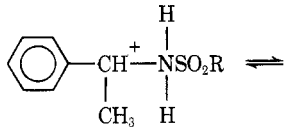
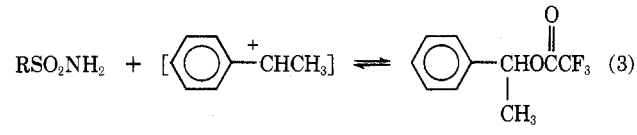
N-alkylsulfonamides such as **19** have been prepared in low yield in concentrated sulfuric acid from primary sulfonamides and alcohols.[10]





In the case of benzamides, the scope of the dealkylation reaction is more limited and only tertiary benzamides cleave under our reaction conditions. Although no cleavage was observed overnight with *tert*-butylbenzamide (**30**), its *N*-methyl derivative **23**, prepared by methylation of **30** with methyl iodide, has a half-life of 2.1 min in trifluoroacetic acid at 37°.

Experimental Section

All new compounds were identified by nmr and ir spectra on Varian A-60A and Beckman IR-18A spectrometers, respectively. Corrected melting points were determined for all new compounds on a Thomas-Hoover capillary apparatus. Elemental analyses were determined with an F & M 185 analyzer by the Physical-Analytical Department, Mead Johnson & Co.

Rate Measurements. Cleavage rates of the amides were measured with a Varian T-60 nmr spectrometer. A sample of the amide was added to prewarmed (37°) trifluoroacetic acid, and this solution was immediately inserted into the pretuned instrument. Scans of the *tert*-butyl region were taken every 30 sec for the first 5 min and every 1 min thereafter until the cleavage was 95% complete. The concentration of starting amide [a] at time t is proportional to the area under the peak due to the *tert*-butyl group in the nmr spectrum. The half-life, $t^{1/2}$, was obtained from the slope of a plot of $-\log$ [a] *vs.* t.

$$t^{1/2} = \log 2/\text{slope}$$

The following experiments represent typical experimental procedure.

Sulfamoylation. *N*-(4-Acetylphenyl)-*N'*-*tert*-butylsulfamide (5). *tert*-Butylsulfamoyl chloride (3.4 g, 0.02 mol) was added dropwise to a solution of 4'-aminoacetophenone (2.7 g, 0.02 mol) and triethylamine (2.0 g, 0.02 mol) in 1.5 l. of ether at −50°. The mixture was stirred for 3 hr during which time it was allowed to warm to room temperature. The mixture was made acidic with ethanolic hydrogen chloride, and the insolubles were collected. The insolubles were washed with water and recrystallized from isopropyl alcohol to give 4.2 g (78%) of **5**.

Sulfonylation. *N*-*tert*-Butyl-4-nitrobenzenesulfonamide (20). A solution of 4-nitrobenzenesulfonyl chloride (22.1 g, 0.10 mol) in CH_2Cl_2 was added dropwise with cooling to a solution of *tert*-butylamine (7.3 g, 0.10 mol) in 50 ml of pyridine. The mixture was stirred overnight at room temperature and then concentrated *in vacuo*. Ether was added to the residue and the mixture extracted with dilute hydrochloric acid. The ether solution was dried over $MgSO_4$ and concentrated *in vacuo*. The residue was recrystallized from benzene to give 11.2 g (44%) of **20**.

Trifluoroacetic Acid Cleavage. *N*-4-Methoxyphenylsulfamide (10). A solution of **4** (3.0 g, 0.011 mol) in 50 ml of trifluoroacetic acid was stirred for 5 hr at room temperature. The solution was concentrated *in vacuo* and the residue recrystallized from ethanol to give 1.5 g (71%) of **10**.

Methylation. *N*-(4-Acetylphenyl)-*N'*-*tert*-butyl-*N,N'*-dimethylsulfamide (14). A mixture of **5** (2.7 g, 0.01 mol), methyl iodide (4.3 g, 0.03 mol), and potassium carbonate (8 g) in 30 ml of acetone was refluxed for 48 hr. Additional methyl iodide was added after 20 hr and 44 hr (0.01 and 0.005 mol, respectively). The insolubles were removed, and the solution was concentrated *in vacuo*. Ether was added, and the insolubles were removed. Cooling furnished pure **14**, 2.2 g (67%).

