parent peak at *m/e* 149 [calcd for $C_{10}H_{15}N$; 149.1204; observed 149.1206].

***p*-Methoxy-*N,N*-dimethylphenethylamine** was prepared from *p*-methoxyphenethylamine in a like manner. It was purified by trapping from glc from a 6 ft SE-30 column at 175°. It was identified by ir and mass spectrum, *m/e* 179(P), 135, 107, 91, 77, 58.

***N,N*-Dimethylpropylamine** was prepared from propylamine by the Leukhart reaction. It was purified by distillation and by trapping the distillate from glc. The material showed refractive index n_0^{25} 1.890 and was unreactive with benzoyl chloride.

1-Phenyl-2-methyl-2-(dimethylamino)propanol was prepared according to a procedure described in the literature[21] by reducing 2-dimethylamino-2-methylpropiophenone hydrochloride with Raney Ni in methanol: bp 82–85° (0.5 mm); mp 54–55° (colorless crystals from methanol); nmr (CCl_4) δ 7.25 (aromatic, m, 5), 4.55 (HC, s, 1), 2.25 [$(CH_3)_2N$, s, 6], 0.75, 0.82 [$(CH_3)_2N$, s, 2 × 3 H]; ir (neat) ν 3400 cm^{-1} (OH). 2-Dimethylamino-2-methylpropiophenone was obtained by a procedure described for similar compounds[22] by reacting α-bromoisobutyrophenone[22] with NaOMe in MeOH to obtain 1-methoxy-1,2-epoxisobutylbenzene.[23] Subsequent reaction with dimethylamine at 200° under pressure yielded 2-dimethylamino-2-methylpropiophenone: bp 71–73° (0.5 mm); nmr (CCl_4) δ 8.55 (aromatic, m, 2), 7.45 (aromatic, m, 3), 2.2 [$(CH_3)_2N$, s, 6], 1.2 [$(CH_3)_2C$, s, 6]; ir (neat) ν 1700 cm^{-1} (C=O) *Anal.* Calcd for $C_{12}H_{17}NO$: C, 74.51; H, 9.9; N, 7.25. Found: C, 74.80; H, 9.78; N, 7.45.

Procedures. Cyclic voltammetry results were obtained using a three-electrode potentiostat with 0.3 V/sec sweep rate. All experiments involved reactions of approximately 10 m*M* amine in 0.1 *M* $NaClO_4$–MeCN at a Pt wire microelectrode. The reference electrode was Ag/$AgNO_3$ (0.1 *M*, MeCN) separated from the reaction solution by an asbestos fiber junction.

Coulometry and preparative electrolyses were performed using apparatus and techniques similar to those previously described.[2] The cells used in this work had anode compartments of 25- and 190-ml capacities. When exclusion of atmospheric oxygen was critical, a cell with high vacuum fittings was used. Its ability to exclude atmospheric oxygen was specifically checked. Comments concerning oxygen consumption in the outline of results should be understood to imply changes significantly larger than those attributable to leakage.

Reactions were generally performed with approximately 10 m*M* initial amine concentration, 40 m*M* water concentration, and 0.1 *M* $NaClO_4$ supporting electrolyte in MeCN. When water was excluded, the procedure previously described was used.[2]

Acknowledgment. The authors wish to acknowledge financial supprort from the National Institutes of Health through Grant NS 10528.

Registry No.—*N,N*-Dimethylphenylacetamide, 18925-69-4; 1,2-epoxyethylbenzene, 20780-53-4; dimethylamine, 124-40-3; ω-bromo-4-methoxyacetophenone, 2632-13-5; veratrole, 91-16-7; dimethylaminoacetonitrile hydrochloride, 3976-11-2; formaldehyde, 50-00-0; formic acid, 64-18-6; 1-methoxy-1,2-epoxyisobutylbenzene, 13694-96-7.

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Formation and Reactions of *N*-Alkyl-2,2-dichlorobenzoylacetanilides

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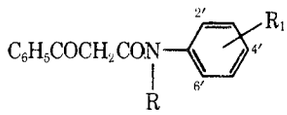
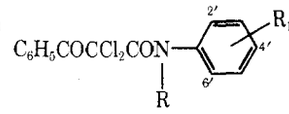
N-Alkylbenzoylacetanilides **1a** exhibit a marked deactivation toward nuclear chlorination by sulfuryl chloride as compared to the corresponding N-dealkylated substrates. The acid-catalyzed cyclization of certain *N*-isopropylbenzoylacetanilides to the corresponding 1-isopropylquinolinones is described. Another example of an apparent intramolecular, two-substituent, migration accompanying indenoquinolinone synthesis is reported.

