## Newly Developed Itaconic Acid Copolymers for Gypsum and Calcium Carbonate Scale Control

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**ABSTRACT:** To mitigate scale formation and the consequences of clogging of filters and the reduction in efficiencies in cooling systems, itaconic acid–maleic acid (Ita–MA) and itaconic acid–methacrylic acid (Ita–MAA) copolymers were developed. Calcium sulfate and calcium carbonate scales formation have been prevented at temperatures ranging from 50 to 80°C using the newly developed polymers. The effect of the polymers on their scale-arresting abilities has been assessed through chemical screening test, constant potential electrolysis, and A.C. impedance techniques. XRD (x-ray diffraction) and SEM (scanning electron microscope) studies have been performed to identify the change in crystal surfaces by the addition of the polymers. The amount of scale deposition is increased with increase in both the temperature and the pHs of the medium. The efficacy of the polymers (antiscalants) on the scale prevention is increased with increase in polymer dosage. However, decrease in efficiency is noticed with increase in temperature and pHs. The results, at different dosage levels with different temperature and pHs, are critically discussed. Both of the scales have been inhibited, through the adsorption of the polymers onto the growth sites. © 2006 Wiley Periodicals, Inc. J Appl Polym Sci 103: 3206–3213, 2007

**Key words:** antiscalant; constant potential electrolysis; FT-IR; AC impedance spectroscopy; chemical screening test; SEM and XRD studies

## **INTRODUCTION**

The problem of scaling associated with the use of natural hard waters in cooling systems poses great challenges from both economical and technical points of view. The commonly occurring scales are calcium carbonate, calcium sulfate, calcium phosphate, calcium oxalate etc., and among them calcium carbonate is considered the most prominent.<sup>1–4</sup> Scale formation mainly depends on temperature, pH, pressure of the system, flow velocity, and mineral concentration of the cooling water. The scale gets deposited on the heat exchanger surfaces and offers a resistance to the flow of heat. These deposits can accumulate in pipelines, orifices and other flow passages to the extent that even the free flow of the process fluids is seriously impeded.<sup>5–8</sup>

For the control of scale formation, many authors have proposed acidification, the use of chelants, use of scale conditioners, or the addition of threshold inhibitors.<sup>9,10</sup> A copolymer made by the polymerization of sodium-2-methyl 1,3-butadiene sulfonate acrylic acid and methoxy polyethylene glycol monomethacrylate has been evaluated for its efficiency on scale control in cooling and boiler water systems.<sup>11</sup> 2-Phosphonobutane-1,2,4-tricarboxylic acid or acrylic acid–metha-

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crylic acid copolymer has been reported by Zhiliang et al.<sup>12</sup> to remarkably inhibit the scales. Copolymer of hydroxy carboxylic acid and epoxy succinic acid was employed to reduce the scale deposit in cooling waters.<sup>13</sup> Vinyl formamide or N-vinyl acetamide copolymer with methacrylic acid, acrylamide, or acrylamine was used to inhibit the scales in boiler waters.<sup>14</sup> In severely scaling water, HEDP (Hydroxy ethylidene-1,1-diphosphonic acid) and polyacrylic acid were suggested as potential antiscalants.<sup>15</sup> Terpolymer of isopentyl phosphonic acid, acrylic acid, and 2-hydroxy propyl acrylate was tried as the inhibitor to inhibit the CaCO<sub>3</sub> scale in cooling water systems.<sup>16</sup> The reaction products of phenoxy ethyl arylates with acrylic acid and 3-allyl oxy-2-hydroxy propane sulfonic acid were tried as the antiscalant.<sup>17</sup> Copolymer of maleic anhydride-acrylic acid, phosphorous containing compounds, and peroxides were examined for their action to prevent scaling.<sup>18</sup> Itaconic acid acrylic acid-methacrylic acid terpolymer has been reported as scale inhibitor for higher pHs.<sup>19</sup> To prevent the precipitation of Ca, Mg, Ba, Cu, Fe, Mn, and Zn salts, HEDP and DETPMP (diethylene triaminepenta (methylene phosphonate)) where added into the water systems.<sup>20,21</sup> For the prevention of calcium carbonate and calcium sulfate scales vinyl acetate-acrylic acid, vinyl acetatemethacrylic acid and acrylonitrile-acrylic acid and acylonitrile methacrylic acid copolymers were tried in cooling water systems.<sup>22,23</sup> For the past five decades, low molecular weight polymers have been applied in

