Pyrethrum Growth in Illinois*

The Pyrethrin Content of Illinois Grown Pyrethrum

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The growth of pyrethrum in this country is receiving increased attention, due to its gradually augmented use as insecticide. At the close of the war the imports of pyrethrum in this country were about 2,000,000 pounds. By 1930 they had increased to 10,000,000, and now exceed 15,000,000 pounds per year. About 90% of this comes from Japan, and the balance from Europe and recently some from British East Africa. The species of pyrethrum which is commercially important is the chrysanthemum (Pyrethrum) cinerariaefolium (Trev.) Bocc. It is a glaucous perennial, slender, 12 to 24 inches high, stems unbranched with a few short scattered hairs below the flower. The leaves are long and petioled, silky beneath with distant segments; involucral scales scarious and whitish at the apex.[1]

The commercial product consists of the air-dried flowers which range from 6 to 24 mm. in width, and from 0.070 to 0.300 grams in weight.[2] The flowers are either powdered, and in admixtures with other ingredients form the active part of the so-called insect powders, or extracted, and the extracts used in sprays.

Too great a volume of literature on pyrethrum has accumulated to warrant any introduction in this paper. An excellent account, as well as an exhaustive bibliography, will be found in C. B. Gnadinger's book *Pyrethrum Flowers*, Second Edition, McLaughlin Gormley and King Company, Minneapolis, Minn., 1936.

The work here reported was begun in the spring of 1932. Its objectives were to determine whether pyrethrum could be grown under the conditions of soil and climate prevailing in northern Illinois, and also to study the influence of various fertilizers and plant catalysts on the pyrethrin content of the flowers.

At the time we began our work, there were few references to the growth of pyrethrum in this country. Since then, a number of reports of attempts to grow it have been published from many states. To my knowledge, this was the first experiment in Illinois. In 1934, another experiment was started at the Tribune Experimental Farm at Wheaton, Ill.

The seed from which the plants were started was imported from France, and was labeled *Dalmatian Pyrethrum*. The germination was extremely poor, the average being about 2%. No attempt was made to prove the truth of the

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[1] United States Department of Agriculture, Departmental Bulletin No. 824, p. 1-2, (1931).

[2] C. B. GNADINGER; Pyrethrum Flowers, p. 1 (1936).

statement that the seed is heated before it is sold so as to destroy its germinating power. From further comparative trials with seed collected from our own plants, it seems safe to state that the imported seed was of very poor quality.

The seed was sown in May, 1932, and the seedlings transplanted in September of the same year. Nine hundred plants were transplanted in one of the experimental fields of the department of Agronomy of the University of Illinois under the supervision and care of Dr. C. Alexopoulos of the Department of Botany (present address Kent State University, Kent, Ohio). This was a test to determine the growth under field crop conditions. The plot was half an acre of brown silt loam located in the south campus and adjoining the agronomy soil bins. Of these nine hundred plants, 855 survived the transplanting operations. No particular care was given the plants during the next three years; according to the report of Dr. Alexopoulos, at the time of the harvest the weeds were two feet high, and on the whole, there was very little cultivation, so that they were grown under the worst possible conditions.

The second group of plants, one hundred in number, was transplanted in a plot 30 x 25 ft., at the back of the author's home, 5558 Ardmore Avenue, Chicago. The field was an empty lot with clay loam over compact light clay subsoil. It had never been cultivated. It was divided into five sections and the plants distributed as follows:

TABLE I—DISTRIBUTION AND TREATMENT OF PLANTS

Plot Number	Number of Plants	Plants surviving, Spring, 1933	Treatment in Spring, 1933
1	20	18	None—Control
2	20	18	Complete fertilizer. $4/N—12/P_2O_5—4/K_2O$
3	21	21	Complete fertilizer plus Uranium Nitrate
4	19	18	Uranium Nitrate alone
5	20	19	Barnyard manure

Plot 2. Fertilizer at the rate of 20 g. per plant, also 0.5 g. $MnSO_4$ per plant.
Plot 3. Fertilizer at the rate of 20 g. per plant; uranium nitrate, 0.1 g. per plant.
Plot 4. Uranium nitrate, 2 g. per plant.
Plot 5. Manure, 400 g. per plant.
The rows were 15 inches apart and the plants spaced at intervals of 8 inches.

