Decreased Synthesis Costs and Waste Product Toxicity for Lead Sulfide Quantum Dot Ink Photovoltaics

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Use of lead sulfide (PbS) colloidal quantum dot (QD) films as photoactive layers in photovoltaic (PV) devices typically requires replacement of native QD ligands with lead-based capping ligands (i.e. PbX_2, X = Br, I) for the best-performing QD PVs. This ligand replacement process often requires additional solvents and toxic reagents. In the present study, an alternative PbS QD PV fabrication method with a lead-free tetrabutylammonium iodide (TBAI) ligand source and lower material requirements and toxicity is demonstrated, yielding 10% power conversion efficient PVs with more than 1000 h of storage stability under ambient conditions. Evaluation of the economic and toxicological benefits of

this new ligand exchange protocol reveal a 72% reduction in synthesis costs, an 80% reduction in solvent volumes, and a nearly 250-fold reduction in lead waste generated compared to the previous PbX_2-based protocol for highest-performing QD PVs. Most significantly, it is shown that unencapsulated PbS QD PVs made from this TBAI method leach less lead than U.S. Environmental Protection Agency limits and thus do not require hazardous waste disposal at the end of life. This finding implies a further decrease in expected device costs, extending progress towards large-scale production of commercial PbS QD PVs with low environmental risk.

Lead sulfide quantum dot (PbS QD) films show potential as photoactive layers in the next generation of photovoltaic (PV) devices. The tunable QD bandgaps,[1-6] high film stability in air,[2,3,5,6] and solution processability[2-6] could allow for the fabrication of low-cost, large-area devices.[2,3,5] During synthesis, PbS QDs are typically capped with long-chain oleic acid (OA) ligands in order to achieve colloidal stability and high monodispersity.[7] OA improves the surface passivation of QDs in organic solutions, however, it also reduces the charge conductivity within the photoactive QD films, leading to poor QD PV device performance.[5] It has been shown that a significant improvement in QD PV performance can be achieved if the deposited PbS QD films are chemically treated to undergo ligand exchange, replacing OA with shorter ligands, such as halides and short-chain thiols, which leads to high charge conductivities within QD films, boosting the power conversion efficiency of QD PVs.[5]

Solution-phase ligand exchange is currently the leading method of preparing the active layers in top-performing PbS QD PVs. In a separate synthetic step, OA-capped PbS QDs are exchanged with short ligands in solution and then suspended at a high concentration in a solvent to form an ink.[1-4] This ink can then be deposited on a substrate to form the QD PV device active layer,[1-4] with recently reported PbS QD PVs achieving 12% power conversion efficiency (PCE).[1] With this advancement the fabrication of the active layer is now more facile, however, the scalability of

PbS QD solar cells is still uncertain. A recent report by Jean *et al.* revealed that current solution-phase QD ligand exchange methods create an added cost of $6.30 per g of QDs, an expense that negatively impacts the commercial viability of QD PV modules.[8] In addition to this added cost, the toxicological properties, environmental impact, and end-of-life disposal of Pb-based PVs, including perovskites, is of critical importance and has received only minimal attention.[9-11] For example, the leading PbS QD ligand exchange procedures employing lead halides such as lead(II) bromide[1-3] and lead(II) iodide[1-4] have yet to be evaluated with regards to U.S. Environmental Protection Agency and European Union regulatory limits.

In this study we present a solution-phase ligand exchange method that reduces the amount of utilized lead by employing tetrabutylammonium iodide (TBAI) as the source of iodide ligands. The resulting PbS QD PVs have a 10% PCE under AM1.5 illumination, with more than 1000 h of storage stability when stored unpackaged in ambient conditions. Importantly, this synthetic methodology is evaluated with regards to its ability to lower costs and toxicity compared to the current champion lead halide (PbX_2) based ligand exchange methods.[1] This work provides a potential pathway towards improved compatibility with industrial scale thin-film PV device production as well as a framework to test the toxicity of new device fabrication procedures for a wide range of PVs.

