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Water determination – scientific and economic dimensions

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In trade,

the value of goods depends on

quality and

on the contents
of certain components.

It is therefore necessary

to analyse products

and determine

the content or
concentration of components

that decide the quality and the price.

Why is this problematic ?

Differences in analytical methods

may lead to

seemingly (!) different concentrations

and different quality grades.

Consequence:

Analytical methods should be harmonised.

Why can this be problematic ?

Official methods are usually adopted by heterogeneous bodies that do not consist of scientists only.

Economic and political reasons may influence decisions.

A certain group
or even a single powerful company

may influence

the introduction or the refusal of a new method,

if this method affects the price of their products.

This may lead to the situation that an official method is scientifically incorrect.

Here we have the clash of

scientific truth

and economic interests !

An example of

such a situation is given.

The price of dairy powders is fixed on the basis of dry matter.

Determination of water content is therefore an important analysis,

particularly for economic reasons:

The vendor wants to „find“
a water content as low as possible,
the buyer as high as possible.

Water content is very often determined by drying techniques.

Principle problems:

1. Drying techniques do not distinguish between water and other volatile substances.

2. Strongly bound water is not detected.

Consequence:

Result of drying techniques is not water content,

but mass loss under the applied (and principally arbitrary) conditions !

This mass loss is often called moisture.

Lactose problem

Scientific aspect:

Lactose exists in different forms.

α-Lactose: 1 mole water of crystallisation per mole
(5% by mass)

β-Lactose: anhydrous

Amorphous lactose: anhydrous, but water in interstices.

Real lactose samples: may be mixtures
(depends on production conditions).

In all cases, additional water can be adsorbed to the surface.

For dairy products, drying at 102 °C for 2 hours is common.

Problem:

Nearly all dairy powders contain lactose.
Water of crystallisation is only partially detected.

Consequence:

Result of drying (mass loss) of dairy powders is neither the surface („free“) water nor the total water content.

Lactose problem

Economic aspect:

Dry matter (*DM*) is calculated from the total mass (m_0) and the water mass (m_W) contained in the product:

$$DM = m_0 - m_W$$

Water mass can be calculated from water content (*WC*):

$$WC = m_W / m_0 \quad \rightarrow \quad m_W = WC \cdot m_0$$

$$\boldsymbol{DM = m_0 - WC \cdot m_0 = m_0 \cdot (1 - WC)}$$

Dry matter (price!) falls with rising water content.

Recently, a reference method for moisture determination of dried milk was adopted by the International Dairy Federation (IDF) and the International Organization for Standardization (ISO).

The introduction of this method was very strongly supported and pushed by parts of the dairy industry and a producer of an apparatus designed particularly for this method.

Scientific arguments brought forward against this method were rigorously denied and ignored.

How does the new reference method work ?

5 g sample are heated in containers
at 87 °C for 5 h
in a stream of dried air (33 ml/min).

The mass loss is defined as moisture content (without checking mass constancy).

As alternative,

a Karl Fischer method,
based on a selective chemical reaction,
was developed and suggested.

Investigations have been made to compare

classical oven drying (OD),

the reference drying method (RD),

the Karl Fischer titration

for various dairy products.

The drying results were observed after various drying times.

Table 1
Results for water content by Karl Fischer titration (KFT) and for mass loss by oven drying (OD) after 2 h and by "reference drying" (RD) after 5 h; n = number of replicates

Sample	Water content by KFT (n=5) [g/100 g]	Mass loss by OD (n=2) [g/100 g]	Mass loss by RD (n=2) [g/100 g]
Lactose	4.45 ± 0.19	2.45 ± 0.13	1.04 ± 0.03
Skimmed milk powder	3.92 ± 0.07	3.85 ± 0.00	3.94 ± 0.13
Full cream milk powder	2.65 ± 0.05	2.46 ± 0.02	2.72 ± 0.14
Whey powder	4.46 ± 0.05	2.12 ± 0.01	2.24 ± 0.07
Calcium caseinate	6.19 ± 0.11	5.62 ± 0.03	5.73 ± 0.02

