Ring-Opening Reaction of 1,3,4-Oxadiazolone and 1,3,4-Oxadiazolinethione: Reaction of 2-Phenyl-1,3,4-oxadiazolin-5-one and 2-Phenyl-1,3,4-oxadiazoline-5-thione with Amines

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The ring-opening abilities of amines toward 1,3,4-oxadiazolines, 2-phenyl-1,3,4-oxadiazolin-5-one (**1a**) and 2-phenyl-1,3,4-oxadiazoline-5-thione (**1b**), were investigated with relation to their basicities or pK_b values. Oxadiazolines **1a** and **1b** were easily reacted with amines such as benzylamine and aniline, but not with *p*-nitroaniline, to form the corresponding ring-opening adducts. The reactions of both **1a** and **1b** with *o*-phenylenediamine produced benzodiazoles with the liberation of benzoylhydrazide, whereas the reactions with *o*-aminobenzamide furnished quinazolines with the liberation of ammonia. *o*-Aminophenol and *o*-aminothiophenol were also reacted with **1a** and **1b** both of them giving 1,5-dibenzoylcarbohydrazide from **1a** and 1,2-dibenzoylhydrazine from **1b**. From the conditions affording the corresponding ring-opening adducts or reaction products, the ring-opening abilities of the amines toward **1a** and **1b** are in good correlation with the strength of their basicities or pK_b values. The ring-opening of oxadiazolines were proved to occur with anilines. Therefore, the other reactions are also supposed to proceed *via* the ring-opening steps.

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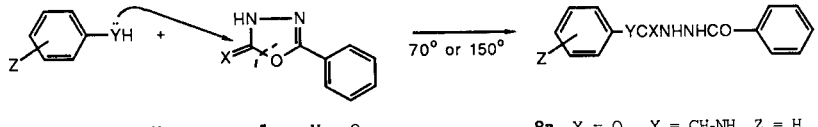
2-Substituted-1,3,4-oxadiazolin-5-ones and 2-substituted-1,3,4-oxadiazoline-5-thiones are of considerable interest for their preparation, chemistry and pharmacological properties [1-37]. We have recently demonstrated that the reaction of 2-aryl-1,3,4-oxadiazolin-5-ones and 2-aryl-1,3,4-oxadiazoline-5-thiones with methyl anthranilate gave novel quinazoline derivatives in one step [38]; the heterocycle formation presumably proceeds through the ring-opening of the oxadiazolines. It has also been reported that the ring-opening reactions of 1,3,4-oxadiazolones and 1,3,4-oxadiazolinethiones occur with water [39], alcohols [39], ammonia [40], amines [39-42] and hydrazines [39,43]. However, no systematic study has been undertaken on the ring-opening abilities of nucleophiles toward the oxadiazolines. In the present report we have investigated in detail the reaction of 2-phenyl-1,3,4-oxadiazolin-5-one (**1a**) [38] and 2-phenyl-1,3,4-oxadiazoline-5-thione (**1b**) [38] with a variety of amines.

With the aim of establishing the correlation between the ring-opening ability and the basicity or pK_b value of amines, the reactions of **1a** and **1b** with benzylamine (**2**) and substituted anilines **3-5** with different pK_b values were carried out in *m*-cresol at 70° for 24 or 48 hours. As shown in Table 1, they were also conducted using acidic nucleophiles, phenol (**6**) and thiophenol (**7**).

In general, it is expected that these reactions result in good yields of the corresponding ring-opening adducts, however, they proceeded with increasing difficulty in the order of nucleophiles **2**, **3** and **4**, and no reaction occurred with *p*-nitroaniline **5** having the largest pK_b value either with **1a** or **1b** under the same reaction conditions. When a mixture of **1a** or **1b** and **6** as well as that of **1b** and **7** were heated in *m*-cresol under more severe conditions, only starting materials could be recovered almost quantitatively. These results indicate that the ring-opening abilities of amines toward 1,3,4-oxadiazolones and 1,3,4-oxadiazoline-

