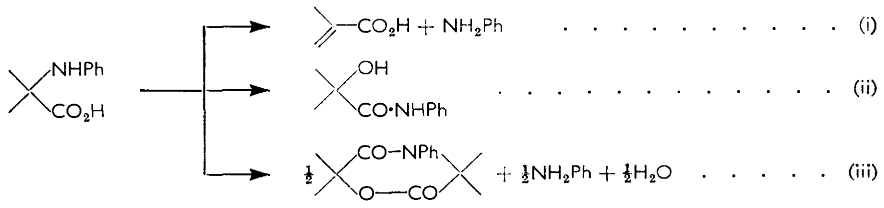
346. *Studies in Pyrolysis. Part XVI.* Pyrolysis of Some Open-chain α-Anilino-carboxylic Acids.*

By J. McGee and P. D. Ritchie.

Several open-chain α-anilino-carboxylic acids ($NHPh{\cdot}CRR'{\cdot}CO_2H$) have been pyrolysed (*ca.* 240°; liquid phase). Where R and R′ are both alkyl, the acids break down by two competitive routes; (i) scission to aniline and an olefinic acid, and (ii) elimination of aniline and water, with formation of a cyclic lactone-lactam. There is no evidence for rearrangement of the anilino-acid to a hydroxy-anilide (contrast Bain and Ritchie[1]). Where $R = Me$ and $R' = Ph$, only reaction (i) is observed: where $R = R' = Ph$, reaction (i) is structurally precluded, and the complex pyrolysate is best explained as arising from primary elimination of aniline with formation of a cyclic dilactone (benzilide).

Reaction mechanisms are discussed.

Bain and Ritchie[1] showed that three homologous 1-anilinocycloalkanecarboxylic acids all break down at *ca.* 250° (liquid phase) by a series of competitive reactions, thus:



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* Part XV, *J.*, **1960**, 4141.

[1] Bain and Ritchie, *J.*, 1955, 4407.

Reaction (i) increases in importance, and reaction (iii) decreases, as the size of the carbocycle is increased ($C_5 \longrightarrow C_6 \longrightarrow C_7$), while the α-hydroxy-anilide produced by the novel rearrangement (ii) remains a fairly constant minor product throughout. Reaction (i) is probably a primary scission, and reaction (iii) the summation of a primary and a secondary reaction. Reaction (ii) appears [1] to be a direct primary isomerisation; and to study its mechanism further, the work has now been extended from cyclic systems to the open-chain α-anilino-acids (I—IV).

$NHPh{\cdot}CMe_2{\cdot}CO_2H$	$NHPh{\cdot}{}^*CMeEt{\cdot}CO_2H$	$NHPh{\cdot}{}^*CMePh{\cdot}CO_2H$	$NHPh{\cdot}CPh_2{\cdot}CO_2H$
(I)	(II)	(III)	(IV)

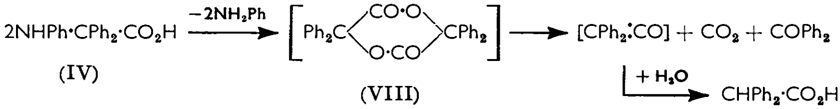
In two of these acids (II, III) the α-carbon atom (*) is dissymmetric; and the intended line of attack was to resolve acids (II) and (III) into their optical antipodes, to pyrolyse these optically active materials, and to find whether detachment of the anilino-group from the dissymmetric carbon atom occurred with or without retention of optical configuration. This stereochemical approach, fruitful in previous studies on various other rearrangements, was here unexpectedly precluded by the observation that the acids (I—IV) gave no detectable quantity of α-hydroxy-anilide on pyrolysis. A separate pyrolysis of the anilide (V), the expected rearrangement product of acid (I), showed it to be stable under the conditions used, so that its absence from the pyrolysate is not due to subsequent thermal breakdown. It seems clear, then, that the novel isomerisation undergone by the three cyclic compounds [1] is not general for all α-anilino-acids.

