

Analysis of Organotin in Paints by GC-MS

1 Experiment

1.1 Instrument and Reagents

GC-MS3100 gas chromatograph-mass spectrometer

Analytical balance, vortex mixer, pH meter, ultrasonic equipment, 0.45 μm organic disposable filter head, pipettor, 25 mL glass test tube with cap, 2 mL glass or disposable syringe.

Reagents: Dibutyl tin dichloride (DBT) standard (purity 97%), sodium tetraethyl borate (purity $\geq 98\%$), glacial acetic acid, anhydrous sodium acetate, methanol (chromatographic reagent), n-hexane (chromatographic reagent), reagent water, etc.

1.2 Sample pretreatment

1g coating powder was weighted and placed in 25mL glass test tube with cap, added with 5mL methanol, vortexed on vortex mixer for 2 min, then placed in ultrasonic equipment up to 20min for ultrasonic extraction. Then, it was vortexed up to 1 min for mixing and extraction, placed at room temperature and added with 5 mL acetic acid-sodium acetate buffer solution (pH 4.5) and 2 mL sodium tetraethyl borate aqueous solution (2%). After that, it was immediately vortexed for 2 minutes, allowed to fully react at room temperature for 30 min, then added with 2 mL n-hexane and vortexed for mixing and extraction, then left for decantation. After that, the upper organic phase was filtered through 0.45 μm disposable organic filter head, and the filtrate was placed in 2 mL small vial with cap for GC-MS analysis.

1.3 Derivatization of standard solution

1 mL organotin standard working solution was placed in 25 mL glass test tube with cap, added with 5 mL acetic acid-sodium acetate buffer solution, then added with 2 mL sodium tetraethyl borate aqueous solution (2%). Then it was immediately vortexed for mixing for 2 min and allowed to fully react at room temperature for 30 min. Then, 2mL n-hexane was added, vortexed for mixing and extraction, then left for decantation. After that, n-hexane layer was taken and placed in 2ml small vial with cap, for GC-MS analysis.

1.4 Analysis conditions

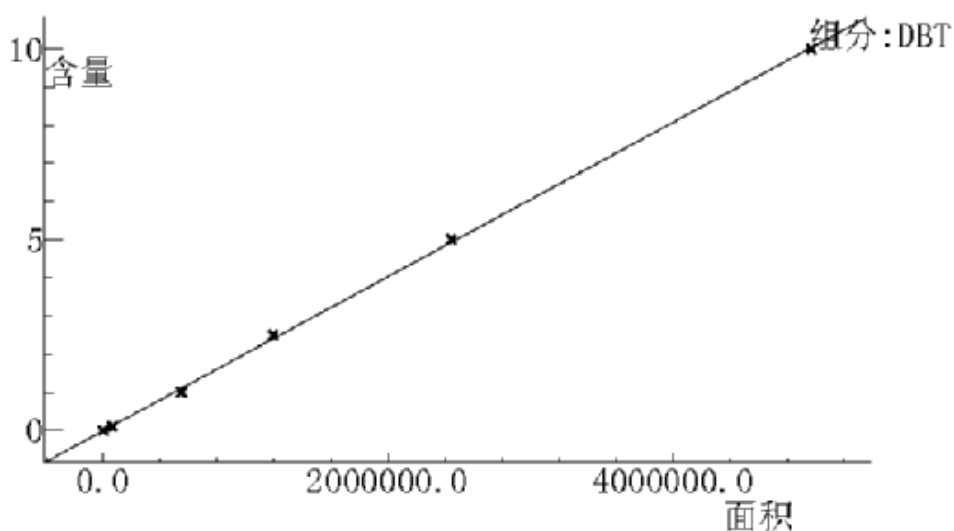
GC conditions: Equity-5 (30m×0.25mm× 0.25μm) quartz capillary column, precolumn pressure 80 kPa, splitless injection, injection volume: 1μL, split ratio: 60:1, purge flow: 3 mL/min, vaporization chamber: 270 °C, column temperature program: hold at 50 °C for 1 min, then ramp up to 250 °C at 10 °C/min, hold for 5 min.

MS conditions: EI source, electron energy 70 eV, ion source temperature 200 °C, interface temperature 270 °C, scanning mode: full scan qualitative, scanning range: 50u~400u, solvent peak time: 10 min, electron multiplier high voltage: 1300V. Selective ion monitoring: m/z 151, 177, 179, 205, 233, 263.

2 Results

2.1 Standard curve

1 mL organotin standard working series methanol solutions with concentration of 0, 0.2, 2, 5, 10, 20μg/mL were respectively taken, derivatized and vortexed with 2mL n-hexane for extraction. Resulting concentration of n-hexane extract were 0, 0.1, 1, 2.5, 5, 10 μg/mL. 1uL n-hexane extract was taken for GC-MS analysis, with quantitative ion m/z 263 peak area as abscissa and n-hexane extracts' concentration as ordinate, standard working curve was plotted.



