

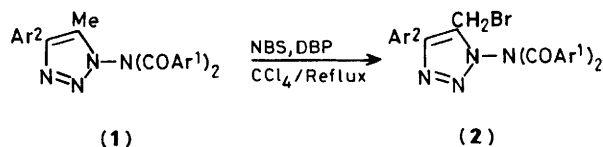
Fused *v*-Triazolo-heterocycles. Synthesis of 4*H*-*v*-Triazolo[1,5-*d*][1,3,4]oxadiazines¹

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The synthesis of some 4*H*-*v*-triazolo[1,5-*d*][1,3,4]oxadiazine derivatives, which constitute a novel heterocyclic ring system, is described. They are prepared by heating of the corresponding 5-bromomethyl-1-(*N,N*-diaroylamino)-*v*-triazoles. The spectroscopic data of these new compounds are also reported.

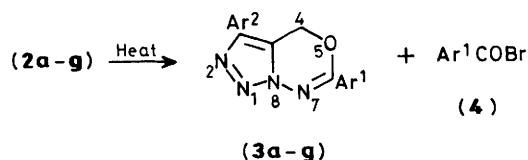
Following our previous studies on *v*-triazole derivatives²⁻⁴ we have now prepared several 4-aryl-5-bromomethyl-1-(*N,N*-diaroylamino)-*v*-triazoles (2) from the corresponding 5-methyl-derivatives (1), in order to use them as starting materials in the synthesis of fused *v*-triazolo-heterocycles.



- a; Ar² = Ar¹ = Ph
 b; Ar² = Ph, Ar¹ = *p*-MeOC₆H₄
 c; Ar² = Ph, Ar¹ = *p*-ClC₆H₄
 d; Ar² = Ph, Ar¹ = *o*-ClC₆H₄
 e; Ar² = Ph, Ar¹ = *o*-O₂NC₆H₄
 f; Ar² = *p*-MeOC₆H₄, Ar¹ = Ph
 g; Ar² = *p*-ClC₆H₄, Ar¹ = Ph

Compounds (2) were prepared in good yield by reaction of (1) with *N*-bromosuccinimide (NBS) in carbon tetrachloride in the presence of dibenzoyl peroxide (DBP). They are stable crystalline compounds. Their i.r. spectra show peaks for $\nu_{\text{C=O}}$ at 1720–1750 cm⁻¹. In the ¹H n.m.r. spectra, the CH₂Br protons appeared at δ 4.6–4.8 and in their mass spectrum they showed the molecular ion peak (*M*⁺), as well as peaks corresponding to the ions *M* – 28⁺, Ar¹CO⁺, Ar²–C≡C–CH₂Br⁺, and Ar²–C≡C–CH₂⁺. Their analytical and spectral data are given in the Table.

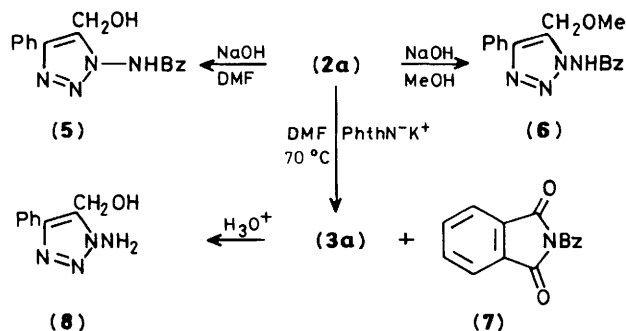
We have found that heating of the compounds (2) at temperatures above their melting points (170–200 °C) leads to the formation of the 4*H*-3,6-diaryl-*v*-triazolo[1,5-*d*][1,3,4]-oxadiazines (3) and the corresponding aryl bromides (4). In the



case of the compounds (2) with melting points higher than 200 °C, as well as for the derivatives with an *ortho*-substituent in the aryl moiety (2d, e), the yields of the *v*-triazolo-oxadiazines (3) were very poor, because of decomposition of the reaction products. Better results were obtained when the reaction was carried out in a tetralin solution in the presence of potassium or sodium carbonate and at lower temperatures, i.e. between 150 and 170 °C. The above method is analogous to that of van

