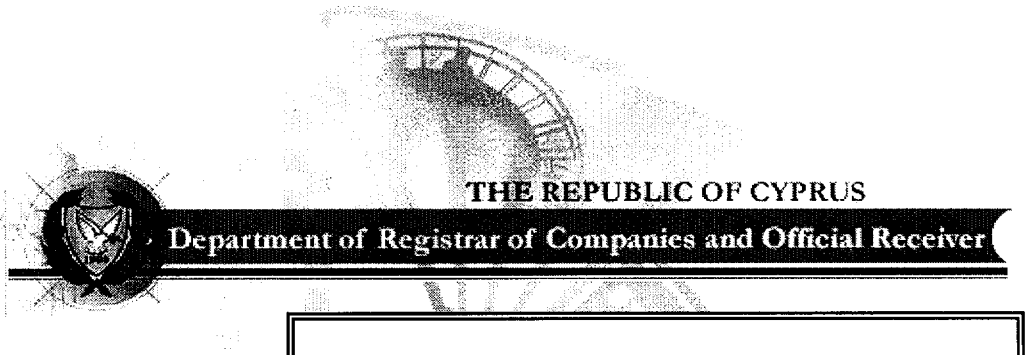

ΚΥΠΡΙΑΚΟ ΓΡΑΦΕΙΟ ΔΙΠΛΩΜΑΤΩΝ ΕΥΡΕΣΙΤΕΧΝΙΑΣ

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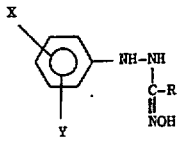
(54) **Arylhydrazo-aldoximes and derivatives thereof exerting a fungicidal activity**

(57) A method of combatting infections due to fungi in plants, and/or of preventing such infections, and/or of immunizing the plants against such infections, in which the plants are treated with an arylhydrazoaldoxime of the general formula:



(I)

or tautomers thereof, in which

X and Y independently represent a hydrogen or halogen atom, an alkyl group having 1 to 5 carbon atoms which may be halogenated, an alkoxyl group or NO_2, and

R represents a hydrogen atom, an alkyl group having 1 to 5 carbon atoms, a phenyl group or a substituted phenyl group, as such or as an organic or inorganic salt at a dosage of 0.01% upwards.

Many of the compounds of formula I are new.

GB 2 038 634 A

SPECIFICATION·

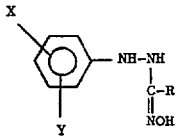
Arylhydrazo-aldoximes and derivatives thereof exerting a fungicidal activity

This invention relates to the use of arylhydrazo-aldoximes as fungicides and to certain new arylhydrazo-aldoximes.

Some arylhydrazo-aldoximes have been disclosed by E. Bemberger et al, in Berichte *35*, 1902, pages 72 to 74; ibid 1085 ibd. *36*, 1903, 57. However, these compounds are known only as intermediates for obtaining other compounds.

We have now found that certain arylhydrazo-aldoximes possess fungicidal activity.

Therefore according to the invention there is provided a method of combatting infections due to fungi in useful plants, and/or of preventing such infections, and/or immunizing the plants against such infections, in which the plants are treated with an arylhydrazo-aldoxime of the general formula:



(I)

or tautomers thereof, in which

X and Y independently represent a hydrogen or halogen atom, an alkyl group having 1 to 5 carbon atoms which may be halogenated, an alkoxyl group or NO_2, and

R represents a hydrogen atom, an alkyl group having 1 to 5 carbon atoms, a phenyl group or a substituted phenyl group, as such or as an organic or inorganic salt or a suitable composition thereof at a dosage of 0.01% upwards.

Arylhydrazo-aldoximes of formula (I) and their tautomers are active as such or in the form of organic or inorganic salts in preventing (i.e. in hindering the arising) and in treating (i.e. in causing the regress) the infection due to fungi in useful plants, as well as in immunizing the plants against the infection (because they do not permit, even if applied at a distance from the infection point, the developing of such infection) in doses of from 0.01% upwards.

Certain of the arylhydrazo-aldoximes within formula (I) are new and are reported in the following Table 1. Table 2 reports the data of known arylhydrazo-aldoximes together with relevant bibliographic data.

