Improving stability of organometallic-halide perovskite solar cells using exfoliation two-dimensional molybdenum chalcogenides

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Organometallic-halide perovskite solar cells (PSCs) are emerging as the most promising next generation solar cell devices. However, the stability is still the main bottleneck of their further development. Here, we introduce two-dimensional (2D) molybdenum chalcogenides (MoS$_2$ and MoSe$_2$) (MCs) nanoflakes as a buffer layer between perovskite layer and hole transport layer (HTL) to improve the stability of the organometallic-halide PSCs. 2D MCs are obtained via liquid-phase exfoliated (LPE) approach, and Glass/FTO/compact-TiO$_2$/mesoporous-TiO$_2$/FA$_{65}$MA$_{15}$PbI$_{85}$Br$_{15}$/2D MCs/Spiro-OMeTAD/Au structured solar cell devices are designed and fabricated. In this system, 2D MCs act both as a protective layer and an additional HTL of PSCs. This kind of PSCs achieve a relatively high-power conversion efficiency (PCE) of 14.9%, along with a much longer lifetime stability compared to the standard PSCs. After 1 h, PCE of the PSC adding a 2D MCs buffer layer could maintain 93.1% of initial value, while the PCE of the standard PSC dropped dramatically to 78.2% of initial efficiency. Our results pave the way towards the implementation of 2D MCs nanoflakes as a material able to boost the shelf life of PSCs and further provide the opportunity to fabricate large-area PSCs in view of their commercialization.

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INTRODUCTION

Traditional solar energy conversion devices based on crystalline silicon face the challenge of resource limitations and their energy intensive production process. Therefore, there is an urgent need to develop cheap and efficient solar cells. High-performance, economically viable, abundant and scalable device components are key to developing the solar cell materials. Organometallic-halide perovskite solar cells (PSCs) are among of the most promising candidates for next generation solar cell technology. Organometallic-halide perovskite have intense light absorption coefficient (1.5 × 10^4 cm$^{-1}$ at 550 nm), long electron and hole diffusion length (up to 1 mm) and high carrier mobility (1–10 cm$^2$ V$^{-1}$ s$^{-1}$). However, the stability of PSCs has become a major barrier for their further development in the market. Because perovskites are very sensitive to environment such as air, UV light, water, thermal stress and other factors. This kind of ETL makes as a buffer layer between perovskite layer and hole transport layer (HTL) to separate and selectively collect the generated charge carriers (holes at the cathode and electrons at the anode electrode). To date, TiO$_2$ is the most widely used ETL material for PSCs to prevent electron-hole recombination at the anode. This kind of ETL typically consists of compact TiO$_2$ layer (c-TiO$_2$, 50–80 nm) and mesoporous TiO$_2$ layer (mp-TiO$_2$, 200–300 nm). On the other hand, 2,2′,7,7′-tetakis-(N,N-di-4-methoxyphenylamo-no)-9,9′-spiro-bifluorene (Spiro-MeOTAD) represents the reference hole transport material (HTM) used for PSCs due to its facile deposition and high performance. It could effectively transfer the photogenerated holes from the perovskite to the cathode while blocking the electrons from reaching the cathode. Unfortunately, Spiro-OMeTAD also has serious degradation issues induced by UV light, temperature, additives, and environmental conditions. Therefore, Spiro-OMeTAD HTL contributes to the poor stability of PSCs solar cell devices as well. For either a HTL or a buffer layer, two-dimensional (2D) molybdenum chalcogenides (MCs) are potential candidates due to their high carrier mobility, low cost and stability. Therefore, many alternative HTMs could hardly compete to replace the ideal band edge positions of the Spiro-OMeTAD with respect to the perovskite absorbing layer. Another strategy to improve the stability of PSCs is inserting a buffer layer into the device structure to optimize the interface between the perovskite layer and Spiro-OMeTAD HTL.

