

***Application of NIR fingerprint
technique in quality control of feed
protein ingredients and feed additives***

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What we talk in this report

- The problems of feed products
- The introduction of NIR fingerprint technology
- Application of NIR fingerprint technique in quality control of feed protein materials
- Application of NIR fingerprint technique in quality control of feed additives



The problems in feed products

More adulterate
Ingredients appear

More difficult
to check

Involve more
products



The problems in feed products

- More and more adulterate ingredients appear in feed products
 - *good protein sources*
 - *expensive additive products*
- They are becoming potential hazards in animal feed products, and endanger the health of animal and human!!!



What we need?

No matter what kind of adulterate ingredients added, it can be recognized at once time

Simple, quick and accuracy

Easy to use and little man-made error

NIR Fingerprint technology



NIR fingerprint technology

- Fingerprint-based identification is the oldest method which has been successfully used in numerous applications.
- Near infrared spectrum mainly reflects chemical bonding information, it is piled up by each component's absorption of compound.
- Every compound is known to have unique, immutable NIR fingerprints.



NIR fingerprint technology

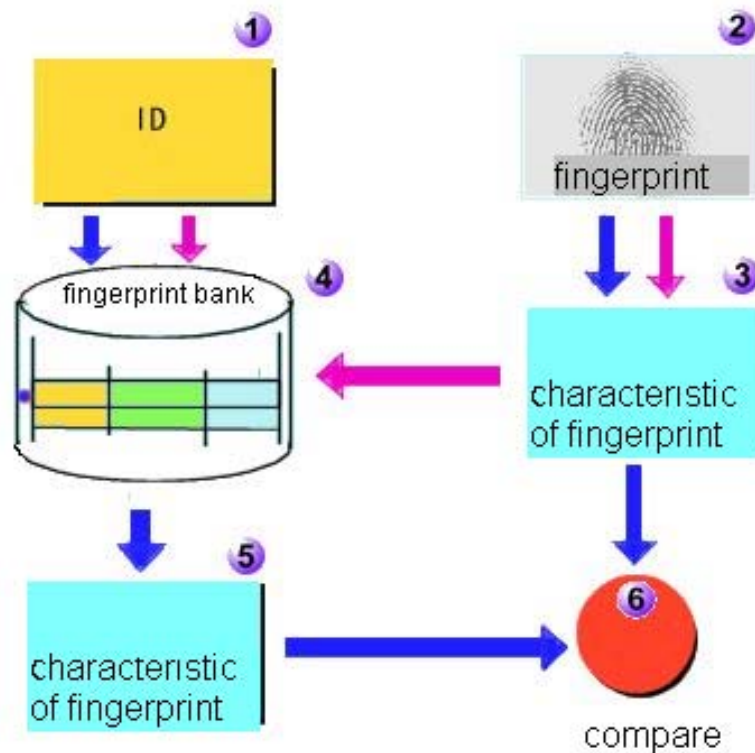
- NIR fingerprint is comprehensive, integrated information of product.
- Each product has its specific spectrum. If only the product has stable quality, its spectrum is steady.
- Feed products
 - ✓ unique, characteristic and repeatable fingerprint spectra
 - ✓ “the uniqueness of groups”——the standard fingerprint characteristic.



Fingerprint identification

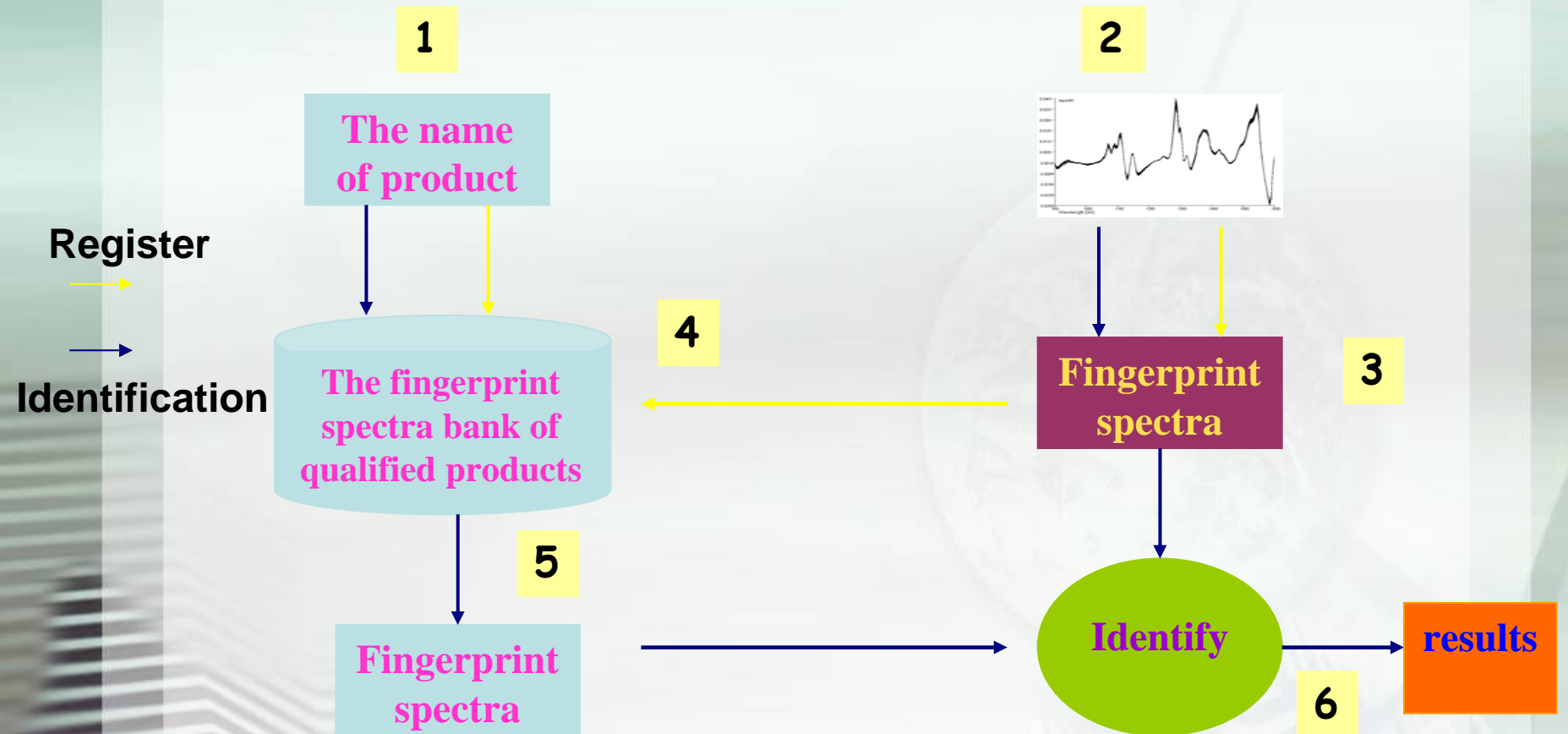
- 1 input ID
- 2 scan fingerprint
- 3 abstract characteristic of fingerprint
- 4 save ID and characteristic of fingerprint
- 5 find the correspondence characteristic fingerprint
- 6 compare the characteristics of two fingerprints

→ register
→ identification



one to one correspondence register procedure of fingerprint

NIR fingerprint identification



- 1、 Input the name of product
- 2、 Scan the spectrum of sample
- 3、 Abstract the fingerprint characters

- 4、 Create fingerprint spectra bank —in this bank fingerprint spectrum and product is one to one correspondence
- 5、 Find the correspondence fingerprint spectra
- 6、 Identify the product according to the compare of fingerprint spectrum or the analysis result of identify model



The features

- The research takes the study of qualified products as the core features.
- Different from traditional method, which try to find out the adulterated ingredient in the sample, our research care about if the sample is corresponding with the qualified product.
- Simple, quick, and environmental friendly



Application

- Application of NIR fingerprint technique in quality control of feed protein materials ——take soymeal as example
- Application of NIR fingerprint technique in quality control of feed additives —— take choline chloride as example



Test Samples

Soybean meal—— from four well-known domestic soymeal manufacturers, almost 2000 soybean meal are analyzed in this research.

