

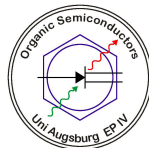
# Polymeric hole injection layers for perovskite-based Light-Emitting Diodes

Colloquium Prepared for the Degree:  
Master of Science  
in  
Physics

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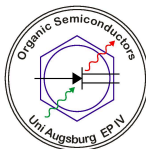
15.11.2018



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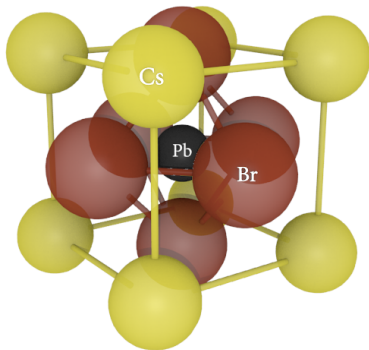
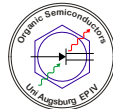
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# What is a perovskite?

A perovskite is a crystal structure, that features unique dielectric properties.

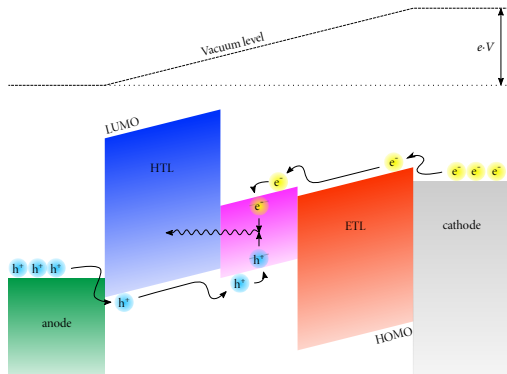


- Perovskite is the retained name for  $\text{CaTiO}_3$ . Calling a material a perovskite refers to its crystal structure being the same as for  $\text{CaTiO}_3$ .
- A perovskite has a structure of  $\text{ABX}_3$ .
- Nobel prize for the first high temperature super conductor.
- High electric conductivity and high absorbance.

**Figure 1:** Illustration of the perovskite structure with the  $\text{CsPbBr}_3$  perovskite as example.

# Basic OLED stack design

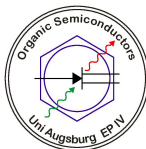
An Organic Light-Emitting Diode (OLED) consists of anode, cathode and functional organic layers in between.



- Electrons and holes are injected by the cathode and anode respectively.
- The ETL and HTL have high mobilities for the respective charge carrier.
- Charge carriers meet to form excitons, which will decay with the emission of a photon.

**Figure 2:** Simplified illustration of processes, leading to light emission, in OLEDs.

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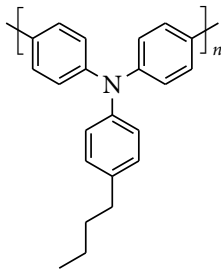


# Developing a suitable hole injection site

poly-TPD has several potential advantages for application in a perovskite based OLED.



Using poly-TPD as additional HIL<sup>a</sup> has advantages:



- Optical optimization by controlling interference effects.
- Electron blocking ability, due to a high LUMO<sup>b</sup>.
- Dried film insoluble to most solvents.
- Enhanced hole injection properties in low HOMO<sup>c</sup> emitting materials, enabled by a low HOMO of poly-TPD itself.
- Chemical shielding between the HIL underneath and the layer above.

Figure 3: poly-TPD

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<sup>a</sup> HIL = hole injection layer

<sup>b</sup> LUMO = lowest unoccupied molecular orbital

<sup>c</sup> HOMO = highest occupied molecular orbital

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# Investigation on optical properties of (MA)PbI<sub>3</sub>

The optical properties are well-suited to verify a perovskite formation.



