On-Line Thermal Desorption Analysis As Replacement for Solvent Extraction Methods

Application Note

Semiconductor Industry

Author:

Raymond Yu & Gary Deger

Abstract

A True Company Time Saver while Improving Worker Health

In the manufacture of modern-day consumer goods, toxic chemicals are still required, potentially putting the workers' health at risk. In the semiconductor and LCD manufacturing industry, one such compound is N-methyl pyrrolidinone (NMP). Because of NMP's non-volatility and its ability to dissolve diverse materials, it is widely used as a cleaning solvent in many industries from petrochemical to electronics to pharmaceuticals.

Figure 1: System Assembly on 19" Rack

In the electronics industry, a wafer must be cleaned after its manufacture to eliminate trace organic contaminants. NMP diluted in 2-Butoxyethanol is a common mixture used in cleaning both wafers and finished chips. However, the latest data from several regulatory sources show that regular exposure to NMP depresses the central nervous system, can cause headache, nausea and vomiting, as well as being a possible reproductive hazard.

Until recently, the current analytical method for monitoring NMP in air was NIOSH Method 1302. This method outlines the collection of samples on a charcoal thermal desorption tube, which is then flushed with a 95:5 methylene chloride:methanol solvent to extract the NMP. The solvent is then injected into a gas chromatograph with flame ionization detector (GC-FID) for final analysis. Although this method adequately meets the necessary longterm and shorterm Threshold Limit Values, there are several shortcomings including:

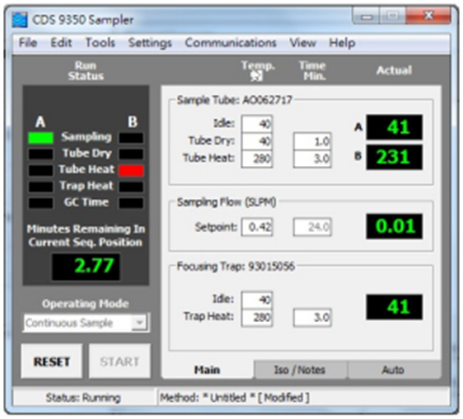
1. Cumbersome sample collection process that can take up to 10 hours per sample
2. Non-environmentally friendly solvent for the extraction
3. A long lag time in obtaining the final results

However, by using an on-line thermal desorption system connected directly into a GC-FID, this analysis can be made in near-real time and on a 24x7 basis, without manual sample preparation. As shown in figure 1, we have a CDS model 9350 Thermal Desorption system connected to a SRI 410 GC with FID detector. A hydrogen generator is also included to feed the FID. Since this site had multiple locations to sample, a 16 position stream selector (Valco) is included that alternates between sampling locations after one has been adequately sampled.

The CDS 9350 contains 2 sampling tubes and one focusing trap. A vacuum pump pulls a few liters of air onto one of the sampling tubes, while the second is being desorbed onto the focus trap, before going into the GC. Once the focus trap is desorbed into the GC, the sampling tubes switch and a fresh sample is then desorbed from the second sampling tube onto the focus trap. This dual tube system allows a fresh sample always to be ready, speeding up the analysis by several minutes/run.

The air samples are continuously drawn from various clean rooms through PTFE tubing. In this example, the distance of the clean rooms are between 100 to 200 meters. By the control of the GC with an event timer, the air sample is sent to the CDS- 9350 for collection at the appropriate time onto a Tenax-TA filled thermal desorption tube. The air flow can be set up to 1.5 LPM, if desired, for maximum speed. The collected sample was then heated and sent to a 1/8" SS focusing trap packed with Tenax-TA. The focus trap is a narrow bore tube packed with adsorbant that aides the GC in producing sharp analytical peaks. Once the transfer is complete, the trap is heated and the components are sent to the GC by a heated transfer line. An RTX-624 wide bore capillary column was used. There are many advantages of an on-line TD system vs a manual prepared sample method, but several of the more important ones include:

1. Fast analysis time allows workers to be moved if there is an upset condition
2. Frees up valuable lab time for other projects
3. Eliminates the need for industrial solvents to make the analysis
4. Keeps a permanent record, documenting safe conditions for workers in the exposed areas



Picture 2: CDS Analytical 9350 DCI

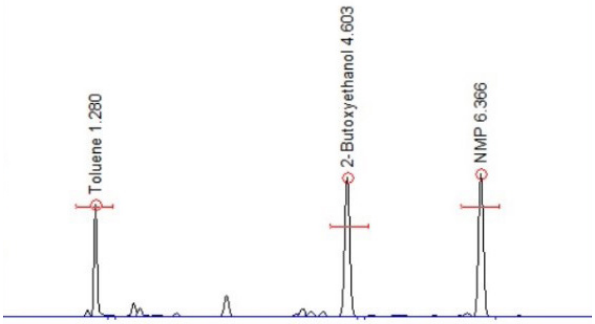
Picture 3: CDS Analytical 9350

CDS 9350 Conditions

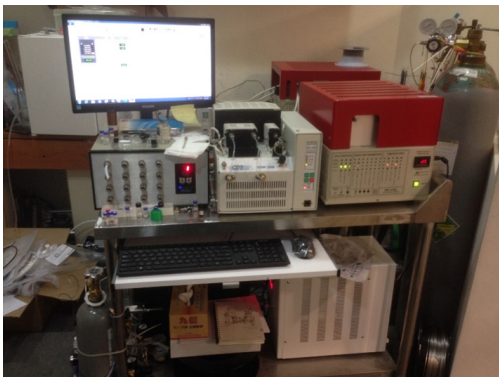
Sample Tube Rest Temp:	40 °C
Tube Dry:	40 °C, 1.0 min
Tube Heat:	280 °C, 3.0 mins
Sample Flow:	.30 SLPM for 10 mins
Focus Trap Tube Heat:	280 °C, 3.0 mins
Valve Oven:	250 °C
Transfer Line: GC:	250 °C

SGE GC 410 Conditions:

Column:	RTX-624; 30M x 0.53mm x 3.0um
Carrier:	Nitrogen, 10ml/min
Oven:	50°C (1 min) -> 20 °C/min -> 240 °C (13mins)
FID Temp:	250°C



Picture 4: Chromotography Data



Picture 5: System Assembly on Table

Picture 6: Valco 16 Position Stream Selector