

ATS-JF060 Ir-ATO

Date: 2025-05-04

Tags: Ir ATO

Category: Synt Autoclave

Status: Success

Created by: Jonas Forner

Goal: Synthesis and analysis of Ir/ATO (50 wt%) (ATS-JF060)

Procedure:

Sample: ATS-JF060

1. The precursor solutions were prepared in the chosen solvent (H₂O) with the following masses:
 - 46.73 mg (0.157 mmol) of Ir(Cl) were dissolved in 5.00 mL of H₂O. This corresponds to a metal concentration of 5.0 mg Ir / ml (31.30 mM).

The solutions were sonicated until the precursors were fully dissolved.

2. Calculated amounts of each stock solution were added to the reaction vessel:

The target composition was Ir_{1.0}.

- 1.000 mL of Ir(Cl) solution were added to the vial. This corresponds to 0.0260 mmol or 4.998 mg of Ir.

3. The 2.0 M reducing agent solution was prepared by dissolving 3999.70 mg of NaOH in 50.00 mL of H₂O.
 - 0.104 mL of the reducing agent solution were added to the vial, corresponding to 0.208 mmol (final concentration: 41.60 mM).

4. No capping agent was added to the reaction.

5. ATO was selected as support with a catalyst loading of 50 wt%. The overall theoretical weight of the nanoparticles

(assuming full reduction) was 5.0 mg. The amount of ATO that had to be added to reach the target loading was 5.00 mg.

6. The vial was filled to the final volume of 5.00 mL with 3.90 mL of H₂O.

7. A stirring bar was added to the PTFE reaction vessel, which was then transferred into an autoclave reactor.

The reactor was filled around the vial with 15 ml of H₂O and afterwards closed carefully.

8. The synthesis process was started. The reactor was heated to a final temperature of 175.0°C with a heat ramp rate of 2.0°C/min,
while stirring at 500.0 rpm. The temperature was held for 240.0 minutes. The reactor was then left to cool down to a reasonable
temperature before opening it. Any remaining pressure was released via the 3-way valve on the panel.
The reaction vessel was carefully retrieved.
9. The solution from the reaction vessel was transferred to a falcon tube. The nanoparticles were washed 2.0 times with 15.0 ml
of Milli-Q water and 2.0 times with 15.0 ml of ethanol. The solution was centrifuged every time at 6000.0 rpm
for 15.0 minutes. The initial supernatant was stored for ICP-MS analysis.
10. The sample was not dried and stored in ethanol until further use.
11. The sample was not heat treated.

Summary Table

| Property | Value | Property | Value | Property | Value |
|---|-------------------|-----------------------------|-----------|-----------------------|-------|
| Author | Jonas Forner | Sample: | ATS-JF060 | | |
| Nanoparticle Mass (mg) | 5.0 | Solution Concentration (mM) | 5.2 | | |
| Molar Weight of Particles (w/o support) (g/mol) | 192.22 | Nanoparticle Moles (mmol) | 0.0260 | | |
| Composition | Ir _{1.0} | Precursor Type | Cl | | |
| Reducing Agent | NaOH | RA Concentration Factor | x 8 | RA Concentration (mM) | 41.6 |
| Capping Agent | None | CA Concentration (wt%) | 0.0 | | |
| Support | ATO | Support Loading (wt%) | 50 | Support Mass (mg) | 5.0 |
| Solvent | H2O | Final Volume (mL) | 5.00 | | |
| Temperature (°C) | 175.0 | Ramp (°C/s) | 2.0 | Synthesis Time (min) | 240.0 |
| Stirring Rate (rpm) | 500.0 | Compound dried? | False | | |
| Heat Treatment | None | HT Temperature (°C) | 0 | HT Duration (h) | 0 |

The catalyst was dispersed in 2 ml of a 2:1 mixture of water:iPrOH, resulting in a metal concentration of 2.5 mg/ml.

extra_fields ⇒

Linked item

Project - [DEMI](#)

Attached files

ATS-JF060_recipe.tdms

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ATS-JF060_recipe.txt

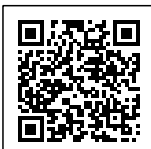
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Link: <https://elabftw.dcbp.unibe.ch/experiments.php?mode=view&id=6>