Scheme I



2, Y = CH_2NH, Z = H
3, Y = NH, Z = H
4, Y = NH, Z = *o*-Cl
5, Y = NH, Z = *p*-NO_2
6, Y = O, Z = H
7, Y = S, Z = H

1a, X = O
1b, X = S

8a, X = O, Y = CH_2NH, Z = H
8b, X = S, Y = CH_2NH, Z = H
9a, X = O, Y = NH, Z = H
9b, X = S, Y = NH, Z = H
10a, X = O, Y = NH, Z = *p*-Cl
10b, X = S, Y = NH, Z = *p*-Cl
11a, X = O, Y = S, Z = H

Table 1

Ring-Opening Reaction of Oxadiazolines **1a**, **1b** with Nucleophiles **2-7**

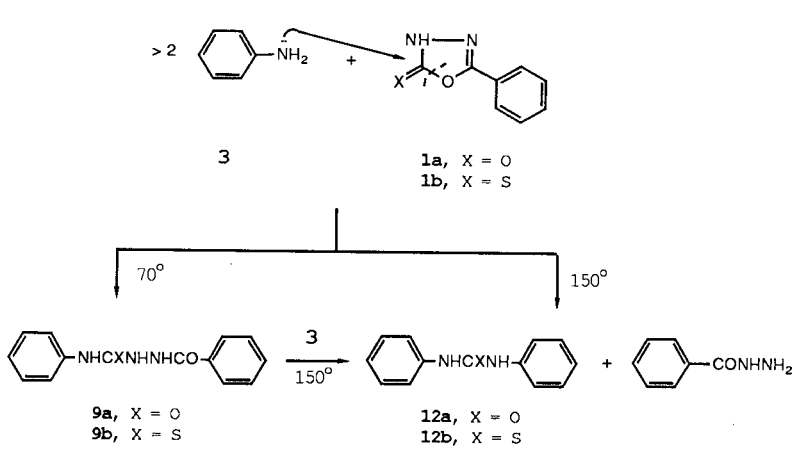
Oxadiazoline	Nucleophile (pK_b) [a]	Reaction temperature °C	Reaction time hours	Product	Yield %	Mp [b] °C	Formula	Analysis % Calcd./Found C	H	N
1a	**2** (4.65)	70	24	**8a**	96	177-179	$C_{15}H_{15}N_3O_2$	66.90	5.61	15.61
								66.98	5.40	15.63
1a	**3** (9.40)	70	48	**9a**	98	209-210 [d]	$C_{14}H_{13}N_3O_2$	65.87	5.13	16.46
								65.94	5.17	16.41
1a	**4** (10.36)	70	48	**10a**	36	203-204	$C_{14}H_{12}ClN_3O_2$	58.04	4.18	14.51
								57.87	4.34	14.63
1a	**5** (13.01)	70	48	[c]	0	---	---	---	---	---
1a	**6**	150	72	[c]	0	---	---	---	---	---
1a	**7**	150	72	**11a**	30	239-241	$C_{14}H_{12}N_2O_2S$	61.74	4.44	10.29
								61.81	4.60	10.20
1b	**2** (4.65)	70	24	**8b**	92	159-160	$C_{15}H_{15}N_3OS$	63.13	5.30	14.73
								63.10	5.10	14.57
1b	**3** (9.40)	70	48	**9b**	92	165-166 [e]	$C_{14}H_{13}N_3OS$	61.97	4.83	15.49
								62.09	4.81	15.69
1b	**4** (10.36)	70	48	**10b**	37	162-163	$C_{14}H_{12}ClN_3OS$	54.99	3.96	13.75
								55.04	3.83	13.70
1b	**5** (13.01)	70	48	[c]	0	---	---	---	---	---
1b	**6**	150	72	[c]	0	---	---	---	---	---
1b	**7**	150	72	[c]	0	---	---	---	---	---

[a] Lit [51]. [b] All compounds were recrystallized from water. [c] Starting materials could be recovered almost quantitatively. [d] Lit [44] gives mp 212-214°. [e] Lit [45] gives mp 164-166°.

