Organic Syntheses

A Publication
of Reliable Methods
for the Preparation
of Organic Compounds

Working with Hazardous Chemicals

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These paragraphs were added in September 2014. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.

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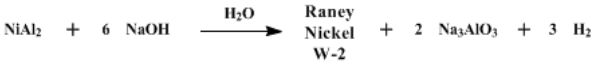
CATALYST, RANEY NICKEL, W-2



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1. Procedure

A solution of 380 g. of sodium hydroxide in 1.5 l. of distilled water, contained in a 4-l. beaker (Note 1) equipped with an efficient stirrer (Note 2), is cooled in an ice bath to 10°, and 300 g. of nickel-aluminum alloy (Note 3) is added to the solution in small portions, with stirring, at such a rate that the temperature does not rise above 25° (Note 4), the beaker being allowed to remain in the ice bath. When all the alloy has been added (about 2 hours is required) the stirrer is stopped, the beaker is removed from the ice bath, and the contents are allowed to come to room temperature. After the evolution of hydrogen becomes slow, the reaction mixture is allowed to stand on a steam bath until the evolution of hydrogen again becomes slow (about 8–12 hours). The heating should not be too rapid at the beginning or the solution may foam over. During this time the volume of the solution is maintained constant by adding distilled water if necessary. After heating, the nickel is allowed to settle and most of the liquid is decanted. Distilled water is then added to bring the solution to the original volume; the nickel is suspended by stirring, again allowed to settle, and the solution is decanted. The nickel is then transferred to a 2-l. beaker (Note 5) with the aid of distilled water, and the water is again decanted. A solution of 50 g. of sodium hydroxide in 500 ml. of distilled water is added; the catalyst is suspended and allowed to settle; and the alkali is decanted. The nickel is washed by suspension in distilled water and decantation until the washings are neutral to litmus and then ten times more to remove the alkali completely (twenty to forty washings are required) (Note 6). The washing process is repeated three times with 200 ml. of 95% ethanol and three times with absolute ethanol; the catalyst is then stored under absolute ethanol in bottles which are completely filled with absolute ethanol and tightly closed (Note 7). The product is highly pyrophoric and must be kept under a liquid at all times. The Raney nickel contained in the suspension weighs about 150 g. (Note 8).

To prepare the catalyst under methylcyclohexane (Note 9), the catalyst, which has been prepared as above and washed free of alkali with water, but to which no ethanol has been added, is covered with 1 l. of methylcyclohexane which is distilled from an oil bath until all the water has been codistilled with the hydrocarbon, more of the methylcyclohexane being added from time to time so that the nickel always remains covered. When the catalyst is free from water it becomes freely suspended in the liquid.

To prepare nickel under dioxane, dioxane (Note 10) is used in place of the methylcyclohexane above and the distillation is continued until the temperature of the vapor reaches 101°. (*Caution. Do not use nickel in dioxane above 210°; the dioxane may react almost explosively with hydrogen and Raney nickel above this temperature.*)

2. Notes

1. A Pyrex battery jar of about 10-l. capacity is also suitable and is sufficiently large for the preparation of a batch of catalyst of two to three times the size given here.
2. The stirrer should be provided with a motor which will not ignite the hydrogen. Either an induction motor or an air stirrer may be used. The stirrer blades may be made of glass, Monel, or stainless steel.
3. The alloy used is "Raney Nickel Aluminum Catalyst Powder" from the Gilman Paint and Varnish Company, Chattanooga, Tenn. It contains about 50% nickel.
4. The thermometer should not be left in the alkali or the bulb may be eaten away. The catalyst is inactivated by mercury. If the mixture foams badly at this point, 2 ml. of *n*-octyl alcohol may be added to prevent excessive foaming; however, this is not usually necessary.
5. A stoppered graduate of 2-l. capacity is somewhat more convenient than a 2-l. beaker.
6. The number of washings required may be materially reduced by allowing time for diffusion of base from the surface of the catalyst into the surrounding wash water. To this end the catalyst is stirred well with 1.5 l. of water for each washing. Diffusion is allowed to proceed for 3–10 minutes, and the mixture is then stirred again and the wash water decanted as soon as the catalyst settles to the bottom. By this method twenty washings should be sufficient to remove all traces of the alkali adsorbed on the catalyst surface.
7. The quantity of catalyst prepared should not be larger than necessary for a 6 months' supply as the catalyst may deteriorate on standing.
8. It is more convenient to measure the catalyst than to weigh it. Raney nickel in alcohol contains about 0.6 g. of the catalyst per milliliter of the settled material. The half and quarter teaspoons used in kitchens are convenient for measuring the catalyst. A level teaspoonful is about 3 g. of nickel.
9. It is necessary that the catalyst be freely suspended in the reaction mixture during a reduction. Therefore, the liquid under which the nickel is placed must be soluble in the reduction mixture at all times, i.e., in both the reactants and products.
10. The dioxane used should be dry, halogen-free, and distilled from sodium.

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3. Discussion

These are summarized in the preceding procedure (p. 176). The previously published procedure [*Org. Syntheses*, **21**, 15 (1941)] which is included here results in the catalyst now known as W-2.

This preparation is referenced from:

- Org. Syn. Coll. Vol. 3, 63
- Org. Syn. Coll. Vol. 3, 148
- Org. Syn. Coll. Vol. 3, 278
- Org. Syn. Coll. Vol. 3, 328
- Org. Syn. Coll. Vol. 3, 358
- Org. Syn. Coll. Vol. 3, 717
- Org. Syn. Coll. Vol. 3, 720
- Org. Syn. Coll. Vol. 3, 794
- Org. Syn. Coll. Vol. 3, 827
- Org. Syn. Coll. Vol. 4, 283
- Org. Syn. Coll. Vol. 4, 298
- Org. Syn. Coll. Vol. 4, 603
- Org. Syn. Coll. Vol. 4, 660
- Org. Syn. Coll. Vol. 5, 743
- Org. Syn. Coll. Vol. 6, 468
- Org. Syn. Coll. Vol. 6, 581
- Org. Syn. Coll. Vol. 6, 601

References and Notes

Appendix
Chemical Abstracts Nomenclature (Collective Index Number);
(Registry Number)

nickel-aluminum alloy

Raney Nickel Aluminum Catalyst Powder

ethanol (64-17-5)

hydrogen (1333-74-0)

sodium hydroxide (1310-73-2)

mercury (7439-97-6)

nickel,
Raney nickel (7440-02-0)

sodium (13966-32-0)

methylcyclohexane (108-87-2)

dioxane (5703-46-8)

n-octyl alcohol (111-87-5)