Under the same conditions but in the absence of sodium methylate the yields were considerably lower.

The reaction appears to be applicable to simple aliphatic esters as would be expected. With ethyl diethylmalonate the same conditions gave only a small yield of the diamide (3.5%). This parallels the experience of Fischer and Dilthey[8] and of Meyer,[10] the latter was not able to detect any noticeable reaction between the substances while the former workers obtained only a 1.1% yield after standing two months. The purely steric situation with this ester should be no more favorable to reaction than with esters of pivalic acid which have been found[6] to react with about one-sixtieth of the speed shown by esters of isobutyric acid.

Experimental

The conversions of the esters to the corresponding diamides are summarized in Table I. To illustrate both the general method and the effect of sodium methylate conversions of ethyl ethylmalonate to ethylmalondiamide in the presence and in the absence of sodium methylate is described.

TABLE I

Ester, ethyl	Time, hours	Amide	M. p., °C.	Yield, %
Ethylmalonate	96	Ethylmalondiamide	215[8]	91
n-Butylmalonate	72	n-Butylmalondiamide[a]	198	87
Benzylmalonate	64	Benzylmalondiamide	224–226[4]	96
Diethylmalonate	336	Diethylmalondiamide	225[8,10]	3.5

^{a} *Anal.* Calcd. for $C_7H_{14}O_2N_2$: C, 53.2; H, 8.9. Found: C, 53.2; H, 8.9.

Conversion of Ethyl Ethylmalonate to Ethylmalondiamide.—(a) In the presence of sodium methylate: The ester (10 g.) was dissolved in methanol (50 ml.) and a solution of ammonia in methanol (100 ml. of a solution saturated at 0°) containing sodium methylate (from 0.1 g. of sodium) was added. The mixture was allowed to stand in a stoppered flask at room temperature. The diamide soon began to separate. It melted at 215° (lit. gives 216°[8]) without further purification. After thirty-six hours the amount of diamide corresponded to a 62% conversion while after ninety-six hours the separated diamide together with a further small amount obtained by evaporation and acidification of the residue weighed 6.3 g. representing a yield of 91%. (b) Without sodium methylate: The above experiment was repeated but no sodium methylate was added. After sixty hours the amount of diamide separated corresponded to a conversion of about 33%. The amount separated after eighty-four hours together with a small amount obtained by removal of the solvent was 4.4 g. (62%).

(10) Meyer, *Ber.*, **39**, 198 (1906).

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Bromination of 9,10-Dihydroanthracene

BY JOHN R. SAMPEY, ANNE K. MCCUEN AND JESSIE M. COX

There is little information in the literature on the bromination of 9,10-dihydroanthracene. Barnett[1] discusses the *cis–trans* isomerism of 9,10-dibromo-9,10-dihydroanthracene, but gives no prep-

(1) Barnett, *Rec. trav. chim.*, **44**, 218–223 (1925).

aration of the same. Oda[2] measured the rates of bromination without identifying any products. Graebe[3] prepared a dibromoanthracene, melting 219–220° with no details or yield given. We have studied both the photochemical and dark room bromination of peroxide free samples of this hydrocarbon under anhydrous conditions.

The addition during two hours of 0.10 mole of bromine in 50.00 ml. of carbon disulfide to 0.05 mole of dihydroanthracene in 70.00 ml. of the same solvent at 0° with a small (2″) mercury arc 6″ distant, resulted in a 51% yield of 9,10-anthracene dibromide, melting at 219–220°, and giving 46.8% bromine (theory 47.0%) by a Rosanoff analysis. Repeating the bromination in carbon tetrachloride on a boiling water-bath for forty-five minutes gave a 44% yield. Using a large arc (6″) at 4″ distance for thirty-five minutes on a water-bath with carbon disulfide as solvent, cut the yield to 24%.

The dark room bromination of dihydroanthracene gave a mixture of poly-bromo derivatives. In an overnight run with 0.10 mole of bromine in carbon disulfide with 1 g. of iodine present, 0.05 mole of dihydroanthracene gave 7% of the anthracene dibromide, melting at 220°, and 30% of tribromoanthracene, melting at 170°. Kaufler[4] gives the melting point of 2,9,10-tribromoanthracene as 171°.

(2) Oda, *Sci. Papers, Tokyo*, **33**, no. 728, 129–208 (1937).
(3) Graebe, *Ann. Chem. Phys.*, **7**, 257–306 (1870).
(4) Kaufler, *Ber.*, **37**, 4707 (1904).

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Spectroscopic Evidence Concerning the Structure of 2-Phenyl-4,4-dimethyl-5(4)-oxazolone Hydrobromide. A Correction

BY CURTIS W. SMITH

The present note is an attempt to correct a mistaken statement concerning the work of Hunter, Hinman and Carter.[1] In our paper[2] "Spectroscopic Evidence Concerning the Structure of 2-Phenyl-4,4-dimethyl-5(4)-oxazolone Hydrobromide" we stated: "During a study of the reaction of β-methylbenzylpenicilloate with phosphorus tribromide, collaborators from the University of Michigan and the Upjohn Company[1] determined the infrared absorption of a solution of the penicilloate and phosphorus tribromide in carefully dried dioxane immediately after combination and then again at short intervals. The oxazolone hydrobromide shown in the accompanying equation

(1) Hunter, Hinman and Carter, "Methyl Benzylpseudopenicillinate" in "The Chemistry of Penicillin," Princeton University Press, Princeton, N. J., 1949.
(2) Smith and Rasmussen, THIS JOURNAL, **71**, 1080 (1949).