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Stabilization of Thin Films of Pyrethrins and Allethrin

Raymond P. Miskus* and Theresa L. Andrews

4 hr.

complete destruction in 4 hr.

The combination of an antioxidant and an ultraviolet screening agent in a mineral oil formulation can significantly stabilize pyrethroids for at least

Nonstabilized formulations show almost

yrethrins and analogous compounds have long been known as powerful insecticides, and are effective against a wide variety of arthropods. The term pyrethroids in this paper includes the natural insecticidal constituents of pyrethrin flowers, the pyrethrin, and the synthetic compound allethrin. For a review of the pyrethroids and their composition see Metcalf (1955) or Elliot (1969).



Most pyrethroids have the important practical aspect of a low order of toxicity to warm-blooded animals, and the advantage of a very low persistence of less than 4 hr. Low persistence is also disadvantageous-pyrethroids are highly unstable in the field; they rapidly convert to products having little insecticidal activity (Crowe et al., 1961).

Their high instability when exposed to air and light (Stahl, 1960; Chen and Casida, 1969), as well as their high cost, have kept pyrethroids from finding much use in agriculture. Their primary use had been in the control of household pests, such as housefly, mosquito, and cockroach.

Many attempts have been made to stabilize pyrethroids and to prolong their effective life. The results evidently were not satisfactory, since they have not been commercially used. A few examples are the addition of trialkylphenols (Smith and Hill, 1947), the addition of 4-aminoazobenzene (Smith and Templin, 1956), and the addition of Food Yellow 10 (2,4dihydroxyazobenzene) (Warner, 1963). Many other patents on stabilization are available, but none have been effective in the field, and are not used. The addition of Food Yellow 10 provides about 50% stabilization after exposure to sunlight for 4 hr, but has the undesirable feature of being a staining dye.

We have eliminated the use of staining dyes, and have discovered that the precise range of ultraviolet radiation that induces pyrethroid destruction is 290 to 320 nm. This range of photoreactivity in the near ultraviolet was found by measuring the transmittance of borosilicate glass and that of soft or window glass. Soft glass transmits radiation only above 300 nm, while borosilicate glass transmits radiation beginning at 290 nm. Pyrethroids were not destroyed when sunlight was filtered through soft glass, but were rapidly destroyed when exposed to sunlight filtered through borosilicate glass. Pyrethroids were not destroyed when exposed to the atmosphere in the dark, nor did any loss occur through evaporation. This difference explains the increased stability of pyrethrins in homes and greenhouses, and also corresponds to the erythermal spectra of sunlight (Das Gupta, 1962). Once this was realized we needed to find materials compatible with pyrethroids that would also be photostable and absorb in the 290 to 320 nm range.

The use of ultraviolet absorbers alone did not provide complete stability (Table II). Even more stabilization was provided by the use of certain oil-soluble antioxidants, although previous trials were not completely successful (Eddy, 1951; Head and Jones-Glynne, 1965; Bell and Kido, 1965).

METHODS AND MATERIALS

The primary materials tested were 20% purified pyrethrins (Fairfield Chemical Co., and the McLaughlin Gormley King Co.) and allethrin (City Chemical Co.). The stock solutions were refrigerated in dark brown bottles; they remained stable

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	Same tot 1	
Composition	Cinerin I	Pyrethrin I
1% pyrethrins in:		
Hexane	6	2
Kerosene	7	4
<i>n</i> -butyl phthalate	7	13
Mineral oil	67	59
Isopar 450	34	34
10% ethylene glycol monobutyl ether in Isopar M (Humble		
Oil)	6	2
10% ethylene glycol monobutyl ether in mineral oil	66	49
1% allethrin in: Hexane	4	

Table I.	Percent of	of Cinerin	I, F	yrethrin	I, a	nd .	Allethrin
Fo	ound after	Exposure	to	Sunlight	for	4 H	ſr

for more than a year. Diluted samples were made daily and discarded after use. Chemical composition was determined by gas-liquid chromatography using a 1/8-in. o.d. glass column containing Chromosorb W 60/80 mesh coated with 5% S.E. 30 at a temperature of 190°C using an electron capture detector (Donegan *et al.*, 1962; Head, 1966). The test formulations were uniformly coated upon 2- \times 8-in. glass slides using 1.0 ml total volume to apply 10 mg of pyrethroid. These slides were exposed to sunlight for 4 hr or until such times as most of the unstabilized pyrethroids had broken down. The light mineral oil we used is available under the trade name Klearol (Sonnenborn Co.).

The test formulations were made to contain 1% weight to volume of each of the additives, such as antioxidants, light absorbers, and pyrethroids.

