Dioxins are a kind of permanent organic pollutants, including polychlorinated dibenzodioxins and polychlorinated dibenzo-furans, with municipal solid waste incineration being one of its main sources. A large number of incinerators encountered in the construction process of “neighborhood avoidance” incidents have had its core appeal to worrying about dioxin pollution. Previously reported results have shown that when the incineration temperature is above 850 °C and the incineration time is over 2 s, the harmful substances will basically decompose. However, when the temperature of the flue gas drops to 200–400 °C, the HCl in the flue gas can be used as a precursor to synthesize dioxins. Besides, the removal of dioxins from the exhaust gas is difficult and unreliable; therefore, the best way is to remove the core element Cl, which produces dioxins.

The traditional HCl removal is often performed together with desulfurization and denitrification. Ca-based sorbents are usually used at low temperature (below 250 °C). The principle involves the acidic gases reacting with active components in sorbents to form stable metal compounds. With the increasing importance of global and higher standards of environmental protection, more and more attention has been drawn to the removal of HCl in high-temperature areas. For example, Dou et al. investigated the reaction between solid sorbents and hydrogen chloride gas at high temperatures in a fixed-bed reactor, with a large range of adsorption temperatures for HCl removal being investigated, while Chyang et al. showed that the HCl removal capacity of CaO was largest at 650 °C. Moreover, Weinell et al. pointed out that both the two sorbents (slaked lime and limestone) have the highest blinding capacities in the range of 500–600 °C.

Generally speaking, Ca-based adsorbent has a certain effect on HCl removal at high temperature (600 °C), but the chlorine capacity of the dechlorinating agent is relatively low, while the temperature range is also narrow. Besides, the adsorption efficiency also decreased obviously once the adsorption reaction deviated from the optimum temperature. Hydrotalcite-like compounds are bimetallic hydroxides with a layered microporous structure. The calcined products are characterized by strong alkalinity and a large specific surface area, in addition to exhibiting good performance as adsorbents, catalysts, and carriers. Layered double hydroxides (LDHs) are currently receiving considerable attention for a wide variety of applications in environmental remediation. The compositions of LDHs have the general expression $\text{M}^{x+}(\text{OH})_2^{(n-x)/2}(\text{A}^{x-})_{x/n}\cdot m\text{H}_2\text{O}$, in which $\text{M}^{x+}$ and $\text{M}^{(n-x)/2}$ are
divalent and trivalent metallic cations located in the main layer, $A^{n-}$ is the interlayer anion, $m$ is the number of interlayer water molecules, and $x$ is the molar ratio of $M^{3+}/(M^{2+} + M^{3+})$. The dechlorination performance and optimum ratio of hydrotalcite-like compounds have been previously studied.\textsuperscript{28–30} Hydrotalcite-like composite is a good dechlorination adsorbent, especially in the water environment, but its dechlorination characteristics in a high-temperature gas environment are yet studied. In some literature studies,\textsuperscript{31,32} the high-temperature dechlorination adsorbent for hydrotalcite-like materials was studied, including the preparation of the dechlorination adsorbent, adsorption test, cross-flow moving bed reactor, and numerical simulation. It was generally considered that the removal efficiency of HCl by a Ca–Mg–Al hydrothermal liquefaction (HTL) adsorbent at high temperature was significantly higher than that by CaO and NaHCO$_3$ adsorbents. Also, Hu et al.\textsuperscript{33} reported the preparation and dechlorination performance of hydrotalcite-like adsorbents containing different metal additions on the basis of the above, and considered that Ca-HTLs have the best comprehensive dechlorination performance under various working conditions. However, these works lack an in-depth study on the characteristics of the hydrotalcite-like high-temperature dechlorination adsorbent, especially for further improving the HCl adsorption at high temperatures.

With increasing attention toward global environmental protection, the standard of environmental protection is also rising, with “near-zero-emission” becoming the goal environmental protection workers strive to pursue.\textsuperscript{34} Therefore, it is more expedient and urgent to study the adsorbents for the effective removal of HCl in the high-temperature region. Furthermore, improvement of the adsorption capacity and efficiency of HCl on the basis of existing Ca–Mg–Al hydrotalcite-like materials is the technical route and innovation of this work. Potassium fluoride is a white monoclinic, crystalline powder with a salty taste and easy moisture absorption, with its aqueous solution being alkaline. Previously reported results from Gao et al.\textsuperscript{35} confirmed that KF/HT is a highly effective catalyst for the transesterification of palm oil with menthol.

