

guaiacyl structures being the main ones. The total yields of degradation products and the ratios of the structures in them have confirmed the greater degree of condensation of the boll DLA.

#### LITERATURE CITED

1. S. Mukhamedova, L. S. Smirnova, and Kh. A. Abduazimov, Khim. Prir. Soedin., 101 (1986).
2. S. Mukhamedova, L. S. Smirnova, and Kh. A. Abduazimov, Khim. Prir. Soedin., 498 (1987).
3. A. A. Geronikaki and Kh. A. Abduazimov, Khim. Prir. Soedin., 242 (1976).
4. N. A. Veksler, L. S. Smirnov, and Kh. A. Abduazimov, Khim. Prir. Soedin., 100 (1977).

#### IDENTIFICATION OF 3-ACETYL- AND 15-ACETYL-SUBSTITUTED DEOXYNIVALENOLS IN A CULTURE OF *Fusarium graminearum*

A. N. Leonov, G. P. Kononenko, and N. A. Soboleva

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Deoxynivalenol (3,7,15-trihydroxy-12,13-epoxytrichothec-9-en-8-one) and its acyl derivatives are known as biosynthetically linked metabolites of a number of isolates of *Fusarium graminearum* (Gibberellaceae) [1].

In the biomass obtained after the growth of the isolate F. sp. 579a VNIIZ of *F. graminearum* Schw. on grain (29°C, 40 days), together with deoxynivalenol, we have identified 3-acetoxy-7,15-dihydroxy-12,13-epoxytrichothec-9-en-8-one (I) and 15-acetoxy-3,7-dihydroxy-12,13-epoxytrichothec-9-en-8-one (II).

The biomass, in an amount of 3.8 kg (equivalent to a dry weight of 0.7 kg) was extracted with acetonitrile-water (5:1). The extract so obtained was passed through a column filled with layers of activated carbon and of neutral alumina, and the eluate was concentrated and was evaporated in vacuum at 50°C to an aqueous phase. Sodium chloride (100 g/liter) was added to this aqueous residue and it was extracted with three volumes of ethyl acetate. The ethyl acetate solution was evaporated in vacuum to a dry residue (65.7 g), and this was subjected to column LC on silica gel L (100/400) with a gradient of the phases benzene and benzene-acetone (95:5 and 9:1). The eluate from the mobile phase benzene-acetone (9:1) was evaporated in vacuum to a dry residue (8.2 g) and this was subjected to column LC on silica gel L (100/400) with a gradient of mobile phases consisting of hexane with successive 5% additions of ethyl acetate. The compositions of the fractions were monitored by TLC on Silufol with the mobile phase ethyl acetate-hexane (3:1), the substances being detected from their blue fluorescence in UV light (366 nm) after the plates had been treated with a 20% solution of aluminum chloride in ethanol and had been heated at 92°C for 10 min.

The dry residue (0.75 g) that had been eluted with hexane-ethyl acetate (55:45) and contained, according to TLC, a substance with  $R_f$  0.26 was subjected to reversed-phase HPLC on the sorbent LiChrosorb  $C_8$  (10  $\mu$ ) with the mobile phase acetonitrile-water (1:4). The eluate containing the main component of the fraction gave 0.21 g of substance (I) (yield 0.03% on the dry biomass).  $[\alpha]_D^{22} + 25.6^\circ$  (c 0.3; methanol). UV spectrum:  $\lambda_{max}^{CH_3OH}$  217 nm ( $\log \epsilon$  3.7330). IR spectrum:  $\nu_{max}^{KBr}$  3548 (OH); 1742 (-O-C=O); 1684 (C=C-C=O). Mol. wt. 338 (mass spectrometry). The PMR spectrum [ $\delta$  2.20 ppm (3 H,  $CH_3CO$ )] and the  $^{13}C$  NMR spectrum (in  $CDCl_3$ ) coincided with those described for 3-acetoxy-7,15-dihydroxy-12,13-epoxytrichothec-9-en-8-one [1, 2].

