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Developing New 2D Materials and Heterostructures for Printed Digital Devices

2D-PRINTABLE - Deliverable report

D6.2 – All-printed, all-nanosheet diodes-type devices

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Project Scientific Abstract

The 2D-PRINTABLE project aims to integrate sustainable large-scale liquid exfoliation techniques with theoretical modelling to efficiently produce a wide range of new 2D materials (2DMs), including conducting, semiconducting, and insulating nanosheets. The focus includes developing the printing and liquid phase deposition methods required to fabricate networks and multicomponent heterostructures, featuring layer-by-layer assembly of nanometer-thick 2DMs into ordered multilayers. The goal is to optimize these printed networks and heterostructures for digital systems, unlocking new properties and functionalities. The project also seeks to demonstrate various printed digital devices, including proof-of-principle, first-time demonstration of all-printed, all-nanosheet, heterostack light-emitting diodes (LEDs). In conclusion, 2D-PRINTABLE will prove 2D materials to be an indispensable material class in the field of printed electronics, capable of producing far-beyond-state-of-the-art devices that can act as a platform for the next generation of printed digital applications.

Public summary

This deliverable reports the first steps toward creating fully printed electronic diodes made entirely from solution-processed two-dimensional (2D) materials. These all-2D devices are important because diodes form the basis of key technologies such as light-emitting diodes (LEDs), photodetectors, and solar cells. Traditionally, 2D devices rely on materials grown under highly controlled conditions, which limits scalability. In contrast, this project aims to enable low-cost, large-area manufacturing by producing nanosheets in liquid form and depositing them using printing and coating methods.

In this work, a novel vertically stacked diode structure was designed using electrochemically exfoliated 2D nanosheets, including p-type niobium-doped WSe_2, n-type MoS_2, and optically active WS_2. A semi-transparent MXene electrode was used as the bottom contact, while a printed silver nanoplatelet film served as the top electrode. Several solution-compatible deposition methods, including liquid–liquid interface assembly, spin coating, and spray coating, were combined to create uniform, multilayer heterostructures over large areas. This demonstrates that complex device stacks can be fabricated entirely from printed nanosheet networks. Further, based on MoS_2 luminescent layer, LEDs based on polymeric and inorganic transport layer materials were fabricated and electroluminescence from MoS_2 was observed, which proves its capability in LEDs applications.

Although the completed all-2D devices did not yet show strong diode rectification or electroluminescence, the results identify the main limitations and the necessary next steps. In particular, the relatively high sheet resistance of the MXene electrode leads to voltage losses, and the photoluminescence quantum yield of the available semiconducting nanosheets remains low. These insights provide clear guidance for improving material quality, energy-level alignment, and electrode performance.

Overall, this deliverable demonstrates the feasibility of constructing multi-layer, all-printed 2D heterostructures and represents an important milestone toward printable LEDs, photodetectors, and solar cells. The work directly supports the project's broader goal of establishing 2D materials as a scalable platform for next-generation printed electronics.

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Abbreviations & Definitions

Abbreviation	Explanation
MXene	Two-dimensional carbides and nitrides of transition metals
LEDs	Light-emitting diodes
SCs	Solar cells
CAN	Acetonitrile
THAB	Tetraheptylammonium bromide
PVP	Polyvinylpyrrolidone
DMF	Dimethylformamide
LLIA	Liquid-liquid interface assembly
AgNPLs	Silver nanoplatelets
EL	Electroluminescence
Eg	Bandgap
q	Elementary charge
Vb	Built-in potential
IE	Ionisation potential
EA	Electron affinity
E	Irradiance
J	Current density
I	Current
V	Voltage
EL	Electroluminescence
PL	Photoluminescence
QY	Quantum yield
ITO	Indium tin oxide
PEDOT:PSS	Poly(3,4-ethylenedioxythiophene) polystyrene sulfonate
poly-TPD	Poly(N,N'-bis-4-butylphenyl-N,N'-bisphenyl)
ZnO	Zinc oxide
PEIE	polyethyleneimine ethoxylated

1 Introduction

Two-dimensional (2D) materials offer exceptional electronic and optical properties, but most all-2D devices demonstrated to date rely on mechanically exfoliated or chemically grown nanosheets. These approaches typically require stringent growth conditions, cleanroom processing, or specialised substrates, making them costly and unsuitable for scalable manufacturing. In contrast, 2D materials produced in liquids from the exfoliation of their bulk counterparts can be processed from solution, enabling low-cost, large-area deposition through printing and coating methods. This compatibility with high-throughput fabrication has already been exploited to produce fully solution-processed transistors, capacitors, and photodetectors. However, one of the most fundamental components in electronic and optoelectronic circuitry, the diodes, has not yet been realised in a fully printed, all-2D architecture.

