evaporated *in vacuo* and two 75-ml portions of toluene were added and removed *in vacuo*. Trituration of the residue with 50 ml of water gave 5.38 g (93%) of **11** as a white crystalline powder, mp 264.5–265.5°, homogeneous and suitable for use in the next step. Two recrystallizations from water afforded the analytical sample of **11**: mp 267–268°; $\lambda_{max}^{pH\ 1}$ 248 mμ (ε 11,900), $\lambda_{max}^{pH\ 7}$ 248 mμ (ε 12,300), $\lambda_{max}^{pH\ 13}$ 253 mμ (ε 13,200); $[\alpha]_{589}^{22}$ −20° (*c* 0.5, pyridine); it moved as one spot in solvent systems A, B, and C with R_{Ad} 1.34, 2.16, and 1.43, respectively.

Anal. Calcd for $C_{15}H_{18}N_4O_7$: C, 49.2; H, 4.95; N, 15.3. Found: C, 49.2; H, 5.03; N, 15.5.

9-(2,5-Di-O-acetyl-3-O-methyl-β-D-ribofuranosyl)-6-mercaptopurine (12).—A suspension of 1.83 g (5.0 mmoles) of **11**, 75 ml of dry pyridine, and 4.44 g (20 mmoles) of phosphorus pentasulfide was heated at reflux and stirred under a nitrogen atmosphere for 4 hr. During this time, complete solution was attained. The solution was evaporated. The residue was triturated with 100 ml of 5% sodium bicarbonate solution for 20 hr, collected, and washed thoroughly with water and absolute ethanol to afford 1.71 g (89%) of **12** as a pink, crystalline powder, mp 218.5–221.5° dec, free of starting material and suitable for the next step. A portion was recrystallized three times from water to afford the analytical sample of **12**: mp 245.5–248° dec; $\lambda_{max}^{pH\ 1}$ 223 mμ (ε 9600) and 321 mμ (ε 24,400), $\lambda_{max}^{pH\ 13}$ 232 mμ (ε 15,500) and 310 mμ (ε 23,800); $[\alpha]_{589}^{25}$ −41° (*c* 0.5, pyridine); it moved as one spot in solvents C and D with R_{Ad} 1.52 and 1.87, respectively.

Anal. Calcd for $C_{15}H_{18}N_4O_6S$: C, 47.1; H, 4.74; N, 14.7; S, 8.39. Found: C, 46.8; H, 5.00; N, 14.7; S, 8.71.

9-(3-O-Methyl-β-D-ribofuranosyl)-6-mercaptopurine (13).—A suspension of 5.56 g (14.5 mmoles) of unrecrystallized **12**, 200 ml of methanol, and 22 ml of 1 *M* sodium methoxide in methanol was stirred and heated at reflux for 2 hr and then evaporated *in vacuo*. The residue was dissolved in 100 ml of water, treated with charcoal, and neutralized to afford 3.07 g (71%) of **13** as a tan powder, mp 196.5–198.5° dec, with a second crop, mp 197.5–199° dec (total 3.49 g, 80%). Two recrystallizations from water afforded the analytical sample of **13** as white, fibrous crystals: mp 200–201° dec; $\lambda_{max}^{pH\ 1}$ 224 mμ (ε 9200) and 322 mμ (ε 23,700), $\lambda_{max}^{pH\ 7}$ 226 mμ (ε 10,100) and 318 mμ (ε 23,200), $\lambda_{max}^{pH\ 13}$ 232 mμ (ε 14,100) and 311 mμ (ε 22,800); $[\alpha]_{589}^{23}$ −77° (*c* 1.0, 0.1 *N* sodium hydroxide); it moved as one spot in solvents B, C, and D with R_{Ad} 2.08, 1.32, and 1.75, respectively.

Anal. Calcd for $C_{11}H_{14}N_4O_4S$: C, 44.3; H, 4.73; N, 18.8; S, 10.8. Found: C, 44.2; H, 5.08; N, 18.7; S, 10.9.

