# **Electronic Supplementary Information**

# Effect of temperature and exfoliation time on the properties of chemically exfoliated MoS<sub>2</sub> nanosheets

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#### 1. EXPERIMENTAL

#### 1.1. Materials

Molybdenum disulphide (MoS<sub>2</sub>, powder, <2  $\mu$ m), *n*-butyllithium solution (*n*-BuLi, 1.6 M in *n*-hexane), (anhydrous) *n*-hexane were purchased from Sigma Aldrich and used as received.

#### 1.2. Production of dispersion of ultrathin MoS<sub>2</sub>

Under argon atmosphere, 2.5 mL of anhydrous *n*-hexane as a solvent was added to 0.250 g (1.56 mmol) MoS<sub>2</sub>. After the addition of 2.5 mL of 1.6 M solution of *n*-BuLi in hexane, the dispersion was stirred for 1, 2 and 3 days at room temperature or heated at 55°C in a Schlenck tube. Then, approximately 30 mL of *n*-hexane and 15 mL of Millipore water (18.2 M $\Omega$ •cm in 25°C) was added to the dispersion at the end of the reaction. After the decrease of the gas formation, the dispersion was filtered through 0.45 µm PTFE Omnipore<sup>TM</sup> membranes (Merck). Then, the solid was collected and the intercalated MoS<sub>2</sub> was suspended in the proper amount of Millipore water (18.2 M $\Omega$ •cm in 25°C, 100 times more than weight of intercalated MoS<sub>2</sub>). Obtained dispersion was bath-sonicated (Bandelin Sonorex, 30 kHz) for 1h and centrifuged at 750 rpm (62 g) for 30 min in order to remove non-exfoliated material as sediment. Finally, the obtained dispersion was dialyzed in distilled water for 24h to eradicate residual lithium compounds.

#### **1.3.** Characterization techniques

Samples for thermogravimetric analysis were prepared by freeze-dried ce-MoS<sub>2</sub> from dispersed solutions. The analysis was performed from 25 to 800°C with a ramp of 10°C under nitrogen, on a Mettler Toledo TGA/SDTA851e.

Zeta potential of *ce*-MoS<sub>2</sub> dispersions was recorded on Delsa Nano C Particle Analyzer (Beckman Coulter, Brea, CA, USA).

Dispersed  $MoS_2$  nanoflakes were transferred in a 10 mm path length quartz cuvette and analyzed by UV-Vis spectrophotometry using a Jasco V670 spectrophotometer.

X-ray photoelectron spectroscopy (XPS) analyses were carried out using a Thermo Scientific KAlpha X-ray photoelectron spectrometer with an aluminum X-ray source (energy 1.4866 keV) and working at pressure of  $10^{-8}$ – $10^{-9}$  mbar in the main chamber. The X-ray spot size was settled at 400 µm. Survey spectra were recorded as result of 10 scans with a pass energy of 200.00 eV and a step size of 1 eV, while high-resolution spectra were an average of 30 scans with a pass energy of 50.00 eV and a step size of 0.1 eV. The samples were prepared by a vacuum filtration and dry-transfer method. An

appropriate volume of obtained  $MoS_2$  dispersions after dialysis (1-4 mL, depending on concentration of solution) were vacuum-filtered through 0.45 µm PTFE Omnipore<sup>TM</sup> membrane followed by mechanically pressing the filtered film on a SiO<sub>2</sub>/Si (oxide thickness 230 nm) substrate. After around 1h, the PTFE membrane was peeled off, leaving the transferred  $MoS_2$  film on the substrate.

Raman spectra were recorded at room temperature by a Renishaw Raman Microscope InVia Reflex equipped with a 2D-CCD camera. All samples, which were prepared the same way as for XPS characterization, were focused with a 100x objective and were excited with green laser ( $\lambda$ =532 nm). To characterize the presence of metallic 1T phase, all samples were irradiated with laser power of 0.1% for 80 s (20 accumulations lasting 4 s each). More than 20 data points were averaged to confirm the homogeneity of the samples.