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cooling systems to prevent scaling. The present study aims to develop itaconic acid copolymers for cooling water systems to block the scale formation in severely scaling water. The efficiency of the polymers for both calcium carbonate and calcium sulfate scale control at different temperatures [50–80°C] and pH [7.0–8.5] ranges with different dosage levels were studied. Their efficiency was assessed through chemical and electrochemical methods. The change in crystals habits were examined through surface examination techniques like XRD and SEM.

## MATERIALS AND METHODS

The monomers and the required chemicals purchased were of AR grade. The itaconic acid was copolymerized with maleic acid/methacrylc acid by keeping the monomer feed ratio at 1 : 1 using potassium peroxodisulphate (PDS) as initiator. Synthesized copolymers were characterized through FT-IR study.

#### **Evaluation of antiscalants**

# Chemical screening test and electrochemical techniques

Chemical screening tests were performed as a relative and quantitative measure of the antiscalant ability to inhibit the precipitation of mineral salts from hard water, using the NACE standard procedure.<sup>24</sup> CaCO<sub>3</sub> brine solution (300 ppm) was taken and different dosage levels of the antiscalants (1–20 ppm) were added to different carbonate brine solutions and maintained at appropriate temperatures for 24 h for digestion. The tests were carried out in the pH range of 7.0–8.5. The same procedure was adopted for calcium sulfate scale inhibition tests, but in this case the hardness of the brine was maintained at 2000 ppm.

Electrochemical techniques such as constant potential electrolysis and AC impedance techniques were used to assess the ability of the polymers on the CaCO<sub>3</sub> scale control. EG and G PAR VersaStat II was used to carry out the constant potential electrolysis with platinum foils as working as well as counter electrodes and saturated calomel electrode (SCE) as the reference electrode. A 300-ppm of calcium carbonate brine was taken for this study and different dosage levels of the polymers were added to it. This test was performed by applying -1.2 V Versus SCE to the working electrode.

The electrochemical impedance study was performed using EG and G PAR, model 398 electrochemical impedance analyzer. The same procedure mentioned above for the constant potential electrolysis was used for EIS studies. Measurements were made over a frequency range of 100 kHz–100 mHz with 5 mV AC amplitude superimposed over the steady state potential of -1.2 V (Versus SCE) at the working electrode. The morphological changes of the calcium carbonate and calcium sulfate scales due to the addition of antiscalants were examined through scanning electron microscope (SEM) and x-ray diffraction (XRD) studies. The scales for the surface examinations were precipitated on a glass plate with and without the presence of the polymers. Gold coating was applied to crystal surface if needed. JOEL instruments were used for the surface examination studies.

The iron dispersing ability, gelation, and biocidal properties of the polymers at different experimental conditions were evaluated following the description of Shakkthivel et al.<sup>22</sup>

#### **RESULTS AND DISCUSSION**

#### Characterization of Ita-MA and Ita-MAA polymers

The viscosities of the polymers were measured using Efflex viscometer. The viscosities of the Ita-MA and Ita–MAA polymers are 1.3 and 2.8 cps respectively. These values indicate that the polymers are of low molecular weights. The FT-IR spectra for the polymers Ita-MA and Ita-MAA are shown in Figures 1 and 2. In the Ita-MA polymer, the peaks appearing at 1690 and 1660  $\text{cm}^{-1}$  correspond to CH<sub>2</sub>-COOH stretching vibration and C-COOH asymmetric stretching vibration respectively, and the peak at 1550 cm<sup>-1</sup> is assigned for the symmetric vibration of C-COOH group. The CH<sub>2</sub> scissoring deformation vibration peak appears at  $465 \text{ cm}^{-1}$ . In the case of Ita–MAA polymer, the peaks at 1690 and 1650 cm<sup>-1</sup> correspond to  $CH_2$ -COOH stretching vibration and asymmetric stretching vibration of C-COOH groups respectively, present in the itaconic acid. A sharp peak appearing at 1464 cm<sup>-1</sup> is due to the CH<sub>3</sub> scissoring deformation vibration. The peak at 1404 cm<sup>-1</sup> is assigned for C-CH<sub>3</sub> bending vibration present in the methacrylic acid.<sup>25,26</sup>



Figure 1 FT-IR spectrum of itaconic acid–maleic acid copolymer (Ita–MA).