Diodes based on 2D materials are of particular interest because they underpin key technologies including light-emitting diodes (LEDs), photodetectors, and solar cells. Their operation depends on carrier injection, extraction, and recombination at metal/semiconductor or semiconductor/semiconductor interfaces. With appropriate band alignment, injected carriers can be confined to an active layer to produce electroluminescence, while photogenerated carriers can be separated by built-in potentials to generate photocurrent.

In this deliverable, we report progress toward fabricating vertically stacked, all-2D, fully solution-processed diodes using networks of electrochemically exfoliated nanosheets. By combining p-type and n-type 2D semiconductors with printable metallic electrodes, we aim to establish a scalable heterostructure platform suitable for LED and solar-cell operation. This work represents the first demonstration of a fully printed multilayer 2D diode stack and provides critical insights into material choice, energy-level alignment, and electrode performance needed to enable functional 2D-based optoelectronic devices.

2 Methods and core part of the report

2.1 Background

All-2D based devices were mostly fabricated based on chemically grown or mechanically exfoliated nanosheets or thin films. (1, 2) These methods have strict requirements on the synthesis environment or substrates. 2D materials produced in liquids are compatible with solution-processing techniques that can obtain large-area and uniform thin films in a much scalable way. (3-5)

All solution-processed, all-2D devices were demonstrated previously, such as transistors (6), capacitors (7), and photoconductors (8), etc. However, diodes, representing one of the most important types of electronic components, were never demonstrated. This type of device utilises the electrical conduction across the metal/semiconductor or semiconductor/semiconductor interfaces, to manipulate the carrier injection and extraction behaviours with external biases. With appropriate design, the injected carriers can be confined in the target emissive layer to produce electroluminescence, which is referred as light-emitting diodes. In another case, the carriers generated in the semiconductor upon illumination can be spatially separated by the built-in potential at the interface, which operates as solar cells.

In this report, we present our progress in using novel 2D materials with various functionalities to fabricate vertical stacked, all-2D, and all-solution-processed diodes. The potential of these diodes as LEDs and SCs were explored.

2.2 Procedures

2.2.1 Materials

All materials were obtained from Merck, unless stated otherwise.

2.2.2 Material exfoliation

The raw crystals including niobium doped WSe_2, WS_2, and MoS_2, were supplied by Prof. Zdenek Sofer. A typical electrochemical exfoliation process is conducted as follows. The 2D crystal was held by a copper clip as the anode and graphite foil was used as the cathode. 12.5 mg mL^{-1} tetraheptylammonium bromide (THAB) solution in acetonitrile (ACN) was used. A DC voltage of 7 V was applied for 30-60 min. The intercalated crystal was removed from the clip and was immediately immersed in 2 wt% Polyvinylpyrrolidone/dimethylformamide (PVP/DMF) solution. The solution was ultra-sonicated in a sonic bath for 30 min. To remove the un-exfoliated nanosheets, the dispersion was centrifuged at 3000 rpm for 30 min and the supernatant was collected. The supernatant was further centrifuged at 6000 rpm for 60 min and the resultant supernatant was decanted. The sediment was washed by DMF to remove excess PVP and was finally re-dispersed by anhydrous DMF. This concentrated ink will be used for spin coating. The ink formulation for liquid-liquid interface assembly (LLIA) use isopropanol (IPA) or DMF as the solvent.

2.2.3 Thin film deposition

Spin coating was performed inside a N_2-filled glovebox. The ink with a volume of 50 µL was dispensed onto the substrate. The spinning was set at 1500 rpm for 60 s. The substrate was baked at 100 °C for

5 min before the next coating of the identical layer. After double depositions of the same material, the substrate was baked at 110 °C for 10 min.
Liquid-liquid interface assembly was performed in ambient. The detailed protocols were given in ref (9).
The spray coating was performed in ambient condition. The silver nanoplatelets isopropanol dispersion was sprayed by a Harder and Steenbeck Infinity airbrush which was attached to a Janome JR2300N mobile gantry to spray the dispersion over a programmed area.

