



## The hidden perils of lead in the lab: Guidelines for containing, monitoring and decontaminating lead in the context of perovskite research

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# The hidden perils of lead in the lab: Guidelines for containing, monitoring and decontaminating lead in the context of perovskite research

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## Abstract

The metal lead is an integral part of mainstream perovskite solar cells. Lead-based compounds in the form of lead-based paint and lead-contaminated dust are known to potentially trigger long lasting health implications when exposure surpasses certain limits and lead is accumulated in the human body. Because it is not clear what the health implications are of lead that is processed in the context of perovskite research and no organization has published specific directives, we have established a set of instructions for lead safety in perovskite research labs. The instructions include best practices for handling, containing, monitoring and decontaminating lead-based materials. Importantly, it is shown that lead can be contained best by adopting strict cleaning and housekeeping protocols, while decontamination can be accomplished with conventional detergents. Reliable testing of contamination levels requires periodic chemical analysis such as ICP (inductively coupled plasma spectroscopy). Conversely, simple lead tests that are currently on the market can lead to misleading assessment. We further recommend periodic medical surveillance in the form blood lead level testing to ensure the well-being of lab users. The directives described here were established such that they can be easily adopted by any lab working on perovskite research.

## Introduction

Perovskite solar cells have garnered enormous excitement over the last decade, arising from their ease of fabrication, high performance and rapid performance improvement.<sup>1,2</sup> As the number of research and development groups actively engaged in this field continues to increase, the safe handling, exposing and disposal of the toxic lead salts used in these devices becomes a more prominent issue. While students and researchers are usually familiar with the general idea that lead can be toxic, very little awareness is often demonstrated towards the potential hazards associated with lead contamination in the lab, particularly around lead-based device fabrication. This article provides guidance on how to best control lead contamination in the form of the most commonly used lead salts in the context of mainstream perovskite research that is happening today across many labs in the world. The procedures described herein emerged from the experience gained in our labs over the past several years. The text reflects our discussions with health, safety and environment (HSE) experts, observations of exposure routes in the lab, quantitative results of lead contamination and spread in perovskite research labs, and interpretation on how to best contain, monitor and decontaminate lead compounds in the form of solids (powders) and solutions. We note that this is not an all-comprehensive safety instruction bulletin, but rather an attempt to highlight less-obvious dangers, and offer practical solutions for researchers to consider and utilize. These guidelines provide laboratory users of lead compounds information on how to implement our decontamination procedures. Utilizing these procedures and methods will reduce lead levels in the lab and thereby reduce lab user's potential exposure to harmful lead.

Lead(II) salts are precursors of lead-based perovskite semiconductors. In a typical perovskite research lab, spillage of trace amounts of lead powder is unavoidable. Unlike a simple spill of a benign chemical, spills of lead compounds can expose researchers to levels of lead that can be harmful.<sup>3</sup> Lead(II) salts can be toxic and are suspected carcinogens.<sup>4,5</sup> Lead can also accumulate in the body through bioaccumulation in bone and other tissues.<sup>6</sup> It can cause a variety of well documented health problems and can even ultimately result in death.<sup>7</sup> Moreover, lead salts are most commonly provided in the form of fine powders that can easily become airborne and spread across large distances in the lab. Solvents which are commonly used to prepare perovskite formulations, such as dimethylformamide (DMF) and

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2  
3 dimethylsulfoxide (DMSO) can significantly exacerbate the intoxication of lead compounds  
4 as they are particularly effective in enhancing skin permeability, thus increasing the risk of  
5 absorption via dermal contact.<sup>8</sup> In addition, it is important to realize that contaminated  
6 clothing can also transport lead from the lab leading to secondary exposure. Therefore,  
7 controlling lead levels at the source is vital in order to reduce lead exposures.  
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13 The motivation to draft these guidelines stems from a current lack of clear directions on lead  
14 work in a laboratory/research setting from a regulatory body, at least from one that is  
15 accessible and widely adopted by the perovskite research community. Instead, there are  
16 publications by a range of health and safety organizations that are based on, e.g., permissible  
17 amounts of lead on surfaces and in the air and reference blood lead levels. However, published  
18 values, and respective directives, differ widely from agency to agency<sup>9,10</sup> and do not account  
19 for the work environment of modern research labs, leading to an existing uncertainty around  
20 lead safety in labs. For instance, the National Institute of Occupational Safety and Health  
21 (NIOSH) and the Occupational Safety and Health Administration (OSHA) have set action  
22 levels of 5 and 50  $\mu\text{g}/\text{dL}$  of lead in blood, respectively. Table 1 compiles the stated permissible  
23 amounts of lead according to different health and safety agencies. Moreover, we found that  
24 current regulations typically refer to lead embedded in a material mixture, e.g., lead paint,  
25 and therefore allude to breathing dust containing lead arising from scraping, grinding, etc. of  
26 such lead paint. According to OSHA, the action level for workers in contact with lead  
27 (measured in  $\mu\text{g}/\text{m}^3/8\text{hr}$  time weighted average) is 30  $\mu\text{g}/\text{m}^3/8\text{hr}$  and the permissible exposure  
28 level is 50  $\mu\text{g}/\text{m}^3/8\text{hr}$ .<sup>11</sup> However, continuous exposure to lead in the air during an 8-hour  
29 workday is not the route of exposure that researchers encounter in common perovskite  
30 research labs.  
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44 Alarmingly, a recent cohort study by Lanphear et al.<sup>12</sup> reported an attributable fraction of  
45 blood lead levels to all-cause mortality among US adults of 18%, pointing to potential health  
46 implications at levels much lower than the guidelines defined, for instance, by OSHA.<sup>12,13</sup> In  
47 this context, we cite text from the Association of Occupational and Environmental Clinics  
48 (AOEC): “Although the Federal Occupational Safety and Health  
49 Administration’s lead standards have provided guidance that has been beneficial for lead-  
50 exposed workers, these regulations have not been substantially changed since the late 1970s  
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and thus are primarily based on health effects studies that are well over three decades old. There is an urgent need to revise them.”<sup>14</sup> As such, current regulations are outdated and do not account for the much more concentrated forms of lead (>97% purity) and its diffusivity (in the form of loose powders). Considering these limitations, we drafted procedures, which are shared here, evolving from experiences within our groups and collaborators.