The PbS QD precursor solution is first synthesized with OA ligands, resulting in QDs with an optical bandgap of 1.3 eV (corresponding to the absorption peak wavelength of λ = 956 nm) and bright photoluminescence that peaks at 1.19 eV (λ = 1043 nm) (**Figure S1**). Next, TBAI ligand exchange is performed by precipitating the OA-capped PbS QDs in octane with a solution of TBAI in ethanol. This precipitation step replaces previous phase transfer exchange methods, which involve a phase transfer of PbS QDs in octane into a solution of lead halides dissolved in *N,N-*

dimethylformamide (DMF).[1-4] The QDs are then resuspended in DMF, precipitated with ethanol a second time for further purification, and finally suspended in DMF to form a concentrated ink solution. The TBAI ligand exchange process leads to a slight red shift in the band-edge absorbance and emission peaks, as would be expected due to the increased dielectric environment from reduced interdot spacing[12,13] as well as an 11% increase in the full-width-at-half-maximum (FWHM) of the luminescence spectrum (Figure S2).

The QD ink formed by the TBAI ligand exchange method can be deposited in a single step to form the active layer of PbS QD PVs. Figure 1a shows a schematic diagram of the layers comprising the QD PVs from TBAI ink, including a thin layer of PbS QDs capped with 1,2-ethanedithiol (EDT) that is often required to suppress interfacial recombination.[6] Figure 1b shows the forward and reverse current-voltage sweeps of the same device, showing minimal hysteresis and demonstrating a current density (J_{sc}) of 26 mA cm^{-2}, open circuit voltage (V_{oc}) of 0.62 V, and fill factor (FF) of 0.62, resulting in a PCE of 10%. This value is approaching the highest-performing PbS QD ink solar cells fabricated to date.[1-4] Key differences in device performance between the TBAI ink device and the current certified champion PbS QD ink solar cell fabricated with PbX_2 based ligand exchange methods likely arise from less efficient QD surface passivation following ligand exchange with the TBAI method. The 30 mV lower V_{oc} for the TBAI ink device compared to the champion PbX_2 ink device is likely due to increased QD polydispersity, nonradiative losses, and aggregation following ligand exchange with the TBAI method compared to the PbX_2 method (Figures S3 and S4), and the 3 mA cm^{-2} lower J_{sc} likely arises from a lower exchange percentage of native OA ligands with iodide (Figure S5, Tables S1 and S2).[1]

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Figure 1c shows that the PV performance is maintained after more than 1,000 h of unencapsulated storage in ambient conditions, matching the storage stability of similar PbS QD PVs.[2] Furthermore, favorable optoelectronic properties, such as a low shunt current and an absence of radiative sub-bandgap states are revealed by dark device J-V characteristics (Figure S6a) and film photoluminescence (Figure S6b), respectively.

The TBAI ligand exchange method replaces several of the key precursors and solvents used in previous PbX_2 based ligand exchange methods.[1-4] Figure 2 outlines the precursor and solvent amounts of the TBAI ligand exchange method and the PbX_2 method reported by Xu *et al.*[1] and estimates the nominal material costs to prepare the PbS QD ink. Material costs are based on the largest purchase volumes available across leading commercial suppliers including Sigma Aldrich, EMD Millipore, and TCI, and draw from the framework for calculating Monte Carlo input parameters provided by Jean *et al.*.[8] A detailed spreadsheet outlining the material cost calculations is provided as Supporting Information. The values in Figure 2 reveal significantly lower synthesis costs for the TBAI QD ink preparation method compared to the PbX_2 method reported by Xu *et al.*,[1] with nominal costs of $1.82/g and $6.40/g, respectively. This lowered cost for the TBAI method is due to factors such as the usage of smaller solvent volumes, lower cost of ink solvent, and lower mass of iodide salt for ligand exchange. In addition to lower synthesis costs, the TBAI method offers improved colloidal stability (Figure S7) and chemical stability (Figure S8) of ligand-exchanged PbS QD ink solutions with storage in ambient conditions due to replacement of the PbX_2 method's amine solvent mixture with DMF (Figure S9). As PbX_2 exchanged QDs cannot be suspended in DMF without aggregating over time[1] the TBAI ligand exchange method offers improved solvent compatibility, which allows for increased air stability of PbS QD ink solutions and potentially lower yield losses during PV

manufacturing. The TBAI-based ligand method thus provides promising progress towards commercially viable QD solar cells.

