

Standard Operating Procedure

Settlement Class: Pyrophorics

Gallium alkyls

This SOP is not complete until it has been signed and dated by the PI and relevant lab personnel.

Print a copy and insert into your
Laboratory Safety Manual and Chemical Hygiene Plan.
Refer to instructions for assistance.

Department:	Chemistry & Biochemistry - Chemical Engineering
Date SOP was written:	December 14, 2012
Date SOP was approved by PI/lab supervisor:	January 18, 2013
Principal Investigator:	Prof. Susannah Scott
Internal Lab Safety Coordinator/Lab Manager:	Stephanie Goubert-Renaudin
Lab Phone:	805-893-8941
Office Phone:	805-893-7403
Emergency Contact:	EH&S 24 hour line: 805-893-3194 (Name and Phone Number)
Location(s) covered by this SOP:	ESB 3324 and 3328 (Building/Room Number)

Type of SOP: ☐ Process ☐ Hazardous Chemical ☒ Hazardous Class

Purpose

Gallium alkyls are spontaneously flammable in air and react violently with water, they are also corrosive. They can cause severe burns to skin, eyes, respiratory tract, and gastrointestinal tract. Gallium alkyls are often used as co-catalysts in the polymerization of olefins and dienes. They are effective alkylating agents and are important in pharmaceuticals and chemical synthesis.

Examples of Gallium alkyls

TrimethylGallium

Physical & Chemical Properties/Definition of Chemical Group

CAS#: Various

Class: **Highly flammable, pyrophoric, corrosive**

Molecular Formula: Various

Form (physical state): Liquid

Color: Colorless

Boiling point: Various

Potential Hazards/Toxicity

Gallium alkyls are spontaneously flammable in air and react violently with water. They can cause severe skin burns and irreversible eye damage. They may be harmful if inhaled, ingested, or absorbed through the skin. They may be extremely destructive to the tissues of the mucous membranes and upper respiratory tract. Ingestion can cause damage to the mouth, throat, and esophagus.

Personal Protective Equipment (PPE)

Respirator Protection

Use a full-face respirator with multi-purpose combination (US) respirator cartridges.

Respirators should be used only under any of the following circumstances:

- As a last line of defense (i.e., after engineering and administrative controls have been exhausted).
- When Permissible Exposure Limit (PEL) has exceeded or when there is a possibility that PEL will be exceeded.
- Regulations require the use of a respirator.
- An employer requires the use of a respirator.
- There is potential for harmful exposure due to an atmospheric contaminant (in the absence of PEL)
- As PPE in the event of a chemical spill clean-up process

Lab personnel intending to use/wear a respirator mask must be trained and fit-tested by EH&S. This is a regulatory requirement.

Hand Protection

Handle with gloves. Nitrile gloves are recommended.

NOTE: Consult with your preferred glove manufacturer to ensure that the gloves you plan on using are compatible with gallium alkyls.

Refer to glove selection chart from the links below:

http://www.ansellpro.com/download/Ansell_8thEditionChemicalResistanceGuide.pdf

OR

<http://www.allsafetyproducts.biz/page/74172>

OR

<http://www.showabestglove.com/site/default.aspx>

OR

<http://www.mapaglove.com/>

Eye Protection

ANSI approved, tight-fitting safety glasses/goggles.

Skin and Body Protection

Flame resistant lab coat preferably made of antistatic material, long pants, and closed-toe shoes.

Hygiene Measures

Avoid contact with skin, eyes and clothing. Wash hands before breaks and immediately after handling the product.

Engineering Controls

Pyrophoric chemicals should be used in a glove box or in a closed system in a certified chemical fume hood.

First Aid Procedures

If inhaled

Move person into fresh air. If not breathing, give artificial respiration. Consult a physician.

In case of skin contact

Take off contaminated clothing and shoes immediately. Wash skin with soap and plenty of water. Consult a physician.

In case of eye contact

Flush eyes with plenty of water for at least 15 minutes lifting lower and upper eyelids and removing contact lenses. Consult a physician.

If swallowed

Never give anything by mouth to an unconscious person. Rinse mouth with water. Consult a physician.

Special Handling and Storage Requirements

Precautions for safe handling: Avoid inhalation and ingestion. Avoid contact with skin, eyes, and clothing. Provide adequate exhaust ventilation. Use explosion-proof equipment. Keep away from sources of ignition - No smoking. Prevent build-up of electrostatic charges.

