

## Englisch

Name, Vorname:

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**Hinweise:**

- Erlaubte Hilfsmittel: gedrucktes oder elektronisches Wörterbuch
- Prüfungszeit: 60 Minuten (Teil 1 ca. 40 Minuten, Teil 2 ca. 20 Minuten)
- Bewertung: Teil 1 zählt 2/3, Teil 2 zählt 1/3 der Note im Englisch. Notenschlüssel gemäss Prüfungskommission.

*Die Prüfungskommission wünscht Ihnen gutes Gelingen und viel Erfolg!*

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### Teil 1: Übersetzung englischer Fachtext ins Deutsche

Bitte übersetzen Sie aus der Kopie der Originalliteratur die mit einem seitlichen Balken markierten Stellen (Titel, „Microscale Procedure“ und „Safety“) und schreiben Sie die Übersetzung auf die 4. Seite und deren Rückseite.

## Microscale Synthesis of 1-Bromo-3-chloro-5-iodobenzene: An Improved Deamination of 4-Bromo-2-chloro-6-iodoaniline

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The multistep synthesis of 1-bromo-3-chloro-5-iodobenzene has appeared both in this *Journal* (1) and in laboratory texts (2–3). The final step in the synthesis is the reductive deamination of 4-bromo-2-chloro-6-iodoaniline using a standard diazonium route, in which the final product is isolated by steam distillation. Not only does this require the use of gram quantities of 4-bromo-2-chloro-6-iodoaniline, but also the product, 1-bromo-3-chloro-5-iodobenzene, “has a tendency to solidify in the condenser” (3).

In an effort to eliminate the lengthy and often inefficient product isolation procedure, we sought to identify an alternative deamination method. Alkyl nitrites in *N,N*-dimethylformamide can be utilized for the reductive deamination of haloanilines (4). In this article, we report that this reaction can be performed successfully in an undergraduate laboratory using microscale techniques. The reaction time has been significantly decreased and the production isolation uses simple extraction techniques. Additionally, the scale of the experiment has been reduced to require less starting material (0.25 g).

The procedure provided utilizes commercially available isoamyl nitrite (Figure 1). Other alkyl nitrites will also work in this procedure. For example, we have obtained excellent results using *n*-butyl nitrite (85–90%), synthesized according to a published procedure (5).

### Microscale Procedure

Under a fume hood, 1.65 mL of a 0.75 M isoamyl nitrite/DMF solution<sup>1</sup> is added to a 10-mL round-bottomed flask containing a magnetic spin vane. A reflux condenser is placed on the flask and the flask is clamped in a sand bath heated to 65 °C. With the isoamyl nitrite solution stirring rapidly, a solution of 4-bromo-2-chloro-6-iodoaniline (0.25 g, 0.75 mmol) dissolved in a minimal amount of DMF (2

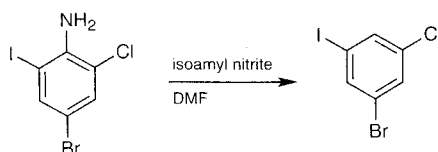


Figure 1. Reductive deamination step—the final step.

mL) is added dropwise to the reaction solution over a period of 5 min.<sup>2</sup> The evolution of nitrogen gas is immediate and continues until all of the aniline solution is added.<sup>3</sup> After the gas evolution is complete (~15 min), the reaction mixture is allowed to cool to room temperature and then carefully transferred to a 25-mL Erlenmeyer flask. Fifteen mL of 20% aqueous hydrochloric acid solution is added to the reaction mixture and the product is extracted from the reaction mixture into two, 10-mL portions of ether. The combined organic extract is washed with 15 mL of 10% aqueous hydrochloric acid solution, dried over anhydrous magnesium sulfate, filtered, and concentrated to yield the product as a pale copper-colored solid.<sup>4</sup> The crude product is recrystallized from methanol, using a ratio of 6 mL of methanol per gram of product, to give 1-bromo-3-chloro-5-iodobenzene as long, colorless needles (75–80%, m.p. 85–86 °C).

### Safety

Wear eye protection at all times in the laboratory. Work in a hood or in a well-ventilated area. Care must be used in handling the alkyl nitrite; inhalation of the vapor may cause severe headache and heart excitation. *N,N*-dimethylformamide is flammable and absorbed through the skin. Diethyl ether is flammable. Hydrochloric acid solutions are corrosive and toxic and can cause burns if they come in contact with your skin. Wash affected areas with copious quantities of water.

### Results and Discussions

We incorporate this experiment into our curriculum at the end of the second semester of organic chemistry laboratory as a part of a multistep synthesis (Figure 2). Our focus in the teaching laboratories is to allow students to learn chemistry by doing experiments similar to those done by practicing chemists. By including a multistep synthesis at the end of the course, students apply a variety of skills and techniques learned during the two-semester laboratory course. They are also able to see firsthand the importance of synthetic design, the importance of high yields and product purity at each step, and the dependence of the overall product yield and quality on the individual transformations. This reaction sequence provides an excellent review of the concept of group effects in electrophilic aromatic substitution reactions. In particular, it demonstrates the use of activating and deactivating groups and the use of a “traceless” directing group.

## Teil 2: Grammatik

1.) Make questions by putting the words in correct order (6 points for correct sentences):

you from are where?

do in Germany live where you?

you living where are in Oxford?

at school did learn you English?

have how long been in England you?

this evening what do you to are going?

2.) Fill in the gaps by finishing the sentence and using the correct past time of the verbs. You can use a verb only once! (8 points).

*2 years ago we all went to Varazze, Italy. When we arrived on Saturday*

- we \_\_\_\_\_ .
- On Sunday we \_\_\_\_\_ .
- On Monday \_\_\_\_\_ .
- On \_\_\_\_\_ .
- On \_\_\_\_\_ .
- On \_\_\_\_\_ .
- On \_\_\_\_\_ .
- On \_\_\_\_\_ .

3.) Questionnaire: Fill in the chart by making a question or a correct answer (8 points).

What do you want to do when you grow up?	
	It's the 25 April.
What are you good at?	
What are your favourite clothes?	
	About 45 minutes.
	No, I haven't.
	How do you do.

Punkte Teil 2

Note Teil 2

Name, Vorname:

Punkte Teil 1

Note Teil 1

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Deutsche Übersetzung (Teil 1):