Alphen,⁵ who first synthesized the 4,5-dihydro-6*H*-1,3,4-oxadiazine ring system by heating *N'*-chloroacetylhydrazides and related compounds with sodium carbonate in acetone in a sealed tube. Other reported preparations of this heterocyclic ring are based on the cyclization of the appropriate derivatives of acylhydrazides.⁶⁻⁹

Reaction of (2a) with sodium hydroxide in dimethylformamide (DMF), by a method analogous to that reported by Gaozza and Laudan,⁷ failed to give the expected (3a), but gave instead the 1-benzamido-5-hydroxymethyl-*v*-triazole (5). Treatment of (2a) with sodium hydroxide in a methanolic solution gave the 5-methoxymethyl-derivative (6). Treatment of (2a) with potassium phthalimide (PhthN⁻K⁺) in DMF gave compound (3a) and *N*-benzoylphthalimide (7).



The structures of compounds (3) have been established by examining their analytical and spectral data. Thus, in the i.r. spectrum they showed a weak peak at 1620 cm⁻¹, which is attributed to the C=N bond stretching vibration. In the ¹H n.m.r. spectrum, taken in CDCl₃ the methylenic protons of the oxadiazine ring resonated at δ 5.8–5.9, whereas the aromatic protons gave the expected pattern for the two aryl rings. The CH₂O carbon appeared in the ¹³C n.m.r. spectrum at ca. 63 p.p.m., whereas a peak at ca. 154 p.p.m. was in accord with the presence of the C=N carbon¹⁰ of the oxadiazine ring. In the mass spectrum, besides the molecular ion peak (*M*⁺), they gave also peaks corresponding to the *M* – 28⁺ ion (which is characteristic for the triazole ring)¹¹ and also peaks corresponding to the Ar¹CO⁺ and Ar¹CN⁺ ions as well as to the Ar²–C≡C–CH₂O⁺ and Ar²–C≡C–CH₂⁺ fragments.

Heating of compound (3a) with concentrated hydrochloric acid gave the 1-amino-4-phenyl-*v*-triazole (8), which is in agreement⁷ with structure (3).

Experimental

M.p.s were determined on a Kofler hot-stage apparatus and are uncorrected. I.r. spectra were recorded as Nujol mulls on

Table. Physical, spectral, and analytical data for compounds (2)

