Ring openings of γ- and δ-lactones to form γ- and δ-hydroxyamides. Cyclodehydration. Mass spectra of δ-hydroxyamides[1]

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Ring openings of γ- and δ-lactones were effected with potassium amide or hydrazine to form γ- and δ-hydroxyamides, which were cyclodehydrated with sulfuric acid to give γ- and δ-lactams, respectively. These products are, respectively, phthalimidines and 3,4-dihydroisocarbostyrils having no substituent on nitrogen or having the N—NH_2 group. The possible linear dehydration of the δ-hydroxyamides was not observed. The δ-hydroxyamides exhibited, on mass spectrometry, a type of carbon–carbon cleavage that has apparently not been reported for ordinary alcohols. An example of the "*ortho* effect" in mass spectra of *o*-disubstituted benzene derivatives is also discussed.

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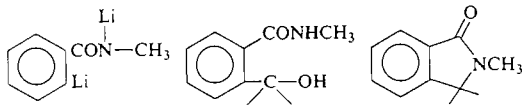
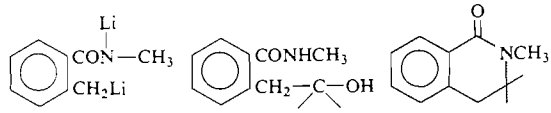
Dilithiations of *N*-methylbenzamide (1) and *N*-methyl-*o*-toluamide (2) have previously been accomplished with *n*-butyllithium to form dilithioamides **1** and **4**, which were condensed with ketones or aldehydes to give γ- and δ-hydroxyamides **2** and **5**, respectively. These compounds have been cyclodehydrated with sulfuric acid to afford γ- and δ-lactams **3** and **6**, respectively (3).



1 **2** **3**



4 **5** **6**

However, similar dilithiations of benzamide (1) and *o*-toluamide[2] have failed. Thus, the above method has not been applicable to preparations of γ- and δ-hydroxyamides and γ- and δ-lactams having no substituent on the nitrogen atom.

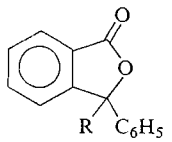
We have now prepared such compounds by ring opening of γ- and δ-lactones with potassium amide, followed by ring closure with acid; similarly, hydroxyamides and lactams having the N—NH_2 group were synthesized employing hydrazine. The γ- and δ-lactones were readily obtained by thermal cyclodeamination of γ- and δ-hydroxyamides of types **2** and **5**, respectively (1, 2).

Results with γ-Lactones

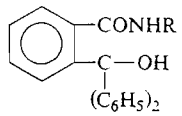
γ-Lactone **7** underwent ring opening with potassium amide in tetrahydrofuran (THF) and with hydrazine (neat) to form γ-hydroxyamide **8** and hydrazide **9**, which underwent cyclodehydrations with cold, concentrated sulfuric acid to give γ-lactams **10***a* and **10***b*, respectively.

Crude hydroxyamide **8** and hydrazide **9** were employed in these reactions, since attempted purification of them resulted in reconversion to lactone **7**. The overall yields of **10***a* and **10***b* from lactone **7** were 25 and 20%, respectively.

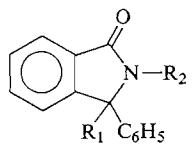
Similarly, treatment of γ-lactone **11** with hydrazine afforded γ-lactam **12** in 4% yield; this yield might be improved since most of lactone **11** was recovered. The intermediate hydrazide was not isolated. γ-Lactone **11** has previously been



7, R = C_6H_5
11, R = H



8, R = H
9, R = NH_2



10*a*, R_1 = C_6H_5, R_2 = H
10*b*, R_1 = C_6H_5, R_2 = NH_2
12, R_1 = H, R_2 = NH_2

[1]Supported by Public Health Service Grant No. CA-04455 from the National Cancer Institute and by the National Science Foundation.

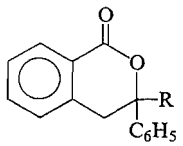
[2]Unpublished observation of R. E. Ludt in this laboratory.

shown to undergo ionization of its benzhydrylic hydrogen atom with potassium amide to form a carbanion which can be alkylated (4).

