TABLE I

SOME HYDROXY PEROXIDES

Peroxide	Formula	Active oxygen, % Calcd.	Found
Hydroxy dicarbethoxymethyl[a]	$C_7H_{12}O_7$	7.7	7.55
Di-[hydroxy dicarbethoxymethyl][b]	$C_{14}H_{22}O_{12}$	4.19	4.24 4.19
α,α'-Dihydroxydiisoamyl	$C_{10}H_{22}O_4$	7.76	7.60
α,α'-Dihydroxydi-*n*-hexyl[c]	$C_{12}H_{26}O_4$	6.84	6.69

[a] d^{29}_{29} 1.226; n^{27}D 1.4392.
[b] d^{29}_{29} 1.216; n^{27}D 1.4388.
[c] M. p. 62–63°.

yields from aqueous 30% hydrogen peroxide and *n*-hexaldehyde.

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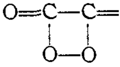
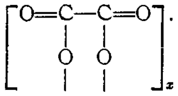
(3) From Part I of Ph.D. Thesis, M. I. T., 1939.

Diperoxalic Acid

When oxalyl chloride[1] was treated at 0° with an ice-sodium peroxide mixture, it was hoped to obtain a carbon peroxide,  $\begin{array}{cccc} O{=}&C&-&C{=}O\\ &|&&|\\ &O&-&O\end{array}$, or one of its polymers,  $\left[\begin{array}{cccc} O{=}&C&-&C{=}O\\ &|&&|\\ &O&&O\\ &|&&|\end{array}\right]_x$. Neither one of these peroxides was obtained, but instead a small yield of diperoxalic acid. A higher yield of this diperacid was obtained by the following, somewhat different, procedure: To a solution of 19 g. of dry pyridine, 285 cc. of anhydrous ether containing 4 g. of hydrogen peroxide and maintained at −20°, was added, dropwise in the course of two hours and with vigorous stirring, a precooled (−10°) solution of 75 cc. of anhydrous ether containing 14.3 g. of oxalyl chloride. At the end of the reaction, the ether layer was decanted and the solid precipitate, which had separated out, extracted with two 100-cc. portions of anhydrous ether. The residue was then treated with an ice-cold mixture of 60 cc. of saturated sodium sulfate solution and 40 cc. of 85% orthophosphoric acid. The resulting mixture, after adding more solid sodium sulfate, was extracted with three 100-cc. portions of acetone which was combined with an equal volume of ether and the mixture dried and filtered. When the solvent was removed under reduced pressure, a highly viscous residue (2.2 g.) remained which failed to crystallize on standing for some time at 0°. This product was free from chlorine and nitrogenous products, and was found to be very soluble in water and chloroform. Diperoxalic acid is a powerful oxidizing agent, and, when treated with potassium iodide, it is reduced rapidly to oxalic acid.

Anal. Calcd. for $C_2H_2O_6$: (O), 26.3. Found: (O), 25.4, 25.7.

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(1) Prepared according to Staudinger, *Ber.*, **41**, 3563 (1908).

(2) From Part I of Ph.D. Thesis, M. I. T., 1939. Present address: Lowell Textile Institute, Lowell, Mass.

COMMUNICATIONS TO THE EDITOR

3-*n*-PENTADECYLCATECHOL

Sir:

Recently the synthesis of 3-*n*-pentadecylcatechol (Hydrourushiol) was described by H. S. Mason.[1] This compound, the dimethyl ether of which was first synthesized in low yield about thirty years ago by Majima and Tahara,[2] and later by Backer and Haack,[3] has attracted considerable interest in view of its close relationship to poison ivy "urushiol." The scheme of synthesis which Mason found gave the best results was essentially that employed by Backer and Haack for the preparation of the dimethyl ether. However, Mason markedly improved the yields over those reported by Backer and Haack, and carried the synthesis one step further by cleaving the dimethyl ether to obtain the catechol compound.

In view of the current interest in 3-*n*-pentadecylcatechol, because of its possible use as a standard agent for the diagnosis and therapy of poison ivy dermatitis, we are prompted to point out that it was synthesized in our laboratory in minimum over-all yield of 57% from *o*-veratraldehyde over two years ago. The synthesis was not published at that time inasmuch as it was a duplication of the work of Backer and Haack except for the improved yields and the additional ether cleavage step. However, the fact that we had synthesized 3-*n*-pentadecylcatechol was made clear in a later article.[4] Furthermore, the data in this article showing the 100% correlation of hypersensitiveness to poison ivy and to standard acetone solutions of 3-*n*-pentadecylcatechol involving patch tests on 21 patients made obvious the possible use of this synthetic compound as a standard agent for the diagnosis and therapy of poison ivy dermatitis. It would appear that Mason overlooked this article since no reference to it was made in his description of the synthesis nor in his statements in regard to the use of 3-*n*-pentadecylcatechol as a standard allergen.

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(1) Mason, THIS JOURNAL, **67**, 1538 (1945).

(2) Majima and Tahara, *Ber.*, **48**, 1606 (1915).

(3) Backer and Haack, *Rec. trav. chim.*, **57**, 225 (1938).

(4) Keil, Wasserman and Dawson, *J. Exptl. Med.*, **80**, 275 (1944.)