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COMMISSION ON FOOD CHEMISTRY*

PHYSICOCHEMICAL DATA FOR SOME SELECTED MYCOTOXINS

Prepared for publication by

A. E. POHLAND[1], P. L. SCHULLER[2] and P. S. STEYN[3]

[1]Food and Drug Administration, Washington D.C., USA
[2]National Institute of Public Health, Bilthoven, the Netherlands
[3]National Chemical Research Laboratory, Pretoria, RSA

in conjunction with

H. P. VAN EGMOND

National Institute of Public Health, Bilthoven, the Netherlands

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†Deceased.

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A.E.POHLAND
Food and Drug Administration, Washington D.C., USA

P.L.SCHULLER
National Institute of Public Health, Bilthoven, the Netherlands

P.S.STEYN
National Chemical Research Laboratory, Pretoria, RSA

Evaluator and Compiler

H.P.VAN EGMOND
National Institute of Public Health, Bilthoven, the Netherlands

CONTENTS

I. ABSTRACT

Physico-chemical data i.e. melting point, specific rotation, circular dichroism, ultraviolet absorption spectrum, infrared absorption spectrum, electron impact mass spectrum and nuclear magnetic resonance spectrum have been determined of the following 15 mycotoxins: aflatoxin B_1, austdiol, citrinin, cytochalasin B, α-cyclopiazonic acid, diacetoxyscirpenol, fumitremorgen B, ochratoxin A, patulin, penicillic acid, roridin A, secalonic acid D, sterigmatocystin, T-2 toxin and zearalenone.

II. INTRODUCTION

Mycotoxicoses, the diseases caused by the ingestion of mycotoxins in man and animals, have been known for centuries. Japanese researchers found in the 1940's that *Penicillium islandicum* isolated from yellow rice produced a number of toxins. These toxins were found to be hepatocarcinogenic to mice and this led to the hypothesis that mould-produced toxins might cause chronic diseases in man. The present interest in mycotoxins arose from the discovery of the aflatoxin problem in Britain in 1960. The aflatoxins are hepatotoxic and hepatocarcinogenic metabolites which are produced by *Aspergillus flavus* and *Aspergillus parasiticus*, common contaminants of groundnuts, pistachio nuts, maize etc. This led to the present international recognition of the real and potential danger of mycotoxin contamination of foodstuffs and feedstuffs.

In recent years an increasing number of chemical analyses have been carried out for the presence of mycotoxins in several commodities. The levels of some of the toxins have been controlled legally as toxic substances. The main purpose of the analyses was to establish the actual state of contamination. Unfortunately the reliability of the data provided by many laboratories is open to criticism, since there was a lack not only of collaboratively studied methods of analysis but also of appropriate standard reference materials. The Food Chemistry Commission of IUPAC recognized both items as problems of international relevance. In cooperation with the Association of Official Analytical Chemists (USA) the Commission undertook the development of reference methods. The reliability of results is a function of precision (repeatability and reproducibility) and of accuracy (which refers to the true value). The accuracy of results depends in the first place on the correctness of (published) physicochemical constants of the compound(s) to be determined.

A survey of the scientific literature indicated that ambiguous physical constants have been reported for some of the important mycotoxins and directed the Commission's interest to the problem. As early as 1971 the IUPAC published Information Bulletin Technical Report number 1 entitled "Collaborative Study of a Method for Determination of Concentration and Purity of Aflatoxin Standards and Use of the Method for Measuring Stability of the Standards". At the IUPAC meeting in 1975 (Madrid) the Commission recognized its responsibility in this regard and decided to collect the physical data for 15 selected mycotoxins of analytical purity. The mycotoxins included are: aflatoxin B_1, austdiol, citrinin, α-cyclopiazonic acid, cytochalasin B, diacetoxyscirpenol, fumitremorgen B, ochratoxin A, patulin, penicillic acid, roridin A, secalonic acid D, sterigmatocystin, T-2 toxin and zearalenone. These compounds are representative of important types of mycotoxins. Dr.P.S.Steyn, National Chemical Research Laboratory, Pretoria, Republic of South Africa and other members of the Commission supplied the toxins.

The document provides the following data: melting point, specific rotation, circular dichroism, ultraviolet absorption, infrared absorption, 100 Mz and 250 Mz ^{1}H nuclear magnetic resonance spectra and mass spectra. These data were measured and recorded in a co-operative study involving the Food and Drug Administration, Washington D.C., USA, the National Institute of Public Health, Bilthoven, the Netherlands and the National Chemical Research Laboratory, Pretoria, Republic of South Africa.
Final editing of the document was done at the National Institute of Public Health, the Netherlands.

P.L.Schuller

III. TECHNIQUES AND EQUIPMENT USED TO CHARACTERIZE THE MYCOTOXINS

As a standard procedure before measuring any physico-chemical data, all samples were dried for 1 hour at 60°C with the exception of aflatoxin B_1 which was dried for 16 hours under high vacuum at 120°C.

III.1 Melting Point [1)]

Melting points were determined with an Olympus melting point microscope, fitted with a Mettler heating stage type FP2 and a matching temperature programmer, able to provide rates of 0.2, 2 an 10°C/minute.

Test samples of about 0.1 mg were placed on the microscope slide by means of a micro spatula. The samples were covered with a coverslip, lined along the edges with just sufficient high-melting vacuum grease, to stick the coverslip to the slide and to keep the test sample in position.

III.2 Specific Rotation

The specific rotation at 21°C was measured with a Perkin Elmer model 241 polarimeter. All mycotoxins were dissolved in chloroform, with the exception of austdiol, which was dissolved in pyridine. Concentrations are given in the data-sheets.

III.3 Circular Dichroism

The differential dichroic absorption was measured with a Jasco Model J-20 instrument. A standard solution of D-10-camphor sulphonic acid in water was used for the calibration of the CD scale.

All mycotoxins were dissolved in spectroscopic grade methanol at concentrations of ca. 1 mg/ml. All solutions were diluted with methanol to the concentrations given in the data-sheets.

III.4 Ultraviolet Absorption Spectra

The spectra were recorded with a Cary model 14 double beam, double monochromator recording spectrophotometer. The spectral band width, determined by the absorbance of methanol in the reference cell, was a few nm for wave-lengths smaller than 220 nm and less than 0.1 nm in the remaining wave-length region. The wave-length scale was calibrated and found to be accurate within 0.2 nm. The absorbance scale was calibrated with the help of potassium-dichromate solutions. The noise of the system was 3.10^{-3} absorbance units within the wave-length interval 200-300 nm and 1.10^{-3} within the interval 300-400 nm. Accurately weighed quantities of ca. 1 mg of each mycotoxin were dissolved in 10 ml methanol. All solutions were diluted with methanol until the absorbance reached the appropriate region for U.V.-measurement (absorbance between 0.2 and 0.8). Concentrations are given in spectra 1a - 15a.

[1)]From the results obtained in this co-operative study it was obvious that the procedure used to determine melting points was not well normalized. The differences in results were of such a degree that, taking into account the (very) good agreement of the other physico-chemical data, one had to conclude, that the determination of melting point depends mainly on the interpretation of the observer.

III.5 Infrared Absorption Spectra

The spectra were recorded with a Perkin-Elmer model 180 Infrared Spectrometer. The resolution was 2 cm^{-1}, the scan ranged from 4000 to 250 cm^{-1}.

Each mycotoxin was thoroughly ground with KBr at a mass fraction of 1.25 mg/g KBr. The mixtures were pressed to 13 mm KBr discs.

III.6 Electron Impact Mass Spectra

The mass spectra were taken on a Varian Mat CH5 mass spectrometer using the following parameters:

1. electron impact ionization
2. electron energy 70eV
3. temperature ion source 200°C
4. resolution M/ΔM = 800, 10% valley definition
5. scan speed 2.5 sec/decade
6. scan range m/z 20 - m/z 700
7. The data were recorded on a Varian MAT SS200 computer
8. The samples were introduced into the ion source by means of the direct inlet probe. The samples themselves were evaporated from gold crucibles inserted in the direct inlet, which was temperature programmed. During the temperature programme mass spectra were taken sequentially, and stored in the computer. The temperatures used for volatilisation of the different mycotoxins are listed below:

Mycotoxins	Direct inlet temp. °C	Mycotoxins	Direct inlet temp. °C
Aflatoxin B_1	150	Patulin	40
Austdiol	75	Penicillic acid	25
Citrinin	75	Roridin A	140
α-Cyclopiazonic acid	85	Secalonic acid D	195
Cytochalasin B	150	Sterigmatocystin	120
Diacetoxyscirpenol	50	T-2 toxin	70
Fumitremorgen B	160	Zearalenone	110
Ochratoxin A	110		

III.7 Nuclear Magnetic Resonance Spectra

100 MHz ^1H NMR spectra were recorded with a Varian XL-100 FT NMR spectrometer. The 90 MHz ^1H NMR spectrum for zearalenone was recorded with a Varian EM-390 NMR spectrometer. 250 MHz ^1H NMR spectra were recorded with a Bruker WM-250 FT NMR spectrometer.

All mycotoxins were dissolved in $CDCl_3$ of 99.5% isotopic purity, with the exception of austdiol, which was dissolved in d_5-pyridine of 99% isotopic purity at concentrations indicated in spectra 1d - 15d and 1e - 15e. TMS (ca. 0.02%) was used as internal standard. Temperatures were 305°K for recording the 100 MHz and 90 MHz spectra and 300°K for the 250 MHz spectra. The samples were not evacuated.

IV. PHYSICO-CHEMICAL DATA

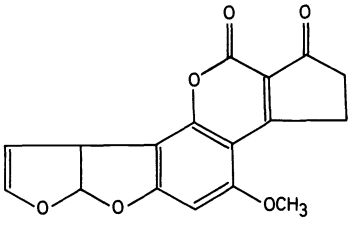
IV.1 AFLATOXIN B_1

I SYNONYMS: none

II CHEMICAL NAME: 2,3,6a,9a-tetrahydro-4-methoxy-,(6a R-cis)-Cyclopenta[c]furo[3',2':4,5]furo[2,3-h][1]benzopyran-1,11-dione

III EMPIRICAL FORMULA: $C_{17}H_{12}O_6$

IV STRUCTURAL FORMULA:



V MOLECULAR WEIGHT: 312.3

VI DESCRIPTION: Aflatoxin is a white, odourless, crystalline solid

VII CHARACTERIZATION DATA:

1.Melting range: 269-271°C, after drying for 16 hours at 120°C in vacuo.

2.Specific rotation: $[\alpha]_D^{21} = -559^o$
conc. 625 µmol/l
solvent: chloroform

3.Circular dichroism:
$\Delta\varepsilon_{(\lambda 400)}$ 0, $\Delta\varepsilon_{(\lambda 354)}$ -5.48, $\Delta\varepsilon_{(\lambda 280)}$ 0
$\Delta\varepsilon_{(\lambda 264)}$ - 2.0, $\Delta\varepsilon_{(\lambda 244)}$ 0, $\Delta\varepsilon_{(\lambda 219)}$ - 19.38, $\Delta\varepsilon_{(\lambda 209)}$ 0
conc. 375 µmol/l
solvent: methanol
temperature: 23°C

4.Ultraviolet absorption spectrum:
see spectrum 1a

Molar absorption coefficients:
$\varepsilon_{(\lambda 213)} = 1816 \pm 2$
$\varepsilon_{(\lambda 224)} = 2200 \pm 5$
$\varepsilon_{(\lambda 250)} = 793 \pm 3$
$\varepsilon_{(\lambda 265)} = 1257 \pm 2$
$\varepsilon_{(\lambda 284)} = 124 \pm 2$
$\varepsilon_{(\lambda 360)} = 2195 \pm 8$
conc. 30.27 µmol/l
solvent: methanol

5.Infrared absorption spectrum:
see spectrum 1b

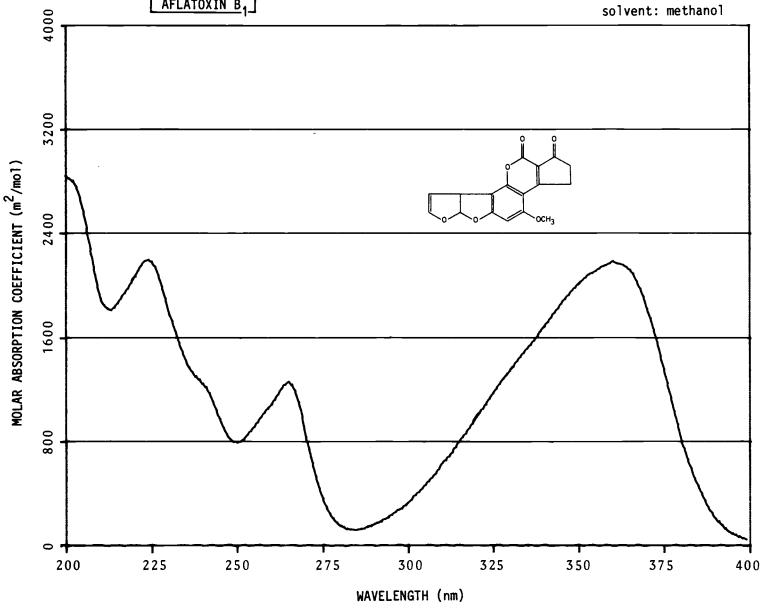
6.Electron impact mass spectrum:
see spectrum 1c

7.Nuclear Magnetic Resonance spectrum:
see spectra 1d and 1e

ULTRAVIOLET ABSORPTION SPECTRUM 1a

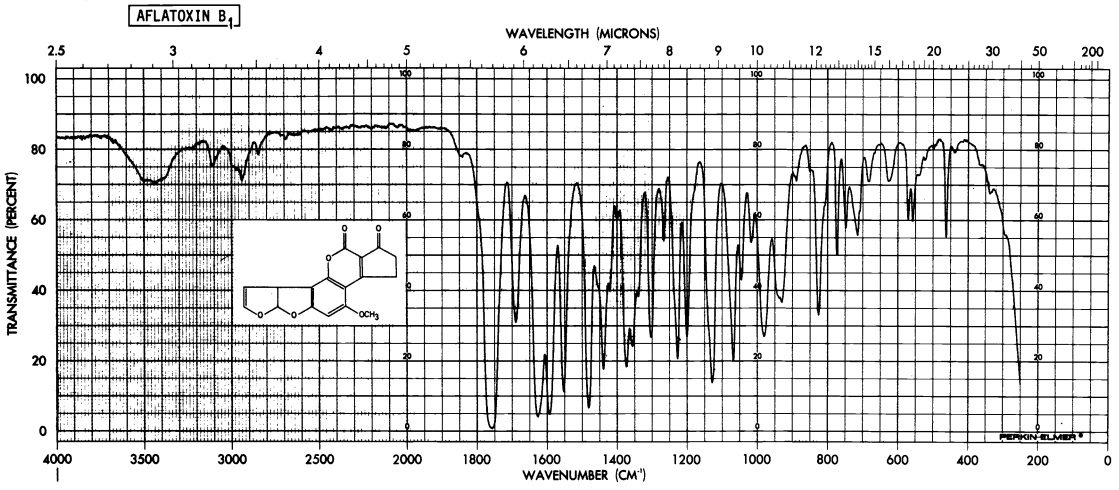
AFLATOXIN B_1

conc. 30.27 µmol/l
solvent: methanol



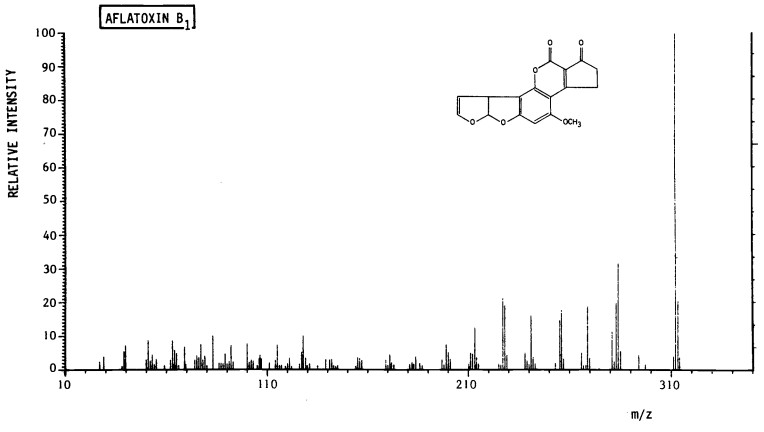
INFRARED ABSORPTION SPECTRUM 1b

AFLATOXIN B_1



MASS SPECTRUM 1c

AFLATOXIN B_1



MASS SPECTRUM 1c

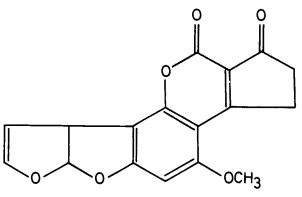
AFLATOXIN B_1

relative intensity	mass	relative intensity	mass	relative intensity	mass
2.57%	27.0	3.35%	107.0	4.71%	212.1
3.90%	29.1	2.05%	111.0	12.60%	213.2
1.14%	38.1	2.82%	114.1	3.77%	214.1
5.67%	39.1	7.41%	115.0	1.90%	215.1
7.31%	39.9	1.39%	116.1	1.82%	225.1
3.12%	50.1	1.40%	117.0	1.78%	226.1
8.83%	51.1	1.03%	119.0	21.30%	227.1
2.73%	52.0	1.85%	120.0	19.15%	228.1
4.55%	53.0	3.66%	121.0	4.49%	229.1
1.61%	54.0	1.02%	122.1	5.00%	238.1
3.25%	55.1	1.78%	126.1	2.79%	239.1
1.34%	59.1	5.28%	127.0	1.74%	240.1
3.06%	62.1	10.00%	127.9	16.08%	241.1
8.71%	63.1	3.58%	129.0	3.83%	242.0
1.68%	63.6	1.35%	129.9	1.80%	243.1
5.83%	64.1	1.83%	131.0	2.15%	253.1
1.11%	64.6	1.28%	135.0	14.81%	255.3
4.96%	65.0	3.10%	139.1	17.74%	256.1
1.40%	66.0	3.03%	141.1	3.30%	257.1
2.32%	69.0	3.10%	142.1	5.01%	265.9
6.82%	69.1	1.22%	143.1	1.47%	266.9
1.76%	69.5	1.08%	144.1	1.45%	268.0
3.09%	74.1	1.31%	145.1	18.87%	268.9
4.23%	75.1	1.15%	154.1	3.50%	269.9
3.73%	76.1	3.63%	155.1	11.31%	280.9
7.51%	77.1	3.41%	156.1	2.53%	281.9
3.06%	78.1	3.09%	157.1	19.88%	282.9
4.12%	79.1	2.93%	169.0	31.72%	283.9
1.35%	80.1	1.34%	170.0	5.51%	285.0
10.26%	83.1	4.68%	171.1	4.33%	294.1
2.04%	86.1	2.11%	171.9	1.68%	297.3
2.05%	87.0	1.55%	173.0	4.09%	311.1
1.94%	88.1	1.78%	180.9	100.00%	312.0
4.77%	89.0	2.33%	182.1	20.51%	313.2
1.83%	90.0	1.84%	182.9	3.80%	314.1
2.52%	91.1	4.14%	183.9		
7.22%	92.1	2.03%	185.9		
2.39%	93.1	1.34%	187.0		
7.79%	100.0	3.02%	196.9		
2.32%	101.0	1.63%	197.9		
2.84%	102.1	7.56%	199.1		
2.76%	103.0	5.29%	200.0		
1.34%	105.1	3.39%	201.1		
3.49%	106.0	1.99%	210.2		
4.31%	106.4	5.04%	211.0		

Most abundant peaks

m/z	312.0	283.9	227.1	313.2	282.9
Intensity	100.00	31.72	21.30	20.51	19.88
m/z	228.1	268.9	256.1	241.1	255.3
Intensity	19.15	18.87	17.74	16.08	14.81

Formula



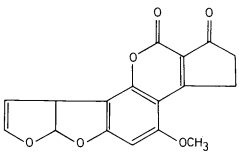
Experimental conditions: see p 2223

100 MHz ^{1}H NMR SPECTRUM 1d

AFLATOXIN B_1

conc. 40.03 mmol/l

solvent: $CDCl_3$

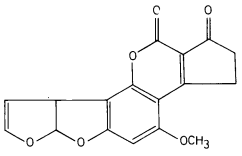


250 MHz ^{1}H NMR SPECTRUM 1e

AFLATOXIN B_1

conc. 40.03 mmol/l

solvent: $CDCl_3$



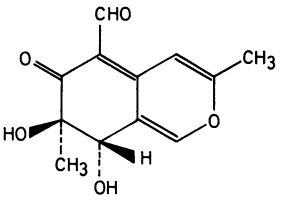
IV.2. AUSTDIOL

I SYNONYMS: none

II CHEMICAL NAME: 7,8-dihydro-7,8-dihydroxy-3,7-dimethyl-6-oxo-,(7R-trans)-6H-2-Benzopyran-5-carboxyaldehyde

III EMPIRICAL FORMULA: $C_{12}H_{12}O_5$

IV STRUCTURAL FORMULA:



V MOLECULAR WEIGHT: 236.2

VI DESCRIPTION: Austdiol is a yellow, odourless, crystalline solid

VII CHARACTERIZATION DATA:

1. Melting range: 245-249°C, after drying for 1 hour at 60°C

2. Specific rotation: $[\alpha]_D^{21} = +156.6^o$
conc. 4234 µmol/l
solvent: pyridine

3. Circular dichroism:

$\Delta\varepsilon_{(\lambda 430)}$ 0, $\Delta\varepsilon_{(\lambda 405)}$ -1.71, $\Delta\varepsilon_{(\lambda 393)}$ 0, $\Delta\varepsilon_{(\lambda 360)}$ +4.29, $\Delta\varepsilon_{(\lambda 330)}$ +0.71,

$\Delta\varepsilon_{(\lambda 312)}$ +2.15, $\Delta\varepsilon_{(\lambda 297)}$ 0, $\Delta\varepsilon_{(\lambda 276)}$ -6.15, $\Delta\varepsilon_{(\lambda 255)}$ 0, $\Delta\varepsilon_{(\lambda 237)}$ +8.01, $\Delta\varepsilon_{(\lambda 215)}$ 0.

conc. 529.2 µmol/l
solvent: methanol
temperature: 22°C
cell length: 2 mm

4. Ultraviolet absorption spectrum:
see spectrum 2a

Molar absorption coefficients:

$\varepsilon_{(\lambda 224)} = 276 \pm 12$
$\varepsilon_{(\lambda 256)} = 1440 \pm 4$
$\varepsilon_{(\lambda 305)} = 81 \pm 6$
$\varepsilon_{(\lambda 381)} = 2428 \pm 11$

conc. 32.50 µmol/l
solvent: methanol

5. Infrared absorption spectrum:
see spectrum 2b

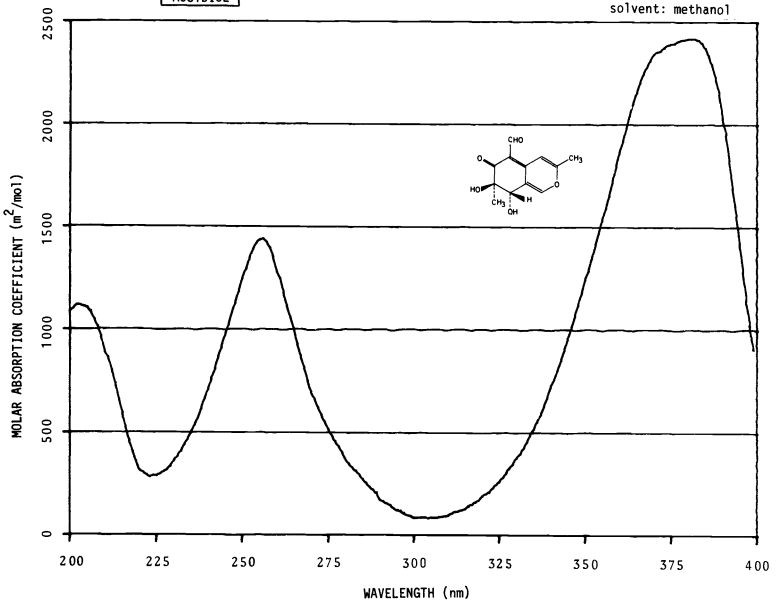
6. Electron impact mass spectrum:
see spectrum 2c

