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New mesoionic amides **4** and **5** with novel tricyclic ring systems have been synthesized by the photolysis of a 5-azido-1-mesityl-3-phenyltetrazolium salt **1c** *via* the corresponding triplet nitrene intermediate. The structure of **5** has been determined by X-ray crystallography.

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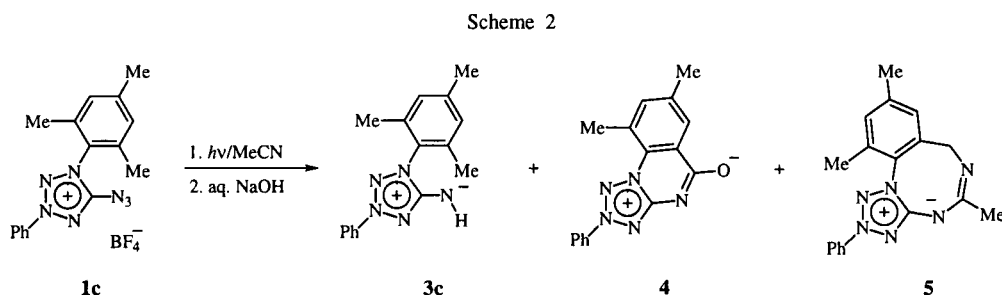
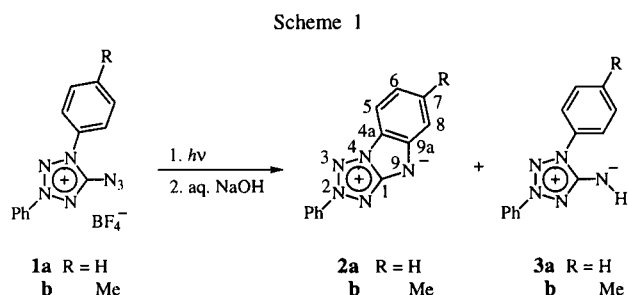
Introduction.

We recently reported the photochemical behavior of 5-azido-1,3-diaryltetrazolium salts [1]. The photolysis of the tetrazolium salts bearing a phenyl (**1a**) or *p*-tolyl substituent (**1b**) at the *N*¹-position gave novel tricyclic mesoionic amides **2a,b** with a tetrazolo[1,5-*a*]benzimidazole structure *via ortho*-hydrogen abstraction from the aryl group at the *N*¹-position by the nitrene intermediates of a singlet state (Scheme 1). On the other hand, the formation of this type of ring systems is not possible using the azidotetrazolium salt **1c** bearing a mesityl group at the *N*¹-position owing to the lack of *ortho*-hydrogens. Instead, **1c** was expected to undergo benzylic hydrogen abstraction from an *ortho*-methyl group by the triplet nitrene intermediate, because it is well known that triplet nitrene easily abstracts a neighboring benzylic hydrogen atom [2]. However, the photolysis of **1c** in methanol gave

only the corresponding tetrazolium-5-amide **3c** in 56% yield *via* hydrogen abstraction from methanol [1]. Now we report that changing the solvent from methanol to acetonitrile altered the reaction course markedly, and that two new tricyclic mesoionic amide derivatives **4** and **5** were obtained *via* intramolecular cyclization of the triplet nitrene intermediate.

Results and Discussion.