## Experimental Details:

- Precursors PbI<sub>2</sub> and (MA)I<sup>a</sup> have been dissolved in DMF<sup>b</sup> with 1 M concentration.
- Two solutions have been chosen for the experiment:
  - ▶ PbI<sub>2</sub> in DMF (1 M).
  - ▶ PbI<sub>2</sub> in DMF (1 M) and (MA)I in DMF (1 M) mixed at 1:1 ratio (volumetric).
- The solutions are spin coated at 5000 rpm for 30 seconds on glass substrates and subsequently heated to 90°C for 5 minutes.
- The reflexion and transmission is measured in air.

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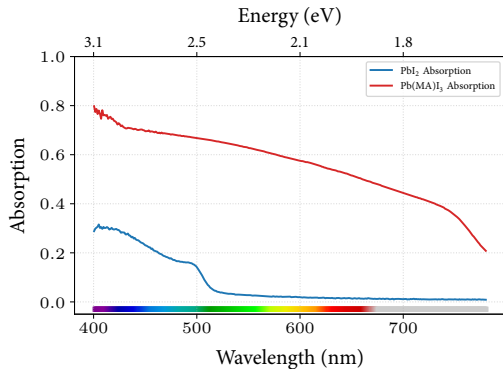
<sup>a</sup> MA = Methylammonium

<sup>b</sup> DMF = Dimethylformamide

# Investigation on optical properties of (MA)PbI<sub>3</sub>



The optical properties of PbI<sub>2</sub> are negligible for the mixed solvent's sample, indicating a full (MA)PbI<sub>3</sub> perovskite formation with few residual PbI<sub>2</sub>.



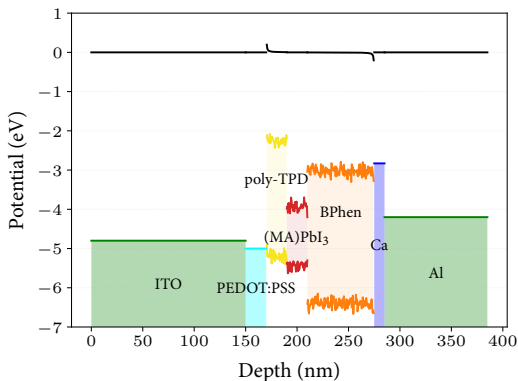
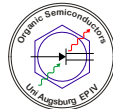
- The PbI<sub>2</sub> absorption:  $\lambda < 500$  nm.
- The (MA)PbI<sub>3</sub> absorption: whole visible spectrum.
- The characteristic trait of (MA)PbI<sub>3</sub> to have blackish color respectively having high absorbance can be verified.<sup>[1]</sup>

Figure 4: Absorption spectra measured by a spectrophotometer.

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# Introduction of (MA)PbI<sub>3</sub> into an OLED stack

A standard electron injection site has been chosen, for hole injection the poly-TPD-based system has been applied.



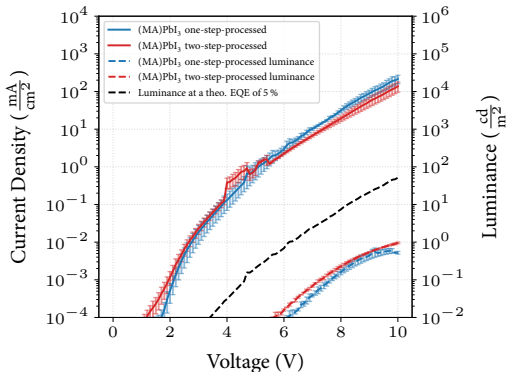
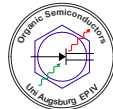
- The hole injection is performed by a PEDOT:PSS and poly-TPD layer of total thickness of 40 nm.
- The emitter is deposited by two different procedures.
  - ▶ The one-step-process: deposition by a (MA)PbI<sub>3</sub> solution.
  - ▶ The two-step-process: subsequent deposition of PbI<sub>2</sub> and (MA)I.
- For electron injection a 70 nm thick BPhen<sup>a</sup> layer was used.

