

Code of Practice G: Disposal of Lithium Aluminium Hydride and Grignard Reagents

1. Recommended procedure for the disposal of LiAlH_4

(a) Powder (1 g quantity) or ether solution (10 ml quantity)

The disposal must be carried out in a fume hood, free of clutter, flammable materials, and solvents. To a flask, equipped with a stirrer bar, a condenser (connected to a nitrogen inlet and a bubbler *via* a T-piece), an addition funnel, a Quick-fit thermometer, and containing a dry (supplied through the departmental SPS) low reactivity, high boiling solvent such as toluene (20 ml) or heptane (20 ml), slowly and cautiously add LiAlH_4 (1 g); see Note 1. Cool the flask containing the suspension in an ice bath. Maintaining the system under a nitrogen atmosphere, slowly and carefully add dry (dried over molecular sieves) ethyl acetate (10 ml) through the addition funnel while stirring the suspension. Continuously monitor the temperature *via* the internal thermometer to ensure that the ester is not being added too fast. Leave the mixture stirring for 1 hr and then slowly add isopropyl alcohol (10 ml) *via* the addition funnel and leave stirring for a further 1 hr. Slowly add methanol (10 ml) *via* the addition funnel and leave stirring for a further 1 hr. Slowly add water (5 ml) *via* the addition funnel and leave stirring for a further 1 hr. Add the resulting mixture from the flask slowly and carefully to ice-cold water (20 ml) while stirring. In order to neutralise the mixture add dilute HCl (check pH). Separate the organic layer and dispose in waste bottle category "C" and pour the aqueous layer down a fumehood sink.

Note 1: Lithium aluminium hydride (LiAlH_4) reacts violently with water and has a significant heat of solvation. Therefore, do not add solvent to dry LiAlH_4 . Instead, slowly add LiAlH_4 to anhydrous solvent in a nitrogen-purged flask as above; the initial small amount of LiAlH_4 will react with residual trace moisture.

Note 2: For the disposal of quantities more than 1 g, the above method is not appropriate and must not be used. A separate protocol must be written, approved, and implemented.

2. Recommended procedure for the disposal of Grignard reagents

(a) Solution (10 ml quantity)

The disposal must be carried out in a fume hood, free of clutter, flammable materials, and solvents. To a flask, equipped with a stirrer bar, a condenser (connected to a nitrogen inlet and a bubbler *via* a T-piece), an addition funnel, and containing a dry (supplied through the departmental SPS) low reactivity, high boiling solvent such as toluene (20 ml) or heptane (20 ml), slowly and cautiously add the solution containing the Grignard reagent (10 ml). Cool the flask in an ice bath. Maintaining a nitrogen atmosphere, slowly and carefully add isopropanol (20 ml) through the addition funnel while stirring the flask's contents. Leave the mixture stirring for 1 hr. Slowly add methanol (10 ml) *via* the addition funnel and leave stirring for a further 1 hr. Slowly add water (5 ml) *via* the addition

funnel and leave stirring for a further 1 hr. Add the mixture slowly and carefully to ice/water with stirring. In order to neutralise the mixture add dilute HCl (check pH). Separate the organic layer and dispose in waste bottle category "C" and pour the aqueous layer down the sink.

(b) **Solid (1 g, in the bottle)**

The disposal must be carried out in a fume hood, free of clutter, flammable materials, and solvents. Extremely cautiously add a dry (supplied through the departmental SPS) low reactivity, high boiling solvent such as toluene (20 ml) or heptane (20 ml) to the bottle containing solid Grignard reagent (1 g) to make a slurry solution. To a flask, equipped with a stirrer bar, a condenser (connected to a nitrogen inlet and a bubbler *via* a T-piece), an addition funnel, and containing a dry (supplied through departmental SPS) low reactivity, high boiling solvent such as toluene (20 ml) or heptane (20 ml), slowly and cautiously add the slurry solution containing the Grignard reagent. Cool the flask in an ice bath. Maintaining a nitrogen atmosphere, slowly and carefully add isopropanol (20 ml) through the addition funnel while stirring the flask's contents. Leave the mixture stirring for 1 hr. Slowly add methanol (10 ml) *via* the addition funnel and leave stirring for further 1 hr. Slowly add water (5 ml) *via* the addition funnel and leave stirring for a further 1 hr. Add the mixture slowly and carefully to ice/water with stirring. In order to neutralise the mixture add dilute HCl (check pH). Separate the organic layer and dispose in waste bottle category "C" and pour the aqueous layer down the sink.

Notes:

The disposal procedure for Grignard reagents can be used for the disposal of organolithium reagents (10 ml quantity).

For the disposal of quantities of either Grignard reagents or organolithiums of more than 10 ml, the above method is not appropriate and must not be used. A separate protocol and risk assessment must be written, approved, and implemented.

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