Little is known of the effect of sulfuryl chloride on substrates of type **1a**.[1] When refluxed with a large excess of sulfuryl chloride for 1 hr the *N*-alkylbenzoylacetanilides **1c–e** afforded the corresponding 2,2-dichloro derivatives **2a** free of trichloro impurity. The more reactive *N*-alkyl-3′,5′-dimethylbenzoylacetanilides **1h** and **1i** gave the 2,2,4′-trichloro products **2k** and **2m**, respectively, contaminated with minor impurity after 15 min of heating, and with appreciable tetrachloro material after reaction for 40 min. Acid hydrolysis of **2k** and **2m** to the corresponding *N*-alkyl-4-chloro-3,5-dimethylaniline confirmed the 4′-substitution. Compounds **1h** and **1i**, when chlorinated under less drastic conditions (in chloroform solution at room temperature with a 3–5 *M* proportion of sulfuryl chloride), provided the 2,2-dichloro derivatives **2j** and **2l** practically free of trichloro anilide.

In contrast, **1b** was converted by hot, excess sulfuryl chloride into **2b**,[1] while **1g**, when treated with a 6 *M* amount of reagent at room temperature, yielded **2i**.[1,2] Evidently, in comparison with the parent compounds **1b** and **1g**, the N-alkylated anilides **1a** are severely deactivated toward nuclear chlorination. The observed nuclear susceptibility of **1b** and **1g** may be explained by N–H hyperconjugation[3] or in terms of the additional tautomeric and reso-



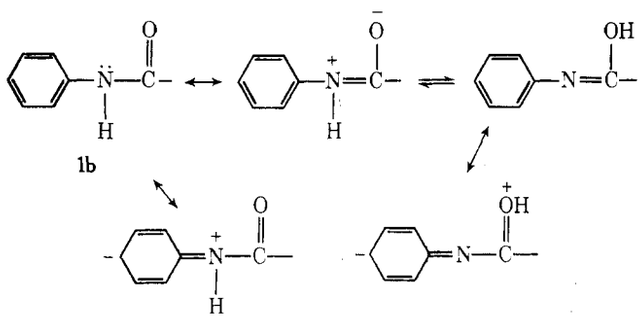
1a, R = alkyl;
R_1 = H or 3′,5′-diCH_3
b, R = R_1 = H
c, R = CH_3; R_1 = H
d, R = C_2H_5; R_1 = H
e, R = *i*-C_3H_7; R_1 = H
f, R = *i*-C_3H_7; R_1 = 2′-CH_3
g, R = H; R_1 = 3′,5′-diCH_3
h, R = C_2H_5; R_1 = 3′,5′-diCH_3
i, R = *i*-C_3H_7; R_1 = 3′,5′-diCH_3
j, R = H; R_1 = 2′-Cl-3′,4′-diCH_3



2a, R = alkyl; R_1 = H
b, R = H; R_1 = 4′-Cl
c, R = CH_3; R_1 = H
d, R = C_2H_5; R_1 = H
e, R = *i*-C_3H_7; R_1 = H
f, R = *i*-C_3H_7; R_1 = 2′-CH_3
g, R = H; R_1 = 3′,5′-diCH_3
h, R = H; R_1 = 4′-Cl-3′,5′-diCH_3
i, R = H;
R_1 = 2′,4′-diCl-3′,5′-diCH_3
j, R = C_2H_5; R_1 = 3′,5′-diCH_3
k, R = C_2H_5;
R_1 = 4′-Cl-3′,5′-diCH_3
l, R = *i*-C_3H_7; R_1 = 3′,5′-diCH_3
m, R = *i*-C_3H_7;
R_1 = 4′-Cl-3′,5′-diCH_3
n, R = H; R_1 = 2′-Cl-3′,4′-diCH_3

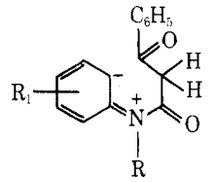
nance contributions that may be derived from **1b** and **1g** (Scheme I). It is anticipated that **1a** will likewise exhibit deactivation toward other electrophilic reagents.