After exposure, the residue was recovered by washing the slides with distilled *n*-hexane and diluting to volume so that

Table II.Percent of Cinerin I and Pyrethrin I Found in Combination of Pyrethrins and Additives after Exposure to Sunlight for
4 Hr.4 Hr.Pyrethrins, Antioxidants, and uv Screens Present in 1% Weight to Volume Concentration

Pyrethrins with:2-Hydroxy-4-(2-hydroxy-3-meth- acryl(xy)propoxybenzophe- none + 2.6-dioctadey) para-cress in hexane2-Hydroxy-4-(2-hydroxy-3-meth- butyl ether in isoparafim-4506782Amyl para-dimethylaminobenzoate in mineral oil7765butyl ether in isoparafim-4506782Glyceryl para-aminobenzoate in bexane20acryl(xy)propoxybenzophenone1718171Ethyl cinnamate in nikarae ethyl cinnamate in mineral oil106259none-5-sulfonic acid + 2.6-di- octadeyl para-cress in 728171Benzyl cinnamate in mineral oil glycol monobutyl ether in mineral oil sobutyl cinnamate in hersane ethyl cinnamate in hersane79682-Hydroxy-4-(2-hydroxy-3-meth- mineral oil9085Benzyl cinnamate in mineral oil loobutyl ether in mineral oil bobutyl cinnamate in nineral oil bobutyl cinnamate in nineral oil loobutyl ether in mineral oil sobutyl cinnamate in nineral oil bobutyl cinnamate in mineral oil bobutyl cinnamate in mineral oil real oil7765CHydroxy-4-methoxy benzophe- mone 5-sulfonic acid in <i>n</i> -butyl phthalate827Bobutyl cinnamate in mineral oil real oil7765CHydroxy-4-methoxy benzophe- mone 5-sulfonic acid in <i>n</i> -butyl ethylen glycol monobutyl ether in mineral oil tootadeyl para-cresol in 10% thylene glycol monobutyl ether in mineral oil sobutyl cinnamate in mineral oil tootadeyl para-cresol in 10% thylene glycol monobutyl ether in mineral oil tootadeyl para-dimethylaminobenzoate in mineral oil776860CHydroxy-4-(2-hydroxy-3-meth- acryls(oxy)moropxybenzop	Composition	Cinerin I	Pyrethrin I	Composition	Cinerin I	Pyrethrin I
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eral oil6259none-5-sulfonic acid $+ 2,6-di-$ Ethyl cinnamate in nineral oil9372octadecyl para-cresol in 1% eth- ylene glycol monobutyl ether in min- mineral oil9085Benzyl cinnamate in mineral oil79682-Hydroxy-4/2-hydroxy-3-meth- acrylyloxy)propoxybenzophe- none in 10% ethylene glycol9085Isobutyl cinnamate in mineral oil79682-Hydroxy-4/2-hydroxy-3-meth- acrylyloxy)propoxybenzophe- none-5-sulfonic acid in <i>n</i> -butyl glycol monobutyl ether in mineral oil9377Isobutyl cinnamate in nimeral oil7765none-5-sulfonic acid in <i>n</i> -butyl phthalate827Isobutyl cinnamate in mineral oil7765none-5-sulfonic acid in 10% ethylene glycol monobutyl ether in mineral oil9634Isobutyl cinnamate in mineral oil7765none-5-sulfonic acid in 10% ethylene glycol monobutyl ether in mineral oil96342-Hydroxy-4-(2-hydroxy-3-meth- acrylyloxy)propoxybenzophe- none in <i>n</i> -butyl phthalate11282.6-Di- <i>terr</i> -butyl-4-methylphenol in hexare ethylene glycol monobutyl ether in mineral oil76632-Hydroxy-4-(2-hydroxy-3-meth- acrylyloxy)propoxybenzophe- none in <i>n</i> -butyl phthalate1128amylenethylenethylenethylene in mineral oil76632-Hydroxy-4-(2-hydroxy-3-meth- acrylyloxy)propoxybenzophe- none in <i>n</i> -butyl phthalate7663632-Hydroxy-4-(2-hydroxy-3-meth- acrylyloxy)propoxybenzophe- none + 2.6-dioctadecyl para-cresol in mineral oil solutyl ether in mineral oil76 <td>glycol monobutyl ether in min-</td> <td></td> <td></td> <td>2-Hydroxy-4-methoxy benzophe-</td> <td></td> <td></td>	glycol monobutyl ether in min-			2-Hydroxy-4-methoxy benzophe-		