Table 1

Properties of the compounds of general formula I

Compound No.	X	Y	R	Form (free base or salt	Melting Point °C	Elemental analysis					
						C		H		N	
						calc.%	found %	calc.%	found %	calc.%	found %
1	4-Cl	H	H	hydrochloride	118	37.87	37.60	4.00	4.01	18.93	19.00
2	2-Me	H	CH_3		116	60.31	59.68	7.31	7.32	23.44	23.49
3	2-Me	H	CH_3	hydrochloride	180	50.12	49.82	6.65	6.54	19.48	19.31
4	4-Me	H	CH_3	hydrochloride	148	50.12	49.96	6.65	6.56	19.48	19.49
5	2-Cl	H	CH_3		128	48.13	47.89	5.05	4.80	21.05	20.85
6	2-Cl	H	CH_3	hydrochloride	185	40.70	40.63	4.70	4.62	17.80	17.46
7	3-Cl	H	CH_3		148	48.13	48.00	5.05	5.04	21.05	21.25
8	3-Cl	H	CH_3	hydrochloride	160	40.70	40.44	4.70	4.60	17.80	17.83
9	2-F	H	CH_3		140	52.45	52.35	5.50	5.61	22.42	22.29
10	2-F	H	CH_3	hydrochloride	180	43.74	43.66	5.05	5.10	19.14	19.22
11	3-CF_3	H	CH_3		115	46.35	46.05	4.32	4.24	18.02	18.10
12	3-CF_3	H	CH_3	hydrochloride	161	40.08	40.18	4.11	4.06	15.58	15.40

Compound No.	X	Y	R	Form (free base or salt	Melting Point °C	Elemental analysis					
						C		H		N	
						calc.%	found %	calc.%	found %	calc.%	found %
13	4-OMe	H	CH_3		105	55.37	55.04	6.71	6.73	21.52	21.38
14	3-Me	5-Me	CH_3		124	62.15	62.45	7.82	7.86	21.14	21.18
15	3-Me	5-Me	CH_3	hydrochloride	170	52.29	52.37	7.02	6.89	18.29	18.15
16	3-Cl	4-Cl	CH_3		150	41.05	41.09	3.87	3.90	17.95	18.24
17	3-Cl	4-Cl	CH_3	hydrochloride	156	35.52	35.47	3.72	3.67	15.53	15.43
18	3-Cl	4-Cl	CH_3	oxalate	160	38.70	39.00	3.60	3.70	15.0	15.0
19	3-Cl	4-Cl	CH_3	p-toluene sulphonate	182	44.30	45.10	4.20	4.40	10.30	10.50
20	3-Cl	5-Cl	CH_3		155	41.05	41.19	3.89	3.83	17.95	17.96
21	3-Cl	5-Cl	CH_3	hydrochloride	114	35.52	35.56	3.72	3.60	15.53	15.44
22	2-Me	H	C_2H_5		103	62.15	62.56	7.82	8.06	21.74	21.97
23	2-Me	H	C_2H_5	hydrochloride	159	52.29	51.86	7.02	6.95	18.29	18.18
24	4-Me	H	C_2H_5	hydrochloride	145	52.29	51.94	7.02	6.95	18.29	18.07

Table 1

Properties of the compounds of general formula I

Compound No.	X	Y	R	Form (free base or salt	Melting Point °C	Elemental analysis					
						C		H		N	
						calc.%	found %	calc.%	found %	calc.%	found %
25	2-Cl	H	C_2H_5		111	50.59	50.56	5.66	5.68	19.67	19.71
26	2-Cl	H	C_2H_5	hydrochloride	160	43.22	43.06	5.24	5.50	16.80	16.09
27	3-Cl	H	C_2H_5		112	50.59	50.68	5.66	5.75	19.67	20.43
28	3-Cl	H	C_2H_5	hydrochloride	148	43.22	43.03	5.24	5.23	16.80	16.97
29	4-Cl	H	C_2H_5	hydrochloride	166	43.22	43.23	5.24	5.27	16.80	16.75
30	3-CF_3	H	C_2H_5		106	48.58	48.44	4.89	4.73	17.00	16.93
31	3-CF_3	H	C_2H_5	hydrochloride	155	42.34	42.53	4.62	4.57	14.81	14.86
32	3-Me	5-Me	C_2H_5	hydrochloride	150	54.21	54.37	7.44	7.45	17.24	17.05
33	2-Me	6-Me	C_2H_5		94	63.74	63.64	8.27	8.48	20.27	20.44
34	2-Me	6-Me	C_2H_5	hydrochloride	145	54.21	53.51	7.44	7.58	17.24	17.02
35	2-Me	4-Cl	C_2H_5		112	52.75	51.75	6.20	6.04	18.45	18.01
36	2-Me	4-Cl	C_2H_5	hydrochloride	175	45.47	45.36	5.72	5.81	15.91	15.66