2-Amino-6-mercapto-9-(3-O-methyl-β-D-ribofuranosyl)purine (15).—A suspension of 18.3 g (41 mmoles) of the mercury derivative[16] of 2-acetamido-6-chloropurine (in admixture with 8.5 g of Celite), in 1300 ml of xylene, was dried by distillation of 200 ml of solvent. To this was added 12.3 g (37.4 mmoles) of the chloro sugar **7** in 75 ml of xylene and 13 g of molecular sieves (Linde Type 4A), and the mixture was refluxed for 4 hr. The reaction was worked up[16] to afford the blocked nucleoside **14** as a yellow gum. This still contained some mercury salts. The gum was dissolved in 400 ml of methanol, treated with a stream of hydrogen sulfide for 30 min,[17b] and filtered. The solvent was evaporated; 100 ml of benzene was added and evaporated to afford 15.1 g (80%) of the blocked nucleoside **14** as a yellow gum that contained small amounts of purine and sugar material according to thin layer chromatography in solvent TB.

To a solution of 4.28 g (8.5 mmoles) of the blocked nucleoside **14** in 100 ml of methanolic hydrogen sulfide was added 26 ml of 1 *M* sodium hydrogen sulfide in methanol, and the solution was maintained at reflux temperature while hydrogen sulfide was bubbled through it for 2 hr. The hydrogen sulfide was replaced by nitrogen, which was bubbled through for 15 min, and finally 13 ml of 1 *M* methanolic sodium methoxide was added. After refluxing for 2 hr more, the solution was evaporated. The residue was dissolved in 40 ml of water, washed with three 15-ml portions of benzene, treated with charcoal, and neutralized with acetic acid to afford 1.07 g (40%) of **15**, mp 226–229° dec. Crystallization from 90 ml of water afforded 0.774 g (29%) of **15**, mp 232–235° dec. Two more crystallizations from water afforded the analytical sample of **15**, as an off-white powder: mp 232.5–235° dec; $\lambda_{max}^{pH\ 1}$ 207 mμ (ε 23,800), 264 (8000), and 344 (22,000); $\lambda_{max}^{pH\ 7}$ 209 mμ (ε 22,600), 225 sh (~13,600), 257 (8150), and 342 (24,300); $\lambda_{max}^{pH\ 13}$ 251 mμ (ε 13,600), 271 (7000), and 319 (20,400); $[\alpha]_{589}^{25}$ −70° (*c* 1, 0.1 *N* sodium hydroxide); homogeneous in solvents B, C, and E with R_{Ad} 2.19, 1.04, and 0.96, respectively.

Anal. Calcd for $C_{11}H_{15}N_5O_4S$: C, 42.2; H, 4.83; N, 22.4; S, 10.2. Found: C, 41.8; H, 4.80; N, 22.1; S, 10.6.

2-Amino-6-hydroxy-9-(3-O-methyl-β-D-ribofuranosyl)purine (16).—To a solution of 4.28 g (8.5 mmoles) of the blocked nucleoside **14** (see procedure for **15** above) in 100 ml of methanol was added a solution of 2.1 ml (30 mmoles) of 2-mercaptoethanol in 25 ml of 1 *M* methanolic sodium methoxide. The solution was refluxed under a nitrogen atmosphere for 3 hr and evaporated. The residue was dissolved in 40 ml of water, washed with three 15-ml portions of benzene, allowed to stand for 1.5 hr, and neutralized with acetic acid, and the resultant gel was kept at 5° overnight. The crystalline product was collected to afford 0.733 g (29%) of **16**, which decomposed at 258–300° without melting. Three recrystallizations afforded the analytical sample of **16** as white fibrous needles, which decomposed at 263–300° without melting: $\lambda_{max}^{pH\ 1}$ 256 mμ (ε 12,300) and 267 mμ sh (ε ~8500), $\lambda_{max}^{pH\ 7}$ 252 mμ (ε 13,200) and 270 mμ sh (ε ~9400), $\lambda_{max}^{pH\ 13}$ 258 mμ sh (ε ~11,300) and 264 mμ (ε 11,400); $[\alpha]_{589}^{25}$ −69° (*c* 1, 0.1 *N* sodium hydroxide); homogeneous in solvents C with R_{Ad} 1.04 (guanosine, R_{Ad} 0.86) and E with R_{Ad} 0.89 (guanosine, R_{Ad} 0.68).

