270 mg (68%) of XIII with mp 167.5-169°. IR spectrum (KBr pellet): 1540, 1550, 1600, and 3280 cm^{-1}. PMR spectrum (in $CDCl_3$): 2.24 (CH_3, s, 3H), 2.58 (CH_2, unresolved multiplet, 4H), 8.64 (NH, broad s, 1H), and 13.56 ppm (OH, s, 1H). Found: C 54.4; H 5.9; N 9.2%; M 155 (mass spectrometrically). $C_7H_9NO_3$. Calculated: C 54.; H 5.8; N 9.2%; M 155.15.

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RECYCLIZATION REACTIONS OF HETEROCYLES.

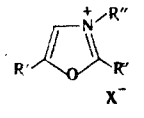
XVII*. HYDRAZINATION OF 1,3,4-OXADIAZOLIUM SALTS

V. I. Fomenko and O. P. Shvaika

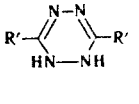
UDC 547.793.4

2,5-Diaryl-3-alkyl-1,3,4-oxadiazolium salts react with hydrazine to give acyclic hydrazinolysis products or products of recyclization with participation of the carbon atom in the 2 position of the oxadiazole ring, i.e., dihydro-sym-tetrazines and N-amino-sym-triazoles (with hydrazine) and 2-phenyl-5-aryl-1,3,4-oxadiazoles (with benzoylhydrazine) or formazans (with phenylhydrazine).

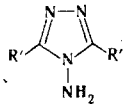
Perfluoroalkyl-, monoaryl-, and amino-1,3,4-oxadiazoles, 1,3,4-oxadiazolones, and oxadiazolethiones react with hydrazines to give acyclic compounds or undergo recyclization to



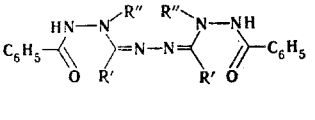
I a-d



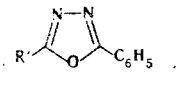
II a-c



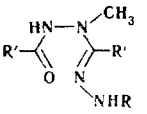
III a-c



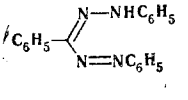
IV a, b



V a, b



VI a, b



VII

I—VI a $R'=C_6H_5$; I—III, V b $R'=p\text{-}CH_3OC_6H_4$; IV b $R'=CH_3$; VI b $R'=C_6H_5$; I—III c $R'=C_{10}H_7$; I d $R'=CH_3$; I, IV a $R''=CH_3$; I b,c $R''=CH_3$; I d IV b $R''=C_6H_5$; VI a R= $=C_6H_5$, b $R=COC_6H_5$; I a-c X=TsO, d $X=ClO_4$

*See [1] for communication XVI.

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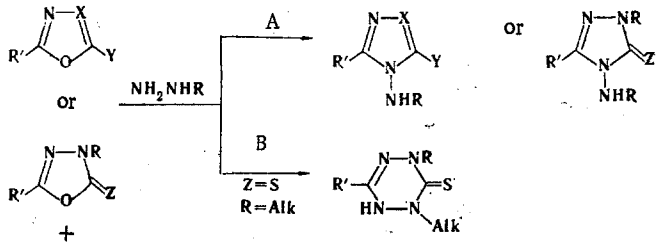
the corresponding N-amino-sym-triazoles (see the review in [2]). Boyd and Summers [3] have shown that oxadiazolium perchlorates can be converted to N-amino-sym-triazolium salts. Under the influence of alkylhydrazines, oxadiazole thiones are also capable of undergoing recyclization to dihydro-sym-tetrazinethiones [2]:



X=N, NR; Y=H, Alk, Ar, NHR; R′=Alk, Ar, hetaryl; R=H, Alk, Ar

All of these recyclizations proceed with participation of the carbon atoms in the 2 and 5 positions of the heteroring and one (pathway A) or both (pathway B) nitrogen atoms of hydrazine.