Compound No.	X	Y	R	Form (free base or salt	Melting Point °C	Elemental analysis					
						C		H		N	
						calc.%	found %	calc.%	found %	calc.%	found %
37	3-Cl	4-Cl	C_2H_5		130	45.58	45.31	4.47	4.42	16.94	16.65
38	3-Cl	4-Cl	C_2H_5	hydrochloride	182	38.00	37.91	4.25	4.26	14.76	14.62
39	3-Cl	4-Cl	C_2H_5		135	43.57	43.06	4.47	4.40	10.93	17.11
40	3-Cl	4-Cl	C_2H_5	hydrochloride	152	38.00	38.05	4.25	4.37	14.76	14.50
41	3-Cl	4-Cl	C_2H_5	oxalate	147	39.70	39.59	3.87	3.96	12.43	12.25
42	3-Cl	4-Cl	C_2H_5	p-toluene sulphonate	166	45.72	45.55	4.56	4.66	10.00	9.67
43	3-Cl	5-Cl	C_2H_5		127	43.57	43.35	4.47	4.41	16.93	16.85
44	3-Cl	5-Cl	C_2H_5	hydrochloride	160	38.00	37.94	4.25	4.23	14.76	14.56
45	H	H	C_6H_4(4-Cl)	hydrochloride	148	52.37	53.13	4.39	4.84	14.09	14.07
46	4-Cl	H	C_2H_5		98	50.59	49.65	5.66	5.60	19.67	19.36
47	H	H	C_6H_3(3,4-Cl_2)	hydrochloride	158	46.94	46.56	3.63	3.59	12.63	12.54

Table 2

Compound No.	X	Y	R	Form (free base or salt)	Reference
48	H	H	CH_3		Bamberger, Frei, Berichte 35, 1088
49	4-Me	H	CH_3		Bamberger, Berichte 35, 756
50	4-Cl	H	CH_3		Bamberger, Berichte 35, 58
51	4-Cl	H	CH_3	hydrochloride	Bamberger, Berichte 35, 59
52	2-Cl	4-Cl	CH_3		Bamberger, Berichte 35, 61
53	2-Cl	4-Cl	CH_3	hydrochloride	Bamberger, Berichte 35, 61
54	H	H	C_2H_5		Bamberger, Frei, Berichte 35, 1092
55	H	H	C_6H_5	hydrochloride	Bamberger, Frei, Berichte 35, 1091

The preparation of the new compounds is analogous to those methods disclosed in the cited literature.

The arylhydrazo-aldoximes can be given to the plant as such or in the form of formulations prepared according to conventional techniques. They can be mixed with inert powders, e.g. kieselguhr, activated carbon, gypsum and urea, or they can be made in the form of wettable powders with surfactants if required, they can also be dissolved, or dispersed, or emulsified in water or in organic solvents.

The activity has been tested on various species of plant artificially infected with noxious fungi before (to determine the curative activity) and after (to determine the preventive activity) the treatment with the arylhydrazo-aldoxime, and by treating said plants with the fungicidal agent in parts far from the point of infection (to determine the immunizing activity).

The invention will now be illustrated by the following Examples, in which Examples 1 and 2 relate to the method of synthetizing the new compounds and

Examples 3 to 10 relate to the biological activity of the arylhydrazo-aldoximes.

Example 1

Preparation of Compound No. 39

a) An aqueous solution containing 44.5 g of 1-nitro-propane and 20 g of NaOH was added, at 0°C over a period of 30 minutes to a water-alcoholic solution of a diazonium salt prepared from 81 g of 3,4-dichloroaniline, 210 cc of concentrated HCl, 35 g of $NaNO_2$ and 225 g of trihydrated sodium acetate. At the conclusion of the addition the mixture was stirred for 3 hours at 0°C, and thereafter the resulting solid product was filtered. After washing with water and drying, 125 g of 1 - nitro - 1 - (3, 4 - dichlorophenyl - hydrazone) - propane were collected in the form of a yellow solid having a melting point of 137°C with decomposition.

b) 125 g of 1 - nitro - 1 - (3, 4 - dichlorophenyl - hydrazone) - propane were added to 400 cc of ethanol saturated at 0°C with gaseous NH_3. Anhydrous H_2S was bubbled through the reaction mixture at a rate to ensure the inside temperature did not exceed 35°C until the evolution of heat was no longer observed. At the conclusion of the reaction the solvent was removed under reduced pressure and the residual solid was washed with water. 75 g of β - (α - oximino - propyl) - 3, 4 - dichlorophenyl - hydrazine were obtained in the form of a white solid having a melting point at 124°C with decomposition.

