6	96	1.0
Control 2	1.0	89	1.2	0.9	97	1.4
DCI 1	0.7	87	1.1	0.6	94	1.0
DCI 2	0.6	87	1.0	0.7	97	0.9

FREEZE-THAW DATA

^a Beams tested in accordance with ASTM C666 Procedure A. Tested in water after two weeks of moist curing.

^b Modified ASTM C666 Procedure A to include 2% NaCl in test water and one week additional dry curing.

^c WL = weight loss; DF = durability factor; SR = surface rating (ASTM C672 rating scale).

Petrographic Examination

The air-void system in the hardened concrete was determined in accordance with the linear transverse method of ASTM C457. The cylindrical specimens were moist cured at least a month, and a vertical slab was cut, lapped, and measured. The values for small, large, total voids, specific surface, and spacing factor are summarized in Table 8. For adequate protection of critically saturated concretes exposed to extreme conditions, specific surface values of 600 in²/in³ or more and spacing factor values of 0.008 in or less are generally required. All of the concretes tested met these criteria. They also had satisfactory resistance when subjected to cycles of freezing and thawing. Concretes containing DCI and the control concretes had similar air-void parameters.

AIR-VOID PARAMETERS OF HARDENED CONCRETE						
Batch	<1mm	>1mm	Total	Specific Surface (in ⁻¹)	Spacing Factor (in)	
Control 1	5.8	0.7	6.5	876	0.0048	
Control 2	5.6	2.5	8.1	617	0.0053	
DCI 3	5.8	2.4	8.2	631	0.0051	
DCI 4	5.9	0.8	6.7	843	0.0048	

Drying Shrinkage

The drying shrinkage of the concretes was determined using ASTM C157. Beams measuring 3 in by 3 in by 11 1/4 in with gage studs at both ends were moist cured for a month and then kept in laboratory air. The shrinkage values at 32 weeks and 2.5 years are summarized in Table 9. The results show that the drying shrinkage values were slightly lower in the experimental concretes, but the differences are not considered important.

DRYING SHRINKAGE DATA IN PERCENT				
32 Weeks	2.5 Years			
0.0563	0.0674			
0.0605	0.0725			
0.0567	0.0637			
0.0517	0.0587			
	32 Weeks 0.0563 0.0605 0.0567 0.0517			

Table 9

Effectiveness of the Corrosion Inhibitor

A test method (ASTM G 109) is being developed to provide a means of predicting the effectiveness of corrosion inhibitors. Tests were made using this proposed test method. Concretes were prepared using Type II cement and coarse aggregate with a maximum size of 0.5 in. The w/c was 0.50, and the cement content was 600 lb/yd³. Three batches of concrete were prepared. The control batch without the corrosion inhibitor had a slump of 2.5 in and an air content of 6.0 percent. The second batch contained 3.5 gal/yd³ of calcium nitrite and had a slump of 4.8 in and an air content of 6.4 percent. The third batch contained 5.4 gal/yd³ of calcium nitrite, and had a slump of 3.5 in and an air content of 5.6 percent. Specimens mea-

suring 11 in by 6 in by 4.5 in were fabricated with one reinforcing bar 1 in from the top and two bars 1 in from the bottom. The bars were deformed #4 bars, which were cleaned with sulfuric acid and wire brushed before the placement of the concrete. A 100 ohm resistor was placed between the top and bottom bars. Three standard test specimens were prepared from each batch of concrete. Three additional samples were prepared from the second batch with 3.5 gal/yd^3 of calcium nitrite. These additional samples were subjected to load so that cracks developed at the surface of the concrete. This was done to determine the level of effectiveness of calcium nitrite as a corrosion inhibitor in the presence of cracks. The width of the cracks ranged from 0.15 to 0.30 mm. It was difficult to control the width of the cracks with the test apparatus available, and most of the cracks happened to be larger than the maximum tolerable crack width of 0.18 mm when deicing chemicals are used (see ACI 224). However, cracks of this magnitude occur in our structures, and the effectiveness of DCI in inhibiting corrosion when such cracks are present needs to be known. The samples were ponded with 3 percent NaCl solution, which contained 3 parts of NaCl by mass in 97 parts of water. After 2 weeks the solution was removed, and the samples were allowed to dry for an additional 2 weeks. The voltage across the resistor at the beginning of the second week of ponding was measured, and the current was calculated. This cycle was repeated until a current of 10 µA or greater was obtained for the control samples. According to the test procedure, an average current of 10 μ A is indicative of corrosion. At this time, the test has been in progress for 2.5 years. However, only one control specimen has reached the 10 µA value, and the other two have negligible values. Thus, corrosion does not appear to be occurring. The uncracked specimens with 3.5 gal/yd³ and 5.4 gal/yd³ calcium nitrite also have negligible current values at 2.5 years.

Two of the cracked specimens with DCI had values in excess of 10 μ A from the beginning. After 2.5 years, they had reached 13 μ A and 17 μ A, indicating minimal change over the years. The third cracked specimen had an initial value of 4 μ A. It reached 10 μ A after 1.5 years, and it has a similar value after 2.5 years.

These results indicate that more time is needed to differentiate between the control and the DCI samples; they also show that when cracks are present, corrosion may occur even with DCI if the cracks are large enough to facilitate the penetration of chlorides into concrete.