Anal. Calcd for $C_{11}H_{15}N_5O_5$: C, 44.4; H, 5.09; N, 23.6. Found: C, 44.1; H, 5.10; N, 23.9.

Registry No.—**3**, 6698-46-0; **5**, 10300-20-6; **6**, 10300-21-7; **9**, 10300-22-8; **11**, 10300-23-9; **12**, 10300-24-0; **13**, 10300-25-1; **15**, 10300-26-2; **16**, 10300-27-3.

Acknowledgments.—We thank Dr. Peter Lim and his staff for the infrared and ultraviolet spectra and paper chromatography and Mr. O. P. Crews, Jr., and his staff for large-scale preparations of some intermediates.

A Novel Synthesis of 1,1-Dimethyl-3,3-diphenylindan[1]

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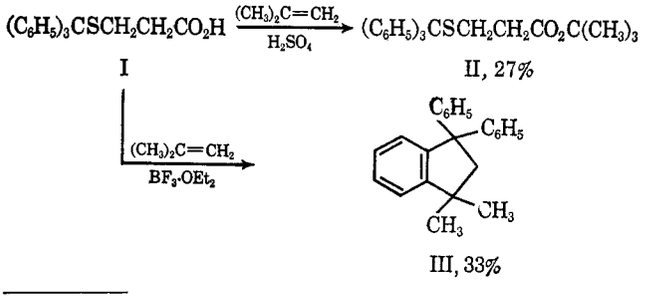
During recent investigations concerning the preparation of esters from acids containing acid-labile sulfur-protecting groups, 5,5,5-triphenyl-4-thiapentanoic acid (I) was converted to *t*-butyl 5,5,5-triphenyl-4-thiapentanoate (II) in 27% yield *via* the classical method involving isobutylene and sulfuric acid in chloroform. In an attempt to improve the yield, the reaction was repeated using an equivalent amount of boron trifluoride etherate and otherwise identical reaction conditions. The only isolable product from this reaction was a crystalline hydrocarbon obtained in 33% yield;

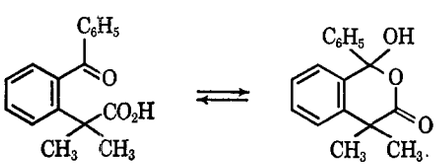


(1) Supported by Research Grant RG-7966 from the National Institute of General Medical Sciences of the National Institutes of Health, U. S. Public Health Service.

(2) National Science Foundation Cooperative Fellow, 1964–1966.

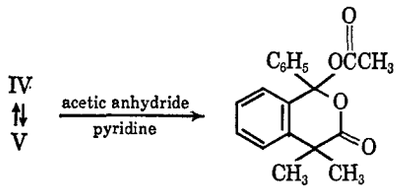
elemental analysis and molecular weight determination[3] indicated the molecular formula to be $C_{23}H_{22}$. The substance could also be prepared by allowing triphenylcarbinol, isobutylene, and boron trifluoride etherate to react under similar conditions. The hydrocarbon has been identified as 1,1-dimethyl-3,3-diphenylindan (III) on the basis of the following data. The infrared spectrum is indicative of an alkylaryl hydrocarbon and exhibits a prominent doublet at 1370 cm^{-1} as well as a weak band at 1310 cm^{-1} which has been identified with the indan structure.[4] The ultraviolet spectrum contained a cluster of aromatic peaks centered at 265 mμ, characteristic of indans.[5] The nuclear magnetic resonance (nmr) spectrum of the substance consisted of an aromatic multiplet (τ 2.80, 14.8 H) and two singlets (τ 7.12, 2.0 H; τ 8.83, 6.1 H). The mass spectrum corroborated the molecular weight ($C_{23}H_{22}$, 298.17) and indicated that the parent ion readily lost methyl and phenyl fragments. In addition, the mass spectrum contained a peak (C_9H_7, m/e 115) which has been associated with indans.[6,7] The absence of benzylic hydrogens in the molecule was suggested by the low intensity of the M − 1 peak.[8]

