COPLEY SCIENTIFIC




Quality Solutions for the Testing of Detergents

2017 EDITION

POWDERS • TABLETS • LIQUIDS • CAPSULES

FOR LAUNDRY, DISHWASHER AND TEXTILE MANUFACTURING APPLICATIONS

Tergotometer

INTRODUCTION

Copley Philosophy			
Robust	✓	Reliable	✓
Easy to use	✓	Compliant	✓

Detergents are ubiquitous within our homes with increasingly sophisticated formulations cleaning our clothes, crockery and cutlery to exacting standards, in a wide array of domestic appliances. The effective removal of different types of soiling is essential but is also critical to maintain the integrity of the item being washed. Reducing the environmental burden of routine cleaning is an ongoing target and drives trends towards enhanced performance at lower wash temperatures, and the use of more environmentally benign ingredients. Detergent re-formulation is an ongoing, continuous activity.

To support formulation, for QC and for comparative performance testing, detergent manufacturers require relevant, reproducible, cost-efficient tests that can be applied easily in the laboratory. For laundry products this need extends to textile manufacturers, since the maintenance of colour and integrity in the face of successive washes is intrinsically associated with higher quality fabrics. Ultimately, the performance and impact of detergents is assessed under real-life conditions using a domestic appliance, but this is time consuming, costly and has a number of other limitations, with respect to routine use within the lab.

UNDERSTANDING TEST REQUIREMENTS

Laundry detergents are complex formulations incorporating a range of functional ingredients that may include: surfactants; buffers; chelating agents; enzymes; polymers; fragrances and optical brighteners. Their physical form has evolved from traditional powders through tablets, to liquid detergents and capsules, and the commercial marketplace is highly competitive. with consumers sensitive to both performance and price. Understanding the relative benefit of incorporating what can be expensive ingredients is crucial in product development, whilst exemplary QC is essential for long term market advantage.

When it comes to assessing product performance there are a number of factors to consider. The issue of stain removal is clearly paramount with a range of different soils routinely tackled - from wine, chocolate and fruit juice through to oil/grease, mud and blood. Effective cleaning relies in the first instance on removal of the soil from the fabric but preventing re-deposition is vital. Enzymes are routinely used to enhance soil breakdown, particularly for biological stains and for low temperature washing, while cellulosic/ polymeric ingredients are often incorporated to inhibit re-deposition.

Other characteristics that directly impact consumer opinion include the ability of the detergent to:

- Maintain whiteness - with optical brightening (OBAs) and fluorescent whitening agents (FWAs) often used to enhance perceived whiteness.
- Maintain colour - with many products now sold on their ability to reduce colour fading.
- Inhibit dye transfer - since this enables a broader range of clothing to be washed together.

Test requirements for **textile manufacturers** are closely aligned. Colour maintenance and dye transfer inhibition (fastness) are the primary issues but there are also special considerations for specific textiles. For example, fabrics for swimming apparel are routinely tested for their ability to withstand exposure to hypochlorite (chlorinated water).

The market for **dishwashing** powders, liquids, gels and tablets is equally competitive and formulations are just as complex, drawing on a range of ingredients with similar functionality including: complexing agents; surfactants; bleaching components; enzymes and wetting agents.

However, the focus of testing is slightly different with soil removal; the avoidance of re-deposition and the resulting finish (clarity and shine) the key characteristics assessed.

Detailed evaluation of the effect of a detergent on crockery and cutlery (for example, corrosion, loss of gloss, cloudiness) is typically more of an issue for those charged with specifying appropriate washing instructions for any given product.

The range of soils encountered is arguably less diverse but presents an equally tough challenge with requirements for oil/grease based soil removal, for example, quite different from those most suitable for the removal of proteinaceous deposits.

ESTABLISHING A TESTING STRATEGY

For all developers there is a need for test methods that are practical, reliable, reproducible and sufficiently differentiating to detect superior performance and/or any failure to meet a specification. Ensuring that testing meets these goals calls for careful consideration and control of the factors that can impact the results obtained.

These can be broadly classified under the headings: test equipment; test conditions and materials; analytical techniques and assessment.

Test Equipment

The most representative way to test a detergent in terms of customer experience, is in a full-scale domestic appliance.

However, for screening and QC this approach has some important limitations. Firstly, the equipment takes up a large amount of lab-space, is noisy, energy consuming and requires a large amount of test materials, water and detergents.

Typical washing cycles are around 60-90 minutes for laundry and 120 minutes for dishwashing so such tests are also lengthy, leading to a low analytical throughput. Furthermore, there is limited opportunity to closely control test conditions and the potential for "carryover" of soil or detergent, from one test to another is a recognised issue.

The **Tergotometer** directly addresses these issues and is a simple solution that enables more rapid, representative testing. It consists of a number of miniature washing machines (eight as standard) in a single bench top instrument.

Each test station comprises a 1000 mL glass test vessel (stainless steel on request) and an agitator.

