This article was downloaded by: [George Washington University]
On: 28 December 2014, At: 17:51
Publisher: Taylor & Francis
Informa Ltd Registered in England and Wales Registered Number: 1072954
Registered office: Mortimer House, 37-41 Mortimer Street, London W1T 3JH, UK

Agricultural and Biological Chemistry
Publication details, including instructions for authors and subscription information:
http://www.tandfonline.com/loi/tbbb19

New Nematicidal Metabolites from a Fungus, Irpex lacteus

Masatoshi Hayashi[a], Kojiro Wada[a] & Katsura Munakata[a]
[a] Department of Agricultural Chemistry, Nagoya University, Chikusa-ku, Nagoya 464, Japan

Published online: 09 Sep 2014.

To cite this article: Masatoshi Hayashi, Kojiro Wada & Katsura Munakata (1981) New Nematicidal Metabolites from a Fungus, Irpex lacteus, Agricultural and Biological Chemistry, 45:6, 1527-1529

To link to this article: http://dx.doi.org/10.1080/00021369.1981.10864743

PLEASE SCROLL DOWN FOR ARTICLE

Taylor & Francis makes every effort to ensure the accuracy of all the information (the "Content") contained in the publications on our platform. However, Taylor & Francis, our agents, and our licensors make no representations or warranties whatsoever as to the accuracy, completeness, or suitability for any purpose of the Content. Any opinions and views expressed in this publication are the opinions and views of the authors, and are not the views of or endorsed by Taylor & Francis. The accuracy of the Content should not be relied upon and should be independently verified with primary sources of information. Taylor and Francis shall not be liable for any losses, actions, claims, proceedings, demands, costs, expenses, damages, and other liabilities whatsoever or howsoever caused arising directly or indirectly in connection with, in relation to or arising out of the use of the Content.

This article may be used for research, teaching, and private study purposes. Any substantial or systematic reproduction, redistribution, reselling, loan, sub-licensing, systematic supply, or distribution in any form to anyone is expressly forbidden. Terms & Conditions of access and use can be found at http://www.tandfonline.com/page/terms-and-conditions

Short Communication

New Nematicidal Metabolites from a Fungus, *Irpex lacteus*

Masatoshi Hayashi, Kojiro Wada
and Katsura Munakata

Department of Agricultural Chemistry,
Nagoya University, Chikusa-ku,
Nagoya 464, Japan

Received February 13, 1981

Many higher plants and fungal species have been found to posses characteristics which were detrimental in populations of specific nematodes.[1~3] Recently, the authors[5~7] and other groups[8,9] isolated nematicidal components from several higher plants, and most of the isolated nematicidal substances were polyacetylenic compounds and diterpenoids. On the other hand, only a few studies on the isolation of the nematicidal substances from microorganisms were reported.[4a,b,c] Our attention was focused to the isolation of the new types of nematicidal substances from fungal metabolites.

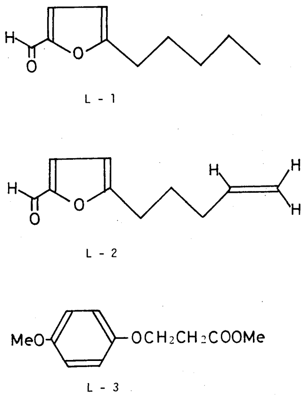


FIG. 1.

In this paper, the authors report the isolation of three new nematicidal substances, L-1, L-2 and L-3 from a culture filtrate of *Irpex lacteus* (IFO 5367) and their structure elucidation. These compounds were isolated for the first time as fungal metabolites.

Irpex lacteus was cultivated at 30°C for a week in Sakaguchi flasks containing each 100 ml of a malt-dextrose medium, which was composed of glucose (2%), peptone (0.1%), malt extract (2%) and tap water. The isolation of the nematicidal substances was guided by immersion test against *Aphelencoides besseyi*.[6]

The nematicidal substances were extracted from the culture filtrates (*ca.* 20 liters) of *Irpex lacteus* with two portions of ethyl acetate (10 liters) at pH 3. The oily extract (2.6 g) was separated into an acidic fraction and a neutral fraction. The neutral fraction which showed a strong nematicidal activity was then subjected to a column chromatography on silica gel (100 g AR 100 mesh, Mallinkrodt) and eluted with benzene and followed by ethyl acetate. The nematicidal activity was found in the benzene fraction. The benzene fraction was separated by a silicagel column chromatography (*n*-hexane–ethyl acetate, 98 : 2), and further purification of the active fractions on preparative TLC gave the nematicidal substances, L-1 (180 mg), L-2 (12 mg) and L-3 (1.4 mg).