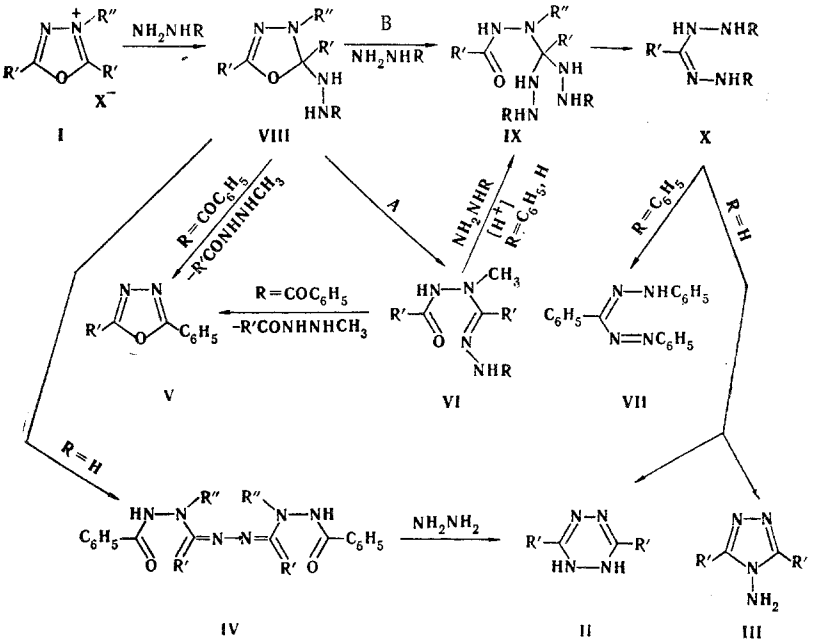
In the present research it was established that 1,3,4-oxadiazolium salts (I) are capable of transformation via a different route, which is delineated below. Oxadiazolium tosylates on reaction with hydrazine are recyclized to dihydro-sym-tetrazines (II) and N-amino-sym-triazoles (III), the ratio of which depends very markedly on the nature of the substituent in the heteroring and on the reaction conditions. Acyclic hydrazone IV is the primary product when insufficient hydrazine is present. Under the influence of benzoylhydrazine, tosylates of the I type are recyclized to 2-phenyl-5-aryl-1,3,4-oxadiazoles (V). An acyclic hydrazinolysis product — hydrazidine VI ($R = C_6H_5CO$) — was also isolated. When salts I are heated in excess phenylhydrazine, they form formazan VII and hydrazidine VI ($R = C_6H_5$); only hydrazidine VI ($R = C_6H_5$) is formed in alcohol.

Acyclic hydrazinolysis products IV and VI may be intermediates in the described transformations. Thus oxadiazole V is obtained when an alcohol solution of hydrazidine VI ($R = C_6H_5CO$) is heated in the presence of perchloric acid, whereas similar treatment of hydrazidine VI ($R = C_6H_5$) in phenylhydrazine gives formazan VII. These transformations do not occur when acid is absent. Prolonged heating of hydrazidine VI ($R = C_6H_5CO$) in piperidine also leads to oxadiazole V, whereas prolonged heating of hydrazone IV with hydrazine in piperidine leads to dihydrotetrazine II and α-methylbenzoylhydrazine. However, these conditions do not completely model the conditions for the transformation of oxadiazolium salts.

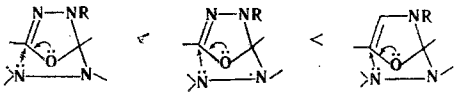
Not one of the above-mentioned recyclization reactions, including the formation of a formazan, is included within the framework of the scheme given above. 2-Phenyl-5-(p-methoxyphenyl)-1,3,4-oxadiazole (V) is obtained by reaction of oxadiazolium salt I ($R' = p\text{-}CH_3OC_6H_4$ $R'' = CH_3$) with benzoylhydrazine, and replacement of the $p\text{-}CH_3OC_4H_6CONNCH_3$ fragment in the starting heterocyclic system by a C_6H_5CONN fragment belonging to the benzoylhydrazine consequently occurs during recyclization. In recyclization via the above scheme one should have expected the formation of the corresponding N-amino-sym-triazolium salts or N-substituted dihydro-sym-tetrazines. However, the N-alkyl substituents present in starting oxadiazolium salts I is absent in tetrazines II and triazoles III, as well as in formazan VII, and this is difficult to explain by transalkylation or hydrazination, inasmuch as an N-alkyl substituent is observed in acyclic hydrazinolysis products IV and VI. In this case also, the hydrazine molecule probably replaces the hydrazine fragment in the ring.

Thus the recyclization reactions under consideration proceed only with participation of the carbon atom in the 2 position of the starting ring, apparently via the scheme on the following page.