In addition to the raw materials costs, the byproduct disposal costs should be considered to achieve a comprehensive understanding of synthesis costs. In the United States, the Environmental Protection Agency (EPA) restricts the disposal of hazardous wastes such as contaminated solvents in municipal landfills based on their potential to leach toxic substances into the surrounding environment.[14] For liquid samples, waste is regulated based on the concentration of hazardous contaminants.[15] Above threshold regulatory limits (5 mg L^{-1} in the case of lead)[16] waste cannot be disposed by using municipal waste streams, and instead requires costly hazardous waste removal methods.[11,17] To determine the concentration of hazardous contaminants, and thus the disposal requirements for the synthesis byproducts of PbS QD ligand exchange using both TBAI and PbX_2 methods, elemental analysis was performed using inductively coupled plasma optical emission spectroscopy (ICP-OES). As lead is the only EPA-regulated contaminant present in the synthesis, only the lead concentrations of the byproducts are reported. Table 1a shows the lead concentration and content of the synthesis byproducts of the TBAI and PbX_2 ligand exchange methods. While the first phase transfer exchange step of the PbX_2 ligand exchange leaves the octane phase uncontaminated with lead, thereby not requiring hazardous waste disposal, the high concentration of lead halides and large solvent volumes used in the second precipitation step cause the total lead content in the PbX_2 ligand exchange byproducts to be nearly 250 times that of the TBAI method for the same mass of PbS QDs. Based on the U.S. EPA's Pollution Prevention (P2) Cost Calculator, the reduction in hazardous waste generated with the TBAI method would lead to further cost savings of $0.48/g

compared to the PbX$_2$ method,[18] making the TBAI method a more scalable alternative for fabricating a deposition-ready PbS QD ink.

Because the ink active layer of PbS QD solar cells also contains lead, the toxicity of the fully fabricated solar cell should also be characterized to understand the end-of-life disposal regulations and requirements and their impact of the commercializability of QD PVs. In the European Union, the disposal of waste electrical and electronic equipment (WEEE) is regulated based on its total lead content, as a higher lead content is likely to pose a higher risk to human or environmental health.[11,19] To characterize the relative total lead content of solar cells fabricated with a device structure of ITO/ZnO/PbS-ink/Au prepared with the TBAI and PbX$_2$ ligand exchange methods, the solid lead in the devices was first fully dissolved into a solution of nitric acid using ultrawave digestion. The concentration of lead in the nitric acid solution was then determined using ICP-OES. Table 1b reveals that QD PVs made with the TBAI ink preparation method have a 46% lower lead content than those made with the PbX$_2$ method. This is likely due to the introduction of additional lead from lead halides during the PbX$_2$ ligand exchange[1] that does not occur during the TBAI ligand exchange. The lower content of lead in PbS QD films prepared with the TBAI method compared to those prepared using the PbX$_2$ method is consistently evidenced by a 60% lower atomic percentage of lead obtained using X-ray photoelectron spectroscopy (Figure S10, Table S1).

Unlike the European Union, the United States regulates solid waste based on leaching potential of lead rather than total lead content. This leaching potential is determined using an analytical method called the Toxicity Characteristic Leaching Procedure (TCLP). It involves a particle size reduction to < 1 cm, an extraction in acetic acid buffer solution, filtration to remove solids remaining in the extract, and finally, chemical analysis of the filtered extract to determine the

analyte concentration.[15] Waste that yields an extraction fluid with analyte concentration greater than the regulatory limits specified by the EPA (5 mg L^{-1} in the case for lead)[16] cannot be disposed of using municipal waste streams, and instead requires more expensive hazardous waste removal methods.[11,17]

To perform a preliminary analysis of the mobility of lead and the subsequent disposal requirements of QD PVs made with TBAI and PbX$_2$ ligand exchange methods, TCLP was performed on solar cells fabricated with a device structure of ITO/ZnO/PbS-ink/Au with matched photoactive layer thickness based on short-circuit current (J_{sc}) values. As shown in Table 1b, QD PV devices made with the PbX$_2$ method leach more than double the EPA lead limit (10.5 mg L^{-1} compared to 5 mg L^{-1}) and would thus be characterized as hazardous waste. The devices made with the TBAI ligand exchange however leach less lead than the EPA limit (4.0 mg L^{-1} compared to 5.0 mg L^{-1}) and as a result are more likely than the PbX$_2$ films to have the option of municipal waste as an end-of-life disposal pathway.