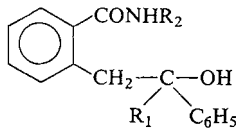
Attempts to prepare γ-hydroxyamides by the action of potassium amide on the γ-lactones obtained from dilithioamide **1** and cyclohexanone or fluorenone were unsatisfactory. Apparently these lactones underwent ring opening but were again regenerated on work-up.

Results with δ-Lactones

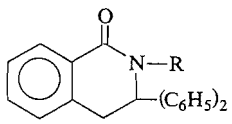
δ-Lactone **13** underwent ring opening with potassium amide in THF and with hydrazine (neat) to form δ-hydroxyamide **14** and the corresponding δ-hydroxyhydrazide (isolated as its acetone derivative **15**) in yields of 69 and 45%, respectively. These compounds were cyclodehydrated with cold, concentrated sulfuric acid to give the 3,4-dihydroisocarbostyrils **16***a* and **16***b* in yields of 89 and 53%, respectively. Interestingly, the possible linear dehydration was not observed.



13, R = C_6H_5
22, R = H



14, $R_1 = C_6H_5$, R_2 = H
15, $R_1 = C_6H_5$, $R_2 = N{=}C(CH_3)_2$
23, $R_1 = R_2$ = H



16*a*, R = H
16*b*, R = $N{=}C(CH_3)_2$

Similarly, δ-lactone **17** underwent ring opening with potassium amide to form δ-hydroxyamide **18** in 82% yield. This compound may have undergone cyclodehydration with cold, concentrated sulfuric acid to give δ-lactam **19** but, surprisingly, olefin-amide **20** was obtained on attempted distillation. That the olefinic product was **20**, and not the possible **21**, was supported by the nuclear magnetic resonance (n.m.r.) spectrum with the vinyl proton absorption at δ 5.28 p.p.m.[3] Had the product been **21**, this absorption should have appeared at lower field. In addition, this n.m.r. spectrum shows benzylic methylene protons at δ 3.47 p.p.m. The infrared (i.r.) spectrum is characteristic of a benzamide and the mass spectrum resembles that of the unsubstituted δ-hydroxyamides in that the most abundant fragment ion appears at *m/e* 135 (see below). The base peak in this spectrum is the molecular ion (*m/e* 215). One might expect to observe the formation of **21** as well as **20**; however, none was isolated.

Likewise, δ-lactone **22** underwent ring opening with potassium amide to form δ-hydroxyamide **23** in 78% yield. Such ring openings appear to be quite general and, in contrast to corresponding γ-lactones, uncomplicated by ring closure during work-up.

It should be mentioned that δ-lactone **13** also underwent ring opening with lithium methylamide in THF to form the corresponding δ-hydroxyamide of type **5**. However, this compound can be prepared more conveniently from dilithioamide **4** and benzophenone (2).

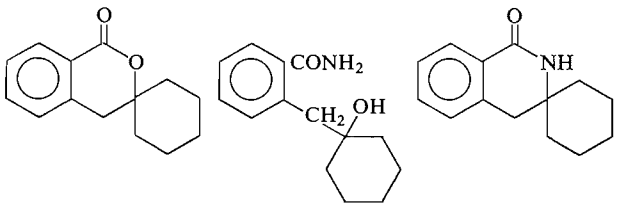
The present method of synthesis of 3,4-dihydroisocarbostyrils having no substituent on nitrogen appears more convenient than earlier ones, which have involved cyclizations of urethanes with polyphosphoric acid (6) and of isocyanates with aluminum chloride (7).