**Table 1.** Permissible amounts of lead on surfaces, in the air, in food, and reference blood lead levels according to different health and safety agencies in the US, Canada, UK, Germany, Europe and global agencies. Adapted from references <sup>9,10</sup>.

Lead Level Action Thresholds for Differing Agencies and Media			
Media	Agency	Lead Levels	Notes
Air (workplace)	ACGIH <sup>a</sup>	150 µg/m <sup>3</sup>	TLV/TWA <sup>b</sup> guideline for lead arsenate
		50 µg/m <sup>3</sup>	TLV/TWA <sup>b</sup> guideline for other forms of lead
Air (workplace)	CCOHS <sup>c</sup>	50 µg/m <sup>3</sup>	TLV-TWA <sup>b</sup>
Air (workplace)	EEC <sup>d</sup> Directive 98/24	150 µg/ m <sup>3</sup>	Occupational exposure limit value, 8 h time-weighted average
Air (ambient)	EPA <sup>e</sup>	0.15 µg/m <sup>3</sup>	Regulation; NAAQS <sup>f</sup> ; 3-moth average
Air (workplace)	HSE <sup>g</sup>	150 µg/ m <sup>3</sup>	Action level (8-h time-weighted average)
Air (workplace)	NIOSH <sup>h</sup>	50 µg/m <sup>3</sup>	REL <sup>i</sup> (non-enforceable)
Air (workplace)	OSHA <sup>j</sup>	50 µg/m <sup>3</sup>	Regulation; PEL <sup>w</sup> (8-hour time weighted average) (general industry)
		30 µg/m <sup>3</sup>	Action level (averaged over an 8-hour period)
Air (workplace)	SCOEL <sup>k</sup>	100 µg/ m <sup>3</sup>	Action level (8-h time-weighted average)
Blood	ACGIH	30 µg/dL	Advisory; indicates exposure at TLV
Blood	EEC	70 µg/dL	Recommended limit for BLL <sup>l</sup>
Blood	Health Canada	10 µg/dL	Blood lead intervention level
Blood	HSE	25 µg/dL	Action levels
		40 µg/dL	Women of reproductive age
		50 µg/dL	People 16-17 years old All other groups

Blood	MAK <sup>m</sup>	300 µg/dL 75 µg/dL	BLW <sup>n</sup> : Women >45 yrs and men BAR <sup>o</sup> : applies to women Does not apply to lead arsenate, lead chromate and alkyl lead compounds
Blood	NIOSH	5 µg/dL	Reference range upper value for children's BLL <sup>l</sup> and reference BLL for adults according to ABLES <sup>p</sup>
Blood	OSHA	40 µg/dL 50 µg/dL and 60 µg/dL	Regulation; cause for written notification and medical exam and return to work after removal. Regulation; cause for medical removal from exposure
Blood	SCOEL	30 µg/dL	Recommended limit for BLL <sup>l</sup>
Drinking water	EPA	15 µg/L 0 µg/L	Action level for public supplies Non-enforceable goal; MCLG <sup>q</sup>
Drinking water	FDA <sup>r</sup>	5 ppb	Bottled water
Food	FDA	Various	Action levels for various foods; example: lead in candy should be limited to a maximum level of 0.1 ppm and in juice to 50 ppb; action level of 0.5 µg/dL for lead in infant food products; lead-soldered food cans now banned;
Food	JECFA <sup>s</sup>	25 µg/kg body weight/week	Provisional Tolerable Weekly Intake (PTWI)
Paint	CPSC <sup>t</sup>	90 ppm (0.009%)	Regulation; by dry weight. New standard for lead in household paint and similar surface coatings in children's products, and some furniture, for adult and children, children's toys, jewelry, etc.
Soil (residential)	EPA	400 ppm (play areas) 1200 ppm (non-play areas)	Soil screening guidance level; requirement for federally funded projects only (40 CFR Part 745, 2001)
Floors	EPA and HUD <sup>v</sup>	108 µg/m <sup>2</sup>	Hard-surfaced and carpeted; New hazard standards for lead in paint, dust and soil, starting June 21, 2019

<sup>a</sup>ACGIH – American Council of Government and Industrial Hygienists

<sup>b</sup>TLV/TWA – Threshold Limit Value/Time Weighted Average

<sup>c</sup>CCOHS – Canadian Centre for Occupational Health and Safety

<sup>d</sup>EEC – European Council

<sup>e</sup>EPA – Environmental Protection Agency

<sup>f</sup>NAAQS – National Ambient Air Quality Standards

<sup>g</sup>HSE – United Kingdom Health and Safety Executive

<sup>h</sup>NIOSH – National Institute of Occupational Safety and Health

<sup>i</sup>REL – Recommended Exposure Limit

<sup>j</sup>OSHA – Occupational Safety and Health Administration

<sup>k</sup>SCOEL – European Union Scientific Committee on Occupational Exposure Limits

<sup>l</sup>BBL – blood lead level

<sup>m</sup>MAK – German Commission for the Investigation of Health Hazards of Chemical Compounds  
in the Work Area

<sup>n</sup>BLW – Biological guidance value (work related exposure)