Atomic force microscopy (AFM) imaging was carried out using a Veeco Dimension 3100 operating on a Nanoscope IV control unit under ambient conditions. The samples were prepared by spin coating MoS<sub>2</sub> dispersions (1 mL of dilute solution) on top of SiO<sub>2</sub>/Si substrates, which were first cleaned with acetone and isopropyl alcohol in an ultrasonic bath. Topographic and phase imaging was performed in tapping mode using antimony (n)-doped silicon tips (resonant frequency = 320 kHz, k = 42 N m<sup>-1</sup>; TESPA-V2, Bruker). The thickness and lateral size of the exfoliated MoS<sub>2</sub> samples were determined by the analysis of AFM images with Gwyddion software (version 2.50).

Morphological characterization was performed using a scanning electron microscope (SEM) and a scanning transmission electron microscope (STEM) with focused ion beam FEI Helios NanoLab 66. The samples for SEM were prepared by the dry transfer method (as for XPS and Raman). On the other hand, the samples for STEM characterization were prepared by drop-casting on a lacey carbon copper grid 0.2 mL of dilute solution of exfoliated MoS<sub>2</sub>, followed by solvent evaporation. The analysis of bulk MoS<sub>2</sub> was conducted by dispersing it in water and deposited onto the lacey carbon grid.

Electrical conductivity of *ce*-MoS<sub>2</sub> films was calculated by measuring: i) the sheet resistance (Rs) using a four-probe system (Jandel, RM 3000); ii) the film thickness using a profilometer (KLA-Tencor Alpha-Step IQ). All the measurements were performed under ambient conditions.

Electrical characteristics of MoS<sub>2</sub> were evaluated by measuring the performance of FETs under N<sub>2</sub>-controlled atmosphere (N<sub>2</sub>-filled glovebox). Bottom-contact/bottom-gate FETs were fabricated by using pre-patterned SiO<sub>2</sub>/n<sup>++</sup>-Si substrates (15 mm x 15 mm, Fraunhofer IPMS, Dresden, Germany). These substrates consist of thermally grown SiO<sub>2</sub> (230 nm thick) having Interdigitated Electrodes (IDEs, 30 thick Au onto 10 nm ITO adhesion layer) spaced 2.5  $\mu$ m, yielding a channel width-length (W/L) ratio of 4000. Prior to use, the substrates were cleaned by ultrasonication in acetone and 2-propanol (10 min each), and dried under N<sub>2</sub> flow afterwards. To complete the device fabrication, MoS<sub>2</sub> dispersion, obtained as a result of 1 day reaction at 55°C, was drop-casted onto the clean substrates. Conversely, top-contact/top-gate FETs were fabricated starting from MoS<sub>2</sub> films deposited onto SiO<sub>2</sub>/Si substrates

*via* dry-transfer method (prepared the same way as XPS samples). Then, IDEs were deposited onto the film surface by shadow mask evaporation approach, having 5 nm of Cr adhesive layer and 90 nm of Au. Finally, a drop of EMIM-TFSI ionic liquid was placed onto the device as electrolyte (liquid dielectric), as well as Pt wire partially immersed in it as gate electrode.

## 2. ANALYSIS

## 2.1. Thermogravimetry



**Fig. S 1** Thermogravimetric analysis of freeze-dried *ce*-MoS<sub>2</sub>. The thermograms show greater decline in weight during thermal annealing for sample (1 day, RT) comparing to the other samples, which have similar thermal stability.



