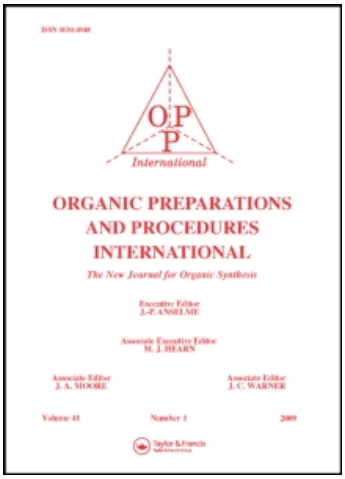


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# SIMPLE SYNTHESIS OF 3-*t*-BUTYLGLUTARIC ACID

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IId, mp. 165-167°. Anal. Calcd for $C_{10}H_{10}O_4$: C, 61.85; H, 5.19. Found: C, 61.58; H, 5.42.

IIe, mp. 182-184°. Anal. Calcd for $C_{27}H_{46}O_4$: C, 74.61; H, 10.67. Found: C, 74.56; H, 10.67.

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1. This is presumably due to the stability of bicyclic anhydrides to hydrolysis in acetic acid at room temperature.
2. F. L. Weinsenborn and H. E. Applegate, J. Am. Chem. Soc., 81, 1960 (1959).



# SIMPLE SYNTHESIS OF 3-t-BUTYLGLUTARIC ACID

Submitted by M. Tichy
7/23/76

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A recent synthesis of 3-t-butylglutaric acid (IIIa)[1] prompts us to report another route of IIIa which was obtained in 45% overall yield from the commercially available 4-t-butylcyclohexanone (I) in 3 steps. I was converted into its 2,6-dibenzylidene derivative II which was ozonized to give a mixture of benzoic acid and the desired acid IIIa, separated by fractionation of their esters.

## EXPERIMENTAL

2,6-Dibenzylidene-4-t-butylcyclohexanone (II).- A solution of sodium hydroxide (4 g) in water (10 ml) was added to a solu-

tion of 4-t-butylcyclohexanone (185 g) and benzaldehyde (294 g) in ethanol (800 ml). The mixture warmed spontaneously and was allowed to stand overnight. The precipitated product was filtered and washed thoroughly with ethanol, yielding 310 g (78%) of the product II as yellow needles, mp. 142-144°. An analytical sample was obtained by crystallization from ethanol, mp. 143.5-144.5°.

Anal. Calcd for $C_{24}H_{26}O$: C, 87.23; H, 7.93.
Found: C, 87.29; H, 7.96.

Diethyl 3-t-Butylglutarate (IIIb).- The dibenzylidene derivative II (310 g) was ozonized in batches as follows. A rapid stream of oxygen, containing about 5% of ozone, was passed through a suspension of finely divided II (30 g) in acetic acid (350 ml), the temperature being held at 10-20° (external cooling with cold water). The mixture became homogeneous and became colorless. After 2-4 hrs., the absorption of ozone ceased. Each batch was then mixed with water (40 ml) and 30% hydrogen peroxide (45 ml) and refluxed for 3 hrs. The reaction mixtures from all the batches were combined and evaporated to dryness *in vacuo*. The residue was azeotropically esterified with anhydrous ethanol (500 ml), benzene (500 ml) and conc. $H_2SO_4$ (10 ml). The usual work-up procedure afforded a mixture of esters which was distilled *in vacuo* and then fractionated on a column, yielding 150 g of ethyl benzoate, bp. 49°/0.13 mm and 154 g (67%) of diethyl 3-t-butylglutarate (IIIb), bp. 80°/0.13 mm., lit.[1] bp. 109-110°/2.5 mm.

3-t-Butylglutaric Acid (IIIa).- The diester IIIb (154 g) was mixed with a solution of potassium hydroxide (150 g) in water

(500 ml) and ethanol (100 ml). Gentle heating of the mixture brought about an exothermic reaction and the resulting homogeneous solution was heated to 85° for 30 min, cooled, extracted with ether, the aqueous layer was acidified and the product was taken up in ether. The ethereal layer was dried, evaporated and the acid was crystallized from benzene to yield 103 g (87%), mp. 148-149°, lit.[1] mp. 146-147.5°.

## REFERENCE

1. W. R. Purdum and K. D. Berlin, Org. Prep. Proced. Int., 7, 283 (1975).



# PARTIAL TRANSALKYLATION OF 3,3', 5,5'-TETRA-t-BUTYL-4,4'-DIHYDROXYBIPHENYL

Submitted by 6/18/76

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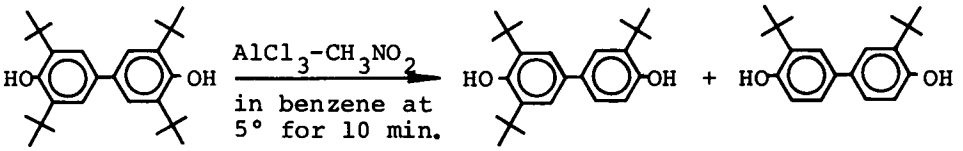
The partial transalkylation of the title compound (I) affords 3,3'-5-tri-t-butyl- (II) and 3,3'-di-t-butyl-4,4'-dihydroxybiphenyl (III) in 76% and 17% yields respectively.[1]




I II III