In these experiments, no attempt was made to study each plant as a unit, but rather each plot was considered as a unit. Inasmuch as the plots were not separated by concrete, this way of regarding them must be taken with some reservation, although the results were quite constant. For the control, that is, plants which were not treated with any fertilizers, etc., the plot selected was at a higher level than the others at the extreme end of the section. Otherwise, the plots were divided by a small ditch of 8 inches depth, and one foot wide, with drainage. Table I shows the distribution of plants and the treatment of each plot.

The treating of the plants with fertilizers, uranium salt and manure, was done during the middle of May, 1933. A similar treatment was given in May, 1934, and after that, no further treatment. In each case, the material was placed in a small circular furrow around the plant and six inches from the roots, then covered with soil.

The first harvesting in 1933 was very small, both in the Urbana and Chicago plots. This is usually the procedure. The flowers in the field crop were collected at half-closed stage, those at Chicago at the fully opened stage. As the amounts from the different experimental plots were too small (i. e., 5.8 to 6.7 g.) for a detailed chemical determination of pyrethrins, they were mixed to form one sample after being dried and weighed.

TABLE II—HARVEST OF 1933

Plot	Number of plants	Number of flowers	Fresh weight g.	Air-dry weight g.	Loss per cent	Average weight of flowers g.	Remarks
Field crop, Urbana	855	350 closed showing petals	1,050	304	71	0.086	
I	18	49	----------	5.8	76.5	0.119	Fully open
II	20	52	----------	6.0	76.4	0.115	Fully open
III	21	50	----------	6.1	77.0	0.122	Fully open
IV	19	51	----------	5.9	78.2	0.116	Fully open
V	20	51	----------	5.8	74.9	0.114	Fully open

TABLE III—HARVEST OF 1934

Plot	Number of plants	Fresh weight g.	Air-dry weight g.	Loss per cent	Average weight of flowers g.	Yield per plant g.
I	18	897	228	74.6	0.141	12.1
II	20	966	242	75.2	0.151	12.1
III	21	1,040	298	76.4	0.153	14.1
IV	19	1,001	248	75.3	0.160	13.0
V	20	796	201	74.8	0.140	10.05

The harvesting in 1934 was done between June 14th and 16th, and June 21st and 27th, while a third small collection was made on July 8th. The flowers were weighed after each collection and placed on racks and allowed to dry indoors. They were weighed again when dried, and then placed in bottles and placed on a shelf where they were kept until analyzed. The field crop of 1934 was not collected, but allowed to form seed.

The harvesting in 1935 both in the experimental and field plots was done between June 15th and July 10th. The flowers from the field crop, after drying, were placed in a large carton where they were kept until they were analyzed. Tables II, III, IV and V show the results of the collections of the three years.

TABLE IV—HARVEST OF 1935

Plot	Number of plants	Fresh weight g.	Air-dry weight g.	Loss per cent	Average weight of flowers g.	Average flowers per plant	Yield per plant g.
I	18	2,140	479	77.6	0.148	132	26.5
II	20	2,224	489	78.1	0.160	152	27.2
III	21	3,005	698	78.6	0.165	182	32.2
IV	19	1,940	450	76.8	0.163	145	25.0
V	20	1,575	360	77.2	0.147	122	19.0
Field crop, Urbana, Illinois	855	Not weighed fresh	12,400	----------	0.188*	----------	14.5

* Weight per flower determined by taking the mean of the weights of five lots picked at random. Each lot consisted of 100 flowers. The flowers consisted of about 75% fully opened and 25% partially opened flowers. The fully opened flowers averaged 20.8 g. per 100, or 0.208 g. per flower. The extra large flowers of this lot were as high as 0.270 g. per flower, and averaged 0.241 g. per flower. The partially opened averaged 13.1 g. per 100 flowers, or 0.131 g. per flower.