Nucleophilic attack in azolium salts, including oxadiazolium salts, takes place at the 2 position of the heteroring [4-8]. In contrast to what occurs in oxazole or thiadiazole systems [1,4], in which dihydrocyclazines are obtained in the same manner, subsequent addition of the β-nitrogen atom of the hydrazine group to the 5 position in intermediate VIII (see the scheme on the following page) does not occur. In this respect there is a substantial difference in the cyclization of 1,3,4-oxadiazolium salts and related compounds — 1,3,4-thia-



diazolium and oxazolium salts — under the influence of hydrazines. This difference may be associated with the fact that the electrophilicity of the carbon atom in the 5 position of the azoline ring increases with the capacity of the oxygen and sulfur atoms to increase their covalent character in such systems, in conformity with Ingold's rules [9], in the following order:



Hence the probability of transannular addition of hydrazine, which leads to cyclazines, is at minimum in the oxadiazoline ring. Ring opening to give hydrazine VI therefore evidently predominates in the latter case (see the scheme above, pathway A), although the possibility of reaction of another molecule of hydrazine at the 2 position of oxadiazoline VIII (pathway B) is not excluded. In its turn, hydrazidine VI, as demonstrated by model experiments, is also capable of reacting with another molecule of hydrazine. The resulting addition product (IX) is converted to X. The latter, when R = H, is converted to dihydrotetrazine II or N-aminotriazole III via the Pinner reaction [10] or, when R = C_6H_5, is apparently oxidized by another molecule of phenylhydrazine to formazan VII. However, the formation of oxadiazoles V is associated with intramolecular cyclization of intermediate VIII (R = C_6H_5CO) or VI (R = C_6H_5CO) in the presence of an acid catalyst with the participation of the oxo atom of the hydrazide fragment.

EXPERIMENTAL

3,6-Diphenyl-1,2-dihydro-1,2,4,5-tetrazine (II, R' = C_6H_5). A) A 5-ml (100 mmole) sample of hydrazine hydrate was added to 4 g (10 mmole) of oxadiazolium tosylate I (R' = C_6H_5, R'' = CH_3, X = TsO), obtained by fusion of 2.2 g (10 mmole) of 2,5-diphenyl-1,3,4-oxadiazole with 1.86 g (10 mmole) of methyl p-toluenesulfonate at 180-190° for 10-15 min. The mixture was refluxed for 10-15 min, after which it was cooled, and water was added. The resulting yellow-orange precipitate was removed by filtration, washed with water, and vacuum dried at 50-60° to give 2.8 g of an orange crystalline substance, which was treated with chloroform and filtered to give 0.2 g (4.5%) of white crystals of triazole III with mp 256-258° (from ethanol; mp 258° [10]). No melting-point depression was observed for a mixture of this product with a sample obtained by the method in [10], and the benzylidene derivative was identical to a sample obtained by the method in [11]. The chloroform extract was vacuum evaporated to give 2.6 g (61%) of yellow-orange crystals of dihydrotetrazine II with mp 191-192° (from ethanol; mp 192° [12]). No melting-point depression was observed for a mixture of this product with a sample synthesized by refluxing benzonitrile, sublimed sulfur, and hydrazine hydrate in ethanol [12].

B) A mixture of 1 g (2 mmole) of hydrazone IV ($R' = C_6H_5$, $R'' = CH_3$), 0.6 ml of absolute hydrazine, and 5 ml of morpholine was refluxed for 1 h, after which it was cooled, and water was added. The solid material was removed by filtration, washed with water, and vacuum dried at 60°. The yield of product with mp 191-192° (from ethanol) was quantitative. No melting-point depression was observed for a mixture of this product with the product obtained in the preceding experiment. Evaporation of the aqueous mother liquor in vacuo gave 0.25 g (95%) of 1-methyl-2-benzoylhydrazine with mp 86-87° [from petroleum ether—chloroform (4:1); mp 87° [13]. No melting-point depression was observed for a mixture of this product with a sample obtained by the method in [13].

Dihydrotetrazine II was obtained in 89% yield by refluxing hydrazone IV ($R' = C_6H_5$, $R'' = CH_3$) in absolute hydrazine for 1.5 h.

