**4-Methyl-3-phenyl-l,4-gentanediol(6**.**)**-Anhydrous ether **(500**

ml) followed by LiAIHa **(2.25** g, **60** mmole) was introduced into

a round-bottom flask equipped with a magnetic stirrer and

heating mantle. In a modified liquid-liquid continuous extractor

there was added **15** g (80 mmoles) of ***5.*** The column was filled

with ether and the pot containing the hydride solution was heated

at reflux for **12** hr (necessary procedure since the solid lactone is

only slightly soluble in ether). After cooling of the reaction

mixture, the hydride solution was cautiously hydrolyzed with

saturated NazSOc solution. The resulting salts were dissolved in

**10yc** hydrochloric acid and the layers separated. The aqueous

solution was extracted with an additional **100** ml of ether. The

combined ether so1ution;j were washed with water, NaHC03

solution, and water again, then dried (NazSOa), and concentrated

to dryness at reduced pressure, giving a white solid which was

recrystallized from benzene yielding **14** g **(91%)** or white needles:

mp **89-90'; *v:::* 3290** (13-bonded OH, broad), **1065** (primary

COH stretch), and **1135** ((tertiary COH stretch).*.* Calcd

for C1~HISOCz:, **74.19;** 13, **9.34.** Found: C, **74.31;** H, **9.31.**

**2,2-Dimethyl-3-phenyltetrahydrofuran(7 )** and 4-Methyl-3-

phenyl-3-penten-1-ol (8) **.--A** solution of **40** ml of **10%** sulfuric

acid containing **3.88** g (20 mmoles) of diol ***6*** was stirred at reflux

for **90** min, cooled to room temperature, and neutralized

with NasCOa. The neutralized solution was extracted twice with

50-ml portions of ether. The extracts were washed with water

twice, dried (NatSOd), arid concentrated at reduced pressure to

yield **3** g **(857,)** of an oil. Analysis by gas chromatography on

an 8-ft **207c** Carbowax **011** Diataport S support at **238'** (helium

flow, **60** rc/min) gave two components in a **3: 1** ratio with retention

times of **4.5** min **(7)** and **8.5** rnin **(8),** respectively. Infrared

absorption of **7** showed no hydroxyl absorption but exhibited

absorption a t **1135** (ether) and was nonviscous with *n Z 7***1.***~***51 60.**

The nmr in CDC13 showed a singlet at 6 **7.07 (5** H), a multiplet

at **8 3 85 (2** H) , a triplet at 6 **2.96** *(J* = **8.5** cps) **(1** H), a multiplet

at **S 2.21 (2** H), a singlet at 6 **1.27 (3** H), and a singlet at 6 **0.81**

**(3** H). Compound **8, *uz::* 3290** (OH, broad), **1595** (C=C), was

viscous with *n " ~***1. 5360.** The nmr of 8 (CDC18) exhibited a

multiplet at 6 **7.01 (3** R), a triplet at 6 **3.40** *( J* = **7** cps) **(2** H), a

triplet at 6 **2.54** *( J* = **7** cps) **(2** H), a singlet at 6 1.78 **(3** H), and

a singlet at 6 **1** .SO **(3** E). The hydroxyl proton absorption occurred

at 6 **2.45,** but it is Re11 known that the hydroxyl frequencies

of alcohols can vary over a wide range according to the nature of

the solvent, concentration of the solute, and temperature.14

*Anal.* Calcd for **7,** C1~H[leO: C, **81.77;** H, **9.15.** Found: C,

**81.87;** H, **9.44.***.* Calcd for *8,* ClzHleO: C, **81.77;** H,

**9.15.** Found: C, **81.51;** H, **9.33.** Similar ratios of products

were obtained when ***6*** was refluxed in **10%** HzS04 for **1.5** min or

in **1** % H2S04 for **2** hr .

**2,2-Dimethyl-3-phenyltetrahydrofuran (7)** .-Diol 6 **(1** g, **5.15**

mmoles) was added to a solution of **10** ml of anhydrous benzene

containing **100** mg of p-toluenesulfonic acid (prepared from the

hydrate by azeotroping with benzene). This mixture was stirred

at reflux for **1** hr. After cooling, the solution was washed with

NaHCOs solution and water and dried (Na2SO4). Then this

was concentrated at reduced pressure to yield 0.8 g **(88%)** of **7.**

Gas chromatographic analysis as presented above showed an

identical retention time with **9701,** purity of compound **7.**

**alfa-Cyano-beta-cyclopropylcinnamonitrile** (9b) -The starting

material, **alfa-cyano-beta-(3-chloropropyl)cinnamonitrile**w**,**a s prepared

in the usual manner as described above. To 50 ml of

benzene was added **6.2** g **(27** mmoles) of or-cyano-fl-(3-chloropropy1)

cinnamonitrile and **2.3** g **(27** mmoles) of piperidine. The

solution was stirred at reflux for **6** hr, then the resulting dark

orange slurry was cooled to about ***5'*** and filtered **(3.1** g **(93** %) of

piperidine hydrochloride was obtained). The filtrate was washed

with saturated NaHCOs solution and water and dried (Na2S04).

The dried solution was concentrated at reduced pressure to dryness

and the resulting solid was taken up into ethanol, treated

with Norit A, filtered while hot, and allowed to crystallize,

yielding **3.2** g **(61%)** of white crystals: mp **110-11 1: :':;Y 3050**

(cyclopropyl CH), **1020** (skeletal CsHs), and **2250** (CN).

Calcd for CI~HIONC~,: **80.39;** H, **5.19; N, 14.42.** Found:

C, **80.44;** H, **5.50;** N, **14.40.**

**alfa-Carboxamido-beta-phenyl-y-spirocyclopropylbutyrolacton**(**10** b).

-Compound 9b **(10** g, **51** mmoles) was added to **100** g of polyphosphoric

acid with stirring. The resulting mixture was heated

to **100'** for **8** hr during which time the reaction mixture turned

dark orange. The mixture was hydrolyzed in **1** 1. of water and the

aqueous solution saturated with NaCl and extracted with three

100-ml portions of CHCL. The combined extract was washed

with a small volume of water, dried (NazSOa), and concentrated

at reduced pressure to yield an orange foam. The foam **(4** g)

was chromatographed over **120** g of acid-washed alumina and

eluted successively with **500** ml of benzene, **300** ml of **loyo** ether

in benzene, **200** ml of **20%** ether in benzene, **500** ml of *SO%* ether

in benzene, and **200** ml of **75%** ether in benzene. The **50** and **75y0**

ether fractions yielded the product as a fluffy white solid. The

solids were combined and recrystallized from ethyl acetate to yield

**0.85** g **(7%)** of colorless needles: mp **179.5-181'; *v:::* 3050**

(CH stretch CaHs), **1720** (CO), **1650** (amide Co), and **3430** (amide

NH); **6** (CFaCOOH), singlet at **7.40 (5** H), triplet at **4.63**

**(2** H) *( J* = **6.0** cps), triplet at **2.91 (2** H) *(J* = 7.0 cps), triplet

at **2.33 (2** H) *(J* = 7.0 cps).16*.* Calcd for C13H13N03:

C, **67.50;** H, **5.67; N, 6.06.** Found: C, **67.38;** H, **5.79;** N,

**6.07.**