The photolysis was carried out using the Pyrex-filtered light of a high-pressure mercury lamp as described previously [1]. The photo-conversion of **1c** in acetonitrile was completed within 1 hour. A base treatment of the photolysate followed by column chromatographic separation gave two new mesoions **4** (8% yield) and **5** (29%) together with known **3c** (10%) (Scheme 2). The colorless product **4** was found to have the molecular formula C₁₆H₁₃N₅O based on the mass spectral and elemental analyses. The characteristic band at 1550 cm⁻¹ in the ir spectrum as well as the low field-shift (δ 8.3 ppm) of one of the mesitylene-ring protons in the ¹H nmr suggest the presence of a carbonyl group which is conjugated with the aryl group at the *N*¹-position. This product was finally deduced to be 5-6-6 tricyclic amide **4** based on spectroscopic data. The very low wave-number of the amide-carbonyl stretching vibration implies that this carbonyl group exists as an enolate form rather than an amide form. These ir and ¹H nmr spectroscopic features of **4** are very similar to those (1552 cm⁻¹ and δ 8.3 ppm, respectively) of the reference compound, 1,3-diphenyltetrazolium-5-benzamide (**6**). The formation of this 5-6-6 tricyclic structure [3] can be rationalized by an intramolecular insertion of the triplet nitrene



to a C-H bond of an *ortho*-methyl group to give compound **7**, which is considered to be converted to enolate **4**.

The other product **5** was obtained as yellow crystals. The molecular formula $C_{18}H_{18}N_6$ suggests that one molecule of the solvent acetonitrile is incorporated into this compound. The structure of **5** was unambiguously established

by X-ray crystallography. The X-ray analysis details, fractional atomic coordinates, bond lengths and bond angles are collected in Tables 1-4. The ORTEP drawing of the structure with atom labels is shown in Figure 1, which reveals the novel 5-8-6-membered tricyclic ring system [4]. The eight-membered ring is not planar, and the bond

Table 1
Crystal and Experimental Data for **5**

Formula	$C_{18}H_{18}N_6$
Formula Weight	318.38
Crystal Color, Habit	pale yellow, prismatic
Crystal size / mm	0.50 x 0.30 x 0.20
Crystal system	monoclinic
Lattice Type	Primitive
Space Group	$P2_1/n$ (#14)
$a / \text{\AA}$	11.807(2)
$b / \text{\AA}$	7.595(2)
$c / \text{\AA}$	18.641(2)
$\beta / ^\circ$	107.314(9)
$V / \text{\AA}^3$	1595.9(4)
Z value	4
Scan mode	ω -2 θ
$2\theta_{\max} / ^\circ$	52.6
$D_{\text{calc}} / \text{g}\cdot\text{cm}^{-3}$	1.325
Radiation	MoK α ($\lambda = 0.71069 \text{\AA}$)
T / K	293
$F(000)$	672.00
No. of refls. obsd.	3662
hkl collected	$-13 \leq h \leq 0, 0 \leq k \leq 8, -21 \leq l \leq 21$
No. of refls. used.	1084
$(I_o > 3\sigma I_o)$	
R	0.055
R_w	0.089

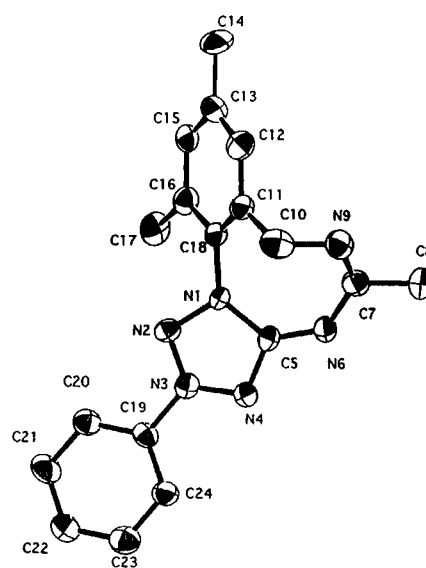


Figure 1. ORTEP view of **5**.