3,6-β-Dinaphthyl-1,2-dihydro-1,2,4,5-tetrazine (II, $R' = \beta-C_{10}H_7$). This compound was obtained as described in experiment A from oxadiazolium tosylate I ($R' = \beta-C_{10}H_7$ $R'' = CH_3$) (4 mmole) and 100 mmole of hydrazine hydrate. The yield of orange crystals with mp 242-244° (from glacial acetic acid; mp 244° [10]) was 90%. No melting-point depression was observed for mixture of this product with a sample obtained by refluxing β-naphthylthioamide and 50% hydrazine in ethanol for 30 min [10, 14].

3,6-Di(p-methoxyphenyl)-1,2-dihydro-1,2,4,5-tetrazine (II, $R' = p\text{-}H_3COC_6H_4$). A 3.2-ml sample of anhydrous hydrazine was added to hot fused 2,5-di(p-methoxyphenyl-3-methyl-1,3,4-oxadiazolium tosylate obtained by fusing 2.82 g (10 mmole) of 2,5-(p-methoxyphenyl)-1,3,4-oxadiazole with an equimolar amount of methyl tosylate. A homogenous solution formed initially, and a precipitate formed after 3-5 min. The mixture was heated for 10 min, after which it was cooled, and water was added. The resulting precipitate was removed by filtration, washed with water, and vacuum dried at 50°. The product (0.9 g) was extracted with hot ethanol. The residue was worked up to give 0.6 g (20%) of yeloww crystals of dihydrotetrazine II with mp 241-242° (from butanol); the product was soluble in hexanol and benzene, slightly soluble in ethanol, and insoluble in ether and water. Found: C 64.8; H 5.6; N 19.0%. $C_{16}H_{16}N_4O_2$. Calculated: C 64.9; H 5.4; N 18.9%.

Oxidation of dihydrotetrazine II ($R' = p\text{-}H_3COC_6H_4$) as a suspension in ethanol with 10% aqueous ferric chloride solution at room temperature for 30 min gave red crystals of 3,6-di(p-methoxyphenyl)-1,2,4,5-tetrazine with mp 242-244° (from acetone). Found: C 65.2; H 5.0; N 18.9%. $C_{16}H_{14}N_4O_2$. Calculated: C 65.4; H 4.8; N 19.1%.

3,5-Diphenyl-4-amino-1,2,4-triazole (III, $R' = C_6H_5$). A 4-g (10 mmole) sample of oxadiazolium tosylate I ($R' = C_6H_5$, $R'' = CH_3$), obtained in the form of a melt, was dissolved in ethanol, 1 ml (20 mmole) of hydrazine hydrate was added, and the mixture was heated for 40 min. After 10-15 min, a colorless crystalline material precipitated. The mixture was cooled, water was added, and the resulting precipitate was removed by filtration, washed with water, and dried. The product (1.5 g) was extracted with chloroform. The chloroform was evaporated, and the residue was worked up to give 1.4 g (61%) of colorless cyrstals of triazole III with mp 256-258° (from ethanol 258° [12]). The N-benzylidene derivative of triazole III was obtained by refluxing triazole III with benzaldehyde in acetic acid in the presence of sodium acetate; workup gave white crystals with mp 207-208° (from ethanol; mp 208° [11]). Evaporation of the chloroform extract gave 0.1 g of yellow-orange crystals of dihydrotetrazine II ($R' = C_6H_5$) with mp 191-192°.

2,5-Di(β-naphthyl)-4-amino-1,2,4-triazole (III $R' = \beta-C_{10}H_7$). The procedure in the preceding experiment was used to obtain this compound from oxadiazolium tosylate I ($R' = \beta-C_{10}H_7$, $R'' = CH_3$) (10 mmole) used in the form of the melt, and hydrazine (20 mmole). The product, with mp 319-321° (from acetic acid), was obtained in 60% yield. Found: C 78.7; H 5.3; N 16.7%. $C_{22}H_{16}N_4$. Calculated: C 87.6; H 4.8; N 16.7%.

