9 on a 20% QF-1 column at 110 °C. Alternatively, 8 could be obtained as the major product by hydrogenation of 23 at high pressure and temperature over Raney nickel.[24b]

2,8-Dimethyl-*cis*-decahydroquinolines 16 and 17. Hydrogenation of 23 in concentrated HCl at 50 psi of H_2 and 70 °C until no further hydrogen uptake was observed[16] gave a mixture of ~80% 16, 17, and 10 and 20% starting material. The mixture was separated by preparative GC on the Carbowax–KOH column at 150 °C. After the decahydro compounds were collected, the starting material was recovered at a 200 °C column temperature. The cis compounds 16 and 17 were obtained in a ratio of ~2:1.

2-Methyl-8-*tert*-butyl-*trans*-decahydroquinolines. Reduction of 27 with sodium–ethanol gave a mixture of 8% 18, 31% 19, 47% 14, and 9% 13 together with traces of 12 and some unidentified compounds. The mixture was separated by column chromatography on aluminum oxide 90 (Merck, 70–230 mesh) with *n*-hexane as solvent. Compounds 18 and then 14 were eluted; addition of increasing amounts of ether gave 19 and 13. The compounds were obtained completely pure by submitting the samples to preparative gas chromatography (Carbowax–KOH, 160 °C). Hydrogenation of 24 at high pressure and temperature as for 22[15] gave a mixture of mainly 18 and 12 (ratio ~3:1) besides small amounts of other isomers. Separation by column chromatography (aluminum oxide, *n*-hexane) gave both isomers GC pure.

Compound 15 was obtained from *N*-nitroso-8β-*tert*-butyl-*trans*-decahydroquinoline[24b] by α-lithiation, α-methylation, and denitrosation of the resulting *N*-nitroso-15.[24c] The cis isomers 20 and 21 were also prepared from their *N*-nitroso derivatives.[24c]

The *N*-methyl derivatives 6m–15m were prepared by the Eschweiler–Clarke procedure.[30] Hydrochlorides were precipitated by passing gaseous HCl into the solutions of the amines in ether. The picrates were made by addition of an ethereal solution of picric acid to the solutions of the amines in ether and recrystallization from ethanol.

Acknowledgment. Part of this work was supported under NSF Grant CHE78-08713. F.W.V. thanks the Fonds zur Förderung der Wissenschaftlichen Forschung for support under Project No. 3241 and the Jubiläumsfonds der österreichischen Nationalbank for the funds for the purchase of an EM 360 spectrometer.

Registry No. 6, 18609-01-3; **6m**, 18609-11-5; **7**, 18610-37-2; **7m**, 18609-07-9; **8**, 73698-32-5; **8**·HCl, 75031-04-8; **8m**, 75031-05-9; **8m**·HCl, 75031-06-0; **9**, 75031-07-1; **9**·HCl, 75031-08-2; **9m**, 75031-09-3; **9m**·HCl, 75031-10-6; **10**, 75031-11-7; **10**·HCl, 75031-12-8; **10m**, 75031-13-9; **10m**·HCl, 75031-14-0; **11**, 75031-15-1; **11**·HCl, 75031-16-2; **11m**, 75031-17-3; **11m**·HCl, 75031-18-4; **12**, 75031-19-5; **12**·HCl, 75031-20-8; **12m**, 75031-21-9; **12m**·HCl, 75031-22-0; **13**, 75031-23-1; **13**·HCl, 75031-24-2; **13m**, 75031-25-3; **13m**·HCl, 75031-26-4; **14**, 75031-27-5; **14**·HCl, 75031-28-6; **14m**, 75031-29-7; **14m**·HCl, 75031-30-0; **15**, 75045-70-4; **15**·HCl, 75031-31-1; **15m**, 75031-32-2; **15m**·HCl, 75031-33-3; **16**, 75031-34-4; **17**, 75031-35-5; **18**, 75031-36-6; **19**, 75031-37-7; **20**, 75031-38-8; **21**, 75031-39-9; **22**, 91-63-4; **23**, 1463-17-8; **24**, 75031-40-2; **26**, 75031-41-3; **26** picrate, 75031-42-4; **27**, 75031-43-5; **27** picrate, 75031-44-6; **28**, 769-92-6; **30**, 1886-57-3; *N*-ethyl-2-*tert*-butylaniline, 75031-45-7.