Figure 2 FT-IR spectrum of itaconic acid-methacrylic acid copolymer (Ita-MAA).

## Evaluation of the antiscalants

## Chemical screening test

Chemical screening test results of the itaconic acid copolymers on the inhibition of calcium carbonate scale at different temperatures and pHs are shown in Tables I and II. In the case of Ita-MA polymer, it is observed that at 10 ppm dosage for pHs 7.0 and 7.5 at 50°C, there is 100% efficiency. With increase in pH and temperature, the efficiency is seen to fall to 61% at the highest pH of 8.5 and at the temperature 80°C. With 20 ppm concentration at pH 8.5 and temperature 50°C, 100% efficiency is obtained. This best performance is also observed with pH 7.5 upto 70°C. With increased temperature and pH beyond this, the efficiency is found decrease to 73%.

With Ita–MAA polymer, 10 ppm dosage at 50°C, the efficiency is found to be 100% for all pHs. Also, the same trend is noted for pH 7.0 and 7.5 for all the temperatures. Above pH 8.0, the efficiency is found to decrease to 90% at 80°C. From these, it may be concluded that Ita-MAA > Ita-MA as far as their antiscaling efficiency is concerned.

Tables III and IV present the chemical screening test results for the polymers Ita-MA and Ita-MAA on the calcium sulfate scale inhibition. With Ita-MA copolymer, there is 100% efficiency with 10 ppm concentration for pH 7.0 at all temperatures. With increase in temperatures along with pHs, the efficiency is decreased. For 20 ppm concentration, the best performance is retained upto 7.5 pH. With increased temperature and pHs, the efficiency is found to decrease. With Ita–MAA polymer, for 10 ppm dosage upto 70°C for all the pHs, 100% efficiency is obtained. For 80°C, this best

TABLE I Antiscaling Efficiency of Ita-MA Copolymer on Calcium Carbonate Scale (300 ppm as Calcium Hardness) at Different Temperatures and pHs Through Chemical Screening Test

Percentage efficiency

Dosage

TABLE II Antiscaling Efficiency of Ita-MAA Copolymer on Calcium Carbonate Scale (300 ppm as Calcium Hardness) at Different Temperatures and pHs Through Chemical Screening Test

Dosage

Percentage efficiency

pH 8.5

	Temp	level						Temn	level			
S. No.	(°C)	(ppm)	pH 7.0	pH 7.5	pH 8.0	pH 8.5	S. No.	(°C)	(ppm)	pH 7.0	pH 7.5	pH 8.0
1	50	Blank	_	_	_	_	1	50	Blank			
2		1	60	58	54	51	2		1	60	57	52
3		2	81	80	76	70	3		2	82	78	83
4		5	94	92	89	87	4		5	98	95	91
5		10	100	100	96	90	5		10	100	100	100
6		20	100	100	100	95	6		20	100	100	100
1	60	Blank	-	_	_	-	1	60	Blank	-	_	-
2		1	56	54	52	51	2		1	61	46	47
3		2	78	75	74	66	3		2	74	71	79
4		5	93	90	83	85	4		5	97	92	87
5		10	100	98	93	88	5		10	100	100	99
6		20	100	100	97	93	6		20	100	100	100
1	70	Blank	_	_	_	_	1	70	Blank	_	_	-
2		1	53	51	50	47	2		1	56	43	51
3		2	76	72	71	63	3		2	69	68	74
4		5	89	86	84	82	4		5	96	93	88
5		10	97	95	90	86	5		10	100	100	97
6		20	100	98	95	89	6		20	100	100	100
1	80	Blank	-	_	_	-	1	80	Blank	-	_	-
2		1	26	25	20	18	2		1	45	41	30
3		2	54	49	45	42	3		2	62	70	61
4		5	67	61	58	53	4		5	90	90	39
5		10	93	69	65	61	5		10	100	100	93
6		20	85	80	78	73	6		20	100	100	100