7. Nuclear Magnetic Resonance spectrum:
see spectra 2d and 2e

ULTRAVIOLET ABSORPTION SPECTRUM 2a

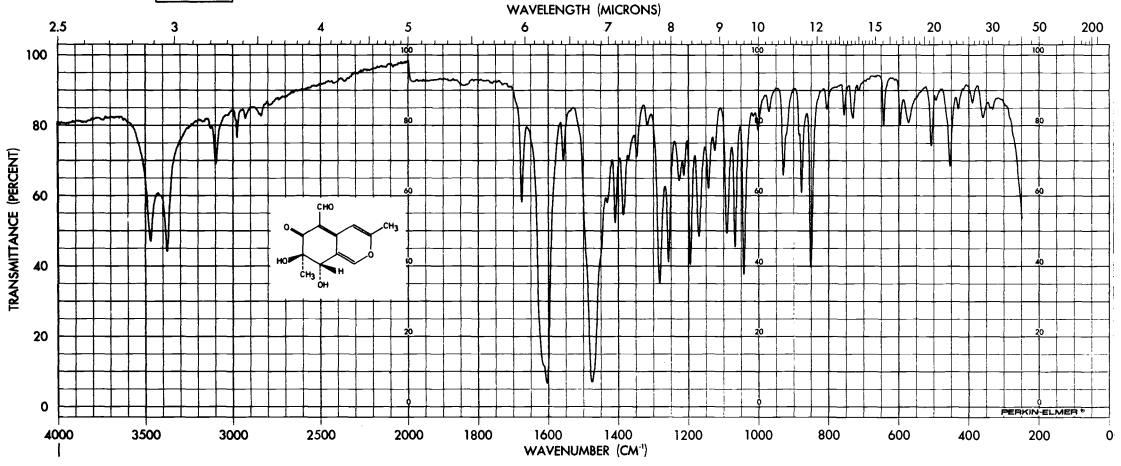
AUSTDIOL

conc. 32.50 µmol/l
solvent: methanol



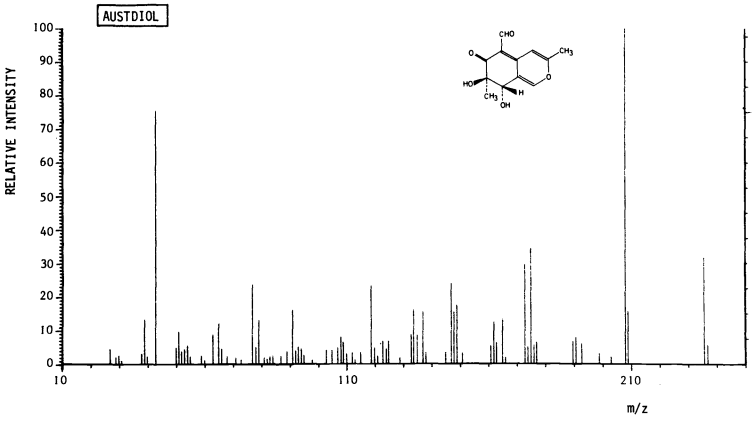
INFRARED ABSORPTION SPECTRUM 2b

AUSTDIOL



MASS SPECTRUM 2c

AUSTDIOL



MASS SPECTRUM 2c

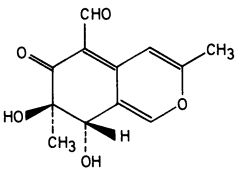
AUSTDIOL

relative intensity	mass	relative intensity	mass
4.40%	27.0	3.32%	115.1
2.08%	29.0	23.26%	118.9
2.52%	30.0	4.65%	120.0
1.00%	31.0	2.20%	121.1
3.02%	38.0	6.63%	123.0
13.21%	39.1	4.22%	124.1
2.22%	40.0	6.70%	125.0
75.44%	43.0	1.74%	129.0
4.84%	50.1	8.69%	133.0
9.54%	51.1	16.29%	133.9
3.62%	52.0	8.44%	135.1
4.22%	53.1	15.48%	137.1
5.34%	54.1	3.39%	138.1
2.15%	55.1	3.32%	145.1
2.45%	59.0	23.93%	147.1
1.00%	60.1	15.44%	148.0
8.62%	63.1	17.34%	149.1
11.93%	65.1	3.00%	151.0
4.40%	66.1	5.27%	161.0
2.20%	68.0	12.39%	162.0
1.85%	71.1	6.14%	163.0
1.28%	73.0	13.40%	165.1
23.51%	77.0	1.72%	166.1
5.09%	78.1	29.83%	173.0
12.94%	79.2	4.95%	174.0
2.01%	81.1	34.32%	175.1
1.37%	82.1	5.94%	176.1
2.15%	83.1	6.26%	177.1
2.38%	84.1	6.56%	189.9
2.45%	87.0	7.66%	190.9
3.74%	89.1	5.94%	192.9
15.92%	91.1	2.96%	199.0
3.87%	92.1	2.04%	203.1
4.97%	93.1	100.00%	208.1
4.42%	94.1	15.55%	209.1
2.63%	95.1	31.75%	235.9
1.00%	98.0	5.57%	237.0
4.06%	103.0		
3.99%	105.0		
4.86%	107.0		
7.91%	108.1		
6.37%	109.0		
2.93%	110.1		
3.25%	112.1		
1.12%	113.1		

Most abundant peaks

m/z	208.1	43.0	175.1	235.9	173.0
Intensity	100.00	75.44	34.32	31.75	29.83
m/z	147.1	77.0	118.9	149.1	91.1
Intensity	23.93	23.51	23.26	17.34	15.92

Formula



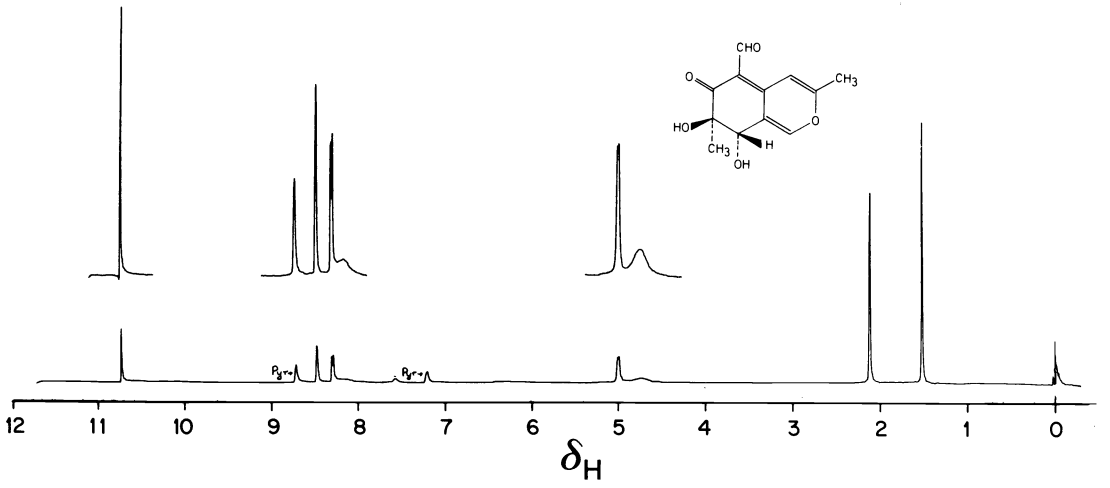
Experimental conditions: see p 2223

100 MHz ^{1}H NMR SPECTRUM 2d

AUSTDIOL

conc. 52.92 mmol/l

solvent: d_5-Pyridine

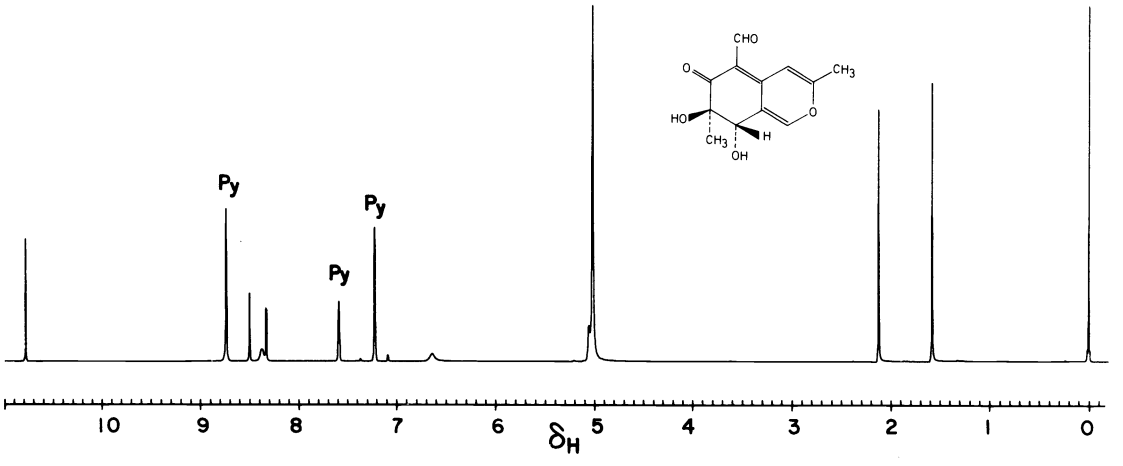


250 MHz ^{1}H NMR SPECTRUM 2e

AUSTDIOL

conc. 52.92 mmol/l

solvent: d_5-Pyridine



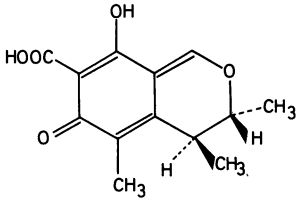
IV,3.CITRININ

I SYNONYMS: antimycin

II CHEMICAL NAME: 4,6-dihydro-8-hydroxy-3,4,5-trimethyl-6-oxo-,(3R trans)-3H-2-Benzopyran-7-carboxylic acid

III EMPIRICAL FORMULA: $C_{13}H_{14}O_5$

IV STRUCTURAL FORMULA:



V MOLECULAR WEIGHT: 250.2

VI DESCRIPTION: Citrinin is a yellow, odourless, crystalline solid

VII CHARACTERIZATION DATA:

1.Melting range: 170-173°C, after drying for 1 hour at 60°C

2.Specific rotation: $[\alpha]_D^{21} = -17.4^\circ$
conc. 3997 µmol/l
solvent: chloroform

3.Circular dichroism: No Cotton effects

4.Ultraviolet absorption spectrum:
see spectrum 3a

Molar absorption coefficients:

$\varepsilon_{(\lambda 225)} = 1580 \pm 10$
$\varepsilon_{(\lambda 250)} = 918 \pm 23$
$\varepsilon_{(\lambda 286)} = 175 \pm 17$
$\varepsilon_{(\lambda 330)} = 907 \pm 13$

conc. 60.07 µmol/l
solvent: methanol

5.Infrared absorption spectrum:
see spectrum 3b

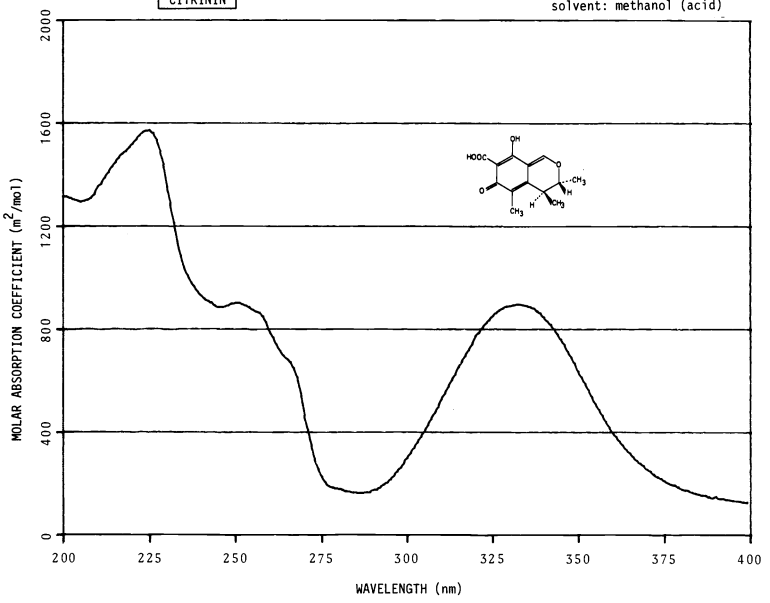
6.Electron impact mass spectrum:
see spectrum 3c

7.Nuclear Magnetic Resonance spectrum:
see spectra 3d and 3e

ULTRAVIOLET ABSORPTION SPECTRUM 3a

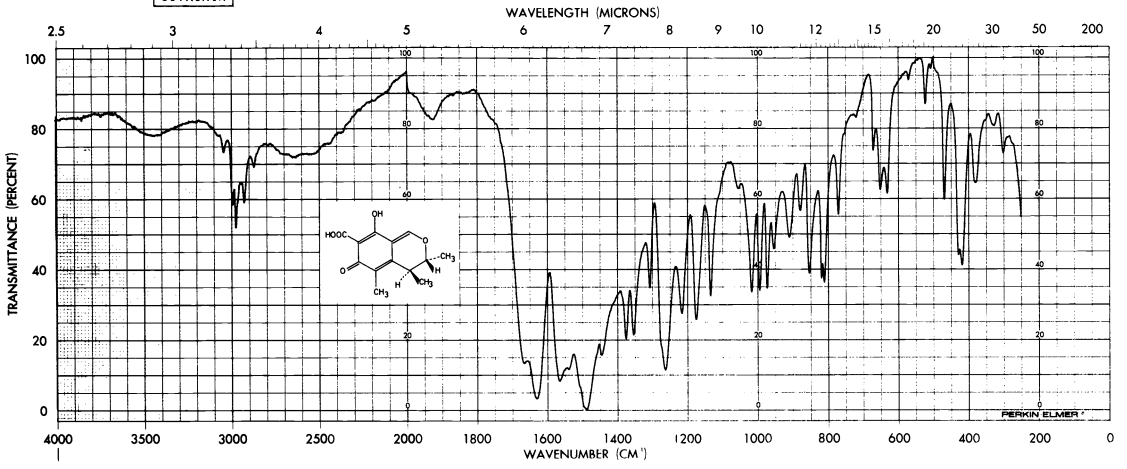
CITRININ

conc. 60.07 µmol/l
solvent: methanol (acid)



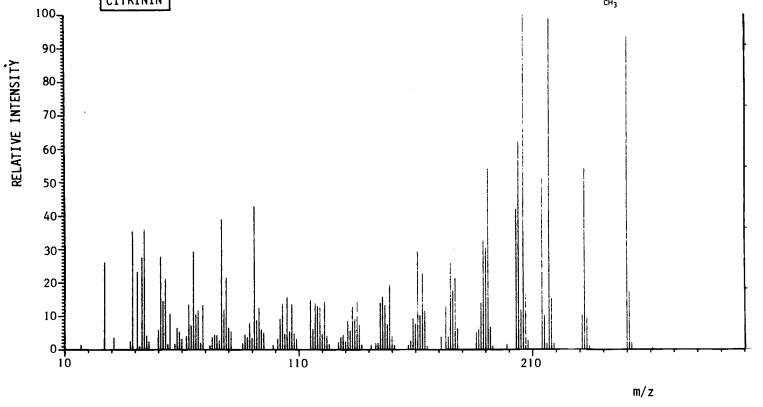
INFRARED ABSORPTION SPECTRUM 3b

CITRININ



MASS SPECTRUM 3c

CITRININ



MASS SPECTRUM 3c

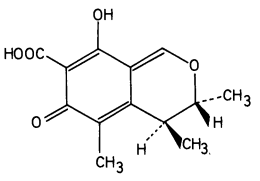
CITRININ

relative intensity	mass	relative intensity	mass	relative intensity	mass
26.16%	27.0	12.48%	93.0	9.28%	159.0
3.70%	31.0	5.91%	94.0	7.75%	160.0
2.67%	38.0	5.06%	95.0	29.25%	161.0
35.45%	39.0	1.30%	99.0	10.15%	162.0
23.32%	41.0	3.28%	101.0	22.74%	163.0
1.03%	42.0	9.14%	102.0	11.56%	164.0
27.65%	43.0	13.67%	103.0	1.08%	165.0
36.04%	44.0	4.70%	104.0	3.80%	171.0
4.23%	45.0	15.72%	105.0	12.80%	173.0
2.41%	46.0	5.43%	106.0	3.93%	174.0
5.81%	50.0	13.49%	107.0	25.80%	175.0
27.89%	51.0	4.79%	108.0	17.70%	176.0
14.50%	52.0	3.29%	109.0	21.46%	177.0
21.10%	53.0	14.79%	115.0	6.34%	178.0
1.79%	54.0	6.16%	116.0	5.41%	186.0
10.67%	55.0	13.88%	117.0	6.51%	187.0
1.74%	57.0	13.02%	118.0	13.96%	188.0
6.56%	58.0	12.44%	119.0	32.61%	189.0
5.30%	59.0	4.73%	120.0	30.42%	190.0
3.29%	60.0	14.26%	121.0	54.23%	191.0
4.02%	62.0	4.16%	122.0	6.78%	192.0
13.49%	63.0	1.62%	123.0	1.19%	193.0
7.29%	64.0	2.31%	127.0	1.60%	199.0
29.39%	65.0	3.79%	128.0	42.17%	203.0
10.43%	66.0	4.45%	129.0	62.28%	204.0
11.61%	67.0	2.49%	130.0	11.81%	205.0
2.01%	68.0	8.65%	131.0	100.00%	206.0
13.33%	69.0	5.74%	132.0	16.47%	207.0
1.38%	72.0	12.87%	133.0	2.71%	208.0
3.75%	73.0	9.21%	134.0	51.12%	214.0
4.39%	74.0	14.36%	135.0	10.29%	215.0
4.32%	75.0	7.50%	136.0	1.92%	216.0
2.88%	76.0	1.50%	137.0	99.06%	217.0
39.05%	77.0	1.58%	141.0	15.28%	218.0
12.03%	78.0	1.90%	143.0	1.91%	219.0
21.54%	79.0	2.02%	144.0	10.49%	231.0
6.56%	80.0	13.93%	145.0	54.19%	232.0
5.41%	81.0	15.78%	146.0	9.45%	233.0
1.93%	86.0	13.20%	147.0	1.16%	234.0
4.51%	87.0	7.58%	148.0	93.83%	250.0
3.77%	88.0	19.35%	149.0	17.20%	251.0
7.96%	89.0	4.19%	150.0	2.24%	252.0
3.45%	90.0	1.39%	151.0		
42.95%	91.0	1.48%	157.0		
8.73%	92.0	2.60%	158.0		

Most abundant peaks

m/z	206.0	217.0	250.0	204.0	191.0
Intensity	100.00	99.06	93.83	62.28	54.23
m/z	232.0	214.0	91.0	203.0	77.0
Intensity	54.19	51.12	42.95	42.17	39.05

Formula



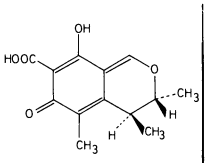
Experimental conditions: see p 2223

100 MHz ^{1}H NMR SPECTRUM 3d

CITRININ

conc. 49.96 mmol/l

solvent: $CDCl_3$

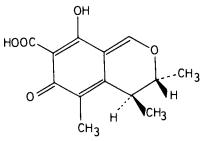


250 MHz ^{1}H NMR SPECTRUM 3e

CITRININ

conc. 49.96 mmol/l

solvent: $CDCl_3$



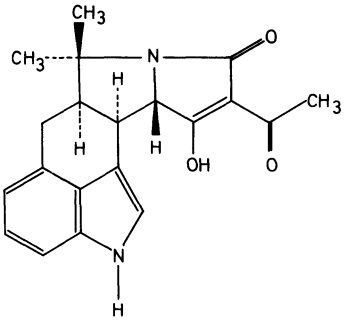
IV.4.α-CYCLOPIAZONIC ACID

I SYNONYMS: none

II CHEMICAL NAME: 10-acetyl-2,6,6a,7,11a,11b-hexahydro-11-hydroxy-7,7-dimethyl-(6aα,11aβ,11bα)-9H-Pyrrolo[1',2':2,3]iso indolo[4,5,6-cd] indol-9-one

III EMPIRICAL FORMULA: $C_{20}H_{20}N_2O_3$

IV STRUCTURAL FORMULA:



V MOLECULAR WEIGHT: 336.4

VI DESCRIPTION: α-Cyclopiazonic acid is a white, odourless, crystalline solid

VII CHARACTERIZATION DATA:

1.Melting range: 214-218°C, after drying for 1 hour at 60°C

2.Specific rotation: $[\alpha]_D^{21} = -109.4^o$
conc. 2973 μmol/l
solvent: chloroform

3.Circular dichroism:

$\Delta\varepsilon_{(\lambda 365)}$ 0, $\Delta\varepsilon_{(\lambda 301)}$ +1.8, $\Delta\varepsilon_{(\lambda 292)}$ 0, $\Delta\varepsilon_{(\lambda 269)}$ -4.56, $\Delta\varepsilon_{(\lambda 253)}$ 0.
conc. 351.1 μmol/l
solvent: methanol
temperature: 22°C
cell length: 2 mm

4.Ultraviolet absorption spectrum:
see spectrum 4a

Molar absorption coefficients:

$\varepsilon_{(\lambda 224)} = 3973 \pm 4$
$\varepsilon_{(\lambda 239)} = 1186 \pm 35$
$\varepsilon_{(\lambda 281)} = 1972 \pm 15$

conc. 21.19 mol/l
solvent: methanol

5.Infrared absorption spectrum:
see spectrum 4b

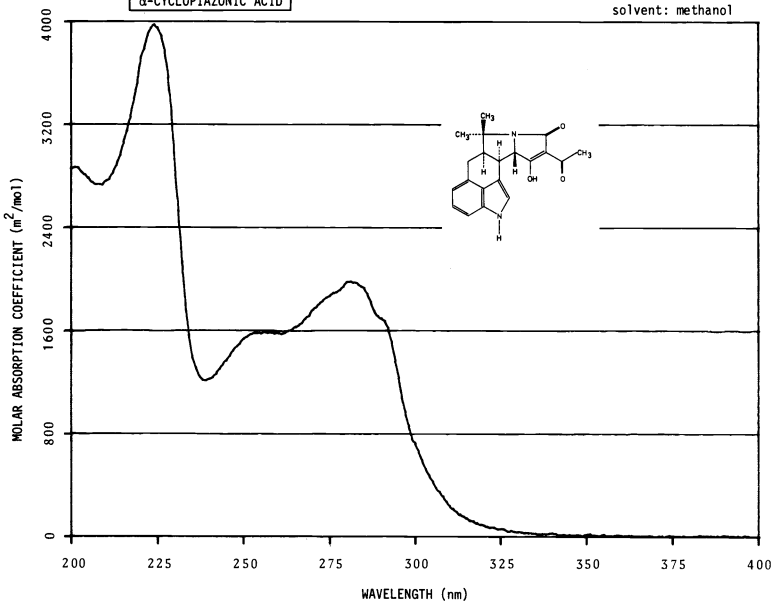
6.Electron impact mass spectrum:
see spectrum 4c

