



A Publication
of Reliable Methods
for the Preparation
of Organic Compounds

Working with Hazardous Chemicals

The procedures in *Organic Syntheses* are intended for use only by persons with proper training in experimental organic chemistry. All hazardous materials should be handled using the standard procedures for work with chemicals described in references such as "Prudent Practices in the Laboratory" (The National Academies Press, Washington, D.C., 2011; the full text can be accessed free of charge at http://www.nap.edu/catalog.php?record_id=12654). All chemical waste should be disposed of in accordance with local regulations. For general guidelines for the management of chemical waste, see Chapter 8 of Prudent Practices.

In some articles in *Organic Syntheses*, chemical-specific hazards are highlighted in red "Caution Notes" within a procedure. It is important to recognize that the absence of a caution note does not imply that no significant hazards are associated with the chemicals involved in that procedure. Prior to performing a reaction, a thorough risk assessment should be carried out that includes a review of the potential hazards associated with each chemical and experimental operation on the scale that is planned for the procedure. Guidelines for carrying out a risk assessment and for analyzing the hazards associated with chemicals can be found in Chapter 4 of Prudent Practices.

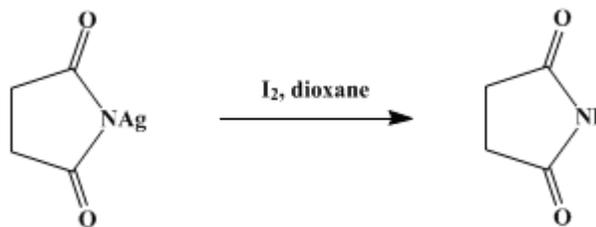
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These paragraphs were added in September 2014. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.

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N-IODOSUCCINIMIDE

[Succinimide, N-iodo-]



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Checked by B. C. McKusick and T. J. Kealy.

1. Procedure

Twenty grams (0.079 mole) of iodine and 90 ml. of dried dioxane (Note 1) are placed in a wide-mouthed, screw-cap, brown bottle of 150–200 ml. capacity. Most of the iodine dissolves. Eighteen grams (0.087 mole) of thoroughly dried N-silver succinimide (Note 2) is added, and the bottle is shaken vigorously for several minutes. The mixture is occasionally shaken in the course of an hour and then is warmed in a water bath at 50° for 5 minutes. It is now filtered hot through a Büchner funnel into a 500-ml. filter flask well wrapped with black paper or aluminum foil. The silver iodide that is collected is washed with a 10-ml. portion of warm dioxane. Carbon tetrachloride (200 ml.) is added to the combined filtrates in the filter flask, and the solution is chilled overnight at –8° to –20°. N-Iodosuccinimide separates as colorless crystals. It is collected on a Büchner funnel with as little exposure to light as possible, washed with 25 ml. of carbon tetrachloride, and dried with suction. After being dried overnight in the dark at 25° (1 mm.) the N-iodosuccinimide weighs 14.3–15.1 g. (81–85% yield) and melts with decomposition at 193–199° (Note 3).

2. Notes

1. The dioxane is purified only by the use of sodium strips and distillation.³ The checkers used a newly opened bottle of "Spectroquality Reagent" dioxane (Matheson, Coleman and Bell) without further treatment.
2. N-Silver succinimide was prepared by the method of Djerassi and Lenk.⁴ The checkers rapidly added a solution of 64 g. (1.6 moles) of sodium hydroxide in 300 ml. of water dropwise to a stirred solution of 249 g. (1.47 moles) of silver nitrate in 700 ml. of water at room temperature. The silver oxide that formed was separated on a Büchner funnel and washed with water. The moist oxide was added in one portion to a boiling solution of 133 g. (1.34 moles) of succinimide in 4 l. of water. The reaction vessel was wrapped with aluminum foil in order to exclude as much light as possible. After 45 minutes, the suspension was filtered through a heated Büchner funnel into a filter flask also wrapped with aluminum foil. The filtrate was allowed to stand at room temperature overnight, during which time N-silver succinimide crystallized. The N-silver succinimide was separated on a Büchner funnel, dried in air under suction, and ground to a powder. After being dried in a vacuum oven for 1 hour at 110°, it weighed 128 g. (47%). N-Silver succinimide should be stored in a brown bottle.
3. This material is pure enough to use in the preparation of α -iodoketones. The checkers found that, after one recrystallization from a mixture of dioxane and carbon tetrachloride, the N-iodosuccinimide melted with decomposition at 195–200°. Pure N-iodosuccinimide is reported to melt at 200–201°.⁴

3. Discussion

N-Iodosuccinimide has been prepared by the action of iodine on N-silver succinimide,^{4,5} and by the action of iodine monochloride on the sodium salt of succinimide.⁶ The present procedure is essentially that of Djerassi and Lenk,⁴ with the modification that dioxane is the reaction medium instead of acetone;

dioxane gives a better yield without formation of a lachrymatory by-product.

4. Merits of Preparation

N-Iodosuccinimide reacts with enol acetates derived from ketones to give α -iodoketones, and the reaction has found application in the steroid field.^{4,6} The iodination of the enol acetates seems to proceed by an ionic mechanism, and preliminary work indicates that N-iodosuccinimide is not capable of at least some of the radical-chain iodinations analogous to radical-chain brominations brought about by N-bromosuccinimide.⁴ N-Iodosuccinimide has also been used for the iodination of purine nucleosides⁷ and of fluorenone.⁸ Iodination of 2-hydroxy-3-(β -alkylvinyl)-1,4-naphthoquinones results in an unusual replacement of a vinyl hydrogen.⁹

This preparation is referenced from:

- [Org. Syn. Coll. Vol. 6, 226](#)

References and Notes

1. Chemistry Department, Colorado State University, Fort Collins, Colo.
2. Department of Chemistry, Purdue University, Lafayette, Ind.
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7. D. Nowotny and D. Lipken, *Monatsh. Chem.*, **96**, 125 (1965).
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Appendix

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

N-silver succinimide

[sodium hydroxide](#) (1310-73-2)

[silver oxide](#) (20667-12-3)

[silver nitrate](#) (7761-88-8)

[carbon tetrachloride](#) (56-23-5)

[iodine](#) (7553-56-2)

[acetone](#) (67-64-1)

[sodium](#) (13966-32-0)

[Succinimide](#) (123-56-8)

dioxane (123-91-1)

iodine monochloride (7790-99-0)

N-bromosuccinimide (128-08-5)

fluorenone (486-25-9)

silver iodide (7783-96-2)

N-Iodosuccinimide,
Succinimide, N-iodo- (516-12-1)

sodium salt of succinimide