Infrared Spectrometric Purity Control of Chemical Substances using R

Fayaz Kondagula[1,*], Karl Molt[1]

1. Universität Duisburg-Essen, Department of Chemistry, 47048 Duisburg, Germany
*Contact author: fayazpharm@yahoo.co.in

Keywords: Infrared Spectroscopy, Difference Spectroscopy, Purity Control

Infrared Spectra of organic compounds offer a wealth of bands which are characteristic for certain structural units. Therefore IR spectroscopy is widely used as "fingerprint method" for identifying unknown compounds. A typical IR spectrum contains about 4000 data points and computer software is needed for processing the spectral data. The instrument manufacturers normally provide proprietary software for this purpose. But this is often limited to a restricted number of common applications. To enable a more flexible and universal numerical and statistical evaluation of spectral data, the authors have developed methods to directly read the spectra into R.

On this basis a method was developed for controlling the purity of chemical substances. If a spectrum of a pure reference substance is known, there are two ways for determining the purity of a potentially contaminated sample. The first is difference spectroscopy and its principle is very simple. After obtaining the spectrum of the sample the reference spectrum is subtracted from it. The resulting difference spectrum is then due to the impurity contained in the sample. However due to noise, baseline shifts and atmosperic disturbances IR spectra normally are far from perfect and so a specific algorithm ("dynamic difference spectroscopy") was developed for determining the optimal difference factor, i.e. the factor by which the reference spectrum has to be multiplied before subtraction to obtain an optimal compensation. This works by calculating a whole series of difference spectra with decreasing difference factors and monitoring how the integral of the difference spectra changes. Shortly before it becomes significantly larger than zero, the optimal compensation has been reached. For finding this critical point the `diff` function was used.

The second way to check for impurities is to calculate the correlation coefficient r between the sample and the reference spectrum [1, 2, 3]. This is performed by regressing the sample on the reference spectrum with `lm`. The correlation coefficient however has the disadvantage that the progress of its deviation from one is very slow with increasing contamination. We therefore transformed the r values into Fisher's z coefficients (using the package **survcomp**) and could show that these react much more sensitive to small impurities [4].

Both methods are demonstrated with the example of a certain plasticizer (Palatinol N) contaminated by increasing quantities of another type of plasticizer (Palatinol 911 P).

References

[1] Horst Weitkamp und Dieter Wortig, Vollautomatische Identitätsprüfung von Arzneimitteln durch rechnergekoppelte IR-Spektroskopie. Mikrochimica Acta 1983 II, 31-57.

[2] Robert A. Hoult, Patent number US5023804: Method and apparatus for comparing spectra.

[3] Peter R. Griffiths and Limin Shao, Self-Weighted Correlation Coefficients and Their Application to Measure Spectral Similarity, Appl. Spectroscopy. 63, 2009, 916-919.

[4] Fayaz Kondagula and Karl Molt, Infrared Spectrometric Purity Control of Organic Liquids and Water, Clean 2009,37(12),955-962.