Determination of Dimethylamine Content in Dimethylhydrazine Based on Partial Least Squares Method

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Abstract. Based on partial least squares, the dimethyl monomethyme content was measured. The original spectroscopy, smooth, first-order differential, second-order differential, multi-scattered correction, standard regular conversion, the main cause of the child were effectively used. The results show that the model performance of 6 main factors established separately, the correction set coefficient and verification set coefficient of correction set is 0.9999 and 0.9991, respectively, and the correlation set prediction standard deviation is 0.00356, respectively. And 0.0178. This optimal model is used to measure the blind sample of modeling. As a result, the deviation of the measurement results is between -0.028% ~ 0.012%, the average deviation is from 0.014%, the standard deviation is 0.014%, and the relative standard deviation is 2.9%. The result is satisfactory.

1. Introduction
Dimethylhydrazine is a strong reducing agent that has a wide range of application prospects in various new material synthesis. Dimethylhydrazine content is an important parameter affecting the performance of bias ferrogen, thus determining dimethylhydrazine content is very important. Near infrared spectroscopy is a rapid, non-destructive, non-contact green analysis technology that has developed in recent years, has been successfully applied to crops, pharmaceutical, tobacco and other fields. In this paper, it is combined with the non-infrared spectroscopy to the partial least squares, and the determination of dimethylamine content is applied to the determination of dimethylhydrazide content, and the rapid, lossless, non-contact assay of dimethylamine content, and the result is satisfactory.

2. Experiment
2.1. Experimental Samples and Standard Preparation
Dimethylhydrazine: commercially available industrial grade. Dimethylamine solution: commercial analysis is pure. Transparent glass bottles with polyethylene cover, Shaoxing Oerces Material Technology Co., Ltd. The dimethylamine content was formulated in a weight method in a weight method of 0.202%, 0.423%, 0.837%, 1.052%, respectively. Each sample was subjected to 5 bottles of 5 independent samples, respectively, to maximize Reduce the effects of different reagents in the bottom of the test results. Each concentration of 5 bottles of samples were randomly selected from modeling as a verification sample. In addition, a concentration of 0.482% of samples were dispensed into 10 glass bottles and did not participate in modeling as blind samples. Evaluation of the optimal model.
2.2. Experimental Equipment and Experimental Methods
Fourier transform Near infrared spectrometer is preheated for 1 hour, the sample is processed nearly infrared transmission scan. Scan resolution is 8cm⁻¹, 32 scan, scanning range 4000-10000cm⁻¹. The data is processed using TQ Analyzer 8.0, and the processing method includes the minimum multiplier (PLS), smooth, first-order differential (1st), second-order differential (2nd), multi-scattered correction (MSC), standard regular transform (SNV); The calculation parameters include correction set coefficient (RC), correction set prediction standard deviation (RMSEC), verification set correlation coefficient (RP), and verification set prediction standard deviation (RMSEP).

3. Results and Discussion
3.1. Modeling Parameter Optimization
There is a large amount of chemical structural information in the near infrared spectrum, but there is a large number of noise information, the optimization of the model is an important guarantee for good prediction effects, which is to maximize the extraction of the extraction from a large amount of information. Optimized methods include differential, smooth and correction, and the parameters of the evaluation are mainly related coefficients RC and RP and predictive standards RMSEC and RMSEP. The closer the correlation coefficient, the smaller the prediction standard, the more parameters are established. The prediction results are shown in Table 1. As can be seen from Table 1, the model performance of the first-order differential establishment is preferably, RC and RP are 0.9999 and 0.9991, respectively, and RMSEC and RMSEP are 0.0036 and 0.0178, respectively. This shows that the first-order differential extracts more effective information. Other various types of treatment methods include smoothing and correction without more extraction effective information, or although more efficient information, more noise information is introduced into the model, resulting in the prediction effect of the model, which does not rise without rising, so It is said that the optimized modeling parameters are used in this study.

