

Safety Note:**Bis(1-benzo[*d*]triazolyl)oxalate and Bis(1-benzo[*d*]triazolyl)carbonate as Potential Explosive Compounds**

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Abstract: The two diesters (**1** and **2**) mentioned in the title are very shock sensitive in dry state. They can be stabilized by adding 30% of 1,1,2-trichloroethane.

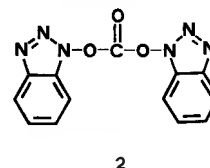
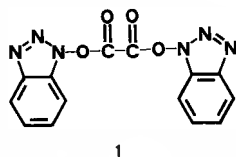
Bis(1-benzo[*d*]triazolyl)oxalate **1** (Di-1-benzotriazolyl oxalate, DBTO)^[1] and Bis(1-benzo[*d*]triazolyl)carbonate **2** (Di-1-benzotriazolyl carbonate)^[2] have been shown to be promising condensating agents in organic synthesis. We wish now to draw attention to the critical properties of these reagents on inadequate handling, and how they can be stabilized for their safe use in synthesis.

- the temperature must not exceed 60°C during synthesis, in particular the wall of the reaction vessel should not be overheated;

- grinding or exposure to heat sources must be avoided (self-ignition);

- the carefully isolated products can be stabilized by producing a preparation containing an inert solvent

- **1** with 40% 1,1,2-trichloroethane,
- **2** with 30% 1,1,2-trichloroethane.



On checking the melting points of both of these reagents, prepared according to literature^[1,2], we had to accept the fact that both substances *decompose explosively* at the published melting points of about 160°C (**1**) and 150°C (**2**) respectively. Further safety tests^[3] have disclosed a high *shock sensitivity* as well as a pronounced combustibility and ignition capacity. Control of thermal stability by differential thermometry in an open tube^[3] revealed moreover a decomposition temperature with explosion at 130°C for **1** and 120°C for **2**.

On observing the following precautions, the oxalate **1** as well as the carbonate **2** can safely be prepared and handled:

The material so obtained is no longer shock sensitive and can be safely handled. Both reagents are now commercially available in this stabilized form and should be used as such without drying. If drying should be necessary, this can be done at 30–40°C under reduced pressure.

Received: December 20, 1984 [FC 7]

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[1] K. Takeda, I. Sawada, A. Suzuki, H. Ogura, *Tetrahedron Lett.* 24 (1983) 4451.

[2] M. Ueda, H. Oikawa, T. Teshirogi, *Synthesis* 1983, 908.

[3] The safety test have been carried out according to the standards published in «Sicherheitstests für Chemikalien, Schriftenreihe der Expertenkommission für Sicherheit in der Chemischen Industrie der Schweiz (ESCIS)», *Chem. Rundsch.* 30 (1977) Nr. 1.