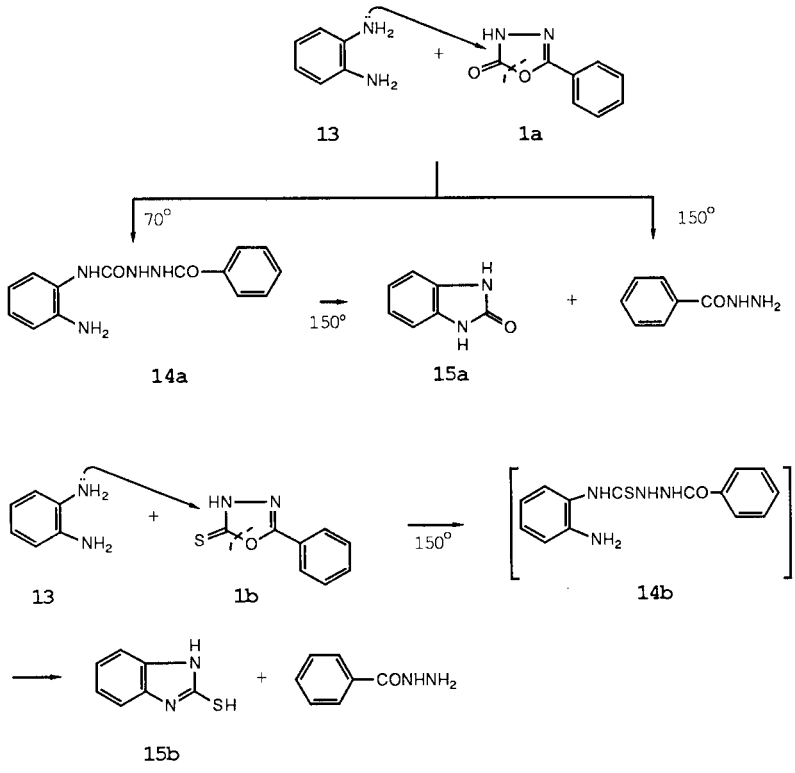
thiones decrease with increasing pK_b value, and that incidentally **1b** is somewhat less reactive toward nucleophiles than **1a**. Ring-opening adducts, 1-benzoyl-4-phenylsemicarbazide (**9a**) [44] and 1-benzoyl-4-phenylthiosemicarbazide (**9b**) [45], were also obtained in high yields from **1a** and **3**, and from **1b** and **3**, respectively, in polar aprotic media such as *N,N*-dimethylacetamide, *N,N*-dimethylformamide, dimethyl sulfoxide and *N*-methyl-2-pyrrolidone.

The treatment of **1a** and **1b** with a large excess of **3** at 70° for 24 hours also afforded quantitative yields of **9a** and **9b**, respectively. At 150°, however, the respective reactions led to the formation of diphenylurea (**12a**) [46] from **1a** and diphenylthiourea (**12b**) [47] from **1b** in a relatively short time. The formation of **12a** and **12b** undoubtedly proceeds through nucleophilic attack of the amino nitrogen of unreacted **3** on the (thio)carbonyl C-3 of ring-opening adducts **9a** and **9b** initially formed with the liberation of benzoylhydrazide. This is firmly supported by the recovery of benzoylhydrazide in high yields from the respective reaction mixtures. As anticipated, both **9a** and **9b** were reacted readily with **3** in *m*-cresol at 150° for 1 hour to produce **12a** and benzoylhydrazide from **9a** and also **12b** and benzoylhydrazide from **9b**, respectively.