Registry No.—3, 49689-92-1; **4**, 49689-93-2; **5**, 49689-94-3; **6**, 49689-95-4; **7**, 49689-96-5; **8**, 49689-97-6; **9**, 15959-53-2; **10**, 10539-83-0; **11**, 49690-00-8; **12**, 49690-01-9; **13**, 710-15-6; **14**, 49690-03-1; **15**, 49690-04-2; **16**, 2512-23-4; **17**, 2849-81-2; **18**, 49690-07-5; **19**, 2512-24-5; **20**, 49690-09-7; **21**, 49690-10-0; **22**, 49690-11-1; **23**, 49690-12-2; $RNHSO_2Cl$, 33581-95-2 (R = *t*-C_4H_9), 7778-42-9 (R = H); NH_2R', 62-53-3 (R' = C_6H_5), 104-94-9 (R' = C_6H_4-4-OCH_3), 99-92-3 (R' = C_6H_4-4-$COCH_3$), 95-76-1 (R' = C_6H_3-3,4-Cl_2), 64-04-0 (R' = $CH_2CH_2C_6H_5$), 7664-41-7 (R' = H); $RNHR'$, 75-64-9 (R = *c*-C_4H_9, R' = H), 14610-37-8 (R = *t*-C_4H_9, R' = CH_3), 21230-07-9 (R = *i*-C_3H_9, R' = $SO_2C_6H_4$-4-CH_3), 32512-24-6 (R = $C_6H_5CH(CH_3)$, R' = CH_3); $ClSO_2R''$, 124-63-0 (R'' = CH_3), 98-59-9 (R'' = C_6H_4-4-CH_3), 98-09-9 (R'' = C_6H_5), 98-74-8 (R'' = C_6H_4-4-NO_2); trifluoroacetic acid, 76-05-1.

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Central Nervous System Depressants. 12. The Reaction of Chlordiazepoxide with Methyl Isocyanate

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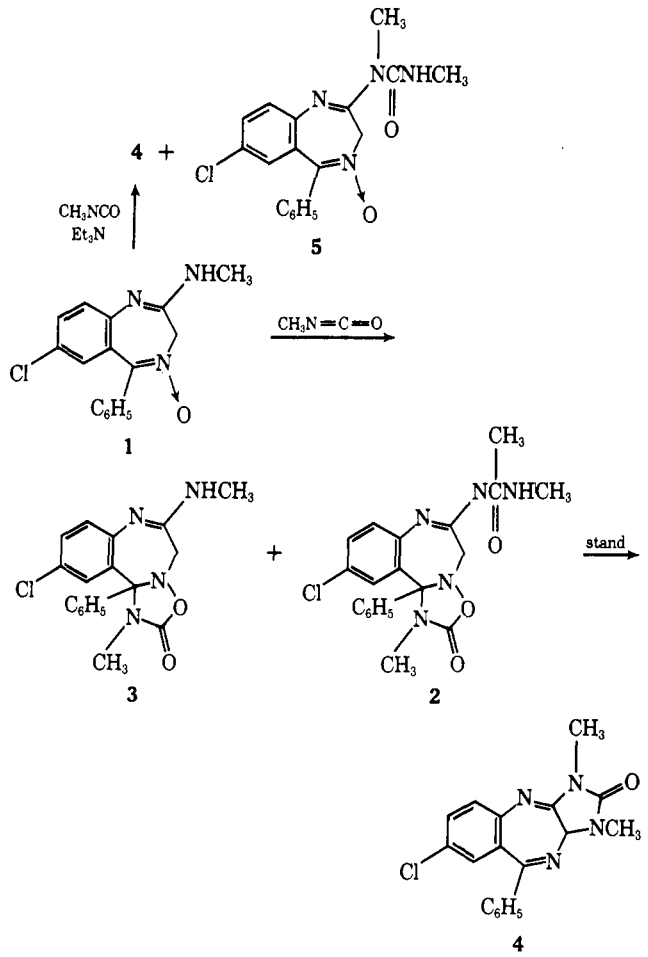
Received August 27, 1973

