Tensile and oil resistance properties of chloroprene added in epoxidized natural rubber, nitrile butadiene rubber, and poly vinyl chloride blends

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Abstract. In order to enhance the oil resistance of natural rubber (NR) and Epoxidized natural rubber (ENR), this research was investigated the effect of chloroprene addition as compatibilizer into natural rubber (NR), nitrile butadiene rubber (NBR), and poly vinyl chlorides (PVC) blends. The study aims to examine the effect of CR addition into NR and ENR blends with NBR, PVC on physical mechanical, tensile and oil resistance properties of the thermoplastic elastomer/ blends. ENR was prepared using natural rubber latex (30% DRC), 1 M formic acid, 1M acetic acid glacial at temperature 70-80°C for about 6 hours. The CR was added 0; 2,5; 5; 7,5; 10 phr into both NR/NBR/PVC and ENR/NBR/PVC blends. The mastication, blending and rubber compounding were conducted based on the ASTM procedures. The results show that at similar blend ratio composition of CR/NBR/PVC, ENR possess slight higher hardness values and better oil resistance compared to NR blends. However, tensile strength, elongation at break, and modulus of NR blends is not significantly different from ENR. In terms of change mass and the changes value of tensile strength and hardness after oil immersion, ENR blends that has oxirane group has better properties rather than NR blends. The CR addition could present a chlorine atom that restrict oil diffusion and oil absorption into the blends, thus it might lower the change mass and enhance the oil resistance. The CR loading also give positive trends in tensile strength, elongation at break for both NR and ENR blends, but an adverse effect on the modulus 100% elongation. In addition, the higher CR added, the smaller changes on tensile strength and hardness after oil immersion in both NR and ENR blends. It could be inferred that CR addition is able to improve compatibility and process-ability of the NBR, ENR, NR, and PVC blends using a proper plasticizer.

Keywords: Chloroprene, Compatibilizer, ENR/NBR/PVC, Oil Resistance, Tensile Properties.

1. Introduction
The utilization of rubber products has become wider, it is not only for interior construction, households, automotive parts, tire production, health care supplies, transportation but also for oil and mining industries [1, 2]. Those rubber products have to comply a certain specification that designed for that purposes. Thus, designing polymer blending and rubber formulation need to be observed properly in order to comply the product standard. Polymer blending aims to develop certain products that cannot be attained from individual polymer, thus two or more rubber/polymer blend in order to obtain very specific properties. The polymer blending is is determined by factors such as chemicals, nature of individual polymer, type of vulcanizing agents and chemicals (plasticizer, filler, tackifiers), and processing parameters. In addition, blend composition, blending condition and its interfacial adhesion

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also affect the properties of polymer blends [3, 4]. Additionally, for blending with some fillers, the distribution of fillers among in the polymer matrixes also affect the mechanical properties [5]. Thus, polymer selection is one of important steps on designing certain rubber products to obtain certain properties.

To illustrate, rubber products for oil industries like belt conveyor, idler roller, hose, gasket and seal need to be produced using properly excellent polymer types in order to comply the specific properties like physical mechanical properties hardness, tensile properties, oil resistance, ozone resistance, organic solvent resistance, heat ageing resistance, and thermo-oxidative ageing resistance. Thus, in this work, a polymer blending that designed for oil industries consists of natural rubber (NR), epoxidized natural rubber (ENR), chloroprene (CR), and nitrile butadiene rubber (NBR) need to observed to examine the tensile properties and oil resistance profiles in order to maintain the usage and its storage.

NR have high tensile strength because of their ability to crystallize under stretching, low compression set and stress relaxation, good electrical insulation and high resistance to abrasion, tear and fatigue [6, 7]. However, NR is susceptible to attack by atmospheric ozone and is not resistant to petroleum based oils and fuels. The natural rubber/isoprene molecules contain carbon double bonds, which are reactive to free radicals, ozone, oxygen, rubber compounds have some disadvantages especially poor thermal properties, low ozone resistance an low oil resistance [3, 8]. The present work was motivated by a desire to know how the epoxidation of NR affects the physical, tensile properties and particularly the oil resistance profiles. Chemically modified of NR as ENR would enhance the oil resistance properties due to the modification of double bonds become oxirane group [2, 6]. ENR can be produced by epoxidation of NR latex with formic acid and hydrogen peroxide, under the proper pH and temperature [9]. In this work, ENR has been prepared using technique based on [9, 10]. It is reported that, the high level epoxidation influences the increasing gas permeability, damping, and compatibility with reinforcement fillers along with the reduction in swelling hydrocarbon oil. ENR has unique properties such as good oil resistance, low gas permeability, improve wet grip and rolling resistance, coupled with high strength [2, 11]. Blending ENR with other polymers such as NR, SBR, BR, PVC have been successfully prepared [11].

