Letter to the Editor

Analysis of Fatty Acid Methyl Esters (FAME) with High Accuracy and Reliability[1]

Sir,

In a recent paper Bannon et al. (1) describe the gas chromatographic (GC) analysis of methyl esters of fatty acids containing four carbon atoms or more. The authors kindly refer to our paper on the same subject, which was published lately (2), and state that this is ". . . the only study to date, with all considerations in mind of the methodology of Christopherson and Glass (3) and the subsequent GC analysis (of FAME). . . recognizing the widespread occurrence of inconsistent and erroneous results." Unfortunately, some of our results have not been interpreted correctly, which is the reason why we wish to add the following information.

In our paper we have put forward two important points (and Bannon et al. state the same): (I) In the GC analyses of fatty acid methyl esters (FAME's), accurate response factors are needed to compensate for the different responses of the various FAME's in the flame ionization detector (FID); (II) response factors must be used that are based on the "theoretical" FID response factors for the individual FAME's; it is improper to use factors other than the "theoretical" ones in order to compensate for systematic errors in the methodology.

Now Bannon et al. state that our methodology must contain such errors because our response factors are considered to be different from the ones which he calculated theoretically. Unfortunately, this conclusion is based on a misinterpretation of our data. In our paper response factors are given for the calculation of fatty acid composition on the basis of mass percent of fatty acids (as is usual in the dairy industry in several countries), whereas Bannon et al. use response factors to express their results as mass percent of fatty acid methyl esters.

If all response factors are converted into those used for expressing results as mass percent of FAME, the factors used by Bannon et al. and by us are, respectively: for methyl butyrate, 1.54 and 1.57; for methyl caproate, 1.31 and 1.26; for methyl caprylate, 1.19 and 1.14, and for methyl caprate, 1.12 and 1.08 (all factors relative to methyl stearate = 1.00). These figures clearly show that the differences between the corresponding response factors used by Bannon et al. and by us are small. Our results should have been studied more carefully before stating that our method contains errors. Another point of interest are the so-called theoretical response factors used by Bannon et al. Indeed, Ackman and Sipos (4) conclude that the FID response of saturated FAME is proportional to the mass percent of carbon content of the ester, excluding the carbonyl C-atom. However, it should be noted that in another publication Ackman and Sipos (5) state: "The deficiency in response for the lower esters (less than nonanoic acid, excepting formic acid) is approximately 1.5 carbon atoms." This carbon atom deficiency was found to be 1.4 for methyl butyrate, 1.4 for methyl caproate and 1.2 for methyl caprylate. Surprising to say, this paper is not cited by Bannon et al., who calculate the "theoretical" response factors also for the lower FAME's on the basis of a carbon atom deficiency of 1, instead of using the value 1.2–1.4 estimated by Ackman and Sipos (5). Since, in the calculation of "theoretical" response factors for FAME's, the carbon atom deficiency depends on the chain length, it is difficult to speak of "theoretical" response factors for the lower FAME's which are calculated according to a general rule. Therefore, it might be better to determine response factors accurately for individual FAME's in carefully conducted experiments using different methods which do not contain systematic errors. These response factors should be generally applicable, i.e., they should not require corrections which are sometimes used to compensate for systematic errors incidental to certain methods.

From our experimental work we have learned that the response factors determined are valid for three different GC systems (with different types of column and nondiscriminating injection techniques), and that they are independent of the type of sample: either FAME's prepared from triglycerides of known composition and purity (2) or samples of FAME's also of known composition and purity.

To ensure accurate and precise quantitative analyses of FAME's by GC, it is essential that none of the steps in the sequence of operations (from isolation and conversion into FAME's to final GC analysis) should affect the qualitative and quantitative results. In order to prevent systematic errors (for which in certain methods corrections are made by using adapted response factors) it is important that a number of essential factors are taken into account which, among others, are: quantitative conversion of TG into FAME's, inert system for GC analysis (injector, capillary columns), nondiscriminative sample introduction (i.e., cold on-column or PTV-injection), and correct detection/data acquisition. Further details have been given elsewhere (2).

REFERENCES

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[1]The Editor apologizes for the delay in publishing this Letter. The original was received on April 15, 1986 and was accepted on April 22, 1986, but was lost in transit.