Deck Evaluations

The first evaluation of the control and the experimental decks was made just after placement on June 15, 1988. The second evaluation was made after the first winter on May 25, 1989, and the third one was made after the third winter on June 18, 1991.

First Evaluation

The visual survey showed some cracks near the parapet at the west end of the deck paved with the control concrete. These were attributed to plastic

Evaluation	A		Ι	В		С	
	WBL	EBL	WBL	EBL	WBL	EBL	
First (1988)	0	0	98	99	2	1	
Second (1989)	0	4	99	96	1	0	
Third (1991)	—	0	—	100	_	0	

PERCENTAGE DISTRIBUTION OF HALF-CELL POTENTIALS

A - More positive than -0.20 V CSE, indicative of no corrosion.

B - In the range of -0.20 to -0.35 V CSE, presence of corrosion uncertain.

C - More negative than -0.35 V CSE, indicative of corrosion.

shrinkage cracking and they were filled with epoxy resin. No cracks were detected in the deck paved with the experimental concrete. There were no delaminations in either deck.

The average depth of cover in the deck containing the control concrete was 2.38 in with a standard deviation of 0.49 in. The deck containing the experimental concrete had a depth of cover of 2.84 in with a standard deviation of 0.45 in. There was no scaling on either of the concrete surfaces.

Electric half-cell potentials were obtained in accordance with ASTM C876 using 4-ft grids in the experimental span, which had uncoated bars. Since the control had epoxy-coated bars, potential values were not determined. Coatings interfere with the formation of the circuit needed to determine the potential values.

The values for the east bound lane (EBL) or the west bound lane (WBL) given in Table 10 are a percentage of the readings in each of the three specified ranges. In accordance with the ASTM methods, there are three ranges: (1) when readings are more positive than -0.20 V, there is a 90 percent probability that no corrosion of the reinforcing steel is occurring; (2) when the readings are between -0.20 V and -0.35 V, it is uncertain whether corrosion is occurring; (3) when the readings are more negative than -0.35 V, there is more than a 90 percent probability that corrosion is occurring. The values after the first evaluation showed a very small area with readings indicative of corrosion.

Second Evaluation

The second evaluation conducted after the first winter showed that a few fine cracks had developed in both the control and the experimental concretes. In the control span, there were two cracks at the transverse joints totaling 5.5 ft in length. In the experimental deck, there were seven cracks (four at the joints) totaling 14 ft. There were no delaminations or scaling in either span. Electrical half-cell potentials given in Table 10 show that areas with more negative values than -0.35 V were smaller than was shown after the first evaluation. None was found in the

CHLORIDE CONTENTS IN LB/YD³

Depth (in)	Control	DCI	
1/4 - 3/4	1.79	1.83	
3/4 - 1 1/4	0.81	0.54	
1.5 - 2.0	0.08	0.08	

EBL. Also, the location of more negative values in the WBL was in a different place than that found in the first evaluation.

Third Evaluation

After the third winter, a continuous longitudinal crack was observed in both the EBL and the WBL in both the control and the experimental spans; it is attributed to the load distribution over the deck and the girders. There were also a few cracks at the joints. The total length of cracks was about 71 ft for the control and 74 ft for the experimental span. There was no delamination or scaling in either span. The electric potentials were determined only for the EBL; they indicated that no corrosion was occurring.

At this time, chloride contents were determined at four locations along the EBL, two in the control and two in the experimental spans. One location was in the right wheel path, and the other was between the wheel paths. The chloride contents were determined at three depths, 1/4 to 3/4 in, 3/4 to 1 1/4 in, and 1 1/2 to 2 in, and the values at each depth were averaged for each concrete and are given in Table 11. The results are similar in both concretes and indicate a reduction in chloride content with depth. Only the depth nearest the surface had values that were in excess of the threshold level of 1.3 lb/yd³. At the 1 1/2- to 2-in depth, values were negligible because of the young age of the deck.

CONCLUSIONS

1. The properties of the hardened experimental concretes containing DCI are satisfactory and are similar to those of the control concretes. Compressive strengths were higher, but flexural strengths at 28 days were similar. The two concretes had equal resistance to damage from cycles of freezing and thawing, comparable drying shrinkage, and similar chloride permeability values when tested with the rapid permeability or the ponding tests. They also had similar absorption values.

2. After 2.5 years of ponding, all but one control specimen and all of the experimental concretes had very low current flow between the upper and lower bars,

which is indicative of the absence of corrosion. Cracked specimens with DCI are exhibiting currents indicative of corrosion, but values have been quite stable during the test period. The significance of this result is not yet clear.

3. The field performance of the control and the experimental bridge spans has been similar for the first 3 years. The amounts of chlorides penetrating into the decks are also similar. However, more time is needed to make definite conclusions regarding the long-term performance of the DCI inhibitor as a means of protecting against corrosion induced by chloride penetration.

RECOMMENDATIONS

The Department should continue the use of epoxy-coated reinforcing bars in bridge decks to protect against corrosion. The evaluation of protective systems containing corrosion inhibitors and uncoated bars should be continued to determine whether they can provide the same level of protection as the epoxy-coated systems and whether they would be cost-effective.

In critical areas where there is heavy traffic and large numbers of salt applications, the possibility of using combinations of more than one system (i.e., lowpermeability concretes containing slag or silica fume, corrosion inhibitors, and epoxy-coated bars) should be considered.

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