

This article was downloaded by:

On: 28 January 2011

Access details: *Access Details: Free Access*

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



Phosphorus, Sulfur, and Silicon and the Related Elements

Publication details, including instructions for authors and subscription information:

<http://www.informaworld.com/smpp/title~content=t713618290>

Efficacy of Organophosphorus Derivatives Containing Substituted Chalcone Thiosemicarbazones and Dithiocarbazates Against Fungal Pathogens of Sugarcane

S. K. Sengupta^a; O. P. Pandey^a; G. P. Rao^b; Alpana Dwivedi^a; Priyanka Singh^a

^a DDU Gorakhpur University, Gorakhpur, India ^b Sugarcane Research Station, Gorakhpur, India

Online publication date: 27 October 2010

To cite this Article Sengupta, S. K. , Pandey, O. P. , Rao, G. P. , Dwivedi, Alpana and Singh, Priyanka(2010) 'Efficacy of Organophosphorus Derivatives Containing Substituted Chalcone Thiosemicarbazones and Dithiocarbazates Against Fungal Pathogens of Sugarcane', *Phosphorus, Sulfur, and Silicon and the Related Elements*, 186: 4, 839 – 849

To link to this Article: DOI: 10.1080/10426500307813

URL: <http://dx.doi.org/10.1080/10426500307813>

PLEASE SCROLL DOWN FOR ARTICLE

Full terms and conditions of use: <http://www.informaworld.com/terms-and-conditions-of-access.pdf>

This article may be used for research, teaching and private study purposes. Any substantial or systematic reproduction, re-distribution, re-selling, loan or sub-licensing, systematic supply or distribution in any form to anyone is expressly forbidden.

The publisher does not give any warranty express or implied or make any representation that the contents will be complete or accurate or up to date. The accuracy of any instructions, formulae and drug doses should be independently verified with primary sources. The publisher shall not be liable for any loss, actions, claims, proceedings, demand or costs or damages whatsoever or howsoever caused arising directly or indirectly in connection with or arising out of the use of this material.

EFFICACY OF ORGANOPHOSPHORUS DERIVATIVES CONTAINING SUBSTITUTED CHALCONE THIOSEMICARBAZONES AND DITHIOCARBAZATES AGAINST FUNGAL PATHOGENS OF SUGARCANE

S. K. Sengupta,^a O. P. Pandey,^a G. P. Rao,^b Alpana Dwivedi,^a
and Priyanka Singh^a

DDU Gorakhpur University, Gorakhpur, India^a and
Sugarcane Research Station, Kunraghat, Gorakhpur, India^b

(Received March 20, 2002; accepted September 29, 2002)

Ten novel organophosphorus derivatives have been prepared by the reactions of O,O-diethylchlorophosphate with two important series of substituted chalcones viz., substituted chalcone thiosemicarbazones and substituted chalcone dithiocarbazates. The derivatives have been characterized on the basis of analysis and spectral (IR, ¹H NMR) data. Fungicidal activity of these derivatives against Colletotrichum falcatum, Fusarium oxysporum, and Curvularia pallescens have been evaluated. The screening results have been correlated with the structural features of the tested compounds. Organophosphorus derivatives containing 2,2'-dihydroxybenzalacetophenone thiosemicarbazone, 2-chlorobenzal-2'-hydroxyacetophenone thiosemicarbazone, 2-chlorobenzal-2'-hydroxy acetophenone-S-benzyl-dithiocarbazate proved more active than some prevalent commercial synthetic fungicides.

