RESEARCH NOTE

The Determination of Alcohol in Wines by Means of Near Infra-red Technology*

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A rapid method for alcohol determination using Near Infra-red technology was investigated and compared to the standard pycnometric method. The apparatus was tested to show wine colour and sugar content had an influence on the analysis. The reproducibility of the technique was also checked and compared to that of the pycnometric method. It was found that colour, turbidity and sugar have little effect on the analysis and the reproducibility of the Near Infra-red apparatus had a standard deviation of ≤ 0,04% as compared to the ≤ 0,06% of the pycnometer.

The pycnometric method used requires volumetric measuring and distillation and is time consuming. With these difficulties in mind an instrument using Near Infra-red (NIR) technology was developed and after several years of research the machine was refined and is capable of determining alcohol in beverages. The advantages of this method are precise, accurate and rapid results (every two minutes), minimum preparation of the sample and the possibility of analysing alcohol in a large variety of beverages (Anon., 1984).

The principle of NIR technology can be summarized as follows. Light may be defined as electromagnetic radiation with wavelengths ranging from Infra-red, through visible to ultra violet. The part of the spectrum visible to the human eye extends from about 400 nm to 700 nm, while the Infra-red extends from about 2 500 nm to 15 000 nm. Near Infra-red is considered as the part of the spectrum lying between the visible region and the Infra-red region. NIR covers the range of wavelengths from 800 nm to 2 800 nm (Weyer, 1985).

The sample to be analysed is irradiated with NIR rays of different wavelengths. These rays are obtained in some instruments by passing a light beam through a series of rotating filters, or in others by passing it through tilting filters of varying wavelengths. The third type is obtained by reflecting the light beam from an oscillating holographic grating. Each type of chemical band within the sample absorbs NIR rays of a specific wavelength. Other rays are reflected as shown in Figure 1 (Groenewald, 1986). The scattered reflected rays of each wavelength are concentrated onto a gold-plated intergrating sphere, as shown in Figure 2 (Weyer, 1985). The analysis is based on a calculation of the amount on NIR light reflected from the sample at different wavelengths using the method of Norris and coworkers as reported by Weyer (1985).

A study was carried out at the KWV analytical laboratories in Paarl to compare the NIR method of alcohol determination in wines with that of the standard pycnometric method.

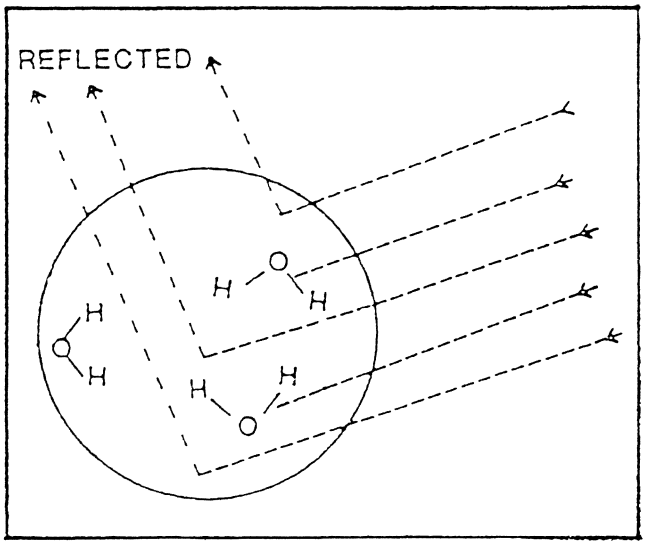

FIG. 1.
Absorption of NIR rays by O-H bond in water molecules.

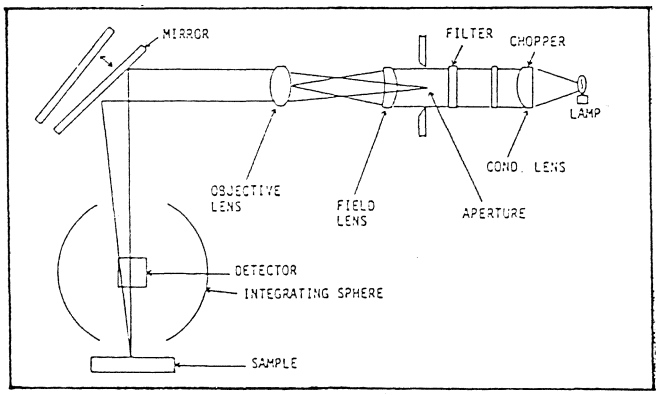

FIG. 2.
Optical system of the Technicon model 400 spectrophotometer (courtesy of Technicon.)