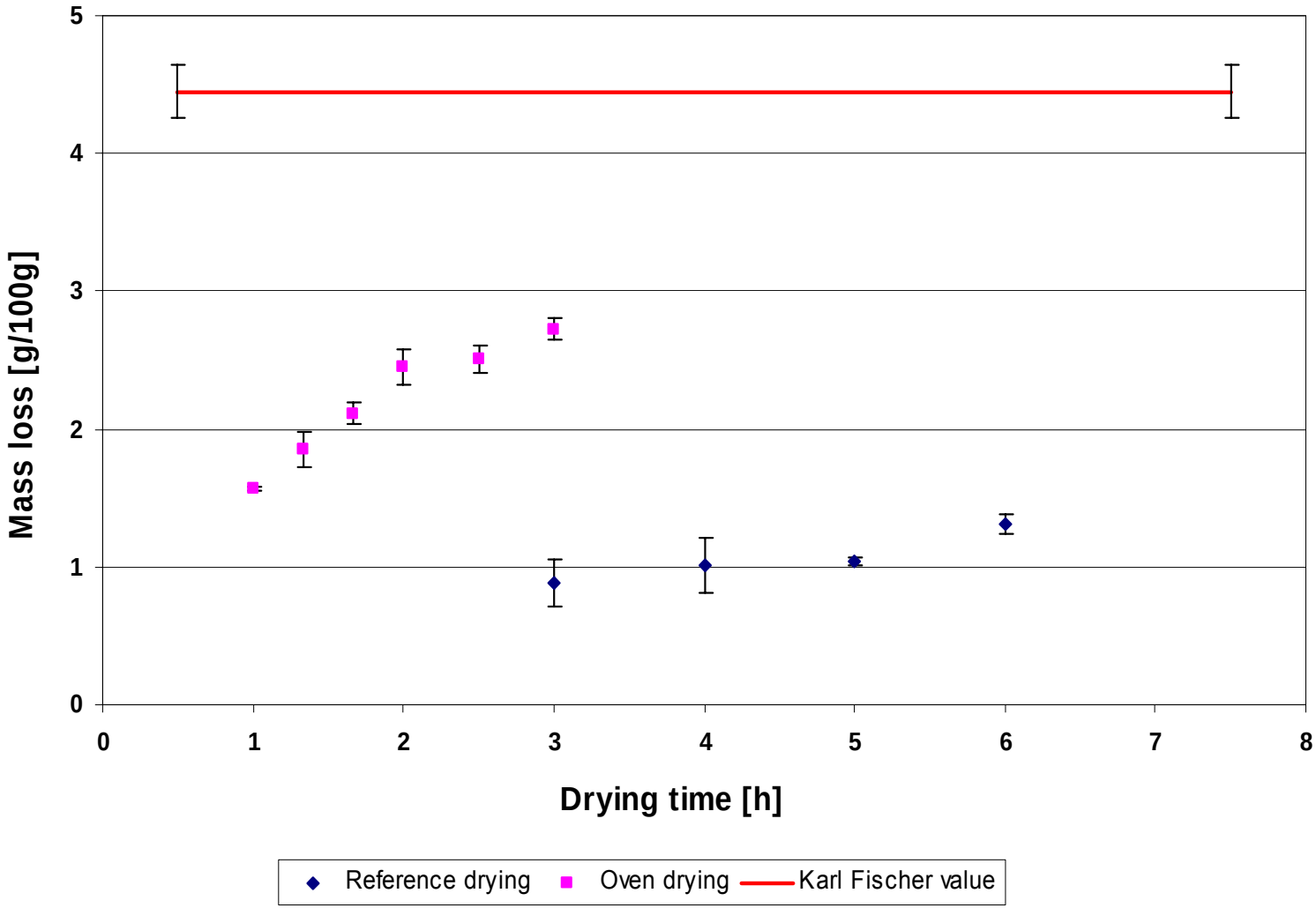

Figure 1. Mass loss by “reference drying” and oven drying after various drying times of ***crystallised lactose*** and – for comparison and reference – the water content by Karl Fischer titration (also in g/100 g).