Scheme I

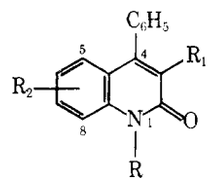


A specimen of 2,2,4′-trichloro-3′,5′-dimethylbenzoylacetanilide (**2h**) was prepared for the present study by dropwise addition of sulfuryl chloride to a dilute chloroform solution of **1g** and repeating this treatment on the 2,2-dichloro intermediate **2g** which was contaminated with both the mono and trichloro derivatives.

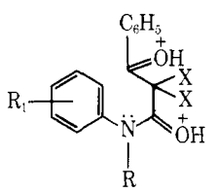
The effect of acid on the newly acquired **1** and **2** was examined. Contrary to the finding of Koelsch and Britain,[4] concentrated sulfuric acid at 95° converted **1e** into 4-phenyl-1-isopropylquinolin-2-one (**4a**), albeit in poor (*ca.* 10–15%) yield, in a slow, incomplete reaction. The synthesis of a 1-isopropylquinolinone from a *N*-isopropyl anilide was effected more successfully with PPA at 135–140°, and **4a** was isolated in 25% yield after separation from unchanged **1e**. The cyclization of anilide **1i** in PPA at 125–135° proceeded more readily, and 5,7-dimethyl-4-phenyl-1-isopropylquinolin-2-one (**4b**) was isolated in 40% yield. In contrast, **1f** and PPA under similar conditions produced little if any quinolinone. The structural assignments **4a** and **4b** were supported by the (nmr and mass) spectral evidence. In addition, when heated with 70% sulfuric acid, **4a** was dealkylated to **4c**. The mass spectra of the 1-isopropylquinolinones **4a** and **4b** both exhibited a substantial peak at *m/e* M − 42, reflecting the loss from the structures of C_3H_6 *via* a McLafferty rearrangement (*cf.* ref 5). A representative 2,2-dichloro-*N*-isopropylanilide, *viz* **2e**, and sulfuric acid furnished several products, including quinolinone **4d** in *ca.* 20% yield.



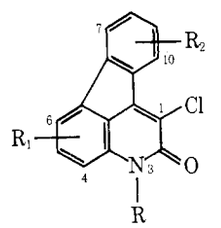
3a, R = *i*-C_3H_7; R_1 = H



4a, R = *i*-C_3H_7; R_1 = R_2 = H
b, R = *i*-C_3H_7; R_1 = H; R_2 = 5,7-diCH_3
c, R = R_1 = R_2 = H
d, R = *i*-C_3H_7; R_1 = Cl; R_2 = H
e, R = H; R_1 = Cl; R_2 = 5,7-diCH_3
f, R = H; R_1 = Cl; R_2 = X-Cl-5,7-diCH_3
g, R = C_2H_5; R_1 = Cl; R_2 = 5,7-diCH_3
h, R = R_1 = H; R_2 = 5,7-diCH_3
i, R = H; R_1 = Cl; R_2 = 8-Cl-6,7-diCH_3



5a, X = H
b, X = Cl



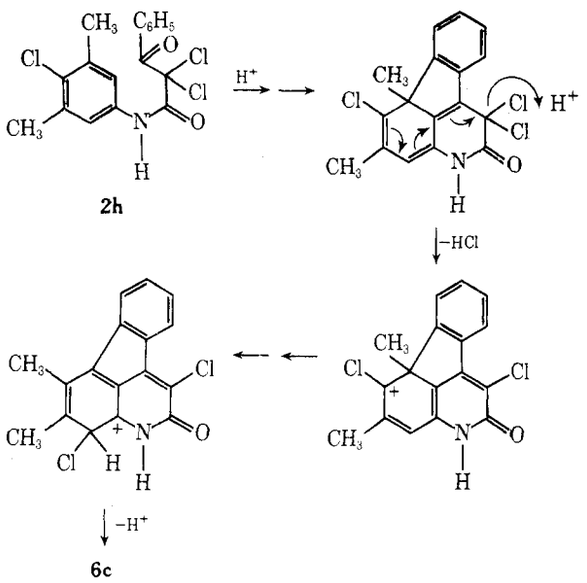
6a, R = R_2 = H; R_1 = 6-Cl-4,5-diCH_3
b, R = R_2 = H; R_1 = 5-Cl-4,6-diCH_3
c, R = R_2 = H; R_1 = 4-Cl-5,6-diCH_3
d, R = R_1 = H; R_2 = 8-Cl-7,9-diCH_3
e, R = C_2H_5; R_2 = H; R_1 = 4-Cl-5,6-diCH_3
f, R = R_2 = H; R_1 = 4-Cl-5-CH_2Cl-6-CH_3