Keywords: Fungal pathogens; organophosphorus derivatives; sugarcane; synthesis

Sugarcane, a major source of sugar and an important cash crop, is grown extensively worldwide. Sugarcane is known to be affected by fungal, viral, bacterial, and phytoplasma pathogens that are responsible for considerable economic losses.^{1,2} Among fungi, *Colletotrichum falcatum*

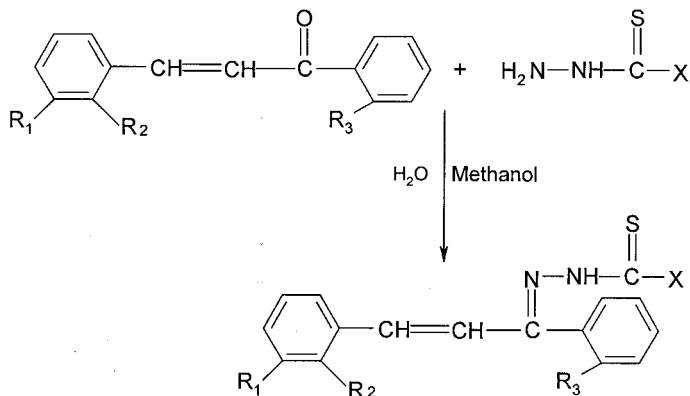
We are grateful to the University Grants Commission, New Delhi for financial support.
Address correspondence to S. K. Sengupta, Department of Chemistry, DDU Gorakhpur University, Gorakhpur-273009, India. E-mail: sengupta2002@yahoo.com

(red rot), *Fusarium oxysporum* (associated with wilt syndrome) are the most important pathogens that may be transmitted primarily by planting infected sets. *Curvularia pallescens* and *Curvularia lunata* (causing leaf spots) are the important foliar pathogens of sugarcane, sometimes causing enormous losses to sugar industry. A number of synthetic organic compounds viz., dithiocarbamates, carbamates, hydrazides are known now to be useful in the control of various fungal diseases of plants.² As an alarming level of loss is being caused to sugarcane by fungal attacks, it has become important to search for new fungicides to minimize disease incidence.

A few recent reports³⁻⁶ from our laboratory have show that on the basis of suitable logic, organic molecules incorporating phosphorus may be designed such that they may be less dangerous in use without losing their value as effective pesticides. One of the useful properties of phosphorus compounds is their relatively low stability and rapid metabolic breakdown in plants, in animal organism, in soil, and in other component of the environment with the formation of products that are safe for human beings and domestic animals. Another important feature of these compounds is the high selectivity of their action. The discovery of the mechanism of action⁷ of organophosphorus compounds make it possible to develop the fundamental principles of the directed synthesis of new substances and to establish the cause of their selective action on an organism. Studies on organophosphorus derivatives could constitute new and promising field of application in the national economy. The present study therefore, was, undertaken to evaluate the antifungal efficacy of some newly synthesized organophosphorus compounds against various important fungal pathogens of sugarcane.

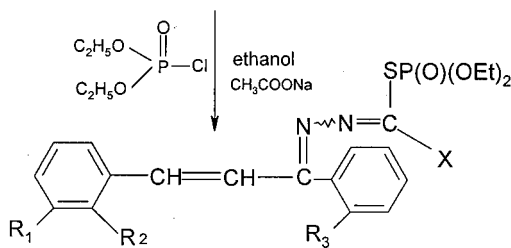
RESULTS AND DISCUSSION

The reactions of O,O-diethylchlorophosphate with thiosemicarbazones of substituted chalcones, derived by the condensation of 2-hydroxybenzalacetophenone/benzal-2'-hydroxyacetophenone/2,2'-dihydroxybenzalacetophenone/2-chlorobenzal-2'-hydroxyacetophenone/3-methoxybenzal-2' hydroxyacetophenone and thiosemicarbazide, have been carried out in ethanol in the presence of sodium acetate and a variety of organophosphorus derivatives (**2-I** to **2-V**) have been isolated according to Scheme 1. Similar reactions of O,O-diethylchlorophosphate with dithiocarbazates derived by the condensation of substituted chalcones and benzyl dithiocarbazate, are also summarized in Scheme 1. Organophosphorus derivatives of type **2-VI** to **2-X** have been isolated.