Mass Spectra of δ-Hydroxyamides

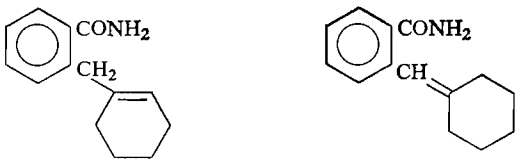
These hydroxyamides exhibited, on mass spectrometry, a type of cleavage that appears not to have been observed previously (Schemes 1 and 2). Thus, compounds **14**, **18**, and **23** underwent not only a normal alcohol cleavage (course *a*), but also an abnormal one (course *b*) that involves migration of hydrogen from oxygen to nitrogen accompanied by carbon–carbon bond cleavage. The latter cleavage gave the base peak at *m/e* 135, the molecular ion of a toluamide, common to the three spectra (see Scheme 1). A minor fragmentation observed in all 3 spectra is the loss of 17 mass units from the molecular ion. This fragmentation, in the case of **23**, was shown by high resolution to be due to the loss of *ammonia*,[4] and not to loss of *hydroxyl radical*. This is due to either

[3]The positions reported for vinyl protons of similar type lie between δ 5.36–5.43 p.p.m. (5).

[4]Anal. Calcd. for $C_{15}H_{12}O_2$: C, 224.0838. Found: C, 224.0837.