TABLE V—COMPARISON

Plot	Weight of flowers per plant			Average weight of flowers		
	1933 1st year g.	1934 2nd year g.	1935 3rd year g.	1933 1st year g.	1934 2nd year g.	1935 3rd year g.
I	0.32	12.1	26.5	0.119	0.147	0.148
II	0.30	12.1	27.2	0.115	0.151	0.160
III	0.29	14.1	30.2	0.122	0.153	0.165
IV	0.31	13.0	25.0	0.116	0.160	0.163
V	0.29	10.5	19.0	0.114	0.140	0.161
Field crop	0.35	----------	14.5	0.086 (closed)	----------	0.188

The results as summarized in Table V indicate:

1. The already known facts that the plants give almost no flowers during the first year, and begin to yield a good harvest the second year.
2. That the yield per plant is not materially increased by the application of fertilizer, but that there is a definite increase in the plants which received small amounts of uranium salts in addition to the fertilizer.
3. The increased yield is due to the more vigorous growth of the plants receiving stimulation. There was more evidence of luxuriant growth in the plants of Plot III, which received fertilizer and uranium salts.
4. The weight per flower is maximum in the field crop, which received very little care or cultivation. This, however, gave also fewer flowers per plant.

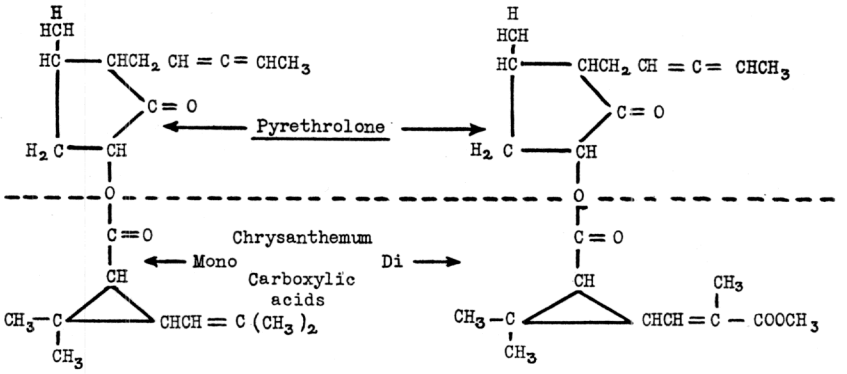
The poorest yield per plant occurred in Plot V which received manure. It must be noted, however, that this plot was at a lower level, and received more water during rains due to imperfect drainage. This is in accord with all previous observations that pyrethrum does not thrive in very humid soil.

The most exhaustive recent work on the effect of environmental conditions is the work of Tattersfield and his co-workers, published in 1931-1934.[3] Their results, so far as the weight of flowers per plant is concerned, point out that rich or fertilized soil does not particularly affect the yield of flowers. The same results also are indicated in their experimental growth of pyrethrum in Colorado by Gnadinger, Evans and Corl.[4] Rippert[5] on the other hand, claims that fertilizers increase the yield of flowers per acre in good years, while they maintain a satisfactory yield in poor years. High nitrogenous fertilizers are not recommended, either by Rippert or by Drain.[6]

It must be noted here that a part of our program was to try the effect of a number of organic plant catalysts, and a beginning was made with a few potted plants in 1935, but the plan was temporarily abandoned; recently we have resumed these experiments.

The Pyrethrin Content of the Flowers

At the outset of this work, the method of estimating the activity of the flowers was considered. The insecticidal activity of pyrethrum was proved by the work of Staudinger and Ruzicka[7] to be due to two compounds, called Pyrethrin I and Pyrethrin II. These are esters of a keto-alcohol-derivative of cyclopentane and two acids named chrysanthemum mono-carboxylic and dicarboxylic acids. Both are cyclopropane-carboxylic acids. Their structure is represented by the following formulae:



Pyrethrin I. Pyrethrin II.

[3] F. TATTERSFIELD; *Pyrethrum Flowers and Their Development*, Ann. Applied Biol. Vol. 18, pp. 602-35 (1931). F. TATTERSFIELD and J. P. MARTIN: The Effect of Environmental conditions upon pyrethrum, *ibid.*, Vol. 21, 670-90.

[4] GNADINGER, EVANS and CORL; Pyrethrum flowers, 2nd editions; C. B. Gnadinger: pp. 280-302.

[5] RIPPERT; J., Finn. fals. Vol. 25, 395-409 (1932).

[6] DRAIN, B. D.; Pyrethrum in Tennessee, Tenn. Agr. Exp. Sta.; Corl 59, 1-4 (1936).

[7] STAUDINGER, H., and RUZICKA, L.; Helv. Chim. Acta; Vol. 7, pp. 177-259 (1924).

