The Effect of a New Acaricide, Phenothiocarb (*S*-(4-phenoxybutyl)*N,N*-dimethyl thiocarbamate)

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Phenothiocarb has been developed by Kumiai Chemical Ind. Co., Ltd. under the code number of BI-5452 as a new acaricide with low toxicity to mammals, fish and beneficial insects. It is effective on mites belonging to *Panonychus*. The LC-50 values of phenothiocarb for eggs, larvae, protonymphs, deutonymphs and female adults of *P. citri* were 8.9, 12.6, 15.2, 32.8 and 81 ppm, respectively. Its acaricidal activity varied according to the temperature at which it was tested. Activity at a low temperature (15°C) was higher than that at a high temperature (30°C). Phenothiocarb was very effective against not only a susceptible strain but also the dicofol or benzoximate resistant strains of *P. citri*. In field trials conducted for 3 years phenothiocarb sprayed at a concentration of 233–500 ppm showed an efficacy equal to dicofol and amitraz for the control of citrus red mite. The control effect in the field was better in autumn than in summer.

INTRODUCTION

MARSHALL (1955) predicted that mites attacking apples against which regular control programs are being carried out will become resistant to many acaricides. Resistance of European red mite to various acaricides has been reported from several areas in the United States and Canada.. JEPSON et al. (1958) reported that citrus red mite, *Panonychus citri* (McGREGOR), had become resistant to chlorfenson and 14 organic phosphate insecticides, such as parathion, demeton etc.. SEKI (1973) reported that in Japan the occurrence of the mite, *Panonychus citri*, which was resistant to organophosphate compounds was found in 1958. Frequent use of the acaricide dicofol after its introduction in Japan in 1962 has caused a resistance problem, which spread through most of the orchards and greenhouses until 1968 (NOMURA et al., 1973). Since the resistance of spider mites to many acaricides was a serious problem much effort was devoted to the development of new acaricides. We found that phenothiocarb was highly effective against the egg stage of the citrus red mite.

Phenothiocarb has been developed by Kumiai Chemical Ind. Co., Ltd. under the code number of BI-5452 as a new acaricide with low toxicity to mammals, fish and beneficial insects. This paper describes some of the results obtained during the course of our laboratory and field investigations.

MATERIALS AND METHODS

Mites. Citrus red mites, *Panonychus citri* (McGREGOR), were reared on satsuma orange trees in an Orchard at Kumiai Chemical Life Science Research Institute, Kikugawa-cho, Shizuoka Prefecture. European red mites, *Panonychus ulmi* KOCH, were reared on apple seedlings and two-spotted spider mites, *Tetranychus urticae* KOCH, were reared on potted kidney bean plants in the greenhouse at Kumiai Chemical Life Science Research Institute, Kogota-cho, Tooda-gun, Miyagi Prefecture. Dicofol and benzoximate resistant strains of citrus red mite were collected from a citrus orchard in Yamagiri, Shimizu City, Shizuoka Prefecture. Susceptible strains of these mites were reared on the Satsuma orange trees at Kumiai Chemical Life Science Institute.

Chemicals. Phenothiocarb (*S*-(4-phenoxybutyl) *N,N*-dimethylthiolcarbamate), technical grade (95%), was synthesized in our institute and 35% emulsifiable concentrate was formulated from the technical material. Dicofol (40% E.C.) was purchased from Nippon Noyaku Co., Ltd. (Osaka), benzoximate (40% E.C.) was formulated in Kumiai Chemical Co., Ltd., amitraz (40% E.C.) was purchased from Nissan Chemical Co., Ltd. (Tokyo), propargite (40% E.C.) was purchased from Nippon Noyaku Co., Ltd. (Osaka), cyhexatin (50% wettable powder) was formulated in our institute as were quinomethionate (50% W.P.) and bromopropylate (45% E.C.).

Toxicity against eggs of citrus red mites. Eggs were obtained by allowing 12–13 female adults to oviposit on the undersurface of a 1.5 cm diameter leaf disk of a mandarin orange held on a wet filter paper for 48 hr. The disk with eggs was dipped in a test solution for 5 seconds and was then placed on another filter paper until the solution evaporated. Three disks were treated at each dosage level and placed together on a wet filter paper on a plastic cup (60 ml). Eggs were incubated at 25°C for 9 days. The number of unhatched eggs was recorded and the mortality was calculated. ABBOTT's formula (ABBOTT, 1925) was used to correct the mortality percent.