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Reaction of Mesoionic Thiazolones with *m*-Chloroperbenzoic Acid. Oxidative Dipole Extensions

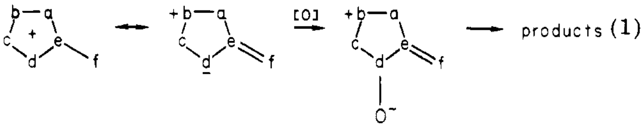
Tuvia Sheradsky* and David Zbaida

Department of Organic Chemistry, The Hebrew University, Jerusalem, Israel

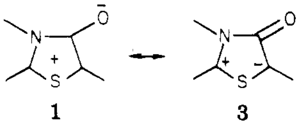
Received May 22, 1980

The oxidation of anhydro-4-hydroxy-2,3,5-triarylthiazolium hydroxides (1) with *m*-chloroperbenzoic acid gave (in primary alcohols) 2,5-dialkoxy-2,3,5-triarylthiazolidin-4-ones (5) and *N*-aroylthiobenzanilides (6). Oxidation of anhydro-5-hydroxy-3-methyl-2-phenylthiazolium hydroxide (2) gave 3-methyl-2-phenylthiazolidine-4,5-dione (8) and *N*-formyl-*N*-methylthiobenzamide (9). The products arise from initial oxidations at the carbon atoms at position 5 of 1 and position 4 of 2, and transformation modes to the final products are discussed. These oxidations are the first examples of oxidative dipole extensions.

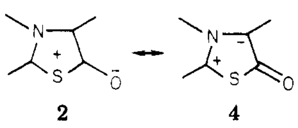
To date, no reactions of mesoionic systems or heteroaromatic betaines with peracids have been described.[1] The electrophilic nature of peracids is revealed in oxidations of amines,[2] sulfides,[3] and double bonds.[4] On this basis the peracid oxidation of dipolar species can be expected to occur at the nucleophilic terminus and to give oxidized intermediates which retain the dipolar character. We report the oxidation of two isomeric mesoionic systems (1[5,6] and 2[7]). Our expectation was that, according to eq 1,[8] the oxidation would occur specifically *on carbon* at position 5 of 1 and position 4 of 2, through the established[9,10] dipolar reactive forms 3 and 4, respectively.



(1)



1 3



2 4

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[5] Ohta, M.; Chosho, H.; Shin, G.; Ichimura, K. *Nippon Kagaku Zasshi* **1969**, *88*, 440; *Chem. Abstr.* **1964**, *61*, 14657.

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Table I. ^{1}H NMR (300 MHz) Chemical Shifts of Alkoxy Protons in 5a[b]

R	α-hydrogens	β-hydrogens	γ-hydrogens
CH_3	3.45, 3.75 (singlets)		
CH_2CH_3	very complex (over 40 lines)	1.46, 1.34, 1.32, 1.26 (triplets, J = 7)	
$CH_2CH_2CH_3$	4.14 and 3.88,[a] 3.66 and 3.50,[a] doublets (J = 8.4) of triplets (J = 7)	1.91, 1.79 (sextets, J = 7)	1.15, 1.01 (triplets, J = 7)

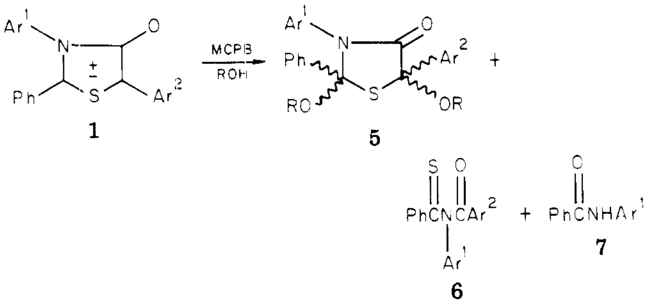
[a] Pairing was made by spin-decoupling. [b] δ values; J values in hertz.