For either a HTL or a buffer layer, two-dimensional (2D) molybdenum chalcogenides (MCs) such as molybdenum disulfide [MoS$_2$] and molybdenum diselenide [MoSe$_2$] are emerging as potential candidates due to their high carrier mobility, low cost (e.g. MoS$_2$ Sigma Aldrich, 0.85 € g$^{-1}$), and solution-processable properties. 2D MoS$_2$ nanoflakes have improved the efficient charge extraction across the HTL reducing recombination at the interfaces. Therefore, combination of 2D MCs and perovskite gives us opportunity to obtain solar cells with good efficiency and longer-term stability. In this work, 2D MCs (such as MoS$_2$ and MoSe$_2$) nanoflakes were prepared via liquid-phase exfoliation (LPE) approach, which is a non-destructive, insensitive (to the environment such as air and water) and solution-processable method. Then, these 2D MCs nanoflakes were utilized as a buffer layer material between perovskite layer and HTL (i.e., Spiro-OMeTAD) of the device to enhance the stability of PSCs. Based on the previous studies, 1School of Chemistry, Centre for Research on Adaptive Nanostructures and Nanodevices (CRANN) and Advanced Materials and BioEngineering Research (AMBER), Trinity College Dublin, Dublin 2, Ireland. 2Qatar Environment and Energy Research Institution (QEERI), Hamad Bin Khalifa University (HBKU), Doha 34110, Qatar. 3Department of Applied Physics and Astronomy, University of Sharjah, P. O. Box 27272, Sharjah, UAE. 4email: liangm@tcd.ie; felmellouhi@hbku.edu.qa; nicolov@tcd.ie

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Glass/FTO/compact-TiO2/mesoporous-TiO2/FA85MA15PbI85Br15/2D MCs/Spiro-OMeTAD/Au structured solar cells were designed and demonstrated. (a sketch of the solar cell and its band energy diagram is shown in Fig. 1a and b)22,29,31,39. We investigated various preparation conditions of 2D MC HTL buffer layers followed with a characterization of the devices’ power conversion efficiency (PCE) and stability.

RESULTS
Preparation of 2D MCs
2D MCs nanoflakes (i.e., MoS2 and MoSe2) were obtained via sonication-assisted LPE approach. The principle of LPE method is quite simple. Because of the characteristic of layered structured materials (i.e., strong chemical bonds in-plane but weak interaction out-of-plane), ultrasonic energy can easily break up weak van der Waals interactions between each layer to obtain exfoliated 2D nanoflakes. As shown in Fig. 2a, taking MoS2 as an example, first, bulk MoS2 was exfoliated in N-Methyl-2-pyrrolidone (NMP), whose Hansen solubility parameters and surface energy are suitable for most 2D layered materials, using a probe sonic tip37. The sonic tip was pulsed for 6 s on and 2 s off with 60% amplitude of power to avoid damage to the processor and reduce solvent heating, and thus, degradation. During sonication, the temperature of the system was cooled to around 5 °C by continuous flow of cooling water. Once sonicated, the dispersion was subjected to centrifugation to remove any unexfoliated materials. Then, the top 60% supernatant was collected. However, although NMP is the most effective solvent for the MoS2 exfoliation, it is toxic (Health code ≥ 2, NFPA 704) and has a high boiling point (202 °C). Moreover, it dissolves the perovskite crystals during the processing. Therefore, exfoliated MoS2 nanoflakes were transferred from NMP to isopropanol (IPA, Health code 1; NFPA 704, boiling point: 82.6 °C), forming environmentally friendly colloidal solutions (inset of Fig. 2b) with concentration up to 3 mg mL\(^{-1}\). As shown in Fig. 2c, UV–vis spectra of exfoliated MoS2 nanoflakes in NMP and IPA solvents, respectively. d HAADF-STEM (Scale bar is 100 nm) and e high-resolution TEM images of exfoliated MoS2, inset in part e is fast Fourier transform pattern of exfoliated MoS2 (Scale bar is 5 nm).

Glass/FTO/compact-TiO2/mesoporous-TiO2/FA85MA15PbI85Br15/2D MCs/Spiro-OMeTAD/Au structured solar cells were designed and demonstrated. (a sketch of the solar cell and its band energy diagram is shown in Fig. 1a and b)22,29,31,39. We investigated various preparation conditions of 2D MC HTL buffer layers followed with a characterization of the devices’ power conversion efficiency (PCE) and stability.
Fabrication of PSCs with a 2D MCs buffer layer

Fig. 3 Three different methods to fabricate the PSCs with a 2D MCs buffer layer. (P-1) MoS2 deposited on annealed perovskite without further annealing, (P-2) MoS2 is deposited on annealed perovskite film and further annealed at 100 °C for 1 h in the glove box, (P-3) MoS2 is deposited on as deposited perovskite film then both films are annealed at 100 °C for 1 h.