$HO{\cdot}CMe_2{\cdot}CO{\cdot}NHPh$	$NHPh{\cdot}CMe_2{\cdot}CO{\cdot}NHPh$	$CH_2{:}CPh{\cdot}CO{\cdot}NHPh$
(V)	(VI)	(VII)

The pyrolysates from acids (I) and (II) contained aniline, water, olefinic acid (see following paragraph), and the curious type of "lactone-lactam" discussed previously.[1,2] In addition, acid (I) gave a trace of the anilino-anilide (VI), presumably by partial condensation of the olefinic acid with aniline from reactions (i) and (iii). Apart, therefore, from the absence of reaction (ii), acids (I) and (II) behave on pyrolysis similarly to their cyclic analogues,[1] even in the increased yield of olefinic acid at the expense of lactone-lactam as molecular weight increases (cf. Table 1). Acid (III), however, yielded only aniline, traces of atropic acid and atropanilide (VII) (the latter presumably by condensation of atropic acid with aniline), and much tar. A reason for the absence of a lactone-lactam is suggested below.

The olefinic acid formed from (II) on pyrolysis is actually a mixture of tiglic and α-ethylacrylic acid. Angelic acid, though a formal possibility, is absent: if formed initially, it must presumably have undergone its known thermal inversion [3] to the more stable geometrical isomer, tiglic acid (cf. an analogous result in the pyrolytic formation of the corresponding olefinic nitriles [4]).

Pyrolysis of acid (IV), which like (III) does not produce a lactone-lactam and is structurally precluded from undergoing primary scission to olefinic acid, yields aniline, benzophenone, carbon monoxide and dioxide, and much tar: also, diphenylacetic acid is obtained from the aqueous alkali used in working-up the products. Previous work [5] on the pyrolysis of benzilide (VIII) suggests that these products are best accounted for by the following reaction, with intermediate formation of the cyclic dilactone (VIII):



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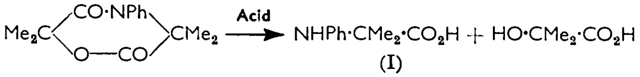
[2] Plant and Facer, *J.*, 1925, **127**, 2037.
[3] Blaise, *Ann. Chim. Phys.*, 1907, **11**, 111.
[4] Bennett, Deans, Harris, Ritchie, and Shim, *J.*, 1958, 4508.
[5] Hurd, "The Pyrolysis of Carbon Compounds," Chemical Catalog Co., New York, 1929, p. 434.

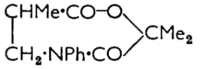
It is possible, in addition, that (VIII) may in part break down directly to carbon monoxide and benzophenone (cf. pyrolysis of simple lactides (6)).

The lactone-lactam (IX) (m. p. 116—120°) from acid (I), unlike those from the cyclic anilino-acids,[1] is readily hydrolysed by sulphuric acid at both the ester and the amide linkage, yielding acid (I) itself and the hydroxy-acid:

(IX)



$NHPh\cdot CH_2\cdot CHMe\cdot CO_2H$ (X)



(XI)

This confirms the view [1,2] that such lactone-lactam rings are six- rather than seven-membered, though the latter structure is suggested by earlier work by Bischoff and Mintz.[7] These authors ascribed the β-anilino-structure (X) to the acid obtained by them from aniline and ethyl α-bromo-α-methylpropionate, and by von Tiemann [8] from aniline, acetone, and hydrogen cyanide. This is clearly incorrect: the acid must be the α-compound (I), as in fact proposed by von Tiemann: [8] and the pyrolysis product (m. p. 120°) which Bischoff and Mintz [7] obtained from it, and to which they ascribed the seven-membered cyclic structure (XI), is now clearly (IX).

