two cleanup procedures is compared in Table I along with the residue data for the Brussels sprouts.

The ability to detect the presence of such extremely small amounts of material provides the advantage of reducing the sample size while still allowing a sensitivity of 0.01 p.p.m. Lower sensitivity could be obtained by using larger samples, but at the expense of the life of the column and detector.

Representative gas chromatograms for picogram amounts of both nematocides are shown in Figures 1 and 4.

The cleanup and analysis procedures are adaptable to other studies involving these nematocides. Studies such as soil penetration, movement in irrigation water, effective dosages, and plant uptake should be possible.

The method sensitivity referred to under sample analysis is depicted for dibromochloropropane in Figure 3. The illustration shows that a linear relationship between detector response and sample size exists at least over the range shown.

Recovery and residue analysis for dibromochloropropane data are shown in Table II for both Brussels sprouts and walnut meats. In no case was a residue above the sensitivity of the method detected. Recovery data were considered to be excellent for the walnuts and Brussels sprouts. The background recorder response for check or treated samples was such that as little as 10 picograms of dibromochloropropane could have been detected.

The extraction, cleanup, and analysis procedures of these diverse crops gave indications of its broader applicability. The speed of analyses would make the analysis of these nematocides in soil, irrigation water, and uptake by plants possible areas for study.

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INSECTICIDE RESIDUES

Insecticide Residues in Peppermint and Their Distillation with Peppermint Oil

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Analyses of peppermint hay and peppermint oil after treatment of the crop with DDT, aldrin, dieldrin, or Dibrom indicate that each of these pesticides will persist through the processing of the hay. Both field and laboratory distillation experience indicate that the amount of residue found in peppermint oil depends in part on the severity of the distillation,—i.e., the amount of steam used. Up to 60 p.p.m. of dieldrin was found in peppermint oil recovered in a small still when excessive amounts of steam were used. Oil recovered with conventional distillation procedures contained less than 1 p.p.m. dieldrin. Peppermint grown in aldrin-treated soil contains more dieldrin than aldrin. Maximum oil residues using commercial stills were: DDT, 10.6 p.p.m.; dieldrin, 1.9 p.p.m.; and Dibrom, 36.4 p.p.m. Microcoulometric gas chromatography has been successfully applied to all samples with sensitivities as low as 0.01 p.p.m. attainable with fresh and spent hay. Special sample preparation methods are described.

During the period of active growth, oil accumulates in the peppermint plant, until at harvest time it constitutes about 0.5% of the fresh weight. At harvest the crop is cut, field cured for 2 to 4 days, chopped, and subjected to steam distillation to remove the oil. The hay residue remaining after this distillation process may be returned to the field and used as green manure or it may be fed to livestock.

The oily nature of this crop and the method of recovering the oil provide special circumstances under which the behavior of pesticide residues may differ from that found in other crops. It might be expected, for example, that residues of oil-soluble pesticides applied to this crop would persist and that the concentrating effect of steam distillation would lead to excessive residues in the final product. The purpose of the study reported here was to investigate this and other questions in the case of aldrin applied to the soil before the peppermint growth begins, and dieldrin, DDT, and *O,O*-dimethyl 1,2-dibromo-2,2 dichloroethyl phosphate (Dibrom, registered trademark of the California Chemical Corp.) applied to peppermint foliage during the growing season.

Residue investigations on peppermint oil have been conducted by Gould (*3*), who reported dieldrin residues up to 4.3 p.p.m., DDT to 9.8 p.p.m., heptachlor to 3 p.p.m., and aldrin to 2.4 p.p.m. No foliage or spent hay analyses were included.

Experimental

Aldrin Soil Treatment. Single 5-acre plots, located in western Oregon, were treated with aldrin emulsible at 2.5, 5, and 10 pounds active per acre in April 1960. At this time, peppermint is in a near dormant stage with growth of not more than 1 or 2 inches. The aldrin was applied with a boom-type weed sprayer and the ground disked in two directions immediately after the application. The plots were harvested 142 days later at which time fresh hay samples were collected for aldrin and

dieldrin analysis. After field curing for 2 days, the hay was chopped, the oil recovered by distillation in a commercial still, and spent hay samples were taken.