Toxicity against adults of citrus red mites. Female adults were transferred onto the upper surface of a mandarin orange leaf placed on 0.4% agar plate in a 9 cm diameter petri dish (MATSUNAGA and FURUHASHI, 1972). The test solutions (3 ml each) were sprayed on the mites by a rotary-type spray tower (Mizuho Scientific Co., Ltd. Nagoya). The treated mites were incubated at 25°C and mortality was recorded. About one hundred mites were tested for each dosage level and mortality was again corrected by ABBOTT's formula.

Toxicity against various stages of citrus red mites. Eggs and larvae were obtained by confining 12–13 female adults to a leaf of mandarin orange placed on 0.4% agar in a 9 cm diameter petri dish for about 12 hr. Those adults were then removed and eggs were incubated at 25°C for several days until the emergence of larvae, protonymph and deutonymph. Just prior to spraying, all unhatched eggs and over-stage mites were removed (STAAL et al., 1975). The mites were treated in the same manner as mentioned above for the adults. Assays were run in 3 replications. The mortality and survivals were recorded at 48 hr after treatments.

Residual effectiveness (foliar residual mitecidal activity). Acaricidal activity by foliar residues was measured by inoculating 30 female adults of citrus red mites onto the upper surface of leaves obtained at appropriate intervals from mandarin orange trees

sprayed with test chemicals. All the sprayed plants were held in a greenhouse at 15–22°C. The solutions of phenothiocarb, dicofol and amitraz were sprayed by a hand sprayer onto the mandarin orange trees at concentrations of 500 ppm, 200 ppm and 200 ppm, respectively. These leaves were placed on 1% agar in a petri dish with a 9 cm diameter. Mortality was recorded 48 hr after confining the mites to the leaves sprayed and was corrected by ABOTT's formula.

Relationship between temperature and mortality of female adult mites. About 30 female adults of the citrus red mite were released onto the upper surface of a mandarin orange leaf on 1% agar in a petri dish with a 9 cm diameter. Treatments of the mites were conducted by spraying 3 ml of test solutions at 100 ppm by a rotary type spray tower. Treated mites were kept at 15°C, 22°C and 30°C respectively, and their mortality and survival were counted at 48 hr after the treatment.

The control effect of phenothiocarb by a pot test. All of the stages of the citrus red mite except eggs were transferred onto the leaves of seedlings of the summer orange. They were kept in a greenhouse and eggs were deposited on the leaves for 24 hr. Then chemicals were applied to those plants at a concentration of 250 ppm with a spray gun. Host plants were held in the greenhouse and the mites were counted just before application and every seventh day after application. The control effects of test compounds were obtained by counting the number of mites. Experiments were also performed for European red mites on leaves of apple seedlings and two-spotted spider mites on leaves of lima beans. All assays were repeated three times. Control effects were calculated by OKUDAI's method (1973). The method of calculation is shown by the following formula

$$\text{Control effect} = \left(1 - \frac{C_b \sum_{i=1}^n T_{ai}}{T_b \sum_{i=1}^n C_{ai}} \right) \times 100$$

n = Times of inquiry after spraying

C_b = Number of mites on non-treatment before spraying

C_{ai} = Number of mites on non-treatment at i-time after spraying

T_b = Number of mites on a treatment before spraying

T_{ai} = Number of mites on a treatment at i-time after spraying

Field test. Mandarin orange trees infested with citrus red mites were sprayed with a handy power sprayer. The mites were counted just before application and every seventh day after the treatment. For each treatment, the number of adults was counted on 100 leaves chosen at random from the fixed trees. Control effects of test compounds were obtained in the same manner as mentioned above for the pot test. Date and weather conditions at time of spraying.

Date: September 20th, 1979

The weather conditions at time of spraying: Fine.

Rainfall during the test:

September 21st; 38 mm, Sep. 24th; 27.4 mm, Sep. 25th; 17 mm, Sep. 26th; 19.8 mm, Sep. 27th; 34.5 mm, Sep. 28th; 38.2 mm, Sep. 29th; 10.3 mm, Sep. 30th; 22.1 mm, Oct. 2nd; 3.7 mm, Oct. 3rd; 4.2 mm, Oct. 4th; 5.0 mm, Oct. 6th; 61.0 mm, Oct. 7th; 171 mm, Oct. 13th; 110 mm.

