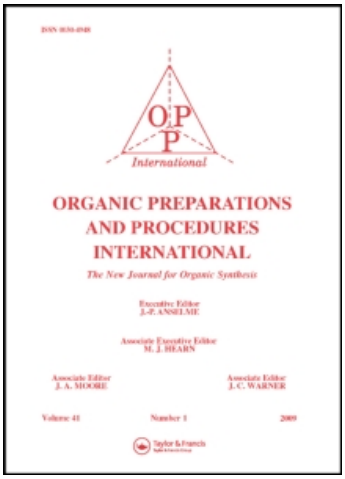

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SIMULTANEOUS PREPARATION OF BROMOACETIC ACID AND ACETYL CHLORIDE

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Silylation of 2-(1H)-pyridone. General Procedure.- Pyridones I (0.015 mol), imidazole (0.06 g., 0.09 mol) and hexamethyldisilazane (15 g., 0.09 ml) were stirred at 70° for 1.5 hr. After removal of excess hexamethyldisilazane at 150 mmHg, the remaining crude products were fractionated in vacuo (see Table 1) to give 2-trimethylsilyloxy pyridines II as colorless liquids. All these compounds are extremely sensitive to moisture.

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SIMULTANEOUS PREPARATION OF BROMOACETIC ACID AND ACETYL CHLORIDE

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(12/26/78)

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The successful use of bromine chloride as a bromination agent has been reported for bromination of arene rings[1-4] and preparation of bromosilane derivatives.[4] Bromination of other organic compounds has not been investigated, owing to supposed side-reactions, especially chlorination since bromine chloride in both liquid and gaseous states always occurs in equilibrium with chlorine and bromine.[5] We now report that bromine chloride reacts easily with acetic anhydride. The reaction proceeds very rapid-

ly above 90° (10-30 min.). If acetyl chloride is removed as it forms, pure bromoacetic acid, free of chloroacetic acid is easily obtained generally in yield greater than 90%.[6]

$$(CH_3CO)_2O + BrCl \longrightarrow BrCH_2COOH + CH_3COCl$$

The method developed for their simultaneous preparation is a good example of rational utilization of raw materials.

EXPERIMENTAL

Bromoacetic acid and Acetyl chloride.- Bromine chloride (116 g., 1 mole) was dropped over 25 min. into boiling acetic anhydride (102 g., 1 mole).[7,8] The acetyl chloride generated, bp. 52-58°, was simultaneously distilled through a Vigreux column.[9] The yield was 62 g. (82%). The reaction mixture was cooled and 10 ml of water was carefully added to hydrolyze mixed acetic bromoacetic anhydride formed in a side-reaction. Vacuum distillation of the resulting mixture gave 132 g. (95%) of bromoacetic acid, bp. 115-118°/15 mm, mp. 46-48°.

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6. G.L.C. analysis after treatment with ether-CH_2N_2. Column length 100 cm, inner diam. 3mm, column packing 5% DC-200 on Varaport, 80-100 mesh, carrier gas Ar 50 ml/min., FID, oven temp. 50°, Willy Giede (DDR) apparatus.

7. Bromine chloride was prepared through dissolution of an equimolar amount of gaseous chlorine in liquid bromine.

8. A dropper with bromine chloride was cooled with Dry Ice to keep the temperature between -20° and -40° (bp. of bromine chloride is +5°). The dropper outlet was immersed in the liquid.

9. The reaction is strongly exothermic. The heat evolved is sufficient to distil the acetyl chloride formed.

1,7-DIMETHYLINDAN

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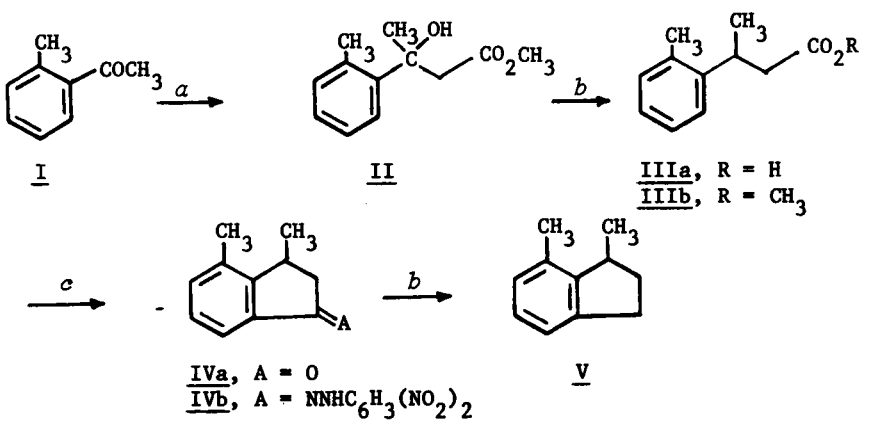
A four step procedure [shown for 1,7-dimethylindan (V, 44% yield)] provides a selective and convenient synthesis of 1,7-substituted indans.



a)$BrCH_2CO_2CH_3$, Zn(Cu), Ph, Δ. *b*)Pd/C, H_2, HOAc, Δ. *c*)PPA, Δ on IIIa.