zene was a commercial one representing the most widely used grade in Western Europe (molecular weight: 242). This solution was used at two use levels—6 gr/lt and 15 gr/lt.

- Heavy-duty powder (H.D.P.). The mixed active ingredient system used was 5 weight percent LAS, 5 weight percent N.I., 2 weight percent soap and 15 weight percent STPP. To avoid interference and to have a detergency result due exclusively to the surfactant system, no other ingredients were considered.
- Unbuilt heavy-duty liquid (H.D.L.). The composition used was 15 weight percent LAS, 20 weight percent N.I. and 10 weight percent soap.

Results and discussion

We also have included the detergency results obtained using not only the Launder-O-Meter but also the Terg-O-Tometer. Both pieces of equipment are used for detergency testing worldwide, and we feel it is more interesting to compare the data on a standard deviation basis than simply to give a reflectance value on a single piece of equipment.

TABLE 3

Detergency Performance (Δ Reflectance)[a]

		Use level			
		0.6 wt%		1.5 wt%	
		Water hardness (ppm $CaCO_3$)			
	Fabric	150	300	150	300
Launder-O-Meter					
	EMPA-101	3.4	3.5	8.1	4.9
	EMPA-104	7.9	8.7	16.3	8.0
Terg-O-Tometer					
	EMPA-101	19.9	19.3	31.3	22.9
	EMPA-104	28.5	29.5	42.5	32.0

[a]Unbuilt H.D.L. (LAS, 15%; N.I., 20%; soap, 10%).

Tables 1–3 depict the Δ R values obtained. The main conclusions are:

- The primary detergency performance in the Terg-O-Tometer is significantly higher than in the Launder-O-Meter.
- The relative standard deviation of the reflectance readings for the Launder-O-Meter is ten times the one found for the Terg-O-Tometer.

This can be summarized briefly as follows:

$$\text{Relative Standard Deviation} = \frac{\text{Standard Deviation} \times 100}{\text{Average of 6 readings}}$$

Terg-O-Tometer = 2%

Launder-O-Meter = 20%

We must point out, however, that this conclusion is valid only with the conditions used for the tests performed.

Radiotracer detergency method

The following is based on a talk given by Nelson E. Prieto of Shell Development Co., Houston, Texas.

The use of radioisotopes in detergency studies goes back many years. Shell Development has used radiolabeled soils and surfactants to study the various processes involved in detergency since the mid-1960s. The radiotracer detergency method makes use of mildly radiolabeled soils for quantitative determination of soil removal, among other properties, by simple standard radiochemical techniques (1). The method has been well developed to include a wide variety of available soils and provide enough flexibility to mimic natural soils and contemporary washing conditions. In general, the method involves soiling fabric swatches with the labeled soil(s), washing the fabric and determining soil removal by counting techniques.

Soil preparation and application

Many suitable labels are available for detergency studies. However, several conditions need to be considered when selecting the label. The choice is usually limited to the chemical elements present in the material of interest. The main determining factors that will affect your decision when selecting an isotope are ease of detection and labeling, purity, commercial availability, half-life, and safety/regulatory considerations. These are discussed in detail by Shebs (1).

Several radioisotopes can be used as labels, depending on the type of soil to be prepared, making the method very flexible, versatile and selective. Polar soils, such as fatty acids and alcohols, are labeled with Carbon-14, whereas nonpolar oily soils, such as mineral oil and triglycerides, use tritium as the label (2). Fabrics are soiled with these materials by applying an aliquot of a toluene carrier solution and allowing them to air-dry.

A particulate soil is also available. It is prepared by mixing a purified kaolinite clay with a clay fraction which has been activated by neutron bombardment (3). Fabric is padded with clay by agitating it in an aqueous suspension. This procedure avoids the difficult task of fixing the radiotracer into the clay matrix. The predominant isotope in the clay is europium-152.

A proteinaceous soil has been developed to test the specificity of enzymes in detergent formulations. It is prepared by binding chromium-51 to red blood cells (4). The test fabric is stained by spraying a labeled RBC suspension to a fabric pretreated with alcohol. The fabric is then heat-set in an oven overnight.

Multicomponent soils can be prepared by blending these soils. For example, a double-label soil simulating sebum can be prepared to study the overall removal as well as the selective removal of the components (Table 1) (2). This sebum soil can be blended with labeled clay soil to simulate more realistic laundering conditions (5). Surfactants can also be labeled with Carbon-14, tritium or sulfur-35 for testing other aspects of the detergency process (6).