Maximum temperature during test: 30.5°C.

Minimum temperature during test: 10.5°C.

Average temperature: 22.0°C.

RESULTS

The properties of phenothiocarb are shown in Table 1. It is stable to both acid and alkali and to heat and melts at a relatively low temperature, 44–45°C. The toxicity of phenothiocarb to mammals and fish is shown in Table 2; its toxicity to mice, to fish like carp, and to beneficial insects like honeybee and silk worm is low.

Table 3 presents data for the LC-50 values of 8 compounds against eggs 0–48 hr after deposit. The LC-50 values of phenothiocarb, dicofol, benzoximate, amitraz, cyhexatin, bromopropylate, quinomethionate and omite were 5.0, 38.6, 5.6, 9.8, 12.7, 141.1, 31.7 and 358.6 ppm respectively.

Table 1. CHEMICAL AND PHYSICAL PROPERTIES OF PHENOTHIOCARB

Chemical name	; S-(4-Phenoxybutyl)N,N-Dimethylthiocarbamate
Code Number	; B1-5452
Chemical structure	;
Melting point	; 44–45°C
Vapor pressure	; 1.25×10^{-6} mmHg at 23°C
Boiling point	; 155°C/0.22 mmHg
Appearance	; colorless crystal
Solubility	; soluble in alcohol, acetone, benzene, xylene, isoprene and methanol, scarcely soluble in water
Stability	; stable to both acid such as HCl(0.1N.) and alkali such as NaCl(0.1N.) stable to heat, no decomposition under 40°C for one month

Table 2. TOXICITY OF PHENOTHIOCARB TO MAMMALS, FISH AND INSECTS

Acute oral	LD-50	Mouse (femle)	more than 1,000 mg/kg
		Mouse (male)	more than 1,000 mg/kg
Fish	Tlm-48 hr	Carp	10–20 ppm
Beneficial insects	LD-50	Honey bee (topical per head)	200–400 µg/insect
		Honey bee (oral)	47 µg/insect
	LC-50	Silkworm (leaf dipping)	more than 500 ppm

Table 3. LC-50 VALUES OF PHENOTHIOCARB AND OTHER ACARICIDES AGAINST EGGS OF *Panonychus citri* (McGREGOR) : (MORTALITY 9 DAYS AFTER TREATMENT)

Chemical	Regression line	LC-50 (ppm)	Confidence limits (ppm)	n	χ^2	Pr
Phenothiocarb	$Y=5.269+3.923(X-0.768)$	5.01	4.51–5.51	3	2.666	$0.20 \ll 0.50$
Dicofol	$Y=5.158+2.023(X-1.664)$	38.55	34.02–43.41	3	3.495	$0.10 \ll 0.20$
Benzoximate	$Y=4.988+2.939(X-0.743)$	5.59	5.13–6.09	3	1.802	$0.50 \ll 0.95$
Amitraz	$Y=5.088+1.686(X-1.042)$	9.75	6.84–12.45	3	12.758	$0.10 \ll 0.20$
Cyhexatin	$Y=5.037+1.916(X-1.125)$	12.74	7.89–17.51	3	24.332	$0.01 \ll 0.02$
Bromopropylate	$Y=4.989+3.182(X-2.146)$	141.05	127.99–155.47	2	2.444	$0.10 \ll 0.20$
Quinomethionate	$Y=4.881+1.294(X-1.409)$	31.70	26.48–38.62	3	6.688	$0.20 \ll 0.50$
Omite	$Y=4.936+2.940(X-2.533)$	358.62	261.53–553.87	2	8.668	$0.20 \ll 0.50$

Fig. 1. Residual effectiveness of phenothiocarb against females of *Panonychus citri* (Compounds sprayed at 13–20°C. Symbols show mortalities 48 hr after treatment.)