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Benzyl cinnamate in 10% ethylene glycol monobutyl ether in min- eral oilylene glycol monobutyl ether in min- mineral oil9085Benzyl cinnamate in mineral oil79682-Hydroxy-4/2(2hydroxy-3-meth- acrylyloxy)propoxybenzophe- nome-sulfonic acid in <i>n</i> -butyl monobutyl ether in mineral oil9087Isobutyl cinnamate in 10% ethylene glycol monobutyl ether in mineral oil16none in 10% ethylene glycol monobutyl ether in mineral oil9377Isobutyl cinnamate in 10% ethylene glycol monobutyl ether in mineral oil1765none-s-sulfonic acid in <i>n</i> -butyl mone-s-sulfonic acid in 10% ethylene glycol monobutyl ether acrylyloxy)propoxybenzophe- none in kerosene96342-Hydroxy-4(2-hydroxy-3-meth- acrylyloxy)propoxybenzophe- none in hetrosene423526-Diotadecyl-para-cresol in 10% atrylene glycol monobutyl ether in mineral oil76632-Hydroxy-4(2-hydroxy-3-meth- acrylyloxy)propoxybenzophe- none in hetrosene122666662-Hydroxy-4(2-hydroxy-3-meth- acrylyloxy)propoxybenzophe- in mineral oil7663632-Hydroxy-4(2-hydroxy-3-meth- acrylyloxy)propoxybenzophe- none + 2,6-dioctadecyl para-cresol in 10% decyl para-cresol in nimeral oil76632-Hydroxy-4(2-hydroxy-3-meth- acrylyloxy)propoxybenzophe- none + 2,6-dioctadecyl para-cresol in 10% decyl para-cresol in nimeral oil76632-Hydroxy-4(2-hydroxy-3-meth- acrylyloxy)propoxybenzophe- none + 2,6-dioctadecyl para-cresol in 10% decyl para-cresol in 10% decyl para-cresol in 10% decyl para-cresol in nimeral oil7663<	Ethyl cinnamate in mineral oil	93	72	octadecyl para-cresol in 1% eth-		
mineral oil9085mineral oil9085eral oilphical colspan="2">mineral oil9085Benzyl cinnamate in mineral oil70682.112085Isobutyl cinnamate in mineral oil15312.14ydroxy-4.4(2hydroxy-3-meth- acrylyloxy)propoxybenzophe- none in mineral oil9085glycol monobutyl ether in mineral oil71Isobutyl cinnamate in mineral oil768015312.14ydroxy-4.(2hydroxy-3-meth- acrylyloxy)propoxybenzophe- none in mineral oil7755Isobutyl cinnamate in mineral oil77Isobutyl cinnamate in mineral oil7714/droxy-4.(2hydroxy-3-meth- acrylyloxy)propoxybenzophe- in mineral oil9634Isobutyl cinnamate + 2.6-diocta- decyl para-cresol in 10% decyl para-cresol in 10% decyl para-cresol in mineral oil76632.6-Dioctadecyl-para- cresol in 10% decyl para-cresol in mineral oil76632.6-Dioctadecyl-para- cresol in 10% decyl para-cresol in mineral oil76632.6-Dioctadecyl-para- 	Benzyl cinnamate in 10% ethylene			ylene glycol monobutyl ether in	•••	0.5
eral of Benzyl cinnamate in mineral oil7968 682-Hydroxy-4(2-hydroxy-s-meth- acrylyloxy)propoxybenzophe- none in 10% ethylene glycol monobutyl ether in mineral oil7768 6071 acrylyloxy)propoxybenzophe- none in 10% ethylene glycol monobutyl ether in mineral oil9377Isobutyl cinnamate in 10% ethylene glycol monobutyl ether in mineral oil acrylyloxy)propoxybenzophe- none in bettyl pithalate15312-Hydroxy-4-(2-hydroxy-4-methoxy benzophe- none-5-sulfonic acid in n-butyl ethylene glycol monobutyl ether in mineral oil8272-Hydroxy-4(2-hydroxy-3-meth- acrylyloxy)propoxybenzophe- none in bettyl pithalate1765none-5-sulfonic acid in 10% ethylene glycol monobutyl ether in mineral oil96342-Hydroxy-4(2-hydroxy-3-meth- acrylyloxy)propoxybenzophe- none in -butyl pithalate112828662-Hydroxy-4(2-hydroxy-3-meth- acrylyloxy)propoxybenzophe- none in -butyl pithalate112828662-Hydroxy-4(2-hydroxy-3-meth- acrylyloxy)propoxybenzophe- reral oil7663632-Hydroxy-4(2-hydroxy-3-meth- acrylyloxy)propoxybenzophe- none + 2,6-dioctadecyl para- cresol in mineral oil76632-Hydroxy-4(2-hydroxy-3-meth- acrylyloxy)propoxybenzophe- in mineral oil76632-Hydroxy-4(2-hydroxy-3-meth- acrylyloxy)propoxybenzophe- none + 2,6-dioctadecyl para- resol in mineral oil76632-Hydroxy-4(2-hydroxy-3-meth- acrylyloxy)propoxybenzophe- in mineral oil76632-Hydroxy-4(2-hydroxy-3-meth- acrylyloxy)propoxybenzophe- non	glycol monobutyl ether in min-	-0	60	mineral oil	90	85