Nitrile Butadiene Rubber (NBR) has a very good basic properties such as oil resistance over a wide range of temperature, heat ageing resistance, and fuel, alcohol resistance, but it has resilience, tensile and tear properties not as good as natural rubber [1, 12]. NBR is commonly extensive use in petroleum and automobile industries. The polar nature of the cyanide group on the polymer chain is the reason for the outstanding chemical resistances and the nitrile content responsible for the oil resistance, but those groups lower the flexibility and hydrophobicity of the material [12, 13]. However, NBR shows no self-reinforcing effect, as there is no crystallinity, thus a properly reinforcing fillers that compatible needs to be combined in order to improve its physical mechanical properties [14]. In terms of designing a certain rubber products that has specific plasticity and rigidity such as for hose rubber or electrical wire, some polyvinyl chloride needs to be compounded as a thermoplastic elastomer along with a compatibilizer and proper plasticizer [15]. The blending of NR with NBR is intended to produce a vulcanizate with the best properties from each component. Ismail reported that the tensile strength and elongation at break decrease, and latter increase is associated with strain-induced crystallization of the respective rubber.

Chloroprene rubber are homopolymers of chloroprene (trans 1-4 configuration) that has basic properties such as excellent in fuel resistance and heat aging resistance [16]. Because of this high degree of the stereo regularity, they are able to crystalize on stretching. Consequently, the gum vulcanizates have high tensile strength and resemble natural rubber gum vulcanizates [17]. In this work, Chloroprene (CR) was applied as a compatibilizer for the blending of ENR, NR, NBR, and PVC. As consequence, blending those rubbers together is the best practical solutions to achieve good mechanical, tensile properties and oil resistance. Additionally, miscibility is an important aspect in polymer blending and blends of ENR, NBR, NR with poly vinyl chloride (PVC) have been shown to be miscible [15]. ENR/PVC blends could be classified as a miscible system characterized by a single glass transition temperature located between that of ENR and PVC [2, 13]. Thus PVC/ENR can be classified as a melt-
processable blend. However, no attempt has been made to investigate the effects if CR as a compatibilizer on the properties of NR, NBR, ENR, PVC filled with Carbon Black.

Bearing in mind the limitation of using NR as a renewable resources polymer compared to synthetic rubber such as NBR, a chemically modified NR, Epoxidized Natural Rubber has been observed in order to improve tensile and oil resistance properties. In terms of developing rubber products like hose, seal and others products for gas and oil industries that has high oil resistance, tensile properties, and physical mechanical, this work concern on blending NR, ENR, NBR with PVC as a thermoplastic elastomer. The effects of using ENR and NR in NBR-PVC blends on tensile properties and oil resistance characteristics is very limited, thus this study would observe the comparison of using NR and ENR to enhance physical mechanical and tensile properties. Meanwhile, the research also would examine the effect of CR addition as the compatibilizer for NR/NBR/PVC and ENR/NBR/PVC blends in order to observe the oil resistance characteristics. Consequently, this research would contribute a new practical information on NR/ENR utilization for rubber hose application in gas and oil industries.

2. Experimental Procedures

2.1. Materials
Natural Rubber as Crumb Rubber with the specification of Standard Indonesian Rubber -10 (SIR 10) was obtained from private factories in Palembang, suspension grade poly (vinyl chloride) PVC in powder form, with a K value of 65 and a degree of polymerization of 920-1060 was supplied by PT Chandra Asri Petrochemical, Nitrile Butadiene Rubber with 40% acrylonitrile and Chloroprene Rubber Bayprene 210 (2-Chloro-1,3-Butadiene) were supplied by PT Chandra Asri. Concentrated latex (55% dry rubber content) was bought from PT Rambang Karet Palembang.