Table 4. LC-50 VALUES OF PHENOTHIOCARB AND OTHER ACARICIDES AGAINST FEMALE ADULTS OF *Panonychus citri* (McGREGOR) : (MORTALITY 48 HR AFTER TREATMENTS)

Chemical	Regression line	LC-50 (ppm)	Confidence limits (ppm)	n	χ^2	Pr
Phenothiocarb	$Y=5.110+3.009 (x-1.948)$	81.64	67.83–97.40	3	0.861	$0.50 \ll 0.95$
Dicofol	$Y=4.972+3.678 (x-1.266)$	18.79	16.50–21.44	2	0.810	$0.20 \ll 0.50$
Benzoximate	$Y=5.033+5.138 (x-0.795)$	6.15	5.60–6.73	3	0.184	$0.95 \ll 0.99$
Amitraz	$Y=4.941+2.164 (x-1.169)$	15.57	12.14–20.31	2	0.209	$0.50 \ll 0.95$
Cyhexatin	$Y=5.346+1.848 (x-1.599)$	25.84	17.30–34.30	3	3.115	$0.20 \ll 0.50$
Bromopropylate	$Y=5.039+2.929 (x-1.362)$	22.33	19.31–25.76	3	3.354	$0.50 \ll 0.95$
Quinomethionate	$Y=5.031+2.166 (x-2.725)$	513.70	337.70–757.72	1	0.338	$0.50 \ll 0.95$
Omite	$Y=5.008+2.599 (x-1.611)$	40.54	33.60–48.88	3	0.886	$0.50 \ll 0.95$

Table 5. LC-50 VALUES OF PHENOTHIOCARB AGAINST EACH STAGE OF *Panonychus citri* (McGREGOR)

Development stage	Regression line	LC-50 (ppm)	Confidence limits (ppm)	n	Pr
Eggs 10 hrs after deposit	$Y=5+1.414 (X-1.218)$	19.3	15.4–25.2	5	$0.02 \ll 0.05$
Eggs 72–96 hr after deposit	$Y=5+1.925 (X-0.999)$	9.9	8.6–11.5	5	$0.02 \ll 0.80$
Eggs 0–24 hr before hatching	$Y=5+1.554 (X-0.982)$	9.6	7.5–10.5	5	$0.08 \ll 0.95$
Larvae	$Y=5+1.442 (X-1.092)$	12.3	10.7–14.2	4	$0.02 \ll 0.80$
Protonymphs	$Y=5+1.344 (X-1.104)$	15.2	12.0–20.0	3	$0.10 \ll 0.20$
Deutonymphs	$Y=5+1.720 (X-1.531)$	32.8	29.5–36.3	3	$0.20 \ll 0.80$
Female adults	$Y=5+2.958 (X-1.934)$	81.0	67.0–99.0	3	$0.50 \ll 0.95$

Comments : Mortality of eggs was examined 9 days after treatment.

Mortality of larvae, nymphs and adults was examined 48 hr after treatment.

Table 4 presents data for the LC-50 values of 8 acaricides against female adults. LC-50 value of phenothiocarb was 81.64 ppm, less than dicofol, amitraz, benzoximate and some others, but more effective than quinomethionate. Figure 1 shows residual effectiveness of phenothiocarb against female adults of *P. citri*. The duration of the

Fig. 2. Relation between temperature and mortality of adult females of *Panonychus citri* (Compounds sprayed at concentration of 100 ppm. Symbols show mortality 48 hr after treatment.)

Table 6. LC-50 VALUES FOR THE DICOFOL AND BENZOXIMATE RESISTANT STRAIN OF *Panonychus citri* (McGREGOR) AND R/S RATIO OF SOME ACARICIDES

Chemical	LC-50 (ppm) ^a		Degree of resistance (R/S)
	Susceptible strain (S)	Resistant strain (R)	
Phenothiocarb	5.0	7.7	1.5
Dicofol	38.6	834.6	21.6
Benzoximate	5.6	939.1	167.7

^a LC-50 values were computed from mortality of eggs examined 9 days after treatments.

residual effect was 5 days after application at 13–20°C. The residual effect of amitraz was weak at this temperature, and the duration of dicofol's effect was estimated to be more than 10 days after application.

Table 5 shows LC-50 values of phenothiocarb for each stage of citrus red mite. In the developmental stage of eggs, the LC-50 values for eggs 10 hr after deposit, 72–96 hr after deposit and 0–24 hr before hatching were 19.3, 9.9 and 9.6 ppm respectively. The LC-50 values for larvae, protonymphs, deutonymphs, and adults were 12.3, 15.2, 32.8 and 81.0 ppm, respectively.