No.	M.p. (°C)	Yield (%)	Solvent*	$\nu_{\max}/\text{cm}^{-1}$ (nujol) (C=O)	^1H n.m.r. (δ , CDCl_3)					Mass spectrum m/z (r.i.)	Formula	Found (Required %)		
					Aroyl groups (Ar^1CO)		4-Aryl group (Ar^2)					C	H	N
(2a)	142—144	70	Benzene-LP	1735 1710	CH_2Br 4.60 (2 H, s)	<i>o</i> - 7.90 (4 H, m)	<i>m-p</i> - <i>ca.</i> 7.5 (11 H, m)	<i>o</i> - <i>ca.</i> 7.5 (11 H, m)	<i>m-p</i> - <i>ca.</i> 7.5 (11 H, m)	462, 460 (M^+) 434, 432 ($M^+ - 28$) 196, 194, 115, 105 (100)	$\text{C}_{23}\text{H}_{17}\text{BrN}_4\text{O}_2$	60.15 (59.88)	3.8 (3.71)	12.15 (12.14)
(2b)	132—134	85	Benzene-LP	1715 1700	4.60 (2 H, s)	7.94 (4 H, d)	6.88 (4 H, d)	7.82 (2 H, m)	7.46 (3 H, m)	494, 492 ($M^+ - 28$) 196, 194, 135 (100), 115	$\text{C}_{25}\text{H}_{21}\text{BrN}_4\text{O}_4$	57.4 (57.59)	4.0 (4.06)	10.55 (10.75)
(2c)	205—207	85	Benzene-LP	1740 1720	4.56 (2 H, s)	7.89 (4 H, d)	7.43 (7 H, m)	7.79 (2 H, m)	7.43 (7 H, m)	530, 528 (M^+) 502, 500 ($M^+ - 28$) 196, 194, 139 (100), 115	$\text{C}_{23}\text{H}_{15}\text{BrCl}_2\text{N}_4\text{O}_2$	52.35 (52.11)	2.7 (2.85)	10.5 (10.57)
(2d)	206—209	90	CH_2Cl_2 - Et_2O	1740 1730 1712	4.68 (2 H, s)	7.78 (4 H, m)	7.29 (6 H, m)	7.78 (4 H, m)	7.49 (3 H, m)	530, 528 (M^+) 502, 500 ($M^+ - 28$) 196, 194, 139 (100), 115	$\text{C}_{23}\text{H}_{15}\text{BrCl}_2\text{N}_4\text{O}_2$	52.25 (52.11)	2.75 (2.85)	10.7 (10.57)
(2e)	207—210	90	CH_2Cl_2 - Et_2O	1750 1740	4.80 (2 H, s)	<i>ca.</i> 7.7 (8 H, m)	8.27 (2 H, m)	<i>ca.</i> 7.7 (8 H, m)	7.46 (3 H, m)	492, 490 (M^+) 464, 462 ($M^+ - 28$) 145, 105 (100)	$\text{C}_{23}\text{H}_{15}\text{BrN}_6\text{O}_6$	50.05 (50.11)	2.75 (2.74)	14.95 (15.24)
(2f)	146—148	80	Benzene-LP	1735 1710	4.59 (2 H, s)	7.93 (4 H, m)	7.41 (6 H, m)	7.75 (2 H, d)	7.02 (2 H, d)	492, 490 (M^+) 464, 462 ($M^+ - 28$) 145, 105 (100)	$\text{C}_{24}\text{H}_{19}\text{BrN}_4\text{O}_3$	58.45 (58.67)	3.85 (3.90)	11.25 (11.40)
(2g)	173—176	75	CH_2Cl_2 - Et_2O	1745 1715	4.58 (2 H, s)	7.92 (4 H, m)	7.45 (8 H, m)	7.75 (2 H, d)	7.45 (8 H, m)	496, 494 (M^+) 468, 466 ($M^+ - 28$) 149, 105 (100)	$\text{C}_{23}\text{H}_{16}\text{BrClN}_4\text{O}_2$	55.75 (55.72)	3.25 (3.25)	11.45 (11.30)

* LP = Light petroleum.

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a Perkin-Elmer 257 spectrometer. ^1H N.m.r. and ^{13}C n.m.r. spectra were obtained on a Varian CFT-20 spectrometer in CDCl_3 with tetramethylsilane (TMS) as internal standard. The mass spectra were obtained on a Hitachi-Perkin-Elmer RMU 6L spectrometer and elemental microanalyses were performed with a Perkin-Elmer 240 analyser. Column chromatography was performed over Merck Kieselgel 60, particle size 0.063–0.200 mm. Light petroleum refers to that fraction of b.p. 60–80 °C. Ether refers to diethyl ether.

Compounds (1).—These were prepared by thermal isomerization from the corresponding triazolyl-isoimides as previously described,^{12,13} and their analytical and spectral data were in agreement with their structure.^{4,13}

4-Aryl-5-bromomethyl-1-(N,N-diaroylamino)-v-triazoles (2). *General Procedure.*—To a refluxing solution of (1) (5 mmol) in carbon tetrachloride (50 ml) were added recrystallized *N*-bromosuccinimide (1.0 g, 5.5 mmol) and dibenzoyl peroxide (0.3 g). The mixture was refluxed for a further 4–6 h and the succinimide formed was filtered off. The filtrate was washed with water (4 × 20 ml), dried and evaporated. The 5-bromomethyl derivative (2) crystallized on treatment with ether, and was isolated by filtration. This product was pure enough for further reaction. For elemental analysis it was recrystallized from CH_2Cl_2 -ether or C_6H_6 -light petroleum. Analytical and spectral data of compounds (2) are given in the Table.