Table 2
Atomic Coordinates and $B_{\text{iso}}/B_{\text{eq}}$

atom	x	y	z	B_{eq}
N(1)	0.8289(4)	0.1413(7)	0.2504(3)	2.3(1)
N(2)	0.7576(4)	0.2049(7)	0.2894(3)	2.4(1)
N(3)	0.8115(4)	0.1540(7)	0.3575(3)	2.4(1)
N(4)	0.9147(4)	0.0711(8)	0.3692(3)	2.6(1)
N(6)	1.0183(4)	-0.0258(8)	0.2889(3)	2.8(1)
N(9)	1.0816(5)	0.1171(9)	0.1915(3)	3.3(1)
C(5)	0.9304(5)	0.0609(9)	0.3004(3)	2.2(1)
C(7)	1.0754(6)	-0.010(1)	0.2345(4)	3.2(2)
C(8)	1.1481(6)	-0.173(1)	0.2321(4)	4.4(2)
C(10)	1.0124(6)	0.276(1)	0.1935(4)	3.7(2)
C(11)	0.8855(5)	0.2461(9)	0.1433(3)	2.6(2)
C(12)	0.8586(6)	0.2744(10)	0.0668(4)	3.3(2)
C(13)	0.7476(7)	0.2327(10)	0.0179(4)	3.7(2)
C(14)	0.7226(7)	0.252(1)	-0.0652(4)	4.9(2)
C(15)	0.6626(6)	0.1661(9)	0.0487(4)	3.2(2)
C(16)	0.6842(5)	0.1401(9)	0.1252(4)	2.9(2)
C(17)	0.5864(5)	0.070(1)	0.1546(4)	3.8(2)

Table 2 (continued)
Atomic Coordinates and B_{iso}/B_{eq}

atom	x	y	z	B_{eq}
C(18)	0.7993(5)	0.1763(9)	0.1718(3)	2.4(1)
C(19)	0.7640(5)	0.1965(9)	0.4183(3)	2.5(2)
C(20)	0.8338(5)	0.1688(9)	0.4913(3)	2.8(2)
C(21)	0.7873(6)	0.2114(10)	0.5489(4)	3.5(2)
C(22)	0.6754(6)	0.2855(10)	0.5329(4)	3.6(2)
C(23)	0.6064(6)	0.313(1)	0.4592(4)	3.7(2)
C(24)	0.6521(6)	0.2662(9)	0.4016(3)	2.9(2)
H(81)	1.0990	-0.2746	0.2269	8.0000
H(82)	1.2092	-0.1871	0.2772	8.0000
H(83)	1.1788	-0.1658	0.1906	8.0000
H(101)	1.0057	0.3253	0.2468	8.0000
H(102)	1.0521	0.3754	0.1684	8.0000
H(121)	0.9204	0.3655	0.0479	8.0000
H(141)	0.6411	0.3311	-0.0920	8.0000
H(142)	0.7749	0.3657	-0.0828	8.0000
H(143)	0.7395	0.1234	-0.0821	8.0000
H(151)	0.5717	0.1219	0.0098	8.0000
H(171)	0.5406	-0.0150	0.1207	8.0000
H(172)	0.5373	0.1623	0.1614	8.0000
H(173)	0.6214	0.0128	0.2023	8.0000
H(201)	0.9144	0.0989	0.4981	8.0000
H(211)	0.8493	0.1684	0.6083	8.0000
H(221)	0.6406	0.3264	0.5754	8.0000
H(231)	0.5045	0.3002	0.4451	8.0000
H(241)	0.6097	0.2908	0.3486	8.0000