Most of the methods of estimation of Pyrethrin I and II that have appeared in the literature may be classified as follows:

1. Methods based upon the semicarbazone derivatives of the carbonyl group of the pyrethrins. The methods of Staudinger and Harder[8] and Tattersfield, Hobson and Gimingham are based on this principle.
2. Methods based upon the hydrolysis of pyrethrins and separation of the carboxylic acids which are then estimated by titration. The methods of Staudinger and Harder, Tattersfield and Martin[9], Seil[10], and Rippert[11] are based upon this principle.
3. Methods based upon the reducing properties of the keto-alcohol pyrethrolone. The methods of Gnadinger and Corl[12], Martin and Tattersfield[13] are based upon this principle.

TABLE VI—DETERMINATION OF PYRETHRINS ON THE SAME SAMPLE OF PYRETHRUM BY VARIOUS METHODS

Sample No. 10 (a)	Seil Method			Gnadinger & Corl	Rippert			Wilcoxon	Moisture per cent (b)
	I	II	Total	Total	I	II	Total	I	
	0.631	0.540	1.221	0.885	0.595	0.614	1.209	0.620	9.51
	0.620	0.540	1.110	0.890	0.588	0.620	1.208	0.620	9.51
	0.578	0.599	1.170	0.901	------	------	------	------	9.51
	0.614	0.594	1.208	0.870	------	------	------	------	9.51
	0.625	0.580	1.205	0.860	------	------	------	------	9.51
	0.627	0.584	1.211	0.880	------	------	------	------	9.51
	0.621	0.591	1.212	0.889	------	------	------	------	9.51
Mean	0.616	0.515	1.191	0.882	0.591	0.617	1.208	0.620	------

(a) Harvest of 1935. Field crop from Urbana, Ill. Age of sample when analyzed 23 months.
(b) Values of Pyrethrins are on air-dried material.

4. Methods which are based upon a characteristic reaction of each of the pyrethrins which permit their independent determination. Haller and Acree[14] determine pyrethrin II from the amount of methyl iodide produced when treated with hydriodic acid. Wilcoxon[15] determines pyrethrin I by its reducing action on mercuric ion (Denige's reagent). The mercurous chloride produced is then determined by titration with standard iodate solution.

At the beginning of our work the Gnadinger-Corl Method alone was used. Later, the Seil method was tried on the samples in addition to the above-mentioned method. However, the results of the two seldom checked. Seil's method gave consistently 30-35 per cent higher total pyrethrins than the Gnadinger-Corl procedure. Consequently publication was withheld to permit a re-examination of all the available samples by both methods side by side.

In addition, the Rippert method and also the Wilcoxon, were tried on one sample which had been exhaustively checked by the two others. Table VI shows a comparison of the various methods.

[8] STAUDINGER, H. and HARDER, H.; Estimation of the Pyrethrin Content of Insect Powder, *Ann. Acad. Fennicae,* A29, No. 18, pp. 1-14 (1927).
[9] TATTERSFIELD and co-workers, *Journal Agr. Sci.* Vol. 19, 266-96 and 433-37 (1929); *ibid.,* Vol. 21, pp. 115-35 (1931).
[10] SEIL, H. A., *Soap* 10, No. 5, Vol. 89 (1934).
[11] RIPPERT, *J. Ann. fals.,* Vol. 29, pp. 344-54 (1936).
[12] GNADINGER, C. B., and CORL, C. S., *Jour. Am. Chem. Soc.* Vol. 51, pp. 3054-64 (1929); also Gnadinger, C. B., *Pyrethrum Flowers,* pp. 52-64 (1936).
[13] MARTIN and TATTERSFIELD, F. J., *Jour. Agr. Sci.,* Vol. 21, pp. 115-35 (1931).
[14] HALLER, H. L. and ACREE, F., *Indust. Eng. Chem.,* Ansl. Ed. I; 343 (1935).
[15] WILCOXON, F., *Contribution of Boyce Thompson Institute,* Vol. 8, No. 3, pp. 175-81 (1936).

The Gnadinger method is simple, but in order to obtain consistent results, it is necessary to observe all the precautions outlined in the method. The Seil method was slightly modified in that in the steam distillation to remove the monocarboxylic acid, 350 cc. of distillate was collected instead of 250 cc. in order to insure the removal of all the monocarboxylic acid. Therefore the solution containing the dicarboxylic acid had to be evaporated. This was done after rendering the solution alkaline with sodium bicarbonate.