Filler such as Carbon Black N-330 and N-774 were bought from PT Renergi Indonesia. While, ZnO, Aflux, TMQ, 6PPD, Sulphur, and DOP (Global Chemical Co, Ltd). Other chemical such as flame retardant, Stearic Calcium were supplied by PT Sumi Asih, Oleochemicals Industry, and MBTS, TMTD, Formic Acid (p.a) Merck , Hydrogen Peroxide (p.a)Merck, Methanol (p.a Merck, Natrium Carbonates (p.a) Merck, and Surfactant LAS, Sulphur (Bratacho Chemical).

2.2. Methods

2.2.1. ENR preparation. High concentrated natural rubber latex was diluted with distilled water so as to reduce its dry rubber content (DRC from about 55% to 40%). 2 phr of surfactant added to stabilize the latex and avoid coagulation during epoxidation. 1 M Formic Acid was added. The stirring was conducted at temperature 50°C prior to the addition of 1 M of Hydrogen peroxide, and then stirring and reaction for about 6 hours at 80°C. After that, the epoxidized latex was coagulated using methanol and continue by soak it in a 5% Natrium Carbonates solution for about 4 -6 hours. Epoxidized was sheeted using a creeper machine with 2-4 mm thickness, and then drying at room temperature about 24 hours before drying at 60-70°C for about 4-6 hours.

2.2.2. Rubber Compounding. The NR or ENR, PVC, NBR, CR and other chemicals blend ratio as listed in Table 1. Before blending the NR was masticate d to reduce the viscosity using a banbury-type internal mixer with a fill factor of 0.6, circulating water at 40°C, and rotor speed of 50 rpm for about 20 minutes. Then, the blending of PVC and ENR or ENR was conducted using brabender plasticorder with some plasticizer. Then, the blends and other chemicals as listed in table 1 were compounded using a laboratory two roll mil XK-160, Shanghai Rubber- Machine Worker China for about 15 minutes and rotor speeds of 55-60 rpm with the circulating water 40°C. The rubber compounding process was in accordance with ASTM D3182 Standard Practice for Rubber (Materials, Equipment, and Procedures) for Mixing Standard Compounds and Preparing Standard Vulcanized Sheets and ASTM D3184-89 Standard Test Methods for Rubber Evaluation of Natural Rubber. The mix was sheeted and compressed into 2 mm thickness sheets and store at room temperature before testing.
Table 1. Formulation of NR/EPR using RHA Filler.

| Chemicals      | Formula phr |
|----------------|-------------|
| NR             | 30          |
| PVC            | 20          |
| ENR-50         | -           |
| NBR            | 50 47.5 45 42.5 40 50 47.5 45 42.5 40 |
| CR             | 0 2.5 5 7.5 10 0 2.5 5 7.5 10 |
| Carbon Black N-330 | 20   |
| Carbon Black N-774 | 40   |
| ZnO            | 5          |
| Aflux          | 1          |
| TMQ            | 1          |
| 6PPD           | 0.5        |
| Flame Retardant| 0.2        |
| Stearic Calcium| 1          |
| DOP            | 6          |
| MBTS           | 1.2        |
| TMTD           | 0.4        |
| Sulphur        | 0.9        |

2.2.3. Physical Mechanical and Tensile Properties. Dumbbell-shaped samples were cut from the moulded sheets according to ASTM D3182. Tensile testing (tensile strength, modulus 100% elongation, and elongation at break) was performed at a cross head speed 500 mm/min using a UTM Instron 3366 in accordance to ASTM D412-06ae2. Hardness measurement was conducted according to ASTM D2240-05 (2010) using a Wallace dead load, with ranges 30-85 IRHD (International Rubber Hardness Degree), the samples was 6 mm thickness at room temperature.

2.2.4. Oil Resistance Measurement. Dumbbell-shaped (punched out using die C, ASRM D412-92) test specimens were immersed in oil (Tellus 100, Shell Co., Ltd) at room temperature for 70 h. Thereafter, the specimens were removed from the oil, quickly dipped in acetone, and blotted lightly with filter paper to eliminate the excess oil on the specimen surfaces. The changes in the tensile strength and hardness of the specimens after oil immersion were used to determine the oil resistance. In addition, the percentage mass changes also observe by comparing the changes mass after oil immersion with mass before oil immersion.