(1)

$\text{R}_1 = \text{R}_3 = \text{H}$, $\text{R}_2 = \text{OH}$, $\text{X} = \text{NH}_2$ (**1-I**) ; $\text{R}_1 = \text{R}_2 = \text{H}$, $\text{R}_3 = \text{OH}$; $\text{X} = \text{NH}_2$ (**1-II**); $\text{R}_1 = \text{H}$, $\text{R}_2 = \text{R}_3 = \text{OH}$, $\text{X} = \text{NH}_2$ (**1-III**); $\text{R}_1 = \text{H}$, $\text{R}_2 = \text{Cl}$, $\text{R}_3 = \text{OH}$, $\text{X} = \text{NH}_2$ (**1-IV**); $\text{R}_1 = \text{OCH}_3$, $\text{R}_2 = \text{H}$, $\text{R}_3 = \text{OH}$; $\text{X} = \text{NH}_2$ (**1-V**); $\text{R}_1 = \text{R}_3 = \text{H}$, $\text{R}_2 = \text{OH}$, $\text{X} = \text{C}_6\text{H}_5\text{CH}_2\text{S}$ (**1-VI**); $\text{R}_1 = \text{R}_2 = \text{H}$, $\text{R}_3 = \text{OH}$, $\text{X} = \text{C}_6\text{H}_5\text{CH}_2\text{S}$ (**1-VII**); $\text{R}_1 = \text{H}$, $\text{R}_2 = \text{R}_3 = \text{OH}$, $\text{X} = \text{C}_6\text{H}_5\text{CH}_2\text{S}$ (**1-VIII**); $\text{R}_1 = \text{H}$, $\text{R}_2 = \text{Cl}$, $\text{R}_3 = \text{OH}$, $\text{X} = \text{C}_6\text{H}_5\text{CH}_2\text{S}$ (**1-IX**); $\text{R}_1 = \text{OCH}_3$, $\text{R}_2 = \text{H}$, $\text{R}_3 = \text{OH}$, $\text{X} = \text{C}_6\text{H}_5\text{CH}_2\text{S}$ (**1-X**)



(2)

$\text{R}_1 = \text{R}_3 = \text{H}$, $\text{R}_2 = \text{OP(O)(OEt)}_2$, $\text{X} = \text{NH}_2$ (**2-I**) ; $\text{R}_1 = \text{R}_2 = \text{H}$, $\text{R}_3 = \text{OP(O)(OEt)}_2$; $\text{X} = \text{NH}_2$ (**2-II**); $\text{R}_1 = \text{H}$, $\text{R}_2 = \text{R}_3 = \text{OP(O)(OEt)}_2$, $\text{X} = \text{NH}_2$ (**2-III**); $\text{R}_1 = \text{H}$, $\text{R}_2 = \text{Cl}$, $\text{R}_3 = \text{OP(O)(OEt)}_2$, $\text{X} = \text{NH}_2$ (**2-IV**); $\text{R}_1 = \text{OCH}_3$, $\text{R}_2 = \text{H}$, $\text{R}_3 = \text{OP(O)(OEt)}_2$; $\text{X} = \text{NH}_2$ (**2-V**); $\text{R}_1 = \text{R}_3 = \text{H}$, $\text{R}_2 = \text{OP(O)(OEt)}_2$, $\text{X} = \text{C}_6\text{H}_5\text{CH}_2\text{S}$ (**2-VI**); $\text{R}_1 = \text{R}_2 = \text{H}$, $\text{R}_3 = \text{OP(O)(OEt)}_2$, $\text{X} = \text{C}_6\text{H}_5\text{CH}_2\text{S}$ (**2-VII**); $\text{R}_1 = \text{H}$, $\text{R}_2 = \text{R}_3 = \text{OP(O)(OEt)}_2$, $\text{X} = \text{C}_6\text{H}_5\text{CH}_2\text{S}$ (**2-VIII**); $\text{R}_1 = \text{H}$, $\text{R}_2 = \text{Cl}$, $\text{R}_3 = \text{OP(O)(OEt)}_2$, $\text{X} = \text{C}_6\text{H}_5\text{CH}_2\text{S}$ (**2-IX**); $\text{R}_1 = \text{OCH}_3$, $\text{R}_2 = \text{H}$, $\text{R}_3 = \text{OP(O)(OEt)}_2$, $\text{X} = \text{C}_6\text{H}_5\text{CH}_2\text{S}$ (**2-X**)

SCHEME 1 Synthetic route of organophosphorus derivatives.