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buffer layer annealed separately from the perovskite layer (P-2) is much smoother than the one with a MoS₂ HTL buffer layer annealed together with the perovskite layer (P-3). It implies a MoS₂ could influence the growth of the perovskite layer, so it can reduce the quality of the perovskite layer. Therefore, it's better to do the deposition of the MoS₂ layer onto the complete perovskite layer by (P-2) method. Figure 4 shows the cross-section TEM image of the PSC with a 2D MoS₂ HTL buffer layer prepared by (P-2) approach. It shows less roughness than expected at interfaces perovskite / 2D MoS₂ / Spiro-OMeTAD which indicates that the inclusion of the MoS₂ layer between the perovskite and the Spiro-OMeTAD did not engender big cracks. This could be seen as a good sign for the solar cell operation.

PV performance of PSCs with additional 2D MCs buffer layer

Figure 5e shows the IV curves of the four devices including standard PSC (without HTL buffer) and PSCs with a 2D MoS₂ buffer layer prepared by three different approaches as described above.

It is clear that the device prepared with sequential annealing (P-2) performs as good as the standard PCS, while the PCS prepared without annealing of MoS₂ (P-1) or annealing MoS₂ together with pristine perovskite film (P-3) performed less efficiently. Interestingly, the fill factor (FF) was strongly affected (45.9% instead of 64.2%, Fig. 5c, f), followed by the open-circuit voltage (Vₜₜ) (0.92 V instead of 1.01 V, Fig. 5a, f) but the short-circuit current density (Jₜₜ) was only slightly affected (21.64 mA cm⁻² instead of 22.22 mA cm⁻², Fig. 5b, f). This indicates that there are serious interface issues under these two preparation conditions (P-1 and P-3). It is worth noting that IPA can partially dissolve the perovskite, so adding 2D MoS₂ buffer layers may lead to a damage of the surface of perovskite and this damage can be more serious in cases of (P-1) and (P-3). In (P-1) because the 2D MoS₂ film is not annealed so the residues of IPA will have more time to get in contact with the perovskite surface. In case of (P-3), the 2D MoS₂ film is added on the as deposited perovskite film so not in form of grains (crystals) which is make it surface more soluble than in the crystalline case. This could be the reason behind the low fill factor of the solar cell devices prepared under these conditions. This is in good agreement with the cross-section TEM images as mentioned before (Supplementary Fig. 5c, d). It is worth noting that compared to a standard PSC, a PSC with a MoS₂ (P-2) layer has slightly higher Vₜₜ and Jₜₜ, so that the efficiency of the device could maintain at 14.9% (Fig. 4d, f). This could be attributed to conducting MoS₂ nanoflake improved charge transport property of the device³⁶. What's more, it is clear from the energy band alignment (Fig. 1b) that MoS₂ can act as an electron blocking layer, which suppresses interfacial charge recombination³⁶. In addition, using 2D MoSe₂ nanoflakes we studied the effect of the nanoflake’s size to devices’ PV performances. According to the IV curves (Supplementary Fig. 6a–d), we could see that smaller size gave the better PV performance due its higher Jₜₜ and FF than the larger size nanoflakes. Therefore, the remaining of this manuscript focuses on small size 2D MCs nanoflakes integrated into PSC.

Fig. 4 TEM characterization of a PSC adding a 2D MoS₂ nanoflakes HTL buffer layer. The cross-section TEM image of a PSC adding a 2D MoS₂ nanoflakes HTL buffer layer prepared by P-2 (Scale bar is 1 μm).