#### 2.2. Zeta potential

Fig. S 2 Values of zeta potential measurements depending on duration of intercalation reaction

#### 2.3. X-ray Photoelectron Spectroscopy (XPS)

The deconvolution of obtained high-resolution Mo3d and S2p XPS spectra was demanding, because there are many various components with similar binding energies. In particular, S2p spectra were challenging due to the fact, that separation between the two doublets from metallic 1T phase and semiconducting 2H phase is ~1.2 eV. On the other hand, in the case of Mo3d spectra, that separation is higher (~3.2 eV), thus the calculation of 1T/1T+2H ratio is more accurate.<sup>1</sup>

*ce*-MoS<sub>2</sub> using excess of *n*-BuLi solution as an intercalation agent is generally described as occurring mainly in its 1T phase. However, it depends on the experimental conditions chosen for the intercalation reaction. The Mo 3d core level spectra present various species. The pair of peaks at ~229.1 eV and ~232.3 eV can be assigned to the molybdenum ion on the fourth oxidation stage occurring in 2H-MoS<sub>2</sub> phase. The spectra after intercalation reaction can be fitted with an additional doublet shifted toward lower binding energies by ~0.8 eV with regard to the main 2H-MoS<sub>2</sub> peaks that can be identified as 1T-phase of MoS<sub>2</sub>.<sup>2</sup> Moreover, small contributions from molybdenum oxides are detected as indicated by the orange and violet traces in the Fig. S 3.<sup>1, 3</sup>



**Fig. S 3** Fitted high-resolution Mo3d XPS spectra of ce-MoS<sub>2</sub> after experiment: (a) bulk MoS<sub>2</sub>; (b) 1 day, RT; (c) 1 day, 55°C; (d) 2 days, RT; (e) 2 days, 55°C; (f) 3 days, RT; (g) 3 days, 55°C.

On the other hand, on the S2p spectra, which are presented on Fig. S 4, more peaks can be observed, due to occurring more amount of various species. Beside doublet at ~162 eV and ~163.2 eV that can be assigned to 2H-MoS<sub>2</sub> phase, on the spectra after intercalation reaction the additional pair of peaks from 1T-MoS<sub>2</sub> phase, shifted towards lower binding energies by ~1.2 eV with respect to the main 2H-MoS<sub>2</sub> peaks, is visible. Moreover, three extra pairs of peaks can be separated from the S 2p region after deconvolution of the peaks, that can be assigned to S<sup>2-</sup>, edge S, and also sulphur oxides.<sup>1</sup> The XPS spectra in both Mo 3d and S 2p regions show that after 1 day of experiment at room temperature, exfoliated material contains less metallic 1T phase than exfoliated MoS<sub>2</sub> obtained as a result of reactions conducted for 1 day at elevated temperature as well as after 2 and 3 days. Furthermore, at higher temperature, usually more product in 1T-MoS<sub>2</sub> is formed. Nevertheless, due to the high number of different components with binding energies that are very close together, it is

difficult to fit properly S2p spectra. Even though the S2p and Mo3d core-levels spectra usually yield similar 2H/1T ratios, results for S2p are with much higher uncertainty.

Table S 1 displays average values of percentage content of components on both high resolution Mo3d and S2p spectra of exfoliated  $MoS_2$  determined based on specific peak's surface area.



**Fig. S 4** Fitted high-resolution S2p XPS spectra of *ce*-MoS<sub>2</sub> after experiment: (a) bulk MoS<sub>2</sub>; (b) 1 day, RT; (c) 1 day, 55°C; (d) 2 days, RT; (e) 2 days, 55°C; (f) 3 days, RT; (g) 3 days, 55°C.