The present work aimed at improving the adsorption capacity of Ca–Mg–Al hydrotalcite via modification with potassium fluoride,\textsuperscript{36} afterward further used as the adsorbent for HCl removal at high temperatures. Moreover, other parameters, including temperature and HCl concentration affecting the adsorption process, were also investigated. In addition, the microstructure and physicochemical properties of the as-prepared samples were also characterized by thermogravimetric (TG), scanning electron microscopy energy-dispersive system (SEM-EDS), X-ray diffraction (XRD), and Brunauer–Emmett–Teller (BET) methods. Finally, a mechanism for the HCl adsorption at high temperature was proposed.

2. RESULTS AND DISCUSSION

2.1. Characterization of the As-Synthesized Samples.

The X-ray diffraction patterns of CaMgAl-LDHs and 25% KF/CaMgAl-LDHs are shown in Figure 1a. It can be clearly seen that both LDHs have diffraction peaks at 11.64, 23.42, and 35.44°, corresponding to (003), (006), and (009) planes of MgAl-LDHs (JCPDS: 89-0460), respectively. Besides, the crystal faces associated with CaAl-LDHs (JCPDS: 87-0493) were detected at $2\theta = 11.70°$ (011), 23.53° (022), 35.62°(033), and 38.47° (114). This result indicated that the layered structure was well-formed, and the characteristic structure consists of both MgAl-LDHs and CaAl-LDHs. The in-plane diffraction peaks at 20 values of 60.76 and 62.10°, attributable to (110) and (113) planes of pristine MgAl-LDHs, demonstrated that the synthesized samples possess a good dispersion of metal ion crystallites and higher interlayer regularity in the hydroxide layers. The thickness layer spacing of the MgAl-LDHs is expressed by $d_{003}$ (0.759 nm). According to the thickness value of a brucite-like layer (0.477 nm),\textsuperscript{37} the calculated gallery height is approximately 0.282 nm. Since this height is comparable to the size of carbanions, it suggests that the carbanions may be arranged in a single layer between brucite-like layers. In addition, MgAl-LDHs belongs to a hexagonal crystal, with the value of $d_{110}$ spacing being 0.152 nm, while the calculated lattice parameters are $a = 2d_{110}$ (0.304 nm) and $c = 3d_{003}$ (2.277 nm). After adding the KF solution, the structure of MgAl-LDHs remains changed with the diffraction peaks of CaF$_2$ being observed additionally in the XRD diagram, a phenomenon, which indicated that Ca$_{2+}$ ions were in excess during the preparation process, with only a limited amount of Ca$_{2+}$ being completely associated with the structural development into CaMgAl-LDHs.

There are two different kinds of calcium carbonate (calcite and aragonite) within the synthetic CaMgAl-LDHs. Calcite is a kind of calcium carbonate mineral, which as the most common natural calcium carbonate, possesses good thermal stability, while aragonite has the advantages of a higher specific surface area and higher solubility.

After calcining at 550 °C, all the samples were converted into LDOs with the XRD patterns shown in Figure 1b. It can...
be observed that the three characteristic peaks of MgAl-LDHs disappeared. Magnesium aluminum hydroxide (JCPDS: 035-0964) and calcium carbonate (JCPDS: 86-2334) consist of the major components of CaMgAl-LDOs. Because both substances are strongly alkaline, CaMgAl-LDOs, as a result, are very suitable as adsorbents for HCl removal. Besides, KCaCO₃F, as a newly formed substance, was also detected by X-ray diffraction. There is a strong possibility that the interlayer CO₃²⁻ ions are spilled over and captured by the active K⁺ ions, once LDH samples are heated to about 400 °C. Importantly, LDOs loaded with potassium fluoride possess better performance in HCl adsorption experiments, which suggested that KCaCO₃F played an important role in this reaction. Furthermore, it can be seen that the product of HCl adsorbed by CaMgAl-LDOs is only CaCl₂·2H₂O (Figure 1c). In contrast, there are some other peaks attributable to chlorides in the XRD pattern of 25% KF/CaMgAl-LDOs, as KCl and MgCl₂ were both detected in the samples after the dechlorination reaction. This can vividly explain that KF/CaMgAl-LDOs have better removal efficiency than CaMgAl-LDOs at 600 °C. Therefore, it can be drawn that the modification of LDHs by adding potassium fluoride is a better way to improving dechlorination performance.