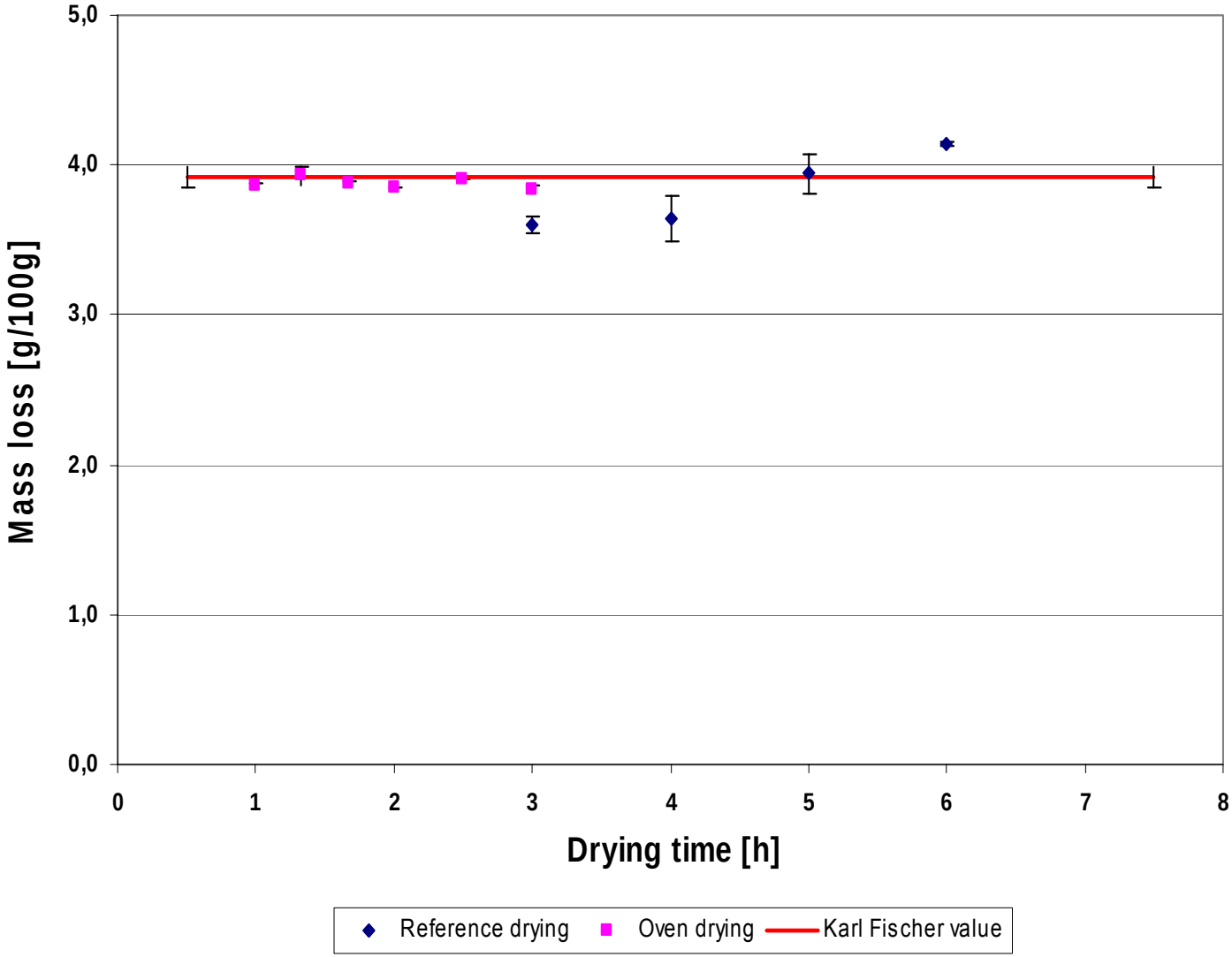

Figure 2. Mass loss by “reference drying” and oven drying after various drying times of ***skimmed milk powder*** and – for comparison and reference – the water content by Karl Fischer titration (also in g/100 g).