| Cond.               | Bulk | 1 day, RT | 1 day, | 2 days, RT | 2 days, | 3 days, RT | 3 days, |  |  |
|---------------------|------|-----------|--------|------------|---------|------------|---------|--|--|
| Comp.               |      |           | 55°C   |            | 55°C    |            | 55°C    |  |  |
|                     | Mo3d |           |        |            |         |            |         |  |  |
| 2H                  | 80%  | 31%       | 20%    | 23%        | 19%     | 21%        | 17%     |  |  |
| 1T                  | 5%   | 50%       | 60%    | 58%        | 62%     | 59%        | 63%     |  |  |
| MoO2                | 7%   | 15%       | 16%    | 16%        | 17%     | 17%        | 18%     |  |  |
| MoO3                | 8%   | 4%        | 4%     | 3%         | 2%      | 3%         | 2%      |  |  |
| 2H/(1T+2H)<br>ratio | 94%  | 38%       | 25%    | 28%        | 25%     | 26%        | 21%     |  |  |
| 1T/(1T+2H)<br>ratio | 6%   | 62%       | 75%    | 72%        | 76%     | 74%        | 79%     |  |  |
|                     | S2p  |           |        |            |         |            |         |  |  |
| 2H                  | 86%  | 28%       | 19%    | 17%        | 17%     | 17%        | 12%     |  |  |
| 1T                  | -    | 39%       | 49%    | 55%        | 53%     | 54%        | 47%     |  |  |
| Edge                | -    | 10%       | 16%    | 10%        | 13%     | 13%        | 18%     |  |  |
| S2-                 | 4%   | 17%       | 16%    | 19%        | 17%     | 16%        | 20%     |  |  |
| SO32-               | 6%   | 4%        | -      | -          | -       | -          | 3%      |  |  |
| SO42-               | 4%   | 2%        | -      | -          | -       | -          | -       |  |  |
| 2H/(1T+2H)<br>ratio | 100% | 42%       | 29%    | 24%        | 24%     | 24%        | 20%     |  |  |
| 1T/(1T+2H)<br>ratio | 0%   | 58%       | 71%    | 76%        | 76%     | 76%        | 80%     |  |  |

Table S 1. The average percentage content of various components on high-resolution Mo3d and S2p XPS spectra of MoS<sub>2</sub>

#### 2.4. UV-Vis Spectrophotometry

Fig. S 5 displays the obtained UV-Vis spectra of dispersion solutions of exfoliated  $MoS_2$  after dialysis. As can be seen in this figure, the spectrum for sample obtained after 1 day of experiment at room temperature differs significantly from other spectra, which are very similar to each other. The UV-Vis spectrophotometry, even visually, additionally confirms that these reaction conditions are insufficient to produce material with suitable content of metallic 1T phase.

In order to further gain information about the content of semiconducting 2H phase and metallic one, the Eqn (S1) were used to calculate the ratio of 2H versus 1T phase. Subsequently, obtained value was subtracted from 100 to determine the 1T vs. 2H ratio, which also support received results (Fig. 1 b). By comparing the results obtained from UV-Vis with XPS a notable discrepancy of 1T% values for the sample prepared in 1 day lasting reaction at RT is observed and it is attributed to the low degree of exfoliation, which hindered the correct quantification of the 1T phase by UV-Vis spectrophotometry.



Fig. S 5 UV-Vis spectra of all  $MoS_2$  dispersions obtained after dialysis

$$\frac{2H}{2H+1T} = 1.29 - (2.562 \cdot 0.287^{x}), x = \frac{I_{410}}{I_{350}}$$
 Eqn (S1)<sup>1</sup>

### 2.5. Raman Spectroscopy

Raman finger-print of ce-MoS<sub>2</sub> changed dramatically upon increasing the laser irradiation power. Even low laser power (*i.e.* 0.33 mW, 10 s) altered the phase composition, making the comparison of the different samples very challenging (Fig. S 6). However, by adjusting the exposure time and intensity power, it is possible to observe differences between samples (Fig. 1 c).



Fig. S 6 Raman spectra at the different laser irradiation power

#### 2.6. Scanning Transmission Electron Microscopy (STEM)



Fig. S 7 STEM images of samples: (a) bulk  $MoS_2$ ; (b) 1 day, RT; (c) 1 day, 55°C; (d) 3 days, RT and (e) 3 days, 55°C.