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MATERIALS AND METHODS

Alcohol determination

1. *NIR Method:* The apparatus used for this method was a Technicon Infra-Alyzer 400. The wavelengths used on the Infra-Alyzer are situated between 1 000 nm and 2 700 nm. Calibration was done for a given substance in a given environment and constants were transferable from one machine to another. A minimum of 30 samples was necessary for calibration and had to be distributed uniformly in a linear fashion over the whole range of alcohol concentrations to be analysed. The apparatus was calibrated for each of these alcohol ranges which, for this experiment, varied from 5 vol % to 8 vol %, 8 vol % to 16 vol %, 16 vol % to 19 vol % and 19 vol % to 23 vol %. The samples were analysed by the Infra-Alyzer and the reflection values were stored in memory for each filter and for each sample simultaneously, the value being obtained by the manual analysis. A cycle of regression was started on the calculator as soon as all the samples had been recorded. The f-values were obtained and entered into the microprocessor.

Each sample was run through the machine which measured the reflection from the wavelength chosen. The alcohol values were automatically displayed on the display screen after the values had been transferred into the microprocessor.

2. *Pycnometric method:* The standard pycnometric method for alcohol determination as described by Amerine & Ough (1974), was used as the comparative method.

Samples

Wines (345) were randomly selected and analysed in duplicate. The samples consisted of the following wine types:

Dry Red wine	- 106
White (sweet and dry) wine	- 113
Rosé (sweet and dry) wine	- 26
Distilling wine	- 62
Fortified wine	- 38

The sugar content of the wines differed from 1 (dry wines) to 200 g/*l* (noble late harvest and jeropigos). The samples were not always clear (some very turbid) and the colours varied dramatically, including such extremes as virtually colourless, rosé, dark red and dark brown.

Repeatability

Repeatability of the two methods was investigated by different analysts analysing twelve wines in duplicate by both methods on two different days ten days apart. A comparison table between the two methods was then set-up.

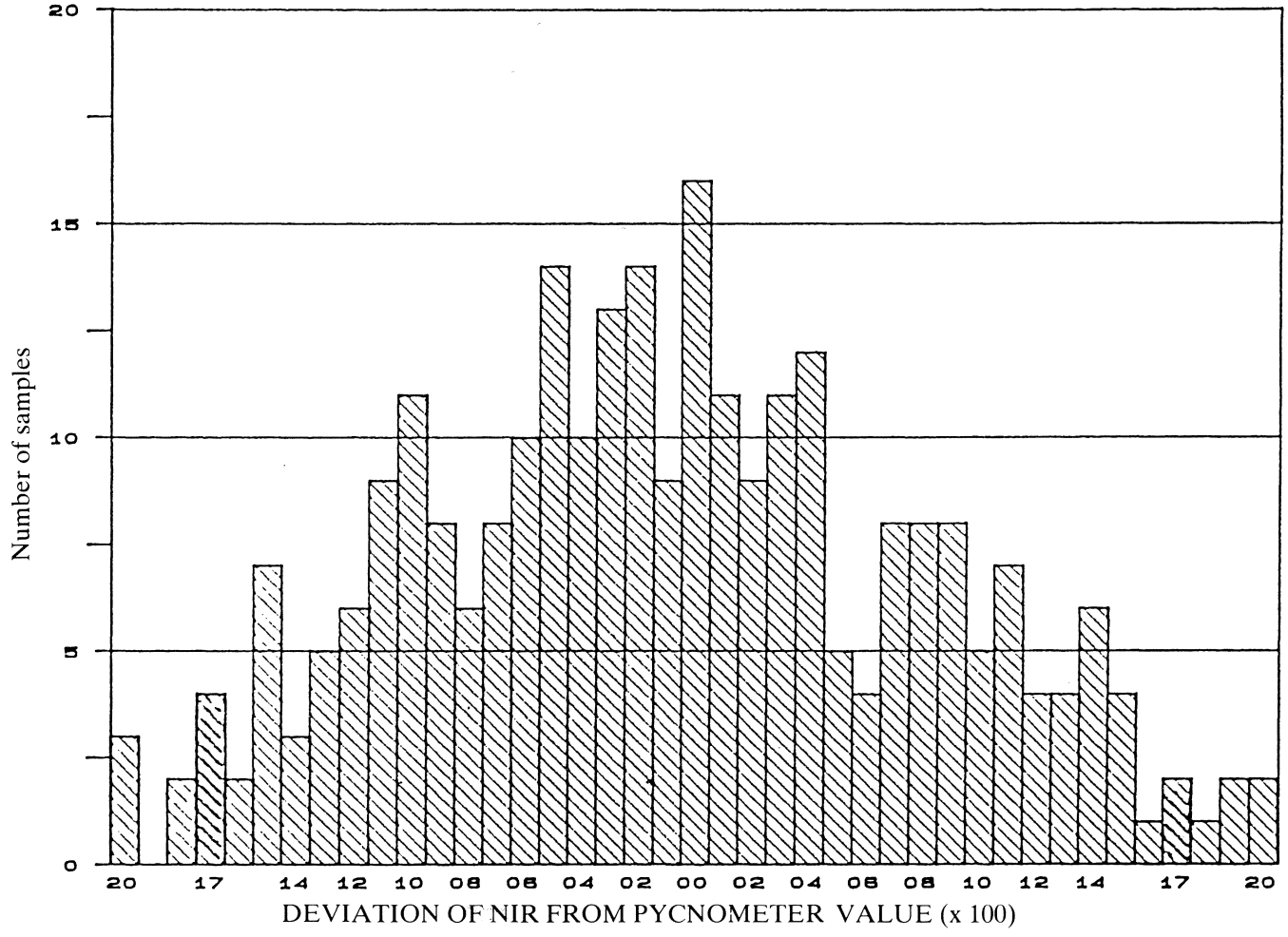

FIG. 3.
Comparison of NIR and pycnometer methods for determination of alcohol in wines.