A strong band at 1193 cm.$^{-1}$, assigned [9] to the isopropylidene group, is present in the infrared absorption spectra of the acid (I), the lactone-lactam (IX), and the hydroxy-anilide (V). This provides further evidence for structure (I) rather than (X), the latter lacking the CMe_2 group.

The lactone-lactam (IX) may formally arise from acid (I) either by (*a*) intermolecular elimination of water, followed by intramolecular elimination of aniline, leading to cyclisation (cf. Bain and Ritchie [1]), or (*b*) acylation of unchanged acid (I) by α-methylacrylic acid formed from the latter, followed by cyclisation of the linear intermediate $CH_2{:}CMe\cdot CO\cdot NPh\cdot CMe_2\cdot CO_2H$ (cf. Bischoff and Mintz [7]). Mechanism (*b*) is now preferred, since it can explain the failure of acids (III) and (IV) to yield a lactone-lactam on pyrolysis. Acid (IV) cannot yield the necessary olefinic acid by elimination of aniline: and although acid (III) can do so, the resulting olefinic acid (atropic acid) here undergoes early pyrolytic conversion [10] into tar, *via* α- and β-isatropic acid.

Pyrolysis of acids (I) and (II) at *ca.* 240° yielded very little gas: and to render the results more comparable with previous studies [1] a few runs were made at *ca.* 540°. At this temperature, acids (I) and (II), and the lactone-lactams formed from them at *ca.* 240°, gave carbon monoxide and carbon dioxide as the major gaseous products, the molar ratio $CO : CO_2$ lying within the range *ca.* 2—5 (cf. Table 2). Bain and Ritchie [1] suggested that the carbon monoxide formed from cyclic anilino-acids may have been due to breakdown of the hydroxy-anilides produced from these by thermal rearrangement (a mode of breakdown observed experimentally [1]): but this cannot be the explanation in the present work, since no such rearrangement is shown by the open-chain anilino-acids. A possible origin for the carbon monoxide lies in the primary formation of lactone-lactams, followed by scission of these to anil, ketone, and carbon monoxide (a breakdown analogous to that of simple lactides,[6] discussed above). This idea, though supported by the fact that acetone and ethyl methyl ketone were formed at *ca.* 540° by the lactone-lactams from acids (I) and (II) respectively, is nevertheless at variance with the failure [1] to detect an anil in the pyrolysate. For the present, the origin of the carbon monoxide remains an open question.

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[6] Blaise, *Compt. rend.*, 1904, **138**, 697; Bagard, *Bull. Soc. chim. France*, 1907, **1**, 317.

[7] Bischoff and Mintz, *Ber.*, 1892, **25**, 2326.

[8] von Tiemann, *Ber.*, 1882, **15**, 2042.

[9] Bellamy, " The Infra-red Spectra of Complex Molecules," Methuen, London, 1958, p. 26.

[10] Fittig and Wurster, *Annalen*, 1879, **195**, 147; McKenzie and Wood, *J.*, 1919, **115**, 828.

EXPERIMENTAL

Apparatus: Procedure: Analytical Methods.—These have been described.[1] Substances were pyrolysed by both the static (Table 1) and the semi-micro (Table 2) flow method.

TABLE 1. *Results of static pyrolyses* (1 *hr.*).

Pyrolysand	Acid (I)		Acid (II)	Acid (III)		Acid (IV)
Temp.	220°	240°	240°	240°	180°	240°
Run no.	1	2	4,5 *	6	7	8
Products % : §						
aniline	18·4(35)	18·4(35)	21·5(44)	14·0(36)	7·5(20)	8·3(27)
olefinic acid	—	0·4(1)	1·0(2) †	‡	3·0(5)	Nil
lactone-lactam	17·0(25)	20·0(29)	17·0(24)	Nil	Nil	Nil
anilino-anilide	3·4(4)	—	Nil	Nil	Nil	Nil
tars, waste, losses	61·2	61·2	60·5	86·0	89·5	91·7

* Average of 2 runs. † Mixture of tiglic and α-ethylacrylic acid. ‡ No acid, but trace of atropanilide formed. — Not observed. § Figures in parentheses represent % yield based on conversion into a single constituent: other figures represent % by wt. based on original acid.

