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# Evolution of Space Charges and Conductivity with DC Aging of Polyethylene-Synthetic and Natural Clay Composites

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## ABSTRACT

The evolution of the space charge density and dc conductivity with dc poling of two types of polypropylene-based nanocomposites containing different concentrations of nanofillers were investigated. The two nanofillers were natural and synthetic organoclays. Determining the optimal concentration of nanofiller within the matrix of polypropylene (PP) can have a positive impact on space charge mitigation with aging time. It was observed that the optimal percentage was 2-wt% for PP-Natural Clay composite and between 2 and 4 wt% for the PP-Synthetic Clay. Above these percentages, charge transport through overlapping of nanoparticles can occur. This fact is due to the interaction zone of double layers formed at the nanoparticle/host material interfaces. Under dc field the overlapping increases the conductivity of polymer nanocomposite (PNC), which could minimize the benefit of incorporating nanofillers into PP. The total charge stored in unfilled PP increased continuously with time, reaching a maximum around 6000 h before decreasing. However, the total charges in all filled specimens only changed slightly. The conductivity with -25 kV/mm dc field of the specimens containing 6-wt% of natural clay and 8-wt% of synthetic clay reached  $\approx 6$  times the level for unfilled PP. This observation could be related to crossing the percolation threshold of these composites. The effect of platelet size on space charge mitigation reported by other authors is confirmed here, i.e. nanofillers with smaller platelets can mitigate space charge more efficiently than nanofillers with larger platelets. In addition to that more than 500 h of aging (with -25 kV/mm DC field) is required before correctly evaluating the impact of filling the PP with nanoparticles.

Index Terms – Polypropylene Nano-Composites (PNC), dc field conductivity, space charge evolution, PEA technique, Natural and Synthetic Organoclay.

## I INTRODUCTION

Polymers such as polypropylene (PP) are extensively used as electrical insulation in power capacitors, cable wraps, and phase separators for

rotating electrical equipment. The impact of filling polymers with a small quantity of nanoparticles is currently a most active subject of research for

universities, research centers and industrial institutions. The main objective of these activities is to develop the next high performance industrial materials with superior properties as compared to unfilled polymers. This could only be realized if the resulting polymer nanocomposites (PNCs) possess as many as possible of the following features: more discharge endurance, better thermal and mechanical properties, lower quantity of accumulated space charges, longer lifetime, etc...

It was reported that the mechanical and thermal properties of polypropylene (PP) [1-3] and Epoxy resin [4] were improved after being loaded with a small weight percentage (wt%) of organoclay. The electrical breakdown strength, surface and volume resistivity to erosion and Partial Discharges (PD) were superior to the unfilled materials [5-8]. It was also stated that the Polyamide-aluminum-nitride composites are more tolerant to high working temperatures [9] and the quantity of space charges was substantially reduced by adding a few wt% of nanoparticles to the polyethylene and polypropylene [10-12]. In addition to that, polymer materials can provide new functions such as gas barrier and flame resistance [13, 14] due to the effects of clay dispersion. Therefore, more understanding of the real impact of filling polymers with nanoparticles will help in obtaining the best possible desired dielectric properties for designing better equipment for high voltage applications.

It seems that the clay platelets limit the molecular motion in the amorphous phase because of the interaction between the platelets and PP matrix [15]. In other words the interface between nanoparticles and neighbouring materials can control the carrier transport and the electric transport properties, which are closely related to the charge injection, retention and bulk dynamics [16]. Nanoparticles can change the depth and density of traps, which in turn change the density and mobility of space charge. Small quantities of nanoparticles could mitigate charge injection (or charge trapping) and slow down material degradation [17]. However, excessive quantities of nanofiller could lead to losing this advantage. This could be related to the overlapping of nanoparticles after crossing the material percolation threshold [18].

In this work two types of organoclay were incorporated in the isotactic polypropylene (PP): Topy synthetic tetrasilic mica from Topy Co., Ltd. and Cloisite®20A powder of Wyoming natural montmorillonite clay from Southern Clay Products. The concentration of nanoparticles was between 1-wt% and 8-wt%. The evolution of space charge

density and conductivity during more than 11000 h of dc poling at -25 kV/mm will be described. This could lead us to define the optimal concentration of nanoclay within the PP that can mitigate of charges and could be the base of developing new insulation materials with high performance for electrical applications.