Table II. Mass Spectral Data (m/e) of Compounds 5

		fragments (relative intensity)					
compd	M^+	$M^+ - OR$	$M^+ - 2OR$	**11**	**12**	**13**	Ar^2CO^+
5a, R = Me	391 (1)	360 (7)	329 (1)	211 (98)	293 (82)	152 (5)	105 (100)
5a, R = Et	419 (0.5)	374 (3)	329 (0.5)	225 (46)	253 (45)	166 (2.5)	105 (100)
5a, R = *n*-Pr	447 (4)	388 (8)	329 (0.5)	239 (26)	267 (52)	180 (42)	105 (100)
5b, R = Me		394 (3)	363 (1)	245 (49)	273 (54)	152 (3)	105 (100)
5c, R = Me	425 (0.3)	394 (3)	363 (0.5)	211 (65)	273 (30)	187 (16)	139 (100)
5d, R = Me	425 (0.5)	394 (6)	363 (9)	211 (85)	273 (52)	187 (16)	139 (100)
5d, R = Et	453 (1)	408 (5)	363 (1)	225 (36)	284 (40)	201 (10)	139 (100)

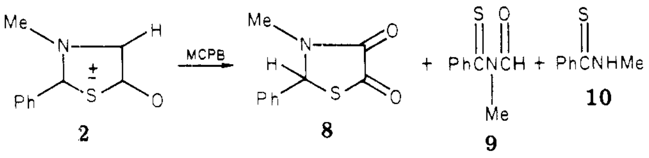
Results

The reaction of anhydro-4-hydroxy-2,3,5-triphenylthiazolium hydroxide (**1a**) with 1 equiv of *m*-chloroperbenzoic acid (MCPB) in methanol proceeded very rapidly (disappearance of the red color) and afforded, after chromatography, 2,5-dimethoxy-2,3,5-triphenylthiazolidin-4-one (**5a**, 45%), *N*-benzoylthiobenzanilide (**6a**, 12%), and benzanilide (**7a**, 25%). Oxidation in ethanol and in *n*-propanol proceeded similarly to give the corresponding 2,5-dialkoxythiazolidones **5** together with **6** and **7**. Three chloro derivatives of **1a** (**1b–d**) reacted in the same manner. In methylene chloride the oxidation of **1a** gave only **6a** (29%) and **7a** (50%).

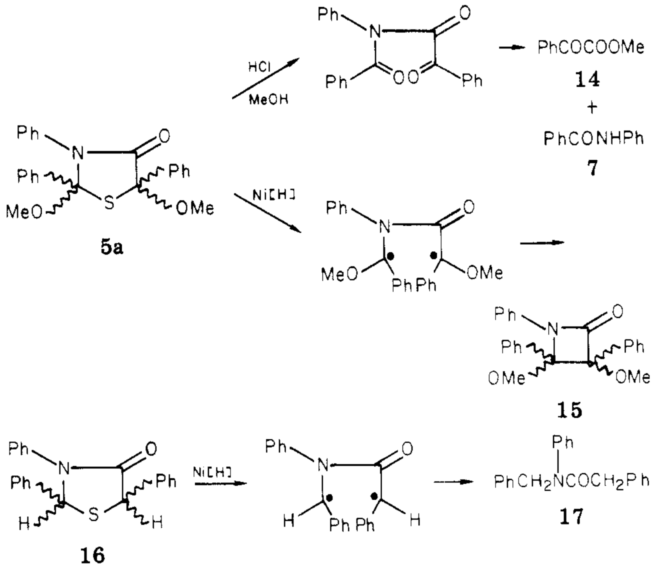


a, $Ar^1 = Ar^2$ = Ph; b, Ar^1 = 4-ClPh, Ar^2 = Ph; c, Ar^1 = Ph, Ar^2 = 2-ClPh; d, Ar^1 = Ph, Ar^2 = 4-ClPh; R = Me, Et, *n*-Pr

The oxidation of anhydro-5-hydroxy-3-methyl-2-phenylthiazolium hydroxide (**2**) with 1 equiv of MCPB in ethanol also afforded three products, identified as 3-methyl-2-phenylthiazolidine-4,5-dione (**8**, 33%), *N*-formyl-*N*-methylthiobenzamide (**9**, 28%), and *N*-methylthiobenzamide (**10**, 25%).