7.Nuclear Magnetic Resonance spectrum:
see spectra 4d and 4e

ULTRAVIOLET ABSORPTION SPECTRUM 4a

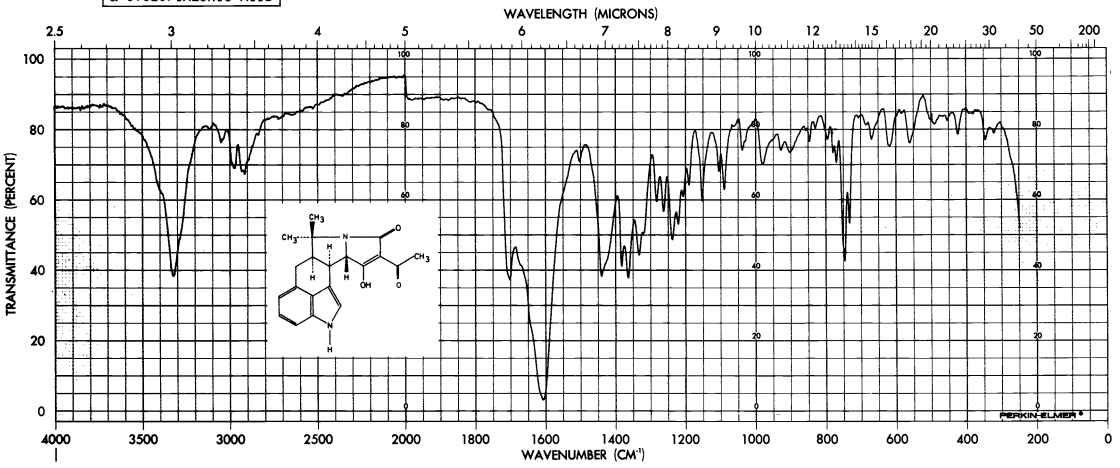
α-CYCLOPIAZONIC ACID

conc. 21.19 μmol/l
solvent: methanol



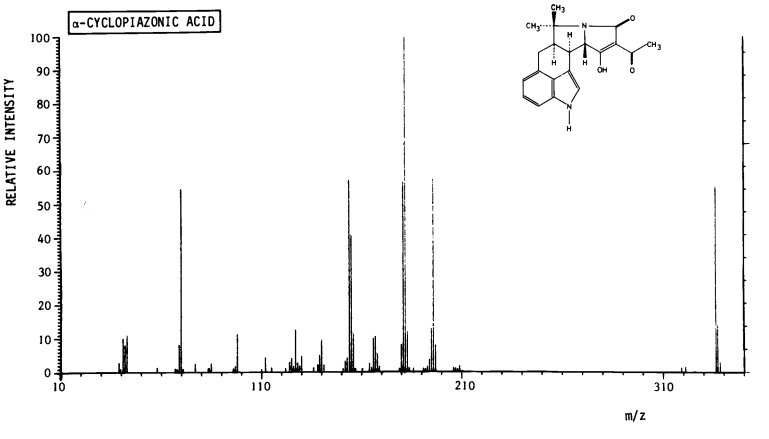
INFRARED ABSORPTION SPECTRUM 4b

α-CYCLOPIAZONIC ACID



MASS SPECTRUM 4c

α-CYCLOPIAZONIC ACID



MASS SPECTRUM 4c

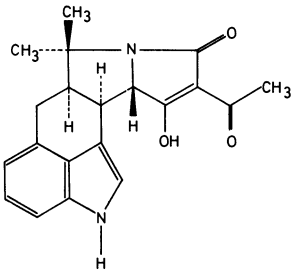
α-CYCLOPIAZONIC ACID

relative intensity	mass	relative intensity	mass
2.97%	39.1	10.66%	167.1
1.15%	39.9	5.52%	168.1
10.18%	41.1	1.82%	169.0
8.16%	42.0	1.10%	179.1
11.09%	43.1	8.11%	180.1
1.53%	58.1	56.67%	181.1
1.29%	67.1	100.00%	182.1
1.00%	68.1	12.10%	183.1
8.21%	69.0	1.34%	184.1
54.51%	70.1	1.29%	186.0
1.24%	71.1	1.24%	191.0
2.68%	77.1	1.15%	192.1
1.39%	83.6	1.87%	193.1
1.48%	84.1	3.84%	194.1
2.73%	85.1	13.20%	195.1
1.39%	96.0	57.54%	196.1
1.96%	97.0	8.11%	197.1
11.38%	98.1	1.48%	206.1
1.00%	110.1	1.39%	207.1
4.46%	112.1	1.05%	208.1
1.53%	115.1	1.92%	209.1
1.29%	122.1	1.44%	319.0
3.07%	124.1	1.63%	321.0
4.22%	125.1	55.18%	336.2
1.96%	126.0	13.92%	337.1
12.63%	127.1	2.97%	338.2
2.97%	128.0		
1.92%	129.1		
4.80%	130.1		
1.48%	136.1		
2.35%	138.1		
5.09%	139.1		
9.55%	140.1		
2.25%	141.2		
1.24%	150.9		
3.31%	151.9		
4.27%	152.9		
57.25%	154.0		
40.77%	155.0		
11.38%	156.1		
1.24%	157.1		
1.20%	160.6		
2.78%	164.1		
1.53%	165.1		
10.08%	166.1		

Most abundant peaks

m/z	182.1	196.1	154.0	181.1	336.2
Intensity	100.00	57.54	57.25	56.67	55.18
m/z	70.1	155.0	337.1	195.1	127.1
Intensity	54.51	40.77	13.92	13.20	12.63

Formula



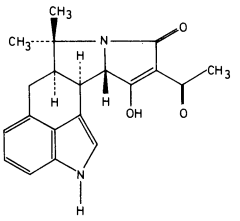
Experimental conditions: see p 2223

100 MHz ^{1}H NMR SPECTRUM 4d

α-CYCLOPIAZONIC ACID

conc. 37.16 mmol/l

solvent: $CDCl_3$

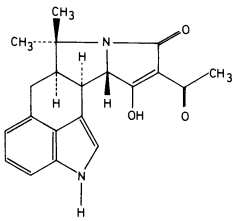


250 MHz ^{1}H NMR SPECTRUM 4e

α-CYCLOPIAZONIC ACID

conc. 22.29 mmol/l

solvent: $CDCl_3$



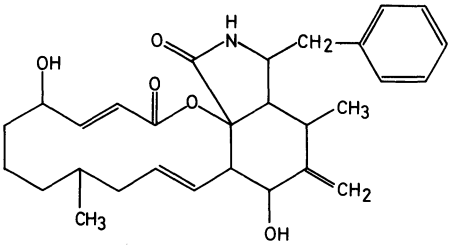
IV.5.CYTOCHALASIN B

I SYNONYMS: phomin

II CHEMICAL NAME: 7,20-dihydroxy-16-methyl-10-phenyl-,(7S,13E,16R,20R,21E)-24-Oxa[14] cytochalasa-6(12),13,21-triene-1,23-dione

III EMPIRICAL FORMULA: $C_{29}H_{37}NO_5$

IV STRUCTURAL FORMULA:



V MOLECULAR WEIGHT: 479.6

VI DESCRIPTION: Cytochalasin B is a white, odourless, crystalline solid

VII CHARACTERIZATION DATA:

1.Melting range: 216-222°C, after drying for 1 hour at 60°C

2.Specific rotation: $[\alpha]_D^{21} = +87.1^\circ$
conc. 2002 µmol/l
solvent: chloroform

3.Circular dichroism: No Cotton effects

4.Ultraviolet absorption spectrum:
see spectrum 5a

No absorption
conc. 23.09 µmol/l
solvent: methanol

5.Infrared absorption spectrum:
see spectrum 5b

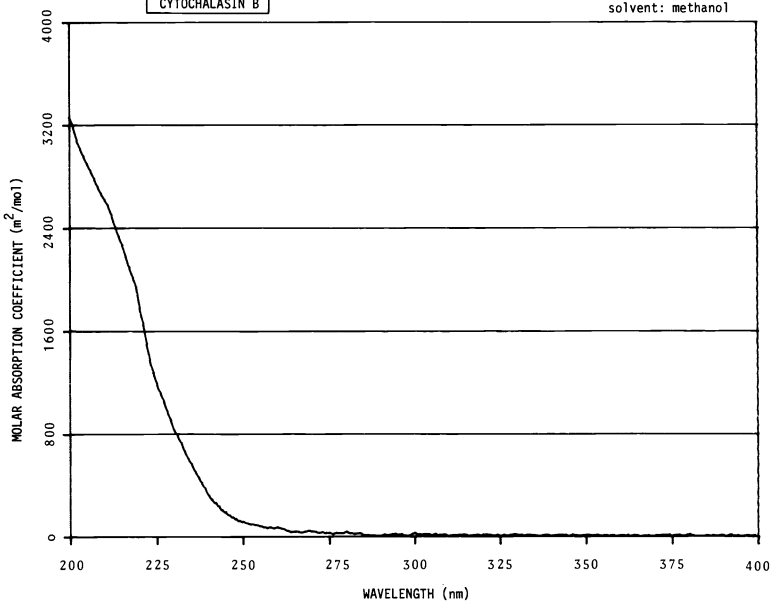
6.Electron impact mass spectrum:
see spectrum 5c

7.Nuclear Magnetic Resonance spectrum:
see spectra 5d and 5e

ULTRAVIOLET ABSORPTION SPECTRUM 5a

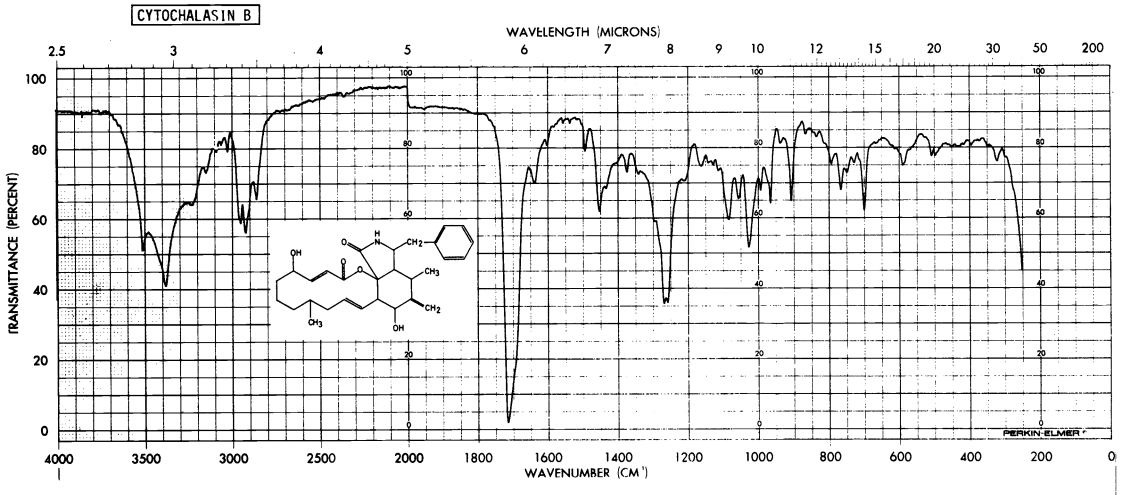
CYTOCHALASIN B

conc. 19.55 µmol/l
solvent: methanol



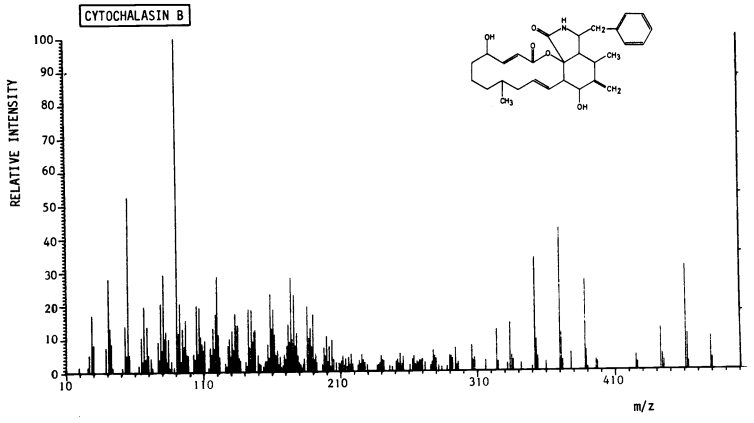
INFRARED ABSORPTION SPECTRUM 5b

CYTOCHALASIN B



MASS SPECTRUM 5c

CYTOCHALASIN B



MASS SPECTRUM 5c

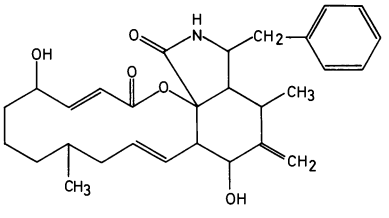
CYTOCHALASIN B

relative intensity	mass	relative intensity	mass	relative intensity	mass	relative intensity	mass	relative intensity	mass
1.84%	19.7	5.48%	103.1	4.27%	158.1	2.65%	209.1	4.65%	279.1
1.79%	25.9	3.96%	104.1	23.33%	159.1	2.31%	210.0	3.92%	280.2
5.31%	26.9	19.96%	105.1	12.96%	160.2	3.26%	211.1	1.70%	282.2
17.20%	28.9	5.46%	106.1	18.65%	161.1	4.69%	212.1	1.36%	284.2
8.31%	30.0	19.37%	107.1	11.11%	162.1	1.96%	213.2	1.58%	288.2
7.54%	39.1	10.57%	108.1	5.17%	163.1	3.81%	214.1	4.60%	290.1
28.18%	41.1	8.49%	109.2	6.38%	164.2	1.54%	215.1	4.51%	291.1
13.28%	42.0	6.53%	110.1	2.22%	165.2	4.21%	216.1	3.84%	292.1
8.57%	43.1	9.47%	111.1	4.56%	166.2	2.19%	217.1	6.90%	294.2
1.55%	44.0	1.39%	114.1	1.30%	167.1	5.28%	218.2	1.90%	295.1
1.47%	51.1	7.29%	115.1	1.90%	168.1	2.27%	219.1	2.70%	296.3
13.90%	53.1	5.28%	116.1	5.12%	169.1	1.94%	223.1	7.65%	306.1
5.19%	54.1	13.11%	117.1	3.86%	170.2	3.37%	224.1	3.34%	307.0
52.50%	55.1	7.02%	118.0	7.85%	171.1	2.31%	225.0	4.05%	308.1
5.36%	56.1	17.29%	119.1	14.23%	172.1	5.15%	226.0	3.10%	316.1
2.10%	63.1	28.61%	120.1	8.97%	173.1	3.41%	227.0	12.30%	324.2
10.57%	65.0	11.21%	121.1	28.18%	174.1	2.74%	228.1	2.82%	325.1
3.34%	66.0	4.48%	122.2	9.71%	175.0	2.03%	229.9	2.39%	332.1
19.74%	67.0	2.61%	126.1	22.93%	176.1	1.99%	237.1	14.20%	334.1
4.15%	67.9	1.70%	127.1	7.81%	177.1	2.07%	238.1	4.60%	335.1
13.78%	69.1	7.90%	128.1	11.66%	178.1	2.54%	239.2	3.44%	336.1
5.27%	70.1	9.95%	129.1	4.87%	179.1	4.67%	240.1	2.39%	342.0
4.24%	72.1	4.69%	130.1	2.74%	180.1	3.41%	241.1	1.23%	350.1
1.42%	73.0	12.24%	131.2	2.18%	181.1	3.25%	242.1	33.75%	352.1
9.19%	77.1	6.78%	132.1	1.20%	182.1	1.67%	246.1	9.38%	353.0
3.93%	78.1	17.41%	133.2	2.37%	183.1	1.47%	248.1	4.45%	354.0
20.60%	79.1	13.95%	134.1	3.80%	183.9	3.09%	251.1	2.61%	360.0
6.59%	80.0	14.02%	135.1	19.58%	185.9	3.61%	252.1	42.44%	370.1
29.20%	81.0	4.20%	136.1	10.04%	187.1	2.31%	253.1	11.21%	371.0
10.10%	82.0	3.41%	137.1	13.01%	188.1	5.22%	254.0	3.13%	372.0
12.13%	82.9	3.01%	141.1	7.42%	188.9	2.39%	255.1	5.36%	378.1
3.26%	84.0	1.84%	142.1	17.04%	190.0	4.48%	256.1	27.06%	388.3
9.98%	84.9	18.70%	143.1	3.65%	191.1	2.02%	261.1	6.03%	389.1
1.30%	86.0	7.38%	144.0	5.46%	192.1	3.68%	263.1	1.08%	390.2
3.37%	86.9	18.51%	145.1	2.79%	192.9	4.69%	264.1	3.22%	396.2
1.66%	88.9	9.16%	146.1	2.19%	196.9	2.19%	265.0	2.79%	397.3
100.00%	90.9	12.06%	147.1	7.25%	198.1	3.09%	266.1	4.67%	425.2
11.66%	92.0	12.65%	148.0	4.87%	199.0	2.67%	267.1	2.46%	426.1
20.50%	93.0	5.05%	150.2	10.57%	200.0	3.37%	268.1	12.52%	443.1
3.20%	94.1	2.54%	151.1	3.06%	201.1	3.20%	269.1	4.81%	444.2
12.95%	95.0	2.10%	152.1	7.57%	202.1	3.74%	270.1	2.82%	445.3
7.78%	96.1	1.70%	154.1	1.67%	203.1	2.70%	272.2	31.10%	461.1
15.54%	97.1	3.26%	155.1	9.47%	204.1	1.87%	276.1	10.75%	462.3
5.24%	98.1	3.26%	156.2	3.72%	205.1	2.82%	277.1	2.54%	463.1
5.05%	99.1	8.21%	157.1	2.67%	207.1	6.29%	278.2	9.90%	479.1
								3.50%	480.1

Most abundant peaks

m/z	90.9	55.1	370.1	352.1	461.1
Intensity	100.00	52.50	42.44	33.75	31.10
m/z	81.0	120.1	174.1	41.1	388.3
Intensity	29.20	28.61	28.18	28.18	27.06

Formula



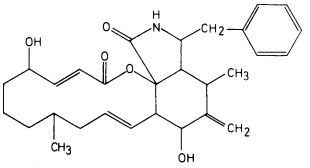
Experimental conditions: see p 2223

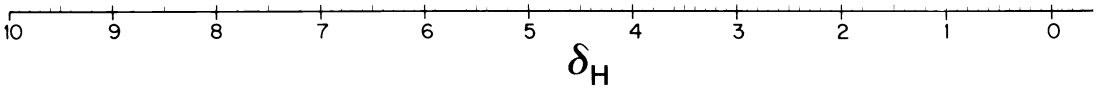
100 MHz ^{1}H NMR SPECTRUM 5d

CYTOCHALASIN B

conc. saturated

solvent: $CDCl_3$





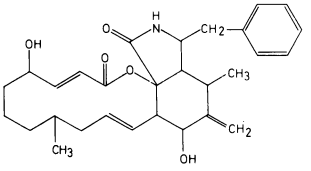
δ_H

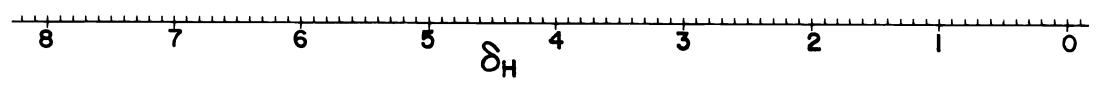
250 MHz ^{1}H NMR SPECTRUM 5e

CYTOCHALASIN B

conc. saturated

solvent: $CDCl_3$





δ_H

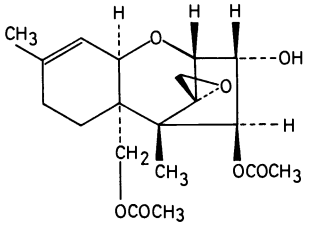
IV.6.DIACETOXYSCIRPENOL

I SYNONYMS: anguidin, anguidine

II CHEMICAL NAME: 12,13-,epoxy-4,15-diacetate,(3α,4β)-Trichothec-9-ene-3,4,15-triol

III EMPIRICAL FORMULA: $C_{19}H_{26}O_7$

IV STRUCTURAL FORMULA:



V MOLECULAR WEIGHT: 366.4

VI DESCRIPTION: Diacetoxyscirpenol is a white, odourless, crystalline solid

VII CHARACTERIZATION DATA:

1.Melting range: 163-164°C, after drying for 1 hour at 60°C

2.Specific rotation: $[\alpha]_D^{21} = -24.3^\circ$
conc. 2593 µmol/l
solvent: chloroform

3.Circular dichroism: No Cotton effects

4.Ultraviolet absorption spectrum:
see spectrum 6a

No absorption
conc. 23.09 µmol/l
solvent: methanol

5.Infrared absorption spectrum:
see spectrum 6b

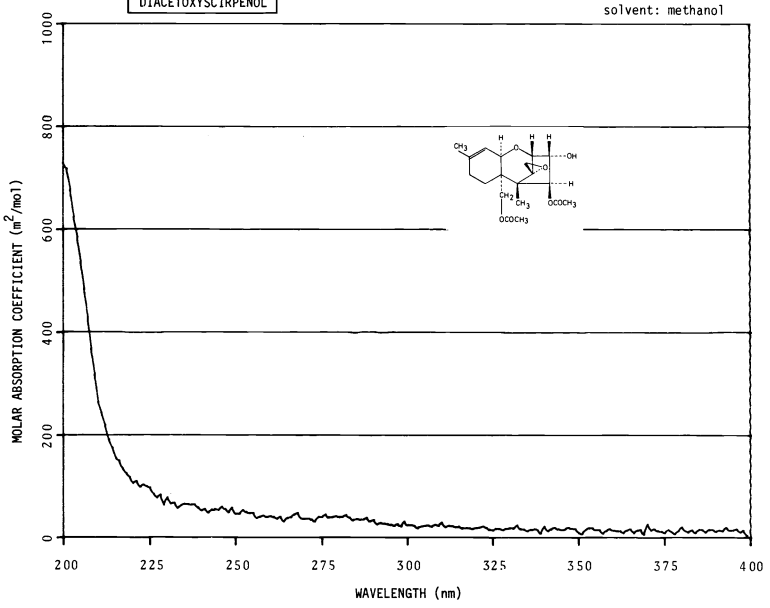
6.Electron impact mass spectrum:
see spectrum 6c

7.Nuclear Magnetic Resonance spectrum:
see spectra 6d and 6e

ULTRAVIOLET ABSORPTION SPECTRUM 6a

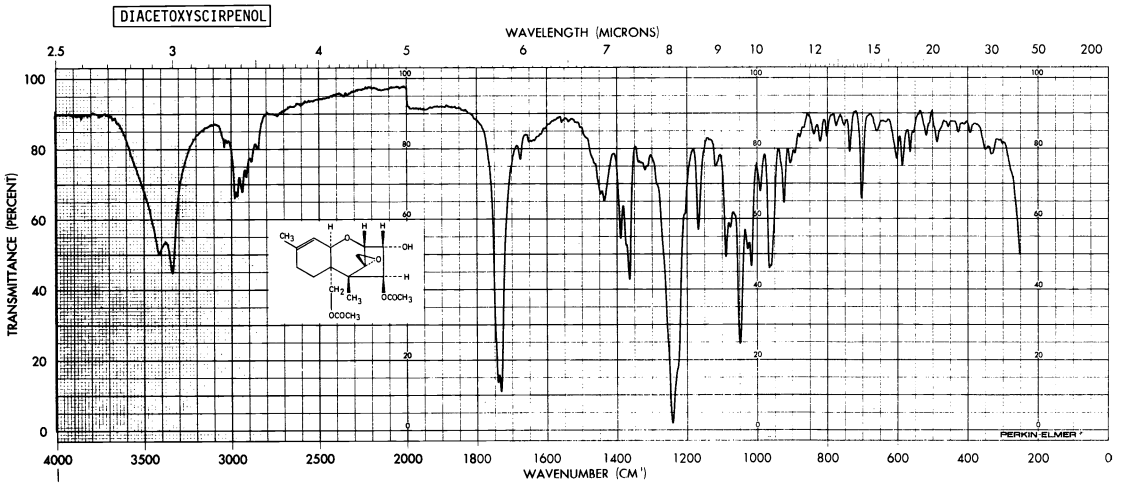
DIACETOXYSCIRPENOL

conc. 23.09 μmol/l
solvent: methanol



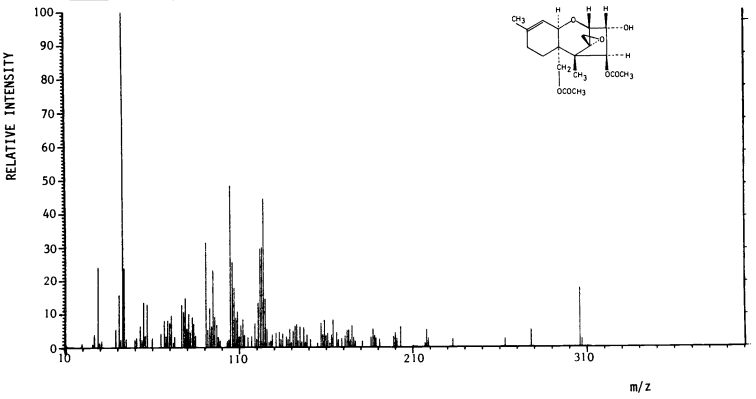
INFRARED ABSORPTION SPECTRUM 6b

DIACETOXYSCIRPENOL



MASS SPECTRUM 6c

DIACETOXYSCIRPENOL



MASS SPECTRUM 6c

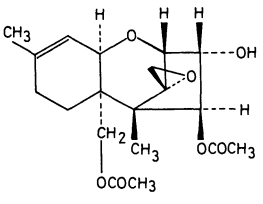
DIACETOXYSCIRPENOL

relative intensity	mass	relative intensity	mass	relative intensity	mass
1.26%	19.7	2.01%	99.1	7.21%	157.1
1.07%	25.9	1.94%	103.1	3.84%	158.1
3.95%	26.9	2.30%	104.1	8.17%	159.1
24.13%	28.9	48.44%	105.2	3.51%	160.1
1.48%	29.9	25.65%	106.2	4.22%	161.1
2.03%	31.0	17.86%	107.2	1.37%	162.1
5.50%	39.1	8.94%	108.1	3.63%	163.2
15.85%	41.1	10.76%	109.2	8.28%	164.1
2.46%	42.0	3.34%	110.1	4.58%	166.2
100.00%	43.1	6.58%	111.1	2.49%	167.1
23.81%	43.9	8.42%	112.1	2.67%	169.2
2.60%	45.1	3.70%	113.1	3.34%	171.1
2.30%	50.1	3.04%	115.1	5.21%	172.1
2.93%	51.1	3.37%	117.1	5.26%	173.1
6.47%	53.1	7.18%	119.1	2.23%	174.1
2.31%	54.1	2.53%	120.1	6.40%	175.1
13.60%	55.1	13.47%	121.1	3.04%	176.1
3.56%	56.1	29.56%	122.1	1.86%	177.0
13.07%	57.1	29.90%	123.2	1.86%	181.1
2.93%	60.1	44.57%	124.1	3.07%	186.0
4.22%	65.1	14.56%	125.1	5.48%	187.1
8.05%	67.0	5.57%	126.1	3.55%	188.1
3.37%	68.0	1.64%	127.1	2.71%	188.9
8.13%	69.0	1.92%	128.1	2.41%	191.0
7.39%	70.1	3.84%	129.1	3.18%	199.1
9.69%	71.1	4.39%	131.2	4.33%	200.0
1.46%	72.1	4.69%	133.1	2.63%	201.1
3.23%	73.0	2.49%	134.1	6.15%	203.1
12.89%	77.0	4.17%	135.1	1.26%	217.2
10.61%	78.1	3.26%	137.1	5.24%	218.1
14.81%	79.1	2.46%	138.1	2.78%	219.1
5.76%	80.1	5.59%	139.1	2.41%	233.1
10.09%	81.1	1.53%	140.1	2.53%	263.2
4.54%	82.0	4.99%	141.1	5.13%	278.1
9.08%	83.1	6.31%	142.1	17.50%	306.1
7.07%	84.0	6.84%	143.2	2.46%	307.1
3.56%	84.9	2.01%	144.0		
31.40%	91.0	6.14%	145.1		
5.26%	92.0	1.70%	146.1		
11.86%	93.1	5.96%	147.1		
6.37%	94.1	1.61%	148.1		
23.17%	95.0	3.81%	149.1		
9.19%	96.1	2.41%	151.1		
6.85%	97.1	1.33%	155.2		
3.15%	98.1				

