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Improved method of preparing Diethyl Furoxan Dicarboxylate

A high-yield, safe, water-based, single-step route to DFD that removes toxic solvents and metals for scalable production.

Diethyl furoxan dicarboxylate (DFD) is a starting material for fields as diverse as drug discovery, energetics, plasticizers, and more. As with many di-substituted furoxans, they are synthesized via the dimerization of the appropriate nitrile oxide. Past procedures to form DFD may have involved low-yield destructive nitrations, which are notoriously exothermic and difficult to control. Alternatives may have required multiple steps, halogenated solvents, complex separations, or heavy or precious metals. Although these methods are functional enough for lab scale preparations of DFD, they have limited capability for economical scale-up in industrial processes. To fulfill this market demand, researchers at Purdue University have developed a new procedure that improves the synthesis of DFD by making it simpler, more cost effective, and safer. With their process, DFD can be made using economical and commercially available starting materials in a single-step, one-pot, high-yield synthesis reaction. This improved procedure requires no halogenated solvents, heavy metals, or difficult-to-store reagents and is the most scalable preparation route for this material to date.

Technology Validation:

Using this novel reaction, researchers were able to confirm the production of a large, easily isolated quantity of pure DFD. On a small scale (7 g starting material), the total yield was measured as 98.5% with 97% purity. Temperature measurements performed using an EasyMax 102 reaction vessel revealed a mild exothermic quality, in the range of 5 K. The researchers verified the safety of the DFD to mechanical stimulus with a BAM Fall Hammer test.

Advantages:

-Reduced production cost

-Simplified synthesis process and trivialized work-up

Technology ID

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Category

Semiconductors/Devices & Components
Semiconductors/Packaging & Integration
Chemicals & Advanced Materials/Materials Processing & Manufacturing Technologies

Further information

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-No need for precious or heavy metals, organic or halogenated solvents, or hard-to-store reagents

-Scalable and economical for large industrial uses

-High purity and yield

-Safe for scale-up

-Water solvent

Applications:

-Chemical supply

-Pharmaceutical production

-Energetic material production

-Plasticizers

-Applied and basic research

Publications:

Economic, One-Pot Synthesis of Diethyl Furoxan Dicarboxylate. Michael Thoenen, Nicholas F. Scherschel, and Davin G. Piercey. Organic Process Research & Development. https://doi.org/10.1021/acs.oprd.4c00191

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