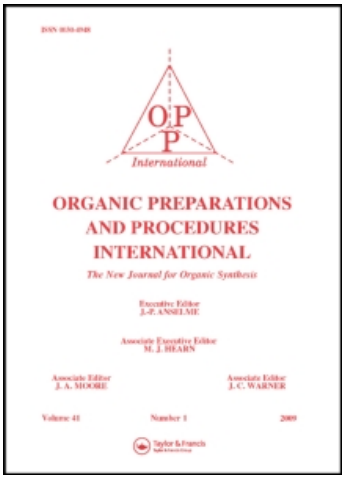


Organic Preparations and Procedures International
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t902189982

3-ARYL-5-CARBOMETHOXY-5-CARBOMETHOXYMETHYL-1,4,2-OXATHIAZOLES

Ned D. Heindel[a]; Gail K. Friedrich[a]; Maria C. Tsai[a]
[a] Department of Chemistry, Lehigh University, Bethlehem, PA

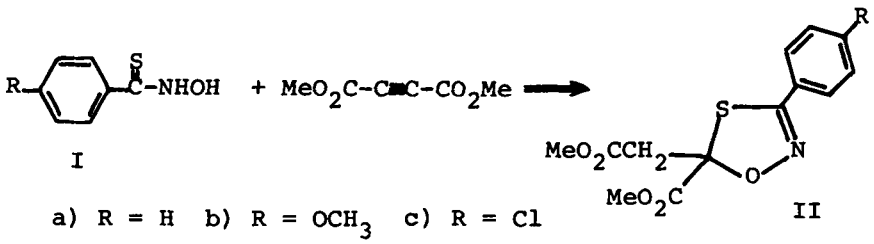
To cite this Article Heindel, Ned D. , Friedrich, Gail K. and Tsai, Maria C.(1980) '3-ARYL-5-CARBOMETHOXY-5-CARBOMETHOXYMETHYL-1,4,2-OXATHIAZOLES', Organic Preparations and Procedures International, 12: 3, 233 — 234
To link to this Article: DOI: 10.1080/00304948009458556
URL: http://dx.doi.org/10.1080/00304948009458556

3-ARYL-5-CARBOMETHOXY-5-CARBOMETHOXYMETHYL-1,4,2-OXATHIAZOLES

Submitted by Ned D. Heindel*, Gail K. Friedrich†, and Maria C. Tsai††
(8/22/79)

Department of Chemistry
Lehigh University
Bethlehem, PA 18015

Low level central nervous system effects (abnormal gait, writhing, loss of body tone) have been observed at 300 mg/kg in a mouse screen of two new (IIa-b) and one reported (IIc) 1,4,2-oxathiazole. These compounds were prepared by an improved modification of the synthesis of that class.[1] Aryl thiohydroxamic acids, known to be labile to autodecomposition,[2] are reacted in the dark under nitrogen atmosphere with dimethyl acetylenedicar-



a) R = H b) R = OCH_3 c) R = Cl

boxylate to give the title compounds. The products are recognized by their non-conjugated ester carbonyls in the infrared (1730-1740 cm^{-1}) and by their AB quartet (J = 15-16 Hz) methylene resonances centered at 3.44 ± 0.04 ppm in the NMR. This coupling has been noted.[1]

EXPERIMENTAL

General Procedure.- An equimolar solution (3.0 mmol) of the requisite thiohydroxamic acid (Ia[2], Ib[2], or Ic[3]) and dimethyl acetylenedicarboxylate was prepared in 25 ml of absolute methanol, briefly degassed by bubbling with a nitrogen stream, sealed under a mercury bubbler, heated at reflux for 0.5 h in a foil-wrapped flask, and then allowed to stand at room temperature for 2 days. The solvent was removed in vacuo and the resulting oil triturated with water:methanol (1:5) to induce crystallization. The crude pro-

ducts were recrystallized from methanol to analytical purity to yield: IIa, 63%, mp. 68-69°; IIb, 57%, mp. 67-69° and IIc, 54%, mp. 85-87°, lit.[1] mp. 88-90°.

Anal. Calcd for $C_{13}H_{13}NO_5S$ (IIa): C, 52.88; H, 4.43; N, 4.74.
Found: C, 52.96; H, 4.38; N, 4.50.

Anal. Calcd for $C_{14}H_{15}NO_6S$ (IIb): C, 51.68; H, 4.64; N, 4.30.
Found: C, 51.69; H, 4.66; N, 4.23.

REFERENCES

† Present address: Rohm and Haas Co., Philadelphia, Pa.

†† Present address: University of the District of Columbia, Washington, D.C.

1. P. Rajagopalan and C. N. Talaty, Heterocycles, 3, 563 (1975).
2. T. Bacchetti and A. Alemagna, Rend. ist. lombardo sci. Pt. I, 91, 30 (1975). See Chem. Abstr., 52, 11749e (1958).
3. T. Bacchetti and A. Alemagna, Atti acad. nazl. Lincei Rend., Classe sci. fis. mat. e nat., 24, 161 (1958). See also Chem. Abstr., 52, 18299 (1958).

IMPROVED PREPARATION OF PYRIDO[3',4'(4',3'):4,5]IMIDAZO [1,2-c] [1,2,3]BENZOTRIAZINES

Submitted by Jessie Lee
(10/15/79)
Department of Chemistry
Philadelphia Community College
Philadelphia, Pennsylvania 19104
and
Anne Guthrie and Madeleine M. Joullié*
Department of Chemistry
University of Pennsylvania
Philadelphia, Pennsylvania 19104

In 1968, we reported a new system, pyrido[3',4',(4',3'):4,5]imidazo-[1,2-c][1,2,3]benzotriazine (1) which could theoretically exist in two iso-