This lowered lead leaching for the TBAI ligand exchange method is due to both a lower total lead content and a lower solubility of lead components in the device. By comparing the concentration of leached lead measured during TCLP analysis to the total lead content of the devices measured following ultrawave digestion, matching the extraction volume and device active layer thickness across measurements, it is revealed that the percentage of lead leached versus the total lead available is lower for devices prepared with the TBAI ligand exchange method compared to the PbX$_2$ method (Table 1b). The percentage of leached lead, and thus the solubility of lead in devices with TBAI ink active layers is 30% lower than those with PbX$_2$ active layers. This decreased solubility is likely due to the absence of a PbI$_2$ matrix element for the TBAI PbS QD ink films, as PbI$_2$ has two

orders of magnitude greater leaching potential than PbS based on TCLP analysis (Table S3). Due to the lower lead content and lead leaching, the TBAI ligand exchange protocol leads to QD PVs that are significantly less toxic compared to the leading PbX_2 ligand exchange protocol.[1]

In summary, a solution phase ligand exchange method with TBAI as the source of iodide ligands improves the scalability of PbS QD solar cells. The lower solvent volumes, lower precursor expenses, and decreased potential yield losses all lower the PV device production costs with the TBAI method compared to lead halide based methods. Further, because the tetrabutylammonium cation is not a source of lead, both synthesis byproducts and the resulting PbS QD PVs are less toxic when using the TBAI QD ink preparation method compared to lead halide based methods, hence are less likely to require costly hazardous waste disposal. This decreased toxicity and cost provides a promising path forward towards scalable deposition-ready QD inks and low-cost, large-area QD PV device production.

Experimental Section

Ligand Exchange of PbS QDs: Oleic-acid-capped PbS QD with a first absorption peak at λ = 956 nm were synthesized using previous methods.[6] The tetrabutylammonium iodide (TBAI) solution-phase ligand-exchange process was carried out in a glass vial in air. TBAI (360 mg) was dissolved in ethanol (1.8 mL). PbS QDs (2.08 mL, 60 mg mL^{-1}) were then added to the TBAI solution. The vial was mixed vigorously for 30 s and then centrifuged to form a pellet of PbS QDs. The QDs were then resuspended in DMF (2 mL) and re-precipitated with ethanol (6 mL), centrifuging to form a pellet. After 5 min of drying, the PbS QDs were then redispersed in DMF (200-400 mg ml^{-1}) to achieve TBAI

ligand-exchanged PbS QD ink. The lead halide (PbX$_2$) ligand-exchanged PbS QD ink was prepared according to the literature.[1]

PbS QD PV Device Fabrication: Patterned ITO glass substrates (Thin Film Device Inc.) were cleaned with solvents and then treated with oxygen plasma. ZnO layers (120 nm) were fabricated by spin-coating a solution of ZnO nanoparticles synthesized according to the literature[6] onto ITO substrates and annealing at 165 °C for 10 min. The ligand-exchanged PbS QD ink was deposited by single-step spin-coating at 1,000 r.p.m. for 60 s and then annealing at 75 °C for 15 min. For the 10% PCE TBAI ink device, a hole transport layer was applied on top of the active layer (440 nm) based on literature methods.[6] Briefly, PbS QD solution (~15 μl diluted to a concentration of 50 mg ml^{-1}) was spincast onto the substrate at 2,500 rpm for 30 s. A 1,2-ethane dithiol (EDT) solution (0.02 vol% in acetonitrile) was then applied to the substrate for 30 s, followed by three rinse-spin steps with acetonitrile. The layer-by-layer spincoating process was repeated twice to achieve a hole transport layer thickness of 46 nm. All the spin-coating steps were performed under ambient conditions. The films were stored in air and then transferred to a nitrogen-filled glovebox for electrode evaporation. Au electrodes (100 nm thick) were thermally evaporated onto the films through shadow masks at a base pressure of 10^{-6} mbar. The nominal device areas are defined by the overlap of the anode and cathode to be 5.44 mm^2.

ICP-OES: Chemical analysis was performed using an inductively coupled plasma analysis system (Agilent 5100). The concentration of the standards were 1, 10, and 100 mg L^{-1} and the Pb emission

wavelengths used for characterization were 179.605, 182.143, 217.000, 220.353, 261.417, 280.199, and 283.305 nm.

TCLP: The Toxicity Characteristic Leaching Procedure was performed according to the literature.[15] Briefly, devices were smashed to reduce particle size to < 1 cm in the widest diameter. The shards where then added to an acetic acid buffer solution (0.1 M, pH = 4.98) in a 20:1 ratio by weight liquid to solid and agitated end-over-end in a polypropylene centrifuge tube for 18 ± 2 hours. The mixture was then filtered with a 0.7 μm glass fiber filter to remove solids for chemical analysis via ICP-OES.

Ultrawave Digestion: Devices were digested in a nitric acid solution (1 M) in a 20:1 ratio by weight liquid to solid using a Milestone UltraWave microwave sample-digestion system at 1500 W. The digestion consisted of two steps: 15 minutes at 180 °C and 120 bar, and 10 minutes at 220 °C and 150 bar.