In their account of the failure of **1e** to give **4a**, Koelsch and Britain[4] postulated a species **3a** as an intermediate in the reaction. We suggest, however, that in excess sulfuric acid or PPA, the anilide **1** (or **2**) is diprotonated[6] to **5a** (or **5b**), thereby militating against the presence of **3**. The reluctance of **1e** to cyclize is now ascribed to the presence of the bulky N substituent in **5a** (R = *i*-C_3H_7; R_1 = H) hindering the attainment of the requisite cyclic conformation for reaction. This steric effect, which apparently is more pronounced in **1f**, is compensated for somewhat in **1i** by the increased susceptibility of the arylamino moiety to undergo electrophilic attack.

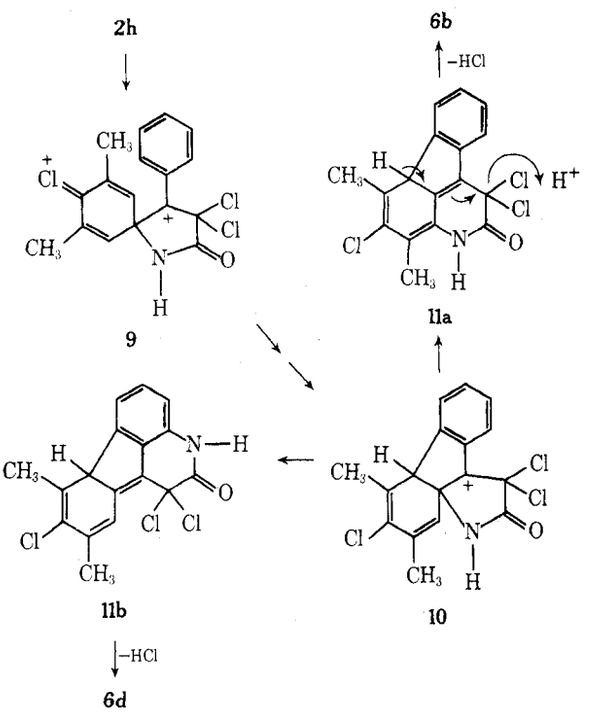
We wish to report here another example of an apparent intramolecular two-substituent migration[2] accompanying indenoquinolinone synthesis. The reaction of **2h** with sulfuric acid yielded (~30%) a product A of molecular formula $C_{17}H_{11}Cl_2NO$. The nmr and ir spectra were consistent with either of the indeno[1,2,3-*de*]quinolin-2-one structures **6a–d.** Compound A was nonidentical with, and was free from (ir spectrum), the known[2] isomeric **6a** and **6b.** This conclusion was confirmed by similarly comparing the respective 2-chloro derivatives. Product A is tentatively regarded as **6c.** In an attempt to prepare **6c,** for comparing directly with A 2,2,2′-trichloro-3′,4′,-dimethylbenzoylacetanilide (**2n**) was treated with sulfuric acid. The reaction, however, afforded mainly quinolinone **4i,** and no indenoquinolinone material was isolated. A possible reaction pathway for the formation of A, involving multiple 1,2 shifts, is outlined in Scheme II. Alternative mechanistic routes may be visualized[7] which involve "spiro" intermediates[8] (Scheme III). However, the cyclization of **2h** *via* **9–11a** would result in A having structure **6b,** while reaction *via* **9–11b** (*i.e.*, "trans" cyclization[9]) would lead to assignment **6d** and is not favored.

A related product B, $C_{19}H_{15}Cl_2NO$, was isolated (~35% yield) when the *N*-ethyl homolog, **2k,** was similarly reacted. The presence of two intact methyl groups in the structure was confirmed from the nmr spectrum, which also

Scheme II



Scheme III



showed the *N*-ethyl moiety. The formulation of B as **6e** is supported by the strong ir absorptions at 1650 cm^{-1} (tertiary amide) and at 760 cm^{-1} (four adjacent aromatic protons). This assignment implies that B, like A, is the product of a "direct cyclization–intramolecular migration" reaction. In the light of the above, an alternative structure **6f**, for the product[2] derived from **2i** and sulfuric acid, merits consideration.