The dry residue (1.28 g) that had been eluted by hexane-ethyl acetate (45:55) and contained, according to TLC, a substance with  $R_f$  0.15 was subjected to HPLC under the conditions described for (I). The eluate containing the main component of the fraction gave 0.32 g of substance (II) (yield 0.05% of the dry biomass).  $[\alpha]_D^{22} + 22.2^\circ$  (c 0.3; methanol). UV

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spectrum:  $\lambda_{\max}^{\text{CH}_3\text{OH}}$  220 nm ( $\log \epsilon$  3.7594). IR spectrum:  $\nu_{\max}^{\text{KBr}}$  3450 (OH); 1744 (-O-C-O); 1684 (C=C-O); mol. wt. 338 (mass spectrometry). The PMR spectrum [1.95 ppm (3 H, CH<sub>3</sub>CO)] and the <sup>13</sup>C PMR spectrum (in CDCl<sub>3</sub>) coincided with those described for 15-acetoxy-3,7-dihydroxy-12,13-epoxytrichothec-9-en-8-one [2, 3].

Thus, the 3-acetate and the 15-acetate of deoxynivalenol have been identified in the biomass from isolate F. sp. 579a VNIIZ of *F. graminearum*. This is the first time that the simultaneous presence of two monoacetates in the culture of an isolate of this species has been reported.

#### LITERATURE CITED

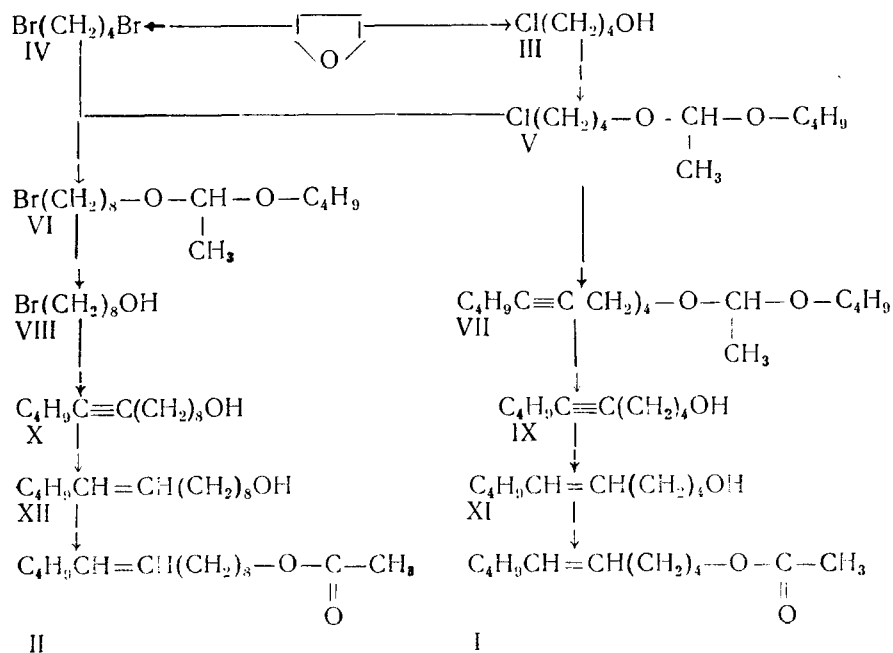
1. T. Toshizawa and N. Morooka, in: *Mycotoxins in Human and Animal Health*, J. V. Rodricks et al. (eds.), Pathotox Publishers, Park Forest South, Ill. (1977), p. 309.
2. B. A. Blackwell, R. Greenhalgh, and A. D. Bain, *J. Agric. Food Chem.*, **32**, 1078 (1984).
3. J. D. Miller, A. Taylor, and R. Greenhalgh, *Can. J. Microbiol.*, **29**, 1171 (1983).

#### SYNTHESIS OF DEC-5Z-ENYL ACETATE AND TETRADEC-9Z-ENYL ACETATE - COMPONENTS OF THE SEX PHEROMONE OF *Agrotis segetum*

G. G. Verba, L. M. Bikulova, and A. A. Abduvakhod

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Dec-5Z-enyl acetate (I) and tetradec-9Z-enyl acetate (II) are among the main components of the pheromone of the turnip moth *Agrotis segetum* [1, 2]. Syntheses of compound (I) have been described with the use of the Wittig reaction [3, 4], the two-stage oxyethylation of lithium hexynylide [5], and the selective ozonolysis of cyclododeca-1,6-diene [6]. Compound (II) has been obtained from methyl myristoleate [7], by an acetylenic route [8], and by the carbocupration reaction [9], and also by a method based on the selective ozonolysis of 1-methylcycloocta-1Z,5Z-diene [10].



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