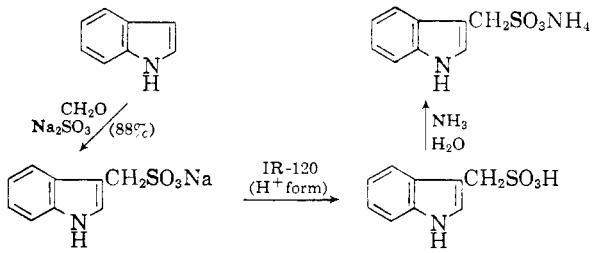
the addition of aqueous ammonia to a solution of the free acid, was found to be comparable in stability to the sodium salt. All efforts to react free 3-indolemethanesulfonic acid or either its sodium or ammonium salt with an alkali cyanide to produce 3-indoleacetonitrile were unsuccessful.



EXPERIMENTAL

Indole (30 g., 0.26 mole) and sodium sulfite (126 g., 1.00 mole) were placed in water (250 ml.) and heated to gentle reflux with stirring. Aqueous formaldehyde (50 ml. 36.2% solution equivalent to 18.1 g. or 0.60 mole) was added and the mixture refluxed gently for 18 hr. The reaction mixture was then cooled and the crystalline precipitate collected by filtration. Several extractions of the product first with ether, then methanol removed the unchanged indole (10 g.). The crystalline residue was dissolved in a minimum amount (800 ml.) of boiling water and saturated sodium bromide solution (200 ml.) added. The resulting solution was cooled to 0–5° and the precipitate of sodium 3-indolemethanesulfonate which formed collected by filtration. After air drying the white crystalline product amounted to 35 g. (0.16 mole, 88% yield at 59% conversion). A second recrystallization from water (300 ml.) and saturated sodium bromide solution (90 ml.) reduced the yield to 29.5 g. (0.13 mole).

Anal. Calcd. for $C_9H_8O_3NSNa$: C, 46.38; H, 3.43; N, 6.01; S, 14.35. Found: C, 46.16; H, 3.86; N, 6.10; S, 14.35.

The Preparation of Diethyl Peroxide. The Use of Dispersing Agents to Increase Yields in Heterogeneous Systems[1]

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The difficulties with regard to the preparation of diethyl peroxide have been described by Leadbeater.[2] According to this investigator, the usual yield of "pure" compound obtained after a number of fractionations was about 2–3%. The methods of preparations reported in the literature as also used by Minkoff[3] were employed to prepare this compound needed for kinetic studies. Unfortunately, all attempts using both methods were met with utter failure. With somewhat modified procedural details, we have been able to prepare the pure product in about 28–30% yields. The significant high yield resulted from the use of sodium stearate as a dispersing agent.

Experimental

To a three-neck Pyrex flask (500 ml.), containing 120 ml. of 30% hydrogen peroxide, was added about 50 mg. of sodium stearate dissolved in a few milliliters of distilled water. The flask was kept cool at or below −35° by using a bromobenzene–Dry Ice slush bath (usually, a simple Dry Ice bath proved equally good and handy, and was mostly used in later preparations). A solution of 56 g. of potassium hydroxide dissolved in 60 ml. of distilled water was added dropwise to the flask whose contents were kept stirred during this process. The result was an almost semifluid mass. The flask was transferred to a common salt-ice bath. Ethyl sulfate, 154 g., was added slowly (a few drops at a time, over a period of about 1 hr.), through another inlet tube. The contents became entirely liquid in the course of the addition of the ethyl sulfate. The temperature of the mixture was kept below −10° during the addition. After the addition, the contents were kept stirred overnight (for about 16 hr.) in the ice bath. No attempt was made to study the effect of period of stirring on the yield. The oily layer was extracted with anisole the next day and washed two to three times with distilled water to remove as much alkali as possible. The extract was transferred to a glass-stoppered flask, 1–2 drops of phenolphthalein were added and sulfuric acid (4 *N*) was added dropwise just to the disappearance of pink color. Anhydrous sodium sulfate (Analar grade), 10 g., was added to the extract. After 1 hr. of drying, the contents were vacuum-distilled (pressure = 20 mm.), raising the temperature of the distillation flask slowly to about 60° toward the end of the operation. The receiving set consisted of a series of traps; the first trap cooled in an ice bath could entrap any anisole that distilled, while the peroxide was found in the two successive traps at liquid nitrogen temperature. The peroxide from both the traps was transferred to a 100 ml. round-bottomed flask fitted with a Vigreux column which had a side-water-condenser and a fraction-cutter of four receivers. The product was fractionated using a water bath; the fraction distilling between 62–63° was collected for subsequent analysis. The infrared spectrum of the product was taken on an Infracord (Perkin-Elmer) and it matched with the one reported by Minkoff.[2] The refractive index also agreed with the value reported in the literature, $n^{20}D$ 1.3698. A yield of 25.6 g. was obtained in one particular trial, and it ranged from 25 to 28 g.

A Stereospecific Photochemical Addition of Acetone to Norbornylene

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Photochemical addition reactions of olefins with carbonyl compounds were reported early in this century,[1] but only in recent years has the scope and variety of these transformations become

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(2) R. J. Leadbeater, *Bull. soc. chim. France*, 1285 (1956).

(3) G. J. Minkoff, *Proc. Roy. Society* (London), **A 224**, 176 (1954).

(1) E. Paterno and G. Chieffi, *Gazz. chim. ital.*, **396**, 341 (1909).