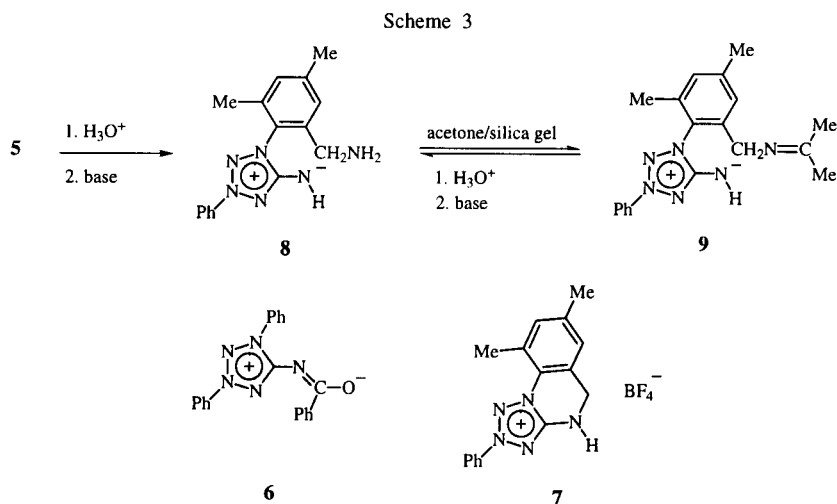
$$B_{eq} = 8/3 \pi^2 (U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}aa^*bb^* \cos \gamma + 2U_{13}aa^*cc^* \cos \beta + 2U_{23}bb^*cc^* \cos \alpha)$$

Table 3
Bond Lengths (Å)

atom	atom	distance	atom	atom	distance
N(1)	N(2)	1.355(6)	N(1)	C(5)	1.419(7)
N(1)	C(18)	1.425(7)	N(2)	N(3)	1.297(6)
N(3)	N(4)	1.331(6)	N(3)	C(19)	1.443(7)
N(4)	C(5)	1.350(7)	N(6)	C(5)	1.300(7)
N(6)	C(7)	1.380(8)	N(9)	C(7)	1.272(9)
N(9)	C(10)	1.463(9)	C(7)	C(8)	1.51(1)
C(10)	C(11)	1.528(9)	C(11)	C(12)	1.381(9)
C(11)	C(18)	1.387(8)	C(12)	C(13)	1.392(10)
C(13)	C(14)	1.497(9)	C(13)	C(15)	1.391(9)
C(15)	C(16)	1.386(9)	C(16)	C(17)	1.516(9)
C(16)	C(18)	1.405(8)	C(19)	C(20)	1.382(8)
C(19)	C(24)	1.370(8)	C(20)	C(21)	1.382(9)
C(21)	C(22)	1.385(10)	C(22)	C(23)	1.389(9)
C(23)	C(24)	1.385(9)			
C(8)	H(81)	0.95	C(8)	H(82)	0.94
C(8)	H(83)	0.95	C(10)	H(101)	1.09
C(10)	H(102)	1.07	C(12)	H(121)	1.14
C(14)	H(141)	1.12	C(14)	H(142)	1.16
C(14)	H(143)	1.07	C(15)	H(151)	1.15
C(17)	H(171)	0.95	C(17)	H(172)	0.94
C(17)	H(173)	0.96	C(20)	H(201)	1.06
C(21)	H(211)	1.18	C(22)	H(221)	1.04
C(23)	H(231)	1.16	C(24)	H(241)	0.98

Table 4
Bond Angles (°)