To characterize the microscopic architectures and composition of the LDHs and LDOs samples, SEM and EDS were employed with the results being displayed in Figure 2. The SEM images of the pristine CaMgAl-LDHs and 25% KF/CaMgAl-LDHs are shown in Figure 2a,b. The surface of both sorbents seems fluffy and porous, with granule-like particles grown on the surface of the crystals. In combination with the XRD diagram (Figure 1a), many agglomerated white particles were observed, as shown in Figure 2b, but not in Figure 2a. This substance most likely consisted of CaF₂ after the addition of KF solution to the LDHs. As a result, there is no obvious difference in the morphology of LDH samples after the KF loading.

Figure 2c,d reveals the surface morphologies of the calcined LDHs at 550 °C. Compared with the uncalcined LDHs as shown in Figure 2a,b, hexagonal and rhombohedral crystals appeared in the LDOs samples. The energy-dispersive X-ray spectroscopy (EDS) spectrum of CaMgAl-LDOs demonstrated the presence of Ca, Mg, Al, and O elements within the solid, with the corresponding weight percentages being 9.3, 17.1, 9.6, and 63.4%, respectively. This result further corroborated the successful preparation of Ca–Mg–Al metal oxides.

With the evaporation of water and evolution of carbon dioxide, some macropores gradually appeared; hence, HCl gas can easily enter into the adsorbent through a macropore. After the adsorption test, the hydrotalcite-like structure of CaMgAl-LDHs was almost destroyed (Figure 2d), with an inhomogeneous aggregation being observed, which is associated with the heaping of tiny particles and bulky lumps. Moreover, many snowflake-like villi were wrapped around the metal oxide crystals, which could play a very positive role in promoting the formation of the pore structure within the synthesized sample. Therefore, calcined hydrotalcite could act as a better adsorbent for enhanced HCl removal.

The morphological characteristics of the samples after the HCl gas adsorption were quite different from the previous samples (Figure 2e,f). The entire adsorbent surface is enclosed with numerous dense, translucent, and glossy crystals. Thus, no particles can be seen from the SEM diagram. Moreover, the XRD characterization showed that the recovered adsorbent was mainly composed of CaCl₂. The porous structure of LDOs was completely blocked, hence could lead to complete
failure of the HCl adsorbent. Because of the strong water absorption of calcium chloride, the samples formed a clear crystal morphology after being exposed in air (Figure 2e), with the EDS spectrograms of the two recovered samples displaying strong Cl signals. This further proved that CaMgAl-LDOs and KF/CaMgAl-LDOs are excellent HCl adsorbents (Scheme 1).

The N2 adsorption was carried out on the LDH and LDO samples to evaluate their porosity. Both samples exhibited reversible Type IV isotherms, revealing some mesoporous structures within the as-synthesized materials. The surface areas of the three samples were calculated using the Brunauer–Emmett–Teller model and are shown in Table 1. It can be observed that the specific surface area and pore volume of the sample obviously increase after calcination. In comparison with the four types of hysteresis loops as classified by IUPAC, Figure 3a–c all show that the hysteresis loop was Type H1, which reflects cylindrical holes with uniform diameter distribution at both ends, and can be used as a criterion to identify the mesoporous materials. This matches well with the pore diameter sizes given in Table 1. The pore diameter of CaMgAl-LDOs after the HCl adsorption reaction was reduced from 18.972 to 12.503 nm, which is very close to that of CaMgAl-LDHs (11.634 nm).

