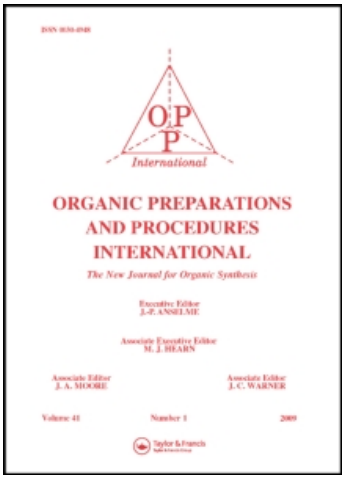

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4-(α,β-DIBROMOETHYL)PHENYLPHOSPHONIC ACID

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JAMES A. MOORE

4-(α,β-DIBROMOETHYL)PHENYLPHOSPHONIC ACID

Submitted by M. A. Rea,[1a] T. L. Slapar[1b] and W. A. Trinler*
(8/23/73)
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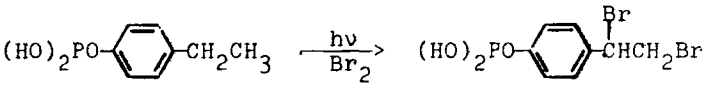
The title compound (II) has been prepared by the free radical bromination of 4-ethylphenylphosphonic acid (I).[2]



I II

EXPERIMENTAL

4-(α,β-Dibromoethyl)phenylphosphonic (II). - A solution of 16 g. (5.5 ml., 0.10 mole) of bromine in 20 ml. of acetic acid was added dropwise over a period of 2 hrs. to a solution of 9.3 g. (0.050 mole) of I (prepared in 38% yield from ethylbenzene and PCl_3, mp. 172-173°)[3] in 50 ml. of acetic acid in a 250 ml. three-neck flask fitted with a reflux condenser and a dropping funnel. The reaction flask was irradiated with a 275 watt G. E. sunlamp placed about 1-2 inches below the flask. The reaction solution was then transferred to a beaker and reduced to one-half its original volume by evaporation to yield 14 g. (82%) of crude II, mp. 179-190°. Recrystallization from dilute hydrochloric acid yielded needles, mp.

189-191°. NMR [$(CD_3)_2SO$] δ 7.58-7.95 (m, 4, aromatic), δ 5.57 (pair of d, 1, CHBr) δ 4.17-4.57 (m, 2, CH_2Br) δ variable with concentration and amount of water contamination (s, 2, OH).

Anal. Calcd. for $C_8H_9Br_2O_3P$: C, 27.94; H, 2.64; P, 9.01.
Found: C, 27.69; H, 2.65; P, 8.68.

REFERENCES

*To whom inquiries should be sent.

1. (a) Undergraduate Student Research Participant;
   (b) Masters' Candidate (Teaching).
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3. G. M. Kosolapoff, "Inorganic Reactions," Vol. VI, Chap. 6, R. Adams, Ed., J. Wiley and Sons Inc., New York, N. Y., p. 273.

3,4-DIALKYLQUINOLINES BY AN EXTENSION OF THE BEYER SYNTHESIS

Submitted by (8/30/73)

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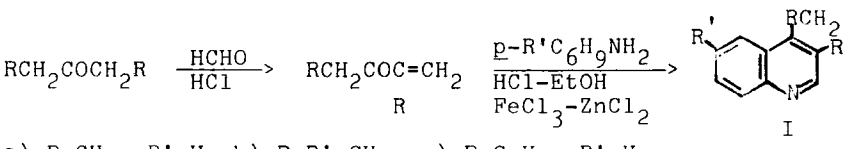
An extension of the Beyer synthesis[1,2] has been developed into a simple one-step procedure, useful for the preparation of 3,4-dialkylquinolines (I), starting from any simple ketone. Prior to this work, only methyl ketones had been used.[1,3-5]



I

a) $R=CH_3$, $R'=H$; b) $R=R'=CH_3$; c) $R=C_2H_5$, $R'=H$