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Synthesis and Structure of 3-Aryl-5-cyano-6-methylthio pyrimidin-2,4-diones

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Abstract: The title compounds of 3-aryl-5-cyano-6-methylthiopyrimidine-2,4-diones were synthesized by the reaction of ethyl 2-cyano-3,3'dimethylthioacrylate with arylureas. The compound (Ar=*p*-$CH_3OC_6H_4$) was recrystallized in acetone/petroleum in space group $P2_{1/n}$ with a=1.1602 (2), b= 1.5921 (3), c=1.3918 (3)nm, β=94.38 (3)° for Z=8, R=0.054.The X-ray analysis showed that there is H-bond interaction among molecules.

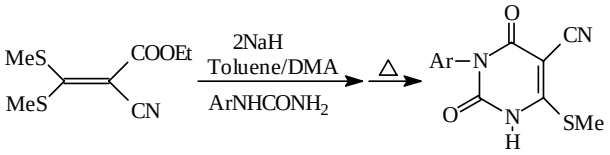
Keywords: Synthesis; crystalline structure.

Polarized ketene dithioacetals **1**, (XY) C=C $(SR)_2$ (X, Y=CN, NO_2, COOEt, $CONH_2$; R=alkyl, benzyl), have been extensively used as building blocks in organic synthesis especially in the preparation of heterocyclic compounds[1, 2].

In an extension of our recent studies[3] in the application of ketoketene N, S-acetals in heterocyclic synthesis, we report herein that the reaction of ethyl 2-cyano-3,3-bis (methylthio) acrylate **1a** with substituted aromatic ureas, under the exitence of sodium hydride and at room temperature, gave the title compound 3-aryl-5-cyano-6-methylthio-pyrimidine-2,4-diones shown in **Scheme 1**.

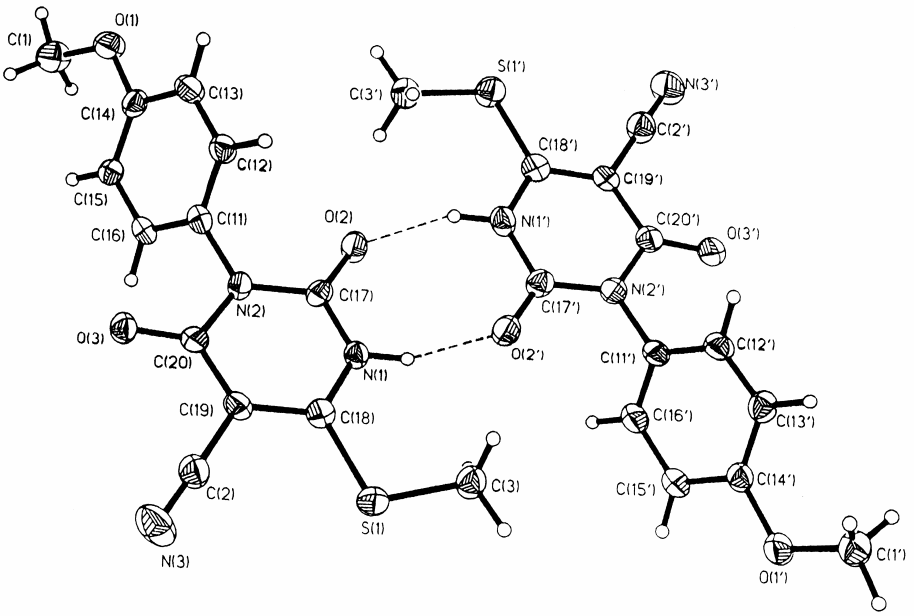
The compound (Ar=*p*-$CH_3OC_6H_4$) was recrystallized in acetone/petroleum, the crystal structure was determined by X-ray. Two molecules of this compound are associated *via* H-bond interaction between amino hydrogen and carbonyl .

Scheme 1. Synthesis of title compounds



Ar: C_6H_5, *p*-ClC_6H_4, *p*-$CH_3OC_6H_4$, *p*-$CH_3C_6H_4$, *o*-$CH_3OC_6H_4$, *o*-$CH_3C_6H_4$, *m*-$CH_3C_6H_4$, *2, 4*- $(CH_3)_2C_6H_3$, *o*-ClC_6H_4, *2,5*- $(CH_3)_2C_6H_3$

Figure 1. Crystal structure of uracil (Ar = $CH_3OC_6H_4$)



General experimental procedure for 3-aryl-5-cyano-6-methylthiopyrimidine-2,4-diones: To a solution of 50 ml of anhydrous toluene and 50 ml anhydrous N, N-dimethylacetamide, 10 mmol of sodium hydride, 5 mmol of ethyl 2-cyano-3,3-dimethylthioacrylate, 5 mmol 4-methoxylphenyl urea was added. The reaction mixture was stirred at room temperature until the reaction was complete monitored by TLC (acetone:petroleum 2:1). Then the mixture was poured into 50 ml of ice-water, the water layer was acidified with 10% hydrogen chloride. The resulting precipitate was collected by filtration under pressure, and was refluxed in 20 ml anhydrous alcohol for 4 hrs. The resulting precipitate was collected by filtration under pressure to give 4.2 mmol of colorless needless, m.p. >300℃, yield 84%. ^{1}H NMR (DMSO-d6, ppm): 2.76 (s, 3H, CH_3S), 7.24~7.47 (m, 5H, C_6H_5), 8.19 (w, 1H, NH).

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