Example 2

Preparation of Compound No. 40

75 g of β - (α - oximino - propyl) - 3, 4 - dichlorophenyl - hydrazine dissolved in a mixture of ethanol - ethyl ether were treated, at 5 to 10°C with an anhydrous HCl to attain an acid pH. The solid which separated was filtered, to yield 75 g of β - (α - oximino - propyl) - 3, 4 - dichlorophenylhydrazine hydrochloride in the form of white crystals having a melting point of 155°C with decomposition.

Example 3

Curative activity on vine mildew — (Plasmopara viticola (B et C.) Berl. et de Toni

The leaves of cv. Dolcetto vine, cultivated in a pot in a conditioned environment at 25°C and 60% relative humidity, were sprayed on their lower faces with an aqueous suspension of conides (200,000 conides/cc). After a residence time of 24 hours in a humidity-saturated ambient environment at 21°C, the plants were divided into three groups. The plants of each group were treated by spraying both faces of their leaves with the products being tested in a water-acetone solution at 20% of acetone (vol./vol.) respectively after 1, 2 and 3 days from the infection.

At the conclusion of the incubation period (7 days) the extent of the infection was evaluated at sight accordingly to an evaluation scale ranging from 100 (sound plant) to 0 (thoroughly infected plant).

Compound No.	Dose % Compound	Activity
49	1.5	75
40	1.5	100
50	1.5	42
51	1.5	100

Example 4

Immunizing activity on vine mildew — (Plasmopara viticola (B et C.) Berl et de Toni)

The leaves of cv. Dolcetto vine, cultivated in a pot in a conditioned ambient environment were sprayed on their upper faces with the product being tested in a water-acetone solution at 20% of acetone (vol./vol.). The plants were then kept in a conditioned ambient environment for 6 days; on the seventh day the lower faces of their leaves were sprayed with a suspension of conides of *Plasmopara viticola* (200,000 conides/cc); after a 24-hour residence time in a humidity-saturated environment the plants were brought again to a conditioned ambient environment. At the conclusion of the incubation period (7 days), the extent of the infection was evaluated at sight according to an evaluation scale ranging from 100 (sound plant) to 0 (fully infected plant).

Compound No.	Dose % Compound	Activity
49	3	100
40	3	100
51	3	100

Example 5

Curative activity on the beet cercospora — (Cercospora beticola Sacc.)

The leaves of beet plants, c.v. KWS polybeta, cultivated in a conditioned ambient environment, were sprayed on both their faces with an aqueous suspension of conides of *Cercospora beticola* (200,000 conides/cc). After 48 hours said leaves were treated with the product being tested in a water-acetone solution at 20% of acetone (vol./vol.) by spraying of both faces. At the end of the incubation period (20 days), the extent of the infection was evaluated at sight according to an evaluation scale range from 100 (sound plant) to 0 (fully infected plant).

Compound No.	Dose % Compound	Activity
40	1	85
50	1	46
51	1	62
17	1	100

Example 6

Immunizing activity on beet Cercospora — (Cercospora beticola Sacc.)

The beet leaves, cv. KWS polybeta, cultivated in a pot in a conditioned ambient environment were sprayed on their upper faces with the product being tested in a water-acetone solution at 20% of acetone (vol./vol.). The plants were then kept in a conditioned ambient environment for 6 days; on the seventh day the lower faces of the leaves were sprayed with a suspension of conides of *Cercospora beticola* (200,000 conides/cc). After a residence time of 48 hours in a humidity-saturated environment, the plants were brought again to a conditioned ambient environment.

At the conclusion of the incubation period (20 days) the extent of the infection was evaluated at sight according to an evaluation scale ranging from

100 (sound plant) to 0 (fully infected plant).

Compound No.	Dose % Compound	Activity
40	1	72
50	1	86
51	1	84
17	1	100

Example 7

Curative activity on cucumber oidium — (Sphaerotheca fuliginea (Schlech) Salmon.)

The leaves of cucumber plants, cv. Marketer, cultivated in a pot in a conditioned ambient environment, were sprayed on their upper faces with an aqueous suspension of conides of *Sphaerotheca fuliginea* (200,000 conides/cc). After 24 hours the leaves were treated with the product being tested in a water-acetone solution at 20% of acetone (vol./vol.) by spraying both faces.

