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# Fundamental Principles of Fibre Fineness Measurement

## Part 8

### Radiometry



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## Scintillation Counting 1

### Liquid Scintillation Cocktail

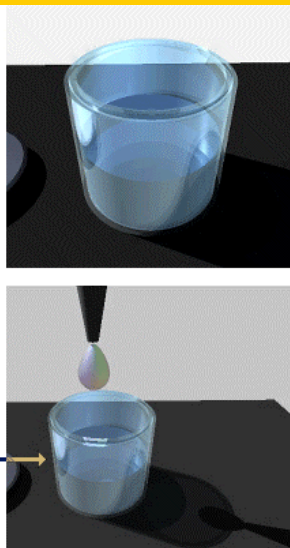
#### Components:

**Solvent:** Typically toluene, xylene pseudocumene, or an alkyl benzene type solvent.

**Emulsifier:** A detergent type molecule that ensures proper mixing of aqueous samples.

**Fluor:** A fluorescent solute.

Process: Radioactive Sample is added to scintillation cocktail.



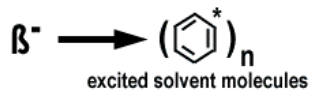
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The process of liquid scintillation involves the detection of beta decay within a sample via capture of beta emissions in a system of organic solvents and solutes referred to as the scintillation cocktail. This mixture is designed to capture the beta emission and transform it into a photon emission, which can be detected via a photomultiplier tube within a scintillation counter. The cocktail must also act as a solubilizing agent, keeping a uniform suspension of the sample.

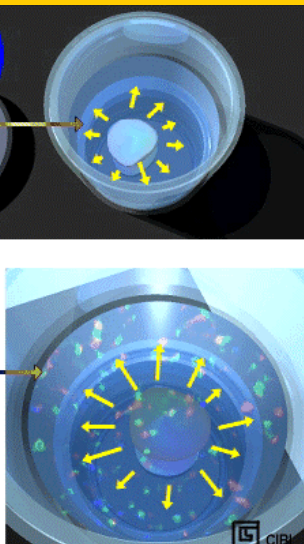
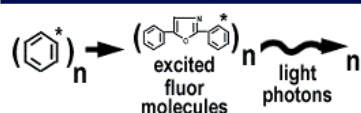
The scintillation counting system consists of three primary components: The radioactive substance, the solvent, and the solute (or fluor).

## Scintillation Counting 2

Beta particles are emitted, which cause solvent molecules to become excited.



The energy of the solvent molecules is transferred to the fluor molecules, which in turn emit light.



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The solvent is the first compound in the scintillation cocktail to capture the energy of the beta particle. The solvent molecule achieves an excited state, and the excess energy is transferred from solvent molecule to solvent molecule. The solvent remains in the excited state for an extended period of time, decaying into the ground state without the emission of light. The solute then absorbs the excitation energy of the solvent, and quickly returns to the ground state by emitting light. If a secondary solute is used, that solute absorbs the signal of the first solute and emits a second burst of light at a longer wavelength.

## RADIOMETRY

### Principle

Radiometric instruments utilise the phenomenon associated with the decay of radioactive substances, and the emissions of sub-atomic particles that is associated with this process, to monitor either rates of decay, or the concentration of the source of the emission.

The luminescence produced when radiation strikes a phosphor represents one of the oldest methods of detecting radioactivity and X-rays, and one of the newest as well. Liquid scintillation is one of the techniques relying on this phenomenon. Liquid scintillation instruments detect scintillations in a suitable liquid such as p-terphenyl in toluene, produced by low energy beta radiation from radioisotopes such as carbon-14, sulphur-35 and tritium.

The sample is generally dissolved in a solution of the scintillating liquid. A vial containing the solution is then placed between two photomultiplier tubes housed in a light tight container. The output from the two tubes is then fed into a coincidence counter, an electronic device that records a count only when pulses from the two detectors arrive simultaneously. The coincidence counter reduces background noise from the detectors and amplifiers because of the low probability of such noise affecting both sensors simultaneously.