Choline chloride samples——from additive manufacturers, quality inspection departments and feed plants, about 550 samples

Check products ——whose qualities are close to the qualified products as check samples

- ✓ *Soymeal check samples: prepared specifically for this research*
- ✓ *Choline chloride check samples: from the feed plants or from the government market detection*



Instrument and Equipment

- Near-infrared analyzer:

DA7200 diode array near-infrared spectrometer Perten

- Software: *Unscrambler V9.8 CAMO Software AS,*
GRAMS / AI V7.02 Thermo Galactic

- Confirm and support equipment:

ion chromatography S135 Sykam GmbH

Rapid N CUBE Elementar

Amino Acid Analyzer S433D of Sykam GmbH



Confirm and proof the authenticity

Product — *the most important concept apart from standard product definition, specialize our product concept according to*

— *cluster results of spectra*

— *the practical application*

To ensure all the products in same database have identical standard fingerprint characteristics.



Test Methods

- Preprocess methods and characteristic wavelengths of the qualified spectra were chosen based on the qualified database.
- Fingerprint identification models of qualified products were established based on spectra analyzing.
- Criteria of choosing the discriminate model

difference between qualified samples MD and check samples MD



Application of NIR fingerprint technique in quality control of feed protein materials



Confirm and proof of soymeal samples

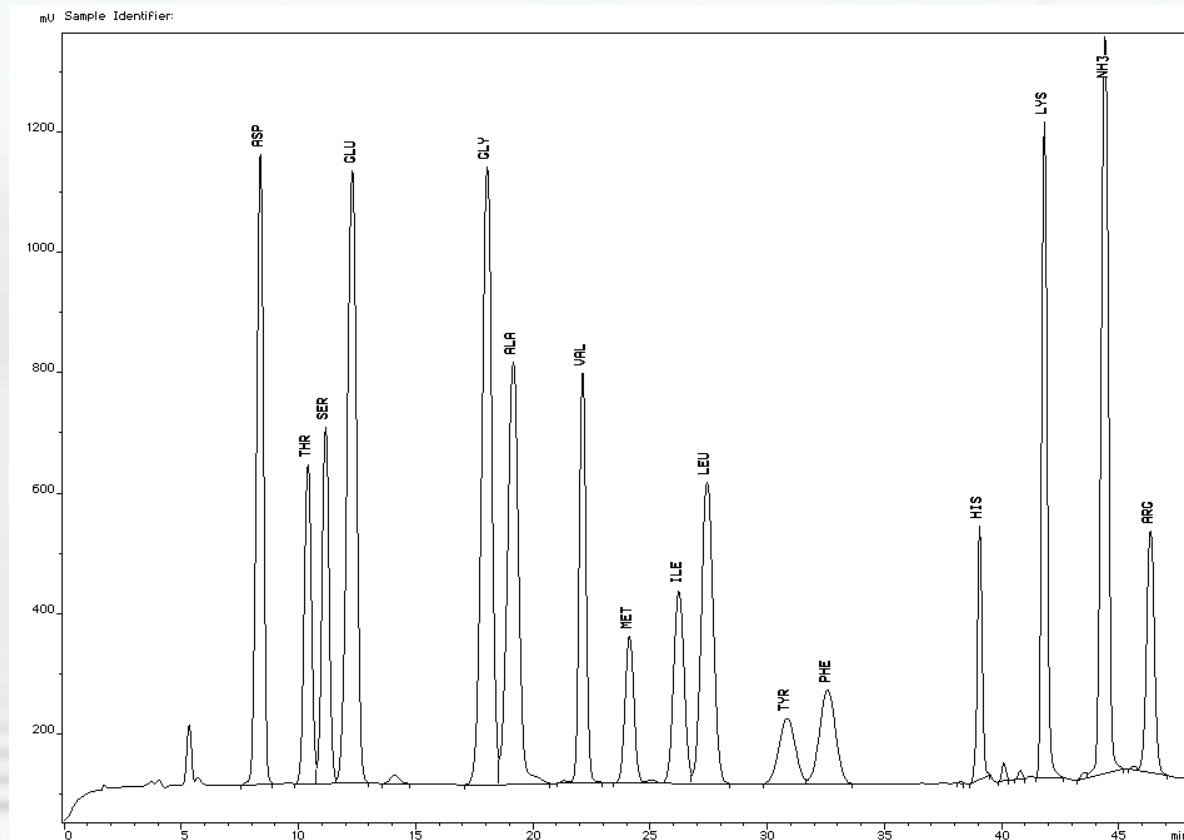


Figure 1 Amino acid analyzing results of soymeal

Classification of soymeal samples

