A Radioactive Evaluation Test for Dairy Cleaning Detergents

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A SATISFACTORY TEST for evaluating detergents used for cleaning dairy equipment has been needed. Such a laboratory test should give the same result as a field test. Therefore the laboratory test must reproduce as nearly as possible the actual field conditions: the soil, the test surface, and the washing operation. In addition, the method of measuring residual soil should be sensitive and accurate.

Some previous evaluating tests for detergents (4, 6) have been based on light transmission or light scattering by films on glass plates. However milk deposits on dairy equipment are usually on metal, not glass, so a modification of these tests would not be completely satisfactory.

A test based on the removal of milk deposits from a metal surface would more nearly approach the actual cleaning operation in a dairy. The best method for finding the degree of soil removal is to determine the residual soil. This requires a sensitive measuring method. Radioactive tracer methods have this sensitivity and have been used to evaluate various cleaning procedures (1, 3, 5). Furthermore the radioactive tracer method by-passes the problems of surface corrosion and cleaner deposition normally encountered in a metal-cleaning evaluation.

The test described in this paper is based on the removal of radioactive deposits from stainless steel. The radioactive milk is prepared by adding radioactive calcium-45 chloride to milk. An exchange takes place between the radioactive calcium-45 and the stable calcium in the milk, yielding "tagged" milk.

Stainless steel planchets are suspended in the "tagged" milk, which is then held at the pasteurization temperature for the usual period of time. A "tagged" milk deposit, which is similar to the actual deposit found in a dairy, forms on the planchets. The planchets are counted in a windowless counter (Figure 1) before and after the cleaning operation. The sensitivity of the radioactive tracer method permits a determination of minute traces of residual milk soil after a cleaning operation, thus making it possible to determine the degree of cleaning efficiency. The test procedure is as follows.

Add 1 ml. of calcium-45 stock solution (containing 0.05 millicuries) to 40 ml. of milk in a 250-ml. beaker and let stand for 10 min. Suspend four stainless steel planchets in the beaker so that they are half immersed in the milk. Place the beaker in a water bath (150 ± 2°F.) for 40 min.

Remove the planchets and rinse off all scum and milk with distilled water, except the firmly held surface deposit. Allow the planchets to dry over-night. Take duplicate radioactive counts of each planchet in a windowless counter.

Add 250 ml. of 0.15% detergent solution (distilled H_2O), fifteen ⅜-in. diameter rubber balls, and one soiled planchet to a pint Launder-Ometer jar (2). Do this with all four planchets. Preheat the four jars in the Launder-Ometer tray (100°F.) for about 10 min., then tumble the jars in the Launder-Ometer (100°F.) for 5 min.

Remove the planchets, rinse thoroughly with distilled water, let dry for 30 min., and take duplicate counts in a windowless counter.

Compare the radioactive counts due to calcium-45 on the planchet, before washing with the counts after washing, to determine the degree of soil removed.

The results obtained by using the above procedure on four detergent formulations are shown in Table I.

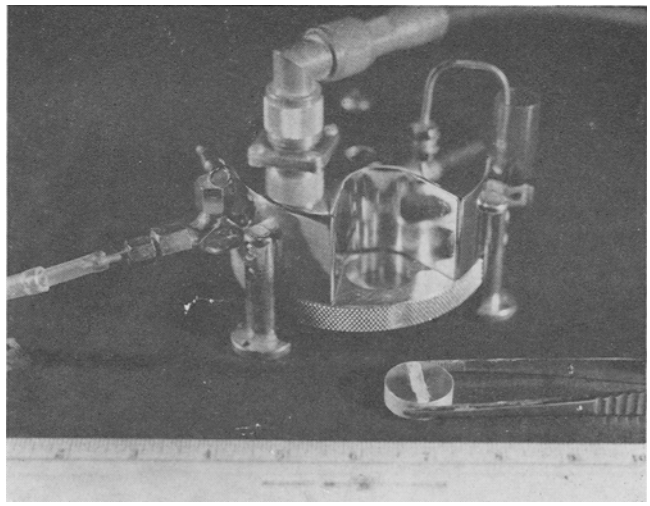

FIG. 1. Windowless counter (RCL Preflush Flow Counter Mark 12, Model 2) and soiled planchet.

The average values are shown in Table II.

Detergents B and D are about equivalent in their cleaning ability, approaching complete removal of milk soil. Detergents A and C are poorer in cleaning ability, leaving considerably more residual milk soil on the stainless steel surfaces.

This laboratory test offers a means of evaluating the relative cleaning abilities of detergent formulations that are to be used in removing milk deposits from dairy equipment.

TABLE I

Determination of Detergency Efficiency, Using Radioactive Calcium-45

Detergent[a] formulation	Planchet No.	Counts before washing	Counts after washing	% Residual soil
A	1	2,420	33	1.4
	2	1,780	37	2.1
	3	1.870	50	2.7
	4	1,510	49	3.2
B	1	2,810	9	0.3
	2	1,750	0	0.0
	3	2,620	5	0.2
	4	1,400	0	0.0
C	1	2,550	56	2.2
	2	3,540	103	2.9
	3	1,850	141	7.6
	4	1,700	76	4.5
D	1	2,320	7	0.3
	2	1,700	0	0.0
	3	1,540	10	0.7
	4	3,060	12	0.4

[a] A—Anionic and nonionic, nonionic predominates.
B—Anionic and nonionics, nonionics predominate.
C—Nonionics.
D—Anionic and nonionic, approximately equal mixture.
All four detergents are liquid, approximately 40% active.