3,5-Di(p-methoxyphenyl)-4-amino-1,2,4-triazole (III, $R' = C_6H_4OCH_3\text{-}p$). A 4.7-g (10 mmole) sample of tosylate I ($R' = C_6H_4OCH_3\text{-}p$, $R'' = CH_3$), obtained in the form of a melt as in the case of tosylate I ($R' = C_6H_5$, $R'' = CH_3$), was dissolved in absolute alcohol, and 0.6 ml (20 mmole) of absolute hydrazine was added. The mixture was allowed to stand at room temperature for 30 min, after which water was added. The oily product cyrstallized after standing for 24 h. Workup gave 1.45 g (50%) of colorless cyrstals with mp 271-272° (from dichloroethane). Found: C 64.8; H 5.6; N 18.7%. $C_{16}H_{16}N_4O_2$. Calculated: C 64.8; H 5.4; N 18.9%. The N-benzylidene derivative had mp 208-209° [from ethanol—petroleum ether (1:3)]. Found: C 72.3; H 5.3; N 14.9%. $C_{23}H_{20}N_4O_2$. Calculated: C 71.9; H 5.2; N 14.6%.

1,4,7,10-Tetraphenyl-3,8-dimethyl-2,3,5,6,8,9-hexaaza-4,6-decadiene (IV, R' = C_6H_5, R" = CH_3). A 5-ml sample of hydrazine hydrate was added to a solution of 4.1 g (10 mmole) of oxadiazolium tosylate (I, R' = C_6H_5, R" = CH_3) in 30 ml of ethanol. A crystalline precipitate formed. The mixture was allowed to stand for 30-40 min, after which water was added, and the precipitate was removed by filtration, washed with water, and vacuum dried at 60° to give 1.4 g of product. This product was extracted with benzene, and the residue was worked up to give 1.1 g (21%) of white crystals of IV with mp 258-260° [from ethanol—benzene (1:2)]. Compound IV was soluble in alcohol and chloroform, less soluble in benzene, and insoluble in ether and petroleum ether. Found: C 71.5; H 5.9; N 16.6%. $C_{30}H_{28}N_6O_2$. Calculated: C 71.4; H 5.6; N 16.7%.

Evaporation of the benzene extract gave 0.3 g (2.4%) of triazole III (R = C_6H_5) with mp 256-258°; no melting-point depression was observed for a mixture of this product with the sample obtained in the experiment described above.

1,3,8,10-Tetraphenyl-4,7-dimethyl-2,3,5,6,8,9-hexaaza-4,6-decadiene (IV, R" = C_6H_5, R' = CH_3). As in the preparation of IV (R' = C_6H_5, R" = CH_3), this compound was obtained by the action of 0.5 ml (10 mmole) of hydrazine hydrate on a suspension of 1.68 g (5 mmole) of 2-methyl-3,5)diphenyl-1,3,4-oxadiazolium perchlorate in 3 ml of ethanol at −1°. The yield of colorless crystals with mp 268-270° (from ethanol) was 32%. The product was soluble in ethanol, dioxane, and dichloroethane but insoluble in water and ether. Found: C 71.7; H 5.7; N 16.7%. $C_{30}H_{28}N_6O_2$. Calculated: C 71.4; H 5.6; N 16.7%.

Action of Phenylhydrazine on 2,5-Diphenyl-3-methyl-1,3,4-oxadiazolium Salts. A) An 11-ml sample of freshly distilled phenylhydrazine was added to 7.8 g (19 mmole) of an oxadiazolium tosylate I (R' = C_6H_5, R" = CH_3) melt obtained as described above, and the mixture was heated at 80-100° for 2-3 min until a vigorous reaction, which was regulated, where necessary, by cooling, began. After 5 min, the viscous dark- red reaction mass was treated with water, and the solid material was removed by filtration, washed with water and vacuum dried at 60°. The product (5 g) was extracted with ether, and the ether extract was evaporated to give dark-red crystals of triphenylformazan VII with mp 172-174° (from ethanol; mp 174-175° [15, 16]) in 30% yield. A dihydrotetrazine ion structure was previously [16] incorrectly assigned to VII obtained via this reaction. No melting-point depression was observed for a mixture of this product with a sample of triphenylformazan obtained by coupling of benzaldehyde phenylhydrazine with a benzenediazonium salt [17].