2.2.4 Characterisations

The transmission spectra of MXene deposited on glass were collected using Cary 50 spectrometer. The sheet resistance of MXene films was tested by 4-probe measurements with parameter analyser 4200A-SCS. The opto-electronic performance of devices was tested by Keithley 2400 with a photodiode (UV-100L).

2.3 Data Analysis

The data is plotted and is analysed in Origin.

3 Results & Discussion

3.1 Results

3.1.1 Design and fabrication of all-2D diodes

In this report, we propose a heterojunction type diode based on solution-processable 2D materials (**Figure 1A**). In this type of device, p-type and n-type transport layer materials are required to facilitate carrier injection or selective extraction. Glass substrate was used as substrate. Thin films of metallic MXene $Ti_3C_2T_x$ were used as the bottom electrode to achieve both transparency and conductivity. Niobium doped WSe_2 (2 atom%, Nb:WSe_2) were used as the p-type material, which was confirmed previously by an electrochemically gated transistor (10). The n-type material is MoS_2 nanosheet, which exhibit n-type transport behaviours (10) possibly due to the presence of sulphur vacancies. WS_2 nanosheets were used as the optical active layer material. Silver nanoplatelets (AgNPLs) were used as the top electrode materials due to their high electrical conductivity. (11)

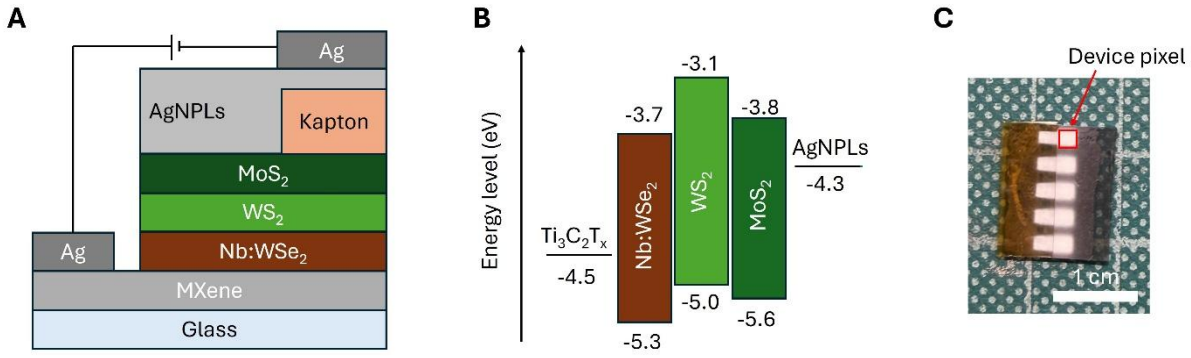

Figure 1 Device design. Schematic demonstrations of device structure (A) and energy band diagrams (B). (C) Photograph of the device after spraying silver nanoplatelets top electrodes.

The energy levels of the rest materials are based on reported values from literature. (12-15) The energy bands of proposed stack of materials are schematically demonstrated in **Figure 1B**. The MoS_2 energy level was determined by Ultraviolet photoelectron spectroscopy and Low Energy Inverse Photoemission Spectroscopy (UPS/LEIPS) by Prof. Georg Düsberg and Sebastian Klenk in UniBw M. The stacking of these materials allows a facile injection and confinement of carriers into WS_2 layer. The light emission from WS_2 could transmit through semi-transparent MXene electrodes.

To realise the proposed stack based on solution-processing techniques, all semiconducting materials were electrochemically exfoliated to obtain large-area flexible nanosheets in liquids. Chemically etched MXene DMF dispersion was obtained from Prof. Valeria Nicolosi. These nanosheets can form conformal junctions and facilitate carrier transport, which will be crucial to maximise the confined carriers in WS_2. Especially, the size-selection protocol after electrochemical exfoliation will yield monolayer-enriched dispersion, so electroluminescence from relatively higher quantum yield monolayer could be obtained. Various methods were used to deposit layers on each other, including liquid-liquid interface assembly (LLIA), spin coating, and spray coating, where LLIA and spin coating can realise uniform large-area coatings (MXene, Nb:WSe_2, MoS_2, and WS_2) and spray coating is able to define the top electrode dimensions.

