

(E)-1,2-Di(2-pyridyl)ethene (5a) was prepared in an identical manner, bp 160–180° (1–2 mm), mp 117–119° (lit.⁹ mp 118–119°).

Bromination of (E)-1,2-Di(6-methyl-2-pyridyl)ethene. Method A.—To a stirred suspension of 1 g (4.8 mmol) of (E)-1,2-di(6-methyl-2-pyridyl)ethene in 10 ml of chloroform, a solution of bromine (770 mg, 4.8 mmol) in 10 ml of chloroform was added dropwise over 1 hr. During the addition, the crude dibromide slowly crystallized, yield 1.4 g (79%), mp 193–195° dec. Recrystallization of 6b from ethanol increased the melting point to 208–209° dec (lit.¹⁰ mp 194–196°).

Method B.—Bromination was identical except that acetic acid was used as solvent, yield 1.4 g (79%), mp 184-185° dec. Recrystallization of 6b from ethanol increased the melting point to 202-203° dec.

1,2-Di(2-pyridyl)1,2-dibromoethane (6a) was prepared (92%) by method A, mp 149-150° (lit.⁹mp 153-154°).

Di(6-methyl-2-pyridyl)acetylene (7b).—To a refluxing solution of potassium hydroxide (3 g) in absolute methanol (20 ml), the above solid dibromide 6b (1.0 g) was rapidly added in 50-mg (or less) quantities. Potassium bromide instantaneously precipitated. The suspension was refluxed for 30 min after completion of addition. The solvent was removed *in vacuo*. The residue was dissolved in ice-water and the organic material was extracted with ether, washed with a saturated salt solution, dried, and concentrated, affording the crude acetylene. Recrystallization from cyclohexane afforded 550 mg (95%) of the white, crystalline acetylene: mp 138-139°; nmr (CDCl₃) δ 2.56 (6-pyr-Me, s, 3 H), 7.1–7.9 (pyr H, m, 4 H); Raman (solid) 2215 cm⁻¹ (sym-acetylene).

Anal. Calcd for $C_{14}H_{12}N_2$: C, 80.74; H, 5.81; N, 13.45. Found: C, 80.45; H, 5.78; N, 13.35.

Di(2-pyridyl)acetylene (7a) was prepared in an identical manner in 93% yield, mp 69-71° (lit.⁸mp 69-70°).

Di(6-methyl-2-pyridyl)acetylene. Rapid Addition and Extended Reflux Times.—The solid dibromide 6b (4.5 g, 12.2 mmol) was added rapidly to a refluxing methanolic potassium hydroxide solution (6 g/20 ml). The reaction mixture was refluxed for 6 hr and cooled, and the potassium bromide precipitate [2.8 g (2.9 g theoretical weight)] was removed by filtration. The solvent was removed *in vacuo* and ice-water was added; the residue was extracted with ether, washed with a saturated salt solution, dried, and concentrated, affording a yellow oil, 2.9 g. Upon standing, the starting olefin 5b crystallized as white needles, yield 350 mg, mp 110-113° (recrystallized from low-boiling petroleum ether, mp 112-113°).

The crude oil (2.55 g) was distilled, affording three major fractions, bp 134-148° (0.6 mm), all of which possess different percentages, as quantitatively determined by vpc (10% OV 101 on Gas-Chrom Q 100-120 mesh, 6 ft \times 0.125 in.), of four major components, which were isolated by thick layer chromatography (Brinkmann silica gel PF, 50% ethyl acetate-50% cyclohexane, 500 mg).

The fastest moving component was the starting olefin 5b: $R_f 0.50$; yield 165 mg (33%), 35% via vpc; mp 111-113°; nmr (CDCl₃) δ 2.58 (6-pyr-**Me**, s) and 7.7 (trans **HC**=C**H**, s).

The second component was (Z)-1-methoxy-1,2-di(6-methyl-2pyridyl)ethene (9): R_1 0.42; yield 200 mg (40%), 45% via vpc; nmr (CDCl₂) δ 2.56 and 2.58 (6-pyr-Me, 2 s, 6 H), 3.73 (-OMe, s, 3 H), 6.82-8.02 (pyr H and >C=CH, m, 7 H); ir (neat) 1640, 1580, 1460, 1330, 1310, 1080, 1020 cm⁻¹.

