Category

Synthesis of Heterocycles

Key words

1,3-dipolar cycloaddition

iodine

benzo(f)isoindole-4,9-diones

naphthoquinones

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The First Iodine Improved 1,3-Dipolar Cycloaddition: Facile and Novel Synthesis of 2-Substituted Benzo[f]isoindole-4,9-diones
Tetrahedron 2013, 69, 9033–9037.

Iodine-Mediated 1,3-Dipolar Cycloaddition to Benzo(f)isoindole-4,9-diones



Significance: Reported is the iodine-mediated synthesis of fused benzo(*f*)isoindole-4,9-diones through the 1,3-dipolar cycloaddition of naphthoquinones (also anthracene-1,4-dione) with azomethine ylides, the latter being generated in situ from N-alkylated glycine esters and paraformaldehyde. Unfortunately, although the optimization work was performed using a standard reaction time of eight hours, the reaction times of the formation of each of the products in the substrate scope was not given. Comment: Substituted benzo(f)isoindole-4,9diones are present in many biologically relevant molecules, some examples of which are given in the introduction of the report. The current work provides a rapid and direct method for their synthesis. This method benefits from operational simplicity, no requirement for air-free conditions, and mostly moderate to excellent yields. The reaction was optimized with respect to iodine stoichiometry, solvent, and base. The substrate scope was modestly examined with respect to the glycine ester; moving from ethyl to more sterically bulky esters resulted in lower yields of products. N-Phenylglycine ethyl ester failed in the reaction, implying that aromatic N-substitution is not tolerated. The substrate scope was poorly examined with respect to the quinone; both 5-hydroxy and 5-nitro naphthoquinones gave almost identical, but modest yields (50% and 53%), although anthracene-1,4-dione was capable of furnishing good yields.

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