Conditions for safe storage: Keep container tightly sealed in a dry and well-ventilated place. Containers must be kept upright to prevent leakage. Never allow contact with water. Store away from oxidizing agents and air. Handle and store under inert gas.

Spill and Accident Procedure

Fire-fighting & Extinguishing media

DO NOT use water

Prior to use, review the Safety Data Sheet for the proper fire extinguisher to use with the given material. Acceptable fire extinguishing media include Metal X, soda ash (lime) or *dry* sand to respond to small fires, and an ABC dry powder extinguisher for large fires. A small beaker of Metal X/LithX, dry sand or soda ash (lime) in the work area is useful to extinguish any small fire that occurs at the syringe tip and to receive any last drops of reagent from the syringe. In general, an ABC dry powder extinguisher will put out the fire, but the pyrophoric reagent may reignite.

Chemical Spill Dial 9-911 and EH&S (805-893-3194)

Spill – Assess the extent of danger. Help contaminated or injured persons. Evacuate the spill area. Avoid breathing vapors. If possible, confine the spill to a small area using a spill kit or absorbent material. Keep others from entering contaminated area (e.g., use caution tape, barriers, etc.).

Small (<1 L) – If you have training, you may assist in the clean-up effort. Use appropriate personal protective equipment and clean-up material for chemical spilled. Double bag spill waste in clear plastic bags, label and take to the next chemical waste pick-up.

Large (>1 L) – Dial 9-911 from campus phones (and 805-893-3446 from a cell phone) and EH&S (893-3194) for assistance.

Chemical Spill on Body or Clothes – Remove clothing and rinse body thoroughly in emergency shower for at least 15 minutes. Seek medical attention. *Notify supervisor and EH&S immediately.*

Chemical Splash Into Eyes – Immediately rinse eyeball and inner surface of eyelid with water from the emergency eyewash station for 15 minutes by forcibly holding the eye open. Seek medical attention. *Notify supervisor and EH&S immediately.*

Medical Emergency Dial 9-911

Life Threatening Emergency, After Hours, Weekends and Holidays – Dial 9-911 (or 805-893-3446 from a cell phone) or go to the Emergency Room of Goleta Valley Cottage Hospital at 351 South Patterson Avenue, Goleta (Phone number: 805-967-3411) *Note: All Serious injuries must be reported to EH&S within 8 hours.*

Non-Life Threatening Emergency – Go to the Student Health Building, Building 588 (phone number: 893-5361, hours: M, T, R, F 8am-4.30pm, W 9am - 4.30pm, R 5pm to 7pm by appointment). After hours go to the Emergency Room of Goleta Valley Cottage Hospital at 351 South Patterson Avenue, Goleta (Phone number: 805-967-3411) *Note: All serious injuries must be reported to EH&S within 8 hours.*

Needle stick/puncture exposure (as applicable to chemical handling procedure) – Wash the affected area with antiseptic soap and warm water for 15 minutes. For mucous membrane exposure, flush the affected area for 15 minutes using an eyewash station. Page the needle stick nurse \ and then enter your extension. After hours go to the nearest emergency room: the Emergency Room of Goleta Valley Cottage Hospital at 351 South Patterson Avenue, Goleta (Phone number: 805-967-3411). *Note: All needle stick/puncture exposures must be reported to EH&S within 8 hours.*

Decontamination/Waste Disposal Procedure

Do not flush with water. Cover with dry sand or other non-combustible material. Call EH&S for assistance. Dispose as hazardous waste after quenching following the protocol/procedure and guidelines below.

General hazardous waste disposal guidelines:

Label Waste

- Affix an on-line hazardous waste tag on all waste containers as soon as the first drop of waste is added to the container

Store Waste

- Store hazardous waste in closed containers, in secondary containment and in a designated location
- Waste must be under the control of the person generating & disposing of it

Dispose of Waste

- Dispose of regularly generated chemical waste within 90 days
- Call EH&S for questions
- Empty Containers
 - Dispose as hazardous waste if it once held extremely hazardous waste (irrespective of the container size)
 - Consult waste pick-up schedule
 - Prepare for transport to pick-up location
 - Check on-line waste tag
 - Write date of pick-up on the waste tag
 - Use secondary containment

Safety Data Sheet (SDS) Location

MSDS can be accessed online: <http://ehs.ucsb.edu/units/labsfty/labrsc/chemistry/lchemmsdsacc.htm>

Protocol/Procedure

**Please see attached SOP “Procedures for Safe Use of Pyrophoric Liquids”
(on line link: http://web.chem.ucsb.edu/~moretto/SOP_Liquid_Reagents.pdf)**

Working with gallium alkyl compounds is not allowed when alone in the Lab. Prior to starting the reaction, locate the extinguisher, eyewash and safety shower.