4H-3,6-Diaryl-v-triazolo[1,5-d][1,3,4]oxadiazines (3): *General Procedure.*—*Method A.* Compound (2) (0.1–1.0 mmol) was heated in an oil-bath at 180–190 °C for 30 min. For compounds (2) which have higher m.p.s, the temperature was raised to their m.p.s; the solids, on melting, turned dark brown. After cooling, the solidified brown mass was washed on a glass filter with ether in order to remove aroyl bromide. The *v*-triazolo-oxadiazine (3) was isolated and recrystallized from dichloromethane-ether. Better purification was achieved by sublimation of the crude product at 180–190 °C at 0.2–0.5 mmHg, or by column chromatography over SiO_2 using ethyl acetate–light petroleum (2:8) as eluant. The aroyl bromide (4) was then either isolated from the ether washings and identified by its i.r. spectrum, or it was hydrolysed and isolated as the corresponding substituted benzoic acid.

Method B. A mixture of (2) (0.5–1.0 mmol) and anhydrous potassium carbonate (0.2 g, 1.5 mmol), (or the equivalent amount of anhydrous sodium carbonate), in tetralin (4 ml) dried over 4 Å molecular sieves, was heated at 160–170 °C for 40 min. After cooling, CH_2Cl_2 (30 ml) was added and the mixture was washed with water (2 × 20 ml). The solution was then dried and the CH_2Cl_2 removed. With time, crystals of the *v*-triazolo-oxadiazine (3) precipitated from the tetralin solution and these were filtered off, washed with ether, and recrystallized from the appropriate solvents.

4H-3,6-Diphenyl-v-triazolo[1,5-d][1,3,4]oxadiazine (3a).—*Method A.* Compound (2a) (0.7 g, 1.5 mmol) was heated at 190 °C for 30 min to give the *v*-triazolo-oxadiazine (3a) (0.22 g, 53%), m.p. 237 °C (subl.) (Found: C, 70.0; H, 4.4; N, 20.25. $\text{C}_{16}\text{H}_{12}\text{N}_4\text{O}$ requires C, 69.55; H, 4.38; N, 20.28%; ν_{max} 1 620 ($\text{C}=\text{N}$) cm^{-1} ; δ_{H} 5.80 (2 H, s, CH_2O), 7.53 (8 H, m), and 8.12 (2 H, m); δ_{C} (20 MHz, CDCl_3) 63.4 (CH_2O), and 154.4 ($\text{C}=\text{N}$); m/z 276 (M^+ , 1.5%), 248 ($M^+ - 28$, 1.3%), 115 (49%), and 105 (Ar^1CO^+ , 100%).

4H-6-p-Methoxyphenyl-3-phenyl-v-triazolo[1,5-d][1,3,4]-oxadiazine (3b).—*Method A.* Compound (2b) (0.52 g, 1 mmol) was heated at 180 °C for 30 min to give the *v*-triazolo-oxadiazine (3b) (0.2 g, 66%), m.p. 230–232 °C (from CH_2Cl_2 -

ether) (Found: C, 67.0; H, 4.55; N, 17.95. $\text{C}_{17}\text{H}_{14}\text{N}_4\text{O}_2$ requires C, 66.66; H, 4.61; N, 18.29%; ν_{max} 1 610 ($\text{C}=\text{N}$) cm^{-1} ; δ_{H} 5.82 (2 H, s, CH_2O), 3.86 (3 H, s, MeO), 6.93 (2 H, d), 7.40 (3 H, m), 7.58 (2 H, m), and 8.18 (2 H, d); δ_{C} (20 MHz, CDCl_3) 63.2 (CH_2O), 153.8 ($\text{C}=\text{N}$); m/z 306 (M^+ , 1.6%), 278 ($M^+ - 28$, 2.5%), 135 (Ar^1CO^+ , 100%), and 115 (24%).