Experimental Section

Melting points were determined with a Kofler hot-stage apparatus and are uncorrected. Infrared spectra for KBr disks were recorded with a Perkin-Elmer Infracord spectrophotometer. Mass spectra were obtained with a Varian CH-5 instrument at 70 eV unless otherwise stated. Nmr spectra (~30 mg, 0.4 ml of solvent) were measured with a Hitachi Perkin-Elmer R-20 spectrometer with tetramethylsilane as internal reference. Solvents and excess SO_2Cl_2 were removed on a rotary evaporator under reduced pressure and at a bath temperature under 40°. Thin-layer chromatography (tlc) was performed on silica gel F-254 precoated plates (E. Merck, Darmstadt) of 0.25 mm thickness. Spots were visualized with an ultraviolet lamp (compounds **4** and **6** fluoresced blue and yellow, respectively, at 366 nm) or in an iodine chamber.

Table I

Compd[a]	Formula	Mp, °C
1f	$C_{19}H_{21}NO_2$	118–120
1i	$C_{20}H_{23}NO_2$	107–108
2e	$C_{18}H_{17}Cl_2NO_2$	143–144
2f	$C_{19}H_{19}Cl_2NO_2$	122–123
2j	$C_{19}H_{19}Cl_2NO_2$	99–100
2l	$C_{20}H_{21}Cl_2NO_2$	128–130
2k[b]	$C_{19}H_{18}Cl_3NO_2$	130–132

[a] Colorless crystals; 70–80% yields, except for **1f** (15%), **1h** ($C_{19}H_{21}NO_2$: isolated as an impure oil, 50–55%), **1i** (50%), **2m** ($C_{20}H_{20}Cl_3NO_2$: 60%; mp 100–103°; the product was contaminated with minor amounts of both dichloro and tetrachloro impurities). Satisfactory nmr, mass spectral, and analytical data for all new compounds were obtained. [b] A trace (*ca.* 5%) of **2j** impurity was revealed from the mass spectrum.

***N*-Alkylbenzoylacetanilides (1a).** A mixture of the appropriate *N*-alkylarylamine[10] (0.05 mol) and ethyl benzoylacetate (0.06 mol) was heated with intermittent stirring at 150–160° for 1.5 hr and then cooled somewhat and treated with 2 *M* NaOH (~30 ml). The reaction product gradually solidified and was removed by filtration, washed with water, and crystallized from aqueous MeOH. New **1** are listed in Table I, and all gave a characteristic purple color with alcoholic $FeCl_3$.

***N*-Alkyl-2,2-dichlorobenzoylacetanilides. A. Preparation at Reflux Temperature (~66°).** A solution of the appropriate **1** (0.5 g) in SO_2Cl_2 (5 ml) was heated at reflux for 1 hr. The excess reagent was evaporated and the residual gum or solid was washed with water and crystallized from aqueous EtOH.

Compounds **2c, 2d,** and (new) **2e** were produced free of trichloro impurity, as evidenced from the mass spectra of the respective crude reaction products. Anilides **2k** and **2m** were obtained, albeit not entirely pure (Table I), when **1h** and **1i,** respectively, were refluxed for 15 min. Reaction of **1i** for 40 min gave **2m** contaminated with (*ca.* 20%) tetrachloro material (*m/e* 445).

A mixture of **2k** (1g), EtOH (5 ml), and 70% (w/w) H_2SO_4 (10 ml) was refluxed for 3 hr, then diluted with water and filtered. Basification with NaOH deposited crude 4-chloro-*N*-ethyl-3,5-dimethylaniline. Purification of the amine was effected by reprecipitation from dilute HCl and yielded, after drying under reduced pressure, a viscous gum (~50 mg): nmr ($CDCl_3$) δ 1.2 (t, 3, CH_2CH_3), 2.28 (s, 6, Ar-CH_3), 3.1 (q, 3, CH_2CH_3 and NH), 6.26 (s, 2, aromatic); mass spectrum *m/e* 183 (M^+).

Acid hydrolysis of **2m,** as for **2k,** afforded 4-chloro-*N*-isopropyl-3,5-dimethylaniline as a viscous gum: nmr ($CDCl_3$) δ 1.15 (d, 6, $CH(CH_3)_2$), 2.3 (s, 6, Ar-CH_3), 3.5 (m, 2, $CH(CH_3)_2$ and NH), 6.35 (s, 2, aromatic); mass spectrum *m/e* 197 (M^+).