Rippert's claim that on aging the pyrethrins are rendered insoluble and therefore have to be extracted by chloroform instead of petroleum ether, were not substantiated in this work. The total pyrethrins with either Seil's or Rippert's method (in the latter, chloroform was used) are substantially the same, (Table VI.) When, however, the chloroform extraction was applied to the Gnadinger-Corl method the total pyrethrins for sample No. 10 rose from 0.880 per cent to 1.35 per cent, which indicates that chloroform extracts other copper-reducing substances.

All extractions were carried through with the type of Soxhlet apparatus made by Rascher and Betzold of Chicago, in which corks are not used, but mercury seals. The petroleum ether was of reagent quality, b.p. range 20 — 40°C.

The data in Table VI show that there is a constant deviation between the copper reduction method and the Seil hydrolysis and subsequent separation of acids. The Gnadinger-Corl method assumes that the rate of reduction by pure pyrethrins at 78°C. is the same as by pyrethrins in presence of other substances which are extracted by petroleum ether, and not precipitated by barium ion.

The deviation between the Gnadinger-Corl and Seil methods becomes as high as 40-50 per cent when closed buds are examined. However, the results by both indicate that the pyrethrins are not fully formed in the closed flowers. The Seil method for Pyrethrin I is considered accurate, assuming, of course, that all the monocarboxylic acid comes over with steam. As shown in Table VI, the results of Pyrethrin I by Seil's method check well with the results obtained by Wilcoxon's method. Further, a sample of Japanese pyrethrum purchased from an importer[16], and according to its label containing 0.43 per cent pyrethrin I, was checked by both Seil's and Wilcoxon's methods, the results checking within 4 parts per hundred.

The Seil method for pyrethrin II is open to an error that tends to give high results. Seil directs that, after the residue from steam distillation is filtered, rendered slightly alkaline, extracted with chloroform, it should be acidified strongly with hydrochloric acid and extracted with ether (total volume 150 cc. in four portions). The amount of acid is not stated. The ether is washed with two 10-cc portions of water, and after removal of ether by distillation the flask is placed in the oven at 100°C. for 10 minutes. The above procedure may well leave some hydrochloric acid in the flask. Both hydrochloric acid and water are soluble in ether. It is assumed that the two washings in water and ten minutes heating when ether has been evaporated will remove all the hydrochloric acid, but it would be better to carry a blank. Ten cc of concentrated hydrochloric acid in 30 cc of water extracted with ether and then washed with two 10-cc portions of water left sufficient residual acidity in the separated ether to require 2.5 cc of 0.02 N sodium hydroxide which in results of the magnitude of our analyses would correspond to an error of 15 to 20 per cent. This of course does not indicate that errors of this magnitude creep in, but merely that the method is not entirely satisfactory.

[16] John Powell and Company, 114 E. 32nd St., N. Y. City, Japanese pyrethrum flowers, Lot 959.

The results of the determination of pyrethrins in the flowers from the harvest of 1933 are given in Table VII.

TABLE VII—DETERMINATION OF PYRETHRINS IN FLOWERS FROM CROP OF 1933

Flowers	Treatment	Age when analyzed (months)	Pyrethrins Seil's Method			Gnadinger-Corl Method	Moisture per cent
			I	II	Total		
Field crop, Urbana, closed flowers	None	9*	--------	--------	--------	0.62	8.55
	----------	40	0.409	0.569	0.978	0.58	8.55
	----------	40	0.393	0.535	0.928	0.55	8.55
Plots 1-5 mixed flowers fully open	4 plots fertilized a month before harvest	9*	--------	--------	--------	0.82	8.84

* Extracted with Petroleum Ether for five hours.
Values of Pyrethrin content are given on air-dried material.

The pyrethrin content of the closed flowers varies to the extent of about 40 per cent, according to the two methods. The flowers from the experimental plots were sufficient for one determination, and this was made before we tried the Seil method. The results, however, in agreement with the findings of a number of investigators, show the pyrethrin content of the flowers to increase as they mature.

Table VIII shows the pyrethrin content of flowers from the 1934 harvest.