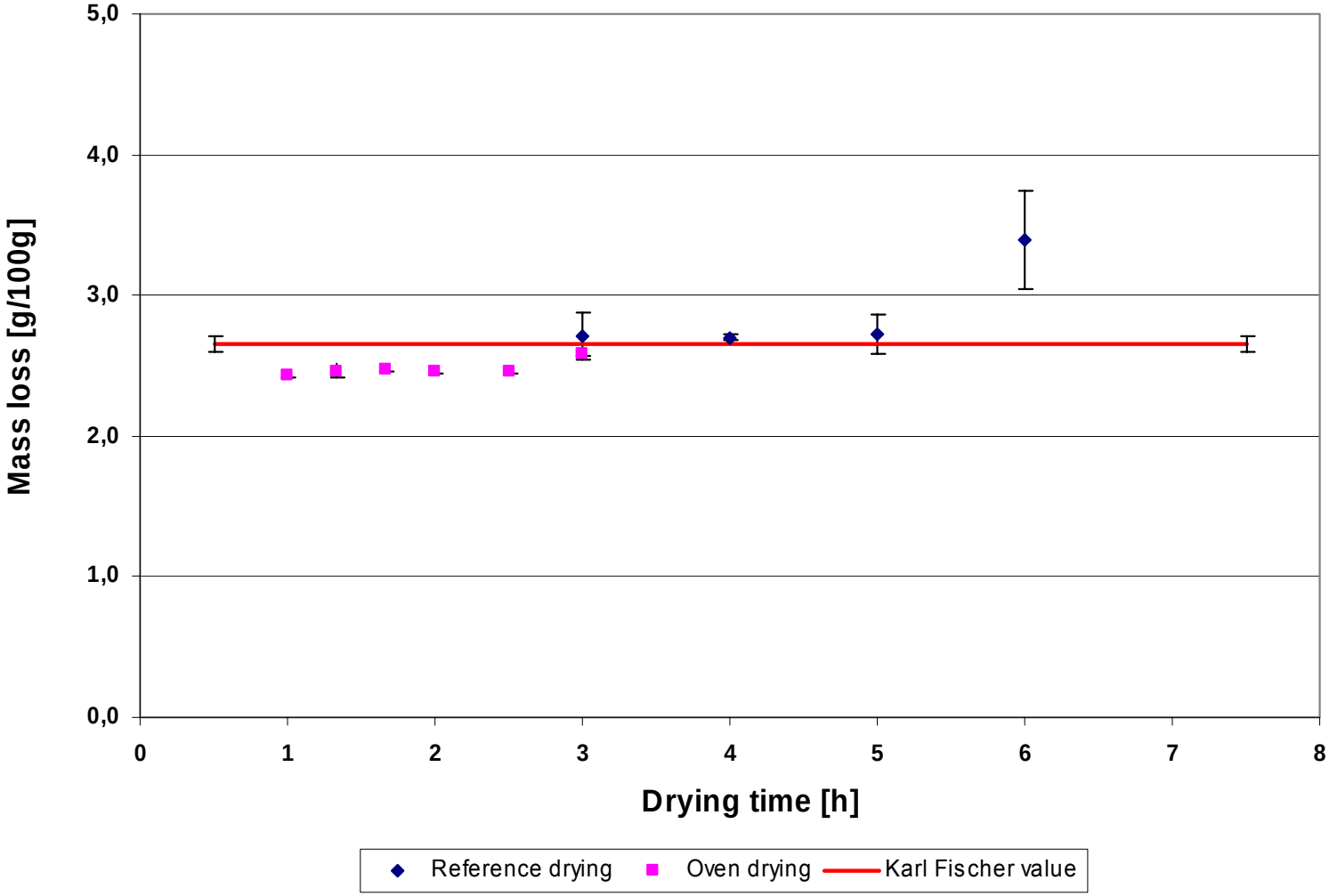

Figure 3. Mass loss by “reference drying” and oven drying after various drying times of ***full cream milk powder*** and – for comparison and reference – the water content by Karl Fischer titration (also in g/100 g).

