

## PREPARATION AND PROPERTIES OF PERLAURIC ACID

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Although the preparation and properties of the lower aliphatic peracids, *e.g.*, peracetic acid, are abundantly described in the literature (1), the higher aliphatic peracids have been studied to a limited extent only.

No adequate description can be found in the literature of the C<sub>12</sub>-C<sub>18</sub> straight chain aliphatic peracids. D'Ans and Frey (2) reported the detection of perpalmitic acid, but no details were given. Small, *et al.* (3) have more recently mentioned perlauric acid, but no description of preparation or properties were noted.

Following the discovery of the bactericidal-fungicidal activity of peracetic acid (4, 5), it was of interest to investigate the higher homologs of peracetic acid, particularly those derived from the solid C<sub>12</sub>-C<sub>18</sub> straight chain acids possessing surface active properties in the form of the alkali soaps. This paper reports studies of perlauric acid.

Perlauric acid has been prepared in good yield by reaction of 90% hydrogen peroxide with molten lauric acid in the presence of a strong acid catalyst. This represents an adaptation to a heterogeneous reaction of one of the standard methods for (6, 7) the preparation of lower aliphatic peracids. The product was a white waxy solid, possessing good stability after recrystallization. On solution in alkali, the perlauric acid formed a germicidal soap with considerably enhanced bactericidal-fungicidal activity over the comparable sodium laurate, although somewhat less active than sodium peracetate. Methyl epoxystearate was formed in good yield (76%) on reacting perlauric acid with methyl oleate in chloroform solution, indicating the general applicability of perlauric acid to epoxidation-hydroxylation reactions, associated with peracetic acid.

### EXPERIMENTAL

*Materials.* The lauric acid and *p*-toluenesulfonic acid monohydrate were obtained from the Eastman Kodak Co. The 90% hydrogen peroxide was the commercial product of the Buffalo Electro-Chemical Company, Inc. Methyl oleate (Iodine number 86) was obtained from Emery Industries, Inc.

*Analytical.* Perlauric acid was determined by a modification of the method of Greenspan, *et al.* (8) using chloroform as the solvent. Oxirane oxygen was determined by the procedure of Swern, *et al.* (9). The Hanus method (10) was employed for iodine number determinations.

*Preparation of perlauric acid.* Hydrogen peroxide (1.5 moles, 56.6 g. of 90%) was added over a 15-minute period to 300 g. of lauric acid (1.5 moles) containing 0.18 g. of an alkyl aryl sulfonate (Nacconol NR) and 3.0 g. of *p*-toluenesulfonic acid monohydrate, maintained at 50°, in a three-neck flask equipped with a reflux condenser, thermometer, and a mechanical stirrer. After 2 hours' reaction, the mixture was slowly poured, with vigorous stirring, into a beaker containing cold water. The finely divided wax-like product was filtered, washed with cold water, and vacuum-dried. Yield, 306 g. of crude perlauric acid, m.p. 32-35°, and analyzing 43.4% perlauric acid, 0.3% hydrogen peroxide. Four successive recrystallizations of the crude product from petroleum ether (2 g./10 ml.) at 20° gave white needle-like crystals of perlauric acid, m.p. 48-49°, and assaying 95.8% perlauric acid.

TABLE I  
COMPARATIVE BACTERICIDAL ACTION OF PERLAURIC ACID; KILLING  
CONCENTRATIONS<sup>a</sup> vs. TEST ORGANISMS

Material	<i>Penicillium roqueforti</i>	<i>Micrococcus pyogenes</i> var. <i>aureus</i>	<i>Fusarium oxysporum</i>
Sodium laurate.	>5%	>5%	—
Sodium per- laurate.....	0.1% ( $4.6 \times 10^{-3}M$ )	0.05% ( $2.3 \times 10^{-3}M$ )	0.01% ( $4.6 \times 10^{-4}M$ )
Sodium perace- tate.....	0.05% ( $6.6 \times 10^{-3}M$ )	0.001% ( $1.3 \times 10^{-4}M$ )	0.0001% ( $1.3 \times 10^{-5}M$ )

<sup>a</sup> Killing in 10 minutes but not in 5 minutes.

*Properties of perlauric acid.* The crude perlauric acid was a white waxy solid, m.p. 32–35°, insoluble in water, and soluble in ethanol, acetone, hexane, benzene, and chloroform. It possessed a slight fatty odor and was not impact sensitive as determined in a Bureau of Mines' Tester.

The recrystallized product showed good stability on storage at 25° with approximately 1% active oxygen loss per month. The crude product was stable at 10° but gave appreciable decomposition losses at 20°. Addition of perlauric acid in chloroform to an aqueous acidified potassium iodide solution liberated iodine quantitatively. A quantitative liberation of iodine was likewise obtained on addition of perlauric acid to a sodium iodide solution in acetone.

Adding dilute sodium hydroxide to an aqueous dispersion of perlauric acid gave the corresponding sodium perlaurate soap. Aqueous solutions of the latter foamed on shaking and generally showed surface active properties associated with lauric acid soaps. The initially clear soluble perlauric acid soap became cloudy on standing and the pH dropped simultaneously. The sodium perlaurate solutions were unstable and showed rapid decomposition losses on storage.

The bactericidal action of perlauric acid was compared with lauric acid and peracetic acid by a modified phenol coefficient method (11) using *Penicillium roqueforti*, *Micrococcus pyogenes* var. *aureus*, and *Fusarium oxysporum* as test organisms. The results are shown in Table I. The initial pH of all the test solutions was 6–7.

*Reaction of perlauric acid with methyl oleate.* The applicability of perlauric acid to epoxidation-hydroxylation reactions with unsaturated fatty chemicals was studied employing methyl oleate as a model compound.

Methyl oleate (20 g., equivalent to 0.0675 mole of ethylenic unsaturation) was dissolved in 180 g. of chloroform and the solution was cooled to 15°. Then 36.8 g. of 43.4% perlauric acid (0.074 mole) was dissolved in 30 g. of chloroform and slowly added to the methyl oleate solution over a 30-minute period. After an additional four hours reaction at 15–25°, the reaction mixture was transferred to a separatory-funnel. The chloroform layer was washed with successive portions of sodium carbonate until acid-free, dried with sodium sulfate, filtered, and stripped of chloroform under a vacuum. The product (23.5 g.) thus obtained showed the following analysis: Oxirane oxygen, % 3.9; Percent (of theory) yield of epoxy ester, 76; Iodine number, 1.0.

#### SUMMARY

Perlauric acid has been prepared from hydrogen peroxide and lauric acid, and some of its properties have been studied. The product is a white waxy solid possessing good stability in the pure state.

Perlauric acid in chloroform reacted with methyl oleate to give methyl epoxy-

stearate, indicating its applicability to epoxidation-hydroxylation reactions associated with lower aliphatic peracids.

Alkaline solutions of perlauric acid were found to be germicidal soaps with considerably enhanced bactericidal-fungicidal activity over the comparable lauric acid soap.

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