

boiled for thirty minutes and then extracted with chloroform. The chloroform extracts, washed thoroughly with water, were concentrated and a low melting solid was obtained which was recrystallized from ethanol (1.5 g., m. p. 38–40°).

2-Methyl-3-*n*-hexadecyl-1,4-naphthoquinone.—The oxidation was accomplished by adding a solution of chromic anhydride to the hydrocarbon in acetic acid at room temperature. After heating for one-half hour at 60°, the product was isolated by diluting with water, extracting with ether, and concentrating the extracts. Purification of the crude quinone was effected by converting it into the hydroquinone, washing the latter thoroughly with petroleum ether and reoxidizing with ferric chloride. The quinone was obtained from ether as pale yellow, fluffy needles melting at 98–98.5°; yield, 35%.

Anal. Calcd. for $C_{27}H_{40}O_2$: C, 81.81; H, 10.10. Found: C, 81.61; H, 10.22.

The dihydrodiacetate from petroleum ether melts at 78–79°.

Anal. Calcd.: C, 77.18; H, 9.54. Found: C, 77.03; H, 9.29.

RESEARCH LABORATORIES

MERCK & CO. INC.

RAHWAY, NEW JERSEY RECEIVED SEPTEMBER 26, 1941

The Densities of Morpholine–Water Solutions¹

BY H. M. TRIMBLE AND ALICE FRANCES BUSE

Morpholine was prepared for this work by the commonly accepted method,² taking special precautions to exclude water and carbon dioxide of the air during its final distillation. The refractive index of several preparations at 20° was 1.4542 ± 0.0002.

TABLE I

DENSITIES OF MORPHOLINE–WATER SOLUTIONS

Morpholine % by weight	25°	Densities 30°	35°
14.17	1.0066	1.0047	1.0026
21.97	1.0128	1.0105	1.0083
26.72	1.0165	1.0140	1.0166
35.55	1.0238	1.0210	1.0180
47.36	1.0325	1.0290	1.0255
53.07	1.0355	1.0317	1.0280
57.05	1.0369	1.0330	1.0291
72.67	1.0352	1.0308	1.0264
84.05	1.0249	1.0204	1.0158
89.65	1.0164	1.0119	1.0073
100.00	0.9947	0.9897	0.9850

Each value of the density given in the table is the mean of four determinations, agreeing to one part in ten thousand or better, made with four different pycnometers.

(1) We wish to express our appreciation to the Carbide and Carbon Chemicals Corp., 30 East 42nd Street, New York, N. Y., who kindly supplied the morpholine used in this study.

(2) Friedman, Barnard, Doe and Fox, *THIS JOURNAL*, **62**, 2366 (1940).

Friedman, Barnard, Doe and Fox² have determined the specific gravities of morpholine–water solutions at 20°. The densities at this temperature, found by interpolation in their data and conversion, fall exactly in line with our own. The variation of density with temperature is nearly exactly rectilinear for all of the solutions. We find that the density of pure morpholine at 20° is 0.9994 as given by Dermer and Dermer,³ not 1.0007 as quoted by Friedman, *et al.*, from Beilstein.

(3) Dermer and Dermer, *THIS JOURNAL*, **59**, 1148 (1937).

CHEMISTRY DEPARTMENT

OKLAHOMA AGRICULTURAL AND MECHANICAL COLLEGE

STILLWATER, OKLAHOMA

RECEIVED AUGUST 20, 1941

NEW COMPOUNDS

Bicyclohexylidene-2,2'-sulfone

Four grams (4.2 cc., 0.025 mole) of dicyclohexenyl (di- $\Delta^{1,1'}$ -cyclohexene)¹ and 10–12 g. (0.16–0.19 mole) of liquid sulfur dioxide plus a few crystals of hydroquinone² were mixed in a glass-lined steel pressure tube. The tube was sealed and heated in a steam jacket for twelve hours. It was then cooled to about 10°, opened, and the excess sulfur dioxide allowed to pass off. The residual liquid was diluted with 6 cc. of petroleum ether (90–100°), the solution cooled in dry-ice and the walls of the tube scratched to induce crystallization. The crystalline mass was kept at 0° for about one hour and then suction filtered. Recrystallization from petroleum ether yielded 2.3 g. of practically colorless crystals, m. p. 76–77°, yield 50%.

Anal. Calcd. for $C_{12}H_{18}S_2$: S, 14.16. Found: S, 14.02.

This procedure for synthesizing a sulfone is substantially that used by Eigenberger³ in preparing the cyclic sulfone from isoprene. Bicyclohexylidene sulfone, like other compounds of this type, decomposes rapidly at 110–120° to form sulfur dioxide and dicyclohexenyl.

(1) Gruber and Adams, *THIS JOURNAL*, **57**, 2555 (1935).

(2) Staudinger, *British Patent* 361,341 (1930).

(3) Eigenberger, *J. prakt. Chem.*, **127**, 307 (1930).

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Aminobenzene-(4-azo-1')-2'-methyl-4'-N-di-(β -hydroxyethyl)-aminobenzene

To a hydrochloric acid solution of 3-methyl-N-di-(β -hydroxyethyl)-aminobenzene prepared by the quantitative condensation of 1 mole of *m*-toluidine and 2 moles of liquid ethylene oxide under pressure, was added an equimolecular quantity of diazotized *p*-nitroaniline, prepared in the usual manner. A 20% solution of sodium acetate