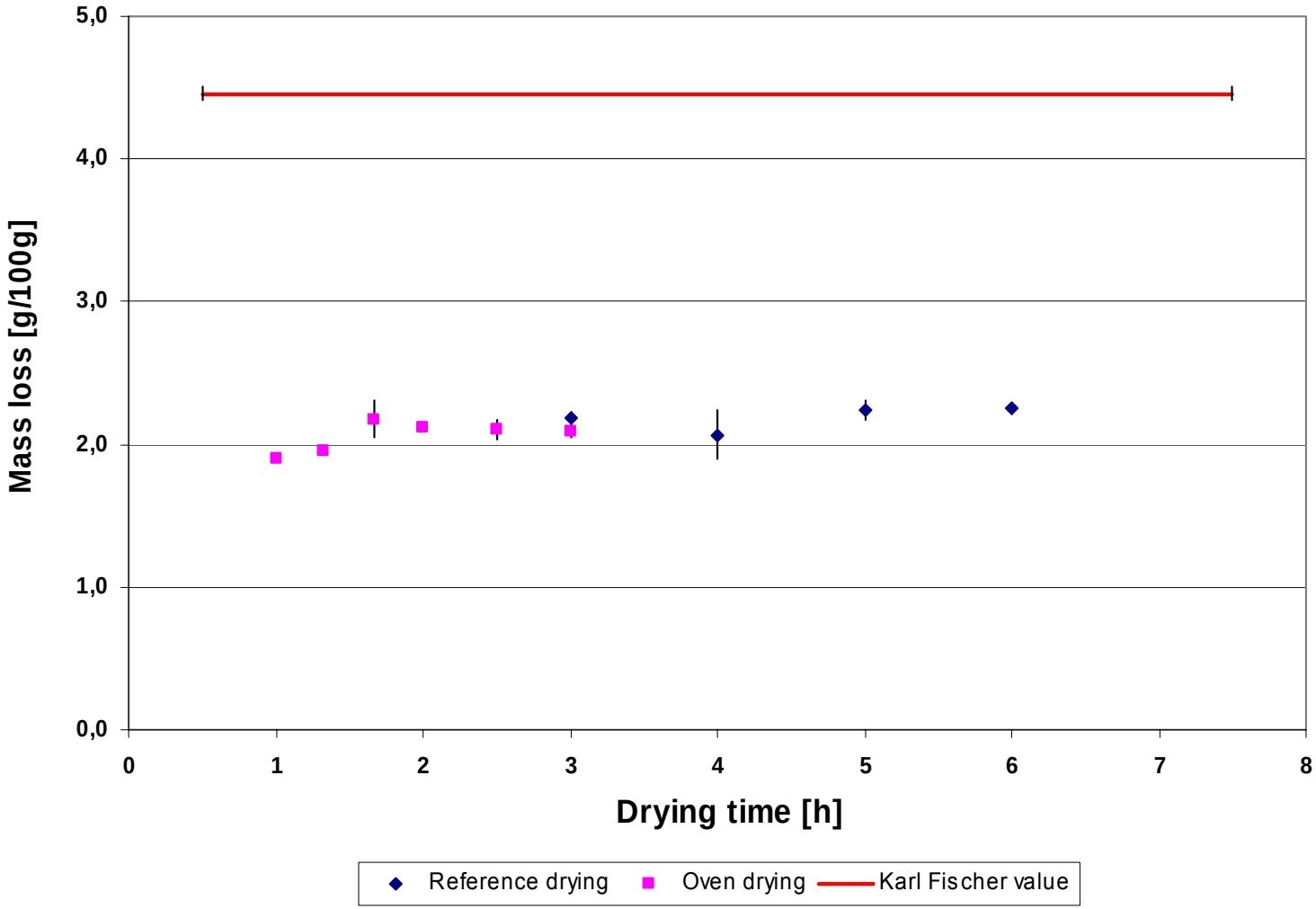

Figure 4. Mass loss by “reference drying” and oven drying after various drying times of ***whey powder*** and – for comparison and reference – the water content by Karl Fischer titration (also in g/100 g).

