The dry solutions were concentrated at reduced pressure to dryness and the resulting products were crystallized from ethyl acetate with subsequent addition of cyclohexane **(2** : 1 to **1** : 1 ratio).

The solid products which precipitated from the aqueous solutions were cooled, filtered, washed well with water, dried in a desiccator under reduced pressure, and recrystallized from ethyl acetate (small volumes of cyclohexane may be added to induce

crystals). The following is a specific example.

**Alfa-Carboxamido-/3-phenyl-~-methyl-r-valerolact(3o)n** .**e**-To **50** g of polyphosphoric acid in a beaker equipped with a mechanicalstirrer was added **5** g **(255** mmoles) of **1.** The mixture was thenheated to 100' for 12 hr with stirring. The viscous mixture washydrolyzed in **500** ml of water while still warm. After the hydrolyzedmixture had cooled to room temperature, the productwhich crystallized was filtered, washed well with water, dried atrediiced pressure, and recrystallized from ethyl acetate and asmall amount of cyclohexane, yielding ***5*** g ***(857,)*** or white crystals:

mp **172-173.5; 3510** (NH), **3390** (NH), **1750** (CO), and

**1700** (amide CO). The nmr spectrum (CF3COOH) showed a

singlet at 6 **7.39 (5** H), a quartet at 6 **4.33 (2** H) *( J* = **13** cps),

a singlet at ***6* 1.64 (3** H), and a singlet at 6 **1.17 (3** H). There

was no ultraviolet absorption above 210 mp. *Anal.* Calcd for

CI3HljNO3: C, **66.93;** H, **6.48;** N, **6.01;** mol wt, **233.** Found:

C, **67.17;** H, **6.43;** N, **5.80;** mol wt (CHCla), **240.** The yields

and properties of other lactones synthesized are reported in

Tables **I1** and 111.

**Alfa-Carboxy-Beta-phenyl--y-methyl-valerolactone( 4)** .-A solution

of **1** g **(4.3** mmoles) of **3** in 15 ml of 10% sodium hydroxide was

stirred at room temperature until no further evolution of ammonia

was detected by litmus tests (about 8 hr), then cooled *to* **0"** in

an ice bath, and acidified with concentrated hydrochloric acid. The resulting white solid was filtered, washed well with water,

and dried at reduced pressure. The dry solid was recrystallized

from ethyl acetate yielding **0.85** g (85%) of white crystals.

**Beta-phenyl-y-methyl-y-valerolactone(5**). A.-In an open reaction

vessel, **2** g **(8.5** mmoles) of **4** was heated in a oil bath to **180-190'** until evolution of carbon dioxide ceased (about **10** min).

Upon cooling of the residue to room temperature, the product solidified and was crystallized from benzene and hexane **(1:l)**

yielding **1.3** g (80%) of white crystals: mp **91-92'; 1765** (CO).Calcd for C12H1402: C, **75.75;** H, **7.43;** mol wt, **190.** Found: C, **75.88;** H, **7.63;** mol wt (CHCla), **189.**

B.-To **50** ml of **20%;;** sulfuric acid was added **4.66** g **(20**

mmoles) of 1. The resulting mixture was heated at reflux with

stirring for **16** hr. After cooling, the resulting oil solidified.

The solid was filtered, washed well with water, and dried at reduced

pressure. The product was dissolved in about **50** ml of

benzene, decolorized with Norit A, filtered, and concentrated

to one-half its initial volume. When **10-15** ml of cyclohexane

was added, the product **(3.45** g, **91%)** crystallized as white plates,

mp **90.5-92.**