cooled to 10°. A cold solution of 0.71 g. of potassium permanganate in 70 ml. of water then was added and the mixture was allowed to warm up to room temperature. After 3.5 hours at room temperature the mixture was filtered through Super-cel and the precipitate was washed well with water. The filtrate was acidified to congo red and the product was filtered, washed with water and dried over sulfuric acid. The crude product, obtained in 94% yield, contained an amorphous impurity. After recrystallization from high-boiling petroleum ether it weighed 0.65 g. (68%) and melted at 106.5–109.5°.

6-Propyl-7-hydroxy-5,8-quinolinequinone (VI) was converted to the phenazine derivative with *o*-phenylenediamine by the procedure used for II (above). The yield was the same. After recrystallization from benzene this phenazine had no definite melting point; it gradually decomposed above 240° and became black at about 250° without melting.

Anal. Calcd. for $C_{18}H_{15}NO_3$: C, 74.72; H, 5.23. Found: C, 74.80, 74.90; H, 5.09, 5.04.

Mannich Reactions.—To a suspension of 0.025 mole of I in 30 ml. of absolute alcohol a 10% excess of amine was added with cooling. When solution was complete, 2.1 ml. of 40% formaldehyde solution was added with stirring at 5–10°. The reaction then was allowed to continue overnight at room temperature although it appeared to be essentially complete in about 4 hours. The crystalline products, which precipitated from the reaction mixture, melted no more than 2° below the melting points of the purest samples. The diethylamino derivative VIII, however, was best isolated after the addition of anhydrous ether to the suspension; the product was washed well with ether and purified by dissolving in methanol at about 40° and precipitating with anhydrous ether. The hexylamino compound X also was recrystallized from this solvent mixture and the piperidino derivative IX from 95% ethanol and ether. Both VIII and IX were very soluble in water, but the hexylamino compound X was insoluble. The piperidino derivative was slightly hygroscopic. Compound VIII was orange; compounds IX and X were deep red.

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NOTES

The Solubility of DDT in Water Determined Radiometrically

BY FRANK H. BABERS

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The solubility of DDT in water has been given by Gauvadan and Poussel[1] as approaching 0.1 p.p.m. at 18°, by Roeder and Weiant[2] as between 0.1 and 0.01 p.p.m., and by West and Campbell[3] as "practically insoluble." Gauvadan and Poussel used a nephelometric method, and Roeder and Weiant used for their calculations the time required for the appearance of symptoms of DDT poisoning in the ventral nerve cord of a roach following treatment with saline-saturated solutions of DDT.

Recently in connection with experiments on the physiological action of DDT on insects, the insecticide labeled with carbon-14 has become available. Because of its physiological interest and the paucity of data on the subject, the water solubility of the material has been determined at three temperatures by radiochemical methods.

Experimental

A solution of the radioactive DDT was prepared by dissolving 3.046 mg. in 2 ml. of acetone. 300 Microliters were transferred to a 100-ml. flask and the acetone evaporated at room temperature. Water redistilled from glass was added and the flask heated on a steam-bath with shaking for 1 hour. After cooling somewhat, the flask was put in a constant-temperature bath and shaken for at least 1 week. Samples were then removed and centrifuged at the bath temperature, a portion was filtered through a fine sintered-glass funnel, and aliquots of each portion were taken for analysis. To 1 ml. of acetone containing 1 mg. of non-radioactive DDT in an aluminum counting cup 0.5 ml. of the water solution of radioactive DDT was added, and most of the solvent was evaporated with an infrared lamp. One milliliter of acetone was then added, followed by a second 0.5-ml. portion of the DDT–water solution. The sample was then evaporated to dryness and its radioactivity determined in a proportional counter.

The solubility results were not altered by filtration through sintered glass. At least 10 replications were made and the results compared with the activity obtained from 10 samples of known size. Because of the reproducibility of the results, correction for self absorption was not made. Each preparation was about 0.35 mg./cm.² in thickness. From the results of these experiments, the solubility of DDT in water is 5.9 ± 0.4 micrograms per liter (0.0059 ± 0.0004 p.p.m.) at 2°, 37.4 ± 0.5 micrograms (0.0374 ± 0.0005 p.p.m.) at 25°, and 45 ± 1 micrograms (0.045 ± 0.001 p.p.m.) at 37.5°.

It was found that in very thin layers and microgram qualities DDT is appreciably volatile at room temperatures. This became apparent in preliminary experiments when after 17 samples to which non-radioactive DDT had not been added had been counted, the background of the counting chamber had risen from 59 counts per minute at the start to 311 at the end due to contamination. By the addition of non-radioactive DDT, the sample size was greatly increased and at the same time, the percentage of active DDT decreased so that no further contamination was noted.

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[1] P. Gauvadan and H. Poussel, *Compt. rend.*, **224**, 683 (1947).

[2] K. D. Roeder and E. A. Weiant, *Science*, **103**, 304 (1946).

[3] T. F. West and G. A. Campbell, "DDT and Newer Persistent Insecticides," 2nd Ed. rev., Chapman and Hall, Ltd., London, 632 pp. (1950).

[4] The radioactive material used in these experiments was procured under the authority of the United States Atomic Energy Commission.

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The Uncatalyzed Thermal Addition of Formaldehyde to Olefins

BY NEAL O. BRACE

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The uncatalyzed thermal addition of formaldehyde (from paraformaldehyde) to dialkyl-substituted terminal olefins such as diisobutylene,[1] β-pinene[2] and methylenecyclohexane,[3] or to an alkyl-

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[1] J. J. Ritter, U. S. Patent 2,335,027; *C. A.*, **38**, 2662 (1944).

[2] J. P. Bain, THIS JOURNAL, **68**, 638 (1948).

[3] R. T. Arnold and J. F. Dowdall, *ibid.*, **70**, 2590 (1948).