The third component was di(6-methyl-2-pyridyl)acetylene (7b): $R_{\rm f}$ 0.25; yield 5 mg (1%), 1% via vpc: mp 133-137°.

The fourth component was (*E*)-1-methoxy-1,2-di(6-methyl-2-pyridyl)ethene (10): $R_{\rm f}$ 0.06; yield 65 mg (13%), 14% via vpc; nmr (CDCl₃) δ 2.42 (6-pyr-**Me**, s, 3 H), 2.53 (6-pyr-**Me**, s, 3 H), 3.88 (-OMe, 3 H), 6.08 (>C==CH, s, 1 H), 6.44-7.6 (pyr H, m, 6 H); ir (neat) 1640, 1580, 1450, 1210, 1140 cm⁻¹. Anal. Calcd for $C_{15}H_{16}N_2O$ (ethers): C, 74.97; H, 6.71; N, 11.66. Found: C, 74.86; H, 6.89; N, 11.58.

Hydrolysis of (E)-1-Methoxy-1,2-di(6-methyl-2-pyridyl)ethene (10).—A solution of the E vinyl ether (55 mg, 0.23 mmol) in methanol (10 ml) and 5 N HCl (5 ml) was refluxed for 8 hr. The solution was cooled, basified with a saturated sodium carbonate solution, and extracted with ether. The extract was washed with a saturated salt solution, dried over anhydrous magnesium sulfate, and concentrated *in vacuo*, giving (100%) the yellow crystalline ketone (as enol): mp 122-123°; nmr (CDCl₃) & 2.50 and 2.56 (6-pyr-Me, 2 s, 6 H), 6.7-7.8 (pyr H, m, 7 H); ir (KBr) 2500-3500 (-OH, broad), 1645, 1600, 1560, 1455, 1305, 830, 805, 765 cm⁻¹.

Anal. Calcd for $C_{14}H_{14}N_2O$: C, 74.31; H, 6.24; N, 12.38. Found: C, 74.33; H, 6.21; N, 12.40.

Hydrolysis of (Z)-1-methoxy-1,2-di(6-methyl-2-pyridyl)ethene (9) under identical conditions as above afforded the ketone, mp 122-123°, in 33% (nmr and isolated) yield and the unreacted Z enol ether.

Hydrolysis of the enol ethers 9 and 10 in the presence of olefin 5b under identical conditions as above initially afforded a viscous oil, which gave colorless crystals upon standing overnight. The crystals were recrystallized from ether-petroleum ether (bp 30-60°), affording an analytical sample of the tetrapyridyl ketone:¹² mp 178-179°; $R_{\rm f}$ 0.1 (50% ethyl acetate-50% cyclohexane); nmr (CDCl₃) δ 2.22, 2.41, 2.51, 2.59 (6-pyr-Me, 4 s, 3 H each), 2.9-3.15 (-CH₂-, J = 7, 11 Hz, 2 H), 4.3-4.8 (>CH²-, <math>J = 7, 7 Hz, 1 H), 6.4 (>CH¹CO-, J = 7 Hz, d, 1 H, echanged slowly with D₂O), and 6.6-7.7 (pyr H, m, 12 H); ir (KBr) 1706 (>C=O), 1592, 1575, 1457, 1378, 1328, 1292, 1263, 1152, 1093, 992 cm⁻¹.

Anal. Calcd for C28H28 N4O: C, 77.04; H, 6.47; N, 12.83. Found: C, 76.83; H, 6.72; N, 12.75.

1,2-Di(6-methyl-2-pyridyl)ethanone (11) was prepared (20.6%) according to the method of Goldberg, et al.,¹¹ from methyl 6-methylpicolinate¹⁴ and 6-methyl-2-picolyllithium, mp 122–123° (cyclohexane). The mother liquor afforded, after chromatography with cyclohexane-ethyl acetate (3:1), 1,2,3-tri(6-methyl-2-pyridyl)propan-1-ol:¹⁵ bp 167–169° (1.2 mm); nmr (CDCl₃) δ 2.40 [1,3-(2-pyr-Me), s, 6 H], 2.45 [2-(2-pyr-Me), s, 3 H], 3.25 (-CH₂-, d, J = 11 Hz), and 6.7-7.5 (pyr H, m, 9 H).