Nematicidal activities of the components, L-1, L-2 and L-3 against *Aphelencoides besseyi* were shown in Fig. 2. The compound (L-3) showed 50% mortality of nematodes in the solution of 25 ppm, and L-1 and L-2 showed the similar activity in the solution of 50 ppm.

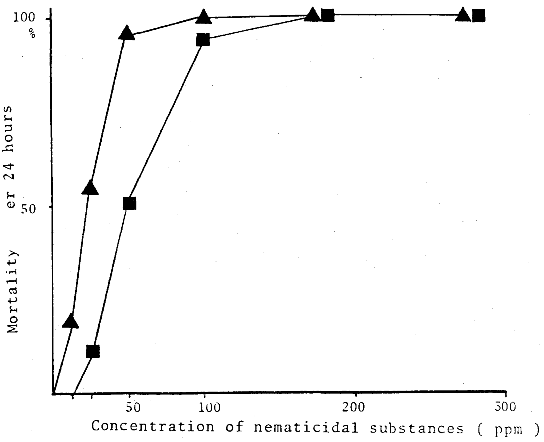
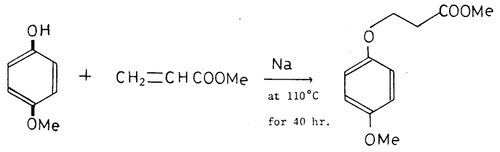


FIG. 2. Nematicidal Activities of L-1, L-2 and L-3.
■, L-1 and L-2; ▲, L-3.

On its mass spectrum, the compound (L-1) showed a molecular ion at m/z 166. Its UV spectrum, [$\lambda_{\max}^{\mathrm{EtOH}}$ (nm); 285 (ε=16,000), 225 (ε=2,500)] and IR spectrum [$\nu_{\max}^{\mathrm{CCl_4}}$ (cm^{-1}); 1680] indicated the presence of a furfural chromophore.[10] The PMR spectrum in $CDCl_3$ exhibited the presence of a methyl group (δ 0.90, 3H, t J=6.6 Hz), four methylene groups [δ 1.2~1.4, (4H, m); δ 1.65 (2H, m); δ 2.72 (2H, t, J=6.8 Hz)], two aromatic protons [δ 6.24 (1H, dt, J=3.6, 0.7, 0.7 Hz), δ 7.19 (1H, d, J=3.6 Hz)] and an aldehyde group [δ 9.51 (1H, s)]. The chemical shifts and coupling constants of the aromatic protons indicated the presence of a 5-substituted furfural structure in the molecule. Its ^{13}C NMR spectrum in $CDCl_3$ showed ten peaks and exhibited the presence of four methylene carbons (δ 22.3, 27.2, 28.2 and 31.3), a methyl carbon (δ 13.9, q), two unsubstituted aromatic carbons (δ 108.6, d; 123.6, d), two substituted aromatic carbons (δ 151.7, s; 161.1, s) and an aldehyde group (δ 176.8, d). Furthermore, the appearance of fragment peaks [m/z 137 ($M-C_2H_5$); 123 ($M-C_3H_7$); 109 ($M-C_4H_9$)] on the mass spectrum indicated the presence of a n-pentyl group in the molecule. Consequently, the compound (L-1) was identified to 5-pentyl-2-furaldehyde.

