The Water Factor in Medical Device Resins

Introduction

The Health Care industry has increased its needs for specialized devices over the past decade, which has led to a new frontier of resin and polymer development designed to keep the quality of care high while minimizing cost. With these goals in mind, the resins being used for medical devices are scrutinized more thoroughly than other resins that require less regulatory compliance. Analyzing a product for outgassing, deformation, and reactivity, among other things, has become part of the daily routine for manufacturers, molders, and final inspection personnel before an item can be shipped or used. This additional testing and control also includes the amount of water that is allowed in the resins, since this will greatly influence the final product's rigidness, consistency, and lifetime, as well as the quality of care that will be provided to the customer. Oftentimes the quality control of the materials is closely monitored using testing equipment defined in an IQ/OQ/PQ: installation qualification (IQ), operational qualification (OQ), and performance qualification (PQ) to ensure that the instruments are effective, and the quality of the product is consistent.

Moisture Determination

As an alternative to the Coulometric Karl Fisher titration, Relative Humidity (RH) sensor moisture detection was first used as a method for determination of water in materials in 1997, with the introduction of the Computrac® 3000 Moisture Analyzer by Arizona Instrument LLC. This method uses a thermoset polymer capacitor that has a selective response when in the presence of water, the same way that many RH sensors work in traditional settings such as houses, laboratory controlled environments, and dry boxes.

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3375 N. Delaware Street, Chandler, AZ 85225
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Medical device resins are sealed in a sample vial, and then transported into an oven chamber with inert gas blown through it. As the material gets hot, water molecules evolve off and are carried to the sensor via the carrier gas. The sensor is exposed to the water molecules and a measurable change in the electronic activity takes place. This method requires no solvents, making it an environmentally friendly alternative to traditional chemical titration. The instrument provides *in-situ* moisture measurements, which allows users to monitor performance in real time. Additionally, it has detection Limit of 10ppm, and is more rugged than Karl Fisher titrators, making it a suitable instrument for moisture analysis in manufacturing facilities, as well as Quality Control and inspection labs. This technology is now being adopted as the standard test method and is described by ASTM D7191, Standard Test Method for Determination of Moisture in Plastics by Relative Humidity Sensor. This instrument also meets the high demands of performance given in an IQ/OQ/PQ.

With major advances in technology, the medical device community is also taking advantage of new RAPID loss-on-drying methods for moisture determination. These instruments use the same principle as traditional loss-on-drying techniques, but address the shortcomings of the method. Sample material is heated on a balance and real time measurements are providing immediate feedback and moisture concentration. The Computrac® MAX® 4000XL instrument, manufactured by Arizona Instrument LLC, provides a parameter development expert program that allows users to optimize testing conditions, such as sample size, test ending criteria, testing temperature, idle temperature, temperature rate, etc. The chassis of this instrument is made of steel, which prevents cracking in the case and cool air from entering the testing chamber, which would influence the results. These new techniques are being adopted as standard testing methods and are described by ASTM D6980-12, Standard Test Method for Determination of Moisture in Plastics by Loss in Weight. Like the Vapor Pro® 3100L, the MAX® 4000XL meets the performance standards set forth in typical IQ/OQ/PQ testing.

Testing

Sample Prep – A medical grade TPU was selected for analysis. The material was stored wet in a 1 gallon plastic Ziploc bag prior to testing. An initial analysis was conducted to determine the water content prior to drying. The material was then dried in the Dri-Air HP4-X 25 plastics drying hopper for 6 hours prior to testing. The material remained in the dryer during testing due to the hygroscopic properties of the material.

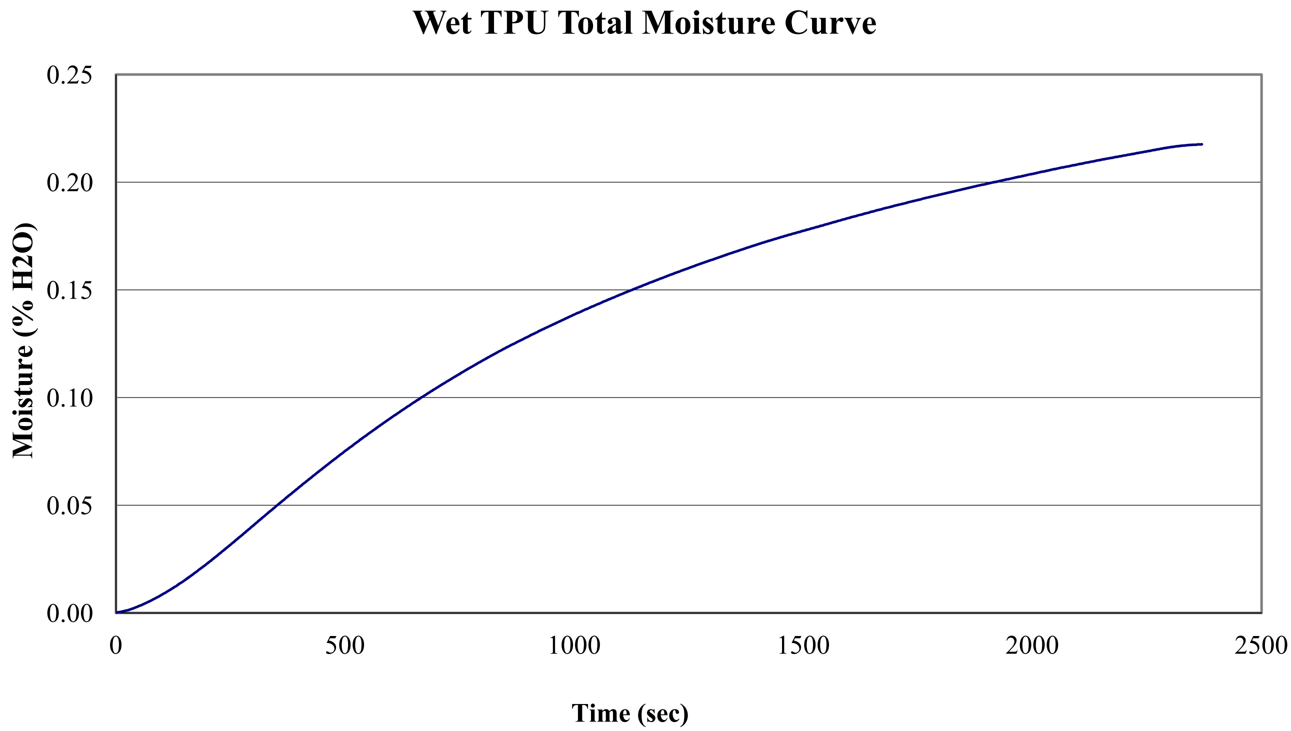
Test Conditions - Reference testing was conducted using the Mitsubishi CA-100 Coulometric Karl Fischer titrator. The parameters were: sample size – 0.5g +/- 0.1g, temperature – 90°C, purge/preheat/cooling – 1/2/2, ending sensitivity – 0.1µg/sec.

