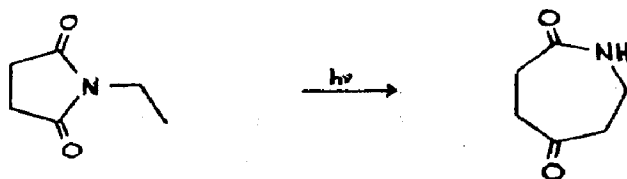


## Organic Photochemical Synthesis

Tetrahydro-1*H*-azepine-2,5-dione

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## 1. Procedure

A solution of *N*-ethylsuccinimide (3.81 g, 30 mmol) (note 1) in acetonitrile (450 cm<sup>3</sup>) (note 2) is placed in the reaction vessel (note 3) and a stream of oxygen-free nitrogen is passed through it for 30 min. The solution is then irradiated (note 4) under nitrogen. The course of the reaction can be followed by gas chromatography (note 5) and the conversion is nearly complete after irradiation for 4 h.

The solvent is removed on a rotary evaporator under reduced pressure at approximately 40 °C. The residual pale yellow solid is subjected to column chromatography on 80 g of silica (Merck 7734) (note 6). With ethyl acetate (250 cm<sup>3</sup>) as eluant a mixture (0.644 g) of *N*-ethylsuccinimide and succinimide (note 7) is obtained, then elution with acetone (600 cm<sup>3</sup>) affords, on evaporation of the solvent, tetrahydro-1*H*-azepin-2,5-dione as almost pure colourless crystals (melting point, 135 - 139 °C; 2.42 g, 64%). Recrystallization from acetone (40 cm<sup>3</sup>) gives colourless prisms (melting point, 139 - 140 °C; 1.93 g, 51%). A second crop (melting point, 137.5 - 139.5 °C; 0.29 g, 8%) is obtained from the mother liquor.

## 2. Notes

(1) *N*-Ethylsuccinimide is prepared from succinic anhydride and ethylamine by adaptation of the procedure for succinimide [1].

(2) Acetonitrile is purified by distillation over phosphorus pentoxide.

(3) The apparatus consists of a cylindrical Pyrex vessel with an inside wall made of quartz and is equipped with a nitrogen inlet tube.

(4) The lamp used was a 60 W Eikosha low pressure mercury arc.

(5) The analysis was performed with the aid of a Hitachi Model 063 gas chromatograph fitted with a 2 m × 3 mm 1% poly(neopentyl glycol adipate) on 60/80 Chromosorb WAW DMCS column at 110 °C.

(6) The checker isolated the product as follows. After evaporation of the solvent, the residual solid was dissolved in acetone to remove insoluble matter. The filtrate was then condensed to half its volume to give a crystalline product as the first crop (52%). The second crop of the product (8%) was obtained from the mother liquor by column chromatography.

(7) Succinimide is a product of a Norrish type-2 elimination [2]. The mixture consists of approximately 1:1 starting material and succinimide.

### *3. Methods and merits of preparation*

The procedure described here is based on that originally published by Kanaoka and Hatanaka [2]. Although tetrahydro-1*H*-azepin-2,5-dione has been claimed to be synthesized by the Beckmann rearrangement of 1,3-dioxan-2-spirocyclohexan-4'-one oxime [3], neither the melting point nor the yield were described. This preparation is offered as an example of a technique that can be used to afford a variety of medium-sized ketolactams which are difficult to prepare [2, 4].

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