The analytical data and physical properties of all organophosphorus derivatives are given in Table I. The methods used for the preparation and isolation of these compounds give materials of good purity as supported by their analysis and TLC.

TABLE I Analytical Data of Organophosphorus Compounds

Compound	Yield (%)	Color	m.p. (°C)	Found (calcd.) (%)			
				C	H	N	S
2-I	50	Dark brown	30–33	56.0 (56.1)	5.5 (5.6)	9.6 (9.8)	7.3 (7.5)
2-II	52	Olive green	90–92	55.9 (56.1)	5.4 (5.6)	9.7 (9.8)	7.4 (7.5)
2-III	52	Dark brown	180–182	54.0 (54.1)	5.2 (5.4)	9.3 (9.5)	7.0 (7.2)
2-IV	50	Reddish brown	120–123	51.8 (51.9)	4.9 (5.0)	9.0 (9.1)	6.7 (6.9)
2-V	45	Dark brown	Semi solid	54.3 (54.5)	5.4 (5.6)	9.0 (9.1)	6.8 (6.9)
2-VI	55	Dark brown	Semi solid	55.0 (55.2)	5.4 (5.6)	4.0 (4.2)	9.3 (9.5)
2-VII	50	Yellow brown	75–78	55.1 (55.2)	5.5 (5.6)	4.0 (4.2)	9.4 (9.5)
2-VIII	52	Dark brown	120–122	52.7 (52.9)	5.7 (5.9)	3.3 (3.5)	8.0 (8.1)
2-IX	60	Yellow brown	85–87	52.4 (52.5)	5.0 (5.2)	3.8 (4.0)	8.8 (9.0)
2-X	50	Reddish brown	Semi solid	54.3 (54.5)	5.6 (5.7)	3.9 (4.0)	9.0 (9.1)

The organophosphorus derivatives are found to be soluble in ethanol, acetone, chloroform (partially), dimethylformamide, and dimethylsulfoxide. These compounds melt in the temperature range 30–180°C. The compounds are stable in air.

Infrared Spectra

The infrared spectral bands of organophosphorus derivatives are given in Table II. The infrared spectra of chalcone thiosemicarbazones/dithiocarbazates show strong C=N vibration⁸ in the 1620–1630 cm⁻¹ region. The configuration about this C=N may be a mixture of *syn* and *anti*. This band appears almost at the same position in the organophosphorus derivatives indicating the noninvolvement of this group into bond formation with phosphorus. The four bands occurring in the regions, 1460–1500 cm⁻¹, 1250–1275 cm⁻¹, 1040–1060 cm⁻¹, and 760–780 cm⁻¹ may be assigned to thioamide-I, -II, -III, and -IV vibrations respectively. The appearance of these four bands indicates the existence of the chalcone thiosemicarbazones/dithiocarbazates in the thione form in the solid state.⁹ The bands of the organic compounds, due to the mixed contributions of $\delta(\text{N-H})$, $\nu(\text{C-N})$, $\nu(\text{C=S})$, and $\delta(\text{C-H})$ vibrations, are found to be absent in the spectra of their corresponding organophosphorus derivatives. The disappearance of thioamide bands in the complexes indicate the possibility of thione \rightleftharpoons thiol tautomerism. This is further confirmed by the appearance of medium intensity band near 720 cm⁻¹ in all organophosphorus derivatives, which may be assigned to $\nu(\text{C-S})$. The formation of bond between thiol sulphur and phosphorus is supported by the appearance of band at ca. 620 cm⁻¹, assignable¹⁰ to $\nu(\text{P-S-C})$. The infrared spectra of chalcone