The above experiment was repeated in 1961 using a single application of 2.5 pounds of aldrin active per acre. Fresh hay, spent hay, and peppermint oil samples were collected.

Dieldrin Applied to Peppermint Foliage. In July 1961, a 1.4-acre plot of peppermint in Madras, Ore., was treated with emulsible dieldrin at 0.5 pound active per acre using a conventional weed sprayer. Hay samples were collected immediately and at 7, 28, and 52 days later (harvest). The harvested crop was steam distilled in a normal manner using commercial distillation apparatus, and spent hay and peppermint oil samples were collected.

Another group of dieldrin-treated peppermint samples were obtained from Michigan State University, East Lansing, Mich. These samples (fresh hay, spent hay, and oil) came from plots which were treated on August 21, 1961, with 0.5 pound dieldrin active per acre and sampled at 0, 7, 14, and 21 days after treatment. At each sampling time, the entire 22- × 40-foot plot was harvested and its oil recovered by steam distillation using a noncommercial, pilot-size still. In this case, therefore, oil and spent hay samples were obtained from plots freshly treated as well as from plots in which 7-, 14-, and 21-day intervals had elapsed.

DDT Applied to Peppermint Foliage. In 1960, an Oregon peppermint field was divided into four large plots which received the following DDT treatments. Plot 1 received one application of a DDT wettable on July 15 at 1.5 pounds active per acre using conventional spray equipment. Samples were collected at 0, 13, 24, and 47 days later (harvest). The same DDT treatment was given plot 2 on July 15 and on August 18, with samples collected at 0, 10, 15, and 23 days after the second application. Plot 3 received 1.5 pounds DDT (active) emulsible per acre on July 14, with the application being made through the irrigation sprinkler system. Samples were collected at 0, 14, 28, and 31 days. Plot 4 received the same treatment on July 14 and on August 8, with samples collected at 0, 7, 15, and 23 days after the second application. Peppermint oil and spent hay samples were collected from each of the plots at harvest using a commercial still for the distillation.

Dibrom Applied to Peppermint Foliage. On July 7, 1961, a 1.4-acre plot of peppermint in Madras, Ore., was treated with 1 pound Dibrom (active) emulsible per acre using a conventional weed sprayer. Samples were collected immediately, and at 7, 14, and 39 days. On August 15, 39 days after the first treatment, the plot was again treated with 1 pound Dibrom (active) emulsible, and samples were collected immediately, and at 3 and 13 days (harvest). The harvested crop was distilled in a normal manner using commercial distillation apparatus. Spent hay and peppermint oil samples were collected.

Analytical Method. DDT. A 400-gram sample of the fresh, chopped hay was extracted by tumbling for 1 hour in a 2:1 mixture of *n*-hexane (Skellysolve B) and isopropyl alcohol using 3 ml. of solvent per gram of crop tissue. Spent hay samples were allowed to stand 24 hours in the solvent before tumbling so as to allow good mixing of the solvent with the dry tissue. After extraction, the isopropyl alcohol was removed from the extracts by washing with water, and the *n*-hexane phase remaining was stored over anhydrous sodium sulfate. Aliquots of the extracts were evaporated to dryness on a steam bath and the residues taken up in carbon tetrachloride. This solution was then washed with sulfuric acid until the acid layers were no longer dark, following the procedure described by Schechter *et al.* (*6*). After sulfonation, the DDT method of Downing and Norton (*2*) was followed without modification. Untreated peppermint hay samples subjected to the analytical procedures described resulted in negligible blank values.