The second nematicidal metabolite (L-2) showed a molecular ion at m/z 164 and its UV spectrum [$\lambda_{\max}^{\mathrm{EtOH}}$ (nm); 283 (ε=16,000) 225 (ε=3,600)] indicated the presence of the same chromophore as L-1. The IR spectrum ($CHCl_3$) showed the presence of carbonyl (1680 cm^{-1}) and double bonds (1640, 999, 920 cm^{-1}). The PMR spectrum ($CDCl_3$) of L-2 exhibited the presence of a vinyl group [δ 5.76 (1H, ddt, J=17.6, 9.8, 6.6 and 6.6 Hz); 5.02 (1H, d with fine splittings, J=17.6 Hz); 5.00 (1H, d with fine splittings, J=9.8 Hz)], an aldehyde group [δ 9.52, (1H, s)], three methylene groups [δ 2.74, (2H, t, J=7.3 Hz); 2.08, (2H, q, J=6.6 Hz); 1.80 (2H, m)], and two aromatic protons [δ 6.24 (1H, d, J=3.4 Hz); 7.17 (1H, d, J=3.4 Hz)]. The PMR decoupling experiments of L-2 showed the presence of a 4-pentenyl group. The PMR and UV spectra of L-2 was very similar to those of L-1 except the presence of a vinyl group. Fragments [m/z 123 ($M-C_3H_5$); 109 ($M-C_4H_7$)] on the MS spectrum confirmed the presence of a 4-pentenyl group. Accordingly, the compound (L-2) was confirmed as 5-(4-pentenyl)-2-furaldehyde.

The third metabolite (L-3) on the mass spectrum showed a molecular ion peak at m/z 210. Its UV spectrum, [$\lambda_{\max}^{\mathrm{EtOH}}$ (nm); 287 (ε=2,300), 230 (ε=4,000)], indicated the presence of an O,O'-dialkylated hydroquinone chromophore.[10] A PMR spectrum ($CDCl_3$) of L-3 exhibited the presence of two methyl groups [δ 3.76, (3H, s); 3.72, (3H, s)], four aromatic protons [δ 6.83 (4H, s)] and two methylene groups [δ 4.20, (2H, t, J=6.3 Hz); 2.71, (2H, t, J=6.3 Hz)]. Its IR spectrum in $CHCl_3$ showed absorption at 1745 (ester carbonyl group), 1510 (aromatic group) and 1235 (carbon-oxygen bond) cm^{-1}. The above data supported methyl 3-p-anisoloxypropionate as the appropriate structure of L-3. Finally, the third metabolite was identified with methyl 3-p-anisoloxypropionate by its synthesis as shown in Scheme 1.[11] The synthesized compound (methyl 3-p-anisoloxypropionate) showed the same nematicidal activity as that of the natural metabolite (L-3).



SCHEME 1.

REFERENCES

1) (a) J. H. Uhlenbroek and J. D. Bijloo, *Recl. Trav. Chim. Pays-Bas Berg.*, **77**, 1004 (1958); (b) J. H. Uhlenbroek and J. D. Bijloo, *Recl. Trav. Chim. Pays-Bas Berg.*, **78**, 382 (1959).

2) Y. Ueno and K. Iatomi, *Sci. Rep. Fac. Agr., Meijo Univ.*, **14**, 7 (1978).

3) N. A. Mektieva, A. A. Radzhavova and S. G. Gasanova, *Mikologia i Fitopatologia*, **11**, 385 (1977).

4) (a) H. Iizuka, K. Komagata, T. Kawamura, Y. Kunii and M. Shibuya, *Agric. Biol. Chem.*, **26**, 199 (1962); (b) R. Mori, *J. Antibiotics, Ser. A*, **14**, 280 (1961); (c)

M. Takashima, H. Sakai and K. Arima, *Agric. Biol. Chem.*, **26**, 660 (1962).

5) S. Kogiso, K. Wada and K. Munakata, *Tetrahedron Lett.*, **1976**, 109.
6) S. Kogiso, K. Wada and K. Munakata, *Agric. Biol. Chem.*, **40**, 2085 (1976).
7) S. Kogiso, K. Wada and K. Munakata, *Agric. Biol. Chem.*, **40**, 2119 (1976).
8) K. Kawazu, Y. Nishii and S. Nakajima, *Agric. Biol. Chem.*, **44**, 903 (1980).
9) F. J. Gommers, *Phytochemistry*, **10**, 1945 (1971).
10) H. E. Ungnade, ed., "Organic Electronic Spectral Data," Vol. II, Interscience Publishers, New York, 1957, p. 27 and 146.
11) R. H. Hall and E. S. Stern, *J. Chem. Soc.*, **1949**, 2045.