2.2 Sample chromatogram

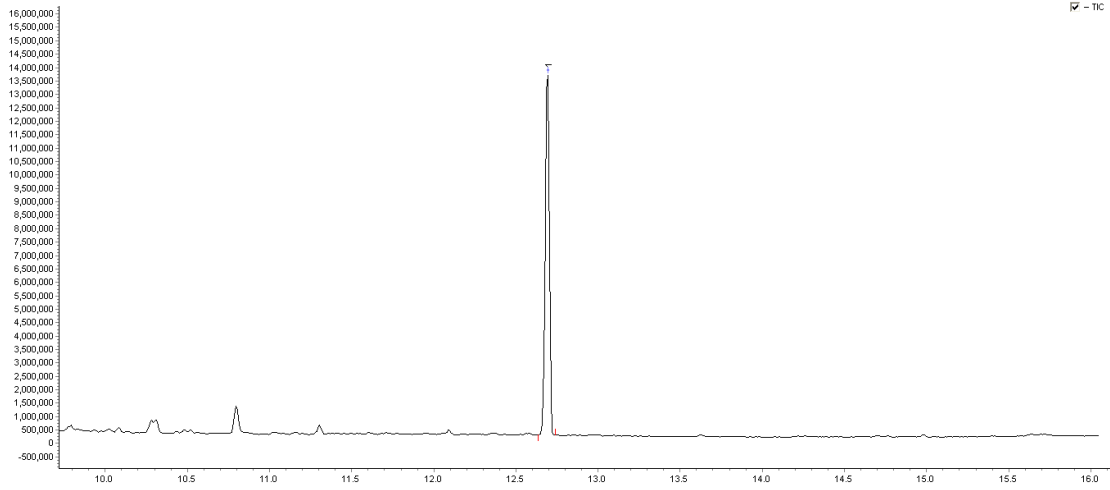


Figure 1 TIC chromatogram of dibutyl tin (DBT) standard sample derivatives

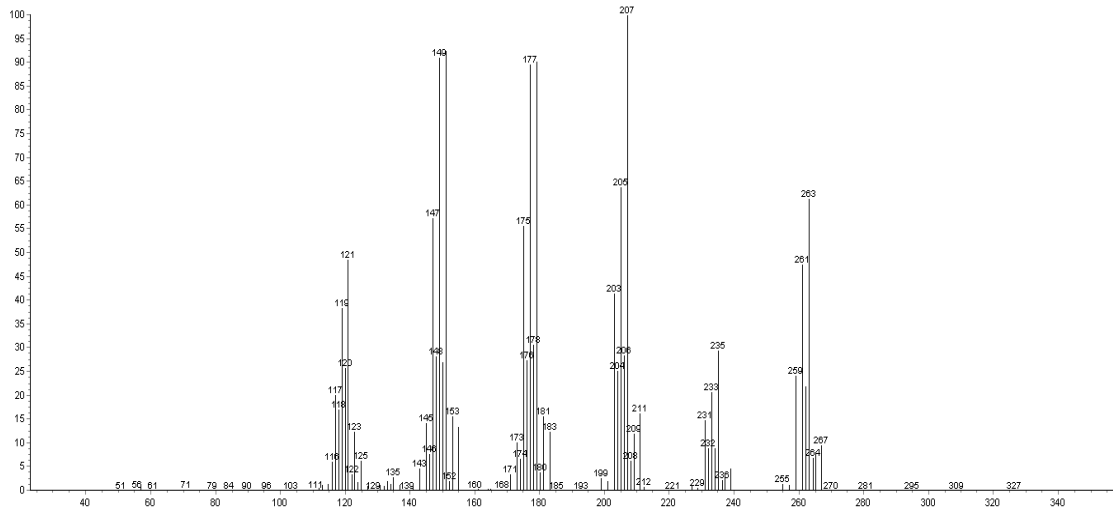


Figure 2 Full scan mass spectra of dibutyl tin (DBT) standard sample derivatives

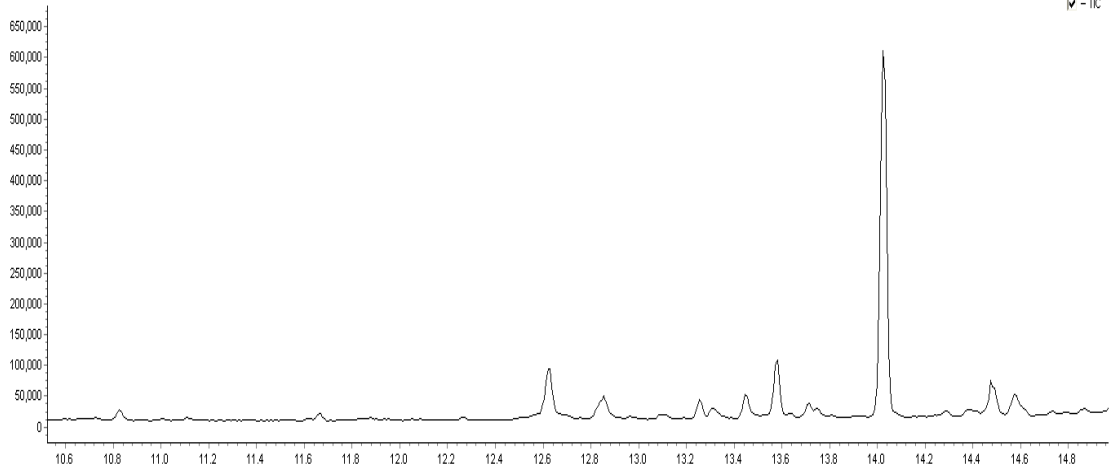


Figure 3 Selective ion scan chromatogram of paint sample