

CONTINUOUS MONITORING OF IMPURITIES DURING THE MANUFACTURE OF TITANIUM DIOXIDE

Titanium dioxide (TiO₂) is a white pigment used in the production of cosmetics, food, paint, paper, pharmaceuticals and plastics.

MEASUREMENT REQUIREMENT

TiO₂ has tight product quality specifications. Titanium tetrachloride (TiCl₄) with limited impurities is required in the manufacturing of TiO₂ to meet the end product specifications. The presence of vanadium, in the form of vanadium oxytrichloride (VOCl₃), in TiCl₄ is an impurity that affects the quality of the TiO₂. TiCl₄ also needs to be completely oxidized

in the formation of TiO₂, as it is an undesired impurity in TiO₂ product. The AMETEK IPS-4 integrated process spectrometer can be used to measure VOCl₃ in the TiCl₄ intermediate step, Cl₂ to TiO₂ ratio prior to pigment separation and TiCl₄ in the TiO₂ product.

PROCESS OVERVIEW

TiO₂ is primarily manufactured using the chloride process, during which rutile (mostly TiO₂) is converted to TiCl₄ by the ore reacting with chlorine (Cl₂) gas around 900°C (1652°F).

Next, other trace metal chlorides are removed using fractional distillation to refine the TiCl₄. VOCl₃ is a common impurity that is difficult to remove, as it has a similar boiling point as TiCl₄. Because the quality of the TiCl₄ produced directly affects the quality of the TiO₂ pigment and titanium metal, it is important to measure the VOCl₃

concentration after distillation and TiCl₄ storage. An IPS-4 analyzer can perform continuous measurements in this process location, to optimize the process feed rate, distillation column temperature and catalyst usage. Common ranges are 0-20 or 0-40 parts per million by weight of VOCl₃.

Once producers have verified the TiCl₄ meets specification requirements, the TiCl₄ is oxidized to produce pure TiO₂.



AMETEK SOLUTION

The AMETEK IPS-4 integrated process spectrometer, a full-spectrum analyzer, is installed after the oxidation reactor, to measure concentrations of both Cl_2 and TiCl_4 . Ideally, the ratio of Cl_2 to TiO_2 is known to manage the pigment separation stage, and the TiCl_4 concentration – which should be at parts per million levels – is identified to also assist in the automated process control. The feed rates of oxygen, other additives and the TiCl_4 can be adjusted if the TiCl_4 concentrations are too high following oxidation to TiO_2 .



PROCESS OVERVIEW

The total time measurement and calculation is less than four seconds. The multivariate calibration accounts for specific interferences caused by the sample stream. The IPS-4 is capable of monitoring specific wavelengths and performing multi-component analysis. For this application, measurements are taken between 365 and 450 nm to avoid inherent interferences of the process.

The IPS-4's unique flow cell design separates the process sample from the spectrophotometer, simplifying installation and eliminating any possibility of sample damage to the electronics.

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