<sup>o</sup>BAR – Biological reference value (not work related)

<sup>p</sup>ABLES – Adult Blood Lead Epidemiology and Surveillance

<sup>q</sup>MCLG – Maximum Contaminant Level Goal

<sup>r</sup>FDA – Food and Drug Act

<sup>s</sup>JECFA – U.N. Food and Agriculture Organization (FAO)/ World Health Organization (WHO)  
Expert Committee on Food Additives

<sup>t</sup>CPSC – Consumer Products Safety Commission

<sup>v</sup>HUD – U.S. Department of Housing and Urban Development

<sup>w</sup>PEL – Permissible Exposure Level



## Preventing exposure to the Hazard

Controlling exposures to lead and lead products begins with the provision of education and training for all lab personnel about the hazards of working with lead. At the KAUST Solar Center (KSC), mandatory training is required before access is granted to the lab, in conjunction with KAUST's Department for Health, Safety and Environment. The training includes an educational text and an online quiz that requires successful completion. In addition, KSC drafted a standard operating procedure (SOP) for the handling of lead-based materials, complementing this training with considerations on the usage of required engineering controls, work practices and handling techniques as well as personal protective equipment (PPE) while working with lead-based materials. Part of the SOP details a mandatory blood lead level surveillance program for those who work with lead-based materials. These combined, compulsory measures help to ensure all lab personnel are alerted to work requirements prior to commencement. All KSC educational material is made available online from our website.<sup>15</sup>

Mandatory personal protective equipment (PPE), consisting of lab coat, goggles, gloves and shoes are required for all lab related activities, be it research or periodic cleaning of lab surfaces. A separate contained space for storing PPE for lead users is advisable. We highlight the necessity of double gloving (ideally with extended cuff) when handling lead solutions because solvents such as DMF and DMSO can permeate conventional nitrile gloves with surprising velocity.<sup>16</sup> Still, while other gloves such as butyl rubber or silver shielded gloves may feature smaller permeation rates (better barriers), depending on solvent and manufacturer, nitrile gloves offer the best general protection and a combination of good affordability, availability and practical disposability.<sup>3</sup> Detailed information about suitable choices for PPE can be found in the online presence of NIOSH under personal protective equipment. Specialized PPE for lead decontamination is mentioned further below.

Beyond PPE, the proficient use of engineering controls is critically important for preventing any exposure to lead solids. Gloveboxes provide a contained (inert) environment for working

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3 with air-oxygen-water reactive reagents, making for an effective control device for working  
4 with lead-based materials by containing dusts. Given the toxicity and airborne nature of lead  
5 powders and the inherent risk for cross-contamination, we recommend all work with lead  
6 powders, especially the preparation of lead salt solutions, to be carried out exclusively inside  
7 gloveboxes dedicated to perovskite use only. Appropriate signage should inform users where  
8 lead-based materials are being worked with. Early on, we observed the highest levels of  
9 contamination and spreading of lead material emanating from areas with balances installed in  
10 open atmospheric locations, even with balance enclosures in place. Moving all balances inside  
11 gloveboxes decisively inhibits the spreading of lead material across the lab. Yet, most  
12 gloveboxes are designed such that antechambers are vented with nitrogen coming from the  
13 inside of the glovebox. This creates the possibility to deposit lead powders inside the  
14 antechamber on the tray and walls. Thus, cleaning the antechamber after every usage will  
15 diminish the chances for contamination. We also recommend fitting the nitrogen purge line  
16 of the antechamber with a particulate filter with suitable pore size (we use Swagelok ultra-  
17 high purity gas filters for which the manufacturer specifies a removal rating of >99.99% for  
18 particulates of 0.003  $\mu\text{m}$ ; note, the filter reduces the nitrogen flow rate). For the same reasons,  
19 disposable gloves should be worn in conjunction with the rubber gloves of the gloveboxes.  
20 Isolating lead waste inside the glove box, with one or more zip-lock bags, and disposing  
21 sharps in a hard-plastic container is an easy and effective way to further reduce contamination.  
22 The waste should be disposed of in dedicated hazardous waste containers and should be  
23 handled in accordance with reigning regulations for hazardous waste treatment.  
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40 We have evaluated the use of secondary containment to prevent lead spreading. We found  
41 that it can be useful, especially for transportation. However, it can also promote spreading  
42 lead, if not handled appropriately. For instance, in antechamber use, if lead powder happens  
43 to deposit inside the antechamber during the venting mechanism, it may promote spreading  
44 of lead contamination out of the glovebox. Frequent wet cleaning of the antechamber is more  
45 effective in this case.  
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51 In fume hoods, good housekeeping with regular cleaning is again vital. We find it helpful to  
52 cover the working area with high liquid absorbency paper (e.g. Whatman high absorbency  
53 protector) which we replace at the end of each day.  
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3 Creating separate lab spaces for designated work practices and handling techniques involving  
4 lead has proven particularly effective, especially in preventing cross-contamination. In areas  
5 of heavy usage, stricter requirements are imposed, for example, by introducing the  
6 requirement for head covers, disposable boots and adhesive dust mats by the entrance doors.  
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8 These labs or lab areas should be clearly labelled for identification. Ideally, sets of  
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10 laboratories should be set up in a manner that isolates work type and equipment, e.g. a Wet  
11 Lab, Device Fabrication Lab, Characterization Lab, etc. Lead clean up material, e.g., wet  
12 wiper, should be placed throughout the labs for quick and periodic cleaning and should be  
13 disposed of in the same way as lead contaminated waste.  
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3 Figure 1. Stepwise graphical representation of how to safely remove used gloves. The first  
4 step requires pinching the glove at the wrist level, pulling it away from the skin and then  
5 peeling it away from the hand, allowing it to turn inside out. After securing the removed  
6 glove, the ungloved hand is used to remove the other glove by sliding the fingers of the  
7 ungloved hand between the glove and the skin of the wrist and rolling the second glove down  
8 the hand. Adapted from Centers for Disease Control and Prevention.<sup>17</sup> Figure 1 was created  
9 by Heno Hwang, scientific illustrator at King Abdullah University of Science and Technology  
10 (KAUST).  
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20 It is reinforced that PPE is only a temporary barrier for protection. Therefore, gloves must be  
21 changed often and in a way that minimizes the chances for skin contact. A safe way of  
22 removing used gloves is depicted in Figure 1. We believe disposal of contaminated lab coats  
23 following the same protocols used with hazardous waste is necessary for the sake of  
24 preventing contamination of drain water, which is a possibility when opting for a laundry  
25 service for cleaning.  
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31 In summary, minimizing the handling of lead in open atmospheric locations (fume hoods,  
32 bench tops, etc.) combined with stringent housekeeping practices, by cleaning up surfaces  
33 after every lead handling process will greatly reduce the risk for lead spreading.  
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## 41 Contamination Testing and Decontamination