**Figure 5:** Schematic energy band diagram of the first (MA)PbI<sub>3</sub>-based OLED sample.<sup>[2-8]</sup>

<sup>a</sup> Bathophenanthroline

# Introduction of (MA)PbI<sub>3</sub> in an OLED stack

The (MA)PbI<sub>3</sub> is deposited in two different ways, yielding comparable results.

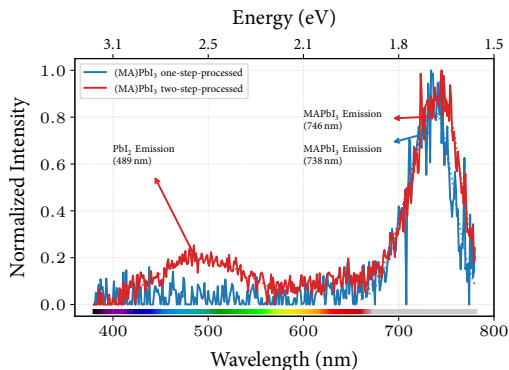


- The two-step-processed OLED shows higher luminance for lower currents.
- The OLED shows no (plain eyed) visible luminance. However devices detect a luminance.

**Figure 6:** Current density and luminance versus driving voltage characteristics of the (MA)PbI<sub>3</sub> based OLEDs.

# Introduction of (MA)PbI<sub>3</sub> in an OLED stack

(MA)PbI<sub>3</sub>'s luminance is at wavelengths reaching the infrared domain.



- Emission in the near infrared.
- Emission energy approx. matching HOMO-LUMO gap of 1.5 eV.
- The green emission can be ascribed to the PbI<sub>2</sub>.

**Figure 7:** Electroluminescent spectrum of the (MA)PbI<sub>3</sub> based OLEDs.

- (MA)PbI<sub>3</sub> films:
  - ▶ A perovskite consisting of PbI<sub>2</sub> and (MA)I, (MA)PbI<sub>3</sub>, can be synthesized.
  - ▶ The absorption of (MA)PbI<sub>3</sub> covers the whole spectrum, increasing towards lower wavelengths.
  - ▶ The (MA)PbI<sub>3</sub> surfaces are partly rough and holey.
  - ▶ Solution processing on hot grounds renders the film more closed.
- (MA)PbI<sub>3</sub> based OLEDs:
  - ▶ The manufactured devices work at low performance, sufficiently for a proof of concept.
  - ▶ The emission maximum is located at the visible spectrum threshold to the infrared spectrum.
  - ▶ For actual light-emitting diodes material systems like (MA)PbBr<sub>3</sub>, are more suited to characterise.

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# Charaterisation of CsPbBr<sub>3</sub> layers

The surface texture and the layer thickness are to be determined.

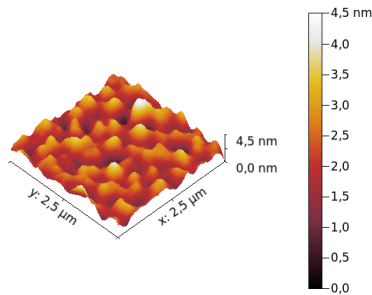
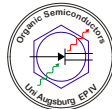


## Experimental Details:

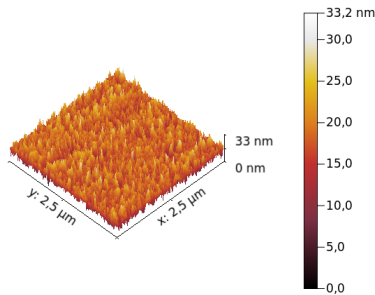
- The CsPbBr<sub>3</sub> quantum dots are present in unknown concentration solved in toluene.
- The solution can be diluted and spin coating speeds can be adjusted.
- Layers of interest are the one grown on poly-TPD. So the standard PEDOT:PSS + poly-TPD layers are applied before spincoating the CsPbBr<sub>3</sub> quantum dots.
- The AFM and profilometer measurement is performed in air.

# Atomic Force Microscopy of CsPbBr<sub>3</sub> layers

The surface texture is controlled by concentration and spin-coating speed.



