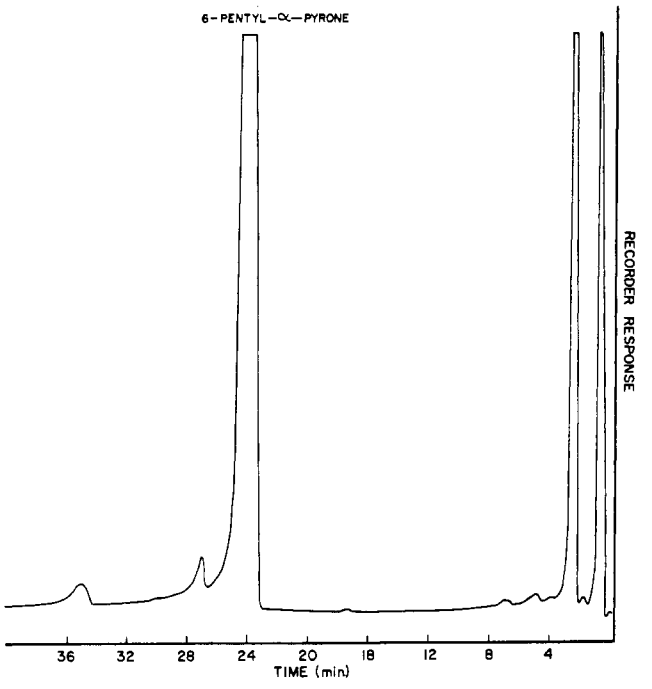
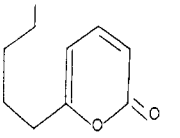


Figure 1. Gas chromatogram of volatiles from *Trichoderma viride* using a Carbowax 20M and PDEAS column

complex multiplet), and 2.48 (2 H, triplet) all consistent with an *n*-pentyl group. The three α-pyrone ring protons formed an ABX pattern with δ_A 5.83, δ_B 6.00, and δ_X 7.19 and J_{AX} = 6.5 and J_{BX} = 9.5 Hz. The ABX protons represent those attached to positions 5, 3, and 4, respectively. All nmr spectra were obtained on a Varian A-60 nuclear magnetic resonance spectrometer.

As an additional proof, the compound was hydrogenated over Adams platinum oxide in ethanol. When the reduction product was subjected to glc (10% W98 column) two peaks were revealed with an approximate peak area ratio of 1:5. The minor peak was identified as *n*-decanoic acid (infrared, retention time) and the major peak was identified as δ-decalactone (infrared, retention time, odor).

The structure of 6-pentyl-α-pyrone is here shown.



This is the first time, insofar as the authors are aware, that 6-pentyl-α-pyrone has been isolated from a fungus; it has been, however, very recently found in peach essence by Sevenants and Jennings (1971). Nobuhara (1969) has synthesized 6-pentyl-α-pyrone along with other unsaturated lactones and a comparison of his published (ir) spectrum with that of the compound reported here shows them to be identical.

It is of interest to note that the origin of this compound in the fungus is primarily the spore rather than the hyphae. This same situation was found by Gehrig and Knight (1958, 1963) in the case of the production of methyl ketones by *Penicillium roqueforti*. Finally it would seem that the yield of this compound by the mold might make it amenable to the flavor industry as a source of a new and interesting natural flavor constituent.

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Determination of Sulfur in Plant Material by Oxygen Flask Combustion

A method is described for the determination of sulfur in plant material. One gram of dry plant material is combusted in a 5-l, oxygen-filled flask. Sulfur is determined turbidimetrically as barium sulfate. Results of analyses of sulfur in plant materials of known sulfur content are presented. The precision of the method was determined (σ = 0.0097) by repeated analysis of one sample of Ladino clover.