#### 2.7. Atomic Force Microscopy (AFM)

Based on AFM analysis, thickness and lateral size distribution of exfoliated  $MoS_2$  were determined. Due to the fact that the experimental conditions used to obtain *ce*-MoS<sub>2</sub> (1 day, RT) are insufficient in order to achieve ultrathin flakes, here we focused on samples (1 day, 55°C), (3 days, RT) and (3 days, 55°C). All exfoliated  $MoS_2$  samples have similar thickness in the range of 3-5 nm. However, in the case of samples at elevated temperature, there is also a second maximum at ~8-9 nm, which is notably evident in the sample (3 days, 55°C). It is known that solution-processed nanosheets spontaneously tend to restack, both in colloidal dispersions as well as after deposition on substrates. Here, *ce*-MoS<sub>2</sub> nanosheets are negatively charged,<sup>4</sup> thus, in order to overcome such a Coulombic repulsion, temperature might act as the required driving force to promote the restacking phenomena, corroborating our AFM results.

In the case of lateral size, the range of distribution in all obtained materials is almost the same. However, they slightly vary in the predominant size of flakes. After the reaction conducted for 1 day at 55°C, the average lateral size is around 200 nm, while for sample (3 days, 55°C) the produced exfoliated MoS<sub>2</sub> mostly has 150-250 nm. From all samples, the largest lateral size has the material produced during the reaction for 3 days at RT, where the average lateral size value is around 290 nm.



**Fig. S 8** (a-c) AFM images of samples (1 day, 55°C), (3 days, RT) and (3 days, 55°C), respectively. (d-f) Thickness and (g-i) lateral size distribution of ce-MoS<sub>2</sub> for (1 day, 55°C) as well as (3 days, RT) and (3 days, 55°C), respectively, based on analyzed of 100 flakes in AFM images. The black lines are distribution curves, lognormal type.

#### 2.8. Electrical Characterization

In order to gain information about the conductivity of obtained *ce*-MoS<sub>2</sub>, the sheet resistance of prepared films was measured. Knowing the thickness of produced films, which was measured by the Alpha-Step IQ profilometer, the resistivity ( $\rho$ ) and, subsequently, conductivity ( $\sigma$ ) of *ce*-MoS<sub>2</sub> were calculated using Eqn (S2) and (S3), respectively.

$$o = t \cdot Rs$$
, Eqn (S2)

where  $\rho$  is resistivity, t is thickness and Rs is sheet resistance

$$\sigma = rac{1}{
ho}$$
, Eqn (S3)

where  $\sigma$  is conductivity.



**Fig. S 9** Dependence of conductivity vs. thickness of the film (deposited by dry-transfer method), measured for the sample (2 days,  $55^{\circ}$ C). The conductivity of *ce*-MoS<sub>2</sub> is independent of the thickness. The conductivity of the thinner sample (110 nm) is lower, because the film is not perfectly homogeneous.

#### 2.9. Field-Effect Transistors (FETs)

The electrical characteristics of *ce*-MoS<sub>2</sub> produced during 1 day lasting reaction at 55°C were assessed by measuring the performance of FETs under N<sub>2</sub>-controlled atmosphere. In this regard, different geometries were exploited to achieve a thorough characterization of the main device figures of merit. Fig. S 10 displays the electrical properties of as-prepared *ce*-MoS<sub>2</sub>-based FETs in bottom-contact/bottom-gate configuration. Typical output and transfer curves (Fig. S 10d and 10e, respectively) reveal no dependence on the applied gate voltage (V<sub>g</sub>), confirming the metallic behavior related to the 1T-phase.