Corollary testing was conducted using the Computrac® Vapor Pro® 3100L. The parameters were: sample size – 2g +/- 0.2g, temperature – 105°C, purge – 50 sec., ending criteria – rate<0.1µg/sec.

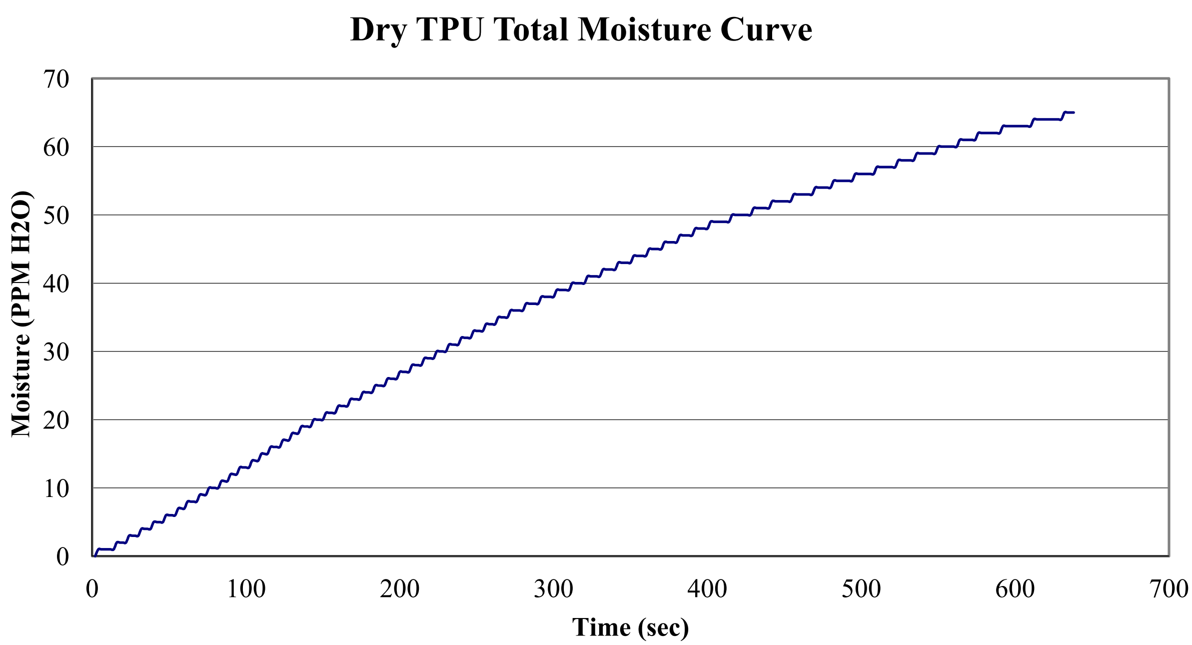
Results

	Karl Fischer		Vapor Pro 3100L	
	Result (ppm)	Test time	Result (ppm)	Test time
	47	6:53	51	12:32
	58	7:30	74	14:36
	67	7:04	53	12:20
	79	7:46	78	15:08
			69	13:18
Average	62.75		65	
S.D.	13.57387196		11.00908716	
RSD	21.63166846		16.93705716	

Table 1. Comparative results of TPU testing



Graph 1. Total moisture curve of pre-dried TPU



Graph 2. Total moisture curve of TPU dried for 6 hours at 200°F

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From the table, the results using the two different instruments with similar testing conditions correlate to each other. The Vapor Pro® did show an improvement in the relative standard deviation, but did require a slightly longer test time than the Karl Fischer. Additionally, the Vapor Pro® provided real time data points that could be used to graph the total moisture curve. This allows for better monitoring of the product, or diagnosing possible problems with the instrument. This feature was not available for Karl Fischer titrator.

Conclusion

The development of an alternative to Karl Fischer moisture analyzer has been achieved, and can be used for moisture specific analysis of medical device grade resins. The Computrac® Vapor Pro® 3100L moisture analyzer successfully uses Relative Humidity sensor technology for selective and accurate moisture measurement. The instrument reduces the use of hazardous organic solvents makes it an environmentally friendly alternative, when compared to current Karl Fischer technology. The results between the two methods of detection of H_2O content in TPU strongly correlate, with the Vapor Pro® 3100L providing real-time data that can be used to provide a complete profile of the TPU.

James Moore, Chemist

Arizona Instrument LLC