Determination of Antioxidant Irganox 1010 in Polypropylene by Infrared Spectrometry

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Abstract. The content of antioxidant 1010 in polypropylene was quantitatively analyzed by infrared spectroscopy. After comparing the IR spectra of antioxidant 1010 and polypropylene, the characteristic absorption peak of antioxidant 1010 in polypropylene is determined to be 1744 cm$^{-1}$. The standard curve of antioxidant 1010 was drawn. The results show that there is a good linear relationship between the content of antioxidant 1010 and the absorption intensity of its characteristic peak, and the correlation coefficient is greater than 0.98. The content of antioxidant 1010 in the sample to be tested was determined by standard curve. The maximum relative error was 4%. Infrared spectroscopy can be used to analyze the content of antioxidant 1010 in polypropylene quickly and accurately. It is of great significance to study the migration of antioxidant in polypropylene and the quality control of products.

Keywords. Infrared spectrometry polypropylene Irganox 1010 quantitative analysis.

1. Introduction
Polyolefin is a polymer formed by addition polymerization of small olefin molecules. Polypropylene is a common polyolefin. Polypropylene has many advantages, such as abundant raw materials, low price, easy processing and molding, which can be widely used in different fields. Although polypropylene has excellent comprehensive properties, it can be affected by oxygen, ultraviolet light, radiation, mechanical stress and other effects in the process of processing, storage and use. The long chain in polypropylene molecules is easy to age and fracture, resulting in deformation, discoloration, mechanical energy reduction of polypropylene products, and losing the original application value. In order to inhibit and slow down the oxidation degradation rate of polypropylene and increase the service life of polypropylene products, the most effective and common method is adding antioxidant [1-5]. Irganox 1010 is a general-purpose antioxidant of hindered phenols commonly used in polypropylene. It has the characteristics of low volatility, high thermal stability, non-toxic, non-toxic, good extraction resistance,
significant and durable antioxidant effect, which can significantly improve the service life of polypropylene products.

Quantitative determination of antioxidant content in polypropylene is an important aspect of monitoring the quality of polypropylene products. At present, the main methods to determine the content of additives in polyolefins are chromatography, nuclear magnetic resonance spectroscopy, mass spectrometry, etc. [6-15] Most of these analysis methods have the problems of long test cycle, high cost, and unable to achieve online detection. The quantitative analysis technology of Fourier transform infrared spectroscopy has the advantages of easy preparation of samples, accurate, rapid and sensitive analysis results. In this study, a quantitative FTIR method for the determination of antioxidant 1010 in PP was established, which is of great significance to the quality control of polypropylene and the study of the migration of antioxidant in polypropylene.

2. Materials and methods
Fourier transform infrared spectrometer, FTIR-650, Tianjin Gangdong Technology Development Co., Ltd.; PP, Maoming Shihua Dongcheng Chemical Co., Ltd.; antioxidant 1010, Ciba Jinghua (Shanghai) Co., Ltd.

In order to prepare polypropylene samples with uniform antioxidant content, antioxidant 1010 was first prepared into a solution with a mass fraction of 1%, and then added to PP powders respectively. After well-mixing, the samples with a thickness of 1mm were pressed on a hot bench at 160 °C. The mass fractions of antioxidant 1010 in PP samples were 0, 0.1%, 0.2%, 0.3%, 0.4% and 0.5% respectively. Three parallel samples were prepared for each sample and FTIR was used to collect the spectra.

3. Results and discussion

3.1. Analysis and determination of characteristic peak of antioxidant 1010
FTIR spectra of antioxidant 1010, PP and PP with antioxidant 1010 samples were analyzed, as shown in Figure 1. In the IR spectrum of antioxidant 1010, the stronger single absorption peak appears at 1744cm⁻¹, which is the characteristic absorption peak of C=O, and there is no other absorption peak nearby. In the IR spectrum of PP, there is almost no absorption peak at 1744cm⁻¹, so the absorption peak of antioxidant 1010 at 1744cm⁻¹ is selected as the characteristic analysis peak of quantitative analysis.

![Figure 1. FTIR spectra of antioxidant 1010, PP and PP + 1010 samples](image)

In the IR spectrum of PP samples, there are a series of strong absorption peaks in 3000-2800cm⁻¹, which are the absorption peaks of C-H asymmetric stretching vibration, 1647cm⁻¹ is the absorption peak of C=C stretching vibration, 1466cm⁻¹ is the absorption peak of methylene and methyl bending vibration, 1370cm⁻¹ is the absorption peak of methyl in PP branched structure, 1165cm⁻¹ is the absorption peak of
methyl plane vibration, 720 cm\(^{-1}\) is the swing vibration peak of methylene plane. After comparison, 1165 cm\(^{-1}\) was selected as PP quantitative analysis peak.