TABLE II IR Spectral Data (cm⁻¹) of Organophosphorus Compounds Containing Substituted Chalcone Thiosemicarbazones/Dithiocarbazates

Compounds	$\nu(\text{C-S})$	$\nu(\text{C=N})$	$\nu(\text{P-S-C})$	$\nu(\text{P-O-C})$ (phenolic)	$\nu(\text{P-O-C})$ (alkyl)	$\nu(\text{P=O})$
2-I	720 m	1620 s	620 m	1150 s	1015 m	1280 m
2-II	710 m	1615 s	610 m	1145 s	1020 m	1285 m
2-III	715 m	1630 s	615 m	1155 s	1030 m	1295 m
2-IV	720 m	1620 s	620 m	1150 s	1030 m	1290 m
2-V	720 m	1635	620 m	1150 s	1020 m	1300 m
2-VI	720 m	1630 s	620 m	1160 s	1030 m	1255 s
2-VII	720 m	1620 s	615 m	1150 s	1040 m	1280 s
2-VIII	720 m	1630 s	625 m	1160 s	1045 m	1260 s
2-IX	710 m	1635 s	620 m	1160 s	1030 m	1260 s
2-X	725 m	1630 s	620 m	1155 s	1040 m	1280 s

thiosemicarbazones/dithiocarbazates show one medium intensity band in the $3350\text{--}3380\text{ cm}^{-1}$ region, which may be assigned to $\nu(\text{O--H})$. In organophosphorus derivatives, this band disappears suggesting the formation of bond between phosphorus and phenolic OH group. This is also confirmed by the appearance of strong band at ca. 1150 cm^{-1} assignable¹⁰ to $\nu(\text{P--O--C})$. The $\nu(\text{C--O})$ (phenolic) band appears near 1240 cm^{-1} in all organophosphorus derivatives. The ethylenic $\text{C}=\text{C}$ stretching vibration¹¹ appears at ca. 1600 cm^{-1} in the spectra of organophosphorus derivatives.

The above observations indicate that possibly the bonding in organophosphorus derivatives is through thiol sulphur and phenolic oxygen atoms. In addition, all organophosphorus derivatives show bands at ca. $1015\text{--}1030\text{ cm}^{-1}$ and $1280\text{--}1295\text{ cm}^{-1}$ assignable³ to $\nu(\text{P--O--C})$ (alkyl) and $\nu(\text{P}=\text{O})$ vibrations respectively.

Proton Magnetic Resonance Spectra

The proton magnetic resonance spectra of substituted chalcone thiosemicarbazones and dithiocarbazates and their corresponding organophosphorus derivatives were recorded in deuterated dimethylsulfoxide. The ^1H NMR spectral data of organophosphorus derivatives are given in Table III. The intensities of all the resonance lines were determined by planimetric integration. Comparing the spectra of substituted chalcone thiosemicarbazones and dithiocarbazates and their corresponding organophosphorus derivatives can draw the following conclusions:

- (i) The signal due to --OH proton appears at about δ 10.5–11.5 in the spectra of chalcone thiosemicarbazones/dithiocarbazates which disappears in the corresponding organophosphorus derivatives indicating the deprotonation of phenolic --OH group.
- (ii) All organophosphorus derivatives show two doublets at ca. δ 4.5–4.8 (J 14.2) and δ 5.1–5.6 (J 15.1) which are due to α and β olefinic protons indicating *trans* configuration.
- (iii) The signal due to --NH group appears at ca. δ 9.30–9.65 in the spectra of thiosemicarbazones and their corresponding organophosphorus derivatives.
- (iv) Benzyl dithiocarbazates and their organophosphorus derivatives show signals for aromatic ring protons at ca. δ 7.65–7.80.
- (v) The signals due to ethoxy group appear at ca. 2.50–2.80 (triplet, due to CH_3 group) and at ca. 3.0–3.4 (quartet, due to CH_2 group) in the spectra of all organophosphorus derivatives.