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43 Lab surface testing for lead can be carried out using 3M's lead testing swabs (*Lead Check<sup>TM</sup>*), and  
44 this is practiced by many labs. This colorless swab contains a proprietary chemical indicator  
45 mixture that changes color from colorless to red in the presence of lead. The manual states that  
46 "pale pink indicates the presence of a minimum of 1-2  $\mu\text{g}$  of lead ion ( $\text{Pb}^{2+}$ ) on the area tested."  
47 Yet, it is not clear if the swab reacts exclusively to  $\text{Pb}^{2+}$  or if other forms of lead, other elements  
48 and material combinations may trigger a color change as well. Importantly, the test was developed  
49 as a "*Qualitative Spot Test Kit for Lead in Paint*" with a sensitivity of 1 mg/cm<sup>2</sup>. A more detailed  
50 report (sponsored by 3M) on the performance evaluation of this qualitative test can be found in  
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3 reference.<sup>18</sup> It is clear from this report that only paint containing  $\text{Pb}^{2+}$  was used as part of the testing  
4 protocol ( $\text{PbCO}_3$ ,  $\text{PbCrO}_4$ ). To evaluate the effectiveness of the 3M testing swabs in our lab  
5 environment we compared the results of this test with a quantitative in-house test that consisted of  
6 rubbing a cotton swab on a contaminated area and post-analysis of the cotton swab using  
7 inductively coupled plasma spectroscopy (ICP). We then tested the same areas with both  
8 techniques after successive decontamination steps using a commercial lead cleaning detergent. A  
9 detailed description of the contamination testing and decontamination procedure can be found in  
10 the Supporting Information. The above two procedures (testing for contamination and  
11 decontamination) were utilized in conjunction with side-by-side comparison of 3M's *Lead Check*  
12 kits on various locations throughout a range of labs. The *Lead Check* swabs were used (as per  
13 manufacturer instructions) after the contamination testing procedure was performed but before the  
14 decontamination procedure was done. The results are listed in Table 2. Additional results are  
15 shown in the Supporting Information. We used other lead testing kits, for example, the *Full*  
16 *Disclosure Kit* from SKC but the results were not conclusive.


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18 In addition to analyzing methods for testing lead contamination and decontamination levels, a  
19 head-to-head comparison to determine the effectiveness of a few chosen cleaners was performed  
20 (Figure 2). Hygenall's *Lead Off* and Esca Tech's *D-Lead* were selected as they have been used in  
21 other Perovskite labs for lead decontamination purposes. Standard *Dial* liquid hand soap (dilution  
22 of 4 mL/L of deionized water) was also used to determine the effectiveness of a standard and  
23 readily available soap/detergent. The results are listed in Table 3.




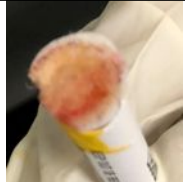
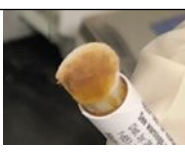


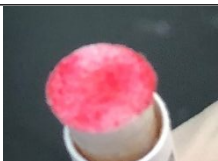
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25 The *Lead Check* swabs show considerable variability in performance. This variability could  
26 possibly be due to the valence state of the lead under evaluation (elemental Pb,  $\text{Pb}^{2+}$  or  $\text{Pb}^{4+}$ ). Even  
27 at triple digit ppm values, as measured by ICP, the swabs barely exhibited a red color, while in the  
28 case of the *fume hood* results, where ICP showed sub-single digit ppm values, the swabs turned  
29 red. This suggests that the swabs may only be specific for a certain ionic species of lead (most  
30 likely  $\text{Pb}^{2+}$ ), or the swabs are detecting another metal. The 3M *Lead Check* swabs have no visible  
31 expiration date and, based on the results of the present study, may not be the most suitable method  
32 of lead detection for laboratory environments or applications. Similarly, a previous study by  
33 Korfmacher et al. reported a false negative rate of 64% and concluded that *Lead Check* swabs  
34 could not reliably detect levels of lead in dust above  $40 \mu\text{g}/\text{ft}^2$ .<sup>19</sup> We note that in order to obtain  
35 quantitative masses of lead per surface area probed, the application of a smear tab (e.g., from  
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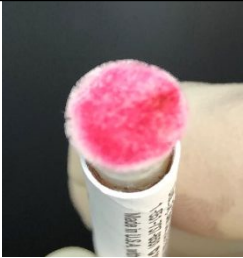
Whatman) across a well-defined area, weigh out of the amount of material adsorbed by the tab and then calculation of the mass of lead on that tab from a ppm measurement like ICP would be required. This would allow a comparison with housekeeping standards for lead as stipulated for example by the U.S. Department of Housing and Urban Development (the permissible amount of leaded dust on floors was just changed from 40 to 10  $\mu\text{g}/\text{ft}^2$ ).<sup>20</sup>

When evaluating the performance of the cleaners (Figure 2 and Table 3), the results show that they are effective in reducing lead contamination levels, with some noticeable differences. The *Dial* liquid hand soap was exceptionally effective and considering its wide availability made it, in our opinion, clearly the best overall choice among the three tested options. Of the specialized cleaners, the *Lead Off* performed best in our tests, whereas the *D-Lead* was not as effective. Importantly, the experimental results clearly illustrate that a single cleaning can significantly reduce the contamination levels. This should reassure lab users that lead decontamination can be effectively accomplished.