TABLE 2. *Results of semi-micro flow pyrolyses* (*at* 540°, *with* 300 *mg. of pyrolysand*).

Pyrolysand	Acid (I)	Lactone-lactam from (I) *	Acid (II)	Lactone-lactam from (II) †
‡ Gas formed (ml.)	37·0(99)	32·0(59)	34·0(97)	48·0(98)
§ Gas analysis (% by vol.):				
CO	66(58)	63(31)	60(43)	72(62)
CO_2	24(21)	22(11)	30(21)	15(13)
Unsat. hydrocarbons	3	7	5	5
H_2	7	8	5	8
CO : CO_2 ratio (moles)	2·8	2·9	2·0	4·8

Acetone (*) and ethyl methyl ketone (†) detected in pyrolysate from lactone-lactam. Figures (‡) in parentheses represent % yield, calculated on assumption that one molecule of acid and one molecule of lactone-lactam yield respectively one and two molecules of gas. Figures (§) in parentheses represent % yield based on conversion into a single constituent.

Preparation of Pyrolysands.—The α-anilino-carboxylic acids (I—III) were prepared by hydrolysing the corresponding 1-anilino-nitriles (made by von Walther and Hübner's method [11]), first to the amide by sulphuric acid (cf. Plant *et al.*[2,12]) and then to the acid by hydrochloric acid. α-Anilino-α-methylpropionic acid (I) (overall yield 17%) had m. p. 184—185° (lit.,[11] m. p. 184—185°). α-*Anilino-α-methylbutyric acid* (II) (10%) had m. p. 144°, forming needles from benzene (Found: C, 68·7; H, 7·9; N, 7·3. $C_{11}H_{15}NO_2$ requires C, 68·4; H, 7·8; N, 7·3%). α-*Anilino-α-phenylpropionic acid* (III) (12%), m. p. 146°, formed needles from carbon tetrachloride (Found: C, 74·3; H, 6·3; N, 6·0. $C_{15}H_{15}NO_2$ requires C, 74·5; H, 6·3; N, 5·8%). α-Anilinodiphenylacetic acid (IV), prepared (6%) as described by Stollé,[13] by the action of aniline on the product formed by treatment of benzilic acid with thionyl chloride, crystallised in needles, m. p. 174°, from benzene (lit.,[13] m. p. 174°). α-Hydroxy-α-methylpropionanilide (V), prepared (16%) by heating α-hydroxy-α-methylpropionic acid with aniline, had m. p. 132—133° (lit.,[14] m. p. 132—133°).

Pyrolysis of Acid (I).—*Run* 1. Pyrolysed in the static system, acid (I) (44 g.: 220°, 1 hr.) yielded on distillation fractions (i) 9·5 g., b. p. 150—200° [containing water, aniline (7·5 g.), and a solid A (0·5 g.), m. p. 116—120°], and (ii) a high-boiling residue (30 g.) which was partly soluble in aqueous sodium carbonate. The aqueous extract, when acidified, yielded 3 g. of crude solid which on recrystallisation gave a further 1·5 g. of material A, m. p. 116—120°; the alkali-insoluble residue was partly soluble in hydrochloric acid. The acidic extract contained aniline (0·5 g.), and when made alkaline gave 1·5 g. of crude α-anilino-α-methylpropionanilide (VI) (m. p. 154—156°, from methanol–water: lit.,[15] m. p. 155°) (Found: C, 75·8; H, 6·8;

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[11] von Walther and Hübner, *J. prakt. Chem.*, 1916, **93**, 119.

[12] Betts, Muspratt, and Plant, *J.*, 1927, 1310.

[13] Stollé, *Ber.*, 1910, **43**, 2471.