TABLE III ^1H NMR Data (δ Scale, ppm) of Organophosphorus Derivatives Containing Substituted Chalcone Thiosemicarbazones/Dithiocarbazates

Compound	Aromatic ring	—NH	—CH=CH—	—S—CH ₂	—OCH ₃	C ₂ H ₅
2-I	7.28–7.45 (m), 7.68 (s)	9.48 (s)	4.55 (d), 5.38 (d)	—	—	2.50 (t), 3.15 (q)
2-II	7.30–7.50 (m), 7.70 (s)	9.30 (s)	4.80 (d), 5.58 (d)	—	—	2.65 (t), 3.00 (q)
2-III	7.35–7.64 (m)	9.65 (s)	4.72 (d), 5.46 (d)	—	—	2.55 (t), 3.28 (q)
2-IV	7.38–7.62 (m)	9.60 (s)	4.66 (d), 5.60 (d)	—	—	2.60 (t), 3.25 (q)
2-V	7.40–7.75 (m)	9.55 (s)	4.75 (d), 5.55 (d)	—	3.72 (s)	2.58 (t), 3.25 (q)
2-VI	7.38–7.55 (m), 7.70 (s)	—	4.50 (d), 5.40 (d)	3.05 (d)	—	2.65 (t), 3.28 (q)
2-VII	7.30–7.45 (m), 7.68 (s)	—	4.65 (d), 5.10 (d)	3.08 (d)	—	2.80 (t), 3.40 (q)
2-VIII	7.28–7.50 (m)	—	4.60 (d), 5.48 (d)	3.12 (d)	—	2.70 (t), 3.25 (q)
2-IX	7.25–7.60 (m)	—	4.55 (d), 5.28 (d)	3.16 (d)	—	2.75 (t), 3.30 (q)
2-X	7.30–7.65 (m)	—	4.50 (d), 5.35 (d)	3.10 (d)	3.68 (s)	2.78 (t), 3.35 (q)

TABLE IV Fungitoxic Screening Data of Organophosphorus Derivatives Containing Substituted Chalcone Thiosemicarbazones/Dithiocarbazates

Compound	Percent mycelial inhibition								
	<i>Colletotrichum falcatum</i>			<i>Fusarium oxysporum</i> compound dose (ppm)			<i>Curvularia pallescens</i>		
	10	100	1000	10	100	1000	10	100	1000
I	48.2	70.6	88.5	40.0	55.3	78.7	39.5	58.2	76.8
II	50.6	74.8	94.3	40.2	58.6	80.8	41.2	60.7	80.6
III	54.2	84.8	100 ^a	48.5	80.2	100 ^a	46.7	75.2	100 ^a
IV	70.6	80.2	100 ^a	68.8	78.5	100 ^a	71.4	80.0	100 ^a
V	38.8	54.6	79.2	32.8	49.5	74.5	55.2	56.6	69.2
VI	18.9	51.2	80.3	19.6	34.2	65.2	20.8	32.7	68.7
VII	21.6	52.6	82.1	42.1	59.5	70.1	10.5	28.9	58.2
VIII	55.8	76.5	95.4	50.6	78.2	91.0	52.8	75.2	90.7
IX	70.2	87.2	100 ^a	67.2	82.8	100 ^a	56.7	79.5	100 ^a
X	41.0	65.1	78.1	33.8	54.3	80.0	30.8	49.1	65.5

^aAbsolute inhibition.