Most abundant peaks

m/z	43.1	105.2	124.1	91.0	123.2
Intensity	100.00	48.44	44.57	31.40	29.90
m/z	122.1	106.2	28.9	43.9	95.0
Intensity	29.56	25.65	24.13	23.81	23.17

Formula



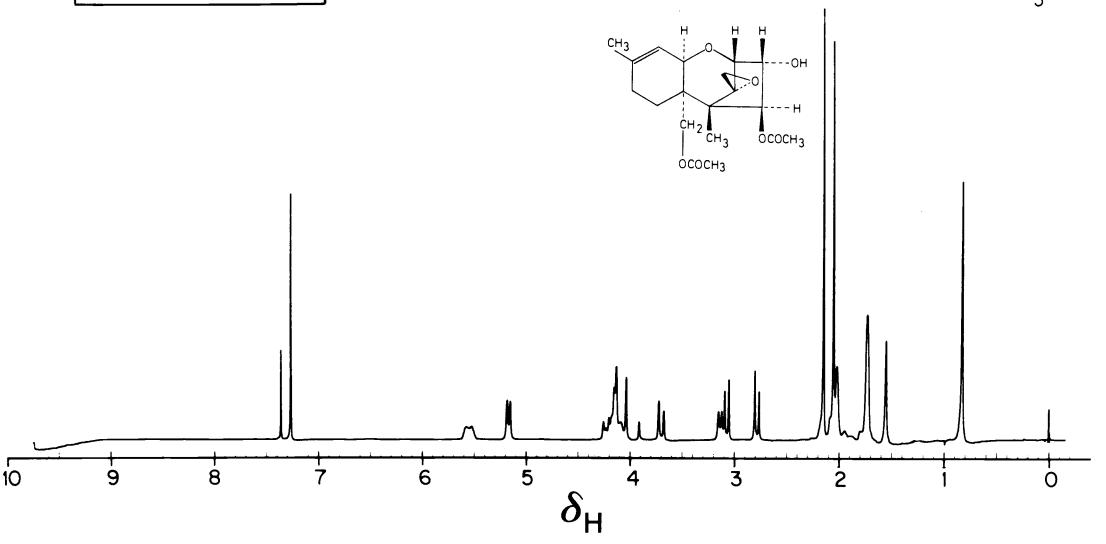
Experimental conditions: see p 2223

100 MHz ^{1}H NMR SPECTRUM 6d

DIACETOXYSCIRPENOL

conc. 34.12 mmol/l

solvent: $CDCl_3$

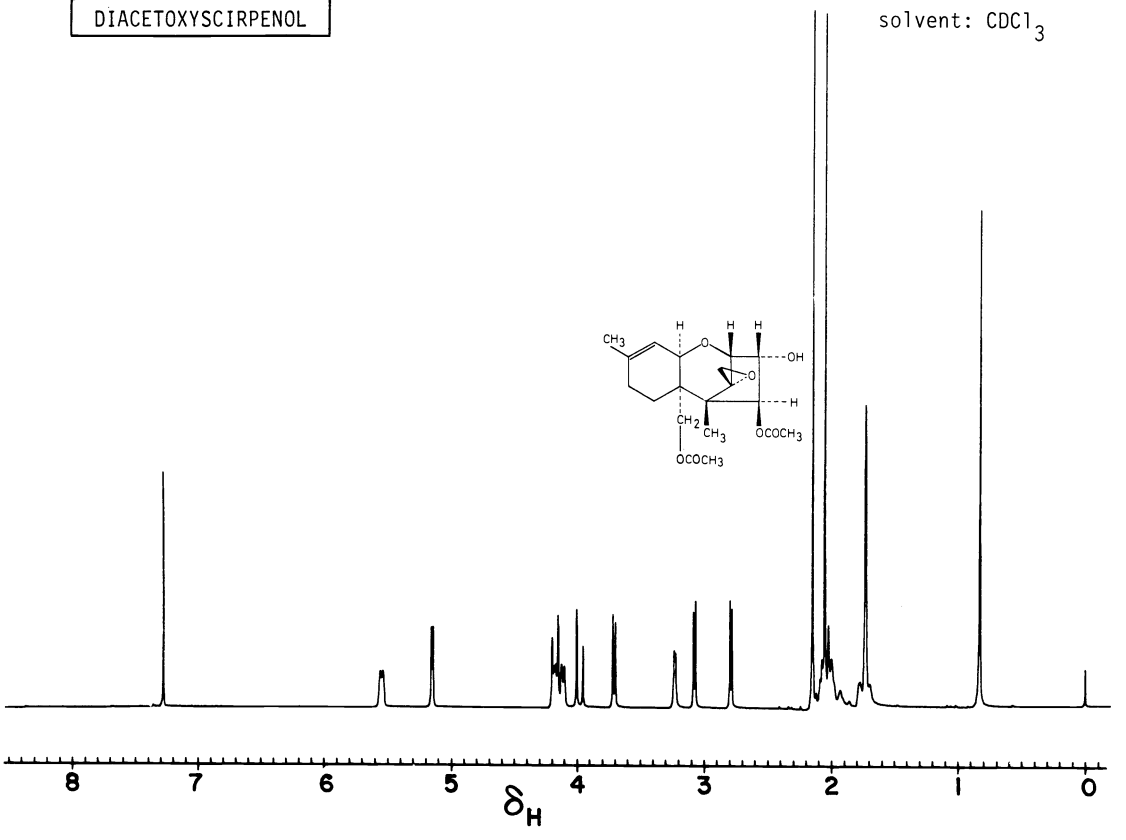


250 MHz ^{1}H NMR SPECTRUM 6e

DIACETOXYSCIRPENOL

conc. 34.12 mmol/l

solvent: $CDCl_3$



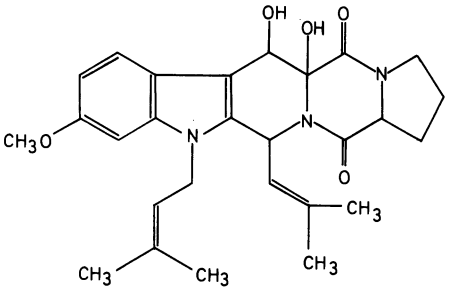
IV.7. FUMITREMORGEN B

I SYNONYMS: fumitremorgin B, lanosulin

II CHEMICAL NAME: 1,2,3,5a,6,11,12,14a-octahydro-5a,6-dihydroxy-9-methoxy-11-(3-methyl-2-butenyl)-12-(2-methyl-1-propenyl)-[5aR-(5aα,6α,12β,14aα)]-5H,14H-Pyrrolo[1",2":4',5']pyrazino[1',2':1,6]pyrido[3,4-b]indole-5,14-dione

III EMPIRICAL FORMULA: $C_{27}H_{33}N_3O_5$

IV STRUCTURAL FORMULA:



V MOLECULAR WEIGHT: 479.6

VI DESCRIPTION: Fumitremorgen B is a white, odourless, crystalline solid

VII CHARACTERIZATION DATA:

1. Melting range: 211-213°C, after drying for 1 hour at 60°C

2. Specific rotation: $[\alpha]_D^{21} = +6.4^\circ$
   conc. 2085 μmol/l
   solvent: chloroform

3. Circular dichroism:
   $\Delta\varepsilon_{(\lambda 320)}$ 0, $\Delta\varepsilon_{(\lambda 300)}$ +3.27, $\Delta\varepsilon_{(\lambda 288)}$ +2.72, $\Delta\varepsilon_{(\lambda 273)}$ +6.43, $\Delta\varepsilon_{(\lambda 252)}$ 0.
   conc. 249.4 μmol/l
   solvent: methanol
   temperature: 22°C
   cell length: 10 mm

4. Ultraviolet absorption spectrum:
   see spectrum 7a

   Molar absorption coefficients:
   $\varepsilon_{(\lambda 213)} = 2969 \pm 55$
   $\varepsilon_{(\lambda 227)} = 3429 \pm 40$
   $\varepsilon_{(\lambda 256)} = 415 \pm 3$
   $\varepsilon_{(\lambda 278)} = 736 \pm 4$
   $\varepsilon_{(\lambda 285)} = 672 \pm 3$
   $\varepsilon_{(\lambda 297)} = 805 \pm 3$
   conc. 21.35 μmol/l
   solvent: methanol

5. Infrared absorption spectrum:
   see spectrum 7b

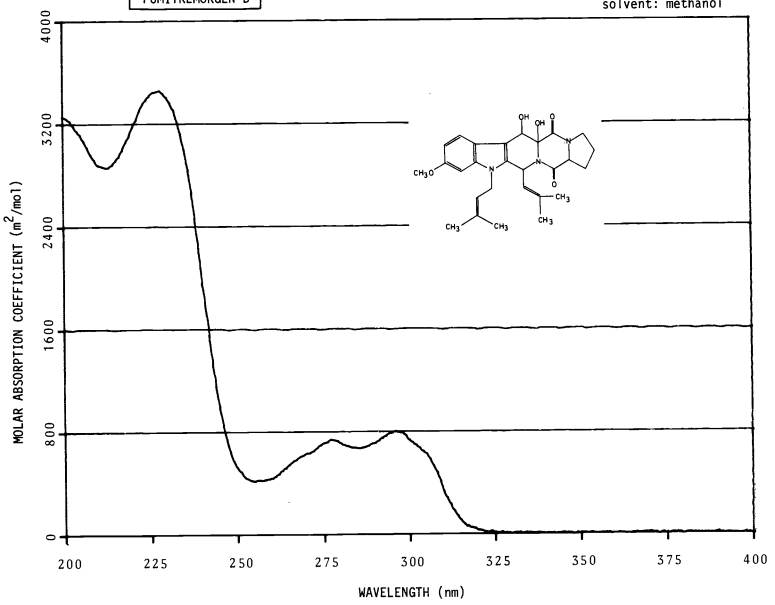
6. Electron impact mass spectrum:
   see spectrum 7c

7. Nuclear Magnetic Resonance spectrum:
   see spectra 7d and 7e

ULTRAVIOLET ABSORPTION SPECTRUM /a

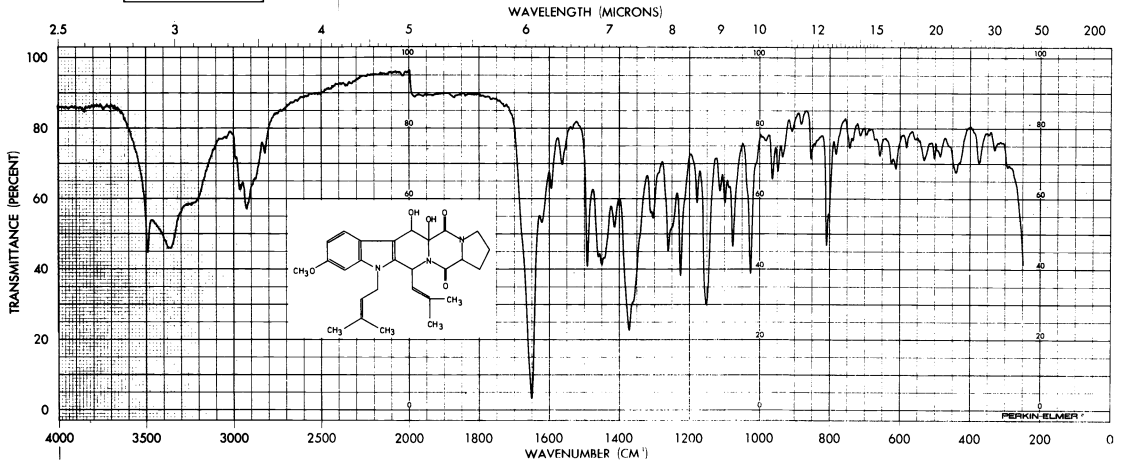
FUMITREMORGEN B

conc. 21.35 µmol/l
solvent: methanol



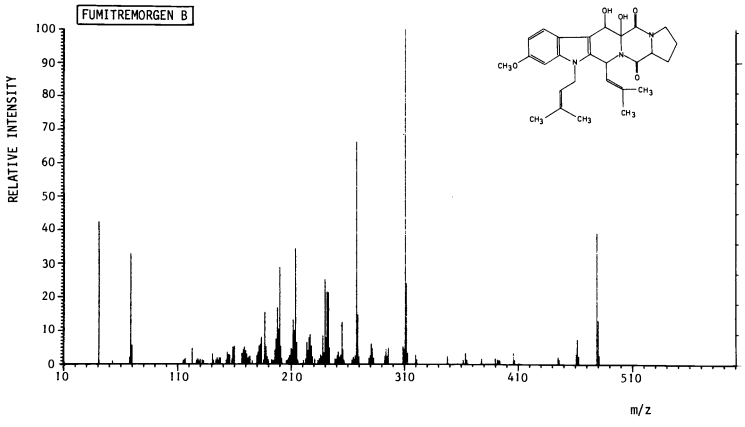
INFRARED ABSORPTION SPECTRUM 7b

FUMITREMORGEN B



MASS SPECTRUM 7c

FUMITREMORGEN B



MASS SPECTRUM 7c

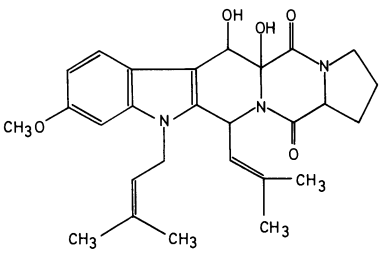
FUMITREMORGEN B

relative intensity	mass	relative intensity	mass	relative intensity	mass	relative intensity	mass
42.51%	41.0	5.29%	188.0	1.52%	250.0	7.33%	462.0
1.10%	53.0	2.31%	189.0	2.74%	251.0	2.48%	463.0
2.19%	68.0	1.30%	190.0	3.62%	252.0	39.17%	479.0
32.99%	69.0	1.40%	193.0	2.03%	253.0	12.93%	480.0
5.71%	70.0	1.02%	194.0	2.79%	254.0	2.50%	481.0
1.15%	115.0	1.22%	195.0	12.53%	255.0		
1.31%	116.0	4.24%	196.0	4.28%	256.0		
1.75%	117.0	7.59%	197.0	1.17%	257.0		
4.79%	123.0	16.95%	198.0	1.30%	264.0		
1.22%	127.0	10.46%	199.0	2.06%	265.0		
1.67%	128.0	28.95%	200.0	1.49%	266.0		
1.05%	129.0	5.39%	201.0	2.51%	267.0		
1.48%	130.0	1.68%	202.0	66.33%	268.0		
1.35%	132.0	1.05%	206.0	14.72%	269.0		
1.13%	133.0	1.27%	207.0	2.24%	270.0		
3.13%	141.0	1.88%	208.0	1.87%	279.0		
1.23%	142.0	2.69%	209.0	2.56%	280.0		
1.35%	144.0	4.71%	210.0	6.01%	281.0		
1.94%	145.0	4.24%	211.0	4.66%	282.0		
1.26%	146.0	13.28%	212.0	1.88%	283.0		
1.99%	147.0	10.04%	213.0	2.33%	293.0		
1.95%	148.0	34.45%	214.0	4.47%	294.0		
1.27%	153.0	6.50%	215.0	2.21%	295.0		
3.60%	154.0	1.22%	216.0	4.82%	296.0		
2.85%	155.0	1.10%	221.0	5.27%	309.0		
2.74%	156.0	1.63%	223.0	4.52%	310.0		
1.66%	158.0	6.45%	224.0	100.00%	311.0		
5.19%	159.0	4.22%	225.0	24.19%	312.0		
5.43%	160.0	8.09%	226.0	3.32%	313.0		
3.32%	167.0	8.81%	227.0	2.77%	320.0		
4.35%	168.0	5.49%	228.0	1.34%	321.0		
5.22%	169.0	2.26%	229.0	2.37%	348.0		
4.01%	170.0	1.68%	231.0	1.23%	362.0		
3.09%	171.0	1.15%	234.0	3.21%	364.0		
1.72%	172.0	1.66%	235.0	1.22%	365.0		
2.12%	173.0	2.85%	236.0	1.69%	378.0		
2.43%	174.0	2.37%	237.0	1.71%	390.0		
1.13%	176.0	8.47%	238.0	1.37%	392.0		
2.50%	180.0	3.46%	239.0	1.12%	393.0		
3.55%	181.0	25.32%	240.0	1.14%	394.0		
5.44%	182.0	8.03%	241.0	3.23%	406.0		
5.96%	183.0	21.52%	242.0	1.25%	407.0		
8.08%	184.0	21.44%	243.0	2.02%	445.0		
1.24%	186.0	4.80%	244.0	1.36%	446.0		
15.46%	187.0	1.68%	249.0	2.96%	461.0		

Most abundant peaks

m/z	311.0	268.0	41.0	479.0	214.0
Intensity	100.00	66.33	42.51	39.17	34.45
m/z	69.0	200.0	240.0	312.0	242.0
Intensity	32.99	28.95	25.32	24.19	21.52

Formula



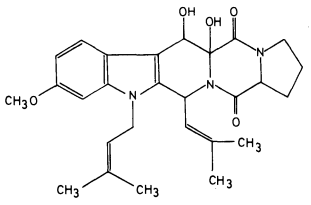
Experimental conditions: see p 2223

100 MHz ^{1}H NMR SPECTRUM 7d

FUMITREMORGEN B

conc. 26.06 mmol/l

solvent: $CDCl_3$

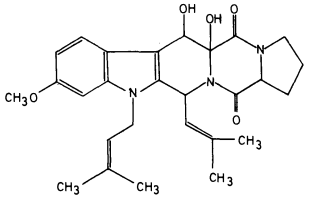


250 MHz ^{1}H NMR SPECTRUM 7e

FUMITREMORGEN B

conc. 26.06 mmol/l

solvent: $CDCl_3$



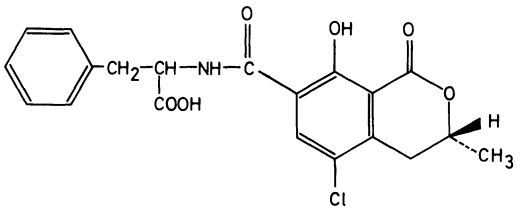
IV.8.OCHRATOXIN A

I SYNONYMS: none

II CHEMICAL NAME: N-[(5-chloro-3,4-dihydro-8-hydroxy-3-methyl-1-oxo-1H-2-benzopyran-7-yl) carbonyl]-,(R)-L-Phenylalanine

III EMPIRICAL FORMULA: $C_{20}H_{18}ClNO_6$

IV STRUCTURAL FORMULA:



V MOLECULAR WEIGHT: 403.8

VI DESCRIPTION: Ochratoxin A is a white, odourless, crystalline solid

VII CHARACTERIZATION DATA:

1.Melting range: 168-173°C, after drying for 1 hour at 60°C

2.Specific rotation: $[\alpha]_D^{21} = -46.8^o$
conc. 2650 µmol/l
solvent: chloroform

3.Circular dichroism:
$\Delta\varepsilon_{(\lambda 395)}$ 0, $\Delta\varepsilon_{(\lambda 327)}$ -1.45, $\Delta\varepsilon_{(\lambda 300)}$ 0, $\Delta\varepsilon_{(\lambda 285)}$ +0.20, $\Delta\varepsilon_{(\lambda 275)}$ 0, $\Delta\varepsilon_{(\lambda 235)}$ -6.37, $\Delta\varepsilon_{(\lambda 218)}$ 0.
conc. 261.3 µmol/l
solvent: methanol
temperature: 22°C
cell length: 2 mm

4.Ultraviolet absorption spectrum:
see spectrum 8a

Molar absorption coefficients:
$\varepsilon_{(\lambda 214)} = 3720 \pm 55$
$\varepsilon_{(\lambda 282)} = 89 \pm 6$
$\varepsilon_{(\lambda 332)} = 633 \pm 7$
conc. 21.81 µmol/l
solvent: methanol

5.Infrared absorption spectrum:
see spectrum 8b

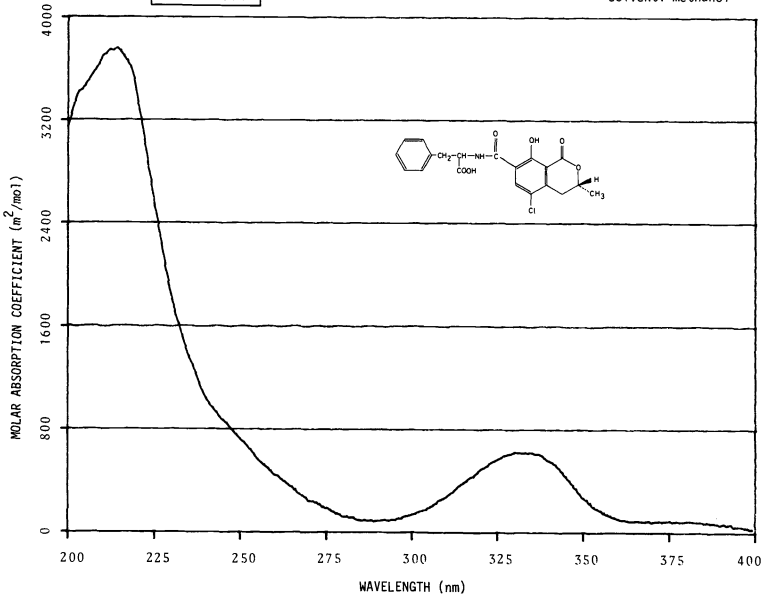
6.Electron impact mass spectrum:
see spectrum 8c