atom	atom	atom	angle	atom	atom	atom	angle
N(2)	N(1)	C(5)	109.7(4)	N(2)	N(1)	C(18)	118.9(5)
C(5)	N(1)	C(18)	131.1(5)	N(1)	N(2)	N(3)	102.5(4)
N(2)	N(3)	N(4)	117.4(5)	N(2)	N(3)	C(19)	120.6(5)
N(4)	N(3)	C(19)	121.9(5)	N(3)	N(4)	C(5)	104.4(5)
C(5)	N(6)	C(7)	131.8(6)	C(7)	N(9)	C(10)	118.3(5)
N(1)	C(5)	N(4)	105.8(5)	N(1)	C(5)	N(6)	132.0(5)
N(4)	C(5)	N(6)	121.9(6)	N(6)	C(7)	N(9)	131.3(7)
N(6)	C(7)	C(8)	110.9(6)	N(9)	C(7)	C(8)	117.6(6)
N(9)	C(10)	C(11)	108.4(6)	C(10)	C(11)	C(12)	119.4(6)
C(10)	C(11)	C(18)	121.3(5)	C(12)	C(11)	C(18)	119.1(6)
C(11)	C(12)	C(13)	121.5(6)	C(12)	C(13)	C(14)	120.9(7)
C(12)	C(13)	C(15)	117.8(6)	C(14)	C(13)	C(15)	121.3(7)
C(13)	C(15)	C(16)	122.8(6)	C(15)	C(16)	C(17)	119.7(6)
C(15)	C(16)	C(18)	117.2(6)	C(17)	C(16)	C(18)	123.1(6)
N(1)	C(18)	C(11)	118.8(5)	N(1)	C(18)	C(16)	119.7(5)
C(11)	C(18)	C(16)	121.5(5)	N(3)	C(19)	C(20)	118.8(5)
N(3)	C(19)	C(24)	118.8(5)	C(20)	C(19)	C(24)	122.4(6)
C(19)	C(20)	C(21)	118.1(6)	C(20)	C(21)	C(22)	120.2(6)
C(21)	C(22)	C(23)	121.1(6)	C(22)	C(23)	C(24)	118.6(6)
C(19)	C(24)	C(23)	119.7(6)				
C(7)	C(8)	H(81)	109.4	C(7)	C(8)	H(82)	111.1
C(7)	C(8)	H(83)	109.6	H(81)	C(8)	H(82)	106.3
H(81)	C(8)	H(83)	109.3	H(82)	C(8)	H(83)	111.1
N(9)	C(10)	H(101)	120.0	N(9)	C(10)	H(102)	104.5
C(11)	C(10)	H(101)	106.7	C(11)	C(10)	H(102)	108.4
H(101)	C(10)	H(102)	108.4	C(11)	C(12)	H(121)	116.4
C(13)	C(12)	H(121)	120.1	C(13)	C(14)	H(141)	113.3
C(13)	C(14)	H(142)	114.1	C(13)	C(14)	H(143)	102.5
H(141)	C(14)	H(142)	86.4	H(141)	C(14)	H(143)	124.9
H(142)	C(14)	H(143)	115.9	C(13)	C(15)	H(151)	119.9
C(16)	C(15)	H(151)	117.3	C(16)	C(17)	H(171)	110.0
C(16)	C(17)	H(172)	110.4	C(16)	C(17)	H(173)	109.1
H(171)	C(17)	H(172)	110.1	H(171)	C(17)	H(173)	108.4
H(172)	C(17)	H(173)	108.8	C(19)	C(20)	H(201)	116.4
C(21)	C(20)	H(201)	124.9	C(20)	C(21)	H(211)	112.7
C(22)	C(21)	H(211)	127.0	C(21)	C(22)	H(221)	121.7
C(23)	C(22)	H(221)	117.3	C(22)	C(23)	H(231)	118.4
C(24)	C(23)	H(231)	114.9	C(19)	C(24)	H(241)	118.0
C(23)	C(24)	H(241)	122.1				



lengths in the tetrazolium ring are in good accord with those of the similar 1,3-diphenyltetrazolium mesoionic systems [5]. A possible mechanism for the formation of **5** is benzylic hydrogen abstraction by the triplet nitrene intermediate to produce the biradical species, which captures the solvent acetonitrile to yield **5**. The eight-membered ring of **5** was readily cleaved by acid-hydrolysis to give benzylamine **8**, which gave imine **9** simply by passing the acetone solution through a silica gel column. An acid-hydrolysis of **9** gave back **8** quantitatively (Scheme 3).

In summary, the photolysis of a 5-azido-1-mesity-3-phenyltetrazolium salt **1c** gave new tricyclic mesoionic amides **4** and **5** with 5-6-6 and 5-8-6 ring systems, respectively. This work as well as our previous paper [1] demonstrate that azidotetrazolium salts **1a-c** are useful precursors to unique mesoionic compounds with novel polycyclic ring systems.