Registry No.—5a, 13341-40-7; 5b, 16552-23-1; 6a, 42296-31-1; 6b, 42296-32-2; 7a, 28790-65-0; 7b, 42296-34-4; 8, 39689-97-9; 9, 42296-37-7; 10, 42296-38-8; 11b, 42296-39-9; i, 42296-36-6; 6-methylpyridine-2-carboxaldehyde, 1122-72-1; 2,6-butadiene, 108-48-5; 2-picoline, 109-06-8; 2-pyridenecarboxaldehyde, 1121-60-4; 1,2,3-tri(6-methyl-2-pyridyl)propan-1-ol, 42447-96-1.

(14) W. Mathes, W. Sauermilch, and T. Klein, Chem. Ber., 86, 584 (1953).
(15) Attempted further purification was thwarted by decomposition to lutidine and 1,2-di(6-methyl-2-pyridyl)ethanone. Additional evidence on this point will be presented in a subsequent communication.

Preparation of Cis and Trans Isomers of 4-Phenylcyclohexyl and 4-Cyclohexylcyclohexyl Bromides

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Received July 5, 1973

Substituted cyclohexane compounds have been extensively used in the investigation of steric and conformational properties of functional groups, and for the elucidation of certain mechanistic aspects of chemical reactions. For a number of these investigations, bulky, inert functional groups having a strong prefer-

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ence for equatorial bonding to the cyclohexane ring in cases of 1,4 substitution such as *tert*-butyl have been utilized. 1,4-Substituted cyclohexanes containing either the phenyl or cyclohexyl group provide another such set of potentially useful compounds. In particular, the syntheses and assignments of the stereochemistry of the cis and trans isomers of 4-phenylcyclohexyl and 4-cyclohexylcyclohexyl bromides have been accomplished. These compounds possess a reactive bromide atom which permits ready access to other 4-phenyl and 4-cyclohexyl substituted cyclohexanes, especially by way of organometallic syntheses. Only mixtures of 4-phenylcyclohexyl bromides,^{2a} and 4-cyclohexylcyclohexyl bromides,^{2b} have been reported.

Results³

The route initially attempted for the preparation of the 4-phenylcyclohexyl bromide isomers was the reaction of Br_2 -CCl₄ with the silver salt of *trans*-4-phenylcyclohexanoic acid,⁴ but only para aromatic ring bromination occurred to produce 4-(4-bromophenyl)cyclohexanoic acid (1), mp 266-267°.

The successful route was the conversion of *trans*-4phenylcyclohexanol (2a) to a 4-phenylcyclohexyl halide, although this was far more difficult than expected. (Reaction with hydriodic acid, hydrobromic acid, anhydrous HI, SOCl₂, SOBr₂, PBr₅, PCl₅, POCl₃ or PCl₃ produced the inorganic esters or starting material.)

Successful reaction of 2a to produce mainly cis-4phenylcyclohexyl bromide (3b) in 82% yield was only accomplished with PBr₃ at 80° for 34 hr with intermittent addition of excess HBr gas. (trans-4-tert-Butylcyclohexanol responded readily at room temperature with PBr₃.) The trans bromide 3a could only be obtained pure and in reasonable amounts by reaction of trans-4-phenylcyclohexylmercuric bromide (4a) with Br_2 -pyridine complex. This reaction has been shown to be stereospecific by Jensen and Gale.⁵ 4a was prepared by recrystallization from benzene of the mixture of 4-phenylcyclohexylmercuric bromides (4a and 4b) obtained by reaction of HgBr₂ with the Grignard from pure 3b or the crude bromide product mixture from the PBr₃ reaction. (This 4-phenylcyclohexylmercuric bromide mixture was 88% 4a and 12%**4b**.) Pure *cis*-4-phenylcyclohexylmercuric bromide (**4b**) was obtained by recrystallization and chromatography of the residue from the benzene mother liquors formed in the isolation of 4a. 4b was cleaved by Br2-pyridine to give the previously obtained cis bromide **3b**. The infrared spectra were the same, and the mixture melting point was not depressed for 3b bromides from both sources.