7.Neclear Magnetic Resonance spectrum:
see spectra 8d and 8e

ULTRAVIOLET ABSORPTION SPECTRUM 8a

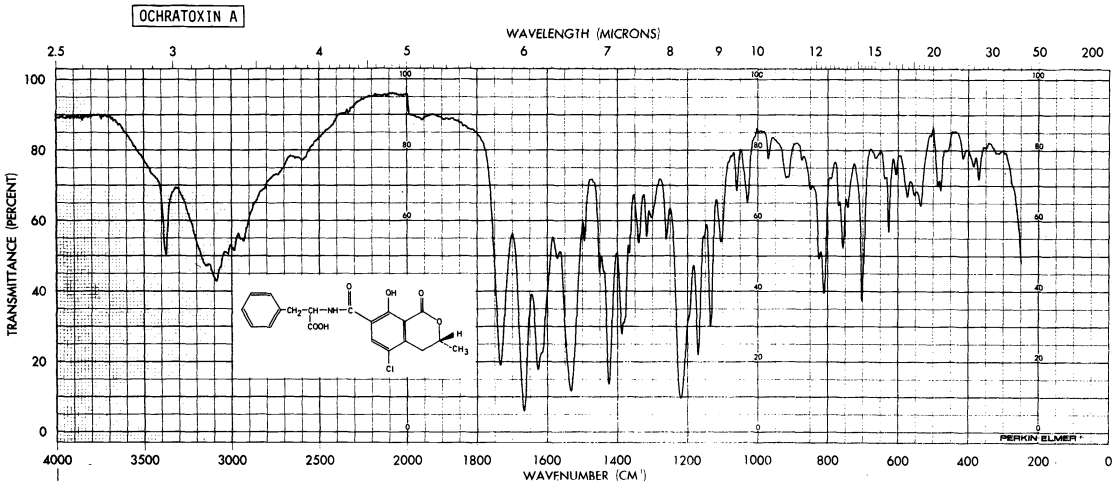
OCHRATOXIN A

conc. 21.81 µmol/l
solvent: methanol



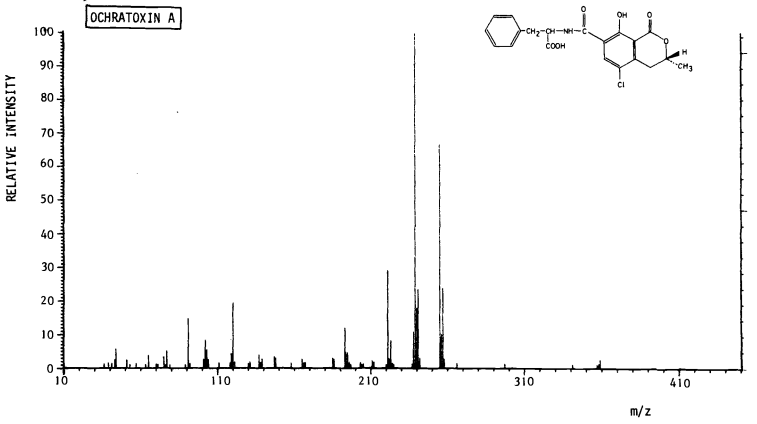
INFRARED ABSORPTION SPECTRUM 8b

OCHRATOXIN A



MASS SPECTRUM 8c

OCHRATOXIN A



MASS SPECTRUM 8c

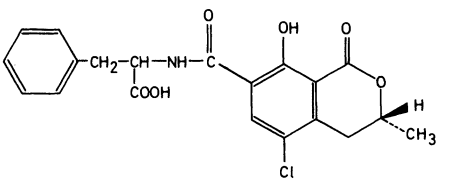
OCHRATOXIN A

relative intensity	mass	relative intensity	mass
1.41%	36.1	1.05%	196.9
1.62%	39.1	1.62%	203.1
1.58%	41.1	1.04%	204.1
2.57%	43.1	1.29%	205.1
5.74%	44.0	2.33%	211.0
2.40%	51.0	1.91%	212.0
1.29%	53.0	1.05%	220.1
1.57%	57.1	29.17%	221.1
1.24%	63.1	2.86%	222.1
3.84%	65.1	8.16%	223.1
1.36%	70.1	1.47%	224.2
1.25%	71.2	1.14%	225.0
3.39%	75.1	1.30%	237.1
1.09%	76.1	10.93%	237.9
5.16%	77.1	100.00%	239.0
1.19%	79.1	17.92%	240.2
1.17%	89.1	23.51%	241.0
14.86%	91.0	3.09%	242.0
1.44%	92.1	66.59%	255.0
2.65%	101.0	10.05%	256.0
8.37%	102.1	23.84%	257.1
5.41%	103.1	2.86%	258.0
2.52%	104.1	1.66%	266.1
1.66%	111.0	1.31%	297.4
1.57%	118.1	1.16%	341.2
4.36%	119.1	1.00%	357.0
19.47%	120.2	1.21%	358.1
1.95%	121.1	2.57%	359.1
1.47%	130.1	0.86%	403.1
1.87%	131.1		
3.93%	137.1		
1.72%	138.1		
2.79%	139.1		
3.35%	147.0		
2.91%	147.9		
1.61%	157.9		
2.69%	165.0		
1.62%	166.1		
1.69%	167.1		
2.99%	185.1		
2.62%	186.0		
11.93%	193.1		
4.73%	193.9		
4.58%	194.9		
1.80%	195.9		

Most abundant peaks

m/z	239.0	255.0	221.1	257.1	241.0
Intensity	100.00	66.59	29.17	23.84	23.51
m/z	120.2	240.2	91.0	193.1	237.9
Intensity	19.47	17.92	14.86	11.93	10.93

Formula



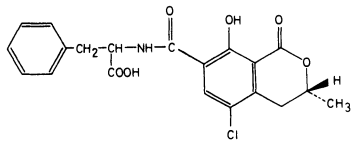
Experimental conditions: see p 2223

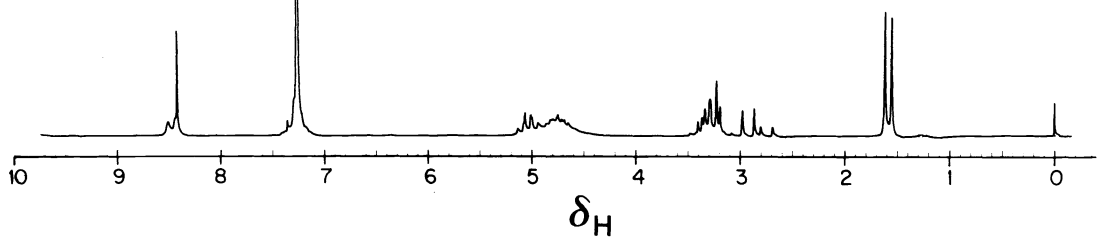
100 MHz ^{1}H NMR SPECTRUM 8d

OCHRATOXIN A

conc. 30.96 mmol/l

solvent: $CDCl_3$



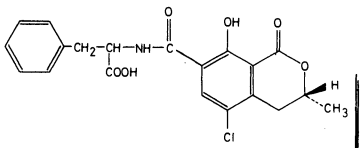


250 MHz ^{1}H NMR SPECTRUM 8e

OCHRATOXIN A

conc. 18.57 mmol/l

solvent: $CDCl_3$



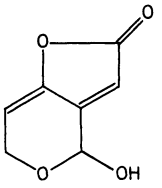
IV.9.PATULIN

I SYNONYMS: clairformin; clavacin; clavatin; claviformin; expansin; expansine; gigantin ; leucopin; mycoin; mycoin C; mycoin C3; mycoine C3; mycosin; patuline; penatin; penicidin; tercinin

II CHEMICAL NAME: 4-hydroxy-4H-Furo[3,2-c]pyran-2(6H)-one

III EMPIRICAL FORMULA: $C_7H_6O_4$

IV STRUCTURAL FORMULA:



V MOLECULAR WEIGHT: 154.1

VI DESCRIPTION: Patulin is a white, odourless, crystalline solid

VII CHARACTERIZATION DATA:

1.Melting range: 105-108°C, after drying for 1 hour at 60°C

2.Specific rotation: $[\alpha]_D^{21} = -6.2^o$
conc. 6489 µmol/l
solvent: chloroform

3.Circular dichroism: No Cotton effects

4.Ultraviolet absorption spectrum:
see spectrum 9a

Molar absorption coefficient :
$\varepsilon_{(\lambda 276)} = 1436 \pm 4$
conc. 44.98 µmol/l
solvent: methanol

5.Infrared absorption spectrum:
see spectrum 9b

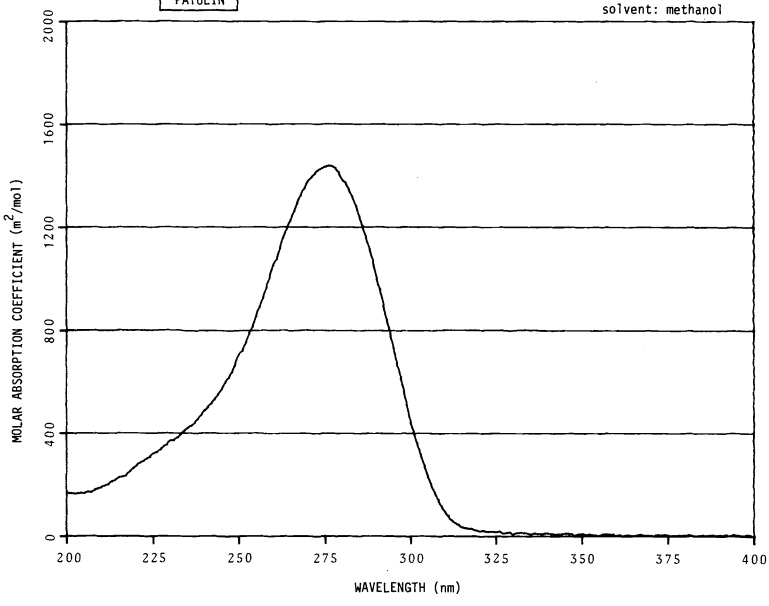
6.Electron impact mass spectrum:
see spectrum 9c

7.Nuclear Magnetic Resonance spectrum:
see spectra 9d and 9e

ULTRAVIOLET ABSORPTION SPECTRUM 9a

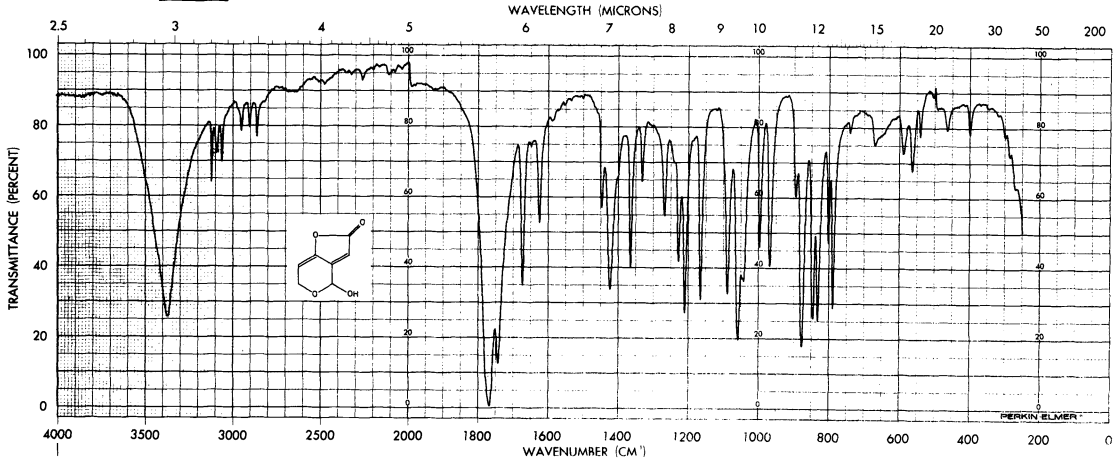
PATULIN

conc. 44.98 µmol/l
solvent: methanol



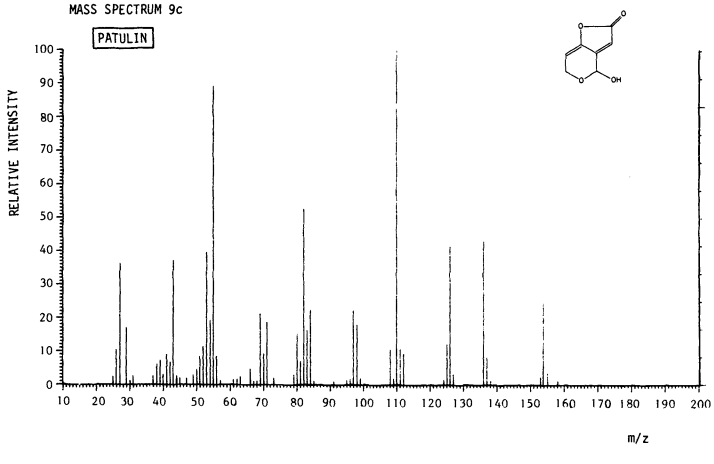
INFRARED ABSORPTION SPECTRUM 9b

PATULIN



MASS SPECTRUM 9c

PATULIN



MASS SPECTRUM 9c

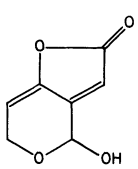
PATULIN

relative intensity	mass	relative intensity	mass
2.35%	25.0	1.72%	96.0
10.50%	26.0	22.10%	97.0
36.44%	27.1	17.94%	98.1
17.00%	29.0	1.95%	99.1
1.25%	30.1	10.50%	108.1
2.66%	31.0	1.88%	109.1
2.66%	37.0	100.00%	110.0
6.11%	38.1	10.73%	111.1
7.28%	39.1	9.32%	112.1
3.13%	40.0	1.41%	124.1
9.09%	41.1	12.30%	125.1
6.73%	42.1	41.37%	126.0
37.22%	43.0	3.13%	127.0
2.11%	44.0	43.02%	136.0
2.66%	44.1	8.22%	137.0
1.95%	45.0	1.17%	138.1
1.88%	47.0	2.35%	153.0
2.82%	49.0	24.52%	153.9
4.54%	50.1	3.44%	155.1
8.54%	51.0		
11.44%	52.0		
39.65%	53.0		
19.27%	54.1		
89.26%	55.0		
8.46%	56.0		
1.25%	57.1		
1.56%	61.0		
1.64%	62.1		
2.35%	63.1		
4.62%	66.1		
1.01%	67.1		
1.17%	68.1		
21.15%	69.1		
9.16%	70.1		
18.65%	71.1		
1.95%	73.2		
2.97%	79.2		
15.36%	80.1		
6.97%	81.1		
52.58%	82.1		
16.22%	83.1		
22.41%	84.1		
1.17%	85.1		
1.09%	91.1		
1.48%	95.0		

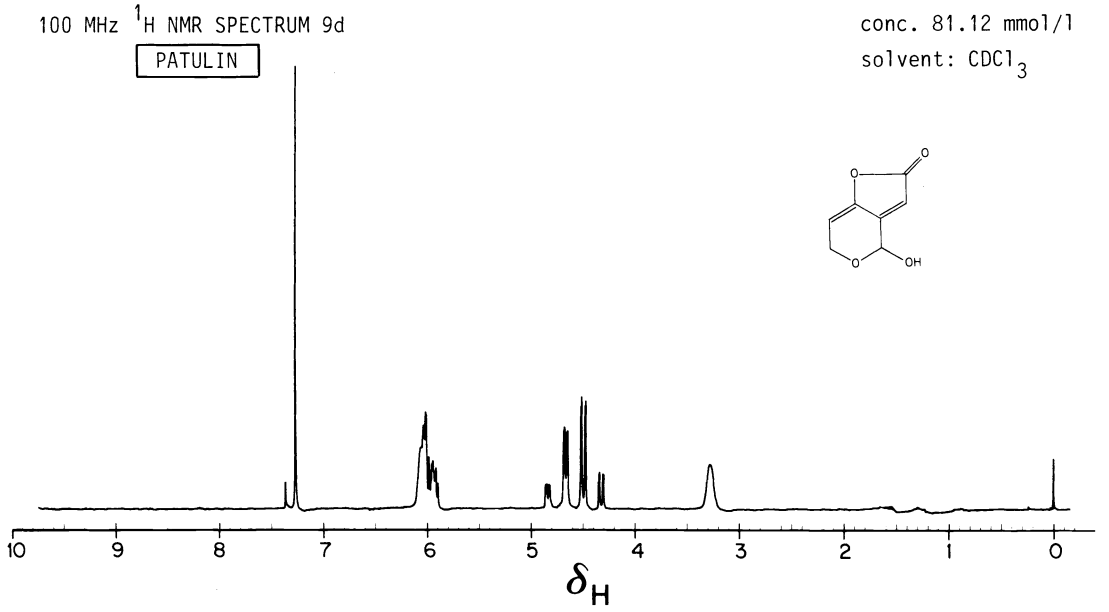
Most abundant peaks

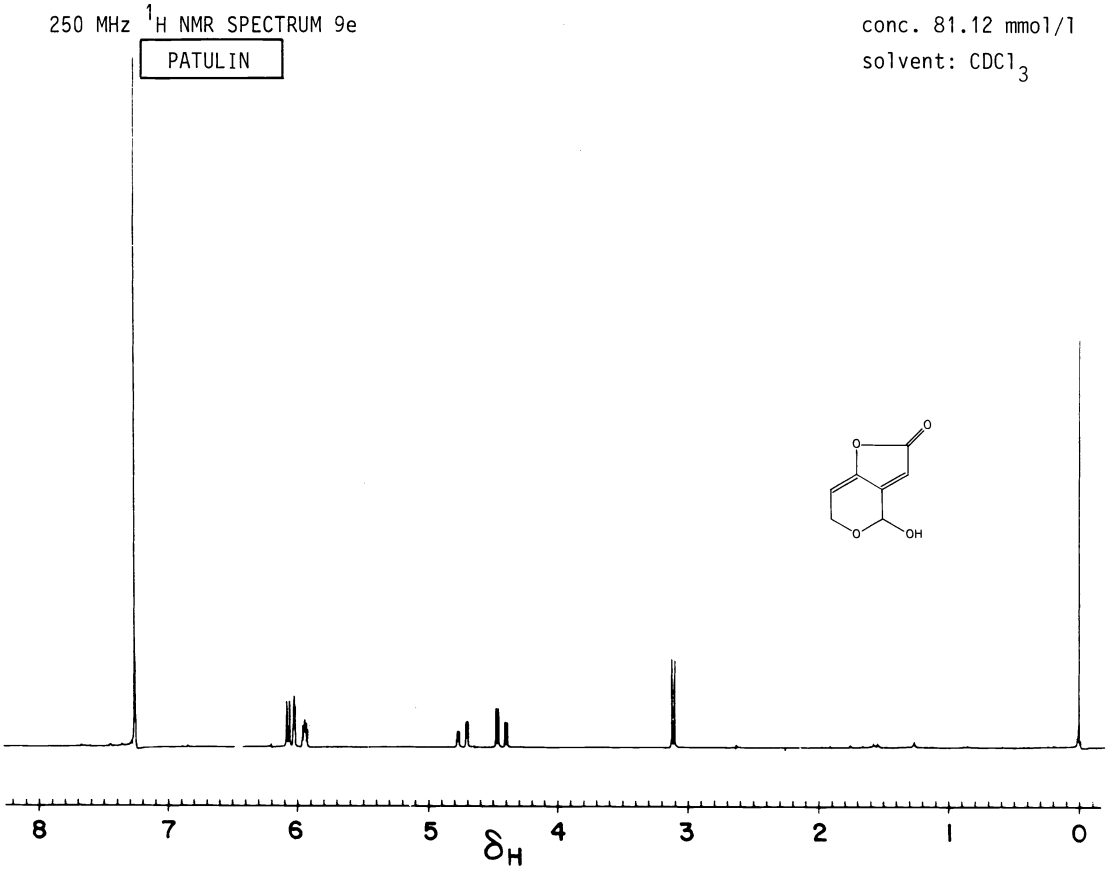
m/z	110.0	55.0	82.1	136.0	126.0
Intensity	100.00	89.26	52.58	43.02	41.37
m/z	53.0	43.0	27.1	153.9	84.1
Intensity	39.65	37.22	36.44	24.52	22.41

Formula



Experimental conditions: see p 2223


100 MHz ^{1}H NMR SPECTRUM 9d
PATULIN
conc. 81.12 mmol/l
solvent: $CDCl_3$
O
O
O
OH
10
9
8
7
6
5
4
3
2
1
0
δ_H


250 MHz ^{1}H NMR SPECTRUM 9e
PATULIN
conc. 81.12 mmol/l
solvent: $CDCl_3$
O
O
O
OH
8
7
6
5
4
3
2
1
0
δ_H

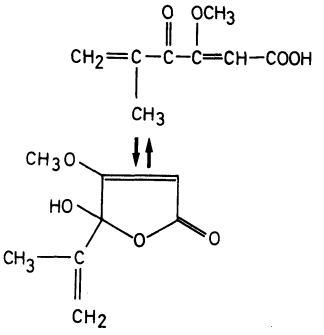
IV.10.PENICILLIC ACID

I SYNONYMS: penicillic acid

II CHEMICAL NAME: 3-methoxy-5-methyl-4-oxo-2,5-Hexadienoic acid

III EMPIRICAL FORMULA: $C_8H_{10}O_4$

IV STRUCTURAL FORMULA:



V MOLECULAR WEIGHT: 170.1

VI DESCRIPTION: Penicillic acid is a white, odourless, crystalline solid

VII CHARACTERIZATION DATA:

1.Melting range: 83-86°C, after drying for 1 hour at 60°C

2.Specific rotation: no specific rotation

3.Circular dichroism: No Cotton effects

4.Ultraviolet absorption spectrum:

see spectrum 10a

Molar absorption coefficients:

$\varepsilon_{(\lambda 224)} = 1063$

conc. 55.13 µmol/l

solvent: methanol

5.Infrared absorption spectrum:

see spectrum 10b

6.Electron impact mass spectrum:

see spectrum 10c

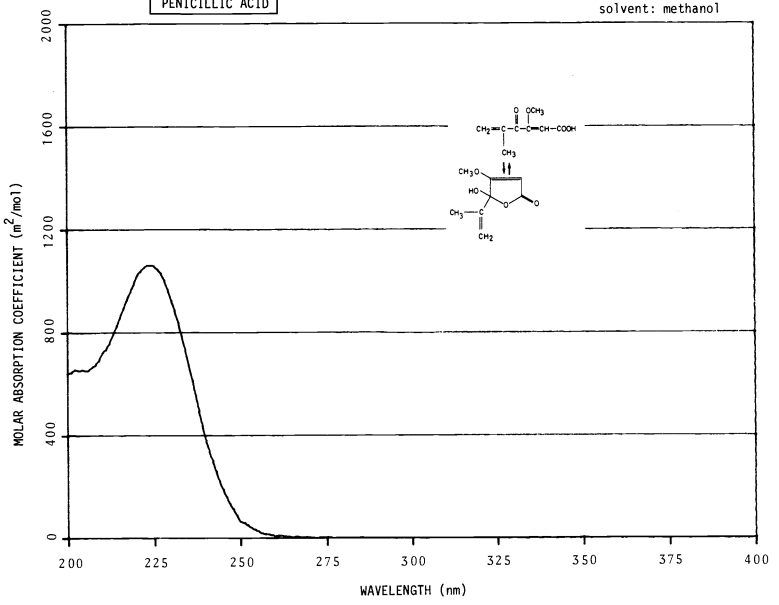
7.Nuclear Magnetic Resonance spectrum:

see spectra 10d and 10e

ULTRAVIOLET ABSORPTION SPECTRUM 10a

PENICILLIC ACID

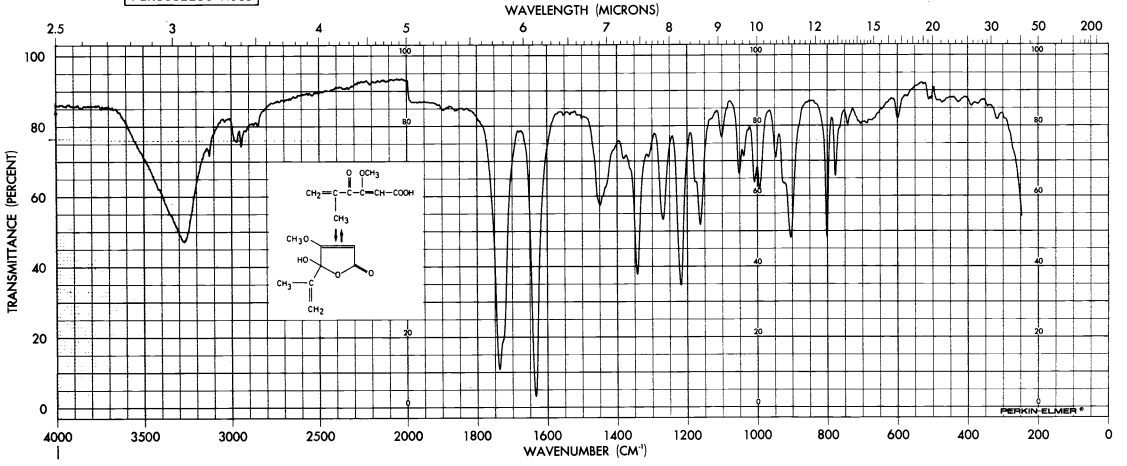
conc. 55.13 µmol/l
solvent: methanol



INFRARED ABSORPTION SPECTRUM 10b

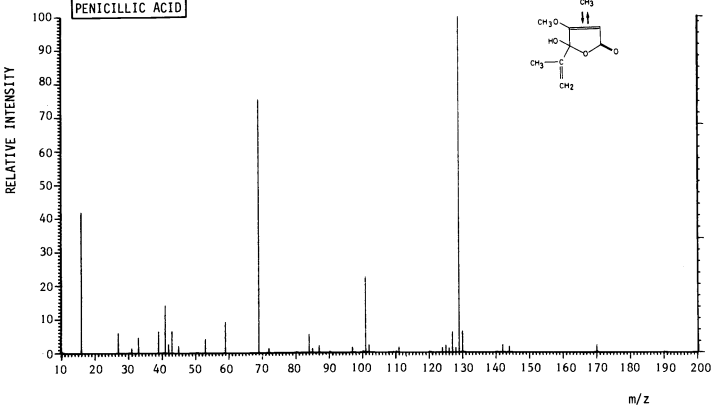
PENICILLIC ACID

52%



MASS SPECTRUM 10c

PENICILLIC ACID



MASS SPECTRUM 10c

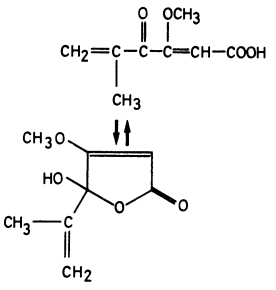
PENICILLIC ACID

relative intensity	mass
5.95%	27.0
1.48%	31.0
4.60%	33.0
6.44%	39.0
14.21%	41.0
2.53%	42.0
6.47%	43.0
2.04%	45.0
4.18%	53.0
9.19%	59.0
75.54%	69.0
1.39%	72.0
5.44%	84.0
1.23%	85.0
2.03%	87.0
1.43%	97.0
22.63%	101.0
2.26%	102.0
1.55%	111.0
1.32%	124.0
2.02%	125.0
1.25%	126.0
6.05%	127.0
1.33%	128.0
100.00%	129.0
6.38%	130.0
2.26%	142.0
1.75%	144.0
2.12%	170.0