Benzyl cinnamate in mineral oil8071acrylyloxylpropoxybenzophe- monobutyl ether in mineral oil9377Isobutyl cinnamate in n-butyl15312-Hydroxy-4, methoxy benzophe- none-5-sulfonic acid in n-butyl9377glycol monobutyl ether in mineral oil68602-Hydroxy-4, methoxy benzophe- none-5-sulfonic acid in 10% ethylene glycol monobutyl ether in mineral oil8272-Hydroxy-4(2-hydroxy-3-meth- acrylyloxy)propoxybenzophe- none in kerosene7653342-Hydroxy-4(2-hydroxy-3-meth- acrylyloxy)propoxybenzophe- none in n-butyl phthalate112826-Di-terr-butyl-4-methylphenol in hexane96342-Hydroxy-4(2-hydroxy-3-meth- acrylyloxy)propoxybenzophe- none in n-butyl phthalate112826-Di-terr-butyl-4-methylphenol in metral oil76632-Hydroxy-4(2-hydroxy-3-meth- acrylyloxy)propoxybenzophe- none in n-butyl phthalate112826-Di-terr-butyl-4-methylphenol in metral oil76632-Hydroxy-4(2-hydroxy-3-meth- acrylyloxy)propoxybenzophe- none + 2.6-dioctadecyl-para-cresol in 10% ethylene glycol monobutyl ether in mineral oil76632-Hydroxy-4(2-hydroxy-3-meth- acrylyloxy)propoxybenzophe- none + 2.6-dioctadecyl para-cresol in 10% ethylene glycol monobutyl ether in mineral oil70802-Hydroxy-4(2-hydroxy-3-meth- acrylyloxy)propoxybenzophe- none + 2.6-dioctadecyl para-cresol in 10% ethylene glycol monobutyl ether in mineral oil79782-Hydroxy-4(2-hydroxy-3-meth- acrylyloxy)propoxybenzophe- none + 2.6-dioctadecyl para-cresol in mineral oil	eral oil	79	68	2-Hydroxy-4-(2-hydroxy-3-meth-		
Isobutyl cinnamate in kerosene1016none in 10% ethylene glycolIsobutyl cinnamate in 10% ethylene15312-Hydroxy-4-methoxy benzophe- none-5-sulfonic acid in n-butylglycol monobutyl ether in min- eral oil68602-Hydroxy-4-methoxy benzophe- none-5-sulfonic acid in 10%Isobutyl cinnamate in mineral oil7765none-5-sulfonic acid in 10%Isobutyl cinnamate in mineral oil7765none-5-sulfonic acid in 10%-Hydroxy-4(2-hydroxy-3-meth- acrylyloxy)propoxybenzophe- none in <i>m</i> -butyl phthalate1765-Attydroxy-4(2-hydroxy-3-meth- acrylyloxy)propoxybenzophe- none in <i>m</i> -butyl phthalate1128Isobutyl cinnamate + 2,6-diocta- decyl para-cresol in 10% ethylene arrylyloxy)propoxybenzophe- real oil76632.Hydroxy-4(2-hydroxy-3-meth- acrylyloxy)propoxybenzophe- none in <i>m</i> -butyl phthalate112828Isobutyl cinnamate + 2,6-diocta- decyl para-cresol in mineral oil7266Amyl para-dimethylaminobenzo- ate + 2,6-dioctadecyl-para- cresol in 10% ethylene glycol94782.Hydroxy-4(2-hydroxy-3-meth- acrylyloxy)propoxybenzophe- none + 2,6-dioctadecyl para- cresol in 10% dibutyl phthalate in mineral oil9094942.Hydroxy-4(2-hydroxy-3-meth- acrylyloxy)propoxybenzophe- none + 2,6-dioctadecyl para- cresol in 10% dibutyl phthalate in mineral oil9094942.Hydroxy-4(2-hydroxy-3-meth- acrylyloxy)propoxybenzophe- none + 2,6-dioctadecyl para- cresol in 10% dibutyl phthalate in mineral oil9094942	Benzyl cinnamate in mineral oil	80	71	acrylyloxy)propoxybenzophe-		
Isobutyl cinnamate in <i>n</i> -butylIsobutyl cinnamate in <i>n</i> -butylIsobutyl cinnamate in <i>n</i> -butyl <i>n</i> Isobutyl cinnamate in 10% ethylene15312-Hydroxy-4-methoxy benzophe- none-5-sulfonic acid in <i>n</i> -butyl827eral oil68602-Hydroxy-4-methoxy benzophe- none-5-sulfonic acid in 10% ethylene glycol monobutyl ether in mineral oil96342-Hydroxy-4(2-hydroxy-3-meth- acrylyloxy)propoxybenzophe- none in <i>n</i> -butyl phthalate11282,6-Di- <i>tert</i> -butyl-4-methylphenol in hexane96342-Hydroxy-4(2-hydroxy-3-meth- acrylyloxy)propoxybenzophe- none in <i>n</i> -butyl phthalate11282826-Di- <i>tert</i> -butyl-4-methylphenol in hexane14552,6-Dictatecyl-para-cresol in 10% ethylene glycol monobutyl ether in mineral oil7663632,6-Dictatecyl-para-cresol in 10% ethylene glycol monobutyl ether in mineral oil947878glycol monobutyl ether in mineral oil7266Amyl para-dimethylaminobenzo- acresol in 10% ethylene glycol monobutyl ether in mineral oil90942-Hydroxy-4(2-hydroxy-3-meth- acrylyloxy)propoxybenzophe- none + 2,6-dioctadecyl para-cresol in in mineral oil9094782-Hydroxy-4(2-hydroxy-3-meth- acrylyloxy)propoxybenzophe- in mineral oil9094942-Hydroxy-4(2-hydroxy-3-meth- acrylyloxy)propoxybenzophe- in mineral oil9094942-Hydroxy-4(2-hydroxy-3-meth- acrylyloxypropoxybenzophe- in mineral oil9094942-Hydroxy-4(2-hydroxy-3-meth- acrylyloxypro	Isobutyl cinnamate in kerosene	10	16	none in 10% ethylene glycol	02	77