4H-6-p-Chlorophenyl-3-phenyl-v-triazolo[1,5-d][1,3,4]-oxadiazine (3c).—*Method B.* A mixture of (2c) (0.22 g, 0.41 mmol) and K_2CO_3 (0.1 g, 0.7 mmol) in tetralin (3 ml) was heated at 180 °C for 50 min to give the *v*-triazolo-oxadiazine (3c) (0.03 g, 24%), m.p. 260–262 °C (Found: C, 62.05; H, 3.25; N, 18.1. $\text{C}_{16}\text{H}_{11}\text{ClN}_4\text{O}$ requires C, 61.85; H, 3.57; N, 18.03%; ν_{max} 1 610 ($\text{C}=\text{N}$) cm^{-1} ; δ_{H} 5.90 (2 H, s, CH_2O), 7.45 (5 H, m), 7.61 (2 H, m), and 8.05 (2 H, d); m/z 310 (M^+ , 1.1%), 282 ($M^+ - 28$, 2.2%), 139 (Ar^1CO^+ , 100%), and 115 (76%).

Method A. Compound (2c) (0.2 g, 0.38 mmol) was heated at 205 °C for 40 min to give a crude product, m.p. 200–250 °C. This was chromatographed on SiO_2 (using CHCl_3 as eluant) to give the *v*-triazolo-oxadiazine (3c) (0.013 g, 11%), m.p. 252–256 °C, which was identical with that isolated by method B.

4H-6-o-Chlorophenyl-3-phenyl-v-triazolo[1,5-d][1,3,4]-oxadiazine (3d).—*Method B.* A mixture of (2d) (0.8 g, 1.5 mmol) and Na_2CO_3 (0.2, 2 mmol) in tetralin (4 ml) was heated at 170 °C for 40 min to give a crude product (0.17 g) which was chromatographed on SiO_2 using ethyl acetate–light petroleum (2:8) as eluant to give the *v*-triazolo-oxadiazine (3d) (0.093 g, 20%), m.p. 172–174 °C (from CH_2Cl_2 -ether–light petroleum) (Found: C, 61.75; H, 3.55; N, 17.95. $\text{C}_{16}\text{H}_{11}\text{ClN}_4\text{O}$ requires C, 61.85; H, 3.57; N, 18.03%; ν_{max} 1 620 ($\text{C}=\text{N}$) cm^{-1} ; δ_{H} 5.90 (2 H, s, CH_2O), 7.44 (5 H, m), 7.62 (2 H, m), ca. 7.8 (1 H, m); m/z 310 (M^+ , 1%), 282 ($M^+ - 28$, 1%), 139 (Ar^1CO^+ , 100%), and 115 (80%).

4H-6-o-Nitrophenyl-3-phenyl-v-triazolo[1,5-d][1,3,4]-oxadiazine (3e).—*Method B.* A mixture of (2e) (0.276 g, 0.5 mmol) and K_2CO_3 (0.15 g, 1 mmol) in tetralin (4 ml) was heated at 170 °C for 50 min to give the *v*-triazolo-oxadiazine (3e) (0.03 g, 19%), m.p. 228–230 °C (from CH_2Cl_2 -ether) (Found: C, 59.7; H, 3.35; N, 21.65. $\text{C}_{16}\text{H}_{11}\text{N}_5\text{O}_3$ requires C, 59.81; H, 3.45; N, 21.8; ν_{max} 1 620 ($\text{C}=\text{N}$) cm^{-1} ; δ_{H} 5.82 (2 H, s, CH_2O), 7.45 (3 H, m), ca. 7.7 (4 H, m), and 8.05 (2 H, m); m/z 321 (M^+ , 0.3%), 293 ($M^+ - 28$, 0.6%), 150 (Ar^1CO^+ , 18%), and 115 (100%).