## II. EXPERIMENTAL

The tested specimens were composed of two sets of PNCs. The first set was composed of an isotactic PP (Profax HL-451H from Basell) filled with different wt% of Topy synthetic tetrasilic mica from Topy Co., Ltd. of Japan. The second set was composed of the same PP material but filled with a different quantity of Cloisite®20A natural montmorillonite clay from Wyoming. The first set was prepared for a collaboration project between the Industrial Materials Institute (IMI) and the Institute for National Measurement Standards (INMS) of the NRC to develop nanocomposite insulations for high voltage capacitor applications. The second set was manufactured for a project within the VAMAS Technical Working Area 33 (TWA-33) concerned with the development of test methods for chemical, morphological, mechanical and electrical characterization techniques for polymer nanocomposites. VAMAS is an international organization that supports world trade in high technology [19].

The percentage of nanofillers, the composition and names of each type of specimens used in this paper is given in detail in Table 1. The two PP host materials PP1-0% and PP2-0% only differ from each other by the percentage of compatibilizers (17.4% and 12.8% respectively). The effect of compatibilizers on some properties of PNCs was reported in [15]. The filled specimens are referred to in this paper as "PP1+Sx%" if they contain synthetic clay and "PP2+Ny%" if they contain natural clay. x% and y% indicate the percentage by weight content of Topy and Cloisite®20A organoclay, correspondingly.

The production of nanocomposites involved three steps: (1) preparation of the Master Batch (MB), (2) dilution of MB, and (3) preparation of films. These three steps were thoroughly explained in [20].

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The individual platelets' aspect ratios were around 6000 and 286 for synthetic and natural clay, respectively [21].

Space charge measurements were performed using the Five Labs PEA measurement system having a resolution of  $\sim 15 \mu\text{m}$  and Tektronix 7404, 4 GHz, 20 GS/s digital oscilloscope. The dc conductivity was measured with a Keithley 6517B electrometer.

**Table 1.** PNC compositions for both sets of tested specimens. Synthetic: for NPC filled with nanoclays from Topy and Natural: for PNC filled with Cloisite®20A.

Given name	PP (%)	Anti-Oxidants (%)	Copatibilizer (%)	Nanoclay	
				(%)	Type
PP1-0%	82.4	0.2	17.4	0	—
PP2-0%	87.0	0.2	12.8	0	—
PP1-S1%	81.6	0.2	17.2	1	Synthetic
PP1-S2%	80.7	0.2	17.1	2	Synthetic
PP1-S4%	79.1	0.2	16.7	4	Synthetic
PP1-S8%	75.8	0.2	16	8	Synthetic
PP2-N2%	85.3	0.2	12.5	2	Natural
PP2-N6%	81.8	0.2	12	6	Natural

During the PEA and conductivity tests, a thin layer of silicon oil was applied on each side of the sample to ensure a good contact between the specimen and the electrodes. A 0.2 mm thick, 30 mm diameter semicon disk was inserted between the specimen and the upper brass electrode during dc poling. Specimen poling and conductivity measurements were performed at the same field, -25 kV/mm dc. However, the PEA measurements were performed at 4 kV/mm and performed first on fresh specimens (0 h of dc poling) to be used as reference, and then after 168 h, 336 h, 1008 h, 2016 h, 3024 h, ... 11088 h of poling time.

Up to three specimens were tested for each condition and the average of the conductivity and the total charge (Q) between both electrodes was performed to calculate the total charge Q. The value of the total charge was the integral of the absolute value of charges in the specimens' volume, i.e. between both electrodes.

### III. RESULTS AND DISCUSSION

#### A. Space Charge Distribution

The figures representing the distribution of space charge depicts the cathode, to which the -25 kV/mm dc

field was applied, on the right side and the anode on the left side.

The double injection of charges (homocharges) is clearly observed in Fig. 1 for the unfilled material PP1-0%. The quantity of injected charge is higher from the cathode (the electrode where the voltage is applied) compared to anode (ground). The injected charge peaks increase with poling time and move deeper towards the central area of the specimen. This in turn leads to the increase of the local electric field in the sample's bulk. It seems that the charge injection is stabilized and even tends to decrease after 5040 h of aging.