**Fig. S 10** Characterization of FET device in bottom-contact/bottom-gate configuration: (a-c) SEM images of IDEs coated by *ce*-MoS<sub>2</sub> deposited *via* drop-casting technique showing the inhomogeneous deposition; (d) Typical output curve for *ce*-MoS<sub>2</sub>, reporting the source-drain current ( $I_{ds}$ ) *vs.* source-drain voltage ( $V_{ds}$ ) at different gate voltage ( $V_{g}$ ) ranging from -30 V to +30

V. The linear trend confirms the ohmic contact between Au IDEs and *ce*-MoS<sub>2</sub>; (e) Typical transfer curve for *ce*-MoS<sub>2</sub>, recorded by measuring the  $I_{ds}$  while sweeping the  $V_g$  from -30 V to +30 V with  $V_{ds}$  = 0.1 V; (f) Optical image of IDEs.



Fig. S 11 XPS spectra of (a) ce-MoS<sub>2</sub> (1 day, 55°C) in FET device as well as after annealing of device at (b) 100°C, (c) 150°C, (d) 200°C, (e) 250°C and (f) 300°C.

As reported in the above-mentioned characterization analysis, the metallic 1T-phase is almost fully converted in the semiconducting 2H-phase upon vacuum thermal annealing at 300°C (Fig. S 11). In order to investigate the electrical performance of 2H-MoS<sub>2</sub>, top-contact/top-gate devices were fabricated as reported in Section 1.3. The overall electrical performance was assessed by means of characteristic device figures of merit, such as field-effect mobility ( $\mu_{FE}$ ) and current ratio ( $I_{ON}/I_{OFF}$ ).  $\mu_{FE}$ is calculated from the transfer curves ( $I_{ds}$  vs.  $V_g$ ) as:<sup>5</sup>

$$\mu_{FE} = \frac{g_m L}{C_v V_{ds} W t}$$
 Eqn (S4)

where  $g_m$  is  $(\partial I_{ds}/\partial V_g)_{MAX}$ , L the channel length (2.5  $\mu$ m), C<sub>v</sub> the volumetric capacitance (1.9 F/cm<sup>3</sup>), V<sub>ds</sub> the source-drain voltage (0.1 V), W the channel width (10<sup>4</sup>  $\mu$ m) and t the film thickness (600 nm).



**Fig. S 12** Typical (a) transfer and (b) leakage curve for *ce*-MoS<sub>2</sub> upon vacuum annealing at 300°C. All the curves are recorded applying  $V_{ds} = 0.1$  V and sweeping  $V_g$  from -2.5 to 2.5 V.

As displayed in Fig. S 12, the ultimate electrical performance of 2H-MoS<sub>2</sub> devices was characterized by an average  $\mu_{FE}$  of  $10^{-2}$  cm<sup>2</sup> V<sup>-1</sup> s<sup>-1</sup> and  $I_{ON}/I_{OFF}$  of  $10^2$ , in perfect agreement with values already reported in literature for solution-processed materials of this kind.<sup>2</sup>

