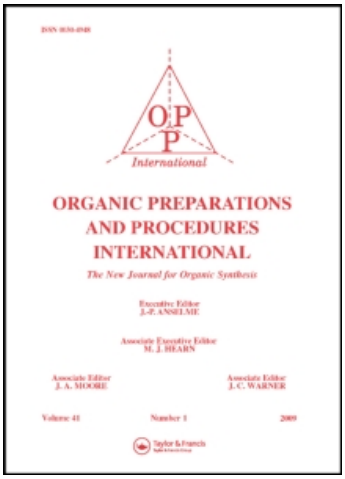

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THE SYNTHESIS OF SOME NEW 3-METHYL-2-PYRAZOLIN-5-ONES

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aryl); ir (KBr) 1270, 1620, 3280, 3400 cm^{-1} (Ar-NH_2).

Anal. Calcd. for $C_7H_6NCl_3$: C 39.93, H 2.85, N 6.66, Cl 50.59
Found: C 39.80, H 2.83, N 6.82, Cl 50.18

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4. Exchanges with D_2O.

THE SYNTHESIS OF SOME NEW 3-METHYL-2-PYRAZOLIN-5-ONES

Submitted by (3/4/76)

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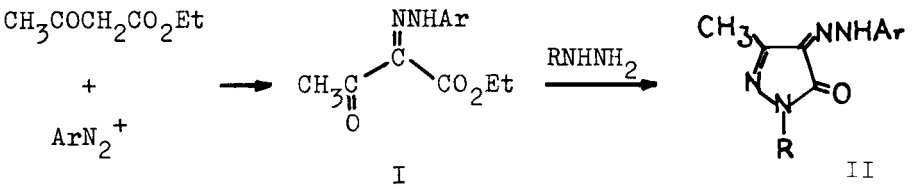
A number of new N-substituted 3-methyl-4-arylhydrazono-2-pyrazolin-5-ones (II) have been prepared by the reaction of ethyl 2,3-dioxobutyrate 2-arylhydrazones (I)[1] with 4-phenylsemicarbazide, benzylsulfonylhydrazine[2] and 4-acetamidobenzenesulfonylhydrazine[3] respectively.



I II

a) R = PhNHCO; b) R = $PhCH_2SO_2$; c) R = 4-(CH_3CONH)$PhSO_2$

Ethyl 2,3-dioxobutyrate 2-arylhydrazones (I) were obtained by coupling the appropriate diazotized aniline with ethyl acetoacetate.[4]

EXPERIMENTAL[5]

N-(Phenylcarbamoyl)-3-methyl-4-arylhydrazono-2-pyrazolin-5-ones (IIa). - An alcoholic solution of 4-phenylsemicarbazide (0.005 mole) containing a few drops of conc. sulfuric acid was added to an alcoholic solution of ethyl 2,3-dioxobutyrate 2-arylhydrazone (0.005 mole) and heated to reflux for 4 hrs. The shiny crystals which separated on cooling were collected and recrystallized from ethanol. The characteristics of the compounds are given in Table I.

N-(Benzylsulfonyl)-3-methyl-4-arylhydrazono-2-pyrazolin-5-ones (IIb). - To a solution of ethyl 2,3-dioxobutyrate 2-arylhydrazone (0.005 mole) in ethanol (40 ml) was added benzenesulfonylhydrazine (0.005 mole)[2] in acetic acid (10 ml) containing a few drops of sulfuric acid. The contents were refluxed for 4 hrs. Upon cooling crystals separated, and were recrystallized from ethanol. Data are given in Table II.

N-(4-Acetamidobenzenesulfonyl)-3-methyl-4-arylhydrazono-2-pyrazolin-5-ones (IIc). - To a solution of ethyl 2,3-dioxo-

butyrate 2-arylhydrazone (0.002 mole) in acetic acid (20 ml), 4-acetamidobenzenesulfonylhydrazine (0.002 mole) in benzene (25 ml) was added. The mixture was refluxed for 4 hrs. Upon cooling shiny crystals separated, were collected and recrystallized from ethanol. The characteristics of these derivatives are listed in Table III.

Table I. N-(Phenylcarbamoyl)-3-methyl-4-arylhydrazono-2-pyrazolin-5-ones (IIa)[a]

Ar	Yield (%)	mp. (°C)	Color
Ph	65	204	Yellow needles
2-NO_2Ph	58	171	Orange needles
4-NO_2Ph	60	197	Dark yellow needles
2-MePh	55	196	Yellow plates
2-MeOPh	40	206	Yellow plates

a) Elemental analyses for nitrogen were within acceptable limits for all compounds.

Table II. N-(Benzylsulfonyl)-3-methyl-4-arylhydrazono-2-pyrazolin-5-ones (IIb)[a]

Ar	Yield (%)	mp. (°C)	Color
Ph	70	194	Orange needles
2-NO_2Ph	63	233	Yellow fibers
3-NO_2Ph	60	126	Yellow fibers
2-MePh	60	186	Yellow needles
2-MeOPh	69	192	Orange needles

a) Elemental analyses for sulfur were within acceptable limits for all compounds.

Table III. N-(4-Acetamidobenzenesulfonyl)-3-methyl-4-aryl-hydrazono-2-pyrazolin-5-ones (IIc)[a]

Ar	Yield (%)	mp. (°C)	Color
Ph	58	257	Yellow plates
2-NO_2Ph	65	250	Yellow plates
3-NO_2Ph	65	248	Yellow plates
2-MePh	60	241	Orange needles
2-MeOPh	65	251	Orange needles

a) See footnote of Table II.

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5. Melting points were taken on a Kofler-hot stage apparatus and are uncorrected.

SYNTHESIS OF OXY AND THIOARYLENE BISNAPHTHALIC ANHYDRIDES

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Bis-1,8-naphthalic anhydride monomers are made by the