**CEM**

ORACLE
Application Note

**Analysis of Whey Protein Concentrates**
Despite Reference Chemistry Limitations
(Extraction of Non-Lipid Contaminants)            ap0253v1

## Introduction

First introduced in the 19th century, gravimetric methods (e.g. solvent extraction) have been widely accepted for crude lipid (fat) determination in a broad range of foodstuffs. Over the years, however, it has been found that gravimetric methods can tend to overestimate the total lipid content in certain matrices due to the extraction of non-lipid contaminants.[1] While this "over-extraction" may be minimal for many matrices, whey protein concentrate (WPC) has been shown to be a matrix where this is especially prevalent using traditional gravimetric methods (Rose-Gottlieb and Mojonnier).[2]

It has been suggested that lipid extraction from whey protein concentrates is fundamentally difficult due to a combination of low lipid concentration, high protein concentration (can affect solvent phase separation), and the presence of stable lipid-protein complexes. Moreover, the accepted extraction methods involve heating in the presence of acid or base in order to dissociate the lipid-protein complexes. This can result in oxidative degradation and/or hydrolysis of particular lipids, thus affecting the recovery. Vaghela and Kilara have proposed an alternative approach which involves a cold extraction using a chloroform/methanol solvent system in addition to a gel filtration step in order to remove non-lipid contaminants. Applying this approach to a 75 % WPC sample, the authors found that the total lipid content was ca. 44 % lower than that determined without using a filtration step. Therefore, non-lipid contaminants comprised ca. 44 % of the crude extract.[2]

Despite the limitations of the current methods, the industry is unlikely to adopt alternative gravimetric approaches since the traditional techniques have gained such widespread acceptance. Since the ORACLE is insensitive to non-lipid contaminants (only sensitive to fat content), this can create an issue when attempting to validate the ORACLE against traditional gravimetric methods (likely a requirement for most installations). Even as CEM has confidence that the ORACLE is accurately determining the true lipid content in WPC, it is clear that there will be many customers that will want the ORACLE to match the traditional methods. In light of this, CEM has determined an approach that can effectively make the ORACLE match the traditional methods for WPC samples.

In an attempt to characterize the ORACLE readings for WPCs, over 40 WPC samples (ca. 1.2 – 7.5 % fat content) were analyzed and compared to the results from traditional methods. It was found that the ORACLE results averaged ca. 64 % of the value found using the traditional methods, which is in rough agreement with the 56 % recovery found by Vaghela and Kilara. Therefore, the ORACLE results for WPC samples can be adjusted to match traditional methods by simply increasing the ORACLE readings by ca. 36 %. It should be noted that although over-extraction of non-lipid contaminants is most significant in pure WPC, this trend has also been observed in other dairy powders. This is most common in dairy powders with high protein content or with WPC as an ingredient.

The remainder of this document describes the software implementation for this adjustment in both possible ORACLE analysis configurations: Fat only (stand-alone ORACLE), and Moisture & Fat (SMART 6 – ORACLE).

[1] Aued-Pimentel et al. *Quim. Nova*, **2010**, 33, 76 – 84
[2] Vaghela, M. N. and Kilara, A. *JAOCS*, **1995**, 72, 1117 – 1121
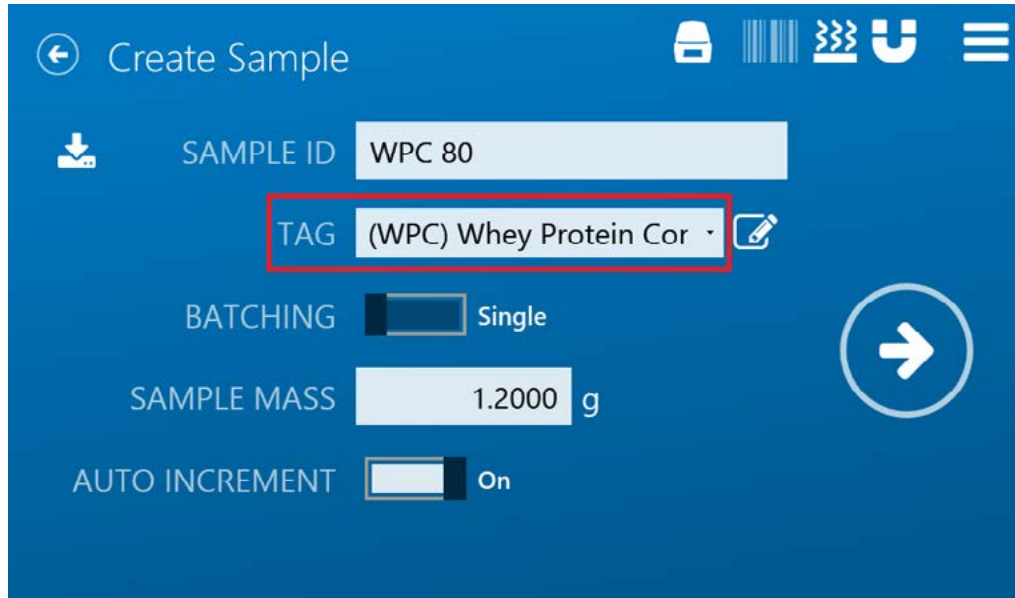
## ORACLE: Fat Only Analysis

Implementation in stand-alone ORACLE systems is done through the use of a specific *Tag*, named "WPC (Whey Protein Concentrate)". Upon selection of this *Tag* in the Create Sample screen, a ratio is applied and the ORACLE result is divided by 0.64. The final displayed ORACLE result is the standard ORACLE result increased by 36 % to account for the average percentage of non-lipid contaminants extracted in WPC by traditional methodologies.

For example, a WPC 80 with an expected solvent extraction result of 4.20 % fat would read 2.69 % fat on the ORACLE with no *Tag* applied. If the same sample is run through the ORACLE with the "WPC (Whey Protein Concentrate)" *Tag* selected, then the final displayed ORACLE result would be 4.20 % fat.

**Step 1: Activate** *Tags*

Select "Menu" → "Settings" → "Tags" → ON

**Step 2: Select "WPC (Whey Protein Concentrate)"** *Tag* **on Create Sample screen**



*Note- the "WPC (Whey Protein Concentrate)" *Tag* comes standard on every ORACLE with software version 1.6.0 and later. The ratio is embedded in the *Tag* and is not editable.

## SMART 6 – ORACLE: Moisture & Fat Analysis

Many customers analyzing WPC also require rapid moisture/solids for their WPC and other dairy products. A *Tag* is only applicable for ORACLE only users (no SMART 6) so the ratio is handled differently for the combination system (SMART 6 – ORACLE).

All method parameters for the combination system (i.e. drying, cooling, QuikPrep, biases) are accessed through the SMART 6, which is where the WPC ratio resides.

**Step 1: Login to Service Mode**

Select "Menu" → "Information" → "System Information" → "Service Mode" → ON (password: service)



**Step 2: Activate "Fat Ratio"**

Select "Menu" → "Settings" → "System" → "Fat Ratio"

**Step 3: Input 0.64 Fat Ratio**

Highlight Method and select the Edit icon → "Details" → "Fat Ratio"



*Note- the Fat Ratio default is always set to "1" unless manually changed. It has been empirically determined by CEM that 0.64 is the optimal ratio for WPCs, however, it may be necessary to modify based on a specific customer's historical results. The Fat Ratio capabilities are in SMART 6 software version 1.11 and later.