The Grignard reagents from trans bromide **3a** and from cis bromide **3b** were carbonated, and the resultant acid mixture was esterified with diazomethane and analyzed by glc. **3a** gave 99.8% methyl*trans*-4-phenylcyclohexanoate (**5a**), and **3b** gave 98.2% trans methyl ester (5a). 5a had the same infrared spectra with no depression of the mixture melting point with authentic methyl *trans*-4-phenylcyclohexanoate prepared from the trans acid.⁴

3a (trans) and 3b (cis) were both reduced selectively in glacial acetic acid with H_2 -PtO₂ to *trans*-4-cyclohexylcyclohexyl bromide (6a) and *cis*-4-cyclohexylcyclohexyl bromide (6b), respectively. (Infrared spectra showed no aromatic hydrogen present in the products.) The reaction of an authentic sample of *cis*-4cyclohexylcyclohexanol (7b) with PBr₃ gave the trans bromide 6a. The same reaction with authentic trans alcohol 7a gave the cis bromide 6b.

6b (cis) and **6a** (trans) bromides produced by reaction with PBr₃ had the same ir, nmr, and physical properties as the corresponding **6b** and **6a** produced by the reduction of the **3b** (cis) and **3a** (trans) bromides.

The nmr absorptions of the bromide carbon methine hydrogen are summarized in the Experimental Section for the cyclohexyl bromides prepared in this investigation, and as expected the cis isomers show this hydrogen as a singlet downfield compared to the multiplet found for the trans isomers by Jensen and Berlin.⁶

Discussion

The cis-trans interrelationship of the two 4-phenylcyclohexyl bromide isomers and the two 4-cyclohexylcyclohexyl bromide isomers was shown by interconversion using organometallic derivatives and comparisons with authentic compounds.

The high preference of the Grignard should be noted for equatorial bonding considering that the *trans*-4phenylcyclohexanoic acid was formed in 98.2% or higher purity by the generally accepted stereospecific carbonation reaction. The corresponding reaction with HgBr₂ gave 88% trans isomer, but there are a number of possible intermediates formed during reaction that can explain this slightly lower preference.

Experimental Section

General.—All melting points are uncorrected. Nmr spectra were run in CS₂ with a Varian Model HR-60 nmr spectrometer. Ir spectra were obtained with a Beckman IR-4 recording spectrometer. Microanalyses were performed by the Microanalysis Laboratory, Department of Chemistry, University of California, Berkeley. Glc analyses were carried out on a 10 ft \times 0.25 in. column packed with 10% DEGS on 60–80 mesh Chromosorb W. All ir spectra were consistent with structure. 4-Cyclohexylcyclohexanol and 4-*tert*-butylcyclohexanol were donated by Dow Chemical Co. All solvents were reagent grade unless otherwise stated.

cis-4-Phenylcyclohexyl Bromide (3b).—To 248.3 g (1.410 mol) of trans-4-phenylcyclohexol⁷ at -70° was added 134 ml (1.410 mol) of PBr₃. The mixture was stirred, initially warmed to 80°, and maintained at this temperature for 34 hr with intermittent addition of HBr gas. The reaction mixture was cooled and poured into ice-isopentane. The isopentane solution was washed with concentrated H₃SO₄, aqueous NAHCO₈, and H₂O, then dried over anhydrous MgSO₄ and filtered. The solvent was removed to give 276.8 g (1.20 mol), 82% yield, of impure 4phenylcyclohexyl bromide (cis was the predominant isomer). **3b** was obtained pure by seven recrystallizations from pentane: mp 46.2-46.4°; ν_{max} 687 cm⁻¹ (C-Br); mrr (CS₂) δ 5.54 (s, =CBrH). Anal. Calcd for C₁₂H₁₅Br: C, 60.28; H, 6.32; Br, 33.40. Found: C, 60.19; H, 6.27; Br, 33.41.

trans-4-Phenylcyclohexylmercuric Bromide (4a).-Triply sub-

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⁽⁶⁾ F. R. Jensen and A. Berlin, unpublished results.

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limed Mg (43.7 g, 1.80 mol) and 200 ml of purified Et₂O under argon were stirred while 348 g (1.46 mol) of 3b in 800 ml of purified Et_2O was added at such a rate as to maintain reflux. The solution was stirred for 30 min after addition and then filtered. The Grignard solution was added slowly to 548 g (1.60 mol) of HgB_{12} in 200 ml of purified Et_4O and stirred overnight. (The slurry had the consistency of paste at this point.) The slurry was then poured into a large excess of H_2O and filtered, and the solid was air dried. The Et_2O filtrate was distilled, and the solid remaining was combined with the precipitate. The material was divided into two batches, and each was recrystallized from ca. 15 l. of benzene. The first crops were pure 4a, mp 182.1-183.0° dec (sealed tube), and weighed 158 g (0.36 mol). The benzene solution was evaporated down in stages, taking

various crops, and the final residue of 90 g was discarded. The last crops were recrystallized from EtOH to yield 130 g (0.226 mol) of impure 4b, mp 109-111°. There was also isolated 158 g (0.360 mol) of additional impure 4a. The total yield was 70%based on **3b**. Anal. Calcd for C₁₂H₁₅HgBr: C, 32.75; H, 3.44. Found: C, 32.47; H, 3.48.