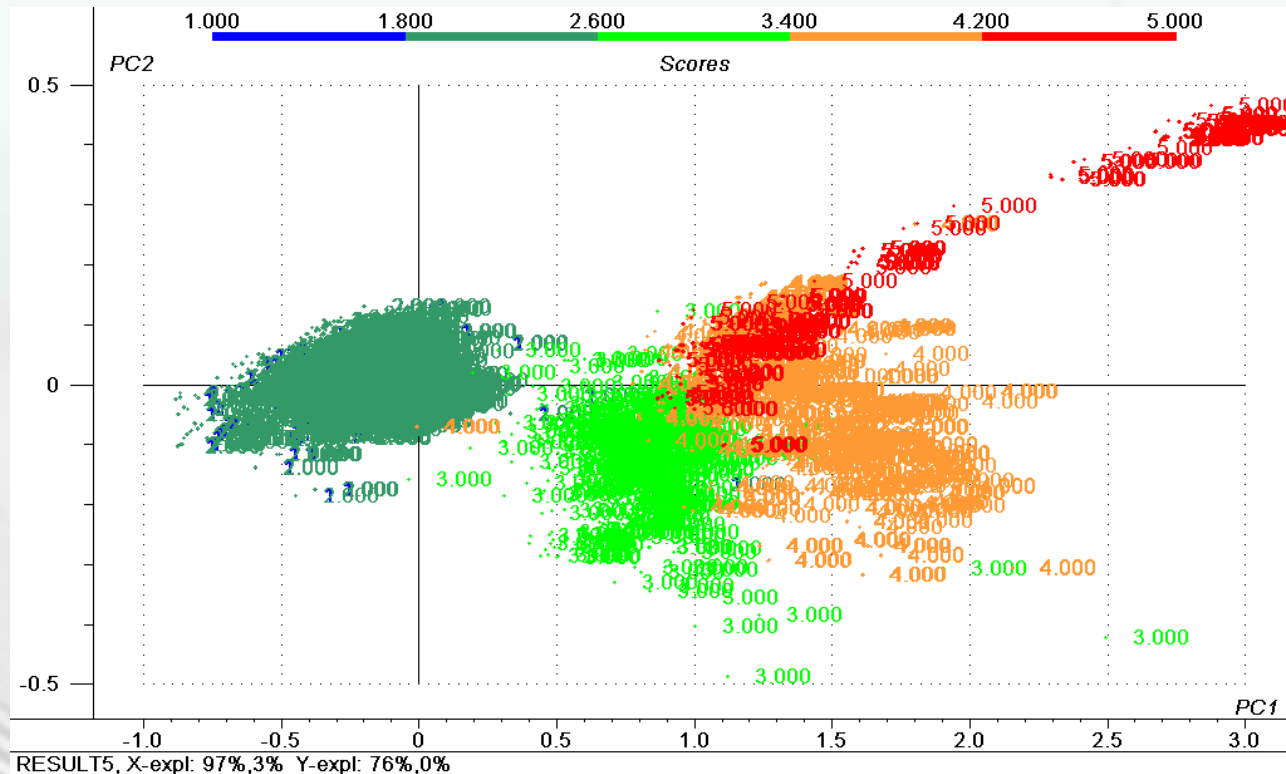


Figure 2 the score cluster result of different soymeal



Preprocess for soymeal samples

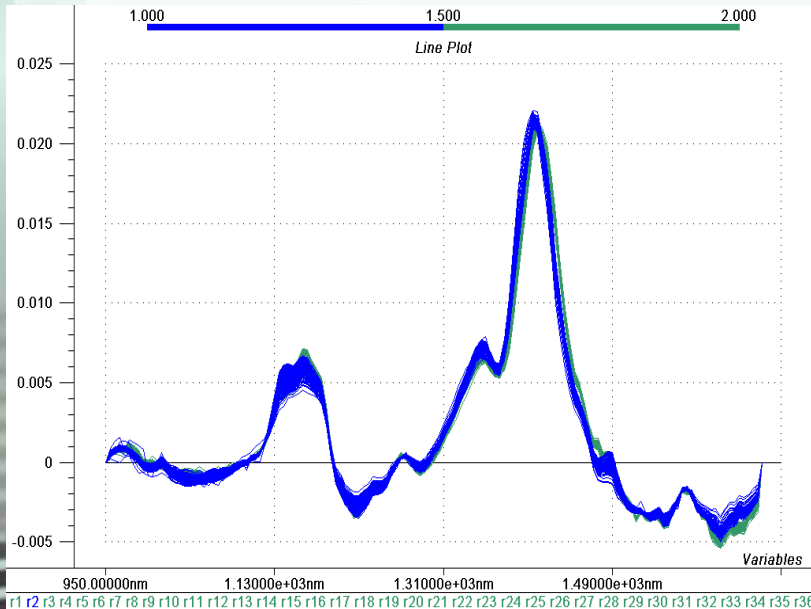


Figure 3 the spectra of soymeal after preprocess

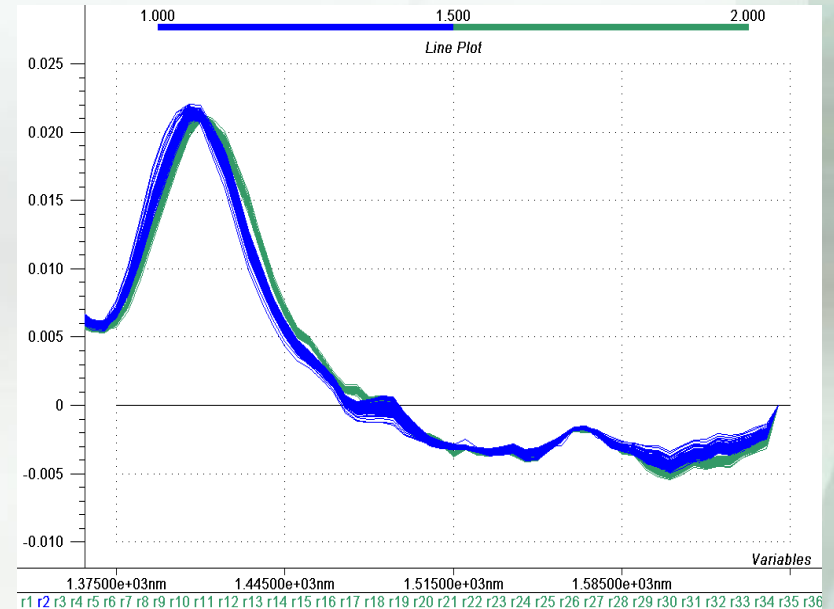


Figure 4 the wavelength which has distinct difference

Fingerprint wavelength selection

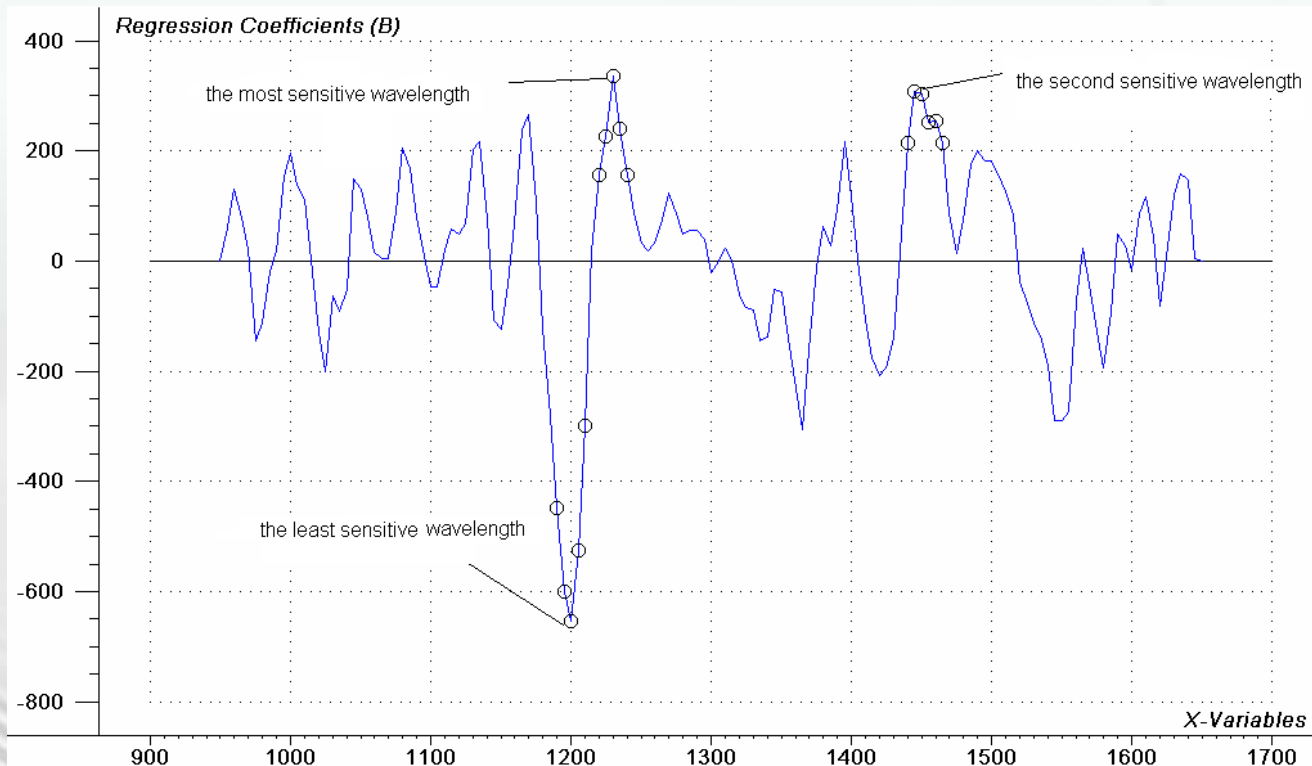


Figure 5 the regression coefficients of soymeal protein and absorption of different wavelength

Criterion

- The discriminate model:
 - preprocess spectra
 - fingerprint wavelength selection
- **Unqualified samples:**

MD is more than maximum MD of the qualified products adding 3 times MD standard deviation of calibration sets
- **Suspicious sample:**

MD is more than the maximum MD adding 2 times MD SD of calibration sets, and less than unqualified criterion.



NIR fingerprint identification results

	Mean of MD	Criterion of unqualified samples is its MD more than 2.2777			rate
Calibration sample	0.9537				
Validation sample	0.8745				
Soymeal made from domestic soybean, 20 samples	58.97	78.49	15.78		100%
Soymeal with 3% other plant meal, 5	2.719	3.791	0.532		100%
Soymeal with 3% meat meal, 5 samples	2.848	3.982	0.443		100%
Soymeal with 0.5% urea, 5 samples	5.024	5.176	0.282		100%
Soymeal with 0.5% melamine, 5 samples	10.85	11.06	0.238		100%



NIR Fingerprint identification results

- Distinguish the soymeal samples which made from different soybean or made by different processing
- Identify unqualified samples which have the protein from other sources
- Recognize unqualified samples which have non-protein nitrogen content



