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A Solid State Photo-Diels-Alder Reaction

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A SOLID STATE PHOTO-DIELS-ALDER REACTION

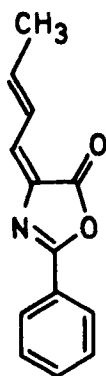
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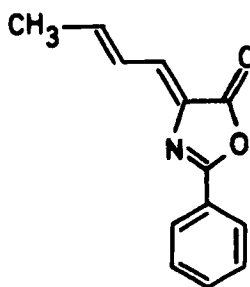
Abstract On irradiation in the solid state, the oxazolone (Z)-1 yields the 4+2-photodimer 2.

On irradiation, substituted 4-cyclohexylidene-5(4H)-oxazolones yield three different non-symmetric solid state dimers. These dimers are formed by complex, unique dimerization reactions between glide plane related monomers¹.

In continuation of this work, we examined the solid state reactivity of the 4-butenylidene-5(4H)-oxazolones (E)-1 and (Z)-1. The (E)-isomer proved to be photostable; this behaviour is rationalized by its packing geometry.

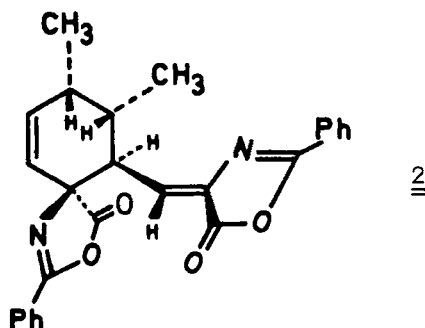


(E)-1



(Z)-1

The planar (E)-1 crystallizes in a layered structure ($a = 7.707$, $b = 7.884$, $c = 10.551$ Å, $\alpha = 100.41$, $\beta = 98.10$, $\gamma = 116.15^\circ$, $P\bar{1}$, $Z = 2$, $R = 0.048$) with unfavourable geometric relations for a solid state reaction.



In contrast, (Z)-1 yields a photodimer 2 ($a = 9.286$, $b = 14.831$, $c = 16.388$ Å, $\beta = 99.31^\circ$, $P2_1/n$, $Z = 4$, $R = 0.047$), isolated in 30 % yield. During the course of this remarkable 4+2-photodimerization, one s-trans-diene unit has to isomerize to the s-cis-conformation in the solid state. Unfortunately, the packing type of the monomer (Z)-1 is still unknown due to twinning problems. A tentative dimerization mechanism again suggests short contacts between glide plane related molecules.

The preparation and solid state photoreactivity of further substituted oxazolones of type (E/Z)-1 is in progress.

REFERENCE

1. S. Mohr, Tetrahedron Lett. 1980, 593.