In our lab, gallium alkyl compounds are mainly used for activation of Ziegler-Natta catalysts for olefin polymerization.

Gallium alkyl compounds are volatile liquids stored in stainless steel containers with a ball valve, inside the N₂-filled double glovebox, to be protected from moisture at all times. Liquid gallium alkyl compounds can leak through the plastic cap of glass containers such as vials, and therefore should NOT be stored in such kind of containers for prolonged period of time. Since gallium alkyl compounds burn spontaneously in air at room temperature, they have to be handled under inert atmosphere, in the glove box, on a vacuum line or in a ventilated fume hood with proper air-free techniques for inert atmosphere. Neat gallium alkyl liquids MUST NOT be brought outside of the glovebox in an open container. Only very small quantities (less than 1 mL) should be brought outside the glovebox, and then they should be handled in a Schlenk flask equipped with Teflon stopcocks to prevent leaks.

Before dispensing any of the compound, determine the exact amount needed and try to minimize unused material. To dispense the liquid from the container, remove the cap from the stainless steel container, then turn the handle to open the valve (parallel to the opening), strictly under an inert atmosphere. Use a pipette or syringe to transfer the liquid to the reaction or a Schlenk flask, which is then capped and cannot be opened in the open-bench: it has to be kept under inert atmosphere. Close the valve (perpendicular to the opening) and place the cap (finger-tight) on the container immediately after use.

Any apparatus used to transfer the compound, including pipette, syringe and needle should be flushed 3 times using non-polar hydrocarbon solvent after use, in order to remove any residual gallium alkyl trapped.

Quenching in the glovebox:

Unreacted gallium alkyl compounds must be properly quenched and disposed upon completion of the experiment. To quench gallium alkyls, the liquid should first be diluted with a copious amount of hydrocarbon solvents (x 200 by volume) such as pentane or hexane. Then, alcohol (isopropanol or butanol) (x 50 by volume) can be added dropwise to the solution inside the glovebox.

Quenching out of the glovebox:

Since gallium alkyl compounds are volatile, they can be pumped and trapped into the cold-trap in vacuum manifolds. Therefore, quenching of gallium alkyl compounds outside the glovebox may be needed and should be performed with great care. Gallium alkyl compounds can ignite spontaneously upon exposure to air, a fire retardant safety goggles and appropriate gloves have to worn at all times. Disposable gloves have to be changed as soon as contaminated. A dewar with liquid nitrogen should also be prepared to stop the quenching process, in case any unexpected amount of heat is evolved. To quench gallium alkyl compounds condensed in the manifold, the cold-trap should be removed at liquid N₂-temperature. The content should then be immediately diluted with 200 mL of hydrocarbon solvents (hexane or pentane). The solution is then quenched by slowly adding alcohol to the mixture, which should always be kept cold until quenching finishes.

When the solution is not reactive anymore, it can be disposed in the appropriate organic waste container, kept closed at all times.

NOTE: Any deviation from this SOP requires approval from PI.

Documentation of Training (signature of all users is required)

- Prior to conducting any work with Gallium alkyls, designated personnel, approved users listed below, must provide training to his/her laboratory personnel specific to the hazards involved in working with this substance, work area decontamination, and emergency procedures.
- The Principal Investigator must provide his/her laboratory personnel with a copy of this SOP and a copy of the SDS provided by the manufacturer.
- The Principal Investigator must ensure that his/her laboratory personnel have attended appropriate laboratory safety training or refresher training as required by EH&S.

I have read and understand the content of this SOP:

Name	Signature	Trainer	Date
Prof. Susannah Scott			
Stephanie Goubert-Renaudin			
Gary Kwanyu Ng			
Alessandro Gallo			

Anthony Crisci			
Haibo Yu			
Taeho Hwang			
Bethany Wigington			
Daniel Coller			
Zachary Jones			
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Jason Fendi			