Fig. 5 Variation of PSC parameters of a standard PSC and a PSCs with a 2D MoS₂ HTL buffer layer prepared by P-1, P-2, and P-3 methods. a Vₜₜ, b Jₜₜ, c FF, d PCE, e IV curves, and f summary of Vₜₜ, FF, Jₜₜ and PCE values of a standard PSC and PSCs adding 2D MCs nanoflakes as P-1, P-2, and P-3.
Stability of PSCs with additional 2D MCs buffer layer

Dark storage studies (ISOS-D), which estimated a cell’s shelf life under ambient conditions when it is not exposed to light were conducted at room temperature (23 ± 4 °C) in ambient air and humidity using sun simulator (AM1.5 G including UV). PCE of the standard PSC, PSC with a 2D MoS2 HTL buffer layer obtained by (P-2) and (p-3) were measured after 1 day, 1 week and 3 weeks. All PSCs were stored in ambient air at room temperature in dark. It is known that the PCE of a standard PSC using Spiro-OMeTAD as a HTL increases after several days because of the oxidation of Spiro-OMeTAD. We observed the same behavior for our PSCs with/without a 2D MoS2 HTL buffer layer. Interestingly, we observed that the efficiencies of these solar cells continue to increase even after 3 weeks as it is shown in Supplementary Fig. 7a-d and Supplementary Table 2. It indicates that the insertion of the MoS2 layer does not have a negative effect on the aging of the PSCs devices and our PSCs are relatively stable.

What’s more, we also tested the stability of PSCs with/without a 2D MoS2 HTL buffer layer under continuous sun illumination (AM1.5 G including UV) in ambient air at room temperature for 1 h. As shown in Fig. 6a, the PCE of the standard PSC reached its lifetime (21.8% of its initial efficiency) in 60 min. However, the PSC with a 2D MoS2 HTL buffer layer only loses 6.9% of PCE after 60 min. This result shows clearly that the 2D MoS2 prepared by exfoliation and inserted as a HTL buffer is able to enhance the stability of the PSC devices. When looking at the variation of each solar cell parameter $V_{oc}$, $J_{sc}$ and FF with the illuminated time, one could observe a gradual decrease of the values of these parameters with the time. However, in comparison to the standard PSC, the main difference is in the decay of the FF and $J_{sc}$ with time, which is more pronounced in the absence of 2D MoS2. This is because the 2D MoS2 buffer layer can cause changes in conductivity, transparency, structure, and/or band alignment with other layers of PSCs. This would result in preventing drastically changes of FF, likely by reducing recombination losses at the interface through blocking electron and accelerating charge transport, simultaneously. In addition, the improvement of the stability also attributed to the stabilization of $J_{sc}$, which is related with the improved charge transfer efficiency.

In addition, IV curves of forward and backward scans for a standard PSC and a PSC adding a 2D MoS2 buffer layer are shown in Supplementary Fig. 8a, b. No appreciable difference was observed between backward and forward scans for the standard PSC (Supplementary Fig. 8a), while $J_{sc}$ increased apparently leading to the increase of PCE for the PSC adding MoS2 buffer layer (Supplementary Fig. 8b). This is because of improvement of the charge transport property by adding a conducting 2D MoS2 HTL buffer layer between the perovskite and Spiro-OMeTAD. It also supports the conclusion that adding a 2D MoS2 HTL buffer layer gives better stability than a standard PSC. X-ray diffraction (XRD) tests of a standard PSC and a PSC adding an additional 2D MoS2 buffer layer after stability test under continuous sun illumination for 1h (characteristic peaks of the perovskite marked as # and MoS2 marked as *, respectively).
characteristic peaks of a PSC with a MoS2 buffer layer much stronger than a standard one. It implies less degradation of the perovskite in the PSC with a 2D MoS2 HTL buffer layer.36,44. What’s more, according to the energy-dispersive X-ray spectroscopy (EDX) mapping of the PSC with a 2D MoS2 HTL buffer layer after IV measurement, it’s apparent that a 2D MoS2 HTL buffer layer efficiently prevents migration of lead (Pb), iodine (I), and bromide (Br) from the perovskite layer to the Spiro-OMeTAD HTL (Supplementary Fig. 9). In the meanwhile, a 2D MoS2 HTL buffer layer also prevents the migration of metals (i.e., Au) from electrode to the Spiro-OMeTAD layer to the perovskite layer (Supplementary Fig. 9). The migration of metal through the HTL can lead to high recombination losses, which in turn cause reduction of FF and Voc during the operation.21,25,29.