Temperature, agitator speed and cycle time can all be controlled to apply the conditions required, and testing is typically complete in around 15-20 minutes (for a laundry detergent).

An important development of the Tergotometer has been the introduction of an accessory that enables it to be used for testing dishwasher detergents; test cycles for these products are even shorter - in the order of 10 to 15 minutes.

In summary, testing with a Tergotometer boosts analytical throughput in laundry detergent testing by around three to eight times compared with using a standard domestic appliance, at the same time enabling reproducible testing under closely controlled conditions. It is therefore extremely useful for generating the statistically significant data needed to justify claims of performance improvement.

Other routine applications include:

- Washability and colour fastness testing.
- The optimisation of washing conditions for specific detergents (e.g. temperature and water hardness).
- Assessment of brightening, softening and foaming.

Standard Agitators

Test Conditions and Materials

A primary consideration with respect to test materials is the source of the soil: "real life" or "laboratory produced". In the USA, for example, ASTM D2960 specifies the use of "real life" soils for home laundering products.

However, the use of "real life" soils does not easily facilitate the development of direct links between specific formulation ingredients and the removal of certain stains, or indeed, other aspects of detergent performance, or offer the level of control that can be achieved using laboratory produced soils. For screening, formulation and QC it can therefore be preferable to use laboratory produced stains, as specified in ASTM D3050 and D4008.

Traditionally, laboratory stains were manufactured and applied in-house - a messy and time-consuming process. Over recent decades, companies specialising in the supply of laundry samples with closely defined stains, pre-applied to a suitable substrate, have become well-established and it is now possible to purchase samples in a range of fabrics, sizes and volumes, for many different stains. Samples are also available in a range of colours, including white, to test colour maintenance and fastness.

Stain samples for dishwasher detergent testing are equally accessible, typically "baked on" to melamine tiles (to mimic the perforance of porcelain), although other substrates such as glass or stainless steel may also be usefully deployed.

Beyond the issue of soil there area number of test conditiona that can impact the results including:

- Detergent concentration.
- Temperature.
- Water hardness, pH, bleach, etc.
- Washing and rinsing times.
- Washing action
- The load.

Tergotometer (fitted with optional refrigeration unit)

Analytical Assessment

Once processed, the samples can be analysed manually (by eye) by comparison with a grey scale. Whiteness or yellowness indices can also be employed. The whiteness scale ranges from 0 (black) to 100 (white), the yellowness from positive (yellow) to negative (blue).

Instruments for direct **quantitative** assessement include the colorimeter whose relatively simple and robust construction makes it ideal for QC applications and the more accurate and sophisticated spectrophotometer which is more suited to high precision QC and research and development applications.

A product or surfactant can normally be considered successful when both criteria - stain removal and whiteness - can be said to have been met.

Similar methods can also be applied to the testing of light duty detergents, as used in domestic dishwashers, using a suitable accessory.

To illustrate the practical implications of testing, it is worth looking in a little more detail at a typical laboratory based procedure, using the Tergotometer, for a laundry detergent, and for a dishwasher detergent.

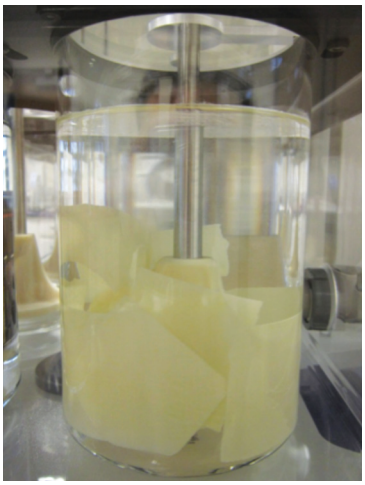
Loose fabric swatches being "washed"

LAUNDRY DETERGENT TESTING

The illustration above shows a Tergotometer set up for routine laundry testing; operation is simple.

Adjust the water bath to the desired temperature, add 1000 mL of water, suitably adjusted for hardness, and pH to each test vessel, and allow to equilibriate until the desired temperature has been achieved. The temperature is adjustable between ambient and 70 degrees C or between 10 and 70 degrees C with the optional refrigeration unit.

Add the prerequisite amount of detergent and operate the agitators at the appropriate speed (between 50 and 200 rpm) until homogenisation is complete.



The agitators operate in a preset pattern: 10 revolutions (360 degrees) clockwise followed by 10 revolutions anti-clockwise "ad infinitum" throughout the test (see Cat. No. 6401+6403).

Alternatively, the unit can be supplied for unidirectional clockwise operation only, if required (see Cat. No. 6401).

Plain 316 stainless steel paddle agitators, or paddle agitators which allow pieces of fabric to be attached to them, are also available on request.

Pre-stained fabric test samples are then dropped into each test station as loose swatches and "washed" for a defined period of time (see photo on previous page).