EXPERIMENTAL

The melting points were determined with a hot-stage apparatus and are uncorrected. The IR spectra were obtained as potassium bromide discs with a JASCO A-102 spectrometer. The ^1H and ^{13}C NMR spectra were run with a Varian Gemini 200 (200 MHz and 50 MHz, respectively) and referenced using either the residual non-deuterated solvent or tetramethylsilane. The J values are given in Hz. The electronic spectra were measured on a Hitachi U-3500 spectrophotometer. The mass spectra were measured with a Hitachi M-2000 instrument. Elemental analyses were performed at Elemental Analysis Centre of Kyoto University. For photochemical reactions, a Riko Kagaku Sangyo UVL-100HA-100P apparatus was used. Column chromatography was carried out on 3-aminopropylsilane-modified silica gel (Fuji Silysia Chemical, NH-DM 1020).

Photochemical Reaction.

A solution of **1c** [1] (0.10 g, 0.25 mmole) in acetonitrile (100 ml) was irradiated using a 100W high pressure mercury lamp with a Pyrex filter at room temperature under an argon atmosphere. A gas evolution started immediately. After 1 hour, the solvent was evaporated and the residue was dissolved in dichloromethane and shaken with aqueous sodium hydroxide (1 N). The organic layer was separated, dried over anhydrous sodium sulfate and the solvent was evaporated. The residue (85 mg) was column chromatographed (dichloromethane:acetone = 10:1, then methanol) to give, in the order of elution, **4** (6 mg, 8%), **3c** [1] (6 mg, 10%) and **5** (23 mg, 29%).

5-6-6 Tricyclic Mesoionic Amide (**4**).

This compound was obtained as colorless crystals, mp $>300^\circ$ dec (ethanol); IR: 1644, 1586, 1550 (C-O^-), 1464, 1358, 1346, 1228, 982, 790 and 774 cm^{-1} ; ^1H NMR (deuteriochloroform): δ 2.51 (3H, s, Me), 2.92 (3H, s, Me), 7.50 (1H, s, aryl-H), 7.65-7.73 (3H, m, m -H and p -H of Ph) and 8.26-8.31 (3H, m, o -H of Ph and aryl-H); ^{13}C NMR (deuteriochloroform): δ 21.3 (Me), 21.5 (Me), 120.4 (o -C of Ph), 121.8 (aryl-C), 127.3 (aryl-C), 127.6 (aryl-CH), 128.7 (aryl-C), 130.1 (m -C of Ph), 132.4

(p -C of Ph), 135.7 (i -C of Ph or aryl-C), 136.9 (aryl-CH), 140.4 (i -C of Ph or aryl-C), 157.6 (C^+) and 167.4 (C-O^-); UV: λ_{max} (acetonitrile) (log ϵ)/nm 228 (4.41), 268 (4.45), 274 (4.43), 294 (4.17) and 360 (3.95); MS: m/z (EI, 70 eV) 291 (M^+ , 100%), 263 (7), 158 (8), 132 (61), 103 (34) and 77 (100, Ph); HRMS: Calcd. for $\text{C}_{16}\text{H}_{13}\text{N}_5\text{O}$: 291.1118. Found: M^+ , 291.1114.

Anal. Calcd. for $\text{C}_{16}\text{H}_{13}\text{N}_5\text{O} \cdot 1/2(\text{C}_2\text{H}_5\text{OH})$ (314.35): C, 64.96; H, 5.13; N, 22.28. Found: C, 65.27; H, 5.04; N, 22.42.

5-8-6 Tricyclic Mesoionic Amide (**5**).

