

General SrO₂ Procedure

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January 11, 2024

1 Introduction

This is the general procedure for producing SrO₂. This procedure was produced by following the 1998 Hummel manual and custom tailoring it to work with our machines and experiences during several pilot batches of SrO₂. There is still room for improvement in procedure, technique, and machinery. Further change in all of these fields should be considered, especially for full scale production. Proper PPE must be worn while handling hazardous materials, specifically the Sr(NO₃)₂, aqua ammonia, and H₂O₂. Refer to the MSDS for information on these materials or to the brief hazard description further in this document.

2 Materials

The materials to be charged to the reactor are as follows:

146 Liters of soft water

68 Kg of Sr(NO₃)₂CAS # 10042-76-9

82 Kg of Aqua AmmoniaCAS # 1336-21-6

68 Kg of H₂O₂CAS # 7722-84-1

The filtered product will be washed outside the reactor with:

20 Liters of 10% H₂O₂

Which should yield around:

30 Kg of SrO₂CAS # 1314-18-7

3 Method

3.1 Stage 1

- Preheat oven to 300°F

- Charge 146 Liters of soft water to the reactor. Refer to mixer-arm to water level chart.
- Set the agitator to low speed (around 10-20 on the VFD)
- Heat the reactor to 60°C.
- Break up the $\text{Sr}(\text{NO}_3)_2$ into chunks no greater than 8" in diameter so as to avoid damage to the thermocouple when charging.
- Add 68 Kg of $\text{Sr}(\text{NO}_3)_2$ (3 50-lb bags) to the reactor.
- Wait until all $\text{Sr}(\text{NO}_3)_2$ has dissolved (around 20-30 minutes) before proceeding to Stage 2.

3.2 Stage 2

- Add 88 Kg of Aqua Ammonia to the reactor.
- Set agitator to high (90 on the VFD)
- Stage the H_2O_2 pumps, ensure enough H_2O_2 is present in the single barrel for the charge. Set the peristaltic pump to 12 LPM (its maximum)
- Clean out the filter, soak the filter membrane with water thoroughly. Ensure the filter has no major vacuum leaks and that the vacuum pump is working as expected. Ensure there are no other active connections to the vacuum line.
- Prepare the H_2O_2 wash liquid by diluting 4 kg of concentrated H_2O_2 with 16 kg of water in a sufficiently large vessel.
- Stage clean oven trays on a pallet nearby the filter and ready to be transported quickly by forklift.

3.3 Stage 3

- Ensure oven has reached 300°F

The following steps should be taken with haste. SrO_2 quality, specifically the free oxygen percentage, diminishes the longer the following steps take until the product is dry:

- Set the chiller temperature to 5°C. (A temperature far below necessary so as to avoid compressor lockout.)
- Catch the reactor on the downslide at 35°C and proceed to the next bullet immediately
- Add 17 Kg of H_2O_2 to the reactor (one-fourth of the total 68 Kg) using the low setting of the drum pump. Be aware you will need to pull the feed end of the pump above the waterline to stop a siphoning effect.

- Observe the peak temperature rise (44-46°C nominal).
- allow the reactor to cool to 44.5°C
- Add another 5.6 Kg to finish off the initial dump using the drum pump (Difference between one-third and one-fourth of total H_2O_2)
- Cool to 44°C.
- Add the remaining 45.3 Kg of H_2O_2 to the reactor using the peristaltic pump at a rate of 12 liters per minute, ensuring the temperature stays below 45°C and above 38°C. If the temperature is falling too rapidly, supplement the peristaltic pump with the drum pump. If the temperature is threatening 45°C, turn off the peristaltic pump and allow cooling to take place.

3.4 Stage 4

The following steps should also be taken with haste:

- Cool to 37°C.
- Discharge the contents from the reactor into the filter.
- Perform vacuum filtration.
- Once all the liquid has been purged close the vacuum filter valve and wash the filter cake with wash solution prepared earlier.
- Perform vacuum filtration again.
- Transfer the rest of the filter cake into the 12 oven trays.

3.5 Stage 5

- Place trays in a preheated oven at 300°F (150°C).
- After kickoff, set temperature to 275°F (135°C).
- After kickoff, stir tray contents with a hoe.
- Repeat stirring every 1.5 hours until dry, no more than 5 hours in oven.
- If the product is still not dry by the end of the five hours, set the temperature to 110°C and leave overnight. The product must not be left at >130°C for extended periods of time as the thermal decomposition of SrO_2 will take place.

3.6 Stage 6

- Remove from oven and break up any SrO_2 clumps.
- Store in bags within Romex containers. Be sure to label the container with the contents and batch number.
- Set aside a sample of around 100 grams in a sample bag for laboratory analysis. Be sure to properly label bag.