Device Characterization: Current density–voltage characteristics of devices were measured using a Keithley 2636A sourcemeter. Simulated solar light illumination (1-Sun, 100 mW cm^{-2}) was generated by a Newport 96000 solar simulator equipped with an AM1.5G filter. The light intensity was calibrated with a Newport 91150 V reference cell before each measurement. The relative error in efficiency measurements is estimated to be below 7%.

XRD: Powder X-ray diffraction patterns were collected with a PANalytical X'Pert Pro diffractometer. The samples were prepared by drop casting PbS QD ink (40 µL) on a silicon wafer. Cu Kα1 irradiation was used.

XPS: X-ray photoelectron spectroscopy was performed on a Thermo Scientific K-Alpha+ using a 400 µm spot size. Sample charging was corrected by shifting peaks to align with the 284.8 eV adventitious carbon peak. Atomic ratios were calculated through integrating and comparing high resolution elemental scans in the Thermo Avantage software.

Supporting Information

Supporting Information is available from the Wiley Online Library or from the author.

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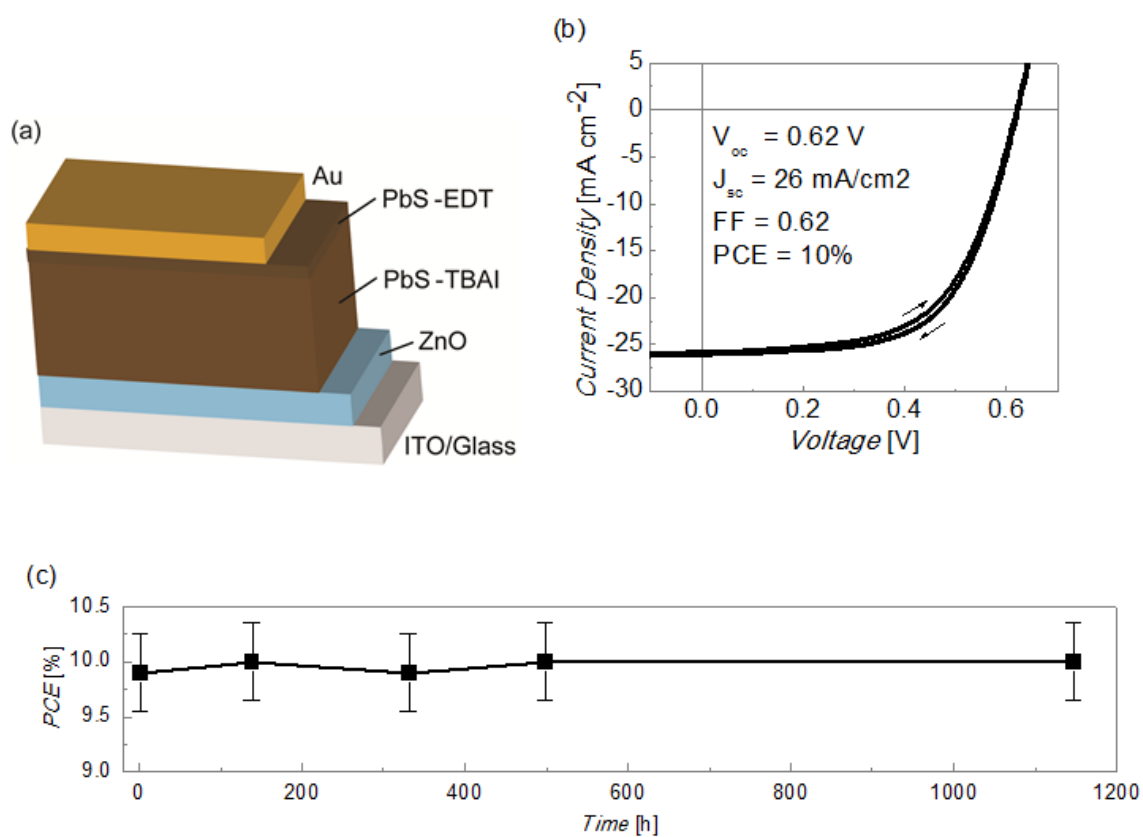[19] European Parliament, *Off. J. Eur. Union* **2011**, *54*, 88.