pinnalate153121-Hydroxy-4-methoxy benzophe- none-5-sulfonic acid in n-butyl phthalateIsobutyl cinnamate in mineral oil68602-Hydroxy-4-methoxy benzophe- none-5-sulfonic acid in 10% ethylene glycol monobutyl ether in mineral oil8272-Hydroxy-4(2-hydroxy-3-meth- acrylyloxy)propoxybenzophe- none in kerosene42352,6-Di- <i>tert</i> -butyl-4-methylphenol in hcxane96342-Hydroxy-4(2-hydroxy-3-meth- acrylyloxy)propoxybenzophe- none in <i>k</i> -butyl phthalate1128282,6-Di- <i>tert</i> -butyl-4-methylphenol in hcxane14552-Hydroxy-4(2-hydroxy-3-meth- acrylyloxy)propoxybenzophe- reral oil76636363126-Diotadecyl-para-cresol in 10% ethylene glycol monobutyl ether in mineral oil7663631107266ate + 2,6-diocta- cresol in 10% ethylene glycol monobutyl ether in mineral oil94782107266ate + 2,6-diocta- cresol in 10% ethylene glycol monobutyl ether in mineral oil90942-Hydroxy-4(2-hydroxy-3-meth- acrylyloxy)propoxybenzophe- none + 2,6-dioctadecyl-para-cresol in 10% ethylene glycol moneral oil9094Para-cresol in 10% ethylene glycol moneb-tyle in mineral oil102832-Hydroxy-4(2-hydroxy-3-meth- acrylyloxy)propoxybenzophe- none + 2,6-dioctadecyl-para-cresol in mineral oil97802-Hydroxy-4(2-hydroxy-3-meth- acrylyloxy)propoxybenzophe- none + 2,6-dioctadecyl para-cresol in mineral oil97802-Hydroxy-4(2-hydroxy-3-meth- acrylyl	Isobutyl cinnamate in <i>n</i> -butyl	1.7	21	monobutyl ether in mineral oil	93	11
Isobutyl cinnamate in 10% ethylene glycol monobutyl ether in min- eral oil68602-Hydroxy-4-methoxy benzophe- none in kerosene7765none-5-sufforic acid in <i>n</i> -bulyl none-5-sufforic acid in <i>n</i> -bulyl2-Hydroxy-4-(2-hydroxy-3-meth- acrylyloxy)propoxybenzophe- none in kerosene42352,6-Di-terr-butyl-4-methylphenol in hexane96342-Hydroxy-4-(2-hydroxy-3-meth- acrylyloxy)propoxybenzophe- none in <i>n</i> -butyl phthalate112814552.Hydroxy-4-(2-hydroxy-3-meth- acrylyloxy)propoxybenzophe- none in <i>n</i> -butyl phthalate11282,6-Di-terr-butyl-4-methylphenol in hexane14551.Sobutyl cinnamate + 2,6-diocta- decyl para-cresol in 10% ethylene glycol monobutyl ether in min- eral oil7663632.Hydroxy-4(2-hydroxy-3-meth- acrylyloxy)propoxybenzophe- none + 2,6-dioctadecyl-para-cresol in 10% ethylene glycol monobutyl ether in mineral oil726666monobutyl ether in mineral oil94782.Hydroxy-4(2-hydroxy-3-meth- acrylyloxy)propoxybenzophe- none + 2,6-dioctadecyl para- resol in 10% dibutyl phthalate in mineral oil90949494782.Hydroxy-4(2-hydroxy-3-meth- acrylyloxy)propoxybenzophe- none + 2,6-dioctadecyl para-cresol in 10% dibutyl phthalate909494782.Hydroxy-4(2-hydroxy-3-meth- acrylyloxy)propoxybenzophe- none + 2,6-dioctadecyl para-cresol in 10% dibutyl phthalate909494782.Hydroxy-4(2-hydroxy-3-meth- acrylyloxy)propoxybenzophe- none + 2,6-dioctadecyl para-cresol in mineral oil9780	phinalate	15	31	2-Hydroxy-4-methoxy benzopne-		
glycol monobutyl ether in min- eral oil6860 2-Hydroxy-4-methoxy benzophe- none-5-sulfonic acid in 10% ethylene glycol monobutyl ether in mineral oil7765 ethylene glycol monobutyl ether in mineral oil96342-Hydroxy-4-(2-hydroxy-3-meth- acrylyloxy)propoxybenzophe- none in kerosene42352.6-Di- <i>tert</i> -butyl-4-methylphenol in hexane96342-Hydroxy-4-(2-hydroxy-3-meth- acrylyloxy)propoxybenzophe- none in <i>n</i> -butyl phthalate1128282.6-Dioctadecyl-para-cresol in 10% ethylene glycol monobutyl ether in mineral oil76632-Hydroxy-4-(2-hydroxy-3-meth- acrylyloxy)propoxybenzophe- none in <i>n</i> -butyl phthalate7266Amyl para-dimethylaminobenzo- ate + 2.6-dioctadecyl-para- cresol in 10% ethylene glycol monobutyl ether in mineral oil94782-Hydroxy-4-(2-hydroxy-3-meth- acrylyloxy)propoxybenzophe- none + 2.6-dioctadecyl para- cresol in 10% dibutyl phthalate in mineral oil7266Amyl para-dimethylaminobenzo- ate + 2.6-dioctadecyl-para- cresol in 10% ethylene glycol monobutyl ether in mineral oil886660782-Hydroxy-4-(2-hydroxy-3-meth- acrylyloxy)propoxybenzophe- none + 2.6-dioctadecyl para- cresol in 10% dibutyl phthalate in mineral oil909494782-Hydroxy-4-(2-hydroxy-3-meth- acrylyloxy)propoxybenzophe- none + 2.6-dioctadecyl para-cresol in mone- sol in 10% dibutyl phthalate in mineral oil9094942-Hydroxy-4-(2-hydroxy-3-meth- acrylyloxy)propoxybenzophe- none + 2.6-dioctadecyl para-cresol in mone- sol in 10% dibutyl phthalate in mineral	Isobutyl cinnamate in 10% ethylene			none-5-sulfonic acid in <i>n</i> -butyl	0	27
eral oil68602-Hydroxy-4-(1-hydroxy-3-meth- acrylyloxy)propoxybenzophe- none in kerosene7765none-5-sulforic acid in 10% ethylene glycol monobutyl ether in mineral oil96342.Hydroxy-4-(2-hydroxy-3-meth- acrylyloxy)propoxybenzophe- none in <i>n</i> -butyl phthalate4235 $2.6-Di-ert-ert-butyl-4-methylphenol inhexane96342.Hydroxy-4-(2-hydroxy-3-meth-acrylyloxy)propoxybenzophe-none in n-butyl phthalate112814552.6Di-otatdecyl-para-cresol in 10% ethyleneglycol monobutyl etherin mineral oil76633.butyl cinnamate + 2,6-diocta-decyl para-cresol in 10% ethyleneglycol monobutyl etherin mineral oil7266Amyl para-dimethylaminobenzo-acresol in 10% ethylene glycol1.Sobutyl cinnamate + 2,6-diocta-decyl para-cresol in mineral oil7266Amyl para-dimethylaminobenzo-acresol in 10% ethylene glycol2.Hydroxy-4.(2-hydroxy-3-meth-acrylyloxy)propoxybenzophe-in mineral oil8866monobutyl ether in mineral oilmonobutyl ether in mineral oil80582.Hydroxy-4.(2-hydroxy-3-meth-acrylyloxy)propoxybenzophe-in mineral oil9094Para-cresol in 10% ethylene glycolmonobutyl ether in mineral oil102832.Hydroxy-4.(2-hydroxy-3-meth-acrylyloxy)propoxybenzophe-none + 2,6-dioctadecyl para-cresol inmineral oil9094782.Hydroxy-4.(2-hydroxy-3-meth-acrylyloxy)propoxybenzophe-none + 2,6-dioctadecyl para-cresol inmineral oil97802.Hydroxy-4.(2-hydroxy-3-meth-acrylyloxy)propoxybenzophe-none$	glycol monobutyl ether in min-		<u>()</u>	prinalate	8	27
Isobutyl cinnamate in mineral oil7765none-5-sultonic acid in 10% ethylene glycol monobutyl ether in mineral oil96342-Hydroxy-4-(2-hydroxy-3-meth- acrylyloxy)propoxybenzophe- none in kerosene42352.6-Di-rerr-butyl-4-methylphenol in hexane96342-Hydroxy-4-(2-hydroxy-3-meth- acrylyloxy)propoxybenzophe- none in <i>n</i> -butyl phthalate1128282.6-Dioctadecyl-para-cresol in 10% ethylene glycol monobutyl ether in mineral oil76632.butyl cinnamate + 2.6-diocta- decyl para-cresol in mineral oil7266att + 2.6-dioctadecyl-para- cresol in 10% ethylene glycol monobutyl ether in mineral oil94782.Hydroxy-4-(2-hydroxy-3-meth- acrylyloxy)propoxybenzophe- none + 2.6-dioctadecyl para- cresol in 10% dibutyl phthalate in mineral oil8866monobutyl ether in mineral oil monobutyl ether in mineral oil902.Hydroxy-4-(2-hydroxy-3-meth- acrylyloxy)propoxybenzophe- none + 2.6-dioctadecyl para- cresol in 10% dibutyl phthalate in mineral oil909494972.Hydroxy-4-(2-hydroxy-3-meth- acrylyloxy)propoxybenzophe- none + 2.6-dioctadecyl para-cresol in 10% ethylene glycol102832.Hydroxy-4-(2-hydroxy-3-meth- acrylyloxy)propoxybenzophe- none + 2.6-dioctadecyl para-cresol in 10% ethylene glycol97802.Hydroxy-4-(2-hydroxy-3-meth- acrylyloxy)propoxybenzophe- none + 2.6-dioctadecyl para-cresol in 10% ethylene glycol97802.Hydroxy-4-(2-hydroxy-3-meth- acrylyloxy)propoxybenzophe- none + 2.6-dioctadecyl para-cresol in 10% ethylene glycol mono- butyl ether in mineral oi	eral oil	68	60	2-Hydroxy-4-methoxy benzophe-		