At the conclusion of the incubation period (8 days) the extent of the infection was evaluated at sight according to an evaluation scale ranging from 100 (sound plant) to 0 (fully infected plant).

Compound No.	Dose % Compound	Activity
49	0.3	80
46	0.3	80
40	0.3	100
50	0.3	100
51	0.3	100
17	0.3	87
47	0.3	100

Example 8

Immunizing activity on cucumber oidium — (Sphaerotheca fuliginear (Schlech) Salmon.)

The leaves of cucumber plants, c.v. Marketer, cultivated in a pot in a conditioned ambient environment, were sprayed on their lower faces with the product being tested in a water-acetone solution at 20% acetone (vol./vol.). The plants were then maintained in a conditioned ambient environment for 6 days; on the 7th day the upper faces of the leaves were sprayed with an aqueous suspension of conides of *Sphaerotheca fuliginea* (200,000 conides/cc) then the plants were brought again into a conditioned ambient environment.

At the conclusion of the incubation period (8 days), the extent of the infection was evaluated at sight according to an evaluation scale ranging from 100 (sound plant) to 0 (fully infected plant).

Compound No.	Dose % Compound	Activity
40	0.1	100
47	0.1	50
51	0.1	74
17	0.1	40

Example 9

Curative activity on bean rust (Uromyces appendiculatus (Pers.) Link)

The leaves of the bean Borlotto di Vigevano, cultivated in a pot in a conditioned ambient environment, were sprayed on their lower faces with an aqueous suspension of spores of *Uromyces appendiculatus* (200,000 spores/cc). After a residence time of 24 hours in a humidity-saturated environment, the leaves were treated with the products being tested in a water-acetone solution at 20% of acetone (vol./vol.) by spraying both faces of the leaves.

At the conclusion of the incubation period (14 days) the extent of the infection was evaluated at sight according to an evaluation scale ranging from 100 (sound plant) to 0 (completely infected plant).

Compound No.	Dose % Compound	Activity
49	0.5	100
46	0.5	100
50	0.5	80
47	0.5	100

Example 10

Immunizing activity on bean rust — (Uromyces appendiculatus (Pers.) Link.)

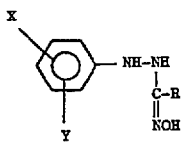
The leaves of the bean cv. Borlotto di Vigevano, cultivated in a pot in a conditioned ambient environment, were sprayed on their upper faces with the product being tested in a water-acetone solution at 20% of acetone (vol./vol.). The plants were then maintained in a conditioned ambient environment for 6 days; on the seventh day the lower faces of the leaves were sprayed with a suspension of spores of *Uromyces appendiculatus* (200,000 spores/cc). After a residence time of 24 hours in a humidity-saturated ambient environment, the plants were brought again into a conditioned ambient environment.

At the conclusion of the incubation period (14 days), the extent of the infection was evaluated at sight, accordingly to an evaluation scale ranging from 100 (sound plant to 0 (fully infected plant).

Compound No.	Dose % Compound	Activity
49	1	100
50	1	70
46	1	28
47	1	95

CLAIMS

1. A method of combatting infections due to fungi in useful plants, and/or of preventing such infections, and/or of immunizing the plants against such infections, in which the plants are treated with an arylhydrazo-aldoxime of the general formula:



(I)

or tautomers thereof, in which

X and Y independently represent a hydrogen or halogen atom, an alkyl group having 1 to 5 carbon atoms which may be halogenated, an alkoxyl group or NO_2, and

R represents a hydrogen atom, an alkyl group having 1 to 5 carbon atoms, a phenyl group or a substituted phenyl group,

as such or as an organic or inorganic salt or a suitable composition thereof at a dosage of 0.01% upwards.

2. A method as claimed in Claim 1 in which the arylhydrazo-aldoxime is applied in the form of a composition.

3. A method as claimed in Claim 1 substantially as herein described with reference to any one of Examples 3 to 10.

4. β - oximinomethyl - 4 - chlorophenyl - hydrazine.

5. β - (α - oximinoethyl) - 2 - methylphenyl - hydrazine.

6. β - (α - oximinoethyl) - 2 - methylphenyl - hydrazine hydrochloride.

7. β - (α - oximinoethyl) - 4 - methylphenyl - hydrazine hydrochloride.