**Table 2.** Lead contamination detection level comparisons – ICP vs. Lead Check swabs. A new test swab was used after every cleaning step. The explanation in parentheses refers to the cleaning method used.

Cleaning order	Detected ppm Pb (via ICP)	Image of 3M Lead Swab Check after wiping surface	Practical Quantitation Limit
<b>Fume Hood Experiment (<i>Lead-off</i> Spray Solution)</b>			
Before cleaning	34.4		0.02 ppm

After 1 <sup>st</sup> cleaning	0.7		
After 2 <sup>nd</sup> cleaning	0.4		
After 3 <sup>rd</sup> cleaning	0.3		
<b>Analytical Balance Area Experiment (<i>Lead-off Spray Solution</i>)</b>			
Before cleaning	182.4		
After 1 <sup>st</sup> cleaning	Not Detected		0.02 ppm
After 2 <sup>nd</sup> cleaning	0.3		
After 3 <sup>rd</sup> cleaning	Not Detected		
<b>Bench Top Surface Experiment (<i>Lead-off Wet Wipes</i>)</b>			
Before cleaning	1.9		0.2 ppm

After 1 <sup>st</sup> cleaning	Not Detected		
After 2 <sup>nd</sup> cleaning	Not Detected		
After 3 <sup>rd</sup> cleaning	0.56		

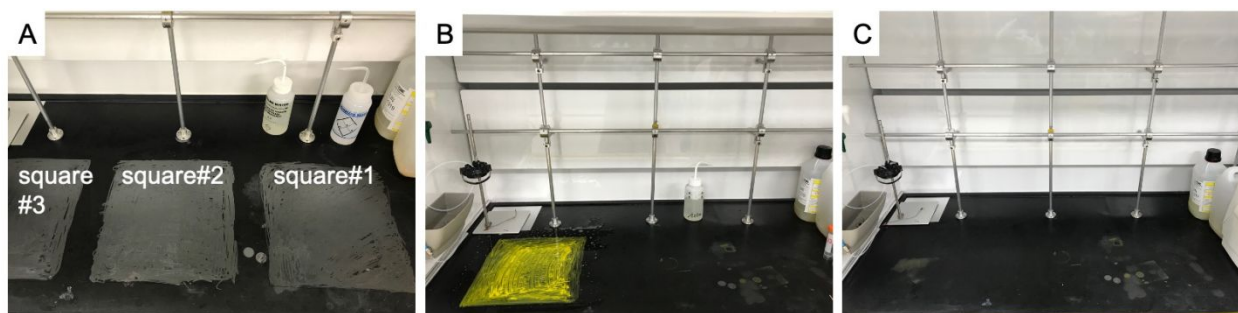


Figure 2. Probing the effectiveness of detergents/soap to remove lead from the surface of a fume hood. A) 1 ml of a perovskite precursor solution in a 4:1 mixture of Dimethylformamide and Dimethyl Sulfoxide containing Lead Bromide, Lead Iodide, Cesium Iodide, Methylammonium Bromide, Formamidinium Iodide (all with a 1.7M concentration) was dropped on and spread across similar areas in the form of squares. The surface was blow dried using a heat gun. B) Photograph after cleaning the right and center piece and before cleaning the left contaminated piece. The contaminated area on the left turned yellowish upon sprinkling the area with soap water (*Dial* soap) but lead was removed effectively after only one cleaning cycle (Figure 2C). C) Photograph of the three purposely contaminated areas after completing the cleaning cycles described in Table 3.



**Table 3.** Comparison of performance of lead decontamination cleaners. Square #1, #2, #3 corresponds to the areas shown in Figure 2A from right to left.

Cleaner	Description	ppm Pb	Practical Quantitation Limit
<b>Square #1</b>			
Lead-Off	Before cleaning	2261	0.02 ppm
Lead-Off	After 1 <sup>st</sup> cleaning	159	
Lead-Off	After 2 <sup>nd</sup> cleaning	88	
Lead-Off	After 3 <sup>rd</sup> heavy cleaning	24	
Lead-Off	After 4 <sup>th</sup> heavy cleaning	<0.2	
<b>Square #2</b>			
D-Lead	Before cleaning	1918	0.02 ppm
D-Lead	After 1 <sup>st</sup> cleaning	316	
D-Lead	After 2 <sup>nd</sup> cleaning	339	
D-Lead	After 3 <sup>rd</sup> cleaning	261	
D-Lead	After 4 <sup>th</sup> cleaning	104	
*After heavy Lead-Off use (2 <sup>nd</sup> square)	After 5 <sup>th</sup> cleaning	<0.2	
<b>Square #3</b>			
Dial Solution	Before cleaning	1878	0.02 ppm
Dial Solution	After 1 <sup>st</sup> cleaning	20	
Dial Solution	After 2 <sup>nd</sup> cleaning	<0.2	
Dial Solution	After 3 <sup>rd</sup> cleaning	<0.2	
Dial Solution	After heavy cleaning with dial solution	<0.2	