| Pre-treatment method   | RC       | RMSEC /% | RP       | RMSEP /% |
|------------------------|----------|----------|----------|----------|
| Raw                    | 0.9989   | 0.0157   | 0.9987   | 0.0187   |
| 1st                    | 0.9999   | 0.0036   | 0.9991   | 0.0178   |
| 2nd                    | 0.99947  | 0.0344   | 0.9966   | 0.0324   |
| 1st+ smoothing         | 0.9996   | 0.0093   | 0.9991   | 0.0176   |
| 2nd+ smoothing         | 0.9958   | 0.0306   | 0.9943   | 0.0386   |
| SNV                    | 0.9988   | 0.0161   | 0.9989   | 0.0158   |
| MSC                    | 0.9988   | 0.0161   | 0.9989   | 0.0161   |
| SNV+ smoothing +1st    | 0.9949   | 0.0339   | 0.9944   | 0.0415   |
| MSC+ smoothing +1st    | 0.9951   | 0.0330   | 0.9945   | 0.0413   |
| SNV+ smoothing +2nd    | 0.9924   | 0.0413   | 0.9925   | 0.0456   |

3.2. Main Factor
The number of children is one of the most important indicators that affect the performance of the model. Table 2 shows the variations of the correlation coefficients RC and RP and predictive standards RMSEC and RMSEP when subduction of different main factors. It can be seen that before the number of primary agents reaches 6, the RC and RP gradually increases to 1, and RMSec and RMSEP gradually decrease. When the number of sub-genes exceeds 6, the RC continues to be compared with RMSEC, and the value of RP and RMSEP is basically unchanged. Considering that the probability of introducing the noise information in the number of the probability of continuing to improve the probability of the model is, it will affect the model applicability, and it is said that for this study, the best primary sub number is selected to 6.
Table 2. Predictive effect of different main factors

| Main factor | Rc  | RMSEC /% | Rp  | RMSEP /% |
|-------------|-----|----------|-----|----------|
| 1           | 0.9787 | 0.0687   | 0.9832 | 0.0618   |
| 4           | 0.9989 | 0.00156  | 0.9993 | 0.0188   |
| 5           | 0.9998 | 0.0069   | 0.9990 | 0.0180   |
| 6           | 0.9999 | 0.0036   | 0.9991 | 0.0178   |
| 7           | 0.9999 | 0.0016   | 0.9992 | 0.0168   |
| 8           | 0.9999 | 0.0011   | 0.9991 | 0.0169   |
| 10          | 0.9999 | 0.0006   | 0.9992 | 0.0167   |
| 20          | 0.9999 | 0.0002   | 0.9992 | 0.0168   |

3.3. Abnormal Value Test

Use the Marswa distance to determine whether there is an abnormal value in the original spectrum, as shown in Figure 1. It can be seen from the figure that all the distance values of all the spectra are less than 1.623, indicating that all spectra is not an exception value, which proves that all spectrum is valid.

3.4. Precision and Accuracy

The use of the best model separately tests the blind sample that does not participate in modeling, calculates the deviation, mean deviation, standard deviation, and relative standard deviation (RSD) of the measurement results (RSD), and the results are shown in Table 3. From Table 3, 10 measurement results can be seen between -0.028% and 0.012%, the average deviation is from 0.014%, the standard deviation is 0.014%, and the relative standard deviation is 2.9%, and the result is satisfactory.
Table 3. Precision and accuracy

|          | 1#  | 2#  | 3#  | 4#  | 5#  | 6#  | 7#  | 8#  | 9#  | 10# |
|----------|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|
| Theoretical value /% |     |     |     |     |     |     |     |     |     | 0.482 |
| Measured /% | 0.473 | 0.469 | 0.492 | 0.456 | 0.461 | 0.496 | 0.475 | 0.483 | 0.467 | 0.459 |
| Measured deviation /% | 0.011 | 0.015 | 0.008 | 0.028 | 0.023 | 0.012 | 0.009 | 0.001 | 0.017 | 0.025 |
| Measured average /% |     |     |     |     |     |     |     |     |     | 0.473 |
| Average deviation /% | -0.011 |     |     |     |     |     |     |     |     |     |
| Standard deviation /% | 0.014 |     |     |     |     |     |     |     |     |     |
| RSD /% | 2.9 |     |     |     |     |     |     |     |     |     |

4. Conclusion

This paper has achieved rapid, lossless, non-contact assay in dimethylamine content in dimethylhydrazine in combination with near infrared spectroscopy and bias. The parameter optimization process demonstrates that the first order differential has the highest signal-to-noise ratio, smooth, differential, multi-scatter correction, standard regular conversion, etc., but will reduce the performance performance. The best performance optimal performance through first-order differential binding 6 main factors, this optimal model is satisfactory to the precision and accuracy test results of unknown sample tests.

5. References

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