Application of NIR fingerprint technique in quality control of feed additives



Confirm and proof the authenticity

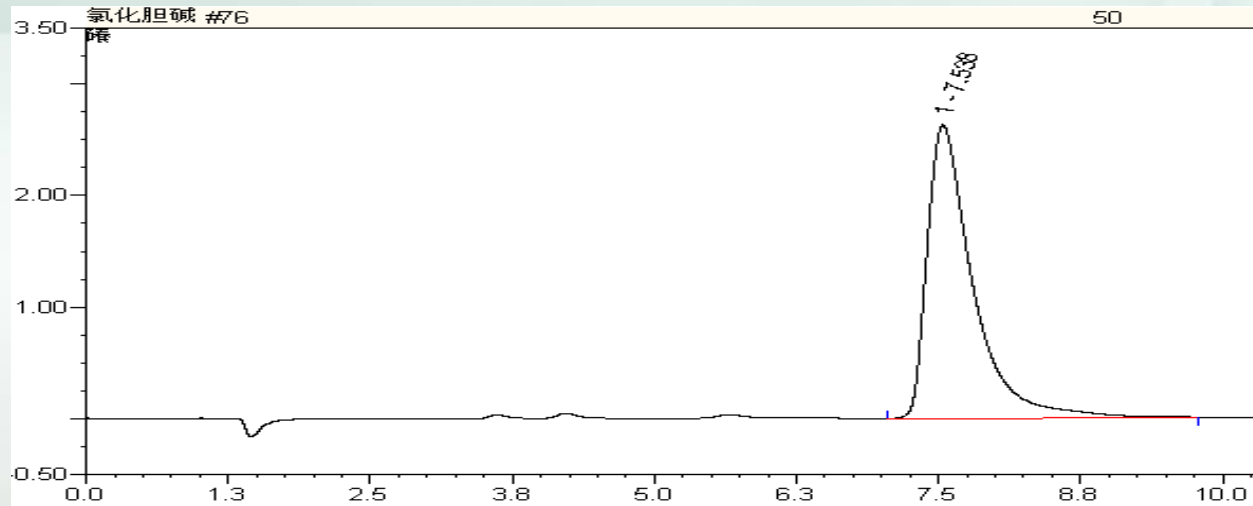


Figure 6 the IC result of 50% standard choline chloride samples

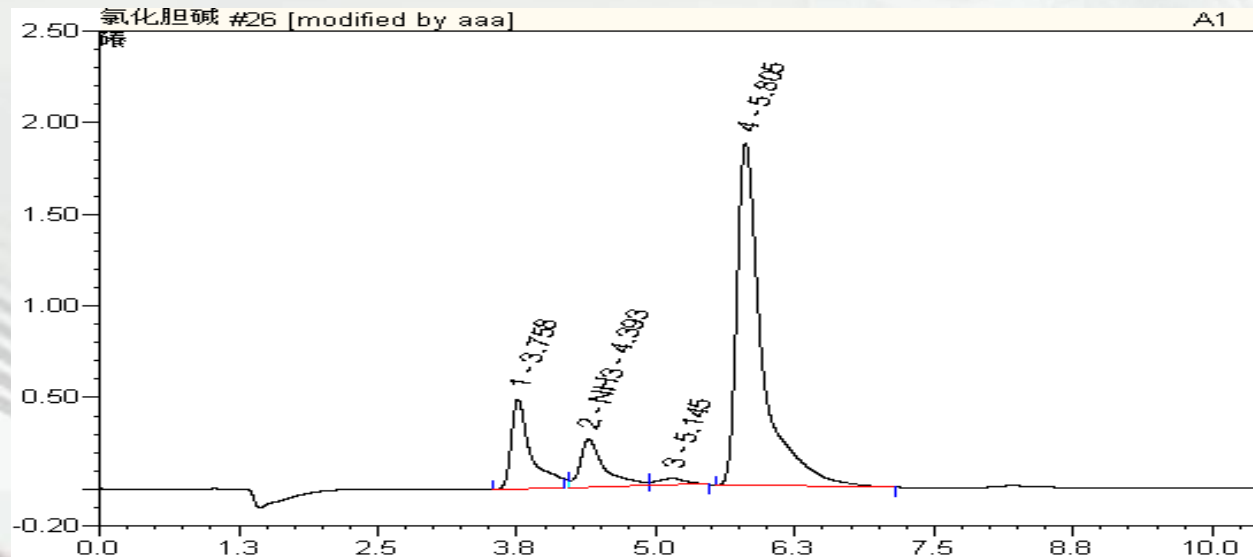


Figure 7 the IC result of 50% fake choline chloride samples



Classification of choline chloride

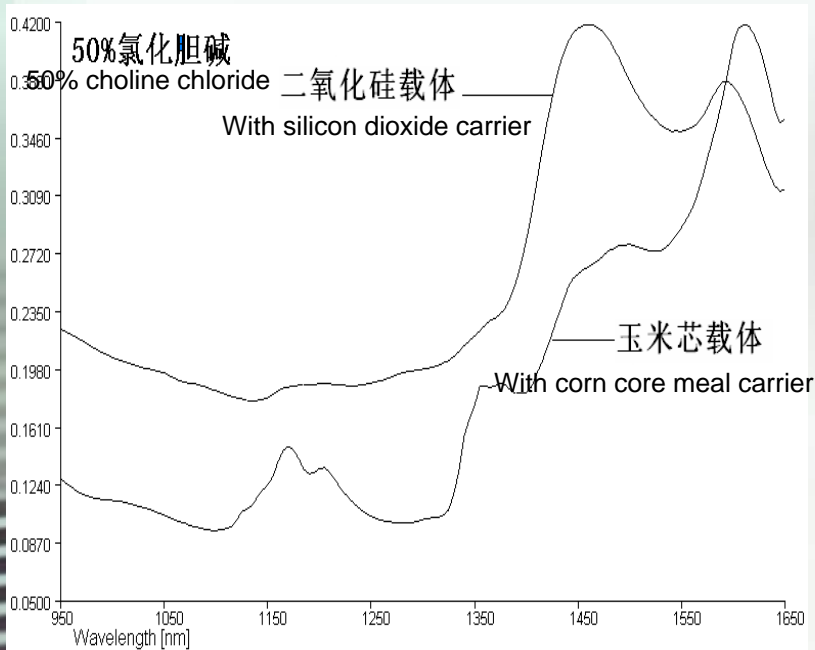


Figure 8 the NIR spectra of 50% choline chloride with different carrier

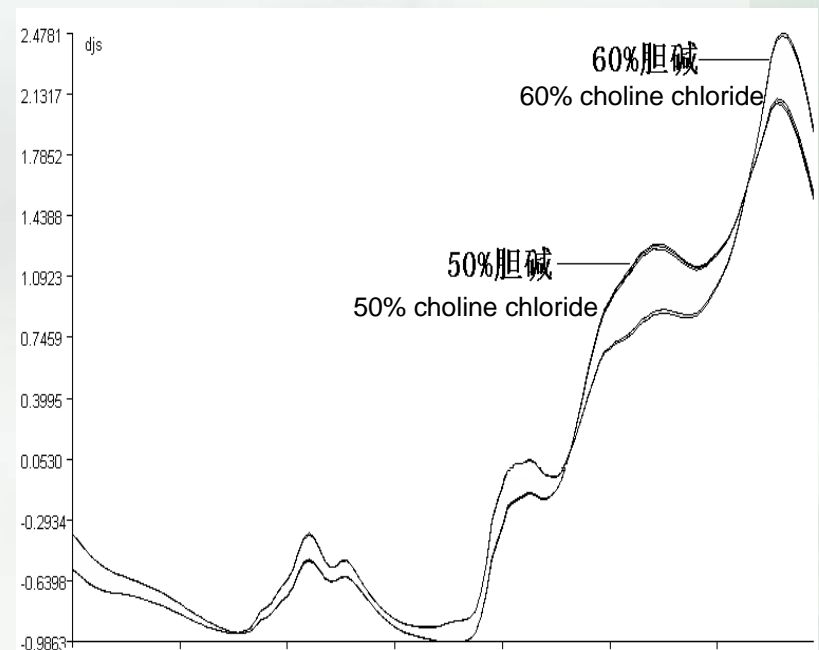


Figure 9 the NIR spectra of 50% and 60% choline chloride samples



Classification of choline chloride

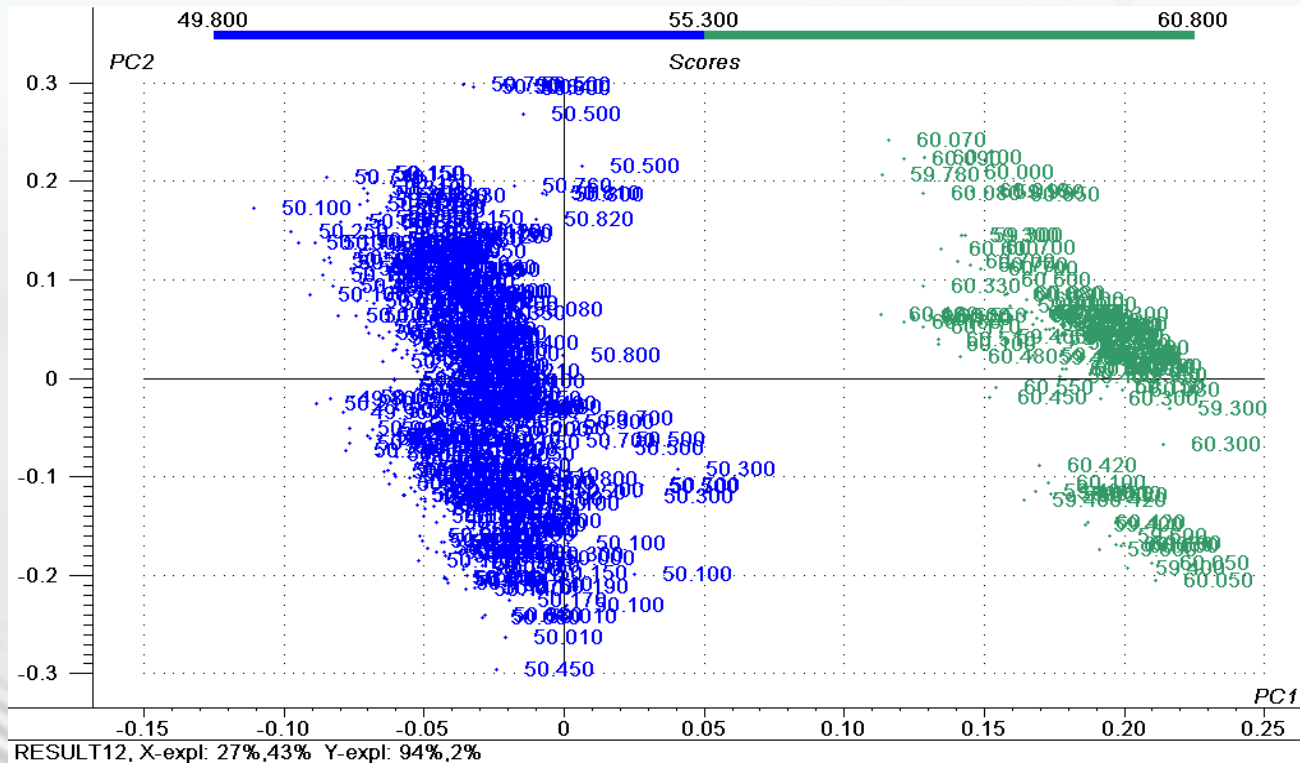


Figure 10 the score cluster result of different content choline chloride