2-Hydroxy-4(2-hydroxy-3-meth- acrylyloxy)propoxybenzophe- none in kerosene42352,6-Di-tert-butyl-4-methylphenol in hexane96342-Hydroxy-4-(2-hydroxy-3-meth- acrylyloxy)propoxybenzophe- none in <i>n</i> -butyl phthalate11282814552.6-Diottadecyl-para-cresol in 10% ethylene glycol monobutyl ether in mineral oil1455563.6-Diottadecyl-para-cresol in 10% ethylene glycol monobutyl ether in mineral oil76633.72.6-Diottadecyl-para-cresol in 10% ethylene glycol monobutyl ether in mineral oil76633.82.6-Diottadecyl-para-cresol in min- eral oil7266Amyl para-dimethylaminobenzo- eresol in 10% ethylene glycol monobutyl ether in mineral oil783.72.6Amyl para-dimethylaminobenzo- eresol in 10% ethylene glycol monobutyl ether in mineral oil78783.72.6-dioctadecyl-para- cresol in 10% ethylene glycol monobutyl ether in mineral oil80583.86666102833.9909494783.10% ethylene glycol mono- butyl ether in mineral oil909494783.10% ethylene glycol popoxybenzophe- none + 2,6-dioctadecyl para- cresol in 10% ethylene glycol mone + 2,6-dioctadecyl para-cresol in 10% ethylene glycol102834.1490949478784.1490949478784.1490949478784.14909494<	Isobutyl cinnamate in mineral oil	77	65	none-5-suitonic acid in 10%		
acrylyloxy)propxybenzophe- none in kerosene4235 $2.6-Di-tert-butyl-4-methylphenol inhexane14552.Hydroxy-4(2-hydroxy-3-meth-acrylyloxy)propxybenzophe-none in n-butyl phthalate11281455Isobutyl cinnamate + 2.6-diocta-decyl para-cresol in 10% ethyleneglycol monobutyl ether in min-eral oil76632.Hydroxy-4(2-hydroxy-3-meth-acrylyloxy)propxybenzophe-none in 2.6-Dioctadecyl-para-cresol in min-eral oil76632.Hydroxy-4(2-hydroxy-3-meth-acrylyloxy)propxybenzophe-none + 2.6-dioctadecyl para-cresol in mineral oil7266Amyl para-dimethylaminobenzo-ate + 2.6-dioctadecyl-para-cresol in 10% ethylene glycolmone + 2.6-dioctadecyl para-cresol in 10% dibutyl phthalatein mineral oil9094782.Hydroxy-4.(2-hydroxy-3-meth-acrylyloxy)propxybenzophe-none + 2.6-dioctadecyl para-cresol in 10% dibutyl phthalatein mineral oil909494782.Hydroxy-4.(2-hydroxy-3-meth-acrylyloxy)propxybenzophe-none + 2.6-dioctadecyl para-cresol inolin 10% ethylene glycol mono-butyl ether in mineral oil909494782.Hydroxy-4.(2-hydroxy-3-meth-acrylyloxy)propxybenzophe-none + 2.6-dioctadecyl para-cresol inolin 10% ethylene glycol mono-butyl ether in mineral oil909494782.Hydroxy-4.(2-hydroxy-3-meth-acrylyloxy)propxybenzophe-none + 2.6-dioctadecyl para-cresol inolin 10% ethylene glycol mono-butyl ether in mineral oil95969696978030909094Benzyl cinnamate + 2.6-dioctad-cer$	2-Hydroxy-4-(2-hydroxy-3-meth-			in mineral oil	06	34
none in kerosene4235 $2.65D:26:26:26:26:10:10:10:10:10:10:10:10:10:10:10:10:10:$	acrylyloxy)propoxybenzophe-			111 Initial Off 26 Di test hutul 4 methylphenel in	90	54
2-Hydroxy-4(2-hydroxy-3-meth- acrylyloxy)propoxybenzophe- nome in <i>n</i> -butyl phthalate112816-Dioctadecyl-para-cresol in 10% ethylene glycol monobutyl ether 	none in kerosene	42	35	2,0-DI-tert-outyi-4-methyiphenorm	14	55
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eral oil7266Initial para initial para in	glycol monobutyl ether in min-			Amyl para-dimethylaminobenzo-		
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butyl ether in mineral oil9596decyl-para-cresol in 10% ethyl- ene glycol monobutyl ether in mineral oil93902-Hydroxy-4-(2-hydroxy-3-meth- acrylyloxy)propoxybenzophenone + 2,6-dioctadecyl para-cresol in 10% ethylene glycol9662decyl-para-cresol in 10% ethyl- ene glycol monobutyl ether in mineral oil93909662decyl-para-cresol in mineral oil10392	sol in 10% ethylene glycol mono-			Benzyl cinnamate + 2,6-diocta-		
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+ 2,6-dioctadecyl para-cresol inBenzyl cinnamate+ 2,6-diocta-10% ethylene glycol9662decyl-para-cresol in mineral oil10392	acrylyloxy)propoxybenzophenone			mineral oil	93	90
10% ethylene glycol 96 62 decyl-para-cresol in mineral oil 103 92	+ 2,6-dioctadecyl para-cresol in			Benzyl cinnamate + 2,6-diocta-	100	0.2
	10% ethylene glycol	96	62	decyl-para-cresol in mineral oil	103	92