The thermal stability is also an important factor for evaluating the synthesized samples. Thermal analysis of the uncalcined LDHs was studied with the results shown in Figure 4. The TG and differential TG (DTG) curves for the pyrolysis of the CaMgAl-LDHs and 25% KF/CaMgAl-LDHs were detected at a heating rate of 5 °C min⁻¹ under a nitrogen atmosphere.
atmosphere. The DTG curves of CaMgAl-LDHs displayed three weight loss stages below 630 °C, as against two endothermic peaks in the 25% KF/CaMgAl-LDHs. Weight loss (5.1%) was detected for stage I, which can probably be attributed to the evaporation of physically absorbed water on the external surface. Potassium fluoride has very strong water absorption and can turn the adsorbed water into structural water, which requires higher temperature for removal. This could be the reason 25% KF/CaMgAl-LDHs DTG curve does not exhibit any endothermic peak in stage I. Moreover, the two DTG curves have the same trend in stage II and stage III. stage II, with two narrow peaks from 170 to 230 °C in the DTG curves, which overlap with the first peak at 125 °C, can be ascribed to the structural water intercalated in the interlayer galleries, as well as a small loss of CO$_2$. Moreover, with the peak at 211 °C, 25% KF/CaMgAl-LDHs has more weight loss than CaMgAl-LDHs. This further shows that the doped potassium fluoride largely influences the increase of interlayer water. Stage III appeared in the range of 240–420 °C with an obvious weight loss at 320 °C, indicating that most of the carbonate anions decomposed via the dehydroxylation of brucite-like sheets. Throughout the whole stage, the weight loss of the 25% KF/CaMgAl-LDHs is very close to the CaMgAl-LDHs, suggesting that the doped sample was also stable during the adsorption test.

2.2. HCl Adsorption Capacity of Various Sorbents. In this study, the operation conditions of different load ratios KF/LDOs on the effect of the HCl removal are listed in Table 2. It has been reported that when waste incineration temperature reaches 850 °C, some harmful substances can basically be decomposed in not more than 2 s during the incineration time. When the tail flue gas temperature drops to 300–450 °C, the HCl as a precursor reactivates dioxins. Therefore, the optimal temperature range for removing HCl at high temperature should be between 450 and 800 °C. Therefore, this work adopted 600 °C for further research. HCl concentration in waste incineration flue gas ranges from hundreds to thousands of ppm. However, with the advancement of garbage classification, the amount of chlorine in domestic garbage decreases accordingly, hence, 500 ppm is selected as the appropriate concentration in this work.

Figure 5 shows the total HCl capacities of KF/LDOs. It could be seen that the adsorption capacity of 0% KF/LDOs is the least (0.200 g HCl/0.5 g sorbent), as the HCl capacities were enhanced for all the samples with the increasing mass ratio of KF, with 25% KF/LDOs showing the highest HCl capacity (0.297 g HCl/0.5 g sorbent). Moreover, the penetration times of the synthesized samples are 665, 730, 870, 980, 820, and 770 min, respectively, with increasing weight percentages of KF. Compared with the other sorbents, 25% KF/LDOs also exhibited the longest breakthrough time, which illustrated that 25% KF/LDOs are an excellent and effective sorbents for removing HCl. The breakthrough curves and removal efficiencies of the worst and the best HCl adsorbents are shown in Figure 6. The removal efficiencies of

| operating parameter | symbol | units | conditions |
|---------------------|--------|-------|------------|
| reaction temperature| $T$    | °C    | 600        |
| initial HCl concentraion| $C$    | ppm   | 500        |
| gas flow rate       | $d$    | L min$^{-1}$ | 0.9       |
| average particle size| $m$    | mm    | 0.355      |
| mass of sample      | $m$    | g     | 0.5        |

LDOs and 25% KF/LDOs can reach more than 95%, but the LDO sample can only maintain such high levels for 40 min, while the removal efficiency of 25% KF/LDOs can be prolonged to 133 min. The potassium fluoride-doped CaMgAl-LDHs possesses better removal performance compared to the CaMgAl-LDH sorbent, especially for the 25% KF per unit loading sample. Hence, 25% KF/CaMgAl-LDH was chosen as the optimal adsorbent for further studies.

The results of the temperature effects on HCl removal are shown in Figure 7. It can be observed that the adsorption capacity of the 25% KF/CaMgAl-LDHs was initially enhanced from 0.2784 g (550 °C) to 0.2968 g (600 °C), followed by a continuous decrease to 0.2661 g (800 °C) with increasing
temperature. This may be due to the increased temperature causing the 25% KF/CaMgAl-LDHs to be activated for an easy adsorption process to occur. When the temperature was above 600 °C, the structure of 25% KF/CaMgAl-LDHs became damaged, leading to the loss of the adsorption sites. Hence, 600 °C can be considered as the optimal adsorption temperature for HCl removal.