TABLE VIII—DETERMINATION OF PYRETHRINS IN FLOWERS FROM CROP OF 1934

Flowers	Treatment	Age when analyzed (months)	Pyrethrins Seil's Method			Gnadinger-Corl Method	Moisture per cent
			I	II	Total		
Plot I----------	None	10 30	0.75	0.550	1.30	1.21 1.05	8.85
Plot II----------	Complete fertilizer	10	--------	--------	--------	1.01	8.50
Plot III----------	Complete fertilizer and uranium salt	10 30 30	-------- 0.781 0.730	-------- 0.580 0.560	-------- 1.371 1.290	1.22 1.05 ----------	8.85 ---------- ----------
Plot IV----------	Uranium salt	10	--------	--------	--------	1.20	9.08
Plot V----------	Manure	10	--------	--------	--------	0.96	8.30

Values of Pyrethrin content are given on air-dried material.

It is to be noted that there is no difference in the pyrethrin content of Plot I which received no treatment, and Plot III which received fertilizer and small amounts of uranium salts, resulting in a higher yield of flowers for this plot. The results are similar and more complete in the data for the harvest of 1935 given in Table IX.

TABLE IX—DETERMINATION OF PYRETHRINS IN FLOWERS FROM CROP OF 1935

Flowers	Treatment	Age when analyzed (months)	Pyrethrins Seil's Method			Gnadinger-Corl Method	Moisture per cent
			I	II	Total		
Field crop, Urbana, Ill.	None	(a) 12	0.605	0.585	1.190	0.890	9.51
		(b) 21	0.616	0.575	1.190	0.880	
Plot I		12	0.548	0.546	1.09	1.06	8.80
		13	0.540	0.530	1.07	1.10	
Plot II		12				0.96	8.65
		13				1.05	
Plot III		12	0.702	0.630	1.32	1.21	8.65
		13	0.685	0.610	1.29	1.15	
Plot IV		12	0.648	0.541	1.18	0.93	8.70
		13	0.655	0.490	1.14	0.98	
Plot V		12	0.522	0.490	1.01	0.91	8.85
		13	0.550	0.501	1.06	0.94	

(a) Average of 4 analyses.
(b) Average of 7 analyses.
Values of Pyrethrin content are given on air-dried material.

It is to be noted that there is a very constant deviation between the two methods of analysis, but either method gives results which indicate that the pyrethrin content is not appreciably changed, either by stimulation with fertilizers or photochemical catalysts, such as uranium. A summary of the analysis and yield per plant of all crops is given in Table X.

TABLE X—SUMMARY OF PYRETHRIN CONTENT FOR ALL SAMPLES CALCULATED ON MOISTURE FREE BASIS

Flowers	Average weight of flowers 2 year g.	Yield per plant 2 year g.	Pyrethrin content—moisture free basis					
			1933 Total pyrethrin		1934		1935	
			G-C	(a) S	G-C	(a) S	G-C (a)	S
			Per cent	Per cent	Per cent	Per cent	Per cent	Per cent
Field crop, Urbana, Illinois	0.188 (b)	14.5 (b)	0.638	1.250			0.978	1.31
Plot I	0.147	19.3	0.899 (c)		1.23	1.42	1.14	1.18
Plot II	0.155	19.65			1.10		1.10	
Plot III	0.159	22.15			1.24	1.45	1.29	1.43
Plot IV	0.161	19.25			1.32		1.04	1.27
Plot V	0.150	14.25			1.04		1.01	1.13

(a) G-C designates the results by the method of Gnadinger and Corl, and S the method of Seil.
(b) These results are on the basis of one year.
(c) This analysis represents all the harvest of the five plots mixed: The amount collected from each was too small to permit analysis.

According to these results stimulation with fertilizer and photochemical catalysts increases the yield per plant, but the pyrethrin content is not appreciably changed. The maximum pyrethrin content of the flowers from Plot III is not appreciably different from the flowers of Plot I which was not treated. This would indicate that the production of pyrethrins depends on genetic factors, which has been pointed out by Tattersfield. The content of pyrethrins is not diminished in the plants which by stimulation produced more flowers.

The effect of other plant stimulants is being further investigated.

SUMMARY

1. The growth of pyrethrum as a field crop in northern Illinois was investigated for three successive years. According to Dr. Alexopoulos under whose care the field crop at Urbana was grown, pyrethrum can be grown successfully in Illinois with no more care than is needed by the ordinary field crop.
2. The growth of pyrethrum in experimental plots at Chicago was investigated for three successive years.
3. Fertilizers do not seem to change appreciably either the yield per plant or the content of pyrethrin. Photochemical catalysts such as uranium salts increase the yield per plant, but not the pyrethrin content.
4. The various methods for the determination of pyrethrins were examined.

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