Preliminary cleanup of the peppermint oil was accomplished by adding 20- to 48-mesh Columbia carbon at the rate of 1 gram per ml. of oil (oil samples not more than 10 ml.) and 100 ml. of benzene. This mixture was shaken for 10 minutes and the benzene removed by filtration. The charcoal remaining was washed two additional times with benzene, the three benzene fractions were combined, and the solvent was removed on the steam bath. The residual oil was taken up in carbon tetrachloride and subjected to the sulfonation and colorimetric procedures described above.

DIELDRIN. A 500-gram sample of fresh, chopped hay was tumbled for 1 hour at 30 r.p.m. in hexane–isopropyl alcohol (2:1) at 3 ml. per gram of hay. The extract was removed by filtration, extracted three times with water to remove the isopropyl alcohol, and dried with anhydrous sodium sulfate.

An aliquot equivalent to 50 grams of peppermint hay was concentrated to 10 ml. on the steam bath, and passed through an *n*-hexane-prewashed column containing 24 grams of magnesia-Celite (4:1) topped with 3 cm. of anhydrous sodium sulfate. The first 200 ml. of *n*-hexane eluate were discarded and the next 500 ml. collected. The sample was concentrated in a Danish-Kuderna evaporator to a small volume. A suitable

Table I. DDT Residues in Peppermint after Foliar Application

DDT Treatment, Pounds Active/Acre	Interval, Application to Harvest, Days	DDT, P.P.M.[a] Fresh hay	Spent hay	Oil
Untreated		0.16	0.15	<1.0
One spray application, 50% wettable, 1.5 lb.				
	0	66		
	13	27		
	24	8		
	47	5.6	2.1	2.6
Two spray applications, 50% wettable, 1.5 lb.				
	0	91		
	10	22		
	15	5		
	23	6.7	3.3	10.6
One sprinkler application, 25% emulsible concentrate, 1.5 lb.				
	0	42		
	14	9		
	28	5		
	31	4.2	2.3	2.9
Two sprinkler applications, 25% emulsible concentrate, 1.5 lb.				
	0	23		
	7	15		
	15	12		
	23	11.5	4.1	3.5

[a] Spectrophotometric method; analytical values corrected for crop blank, represent one to four analyses of each sample. Method sensitivities, 0.05 p.p.m. for hay and 1.0 p.p.m. for oil. Recoveries averaged 89% for fresh hay, 87% for spent hay, and 82% for oil.

aliquot was injected into the microcoulometric gas chromatograph (MCGC). The column was 20% silicon on Chromosorb, and the instrument settings were: block temperature, 270° C.; column temperature, 250° C.; carrier gas rate, 180 ml. per minute; and sensitivity setting, 64 to 128 ohms.

A 10-gram sample of peppermint oil was analyzed for dieldrin by concentration from a solution with 50 ml. of *n*-hexane. The oil-hexane mixture was placed on a steam bath and evaporated under a current of air. When the hexane and about one fourth of the oil had evaporated (about 30 minutes), an additional 50 ml. of *n*-hexane were added, and the evaporation was continued. This process was repeated until about 1 ml. of resinous material remained.

The residue in the beaker was dissolved in 25 ml. of ethanol and transferred to a separatory funnel using an additional 25 ml. of ethanol. Fifty milliliters of water were added, and the mixture was extracted three times with 50-ml. portions of *n*-hexane. The combined hexane extracts were backwashed three times with 50-ml. portions of water and dried with sodium sulfate. At this point, the sample was suitable for analysis in the microcoulometric gas chromatograph apparatus, but improved sensitivity could be attained by passage through the magnesia-Celite column described earlier. A few samples were given this additional cleanup.

A few of the peppermint hay and oil samples containing dieldrin were analyzed by means of the phenyl azide method of O'Donnell *et al.* (*4*). The cleanups used were those described in the above paragraphs including the more thorough processing of the oil samples. Recoveries of added dieldrin were 72% for peppermint hay and 56% for peppermint oil. This method was not satisfactory for samples containing aldrin.