The hydrocarbon did not decolorize bromine in carbon tetrachloride and failed to react with N-bromosuccinimide (NBS) or boiling aqueous potassium permanganate. However, when III was subjected to chromium trioxide in glacial acetic acid, 2-methyl-2-(*o*-benzoyl)phenylpropanoic acid (IV) was isolated in 10.4% yield. This product appears to be identical with the material reported by Koelsch and Johnson,[9] but the spectrum contained two methyl signals and a hydroxyl signal. Integration of these signals gave values which indicate the presence of an equilibrium between the keto acid IV and the lactol V.



IV V

This equilibrium has been verified by converting V into 1-acetoxy-1-phenyl-3-oxo-4,4-dimethyl-3,4-dihydro-1H-2-benzopyran (VI) in 87% yield.



VI, 87%

Although 1-methyl-1,3,3-triphenylindan[10] (VII) could be prepared by substituting α-methylstyrene into the general procedure, attempts to substitute either benzhydrol or 1,1-diphenylethanol for triphenylcarbinol were unsuccessful.

Experimental Section[11]

5,5,5-Triphenyl-4-thiapentanoic acid (I) was prepared in 93% yield by the procedure of Gregg, *et al.*,[12] mp 211–212°, lit.[13] mp 211–212°.

***t*-Butyl 5,5,5-triphenyl-4-thiapentanoate** (II) was prepared from 3.36 g (0.01 mole) of I in 200 ml of chloroform. After the solution was saturated with isobutylene, 0.60 ml (0.011 mole) of concentrated sulfuric acid was added and the reaction mixture was stirred for 24 hr. The yellow solution was washed with 100 ml of 5% sodium bicarbonate solution and 200-ml portions of water and saturated sodium chloride solution. After drying, the solvent was removed *in vacuo* and the resulting oil was applied to a 1.5 × 50 cm column of silica gel (40 g, 0.05–02 mm). The desired material was removed with chloroform–carbon tetrachloride (1:9) and the solvent was removed *in vacuo* to yield a viscous oil which was crystallized from *n*-hexane. The product appeared as 1.05 g (27%) of colorless needles, mp 80.5–81.5°, homogeneous by tlc (systems A and B).

Anal. Calcd for $C_{26}H_{28}O_2S$: C, 77.19; H, 6.98; S, 7.93. Found: C, 77.16; H, 7.03; S, 7.98.

1,1-Dimethyl-3,3-diphenylindan (III) was prepared by the general procedure previously described for II, using 3.36 g (0.01 mole) of I and 1.4 ml (0.011 mole) of boron trifluoride etherate. After washing and drying, the solvent was removed *in vacuo* to yield a mixture of crystals and oil. The oil was decanted from the crystals and these were recrystallized from *n*-hexane. The product appeared as 0.99 g (33.2%) of colorless crystals: mp 109–110°; homogeneous by tlc (system B); ν_{max}^{KBr} 3055, 2960, 2860, 1594, 1490, 1440, 1380, 1360, 1310, 750, 696 cm^{-1}; $\lambda_{max}^{CHCl_3}$ 235 (ϵ 613), 260 (1190), 266 (1420), 273 mμ (1235); molecular weight (vapor pressure osmometry; benzene, 37°), 304; τ 2.80 (14.8 H, m), 7.12 (2.0 H, s), 8.83 (6.1 H, s); m/e 298 (100%), 283 (73%), 221 (99%), 205 (47%), 143 (38%), 91 (61%), 28 (53%).