Table 6 shows the acaricidal activity of phenothiocarb against citrus red mites resistant to dicofol and benzoximate. Although phenothiocarb, dicofol and benzoximate were effective against the sensitive strain, only phenothiocarb showed high efficacy to the resistant strain.

Fig. 2 shows the relation between temperature and mortality of female adults of *Panonychus citri*. An acaricide like amitraz generally shows a high control effect on citrus red mites when the temperature rises, however, phenothiocarb showed an increased effect on the adults when the temperature fell, especially to 15°C or 22°C. The effect of dicofol was not influenced by this sprayed concentration.

Table 7 shows the control effect of phenothiocarb against *Panonychus citri* (McGREGOR), *Panonychus ulmi* (Koch) and *Tetranychus urticae* (Koch). The control effect

Table 7a. CONTROL EFFECT OF PHENOTHIOCARB AGAINST *Panonychus citri*

Chemical	Conc. (ppm)	Number of mites						Control effect
		Pre-treatment	Post-treatment					
			2-days	7	14	19	21	
Phenothiocarb	250	72.3/pot	0.7	1	3.7	609	6.7	98
Dicofol	200	56.3	0.7	0.3	0.3	23.0	24.7	93
None	—	43.7	45.3	56.3	141.0	155.3	177.0	

Table 7b. CONTROL EFFECT OF PHENOTHIOCARB AGAINST *Panonychus ulmi*

Chemical	Conc. (ppm)	Number of mites					Control effect
		Pre-treatment	Post-treatment				
			3-days	8	15	22	
Phenothiocarb	250	93.0/pot	0	0	3.2	1.1	99
Dicofol	200	97.0	0	0	0	0	100
None	—	94.0	43.6	294.7	521.3	274.5	—

Table 7c. CONTROL EFFECT OF PHENOTHIOCARB AGAINST *Tetranychus urticae*

Chemical	Conc. (ppm)	Number of mites					Control effect
		Pre-treatment	Post-treatment				
			2-days	7	10	15	
Phenothiocarb	250	66.0/pot	43.9	16.7	9.1	18.2	83
Dicofol	200	36.0	36.0	0	0	2.8	98
None	—	43.7	45.3	56.3	141.0	155.3	—

Table 8 CONTROL EFFECT OF PHENOTHIOCARB AGAINST CITRUS RED MITE IN FIELD TRIALS

Chemical	Dosage (g/5,000 l/ha)	Number of mites					Control effect
		Pre-treatment	Post-treatment				
			4-days	11	18	25	
Phenothiocarb	1750	351.3/tree	3.3	1.3	2.3	2.3	99
Phenothiocarb	1450	184.5	1.7	0	0.3	2.0	99
Amitraz	1000	306.0	4.3	3.0	13.3	22.7	93
Dicofol	1500	359.3	8.7	1.3	41.3	43.7	92
None	—	324.0	418.3	190.0	238.7	192.7	—

was 98, 99 and 83 against *P. citri*, *P. ulmi* and *T. urticae* respectively. As this compound showed phytotoxicity to apple trees (Kokko strain), it cannot be sprayed on these trees.

Table 8 shows the control effect of phenothiocarb against citrus red mites in field trials. Phenothiocarb (1750 g and 1450 g/ha) was more effective than amitraz (100 g/ha) and dicofol (1500 g/ha).