Alcohol recovery by the NIR method

The suitability of the NIR method for determining alcohol in wines was confirmed by adding 1 volume percent of pure alcohol to six wines and measuring the recovery of the added alcohol.

RESULTS AND DISCUSSION

The deviation of the Near Infra-red alcohol as compared to that of the pycnometric method is shown in Figure 3. It can be seen that there is a normal distribution in the deviations obtained. Deviation in relation to the pycnometric method is as follows:

$\leqslant \pm 0{,}05$ Vol % – 45 % of the samples
$\leqslant \pm 0{,}10$ Vol % – 79 % of the samples
$\leqslant \pm 0{,}17$ Vol % – 89 % of the samples
$\leqslant \pm 0{,}20$ Vol % – 100 % of the samples

The effect of wine type on the NIR method as compared to the pycnometric method was minimal as can be seen from Figure 3 which was constructed from five different types of wine. It must be stated that each wine type is analysed within a certain alcohol concentration range. Data of the effect of the type of wine in each of these alcohol ranges is not shown.

During the initial trials run at the KWV Laboratory it was found that red wine colour had an effect on the NIR method. The alcohol readings obtained by NIR method were 0,3 volume percent higher than the analysis obtained by the pycnometric method. Due to the flexibility of the NIR method and apparatus used this problem was overcome by calibrating the NIR apparatus using a different channel just for red wine alcohol analysis. It can be seen in Figure 3 from the data obtained that colour had minimal effect on the alcohol analysis by the NIR method during this study, after the initial re-calibration of the Infra-Alyzer for colour. All the other colours mentioned earlier had an insignificant effect on the NIR method of alcohol analysis. Figure 3 also demonstrates that sugar content has little or no effect on the NIR method.

Although sugar content of the wines differed from low to very high concentration we could not note a difference in the comparisons between the two methods of alcohol determination. This specific data is not shown. Turbidity of the samples was judged visually. The two categories were turbid or clear. The distilling wines were used to study the effect of turbidity and little ($\leqslant 0{,}2$ volume %) or no effect was detected as can be seen in Figure 3.

As far as reproducibility is concerned the same wines measured at different times on different days by the Infra-Alyzer showed a variation in the measured alcohol concentration of less than 0,04 volume % (Table 1). Similar results have been attained by other laboratories using NIR (Anon., 1984).

The recovery tests carried out by adding one volume percent absolute alcohol to wines of known alcohol concentration showed good recoveries and minimal or no losses of alcohol when analysed by NIR (Table 2). As far as precision is concerned the standard deviation of the NIR method obtained in practice is $\leqslant 0{,}04\%$.

TABLE 1

Reproducibility of alcohol determination by NIR and pycnometric methods.

Wine	Pycnometer	Infra-Alyzer
	Alcohol concentration in vol. %	
1a	6,84	6,71
b	6,84	6,72
2a	7,63	7,71
b	7,79	7,81
3a	10,50	10,43
b	10,42	10,48
4a	11,59	11,52
b	11,59	11,46
5a	11,00	10,92
b	10,92	11,00
6a	12,02	12,02
b	11,85	12,05
7a	12,88	12,85
b	12,80	12,75
8a	13,85	13,82
b	13,76	13,85
9a	11,00	11,06
b	11,00	11,08
10a	11,93	12,01
b	12,02	11,98
11a	17,00	17,00
b	16,91	17,06
12a	18,88	18,98
b	18,98	18,94
	Average S = 0,06	Average S = 0,04

S = Standard deviation
a and b are duplicate analysis on the same sample

TABLE 2

Determination of alcohol with the Infra-Alyzer in six wines and recovery after an addition of one volume percent alcohol.

Wine	Original alcohol content of sample (a) vol. %	Alcohol determined after alcohol addition vol. %	% Recovery
1	6,72 ± 0,01	7,76	104
2	10,46 ± 0,04	11,48	102
3	10,96 ± 0,06	12,04	108
4	12,80 ± 0,07	13,83	103
5	11,07 ± 0,01	12,02	95
6	17,03 ± 0,04	17,99	96

(a) Average of 5 independent determinations ± standard deviation

CONCLUSION

It is evident from this study that the analysis of alcohol concentration in wines with the NIR method is accurate enough for routine laboratory analysis. The instruments are relatively expensive and savings due to better production control, usually result in the cost being covered within a year. No reagents are necessary for use with this apparatus. A large variety of substances can be analysed by this machine which makes it more cost effective. The most important aspect to consider in evaluating Near Infra-red technology is the calibration support that is available in the form of a computer from the supplier.

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