To further explore the HCl concentration affecting the adsorption process, HCl concentration was increased from 250 to 1000 ppm, and the results are displayed in Figure 8.

Interestingly, the final adsorption capacities were equal to a constant for the various HCl concentrations, and the higher the HCl concentration, the shorter the time was required. This result indicated that the HCl concentration was not the determining factor for final adsorption capacity, implying that the HCl removal is attributed to chemical adsorption.

To study the kinetics in the adsorption process, the isothermal adsorption was carried out with the results shown in Figure 9a, while the corresponding kinetic curve is plotted in Figure 9b. It can be seen from the kinetic curve that the HCl removal is very close to the first-order adsorption kinetics.

3. CONCLUSIONS

In this study, the potassium fluoride (KF) doped Ca–Mg–Al layered double hydroxides (LDHs) were synthesized, and further converted into KF/Ca–Mg–Al layered double oxides (LDOs) via calcination. The doped samples exhibited enhanced HCl removal capacity compared to CaMgAl-LDOs. Moreover, 25% KF/CaMgAl-LDOs was optimized as the best adsorbent, with the best adsorption capacity achieved with 0.2968 g under optimal reaction parameters (600 °C, 500 ppm HCl concentration, 0.5 g adsorbent). In addition, the neutralization reaction is predominant for the removal of HCl by CaMgAl-LDOs according to the characterization results from TG, XRD, SEM, and BET, hence, the proportion of chemical adsorption is more important than that of physical adsorption. Furthermore, CaMgAl-LDHs possess good mechanical strength and sintering resistance when comparing the adsorbent sample after the reaction.

4. EXPERIMENTAL SECTION

4.1. Materials and Experimental Facilities. Ca(NO₃)₂·4H₂O, Mg(NO₃)₂·6H₂O, Al(NO₃)₃·9H₂O, NaOH, Na₂CO₃, and KF·2H₂O were all purchased from Sinopharm Chemical Reagent Co. Ltd. (Shanghai, China). All of the chemicals were of analytical grade reagents and used without further purification. Milli-Q water was procured from Shenzhen Yiliyuan Water Treatment Equipment Co. Ltd., while hydrogen chloride (3000 ppm) and nitrogen (99.999%) cylinder gas were obtained from Nanjing Shangyuan Gas Product Co. Ltd. A DF-101S collector with constant heating, charged magnetic stirrer and filled with methyl silicone oil was used as the heating source in the synthesis process, while a water-circulation multifunction vacuum pump used in the drainage and filtration process was supplied by Henan Yuhua Experimental Instruments Co. Ltd.

4.2. Synthesis of Ca–Mg–Al LDHs Sorbents. The Ca–Mg–Al LDHs were synthesized by the traditional coprecipitation method. Solution A was prepared by dissolving Na₂CO₃ (1.5 mol) and NaOH (2.0 mol) in 400 ml Milli-Q water, while Ca(NO₃)₂·4H₂O (0.4 mol), Mg(NO₃)₂·6H₂O (0.4 mol), and Solution B were prepared by dissolving Al(NO₃)₃·9H₂O (0.2 mol) in 300 mL Milli-Q water. A four-necked flask containing 100 mL ultra-pure water was fixed in an oil bath filled with methyl silicone oil. The two solutions (Solution A and Solution B) were simultaneously poured into two 500 mL pear-shaped separator funnels, which were thereafter inserted into two symmetrical inlets of flasks. The other two flasks were charged with a PTFE agitator and a pH meter (Sinomax, SIN-pH6.0). The pH of the mixture within the flask was maintained at about 10.5. The resulting slurry was continuously stirred at 65 °C for 24 h and then kept unstirred for another 12 h. Thereafter, the obtained slurry was filtered and washed continuously with deionized water until the pH reached 7. Subsequently, the precipitates were dried at 95 °C for 12 h to eliminate redundant water, and the final obtained sample was named CaMgAl-LDHs.

Five beakers containing an average 20 g of hydrotalcite-like samples were poured into KF·2H₂O solution with the ratios of

![Figure 8](image-url)  
Figure 8. Effects of HCl concentration on the removal process.