Preprocess for choline chloride

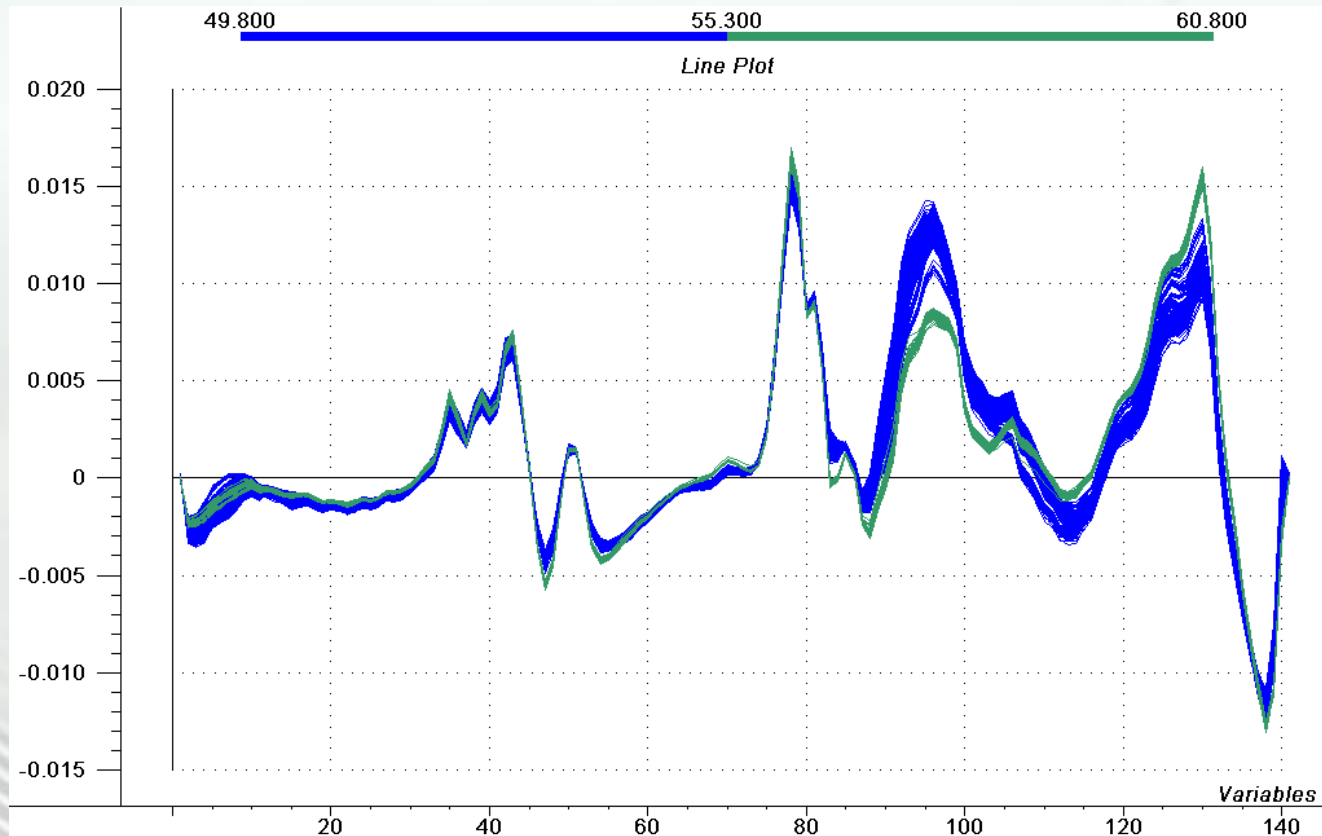


Figure 11 the spectra of choline chloride samples after preprocess

Fingerprint wavelength selection

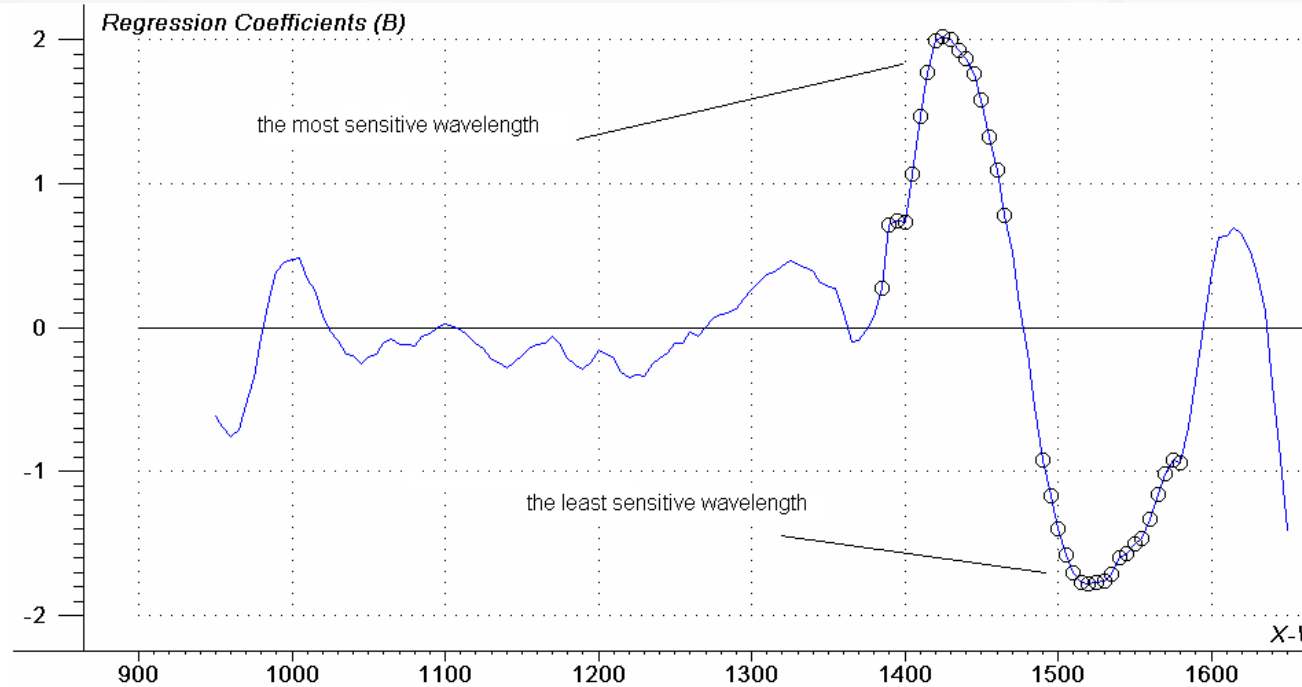


Figure 12 the regression coefficients of choline chloride content and absorption of different wavelength

NIR Fingerprint identification results

	Mean of MD	Max of MD	SD of MD	Accuracy rate
Calibration sample	0. 5743	1. 069	0. 3374	100%
Validation sample	0. 4796	0. 976	0. 1521	100%
Unqualified sample, 20	2. 631	3. 890	0. 5284	95%
Fake sample, 10	5685	14759	3870	100%



NIR Fingerprint identification results

- Only one suspicious sample:
choline chloride content is 48.45%——very close to the qualified samples, Mahalanobis distance is 2.011