This compound was obtained as yellow crystals, mp $228-229^\circ$ dec (acetonitrile); IR: 1604, 1594, 1526, 1466 and 764 cm^{-1} ; ^1H NMR (deuteriochloroform): δ 2.04 (3H, s, Me), 2.35 (3H, s, Me), 2.36 (3H, s, Me), 4.25 (1H, d, $J = 12$, CH_2), 5.12 (1H, d, $J = 12$, CH_2), 7.08 (1H, s, aryl-H), 7.14 (1H, s, aryl-H), 7.59-7.63 (3H, m, m -H and p -H of Ph) and 8.09-8.14 (2H, m, o -H of Ph); ^{13}C NMR (deuteriochloroform): δ 19.1 (Me), 21.2 (Me), 27.9 (Me), 50.6 (CH_2), 119.8 (o -C of Ph), 129.3 (aryl-C), 129.9 (m -C of Ph), 130.1 (aryl-CH), 131.4 (aryl-CH), 131.9 (p -C of Ph), 133.7 (aryl-C), 135.9 (i -C of Ph), 136.0 (aryl-C), 141.1 (aryl-C), 157.1 (C^+) and 163.0 ($=\text{C}$); UV/vis: λ_{max} (acetonitrile) (log ϵ)/nm 250 (4.36), 275 (4.30) and 402 (3.25); MS: m/z (EI, 70 eV) 318 (M^+ , 49%), 277 (48), 276 (100), 265 (15), 264 (82), 158 (14), 117 (24) and 77 (100, Ph); HRMS: Calcd. for $\text{C}_{18}\text{H}_{18}\text{N}_6$: 318.1592. Found: M^+ , 318.1599.

Anal. Calcd. for $\text{C}_{18}\text{H}_{18}\text{N}_6$ (318.38): C, 67.91; H, 5.70; N, 26.40. Found: C, 67.70; H, 5.48; N, 26.62.

1,3-Diphenyltetrazolium-5-benzamide (**6**).

1,3-Diphenyltetrazolium-5-amide (**3a**) (0.10 g, 0.42 mmole) was dissolved in acetonitrile (2 ml) and benzoyl chloride (48 μl , 0.42 mmole) was added. The solution turned from yellow to colorless and a precipitate was formed. After 20 minutes, the solvent was evaporated and the residue was dissolved in dichloromethane and treated with aqueous sodium hydroxide (0.5 N). The organic layer was separated, the solvent was removed and the residue was recrystallized from ethanol to give **6** (0.12 g, 84%); colorless crystals; mp $228-229^\circ$ dec; IR: 2950, 1616, 1592, 1552 (C-O^-), 1484, 1458, 1340, 1240, 760, 706 and 690 cm^{-1} ; ^1H NMR (deuteriochloroform): δ 7.36-7.48 (3H, m, o - and p -H of benzoyl), 7.57-7.70 (m, 6H, m -H and p -H of Ph) and 8.21-8.33 (m, 6H, o -H of Ph and benzoyl); ^{13}C NMR (deuteriochloroform): δ 120.5 (o -C of Ph), 123.1 (o -C of Ph), 127.3 (m -C of Ph or o - or m -C of benzoyl), 129.2 (m -C of Ph or o - or m -C of benzoyl), 129.6 (m -C of Ph or o - or m -C of benzoyl), 129.8 (m -C of Ph, or o - or m -C of benzoyl), 130.7 (p -C of Ph or p -C of benzoyl), 131.9 (p -C of Ph or p -C of benzoyl), 133.8 (i -C of benzoyl), 135.8 (i -C of Ph), 138.3 (i -C of Ph), 159.4 (C^+) and 173.2 (C-O^-); UV: λ_{max} (acetonitrile) (log ϵ)/nm 203 (4.99), 232 (4.46), 279 (4.65), 287 (4.66) and 348 (3.97); MS: m/z (EI, 70 eV) 341 (M^+ , 14%), 314 (25), 313 (98), 285 (10), 272 (8), 266 (10), 265 (96), 264 (99), 248 (49), 208 (6), 181 (10), 145 (11), 105 (100) and 77 (98, Ph); HRMS: calcd. for $\text{C}_{20}\text{H}_{13}\text{N}_5\text{O}$: 341.1240. Found M^+ , 341.1280.

