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VARIATION OF CHEMICAL CONCENTRATION IN SURFACE-COATED AND TABLETED GRAIN BAITS

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VARIATION OF CHEMICAL CONCENTRATION IN SURFACE-COATED AND TABLETED GRAIN BAITS

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Abstract: Liquid scintillation spectrometry was used to determine the quantity of recovered carbon[14]-labelled Mesurol (4-[methylthio]-3,5-xylyl *N*-methylcarbamate) from individual baits. The six grain baits tested were cracked corn, wheat kernels, and oat groats, each formulated as surface-coated baits and as tablets (ground grain mixed with the chemical and tableted). The concentration of Mesurol on the surface-coated baits was highly variable, with coefficients of variation (CV) ranging from 21.0 percent (wheat) to 48.1 percent (cracked corn); Mesurol in individual cracked corn baits ranged from 99–532 μg. Such variation could easily explain erratic results in field baiting programs or laboratory tests. The tableted baits were more uniform (CV, 4.6–8.0 percent; greatest range, 274–360 μg) and are recommended for baiting situations where chemical concentration is critical.

Surface-coated baits often produce erratic and unsatisfactory results in field experiments and pest control programs. Many physiological, nutritional, and ecological factors contribute to this variability, but formulation is also potentially important. In baiting programs for wildlife, anomalous results can often be attributed mainly to inadequate or variable chemical concentration on the baits. For example, unpublished progress reports from this laboratory reveal such problems in baiting programs to control *Peromyscus* and to frighten blackbirds (Kverno et al. 1967, Guarino et al. 1968, unpublished reports, Denver Wildlife Research Center).

Lord et al. (1967) and Jeffs et al. (1968) investigated the uniformity of insecticidal seed dressings on individual seeds, and with minor exceptions, found the treatments to be low in chemical concentration and quite variable, especially in commercial preparations. The present experiment was conducted to determine the extent of such variation in grain baits for animal damage control by comparing the chemical concentration in three kinds of surface-coated baits, all prepared in the same manner, with that in bait tablets made from the same materials.

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MATERIALS AND METHODS

Mesurol (also called Bay 37344[1]) was chosen as the test chemical because of our

[1] Trade name of Chemagro Corp., Kansas City, Missouri. Reference to trade names does not imply endorsement of commercial products by the Federal Government.

current interest in it as an immobilizing agent for bird control (Schafer et al. 1967). The quantity in baits was ascertained by liquid scintillation spectrometry. Three commonly used baiting materials: cracked corn, whole wheat kernels, and whole oat groats, were chosen for testing as surface-coated treatments and as tablets.

Carbon[14]-labelled Mesurol[2] (specific activity, 1.95 mc/mm) was diluted to 0.2 percent with technical grade Mesurol, and this mixture ($M\text{-}C^{14}$) was used for all formulations. All baits were prepared to contain 1 percent $M\text{-}C^{14}$ by weight.

Surface-coated baits were prepared by mixing the grain with an acetone solution of the $M\text{-}C^{14}$ (1 ml of solution per 10 g of grain) in a pint jar. Frequent shaking and a stream of air were used to evaporate the acetone and coat the particles. Cracked corn was screened to 5-mesh size before formulation. Cracked corn particles weighed about 20 mg, wheat kernels about 45 mg, and oat groats about 24 mg. Tableted baits were prepared from treated granules. Granules were formed by mixing the ground grain with a binder composed of a 1 : 1 Plasdone[3]-Sorbo[4] mixture (31.5 percent by weight), forcing the wet mixture through a 9-mesh screen, and spreading this in a thin layer for drying at 40 C in a forced air oven. Screened 20-mesh granules were mixed with a diethyl ether solution of the $M\text{-}C^{14}$, and then the ether was evaporated. This formulation was tableted with a Stokes Model 518-1[5] tableting press (a punch-and-die system) to produce ⅛-inch diameter tablets of 32 mg weight. A Jones type sample splitter (a mechanical device for randomly partitioning large quantities of material into samples of usable size) was then used to select 27 baits from each of the six treatments (one surface-coated and one tableted for each bait material) for analysis.