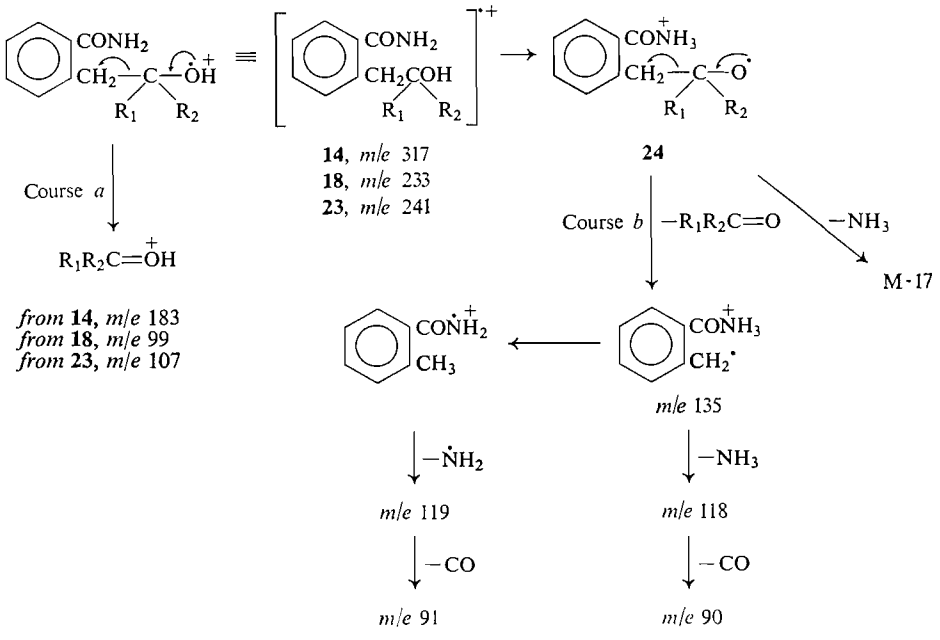
17 18 19



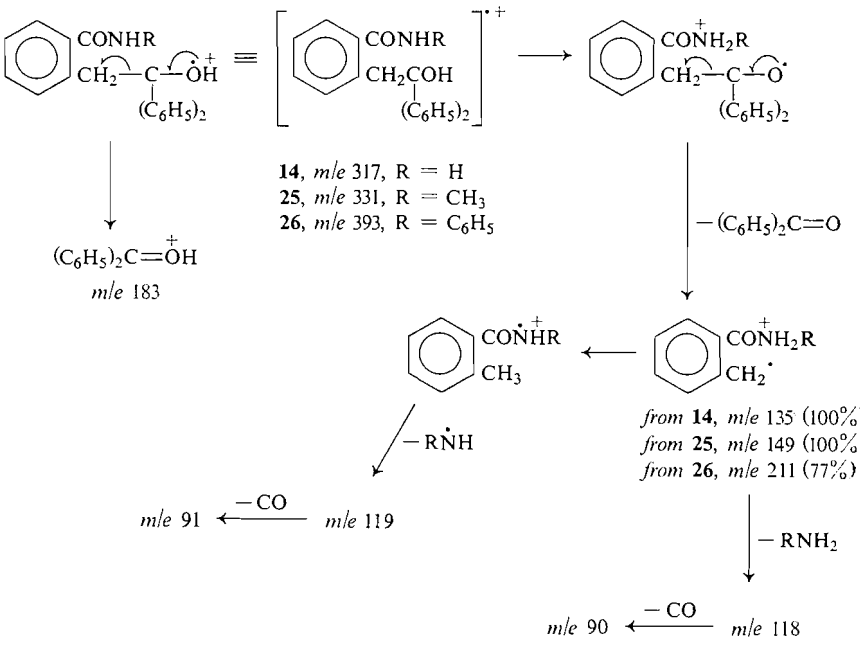
20 21

cyclodeamination before ion impact, loss of ammonia from ion **24** (Scheme 1), or a combination of these effects. The fragmentation from the base peak ion is interesting in that it is another example of the "*ortho* effect" in *o*-disubstituted benzene derivatives. This effect has been observed in the mass spectra of *o*-ethoxybenzamide (8) and *o*-methylbenzoates (9) when compared to the spectra of the *meta* and *para* isomers. The "normal" fragmentation of a toluamide would be expected to follow the pathway *m/e* 135 to *m/e* 119 to *m/e* 91; i.e. loss of an amine radical, followed by loss of carbon monoxide (10). In the mass spectra of the δ-hydroxyamides, part of the fragmentation occurs *via* the pathway *m/e* 135 to *m/e* 118 to *m/e* 90; i.e. loss of ammonia followed by loss of carbon monoxide, as well as following the "normal" pathway.

These rationalizations of the fragmentation pathways are supported by the mass spectra of the *N*-methyl and *N*-phenyl substituted derivatives **25** and **26** (Scheme 2). In these spectra, as in the spectrum of **14**, the major fragmentation follows the abnormal course described above and



SCHEME 1



SCHEME 2

is confirmed by shifting of the toluamide peak to *m/e* 149 and *m/e* 211, respectively. The normal elimination of the protonated benzophenone ion is also observed, but to a much lesser extent. The fragmentation from the toluamide ions again follows both the "normal" and the "*ortho* effect" routes. These latter 2 pathways are roughly comparable in terms of ion current carried for all of the 5 spectra examined. The supporting metastable peaks for all of the fragmentations were observed (see Experimental), although not all were observed in each spectrum.

Experimental

Melting points are uncorrected. Infrared spectra were recorded on a Perkin–Elmer Model 137 Infracord and were calibrated against the 6.24 μ band of polystyrene. Nuclear magnetic resonance spectra were taken on a Varian A-60 instrument, operating at 60 MHz, and are reported as downfield shifts in p.p.m. for tetramethylsilane employed as an internal standard.[5] Mass spectra were obtained on an Associated Electrical Industries MS-902 at 70 eV.[6] Tetrahydrofuran (THF) was dried at reflux temperature over lithium aluminum hydride and was freshly distilled before use. Analyses were performed by M-H-W Laboratories, Garden City, Michigan.

2-[(Diphenylhydroxy)methyl]benzamide (8)

A solution of 14.3 g (0.05 mole) of 3,3-diphenylphthalide (**7**) (1) in dry THF (50 ml), was slowly added to a solution of potassium amide prepared from 7.8 g (0.2 g-atom) of potassium in *ca.* 250 ml of liquid ammonia. The reaction mixture was stirred at the b.p. of ammonia for 3 h, 150 ml of dry THF was added, and the ammonia was removed under a slow stream of nitrogen. After stirring for an additional 18 h at room temperature, the reaction mixture was poured into iced hydrochloric acid. The resulting mixture was filtered, and the solid washed well with water, then with acetonitrile to remove lactone **7**. The crude hydroxyamide (10.0 g, 65%), m.p. 170–162° decomp., was recrystallized twice from acetonitrile to give, apparently, pure **8**, m.p. 168–170° decomp.; λ_{max} ($CHCl_3$): 2.90, 3.00, and 6.02 μ.

