United States Patent [19]

Adams, Jr. et al.

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[54] **1-FLUORO-1,1,5-TRINITRO-3-OXA-5-AZAHEXANE AND METHOD OF PREPARATION**

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[52] **U.S. Cl.** **564/111**; 149/92; 560/227; 560/251; 562/605; 562/607

[58] **Field of Search** 560/227, 251; 564/111

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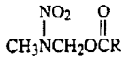
[57] **ABSTRACT**

1-Fluoro-1,1,5-trinitro-3-oxa-5-azahexane which is prepared by reacting one mole of 2-fluoro-2,2-dinitroethanol with one mole of an acetate of the formula



wherein R is —CF_3 or CH_3.

4 Claims, No Drawings

After 3 hours the solution was allowed to cool and was then triturated with 50 ml of ice water. The organic phase was separated, dried (MG SO_4), and distilled. The product 2-nitro-2-azapropyl acetate had b.p. 56°–57° C. at 0.07 mm Hg.

EXAMPLE 2

1-fluoro-1,1,5-trinitro-3-oxa-5-azahexane prepared from 2-nitro-2-azapropyl acetate

2-Fluoro-2,2-dinitroethanol (9.86 g, 64.0 mmol) and 2-nitro-2-azapropyl acetate (8.88 g, 60.0 mmol) in dichloromethane (30 ml) were stirred under nitrogen at 0° C. to 5° C. Boron trifluoride etherate (7.52 ml, 8.46 g, 60 mmol) was added during 5 minutes, and stirring was continued for 70 minutes. The mixture was then poured on to a saturated solution of sodium chloride (10 ml/mmol). The yield was 10 g of an oil.

Chromatography on 350 g silica gel (Merck, Kieselgel 40) eluting with 3:2 dichloromethane/hexane to dichloromethane gave 1-fluoro-1,1,5-trinitro-3-oxa-5-azahexane; yield: 4.5 gm (31%). Recrystallization from ether/hexane at −78° C. gave 1-fluoro-1,1,5-trinitro-3-oxa-5-azahexane; yield: 4.1 gm (28%); m.p. 13.0°–14.2° C.

H-N.M.R. ($CDCl_3$/TMS_{intl}): $\delta=3.5$ (s, 3 H); 4.87 (d, J=19 Hz, 2 H); 5.4 ppm (s, 2 H).

EXAMPLE 3

2-nitro-2-azapropyl trifluoroacetate

Trifluoroacetic anhydride (20 ml, 103.6 g, 0.49 moles) was added to a stirred solution of 1-hydroxy-2-nitro-2-azapropane (40.0 g, 0.38 moles) in dichloromethane (60 ml) at −5° to 0° C. during 1½ hours. The resulting solution was then distilled without warming at 20 mm, then to 3 mm. The product was distilled at 1.5 mm, 65° C. to give 2-nitro-2-azapropyl trifluoroacetate; yield: 48 g (63%).

^{1}H-N.M.R. ($CDCl_3$/TMS_{intl}): $\delta=3.58$ (s, 3 H), 6.08 ppm (s, 2 H).

EXAMPLE 4

1-fluoro-1,1,5-trinitro-3-oxa-5-azahexane prepared from 2-nitro-2-azapropyl trifluoroacetate

2-Fluoro-2,2-dinitroethanol (7.62 g, 49.5 mmol) was stirred with acetonitrile (2.5 ml) under nitrogen as 2-nitro-2-azapropyl trifluoroacetate (10.0 g, 49.5 mmol) in acetonitrile (3.5 ml) was added in one portion. The reaction flask was immediately connected to a vacuum pump and brought to 1–2 mm during several minutes. After stirring 2⅔ hours, the solution was stirred ⅓ hour at ambient pressure before pouring onto a saturated sodium chloride solution (50 ml), which was extracted with dichloromethane (3×25 ml). The organic portion was washed with a 1:1 mixture of saturated sodium chloride solution: 0.04 N sodium hydroxide solution (10×150 ml), dried with magnesium sulfate, and concentrated to crude 1-fluoro-1,1,5-trinitro-3-oxa-5-azahexane; yield: 8.67 g (72%). Recrystallization from ether/hexane at −78° C. gave 1-fluoro-1,1,5-trinitro-3-oxa-5-azahexane; yield: 7.5 g (63%).

^{1}H-N.M.R. ($CDCl_3$/TMS_{int}) : δ3.5 (s, 3 H); 4.87 (d, J=19 Hz, 2 H); 5.4 ppm (s, 2 H).

Obviously, numerous modifications and variations of the present invention are possible in light of the above teachings. It is therefore to be understood that within the scope of the appended claims the invention may be practiced otherwise than as specifically described herein.

What is claimed as new and desired to be secured by Letters Patent of the United States is:

1. 1-fluoro-1,1,5-trinitro-3-oxa-5-azahexane.

2. A method of preparing 1-fluoro-1,1,5-trinitro-3-oxa-5-azahexane comprising:

(1) reacting one mole of 2-nitro-2-azapropyl trifluoroacetate with one mole of 2-fluoro-2,2-dinitroethanol in a polar solvent at a temperature of from 0° C. to 40° C. to form 1-fluoro-1,1,5-trinitro-3-oxa-5azahexane, wherein the reaction is run under vacuum to remove trifluoroacetic acid generated by the reaction; and

(2) isolating the product 1-fluoro-1,1,5-trinitro-3oxa-5-azahexane.

3. The method of claim **2** wherein the reaction is run at a temperature of from 20° C. to 25° C.

4. The method of claim **2** wherein the polar solvent is acetonitrile.

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