Laundering methods

Many aspects of the detergency process can be studied using the radiotracer method (1-8). Soil removal is determined on an absolute basis for the overall process and/or the selective removal of individual components. It will give true soil removal and does not rely on an indirect method such as change in reflectance. Reflectance measurements mostly show trends in overall detergency. Quantitative determination of selective removal of components is impossible. The removal of polar and nonpolar soils, and blends of these, from fabric swatches is determined by analyzing the wash liquor (2). The removed soft β-ray emitting tritium and Carbon-14 labeled soils are detected using scintillation counting along with an internal standard. The analyses of clay and proteinaceous soils are done by counting directly the activity on the fabric swatches using γ-ray counters. The amount of proteinaceous soil on the fabric is determined by comparison with standards (4), whereas for clay soil the amount is obtained by difference, i.e., the fabric is counted before and after washing (3).

TABLE 1

Composition of Radiolabeled Multisebum Soil

Components	Label	wt %
Nonpolar		
Cetane	Tritium	12.5
Squalane	Tritium	12.5
Tristearin	Tritium	10.0
Arachis (peanut) oil	None	20.0
Polar		
Cholesteol	Carbon-14	7.0
Octadecanol	Carbon-14	8.0
Oleic acid	Carbon-14	15.0
Stearic acid	Carbon-14	15.0
Total		100.0

The radiotracer detergency method also has been used to determine soil redeposition (2,8), enzyme efficacy (4) and surfactant adsorption and rinsability in fabrics(6), in addition to soil removal. Some of these experiments were conducted using washing machines (6). The results obtained by the radiotracer method usually parallel those obtained using the reflectance technique but with somewhat greater precision. Customers' bundle test data also have confirmed results obtained using the radiotracer method. Overall, the method is very sensitive, assays require only two replicates, and it provides high precision (ca. 2%). Automation of the method using robotics increases its efficiency significantly, reducing manual labor dramatically (9). Experiments can be performed unattended around the clock. The method is very cost-effective since it yields a large number of analyses at a very low cost.

Safety considerations

The use of radioactive materials is regulated in the U.S. by federal guidelines. One must become familiar with these regulations to carry out radiotracer experiments. A license is needed to use radioisotopes in the laboratory. Training, experience and facilities are necessary and will depend on the broadness of the project. At Shell Development, only people with the necessary training, knowledge and license handle the stock soil solutions, which are stored in a designated area. Due to the low levels of activity of these solutions, dosimeter badges are not required throughout the method. The activity of the soils in the wash liquor is even lower, requiring long counting hours. Thus, the use of these materials for detergency testing is very safe (1).

Advantages and limitations

The radiotracer detergency method has advantages and some limitations. The main advantages are its sensitivity and analytical selectivity, capability to provide clear-cut results (overall or for specific components), high precision, use of low levels of activity, experimental flexibility, automation of complete method, and applicability to large-scale practical tests. Some limitations are the need for radiochemical technology (knowledge, equipment, safety license, experience), commercial availability and purity of labels and initial cost of investment (1). These limitations are being addressed by ongoing developments in radiochemical methodology.

Reflectance and radiotracer techniques can be complementary. Although reflectance methodology does not enjoy the precision, selectivity, sensitivity and some of the flexibilities of the radiotracer method, it is widely used to detect the removal of color bodies. The radiotracer method does not measure removal of color bodies, unless these are properly labeled. However, color body removal does not necessarily represent removal of the other components which can be removed, especially complex soils. Reflectance methodology is widely used in large-scale tests and offers the advantage of a low initial investment. The use of multiple labels in the radiotracer method reduces the number of experimental runs while increasing the amount of data collected, an important consideration when conducting a large number of tests.

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Soiled cloths are used in testing

The following is based on a talk given by George C. Feighner of Scientific Services, Oakland, New Jersey.

Throughout time, people have tested detergents. Until recently, testing was done subjectively by housewives using soap on their laundry. Now, there is a science dedicated to correlating and predicting consumer acceptance of laundry products with laboratory tests.

Standard soiled cloths are an important part of testing. The diversity of soils and stains is one problem; the variety of fabrics and laundry practice is another. Controlled conditions of laundering standard soiled cloths in a Terg-O-Tometer or full-scale washing machine produce results which can predict to some extent results in the home and, more importantly, allow scientists to evaluate solutions and innovations in the laboratory.

Since the discovery of soap, people have been comparing their laundry results. Application of scientific principles to study of the cleaning process has resulted in a considerable body of knowledge about testing new detergent developments. Laboratory testing of detergents requires controlled conditions and artificially soiled cloths which predict cleaning of real-world soils and stains.

In the laboratory, we like to see really dirty fabric become nearly white. With real-life laundry, soils are relatively light and any one stain uncommon. Redeposition, whether perceived or not, is probably the most serious laundry problem. Real life needs to be kept in mind in selecting soiled cloths for testing detergents, and the developer should remember that laboratory tests are an abstraction of