The near infrared paradigm: will 25 years of research/knowledge hinder near infrared's future?

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Introduction

We thought that this question could best be addressed by listening to the views of a randomly selected segment of the vast near infrared (NIR) community. Accordingly, a set of questions was devised and circulated to 100 NIR users by one of us (RDR). This list included "real-world" workers and "researchers". The survey achieved a 53% response, of which 54% came from researchers and 46% from users, for an approximately 50 : 50 split.

The questions were:

1. "In the majority of NIR applications, use of factor-based calibrations [e.g. partial least squares (PLS)] will provide more accurate prediction of unknown samples than traditional multiple linear regression (MLR) methods". True or false?
2. "In the majority of applications, the use of an experienced NIR analyst is highly desirable to derive the calibration and to provide long-term monitoring of instrument performance and accuracy". True or false?
3. "In order for an NIR instrument to measure accurately, the calibration must be performed using samples that have accurate laboratory methods". True or false?
4. Estimate how many NIR instruments *are currently* in use throughout the world.
5. Project how many instruments will be in use worldwide in the year 2000.
6. What do you think are the major current limitations of NIR instruments?

(Please list in order 1–4, 1 is most important)

- Difficulty in calibrating the instrument.
- NIR instruments are too expensive.
- NIR instruments have insufficient versatility.
- NIR instruments have insufficient sensitivity.
- Speed of instrument is too slow.
- Other.

7. Do you think that in the near future NIR instruments will become very simple to calibrate (e.g. only require one or two samples to perform an accurate calibration)?
8. Assuming that there is sizable funding available, what type of research should be performed to make NIR technology more useful to more people?

From Near Infrared Spectroscopy: The Future Waves

Results

This paper, the first of the conference proper, will summarize the response to the questions and also our reaction to the responses.

Question 1. In the majority of applications, use of factor-based calibration techniques (e.g. PLS) will provide more accurate prediction of unknown samples than traditional MLR methods. True or False?

About 55% of the responses were affirmative. Over 60% of the researchers considered that PLS would provide more accurate results than MLR, whereas the reverse was true for the users, most of whom believed that MLR would do as well or better than PLS-type calibrations. The approximately equal "Yes"/"No" answers indicated that opinions are certainly divided on this issue. One researcher summed up the situation (in our opinion)—this worker believed that in general the answer was "true", but that it depended on the sample set and assay. Bob and I agree that in most applications PLS and MLR give essentially the same predictions and reproducibility. Where one method prevails, it is usually by small differences in the correlation coefficient (r^2) and standard error of prediction (*SEP*).

Question 2. In the majority of applications, the use of an experienced NIR analyst is highly desirable to derive the calibration and to provide long-term monitoring of instrument performance and accuracy. True or False?

Over 80% of the responses were positive from both researchers and users, which indicates (i) that calibration and particularly the interpretation of the statistics is considered to be very important in NIR technology and (ii) that this is probably the single most important reason underlying the relatively slow acceptance of the technology. The need for separate calibrations for *everything* still acts as a deterrent to would-be users. Continuous monitoring is also a factor, but a point which is often overlooked is that reference methods also require monitoring and NIR instruments are in general more precise in their analysis than most reference methods.

Question 3. In order for a NIR instrument to measure accurately, the calibration must be performed using samples that have accurate laboratory methods. True or False?

Again, both groups were convinced that this is true. Bob did not agree with this, but here he and I (for once!) are not in true accord. For the first part of the question, "In order for a NIR instrument to measure accurately…" I agree with Bob 100%—NIR instruments themselves are quite capable of measuring accurately—but the laboratory which performs the tests to evaluate performance, whether immediately after calibration or over the long term, is likely to be the laboratory which did the analysis on the calibration set. If the results from this laboratory are not reliable the operators will most likely blame the instrument, rather than the laboratory. But some laboratory analyses are very expensive—so a compromise—the analysis on the samples should at least be of acceptable accuracy.

Question 4. Estimate how many instruments *are currently* in use throughout the world.

An interesting question. Perhaps someone was enterprising enough to call all of the instrument companies! The researchers reported an average figure of about 16,000, with a high standard deviation. The users came up with a figure of about 18,000, with a lower standard deviation. Bob made the point that, no matter which is closest, this would still represent only a very small fraction (about 2%) of the total number of spectrophotometric instruments of any type used in analytical chemistry, worldwide—and NIR has been around for well over 20 years!

Question 5. Project how many NIR instruments will be in use worldwide in the year 2000.

The researchers projected about 41,000 and the users about 58,000, rather a more optimistic approach. Both groups had large standard deviations, so, as in the case of Question 4, there was a wide diversity of opinion. The annual compounded growth rate was about 42%.

A healthy increase in the use of NIR will depend on the instrument manufacturers (with help from us users) unraveling the knots nestling in Question 6!

Question 6. What do you think are the major current limitations of NIR instruments?

a) Difficulty in calibrating
b) Instruments too expensive
c) Instruments have insufficient versatility
d) Instruments have insufficient sensitivity
e) Speed of testing too slow
f) Other (please specify).

Please list in order 1–4 (1 is most important);
Rank 1 = 10 points
Rank 2 = 5 points
Rank 3 = 3 points
and Rank 4 = 1 point.

