TABLE I

BIS-DIALKYLAMINOALKYL 5-METHOXYISOPHTHALATE DIHYDROCHLORIDES

Bis-dialkylaminoalkyl 5-methoxy-isophthalate dihydrochloride	Yield, %	M. p., °C.	Formula	Nitrogen, % Calcd.	Nitrogen, % Found
β-(Dimethylamino)-ethyl	46.0	224–225	$C_{17}H_{28}O_5N_2Cl_2$	6.81	6.71
β-(Diethylamino)-ethyl	59.5	227	$C_{21}H_{36}O_5N_2Cl_2$	5.99	6.07
γ-(Diethylamino)-propyl	72.5	178–179	$C_{23}H_{42}O_5N_2Cl_2$	5.65	5.64
β-(Di-n-propylamino)-ethyl	50.0	151–152	$C_{25}H_{44}O_5N_2Cl_2$	5.35	5.28
γ-(Di-*n*-propylamino)-propyl	65.8	139–140	$C_{27}H_{48}O_5N_2Cl_2$	5.07	4.94
β-(Di-isopropylamino)-ethyl	52.0	188–189	$C_{25}H_{44}O_5N_2Cl_2$	5.35	5.23
γ-(Di-isopropylamino)-propyl	63.8	119	$C_{27}H_{48}O_5N_2Cl_2$	5.07	5.00
β-(Di-*n*-butylamino)-ethyl	69.1	137.5–138.5	$C_{28}H_{52}O_5N_2Cl_2$	4.83	4.74
γ-(Di-*n*-butylamino)-propyl	63.5	127–128	$C_{31}H_{56}O_5N_2Cl_2$	4.59	4.50
β-(Di-*n*-amylamino)-ethyl-[a]	65.2	....	$C_{33}H_{58}O_5$	4.96	4.78
γ-(Di-*n*-amylamino)-propyl	35.8	70–72	$C_{35}H_{64}O_5N_2Cl_2$	4.22	4.22
β-(Di-*n*-hexylamino)-ethyl[b]	62.1	64–65	$C_{37}H_{66}O_5N_2Br_2$	3.59	3.63
γ-(Di-*n*-hexylamino)-propyl	77.5	75–76	$C_{39}H_{74}O_5N_2Cl_2$	3.89	3.96

[a] Free base b. p. 205–210° (0.05 mm.). [b] Dihydrobromide.

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RECEIVED[6] AUGUST 17, 1949

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[6] Original manuscript received July 16, 1947.

The Formation of Biphenyl in the Decomposition of Benzoyl Peroxide in Nitrobenzene

BY DELOS F. DETAR

In the decomposition of diacyl peroxides $(ArCOO)_2$ in aromatic solvents it has been observed[1] that the principal product is the unsymmetrical biaryl ArAr[1] formed by attack on the solvent. In the decomposition of benzoyl peroxide in nitrobenzene Wieland, Schapiro and Metzger[2] were unable to detect any of the symmetrical product, biphenyl. With the idea that more biphenyl might be formed in more concentrated solutions, the experiment[2] was repeated with 20–40% solutions. Biphenyl was definitely formed and probably in at least 3–4% yields since the isolation method is known to involve losses. The biphenyl may have been formed by either a coupling of two phenyl radicals, or by an induced decomposition of the peroxide by a phenyl radical.[3,4] No more than a trace of the symmetrical 4,4′-dinitrobiphenyl could have been present since none could be found. It is much less soluble than the 4-nitrobiphenyl and is detectable in mixtures with it.

Experimental

Decomposition of Benzoyl Peroxide in Nitrobenzene.—Benzoyl peroxide (5.00 g.) was added to 15 g. of nitrobenzene and the mixture warmed at 110° for one-half hour (homogeneous solution). The reaction mixture was steam distilled, the distillate extracted with alkali and then the nitrobenzene layer was reduced with tin and hydrochloric acid.[5] Steam distillation of the reduction mixture followed by extraction with peroxide-free ether gave a neutral residue. This was refluxed with 10% sodium hydroxide solution to assure removal of phenyl benzoate. The residue amounted to 44 mg. (1.4%). It was identified as biphenyl by microscopic comparison with authentic biphenyl by identity of the 70° angle of the rhombic-shaped plates obtained from ethanol and the extinction angles which bisected the vertices. Other runs at 90° and one decomposition of a 40% solution of benzoyl peroxide gave similar results.

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RECEIVED AUGUST 26, 1949

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[1] *Cf.* Waters, "The Chemistry of Free Radicals," Oxford University Press, London, 1946, p. 165.

[2] Wieland, Schapiro and Metzger, *Ann.*, **513**, 105 (1934).

[3] Nozaki and Bartlett, THIS JOURNAL, **68**, 1686 (1946).

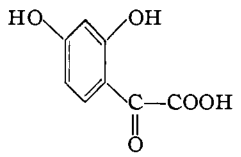
[4] DeTar and Sagmanli, *ibid.*, **72**, 965 (1950).

[5] This reduction method was used by Wieland, Schapiro and Metzger.[2]

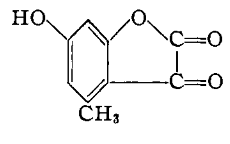
The Ultraviolet Absorption Spectra of Several Substituted Phenylglyoxylic Acids

BY DONALD J. CRAM

In connection with a study on the structure of citrinin, the compounds I,[1] II,[1] III[1] and IV[1] were synthesized as models for an ultraviolet absorption spectra comparison with citrinin. Although the recent resynthesis of this natural product[2] made such a study unfeasible, an examination of the spectra of these molecules reveals some interesting structural relationships.



I



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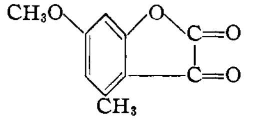
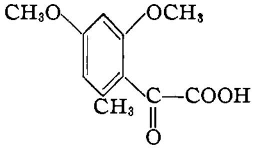


III



IV

Hunsberger, *et al.*,[1] reported that methylation of II with dry diazomethane produced a monomethylated compound that possessed the hemiquinone

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[1] Hunsberger and Amstutz, THIS JOURNAL, **70**, 671 (1948).

[2] Robertson, *et al.*, *Nature*, **163**, 94 (1949).