The application of this technology to the measurement of the fineness of wool relies on the fact that the surface area of a wool fibre increases as the fibre diameter increases. This affords the possibility of absorbing the active isotope from a standard solution onto the wool fibre, separating the fibre from the solution and measuring the concentration of the isotope in the liquid. Alternatively the fibres can be labelled by immersing them into a suitable solution containing the active radioisotope, waiting until the isotope is distributed uniformly throughout the fibre, and then measuring the beta emissions directly, with the fibre immersed in a suitable scintillator. The beta particles from the isotope within the fibre will be absorbed and therefore not detected. The visible emissions will originate only from those atoms that are located in an annulus under the surface of the fibre. The fibres can then be

oxidised and re-measured to determine the concentration of isotope within the fibre. The ratio of counts before and after oxidation can be shown to be proportional to diameter as follows:



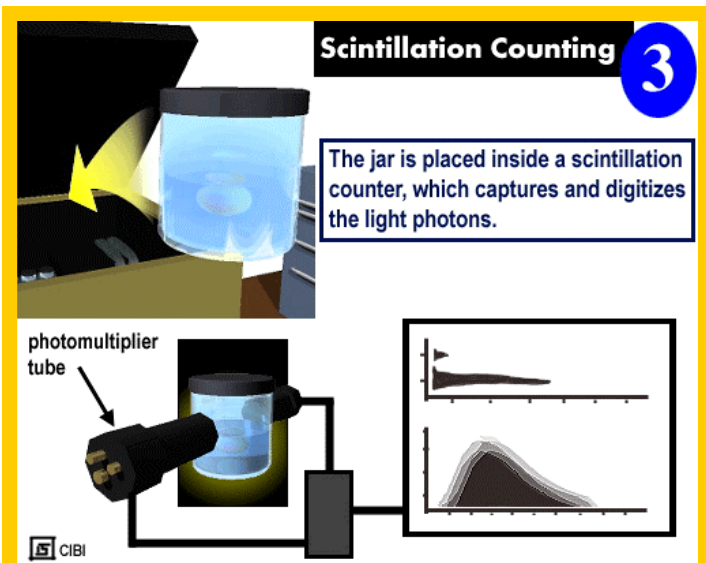
$$\frac{C_{raw}}{C_{oxid}} = \frac{4K(RD_s - R^2)}{D_s^2} \quad 1$$

where

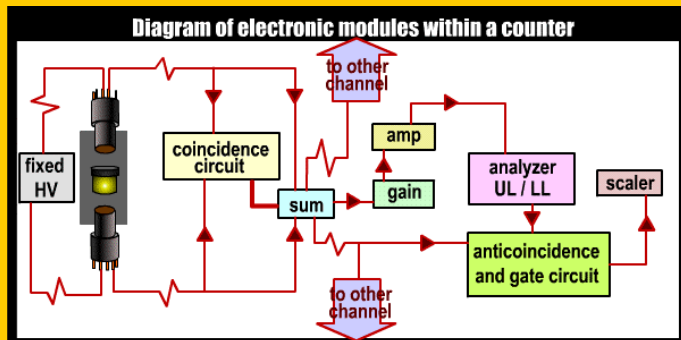
- $C_{raw}$  = count rate of the raw fibre
- $C_{oxid}$  = count rate of the oxidised solution of fibre
- $D_s$  = diameter of the fibre
- $K$  = a constant
- $R$  = the thickness of the annulus

Beta particles are low range and therefore the thickness of the annulus will be very much smaller than the diameter. Equation 1 therefore simplifies to

$$\frac{C_{raw}}{C_{oxid}} = \frac{K}{D_s} \quad 2$$



Soon after the discovery of the basic principles of liquid scintillation in 1950, instruments designed for counting began appearing, with the first commercial model becoming available in 1954. A schematic diagram of a scintillation machine can be seen below:



Most commercial scintillation counters are coincidence systems utilizing Photo Multiplier Tubes (PMT's) in tandem to monitor for a photon event. A pulse is not registered unless both PMTs view the incident photons within the predetermined time interval usually 20-30 nsec. If a pulse is recorded by the two PMTs within the 20-30 nanosecond window, a coincidence pulse is recorded that is a measure of the number of single events which occurred during the window. If an event occurs within only one of the PMTs, a coincidence pulse will not be recorded.

