mersed in an ice-bath and shaken vigorously at intervals of a few minutes until a solid formed. Purification of the solid was effected by recrystallization from ligroin.

(b) **Monohalogenated Esters.**—Although the derivatives of monohalogenated esters may be prepared as in (c), a better procedure is to heat 2 mols of the amine with 1 mol of the ester for a few minutes when the hydrohalide forms. To obtain the salt in pure form, it was dissolved in hot ethyl acetate containing sufficient ethyl alcohol to effect solution, after which the solid crystallized from the cooled solution upon the addition of a few cc. of ether.

(c) **Non-halogenated Esters.**—The ester, 2 cc., mixed with 4 cc. each of benzylamine and water and, when necessary, sufficient ethyl alcohol or dioxane to effect solution, was refluxed one hour (for formates), two hours (for acetates) or from four to five hours (for higher esters like caproates). The excess of reagents was removed by distilling up to 200°, after which the derivative[2] in the distillation flask was crystallized from hot ligroin by cooling to room temperature or lower.

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[2] Before final crystallization, derivatives melting below 50° were distilled off over a range beginning at 290° and continuing until decomposition occurred in the distillation flask.

DABNEY CHEMICAL LABORATORY
UNIVERSITY OF TENNESSEE
KNOXVILLE, TENN.
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The Thermal Decomposition of Gaseous Hydrogen Peroxide

BY G. B. KISTIAKOWSKY AND S. L. ROSENBERG[1]

The thermal decomposition of gaseous hydrogen peroxide was investigated semi-quantitatively by a static method at 85 and 98°. A very pure, 70% peroxide was used. The reaction in a quartz flask is heterogeneous, with an almost negligible temperature coefficient. Times for 50% reaction varying from one to thirty minutes were obtained, the reaction rate increasing with time due to changes in the reaction vessel surface. No stopping of the reaction at about 15% completion as described by Elder and Rideal[2] could be detected. Furthermore, the pressure increase accompanying the reaction proved to be the theoretical value of 50% (based on hydrogen peroxide present), rather than the somewhat astonishing value of about 1000% found by these authors.

Some experiments were tried, where the reaction vessel was flushed with oxygen prior to the reaction. This had no effect, so that neither oxygen from the reaction nor other oxygen leads to a stoppage of the reaction before completion. In another experiment a thin coating of paraffin wax was applied to the interior of the quartz reaction bulb. This slowed down the reaction somewhat, demonstrating its heterogeneity.

We take this opportunity of thanking Mr. P. Giguere of McGill University for the gift of some very pure 70% hydrogen peroxide.

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[1] McGill University Moyse traveling fellow.

[2] Elder and Rideal, *Trans. Faraday Soc.*, **23**, 545 (1927).

HARVARD UNIVERSITY
CAMBRIDGE, MASS.
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Syntheses from Ethanolamine. IV. A Synthesis of β-Chloroethyl Phthalimide

BY HENRY WENKER

This paper presents a convenient method of preparing β-chloroethyl phthalimide, an intermediate which can be used for most purposes in place of the less readily accessible β-bromo analog.[1] The reactions involved are: phthalic anhydride and ethanolamine react in molar proportions to form β-hydroxyethylphthalimide; this compound reacts with one mole of phosphorus pentachloride to yield β-chloroethyl phthalimide.

Experimental

Garelli and Racciu[2] obtained β-hydroxyethyl phthalimide from phthalic anhydride and ethanolamine, using absolute ethanol as a solvent. It has been found that by omitting the solvent and heating both components[3] for about thirty minutes to 210°, a quantitative yield is obtained. On cooling, the product solidifies in crystalline form, melting at 127–128° (lit. 127–128°). Through the action of phosphorus pentachloride, β-chloroethyl phthalimide was obtained from this product in the usual way. The yield was 94%, the melting point 81° (lit. 79–81°).

ELIZABETH, N. J.
RECEIVED DECEMBER 4, 1936

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[1] "Organic Syntheses," John Wiley and Sons, Inc., 1932, Coll. Vol. I, p. 114.

[2] Garelli and Racciu, *Atti acad. sci. Torino, Classe sci. fis, mat. nat.*, **69**, I, 358–63 (1934).

[3] Technical monoethanolamine, boiling at 169–171°, from Carbide and Carbon Chemicals Corporation, was used.