Anal. Calcd for $C_{23}H_{22}$: C, 92.58; H, 7.43. Found: C, 92.24; H, 7.42.

Material which is identical in all respects with that from the above procedure was prepared by substituting an equivalent amount of triphenylcarbinol for I. Table I lists the various modifications which produced yields of 42–68%.

1-Methyl-1,3,3-triphenylindan (VII) was prepared in 77% yield by the method of Schoepfle and Ryan,[10] mp 140–141°, lit.[10] mp 143°.

This compound was also prepared by the general procedure used for III, except that α-methylstyrene was the olefin. The product appeared as 1.5 g (21%) of colorless crystals, mp 141–142.5°. A mixture melting point was not depressed and tlc behavior (system B) is identical with authentic 1-methyl-1,3,3-triphenylindan.

Attempted Bromination of III with NBS.—A carbon tetrachloride solution of III was refluxed for 1 hr with NBS and a trace of α,α'-azobisisobutyronitrile; no succinimide was produced and 95% of the starting material was recovered.

(3) Determined in benzene at 37° using Mechrolab vapor pressure osmometer, Model 310A.

(4) T. F. Wood and J. Angiolini, *Tetrahedron Letters*, No. 1, 1 (1963).

(5) "Ultraviolet Spectral Data," American Petroleum Institute Research Project 44, Carnegie Institute of Technology, Pittsburgh, Pa.

(6) F. W. McLafferty, "Mass Spectral Correlations," American Chemical Society, Washington, D. C., 1963, p 49.

(7) "Catalog of Mass Spectral Data," American Petroleum Institute Research Project 44, Carnegie Institute of Technology, Pittsburgh, Pa.

(8) H. Budzikiewicz, C. Djerassi, and D. H. Williams, "Interpretation of Mass Spectra of Organic Compounds," Holden-Day, Inc., San Francisco, Calif., 1964, p 170.

(9) C. F. Koelsch and P. R. Johnson, *J. Am. Chem. Soc.*, **65**, 565 (1943).

(10) C. S. Schoepfle and J. D. Ryan, *ibid.*, **52**, 4021 (1930).

(11) Melting points are uncorrected and were obtained in capillary tubes. Elemental analyses were performed by Micro-Tech Laboratories, Skokie, Ill. Thin layer chromatograms were conducted on microscope slides and 5 × 20 cm Pyrex plates using uniform coatings of silica gel GF_{254}. The chromatograms were developed with iodine vapor and/or viewed under ultraviolet light. Solvent systems for thin layer chromatography (tlc) were benzene–dioxane–acetic acid (90:25:4, system A) and chloroform–benzene (1:1, system B). Commercial reagents were of the highest quality available. Infrared spectra were recorded on a Perkin-Elmer 421 grating spectrophotometer. Ultraviolet spectra were recorded on a Cary recording spectrophotometer, Model 14. Nmr spectra were obtained on a Varian Associates A-60 spectrometer using tetramethylsilane as the internal standard. Mass spectra were recorded on a Hitachi Perkin-Elmer RMU-6E mass spectrometer and intensities are relative to the highest peak. Unless noted, all spectral data were consistent with the proposed structures.

(12) D. C. Gregg, H. A. Iddles, and P. W. Stearns, *J. Org. Chem.*, **16**, 246 (1951).

(13) J. E. Reece, Ph.D. Thesis, University of North Carolina, Chapel Hill, N. C., 1965.

TABLE I

PREPARATION OF $C_{23}H_{22}$ WITH $BF_3 \cdot (C_2H_5)_2O$[a]

$(C_6H_5)_3COH$ concn, M	Reaction time, hr	Yield,[b] %
0.04	8	51
0.05	4	43
0.05	36	68
0.08	15	68

[a] Both $SnCl_4$ and H_2SO_4 proved to be inferior under analogous conditions. [b] Based on carbinol.