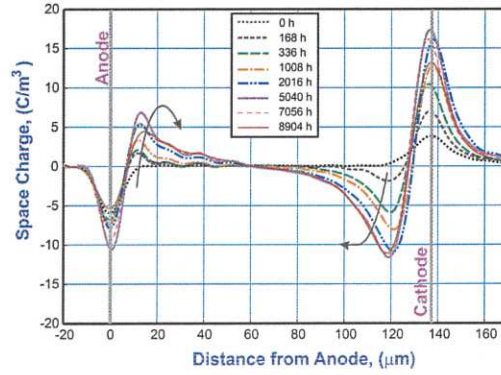


Fig. 1. Effect of the poling time at -25 kV/mm dc field on space charge distributions in PP1-0%.

Just by loading PP1-0% with 1-wt% of synthetic clay (PP1-S1%), the value and profile of the charges dramatically change, see Fig. 2. The injected charges from the cathode side substantially decreased compared to the unfilled material and the majority of

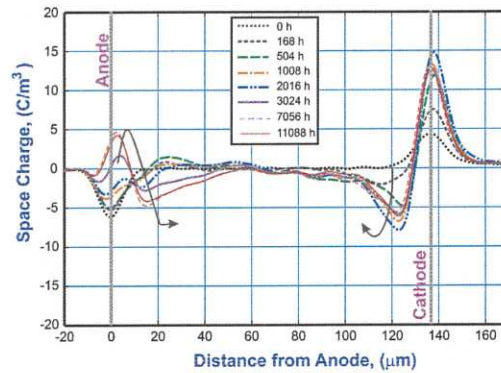


Fig. 2. Effect of the poling time at -25 kV/mm dc field on space charge distributions in PP-S1%.



the homocharges in the vicinity of anode were mitigated and replaced by heterocharges. With aging time the homocharges were substantially mitigated and a small quantity of heterocharges are still located in the vicinity of anode.

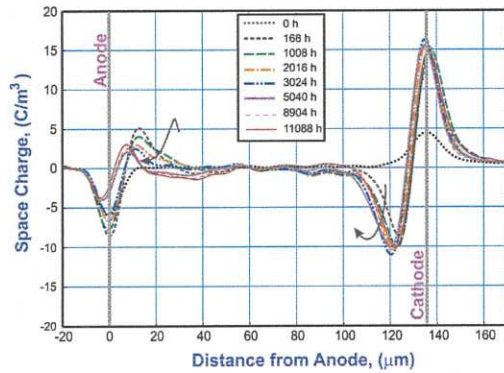


Fig. 3. Effect of the poling time at -25 kV/mm dc field on space charge distributions in PP-S2%.

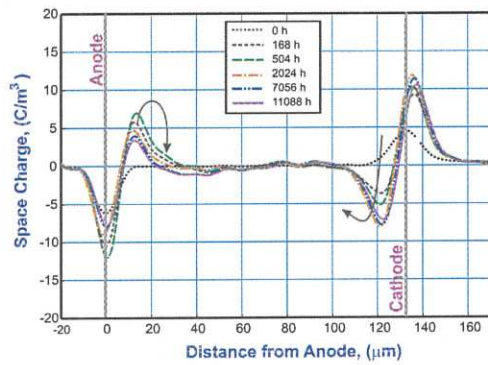


Fig. 4. Effect of the poling time at -25 kV/mm dc field on space charge distributions in PP-S4%.

Two-wt% of synthetic clay was able to mitigate the majority of charges in the central zone of the specimens so called PP1-S2%, see Figure 3. The amplitude space charge peak near the anode decreased with poling time for PP1-S2% while it increased for the PP1-0%. A small increase of homocharge close to the cathode is observed. This is illustrated by the arrow in Fig. 3. The low quantity of charge in the central zone is only slightly changed after 11088 h of dc poling.

Increasing nanofiller content to 4-wt% further decreases the amount of charge, especially in the central area (Fig. 4).

When the PP1-0% is loaded with 8-wt%, sample PP1-S8%, significant charges were observed to build up in the central area, see Fig. 5. It was previously reported that the concentration of charges in the central zone was a sign of degradation of the specimens' condition [22]. Hence, from the results shown in all the figures above, the optimal percentage for the synthetic filler seems to be between 2 and 4-wt%.

