Parallel evaporation after extraction of liquid dairy samples following the Mojonnier method (ISO 1211:2010) – gravimetric fat determination

Syncore® Polyvap R-6, Vacuum Pump V-300, Interface I-300 Pro, Recirculating Chiller F-314, Refrigerated receiving vessel, R-6 sample holder: Fast drying under mild conditions

The fat in liquid dairy samples is usually extracted with an organic solvent that is then evaporated to complete dryness prior to gravimetric analysis.

In this short note, initial conditions and evaporation settings for the parallel evporation of milk fat dissolved in diethyl ether / light petroleum are presented. These settings have been optimized for fast and accurate results with simultaneously high solvent recoveries.

1. Introduction

The quantitative fat content determination of food is usually done *via* extraction using a lipophilic solvent. For milk and dairy products there are specific regulations for the quantitative determination since a protein shell generally encloses the fat in such products. This means that fat needs to be released, *i.e.*, by denaturing and removing the protein shell, prior to the actual analysis.

In the *Röse-Gottlieb* and the *Mojonnier* methods, this denaturation is done applying alkaline conditions. The sample protein is digested with ammonia (25 % solution) and the realeased fat is subsequently extracted with a mixture of diethyl ether (*bp* 34-35 °C, peroxide-free) and light petroleum (*bp* 30-60 °C). The fat is extracted twice (three times for high fat contents) with the diethyl ether / light petroleum mixture. Ethanol (96 vol%) acts as anti-gelling compound and is added to the denaturated sample prior the first two extraction steps.

The organic solvents are distilled off after complete phase separation and the isolated fat is dryed and weighted. After distillation of the organic solvents (including ethanol), the drying (1 h at 102 ± 2 °C) is repeated until the mass decrease is less than 1 mg.

The percental fat content w_f is calculated as follows:

$$w_f\ [\%] = \frac{(m_1 - m_2) - (m_3 - m_4)}{m_0} \cdot 100$$

m_0 weighted sample [g]
m_1 mass of the flask with extracted, dried fat [g]
m_2 mass of the empty, dried flask [g]
m_3 mass of the flask with extracted, dried blank [g]
m_4 mass of the empty, dried blank flask [g]

In the following paragraphs, the use of a Syncore® Polyvap for the parallel solvent evaporation of up to 6 extracted milk fat samples is shown in detail.

2. Experimental

Equipment: Syncore® Polyvap R-6 with primary S-condenser / Vacuum Pump V-300 including Woulff bottle and secondary condenser / Interface I-300 Pro / Recirculating Chiller F-314 / refrigerated receiving vessel (3000 mL) / R-6 sample holder.

Table 1: Syncore® Polyvap R-6 settings

Platform temperature	60 °C
Coolant temperature	5 °C
Vacuum cover temperature	50 °C
Rotational speed	350 rpm

Table 1 shows the settings for the evaporation step 9.5.12 of the ISO 1211:2010(E) using the Syncore® Polyvap R-6.[1] In combination with the pressure gradients programmed on the vacuum controller, a semi-automated and fast evaporation of the diethyl ether/ light petroleum mixture (1:1, Table 2) and the residual ethanol (Table 3) from the extracted sample is provided.

Table 2: Pressure gradient for the ether evaporation, V_{start} 130-150 mL

step	p_{start} [mbar]	p_{end} [mbar]	t [min]
1	930	930	12
2	930	750	5
3	750	750	10
4	750	600	2
5	600	600	18
6	600	1000	1
		total time	**48**

Despite the secondary condenser, it is recommended to empty the receiving flask after the ether evaporation to avoid that solvent passes the pump when further decreasing the pressure.

Table 3: Pressure gradient for the ethanol evaporation, V_{start} 15-20 mL

step	p_{start} [mbar]	p_{end} [mbar]	t [min]
1	950	150	2
2	150	60	1
3	60	60	3
4	60	1000	1
		total time	**7**

3. Results

The extracted milk fat samples could be evaporated to dryness fast and under mild conditions. After complete evaporation, the additional drying at 102 ± 2 °C as described in the ISO norm leads to precise results.

Concurrently, more than 90 % of the evaporated solvents are recollected in the refrigerated receiving vessel and at the secondary condenser and can be recycled for further use.

4. Conclusion

The Syncore® Polyvap equipped with a refrigerated receiving vessel connected to a recirculating chiller and a vacuum system including a secondary condenser provides a fast and mild method of semi-automated sample evaporation for extracted dairy probes. Precise gravimetric results and high solvent recoveries are achieved.

Thus, the Syncore® Polyvap is a versatile and economic solution for the effective parallel sample evaporation of extracted dairy samples.

5. References

[1] ISO 1211:2010(E), *Milk – Determination of fat content – Gravimetric method (Reference method)*