Attempted Oxidation of III with Aqueous Potassium Permanganate.—A modification of the method used by Bogert and Davidson[14] to oxidize 1,1-dimethylindan failed to affect III and 91% of the starting material was recovered.

Oxidation of III with Chromium Trioxide in Acetic Acid.—A slurry of 14.9 g (0.05 mole) of III in 300 ml of glacial acetic acid was stirred rapidly at room temperature while 75 g (0.75 mole) of chromium trioxide was added in three equal portions. The additions required 25 min and an ice bath was used to maintain the temperature below 30°. After stirring at room temperature for 12 hr, the reaction mixture was added to 500 ml of ice water and partially neutralized (pH 5) by the addition of 150 g (3.75 moles) of sodium hydroxide in small portions. The mixture was stirred for 1 hr, filtered, and the amorphous residue was washed with water and air dried. The filtrate and washings were combined and extracted with ethyl ether. The ether solution was extracted with saturated sodium carbonate solution and the aqueous layer was separated and acidified with concentrated hydrochloric acid. The white precipitate was collected, washed with water, and recrystallized from ethanol–water. The product appeared as 1.3 g (10.4%, based on III consumed) of IV, mp 196–198°, lit.[9] mp 198°. An additional recrystallization produced material with mp 196–197°; homogeneous by tlc (system A); ν_{max}^{KBr} 3055, 3000–2500, 1690, 1670, 1595, 1475, 1450, 1380, 1360, 1285, 1255, 1155, 940, 920, 755, 700 cm^{-1}; $\lambda_{max}^{CHCl_3}$ 239 mμ (ε 6.93 × 10³), 252 mμ (ε 9.72 × 10³); τ 2.50 (two signals, 9.48 H), 7.05 (0.58 H, s), 8.70 (two signals, 6.00 H); *m/e* 268 (22%), 224 (61%), 223 (100%), 222 (57%) 209 (90%), 208 (78%), 207 (65%), 206 (73%), 194 (61%), 180 (98%), 165 (59%), 105 (82%), 77 (77%), 28 (61%).

Anal. Calcd for $C_{17}H_{16}O_3$: C, 76.10; H, 6.01. Found: C, 75.78; H, 6.17.

The filtration residue from the original reaction mixture was transferred to a Soxhlet thimble and extracted with *n*-hexane for 2 hr. The *n*-hexane solution was concentrated *in vacuo* to recover 0.96 g (6.4%) of III, mp 109–110°. A mixture melting point was not depressed.

1-Acetoxy-1-phenyl-3-oxo-4,4-dimethyl-3,4-dihydro-1H-2-benzopyran (VI) was prepared from 134 mg (0.5 mole) of IV dissolved in 0.5 ml (5.5 mmoles) of acetic anhydride, and 1.26 ml (15.5 mmoles) of pyridine. The reaction mixture was stirred at room temperature for 1 hr, heated at reflux for 5 min, and allowed to cool while stirring for an additional hour. The solution was poured into 20 ml of ice water and the gum which precipitated was separated by decantation. Acidification of the supernatant provided no starting material. The gum was triturated with 30 ml of ice water to produce an off-white solid which was collected, washed with water, and dried *in vacuo* overnight. The dry product, 131 mg (87.4%), mp 130–134°, was homogeneous by tlc (system B). One recrystallization from *n*-hexane raised the melting point to 135–136°; ν_{max}^{KBr} 3065, 2985, 2950, 1760, 1601, 1490, 1445, 1380, 1365, 1235, 1210, 1130, 1060, 956, 800, 760, 695 cm^{-1}; $\lambda_{max}^{CHCl_3}$ 252 (ε 465), 257.5 (590), 262 (558), 264 (574), 267 (419), 272 mμ (326); τ 2.53 (9.3 H, m), 7.89 (3.0 H, s), 8.14 (3.0 H, s), 8.36 (3.2 H, s); *m/e* 310 (20%), 267 (47%), 251 (91%), 224 (64%), 223 (100%), 209 (43%), 208 (62%), 105 (35%), 77 (52%), 43 (56%).

