unground samples were evacuated at 290° for four hours. The surface area of the unground catalyst, as determined from the nitrogen adsorption isotherm, using the weight after evacuation, was 1061 sq. m./g., whereas that of the ground catalyst was 991 sq. m./g., a difference of 6.7%. Little, if any, effect was observed on the average Kelvin pore radius, as determined from the relative pressure corresponding to the steepest portion of the desorption curve. The unground sample had an average pore size of 21.1 A., whereas the ground sample had an average pore radius of 20.7Å.

STANDARD OIL DEVELOPMENT CO.
ELIZABETH, NEW JERSEY RECEIVED DECEMBER 4, 1947

---

## Preparation and Characterization of β-Dibutylaminoethyl Chloride Hydrochloride

BY HOWARD C. KLEIN[1] AND IRVING A. KAYE

β-Dibutylaminoethyl chloride hydrochloride has been prepared by the dropwise addition of 47.2 g. (0.4 mole) of thionyl chloride to 51.9 g. (0.3 mole) of β-dibutylaminoethanol, dissolved in 150 ml. of benzene. After removal of the solvent and excess thionyl chloride under diminished pressure, the tarry residue was dissolved in dry acetone, the solution decolorized with Darco G-60, and then cooled with Dry Ice. The precipitated white hydrochloride (57.9 g., 85%) melted at 86–88°.

*Anal.* Calcd. for $C_{10}H_{23}NCl_2$: $Cl^-$, 15.41; Cl (total), 30.82. Found: $Cl^-$, 15.30; Cl (total), 30.92.

Recrystallization of the hydrochloride from acetone–hexane gave pearly leaflets, m.p. 87–89°.

The basic chloride boiled at 119–120° (30 mm.).[2]

[1] Nopco Chemical Company, Fine Chemicals Division, Harrison, New Jersey.

[2] Blicke and Maxwell, THIS JOURNAL, **64**, 429 (1942), found 114–115° (23 mm.).

DEPARTMENT OF CHEMISTRY
BROOKLYN COLLEGE
BROOKLYN, NEW YORK RECEIVED NOVEMBER 10, 1947

---

## Synthesis of Amino-substituted Phosphonic Acids. II. α-Aminobenzylphosphonic Acid

BY GENNADY M. KOSOLAPOFF

As it was reported earlier[1] the synthesis of α-aminobenzylphosphonic acid by the reduction of *p*-nitrophenylhydrazone of diethyl benzoylphosphonate by means of hydrogen, followed by sodium sulfide, failed to yield the pure product. The contamination of the final product was caused principally by the products of incomplete reduction and led to the unusually low melting point of the substance. In order to secure the pure material it was necessary to find another reduction method which was usable in this instance. Such a procedure using the method of Fischer and Groh[2] for the reduction led to the pure substance.

The hydrazone (4 g.) in 500 cc. of ethanol was added to 10 g. of amalgamated aluminum foil immersed in 1 liter of ethanol and 200 cc. of water. The mixture was allowed to stand overnight in a loosely stoppered flask. After filtration, the precipitate was washed with 500 cc. of ethanol and the combined filtrates were treated with 300 cc. of concentrated hydrochloric acid. The solution was slowly distilled (six hours) through a Vigreux column until the volume in the still-pot amounted to approximately 50 cc. On cooling, the *p*-phenylenediamine hydrochloride was filtered off, the filtrate was evaporated to dryness by an infrared lamp and the residue was taken up in 200 cc. of ethanol. Neutralization with aniline gave the crude product which was taken up in 30 cc. of warm water, made strongly alkaline with 10% sodium hydroxide and extracted twice with 50 cc. of benzene. The aqueous solution was made just acid to congo red with 1:1 hydrochloric acid and concentrated to incipient crystallization. After cooling, the product was filtered off, washed with 5 cc. of ice-water, dissolved in 50 cc. of hot water, treated with charcoal, filtered and concentrated to 10 cc. On cooling there was obtained 1.0 g. (50%) of *dl*-α-aminobenzylphosphonic acid, in the form of tiny bunched colorless needles, which melted at 272–273°.

*Anal.* Calcd.: N, 7.5; P, 16.6. Found: N, 7.46; P, 16.7.

[1] Kosolapoff, THIS JOURNAL, **69**, 2112 (1947).

[2] Fischer and Groh. *Ann.*, **383**, 363 (1911).

CENTRAL RESEARCH DEPARTMENT
MONSANTO CHEMICAL COMPANY
DAYTON 7, OHIO RECEIVED SEPTEMBER 27, 1947

---

## Copolymerization Rate Constants in the System 2,5-Dichlorostyrene–Methyl Acrylate

BY F. LEONARD, W. P. HOHENSTEIN AND E. MERZ

During the course of an investigation of the copolymerization of the isomeric dichlorostyrenes and acrylic esters, it became of interest to determine the copolymerization rate constants in the system 2,5-dichlorostyrene and methyl acrylate. The results of this determination are herein presented.

### Experimental

**2,5-Dichlorostyrene.**—This monomer was obtained from the Monsanto Chemical Co. and was purified by washing with 5% sodium hydroxide solution and then with distilled water until the washes were neutral to litmus. The monomer was allowed to dry over calcium chloride overnight. It was then filtered and checked for the presence of polymer by adding a sample to methanol. If no turbidity was observed, the monomer was used *per se*.

**Methyl Acrylate.**—This monomer obtained from Rohm and Haas was distilled *in vacuo* through a 12″ column packed with glass helices in an all-glass system. The first 10 cc. to distil was rejected and a residue of 50 cc. was left in the distilling flask.

**Polymerization and Purification.**—The requisite quantities of 2,5-dichlorostyrene, methyl acrylate and benzoyl peroxide (0.5% by weight based on monomers) were weighed into Pyrex glass test-tubes. The tubes were sealed, and immersed in a water-bath held at 70 ± 0.1°. The per cent. conversion was estimated approximately by the rate of rise of air bubbles through the polymerizing mixture when the tubes were inverted. When it appeared that the desired conversion, <5%, had been reached, the tubes were immediately chilled, and the