The insoluble residue remaining after ether extraction was worked up give 1,3,6-triphenyl-4-methyl-1,2,4,5-tetrazahexaen-2-one (VI, R = R' = C_6H_5) with mp 172-174° (from ethanol) in 60% yield. The white crystals were soluble in ethanol and acetone, less soluble in benzene, and insoluble in water and ether. Found: C 73.6; H 6.0; N 16.2%. $C_{21}H_{20}N_4O$. Calculated: C 73.3; H 5.8; N 16.3%. No melting-point depression was observed for a mixture of this product and the product obtained by heating 5 mmole of 2,5-diphenyl-3-methyl-1,3,4-oxadiazolium tosylate with 10 mmole of phenylhydrazine in alcohol for 10-15 min (the yield of product with mp 173° was quantitative). The perchlorate of VI (R = R' = C_6H_5) was obtained by the addition of 70% perchloric acid to dioxane solution of VI; the white crystals of the perchlorate, with mp 175-176° (from ethanol), were soluble in acetone, nitromethane, alcohol, and dioxane but insoluble in water, ether, and benzene. Found: C 56.2; H 5.1; Cl 8.0%. $C_{21}H_{21}ClN_4O_5$. Calculated: C 56.6; H 4.7; Cl 8.1%.

B) A 0.7-g (2 mmole) sample of 2,5-diphenyl-3-methyl-1,3,4-oxadiazolium perchlorate was added to 2 ml of phenylhydrazine heated to 100°, and the mixture was heated at this temperature for 5 min, after which water was added. The resulting precipitate was extracted with ether, and the extract was evaporated to give formazan VII with mp 172-173° (from ethanol) in 31% yield. Hydrazidine VI (R = R' = C_6H_5), with mp 172-174° (from ethanol), was obtained in 45% yield. Both products were identified by mixed-melting-point determinations with the appropriate samples obtained in the preceding experiment.

Reaction of Hydrazidine VI (R = R' = C_6H_5) with Phenylhydrazine. A 0.62-g (3 mmole) sample of phenylhydrazidinium perchlorate was added to a suspension of 1 g (3 mmole) of hydrazine VI in 3.5 ml of phenylhydrazine, and the mixture was heated at 100° for 15 min, during which a red solution formed. The solution was cooled, water was added, and the resulting oily product crystallized after trituration. The solid material was removed by filtration, washed with water, vacuum dried at 60°, and extracted with ether. The residue was worked up

to give 0.42 g (42%) of a colorless crystalline substance with mp 173° (from ethanol), which, according to a mixed-melting-point determination, was identical to the starting hydrazidine. Evaporation of the ether extract gave 0.3 g (35%) of dark-red crystals of triphenylformazan VII with mp 172° (from ethanol); the product was identified by a mixed-melting-point determination with a sample synthesized in the experiment described above.

Action of Benzoylhydrazine on Oxadiazolium Salts I. Triethylamine (10 mmole) and 20 mmole of benzoylhydrazine were added to a solution of 10 mmole of oxadiazolium tosylate I obtained as described above. A) In the case of oxadiazolium tosylate I, a homogeneous solution was formed initially, and a white crystalline precipitate formed after 15 min. Water was then added, and the precipitate was removed by filtration, washed with water, vacuum dried at 60°, and crystallized from ethanol. Dibenzoylbenzhydrazidine VI ($R = C_6H_5CO$, $R' = C_6H_5$) was obtained as white crystals with mp 234-235° (from ethanol) in 30% yield. Found: C 70.5; H 5.1; N 15.3%. $C_{22}H_{20}N_4O_2$. Calculated: C 70.9; H 5.4; N 15.1%. Water was added to the alcohol liquor, and the resulting precipitate was removed by filtration and dried to give white crystals of 2,5-diphenyl-1,3,4-oxadiazole (40%) with mp 136-138° (from alcohol; mp 138° [18]). The product was identified by mixed-melting-point determination with a sample obtained by the method in [18].

B) When oxadiazolium tosylate I ($R' = p\text{-}H_3COC_6H_4$, $R'' = CH_3$) was used in the reaction, the precipitate that formed after the reaction mixture had stood at room temperature for 36 h was removed by filtration, washed with 60% ethanol, and dried to give white crystals of 2-(p-methoxyphenyl)-5-phenyl-1,3,4-oxadiazole (50%) with mp 145-147° (from ethanol; mp 146, [19]). The product was identified by a mixed-melting-point determination with a sample obtained by the method in [19].

2,5-Diphenyl-1,3,4-oxadiazole (mp 138°) was also formed in 70% yield by refluxing an alcohol solution of hydrazidine VI ($R' = C_6H_5$, $R = C_6H_5CO$) for 30 min in the presence of benzoylhydrazinium perchlorate and also (in 99% yield; mp 138°) by refluxing hydrazidine VI ($R' = C_6H_5$, $R = C_6H_5CO$) for 2 h, but hydrazidine VI was recovered completely unchanged after refluxing in benzene for 5 h.

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