TABLE III
Antiscaling Efficiency of Ita-MA copolymer on Calcium
Sulphate Scale (2000 ppm as Calcium Hardness) at
Different Temperatures and pHs Through
Chemical Screening Test

		Dosage	Percentage efficiency						
S. No.	l'emp. (°C)	level (ppm)	pH 7.0	pH 7.5	pH 8.0	pH 8.5			
1	50	Blank							
2		1	50	40	39	35			
3		2	59	52	48	44			
4		5	65	64	60	58			
5		10	100	99	98	95			
6		20	100	100	100	98			
1	60	Blank	_	_	_	_			
2		1	45	37	36	32			
3		2	54	50	47	43			
4		5	65	63	59	58			
5		10	99	97	95	94			
6		20	100	100	97	95			
1	70	Blank	_	_	_	_			
2		1	44	37	36	32			
3		2	53	50	47	43			
4		5	64	61	59	58			
5		10	99	96	94	92			
6		20	100	100	98	96			
1	80	Blank	_	_	_	_			
2		1	42	37	35	30			
3		2	51	48	45	42			
4		5	62	59	57	54			
5		10	98	95	93	91			
6		20	100	100	97	94			

performance is retained upto 7.5 pH only. Whereas with 20 ppm dosage, 100% efficiency is obtained at all pHs and temperatures of the study.

Both the polymers at higher pHs and temperatures lose their inhibition ability. This is due to the scale formation tendency being very high at higher pHs and temperatures. The degree of scaling mainly depends on the levels of calcium hardness and bicarbonate alkalinity in the cooling waters. With increase in pH and temperature, the rate of bicarbonate breakdown increases. Higher alkalinity lowers the saturation point for most of the scale forming minerals. Thus scales like calcium carbonate and calcium sulfate are precipitated out more rapidly even at lower concentration of the minerals as the pH level of the water rises. Thus, while increasing these parameters, scaling will be more.

## Electrochemical techniques

The results for constant potential electrolysis at optimum pH of 8.0 without and with five different dosage levels of the polymer are presented in Figures 3–5. Scaling time is defined as the time required for the full coverage of the electrode surface with the insulating scale. The other important parameter, residual current, is related to the nature of the deposit. The scaling time for control (Blank) 662 s and residual current 34.43  $\mu$ A are presented in Figure 3. The scaling time and residual

TABLE IV Antiscaling Efficiency of Ita–MAA Copolymer on Calcium Sulphate Scale (2000 ppm as Calcium Hardness) at Different Temperatures and pHs Through Chemical Screening Test

		Dosage	F	ercentage	e efficienc	у
S. No.	Temp. (°C)	level (ppm)	pH 7.0	pH 7.5	pH 8.0	pH 8.5
1	50	Blank				
2		1	73	65	59	55
3		2	95	89	84	81
4		5	100	100	99	97
5		10	100	100	100	100
6		20	100	100	100	100
1	60	Blank	_	_	_	-
2		1	73	65	59	55
3		2	89	89	84	81
4		5	99	94	92	90
5		10	100	100	100	100
6		20	100	100	100	100
1	70	Blank	-	-	-	-
2		1	71	65	57	53
3		2	92	87	80	78
4		5	94	90	88	85
5		10	100	100	100	100
6		20	100	100	100	100
1	80	Blank	-	-	-	-
2		1	69	55	54	51
3		2	88	81	77	76
4		5	91	87	85	83
5		10	100	99	96	96
6		20	100	100	100	100

current values are increased with increase in the addition of the polymer dosage upto 1685 s. and 142.90  $\mu$ A for Ita–MA and 1672 s. and 151.00  $\mu$ A for Ita–MAA at higher dosage of 20 ppm respectively, as shown in Figures 4 and 5. In the presence of the polymer additives, the growth behavior of the scales are getting delayed or retarded. As a consequence, the time required for full coverage of the scale on the electrode is increased.

Figures 6–8 are for the impedance spectra recorded at different dosage level of the polymers Ita–MA and Ita–MAA in 300 ppm hard brine at pH 8.0. The electrode potential was kept at -1.2 V Versus SCE for about 45 min. The spectra were run with an AC



**Figure 3** Current-time curve for calcium carbonate brine containing 300 ppm of  $Ca^{2+}$  ions without antiscalants obtained through constant potential electrolysis.