B. Preparation at Room Temperature (~20°). A solution of SO_2Cl_2 (0.03 – 0.05 mol) in $CHCl_3$ (10 ml) was added dropwise to a solution or suspension of the appropriate **1** (0.01 mol) in $CHCl_3$ (20 ml) with intermittent swirling over 2–3 min. The reaction was allowed to proceed for 15–20 min. Solvent and excess reagent were evaporated and the residue was washed with water and crystallized from aqueous EtOH. Details of the new compounds, **2f, 2j,** and **2l,** are listed in Table I.

2,2-Dichloro-3′,5′-dimethylbenzoylacetanilide (2g). A solution of **1g** (2.7 g) in $CHCl_3$ (40 ml) was stirred at room temperature and treated dropwise with a solution of SO_2Cl_2 (2.5 ml; 3 *M* proportion) in $CHCl_3$ (20 ml) over 1 hr. The product was isolated as in B; colorless crystals (2.4 g) (from aqueous EtOH) composed chiefly (mass spectrum; tlc) of **2g** (*m/e* 335; *ca.* 75%), admixed with the trichloro (*m/e* 369; *ca.* 10%), and monochloro (*m/e* 301; *ca.* 15%) derivatives of **1g.**

2,2,4′-Trichloro-3′,5′-dimethylbenzoylacetanilide (2h). A solution of the impure **2g** (1.7 g) in $CHCl_3$ (15 ml) was stirred at room temperature and treated dropwise with a solution of SO_2Cl_2 (1 ml; 2.5 *M* proportion) in $CHCl_3$ (15 ml) over a period of 30 min. Stirring was continued for an additional 20 min at 40–45°; the progress of the chlorination was monitored by tlc [toluene–chloroform (3:2)]. The solvent and excess reagent were removed and the residual gum was triturated with MeOH when it solidified. Crys-

tallization from aqueous MeOH gave a colorless solid (1.2 g) composed chiefly of **2h** (*m/e* 369; *ca.* 80%) admixed with some **2g** (*m/e* 335; *ca.* 20%) and negligible **2i** (*m/e* 403).

The impure **2h** (0.7 g) was refluxed with 2 *M* NaOH (10 ml) for 1 hr to give 4-chloro-3,5-dimethylaniline (0.2 g): colorless crystals (from aqueous EtOH); mp 57–58° (lit.[11] mp 58–59°); nmr ($CDCl_3$) δ 2.25 (s, 6, Ar-CH_3), 3.40 (broad s, 2, NH_2), 6.35 (s, 2, aromatic); mass spectrum *m/e* 155 (M^+).

5,7-Dimethyl-4-phenyl-1-isopropylquinolin-2-one (4b). A mixture of **1i** (0.27 g) and PPA (5 g) was heated at 125–135° with intermittent stirring for 1 hr and then poured into water. The crude acid-insoluble product contained negligible **1i** (tlc, alcoholic $FeCl_3$) and was chromatographed on silica gel (Kieselgel, Merck; 25 g) with ethyl acetate–petroleum ether (bp 80–100°) (1:5) as the eluent. Evaporation of the appropriate fractions gave **4b** as a gum (0.10 g; 40%). This was distilled at 165–175° (0.1 mm) to afford a colorless glass which eventually solidified: mp 106–108°; ir 1650 cm^{-1} (s, amide CO); nmr ($CDCl_3$) δ 1.68 (d, 6, CH(CH_3)$_2$), 1.80 (s, 3, Ar-CH_3), 2.40 (s, 3, Ar-CH_3), 5.45 (m, 1, CH(CH_3)$_2$), 6.40 (s, 1, aromatic), 6.72 (s, 1, aromatic), 7.0–7.5 (m, 6, aromatic); mass spectrum [*m/e* (rel intensity, species)] taken at 17 eV [291 (1, **4b**), 249 (0.7, **4h**)] and 70 eV [291 (1, **4b**), 249 (2, **4h**)]. *Anal.* Calcd for $C_{20}H_{21}NO$: C, 82.42; H, 7.27; N, 4.81. Found: C, 82.19; H, 7.21; N, 4.93.

4-Phenyl-1-isopropylquinolin-2-one (4a). The reaction of **1e** (0.9 g) with PPA (10 g) at 135–140° for 30 min gave a mixture (tlc) of **4a** and substantial unchanged **1e.** Chromatography or silica gel (40 g), as for **4b,** afforded **4a** as a colorless glass (0.2 g; ~25% yield): nmr ($CDCl_3$) δ 1.80 (d, 6, CH(CH_3)$_2$), 5.65 (m, 1, CH(CH_3)$_2$), 7.3–8.3 (m, 10, aromatic); mass spectra [*m/e* (rel intensity, species)] 12 eV [263 (1, **4a**), 221 (0.3, **4c**)], 16 eV [263 (1, **4a**), 221 (1, **4c**)], and 70 eV [263 (1, **4a**), 221 (2, **4c**)]; *ca.* 10% **1e** impurity (*m/e* 281) was present.