Firstly, MXene was deposited by LLIA at the water/hexane interface. Multi-layer of MXene was deposited, and it results in an average transmittance of 51.55% in the range of 400-700 nm and a sheet resistance of 28.29 KΩ/sq. Based on the same deposition method, Nb:WSe_2 was deposited twice on top of MXene to maximise surface coverage. WS_2 nanosheets in DMF were spun on Nb:WSe_2 twice, followed by an annealing at 110 °C for 30 min. Due to the relative low mass yield of WS_2 nanosheets, it is time-consuming and wasteful to obtain the concentration of the dispersion by filtration and weighing. Therefore, we recorded the extinction intensity of the pristine WS_2 dispersion at 638 nm (A excitonic absorption peak) to be 0.53 after 210 times dilution. MoS_2 nanosheets (20 mg/mL) were deposited in the same way as WS_2. AgNPLs were spray coated through a shadow mask to define the top electrode area. The silver electrodes were baked at 120 °C for one hour in glovebox. The final device area is defined as the top and bottom electrode overlapping area, which is around 4 mm^2. The complete device is shown in **Figure 1C**.

3.1.2 Opto-electronic measurements of all-2D diodes

The obtained device was measured by conducting I-V sweep from 0-20 V and the electroluminescence (EL) signals were simultaneously recorded by a photodiode on top of the device. The I-V characteristics of the device is shown in **Figure 2A,** and it displayed no detectable EL signals coming from the device. To understand the behaviour, a circuit is schematically drawn in **Figure 2B**. This device includes a resistor R_e owing to the lateral transport in the MXene layer, a shunt resistor owing to the carrier selectivity by energy band alignment when materials are in contact as well as electrical leakage via imperfect contacts, a series resistor due to the intrinsic resistance of semiconductors, and a diode formed by different types of 2D materials at interfaces. The resistance from silver electrodes is negligible. The device did not display the typical exponential increase of current after turn-on voltages. The applied voltage is already much higher than the theoretical voltage required to turn on the device (V_b/q, V_b is the built-in potential formed at interface and q is the elementary charge). For semiconductors used in the current report, their bandgaps are in the range of 1.5-2 eV, so V_b should never exceed the bandgap values. We differentiate the current with respect to voltage to obtain the resistance at each voltage. The device displayed a resistance of around 4 kiloohms even at 20 V. The result indicates that the high sheet resistance of MXene layer will impose a large voltage drop. Moreover, the resistance is not constant but drops with the applied voltage. This indicates that there are no direct contact between top and bottom electrodes, which will result in a constant resistance coming from electrodes otherwise.

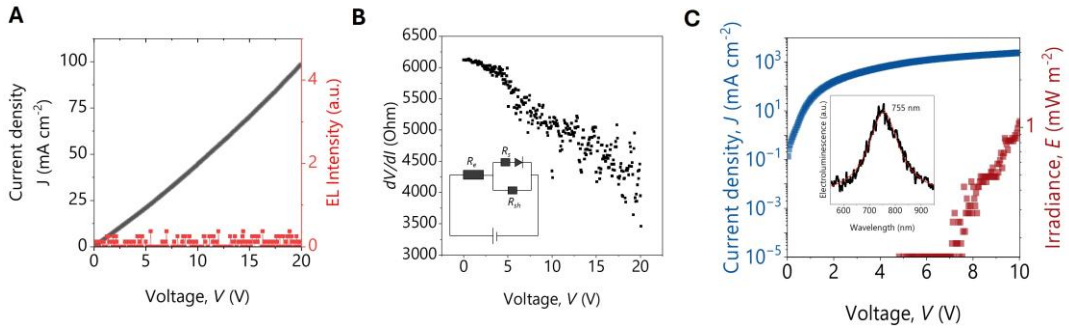

Figure 2 Opto-electronic characterisation. (A) A typical *I-V-EL* curve and (B) *dV/dI* is plotted agianst *V* . The inset is the schematic demonstration of the equivalent circuit of the fabricated device. (C) A typical I-E-V curve of LEDs based on a structure of ITO/PEDOT:PSS/poly-TPD/MoS_2/ZnO/PEIE/Al. The inset is the EL spectrum of the device at 10 V.