3. Results and Discussion

3.1. Physical Properties

Fig. 1 illustrates the effect of CR/NBR composition on hardness for ENR/PVC and NR/PVC blends. It can be seen clearly that in ENR/PVC blends, the CR addition give a negative effect on hardness value. The CR loading of from 0 to 10 phr decreases the NBR composition from 50 to 40 phr and influence the decreasing of hardness from about 73 to 67 shore A. Similarly, the negative deviation of hardness is observed for NR/PVC blends. Thus, it could be concluded that CR addition in NR/PVC/NBR and ENR/PVC/NBR blending cause a slight decreasing of hardness. The hardness decreased as NBR decreased along with the CR addition for the NR and ENR blends. As the content of NBR in the blends is increased, the curative agent such as sulphur and other chemicals like activator, accelerator becomes
less soluble in NR compared with in the ENR [18]. Consequently, the curing rate of the blend decreases with the increasing content of NBR. Another reason, this was due to the improvement in cross link density of the blends. Theoretically, the increase of hardness could be attributed to the cross link density and shorter and more rigid network chains as the level of cross linking increases [13, 15]. The flexibility and elasticity of the rubber chain were less when more NBRs was incorporated into NR rather than ENR [2, 11], which also results in more rigid rubber vulcanizates and an increase in hardness of ENR compared to NR blending [12]. In addition, instead of individual polymer properties, for a certain blends, the processability of the blend becomes easier with the increase in CR [7, 17] when those have nearly polarity. This is due to the different polarity and hardness of CR among other polymers since the presence of chlorine [16, 19].

![Figure 1](image.png)

**Figure 1.** Hardness of ENR/PVC and NR/PVC blends with various composition CR/NBR.

Meanwhile, it could be observed that ENR blends has a little bit higher hardness values for the same polymer compositions rather than NR blends. This phenomena may be attributed to a greater enhancement of ENR compared to NR on NBR/PVC/CR blending [6, 15]. The NBR, PVC, and CR are both polar rubber whereas NR is a non-polar rubber and the ENR has higher polarity than NR [2]. Moreover, ENR was harder than NR but as hard as NBR. The physical mechanical blends of those polymer would be more compatible due to the same polarity and the synergistic effect could be obtained with miscible or partially compatible blends of ENR/NBR/PVC/CR. Consequently, the ENR may enhance the hardness value of the blends rather than the NR. CR/ENR50 blend is miscible and its miscibility is unaffected by the incorporation of high abrasion furnace carbon black [20]. In addition, it might due to the interaction between CR and the epoxy groups of ENR from hydrogen bonding [21]. CR and ENR blends are miscible in all ratios when the blends are prepared by solvent casting Carbon Tetrachloride solution [7]
3.2. Tensile Properties

Fig. 2 depicts the effect of various composition of CR/NBR in ENR/PVC and NR/PVC blends on tensile properties such as tensile strength, elongation at break, and modulus 100% elongation. For both blends, a slight increase of tensile strength, and elongation at break due to the loading of CR while decreasing the NBR. Conversely, for both blends, the modulus 100% elongation decreases slightly. The blends without CR has the lowest tensile strength and elongation at break 10 MPa and 310 % respectively, while the CR addition of 2.5 up to 10 phr causes the increasing of those properties up to 13 MPa and 400%. Aforementioned that NBR and CR influence the cross linking density of the polymers and the CR/NBR composition would a slight gradually increasing on tensile strength and elongation at break. The blending of CR, NR or ENR, PVC and NBR undergo strain-induced crystallization; the rubber reinforced each other when subjected to tensile stress, as reflected by a higher tensile modulus obtained in the blend [1, 16]. The rubber mixing and blending is affected by the composition, hardness, and polarity each individual [3, 4]. Thus, mastication, plasticizer types, compatibilizer agent, and blending condition determine the blending polymer along with the filler chemical dispersion within the rubber matrix [15].