**Figure 8:** Surface morphology of a CsPbBr<sub>3</sub> quantum dots solution spin-coated at 5000 rpm on a poly-TPD coated substrate.

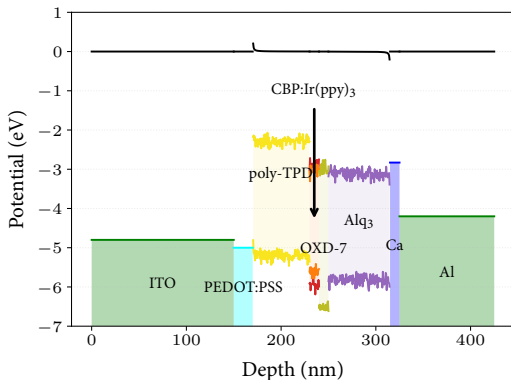
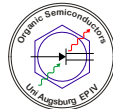


**Figure 9:** Surface morphology of a CsPbBr<sub>3</sub> quantum dots solution spin-coated at 5000 rpm on a PMMA coated substrate.

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# Benchmark of the carrier injection in the OLED.

The charge carrier transport layers are tested with a well known phosphorescent emitter.



- The CBP<sup>a</sup>:Ir(ppy)<sub>3</sub><sup>b</sup> Host-Emitter system is known to have a high efficiency.
- A doping ratio of 8% Ir(ppy)<sub>3</sub> to CBP has been determined to be the most efficient by colleagues of the chair.

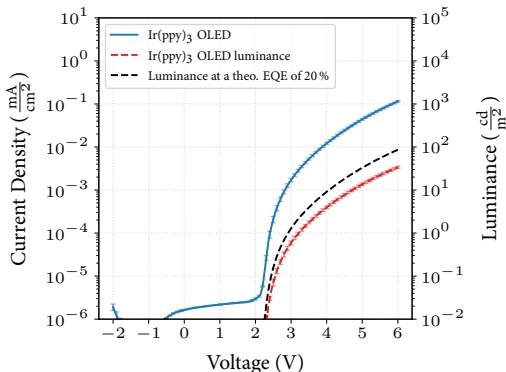
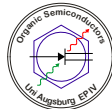
Figure 10: Schematic energy band diagram of the reference OLED sample.<sup>[9, 10]</sup>

<sup>a</sup> CBP = 4,4'-Bis(N-carbazolyl)-1,1'-biphenyl

<sup>b</sup> Tris[2-phenylpyridinato-C2,N]iridium(III)

# Benchmark of the carrier injection in the OLED.

The CBP:Ir(ppy)<sub>3</sub> device is performing well; so the charge transport layers do not have inherently an issue.



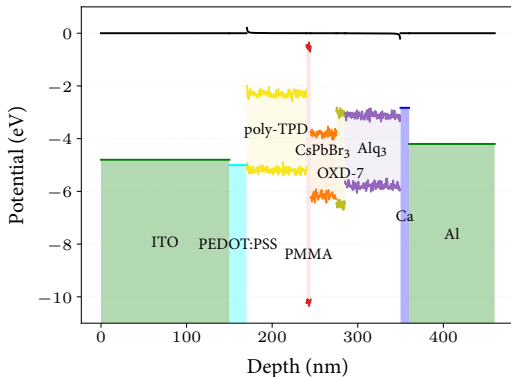
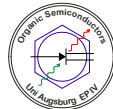
- The luminance is about half as high as the one for an ideal (isotropic) phosphorent emitter.<sup>a</sup>
- Around the onset the sample is nearly ideal.
- No fast degradation for driving voltages up to 10 V can be observed.

**Figure 11:** Current density and luminance versus driving voltage characteristics of the Ir(ppy)<sub>3</sub>-based OLED.

<sup>a</sup> A theoretical device with a constant EQE of 20 %, the same voltage-current dependence, and the same emission spectrum.

# Introduction of CsPbBr<sub>3</sub> in an OLED stack

For electron injection an Alq<sub>3</sub>-OXD7 based system, and for hole injection the poly-TPD-based system has been applied.