Anal. Calcd. for $\text{C}_{20}\text{H}_{15}\text{N}_5\text{O}$ (341.37): C, 70.37; H, 4.43; N, 20.52. Found: C, 70.67; H, 4.51; N, 20.81.

1-(2-Aminomethyl-4,6-dimethylphenyl)-3-phenyltetrazolium-5-amide (**8**).

Compound **5** (18 mg, 0.057 mmole) was refluxed in concentrated hydrochloric acid (2 ml) for 2 hours. After being cooled to

room temperature, aqueous sodium hydroxide (10 N, 2 ml) was added and the product was extracted with dichloromethane. The solvent was removed *in vacuo* and the residue was extracted with hot hexane. Evaporation of the solvent left **8** (13 mg, 78%), yellow crystals; mp 92-94°; ir: 3420, 1620, 1462, 764 and 678 cm⁻¹; ¹H nmr (deuteriochloroform): δ 2.22 (3H, s, Me), 2.40 (3H, s, Me), 3.62-3.80 (2H, m, CH₂), 7.12 (1H, s, aryl-H), 7.22 (1H, s, aryl-H), 7.55-7.58 (3H, m, *m*- and *p*-H of Ph) and 8.04-8.09 (2H, m, *o*-H of Ph); ¹³C nmr (deuteriochloroform): δ 17.8 (Me), 21.3 (Me), 42.7 (CH₂), 119.9 (*o*-C of Ph), 127.6 (aryl-CH), 129.6 (*m*-C of Ph), 130.7 (aryl-CH), 130.8 (aryl-C or *i*-C of Ph), 131.1 (*p*-C of Ph), 136.4 (aryl-C or *i*-C of Ph), 136.7 (aryl-C or *i*-C of Ph), 141.4 (aryl-C or *i*-C of Ph), 141.8 (aryl-C or *i*-C of Ph) and 162.8 (C⁺); ms: m/z (EI, 70 eV) 294 (M⁺, 16%), 278 (6), 277 (8), 276 (100), 133 (16), 105 (10), 91 (9) and 77 (42, Ph); hrms: calcd. for C₁₆H₁₈N₆: 294.1592. Found M⁺: 294.1600. This compound is unstable and satisfactory elemental analysis data were not obtained.

1-[2-(*N*-Isopropylideneaminomethyl)-4,6-dimethyl]phenyl-3-phenyltetrazolium-5-amide (**9**).

Compound **8** (13 mg, 0.041 mmoles) dissolved in dichloromethane/acetone (10:1) was passed through an aminated silica gel column. The solvent was removed *in vacuo* to leave imine **9** (8 mg, 54%); ¹H nmr (deuteriochloroform): δ 1.81 (3H, s, Me), 1.94 (3H, s, Me), 2.21 (3H, s, Me), 2.39 (3H, s, Me), 4.37 (2H, AB q, J = 7.6, CH₂), 7.10 (1H, s, aryl-H), 7.22 (1H, s, aryl-H), 7.54-7.58 (3H, m, *m*- and *p*-H of Ph) and 8.06-8.11 (2H, m, *o*-H of Ph).

Acid Hydrolysis of **9**.

Compound **9** (8 mg, 0.024 mmole) was refluxed in concentrated hydrochloric acid (1 ml) for 2 hours. After being cooled to room temperature, aqueous sodium hydroxide (10 N, 1 ml) was added and the product was extracted with dichloromethane. The solvent was removed *in vacuo* to give **8** (7 mg, 99%).

X-ray Crystallography of **5**.

A crystal grown from acetonitrile was mounted in a glass capillary. All measurements were made on an Enraf-Nonius CAD4 diffractometer with graphite monochromated Mo-K α radiation. The structure was solved by direct methods [6] and expanded using Fourier techniques [7]. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were included but not refined. The detailed analysis results are listed in Tables 1-4.

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