It can be seen that at the same CR/NBR composition, ENR and NR blends has very similar tensile properties. However, ENR has slight higher properties of modulus and tensile strength compared to NR blends. This phenomenon might be attributed to the induction effect of ENR that is higher than NR. It is possible that ENR possess more activated precursors to crosslink are formed as a result of the activation of the double bond by the epoxide groups [2, 13]. The availability and compatibility of activated precursors will accelerate vulcanization, not only between ENR and NBR, but also induces faster inter phase cross linking between ENR, CR, PVC and NBR molecules filled by carbon black [17, 21]. The CR addition within the blends could influence a positive increase on NR and ENR blends in the same way. It is indicated that a stronger interaction and bonding between PVC powder particles and NBR, ENR or NR in the rubber matrix. This is due to the presence of chloroprene that enable to enhance the compatibility of the blends using carbon black as fillers and some plasticizers [14]. In addition, due to the greater polarity of NBR compared to NR and ENR, the reduction NBR/CR composition would
affect the physical mechanical properties like depicted in Fig. 1 and Fig. 2. However, by the incorporation of CR in those blends, the tensile properties improved instead of the modulus 100% elongation. It is inferred that CR might acts as a compatibilizer to improve interfacial adhesion between otherwise gross-phase-separated polymer pairs but reducing the interfacial energy between phases [19, 21]. Thus, interfacial interaction reduction between NR, ENR, PVC, NBR due to the CR addition could hinder agglomeration of rubber filler or rubber.

The effect of CR/NBR ratio on NR and ENR blends on modulus is shown in the Fig. 2, as the content CR increases, it make the blends stiffer, so there would be an increase in modulus, while the elongation would experience a decreasing [11, 12]. Compared to ENR blends, NR blends have a higher modulus even though not so significantly. However, for the tensile strength and elongation at break, ENR exhibited higher value due to the epoxide groups that contributed to the chain rigidity that resulting in dynamic fatigue resistance [13]. Furthermore, strain induces crystallinity was decreased when the amount of epoxies increased, but it was not observed in single NBR [20]. Other researchers reported that increasing in tensile strength can be related to the ability of ENR to crystallize when stretched due to the orientation of inter molecular chains [22, 23]. Those crystals will increase the number of inter molecular network chains per unit volume in the direct extension [20]. Hence, it becomes firmer and account for the enhancement in tensile strength.

At the same CR/NBR composition, the ENR has higher value of elongation at break compared to NR blends. Aforementioned, ENR has epoxide groups that could affect the cross linking density and the tensile properties on the blends [6]. The CR addition also give slight gradual increasing on elongation at break. It is confirmed that CR and the plasticizers may act as compatibilizer agent between PVC, NBR, and ENR or NR filled by carbon black. Thus, those addition cause minimize the uneven distribution of filler rubber, enhance rubber networking, and improve rubber filler interaction [20]. Consequently, it produces more interfacial bonding and cause increasing molecular mobility and visco-elasticity of the matric and improve the elongation at break.

3.3. Oil Resistance Properties.

![Figure 3](image-url)

**Figure 3.** Changes Tensile Strength and Hardness of ENR/PVC and NR/PVC Blends with various composition CR/NBR after oil immersion.

Oil resistance in polymeric materials is a key important properties for automotive industry because exposure to oil and greases. According to [23] the changes in properties after oil immersion could be used as an indicator of the resistance to oil. Knowing the oil resistance also minimize the mastication effect that might occur during blending and could interfere with the results of oil swelling tests. Fig. 3 illustrates the effect CR/NBR composition and oil immersion at room temperature for 70 hours on the tensile strength and hardness of ENR/PVC and NR/PVC blends. It could be seen clearly that the CR addition and NBR reduction cause an increasing on tensile strength and hardness value for both ENR/PVC and NR/PVC. At the same CR/NBR composition, NR/PVC blends experience a decreasing value of tensile strength due to the oil immersion, but not so significantly. This indicates that there is no
significant change in the microstructure of NR/PVC/NBR/CR and ENR/PVC/NBR/CR blends. The closeness of tensile strength values for the samples before and after oil immersion, is an indication of good oil resistance. Compared to ENR/PVC blends, the decreasing value of tensile strength is relatively smaller than NR/PVC. The oil resistance rubber blends mainly from their polarity, whereby the oil resistance is expected to increase with increase in polarity [14, 21]. Aforementioned, ENR is a polar rubber due to the presence of the oxirane groups and it has been proven to show oil resistance similar to medium grade NBR [3, 22]. Furthermore, the good oil resistance may be related to the formation of new cross links which increase the cross link density and consequently increase the rigidity of the vulcanizates [23].