cis-4-Phenylcyclohexylmercuric Bromide (4b).-Impure 4b from above was recrystallized from EtOH until the melting point was 120°. The material was then chromatographed using Woelm activity I neutral Al₂O₃ with Et₂O-benzene as eluent, followed by recrystallization from EtOH, mp 134.0-134.3° Anal. Calcd for C12H15HgBr: C, 32.75; H, 3.44. Found: C. 32.71: H. 3.44.

trans-4-Phenylcyclohexyl Bromide (3a).—After 132.2 g (0.301 mol) of 4a was dissolved in 500 ml of pyridine, a solution of 15.5 ml (0.301 mol) of Br_2 in 100 ml of pyridine was added slowly with stirring over a 15-min period at 25° with cooling. The solution was cooled to 15° for 1 hr and then poured into a mixture of ice, 200 ml of pentane, and 1200 ml of 6 N HCl. The organic layer was separated, washed with NaHSO₂ solution, washed several times with H₂O, and dried over anhydrous MgSO₄. After removal of the pentane, 54.2 g (76% yield) was obtained. After recrystallization from pentane, the melting point was 62.8-63.2°, $\nu_{\rm max}$ 687 cm⁻¹ (C-Br), nmr δ 4.66 (m, =-CBrH). Anal. Calcd for C₁₂H₁₅Br: C, 60.28; H, 6.32; Br, 33.40. Found: C, 60.04; H, 6.45; Br, 33.43.

Reaction of the Grignard Reagent Prepared from Pure cis-4-Phenylcyclohexyl Bromide (3b) with Mercuric Bromide.—3b (12.0 g, 0.0502 mol) in 40 ml of Et₂O was added slowly to just maintain reflux to 1.80 g (0.0741 mol) of sublimed Mg and 25 ml of Et_2O under N_2 . The soln, was refluxed for 1 hr after addition. The Grignard formed in 69% yield. This solution was added to $22.6 \text{ g} (0.0618 \text{ mol}) \text{ of HgBr}_2 \text{ in } 50 \text{ ml of Et}_2 \text{O}$. After the mixture was stirred for 20 hr, 40 ml of H₂O was added, and the precipitate was dissolved in benzene, washed with H₂O, dried, and filtered. After the volume of the filtrate was reduced to 75 ml, 5.28 g of 4a was isolated and recrystallized, mp 182.5–184.0° dec. The 4a was isolated and recrystallized, mp 182.5-184.0° dec. impure residue, 5.6 g, was chromatographed over Woelm activity I neutral Al₂O₃ with Et₂O-benzene eluent. The combination of the latter fractions gave 1.22 g of 4b, mp 133-134°. The total weight of 4-phenylcyclohexylmercuric bromide obtained from the Grignard reaction was 10.9 g (72%). 4b, therefore, constituted 12% of the alkylmercuric bromide compounds formed.

Carbonation of the Grignard Reagent from cis-4-Phenylcyclohexyl Bromide (3b).-Sublimed Mg (0.35 g, 0.0144 mol) in 12 ml of purified Et_2O was refluxed and stirred under N_2 , while 2.00 g (0.0084 mol) of 3b in 22 ml of purified Et₂O was added slowly. The reaction mixture was refluxed for 1.75 hr and poured into an Et₂O-Dry Ice mixture. This mixture was acidified and CH₂Cl₂ was added. The organic layer was washed several times with H_2O and dried over anhydrous Na_2SO_4 ; the solvent was removed to give 1.59 g (93% yield) of the acids. The ir showed the mixture to be only carboxylic acids. After esterification with CH_2N_2 , the ester mixture was analyzed by glc. 5a was found to be 98.2% of the total methyl ester mixture. The glc retention time and the ir spectra of authentic samples corresponded with those obtained by the Grignard carbonation. Pure 5a was obtained by recrystallization from pentane, mp 29-31°. The same Grignard formation and carbonation procedure using trans-4-phenylcyclohexyl bromide (3a) gave 99.8% 5a after esterification followed by glc analyses

cis-4-Cyclohexylcyclohexyl Bromide (6b).-7a was obtained by the recrystallization of commercial 4-cyclohexylcyclohexanol from cyclohexane until the melting point was 103.8-104.2° (lit.³ mp_103-104°)