Source: Principles of Autoradiography, Dr S Gambhir, UCLA.

### Development

Downes and Till (1963) described the application of the liquid scintillation technique to analyse the concentration of tritium, carbon-14 and sulphur-35 in wool. They reported their finding that direct measurement of previously labelled samples, counted only those isotopes that were absorbed into an annular layer on the surface of the fibre and proposed that this phenomenon could enable estimates to be made of the fibre diameter.

In a further study in 1965 these authors reported on the effects of fibre length, fibre diameter, moisture and air bubbles on the efficacy of counting scintillations produced by wool labelled with tritium and sulphur-35. They reported that tritium provided more analytical sensitivity than sulfur-35 for the estimation of diameter. They also described an investigation showing that the method could be used to estimate the degree of yellowing of the fibre. The chemical reactions associated with the yellowing process quenched the beta emissions from the labelled samples to an extent that correlated with the degree yellowing.

Downes and Till (1968) reported further studies, using formic acid labelled with carbon-14, to measure the diameter of wool. They observed a linear relationship in the count ratio with increasing fibre diameter. This fall in count ratio was small (about 1% per micron) but the sensitivity was sufficient to suggest that the technique could be suitable for the rapid measurement of mean fibre diameter for a large number of samples. They identified some disadvantages of the method:



- Pigmented and/or medullated fibres would likely interfere with the measurement and produce a bias.
- The method depended on the volume or mass per unit length of fibre and therefore the derived diameter value would be volume biased, a factor that could cause unusual results for samples with unusual fibre diameter distributions.

In a further extension of their work Downes and Till (1968) examined wool samples labelled with tritium and carbon-14 by aqueous reaction with iodoacetic acid that had been labelled with one of these isotopes. The counting rate was measured firstly, with the wool suspended in the liquid scintillation solution (the direct method) or secondly, after oxidising the same sample and dissolving the product in another liquid scintillation solution. The counting rate determined by the direct method depended on the fibre diameter, because of self-absorption of beta particles by the wool whereas the counting rate after oxidation was constant. With the wools labelled with carbon-14 the ratio of the counting rates changes by 35 % for a diameter change from 14 to 35 microns. For the tritiated samples the analytical sensitivity was considerably greater, with a 100% change in the ratio of the counting rates, for the same range in diameter. The authors reported that the results were the basis for a new method for measuring mean fibre diameter.

Finally Downes (1971) described a method using liquid scintillation for determining the mean fibre diameter of wool. A precision of 0,2 microns was reported. The instrument was calibrated against samples where the mean diameter had been previously determined by Airflow.

### **Technical Issues**

The liquid scintillation system is a calibrated system, where the diameter obtained is directly related to the mean surface area of the fibres. It cannot provide information about distribution. It is unique among the methods that have been developed in that it is the only method that is directly related to surface area. The Airflow system also has a relationship to the surface area of the fibre, but it is less direct. Although Downes (1971) did not explicitly state it, this is probably why the Airflow measurements were used to calibrate the method.

The fact that the system relies on radioactive isotopes, may be of concern today, particularly with regard to occupational health and safety. However the isotopes that were used are handled routinely and safely in many clinical laboratories all over the world, and provided normal laboratory practice is followed they can be handled quite safely

The precision quoted by Downes is really quite amazing, but no documented studies based on inter-laboratory studies have been located. The Australian Wool Testing Authority evaluated the system during 1970's, with a view to utilising the method for flock testing services, but this work was abandoned before 1980.

The advantage of the system in this particular application is the possibility of automated analysis of large numbers of samples, where the major application of the data is for ranking animals. The Department of Agriculture, New South Wales, Australia used the method for many years, in the Department's Trangie laboratories.

### **Commercial Application**

The technology has never been used for the commercial trading of greasy or semi-processed wool, although on the limited data available it does appear to have adequate precision. Its use for testing of Fleece Samples has also ceased, simpler, faster and more precise technologies now being available.



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