Fig. 3 shows that ENR/PVC has higher value of hardness compared to NR/PVC as mentioned in Fig. 2. Generally, oil immersion at room temperature for 70 hours decrease the hardness for NR and ENR blends. It can be inferred that at the same CR/NBR composition, the differences of hardness value before and after oil immersion is wider rather than the changes in tensile properties. In addition, ENR has a better oil resistance on hardness compared to the NR blends. In addition, the CR loading for both NR/PVC and ENR/PVC blends attributed a significant effect on the oil resistance for tensile strength and hardness. This is due to the presence of chlorine atoms in the structure which could have increased the polarity of rubber, gave an improved oil resistance [7, 17].

The changes in weight of samples were calculated before and after oil immersion and the low changes represent high oil resistant of the blends. It can be seen in Fig. 4, at the same CR/NBR composition, the change mass of ENR/PVC is lower rather than NR/PVC. It could be concluded that ENR/PVC blends has a better oil resistance compared to the NR/PVC blends. NR is known to have poor oil resistance and epoxidation has brought substantial improvement in oil resistance [2, 20]. The ASTM oil used in this testing is a low polar hydrocarbon oil. As the amount of epoxidized groups in ENR increased, swelling degrees were decreased markedly and oil resistance were improved [22, 23]. As both ENR and NBR are high polar rubber and the ASTM oil is low polar hydrocarbon, ENR and NBR were found to have smaller changes in volume and mas due to the difference polarity during immersion at a room temperature [3]. In addition, the size of NBR agglomerate decreased leading to the increase in the density NBR phase and strengthening in the rubber network. Thus the number of crystal nucleus increases, facilitating the crystallization of ENR/NBR/CR/PVC and improve the oil resistance properties. In addition, the declination in oil absorption could be explained on the basis of the increased polarity of NBR/CR/ENR/PVC blends which leads to the ordering macro molecular chains in amorphous structure of NBR.
Furthermore, the more CR added in both NR/PVC and ENR/PVC blending, the smaller mass change. In the other words, the CR addition give positive effects on oil resistance as reported by [7, 21]. Aforementioned, the negative induce effect of chlorine will generate a negative charge on it and a delta positive charge on the nearby carbon [2, 17]. Instead of the presence of chlorine from PVC also enhance the oil resistance, the CR also contributed on these properties. The presence of a high presence of chloroprene unit induces dipole moment to the elastomer chain and the chlorine atoms act as cross linking points through which the chain network is formed [1, 16]. This results in restricted diffusion of oil molecules into rubber matrix and oil absorption might be avoided.

Generally, because of dilution effect, the sharp decrease of tensile and physical mechanical properties were obtained after oil immersion [5]. It is interesting that after oil immersion those properties of ENR are not much significantly changes. It could be explained that compared to NR, ENR possess better excellent resistance to hydrocarbon liquids due to the presence of oxirane groups. Moreover, the increasing CR also add more presence of chlorine atoms in the blends, and the oil resistance is expected to increase with increase in polarity the ENR/PVC/NBR blends.

4. Conclusions
The tensile and oil resistance of thermoplastic elastomer based on the blend of NR/PVC/NBR and ENR/PVC/NBR using CR has been studied. At a similar blend ratio of CR/NBR/PVC, ENR shows slightly higher hardness value compared to the NR blending. Conversely, the NR shows slight better tensile properties like elongation at break, tensile strength and modulus 100% elongation. In addition, the ENR blends also possess a better oil resistance and lower change mass after oil immersion rather than the NR blends. This could be explained that the ENR has oxirane groups so it might improve crystallinity and enhance the oil resistance properties. However, those differences were not so significantly. It could be inferred that CR might be act as compatibilizer between those individual polymer (NBR, ENR, NR, and PVC) using a proper plasticizer. Incorporation of CR into PVC/ENR/NBR and PVC/NR/NBR blends give different levels of improvement in tensile properties, hardness, and swelling behaviour. With the CR addition, tensile properties, hardness, and oil resistance increase gradually, but the hardness and change mass after oil immersion decrease less significantly due to the CR addition. In the other words, the high presence of chlorine atoms from CR could restrict oil diffusion and absorption into the rubber blends.

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