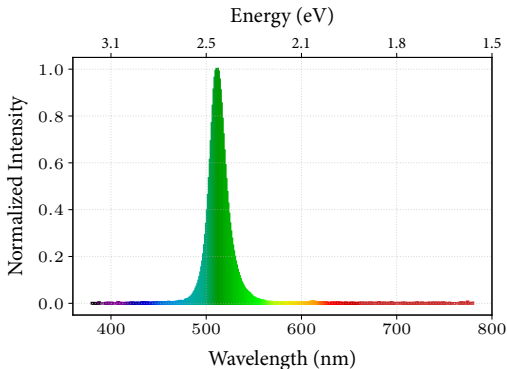
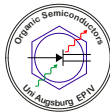


- The hole injection is performed by a PEDOT:PSS and poly-TPD layer of total thickness of 90 nm.
- An optional PMMA spacer is added to the system for better film growth.

**Figure 12:** Schematic energy band diagram of the first CsPbBr<sub>3</sub>-based OLED sample.<sup>[11, 12]</sup>

# Introduction of CsPbBr<sub>3</sub> in an OLED stack

The CsPbBr<sub>3</sub> device's color is as expected a green-cyan tone.



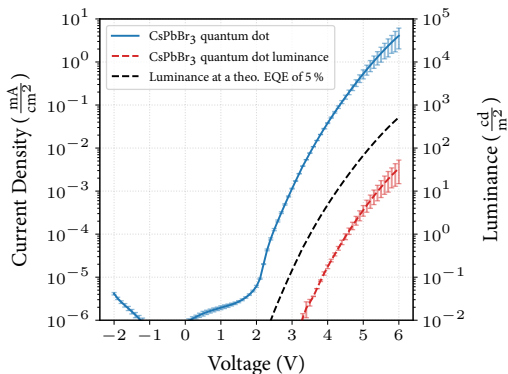
- The peak at 513 nm has a FWHM<sup>a</sup> of approximately 20 nm, which is quite good for application in display devices.
- Other groups get very similar electroluminescent spectra.<sup>[12–14]</sup>

**Figure 13:** Electroluminescent spectrum of the CsPbBr<sub>3</sub> based OLEDs.

<sup>a</sup>FWHM = full width half maximum

# Introduction of CsPbBr<sub>3</sub> in an OLED stack

The CsPbBr<sub>3</sub> device is not performing very well, when considering the high quantum yield of CsPbBr<sub>3</sub> quantum dots in general.<sup>[15]</sup>



- The luminance is an order of magnitude smaller than for an ideal fluorescent emitter.<sup>a</sup>
- The currents are two orders of magnitude higher than the ones for the Ir(ppy)<sub>3</sub> based device.
- The device starts degrading within seconds when driven at voltages higher than 6 volts.

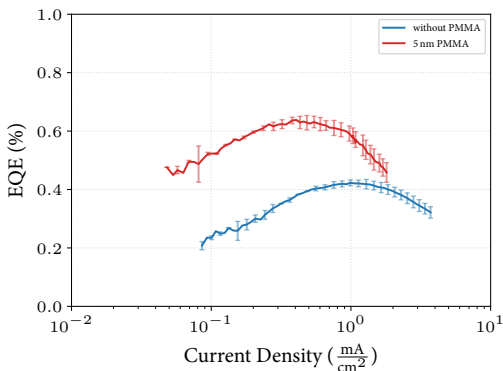
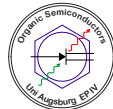
**Figure 14:** Current density and luminance versus driving voltage characteristics of the CsPbBr<sub>3</sub>-based OLEDs.

<sup>a</sup> A theoretical device with a constant EQE of 5 %, the same voltage-current dependence, and the same emission spectrum.



# Influence of PMMA in the CsPbBr<sub>3</sub>-OLED

The PMMA improves the EQE of the devices by a factor of 1.5.