Anal. Calcd for $C_{19}H_{18}O_4$: C, 73.53; H, 5.85. Found: C, 73.63; H, 5.72.

Attempted Preparation of Analogous Indans Using Carbinols Other Than Triphenylcarbinol. A. Benzhydrol.—The general procedure with 1.84 g (0.01 mole) of benzhydrol provided a viscous oil after the usual work-up. This material was shown to be heterogeneous by vapor phase chromatography (silicon rubber column, 2 ft): ν_{max}^{KB} 3060, 3020, 2960 (very strong), 1635, 1598, 1385, 1360, 1225, 890, 695 cm^{-1}; τ, very complex, including a triplet at τ 8.96 with an area equal to 2.5 times the aromatic multiplet at τ 2.60. No solid could be obtained.

B. 1,1-Diphenylethanol.—The procedure described above, using 1.98 g (0.01 mole) of 1,1-diphenylethanol, produced an oil which exhibited almost identical retention times and spectral properties when compared with the product from the benzhydrol experiment.

Registry No.—II, 10271-31-5; III, 10271-32-6; IV, 10271-33-7; VI, 10271-34-8.

Acknowledgment.—We are indebted to Professor M. M. Bursey for his kind assistance in interpreting the mass spectra.

(14) M. T. Bogert and D. Davidson, *J. Am. Chem. Soc.*, **56**, 185 (1934).

Unusual Dimeric Salts of 2-(3-Pyridyl)-1,3-indandione

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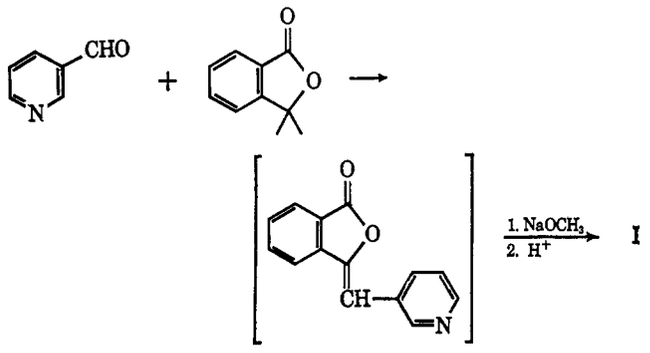
As part of a general investigation of 1,3-indandiones, the preparation 2-(3-pyridyl)-1,3-indandione (I) was undertaken. Lespagnol,[1] *et al.*, have published a preparation for I but did not analyze their product. In place of the method employed by Lespagnol and coworkers[1] to prepare I, the simpler, more general technique of Shapiro[2] for preparing 1,3-indandiones was used to obtain high yields of I from 3-pyridylaldehyde and phthalide.



However, on final acidification of the basic reaction, an excess rather than 1 equiv[1] of hydrochloric acid was used producing an unusual salt (Ia) of I. Elemental analysis and titration of Ia with sodium hydroxide clearly indicated that only one chloride ion was present for every two pyridylindandione moieties.

It was further found that acidification of a basic aqueous solution of I with either hydrobromic, hydriodic, nitric, or picric acid gave similar dimeric salts of I (Ib–e, respectively). In each case titration and analytical data indicated 2:1 adducts (Table I). Acidification with either excess acetic, trifluoroacetic, or sulfuric acid, however, precipitated in each case only the highly insoluble, zwitterionic, internal salt I as indicated by an infrared spectrum, titration, nuclear

(1) A. Lespagnol, C. Lespagnol, and N. Traisnel, *Bull. Soc. Pharm. Lille*, 89 (1961); *Chem. Abstr.*, **58**, 8332 (1963).

(2) S. Shapiro, K. Geiger, J. Youlus, and L. Freedman, *J. Org. Chem.*, **26**, 3580 (1961).