ALDRIN. The aldrin samples were extracted and prepared for cleanup in the same manner as that described for dieldrin. Cleanup was performed by passing the concentrated aliquot through a hexane-prewashed charcoal–Attasol–Dicalite column. The ingredients of the column were, from the bottom, respectively: 1 cm. of anhydrous sodium sulfate, 5 cm. of Columbia activated carbon 20-48- to mesh, 20 cm. of Attasol-Dicalite (3:2), and 2 cm. of anhydrous sodium sulfate. The first 250 ml. of hexane eluate were collected for analysis. From this point on, the procedure followed that of dieldrin including microcoulometric gas chromatograph settings.

The method used for aldrin in peppermint oil was identical with that described for dieldrin except that a final cleanup through a column was not performed.

DIBROM. To a 1000-gram sample of fresh, chopped hay, 40 ml. of concentrated HCl were added, and the sample was tumbled for one-half hour in hexane at 2 ml. per gram of hay. The extract was then removed by decantation and filtered through anhydrous sodium sulfate. An aliquot equivalent to 50 grams of peppermint hay was concentrated to 10 ml. at 30° C. in a rotary evaporator. Oil samples (10 ml.) were diluted with 40 ml. in hexane.

The hay or oil samples were passed through a hexane-washed silicic acid column prepared by the method of Pack and Ospenson (*5*). Two hundred milliliters of hexane-redistilled ether (3 + 1) were passed through the column and discarded, followed by 400 ml. of hexane-redistilled ether (1 + 3). This removed the Dibrom and its principle metabolic product, *O,O*-dimethyl 2,2-dichlorovinyl phosphate (DDVP), from the column. From this point on, the method of Pack and Ospenson was followed. This method depends upon the conversion of Dibrom to DDVP and reflects the total of these two compounds present in the sample.

Results and Discussion

After an early loss of the major portion of the DDT present on peppermint foliage, the remainder appears to persist for several weeks (Table I). Solution of DDT in the oil glands of the leaves may explain the prolonged residual period. As indicated in Tables III and IV, this same persistence pattern is found with dieldrin and Dibrom.

Another example of the well-known conversion of aldrin to dieldrin is seen in Table II. It is evident that most of the aldrin taken up by the plant has been oxidized to dieldrin. Since these treatments were made early in the season before active growth, the residues present in the harvested oil probably result from systemic action although it is possible that soil contamination at harvest could have contributed some of the observed residues. Of interest is the fact that dieldrin residues in the mint oil (Table II) were greater as a result of this early season application of aldrin to the soil than from the mid-season foliage treatment with dieldrin itself (Table III).

A comparison of the Oregon and Michigan experiments with dieldrin (Table III) suggests that growth was not the major cause of the rapid early decline of dieldrin residues. This is indicated by the fact that although the two residue-decline patterns are similar, the Oregon treatments were made in mid-season

Table II. Aldrin and Dieldrin Residues in Oregon Peppermint after Soil Application of Aldrin

Aldrin[a] Treatment, Lb. Active Per Acre	Aldrin, P.P.M.[b] Fresh hay	Aldrin, P.P.M.[b] Spent hay	Aldrin, P.P.M.[b] Oil	Dieldrin, P.P.M.[b] Fresh hay	Dieldrin, P.P.M.[b] Spent hay	Dieldrin, P.P.M.[b] Oil
			1960			
Untreated	<0.01	0.01	<0.5	<0.01	0.03	<0.5
2.5 Lb./acre	0.01	<0.01	<0.5	0.18	<0.01	0.8
5 Lb./acre	<0.01	<0.01	<0.5	0.05	0.03	1.0
10 Lb./acre	0.03	0.01	0.9	0.15	0.06	1.9
			1961			
2.5 Lb./acre	<0.01	<0.01	0.3	0.04	0.10	0.7

[a] Interval application to harvest 1960, 142 days; 1961, 105 days.
[b] Gas chromatographic method; analytical values corrected for crop blank, represent one to five analyses of each sample. Method sensitivities, 0.01 p.p.m. for hay, 0.5 p.p.m. for oil. Aldrin recoveries averaged 93% for fresh hay, 72% for spent hay, and 86% for oil. Dieldrin recoveries averaged 87% for fresh hay, 88% for spent hay, and 74% for oil.