Most abundant peaks

m/z	129.0	69.0	101.0	41.0	59.0
Intensity	100.00	75.54	22.63	14.21	9.19
m/z	43.0	39.0	130.0	127.0	27.0
Intensity	6.47	6.44	6.38	6.05	5.95

Formula



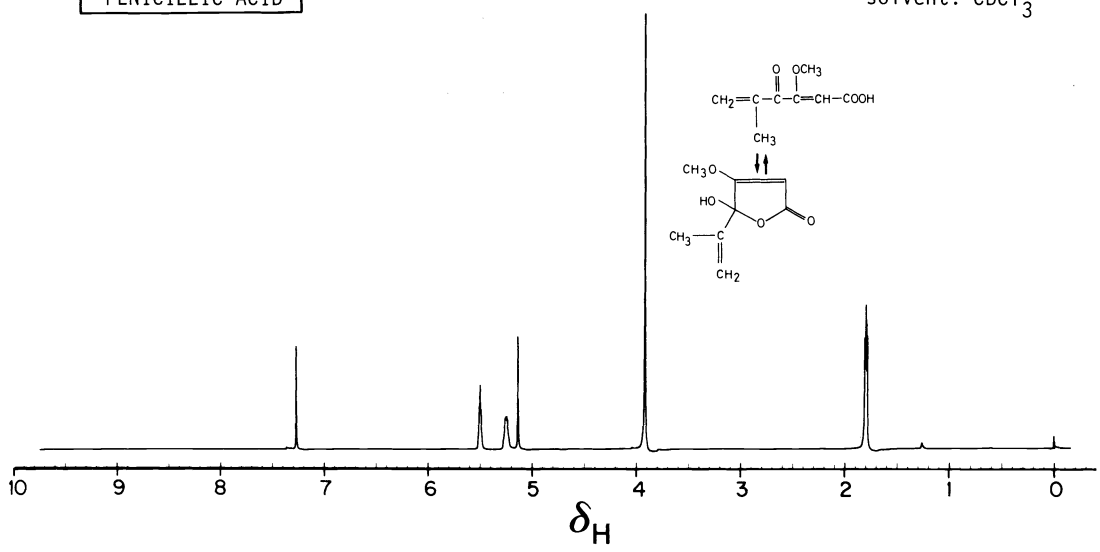
Experimental conditions: see p 2223

100 MHz ^{1}H NMR SPECTRUM 10d

PENICILLIC ACID

conc. 73.49 mmol/l

solvent: $CDCl_3$

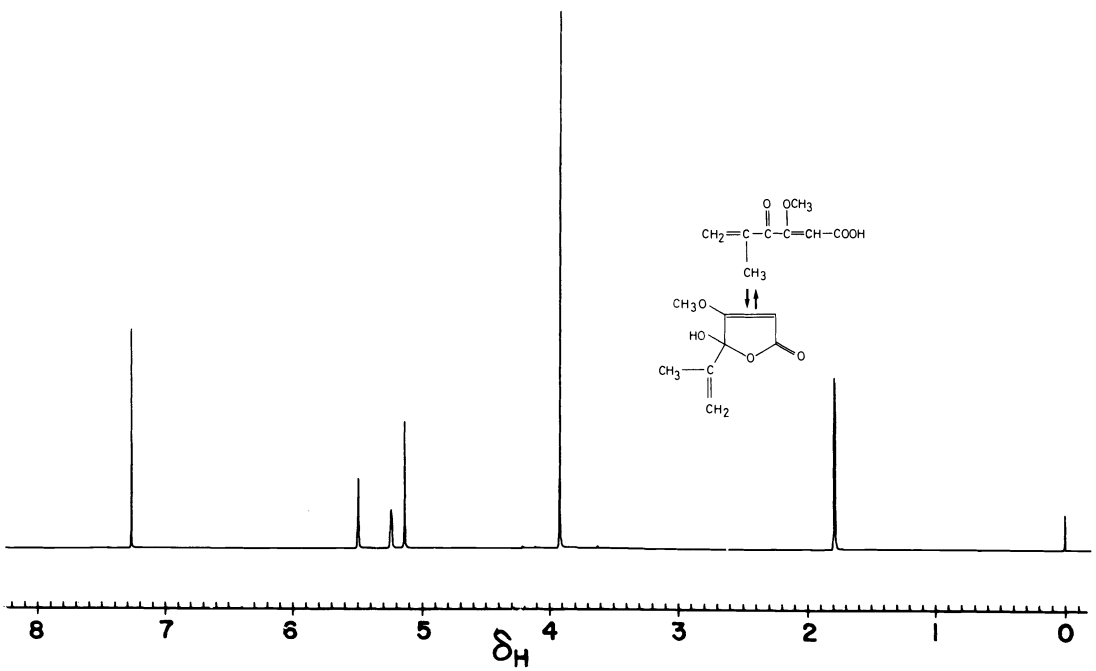


250 MHz ^{1}H NMR SPECTRUM 10e

PENICILLIC ACID

conc. 73.49 mmol/l

solvent: $CDCl_3$



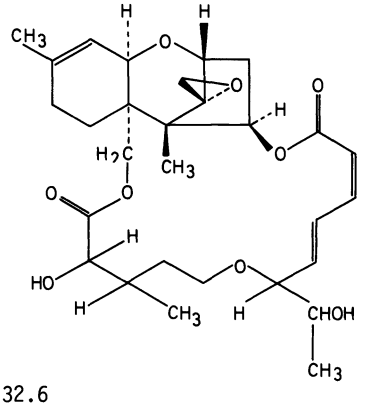
IV.11.RORIDIN A

I SYNONYMS: 7'-deoxo-7'-(1-hydroxyethyl)-verrucarin A, roridan A

II CHEMICAL NAME: 7'-deoxo-7'-(1-hydryethyl)-(stereoisomer of 4,5,6,7,16,16a,19a,22-octahydro-4-hydroxy-5,16a,21-trimethylspiro[16,18-methano-1H,3H,23H-[1,6,12]trioxacyclooctadecino[3,4-d][1]benzopyran-17(18H),2'-oxirane]-3,9,14-trione)

III EMPIRICAL FORMULA: $C_{29}H_{40}O_9$

IV STRUCTURAL FORMULA:



V MOLECULAR WEIGHT: 532.6

VI DESCRIPTION: Roridin A is a white, odourless, crystalline solid

VII CHARACTERIZATION DATA:

1.Melting range: 199-201°C, after drying for 1 hour at 60°C

2.Specific rotation: $[\alpha]_D^{21} = +126.0^\circ$
conc. 1878 µmol/l
solvent: chloroform

3.Circular dichroism:

$\Delta\varepsilon_{(\lambda 300)}$ 0, $\Delta\varepsilon_{(\lambda 261)}$ +19.90, $\Delta\varepsilon_{(\lambda 222)}$ 0
conc. 187.8 µmol/l
solvent: methanol
temperature: 22°C
cell length: 2 mm

4.Ultraviolet absorption spectrum:
see spectrum 11a

Molar absorption coefficients:

$\varepsilon_{(\lambda 217)} = 418 \pm 11$
$\varepsilon_{(\lambda 263)} = 2200 \pm 5$
conc. 35.84 µmol/l
solvent: methanol

5.Infrared absorption spectrum:
see spectrum 11b

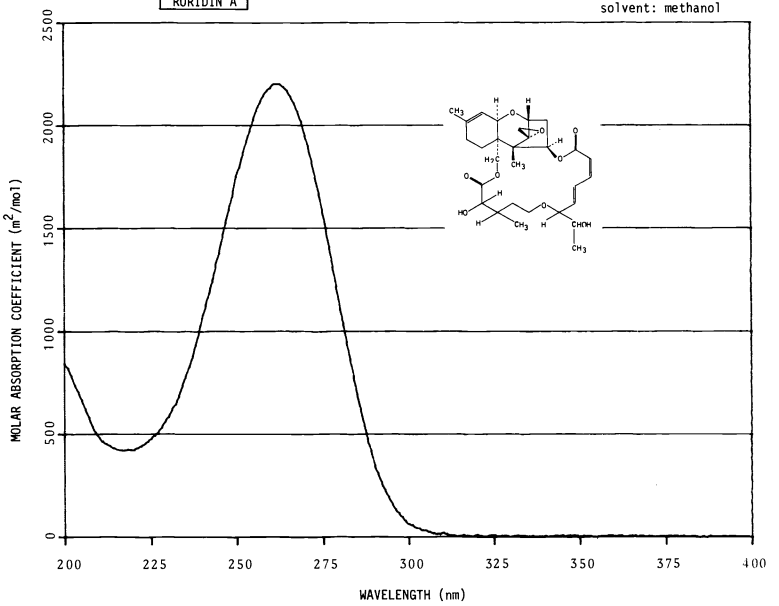
6.Electron impact mass spectrum:
see spectrum 11c

7.Nuclear Magnetic Resonance spectrum:
see spectra 11d and 11e

ULTRAVIOLET ABSORPTION SPECTRUM 11a

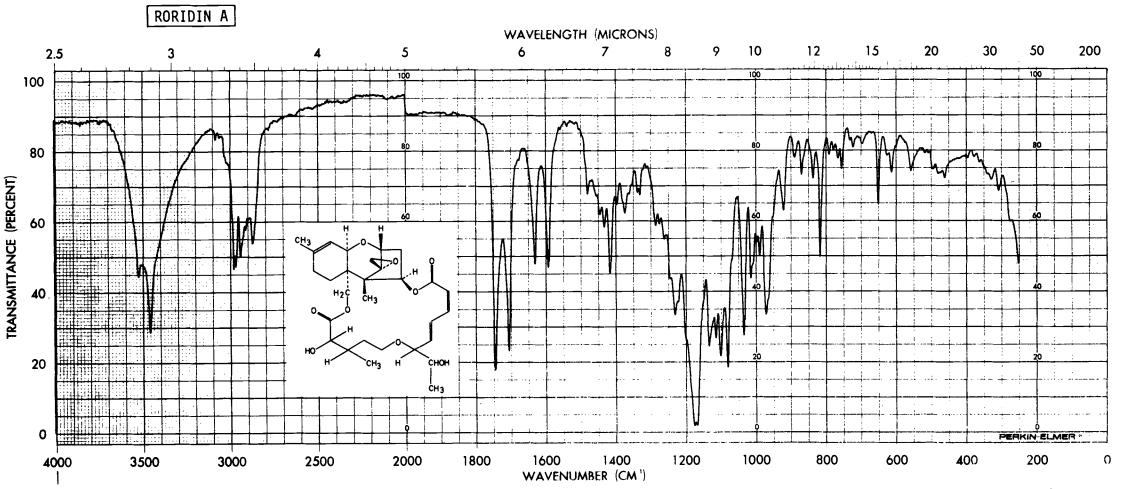
RORIDIN A

conc. 35.84 µmol/l
solvent: methanol



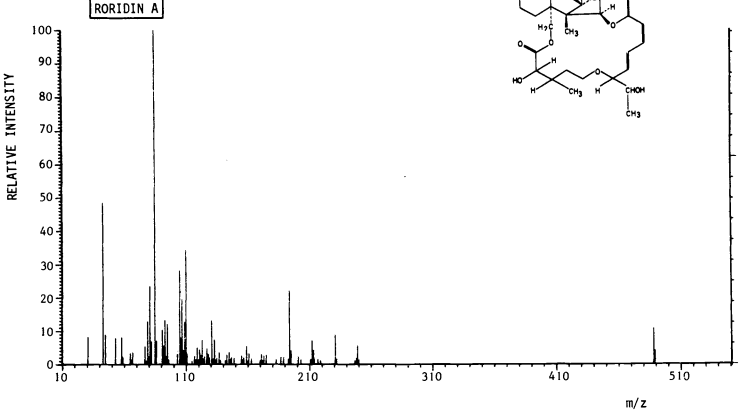
INFRARED ABSORPTION SPECTRUM 11b

RORIDIN A



MASS SPECTRUM 11c

RORIDIN A



MASS SPECTRUM 11c

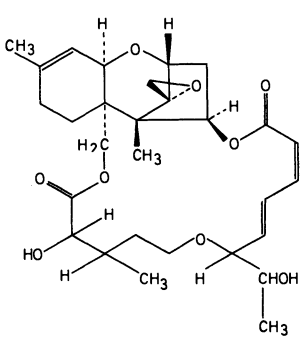
RORIDIN A

relative intensity	mass	relative intensity	mass	relative intensity	mass
8.27%	31.0	1.74%	132.0	4.10%	489.0
48.56%	43.0	7.38%	133.0		
9.08%	45.0	1.41%	134.0		
8.08%	53.0	1.93%	135.0		
8.19%	58.0	3.58%	137.0		
2.23%	59.0	1.31%	138.0		
3.36%	65.0	1.22%	142.0		
1.49%	66.0	2.86%	143.0		
3.53%	67.0	3.65%	145.0		
5.41%	77.0	1.53%	146.0		
1.14%	78.0	2.09%	147.0		
12.87%	79.0	1.83%	149.0		
2.48%	80.0	2.51%	155.0		
23.51%	81.0	1.46%	156.0		
6.87%	82.0	1.97%	157.0		
100.00%	85.0	5.46%	159.0		
7.07%	86.0	1.10%	160.0		
10.38%	91.0	3.28%	161.0		
5.60%	92.0	1.72%	163.0		
13.38%	93.0	1.23%	170.0		
2.57%	94.0	2.90%	171.0		
12.11%	95.0	1.20%	172.0		
1.57%	96.0	2.54%	173.0		
3.18%	103.0	2.84%	175.0		
28.14%	105.0	1.40%	183.0		
8.08%	106.0	2.19%	187.0		
19.54%	107.0	2.04%	189.0		
4.31%	108.0	1.55%	193.0		
12.76%	109.0	22.08%	194.0		
34.40%	110.0	4.15%	195.0		
3.36%	111.0	2.20%	201.0		
1.02%	115.0	1.38%	203.0		
2.55%	117.0	1.55%	211.0		
1.57%	118.0	7.01%	212.0		
5.00%	119.0	4.21%	213.0		
1.58%	120.0	1.79%	214.0		
4.49%	121.0	1.54%	217.0		
2.87%	122.0	1.18%	219.0		
7.40%	123.0	8.86%	231.0		
1.77%	124.0	1.60%	232.0		
2.37%	125.0	1.09%	247.0		
4.86%	127.0	1.75%	248.0		
3.18%	128.0	5.60%	249.0		
2.16%	129.0	1.23%	250.0		
13.19%	131.0	10.61%	488.0		

Most abundant peaks

m/z	85.0	43.0	110.0	105.0	81.0
Intensity	100.00	48.56	34.40	28.14	23.51
m/z	194.0	107.0	93.0	131.0	79.0
Intensity	22.08	19.54	13.38	13.19	12.87

Formula



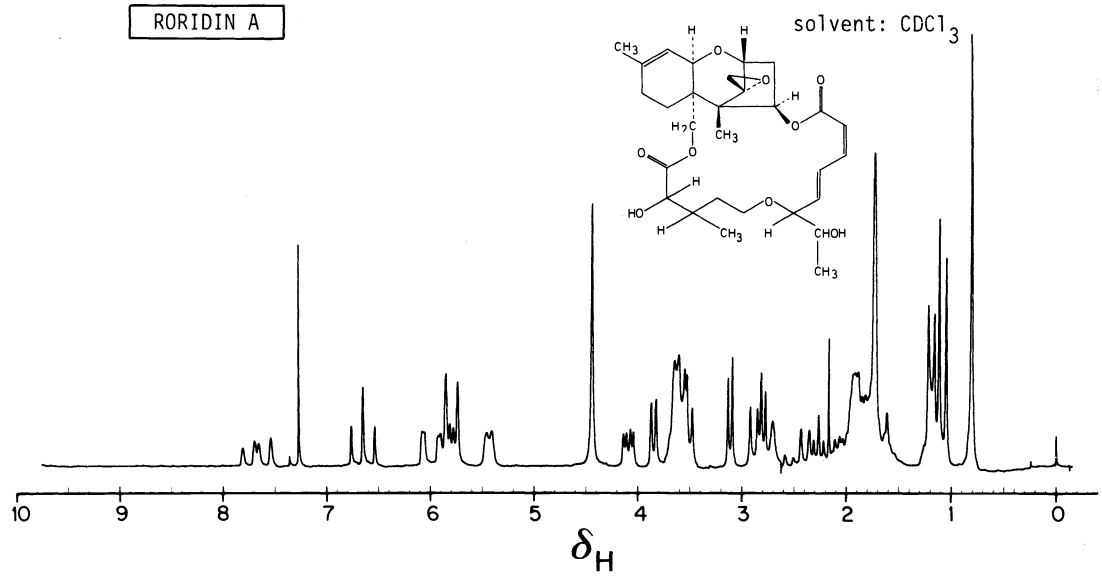
Experimental conditions: see p 2223

100 MHz ^{1}H NMR SPECTRUM 11d

RORIDIN A

conc. 23.47 mmol/l

solvent: $CDCl_3$

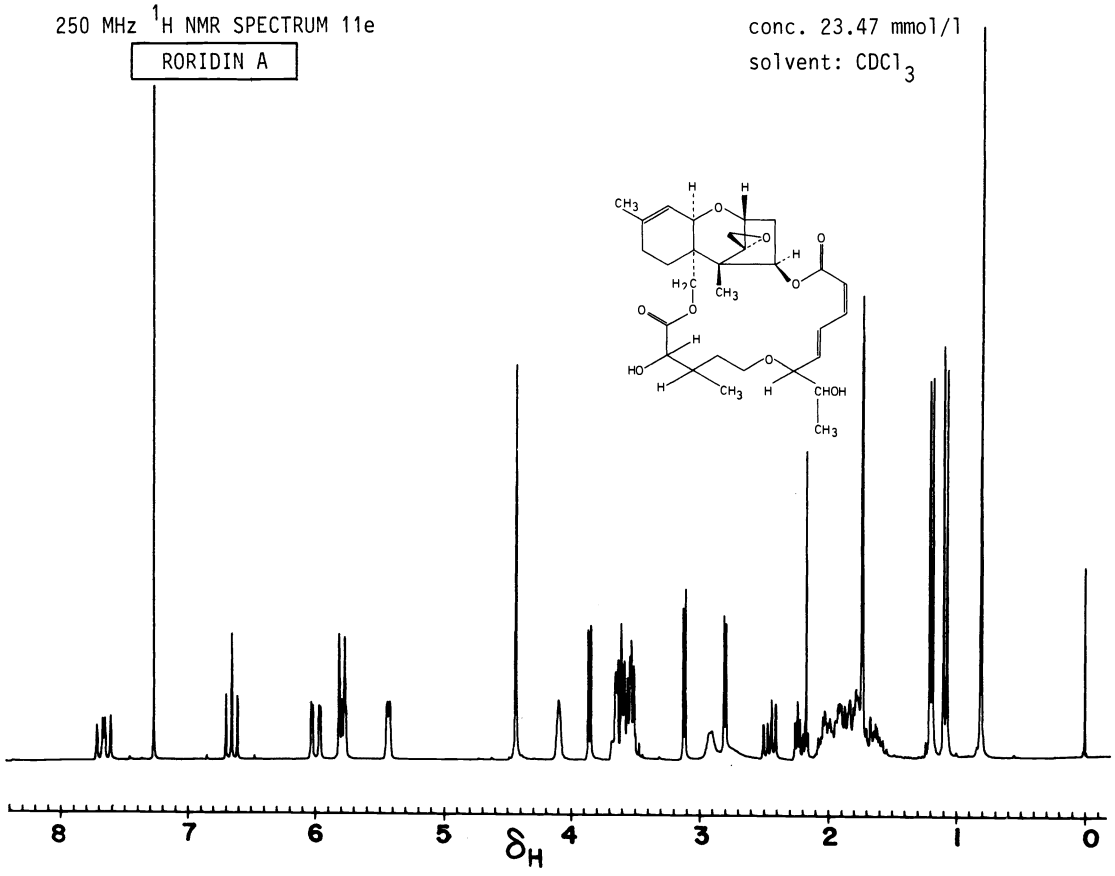


250 MHz ^{1}H NMR SPECTRUM 11e

RORIDIN A

conc. 23.47 mmol/l

solvent: $CDCl_3$



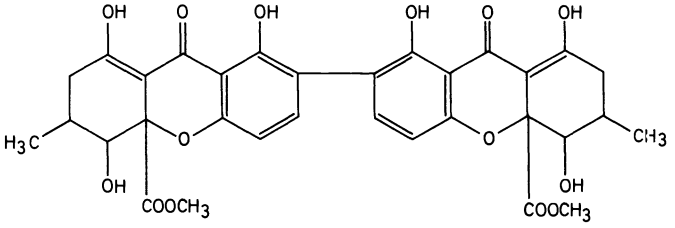
IV.12.SECALONIC ACID D

I SYNONYMS: none

II CHEMICAL NAME: dimethylester of 2,2', 3,3', 4,4', 9,9'-octahydro-1,1', 4,4', 8,8'-hexahydroxy-3,3'-dimethyl-9,9'-dioxo-[7,7'-bi-4aH-xanthene]-4a,4'a-dicarboxylic acid

III EMPIRICAL FORMULA: $C_{32}H_{30}O_{14}$

IV STRUCTURAL FORMULA:



V MOLECULAR WEIGHT: 638.6

VI DESCRIPTION: Secalonic acid D is a yellow, odourless, crystalline solid

VII CHARACTERIZATION DATA:

1.Melting range: 230-240°C, after drying for 1 hour at 60°C

2.Specific rotation: $[\alpha]_D^{21} = +77.9^o$
conc. 1566 µmol/l
solvent: chloroform

3.Circular dichroism:
$\Delta\varepsilon_{(\lambda 400)}$ 0, $\Delta\varepsilon_{(\lambda 370)}$ +3.50, $\Delta\varepsilon_{(\lambda 340)}$ +2.20, $\Delta\varepsilon_{(\lambda 330)}$ +3.06, $\Delta\varepsilon_{(\lambda 287)}$ 0,
$\Delta\varepsilon_{(\lambda 269)}$ -2.63, $\Delta\varepsilon_{(\lambda 263)}$ -2.85, $\Delta\varepsilon_{(\lambda 224)}$ -12.7, $\Delta\varepsilon_{(\lambda 210)}$ 0 .
conc. 172.6 µmol/l
solvent: methanol
temperature: 22°C
cell length: 2 mm

4.Ultraviolet absorption spectrum:
see spectrum 12a

Molar absorption coefficients:
$\varepsilon_{(\lambda 289)} = 929 \pm 28$
$\varepsilon_{(\lambda 338)} = 3585 \pm 52$
conc. 21.33 µmol/l
solvent: methanol

5.Infrared absorption spectrum:
see spectrum 12b

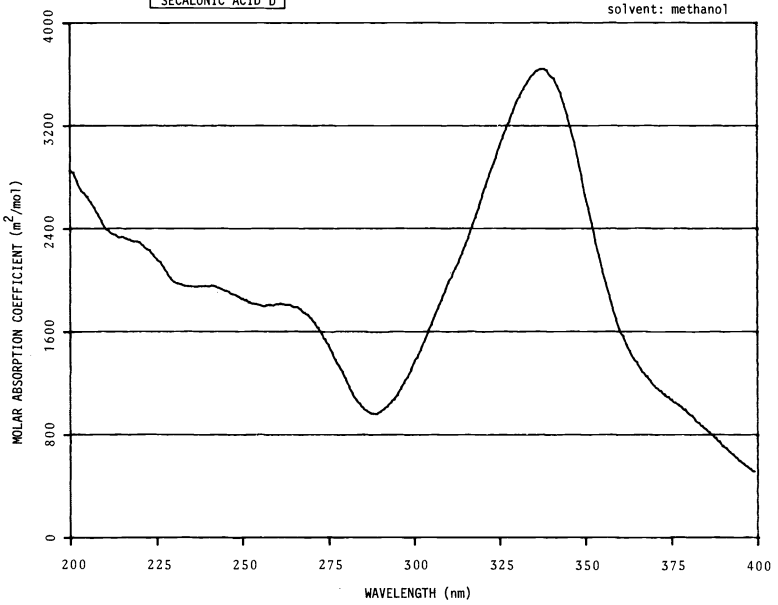
6.Electron impact mass spectrum:
see spectrum 12c