A white solid (possibly **4a** HCl), mp 85–95° dec, was obtained on on addition of concentrated HCl to **4a.**

A mixture of **4a** (50 mg) and 70% (w/w) H_2SO_4 (4 ml) was refluxed for 2.5 hr and then poured into water. The precipitated material (~25 mg) proved to be crude **4c** (ir spectrum, tlc).

The reaction of **1e** (0.5 g) with concentrated H_2SO_4 (1.5 ml) at 95° for 1 hr likewise produced **4a** (mass spectrum; tlc); a 10–15% yield was estimated from the mass spectra (12 eV, 70 eV) of the crude reaction product which contained much unchanged **1e.**

2′-Methylbenzoylacetanilide (**1f;** 0.2 g) was reacted with PPA as for **4b** and also with concentrated H_2SO_4 (1 ml) as for **4a.** Examination of the crude product in each case showed (mass spectrum, tlc, $FeCl_3$) much unchanged **1f** and little if any quinolinone material.

A mixture of **2e** (1g) and concentrated H_2SO_4 (2.5 ml) was heated at 95° for 10 min; reaction occurred with evolution of HCl. The crude acid-insoluble material (0.8 g) contained at least five different products [tlc (benzene–acetone (20:1)] including suspected **4d** [*m/e* 297 (M^+); *ca.* 20% yield].

Indenoquinolinone A. Concentrated H_2SO_4 (1.6 ml) was added to **2h** (0.8 g) when a fair exothermic reaction occurred and HCl was evolved from the permanganate-colored mass. The mixture was heated at ~90° for 4 min and then poured into water. The acid-insoluble product was washed with water and extracted with warm (~50°) EtOH (3 × 50 ml). The residue of **A** (0.2 g; ~30% yield) was crystallized from DMF: yellow crystals; mp > 250°; ir 2900–3200 (broad, NH), 1650 cm^{-1} (s, amide CO), 750 (s, four adjacent aromatic protons); nmr [CF_3COOH–H_2SO_4 (10:1)] δ 2.60 and 2.67 (2s, 6, Ar-CH_3), 7.2–8.3 (m, 4, aromatic); the mass spectrum [*m/e* 315 (M^+)] revealed some (*ca.* 10%) trichloro (*m/e* 349) impurity.

Compound A (0.15 g) was refluxed with $SOCl_2$ (7 ml) and DMF (0.1 g) for 6 hr. The excess reagent was evaporated and the residue of crude 2-chloro derivative was purified by chromatography on silica gel (Kieselgel, Merck; 20 g) with ethyl acetate–petroleum ether (bp 80–100°) (1:4) as eluent: yellow crystals (from DMF); mp 240–250°; ir 745 cm^{-1} (s, four adjacent aromatic protons); nmr (CF_3COOH) δ 2.21 and 2.32 (2s, 6, Ar-CH_3), 6.9–7.6 (m, 3, aromatic), 7.7–8.0 (dd, 1, aromatic); mass spectrum *m/e* 333 (M^+), 298 (M − 35); some (*ca.* 10%) tetrachloro (*m/e* 367) impurity was present.

A similar reaction of **2g** (0.8 g) as for **2h** yielded chiefly the EtOH-soluble quinolinones **4e** [*m/e* 283 (M^+)] and **4f** [*m/e* 317 (M^+)]; a trace (0.02 g) of EtOH-insoluble material [*m/e* 315 (M^+); possibly A] was isolated.

Indenoquinolinone B. Anilide **2k** (0.5 g) was reacted with concentrated H_2SO_4 (1 ml) as for A. The insoluble product obtained after pouring into water was purified by chromatography on silica gel (20 g), eluting with $CHCl_3$–benzene (1:3). Evaporation of the appropriate fractions afforded B (0.15 g; 35% yield): yellow crystals (from DMF); mp 218–220°; ir 1650 (s, amide CO), 760 cm^{-1} (s, four adjacent aromatic protons); nmr ($CDCl_3$) δ 1.41 (t, 3, CH_2CH_3), 2.32 (s, 6, Ar-CH_3; the superimposed singlets were resolved into two distinct peaks in a more dilute solution), 4.55 (q, 2, CH_2CH_3), 7.0–7.7 (m, 3, aromatic), 7.8–8.1 (m, 1, aromatic); mass spectrum *m/e* 343 (M^+), 328 (M − CH_3), 315 (M − C_2H_4, *via* McLafferty rearrangement), 308 (M − Cl), 300 (315 − CH_3), 280 (315 − Cl).