- Significant discriminate effect of choline chloride samples



Conclusions

Soybean meal samples:

non-protein nitrogen

100% correct

cheap protein from other origins

100% correct

Choline chloride samples:

deficient samples

95% correct

adulterate samples

100% correct



Conclusions

NIR fingerprint identification technology is approved to be an objective and accurate method for discriminating many feed ingredients in common use.

Rapid and accurate!!!



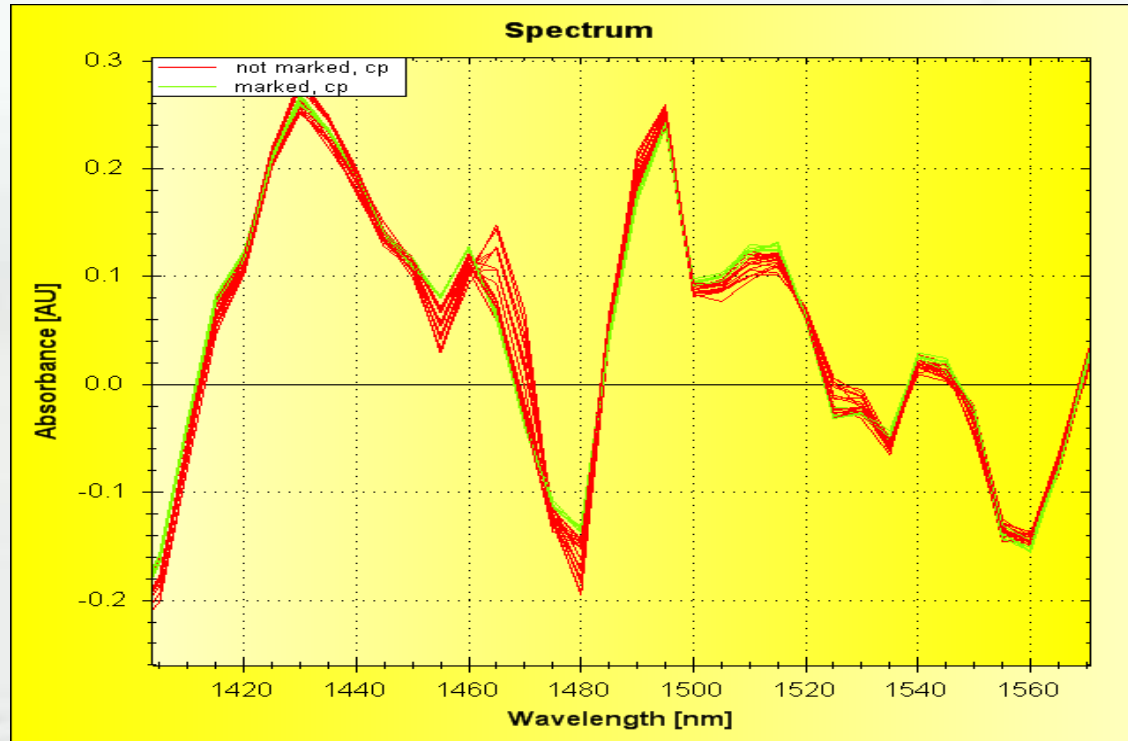
Other examples

1. Corn meal
2. Betain
3. Vitamin
4. Amino acid
5. Organic mineral
6. Organic arsenic



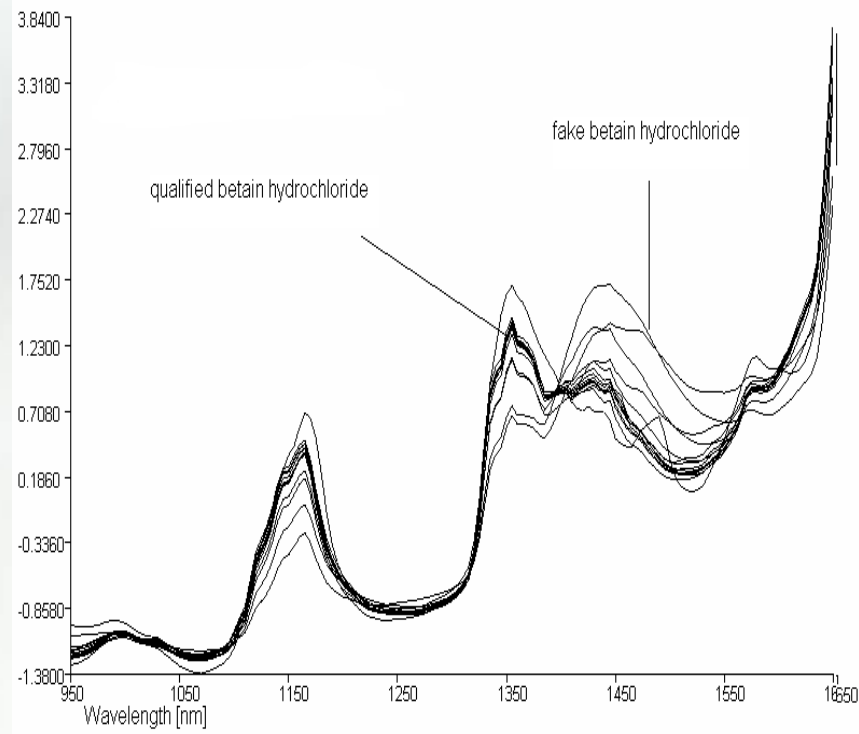
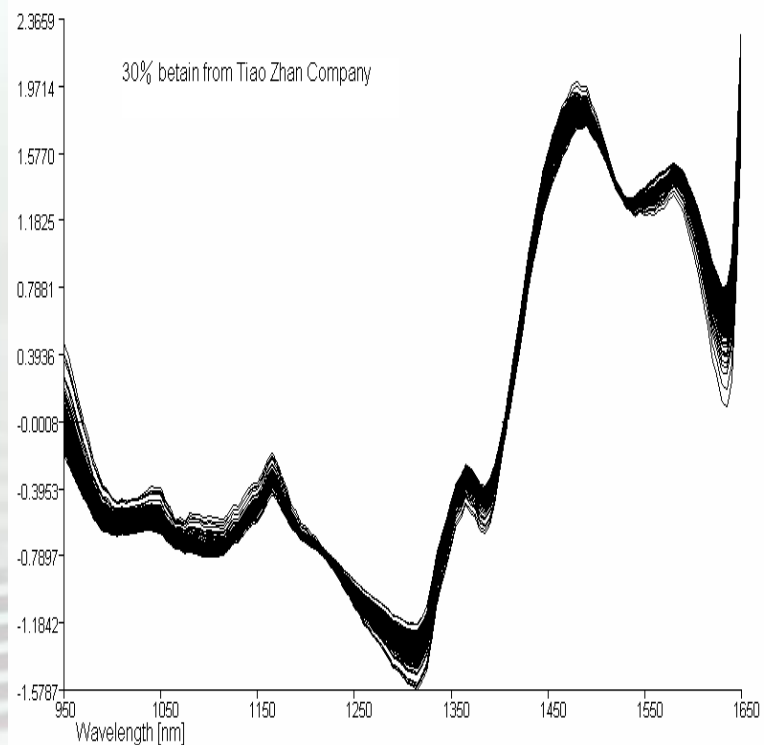
Example 1