Scheme II



Scheme III

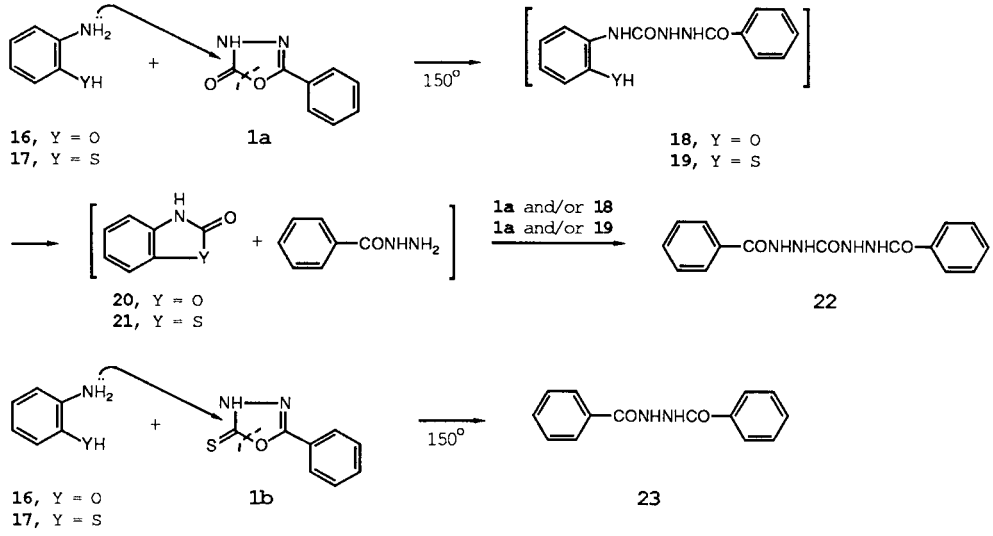


Oxadiazolone **1a** also underwent ring-opening reaction upon treating with *o*-phenylenediamine (**13**) in *m*-cresol at 70° for 48 hours to furnish excellent yield of 1-benzoyl-4-(*o*-aminophenyl)semicarbazide (**14a**), whereas **1b** scarcely reacted with **13** under the same reaction conditions. Ring-opening adduct **14a** was easily cyclized to 1,3-benzodiazol-2-one (**15a**) [48] with the elimination of benzoylhydrazide by heating in *m*-cresol at 150° for 5 hours. Heterocyclic compounds **15a** and 2-mercapto-1,3-benzodiazole (tautomer of 1,3-benzodiazole-2-thione initially formed) (**15b**) [49], however, were both obtained directly in good yields by treating **1a** and **1b** with **13**, respectively, in *m*-cresol at 150° for 5 hours. The formation of **14a** supports the formation of analogous intermediate 1-benzoyl-4-(*o*-aminophenyl)thiosemicarbazide (**14b**) in the reaction sequence of **1b** and **13** leading to **15b** and also points to **14b** as an unstable intermediate compared to **14a**, which is isolated even at relatively high temperature.

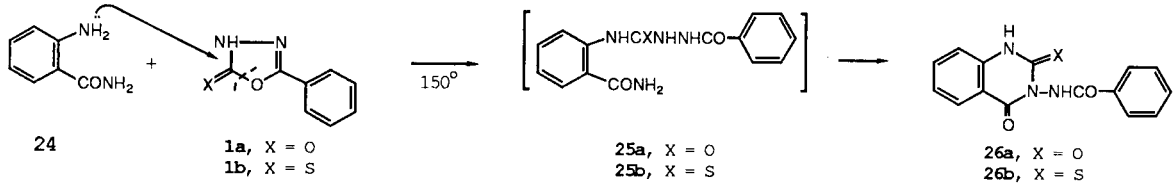
The treatment of *o*-aminophenol (**16**) and *o*-aminothiophenol (**17**) with **1a** or **1b** in *m*-cresol at 150° for 5 hours yielded 1,5-dibenzoylcarbohydrazide (**22**) [39] from **1a** and 1,2-dibenzoylhydrazine (**23**) [50] from **1b**, respectively, as exclusive products. The former reactions giving **22** take place in the following manner: (1) the ring-opening reactions of **16** and **17** with **1a** to afford 1-benzoyl-4-(*o*-hydroxyphenyl)semicarbazide (**18**) and 1-benzoyl-4-(*o*-mercaptophenyl)semicarbazide (**19**), respectively, (2) losses of 1,3-benzoxazol-2-one (**20**) from **18** and of 1,3-benzothiazol-2-one (**21**) from **19** by nucleophilic attack of the intramolecular hydroxy oxygen or mercapto sulfur on the carbonyl C-3 of **18** and **19** to produce benzoylhydrazide, and (3) the latter reacts with unreacted **1a** and/or **18** and with unreacted **1a** and/or **19**, respectively, to furnish the same final product **22**. All attempts to isolate intermediates **18** and **19**, however, were unsuccessful. The route of the reaction leading to **23** remain unclear.