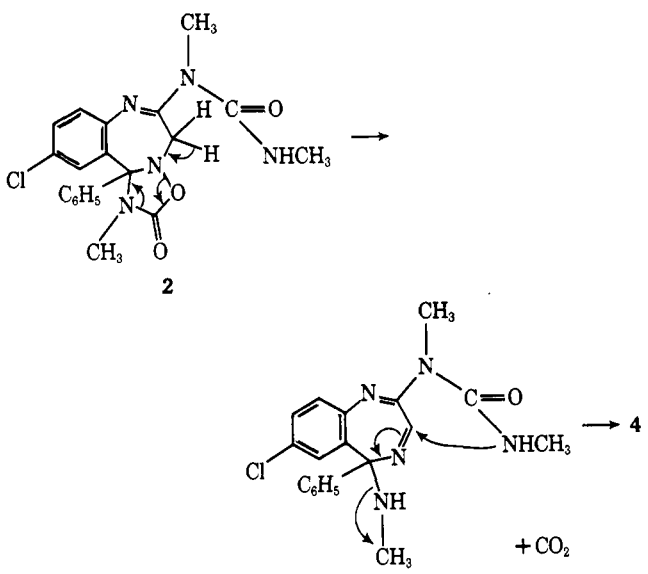
Although the reaction of isocyanates with other benzodiazepines is in the literature,[1,2] the reaction of 7-chloro-2(methylamino)-5-phenyl-3*H*-1,4-benzodiazepine 4-oxide (chlordiazepoxide) (**1**) with isocyanates does not seem to have been reported. We have investigated this reaction and its unexpected course prompts us to report the results.

Treatment of chlordiazepoxide[3] (**1**) with an excess of methyl isocyanate yielded essentially one product as shown by tlc. This contained two isocyanate moieties and was assigned the structure **2**. The oxadiazolo compound **3** was isolated as a minor component from another run. The product ratio suggests that the 1,3 addition of the isocyanate to the nitrone is more rapid than the reaction with the $-NHCH_3$ group. The opposite may be true of the 2-NH_2 analog. Meguro, *et al.*,[1] report only the simple *N*-methylurea derivative from the reaction of methyl isocyanate with 7-chloro-2-amino-5-phenyl-3*H*-1,4-benzodiazepine 4-oxide, but they gave no yield.

Compound **2** proved to be unstable. Repeated crystallization lowered the melting point, but a run, quickly crystallized from CH_2Cl_2–cyclohexane, gave material with an essentially correct analysis. On standing for many months, the elements of $MeNH_2$ and CO_2 split out and the cyclic urea **4** was formed essentially quantitatively. This conversion seemed to take place faster in a bottle than in an open dish and $CH_3NHCOO^-CH_3NH_3^+$ sublimed into the upper part of the bottle. That this conversion is base catalyzed was shown by adding Et_3N to the reaction of **1** with CH_3NCO, giving an increased yield of **4**, and **5** as a minor product. It is believed that the reason the conversion into **4** took place more rapidly in the bottle was due to the $MeNH_2$ being retained in the mixture.

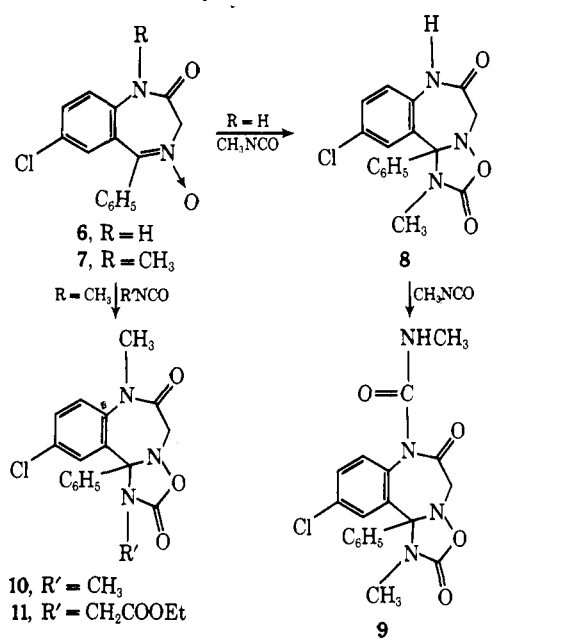


A reasonable mechanism for this conversion is postulated as



Another possible mechanism might involve a Polonovski rearrangement with 7-chloro-3-hydroxy-2-(methylamino)-5-phenyl-3*H*-1,4-benzodiazepine as an intermediate. This was prepared by the method of Bell, *et al.*,[4] and allowed to react with MeNCO. Several products were obtained as shown by tlc, none of which were 4.