Corn protein meal



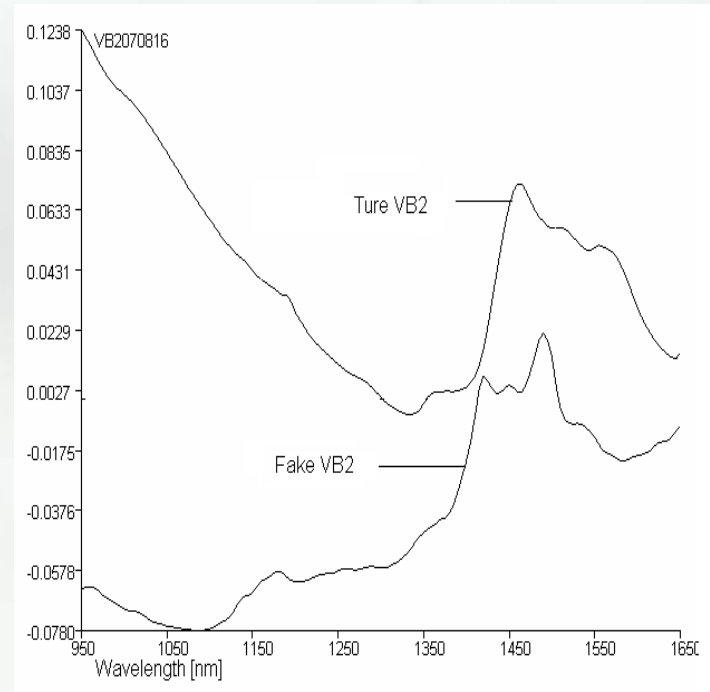
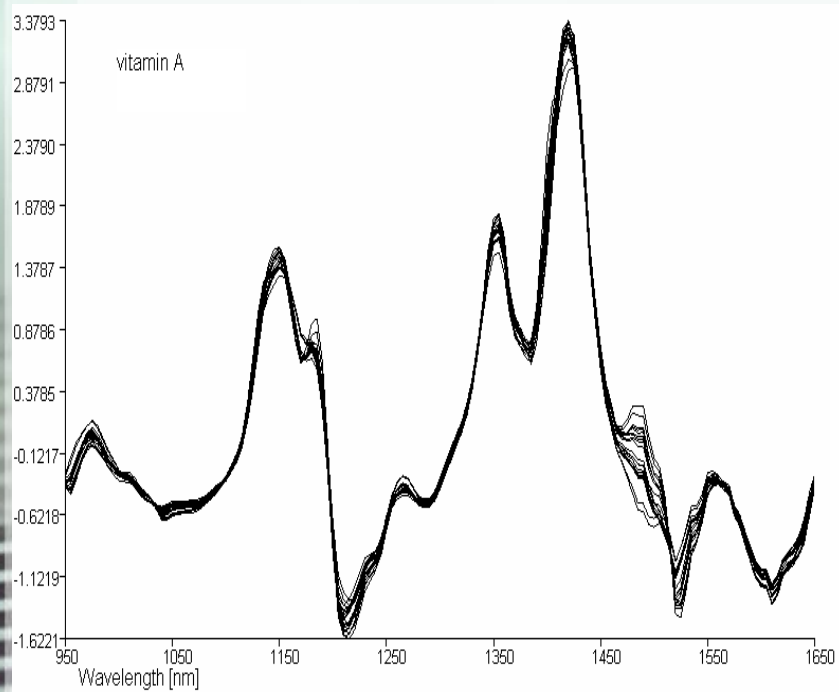
Example 2

Betaine



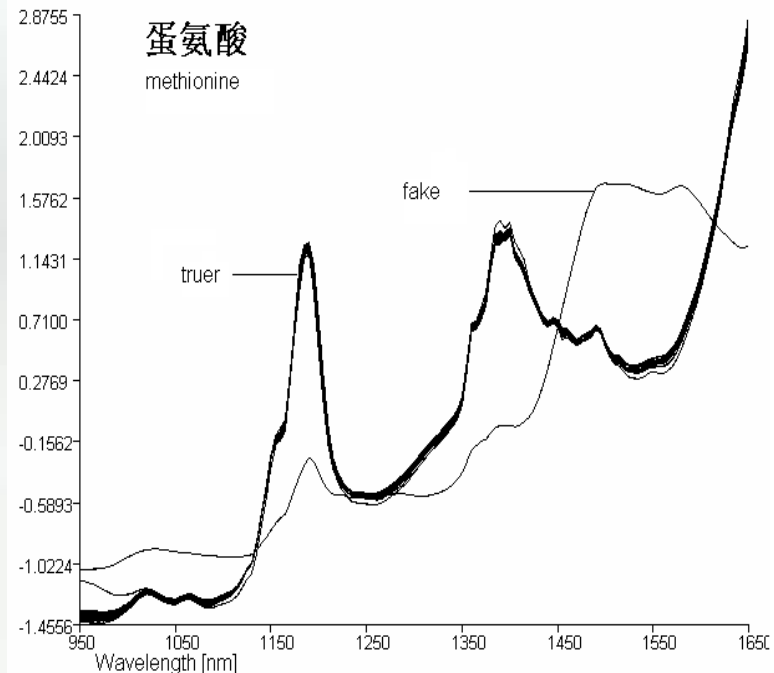
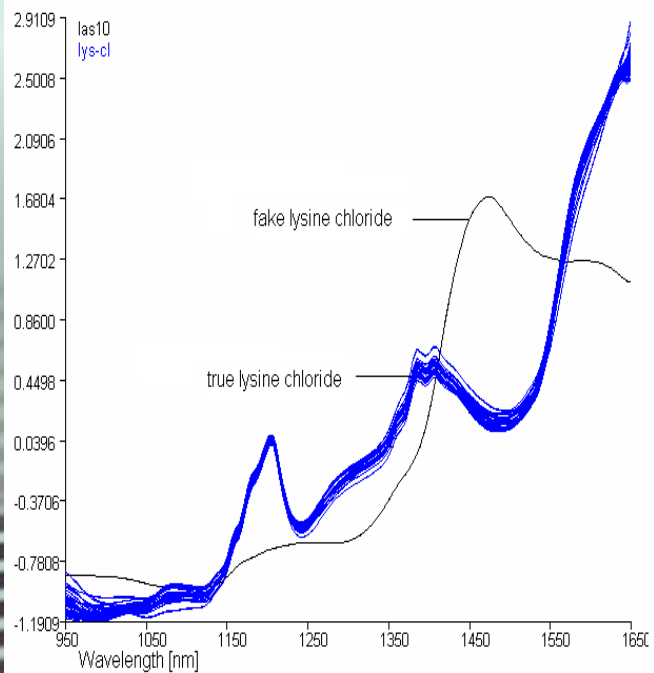
Example 3