8. β - (α - oximinoethyl) - 2 - chlorophenyl - hydrazine.

9. β - (α - oximinoethyl) - 2 - chlorophenyl - hydrazine hydrochloride.

10. β - (α - oximinoethyl) - 3 - chlorophenyl - hydrazine.

11. β - (α - oximinoethyl) - 3 - chlorophenyl - hydrazine hydrochloride.

12. β - (α - oximinoethyl) - 2 - fluorophenyl - hydrazine.

13. β - (α - oximinoethyl) - 2 - fluorophenyl - hydrazine hydrochloride.

14. β - (α - oximinoethyl) - 3 - trifluoromethylphenyl - hydrazine.

15. β - (α - oximinoethyl) - 3 - trifluoromethylphenyl - hydrazine hydrochloride.

16. β - (α - oximinoethyl) - 4 - methoxyphenyl - hydrazine.

17. β - (α - oximinoethyl) - 3, 5 - dimethylphenyl - hydrazine.

18. β - (α - oximinoethyl) - 3, 5 - dimethylphenyl - hydrazine hydrochloride.

19. β - (α - oximinoethyl) 3, 4 - dichlorophenyl - hydrazine.

20. β - (α - oximinoethyl) - 3, 4 - dichlorophenyl - hydrazine hydrochloride.

21. β - (α - oximinoethyl) - 3, 4 - dichlorophenyl - hydrazine oxalate.

22. β - (α - oximinoethyl) - 3, 4 - dichlorophenyl - hydrazine p-toluene sulphonate.

23. β - (α - oximinoethyl) - 3, 5 - dichlorophenyl - hydrazine.

24. β - (α - oximinoethyl) - 3, 5 - dichlorophenyl - hydrazine hydrochloride.

25. β - (α - oximinopropyl) - 2 - methylphenyl - hydrazine.

26. β - (α - oximinopropyl) - 2 - methylphenyl - hydrazine hydrochloride.

27. β - (α - oximinopropyl) - 4 - methylphenyl - hydrazine hydrochloride.

28. β - (α - oximinopropyl) - 2 - chlorophenyl - hydrazine.

29. β - (α - oximinopropyl) - 2 - chlorophenyl - hydrazine hydrochloride.

30. β - (α - oximinopropyl) - 3 - chlorophenyl - hydrazine.

31. β - (α - oximinopropyl) - 3 - chlorophenyl - hydrazine hydrochloride.

32. β - (α - oximinopropyl) - 4 - chlorophenyl - hydrazine.

33. β - (α - oximinopropyl) - 4 - chlorophenyl - hydrazine hydrochloride.

34. β - (α - oximinopropyl) - 3 - trifluoromethylphenyl - hydrazine.

35. β - (α - oximinopropyl) - 3 - trifluoromethylphenyl - hydrazine hydrochloride.

36. β - (α - oximinopropyl) - 3, 5 - dimethylphenyl - hydrazine hydrochloride.

37. β - (α - oximinopropyl) - 2, 6 - dimethylphenyl - hydrazine.

38. β - (α - oximinopropyl) - 2, 6 - dimethylphenyl - hydrazine hydrochloride.

39. β - (α - oximinopropyl) - 2 - methyl - 4 - chlorophenyl - hydrazine.

40. β - (α - oximinopropyl) - 2 - methyl - 4 - chlorophenyl - hydrazine hydrochloride.

41. β - (α - oximinopropyl) - 2 , 4 - dichlorophenyl - hydrazine.

42. β - (α - oximinopropyl) - 2, 4 - dichlorophenyl - hydrazine hydrochloride.

43. β - (α - oximinopropyl) - 3, 4 - dichlorophenyl - hydrazine.

44. β - (α - oximinopropyl) - 3, 4 - dichlorophenyl - hydrazine hydrochloride.

45. β - (α - oximinopropyl) - 3, 4 - dichlorophenyl - hydrazine oxalate.

46. β - (α - oximinopropyl) - 3, 4 - dichlorophenyl - hydrazine p - toluene sulphonate.

47. β - (α - oximinopropyl) - 3, 5 - dichlorophenyl - hydrazine.

48. β - (α - oximinopropyl) - 3, 5 - dichlorophenyl - hydrazine hydrochloride.

49. β - (α - oximino - 4 - chlorobenzyl) - phenyl - hydrazine hydrochloride.

50. β - (α - oximino - 3, 4 - dichlorobenzyl) - phenyl - hydrazine hydrochloride.

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