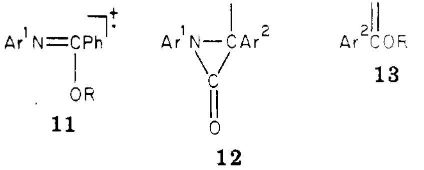


Scheme I



Structural Assignments. Compounds **6**, **7**, and **10** were identified by direct comparisons with authentic samples. The structure of **9** (C_9H_9NOS) was established by the spectra which displayed an IR carbonyl absorption at 1690 cm^{-1} and three NMR singlets at δ 3.60 (3 H, methyl) 7.38 (5 H, phenyl), and 9.00 (1 H, formyl). Compounds **5** were identified by both spectral and chemical means. The IR carbonyl peaks appeared at 1700–1710 cm^{-1} (five-membered cyclic amides). The NMR spectra showed only aromatic and alkoxy protons with appreciable nonequivalence of the α-methylene hydrogens. The NMR also suggests that the dimethoxy and the di-*n*-propoxy derivatives were obtained as single stereoisomers, while the diethoxy compounds were 1:1 isomer mixtures (Table I). This was also evident from the wide melting range of the diethoxy derivatives.

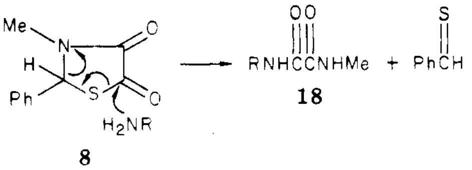
The positions of the two alkoxy groups were established from the mass spectra, which included the significant fragments represented as **11–13** (Table II).



Treatment of **5a** (R = Me) with methanolic hydrogen chloride gave benzanilide (**7a**) and methyl benzoylformate (**14**). Raney nickel desulfurization gave, surprisingly, only the β-lactam **15** (single stereoisomer). Ring contractions of this type have been encountered previously as side reactions only.[11,12] A possible reason for the formation of

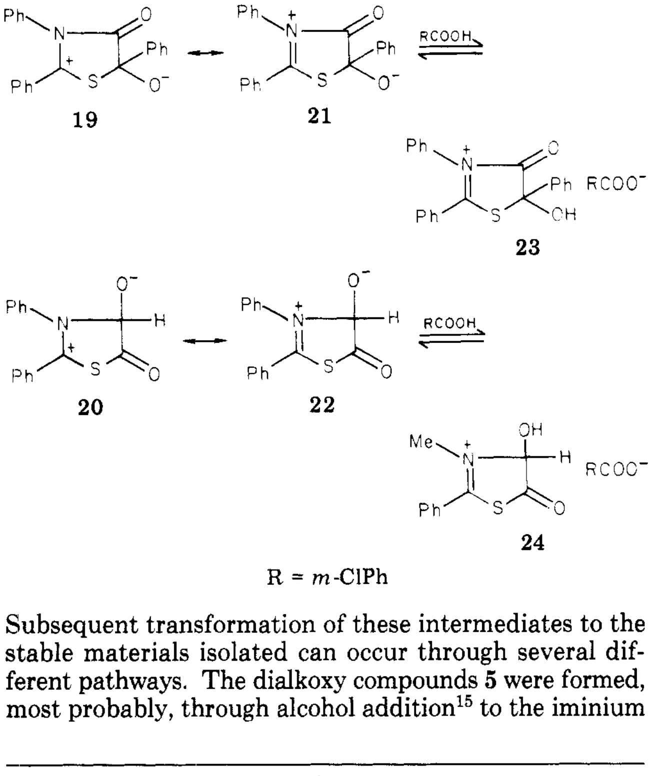
15 is the overcrowding at the radical centers of the intermediate, which did not allow contact with the catalyst surface and promoted radical coupling. For comparison we have desulfurized the thiazolone **16**[13] under the same conditions and it gave only **17**[12] with no trace of β-lactams (Scheme I). The available data, however, do not allow determination of the stereochemistry of **5** and **15**.