In order to further establish the presence of the oxadiazolo ring in 2 and 3, MeNCO was allowed to react with two benzodiazepine 4-oxides, 6 and 7, which do not have the 2-amino group. The oxadiazolo derivatives 8, 9, and 10 were obtained in excellent yields, and spectral properties were consistent with those of 2 and 3. The fact that 8 was isolated as an intermediate in the formation of 9 confirms the high reactivity toward isocyanates of the 4,5 double bond of the 4-oxides. To show the generality of this reaction, 7 was allowed to react with ethyl isocyanatoacetate giving the corresponding carboethoxymethyl oxadiazolo compound 11 in excellent yield.



All these compounds were tested in the battery of tests generally used for our CNS depressants.[2] All showed some activity, but none was as active as chlordiazepoxide.

Experimental Section[5]

10-Chloro-5,11b-dihydro-1-methyl-6-(methylamino)-11b-phenyl[1,2,4]oxadiazolo[2,3-*d*][1,4]benzodiazepin-2(1*H*)-one (3) and 1-[10-Chloro-1,2,5,11b-tetrahydro-1-methyl-2-oxo-11b-phenyl-[1,2,4]oxadiazolo[2,3-*d*][1,4]benzodiazepin-6-yl]-1,3-dimethylurea (2). To a solution of 6.65 g (0.0214 mol) of chlordiazepoxide (base) (1) in 75 ml of tetrahydrofuran, under N_2, was slowly added 15 ml of methyl isocyanate. After stirring for 3 days at room temperature and refluxing for 2.5 hr, the solution was evaporated to dryness *in vacuo*. The residue was dissolved in PhH and on standing crystalline solid separated. This was collected and recrystallized from 2-propanol yielding 0.63 g (8.5%) of white crystals, mp 146–148° dec. This was found by ir, nmr, mass spectrum and analysis to be 3. The principal spectral bands are: ir (Nujol mull) 3340 (NH), 1760 (C=O), 1620, 1590, 1560 (C=N, C=C), 1245, 1210, 1115, 1000, 885, 835, 760, 705 cm^{-1} (arom and other); nmr ($CDCl_3$) δ 2.75 (s, 6, NCH_3), ab centered at 3.57 and 3.85 (2, J = −14 Hz, 5 CH_2), 5, 6 (br s, 1, NH) and between 5.7 and 7.6 (m, 8, arom H's); mass spectrum M^+ 356 (1 Cl).

Anal. Calcd for $C_{18}H_{17}ClN_4O_2$: C, 60.59; H, 4.80; Cl, 9.94; N, 15.70. Found: C, 60.50; H, 5.14; Cl, 10.08; N, 15.43.

The benzene filtrate from 3 was concentrated and diluted with cyclohexane giving 6.77 g of nearly white solid which was recrystallized from aqueous ethanol yielding 5.3 g (60%) of white crystalline solid, mp 141–143° dec. Tlc (SiO_2, 20% MeOH in PhH) indicated this was a nearly pure compound different from 3. In other runs, this same compound was obtained as the only product as shown by tlc (SiO_2, 60% EtOAc in cyclohexane). Rapid recrystallization from CH_2Cl_2–cyclohexane yielded 85% of 2, but repeated crystallization caused decomposition and lowered the melting point. It was found by ir, nmr, mass spectrum and analysis to have the structure 2. The principal spectral bands are: ir (Nujol mull) 3410 (NH), 1755, 1675 (C=O), 1620 (C=N), 1525 (amide II), 1235, 1075, 995, 850, 750, 700 cm^{-1} (arom and other); nmr ($CDCl_3$) δ 2.62 (d, 3, $NHCH_3$), 3.40 (s, 3, CH_3), ab centered at 3.75 and 4.25 (2, J = −11 Hz, 5 CH_2), between 7.0 and 7.65 (m, 8, arom H's), 8.65 (br s, 1, NH); mass spectrum M^+ (not observed) 369, 371, 354, 356, 338, 340 (1 Cl).