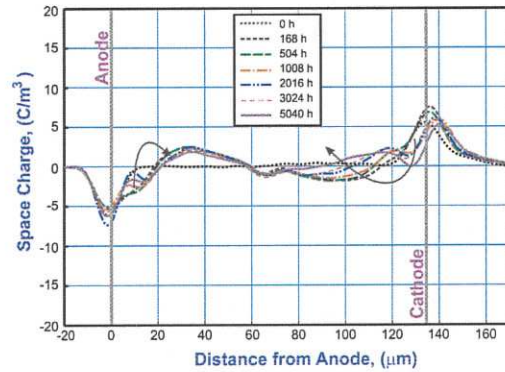


Fig. 5. Effect of the poling time at -25 kV/mm dc field on space charge distributions in PP-S8%.

Figure 6 shows the space charge distribution in the second PP base material, PP2-0%. This figure demonstrates the anti-symmetric peaks of homocharges built-up from both electrodes. The amplitude of these peaks increases and advances deeper into the specimen bulk. After 3024 h of aging the amplitude decreases, but the advance towards the central area continued. Since the two types of PP only differ from each other by the percentage of compatibilizers, the process of preparing the material should receive more attention to figure out the best percentage of compatibilizers that can provide the lowest quantity of charge possible. The optimal quantity of compatibilizers can help for better dispersion of nanoparticles in the PP matrix.

The addition of 2-wt% of natural clay, PP2-N2%, drive to decrease the charges all over the sample bulk as depicted in Fig. 6. As for PP1-S2% (Fig. 2), the small homocharges' peaks in this composition decreases with the poling time at the anode side. The charges in the central zone were very small and independent of poling time. Although PP1-S2%, PP1-S4% and PP2-N2% were all able to suppress

charges in the central zone, PP2-N2% was the most effective. As shown in [12] and [22], the amount of space charge in the central zone of a sample affects its life-time. The wider the zone of no-charge, or of a small quantity of charge in the center of the sample, the longer is the expected sample life-time.

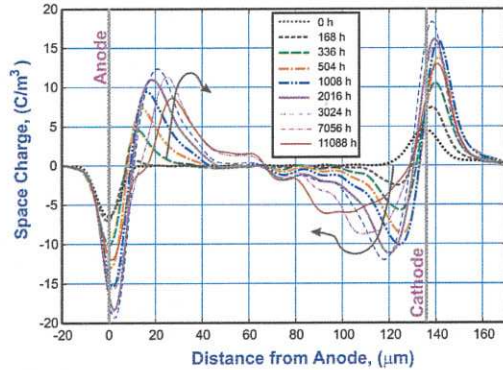


Fig. 6. Effect of the poling time at -25 kV/mm dc field on space charge distributions in PP2-0%.

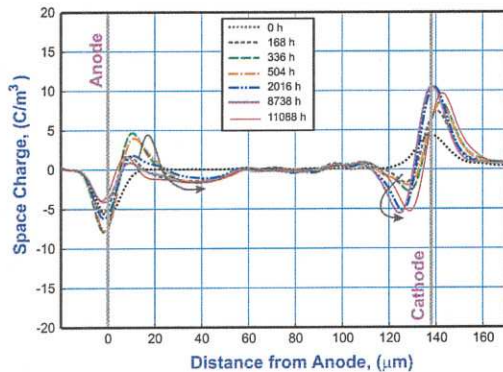


Fig. 7. Effect of the poling time at -25 kV/mm dc field on space charge distributions in PP-N2%.

Loading PP with 6-wt% of natural clay also leads to build-up charges in the sample's central zone. This could generate a high local electric field in that zone, sharply increase conductivity and could lead to specimen failure at any time.

Electric field distributions after 5040 h of aging with -25 kV/mm dc are depicted in Fig. 9. They show higher fields in the specimens' central zone for PP2-N6% and PP-S8% than for PP1-S2% and PP2-N2%. One of the PP-S8% specimens failed after 2016 h and two PP2-N6% specimens failed after 6384 h and 7056 h, indicating some correlation between higher local

electric fields in the sample and shorter times to breakdown. None of the samples containing 2-wt% or 4-wt% of PNC with synthetic nanoclay or 2-wt% of the PNC with Natural nanoclay failed after 11088 h of a dc field poling.

