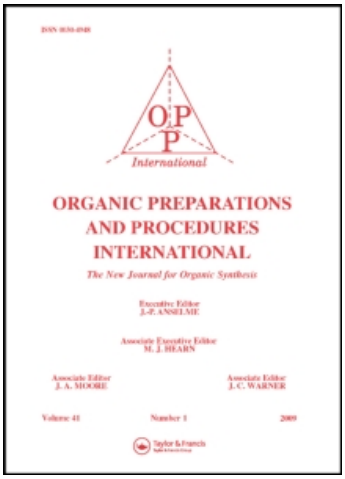

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3-(2'-FORMYL-3'-METHOXYPHENYL)PROPIONIC ACID

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3-(2'-FORMYL-3'-METHOXYPHENYL)PROPIONIC ACID

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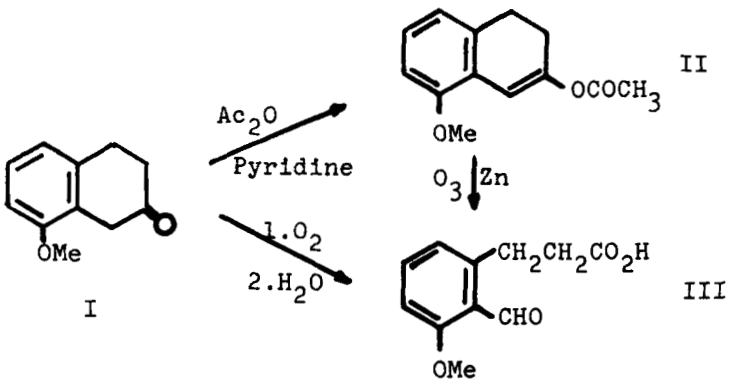
3-(2'-Formyl-3'-methoxyphenyl)propionic acid (III) has been obtained by reduction of the ozonide of the enol acetate (II) of 8-methoxy-2-tetralone (I)[1] or more conveniently by the aerial oxidation of I.



EXPERIMENTAL

2-Acetoxy-8-methoxy-3,4-dihydronaphthalene (II). - A solution of 26 g of 8-methoxy-2-tetralone (I) in 500 ml acetic anhydride and 18 ml of pyridine was refluxed under nitrogen for 72 hrs. After removal of the excess reagents in vacuo, the crude product was distilled to give 23.3 g (73%) of the enol acetate II, as a pale yellow liquid, bp. 108-110/0.02 mm, ν_{max} (CH_2Cl_2) 1750, 1660, 1580 cm^{-1}; nmr ($CDCl_3$) τ 3.15 (3H,

m, aromatic H), 3.50 (1H, s, -CH=C-), 6.30 (3H, s, CH_3O), 7.10 (2H, t, J=7 Hz, $-CH_2-$), 7.60 (2H, t, J=7 Hz, $-CH_2-$), 7.85 (3H, s, CH_3COO-).

Anal. Calcd. for $C_{13}H_{14}O_3$: C, 71.54; H, 6.47.
Found: C, 71.32; H, 6.20.

Ozonolysis of Enol acetate II. - Ozone was bubbled through an ice-cooled solution of 13 g enol acetate II in 100 ml of hexane for 6 hrs. The reaction mixture was flushed with nitrogen and the solvent was evaporated under reduced pressure. The ozonide was treated with 3 g zinc dust in methanol at room temperature for 30 min., then filtered. After evaporation, the product was dissolved in a mixture of 30 ml of chloroform and 20 ml of water and treated with hydrogen sulfide. The organic layer was separated and the aqueous phase was extracted once with 30 ml. of chloroform. The dried (Na_2SO_4) extracts were evaporated and the residue was crystallized from chloroform-methanol to give 4.2 g (34%) of III, mp. 142.5-144°; ν_{max} (CH_2Cl_2) 1705, 1680, 1590, 1580 cm^{-1}; nmr τ -0.63 (1H, s, -CHO), 2.95 (3H, m, aromatic H), 6.20 (3H, s, CH_3O), 6.5-7.5 (4H, m, $-CH_2CH_2-$).

Anal. Calcd. for $C_{11}H_{12}O_4$: C, 63.45; H, 5.81.
Found: C, 63.39; H, 5.82.

Autoxidation of 8-Methoxy-2-tetralone. - Compressed air was passed through a solution of 420 mg of I in 30 ml of benzene maintained at 50° for 4 days under a condenser. The mixture was extracted with aq. $NaHCO_3$ which upon acidification yielded 117 mg. of III, mp. 141-143° ($CHCl_3-CH_3OH$), identified

by comparison with the substance obtained above. From the benzene solution 312 mg of the tetralone I was recovered. The yield of III amounts to 90% based on consumed material.

REFERENCE

1. P. A. Robins and J. Walker, J. Chem. Soc., 409 (1958).

ZINC ETHYL ACETOACETATE

Submitted (6/23/72) by O. Grummitt*, J. Perz and J. Mehaffey
The Sherwin-Williams Company
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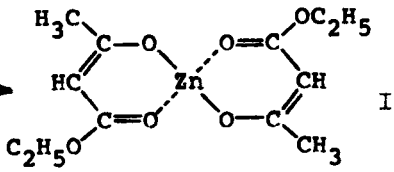
The preparation of zinc ethyl acetoacetate (I)[1] has been successfully repeated by the authors and the zinc content of I

$$ZnCl_2 + 2\ NaOCH_3 \longrightarrow Zn(OCH_3)_2 + NaCl$$

$$Zn(OCH_3)_2 + 2\ CH_3COCH_2CO_2Et \longrightarrow$$



I

was determined by EDTA analysis (Calcd.: Zn, 20.1. Found: 20.2)[2] after acid hydrolysis. Purification of I by crystallization from hot methanol results in extensive decomposition. The pure product is obtained by dissolving I at room temperature in a 95:5 mixture of methanol-ethyl acetoacetate (2.7 g/280 ml) and cooling to 0°;[3] in this fashion, pure I (64% recovery), mp. 147-150°, is obtained.

REFERENCES

1. A. C. Backus and L. L. Wood, U. S. Pat. 3,453,300; Fr.