Anal. Calcd for $C_{19}H_{15}Cl_2NO$: C, 66.29; H, 4.39; N, 4.07. Found: C, 66.51; H, 4.34; N, 3.89.

A similar reaction of **2j** (0.3 g) as for **2k** afforded several products (tlc) including **4g** (*m/e* 311) and a trace of indenoquinolinone material (*m/e* 309).

Attempted Preparation of Indenoquinolinone 6c. 2,3-Dimethylaniline (Schuchardt; 24 g) was transformed into a mixture (5.5 g; 3 spots on tlc) of isomeric chloroxylidines by the procedure described[2] for 3-chloro-2,4-dimethylaniline. The mixture (4.5 g) was separated on a silica gel (130 g) column using ethyl acetate–petroleum ether (bp 80–100°) (1:3) as eluent. First eluted was 2-chloro-3,4-dimethylaniline [oil; 1.2 g; nmr ($CDCl_3$) 2.16 (s, 3, Ar-CH_3), 2.26 (s, 3, Ar-CH_3), 3.7 (broad s, 2, NH_2), 6.46 (d, 1, J = 8 Hz, aromatic 6-H), 6.80 (d, 1, J = 8 Hz, aromatic 5-H); acetyl derivative, mp 108–110° (lit.[12] mp 114°)] followed by 4-chloro-2,3-dimethylaniline [oil; 2.5 g; nmr ($CDCl_3$) 2.03 (s, 3, Ar-CH_3), 2.28 (s, 3, Ar-CH_3), 3.45 (broad s, 2, NH_2), 6.40 (d, 1, J = 8 Hz, aromatic 6-H), 7.0 (d, 1, J = 8 Hz, aromatic 5-H); acetyl derivative, mp 151–152° (lit.[13] mp 150°)]. The former amine (1.2 g) was condensed[2] with ethyl benzoylacetate to give **1j** (1.2 g; mp 168–170°), sparingly soluble in 2 *M* NaOH. Anilide **1j** (1.0 g) was treated[2] with SO_2Cl_2 to give **2n** (0.9 g; mp 140–142°). Nmr and mass spectra served to substantiate the structures **1j** and **2n.** A mixture of **2n** (0.7 g) and concentrated H_2SO_4 (1.5 ml) was reacted at 95° for 4 min and poured into water. The acid-insoluble product (~0.2 g) was shown from its mass spectrum and tlc to contain much **4i** (*m/e* 317). Negligible **6c** was produced in the reaction.

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Registry No.—**1e,** 52827-48-2; **1f,** 52827-49-3; **1g,** 40624-75-7; **1h,** 52827-50-6; **1i,** 52827-51-7; **1j,** 52827-52-8; **2e,** 52827-53-9; **2f,** 52827-54-0; **2g,** 52827-55-1; **2h,** 52827-56-2; **2j,** 52827-57-3; **2k,** 52827-58-4; **2l,** 52827-59-5; **2m,** 52827-60-8; **2n,** 52873-71-9; **4a,** 52827-61-9; **4a** HCl, 52827-62-0; **4b,** 52827-63-1; **6c,** 52827-64-2; **6e,** 52827-65-3; *N*-isopropylaniline, 768-52-5; *N*-isopropyl-2-methylaniline, 2100-43-8; 3,5-dimethylaniline, 108-69-0; *N*-ethyl-3,5-dimethylaniline, 13342-22-8; *N*-isopropyl-3,5-dimethylaniline, 5287-66-4; ethyl benzoylacetate, 94-02-0; 4-chloro-*N*-ethyl-3,5-dimethylaniline, 52827-67-5; 4-chloro-*N*-isopropyl-3,5-dimethylaniline, 52827-68-6; 4-chloro-3,5-dimethylaniline, 51719-61-0; 2,3-dimethylaniline, 87-59-2; 2-chloro-3,4-dimethylaniline, 52827-69-7; 4-chloro-2,3-dimethylaniline, 52827-70-0.

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