The analytical data for this compound were unsatisfactory because of the ease with which it decomposed. On standing for about 1 week at room temperature, it was quantitatively reconverted to lactone **7**.

3,3-Diphenylphthalimidine (10a)

Freshly prepared, crude hydroxyamide **8** (10 g) was slowly added to 50 ml of cold (0°), concentrated sulfuric acid (50 ml), giving a dark red solution which was stirred at 0° for 3 h. The reaction mixture was poured onto ice. The resulting mixture was filtered and the solid washed well with water, then with ether to remove lactone **7**. The remaining crude hydrogen sulfate salt of lactam **10***a* was stirred with 20% aqueous sodium

[5]In n.m.r. descriptions: s = singlet, d = doublet, m = multiplet, and b = broad.

[6]We thank Dr. David Rosenthal for the mass spectral determinations, which were done at the Research Triangle Mass Spectrometry Center Supported by Special Facility Grant No. FR-00330-01, National Institutes of Health.

hydroxide for 2 h. The resulting precipitate was collected and treated with aqueous hydrochloric acid to give 4.05 g (38%) of the hydrogen chloride salt of lactam **10***a* which, after washing with ether to remove traces of lactone 7 still present, was stirred with aqueous sodium hydroxide solution for 1 h. The precipitate was collected to give lactam **10***a*, m.p. 105–109°. Two recrystallizations from acetonitrile afforded pure **10***a*, m.p. 125–126°; λ_{max} ($CHCl_3$): 3.03 (NH) and 5.97 μ (C=O); n.m.r. ($CDCl_3$): δ 6.82 (b, 1H, NH), 7.30–7.75 (m, 13H), and 7.75–7.95 p.p.m. (m, 1H).

Anal. Calcd. for $C_{20}H_{15}NO$: C, 84.18; H, 5.30; N, 4.91. Found: C, 84.02; H, 5.26; N, 4.93.

*2-Amino-3,3-diphenylphthalimidine (**10b**)*

A solution of γ-lactone **7** (2.0 g) and hydrazine (95%, 10 ml) was refluxed for 5 days. Hydrazide **9**, m.p. 105–107°, separated on cooling. On standing for 24 h at room temperature, the product had m.p. 115–117°. The odor of hydrazine was detected emanating from the sample and the i.r. spectrum was that of lactone **7**. Attempted crystallization of crude hydrazide **9** also led to lactone **7**. Compound **9** was therefore used immediately upon isolation without further purification.

Freshly-prepared crude hydrazide **9** (from 2 g lactone **7**) was stirred with concentrated sulfuric acid to 0° for 6 h. The solution was poured onto ice, and the crude aminolactam **10***b* was recrystallized twice from ethanol to give 0.42 g (20%) of pure **10***b*, m.p. 174.5–176° decomp.; λ_{max} ($CHCl_3$): 2.90, 3.00 (NH_2), and 5.95 μ (C=O); n.m.r. ($CDCl_3$): δ 3.95 (b, 2H, NH_2), 7.0–7.3 (m, 13H), and 7.7–8.0 p.p.m. (m, 1H); mass spectrum, *m/e* (relative intensity): 300 (67), 223 (100), 130 (11).

Anal. Calcd. for $C_{20}H_{16}N_2O$: C, 79.98; H, 5.37; N, 9.33. Found: C, 79.97; H, 5.07; N, 9.27.

*2-Amino-3-phenylphthalimidine (**12**)*

A solution of 10 g of 3-phenylphthalide (4) (**11**) in hydrazine (95%, 20 ml) was refluxed for 4 days. The reaction mixture was cooled overnight to give 0.5 g of white needles, m.p. 150–155°. Two recrystallizations from ethanol gave 0.4 g (4%) of pure **12**, m.p. 155–156°; λ_{max} ($CHCl_3$): 2.90, 3.00 (NH_2), and 5.94 μ (C=O); n.m.r. ($CDCl_3$): δ 3.83 (b, 2H, NH_2), 5.47 (s, 1H, ArCH), and 7.0–8.0 p.p.m. (m, 9H).