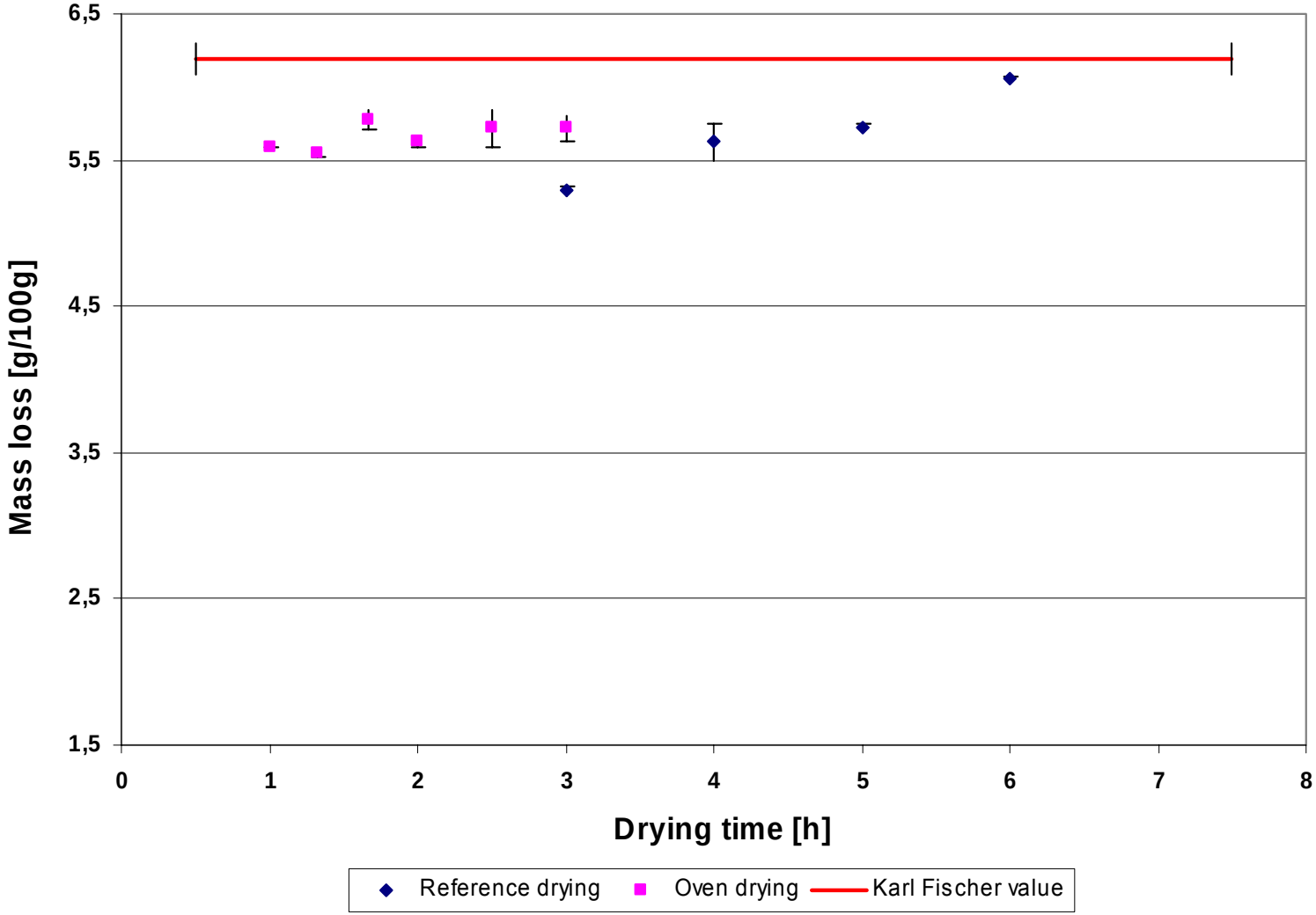

Figure 5. Mass loss by “reference drying” and oven drying after various drying times of ***calcium caseinate*** and – for comparison and reference – the water content by Karl Fischer titration (also in g/100 g).

Conclusions:

1. **The mass loss (“moisture”)**
   determined with the
   new reference method (by drying)
   corresponds approximately to the water content
   found by Karl Fischer titration

   in the case of ordinary milk powders,

   but not for lactose and products
   with high lactose content.

2. **The reference drying method does not determine the „free“ water only (which was claimed by the defenders of this method), but includes a part of the „bound“ water.**

   This had already been proven by an international inter-laboratory trial !

3. **The entity measured by the reference drying method is, therefore, not defined.**

4. **The Karl Fischer titration detects water, all the water and nothing but the water.**

5. **The Karl Fischer titration is generally applicable on all dairy powders.**

6. **The Karl Fischer titration would be a better (and scientifically correct) reference method.**

This example shows that

scientific evidence and argumentation may be not convincing enough to introduce standards or reference methods against commercial and economic interests.

This is a very unfortunate situation.