Antifungal Activity

Results of the antifungal assay of the organophosphorus derivatives are summarized in Table IV. Ten newly synthesized organophosphorus compounds of substituted chalcone thiosemicarbazones and substituted chalcone dithiocarbazates were screened for their antifungal properties against *Colletotrichum falcatum*, *Fusarium oxysporum*, and *Curvularia pallescens* (all parasitic on sugarcane). The antifungal activity of organophosphorus derivatives was found to be greater than that of the corresponding starting materials. O,O-Diethylphosphate derivatives containing 2,2'-dihydroxychalcone thiosemicarbazone (**2-III**), 2-chloro-2'-hydroxychalcone thiosemicarbazone (**2-IV**), and 2-chloro-2'-hydroxychalcone dithiocarbazate (**2-IX**) exhibit absolute inhibition against all test fungi at 1000 ppm concentration. These compounds showed superiority over the commercial fungicides—Bavistin, Blitox 50, Topsin M, and Dithane M-45 which are being used in sugarcane fungal disease management. Compounds were upto four times more active than the tested commercial fungicides (Table V). Other derivatives displayed different levels of mycelial inhibition of the test fungi at different concentrations. The minimum fungal inhibition property was recorded with O,O-diethylphosphate derivative containing 3-methoxy-2'-hydroxychalcone dithiocarbazate (**2-X**).

TABLE V Efficacy of Organophosphorus Derivatives Compared with Synthetic Fungicides against Sugarcane Pathogens

Common names of fungicide/chemical	Trade name	MIC (ppm) against		
		<i>C. falcatum</i>	<i>F. oxysporum</i>	<i>C. pallescens</i>
Carbendazim	Bavistin	4000	3000	4000
Copper oxychloride	Blitox 50	2000	2000	2000
Mancozeb	Dithane M-45	4000	3000	4000
Thiophanate methyl	Topsin M	4000	4000	4000
2-III	—	1000	1000	1000
2-IV	—	1000	1000	1000
2-IX	—	1000	1000	1000

Screening Data Conclusions

1. There is significant alteration in the antifungal activity with the change in the nature of organic group attached to O,O-diethylphosphate moiety.
2. For any particular species of fungus, organophosphorus derivatives containing thiosemicarbazones or dithiocarbazates are found to be more effective as compared to other reported derivatives. This indicates that the presence of sulphur imparts better inhibition against fungi.
3. For any particular series of organic compounds, the compounds containing chloro group in the chalcone ring show better activity.
4. The activity decreases on dilution.

EXPERIMENTAL

The reactions of O,O-diethyl chlorophosphate were carried out under inert atmosphere and anhydrous conditions. Special precautions were taken to exclude moisture from the apparatus and chemicals as the starting material (O,O-diethylchlorophosphate) and reactions were susceptible to hydrolysis. Glass apparatus with interchangeable joints were used throughout the work. The solvents were purified and dried using the method described in the literature.¹² O,O-Diethylchlorophosphate was prepared according to the reported method.¹³ Chalcone thiosemicarbazones/dithiocarbazates were prepared as described.¹⁴ All reactions were carried out in the hood. A hood is a specially constructed workplace that has, at least, a powered vent to suck noxious fumes outside. The details of analyses and physical measurements were the same as reported earlier.³ The infrared

spectra were recorded in potassium bromide medium, in the range 4000–200 cm^{-1} , on Perkin Elmer 621 and Beckman AccuLab-9 Spectrophotometer. The ^1H NMR spectra were recorded on Perkin Elmer R32 Spectrometer at a sweep width of 90 MHz.

For antifungal activity, all of the compounds were tested against all of the test fungi by the food poison technique¹⁵ at three concentrations (10, 100, and 1000 ppm). For this, the desired amount of chemical was dissolved in 0.5 cm^3 of solvent and mixed with the culture medium, on the basis of the volume of medium in each petriplate (80 mm diameter). Oatmeal-agar medium¹⁶ was used for all test fungi. In controls, the same amount of medium containing the requisite amount of solvent was poured in place of test chemicals. A mycelial disk (5 mm diameter) obtained from the periphery of 2-week-old culture was taken and transferred to the center of each petriplate. Plates were incubated for 7 days at $28 \pm 2^\circ\text{C}$. Each treatment was repeated three times, and the inhibition was recorded relative to percent mycelial inhibition calculated using the formula.

$$[(dC - dT)/dC] \times 100,$$

where dC is the average diameter of the mycelial colony of the control and dT is the average diameter of the mycelial colony of the treatment.