Table III. Dieldrin Residues in Oregon and Michigan Peppermint after Foliar Application of Dieldrin

Dieldrin Treatment, Lb. Active Per Acre	Interval, Application to Horvest, Doys	Dieldrin, P.P.M.[a] Fresh hay	Dieldrin, P.P.M.[a] Spent hay	Dieldrin, P.P.M.[a] Oil
	Oregon, 1961			
Untreated	..	0.02	0.06	<0.5
0.5 Lb., emulsible concentrate				
	0	13.30		
	7	0.82		
	28	0.25		
	52	0.01	0.08	<0.5
	Michigan, 1961			
Untreated	..	0.01	0.04	0.9
0.5 Lb., emulsible concentrate				
	0	3.48	1.03	61.5
	7	...	...	56.7
	14	0.47	...	52.4
	21	0.34	0.19	36.8

[a] Gas chromatographic method; analytical values corrected for crop blank, represent one to three analyses of each sample. Method sensitivities, 0.01 p.p.m. for hay, 0.5 p.p.m. for oil. Recoveries averaged 87% for hay, 74% for oil.

Table IV. Dibrom Residues in Peppermint after Foliar Application

Dibrom Treatment, Lb. Active Per Acre	Interval, Application to Harvest, Days	Dibrom, P.P.M.[a, b] Fresh hay	Spent hay	Oil
Untreated		<0.2	<0.2	<1.0
One application, 1 lb. emulsible concentrate				
	0	2.88		
	7	0.47		
	14	<0.2		
Two applications, 1 lb. emulsible concentrate				
	0	8.23		
	3	4.10		
	13	3.02	<0.2	36.4

[a] Calculated as Dibrom; represents both Dibrom and DDVP. [b] Gas chromatographic method; analytical values corrected for crop blank, represent two or more samples. Method sensitivities, 0.2 p.p.m. for hay, 1.0 p.p.m. for oil. Recoveries averaged 71%.

Table V. Steam Distillation in the Laboratory

Insecticide	Mol. Wt.	Vapor Pressure, Mm. Hg at 20° C.	Fortification Level, P.P.M.	Recovery, % 200-Ml. distillate	1000-Ml. distillate
Dibrom	381	$<1 \times 10^{-4}$ [a]	6	31	
			3	34	
DDT	352	1.9×10^{-7} (7)	15	3.4	
			20	1.6	6.3
			10	3.0	11.1
Aldrin	362	0.6×10^{-5} (7)	5	31	56
			2	22	51
Dieldrin	388	1.8×10^{-7} (7)	5	17	34
			3	14	27

[a] Estimated.

Table VI. Comparison of Peppermint Hay to Peppermint Oil Residues

Treatment	Residue, P.P.M. Fresh hay	Oil	Residue in Oil, % of Theoretical[a]
Dieldrin, foliar	3.48	61.5	8.8
	0.47	52.4	55.7
	0.34	36.8	54.0
Dieldrin, soil[c]	0.04	0.18	2.2
Aldrin, soil	0.15[d]	1.9	6.3
	0.05[d]	1.0	10.0
Dibrom, foliar	3.02	36.4	6.0
DDT, foliar	6.7	10.6	0.8
	11.5	3.5	0.2

[a] Assuming complete distillation will result in an oil residue 200 times that of the hay residue. [b] Pilot still. [c] Indiana sample. [d] Dieldrin Residue.

during active growth while the Michigan tests were conducted when the plants were near maturity.