Anal. Calcd for $C_{20}H_{20}ClN_5O_3$: C, 58.04; H, 4.87; Cl, 8.57; N, 16.92. Found: C, 57.58; H, 5.03; Cl, 8.64; N, 16.87.

7-Chloro-3,10a-dihydro-1,3-dimethyl-9-phenylimidazo-[4,5-*b*][1,4]benzodiazepin-2(1*H*)-one (4). A sample of 2, recrystal-

lized from 2-propanol, was found by tlc (SiO_2, MeOH in $CHCl_3$), ir, nmr and analysis to be nearly pure, and on standing in an open dish for 8 months showed little change, but on standing for 2 yr in a sealed bottle crystals had sublimed into the upper part of the bottle. These crystals were found to be methylamine *N*-methylcarbamate salt ($CH_3NH_3^+$ CH_3NHCOO^-) by ir comparison with an authentic sample. The solid remaining in the bottom of the bottle was found by tlc (SiO_2, 60% EtOAc in cyclohexane or 20% MeOH in PhH) to be essentially all **4**. This was kept under vacuum (<0.1 mm) overnight at room temperature to remove any remaining *N*-methylcarbamate salt and then a 3.14-g sample was recrystallized from 2-propanol giving 3.09 g of white crystals, mp 204–208°. This is equivalent to an 80% overall yield from chlordiazepoxide. The assignment of structure **4** is based on ir, uv, nmr, mass spectrum, and analysis. The principal spectral bands are: ir (Nujol mull) 1750 (C=O), 1660, 1605 (C=N), 1325, 1220, 1040, 1010, 850, 835, 755, 705 cm^{-1} (arom and other); uv (EtOH) 218 mμ (ε 95.82) 240 (74.74), 260 (53.91), 285 (33.43), 332 (ε 6.30); nmr ($CDCl_3$) δ 3.21 and 3.27 (s, 6, CH_3), 4.87 (s, 1, CH), between 7.2 and 7.7 (m, 8, arom H's); mass spectrum M^+ 338 (1 Cl).

Anal. Calcd for $C_{18}H_{15}ClN_4O$: C, 63.81; H, 4.46; Cl, 10.46; N, 16.53. Found: C, 63.40; H, 4.38; Cl, 10.45; N, 16.51.

1-(7-Chloro-5-phenyl-3*H*-1,4-benzodiazepin-2-yl)-1,3-dimethylurea 4-Oxide (5) and 4. A solution of 3.0 g (0.01 mol) of chlordiazepoxide (base), 1.4 ml (0.01 mol) of Et_3N, and 5.9 ml (0.1 mol) of MeNCO in 50 ml of tetrahydrofuran was stirred, under N_2, at room temperature for 4.5 hr and allowed to stand at 0° for 11 days. Tlc (SiO_2, 60% EtOAc in cyclohexane) showed two spots, one moving the same as **4**. The mixture was evaporated *in vacuo*, dissolved in $CHCl_3$, washed with water, and again evaporated. The residue was crystallized first from EtOAc and then from EtOAc–$CHCl_3$ yielding 0.4 g (11%) of white crystalline **5**, mp 175–177° dec. This was shown to have the structure **5** by ir, uv, nmr, mass spectrum, and analysis. The principal spectral bands are: ir (Nujol mull) 3270 (NH), 1665 (C=O), 1610, 1545, 1510 (C=N/C=C or "amide II"), 1260, 1230, 1170, 1070, 850, 830, 765, 715, 695 cm^{-1} (arom and other); uv (EtOH) 243 mμ (ε 72.69), 277 (91.75), 320 (21.75); nmr ($CDCl_3$) δ 2.9 (d, 3, $NHCH_3$), 3.55 (s, 3, NCH_3), between 7.0 and 7.6 (m, 8, arom H's), 9.0 (br s, 1, NH); mass spectrum M^+ 356 (1 Cl).