In addition, we tested the application of exfoliated MoS_2 nanosheets as the light emissive material in a widely used device structure. Indium tin oxide (ITO) coated glass was used as the transparent cathode. Poly(3,4-ethylenedioxythiophene) polystyrene sulfonate (PEDOT:PSS) with a work function of -5.1 eV was used as hole injection layer material. Poly(N,N'-bis-4-butylphenyl-N,N'-bisphenyl)benzidine (poly-TPD) was used as the hole transport layer material with an ionisation potential (IE) of -5.2 eV and electron affinity (EA) of -2.3 eV. The zinc oxide (ZnO) nanoparticles were used as the electron transport layer material and an ultra-thin polyethyleneimine ethoxylated (PEIE) was used as the interfacial layer to modify the energy levels of ZnO. Aluminium with a work function of -4.2 eV is used as a shallow work function metal to form Ohmic contact with PEIE modified ZnO layer for electron injection. With the proposed device structure ITO/PEDOT:PSS/poly-TPD/MoS_2/ZnO/PEIE/Al, electron injection can be easily injected into MoS_2, while hole injection from poly-TPD may face a potential barrier of around 0.4 eV.

The typical *J*-*V* and radiance L_r-*V* curve are shown in **Figure 2C**. The current density is much higher than that in all-2D devices, which can be ascribed to the high conductivity of ITO substrates. With increasing voltages, the device can be turned on at around 7 V and the irradiance reached to around 1 mW m^{-2}. The EL spectrum was taken, and it has shown a peak at 755 nm from fitting. The result demonstrates the electroluminescence capability of the exfoliated colloidal nanosheets. This type of device will serve as a benchmark for assessing the performance of 2D-material-based LEDs that rely on heterostructures. Continued development of fully 2D-based LEDs will further enhance their efficiency and demonstrate their potential for advanced electroluminescent applications.

3.2 Contribution to project (linked) Objectives

The current deliverable describes the first design and fabrication of all-2D, all-solution-processed diodes. It contributes to the project that 1) it identified the suitable and available 2D materials for forming heterojunction, 2) multi-layered heterostructured devices were fabricated that each layer was fabricated with the most suitable and compatible technique, 3) it verified that the fabrication of this type of device is feasible but more care should be focused on material properties, such as sheet-resistance and photoluminescence, and 4) it proves the electroluminescent capability of electrochemically exfoliated nanosheets.

3.3 Contribution to major project exploitable result

N/A

4 Conclusion and Recommendation

In this report, we demonstrate the design and fabrication of all-solution-processed, all-2D diode type device structures. Firstly, fabricating this type of device should use those atomically thin and large area nanosheets obtained from electrochemical exfoliation. These nanosheets can form closed structures without pinholes and nanosheet alignment is nearly perfect that facilitate the carrier transport. Secondly, doped materials such as niobium doped WSe_2 as used in this report will be necessary as a vast majority of previously investigated materials exhibit n-type transport behaviours. It is of vital importance to have p-type doped material to construct p-n type heterojunction. Thirdly, deposition of various types of nanosheet can be versatile, facile, and industrially viable. In this report, as a demonstration, we used liquid-liquid interface assembly, spin coating, and spray coating. Their individual merits can be utilised, for example, water-hexane interface assembly can form atomically smooth and thickness tuneable nanosheet networks, which is desirable to be used for bottom electrode deposition to form a smooth buried surface. Spin coating can be used in glovebox in the case in which the material's photoluminescence properties require the inert environment protection. Spray coating can be used to pattern the top electrode and define the device area. It is anticipated that other techniques such as aerosol-jet printing or ink-jet printing, could also be used in the future. Fourthly, by fabricating a LED based on polymeric and inorganic zero-dimensional transport layer materials with luminescent MoS_2 nanosheets, electroluminescence can be observed, showing the potential of applications in electroluminescent devices. Fifthly, based on the electrical characterisations on the fabricated devices, the main limiting factor is the relatively high sheet resistance of the semi-transparent MXene layer. The diode rectifying behaviour was not observed due to the high resistance arising from the lateral transport in the bottom electrodes. Further work will be required to improve this metallic layer to obtain comparable low sheet resistance and high optical transmittance to those of indium tin oxide glass. Sixthly, except MoS_2, the energy levels of all other materials are relying on the literature values. These values may not be accurate enough because the starting raw materials and the preparation conditions are not identical. Precise determination of energy levels will be needed to design the device stack and understand the electrical behaviours.