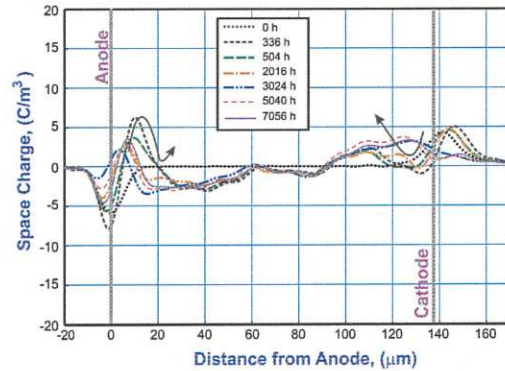


Fig. 8. Effect of the poling time at -25 kV/mm dc field on space charge distributions in PP-N6%.

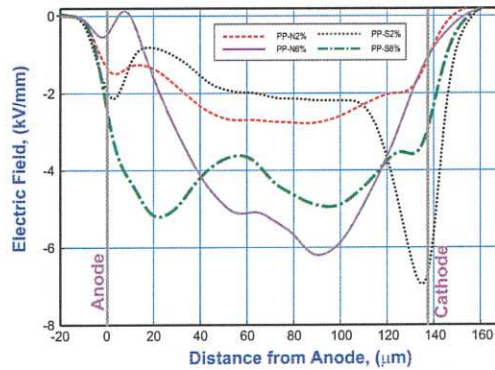


Fig. 9 . Electric field distributions after 5040 h of dc poling at -25 kV/mm.

Fig. 10 illustrates the charge distribution in the two types of host materials PP1-0% and PP2-0%, and PP1-S2% and PP2-N2% after 11088 h of aging. The peaks of charges are much higher and deeply extended into the central areas for the host materials compared to both filled materials. Lower charges in the PNC with natural clay, PP2-N2%, compared to the PNC with synthetic clay, PP1-S2% is also observed. This fact could be related to the ratio of the individual dimensions of the filler nanoplatelets. The individual

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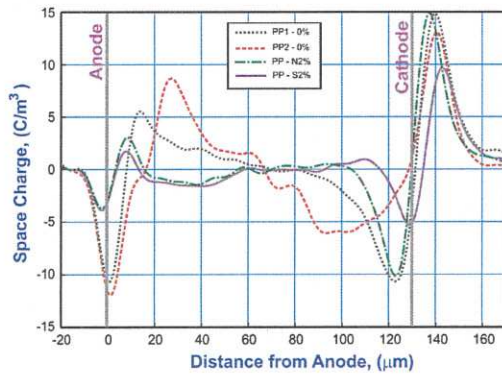


Fig. 10. Space Charge distributions after 66 W of dc poling at -25 kV/mm for the unfilled and filled with 2 wt% of natural and synthetic clay.

platelets' aspect ratios were  $\leq 6000$  and  $\approx 286$  for synthetic and natural, respectively [19, 21]. The smaller the nanoplatelets the larger is the interfacial interaction zone between nanoparticles and the host material. Therefore PP2-N2% would block charge injection more efficiently than PP1-S2% (Figs. 2 and 7). This result is in agreement with the findings of other research groups [6, 17, 23].

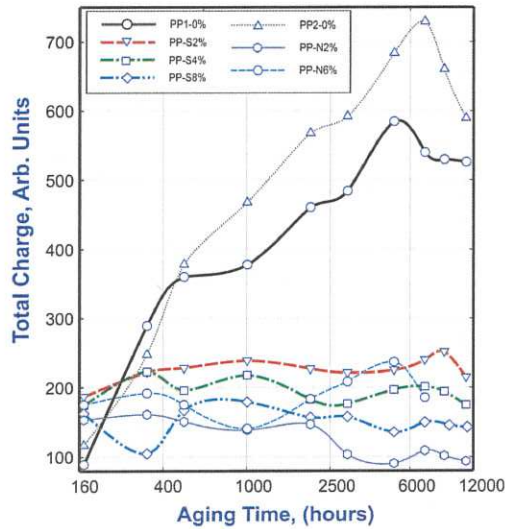


Fig. 11. Total charge,  $Q$ , versus poling time for PPs and their nanocomposites. Effect of the organoclay type and concentration.