Anal. Calcd. for $C_{14}H_{12}N_2O$: C, 74.99; H, 5.38; N, 12.49. Found: C, 75.05; H, 5.10; N, 12.65.

The original reaction mixture was evaporated to an oil. The i.r. spectrum of the residue was indistinguishable from that of lactone **7**.

Reactions of 3,4-Dihydroisocoumarins with Potassium Amide

These δ-lactones (2) were converted to δ-hydroxyamides in the following manner. A solution of the lactone (1 equivalent) in dry THF was added to a solution of potassium amide (4 equivalents) prepared from potassium (4 equivalents) in liquid ammonia (200–300 ml) under dry nitrogen. The ammonia was removed under dry nitrogen, additional dry THF was then added, and the resulting mixture was stirred overnight. The reaction mixture was poured into iced concentrated hydrochloric acid. The product was collected by filtration and recrystallized.

*A. 2-(β,β-Diphenyl-β-hydroxy)ethylbenzamide (**14**)*

This compound was prepared from 4.5 g (0.015 mole) of δ-lactone **13** and 0.06 mole of potassium amide in 250 ml (total volume) of dry THF. The crude product was recrystallized from benzene–heptane to give 3.27 g (69%) of hydroxyamide **14**, m.p. 132–135° decomp. Two additional crystallizations from benzene–heptane gave the analytical sample of **14**, m.p. 134–135° decomp.; λ_{max} ($CHCl_3$): 2.87 (sh), 2.96 (sh), 3.03 (NH_2 and OH), and 6.06 μ (amide C=O); n.m.r. ($CDCl_3$): δ 3.72 (s, 2H, $ArCH_2$), 6.1–6.4 (b, 2H, NH_2), 6.72 (b, 1H, OH), 7.0–7.6 (m, 14H); mass spectrum, *m/e* (relative intensity): 317 (7), 300 (2), 223 (2), 183 (28), 135 (100), 119 (21), 118 (22), 91 (6), 90 (4), 77 (4), metastable peaks, m* (parent, daughter), 283.9 (317, 300), 105.6 (317, 183), 103.1 (135, 118), 69.6 (119, 91), 68.6 (118, 90), 57.5 (317, 135).

Anal. Calcd. for $C_{21}H_{19}NO_2$: C, 79.47; H, 6.03; N, 4.41. Found: C, 79.55; H, 5.97; N, 4.24.

*B. 2-(1-Hydroxycyclohexyl)methylbenzamide (**18**)*

This compound was prepared from 10.80 g (0.05 mole) of lactone **17** and 0.20 mole of potassium amide in 250 ml (total volume) of dry THF. Crude hydroxyamide **18** (10.05 g, 82%), m.p. 182–183° decomp., was recrystallized twice from acetonitrile to give the analytical sample, m.p. 184–185° decomp.; λ_{max} ($CHCl_3$): 2.89 (sh), 2.96 (sh), 3.04 (NH_2 and OH), and 6.05 μ (amide C=O); mass spectrum, *m/e* (relative intensity): 233 (2), 216 (8), 173 (7), 160 (8), 135 (100), 119 (35), 118 (35), 99 (2), 91 (10), 90 (5), 81 (4), 77 (3), metastable peaks, m* (parent, daughter), 200.2 (233, 216), 118.5 (216, 160), 104.9 (135, 119), 103.1 (135, 118), 78.2 (233, 135), 69.6 (119, 91), 68.6 (118, 90).

Anal. Calcd. for $C_{14}H_{19}NO_2$ (mol. wt. 233.1416): C, 72.07; H, 8.21; N, 6.00. Found (mol. wt. 233.1415, mass spectrum): C, 72.44; H, 8.14; N, 5.91.

