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Prior to performing each reaction, a thorough hazard analysis and risk assessment should be carried out with regard to each chemical substance and experimental operation on the scale planned and in the context of the laboratory where the procedures will be carried out.”

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Organic Syntheses – Search Results for DMSO Anywhere in the Text

Preparation of MIDA Anhydride and Reaction with Boronic Acids

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Colloquial PDF: Rich HTML

Figure 1. Preparation of MIDA and Reaction with Boronic Acids

1. Procedure (Note 1)

A. MIDA anhydride (14). A 400-mL three-necked, 250-mL round-bottomed flask equipped with a 5-L 3-way stopcock was charged with t-butyldimethylsilyl chloride (14 g, 120 mmol, 3.6 equiv) and dry toluene (40 mL). The flask was then heated at reflux for 1 h.

B. Reaction with Boronic Acids

1. Procedure (Note 2)

A. Reformulation of MIDA anhydride (14). A 40-mL two-necked, 100-mL round-bottomed flask equipped with a 5-L 3-way stopcock was charged with t-butyldimethylsilyl chloride (14 g, 120 mmol, 3.6 equiv) and dry toluene (40 mL). The flask was then heated at reflux for 1 h.

2. Procedure (Note 3)

A. Reaction mixture after heating at 70°C for 1.5 h. The reaction mixture is filtered through a calcium carbonate (500 mg) and washed with ether (50 mL) before being concentrated by sublimation.

Figure 2. A. Drying MIDA using reduced pressure to remove solvent (See Note 2). B. Reaction mixture after addition of pyridine and acetic anhydride. C. Heating reaction mixture at 70°C for 2 min in an oil bath (See Note 3).

Figure 3. A. Reaction mixture after heating at 70°C for 1.5 h. B. Graded mixture following anisotropic recrystallization of boronate. C. Appearance of crude material after recrystallization (See Note 3).