The main tool for the identification of **8** was ^{13}C NMR. The three ring carbons resonated at (parts per million from CDCl$_3$) 185.12 (C-4), 158.15 (C-5), and 61.82 (C-2, coupled to H). The assignment was also supported by ^{1}H NMR which showed three singlets at δ 3.03 (3 H, N-Me), 5.93 (1 H, C-H), and 7.40 (5 H, Ph) and the IR spectrum which showed (CHCl$_3$ solution) two carbonyl peaks at 1710 and 1720 cm^{-1}. The parent peak in the mass spectrum was at *m/e* 122 (PhCHS$^+$). Chemical proof for structure **8** was obtained by its reaction with primary amines, which gave *N*-alkyl-*N*-methyloxamides (**18**).



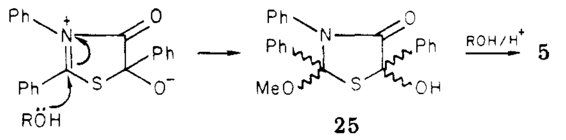
Discussion

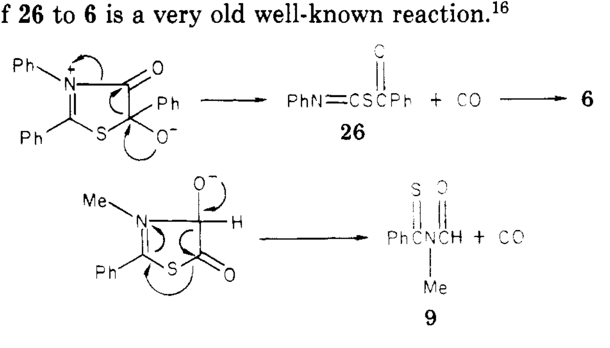
The products obtained clearly arise from oxidations at the anionic centers, as indicated in the introduction.[14] The initially formed intermediates would thus be the extended dipoles **19** and **20**, better represented in the zwitterionic forms **21** and **22**, and possibly also as the salts **23** and **24**.



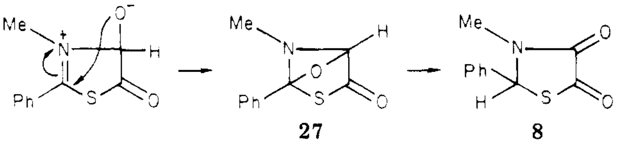
R = *m*-ClPh

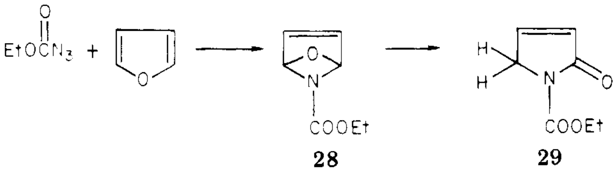
Subsequent transformation of these intermediates to the stable materials isolated can occur through several different pathways. The dialkoxy compounds **5** were formed, most probably, through alcohol addition[15] to the iminium ion portion of the molecule, followed by acid-catalyzed transformation of the hemiketal **25** to a ketal. Compounds **6** and **9** were formed in an identical process of carbon monoxide elimination. The spontaneous rearrangement of **26** to **6** is a very old well-known reaction.[16]





The formation of **8** involves stabilization through hydrogen transfer from C-4 to C-2, and a likely pathway is a cyclization to the bicyclic intermediate **27** and ring cleavage with hydrogen transfer. A similar interpretation was offered by Hafner[17] for the formation of **29** in the addition of carbethoxynitrene to furan, which probably proceeds through **28**.





Conclusions. The carbon oxidations at the predicted positions indicate that oxidative dipole extensions indeed took place, and a broader study of dipoles oxidations can now be undertaken. This conclusion is not affected by the fact that in the specific cases studied here the problems of the stereochemistry of **5**, the course of hydrogen transfer in the formation of **8**, and the ultimate origins of the cleavage products **7** and **10** remain unsolved.

Experimental Section

Melting points were taken on a Thomas-Hoover apparatus. IR spectra (Nujol mulls) were recorded on a Perkin-Elmer 157 spectrometer and NMR spectra on a Brucker WH-300 spectrometer. Mass spectra were recorded on a Varian MAT-311 instrument.