Anal. Calcd for $C_{18}H_{17}ClN_4O_2$: C, 60.59; H, 4.80; Cl, 9.94; N, 15.70. Found: C, 60.39; H, 4.87; Cl, 10.09; N, 15.49.

The filtrate from the first crystallization of **5** was evaporated and crystallized from 2-propanol giving 0.93 g (27%) of yellow crystals whose ir was identical with that of **4** above.

10-Chloro-7,11b-dihydro-1-methyl-11b-phenyl[1,2,4]oxadiazolo[2,3-*d*][1,4]benzodiazepine-2,6(1*H*,5*H*)-dione (8) and 10-Chloro-7,11b-dihydro-1-methyl-7-(methylcarbamoyl)-11b-phenyl[1,2,4]oxadiazolo[2,3-*d*][1,4]benzodiazepine-2,6(1*H*,5*H*)-dione (9). A solution of 2.87 g (0.01 mol) of 7-chloro-1,3-dihydro-5-phenyl-2*H*-1,4-benzodiazepin-2-one 4-oxide (**6**) and 20 ml of MeNCO in 70 ml of tetrahydrofuran was stirred under N_2 at room temperature for 22 hr. The resulting solid was collected, yielding 1.4 g (41%) of white crystals, mp 213.5–214.5° dec. Recrystallization from 2-methoxyethanol gave white crystals with the same melting point. Ir, nmr, mass spectrum, and analysis showed this to have the structure **8**. The principal spectral bands are: ir (Nujol mull) 3220 (NH), 1740, 1690 (C=O), 1485, 1240, 1215, 815, 755, 700 cm^{-1} (arom and other); nmr (at 100° in DMF-d_7) δ 2.8 (s, 3, CH_3), 5.2 (br s, 2, CH_2), 8.2 (s, 1, NH), between 6.6 and 8.1 (m, 8, arom H's); mass spectrum M^+ 5343 (1 Cl).

Anal. Calcd for $C_{17}H_{14}ClN_3O_3$: C, 59.40; H, 4.10; Cl, 10.31; N, 12.22. Found: C, 59.37; H, 3.97; Cl, 10.42; N, 12.57.

To the filtrate from **8** was added 10 ml more of CH_3NCO and the solution was allowed to stand at room temperature for 6 days. Evaporation *in vacuo* gave a residue showing only one spot on tlc (SiO_2, 60% EtOAc in cyclohexane) moving differently from **8** and starting material. Crystallization from 2-propanol yielded 2.0 g (50%) of white crystals, mp 158–161° dec, which was found by ir, nmr, mass spectrum, and analysis to have the structure **9**. The principal spectral bands are: ir (Nujol mull) 3340 (NH), 1775, 1730, 1690 (C=O), 1505, 1190, 745, 700 cm^{-1} (arom and other); nmr ($CDCl_3$) δ 2.5 (d, 3, $NHCH_3$), 2.7 (s, 3, NCH_3), ab centered at 3.4 and 4.1 (s, J = −10 Hz, CH_2), between 7.2 and 7.75 (m, 8, arom H's); mass spectrum M^+ 400 (1 Cl).

Anal. Calcd for $C_{19}H_{17}ClN_4O_4$: C, 56.93; H, 4.28; Cl, 8.84; N, 13.98. Found: C, 56.98; H, 4.67; Cl, 8.90; N, 14.32.

10-Chloro-7,11b-dihydro-1,7-dimethyl-11b-phenyl[1,2,4]oxadiazolo[2,3-*d*][1,4]benzodiazepine-2,6(1*H*,5*H*)-dione (10). A solution of 6.0 g (0.02 mol) of 7-chloro-1,3-dihydro-1-methyl-5-phenyl-2*H*-1,4-benzodiazepin-2-one (**7**) and 25 ml of MeNCO in 100 ml of tetrahydrofuran under N_2 was stirred under reflux for 25 hr. Evaporation *in vacuo* gave a gum which crystallized from 2-propanol yielding 6.97 g (97.5%) of white crystals, mp 166–168° dec. Ir, nmr, mass spectrum, and analysis confirm the structure **10**. The principal spectral bands are: ir (Nujol mull) 1770, 1675 (C=O), 1595, 1485, 1120, 1075, 830, 760, 745, 705 cm^{-1} (arom and other); nmr ($CDCl_3$) δ 2.58 (s, 3, NCH_3), 2.70 (s, 3, NCH_3), ab centered at 3.5 and 4.55 (2, J = −10 Hz, CH_2), between 7.15 and 7.75 (m, 8, arom H's); mass spectrum M^+ 357 (1 Cl).