As the concentration of nanofillers increases over the percolation threshold the interaction zones, or Gouy-Chapman-Stern layers, which are highly

conductive, could overlap and the overall conductivity of the composites considerably increases [24-26].

The integral of the absolute value of charges measured with the PEA technique between both electrodes is considered the total charge stored in a sample,  $Q$ . When  $Q$  is plotted versus time in a logarithmic scale, both unfilled materials demonstrate a dramatic increase of charges with aging time, see Fig. 11. However, all the filled specimens showed steady values of  $Q$  with time and the lowest accumulated  $Q$  was observed in PP2-N2%. Note that for poling times shorter than  $\sim 300$  h all nanocomposites exhibit a higher quantity of charge than PP1-0% and PP2-0%. Hence, short poling times may not reveal the real advantage of the nanocomposite structures as compared with unfilled polymer. At least  $\approx 1000$  h of poling time is required to clearly see the benefit of incorporating nanoparticles in PP material on charge mitigation characteristics.

### B. Conductivity Measurements

Figure 12 shows the conductivity measurement results. The base material, PP2-0% has the lowest conductivity. For the same particle concentration synthetic clay gives lower conductivity than the natural one. This could be due to impurities in the natural clay such as iron, dolomite and gypsum, especially in the presence of moisture. The increase of nanoclay concentration increases conductivity for both types of PNC. PP2-N6% and PP1-S8% showed almost

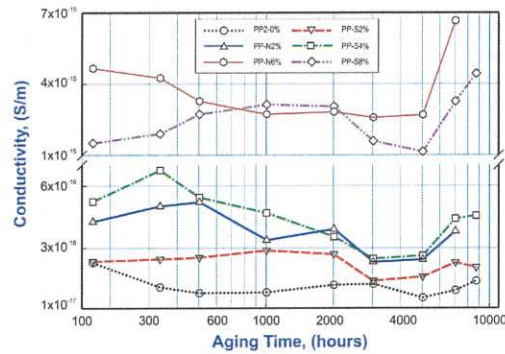


Fig. 12. The effect of nanoparticle concentration dc conductivity measured at -25 kV/mm.

6 times higher conductivity than PP2-0% (for convenience only PP2-0% is drawn in the figure).

This effect could be due to overlapping of the interaction zones around nanoparticles, which could lead to a dominant charge transport process throughout the system via the charge double layers [18, 24, 25].

The lowest value of the conductivity was observed for PP2-0% despite having the highest quantity of accumulated charge could be due to deeply trapped charges. The applied field of -25 kV/mm DC during the conductivity measurements would not be high enough to free these charges from their deep traps.

It is to be noted that the conductivity tends to increase after 5000 h and more tests and longer poling time are required to better understand the effect of dc field on space charge effects in polymer nanocomposites.

### I. CONCLUSIONS

The evolution of the space charge density and quantity as well as the conductivity with dc poling in PP-based nanocomposites containing natural and synthetic organoclays was investigated.

PP material can be affected by the manufacturing process where the percentage of compatibilizers can lead to different space charge evolution.

It was observed that loading PP with certain low percentages per weight of nanoclay can efficiently mitigate space charge in the PP. The optimal concentrations of clay was 2-wt% for the natural clay and between 2 and 4-wt% for the synthetic clay.

The accumulated space charge in PP1-0% and PP2-0% increases continuously with time of dc poling until 5000 h, whereas in both nanofilled composites it is almost independent of poling time.

At least 1000 h of DC poling is required to clearly see the benefit of charge mitigation in PNC specimens.

Samples containing natural clay had less space charge than the samples containing synthetic clay. This behaviour could be related to the difference in the individual platelets aspect ratios of the synthetic and natural organoclays.

The high conductivity of PP1-S8% and PP2-N6% could be related to excessive nanoparticle concentrations that may increase the overlapping of

the diffused double layer charge clouds and promote charge transport throughout those layers. To obtain the best results of filling PP with nanoclays, the nanoclay's wt% incorporated in the PP materials should be less than the percolation threshold.

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