*C. 2-(β-Hydroxy-β-phenyl)ethylbenzamide (**23**)*

This compound was prepared from 2.24 (0.01 mole) of lactone **22** and 0.04 mole of potassium amide in 150 ml (total volume) of dry THF. Crude hydroxyamide **23** (1.85 g, 78%), m.p. 145–147° decomp., was recrystallized 3 times from acetonitrile to give the analytical sample of **23**, m.p. 149–150° decomp.; λ_{max} ($CHCl_3$): 2.84 (sh), 2.97 (sh), 3.03 (NH_2 and OH), and 6.06 μ (amide C=O); mass spectrum, *m/e*, (relative intensity): 241 (1), 224 (2), 178 (2), 146 (1), 135 (100), 119 (34), 118 (31), 107 (4), 105 (3), 91 (10), 90 (6), 77 (8), 51 (6), metastable peaks, m* (parent, daughter), 208.2 (241, 224), 104.9 (135, 119), 103.1 (135, 118), 75.6 (241, 135), 69.6 (119, 91), 68.6 (118, 90).

Mol. Wt. Calcd. for $C_{15}H_{15}NO_2$: 241.1103. Found: 241.1104.

*N-[2-(β,β-Diphenyl-β-hydroxy)ethyl]benzoyl Acetone Hydrazone (**15**)*

A solution of 5 g of lactone **13** in 20 ml of hydrazine (95%) was refluxed for 3 days. The reaction mixture was evaporated at reduced pressure to leave an oil. Acetone (50 ml) was added to precipitate 2.8 g (45%) of the crude acetone derivative **15**, which was collected by filtration. This compound, m.p. 232–234° decomp., was washed well with benzene and acetonitrile, and a sample recrystallized from chloroform to give the analytical sample

of **15**, m.p. 242–244° decomp., λ_{max} ($CHCl_3$): 3.16 (NH) and 6.15 μ (C=O).

Anal. Calcd. for $C_{24}H_{24}N_2O_2$: C, 77.39; H, 6.50; N, 7.21. Found: C, 76.94; H, 6.58; N, 7.17.

Cyclodehydration of δ-Hydroxyamides

The compound was dissolved in 25 ml of concentrated sulfuric acid that had been cooled to 0°. The solution was stirred for 3 h at 0° and then poured onto ice. The product was isolated as described below.

A. 3,3-Diphenyl-3,4-dihydroisocarbostyril (16a)

Crude δ-lactam **16***a*, prepared from 2.38 g (0.075 mole) of δ-hydroxyamide **14**, was collected by filtration and recrystallized from acetonitrile to give 1.98 g (89%) of pure **16***a*, m.p. 243–245°; λ_{max} ($CHCl_3$): 2.98 (NH) and 6.05 μ (C=O); n.m.r. ($CDCl_3$): δ 3.28 (s, 2H, ArCH), 6.5–6.7 (b, 1H, NH), 7.3–7.5 (m, 13H), and 7.9–8.1 p.p.m. (m, 1H); mass spectrum, *m/e* (relative intensity): 299 (34), 271 (4), 270 (14), 223 (17), 222 (100), 204 (6), 203 (10), 195 (3), 194 (6), 179 (4), 178 (5), 165 (4), 119 (13), 118 (81), 104 (6), 90 (27), 89 (8), 77 (8), 57 (7), 55 (5), metastable peaks, m*, (parent, daughter), 269.0 (271, 270), 245.6 (299, 271), 187.5 (222, 204), 177.0 (179, 178), 171.2 (222, 195), 164.8 (299, 222), 144.3 (222, 179), 68.6 (118, 90), 57.0 (104, 77).

Anal. Calcd. for $C_{21}H_{17}NO$: C, 84.25; H, 5.72; N, 4.68. Found: C, 84.24; H, 5.71; N, 4.78.

B. 2-(2-Propylideneamino)-3,3-diphenyl-3,4-dihydroisocoumarin (16b)

Crude δ-lactam **16***b*, prepared from 1.75 g of δ-hydroxyhydrazide **15**, was collected by filtration and recrystallized 3 times from acetonitrile to give 0.90 g (53%) of analytically pure **16***b*, m.p. 197–199°; λ_{max} ($CHCl_3$): 6.06 μ (C=O); n.m.r. ($CDCl_3$): δ 1.82, 1.85 (2s, 6H, $2CH_3$), 3.77 (s, 2H, $ArCH_2$), 7.0–7.5 (m, 13H), and 7.7–8.1 p.p.m. (m, 1H).