5 Risks and interconnections

5.1 Risks/problems encountered

Risk No.	What is the risk	Probability of risk occurrence[1]	Effect of risk[1]	Solutions to overcome the risk
	The metallic layer cannot main high transmittance at a low sheet resistance	1	2	The MXene could be blended with high conductivity one-dimensional silver nanowires to increase its conductivity.
	The quantum yield of exfoliated nanosheet is too low	1	1	The quantum yield of nanosheets could be improved by chemical functionalisation mentioned in Task 2.

[1)] Probability risk will occur: 1 = high, 2 = medium, 3 = Low

5.2 Interconnections with other deliverables

The deliverable applies the techniques and knowledge developed in Task 3.4, Fabrication of stacked heterostructures by printing, to Task 6.3, Diodes, photodetectors and solar cells.

6 Deviations from Annex 1

In this report, we identified that the metallic MXene layer to be a limiting factor that the diode device cannot fully develop rectifying behaviours. Further, through collaboration with Prof. Ali Javey at UC Berkeley, we found that the photoluminescence quantum yield of semiconducting materials such as MoS_2 is low (below 0.01%). The low quantum yield can be attributed to the n-doping effect from PVP and DMF, in which photoluminescence of the heavily n-doped MoS_2 will be dominated by the low quantum yield negatively charged trions. Therefore, fabricating light-emitting diodes with external quantum efficiency exceeds 10% with the currently available low quantum yield nanosheets are challenging. Further solution-compatible improvements on the photoluminescence properties of nanosheets will be required.

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Project partners:

#	Partner short name	Partner Full Name
1	TCD	TCD THE PROVOST, FELLOWS, FOUNDATION SCHOLARS & THE OTHER MEMBERS OF BOARD, OF THE COLLEGE OF THE HOLY & UNDIVIDED TRINITY OF QUEEN ELIZABETH NEAR DUBLIN
2	UNISTRA	UNIVERSITE DE STRASBOURG
3	UKa	UNIVERSITAET KASSEL
4	BED	BEDIMENSIONAL SPA
5	TUD	TECHNISCHE UNIVERSITAET DRESDEN
6	VSCHT	VYSOKA SKOLA CHEMICKO-TECHNOLOGICKA V PRAZE
7	UNR	UNIRESEARCH BV
8	UniBw M	UNIVERSITAET DER BUNDESWEHR MUENCHEN
9	EPFL	ECOLE POLYTECHNIQUE FEDERALE DE LAUSANNE

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9 Appendix A - Quality Assurance Review Form

The following questions should be answered by all reviewers (WP Leader, reviewer, Project Coordinator) as part of the Quality Assurance procedure. Questions answered with NO should be motivated. The deliverable author will update the draft based on the comments. When all reviewers have answered all questions with YES, only then can the Deliverable be submitted to the EC.

NOTE: This Quality Assurance form will be removed from Deliverables with dissemination level "Public" before publication.

Question	**WP Leader**	**Reviewer**	**Project Coordinator**
	NAME (Organisation)	NAME (Organisation)	Jonathan Coleman (TCD))
1. *Do you accept this Deliverable as it is?*	Yes	Yes	Yes
2. *Is the Deliverable complete?* - *All required chapters?* - *Use of relevant templates?*	Yes	Yes	Yes
3. *Does the Deliverable correspond to the DoA?* - *All relevant actions preformed and reported?*	Yes	Yes	Yes
4. *Is the Deliverable in line with the 2D-PRINTABLE objectives?* - *WP objectives* - *Task Objectives*	Yes	Yes	Yes
5. *Is the technical quality sufficient?* - *Inputs and assumptions correct/clear?* - *Data, calculations, and motivations correct/clear?* - *Outputs and conclusions correct/clear?*	Yes	Yes	Yes
6. *Is created and potential IP identified and are protection measures in place?*	Yes	Yes	Yes
7. *Is the Risk Procedure followed and reported?*	Yes	Yes	Yes
8. *Is the reporting quality sufficient?* - *Clear language* - *Clear argumentation* - *Consistency* - *Structure*	Yes	Yes	Yes