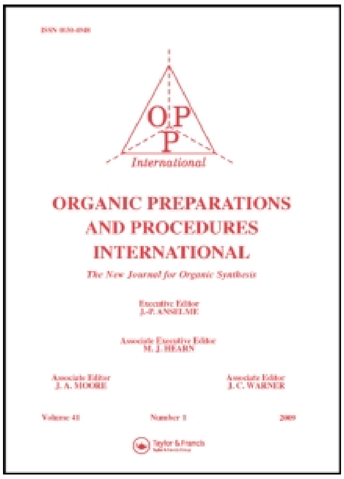

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trans-3,5-DIPHENYL-1-PYRAZOLINE

J. -P. Anselme[a]
[a] Department of Chemistry, University of Massachusetts, Boston

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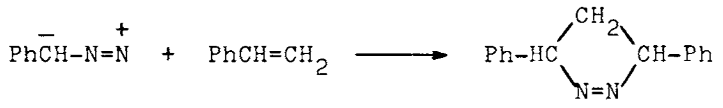
J.-P. Anselme
Department of Chemistry
University of Massachusetts, Boston 02116



The present procedure describes the preparation of *trans*-3,5-diphenyl-1-pyrazoline.[1] It is essentially similar to that previously reported but the preparation of phenyldiazomethane is also described in detail and the yield of pyrazoline obtained has been improved from 24 to 54%.

Experimental

Phenyldiazomethane[2] To a solution of 66 g. (2.0 moles) of anhydrous hydrazine in 200 ml. of ether, is added over a period of 5 hrs. a solution of 106 g. (1.0 mole) of benzaldehyde in 300 ml. of ether. The temperature is maintained at or near 0° throughout the addition. The cloudy white reaction mixture is allowed to stir for 24 hrs. after the addition of benzaldehyde is completed. The ether layer is separated and washed once with 500 ml. of cold water. The resulting ether solution of benzalhydrazone is then added to a 2 l. three-necked round bottom flask containing 200 g. of anhydrous sodium sulfate. The flask, fitted with a mechanical stirrer, a reflux condenser and a solids addition funnel, is then immersed in an ice-salt bath. After the addition of 50 ml. of a saturated methanolic solution of potassium hydroxide, 300 g. of yellow mercuric oxide is added portionwise over a period of 2 hours. The resulting deep wine-red solution of phenyldiazomethane is filtered through a pad of filter aid to separate it from the inorganic solids and mercury. To the reaction flask still containing inorganic solids and mercury is added about 300 ml. of anhydrous ether and the ether solution filtered. The material collected on the Buchner funnel is then washed with 200 ml. of anhydrous ether to remove adhering phenyldiazomethane.[3]

<u>3,5-Diphenyl-1-pyrazoline</u> To the solution of phenyldiazomethane[4] in a 2 l. three-necked round bottom flask, equipped with a condenser and a calcium chloride tube is added 94 g. (0.9 mole, 104 ml.) of styrene.[5] The contents of the flask can be conveniently stirred by means of a magnetic bar. The flask is then wrapped <u>completely</u> with aluminum foil and the reaction allowed to proceed at room temperature for 24 hrs. After this time, some white crystals are visible. The flask is then cooled in a Dry Ice - acetone bath for 1 hr. and the precipipated product, weighing 25 g.[6] is collected and washed with cold pentane to remove the red color. Upon two successive concentrations and overnight cooling of the filtrates, an additional 10 g. of product[6] can be obtained. The total yield of 3,5-diphenyl-1-pyrazoline[7] amounts to 36 g. (54%).

References

1. C. G. Overberger and J.-P. Anselme, J. Am. Chem. Soc., 86, 658 (1964).
2. CAUTION! Although no accident occurred during this sequence of reactions, adequate precautions (Hood) should be taken when handling anhydrous hydrazine and phenyldiazomethane.
3. Adhering phenyldiazomethane (both on the Buchner funnel and in the flask) should be destroyed by the cautious addition of dilute acetic acid as soon as possible.
4. The concentration and yield of phenyldiazomethane were not determined. The percentage yield of the final addition product 3,5-diphenyl-1-pyrazoline is based on a minimum of 0.30 mole of phenyldiazomethane (C. D. Gutsche and E. F. Jason, J. Am. Chem. Soc., 78, 1184 (1956)).
5. Commercial styrene was used without purification.
6. All yields given are for dried, recrystallized product, m.p. 108-109° (dec.). The product can be purified by the addition of boiling methanol until solution occurs (adding charcoal if necessary) followed by filtration. It crystallizes as shiny white flakes. The analytically pure product melts at 109-110° (dec.)[1].
7. The product deteriorates even when kept cold in a dark bottle and must be recrystallized just prior to use.

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