

SYNTHESIS OF 3-ARYLFERVENULINS

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Treatment of 6-hydrazino-1,3-dimethyl-5-nitrosouracil with benzyl halides (or phenacyl halides) in dimethyl sulfoxide gave the corresponding 3-arylfervenulins.

3-Arylfervenulins (3-aryl-6,8-dimethylpyrimido[5,4-e]-as-triazine-5,7(6H,8H)-diones) have been prepared by several routes.¹ We wish to report a new synthesis of these derivatives by the reaction of 6-hydrazino-1,3-dimethyl-5-nitrosouracil (I)² with benzyl halides (or phenacyl halides) in dimethyl sulfoxide (DMSO).

Treatment of (I) (0.001 mol) with the benzyl halides (or phenacyl halides) (0.0015 mol) in DMSO (0.2 ml) at 95° for 5 min, followed by dilution with ethanol caused the separation of the corresponding 3-arylfervenulins (IIa-g) (Table).³

As depicted in the Scheme, the formation of (IIa-g) presumably proceeds by the condensation of (I) with aryl aldehydes (or arylglyoxals) which arise from the oxidation of benzyl halides (or phenacyl halides) with DMSO,⁴ followed by cyclization and subsequent aromatization by loss of water (or formic acid).

Scheme

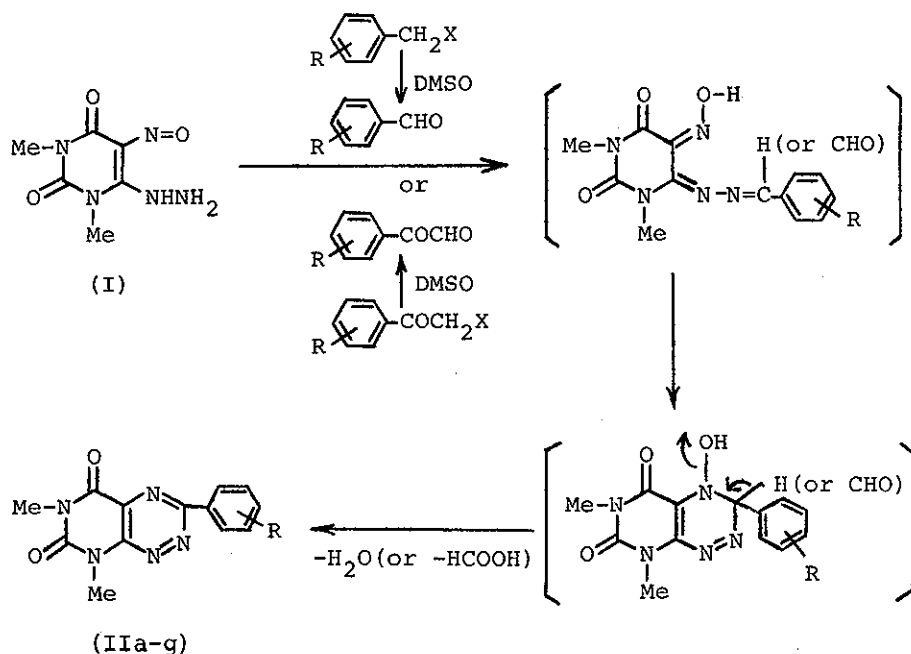


Table 3-Arylfervenulins

Halide	Product	R	Mp (°C) (Lit. ^{1d} Mp) ^a	Yield (%)
C ₆ H ₅ -CH ₂ Br	IIa	H	273-275 (270)	56
C ₆ H ₅ -COCH ₂ Br	IIa	H		30
4-Br-C ₆ H ₄ -COCH ₂ Br	IIb	4-Br	>300 (303)	20
4-Cl-C ₆ H ₄ -CH ₂ Cl	IIc	4-Cl	280-283 (280)	20
4-Cl-C ₆ H ₄ -COCH ₂ Br	IIc	4-Cl		19
4-NO ₂ -C ₆ H ₄ -CH ₂ Br	IIId	4-NO ₂	>300 [*] (323)	47
3,4-Cl ₂ -C ₆ H ₃ -CH ₂ Cl	IIe	3,4-Cl ₂	259-260 (249)	36
4-Me-C ₆ H ₄ -CH ₂ Cl	IIIf	4-Me	286-287	21
4-Me-C ₆ H ₄ -COCH ₂ Br	IIIf	4-Me		11
4-MeO-C ₆ H ₄ -COCH ₂ Br	IIg	4-MeO	263-264 (268)	23

a) All products were recrystallized from ethanol.

REFERENCES AND NOTES

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- 2 W. Pfleiderer and K.-H. Schünkehütte, Annalen, 1958, 615, 42.
- 3 Satisfactory elemental analyses and spectral data were obtained for all products.
- 4 Benzyl halides and phenacyl halides have been reported to undergo oxidation with DMSO to give the corresponding carbonyl compounds: a) N. Kornblum, J.W. Powers, G.J. Anderson, W.J. Jones, H.O. Larson, O. Levand, and W.M. Weaver, J. Am. Chem. Soc., 1957, 79, 6562; b) N. Kornblum, W.J. Jones, and G.J. Anderson, J. Am. Chem. Soc., 1959, 81, 4113.

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