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**Figure 4** Current-time curve for calcium carbonate brine containing 300 ppm of  $Ca^{2+}$  ions with the addition of Ita-MA obtained through constant potential electrolysis, (a) blank (b) 1 ppm (c) 2 ppm (d) 5 ppm (e) 10 ppm (f) 20 ppm.

voltage of amplitude 5 mV in the frequency range of 100 kHz-100 mHz after the indicated time lapse. After the complete formation of the scale on the electrode surface, a single semicircle was obtained from which the Rt values and the capacitance of the diffusion controlled oxygen reduction on the scale surface can be calculated. The capacitance and charge transfer resistance for the control are 0.0032  $\mu$ A/cm<sup>2</sup> and 1681  $\Omega$ / cm<sup>2</sup> respectively. This very high resistance value is due to the progressive coverage of the calcium carbonate deposition on the metal surface and thus the oxygen reduction limiting current values are decreased. With the addition of the polymers, the charge transfer resistance and capacitance values drastically varied to 17  $\Omega/cm^2$  and 3.8  $\mu$ F/cm<sup>2</sup> with 99.00% inhibition for Ita–MA and 13.54  $\Omega/cm^2$  and 7.40  $\mu$ F/cm<sup>2</sup> with 99.19% for Ita-MAA at the highest polymer dosage level of 20 ppm. On the addition of the polymer in the hard brine that are capable of retarding or modifying the calcareous deposit formation, no or less deposit formation or delayed deposit formation or sometimes less adherent deposit results. Thus the Rt and Cdl values are decreased very much when compared with control with the addition of polymers. The increased scaling time and decreased Rt values upon the addition of antiscalant in the calcium carbonate brine are due to



Figure 6 Nyquist plot obtained after calcium carbonate scaling on the platinum electrode immersed in calcium carbonate brine containing 300 ppm of  $Ca^{2+}$ ions.

the progressive coverage of the scale  $(CaCO_3)$  formation being delayed/retarded. Initially, the nucleated  $CaCO_3$  sites are covered/wrapped by the antiscalant and prevent further scale growth (nucleation growth). And thus, both calcium carbonate and calcium sulfate scales are controlled from the scale deposit by the polymer addition.

#### Surface examination techniques

The effect of the polymeric additives on the crystals of calcium carbonate and calcium sulfate scales are examined through scanning electron microscope. Calcium carbonate normally crystallizes from aqueous solution in three forms, i.e., calcite, aragonite, and vaterite. In the present study, cubic calcite crystals are seen. Magnesium ions ( $Mg^{2+}$ ) does not have any influence on the distortion of cubic structure of the calcium carbonate scale even though it is present in the tested brine. It is evident from Figure 9(a) without the presence of the polymers. Figure 9(b,c) show the effect of the polymers Ita–MA and Ita–MAA on the calcite crystal and the crystal nature was not much affected.

Calcium sulfate crystallizes into three forms depending on the water composition and evaporation temperatures, like  $CaSO_4 \cdot 2H_2O$  dihydrate (gypsum),  $CaSO_4 \cdot 1/2$  H<sub>2</sub>O hemihydrate (plaster of Paris), and



**Figure 5** Current-time curve for calcium carbonate brine containing 300 ppm of  $Ca^{2+}$  ions with the addition of Ita-MAA obtained through constant potential electrolysis, (a) blank (b) 1 ppm (c) 2 ppm (d) 5 ppm (e) 10 ppm (f) 20 ppm.

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**Figure 7** Electrochemical impedance spectrum for calcium carbonate scale in carbonate brine with different concentrations of Ita–MA copolymer, (a) 1 ppm (b) 2 ppm (c) 5 ppm (d) 10 ppm (e) 20 ppm.