[14] Tiegerstedt, *Ber.*, 1892, **25**, 2919.

[15] " Dictionary of Organic Compounds," eds. Heilbron and Bunbury, Eyre and Spottiswoode Ltd., London, 1934.

N, 11·0. Calc. for $C_{16}H_{18}N_2O$: C, 75·6; H, 7·1; N, 11·0%). The acid-insoluble residue, crystallised from benzene–light petroleum, yielded 15 g. of crude solid which on recrystallisation gave 7 g. of pure substance A, as large plates, m. p. 116—120°. (There was also a tarry residue, not studied further.) Substance A was the lactone-lactam (IX), *tetrahydro*-2,2,5,5-*tetramethyl*-3,6-*dioxo*-4-*phenyl*-1,4-*oxazine* [Found: C, 68·3; H, 7·1; N, 5·6%; *M* (cryoscopic in benzene), 243. $C_{14}H_{17}NO_3$ requires C, 68·0; H, 6·9; N, 5·7%; *M*, 247]. Hydrolysis of this compound (IX) by 4N-sulphuric acid for 25 hr., followed by neutralisation and extraction with ether, yielded acid (I) (mixed m. p.): the liquid remaining after ether-extraction, evaporated to dryness, yielded α-hydroxy-α-methylpropionic acid, m. p. and mixed m. p. 79° (from benzene).

Run 2. A second run was made in an attempt to detect the predicted olefinic acid, not identified in Run 1. Acid (I) (25 g.; 240°, 1 hr.), when pyrolysed and distilled, yielded a liquid (i) (5 g.), b. p. 140—220°, and (ii) a high-boiling residue. Liquid (i) was dissolved in ether and freed from aniline (4·1 g.) by extraction with acid. The ether layer yielded a semi-solid residue, treatment of which with benzene removed α-methylacrylic acid (infrared) and left a resinous residue of (presumably) poly-(α-methylacrylic acid). Product (ii) yielded further aniline (0·5 g.) and pure lactone-lactam (IX), m. p. 116—120° (5 g.): but material (ii) was completely soluble in benzene, unlike the hydroxy-anilide (V), and on examination by infrared spectrometry showed no evidence (*e.g.*, the strong hydroxyl band at 3279 cm.$^{-1}$) that any of this anilide (V) was present.

Run 3. Bain and Ritchie [1] observed that rearrangement of 1-anilinocycloalkanecarboxylic acids is enhanced by prolonged heating at lower temperatures. However, an additional run with acid (I) at 160° for 48 hr. yielded only a large amount of tar, from which a little of the anilino-anilide (VI), m. p. 155—156°, was isolated, but no trace of the α-hydroxy-anilide (V).

Pyrolysis of Acid (II).—*Run* 4. When pyrolysed in the static system, acid (II) (40 g.; 240°, 1 hr.) yielded by distillation a liquid (i) (9·7 g.), b. p. 140—190°, and (ii) a high-boiling residue. Liquid (i), which contained water, was dissolved in ether: treatment with acid removed aniline (7·7 g.), and the residual brown liquid from the ether layer, cooled to 0°, deposited crude tiglic acid which on recrystallisation from hot water gave the pure acid, m. p. and mixed m. p. 58—62° (lit.,[16] m. p. 64°). Product (ii), extracted with hydrochloric acid, yielded further aniline (0·5 g.): the residue, treated with benzene–light petroleum, gave a crude lactone-lactam, m. p. 117° (7 g.), which on recrystallisation yielded 2,5-*diethyltetrahydro*-2,5-*dimethyl*-3,6-*dioxo*-4-*phenyl*-1,4-*oxazine* (5 g.) as plates, m. p. 124—125° [Found: C, 69·8; H, 7·9; N, 5·0%; *M* (cryoscopic in benzene), 270, 279. $C_{16}H_{21}NO_3$ requires C, 69·8; H, 7·7; N, 5·1%; *M*, 275].