TABLE II

Average Values for Determination of Detergent Efficiency

Detergent formulation	Average counts before washing	Average counts after washing	Average % residual soil
A	1,900	42	2.2
B	2,150	4	0.2
C	2,410	94	3.9
D	2,160	7	0.3

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Incorporation of Gossypol Into Eggs of Hens Fed Gossypol Schiff Bases

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IT HAS BEEN REPORTED (4) that gossypol fed to laying hens is deposited in the yolks as gossypol-cephalin and as gossypol-protein complexes. The amount of gossypol present in the yolk is reported to be a sensitive measure of dietary gossypol that is available to the hen, and it forms the basis for a useful definition of available gossypol units (AGU):

$$AGU = \frac{(Abs_{400} - Abs_{445})\text{diet egg} - (Abs_{400} - Abs_{445})\text{pre-diet egg}}{\%\text{ of material tested in diet}} \times 100$$

Where Abs_{400} = Absorbance at 400 mμ of a crude cephalin solution (see Experimental)

Abs_{445} = Absorbance of solution at 445 mμ.

This definition of available gossypol was proposed by Grau and coworkers (1) in their studies of the distinctive yolk component of eggs from hens fed gossypol. The gossypol was administered in cottonseed meal and also as gossypol added to purified diets.

Eggs with absorbance differences of less than 0.03 (that is, diet egg absorbances minus pre-diet egg absorbances) have been found not to discolor either on exposure to an ammonia atmosphere or during storage.[2]

There are two promising approaches to the gossypol-in-egg problem. These are either destruction of gossypol in the cottonseed meal, or its binding through nondigestible linkages. Cooking and screw-pressing the cottonseed reduce the available gossypol, but usually these operations are not sufficiently effective in reducing the gossypol to the point where significant amounts of processed meal may be fed to laying hens. Phloroglucinol can be used to destroy the gossypol in cottonseed meal, but the treatment may reduce the nutritive value of the meal (2). Preliminary experiments show that aniline does not bind the gossypol in cottonseed meals tightly enough to prevent some incorporation into the yolk.

In the present study gossypol Schiff bases prepared from aromatic and aliphatic amines were fed to laying hens at 0.1–0.5% equivalent dietary gossypol levels, and the eggs were examined by spectrophotometry for gossypol-cephalin content. Some of these compounds are apparently stable enough to preclude gossypol incorporation and subsequent discoloration of the eggs.

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[2] This specification applied only to cottonseed meals and to gossypol derivatives, not to apogossypol and its oxidation products.

Experimental

The examination of the eggs used in this study was restricted to the determination of the available gossypol units as defined above.

The crude cephalin solution was obtained by extracting 10 ml. of yolk exhaustively with acetone, then extracting the residue once with hexane-acetone (3:1), and bringing the volume to 15 ml. The absorbance of this solution was determined with a Beckman [3] Model B Spectrophotometer (1).

Each hen was its own control in that eggs were taken for comparison before the gossypol-containing diet was fed. These were the prediet eggs. The birds were then fed the gossypol-containing diet for at least 10 days before a "diet egg" was taken and examined for the presence of the gossypol-cephalin complex.

AGU values for a series of gossypol Schiff bases are recorded in Table I. Some of the gossypol Schiff bases listed in Table I have not been previously reported and are described in Table II. These compounds were prepared by heating for a few minutes under reflux an isopropyl alcohol solution containing one mole of gossypol and two and one-half moles of the amine (3). The reaction mixture was chilled in the ice-box over-night, and the precipitated base was collected, washed thoroughly with cold solvent, and air-dried. Some of the products were recrystallized from the appropriate solvent before analysis.

[3] It is not the policy of the Department to recommend the products of one company over those of any others engaged in the same business.

TABLE I

Available Gossypol in Eggs from Hens Fed Gossypol Schiff Bases

Gossypol Schiff base	Millimoles equivalent gossypol [c]	% of the diet as gossypol	AGU
Bis(phenylimino)gossypol	38.6	0.5	10.3
(Dianilinogossypol)	12.9	0.17	4.0
Bis(p-carboxyphenylimino)	11.2	0.15	35.5
Bis(benzylimino)	12.4	0.16	.....[b]
Bis(furfurylimino)[a]	12.4	0.16	.....[b]
Bis(cyclohexylimino)[a]	38.6	0.50	4.2
Bis(n-octylimino)[a]	38.6	0.50	0.0
Bis(n-decylimino)	38.6	0.50	0.0
Bis(n-dodecylimino)	38.6	0.50	0.0
Bis(n-tetradecylimino)	38.6	0.50	0.0
Bis(n-octadecylimino)	38.6	0.50	0.2
Bis(dehydroabietylimino)[a]	38.6	0.50	26.2–34.7
Bis(oleylimino)[a]	6.2	0.08	.....[b]

[a] New compounds not previously reported and described in Table II.

[b] Dietary gossypol level was too low to permit a reliable measure of the gossypol-cephalin content of the egg.

[c] Amount ingested per hen per test period.