## 3. LITERATURE REVIEW

| Ref                                                                   | Time<br>(h) | T (°C)   | Intercalating<br>agent                  | Comment                                                                             | Application                                                  |
|-----------------------------------------------------------------------|-------------|----------|-----------------------------------------|-------------------------------------------------------------------------------------|--------------------------------------------------------------|
| Nano Lett. <b>2015</b> , <i>15</i> , 5956-<br>5960 <sup>6</sup>       | 1.5         | RT       | <i>n</i> -BuLi (4.0 eq)                 | The intercalation was done under ultrasonication                                    |                                                              |
| <i>RSC Adv.</i> <b>2014</b> , <i>4</i> , 32570-<br>35578 <sup>7</sup> | 1-2         | 100      | <i>n</i> -BuLi (3.2 to<br>4.5 eq)       | The intercalation was done<br>in an autoclave                                       | Surface functionalization                                    |
| Nano Lett. <b>2015</b> , 15,<br>2194–2202 <sup>4</sup>                | 8           | RT       | <i>n</i> -BuLi (excess)                 | Detailed information about<br>chemical exfoliation of<br>MoS <sub>2</sub> - unknown | MoS <sub>2</sub> -PEO for<br>electrochemical Mg<br>storage   |
| ACS Nano <b>2015</b> , 9, 6018-<br>6030 <sup>1</sup>                  | 16          | 65       | <i>n</i> -BuLi (0.3 or<br>3.0 eq)       |                                                                                     | Functionalization with<br>electrophiles                      |
| Chem. Mater <b>. 2019</b> , 15,<br>5725-5734 <sup>8</sup>             | 24          | RT       | <i>n</i> -BuLi (6.0 eq)<br>NaK (1.5 eq) |                                                                                     | SERS                                                         |
| Chem. Commun. <b>2020</b> , 56, 6878-6881 <sup>3</sup>                | 24          | 55       | <i>n</i> -BuLi (2.5 eq)                 |                                                                                     | Supramolecular functionalization of MoS <sub>2</sub>         |
| Nat. Chem. <b>2015</b> , 7, 45-59 <sup>9</sup>                        | 24          | Reflux   | <i>n</i> -BuLi (2.5 eq)                 |                                                                                     | Functionalization with<br>electrophiles                      |
| Nano Energy <b>2016</b> , 26,<br>172-179 <sup>10</sup>                | 48          | 80       | <i>n</i> -BuLi (2.5 eq)                 |                                                                                     | Thermoelectric energy<br>harvesting                          |
| ACS Catal. <b>2019</b> , 9, 7527-<br>7534 <sup>11</sup>               | 48          | RT       | <i>n</i> -BuLi (2.0 eq)                 |                                                                                     | Catalysis                                                    |
| Chem. Eur. J. <b>2020</b> , 26,<br>6535-6544 <sup>12</sup>            | 48          | RT       | <i>n</i> -BuLi (3.2 eq)                 |                                                                                     | Fundamental study for the defect engineering                 |
| Chem. Mater. <b>2020</b> , 32,<br>148-156 <sup>13</sup>               | 67          | RT       | <i>n</i> -BuLi (2.5 eq)                 |                                                                                     | Study of the stability of<br><i>ce</i> -MoS <sub>2</sub>     |
| Chem. Mater. <b>2017</b> , 29,<br>2066-2073 <sup>14</sup>             | 72          | RT       | <i>n</i> -BuLi (1.8 eq)                 |                                                                                     | Covalent modification of MoS <sub>2</sub>                    |
| Chem. Eng. J. <b>2017</b> , 330,<br>462-469 <sup>15</sup>             | 72          | RT       | <i>n</i> -BuLi (1.3 eq)                 |                                                                                     |                                                              |
| J. Phys. Chem. C <b>2019</b> , 123,<br>12126-12134 <sup>16</sup>      | 72          | RT       | <i>n</i> -BuLi (2.5 eq)                 |                                                                                     | Solid-state lithium-ion<br>batteries                         |
| Angew. Chem. Int. Ed.<br><b>2010</b> , 49, 4059-4062 <sup>17</sup>    | 72          | 100      | <i>n</i> -BuLi (25.0<br>eq)             |                                                                                     | Fundamental study of<br>MoS <sub>2</sub> and WS <sub>2</sub> |
| Adv. Funct. Mater. <b>2017</b> ,<br>27, 1604923 <sup>18</sup>         | 72          | RT       | <i>n</i> -BuLi (1.8 eq)                 |                                                                                     | Biosensing                                                   |
|                                                                       | 24          | RT<br>55 |                                         |                                                                                     |                                                              |
| This work                                                             | 48          | RT<br>55 | <i>n</i> -BuLi (2.5 eq)                 |                                                                                     | Fundamental study                                            |
|                                                                       | 72          | RT       | •                                       |                                                                                     |                                                              |

**Table S 2** Compilation of reaction conditions for the preparation of *ce*-MoS<sub>2</sub>. The aim of the table is to describe the inhomogeneity of the experimental conditions reported in the literature and not to give a detailed review of all reported conditions.

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