Oxidation of 1a. To a solution of anhydro 4-hydroxy-2,3,5-triphenylthiazolium hydroxide (**1a**, 658 mg, 2 mmol) in methanol (500 mL) was added *m*-chloroperbenzoic acid (2 mmol). After the solution was stirred for a few minutes the red color disappeared. The solution was evaporated and the solid residue was chromatographed on silica gel (50 g). Elution with benzene gave *N*-benzoylthiobenzanilide (72 mg, 12%), mp 109 °C, identical with authentic material obtained by benzoylation of thiobenzanilide.[16] Further elution with benzene-chloroform (1:3) gave 350 mg (45%) of 2,5-dimethoxy-2,3,5-triphenylthiazolidin-4-one (**5a**, R = Me),

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[14] An alternative pathway which involves oxidation of sulfur followed by a Pummerer-type rearrangement was suggested by a referee. This can however, be excluded in light of our recent results on oxidation of compounds which do not contain sulfur.

[15] Bohme, H.; Hartke, K. *Chem. Ber.* **1963**, *96*, 600, 604.

[16] Jamieson, G. S. *J. Am. Chem. Soc.* **1904**, *26*, 177.

[17] Hafner, K.; Kaiser, W. *Tetrahedron Lett.* **1964**, 2185.

Table III. 2,5-Dialkoxy-2,3,5-triarylthiazolidin-4-ones (5)

compd, R =	mp	% yield	formula[b]
5a, Me	168	45	$C_{23}H_{21}NO_3S$
5a, Et	121-140[a]	49	$C_{25}H_{25}NO_3S$
5a, *n*-Pr	138	41	$C_{27}H_{29}NO_3S$
5b, Me	145	42	$C_{23}H_{20}ClNO_3S$
5c, Me	174	43	$C_{23}H_{20}ClNO_3S$
5c, Et	48-80[a]	43	$C_{25}H_{24}ClNO_3S$
5d, Me	149	47	$C_{23}H_{20}ClNO_3S$

[a] Mixture of stereoisomers. [b] Satisfactory analytical values (±0.3% for C, H, N, Cl) were reported for all compounds in the table.

mp 168 °C. Further elution with chloroform gave first benzanilide (94 mg, 24%), followed by an acidic fraction, mainly *m*-chlorobenzoic acid.

In an alternative procedure, the evaporation residue was dissolved in chloroform (50 mL), washed with saturated $NaHCO_3$ solution (3 × 150 mL), dried, and evaporated. Crystallization of the residue gave directly compound **5** (350 mg). Chromatography of the mother liquor gave **6** and **7**.

Other oxidations of compounds **1** in alcohols were carried out similarly. Data of compounds **5** obtained are listed in Table III.

3,4-Dimethoxy-1,3,4-triphenylazetidin-2-one (15). Raney nickel (3 g) was added to a solution of **5a** (R = Me, 375 mg) in ethanol (50 mL). After being stirred at room temperature for 20 min the mixture was filtered and evaporated and the residue was crystallized from ethanol to give 88 mg (31%) of **15**: mp 163 °C; IR ν_{max} 1750 cm^{-1} (C=O); NMR δ 3.50, 3.84 (s, 3 H each, OCH_3), 7.1–7.8 (m, 15 H, Ph); mass spectrum, *m/e* (relative intensity) 359 (4, M^+), 240 (3, PhC(OMe)=C(OMe)Ph), 211 (40, PhN=C(OMe)Ph), 119 (6, PhNCO), 105 (100, PhCO).

Anal. Calcd for $C_{23}H_{21}NO_3$: C, 76.86; H, 5.89; N, 3.90. Found: C, 76.46; H, 6.00; N, 3.66.

Oxidation of 2. To a solution of anhydro-5-hydroxy-3-methyl-2-phenylthiazolium hydroxide (**2**, 382 mg, 2 mmol) in ethanol (150 mL) was added *m*-chloroperbenzoic acid (2 mmol). After being stirred for 5 min the solution was evaporated and the residue chromatographed on silica gel. Elution with benzene gave 110 mg (28%) of *N*-formyl-*N*-methylthiobenzanilide (**9**), mp 70–71 °C (see text for spectral properties).

Anal. Calcd for C_9H_9NOS: C, 60.33; H, 5.06; N, 7.82; S, 17.86. Found: C, 60.11; H, 5.14; N, 7.66; S, 17.62.

