Anal. Calcd. for $C_{12}H_8ClN_3O_2$: C, 55.08; H, 3.08. Found: C, 55.28; H, 3.34.

5-Chloro-2-nitroazobenzene.—The procedure was as described above. From 1.9 g. (0.01 mole) of 5-chloro-2-nitronitrosobenzene[13] and 1.0 ml. (0.9 g., 0.01 mole) of aniline was obtained after sublimation 2.4 g. (92%) of bright red needles, m.p. 92.6–94.2°. The sublimate was recrystallized from 95% ethanol to give 2.2 g. (84%) of orange needles, m.p. 93.6–94.2° (lit.,[16] m.p. 94°).

6-Chloro-2-phenylbenzotriazole Oxide.—A solution prepared by warming 2.6 g. (0.01 mole) of 4-chloro-2-nitroazobenzene in 10 ml. of 95% ethanol was stirred magnetically and a solution of 9.6 g. (0.04 mole) of sodium sulfide nonahydrate in 30 ml. of water was added all at once. After stirring at room temperature for 17 hr. the mixture was cooled in an ice bath and the light tan solid was collected by suction filtration. The crude material was sublimed at 125° (0.05 mm.) to give 2.4 g. (97%) of pale yellow crystals. Two recrystallizations from 95% ethanol (Norit) gave 2.2 g. (90%) of white needles, m.p. 141.6–142.4°. A small sample which had been recrystallized twice more from ethanol melted at 142.2–142.8°.

Anal. Calcd. for $C_{12}H_8ClN_3O$: C, 58.67; H, 3.28. Found: C, 58.91; H, 3.15.

5-Chloro-2-phenylbenzotriazole Oxide.—The procedure was as described above. From 2.6 g. (0.01 mole) of 5-chloro-2-nitroazobenzene and 9.6 g. (0.04 mole) of sodium sulfide nonahydrate was obtained after sublimation 2.3 g. (94%) of light yellow crystals. Two recrystallizations from 95% ethanol (Norit) gave 2.0 g. (82%) of white needles, m.p. 139.8–140.4° (lit.,[16] m.p. 142.5°).

Attempted Isomerization of Benzotriazole Oxides.—Separate 0.1-g. portions of 6-chloro-2-phenylbenzotriazole oxide and 5-chloro-2-phenylbenzotriazole oxide were heated for 2.5 hr. in an oil bath maintained at 145°. The 5-chloro isomer became dark on heating while the 6-chloro compound underwent no visible change. In each case, the infrared spectrum of the sample after heating was identical with that of the pure compound.

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Naphthalene as a Photoquencher

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Our interest in a photoadduct between naphthalene and maleic anhydride was intensified by reports of an analogous reaction proceeding with benzene.[1-3] Preliminary evaluation led us to the conclusion the former reaction should proceed in the same manner as the latter and, as a consequence, the reaction would be of lesser significance. However, upon further consideration it was observed that a decision could be made between a 1,2- and a 1,4-addition as the initial reaction of maleic anhydride with naphthlene if the reaction occurred. An analogy could then be made to the benzene photoproduct formation. Two quite different products would be expected depending on the mode of the first addition.

Upon irradiation of naphthalene and excess maleic anhydride in tetrahydrofuran, no photoadduct was found. Naphthalene was recovered quantitatively and the maleic anhydride nearly so with an uncharacterized polymeric material. In an irradiation under identical circumstances without naphthalene, the polymer was obtained exclusively.

On the basis of these results we conclude that naphthalene has a quenching effect on the photoinduced polymerization of maleic anhydride. A similar observation has recently been reported by Moore and Ketchum[4] for the influence of naphthalene on the photoreduction of benzophenone.

Experimental

The following reaction solutions were irradiated simultaneously in identical quartz vessels with a 140-watt Hanovia high pressure mercury arc lamp.

Irradiation of Naphthalene and Maleic Anhydride.—A solution of 5.6 g. (0.057 mole) of maleic anhydride and 1.44 g. (0.011 mole) of naphthalene in 30 cc. of purified tetrahydrofuran was irradiated for 8 hr. Distillation of the reaction mixture provided 4.88 g. (87%) of maleic anhydride. Chromatography of the residue on alumina produced 1.40 g. (97%) of naphthalene. On further elution a gum appeared which was not completely eluted until ether was passed over the column.

Irradiation of Maleic Anhydride.—The above procedure was duplicated omitting the naphthalene. Distillation provided no maleic anhydride and alumina chromatography failed to yield material other than an amorphous gum.

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Formation of Dibenzothiophene by a Disulfide Ring Closure[1]

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Disulfides, derived by oxidation of β-aryl-α-mercaptoacrylic acids, have been shown in earlier reports[2,3] to undergo cyclization in the presence of a Lewis acid to give the corresponding condensed

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