5 μ l contained approximately 50 ng. The pyrethrins were quantized by using a disc integrator and the cinerin I and the pyrethrin I peaks from previously prepared calibration samples. Cinerin II and pyrethrin II peaks were not quantized. Tables I-IV showing the results of the additives therefore are characterized by the larger cinerin I and pyrethrin I peaks; allethrin is characterized by a single major peak.

Tables I-IV do not include the large number of antioxidants and light absorbers that did not show adequate stabilization, although they met the criteria of being antioxidants or uv screens in the critical 290 to 320 nm range.

DISCUSSION

The mechanism for photodecomposition of pyrethroids has not been elucidated completely by Chen and Casida (1969), however, they state that the acid portion of the molecule is oxidized stepwise to yield esters of trans-caronic acid. This mechanism is preferred over that proposed by Sasaki et al. (1968) for methyl trans-chrysanthemumate. Chen and Casida did not find cis-chrysanthemumic acid or meso, ciscaronic acid. Since oxidation occurs in the acid, the use of an antioxidant and a photoscreen seemed desirable to preserve pyrethroids.

For optimum stabilization of pyrethroids, a combination of antioxidants, solvent, and ultraviolet absorbant should be used. Many combinations of antioxidants and uv absorbers will provide enough stability (Table II), but not if each agent is used singly. The solvent selected is an equally important agent. In these tests, mineral oil was the preferred solvent for stability. It may be that a saturated paraffin is quite nonreactive and does not produce peroxides as do ketones or ethers.

The most useful of the uv screening agents are: first, the aromatic ketones in which two aromatic nuclei are attached directly to an oxogroup, e.g., the derivates of benzophenone; and second, the esters of aromatic acids, e.g., the esters of substituted benzoic acids. When antioxidants were incorporated into mixtures of pyrethroid toxicants and uv screening agents, we observed the greatest stabilization with those possessing an hydroxyl group attached directly to an aromatic nucleus and having 14 or more carbon atoms, e.g., 4-methyl-2,6-di-tert-butylphenol or 2,6-dioctadecyl-paracresol. Table IV shows the effect of sunlight on the toxicity of formulation with and without stabilization additives. It can also be seen that sun exposure readily destroys the toxic action of pyrethrins.

Stabilization of allethrin does not use mineral oil as a solvent (Table III). But if the other work with pyrethrins is comparable, stability should greatly improve with mineral oil as a solvent.

Combinations of 2,6-dioctadecyl-para-cresol and 2-hydroxy-4-(2-hydroxy-3-methacrylyloxy)propoxybenzophenone with pyrethrins have been field-tested. In 1969, applications of this formulation gave good control of hemlock looper (Mason, 1970). In the summer of 1970, the Canadian Department of Forestry and Fisheries obtained satisfactory control of spruce budworm (Choristoneura fumiferana), a major defoliator of eastern forests (Fettes, 1971).

Table III. Percent of Allethrin Remaining after 4 Hr Exposure to Sunlight. Allethrin, Antioxidant, and uv Absorber Present in 1% Weight to Volume Concentration

Composition	Allethrin
Allethrin $+$ 2-hydroxy-4-(2-hydroxy-3-meth- acrylyloxy)propoxybenzophenone in 10% ethylene glycol monobutyl ether in isopar	
"M"	66
Allethrin $+$ 2,6-dioctadecyl-para-cresol in 10% ethylene glycol monobutyl ether in isopar	
"M"	42
Allethrin + 2-hydroxy-4-(2-hydroxy-3-meth- acrylyloxy)propoxybenzophenone in 1%	
ethylene glycol ether in isopar "M"	84

Table IV. Effect of 4 Hr of Sunlight Exposure on Toxicity of			
Pyrethrins Formulation to Western Spruce Budworm. Pyre-			
thrins, Antioxidants, and uv Absorber Present in 1% Weight			
to Volume Concentration			

	after 7 days			
Composition	Unexposed	Exposed		
Pyrethrins in combination with: Hexane (control) 2-Hydroxy-4-(2-hydroxy-3-methacryl- yloxy)propoxybenzophenone + 2,6-dioctadecyl-para-cresol in 10% ethylene glycol monobutyl in min-	100	10		
eral oil Amyl para-dimethylaminobenzoate + 2,6-dioctadecyl-para-cresol in 10% ethylene glycol monobutyl	100	100		
ether in mineral oil	100	100		

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