DISCUSSION

HUISMAN et al. (1948) have reported that the most outstanding compound in variously substituted diphenylsulphones, diphenylsulphides and diphenylsulphoxides was proved to be tetradifon, which combined a high activity at all stages of spider mites except the adult stage. KENAGA and HUMMER (1949) discovered that the effect of chlorfenson against the eggs of two-spotted spider mites had the characteristic of having acaricidal activity against larvae just after hatching. In the present experiments, phenothiocarb was most active against the eggs of *P. citri*. Eggs to which the compound had been applied did not hatch. When eggs were treated at a concentration of less than LC-50, a few hatched larvae grew up to the adult stage, whereas eggs treated by chlorfenson died just after hatching. Therefore phenothiocarb showed an ovicidal and a larvicidal activity different from chlorfenson. The acaricidal activity of phenothiocarb against female adults was not as strong and lasted on the leaf surface for only a few days. SUGAWARA (1973) reported that an acaricide like chlordimeform showed the highest toxicity against eggs of mites. HOPP (1954) observed that the susceptibility of eggs of *Tetranychus urticae* and *Panonychus ulmi* against chlorfenson decreased as the stage of eggs developed. The acaricidal activity of phenothiocarb was the most effective against eggs just before hatching and decreased with the progress of the mite stages. The mode of action of phenothiocarb was similar to that of chlordimeform which showed the highest activity to the last stage of eggs but was different from that of PPPS. NISHINO (1973) reported that benzoximate showed a residual effectiveness of more than 12 days, which was longer than that of dicofol, against the eggs of *Panonychus citri*. Phenothiocarb has a longer residual effectiveness than amitraz but shorter than dicofol at 17–20°C. The duration of the residual effectiveness of phenothiocarb was thought to be 5 days, sufficient to control a mite population at low levels for about a month after spraying. SUGAWARA (1973) found that the efficacy of some acaricides was strongly influenced by temperature and that of dicofol, dimethoate and chlorfenson against citrus red mites was excellent at both a high temperature of about 20°C and at a low temperature. From these facts, the timings preferable for spraying to utilize the efficacy of the acaricides were suggested. In the case of phenothiocarb, it showed a higher effectiveness on the female adult at 15–22°C than at 30°C. This suggests that phenothiocarb is more suitable for the control of *P. citri* in spring or autumn than in summer in Japan.

SEKI (1973) reported that in Saga prefecture LC-50 value of a susceptible strain of citrus red mite (Ogi-strain) against tetradifon was 48 ppm and that of a resistant strain (Motooka-strain) was 1,120 ppm. The efficacy of dimethoate against the citrus red mite has disappeared completely since 1965. The resistance of the mite to dicofol has developed since 1968. SARTO (1978) reported that the benzoximate resistant strain of citrus red mites bred at the Fruit Tree Research Station, Akitsu Branch showed an LC-50 value of 5,000 ppm for benzoximate for the female adults and R/S ratio of more than 1250 times. Thus in orchards of Japan many species of mites such as *Panonychus citri* (McGREGOR) and *Panonychus ulmi* (KOCH) have developed resistance to acaricides such as dimethoate, dicofol, tetradifon, phenkapton, and benzoximate. MATSUMOTO and SHINKAJI (1974) discussed the cross-resistance of citrus red mites by testing the resistant strain against dicofol. The R/S ratio of the strain was 13.0 against dicofol, 3.43 against chlorobenzilate, 46.38 against dimethoate and 4.14

against bromopropylate. In our investigation, phenothiocarb did not meet with cross-resistance in the dicofol and benzoximate resistant strains of citrus red mite and showed excellent efficacy against eggs of the resistant strains, suggesting that it can be expected to control those resistant strains of citrus red mite. METCALF (1948) reported that DCPM showed a higher efficacy against *Panonychus citri* than chlorfenethol but lower efficacy against *Tetranychus urticae*. SATO (1976) also reported that benzoximate showed high efficacy against *Panonychus* and a low efficacy against *Tetranychus*. DCPM and benzoximate showed a high efficacy against only *Panonychus* and a low efficacy against *Tetranychus*; phenothiocarb also showed a high efficacy against *Panonychus* and lower efficacy against *Tetranychus*. OATMAN (1959) studied the control of European red mite on apple plants in Wisconsin. Genite, mitox and chlorfenson applied as pre-blossom acaricides provided season-long control of this mite on apple trees.

Aramite, chlorobenzilate, dimeton, dicofol and mitox used as summer foliar sprays were effective in reducing mite populations and maintaining them at low levels for the entire season. CLEVELAND (1958) studied the effectiveness of early-season sprays against the European red mite and of summer sprays against both this mite and two-spotted spider mites on apple trees. He reported that application of dicofol and Guthion in July gave less than one mite per leaf and thimet, trithion, aramite, chlorobenzilate and ethion less than four mites per leaf after 4 weeks; chlorfenson, however, was not as effective as the other materials. As stated above, those acaricides have a different control effect because of their chemical characteristics at spraying time. In the field trial to control the citrus red mite in September in Japan, phenothiocarb showed excellent control effect.

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