The abnormal behavior of Dibrom residues (Table IV) is further evidence that the oil content of peppermint contributes to the slowdown in residue loss. This insecticide, generally considered short-lived as a residue, is seen to persist at least 2 weeks when applied to mature peppermint. Although the residues are indicated as Dibrom, it is likely that part of those present on the plant and most of those found in the oil are actually DDVP, the principal conversion product of Dibrom (*4*). Laboratory tests of the steam volatility of Dibrom have indicated that this conversion is nearly complete during steam distillation.

The tendency of peppermint-borne pesticides to steam distill largely determines the residue hazard encountered with this crop. Since this plant contains approximately 0.5% oil when mature, oil residues 200-fold those found on the plant could occur as a result of distillation. DDT residues in mint oil could thus have been as great as 2300 p.p.m. if complete transfer during distillation occurred.

In most of the cases tested during these experiments, only a small percentage of the pesticide present on the hay actually transferred to the oil. However, in one series of samples, those obtained from the Michigan experiments (Table III), up to 55% of the dieldrin present on the mint hay was found in the oil at completion of distillation. The explanation for these results seems to lie in the type of distillation equipment or the manner of its use. An examination of the distillation data and a comparison of the equipment showed that in the small-scale still used in the Michigan tests the peppermint was subjected to approximately five times more steam than with the commercial still used in the Oregon experiments. According to the theory of steam distillation of water-insoluble materials, the amount of such material volatilizing with steam is proportional to its vapor pressure, its molecular weight, and the weight of steam used. The increased quantities of steam used in the smaller still could thus have contributed greatly to the increased residues found.

Tests of these ideas were made under laboratory conditions using a 12-liter, all-glass still. Chopped peppermint hay was fortified with acetone solutions of aldrin, dieldrin, DDT, or Dibrom and distilled with live steam. Gas chromatographic analysis of the oil phase allowed a comparison to be made between distillate volume and amount of pesticide distilling. As shown in Table V, where these results are summarized, a fivefold increase in distillate volume resulted in as much as a fourfold increase in pesticide distilling. Although the expected relationship between volatility and steam distillability is supported by the data, there appear to be other factors involved. For example, the vapor pressures and molecular weights of DDT and dieldrin have nearly the same values, but dieldrin displays a much greater tendency for steam distillation.

A review of typical mint hay and mint oil pesticide levels (Table VI) leads to the conclusion that the amount of pesticide distilling with the oil is only slightly related to that present on the hay. In the Michigan tests, for example, a 90% decrease in fresh hay dieldrin levels resulted in only a 50% decrease in dieldrin residues in the processed oil. Similar examples of this lack of correlation are found in the laboratory tests.

In some peppermint-growing regions, it is customary to feed the spent or oil-free hay to livestock. The pesticide residue content of this waste product thus becomes of interest. According to the results obtained in this study, both DDT and dieldrin may be present on spent hay, probably in amounts sufficient to lead to deposition of such residues in the fat of animals consuming it.

In comparing the pesticide content of undistilled peppermint with that of the recovered oil and the spent hay, one often finds that the majority of the residue is not accounted for. Although the aqueous portion of the distillate was not analyzed in these experiments, it is not likely that this fraction could accomodate the missing pesticide. Water solubilities of 10 p.p.m. or greater would be necessary if all of the missing residues were to be present here. Two other possibilities can be offered: destruction of the

pesticide during distillation, perhaps catalyzed by the metal surfaces of the still; and deposition of vaporized residues in the condenser coils. Rinses of the water-cooled condensers used in the laboratory experiments show that such deposition can occur although not in sufficient quantity to account for all of the missing residues.

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SAFETY EVALUATION OF CHEMICALS

Relationship between Short- and Long-Term Feeding Studies in Designing an Effective Toxicity Test

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DURING more than 15 years, in the two laboratories involved, chemicals have been tested by mixing them in the diet and feeding these diets to rats. The usual procedure is first to determine an LD_{50}, or the amount of chemical expected to kill half of a group of small animals, usually rats, after a single dose. Second, if the chemical is a potential food ingredient or perhaps will become a residue on food crops, these acute oral toxicity data are used to plan dosage levels, and the material is then included in the diet of rats for a short-term experiment of 30- to 90-day duration. If the no-ill-effect dosage level of the material fed to the test rats is such as to sustain economic interest, a life-span or 2-year test of the chemical in the diet of rats will be started. Additional long-term study will also be undertaken using a nonrodent species, probably the dog.