Figure 1 illustrates the basic answers graphically. Calibration remains the most important limitation to the acceptance of the technology, followed fairly closely by the cost of the instruments. Many would-be users, who are convinced of the value of the technology, believe that the instrument manufacturers have an exaggerated opinion as to the value of their instruments! There were few complaints as to the speed of testing, versatility, sensitivity or physical size of the instruments.

As to the response to the "other" limitations of the technology, there was a wide diversity of answer. One point which recurred was the need for education in NIR technology. This is a need of long-standing and is not addressed by the elegant papers on chemometrics which have appeared.

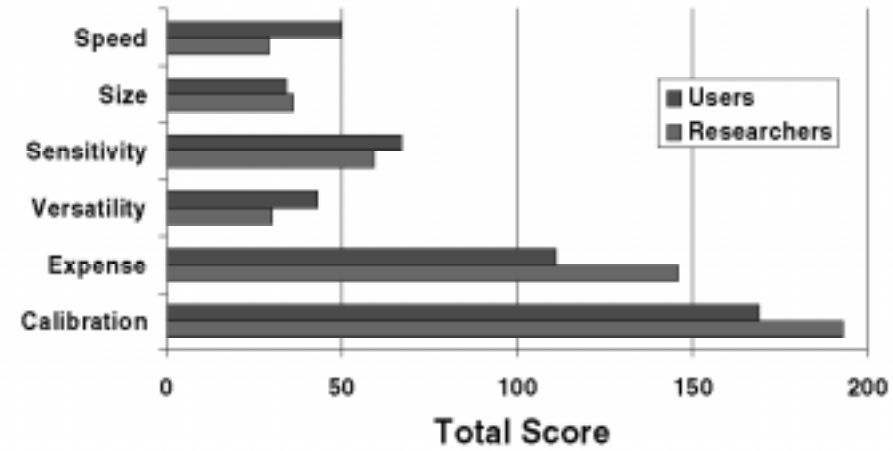

Figure 1. Graphic representation of answers to Question 6—What do you think are the major current limitations of NIR instruments?

There is a lack of understanding of the interpretation of simple statistics, even such simple ones as the r^2 and *SEP*. Some users believe (probably from bitter experience) that NIR companies tend to over-sell the virtues of their instruments. Others complained about the need for bias adjustments. Another source of concern is that the technology is not regarded as official by many authorities, who insist on approved methods of analysis.

Bob's conclusion, with which I heartily concur, is that we *must* simplify the calibration process, and make calibrations more robust. Modem control and networking would improve this. To his sage comments may I add that education in NIR technology is desperately needed. Despite the efforts of people like Dr Dave Wetzel, there is still no university which offers formal education in NIR technology. Another area which has to be improved is the stability of the instruments. This is related to factors such as temperature and humidity and to the complexity of the material being analyzed. In general, agricultural products, including materials of plant and animal origin, cause more concern than most industrial materials. Finally [my own (PW) opinion] the manuals issued by instrument manufacturers are often not clear in their instructions as to how to use their instruments. Ideally the operator should be able to calibrate and operate the instrument without any further assistance.

No one mentioned improvements in software, or reference analysis, which is, and always will be, a fundamental in NIR technology (with sometimes unfortunate overtones!). While several answers drew attention to the need for education, no one mentioned funding of NIR research, most of which is still carried out by instrument companies and government laboratories and not in universities (where all of that untapped talent is waiting!).

Question 7. Do you think that in the near future NIR instruments will become very simple to calibrate (e.g. only require one or two samples to perform an accurate calibration)?

Here the consensus was that this was not likely to happen, with over 70% of users and over 60% of researchers being of the opinion that this was not likely to happen in the near future. Ironically, an instrument *has* been available that could actually be calibrated with two samples. This was the Neotec Model 101 which used a quotient version of the first derivative of the log $1/R$ signal and could be successfully calibrated with two samples. Instruments are available at present which can be calibrated with a small number of samples (12–20) for many industrial applications, e.g. in the pharmaceutical and other chemical industries. More complex materials, such as grains and derived products, require many more and ideally, for the most robust calibrations, at least 200. Whole-grain analyzers require even more. My own view (PW) is that instruments *will* become easier to calibrate, simply because they *must*! This will greatly increase the acceptability of NIR technology. Also, there will be more factory calibration so that instruments will be able to be purchased fully calibrated. This is already in place for some applicaions.

Question 8. Assuming that there is sizable funding available, what type of research should be performed to make NIR technology more useful to more people?

This question predictably brought forth a plethora of ideas! Most popular were demands for research on chemometrics, calibration transfer and software (amazing how many people still believe that software will answer everything), networking, non-invasive clinical research, NIR imaging, "miniaturization", discriminant analysis, spectral calibration methods and fiber optics research were all mentioned. Two responses each favored research on instrument stability and efforts to "legalize" NIR (e.g. by acquiring certification under ISO 9000 for certain applications). I (PW) believe that research into determination of what causes outliers (as distinct from detecting them) would be well rewarded. The differences between two samples of the same material which

predict with small and large errors is probably quite subtle but they have to be caused by *something*. Samples which modern software identifies as outliers on the basis of spectral characteristics often predict with smaller errors than other samples in the same sample sets which are not identified as outliers. It would be very useful to know why.

Conclusions

So—will 25 years of research/knowledge hinder NIR's future?

Of course not—and the next 25 years will make it even better!