

## The Action of Diphenyl Triketone and of Ninhydrin on Certain Carboxamide Hydrazones

Francis H. Case

Department of Chemistry, Temple University, Philadelphia, Pennsylvania 19122

Received November 1, 1971

The preparation of 3-substituted-5,6-diphenyl- and bis-(2-pyridyl)-*as*-triazines from the action of the diketones, benzil and  $\alpha$ -pyridil on carboxamide hydrazones has previously been described (1,2). Recently the use of the triketones diphenyltriketone and ninhydrin to make related compounds; namely, a number of 3-substituted-5-phenyl-6-benzoyl-1,2,4-triazines and 3-substituted-9*H*-indeno[1,2-*e*]-*as*-triazin-9-ones, has been described by Ried and Schomann (3).

In this laboratory, with the idea of providing new compounds with metal-chelating properties, the above triketones have been caused to undergo a similar reaction with the following compounds: 4-methyl- and 4-phenyl-picolinamide hydrazone, 1,10-phenanthroline-2-carboxamide hydrazone, thiazole-2-carboxamide hydrazone, and 6-(2,2') bipyridine carboxamide hydrazone, all previously described (1,2).

Preliminary tests show that compounds I, III, IV, VI, VIII, and IX give a deep blue color with Fe(II).

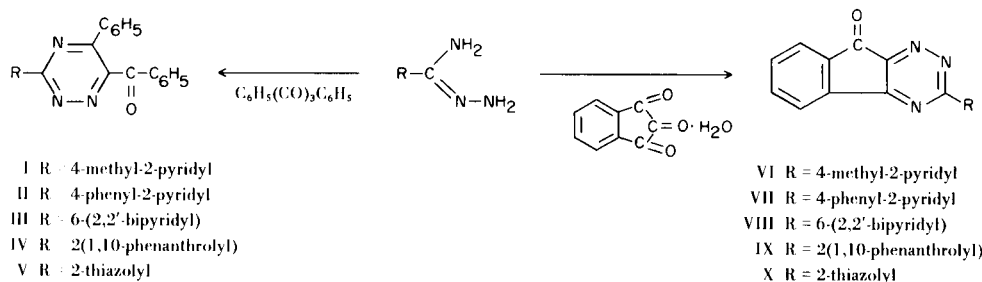
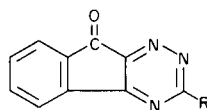


TABLE I

3-Substituted 5-Phenyl-6-benzoyl-1,2,4-triazines

R	Yield %	M.p., °C	Crystallization Solvent	Formula	Analysis						
					Calcd., %			Found, %			
					C	H	N	C	H	N	
I	4-methyl-2-pyridyl	29.2	158-159	ethanol	C <sub>22</sub> H <sub>16</sub> N <sub>4</sub> O	74.98	4.58	15.90	74.77	4.47	15.64
II	4-phenyl-2-pyridyl	45.0	156	ethanol	C <sub>27</sub> H <sub>18</sub> N <sub>4</sub> O	78.24	4.38	13.52	78.41	4.26	13.65
III	6-(2,2'-bipyridyl)	59.8	194	methyl cellosolve	C <sub>26</sub> H <sub>17</sub> N <sub>5</sub> O	75.17	4.12	16.86	74.89	4.30	16.76
IV	2(1,10-phenanthrolyl)	59.5	182	methyl cellosolve	C <sub>28</sub> H <sub>17</sub> N <sub>5</sub> O·H <sub>2</sub> O	73.51	4.19	15.31	73.09	3.83	15.20
V	2-thiazolyl	29.2	200-201	ethanol	C <sub>19</sub> H <sub>12</sub> N <sub>4</sub> OS	66.28	3.51	16.27	66.33	3.57	16.31

TABLE II  
3-Substituted-9*H*-indeno[1,2-*e*]-*as*-triazin-9-ones



	R	Yield %	M.p., °C	Crystallization Solvent	Formula	Analysis					
						Calcd., %		Found, %			
						C	H	N	C	H	N
VI	4-methyl-2-pyridyl	40.7	200	ethanol	C <sub>16</sub> H <sub>10</sub> N <sub>4</sub> O	70.07	3.67	20.43	69.94	3.79	20.63
VII	4-phenyl-2-pyridyl	45.5	229-230	methyl cellosolve	C <sub>21</sub> H <sub>12</sub> N <sub>4</sub> O	74.99	3.60	16.66	74.75	3.75	16.86
VIII	6-(2,2'-bipyridyl)	35.5	256	methyl cellosolve	C <sub>20</sub> H <sub>11</sub> N <sub>5</sub> O	71.21	3.29	20.76	71.06	3.22	20.72
IX	2-(1,10-phenanthrolyl)	52.6	285	dimethyl formamide	C <sub>22</sub> H <sub>11</sub> N <sub>5</sub> O·H <sub>2</sub> O	69.65	3.45	18.46	69.37	3.45	18.92
X	2-thiazolyl	73.7	256-257	methyl cellosolve	C <sub>13</sub> H <sub>6</sub> N <sub>4</sub> OS	58.64	2.26	21.04	58.32	2.49	21.04

#### EXPERIMENTAL

##### Preparation of 3-Substituted 5-Phenyl-6-benzoyl-1,2,4-triazines.

A mixture of 0.005 mole each of the appropriate carboxamide hydrazone (hydrazidine) and diphenyltriketone and 25 ml. of ethanol was heated at reflux for 2.5 hours. After cooling, the resulting precipitate was removed by filtration and crystallized from the solvent indicated in Table I. In the case of compounds I and II, where no precipitate formed, the solvent was first removed by evaporation before crystallization of the residue.

##### Preparation of 3-Substituted 9*H*-Indeno[1,2-*e*]-*as*-triazin-9-ones.

A mixture of 0.005 mole each of the appropriate carboxamide hydrazone and ninhydrin monohydrate and 25 ml. of ethanol was refluxed as above for 2.5 hours. In all cases except that of compound VI the precipitate formed on cooling was removed by filtration and crystallized from the solvent indicated in Table II. Compound VI was obtained by first removing the solvent by evaporation before crystallization.

#### REFERENCES

- (1) F. H. Case, *J. Org. Chem.*, **30**, 931 (1965).
- (2) F. H. Case, *ibid.*, **31**, 2398 (1966).
- (3) W. Ried and P. Schomann, *Ann. Chem.*, **714**, 122 (1968).