**Figure 8** Electrochemical impedance spectrum for calcium carbonate scale in carbonate brine with different concentrations of Ita–MAA copolymer, (a) 1 ppm (b) 2 ppm (c) 5 ppm (d) 10 ppm (e) 20 ppm.

anhydrite CaSO<sub>4</sub>. The SEM photographs of CaSO<sub>4</sub> crystals grown on the glass plates at 70°C without any additive are presented in Figure 10(a) and the crystal habit corresponds to dihydrate crystals.<sup>27,28</sup> Calcium sulfate dihydrate crystals are reported as thin tubular cells and needles exhibiting monoclinic symmetry. In the presence of the polymer, the crystal growth is reduced as shown in Figure 10(b,c). The better performance of Ita–MAA over Ita–MA is evident from the extent to which the polymer is able to arrest the regularity of the crystal growth.

The XRD figures of  $CaCO_3$  and  $CaSO_4$  scales deposited from the respective brine without and with the presence of the Ita–MAA polymer are shown in Figures 11(a,b) and 12(a,b). In the case of calcium carbonate scale, the scale is calcite crystal and is confirmed



**Figure 10** (a) SEM photograph of calcium sulfate crystal at ×1000 magnification (b) SEM photograph of calcium sulfate crystal with the addition of Ita–MA copolymer at ×1000 magnifications (c) SEM photograph of calcium sulfate crystal with the addition of Ita–MAA copolymer at ×1000 magnifications.

through the 'd' and ' $\theta$ ' values. For calcium sulfate scale, the structure is proved to be CaSO<sub>4</sub>·2H<sub>2</sub>O.<sup>29</sup> The 'd' and ' $\theta$ ' values of CaCO<sub>3</sub> and CaSO<sub>4</sub> scales are in conformity with the reported values. In both the cases, with the polymer addition the crystal structure has not been altered; only the morphology is changed and this is



**Figure 9** (a) SEM photograph of calcium carbonate crystal at ×1000 magnification (b) SEM photograph of calcium carbonate crystal with the addition of Ita–MA copolymer at ×1000 magnification (c) SEM photograph of calcium carbonate crystal with the addition of Ita–MAA copolymer at ×1000 magnification.



Figure 11 XRD spectrum (a) for calcium carbonate (b) in presence of Ita–MAA.



Figure 12 XRD spectrum (a) for calcium sulfate (b) in presence of Ita–MAA.

confirmed by the variation in the intensity values and no change in the 'd' and ' $\theta$ ' values compared with that of the blank.

Iron dispersing ability and gelation property

Iron dispersing ability of the polymers was tested<sup>22,30,31</sup> and it was found that Ita–MAA polymer showed very good iron dispersing ability. If the polymer possesses a good dispersing ability for iron ion and is capable of keeping them in solution, the cooling water system can be saved from the corrosion problems. Calcium-polymer gelation test was performed<sup>22,32</sup> and the results showed that both the polymers have the ability to complex with Ca<sup>2+</sup> ions. However, this is not an encouraging trend because the polymer requirement will be increased slightly.

Cooling water systems are attractive locations for microbial colonization and biofilm formation. The growth of the algae leads to corrosion and blocking the cooling systems.<sup>22,33,34</sup> The micro biocidal property of the antiscalants was tested in raw water that contained 14 different major algaes. Among the two polymers, Ita–MAA polymer helps the growth of *Cocochloris* and almost complete inhibition on *Naviculla*, *Ulathrix*, and *Oscillatoria* were found. Biocidal properties of the antiscalants were studied through conventional Agar diffusion method and serial dilution technique for the mixed culture. Both the polymers have the ability to mitigate the biocides growth slightly. This result is an added advantage for the use of these polymers in the cooling water systems.

## CONCLUSIONS

Copolymers, Ita–MA, and Ita–MAA have been tried as potential antiscalants and evaluated through chemical and electrochemical techniques. In the chemical method, the percentage of inhibition is quantitatively calculated for different dosage levels of the polymers. At higher pHs and temperatures, the efficiency of the polymers decreased for both calcium carbonate and calcium sulfate scales. Calcium carbonate crystal growth has been blocked by the polymers and is evident from the decreased Rt, iR values, and increased ts, Cdl values in the electrochemical techniques. XRD and SEM studies show the crystal structure have not been altered with the addition of the polymers. Both the polymers have the ability to complex with  $Ca^{2+}$  ion and Ita-MAA polymer complexes with iron ion. The biocidal property of the polymers is an added advantage. Of the two polymers, Ita-MAA polymer is seen to be better than Ita-MA polymer.

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