THE CHALLENGE OF SO_2 DETERMINATION IN GRAPE JUICE II

M. Hofsommer

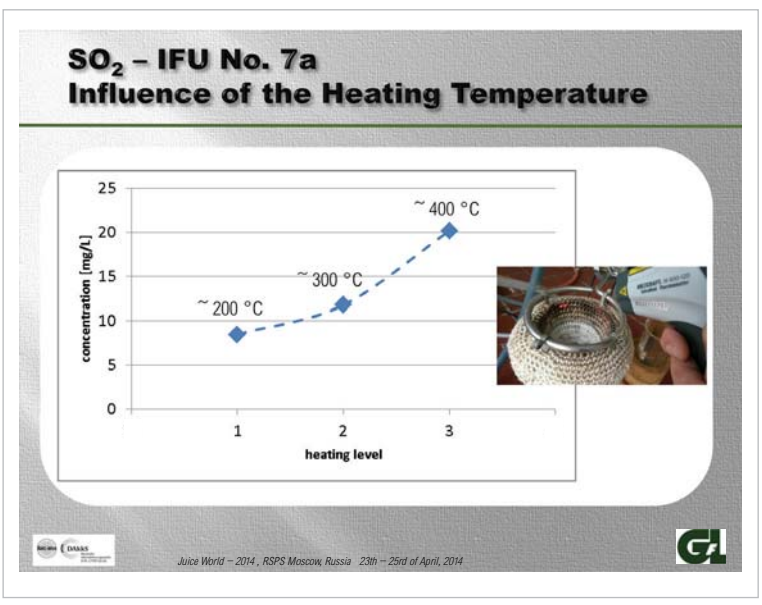
It is now 25 years since Hans-Jürgen Hofsommer published his article on „The Challenge of SO_2 Determination" in the German magazine FLÜSSIGES OBST (Issue 1, 1989, p. 22). Since then, analysis techniques have undergone some rapid developments. LC-MS/ MS and NMR are no longer scientific mysteries. Other things have changed very little. The next two paragraphs are taken verbatim from the introduction to the cited publication.

„The sulphurous acid content (total SO_2) is without doubt an essential assessment criterion for grape juices. According to the German federal food regulations, the total sulphurous acid content must not exceed 10 mg per litre. This limit is always a problem for sulphur neutralising or the delivery of desulphurised grape juices and thus puts SO_2 measurement in the limelight.

Many methods exist to determine the SO_2 content, and the details differ depending on the type of food being analysed. It is not possible to transfer a method dedicated to a particular type of food to another product. Even minor modifications of the method usually produce inconsistent results."

Since that was written, several round robin tests have been conducted, confirming that, if performed correctly, the IFU 7a method is reliable within the anticipated margin of error. It may not be obvious to the non-analyst that 9 mg per litre and 11 mg per litre are „identical" readings, even though one is below the magic limit and the other above it. This is where a revision dating back to 1989 becomes relevant: allergen labelling is regulated in Directive 2003/89/EU Annex IIIa (now known as the Food Allergen Directive), which also sets the limit at 10 mg SO_2 per litre. Some people erroneously assume that anything in excess of this limit must automatically constitute a health risk to the consumer. However, this limit is based exclusively on the *de facto* detection limit of the method, and not on systematic toxicological studies (see draft scientific opinion on the evaluation of allergenic foods, EFSA Journal 2014). The margin of measurement error mentioned above is illustrated in a theoretical experiment, in which someone tries to count the hairs on their head with the greatest possible precision and care. We realise, of course, that the result of a second count may differ slightly from the first one. Some hairs may have been counted twice, others might have fallen out between counts, etc. The same applies to all measurements – there will be slight variations, and the result looks „inaccurate". There is nothing wrong with that, but it is important to know how much variation there might be. To be unacceptable, the result must be clearly over the limit including the margin of error. The principle of *„in dubio pro reo"* (benefit of the doubt) applies. In everyday scientific practice, SO_2 measurement unfortunately often shows that results from juices stored with sulphur often diverge more than the laboratory limit comparisons (R = ± 4 mg/l) anticipate. There are two main reasons for this:

1. Laboratories do not obey the rules to the letter. This is all the more dramatic as the quality assurance measures expected of ISO 17025 accredited test laboratories



are not always carried out. For instance, a recovery ought to be determined regularly, for example by weighing approximately 30 g of analytically pure sodium bisulphite ($Na_2S_2O_5$) into a 100 ml. flask and filling up to the mark with distilled water. This solution is diluted 1:10 (10ml // 100 ml) and can then be used as a normal standard to determine SO_2. The recovery can be calculated using the formula:

$$\text{Recovery (\%)} = \frac{SO_2 \text{ analysis result (mg/l)} \times 100}{0{,}647 \times \text{weighed quantity sodium bisulphite (mg)}}$$

The recovery should be over 85 %, otherwise error analysis is needed and the results of real samples are untrustworthy.

2. A second influence that was underestimated for many years is the temperature at which the sample is distilled. Experiments prove that higher temperatures can produce higher values.

Although the method prescribes a 130 watt mushroom heater, this may only make an inaccurate result look accurate. The IFU Analysis Commission is already discussing an appropriate update or refinement of the definition, so here's hoping that there will be no need for another report on SO_2 in 2039.

AUTHOR

Mikko Hofsommer
GfL Gesellschaft für Lebensmittel-Forschung mbH
D-10787 Berlin, Germany

www.gfl-berlin.de

About GfL

GfL is one of the world wide leading laboratories in the field of fruit and vegetable juice. Theye analyse about 15.000 samples per year on adulterations and authenticity. The percentage of international customers is near 40 %. GfL works for growers and distributors in all major fruit exporting countries. Ranging from Brasil and Argentina to Florida, from Finland to Greece, Northern Africa, Middle East via China, Vietnam, Thailand to Australia. Furthermore the processing and bottling industry makes use of our services as well as retailers throughout Europe and other parts of the world.

GfL was founded in 1984 as a private counselling and service company which is autonomously and independently active for the entire food industry. The objective target is to provide comprehensive services beyond analytical measurements but to give professional solutions for bordering fields.

Legal requirements on quality and security of food and the obligation for executive care of the producer are growing. Fast, qualified analyses and capable consultancy are provided.