For extraction, each bait was wrapped in a packet of aluminum foil lined with three layers of laboratory tissue, thoroughly crushed with pliers, and placed in a micro-soxhlet extraction thimble, where the packet was loosened so that acetone could penetrate. The sample was then extracted for 2 hours. The extract was concentrated to approximately 1 ml, and was transferred, along with three rinsings, to glass scintillation vials. The acetone was evaporated, and 15 ml of scintillator (4 g of 2,5-diphenyloxazole and 50 mg of *p*-bis[2-(5-phenyloxazolyl)]-benzene per liter in toluene) was mixed with the contents of the vial. Each sample was counted for 5 minutes on a Nuclear-Chicago Model 181B liquid scintillation counter; a 30-minute background count was taken daily.

To determine the relationship between sample activity and amounts of Mesurol present, regression equations were calculated for the three bait materials. For each material, three replicate 40-mg samples of 60-mesh ground grain were spiked with 50, 100, 200, 400, 600, and 800 μg of $M\text{-}C^{14}$ and the recovery was determined. In all three cases, the best-fitting regression curve was linear. Analysis of covariance showed that there was no difference between the lines in slope or intercept (indicating no differences in recovery due to the bait material), and that a single standard regression equation was appropriate for determining amounts of Mesurol in all three

[2] Supplied by New England Nuclear Corp., Boston, Massachusetts.

[3] Trade name of General Aniline and Film Corp., Dyestuff and Chemical Division, New York, New York.

[4] Trade name of Atlas Chemical Industries, Wilmington, Delaware.

[5] Pennsalt Chemicals Corp., Stokes Pharmaceutical Equipment Department, Warminster, Pennsylvania.

Table 1. Recovery when known amounts of M-C^{14} were added to grain baits or ground grain.

Material	Number of Baits or Samples Treated	M-C^{14} Added per Bait or Sample (μg)	M-C^{14} Mean ± S.D. (μg)	M-C^{14} Coef. of variation (percent)
Cracked corn	3	200	192.29 ± 5.50	2.9
	3	600	591.51 ± 7.89	1.3
	9	400	390.66 ± 8.43	2.2
Wheat kernels	9	400	404.65 ± 11.08	2.7
Oat groats	9	400	395.55 ± 12.51	3.2
Ground grain[a]	9	200	199.54 ± 3.86	1.9
	9	400	393.94 ± 11.82	3.0
	9	600	573.04 ± 11.96	2.1

[a] Mean of three replicate 40-mg samples for each type of grain used in establishing standard regression equation.

materials: $Y = 16.58 + 0.036\ X$, where $Y = \mu g$ M-C^{14} and X = counts per minute.

As a further check of the method (especially the maceration technique), whole bait materials were surface-coated with known amounts of M-C^{14} and recovery was determined. The results are summarized in Table 1. In all cases, the CV in the amount recovered was small, and recovery was essentially the same as that obtained from ground bait materials (mean recoveries from ground grain spiked at 200, 400, and 600 μg are included in Table 1 for comparison). Recoveries from ground grain represented those expected from tablets. Internal standardization of samples showed that quench was not affected by the presence of Plasdone-Sorbo binder or differences in sample weight.

RESULTS AND DISCUSSION

The concentrations of Mesurol found in the six treatments are given in Table 2. The three surface-coated treatments were the most variable. Wheat was the most uniform of the three, but still had a range of 255–598 μg Mesurol and a CV of 21.0 percent. Cracked corn was the most variable, with a range of 99–532 μg and a CV of 48.1 percent. The greater variation in the cracked corn was almost certainly due to the nonuniformity of the bait particles. Although they were all screened to 5-mesh

Table 2. Mesurol concentration in six bait formulations prepared to contain 1 percent M-C^{14} by weight; each mean represents 27 individual baits.