The minimum inhibitory concentration (MIC) of O,O-diethylphosphate derivatives showing absolute inhibition was determined by poison food technique. Four concentrations, 500, 1000, 2000, 3000, and 4000 ppm of each test compound with respect to the culture medium, were prepared. The fungistatic/fungicidal natures of the active chemical were determined in three replicates against the test fungi following the procedure of Garbour and Houston (1959). This was done by observing if revival of growth of the inhibited mycelial disks occurred following transfer to a chemical-free medium.

Reactions of O,O-Diethylchlorophosphate with Substituted Chalcone Thiosemicarbazones/ Dithiocarbazates

A mixture of the thiosemicarbazones/dithiocarbazates of substituted chalcone (0.01 mmol) and sodium acetate (0.02 mmol/0.03 mmol) was refluxed in ethyl alcohol (40 cm^3 for one-half hour. After cooling, O,O-diethyl chlorophosphate (0.02 mmol/0.03 mmol) was added and again refluxed it for 20–25 h. The reaction mixture was then cooled and poured in ice. The precipitate, thus obtained, was filtered off. The compound was recrystallized from alcohol.

REFERENCES

- [1] V. P. Agnihotri, *Diseases of Sugarcane and Sugarbeat* (Oxford & IBH, New Delhi, 1990), p. 4.
- [2] S. K. Sengupta, O. P. Pandey, and G. P. Rao, *Sugarcane Pathology, Vol. I: Fungal Diseases* (Science Publishers: New Hampshire, 2000), p. 279.
- [3] A. K. Jaiswal, G. P. Rao, O. P. Pandey, and S. K. Sengupta, *J. Agric. Food Chem.*, **46**, 1609 (1998).
- [4] P. Vishen, S. P. Shahi, G. P. Rao, O. P. Pandey, and S. K. Sengupta, *Pestology*, **24**, 54 (2000).
- [5] S. K. Sengupta, O. P. Pandey, G. P. Rao, R. Chandra, and P. Vishen, *Sugartech*, **1**, 77 (1999).
- [6] K. Chaturvedi, A. K. Jaiswal, K. N. Mishra, O. P. Pandey, and S. K. Sengupta, *A. C. H-Models in Chemistry*, **135**, 93 (1998).
- [7] C. Feast and K.-J. Schmidt, *The Chemistry of Organophosphorus Pesticides* (Springer-Verlag, New York, 1982).
- [8] D. X. West, A. M. Stark, G. A. Bain, and A. E. Liberta, *Transition Met. Chem.*, **21**, 289 (1996).
- [9] S. K. Sengupta, O. P. Pandey, V. K. Sharma, and B. K. Srivastava, *Transition Met. Chem.*, **23**, 349 (1998).
- [10] S. K. Sengupta, O. P. Pandey, G. P. Rao, S. P. Shahi, and A. K. Jaiswal, *Sugarcane*, **4**, 17 (1998).
- [11] T. M. Aminabhavi, N. S. Biradar, and S. D. Angadi, *Inorg. Chem. Acta*, **118**, L-17 (1996).
- [12] A. I. Vogel, *A Textbook of Practical Organic Chemistry Including Qualitative Organic Analysis* (Longmans, London, 1956), 3rd ed.
- [13] K. Chaturvedi, *Organic Derivatives of Phosphorus as Pesticides*, Ph.D. Thesis, University of Gorakhpur, 1993.
- [14] P. Vishen, *Efficacy of Organophosphorus Derivatives against Fungal Pathogens of Sugarcane*, Ph.D. Thesis, University of Gorakhpur, 1999.
- [15] R. K. Grover and J. D. Moore, *Phytopathology*, **52**, 876 (1962).
- [16] A. Johnston and C. Booth, *Plant Pathologists Pocket Book* (CMT, Oxford, & IBH, New Delhi, 1983), p. 896.