## Lab Decontamination and Waste Management

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3 We faced the challenge of decontaminating a lab and several gloveboxes that were previously  
4 heavily used for perovskite research. Several conclusions emerged from this experience. Perhaps  
5 the most important one is that it is possible to decontaminate lead salts on a large scale to the  
6 limit of the ICP's detection ( $<0.1$  ppm) using conventional cleaning methods. As shown in the  
7 previous section, commercial lead detergent or even hand soap can remove lead stains. Other  
8 reports suggest adding a lead binding agent to soap solution to enhance the efficiency with which  
9 lead is removed. A common binder is TSP (trisodium phosphate). This is a component of  
10 most lead detergents. We advise rinsing off the surface with water after cleaning with detergent  
11 to fully remove the soap and thus any remaining lead residue. Several wet cleaning cycles with  
12 detergent might be necessary until left with an untraceable amount of lead. Planning the  
13 decontamination is critical, starting with cleaning supply, contamination detection, waste  
14 handling and personal protective lab gear that is impermeable to particulates and chemically  
15 resistant, much beyond common lab PPE. We opted for full body hooded coveralls and boot  
16 covers from DuPont's Tyvek IsoClean product line. We used NIOSH approved respiratory  
17 masks with solvent and particulate (P100) filter from Honeywell. It is advisable to choose  
18 respiratory filters consisting of a HEPA cartridge and a charcoal filter. Here, PPE should be  
19 considered as one-time use only. Respiratory filters can be reused and should be replaced  
20 following manufacturer instructions.

21  
22 Usually, the decontamination process will accumulate a large amount of contaminated waste and  
23 it is important to have a waste management strategy in place that will allow appropriate  
24 hazardous waste disposal. After decontaminating an area, a reliable test should be applied to  
25 ensure that lead has been effectively removed. For a quick qualitative test, 3M swabs are an  
26 acceptable option. More reliable testing will require sophisticated chemical analytics like ICP.  
27 Finally, protective clothing should be removed with great care before exiting the lab. It should be  
28 disposed of together with other contaminated waste material.

29  
30 In a future perspective, we emphasize that the recovery of lead as well as other hazardous  
31 materials such as cadmium, antimony, fluorine-based polymers and other elements/components  
32 (e.g., aluminum, silver, copper, indium, gallium, tellurium, glass) used in manufacturing solar  
33 panels is critically important from an environmental, safety and global health perspective. A  
34 1GW installation of perovskite solar panels would generate an estimated 3450 kg of lead waste  
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3 at the end of its lifecycle (assuming a 500 nm film and 20% power conversion efficiency). In  
4 most countries, waste associated with PV panels falls under the classification of “general waste”,  
5 which may be classified as hazardous waste. In the US, for instance, there is no dedicated  
6 national program or requirement to safely dispose of solar panels. The regulations fall under the  
7 Resource Conservation and Recovery Act (RSCA), which is enforced by the US Environmental  
8 Protection Agency (EPA). The European Union (EU) was the first region to adopt PV-specific  
9 waste regulations. The EU imposes that PV module manufacturers that sell to the European  
10 market finance the costs of collecting and recycling end-of-life PV panels. This mandate is  
11 expected to create a reliable and economically viable recycling industry. The International  
12 Renewable Energy Agency (IRENA) and the International Energy Agency’s Photovoltaic Power  
13 Systems Programme (IEA-PVPS) are taking leading roles in the context of regulating the  
14 disposal of end-of-life photovoltaic panels.<sup>21</sup>  
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## 27 **Medical Surveillance**

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30 Blood testing is a necessary means to monitor the levels of lead in the body.<sup>22</sup> It is a predictive  
31 indicator for medical anomalies associated with lead. Recent results from studies looking into  
32 health implications at low lead concentrations in blood carried out under the supervision of  
33 NIOSH and AOEC, prompted our institution to adopt guidelines and reference standards based  
34 on these two organizations to monitor occupational exposure to lead in our labs.<sup>23</sup> Based on these  
35 recent studies, NIOSH advises 5 µg/dL (five micrograms per deciliter) as the reference blood  
36 lead level for adults. At our institution, every user of our facilities working with lead-based  
37 materials is required to undergo medical surveillance and commits to adhering to the following  
38 procedure:  
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46 a) Blood Lead Level (BLL) testing:

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49 i. Shall be performed before the first assignment to an area that contains lead-based  
50 materials. This value shall be used as a future reference baseline for the  
51 individual.  
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55 ii. After 6 months of the first assignment.  
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3           iii.    Once a year for individuals who had a Blood Lead Level (BLL) of less than 5  
4                     $\mu\text{g}/\text{dL}$  in two consecutive tests or every 6 months for individuals with BLL > 5  
5                     $\mu\text{g}/\text{dL}$  during the preceding 6 months.  
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9           iv.    Individuals will not be permitted to carry out lead-based work, if a BLL of more  
10                    than 10  $\mu\text{g}/\text{dL}$  is obtained during medical surveillance. Re-exposure to lead-based  
11                    work should not begin again until the value has dropped to below 10  $\mu\text{g}/\text{dL}$ .  
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15 In our institution's SOP,<sup>15</sup> we ensure that the blood test analysis only monitors levels of lead and  
16 no other vital factors.  
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19 So far, we have performed 62 blood tests within a period of 2 years. The highest reported value  
20 was 3.6  $\mu\text{g}/\text{dL}$ , still well below the established reference lead blood level. We believe it is  
21 important to understand whether under a perovskite research environment such as in our labs any  
22 accumulative effects are observed within at least one full PhD cycle.  
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## 29           **Recommendations**