DISCUSSION

Here, we demonstrated the use of 2D MCs nanoflakes (mainly focus on MoS2) as a HTL buffer layer of PSCs (Glass/FTO/compact-TiO2/mesoporous-TiO2/FA0.8MA0.2PbI2Br0.7/2D MC/Spiro-OMeTAD/Au). Guided by our computational simulation of the device performance, we reveal that inserting suitable 2D MCs HTL buffer results in better interface quality, absorption of photon energy, and improving the stability by working as a scaffolding layer between perovskite and HTL in PSCs. 2D MCs nanoflakes are obtained via sonication-assisted LPE approach. Then, these exfoliated 2D MCs in IPA dispersions are spin-coated onto the perovskite layer and immediately annealed to remove IPA solvent to obtain the 2D MCs HTL buffer layer. Interestingly, when a 2D MCs buffer layer is added between the perovskite layer and HTL (Spiro-OMeTAD), the stability of the PSC improved apparently. PCE of the standard PSC dropped to 78.2% of original value after endurance test of 1 h under solar simulator (AM1.5 G including UV) illumination in ambient air at room temperature. However, PCE of the PSC adding a 2D MoS2 HTL buffer layer could maintain 93.1% of PCE after 1 h under continuously illumination. Forward and backward IV scans and XRD, and EDX measurements of PSCs with/without a 2D MoS2 HTL buffer layer support the conclusion that adding a 2D MoS2 HTL buffer layer into the PSCs structure could efficiently enhance the stability of them. However, further investigations are underway to deeply understand the reasons behind this stability enhancement using prolonged tests followed by analytical experiments such as XPS and time-of-flight SIMS.

METHODS

Preparation of 2D molybdenum chalcogenides nanoflakes

Here, a typical methodology for the preparation of an exfoliated 2D molybdenum chalcogenides dispersion is described. For MoS2 as an example, in the initial MoS2 experiments, 4 g of MoS2 powder were added to 80 mL of NMP, and a probe sonic tip was utilized to sonicate the solution for a certain number of hours (power: 60% amplitude). The sonic tip was pulsed for 6 s on and 2 s off to avoid damage to the processor and reduce solvent heating, and thus, degradation. The beaker was connected to a cooling system that allowed for cold water (5 °C) to flow around the dispersion during sonication. Then, exfoliated MoS2 nanoflakes in NMP solvent were centrifuged at high spin rate (10,000 rpm) to obtain the MoS2 nanoflakes. Then, these MoS2 nanoflakes were dispersed in IPA through bath sonication, so exfoliated molybdenum chalcogenides nanoflakes dispersion in IPA were obtained. Afterwards, they were then centrifuged at different centrifugation rates (1000 rpm, 3000 rpm, and 5000 rpm) for 1 h, and the supernatant was retained (size selection of MoS2). The same methodology was used to obtain the exfoliated MoSe2 dispersions.

Fabrication of perovskite solar cells

Substrate and ETL preparation: Solar cells were fabricated on FTO glass substrates with a sheet resistance of ~8 Ω·sq⁻¹. The impurities were removed from the FTO-coated substrate via a three-step cleaning process (soapy water, deionized (DI) water and isopropanol (IPA) for 15 min each). The back side of the FTO glasses were covered with tapes. One side of the FTO glasses were etched using zinc powder and 4 M HCl solution. Then, compact TiO2 layer was deposited on FTO via dip-coating twice and annealing at 200 °C for 10 min and 450 °C for 30 min, respectively. A precursor dip-coating solution was prepared by mixing 6 mL of titanium disopropoxidebis(acetylacetone) (TiAcAc) solution with 54 mL of IPA. Then, mesoporous TiO2 layer was deposited by spin coating for 20 s at 4000 rpm resulted a ramp of 2000 rpm s⁻¹ using 30 nm particle Ti paste (mixed titaniumoxide paste (Dyesol 30 NR-D) and ethanol with the ratio of 150 mg: 1 mL). After the spin-coating, the substrates were immediately annealed in a hot plate.