4 Discussion

In our experience it appears that the oven / drying process is the weakest link in our production chain. While special accommodations (additional thermal mass, oven influx air preheating, a added exhaust fan and smoke stack) have been made and have a positive effect, the amount of time needed for kick off and drying yield an inferior product and limit not only the number of batches per day but also the size of each batch. The wetlab oven sets the standard for what is possible with adequate drying. Samples placed in the wetlab oven consistently yield free oxygen percentages of 12.8% - 12.9% while samples dried by the production yield around 12.4% - 12.6% which is a critical problem as it borders on the edge of the required specification.

The theoretical maximum for free oxygen in SrO_2 is 13.4%. Taking the wetlab oven samples to be indicative of the quality of the SrO_2 immediately out of the reactor, there is still work to be done in the reactor process. Three avenues of possible improvement include:

- Chilling of H_2O_2 prior to charging
- Precise flow rate control so as to optimize a function of time and reaction temperature
- Additional reactor chilling capability either in the form of a higher power chiller or larger coolant reservoir.

While the drying capability is the more obvious low hanging fruit for improvements to the process compared to the reactor process portion. A combination of the two is bound to yield better and more consistent results. While difficult to estimate, I suspect a consistent free oxygen percentage of 12.8 - 13.2% and 2 batches per day is possible with further investment, both in machinery and process procedure.

5 Chemical Safety Information

5.1 Aqua Ammonia (26% Solution)

Aqua Ammonia, particularly a 26% solution, is a clear, colorless liquid. It emits a strong, pungent odor characteristic of ammonia. Be mindful not to accidentally mix aqua ammonia and hydrogen peroxide outside of the reactor. Be mindful that transferring of filter contents can take place at the vacuum line junction, if both valves are open, causing this reaction to take place.

- **Composition:** An aqueous solution of ammonia, typically containing 26% ammonia by weight.
- **Corrosive Nature:** Can cause burns to skin and eyes. Requires the use of personal protective equipment, including gloves and eye protection.
- **Vapors:** Releases ammonia vapors, which can irritate the respiratory system and eyes. Ensure good ventilation in handling and storage areas.
- **Toxicity:** High levels of exposure can lead to serious respiratory distress and other health issues.
- **Storage:** Store in a cool, well-ventilated area. Keep container tightly closed when not in use.
- **Environmental Impact:** Can be harmful to aquatic life. Measures should be taken to prevent environmental contamination.

5.2 Hydrogen Peroxide (H₂O₂)

Hydrogen peroxide is a clear, colorless liquid used in various applications due to its oxidizing properties. In industrial settings, it is used as a bleaching agent and for pollution control. It is also used in low concentrations in medical applications for disinfection. Hydrogen peroxide is a strong oxidizer and reacts violently with organic materials. Be mindful not to mix aqua ammonia and hydrogen peroxide outside of the reactor. Be mindful that transferring of filter contents can take place at the vacuum line junction, causing this reaction to take place.

- **Corrosive Nature:** Concentrated H₂O₂ is highly corrosive, causing severe skin burns and eye damage. Dilute solutions can irritate skin and eyes.
- **Oxidizing Agent:** Strong oxidizing properties. Can cause fire and explosion hazards, especially with organic substances or metals.
- **Decomposition Risk:** Prone to rapid decomposition, especially when in contact with catalysts or contaminants. Releases oxygen, increasing fire or explosion risk in confined spaces.

- **Respiratory Hazard:** Inhalation of vapors can cause respiratory irritation or pulmonary edema.
- **Storage and Handling:** Store in a cool, dry, well-ventilated area, away from sunlight and incompatible materials. Avoid contamination and rapid decomposition.

5.3 Strontium Nitrate ($\text{Sr}(\text{NO}_3)_2$)

Strontium Nitrate is typically found as a white, odorless, crystalline powder. It may also appear in granular form.

- **Oxidizer:** Acts as an oxidizing agent. Keep away from flammable and combustible materials to prevent combustion.
- **Health Risks:** Can cause eye, skin, and respiratory irritation. Long-term exposure poses more serious health risks, particularly to the lungs.
- **Environmental Concerns:** Harmful to aquatic life. Avoid environmental contamination.
- **Reactivity:** Store properly to avoid contact with acids, reducing agents, or incompatible materials to prevent hazardous reactions.

5.4 Strontium Peroxide (SrO_2)

Strontium Peroxide is usually a white or pale yellow powder. It is odorless and has an oxidizing nature.

- **Uses:** Commonly used in pyrotechnics for producing a bright red flame, in the production of other strontium compounds, and in oxygen generation systems. It is notable for its ability to release oxygen upon decomposition.
- **Oxidizing Agent:** Potent oxidizer, careful handling required to avoid hazardous reactions with reducing agents.
- **Storage:** Store in a cool, dry place, away from incompatible materials.
- **Exposure Risks:** Can cause irritation to the skin, eyes, and respiratory tract. Appropriate protective measures should be taken during handling.