4H-3-p-Methoxyphenyl-6-phenyl-v-triazolo[1,5-d][1,3,4]-oxadiazine (3f).—*Method A.* Compound (2f) (0.66 g, 1.3 mmol) was heated at 170 °C for 30 min to give the *v*-triazolo-oxadiazine (3f) (0.16 g, 39%), m.p. 223–225 °C (subl.) (Found: C, 66.35; H, 4.55; N, 18.4. $\text{C}_{17}\text{H}_{14}\text{N}_4\text{O}_2$ requires C, 66.66; H, 4.61; N, 18.29%; ν_{max} 1 615 ($\text{C}=\text{N}$) cm^{-1} ; δ_{H} 3.82 (3 H, s, MeO), 5.82 (2 H, s, CH_2O), 6.94 (2 H, d), 7.50 (5 H, m), and 8.06 (2 H, m); δ_{C} (20 MHz, CDCl_3) 63.3 (CH_2O), and 154.4 ($\text{C}=\text{N}$); m/z 306 (M^+ , 2.8%), 278 ($M^+ - 28$, 1%), 145 (100%), and 105 (Ar^1CO^+ , 74%).

4H-3-p-Chlorophenyl-6-phenyl-v-triazolo[1,5-d][1,3,4]-oxadiazine (3g).—*Method B.* A mixture of (2g) (0.496 g, 1 mmol) and K_2CO_3 (0.2 g, 1.5 mmol) in tetralin (4 ml) was heated at 170 °C for 50 min to give the *v*-triazolo-oxadiazine (3g) (0.1 g, 33%), m.p. 289–291 °C (from CHCl_3) (Found: C, 61.5; H, 3.5; N, 17.95. $\text{C}_{16}\text{H}_{11}\text{ClN}_4\text{O}$ requires C, 61.85; H, 3.57; N, 18.03%; ν_{max} 1 620 ($\text{C}=\text{N}$) cm^{-1} ; δ_{H} 5.86 (2 H, s, CH_2O), 7.40 (2 H, d), ca. 7.5 (5 H, m), and 8.07 (2 H, m); m/z 310 (M^+ , 1.5%), 282 ($M^+ - 28$, 0.3%), 149 (20%), 105 (Ar^1CO^+ , 100%).

Reaction of (2a) with Potassium Phthalimide in DMF.—A mixture of (2a) (0.23 g, 0.5 mmol) and potassium phthalimide (0.13 g, 0.7 mmol) in DMF (5 ml) was stirred at 25 °C for 40 min

and then heated at 70 °C for 20 min. The mixture was cooled, diluted with water (70 ml) and extracted with CHCl_3 (30 ml). The chloroform solution was washed with 0.2M-NaOH (20 ml) and water (20 ml), and upon evaporation gave an oily mass; this, on treatment with ethyl ether, gave white crystals (0.027 g, 20%), m.p. 235–237 °C, of the *v*-triazolo-oxadiazine (**3a**) identical with an authentic specimen by i.r., ^1H n.m.r., and mass spectroscopy. From the ether solution a crystalline product (0.025 g) was precipitated, which was identified as the *N*-benzoyl-phthalimide (**7**), m.p. 158–163 °C (lit.,¹⁴ 169 °C); ν_{max} , 1790, 1760, 1740, and 1700 cm^{-1} ; m/z 251 (M^+ , 88%), 223 ($M^+ - 28$, 28%), and 105 (PhCO^+ , 100%).