Treatment	Quantity of M-C^{14} Mean ± S.D. (μg)	Quantity of M-C^{14} Range (μg)	Quantity of M-C^{14} Coef. of variation (percent)	Proportion of M-C^{14} Mean ± S.D. (percent by weight)[a]	Proportion of M-C^{14} Range (percent by weight)	Proportion of M-C^{14} Coef. of variation (percent)
Surface-coated cracked corn	230.47 ± 110.84	99.34–532.39	48.1	1.23 ± 0.56	0.64–2.73	45.3
Surface-coated wheat	430.73 ± 90.52	254.94–597.91	21.0	0.97 ± 0.17	0.72–1.40	17.7
Surface-coated oat groats	238.84 ± 60.13	141.25–373.09	25.2	1.05 ± 0.37	0.60–2.42	35.2
Corn tablets	326.39 ± 20.89	279.52–356.92	6.4	0.98 ± 0.06	0.83–1.11	6.5
Wheat tablets	328.15 ± 26.29	274.41–360.85	8.0	1.03 ± 0.07	0.91–1.20	6.5
Oat groat tablets	335.87 ± 15.40	301.08–359.66	4.6	1.02 ± 0.05	0.91–1.10	4.6

[a] Mean of percent by weight calculated for each individual bait.

size, the irregular shapes and different ratios of endosperm to pericarp in the fractured pieces of corn seed resulted in variations in weight, in total surface area, and in surface area of exposed endosperm (which sorbs dissolved chemicals more readily than the pericarp). The surface-coated oat groats were also somewhat affected by morphological variation. There appeared to be little relationship between seed weight and chemical concentration for this treatment, and the CV for percent Mesurol by weight was actually higher than that for micrograms of Mesurol.

Judging from these data, laboratory formulation of surface-coated seeds can be expected to yield at least a 20 percent CV in chemical concentration, and this value becomes larger with apparent increasing morphological variation in the bait material. In field baiting programs, which normally involve "batch" formulation in large mixers, the variation is probably increased considerably. Such a large range in the amount of active ingredient in individual baits could easily explain erratic results in some baiting programs, particularly where the desired physiological effect depends on the consumption of a relatively few treated bait particles or, even more important, where the effect is lost by ingestion of a slight excess in active ingredient.

Mesurol, which is effective as an avian immobilizing agent at low levels but which has a relatively narrow safety margin for most species, is an example. Schafer et al. (1967:288) gave the median effective dose of Mesurol for starlings (*Sturnus vulgaris*) as 2 mg/kg and the LD_{50} as 12 mg/kg. In a baiting program, if surface-coated cracked corn formulated to contain one median effective dose per bait particle showed the same variation as in these tests, one would expect 32 percent of the baits to vary in chemical concentration by more than one standard deviation (47 percent of the mean). This would mean that about one-third of the baits would contain either less than half an effective dose or more than one and a half effective doses, and that a starling could eat a low-level bait and be unaffected or could theoretically eat three or four unusually high-level baits in quick succession and receive a lethal dose. Since this last possibility is unlikely, formulations with a 47 percent CV would probably be adequately safe. However, field-mixed baits could conceivably show enough additional variation to occasionally contain a lethal dose in two baits, or even one. This possibility may account for at least some of the mortality seen in field baiting programs with Mesurol (Woronecki et al. 1967, 1970).

In contrast to these results with surface-coated treatments, the three tableted baits analyzed were all quite uniform, with CV ranging only from 4.6–8.0 percent (Table 2). This low variability was possible because the chemical was mixed with homogeneous granular bait material before tableting (greatly lessening differential sorption related to morphological variations), and all tablets had the same size and weight. Tableting thus appears to be an excellent alternative for overcoming the variability of surface-coated treatments.

Tableting has additional advantages. For one, it provides a possible solution to the acceptance problems that often occur with surface-coated baits. Since the chemical is uniformly dispersed through the tablet, much less is concentrated on the surface where it may influence palatability by contacting the taste buds and reaching the olfactory receptors through volatilization. In addition, additives such as masking agents,

attractants, dyes, etc., can be incorporated without greatly altering the tablet's physical properties, and weathering characteristics can be controlled by the proper choice of binders and coating materials. These techniques for tableting, which are well documented and now routinely used in pharmacology, would allow the investigator to make uniform baits with almost any properties desired.

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