### 3.2. Drawing of standard curve

FTIR analysis was carried out on PP samples with 0-0.5% antioxidant 1010 mass fraction. The relationship between the characteristic absorption peak intensity at 1744 cm\(^{-1}\) and the added content is shown in Figure 2.

![Figure 2. Relationship between characteristic absorption peak strength and content of antioxidant 1010 in PP](image)

In order to eliminate the influence of the thickness difference of PP sample on the characteristic peak area of antioxidant 1010, the ratio of the characteristic peak area \(A_1\) (1744 cm\(^{-1}\)) of antioxidant 1010 to the characteristic peak area \(A_2\) (1165 cm\(^{-1}\)) of PP is taken as the ordinate, and the standard curve is established with the added content of 1010 as the abscissa, as shown in Figure 3.

![Figure 3. Standard curve of antioxidant 1010 in PP](image)

It shows that in the selected range of the experiment, \(A_1 / A_2\) gradually increases with the increase of antioxidant 1010 content, which is in direct proportion, in line with Lambert Beer law from Figure 3. The linear regression equation of the standard curve is \(y = 0.04248 + 0.31376x\), and the correlation
The coefficient of the standard curve is 0.99939. It shows that the standard curve has a high linear correlation and can be used for the quantitative determination of antioxidant 1010 in PP samples.

### 3.3. Verification of precision and accuracy of measurement results

In order to verify the precision of the established detection method, PP samples with antioxidant 1010 mass fraction of 0.4% were measured by FTIR for 5 consecutive times under the same conditions. The content of antioxidant 1010 in PP was calculated by standard curve. The results are shown in Table 1. It can be seen that the maximum relative error is 3.50%, which proves that the measurement results of this method have good reproducibility and can meet the analysis and determination requirements in actual production.

**Table 1.** Precision verification results of antioxidant 1010

| Sample | Mass fraction (%) | True value | Measured value | Relative error (%) |
|--------|------------------|------------|---------------|-------------------|
| PP     | 0.4              | 0.389      |               | 2.75              |
| PP     | 0.4              | 0.386      |               | 3.50              |
| PP     | 0.4              | 0.391      |               | 2.25              |
| PP     | 0.4              | 0.405      |               | 1.25              |
| PP     | 0.4              | 0.391      |               | 2.25              |

In order to verify the accuracy of the established detection method, PP samples with the mass fraction of antioxidant 1010 of 0.05%, 0.15%, 0.25%, 0.35% and 0.45% were respectively made according to the same method. After FTIR measurement and analysis, the content of antioxidant 1010 in PP was calculated with the standard curve. The results are shown in Table 2. The relative error of the determination of antioxidant 1010 in PP by this method is less than 4%, and the determination result is accurate and reliable.

**Table 2.** Validation results of the accuracy of antioxidant 1010 test results

| Sample | Mass fraction (%) | True value | Measured value | Relative error (%) |
|--------|------------------|------------|---------------|-------------------|
| PP-1   | 0.05             | 0.0480     |               | 4                 |
| PP-2   | 0.15             | 0.1456     |               | 2.93              |
| PP-3   | 0.25             | 0.2524     |               | 0.96              |
| PP-4   | 0.35             | 0.3392     |               | 3.08              |
| PP-5   | 0.45             | 0.4590     |               | 2                 |

### 4. Conclusion

A method for quantitative analysis of antioxidant 1010 in PP by FTIR was established. Through comparative analysis, $1744 \text{cm}^{-1}$ absorption peak was selected as the quantitative analysis peak of antioxidant 1010. The linear regression equations of the standard curves of antioxidant 1010 in PP were $y = 0.04248 + 0.31376x$. The correlation coefficient was 0.99939. The ratio of the characteristic peak area of antioxidant 1010 to the characteristic peak area of polypropylene instead of the characteristic peak area of antioxidant is taken as the measurement parameter, and the standard curve is drawn to eliminate the influence of the difference of sample thickness on the characteristic peak area of antioxidant, so that the samples with different thickness can be directly compared with the antioxidant content. This method can be used to analyze the mobility of antioxidant in polypropylene and the quality of products in the production process.
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