Acid Hydrolysis of (3a).—A solution of *v*-triazolo-oxadiazine (**3a**) (0.14 g, 0.5 mmol) in concentrated hydrochloric acid (7 ml) was refluxed for 2 h, after which it was made alkaline (Na_2CO_3 , pH 8) and extracted with CH_2Cl_2 (60 ml). Evaporation of the solvent from the extract gave the 5-hydroxymethyl-4-phenyl-1-amino-*v*-triazole (**8**) (0.034 g, 37%), m.p. 161–163 °C (from $\text{MeOH-Et}_2\text{O}$) (Found: C, 56.75; H, 5.3; N, 29.3. $\text{C}_9\text{H}_{10}\text{N}_4\text{O}$ requires C, 56.83; H, 5.30; N, 29.45%; ν_{max} , 3370, 3340, 3200br, and 1610 cm^{-1} ; δ_{H} [$\text{CDCl}_3 + [(\text{CD}_3)_2\text{SO}]$] 4.77 (2 H, s, CH_2O), 6.20 (1 H, s, exchangeable with D_2O , OH), 7.40 (3 H, m), and 7.80 (2 H, m); m/z 191 ($M^+ + 1$, 2%), 190 (M^+ , 1.6%), 189 ($M^+ - 1$, 2.3%), 175 ($M^+ - 15$, 1.4%), 173 ($M^+ - 17$, 1.4%), 162 ($M^+ - 28$, 11%), 149 (64%), 131 ($\text{PhC}\equiv\text{CCH}_2\text{OH}^+$, 100%), and 115 (93%).

Reaction of (2a) with NaOH in MeOH.—To a mixture of NaOH (1.0 g) in MeOH (10 ml) a solution of (**2a**) (0.115 g, 0.25 mmol) in MeOH (5 ml) was added and the mixture was heated at 60 °C for 3 h; it was then diluted with water (70 ml) and extracted with CHCl_3 (30 ml). The alkaline solution was acidified (pH 2) and extracted with CHCl_3 (50 ml). The extract was evaporated to give an oily mass which, on treatment with ether–light petroleum, gave the 1-benzamido-5-methoxymethyl-4-phenyl-*v*-triazole (**6**) (0.055 g, 71%), m.p. 128–131 °C (from ether–light petroleum) (Found: C, 66.1; H, 5.2; N, 18.45. $\text{C}_{17}\text{H}_{16}\text{N}_4\text{O}_2$ requires C, 66.22; H, 5.22; N, 18.17%; ν_{max} , 3200 (NH), and 1705 (C=O) cm^{-1} ; δ_{H} 3.26 (3 H, s, MeO), 4.49 (2 H, s, CH_2O), 7.40 (6 H, m), 7.66 (2 H, m), 7.93 (2 H, m), and 11.0 (1 H, s, NH); m/z 308 (M^+ , 0.4%), 280 ($M^+ - 28$, 6%), 146 ($\text{PhC}\equiv\text{CCH}_2\text{OME}^+$, 8%), 115 (46%), and 105 (100%).

Reaction of (2a) with NaOH in DMF.—A mixture of (**2a**) (0.3 g, 0.65 mmol) and NaOH (0.2 g, 5 mmol) in DMF (5 ml) was heated at 100 °C for 2 h. The DMF was then removed on an evaporator and the residue washed repeatedly with CH_2Cl_2 and ether, after which the remaining solid was dissolved in water (20 ml) and acidified (pH 2), to precipitate 1-benzamido-5-hydroxymethyl-4-phenyl-*v*-triazole (**5**); this was filtered off (0.125 g, 65%), m.p. 214–216 °C (from MeOH) (Found: C, 65.1; H, 4.75; N, 19.35. $\text{C}_{16}\text{H}_{14}\text{N}_4\text{O}_2$ requires C, 65.30; H, 4.79; N, 19.04%; ν_{max} , 3210br, 1715, and 1710 (C=O) cm^{-1} ; δ_{H} [$\text{CDCl}_3 + [(\text{CD}_3)_2\text{SO}]$] 4.20 (1 H, br, exchangeable with D_2O , OH), 4.65 (2 H, s, CH_2O), 7.50 (6 H, m), 7.98 (4 H, m), and 12.2 (1 H, s, exchangeable with D_2O , NH); m/z 294 (M^+ , 0.3%), 266 ($M^+ - 28$, 3.7%), 132 ($\text{PhC}\equiv\text{CCH}_2\text{OH}^+$, 19%), 131 ($\text{PhC}\equiv\text{CCH}_2\text{O}^+$, 22%), 115 (25%), 105 (98%), and 103 (100%).

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