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31 Perovskite labs should draft Standard Operating Procedures for working with lead compounds to  
32 ensure a safe working environment. We emphasize the importance of a thorough cleaning, ideally  
33 after every use of a surface or end of the day, similar to those employed in a microbiology lab for  
34 example. This will decrease lead contamination levels and therefore decrease the risk of lead  
35 exposure as well.  
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40 Perovskite labs must monitor and supervise their lead use and cleanup to include reliable lead  
41 testing like ICP swab tests. Labs should institute random sampling of surfaces to be performed  
42 semi-annually with results recorded in their Laboratory Safety Plans and be acted upon. Results  
43 should also be sent to EHS/HSE colleagues for further review.  
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47 Based on the results of the tests performed in our laboratories, and the ease at which the proven  
48 levels can be achieved in just one cleaning, we recommend that a threshold level of <5 ppm lead  
49 be stated as the allowable level of lead contamination. Anything higher should require immediate  
50 cleaning.  
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55 This contamination action level (<5 ppm lead) was chosen, based on 3 factors:  
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1. The absence of clear, accepted, regulatory standard for this type of lead work.
2. It is conceivable that the pure form of lead salts that researchers are using in perovskite research is more bioactive and therefore more hazardous than lead contaminated paint, where lead  $\text{Pb}^{2+}$  is embedded in a material mixture.
3. The ongoing experimental evidence to date shows that  $<5$  ppm lead can be achieved in just one cleaning step.

Although it can be argued that such a threshold is rather arbitrary because no threshold for the health effects of lead is demonstrated, we argue that analytically a contamination level of 5 ppm can be readily and accurately measured and provides a benchmark for successful prevention. These guidelines should be reevaluated if further information is forthcoming.

Overall, the set of data presented here clearly illustrates the importance of cleaning frequently to reduce lead contamination and exposures. We believe that by implementing a good housekeeping plan paired with the recommendations put forward in this text, it is possible to create the level of awareness needed to establish a safe working environment in the context of perovskite research. It is our expectation that labs around the world will use these guidelines to review and elevate their own safety standards.

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## Supporting Information Available:

Detailed contamination Testing Procedures; additional lead contamination detection level comparisons. This material is available free of charge via the Internet at <http://pubs.acs.org>.

## Ethics declarations/Competing interests

We note that we do not have any commercial interest in the products mentioned here and that other alternatives may be available on the market.

## References

- (1) Snaith, H. J. Present Status and Future Prospects of Perovskite Photovoltaics. *Nat. Mater.* **2018**, 17 (5), 372–376.
- (2) Correa-Baena, J.-P.; Abate, A.; Saliba, M.; Tress, W.; Jacobsson, J. T.; Grätzel, M.; Hagfeldt, A. The Rapid Evolution of Highly Efficient Perovskite Solar Cells. *Energy & Environmental Science* **2017**, 10 (3), 710–727.
- (3) Plog, B. A.; Quinlan, P. J. *Fundamentals of Industrial Hygiene*, 6th Edition.; Plog, B. A., Quinlan, P. J., Eds.; National Safety Council, **2012**.
- (4) Tchounwou, P. B.; Yedjou, C. G.; Patlolla, A. K.; Sutton, D. J. Molecular, Clinical and Environmental Toxicology, Volume 3: Environmental Toxicology. **2012**, 133–164.
- (5) Agency for Toxic Substances and Disease Registry. *Toxicological Profile for Lead*, **2019**. Available online at: <https://www.atsdr.cdc.gov/toxprofiles/tp13.pdf> (accessed July 25, 2020)
- (6) Babayigit, A.; Ethirajan, A.; Muller, M.; Conings, B. Toxicity of Organometal Halide Perovskite Solar Cells. *Nat. Mater.* **2016**, 15 (3), 247–251.
- (7) National Toxicology Program Monograph. *Health Effects of Low-Level Lead*, **2012**. Available online at: [https://ntp.niehs.nih.gov/ntp/ohat/lead/final/monographhealtheffectslowlevellead\\_newissn\\_508.pdf](https://ntp.niehs.nih.gov/ntp/ohat/lead/final/monographhealtheffectslowlevellead_newissn_508.pdf) (accessed July 25, 2020)
- (8) Stoughton, R. B.; Fritsch, W. Influence of Dimethylsulfoxide (DMSO) on Human Percutaneous Absorption. *Arch. Dermatol.* **1964**, 90 (5), 512.