The determination of sulfur in plant tissue is subject to error due to volatilization of reduced sulfur compounds during ashing. In open systems, a basic oxidizing substance such as sodium peroxide or basic magnesium nitrate must be added to prevent such losses. These procedures are tedious and must be carried out slowly to prevent ignition which makes the determination worthless. The amount of salt added can also lead to errors in the subsequent precipitation of barium sulfate. Open systems are also subject to errors due to sulfur absorption from flames if a gas burner is used for ashing or from the walls of furnaces routinely used for dry ashing of plants. A comprehensive literature survey on the subject of the determination of sulfur in agricultural samples has been prepared (Beaton *et al.*, 1968). The determination of sulfur in drugs employing an initial combustion in a closed 500-ml flask to obviate vaporization losses was first reported by Schoniger in 1956. Combustion of plant material using this same flask prior to the determin-

ation of sulfur has been reported (Iismaa, 1959) but the small oxygen volume limited the plant sample size to about 100 mg. In the work reported 1 g of dry plant material is combusted in a closed 5-l. oxygen-filled flask (Gutenmann and Lisk, 1960) followed by turbidimetric determination of sulfur as barium sulfate.

EXPERIMENTAL PROCEDURE

One gram of dry plant material was pressed into a pellet using a Parr pellet press having a 0.5 in. bore. The pellet was wrapped in a 1.5-in. square of Whatman No. 41 filter paper and placed in the platinum holder of the 5-l. combustion flask. Gases were absorbed in the flask using the solution of Lysyj and Zarembo (1958). This consisted of 100 ml of a solution containing 6% hydrogen peroxide and adjusted with 0.02 *N* sodium hydroxide to the methyl red endpoint. Combustion was conducted as previously described (Gutenmann and Lisk, 1960). The flask was rinsed with 50 ml of distilled water, which was then combined with the original absorbing solution and made to a total volume of 150 ml with water. An aliquot of this solution up to 50 ml was taken for analysis. The determination of sulfur was performed by the published method (Standard Methods, 1965).

DISCUSSION

The method was applied to the analysis of sulfur in a variety of plant materials. In past years these samples had been repeatedly analyzed by the A.O.A.C. (Official Methods, 1965) method for sulfur in plant material involving magnesium nitrate ashing followed by the turbidimetric determination of sulfur. Table I lists duplicate analyses of sulfur in plant material by the method described and the corresponding percent sulfur determined following the A.O.A.C. (Official Methods, 1965) ashing procedure. Ten replicated analyses of the same sample of Ladino clover showed that the relative standard deviation of the method was 4.04%. We believe the method described should be useful for the rapid determination of sulfur in plant material.

Table I. Percent Sulfur in Plant Material

Plant material	Ashing procedure	
	A.O.A.C.	Oxygen flask
Brome grass	0.22	0.19, 0.21
Ladino clover	0.27	0.25, 0.24
Orchard grass	0.24	0.21, 0.22
Sugar beet leaves	0.61	0.59, 0.59
Timothy	0.24	0.23, 0.24
White pine needles	0.13	0.14, 0.13

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A Potent Juvenile Hormone Mimic, 1-(4′-Ethylphenoxy)-6,7-epoxy-3,7-dimethyl-2-octene, Labeled with Tritium in either the Ethylphenyl- or Geranyl-Derived Moiety

Reduction of citral with sodium borotritide, conversion of the alcohol product to the bromo derivative, formation of the ether by reaction with 4-ethylphenol, and epoxidation yields 1-(4′-ethylphenoxy)-6,7-epoxy-3,7-dimethyl-2-octene-*1*-^{3}H. Alternatively, tritiation of 4-ethylphenol with tritium water in sulfuric acid, reaction of the recovered phenol with geranyl bromide, and epoxidation yields 1-(4′-ethylphen-^{3}H-oxy)-6,7-epoxy-3,7-dimethyl-2-octene. The products have a high specific activity (33 to 654 mCi per mmol) and are useful in studies on the degradation and mode of action of this potent juvenile hormone mimic.

Interest in the potential use of juvenile hormones or related compounds eliciting a similar biological response (juvenoids) for insect control has led to the synthesis of several types of materials having a relatively simple structure compared to the natural product, but which also have equal or higher morphogenetic activity. There is a need for information on the biotransformations which occur with juvenoids of interest in various organisms and in the environment, and on the binding characteristics of these compounds at pertinent hormone receptor sites. These studies are greatly facilitated by the availability of radio-labeled preparations.

1-(4′-Ethylphenoxy)-6,7-epoxy-3,7-dimethyl-2-octene (compound I, Figure 1; code R-20458 of Stauffer Chemical Co., Mountain View, Calif.) combines a relatively simple structure with very high morphogenetic activity (Pallos *et al.*, 1971). It is desirable to have separate preparations of this