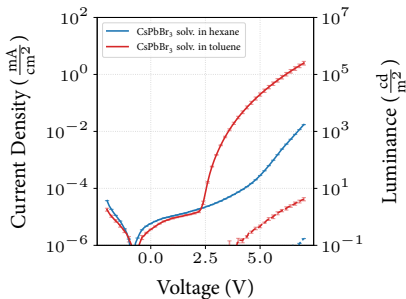


- The PMMA layers leads to an increased EQE.
- However, the EQEs of both devices are an order of magnitude lower than the theoretical limit.

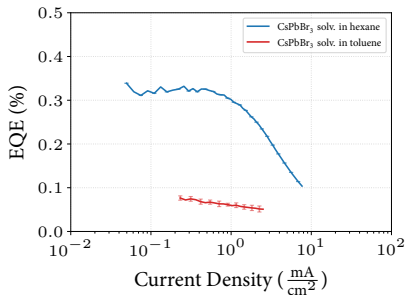
**Figure 15:** EQE versus driving voltage characteristics of the CsPbBr<sub>3</sub>-based OLEDs.

# Performance of toluene-free CsPbBr<sub>3</sub>-OLEDs

Hexane based samples achieve higher efficiencies.



**Figure 16:** Current density and luminance versus driving voltage characteristics of the hexane-CsPbBr<sub>3</sub>-based OLEDs.



**Figure 17:** EQE versus driving voltage characteristics of the hexane-CsPbBr<sub>3</sub>-based OLEDs.

- The hexane based QDs show worse injection, while showing higher EQE.

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# Summary

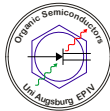
Perovskite based OLEDs could be created. However the efficiency is very low.



- (MA)PbI<sub>3</sub> based OLEDs:
  - ▶ Nearly infrared light is emitted.
  - ▶ Highly depending on environmental conditions during manufacturing.
  - ▶ Very poor performance.
- CsPbBr<sub>3</sub> based OLEDs:
  - ▶ Green-cyan color, small peak width.
  - ▶ Impaired hole injection, which is a major shortcoming in present devices.
  - ▶ Performance depending on used solvent.

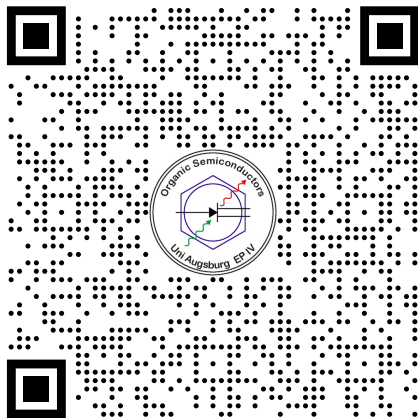
# Outlook

(MA)PbI<sub>3</sub> based OLEDs will be dropped. CsPbBr<sub>3</sub> based OLEDs, need further investigation.



- (MA)PbI<sub>3</sub> based OLEDs:
  - ▶ Switching to (MA)PbBr<sub>3</sub> to get actual optical devices.
  - ▶ For further investigation as IR-diode the initial results were too poor and the required equipment is not present.
- CsPbBr<sub>3</sub> based OLEDs:
  - ▶ Some optical measurement of the CsPbBr<sub>3</sub> layer may help to find other limiting factors.
  - ▶ Easing the hole-injection with new or new combination of the HILs
  - ▶ New batches of CsPbBr<sub>3</sub> with different solvents and ligand density should help finding a suitable hole injection system.
  - ▶ An inverted device, where ITO-ZnO is used for electron injection and hole injection can be done by well-known evaporated organics.

Download this presentation:<sup>a</sup>



Thousand thanks to:

- Professor Wolfgang Brütting.
- Manuel Engelmayer, Florian Graßl and Thomas Morgenstern, for mentoring.
- Matthew Jurow (University of Berkeley) for supplying the CsPbBr<sub>3</sub> quantum dot solutions.
- All other colleagues of the Chair of Experimental Physics IV.
- and You for Your attention.