Figure 1. TBAI ink device. (a) Device architecture, (b) J-V curve, (c) Stability test of device stored in air.
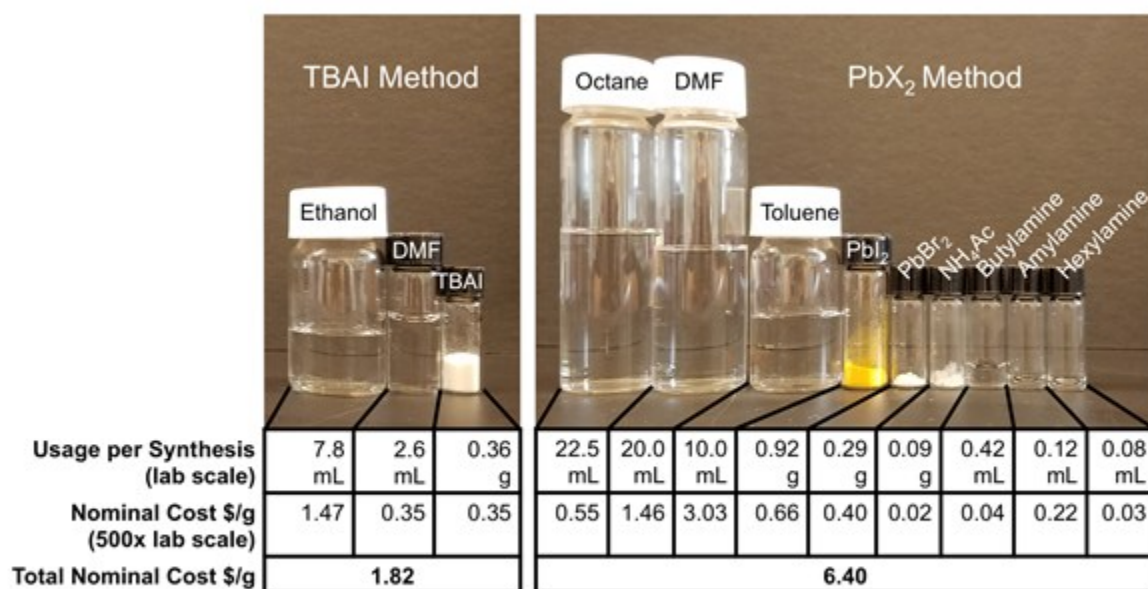
	TBAI Method			PbX$_2$ Method								
	Ethanol	DMF	TBAI	Octane	DMF	Toluene	PbI$_2$	PbBr$_2$	NH$_4$Ac	Butylamine	Amylamine	Hexylamine
Usage per Synthesis (lab scale)	7.8 mL	2.6 mL	0.36 g	22.5 mL	20.0 mL	10.0 mL	0.92 g	0.29 g	0.09 g	0.42 mL	0.12 mL	0.08 mL
Nominal Cost $/g (500x lab scale)	1.47	0.35	0.35	0.55	1.46	3.03	0.66	0.40	0.02	0.04	0.22	0.03
Total Nominal Cost $/g	1.82			6.40								

Figure 2. Precursor and solvent amounts and costs required to process PbS QDs using TBAI (left) and PbX$_2$ (right) ligand exchange protocols.

Table 1a. Lead content of ink synthesis byproducts.

	Byproduct Description	Concentration Pb [mg L^{-1}]	Solution Volume [mL]	Total Pb from Byproduct [mg]
TBAI Method	Supernatant from 1st Crash Out	380± 30	3.88	1.5
	Supernatant from 2nd Crash Out	140± 30	8	1.1
PbX$_2$ Method	Supernatant from Octane Rinse	≈0	25	≈0
	Supernatant from Crash Out	21600 ± 100	30	648

Table 1b. Relative total lead content and lead leached from PbS QD solar cells (ITO/ZnO/PbS ink/Au) made with TBAI and PbX$_2$ ink preparations.

Ink Type	Total Pb [mg L^{-1}]	Leached Pb [mg L^{-1}]	Percentage Leached Pb
TBAI	12.5 ± 0.6	4.0 ± 0.3	32%
PbX$_2$	23 ± 2	10.5 ± 0.4	46%

A PbS quantum dot solution-phase ligand exchange method employing tetrabutylammonium iodide (TBAI) rather than previously used lead halides (PbX$_2$) allows for the rapid solution processing of high-performing, air stable photovoltaics with low solvent volumes and synthesis costs. Solar cells made with this method are shown to leach less lead than the U.S. Environmental Protection Agency limit.

Keyword solar cells

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