- 1  
2  
3 (9) Agency for Toxic Substances and Disease Registry. *Case Studies in Environmental*  
4 *Medicine*, **2017**. Available online at: <https://www.atsdr.cdc.gov/csem/lead/docs/CSEM->  
5 [Lead\\_toxicity\\_508.pdf](https://www.atsdr.cdc.gov/csem/lead/docs/CSEM-) (accessed July 25, 2020)  
6  
7  
8  
9 (10) National Research Council; Division on Earth and Life Studies; Board on  
10 Environmental Studies and Toxicology; Committee on Potential Health Risks from  
11 Recurrent Lead Exposure of DOD Firing Range Personnel; Committee on Toxicology.  
12 *Potential Health Risks to DOD Firing-Range Personnel from Recurrent Lead Exposure*,  
13 **2012**. Available online at: <https://doi.org/10.17226/18249> (accessed July 25, 2020)  
14  
15  
16  
17  
18 (11) Occupational Safety and Health Administration (OSHA). *Lead in Construction*, **2004**.  
19  
20  
21 (12) Lanphear, B. P.; Rauch, S.; Auinger, P.; Allen, R. W.; Hornung, R. W. Low-Level  
22 Lead Exposure and Mortality in US Adults: A Population-Based Cohort Study. *Lancet*  
23 *Public Heal.* **2018**, 3 (4), e177–e184.  
24  
25  
26 (13) O’Connor, D.; Hou, D.; Ok, Y. S.; Lanphear, B. P. The Effects of Iniquitous Lead  
27 Exposure on Health. *Nat. Sustain.* **2020**, 3 (2), 77–79.  
28  
29  
30 (14) Association of Occupational and Environmental Clinics (AOEC). *Medical Management*  
31 *Guidelines for Lead-Exposed Adults*, **2007**. Available online at:  
32 [http://www.aoec.org/documents/positions/MMG\\_FINAL.pdf?id=10.26616/NIOSH PUB2012](http://www.aoec.org/documents/positions/MMG_FINAL.pdf?id=10.26616/NIOSH PUB2012)  
33 164 (accessed July 25, 2020)  
34  
35  
36  
37 (15) <https://ksc.kaust.edu.sa/Pages/Lead-Safety.aspx> (accessed July 25, 2020)  
38  
39  
40 (16) Schwoppe, A. D.; Randel, M. A.; Broome, M. G. Dimethyl Sulfoxide Permeation  
41 through Glove Materials. *Am. Ind. Hyg. Assoc. J.* **1981**, 42 (10), 722–725.  
42  
43  
44 (17) Center for Disease Control and Prevention. *How to Remove Gloves*. Available online  
45 at: <https://www.cdc.gov/vhf/ebola/pdf/poster-how-to-remove-gloves.pdf> (accessed July 25,  
46 2020)  
47  
48  
49 (18) Buehler, S.; Rhoda, D. *3M Leadcheck Swabs Qualitative Spot Test Kit for Lead in*  
50 *Paint*, **2012**. Available online at: <https://www.epa.gov/sites/production/files/documents/3M->  
51 [leadcheck-report.pdf](https://www.epa.gov/sites/production/files/documents/3M-) (accessed July 25, 2020)  
52  
53  
54  
55  
56  
57  
58  
59  
60

1  
2  
3 (19) Korfmacher, K. S.; Dixon, S. Reliability of Spot Test Kits for Detecting Lead in  
4 Household Dust. *Environ. Res.* **2007**, 104 (2), 241–249.

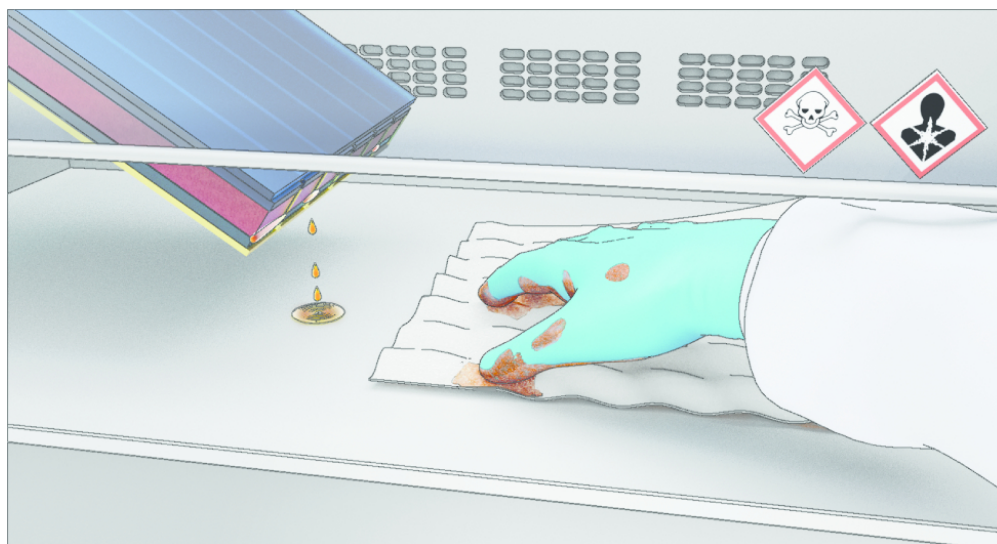
6  
7 (20) U. S. Department of Housing and Urban Development. Office of Healthy Homes and  
8 Lead Hazard Control. *Guidelines for the Evaluation and Control of Lead-Based Paint*  
9 *Hazards in Housing*, **2012**. Available online at:  
10 [https://www.hud.gov/sites/documents/SECOND\\_EDITION\\_2012.PDF](https://www.hud.gov/sites/documents/SECOND_EDITION_2012.PDF) (accessed July 25,  
11 2020)

12  
13 (21) IRENA and IEA-PVPS. *End-of-Life Management: Solar Photovoltaic Panels*.  
14 International Renewable Energy Agency and International Energy Agency Photovoltaic  
15 Power Systems, **2016**. Available online at:  
16 [https://www.irena.org/documentdownloads/publications/irena\\_ieapvps\\_end-of-](https://www.irena.org/documentdownloads/publications/irena_ieapvps_end-of-life_solar_pv_panels_2016.pdf)  
17 [life\\_solar\\_pv\\_panels\\_2016.pdf](https://www.irena.org/documentdownloads/publications/irena_ieapvps_end-of-life_solar_pv_panels_2016.pdf) (accessed July 25, 2020)

18  
19 (22) Kosnett, M. J.; Wedeen, R. P.; Rothenberg, S. J.; Hipkins, K. L.; Materna, B. L.;  
20 Schwartz, B. S.; Hu, H.; Woolf, A. Recommendations for Medical Management of Adult  
21 Lead Exposure. *Environ. Health Persp.* **2007**, 115 (3), 463–471.

22  
23 (23) The National Institute for Occupational Safety and Health (NIOSH). Adult Blood Lead  
24 Epidemiology and Surveillance (ABLES). Available online at:  
25 <https://www.cdc.gov/niosh/topics/ables/description.html> (accessed July 25, 2020)





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