

Monitoring TiCl_4 and Vanadium In the TiO_2 Manufacturing Process

The Process

Titanium dioxide is used as a white pigment and is usually manufactured through the hydrolysis of titanium tetrachloride. Two critical measurement points were identified in this process, one in the incoming stream where the TiCl_4 needs to be Vanadium free, and the other at the final stage after the TiO_2 particles have been removed. The gas stream at this point contains nitrogen, HCl, and carbon dioxide. Monitoring the TiCl_4 contents is of great importance at this stage since if present it indicates that not all the TiCl_4 has reacted with oxygen to create TiO_2 .

Detection Method

An UV-VIS-SWNIR diode array spectrophotometer was used to detect and monitor the concentrations of both the vanadium and titanium tetra chloride at different stages of the process.

The full spectrum advantage

In this particular application there are several reasons for full spectrum analysis: when monitoring vanadium in TiCl_4 (see figure 1), the TiCl_4 itself is a very strong absorbent at the wavelength of interest. The vanadium, therefore, is monitored at the shoulder of the TiCl_4 peak. For reliable and accurate measurements, only a full high resolution spectrophotometer can be utilized. In monitoring titanium tetra chloride, HCl or Cl_2 if present, might interfere with the spectral features of TiCl_4 . Again, a full spectrum analysis allows for the flexibility of monitoring specific wavelengths or performing multi-components analysis. Both applications can be performed by the same analyzer.

Vanadium in TiCl_4

Figure 1 shows the spectrum of vanadium in TiCl_4 . The wavelengths shown were found to be the optimal ones for this application, since at lower wavelengths the TiCl_4 absorbance features interfere and at higher wavelength the absorbance signal of vanadium is too low to comply with the detection levels (vanadium at low PPM) required for this application. It should be noted that in other applications where vanadium is measured without the strong interference of titanium tetra chloride, very accurate measurements even at minute



amounts of vanadium can be achieved. This is done by scanning at lower wavelengths at which maximum sensitivity to vanadium is found. At high levels of vanadium the SWNIR spectral region can be used. See application note on Cu and V ions.

Titanium tetra chloride in the process gas stream

The following graphs show the absorbance spectra of titanium tetra chloride. Figure 2 shows the absorbance spectra of low levels TiCl_4 in a gas stream containing chlorine gas and other non-absorbing components. Note that the chlorine absorbance feature overlaps with that of the TiCl_4 (Figure 3). A multi component analysis method is therefore essential.

Fig 1: absorbance spectra of Vanadium in TiCl₄ (low ppm)

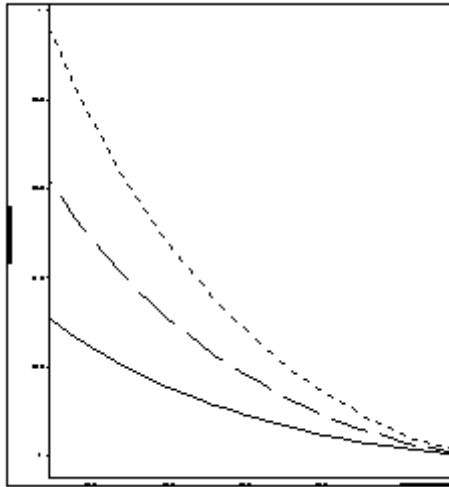


Fig 2: absorbance spectra of TiCl₄

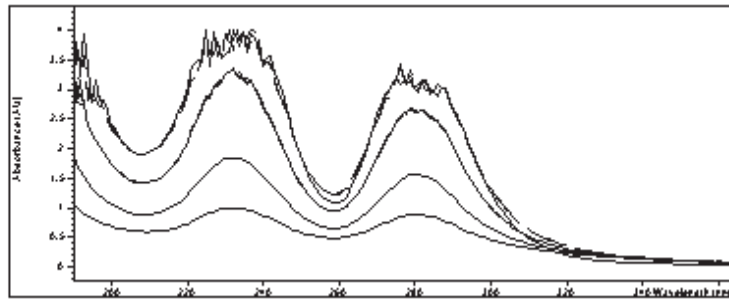


Fig 3: absorbance spectra of TiCl₄ in chlorine stream, note the Cl₂ interference

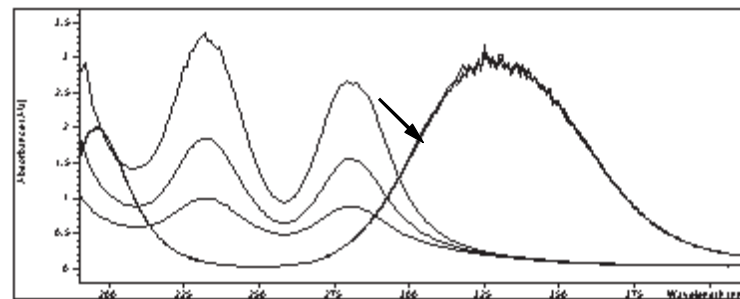


Fig 4: Three valve sampling system

Applied Analytics, Inc.

29 Domino Drive
Concord, MA 01742
Tel: 978-287-4222
Fax: 978-287-5222
e-mail: sales@a-a-inc.com
web: www.a-a-inc.com

Application note - *OMA-300* #Publication #P991018
© Copyright Applied Analytics Inc. , 2004
The information in this publication is subject to change without notice
Microsoft® is a U.S. registered trademark of Microsoft corporation.

<http://www.a-a-inc.com>