7.Nuclear Magnetic Resonance spectrum:
see spectra 12d and 12e

ULTRAVIOLET ABSORPTION SPECTRUM 12a

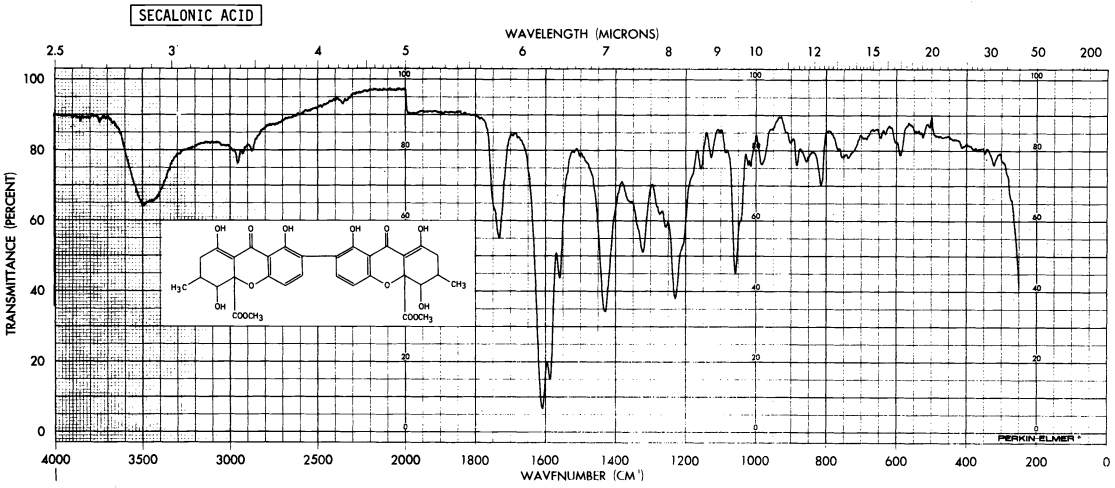
SECALONIC ACID D

conc. 21.33 µmol/l
solvent: methanol



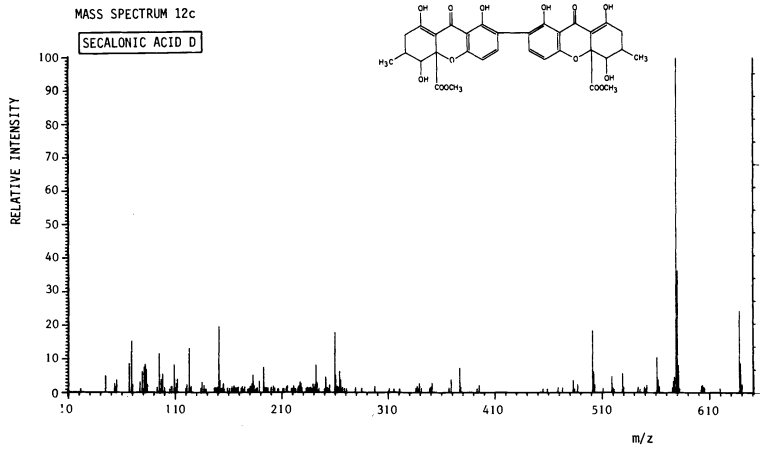
INFRARED ABSORPTION SPECTRUM 12b

SECALONIC ACID



MASS SPECTRUM 12c

SECALONIC ACID D



MASS SPECTRUM 12c

SECALONIC ACID D

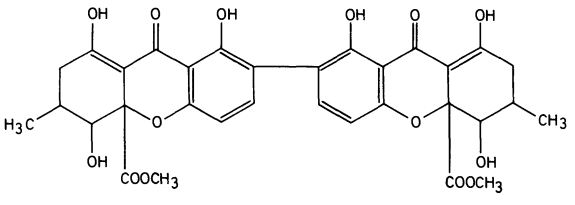
relative intensity	mass	relative intensity	mass	relative intensity	mass	relative intensity	mass	relative intensity	mass
1.25%	21.8	2.70%	155.1	2.01%	226.0	1.73%	350.0	8.75%	638.9
5.06%	44.9	1.04%	156.1	3.26%	227.0	2.84%	351.0	2.50%	640.1
2.70%	53.1	1.45%	159.1	2.77%	227.9	1.45%	367.1		
1.66%	54.1	1.31%	161.1	1.38%	228.9	3.95%	368.9		
3.81%	55.1	1.80%	163.1	1.38%	232.9	7.36%	376.9		
8.68%	67.0	1.11%	164.2	1.59%	234.1	1.73%	377.9		
15.34%	69.1	2.01%	165.1	1.11%	234.9	1.25%	393.1		
2.50%	70.0	1.59%	166.2	1.45%	236.1	2.29%	395.0		
3.26%	76.9	1.11%	167.0	1.59%	237.1	1.25%	454.9		
6.38%	79.1	1.66%	168.1	1.04%	238.1	1.11%	458.9		
1.11%	79.9	1.66%	169.1	2.50%	239.2	1.52%	468.9		
7.63%	81.0	1.38%	172.1	1.45%	240.1	1.45%	472.9		
8.47%	81.9	1.80%	173.0	2.43%	241.0	3.68%	482.9		
6.94%	83.0	1.04%	173.9	8.19%	242.0	1.11%	484.1		
2.50%	83.9	1.73%	175.0	2.84%	242.5	2.43%	487.1		
1.66%	93.1	1.18%	178.1	2.84%	243.0	18.40%	501.0		
11.66%	95.1	1.18%	179.0	1.25%	244.9	6.25%	502.1		
1.11%	96.0	1.87%	180.0	1.18%	250.1	2.43%	502.8		
4.09%	97.1	1.94%	181.1	4.79%	251.0	1.45%	510.9		
5.62%	98.1	2.63%	182.0	1.18%	251.6	4.86%	518.9		
1.66%	100.1	5.20%	183.0	1.52%	252.0	1.59%	519.9		
1.18%	105.1	2.29%	184.0	1.31%	252.9	1.11%	521.0		
1.94%	107.1	1.25%	185.9	2.22%	254.9	5.76%	528.9		
8.33%	109.1	1.52%	186.9	17.84%	259.9	1.66%	529.9		
1.31%	110.1	3.40%	188.8	5.13%	260.5	1.73%	543.1		
2.70%	111.1	7.50%	192.9	1.94%	262.1	1.04%	545.1		
4.09%	112.1	1.66%	193.9	1.45%	263.1	1.73%	549.3		
1.38%	120.1	1.31%	194.9	6.38%	264.3	1.11%	550.3		
2.43%	121.1	1.59%	195.9	3.81%	265.2	2.22%	551.4		
13.12%	123.1	1.45%	196.9	1.11%	266.1	10.48%	561.1		
1.45%	124.1	1.66%	200.1	1.52%	267.1	3.95%	562.1		
1.94%	125.1	1.94%	201.9	1.31%	269.0	1.87%	563.1		
1.38%	134.1	1.31%	203.1	1.11%	270.9	1.59%	575.4		
3.19%	135.1	1.04%	206.0	1.59%	279.1	3.40%	576.3		
2.29%	137.1	1.18%	207.1	1.31%	285.2	4.65%	577.4		
1.11%	138.1	1.31%	211.0	1.87%	297.2	100.00%	579.0		
1.18%	139.1	1.11%	212.0	1.25%	311.0	36.31%	580.1		
1.59%	147.0	1.66%	214.1	1.04%	315.3	8.12%	581.0		
1.52%	148.1	2.08%	215.1	1.11%	320.9	1.25%	582.1		
1.31%	149.1	1.25%	219.1	1.31%	336.0	2.01%	602.4		
1.73%	150.1	1.11%	220.0	1.80%	337.0	2.22%	603.4		
19.65%	151.1	2.01%	221.0	1.18%	338.1	1.66%	604.4		
3.61%	152.1	1.25%	222.1	2.77%	339.2	1.31%	605.5		
1.25%	153.1	1.18%	223.0	1.31%	341.2	1.31%	619.9		
1.38%	154.1	1.66%	225.0	1.38%	348.9	24.37%	637.9		

Most abundant peaks

m/z	579.0	580.1	637.9	151.1	501.0
Intensity	100.00	36.31	24.37	19.65	18.40
m/z	259.9	69.1	123.1	95.1	561.1
Intensity	17.84	15.34	13.12	11.66	10.48

Formula

Experimental conditions: see p 2223

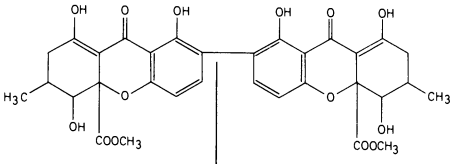


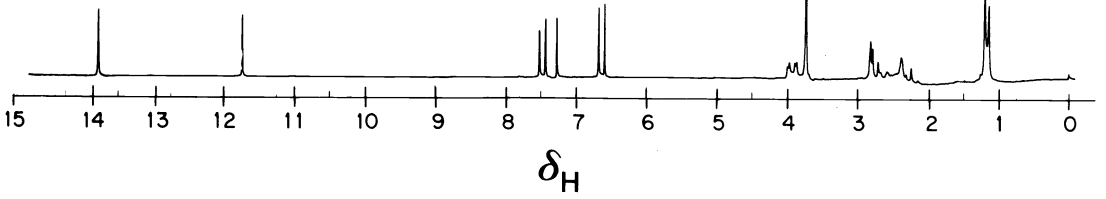
100 MHz ^{1}H NMR SPECTRUM 12d

SECALONIC ACID D

conc. 19.57 mmol/l

solvent: $CDCl_3$





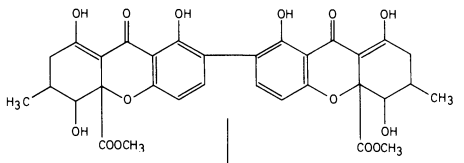
δ_H

250 MHz ^{1}H NMR SPECTRUM 12e

SECALONIC ACID D

conc. 19.57 mmol/l

solvent: $CDCl_3$



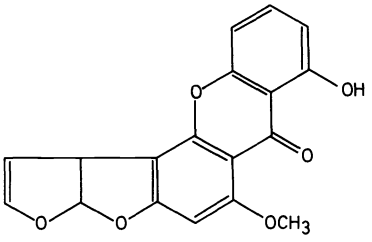
IV.13. STERIGMATOCYSTIN

I SYNONYMS: none

II CHEMICAL NAME: 3a,12c-dihydro-8-hydroxy-6-methoxy-(3aR-cis)-7H-Furo[3',2':4,5]furo[2,3-c]xanthen-7-one

III EMPIRICAL FORMULA: $C_{18}H_{12}O_6$

IV STRUCTRUAL FORMULA:



V MOLECULAR WEIGHT: 324.3

VI DESCRIPTION: Sterigmatocystin is a yellow, odourless, crystalline solid

VII CHARACTERIZATION DATA:

1. Melting range: 242-244°C, after drying for 1 hour at 60°C
2. Specific rotation: $[\alpha]_D^{21} = -369.0^o$
   conc. 3084 µmol/l
   solvent: chloroform
3. Circular dichroism:
   $\Delta\varepsilon_{(\lambda 375)}$ 0, $\Delta\varepsilon_{(\lambda 325)}$ -2.58, $\Delta\varepsilon_{(\lambda 297)}$ 0, $\Delta\varepsilon_{(\lambda 290)}$ +1.42, $\Delta\varepsilon_{(\lambda 280)}$ 0.90, $\Delta\varepsilon_{(\lambda 270)}$ +1.42, $\Delta\varepsilon_{(\lambda 263)}$ 0.
   conc. 2923 µmol/l
   solvent: methanol
   temperature: 22°C
   cell length: 2 mm
4. Ultraviolet absorption spectrum:
   see spectrum 13a

   Molar absorption coefficients:

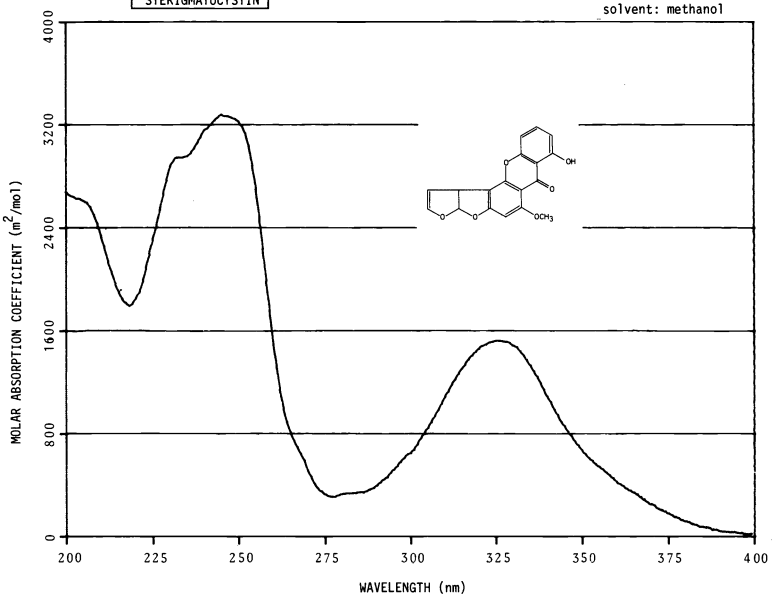
   $\varepsilon_{(\lambda 218)} = 1842 \pm 60$
   $\varepsilon_{(\lambda 246)} = 3287 \pm 13$
   $\varepsilon_{(\lambda 277)} = 304 \pm 10$
   $\varepsilon_{(\lambda 326)} = 1531 \pm 7$

   conc. 27.12 µmol/l
   solvent: methanol
5. Infrared absorption spectrum:
   see spectrum 13b
6. Electron impact mass spectrum:
   see spectrum 13c
7. Nuclear Magnetic Resonance spectrum:
   see spectra 13d and 13e

ULTRAVIOLET ABSORPTION SPECTRUM 13a

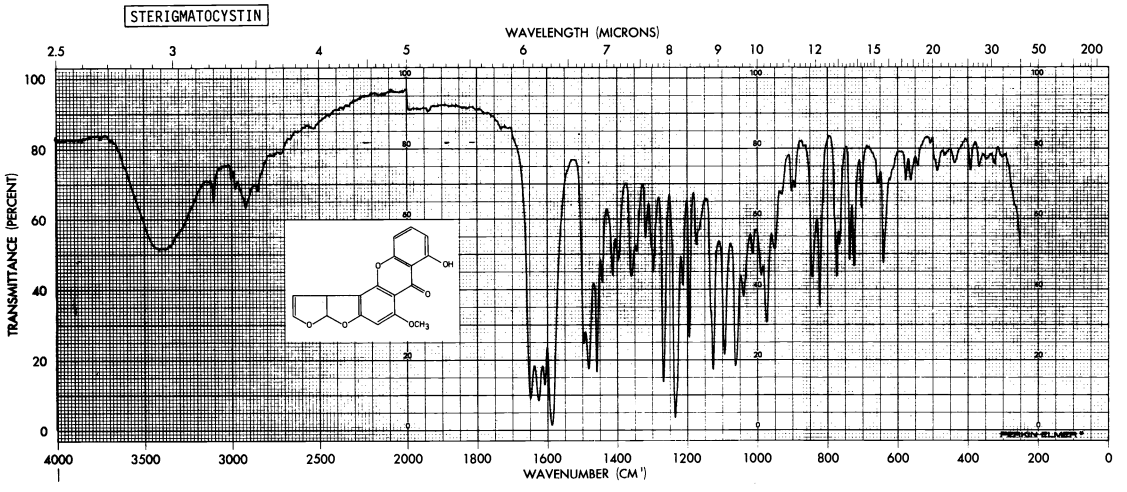
STERIGMATOCYSTIN

conc. 27.12 µmol/l
solvent: methanol



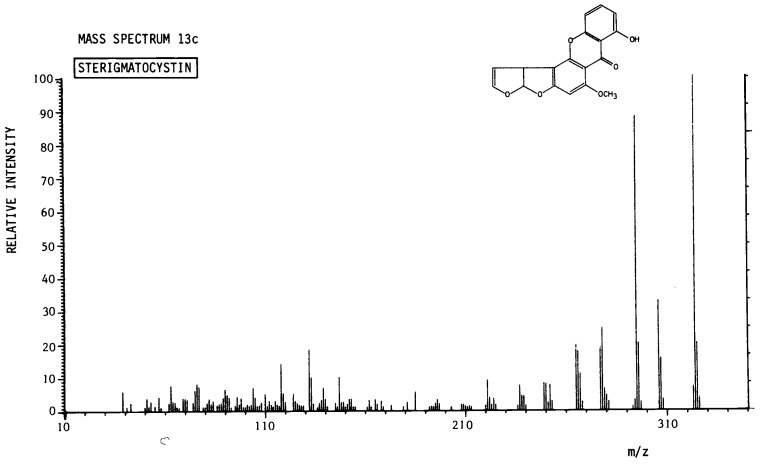
INFRARED ABSORPTION SPECTRUM 13b

STERIGMATOCYSTIN



MASS SPECTRUM 13c

STERIGMATOCYSTIN



MASS SPECTRUM 13c

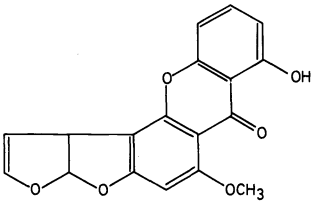
STERIGMATOCYSTIN

relative intensity	mass	relative intensity	mass	relative intensity	mass	relative intensity	mass
5.99%	39.0	6.99%	104.0	2.07%	166.0	3.16%	294.0
1.35%	41.0	4.06%	105.0	2.90%	168.0	88.17%	295.0
2.51%	43.0	1.46%	106.0	1.30%	169.0	20.12%	296.0
1.31%	50.0	1.71%	107.0	1.68%	173.0	2.57%	297.0
3.86%	51.0	2.59%	108.0	1.23%	179.0	32.92%	306.0
1.36%	52.0	5.08%	110.0	2.58%	181.0	15.66%	307.0
2.87%	53.0	1.58%	111.0	5.63%	185.0	3.44%	308.0
1.58%	55.0	3.06%	112.0	1.17%	192.0	7.13%	323.0
4.24%	57.0	1.87%	113.0	1.30%	193.0	100.00%	324.0
1.15%	58.0	1.38%	114.0	1.23%	194.0	20.20%	325.0
2.41%	62.0	3.01%	115.0	2.21%	195.0	3.86%	326.0
7.69%	63.0	1.93%	116.0	3.40%	196.0		
2.93%	64.0	1.53%	117.0	2.11%	197.0		
2.69%	65.0	14.11%	118.0	1.22%	203.0		
1.24%	66.0	5.33%	119.0	1.91%	208.0		
1.03%	67.0	2.71%	120.0	1.83%	209.0		
3.84%	69.0	5.21%	124.0	1.38%	210.0		
3.84%	70.0	2.96%	125.0	1.19%	211.0		
3.51%	71.0	2.32%	126.0	1.53%	212.0		
2.56%	74.0	1.75%	127.0	1.28%	213.0		
6.24%	75.0	1.68%	128.0	1.66%	220.0		
8.14%	76.0	1.69%	129.0	9.19%	221.0		
7.30%	77.0	18.31%	132.0	3.98%	222.0		
1.21%	79.0	10.05%	133.0	1.87%	223.0		
1.25%	80.0	2.24%	134.0	3.64%	224.0		
2.44%	81.0	1.07%	136.0	1.82%	225.0		
3.60%	82.0	2.48%	137.0	1.55%	236.0		
2.14%	83.0	3.38%	138.0	7.55%	237.0		
2.99%	84.0	6.91%	139.0	4.44%	238.0		
1.73%	86.0	3.65%	140.0	4.43%	239.0		
2.15%	87.0	1.34%	141.0	1.57%	240.0		
2.49%	88.0	2.35%	145.0	8.13%	249.0		
3.93%	89.0	1.30%	146.0	8.05%	250.0		
6.55%	90.0	10.17%	147.0	2.42%	251.0		
4.86%	91.0	2.57%	148.0	7.66%	252.0		
4.07%	92.0	2.66%	149.0	2.84%	253.0		
1.11%	93.0	1.26%	150.0	19.66%	265.0		
1.64%	95.0	2.08%	151.0	17.77%	266.0		
4.21%	96.0	3.52%	152.0	11.09%	267.0		
2.09%	97.0	3.63%	153.0	2.67%	268.0		
3.82%	98.0	1.25%	154.0	18.98%	277.0		
1.22%	99.0	1.26%	155.0	24.61%	278.0		
1.18%	100.0	1.15%	161.0	6.69%	279.0		
1.99%	101.0	3.22%	162.0	4.60%	280.0		
1.56%	102.0	1.20%	163.0	2.70%	281.0		
1.90%	103.0	3.49%	165.0	1.24%	293.0		

Most abundant peaks

m/z	324.0	295.0	306.0	278.0	325.0
Intensity	100.00	88.17	32.92	24.61	20.20
m/z	296.0	265.0	277.0	132.0	266.0
Intensity	20.12	19.66	18.98	18.31	17.77

Formula



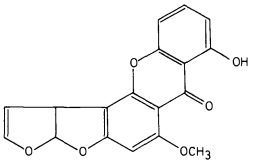
Experimental conditions: see p 2223

100 MHz ^{1}H NMR SPECTRUM 13d

STERIGMATOCYSTIN

conc. 38.54 mmol/l

solvent: $CDCl_3$

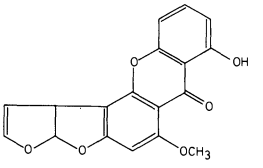


250 MHz ^{1}H NMR SPECTRUM 13e

STERIGMATOCYSTIN

conc. 38.54 mmol/l

solvent: $CDCl_3$



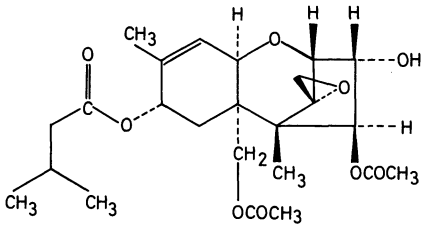
IV.14.T-2 TOXIN

I SYNONYMS: 8-(3-methylbutyryloxy)-diacetoxyscirpenol, fusariotoxin T-2, insariotoxin, T-2 mycotoxin

II CHEMICAL NAME: 12,13-epoxy-,4,15-diacetate 8-(3-methylbutanoate),(3α,4β,8α)-Trichothec-9-ene-3,4,8,15-tetrol

III EMPIRICAL FORMULA: $C_{24}H_{34}O_9$

IV STRUCTURAL FORMULA:



V MOLECULAR WEIGHT: 466.5

VI DESCRIPTION: T-2 toxin is a white, odourless, crystalline solid

VII CHARACTERIZATION DATA:

1.Melting range: 145-149°C, after drying for 1 hour at 60°C

2.Specific rotation: $[\alpha]_D^{21} = -15.5^\circ$
conc. 2144 µmol/l
solvent: chloroform

3.Circular dichroism: No Cotton effects

4.Ultraviolet absorption spectrum:
see spectrum 14a

No absorption
conc. 169.4 µmol/l
solvent: methanol

5.Infrared absorption spectrum:
see spectrum 14b

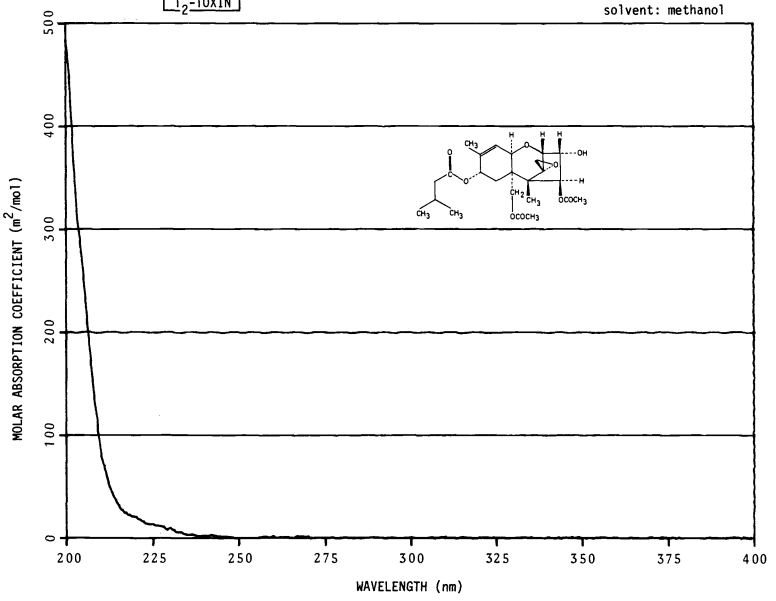
6.Electron impact mass spectrum:
see spectrum 14c