Run 5. To find whether tiglic acid was accompanied by either of its isomers (angelic and α-ethylacrylic acid), a further run was made. The total pyrolysate from acid (II) (21 g.; 240°, 1 hr.) was dissolved in ether and extracted first with dilute aqueous sodium carbonate and then with dilute hydrochloric acid. The alkaline extract was divided into two portions. One was oxidised by potassium permanganate: the product was extracted with ether, and contained acetic and propionic acid (gas–liquid chromatography), the latter confirming the presence of α-ethylacrylic acid. The other portion, acidified and extracted with ether, yielded tiglic acid (*ca.* 0·05 g.; mixed m. p.) and a brown liquid (*ca.* 0·1 g.) which yielded no definite information by infrared spectrometry. The acid extract from the original pyrolysate yielded aniline (4·7 g.) and crude lactone-lactam (6 g.), the latter giving 4 g. of pure product, m. p. 124—125°, on recrystallisation. There was a tarry residue. No evidence could be obtained for the presence of either angelic acid or the hydroxy-anilide in the pyrolysate.

Pyrolysis of Acid (III).—*Run* 6. When pyrolysed in the static system, acid (III) (50 g.; 240°, 1 hr.) yielded by distillation (i) a liquid (10 g.) and (ii) a large tarry residue. The liquid (i) consisted of water (*ca.* 1·5 g.) and aniline, the latter being separated (*ca.* 7 g.) by treatment with ether and hydrochloric acid. The acid extract, rendered alkaline, yielded 0·2 g. of atropanilide, m. p. 132° (from methanol–water) (lit.,[16] m. p. 134°) (Found: C, 80·8; H, 5·9; N, 5·9. Calc. for $C_{15}H_{13}NO$: C, 80·8; H, 5·9; N, 6·3%). The tarry residue (ii) was treated with benzene: the only solid product isolated was a trace of α-isatropic acid, m. p. 238° [lit.,[15] m. p. 239° (decomp.)], no atropic acid being detected.

Run 7. Since it is known [10] that a viscous residue of α- and β-isatropic acid is formed on slow distillation of atropic acid, a further run was made at a much lower temperature in an effort to reduce tar formation. The total pyrolysate from acid (III) (40 g.; 180°, 1 hr.) was

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[16] von Braun and Rudolph, *Ber.*, 1934, **67**, 269.

treated with ether, without previous distillation. There was a resinous residue (5·6 g.), from which nothing definite could be isolated; the ether extract, treated with acid, yielded aniline (3 g.), and on further treatment with aqueous sodium carbonate yielded crude atropic acid (1·2 g.), m. p. 106° (from ethanol) (lit.,[15] m. p. 106—107°). (This acid, heated in a test tube and then extracted with benzene, yielded α-isatropic acid, m. p. 238°.) The residual ethereal solution, evaporated to dryness, left a tar: this was dissolved in benzene–light petroleum, and after a week deposited traces of a solid (A), m. p. 204°. Evaporation of the mother-liquor yielded traces of a second solid (B), m. p. 164°. Recrystallised from methanol, these yielded pure nitrogen-free products A, m. p. 206°, and B, m. p. 168° [Found, for A: C, 78·7; H, 5·8; O (direct), 15·7. Found for B: C, 78·7, 78·4; H, 5·8, 6·2; O (direct), 14·3. $C_{7n}H_{6n}O_n$ requires C, 79·2; H, 5·7; O, 15·1%]. Both *substances* were saturated, and yielded no 2,4-dinitrophenylhydrazone: and compound B resisted attenpts at acidic or alkaline hydrolysis. The quantities were too small to permit further study and the compounds remain unidentified.