To 4.00 g (0.022 mol) of 7a was added 5.96 g (0.022 mol) PBr₃

at -40° . The solution was warmed slowly to room temperature and stirred for 3 days. The solution was then poured over iceisopentane. (A large amount of phosphite ester was still present, indicating that the reaction had not gone to completion.) The yield was 1.81 g (35%), mp 29–32°. Anal. Calcd for C₁₂H₂₁Br: C, 58.87; H, 8.66. Found: C, 58.76; H, 8.61.

trans-4-Cyclohexylcyclohexyl Bromide (6a).--7b was prepared by the reduction of 21.0 g (0.124 mol) of *p*-phenylphenol in 150 ml of HOAc with H₂ and Rh-Al₂O₃, at 50 psi and 70°. After H₂ absorption ceased, the solution was cooled and poured into H_2O . The precipitate was dissolved in CH₂Cl₂, washed with aqueous base, and dried over anhydrous MgSO4 and the solvent was removed to yield 19.2 g (85%) of impure 7b. A small amount of this alcohol was chromatographed over Woelm activity II neutral Al₂O₃ with pentane-Et₂O eluent, mp 94.0-94.5° (lit.³ mp 92-93°).

To 1.05 g (0.0058 mol) of 7b at -40° was added 1.60 g (0.0059 mol) of PBr₃ and the solution was stirred for 3 days at room temperature. The solution was poured into ice-pentane, washed with concentrated H₂SO₄ and H₂O, and then dried over anhydrous MgSO₄. After the solvent was removed, 0.49 g (0.0020 mol) of 6a was obtained (35% yield). The material could not be crystallized at room temperature. Anal. Calcd for $C_{12}H_{21}Br$: C, 58.87; H, 8.66. Found: C, 59.17; H, 8.87.

Reduction of the cis- and trans-4-Phenylcyclohexyl Bromides (3b and 3a).—To 30 ml of HOAc were added 0.300 g (0.0013 mol) of 3a and 0.088 g of PtO₂. The H₂ pressure was kept at 40 mm for 12 hr. The ir spectrum showed complete reduction of 3a bromide, and was identical with that prepared using 7b. The material could not be crystallized at room temperature, nmr δ 4.56 (m, = CBrH).

The same procedure was used with 3b. The ir spectrum was identical with that of the cis bromide 6b prepared from 7a and the mixture melting point was not depressed. The yield was 54%, mp 33-34°, nmr δ 5.40 (s, =-CBrH). Summary of Nmr. Bromide-Substituted Carbon Methine

Absorption for Cyclohexyl Bromides: cis-4-phenylcyclohexyl bromide (3b), 5.54 (s); trans-4-phenylcyclohexyl bromide (3a), 4.66 (m); cis-4-cyclohexylcyclohexyl bromide (6b), 5.40 (s); trans-4-cyclohexylcyclohexyl bromide (**6a**), 4.56 (m); axial methine in cyclohexyl bromide at -81° , 4.70^s (m)⁶; equatorial methine in cyclohexyl bromide at -81° , 5.56^s (s).⁶

Acknowledgment.—I wish to thank Professor F. R. Jensen for his guidance and support during the course of this research.

Registry No.-2a, 5769-13-1; 3a, 42367-11-3; 3b, 42367-12-4; 4a, 42367-13-5; 4b, 42367-14-6; 5a, 36296-69-2; 6a, 42367-15-7; 6b, 42367-16-8; 7a, 7335-42-4; 7b, 7335-11-7.

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The Addition of Dichloroketene to **2-Aryl-\Delta^2-oxazolines**

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Received July 13, 1973

Dichloroketene formed by the in situ dehydrohalogenation of dichloroacetyl chloride¹ has been shown² to react with many Schiff bases to form α, α -dichloro- β -lactams. In our hands, however, many variations of this reaction with the substrate 4,4-dimethyl-2phenyl- Δ^2 -oxazoline (1) have not produced the desired oxygen-containing penicillin-like lactam, 4, but rather a 2:1 ketene diadduct, 3.

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