7.Nuclear Magnetic Resonance spectrum:
see spectra 14d and 14e

ULTRAVIOLET ABSORPTION SPECTRUM 14a

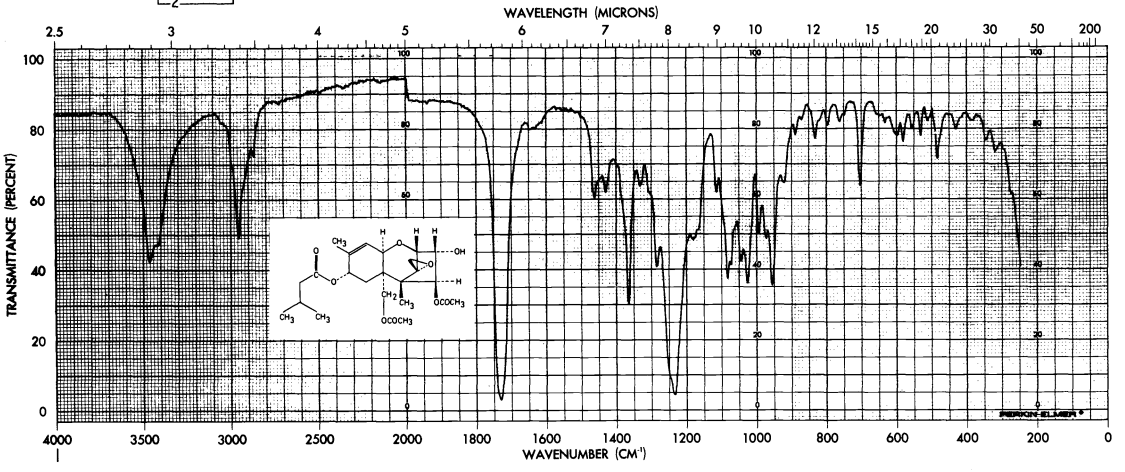
T_2-TOXIN

conc. 169.4 µmol/l
solvent: methanol



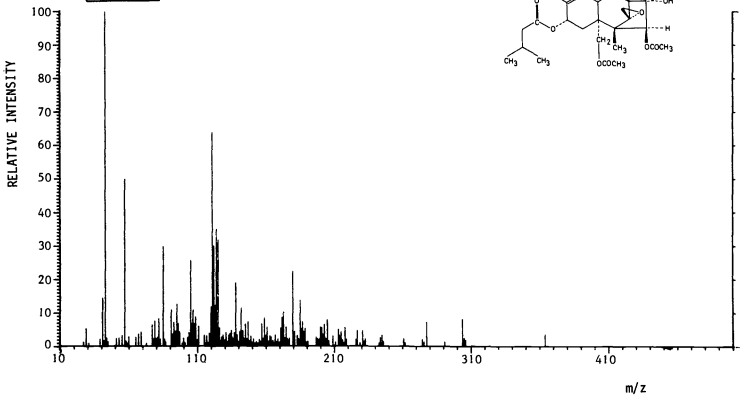
INFRARED ABSORPTION SPECTRUM 14b

T_2-TOXIN



MASS SPECTRUM 14c

T-2 TOXIN



MASS SPECTRUM 14c

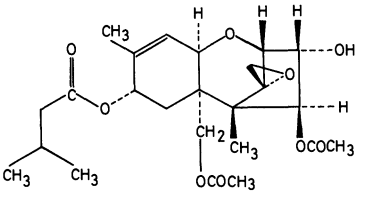
T-2 TOXIN

relative intensity	mass	relative intensity	mass	relative intensity	mass	relative intensity	mass
1.36%	27.1	6.85%	107.9	1.59%	156.1	5.14%	213.2
5.44%	29.1	8.87%	109.0	6.74%	157.1	3.25%	214.3
1.06%	31.0	2.01%	110.1	2.48%	158.1	4.31%	215.1
2.36%	39.1	6.09%	111.0	8.45%	159.1	1.95%	216.1
14.54%	41.0	3.31%	115.1	3.31%	160.1	1.18%	217.3
1.89%	42.1	1.18%	116.0	5.73%	161.1	5.67%	218.1
100.00%	43.0	3.25%	117.0	1.59%	162.1	2.18%	219.2
2.66%	44.0	1.24%	118.1	3.19%	163.1	2.18%	226.1
1.59%	45.0	4.02%	119.0	2.89%	164.0	4.73%	227.1
2.48%	51.1	11.82%	120.0	1.77%	165.1	1.18%	229.1
2.54%	53.1	63.92%	121.1	1.00%	166.1	4.79%	231.0
3.31%	55.1	30.10%	122.1	3.37%	167.0	1.53%	232.1
49.91%	57.1	12.30%	123.1	1.41%	168.0	2.24%	233.1
1.71%	58.1	35.12%	124.1	2.18%	169.0	1.06%	243.1
1.12%	59.1	31.93%	125.1	1.30%	170.1	2.60%	244.1
3.01%	60.1	5.55%	126.1	5.44%	171.1	3.31%	245.1
2.89%	65.1	1.83%	127.0	8.57%	172.0	1.53%	246.1
3.78%	67.1	2.89%	127.9	10.17%	173.0	2.36%	261.1
4.31%	69.1	3.42%	129.0	2.54%	174.0	1.12%	262.1
1.06%	73.1	1.95%	130.1	5.79%	175.0	2.18%	274.9
6.44%	77.1	4.13%	131.0	2.30%	176.0	1.47%	276.0
2.24%	78.1	1.41%	132.0	2.42%	177.1	7.21%	277.9
7.62%	79.1	3.37%	133.0	22.47%	179.9	1.47%	291.0
2.30%	80.1	3.84%	134.1	4.55%	181.1	8.16%	304.1
2.77%	81.1	4.73%	135.0	3.37%	183.1	2.54%	305.3
8.39%	82.1	2.54%	136.0	2.36%	184.0	2.06%	306.2
2.24%	83.1	4.08%	137.1	13.89%	185.1	3.78%	364.1
29.86%	85.1	18.92%	138.1	5.20%	186.1		
2.24%	86.1	3.48%	139.1	7.33%	187.0		
1.41%	87.0	1.47%	140.1	4.49%	188.0		
11.05%	91.1	4.43%	141.1	5.32%	189.0		
3.78%	92.1	11.41%	142.1	1.30%	190.1		
7.27%	93.1	4.67%	143.2	1.53%	190.9		
4.61%	94.1	2.54%	144.1	2.72%	196.9		
12.71%	95.1	6.50%	145.1	2.36%	198.0		
6.91%	96.1	2.48%	146.1	2.12%	199.1		
4.43%	97.1	7.33%	147.1	5.73%	200.0		
1.06%	99.1	1.71%	148.0	5.67%	201.0		
2.54%	100.1	3.07%	149.1	3.60%	202.1		
1.12%	101.0	1.36%	150.0	6.56%	203.1		
2.77%	103.0	2.24%	151.1	2.30%	204.1		
4.19%	104.0	1.06%	152.1	7.86%	205.1		
26.07%	105.1	1.41%	153.1	1.71%	206.2		
6.91%	106.0	1.00%	154.1	3.19%	209.1		
10.94%	107.0	2.06%	155.1	1.30%	211.1		

Most abundant peaks

m/z	43.0	121.1	57.1	124.1	125.1
Intensity	100.00	63.92	49.91	35.12	31.93
m/z	122.1	85.1	105.1	179.9	138.1
Intensity	30.10	29.86	26.07	22.47	18.92

Formula



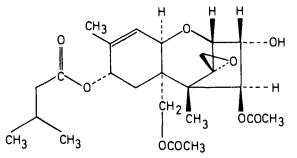
Experimental conditions: see p 2223

100 MHz ^{1}H NMR SPECTRUM 14d

T-2 TOXIN

conc. 26.80 mmol/l

solvent: $CDCl_3$

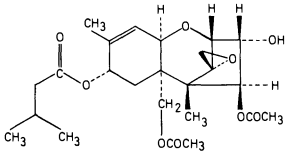


250 MHz ^{1}H NMR SPECTRUM 14e

T-2 TOXIN

conc. 26.80 mmol/l

solvent: $CDCl_3$



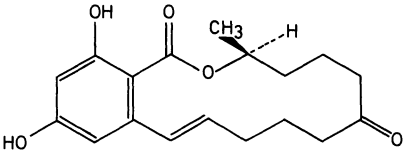
IV.15. ZEARALENONE

I SYNONYMS: 6-(10-hydroxy-6-oxo-trans-1-undecenyl)-beta-resorcylic acid-n-lactone; F-2 toxin; trans zearalenone

II CHEMICAL NAME: 3,4,5,6,9,10-hexahydro-14,16-dihydroxy-3-methyl-[S-(E)]-1H-2-Benzoxacyclotetradecin-1,7(8H)-dione

III EMPIRICAL FORMULA: $C_{18}H_{22}O_5$

IV STRUCTURAL FORMULA:



V MOLECULAR WEIGHT: 318.4

VI DESCRIPTION: Zearalenone is a white, odourless, crystalline solid

VII CHARACTERIZATION DATA:

1. Melting range: 161-164°C, after drying for 1 hour at 60°C
2. Specific rotation: $[\alpha]_D^{21} = -189.0^\circ$
   conc. 3141 µmol/l
   solvent: chloroform
3. Circular dichroism:
   $\Delta\varepsilon_{(\lambda 340)}$ 0, $\Delta\varepsilon_{(\lambda 272)}$ -15.77, $\Delta\varepsilon_{(\lambda 247)}$ 0.
   conc. 436.6 µmol/l
   solvent: methanol
   temperature: 22°C
   cell length: 2 mm
4. Ultraviolet absorption spectrum:
   see spectrum 15a

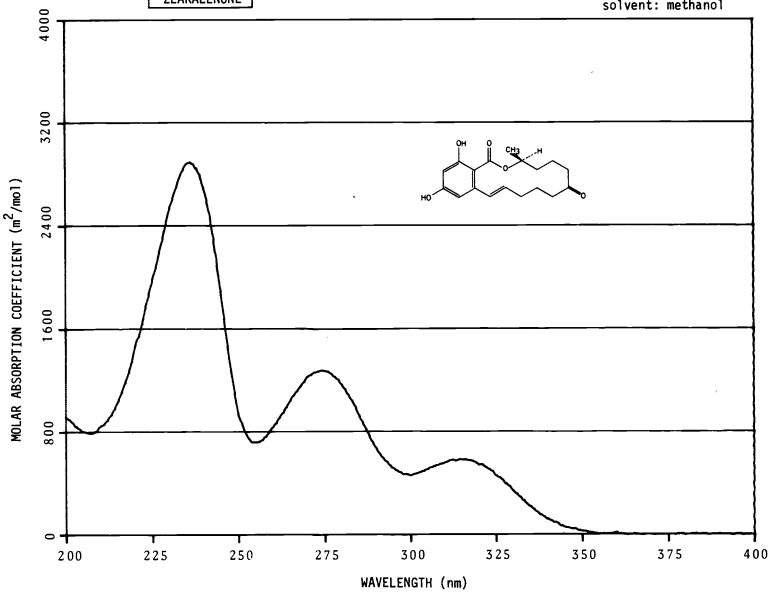
   Molar absorption coefficients:

   $\varepsilon_{(\lambda 236)} = 2885 \pm 12$
   $\varepsilon_{(\lambda 255)} = 713 \pm 11$
   $\varepsilon_{(\lambda 274)} = 1271 \pm 1$
   $\varepsilon_{(\lambda 300)} = 465 \pm 5$
   $\varepsilon_{(\lambda 315)} = 584 \pm 4$
   conc. 26.12 µmol/l
   solvent: methanol
5. Infrared absorption spectrum:
   see spectrum 15b
6. Electron impact mass spectrum:
   see spectrum 15c
7. Nuclear Magnetic Resonance spectrum:
   see spectra 15d and 15e

ULTRAVIOLET ABSORPTION SPECTRUM 15a

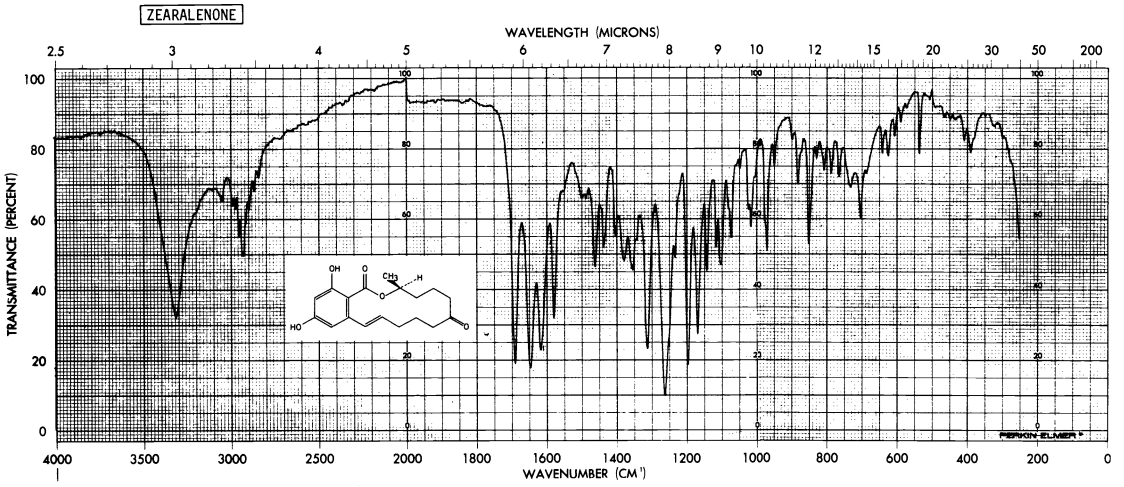
ZEARALENONE

conc. 26.12 µmol/l
solvent: methanol



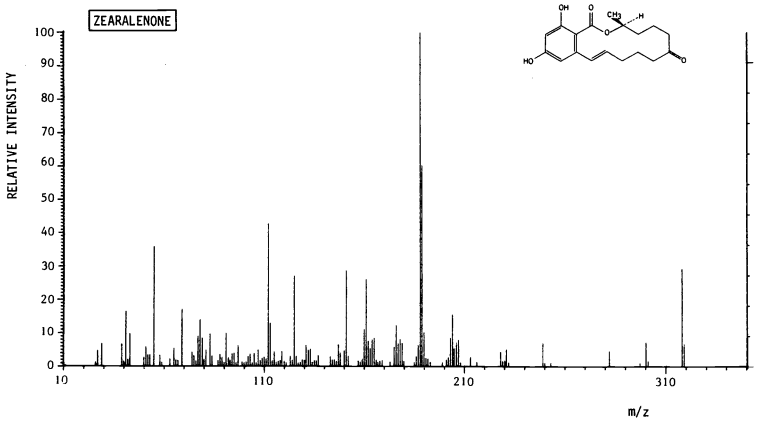
INFRARED ABSORPTION SPECTRUM 15b

ZEARALENONE



MASS SPECTRUM 15c

ZEARALENONE



MASS SPECTRUM 15c

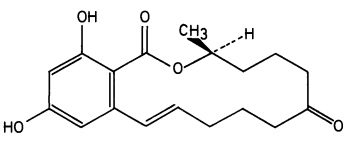
ZEARALENONE

relative intensity	mass	relative intensity	mass	relative intensity	mass	relative intensity	mass
1.20%	26.0	2.97%	102.1	1.32%	158.1	1.01%	250.1
4.74%	27.1	3.66%	103.0	2.33%	159.1	1.07%	253.1
6.89%	29.1	1.07%	104.0	11.06%	160.1	4.67%	282.1
6.76%	39.1	3.85%	105.1	26.10%	161.1	1.01%	297.3
1.51%	40.0	1.07%	106.0	7.64%	162.1	7.20%	300.2
16.49%	41.1	4.86%	107.0	5.43%	163.1	1.64%	301.2
2.02%	42.1	1.76%	108.0	8.09%	164.0	29.20%	318.1
9.79%	43.0	2.46%	109.0	8.59%	165.0	6.70%	319.1
2.65%	50.1	2.78%	110.1	1.95%	166.0		
5.87%	51.1	2.21%	111.0	1.20%	166.9		
3.47%	52.1	42.79%	112.1	1.64%	167.9		
3.41%	53.1	12.95%	113.1	1.89%	169.0		
35.96%	55.1	1.70%	114.1	1.39%	173.0		
3.35%	58.1	4.36%	115.0	5.87%	175.1		
1.26%	59.1	1.20%	116.1	12.38%	176.0		
2.21%	63.1	1.58%	117.0	6.82%	177.0		
5.43%	65.1	1.76%	118.1	8.15%	178.0		
1.95%	66.1	4.42%	118.9	7.01%	179.0		
1.83%	67.1	1.58%	120.0	1.51%	179.9		
17.13%	69.0	1.01%	121.0	1.20%	185.0		
13.90%	69.1	3.03%	123.0	3.03%	186.0		
4.23%	74.0	1.89%	124.1	6.38%	187.0		
3.28%	75.1	27.05%	125.1	100.00%	188.0		
1.51%	76.1	3.09%	126.1	60.30%	188.9		
8.91%	77.1	1.07%	127.0	10.30%	189.9		
13.90%	78.1	1.13%	128.0	2.59%	190.9		
8.40%	79.2	1.95%	129.0	2.33%	191.9		
2.02%	80.1	1.83%	130.0	1.32%	193.0		
4.86%	81.1	6.32%	130.9	1.26%	199.0		
9.60%	83.1	4.99%	132.0	2.02%	201.1		
3.09%	84.1	5.12%	133.1	2.78%	202.1		
1.70%	87.0	1.20%	134.0	8.53%	203.1		
3.53%	88.1	1.76%	135.1	15.54%	204.1		
2.65%	89.1	1.70%	136.1	5.43%	204.9		
1.13%	90.1	3.28%	137.1	7.20%	206.0		
9.86%	91.1	2.97%	143.1	7.96%	207.1		
2.59%	92.2	2.02%	144.1	1.13%	208.1		
1.89%	93.1	2.08%	145.1	2.84%	213.1		
3.72%	94.1	1.51%	146.0	1.32%	216.2		
3.98%	95.1	6.51%	147.1	4.36%	228.0		
1.07%	96.1	4.04%	148.0	1.64%	229.1		
6.19%	97.1	4.74%	150.0	1.83%	230.0		
1.26%	99.1	28.76%	151.1	5.12%	230.9		
1.01%	100.0	3.16%	152.1	1.13%	232.0		
1.26%	101.0	1.70%	157.1	7.07%	249.1		

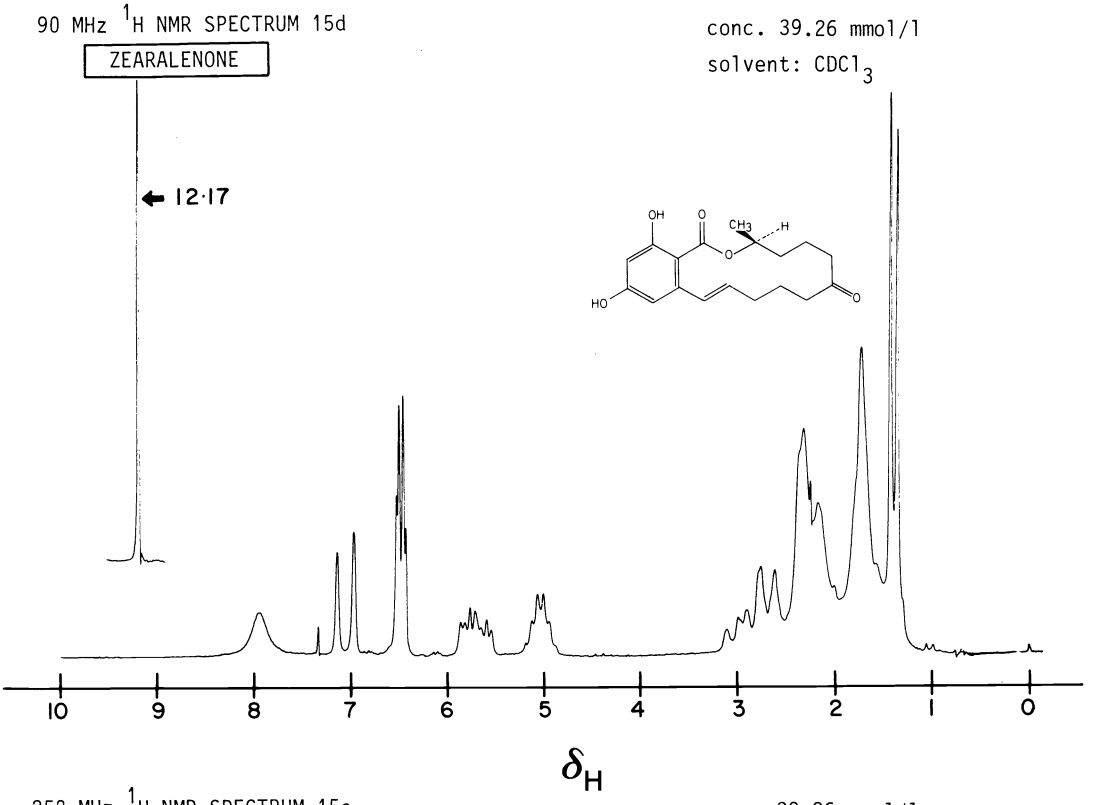
Most abundant peaks

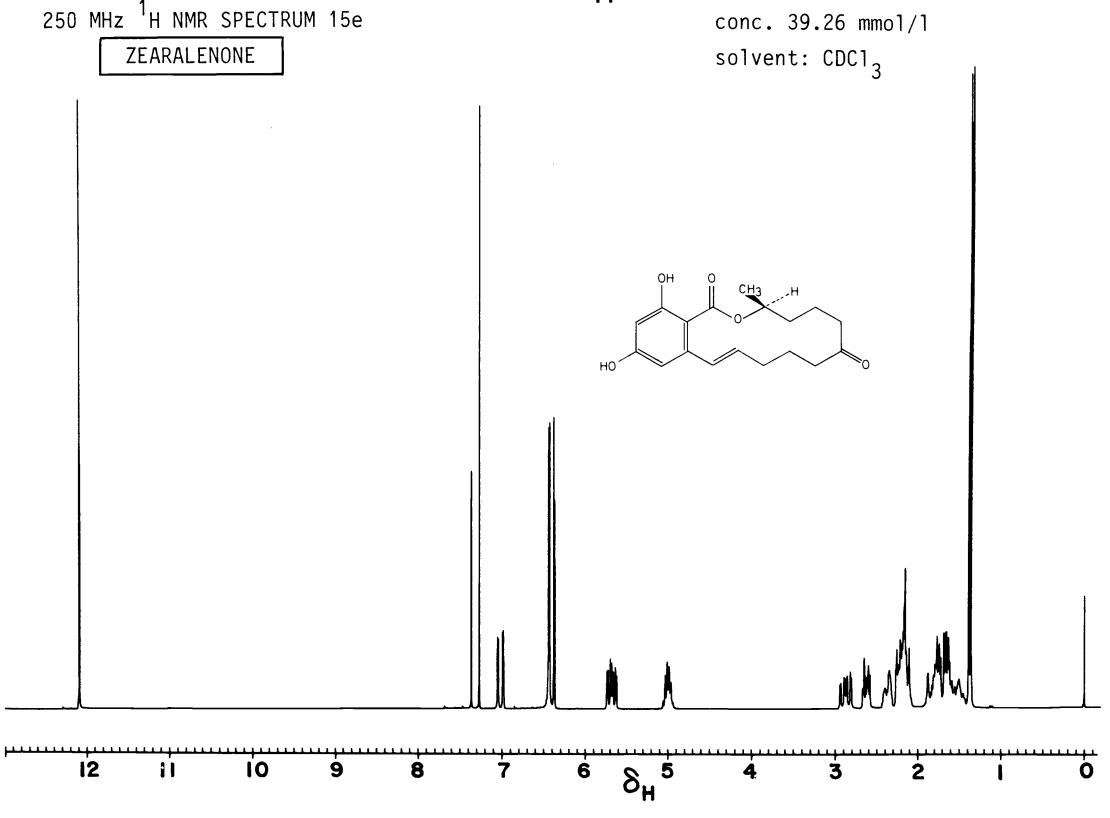
m/z	188.0	188.9	112.1	55.1	318.1
Intensity	100.00	60.30	42.79	35.96	29.20
m/z	151.1	125.1	161.1	69.0	41.1
Intensity	28.76	27.05	26.10	17.13	16.49

Formula



Experimental conditions: see p 2223


90 MHz ^{1}H NMR SPECTRUM 15d
ZEARALENONE
conc. 39.26 mmol/l
solvent: $CDCl_3$
12·17
OH
O
CH_3
H
O
HO
O
10
9
8
7
6
5
4
3
2
1
0
δ_H


250 MHz ^{1}H NMR SPECTRUM 15e
ZEARALENONE
conc. 39.26 mmol/l
solvent: $CDCl_3$
OH
O
CH_3
H
O
HO
O
12
11
10
9
8
7
6
5
4
3
2
1
0
δ_H

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