The reactions of **1a** and **1b** with *o*-aminobenzamide (**24**) in *m*-cresol at 150° for 24 hours resulted in reasonable yields of *N*-(2,4-dioxo-1,2,3,4-tetrahydroquinazolinyl)benzamide (**26a**) [38] and *N*-(2-thiono-4-oxo-1,2,3,4-tetrahydroquinazolinyl)benzamide (**26b**) [38], respectively. The formation of **26a** and **26b** presumably proceeds through nucleophilic attack of the *o*-amino nitrogen of **24** on the (thio)carbonyl C-5 of **1a** and **1b** to give ring-opening adducts, 1-benzoyl-4-(*o*-carbamoylphenyl)semicarbazide (**25a**) and 1-benzoyl-4-(*o*-carbamoylphenyl)thiosemicarbazide (**25b**), respectively. Subsequent ring closure accompanied with the elimination of ammonia completes the quinazoline structure. Intermediates **25a** and **25b** were attempted to be isolated by shortening the reaction time or by lowering the reaction temperature. However, trace amounts of **26a** and **26b** or only starting materials were

Scheme IV

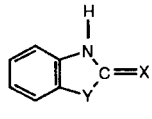


Scheme V



recovered. Therefore, the ring-opening of **1a** and **1b** by **24** is the rate-determining step in the proposed sequence of reaction.

In conclusion, in a series of nucleophiles used, their ring-opening abilities toward oxadiazolines **1a** and **1b** decrease in the following order: **2** > **3** > **13** ≫ **4** ≫ **16**, **17** > **24** ≫ **7**. This order is in well accord with that of the basicities or pK_b values of the nucleophiles. The reactions of 1,3,4-oxadiazolones and 1,3,4-oxadiazolinethiones with *o*-substituted anilines are very promising as a novel system to prepare a variety of heterocyclic compounds possessing the following ring structures:



where X is O and S and Y is, for example, NH, O, S and CONNHCOAr.

EXPERIMENTAL

Melting points were determined using an electrothermal melting point apparatus without correction. The ir spectra were recorded in potassium bromide on a JASCO IR-810 spectrophotometer. The oxadiazolines 2-phenyl-1,3,4-oxadiazolin-5-one (**1a**) and 2-phenyl-1,3,4-oxadiazoline-5-thione (**1b**) were prepared as reported previously [38]. All the nucleophiles and the solvents were obtained commercially and were purified by conventional procedures prior to use. The products obtained were identified by mp, mixed mp and spectral comparison with the corresponding authentic samples unless otherwise noted.

Reactions of **1a** with Nucleophiles **2-7**.

A mixture of **1a** (5 mmoles) and **2-7** (5 mmoles) in a solvent (5 ml) was heated with stirring for a predetermined duration and then allowed to cool to room temperature. Dilution with ether (*ca.* 30 ml) caused separation of a precipitate which was filtered off, rinsed thoroughly with ether and dried.

The reaction conditions and the results are summarized in Table 1.

Reactions of **1b** with Nucleophiles **2-7**.

To a stirred solution of **2-7** (5 mmoles) in a solvent (5 ml) **1b** (5 mmoles) was added. After heated for a prescribed period, the solution was stripped under reduced pressure and the residue was treated with dilute aqueous sodium hydroxide. The solid, so formed, was collected, washed successively with dilute aqueous sodium hydroxide and with water, and then dried.

The reaction conditions and the results are compiled in Table 1.

Reactions of **1a** and **1b** with Excess Aniline (**3**).

A suspension of **1a** or **1b** (5 mmoles) in **3** (10 ml) was stirred at 150° for 2 hours. Removal of excess **3** provided a solid material which was triturated with a small amount of water. The product thus obtained was filtered off, washed with a minimum quantity of water and dried.

Compound **1a** gave a 93% yield of **12a**, mp 240-241° (water) [lit [46], mp 239-240°]; ir: 3340, 3290 (NH), 1650 cm^{-1} (C=O).

Compound **1b** furnished **12b** in 93% yield, mp 154-155° (water) [lit [47], mp 153-154°]; ir: 3200 (NH), 1330 cm^{-1} (C=S).

Evaporation of each of the combined filtrate and washings both afforded benzoylhydrazide in 86% and 83% yields.