Anal. Calcd for $C_{18}H_{16}ClN_3O_3$: C, 60.43; H, 4.51; Cl, 9.91; N, 11.74. Found: C, 60.63; H, 4.90; Cl, 9.74; N, 11.34.

Ethyl 10-Chloro-1,2,5,6,7-11b-hexahydro-7-methyl-2,6-dioxo-11b-phenyl[1,2,4]oxadiazolo[2,3-*d*][1,4]benzodiazepineacetate (11). Similarly 6.0 g (0.02 mol) of **7** in 100 ml of tetrahydrofuran was treated under N_2 with 12.9 g (0.01 mol) of ethyl isocyanatoacetate, stirred under reflux for 7 hr, and allowed to stand at room temperature for 18 hr. The solution was filtered, evaporated at 50° (0.1 mm), mixed with xylene, and again evaporated. The resulting gum crystallized on triturating with ether and was recrystallized from 2-propanol yielding 7.67 g (95%) of white crystals, mp 160.5–162°. Ir, nmr, mass spectrum and analysis confirm the structure **11**. The principal spectral bands are: ir (Nujol mull) 1775, 1745, 1680 (C=O), 1485, 1310, 1225, 1185, 1170, 1145, 830, 790, 700 cm^{-1} (arom and other); nmr ($CDCl_3$) δ 1.05 (t, 3, CH_2CH_3), 2.53 (s, 3, NCH_3), between 3.4 and 4.5 (m, 6, CH_2's), between 7.15 and 7.75 (m, 8, arom H's); mass spectrum M^+ 429 (1 Cl).

Anal. Calcd for $C_{21}H_{20}ClN_3O_5$: C, 58.68; H, 4.69; Cl, 8.25; N, 9.78. Found: C, 58.33; H, 4.64; Cl, 8.54; N, 9.77.

Acknowledgments. The author wishes to thank The Upjohn Company Physical and Analytical Chemistry unit for analyses and spectral data; and Dr. Allan D. Rudzik and Mr. Walter Friis for biological screening of the compounds.

Registry No. 1, 58-25-3; **2,** 49626-75-7; **3,** 49626-76-8; **4,** 49626-77-9; **5,** 49626-78-0; **6,** 963-39-3; **7,** 2888-64-4; **8,** 49626-81-5; **9,** 49626-82-6; **10,** 49626-83-7; **11,** 49626-84-8.

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(3) The author thanks Hoffmann-La Roche, Inc., Nutley, N. J., for a generous sample.
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(5) Melting points were taken in capillary tubes with a partial immersion thermometer. Calibration of the apparatus against standard compounds showed no need for correction.

Preferential Complexation of One of the Diastereomers of 1,2-Diazido-1,2-di-*tert*-butylethane with an Europium Nuclear Magnetic Resonance Shift Reagent[1]

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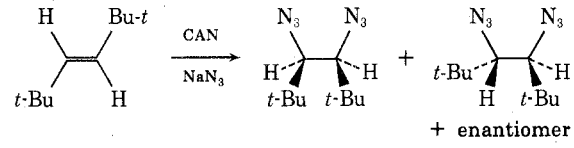
Received July 31, 1973

The reaction of olefins, ceric ammonium nitrate (CAN), and azide ion usually gives α-azido-β-nitratoalkanes;[2] however, α,β-diazidoalkanes have been obtained from sterically hindered olefins.[3] The reaction of 1 mol of *trans*-1,2-di-*tert*-butylethene with 2 mol of CAN and 1 mol of sodium azide in 10% aqueous acetonitrile at 0° gives a *ca.* 5% yield of comparable amounts of *meso*- and *dl*-1,2-diazido-1,2-di-*tert*-butylethane.[4] These diastereo-



+ enantiomer