Vitamin



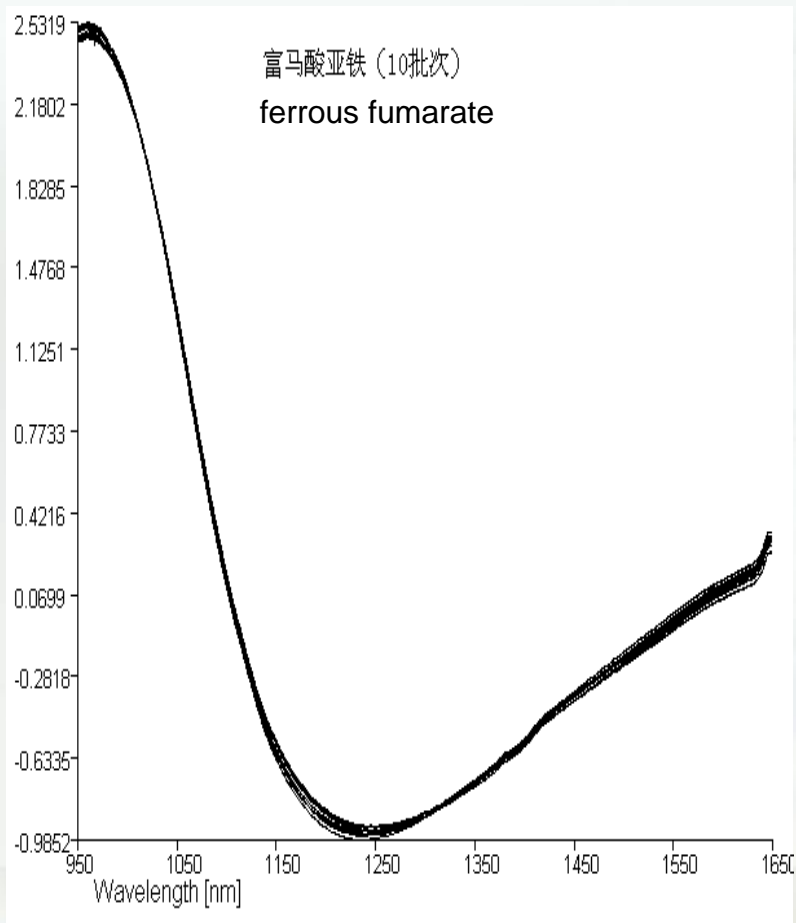
Example 4

Amino acid



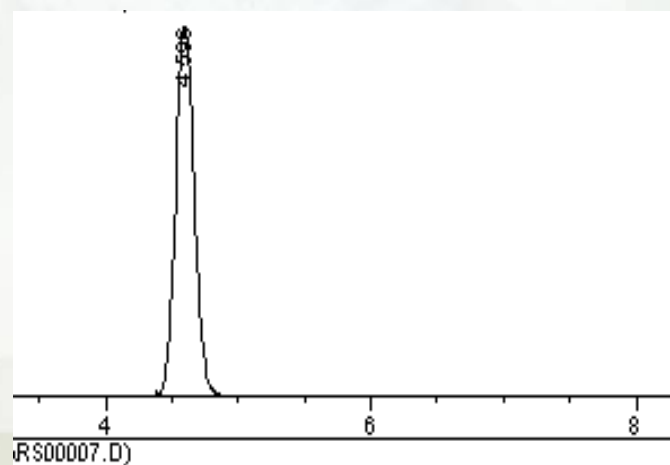
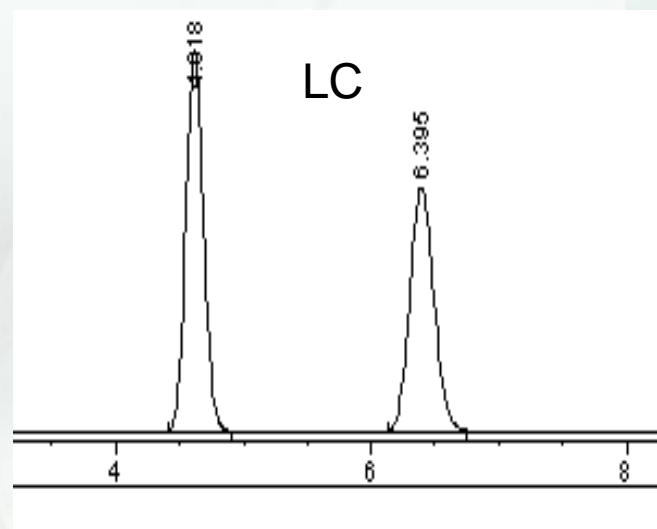
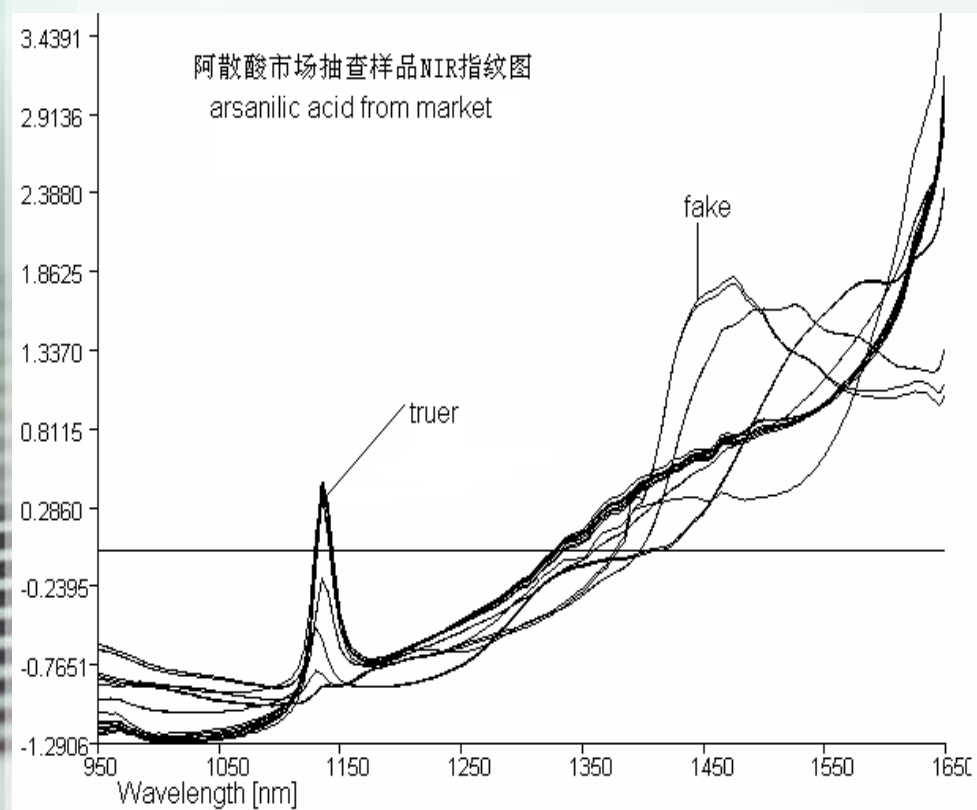
Example 5

Organic mineral

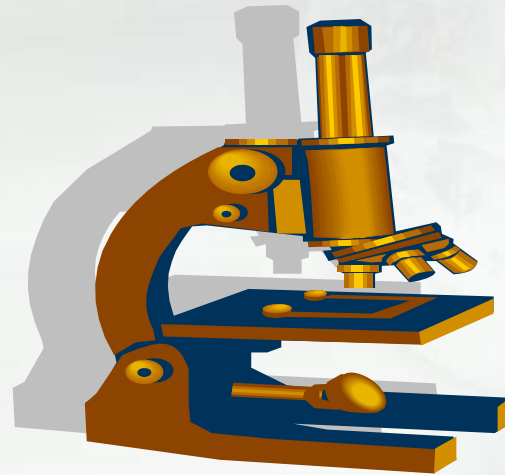
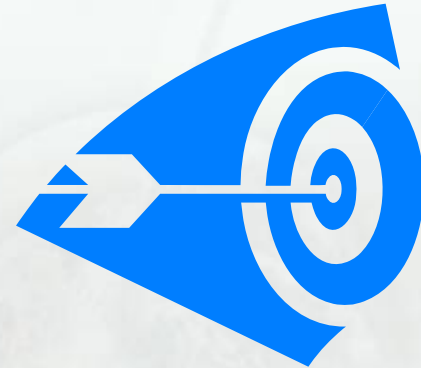


Example 6

Organic arsenic



Let's try something new!



Do not keep to the traditional one only



Thanks for your attention!!