A no ill-effect level determined as a result of these dietary feeding studies in laboratory animals is defined variously as one which shows no measurable effect, no ill effect, or no evidence of adverse effect attributable to the test material when judged by any of the toxicological or biochemical criteria employed. The term maximum no-effect level, used below, thus refers to the highest dietary concentration having no ill effect. Conversely, the minimum effect level is defined as the lowest dietary concentration at which any significant ill effect attributable to the test material was produced.

Table I. Relationship of Dosage Levels of Short-Term and 2-Year Feeding of Materials in the Diet of Rats

Material Number	Duration of Short-Term Test	Percentage of Material in Diet: Short-term, Minimum effect	Short-term, Maximum no-effect	2-Years, Minimum effect	2-Years, Maximum no-effect	Ratio: Short-Term/2-Years, Minimum effect	Ratio: Short-Term/2-Years, Maximum no-effect
1	105	0.015	0.005	0.03	0.01	0.5	0.5
2	90	4.0	2.0	8.0	4.0	0.5	0.5
3	90	1.0	0.3	2.0	0.2	0.5	1.5
4	120	3.0	1.0	5.0	0.5	0.6	2.0
5	90	0.25	0.0625	0.256	0.064	1.0	1.0
6	90	0.01	0.003	0.01	0.003	1.0	1.0
7	97	0.1	0.03	0.1	0.03	1.0	1.0
8	90	8.0	4.0	8.0	4.0	1.0	1.0
9	130	1.0	0.3	1.0	0.2	1.0	1.5
10	30	0.05	0.012	0.04	0.01	1.2	1.2
11	30	25.0	10.0	20.0	5.0	1.2	2.0
12	90	0.75	0.375	0.40	0.13	1.9	2.9
13	90	10.0	3.0	5.0	1.0	2.0	3.0
14	90	0.03	0.01	0.0125	0.0062	2.4	1.6
15	130	3.0	1.0	1.0	0.2	3.0	5.0
16	50	0.3	0.1	0.1	0.03	3.0	3.3
17	98	0.01	0.003	0.003	0.001	3.3	3.0
18	90	16.0	8.0	4.0	2.0	4.0	4.0
19	29	0.25	0.06	0.06	0.02	4.2	3.0
20	210	0.25	0.05	0.05	0.01	5.0	5.0
21	90	0.225	0.15	0.04	0.02	5.6	7.5
22	130	0.1	0.03	0.005	0.0025	20.0	12.0
23	90	0.5	0.25	M[a]	0.5	...	0.5[b]
24	90	0.009	0.003	M[a]	0.004	...	0.8[b]
25	30	0.3	0.1	M[a]	0.1	...	1.0[b]
26	90	8.0	4.0	M[a]	2.0	...	2.0[b]
27	90	16.0	8.0	M[a]	4.0	...	2.0[b]
28	90	M[a]	3.0	3.0	1.0	...	3.0[c]
29	93	M[a]	5.0	5.0	1.0	...	5.0[c]
30	91	M[a]	0.18	0.06	0.02	...	9.0[c]
31	90	M[a]	1.0	M[a]	0.3	...	3.3[d]
32	90	M[a]	2.5	M[a]	0.5	...	5.0[d]
33	142	M[a]	25.0	M[a]	5.0	...	5.0[d]

[a] M = the maximum no-effect level was the highest dosage level fed.
[b] As the M level was on the 2-year test, the ratios are a maximum.
[c] As the M level was on the short-term test, the ratios are a minimum.
[d] As the M levels were on both the short-term and 2-year tests, the ratios are indicative only of which levels were used.