Further elution with benzene gave 83 mg (25%) of *N*-methylthiobenzamide (**10**), mp 77 °C (lit.[18] mp 79 °C). Elution with chloroform gave 144 mg (33%) of 3-methyl-2-phenylthiazolidine-4,5-dione (**8**), mp 155 °C (from ethanol) (see text for spectral properties).

Anal. Calcd for $C_{10}H_9NO_2S$: C, 57.97; H, 4.38; N, 6.76; S, 15.45. Found: C, 58.27; H, 4.48; N, 6.53; S, 15.23.

Reaction of 8 with Primary Amines. A solution of **8** (60 mg) and benzylamine (40 mg) in benzene (10 mL) was refluxed for 2 min and evaporated. Crystallization of the residue from ethanol afforded 53 mg (85%) of *N*-benzyl-*N*-methyloxamide, mp 185 °C (lit.[19] mp 184–185 °C).

In the same manner the reaction with cyclohexylamine gave 90% *N*-cyclohexyl-*N*-methyloxamide, mp 214 °C.

Anal. Calcd for $C_9H_{16}N_2O_2$: C, 58.67; H, 8.75; N, 15.20. Found: C, 58.75; H, 8.61; N, 15.10.

Registry No. 1a, 18100-80-6; **1b**, 68236-17-9; **1c**, 73308-33-5; **1d**, 59208-06-9; **2**, 1280-28-0; **5a** (R = Me), 75111-01-2; **5a** (R = Et), isomer 1, 75102-33-9; **5a** (R = Et), isomer 2, 75102-34-0; **5a** (R = *n*-Pr), 75102-35-1; **5b** (R = Me), 75102-36-2; **5c** (R = Me), 75102-37-3; **5c** (R = Et), isomer 1, 75102-38-4; **5c** (R = Et), isomer 2, 75102-39-5; **5d** (R = Me), 75102-40-8; **5d** (R = Et), isomer 1, 75102-41-9; **5d** (R = Et), isomer 2, 75102-42-0; **6a**, 17570-85-3; **7a**, 93-98-1; **8**, 75102-43-1; **9**, 75102-44-2; **10**, 5310-14-5; **15**, 75102-45-3; **18** (R = CH_2Ph), 7666-51-5; **18** (R = cyclohexyl), 75102-46-4; benzylamine, 100-46-9; cyclohexylamine, 108-91-8.

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[18] Sachs, F.; Loevy, H. *Chem. Ber.* **1904**, *37*, 874.

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Reactions of 2-Fluoro-2-nitro-1,3-propanediol. Trifluoromethanesulfonates and 3-Fluoro-3-nitrooxetane[1]

Phillip T. Berkowitz and Kurt Baum*

Fluorochem, Inc., Azusa, California 91702

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The reaction of diethyl fluoronitromalonate with base and formaldehyde provided a convenient synthesis of 2-fluoro-2-nitro-1,3-propanediol. This diol reacted with triflic anhydride to give 2-fluoro-3-hydroxy-2-nitro-1-propyl triflate and 2-fluoro-2-nitro-1,3-propylene ditriflate. The monotriflate reacted with base to give 3-fluoro-3-nitrooxetane. The ditriflate underwent displacement reactions with sodium azide, 2-fluoro-2,2-dinitroethanol, and methanol. Reactions of 3-fluoro-3-nitrooxetane with strong acids resulted in ring opening to give 3-substituted 2-fluoro-2-nitropropanols. The oxetane was polymerized with phosphorus pentafluoride. Triflates derived from 2,2-dinitro-1,3-propanediol and 2-(hydroxymethyl)-2-nitro-1,3-propanediol did not cyclize.

Primary 2-nitro and 2,2-dinitro alcohols readily undergo the reverse Henry reaction under basic conditions to give nitronate salts and formaldehyde.[2] This reaction is markedly inhibited by a fluorine α to nitro as a manifestation of the "fluorine effect" or the destabilization of a nitronate salt by an α fluorine.[3] Thus, 2-fluoro-2,2-dinitroethanol can be alkylated under basic conditions,[4–6] and 2-fluoro-2-nitro-1,3-propanediol has even been reported to give a stable dialkoxide salt. In order to explore further the chemistry of 2-fluoro-2-nitro-1,3-propanediol, we have developed an improved method for its preparation, and the present paper describes reactions of its trifluoromethanesulfonates (triflates).

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