<sup>a</sup> [https://github.com/TassiloNaujoks/MSc/raw/master/TassiloNaujoks\\_MScColl\\_handout.pdf](https://github.com/TassiloNaujoks/MSc/raw/master/TassiloNaujoks_MScColl_handout.pdf)

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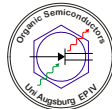


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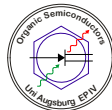
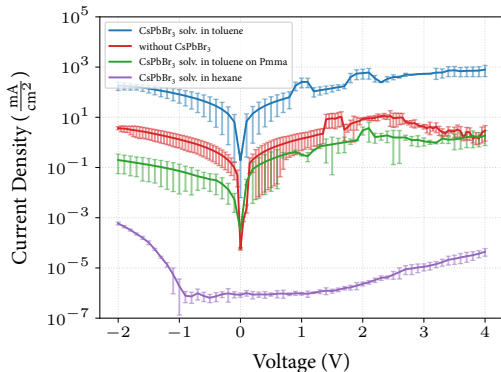
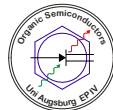


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# Hole Only Devices

The hole conductivity is greatly increased by CsPbBr<sub>3</sub> deposition.

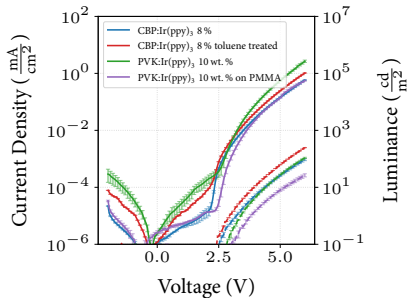
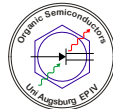


- The CsPbBr<sub>3</sub> solv. in toluene device has a resistance of only 120  $\Omega$ .
- PMMA intervenes, so that the resistance is higher than it is for the CsPbBr<sub>3</sub> free.
- Hexane based CsPbBr<sub>3</sub> devices show extremely low hole conductivity.

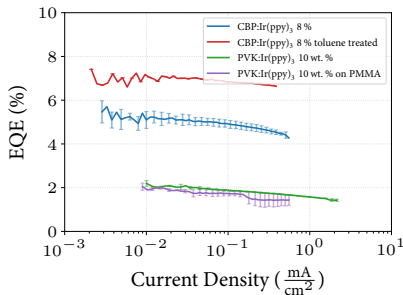
**Figure 18:** Current density versus driving voltage characteristics of hole only devices.

# Solution Processed Ir(ppy)<sub>3</sub>

The toluene susceptibility is still present, but it enhances the efficiency.



**Figure 19:** Current density versus driving voltage characteristics of solution processed Ir(ppy)<sub>3</sub> based devices.

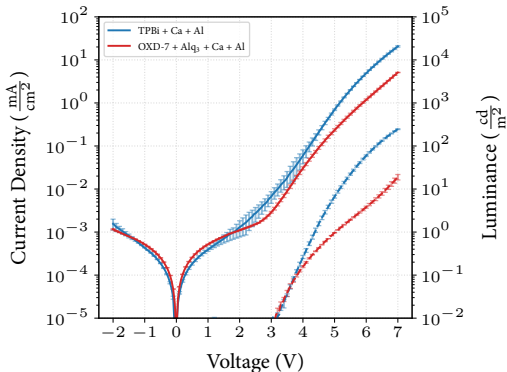
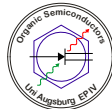


**Figure 20:** EQE versus current density characteristics of solution processed Ir(ppy)<sub>3</sub> based devices.

- PMMA does not increase the EQE, but toluene on poly-TPD does.

# Switching ETL

TBPi increases the current and the luminance.



- TPBi leads to higher currents.
- But it also leads to overproportionally more luminance.
- This matter of facts contradicts the with a bad hole injection.

**Figure 21:** Current density versus driving voltage characteristics of TPBi based devices.

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<https://www.sigmaaldrich.com/catalog/product/aldrich/694924>.
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