The respective reactions carried out at 70° for 24 hours produced a 98% yield of **9a** from **1a** and a 92% yield of **9b** from **1b**.

Reactions of **9a** and **9b** with Aniline **3**.

A solution of **9a** or **9b** (5 mmoles) and **3** (5 mmoles) in *m*-cresol (5 ml) was reacted with stirring at 150° for 1 hour. The solvent was evaporated to dryness under reduced pressure and the residue was triturated with a small quantity of water to yield a solid which was gathered, washed with a small amount of water and dried.

Compound **9a** provided a 96% yield of **12a**.

Compound **9b** gave **12b** in 93% yield.

Each of the combined filtrate and washings was evaporated *in vacuo* both affording benzoylhydrazide in 94% and 89% yields.

Reactions of **1a** and **1b** with *o*-Phenylenediamine (**13**).

A stirred mixture of **1a** or **1b** (5 mmoles) and **13** (5 mmoles) in *m*-cresol (5 ml) was heated at 150° for 5 hours, then chilled to ambient temperature. Upon dilution with ether (*ca.* 30 ml), a solid product precipitated out. It was taken up with ether, rinsed several times with ether and dried.

Compound **1a** produced a 98% yield of **15a**, mp 306° (ethyl acetate) [lit [48], mp 304-305°]; ir: 3190 (NH), 1740 cm^{-1} (C=O).

Compound **1b** yielded **15b** in 94% yield, mp 307° (ethanol) [lit [49], mp 303-304°]; ir: 3170 (NH), 1520 cm^{-1} (C=N).

The reaction of **1a** with **13** conducted at 70° for 48 hours provided **14a** in 98% yield, mp 189-190° (water); ir: 3330, 3230 (NH), 1680 (semicarbazide C=O), 1650 cm^{-1} (benzoyl C=O).

Anal. Calcd. for $C_{14}H_{14}N_4O_2$: C, 62.21; H, 5.22; N, 20.73. Found: C, 62.46; H, 5.10; N, 20.65.

Heating **14a** (5 mmoles) in *m*-cresol (5 ml) at 150° for 5 hours gave a 90% yield of **15a**.

Reactions of **1a** and **1b** with *o*-Aminophenol (**16**) or *o*-Aminothiophenol (**17**).

To a solution of **16** or **17** (5 mmoles) in *m*-cresol (5 ml) was added **1a** or **1b** (5 mmoles) and the mixture was stirred at 150° for 5 hours. After cooling to room temperature, the mixture was poured into ether (30 ml). The precipitate formed was collected, rinsed well with ether and dried.

The reactions of **16** and **17** with **1a** furnished **22** in 92% yield from **16** and in 91% yield from **17**, the yield being calculated on the basis of half-molar quantities of **1a**, mp 215-216° (water) [lit [39], mp 217-218°]; ir: 3420, 3250 (NH), 1720 (carbohydrazide C=O), 1670 cm^{-1} (benzoyl C=O).

In the reaction with **1b**, **16** and **17** afforded **23** in 83% yield from **16** and in 81% yield from **17**, based on the half-molar quantities of **1b**, mp 239-241° (ethanol) [lit [50], mp 237-238°]; ir: 3200 (NH), 1660 cm^{-1} (C=O).

Reactions of **1a** and **1b** with *o*-Aminobenzamide (**24**).

A mixture of **1a** or **1b** (5 mmoles) and **24** (5 mmoles) in *m*-cresol (5 ml) was reacted with stirring at 150° for 24 hours and then left to cool to room temperature. Evolution of ammonia was continued for most of the stirring period. The product was isolated by pouring the mixture into ether (*ca.* 30 ml). It was filtered off, washed repeatedly with ether and dried.

Compound **1a** afforded a 94% yield of **26a**, mp 281-282° (ethanol/water) [lit [38], mp 281-282°]; ir: 3250, 3160 (NH), 1730 (quinazoline 4-C=O), 1660 cm^{-1} (quinazoline 2-C=O and amide C=O).

Compound **1b** produced **26b** in 91% yield, mp 263-265° (ethanol/water) [lit [38], mp 264-265°]; ir: 3240, 3180 (NH), 1715 (quinazoline C=O), 1670 cm^{-1} (amide C=O).

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