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

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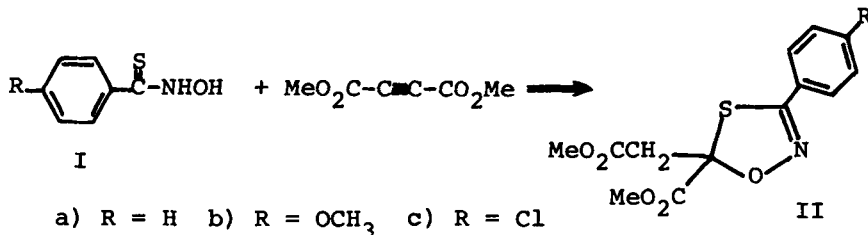
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3-ARYL-5-CARBOMETHOXY-5-CARBOMETHOXYMETHYL-1,4,2-OXATHIAZOLES

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(8/22/79)

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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-



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 (Ia², Ib², or Ic³) 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-

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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 C₁₃H₁₃NO₅S (IIa): C, 52.88; H, 4.43; N, 4.74.

Found: C, 52.96; H, 4.38; N, 4.50.

Anal. Calcd for C₁₄H₁₅NO₆S (IIb): C, 51.68; H, 4.64; N, 4.30.

Found: C, 51.69; H, 4.66; N, 4.23.

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IMPROVED PREPARATION OF PYRIDO[3',4'(4',3'):4,5]IMIDAZO

[1,2-c] [1,2,3]BENZOTRIAZINES

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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-