![Figure 9](image-url)  
Figure 9. Isotherm adsorption curve (a) and the kinetic curve (b) of the HCl removal process.
S, 10, 25, 50, and 100% (w/w HT) while adding 100 mL Milli-Q water into each. Thereafter, all the precipitates were heated at 150 °C until the sample was steam dried. The dried solid was further treated at 95 °C for 12 h, and finally calcined at 550 °C for 5 h to get converted into KF/CaMgAl mixed oxides. The calcined KF-doped LDH (hereafter referred to as LDOs) samples were ground and sieved to 0.180–0.355 mm, after which they were further used for the dechlorination performance test.

4.3. Characterization. The phase components and crystalline structure of the synthesized adsorbent and recovered samples were determined by a Rigaku Smartlab X-ray diffractometer (XRD) at a scanning rate of 10° min⁻¹ using Cu Kα radiation (40 kV, 100 mA, λ = 0.154056 nm) in a 2θ range from 5 to 80°, while the surface morphologies of LDHs and LDOs were observed using a scanning electron microscope (SEM, Hitachi Regulus 8220) equipped with an energy-dispersive X-ray spectrometer (EDS, Oxford Instruments Ultim Extreme) at an accelerating voltage of 15 kV. Moreover, thermal gravimetric analysis (TG, NETZSCH STA 449F3) was used to investigate the thermal properties of CaMgAl-LDH and KF/CaMgAl-LDH samples at a heating rate of 5 °C min⁻¹ from room temperature to 650 °C under a N₂ atmosphere, while the specific surface area and pore-size distribution were obtained using Brunauer–Emmett–Teller (BET) and Barrett–Joyner–Halenda (BJH) methods. The nitrogen adsorption–desorption measurements were performed on a physisorption BELSORP-mini (Microtrac BET, Japan) instrument at 77 K.

4.4. Hydrogen Chloride Removal Experiment. It has been previously reported that the hydroxalite-like oxides were found to possess excellent adsorption capacity in the removal of HCl. In the adsorption performance system, HCl and N₂ cylinders were controlled with a separate mass flowmeter (Beijing HORIBA METRON Instruments Co. Ltd.). The assessment of KF/CaMgAl-LDOs for dechlorination capacity was conducted on a vertical fixed quartz tubular bed (20 mm in diameter, 500 mm in length). In each experiment, 0.5 g KF/CaMgAl-LDOs was placed evenly on a 200-mesh stainless steel wire at the center of a quartz glass tube. After the pipeline leak detection was completed, the furnace was heated to 600 °C under a nitrogen atmosphere at a rate of 0.75 L min⁻¹. The reactor was then heated to the target temperature, followed by opening the solenoid valve for the HCl gas with the mass flowmeter set at a rate of 0.15 L min⁻¹. The total flow rate (0.9 L min⁻¹) of the mixed gases contained the initial HCl concentration (500 ppm). The hot, mixed gases passed through the sample, followed by condensation and drying, and then detection by the HCl infrared analyzer. The residual HCl concentration was determined and recorded by a Model 7900FM HCl GFC analyzer online. The exhaust gas was then released to open space through the NaOH solution. The experiment was carried out until the concentration of HCl in the outlet gases reaches the inlet content (500 ppm). The removal efficiencies and the HCl adsorption capacity were calculated using eqs 1 and 2, respectively.

\[
\eta = \left( \frac{C_{in} - C_{out}}{C_{out}} \right) \times 100\%
\]  

(1)

\[
q_T = \int_0^T (C_{in} - C_{out}) \times \frac{M}{22.4} \times 10^{-6} V \, dt
\]  

(2)

where \(\eta\) stands for the removal efficiency of the sorbent, \(C_{in}\) and \(C_{out}\) are the concentration of HCl (ppm) in the inlet and out gases, \(q_T\) represents the mass of HCl adsorbed by the LDOs from time 0 to \(T\) (g), \(T\) is the reaction time of dechlorination process (min), \(V\) denotes the flow velocity of the mixed gases (L min⁻¹), and \(M\) is the relative molecular mass of HCl (46).

ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acsomega.9b02069.

Deduction process for the kinetic model is given in the Supporting Information (PDF).

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Notes

The authors declare no competing financial interest.

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