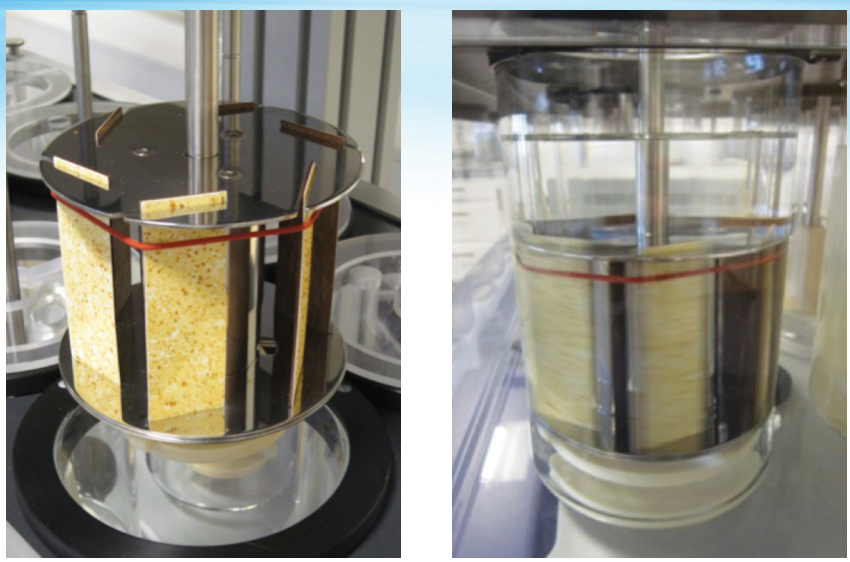
Each test station accepts **up to 12, 5 cm x 5 cm** or **6, 8 cm x 8 cm s**watches. This means that a total of 48 or 96 individual swatches can be processed simultaneously (42 or 84 where refrigeration unit used).

Note: A wide range of standardised pre-soiled test fabrics of suitable dimensions are available from the Center for Testmaterials B.V (www.cftbv.nl).

Once washing is complete the samples are removed from the vessels, wrung out, and rinsed in fresh water for a defined period of time. This washing and rinsing cycle may be repeated, as required.

All test swatches are assessed, both before and after washing/drying (and ironing if necessary). It is common practice to use an equal number of test and clean fabrics so that stain removal efficiency and re-deposition can be assessed in a single test. Triplicate testing is commonplace to safeguard the statistical significance of the results.

The Tergotometer measures 950 x 450 x 640 mm (w x d x h) including heater/circulator.



Dishwasher Testing Accessory (before and during the test)

DISHWASHER DETERGENT TESTING

Dishwasher Detergents can be tested in an exactly analogous way using the accessory (see above) which is affixed to the conventional agitator in each Tergotometer vessel.

Each accessory holds **six pre-stained sample tiles** (7.5 cm x 2.5 cm) mounted at an angle close to the side of the test vessel and held in position by a suitable O-ring.

This means that a total of 48 samples can be processed simultaneously (42 where refrigeration unit used).

The tiles concerned are available in various materials, melamine (to simulate porcelain), glass, stainless steel, etc. from either Copley Scientific or the Center for Testmaterials B.V. (www.cftbv.nl) and are pre-stained to customer selection e.g. Creme Brulee, Egg Yolk, Shepherd's Pie, Spaghetti Bolognese.

The coated/stained tiles are placed such that no two adjacent tiles have the same soil to improve test relevance.

Water volume and temperature, detergent concentration, agitator speed and test time are controlled, as with laundry detergent testing, and a rinsing cycle is similarly applied.

The tiles are then removed for drying and analysis, typically by spectrophotometry.

Vessels (Stainless Steel/Glass)

Cat. No.	Description
6401	Tergotometer (Ambient - 70 degrees C) - Unidirectional
6402	Refrigeration Unit (Ambient to 10 degrees C)
6403	Modification to allow for bidirectional rotation of the Stirrers
6404	Set of 8 Stainless Steel Paddles (option)
6404A	Set of 8 SS Paddles with holes for fabric attachment (option)
6405	Vessel, Glass, Flat Bottomed, 1000 mL with **"Easy-Centre"**
6406	Vessel, Stainless Steel, 1000 mL with **"Easy-Centre"** (option)
6407	Stainless Steel Bath (option)
6408	Dishwasher Slide Accessory for Tergotometer
6409	Pack of 10 O-Rings (spare)
6410	Pack of 60 Glass Slides (spare)

COPLEY
SCIENTIFIC

UK, Ireland & International Sales:

Copley Scientific Limited
Colwick Quays Business Park
Private Road No. 2, Colwick
Nottingham NG4 2JY
United Kingdom

Tel: +44 (0)115 961 6229
Fax: +44 (0)115 961 7637

e-mail: sales@copleyscientific.co.uk
web site: www.copleyscientific.com

Austria, France, Germany, Italy & Switzerland:

Copley Scientific AG
Erlenstrasse 27
Postfach 152
CH-4106 Therwil
Switzerland

Tel: +41 (0)61 725 25 35
Fax: +41 (0)61 721 31 87

e-mail: sales@copleyscientific.ch
web site: www.copleyscientific.com