Anal. Calcd. for $C_{24}H_{22}N_2O$: C, 81.32; H, 6.26; N, 7.90. Found: C, 81.38; H, 6.56; N, 7.90.

C. 2-(1-Cyclohexenyl)methylbenzamide (20)

δ-Lactam **19**, prepared from 9.1 g of δ-hydroxyamide **18** was apparently present as its hydrogen sulfate salt in the aqueous acidic solution. Extraction of the acidic solution with ether gave 0.95 g of a mixture of lactone **17** and δ-hydroxyamide **18**. The aqueous acidic solution was made basic with solid sodium hydroxide, and the resulting mixture was extracted with ether, dried, and evaporated. The residual oil (6.35 g, 75%) had λ_{max} (CCl_4): 3.06 (NH) and 6.14 μ. Attempted purification by distillation (>200°) gave the olefinic product **20**, which did not distil and solidified on cooling. Crystallization from carbon tetrachloride gave 5.25 g (70%) of pure **20**, m.p. 130–131°; λ_{max} ($CHCl_3$): 2.87, 2.97 (NH_2), and 6.02 μ; n.m.r. ($CDCl_3$): δ 1.4–2.2 (m, 8H, cyclohexyl protons), 3.47 (s, 2H, $ArCH_2C{=}C$), 5.28 (b, 1H, $CH_2C{=}CH{-}CH_2$), and 7.0–7.7 p.p.m. (m, 4H); mass spectrum, *m/e* (relative intensity): 215 (100), 198 (57), 172 (35), 170 (15), 135 (75), 119 (27), 118 (24), 91 (14), 81 (21), metastable peaks, m* (parent, daughter), 197.0 (199, 198), 182.3 (215, 198), 170.0 (172, 171), 169.0 (171, 170), 137.6 (215, 172), 104.9 (135, 119), 103.1 (135, 118).

Anal. Calcd. for $C_{14}H_{17}NO$: C, 78.10; H, 7.96; N, 6.51. Found: C, 78.38; H, 7.85; N, 6.39.

Reaction of δ-Lactone 13 with Lithium Methylamide

n-Butyllithium (34 ml of 2.35 *M*, 0.08 mole) in hexane[7] was slowly added to a solution of methylamine (*ca.* 40 ml) in dry THF (100 ml) under nitrogen and the excess amine was then removed under nitrogen. A solution of 6.0 g (0.02 mole) of lactone **13** in dry THF (100 ml) was added and the resulting mixture was stirred at reflux overnight. The crude mixture was cooled and poured onto iced concentrated hydrochloric acid. The precipitate was collected and recrystallized from 95% ethanol to give 4.5 g (66%) of δ-hydroxyamide **25**, m.p. and mixture m.p. 190–191° (lit. (2), m.p. 190–191°); mass spectrum, *m/e* (relative intensity): 331 (1), 283 (1), 183 (7), 182 (1), 149 (100), 148 (13), 134 (4), 119 (37), 118 (13), 105 (15), 91 (9), 90 (2), 77 (12), metastable peaks, m* (parent, daughter), 147.0 (149, 148), 120.5 (149, 134), 93.4 (149, 118), 60.5 (182, 105), 69.6 (119, 91).

N-Phenyl-2-(β,β-diphenyl-β-hydroxy)ethylbenzamide (26)

The mass spectrum of δ-hydroxyamide **26** was obtained on a sample prepared from *N*-phenyl-*o*-toluamide by a method similar to that previously described (2); mass spectrum, *m/e* (relative intensity): 393 (1), 300 (13), 283 (8), 223 (3), 211 (77), 194 (13), 183 (12), 119 (85), 118 (100), 105 (23), 93 (37), 91 (11), 90 (17), 77 (20), metastable peaks, m* (parent, daughter), 113.5 (393, 211), 66.0 (211, 118), 69.6 (119, 91), 68.6 (118, 90), 67.1 (211, 119).

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[7]Obtained from Alfa Inorganic Inc., Beverly, Massachusetts.