Perovskite precursor solution and film preparation: The perovskite solution was prepared first. Typically, 1.1 mM (508 mg mL⁻¹) of PbI2, 0.183 mM (67.1 mg) of PbBr2, 1.05 mM (180.5 mg) of FAI, and 0.183 mM (20.4 mg) of MABr were dissolved into a mixture of DMF: DMSO (800:200 μL) solution. Then, the perovskite solution was spin coated in a two-step program at 1000 and 4000 rpm for 10 and 30 s, respectively. During the second step, 300 μL of chlorobenzene was poured onto the spinning substrate 20 s prior the end of the program to smooth the film. Finally, the substrate was annealed at 100 °C for 1 h in nitrogen-filled glove box.

Preparation of 2D molybdenum chalcogenides buffer layer: Three different approaches were tried to prepare the 2D TMD buffer layer. First method was to dip coating the 2D TMD buffer layer at 100 °C without any other treatment. Second and third methods added the heat treatment (100 °C for 1 h) after spin coating the 2D TMD dispersion onto the substrate. In the second method, the TMD layer was spin coated and annealed after annealing the perovskite layer at 100 °C for 1 h. But in the third method, the TMD layer was annealed together with the perovskite layer at 100 °C for 1 h.

HTL and top electrode preparation: After the deposition of the a 2D molybdenum chalcogenides buffer layer, a spirofluorene-linked methoxy triphenylamines (Spiro-OMeTAD, from Sigma-Aldrich) solution (70 mM in chlorobenzene) was spun at 4000 rpm. 2 s. The Spiro-OMeTAD was doped with bis(trifluoromethylsulfonyl)imide lithium salt (Li-TFSI, from Sigma-Aldrich), tris(2-(1H-pyrazol-1-yl)-4-tert-butylyridine)-cobalt(III) tris (bis(trifluoromethylsulfonyl)imide) (FK209, from Sigma-Aldrich) and 4-tert Butylpyridine (TBP, from Sigma-Aldrich)45,46. The molar ratio of additives for Spiro-OMeTAD was: 0.5, 0.03 and 3.3 for Li-TFSI, FK209 and TBP, respectively. Finally, around 20 nm of gold top electrode was thermally evaporated under high vacuum.

Materials and devices characterization

Characterization of 2D MCs nanosheets: The SEM images of all samples were acquired on a Zeiss Ultra Plus (Carl Zeiss, Germany) with an acceleration voltage of 2 keV. The TEM images of exfoliated 2D MCs nanosheets were performed on a FEI Titan at 300 kV (FEI, USA). Prior to the TEM study, the nanosheet dispersion was drop-casted onto holey carbon grids (Agar Scientific, UK). The UV-vis of 2D MCs dispersion was carried out with a Cary Varian 5000i UV-vis-NIR spectrometer. The dispersion was diluted to 1:10 in ethanol/water (60:40).

Characterization of PSCs: The numerical analysis has been carried out using 1D-solar cell capacitor simulator (SCAPS-1D) software. The computations were carried out using material parameters that were extracted from relevant literatures48-50. During the simulation, 2D MCs materials have been used as layered structured to mimic the real device structures. The highest reported electron mobility of MoS2 is 200 cm²/V·s 15,62. However, the layered structure of these materials induces an anisotropy of the carrier transport. The electron mobility within the layers is excellent but it is much lower across the layers. In our device simulation, we have considered the effective mobility of 50 cm²/V·s 1 as a typical mobility value based on at least two experimentally reported mobilities for MoS2 layer having thickness varying from 1 to 23 mono-layers.51,52. FIB-TEM technique was used to get the cross-section TEM image. Characterization of PSCs. XRD measurements were performed by means of Rigaku SmartLab X-Ray Diffractometer operating at room temperature. Current-voltage (I-V) tests were conducted using Newport Corporation IV tester under sun simulator (AM1.5G including UV).

DATA AVAILABILITY

The datasets generated during and/or analyzed during the current study are available from the corresponding author on reasonable request.
AUTHOR CONTRIBUTIONS
V.N., F.E. and M.L. conceived and designed the study. M.L. and A.A. performed the experiments. M.H. and O.R. are also acknowledged and thanked for the help provided for computational simulation and characterization parts, respectively. A.B. and C.D. are appreciated for the experimental help. M.L. wrote the paper. V.N., F.E., S.S., N.T., A.B., A.A., M.H. and O.R. reviewed and edited the manuscript. All authors read and approved the manuscript.

COMPETING INTERESTS
The authors declare no competing interests.

ADDITIONAL INFORMATION
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