Pyrolysis of Acid (IV).—*Run* 8. When pyrolysed in the static system, acid (IV) (6 g.; 240°, 1 hr.) yielded a total pyrolysate which on treatment with ether left a resinous residue (0·6 g.). Extracted with hydrochloric acid, the ethereal solution yielded aniline (0·5 g.); extracted with aqueous sodium carbonate, it yielded diphenylacetic acid (0·8 g.), m. p. 148° (mixed m. p.; infrared). The ethereal residue finally yielded a tar, not further studied. There was no evidence for the presence of benzilide (VIII) or a lactone-lactam.

Run 9. A further run was made in a search for benzophenone. The total pyrolysate from acid (IV) (1 g.; 240°, 1 hr.) was taken up in methanol and treated with 2,4-dinitrophenylhydrazine: the appropriate derivative of benzophenone was formed (mixed m. p.).

Pyrolysis of α-Hydroxy-α-methylpropionanilide (V).—Substance (V), when heated at 240° (1 hr.) in the static reactor, remained unchanged.

Semimicro-pyrolyses.—Acids (I) and (II), and the corresponding lactone-lactams, were pyrolysed in the semimicro-apparatus (Table 2).

New Infrared Data.—The following are the principal absorption bands of the compounds studied.

Acid (I): 1618s, 1495m, 1404s, 1359s, 1218m, 1193m, 1156m, 1030m, 917m, 897m, 819m, 806m, 782m, 741s, 693s cm.$^{-1}$ (Assignments: 1618 cm.$^{-1}$, CO_2^-; 1193 cm.$^{-1}$, CMe_2 [9]).

Acid (II): 1616s, 1486m, 1391s, 1359s, 1325m, 1305w, 1284m, 1183w, 1145m, 1135w, 1025m, 885m, 797m, 763w, 735s, 692s cm.$^{-1}$ (Assignment: 1616 cm.$^{-1}$, CO_2^-).

Acid (III): 1650s, 1613s, 1558w, 1495s, 1389s, 1361s, 1511w, 1282w, 1258w, 1208w, 1183w, 1156w, 1130w, 1075w, 1029m, 912w, 881m, 797w, 766w, 739s, 694s cm.$^{-1}$ (Assignment: 1650 cm.$^{-1}$, acid C=O; 1613 cm.$^{-1}$, CO_2^-; 1558 cm.$^{-1}$, benzene ring).

Acid (IV): 1626s, 1493sh, 1326s, 1279sh, 1220w, 1183sh, 1170w, 1156sh, 1081w, 1026sh, 985w, 905w, 890w, 833w, 785w, 782w, 746s, 722s, 687s cm.$^{-1}$ (Assignment: 1626 cm.$^{-1}$, CO_2^-).

Lactone-lactam from acid (I): 1745s, 1661s, 1595w, 1490w, 1406s, 1389sh, 1305s, 1250m, 1220m, 1190m, 1147s, 1053m, 759s, 702s cm.$^{-1}$.

Lactone-lactam from acid (II): 1727s, 1667s, 1590m, 1495m, 1437m, 1416s, 1340s, 1309s, 1259s, 1233s, 1211m, 1176m, 1156s, 1130s, 1080m, 1070m, 1036s, 1030sh, 1022 sh, 1000m, 976w, 961m, 855m, 848sh, 812w, 806w, 761s, 735w, 702s cm.$^{-}$ (Assignments: 1745, 1727, 1667, 1661cm.$^{-1}$, lactone C=O; 1595, 1590 cm.$^{-1}$, benzene ring).

Hydroxy-anilide (V): 3279s, 1667s, 1608s, 1555s, 1504w, 1490w, 1449s, 1416w, 1361w, 1333w, 1325m, 1235m, 1190s, 1167sh, 1156s, 976s, 926m, 908m, 761s, 751s, 693s cm.$^{-1}$ (Assignments: 3279 cm.$^{-1}$, OH; 1667 cm.$^{-1}$, NH).

The authors thank the Department of Scientific and Industrial Research for the award of a Research Studentship (to J. McGee).

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[*Received, September* 13*th*, 1960.]