Qualitative Analysis of Volatile Components in Cement Retarder

1 Experiments

1.1 Instrument and apparatus

GC-MS3100 gas chromatograph-mass spectrometer

Constant temperature oven or other temperature control device

5mL pipettor

20 mL headspace vial, and its cap, septum, crimper

1 mL disposable medical syringe and 5# needle

Sample pretreatment

Specified amount of samples were respectively taken and placed in 20 mL headspace vial, capped, placed in oven at 100 °C for 40 min for heating and stabilization. Then, specified amount of headspace gas was collected with 1 mL disposable medical syringe and injected for GC-MS analysis.

1.3 Analysis Conditions

Sample size: 7mL sample is placed in headspace vial

GC conditions: Equity-5 (30m×0.25mm×0.25μm) quartz capillary column, precolumn pressure 30 kPa, split injection, split ratio: 25:1, injection volume: 0.4 mL, vaporization chamber temperature: 150 °C, column temperature program: hold at 35 °C for 4 min, then ramp up to 205 °C at 10 °C/min, hold for 5 min.

MS conditions: EI source, electron energy 70 eV, ion source temperature 150 °C, interface temperature 150 °C, scanning mode: full scan qualitative, scanning range: 25u~280u; solvent peak time: 1.55 min (avoiding air peak), electron multiplier high voltage: 1100V.

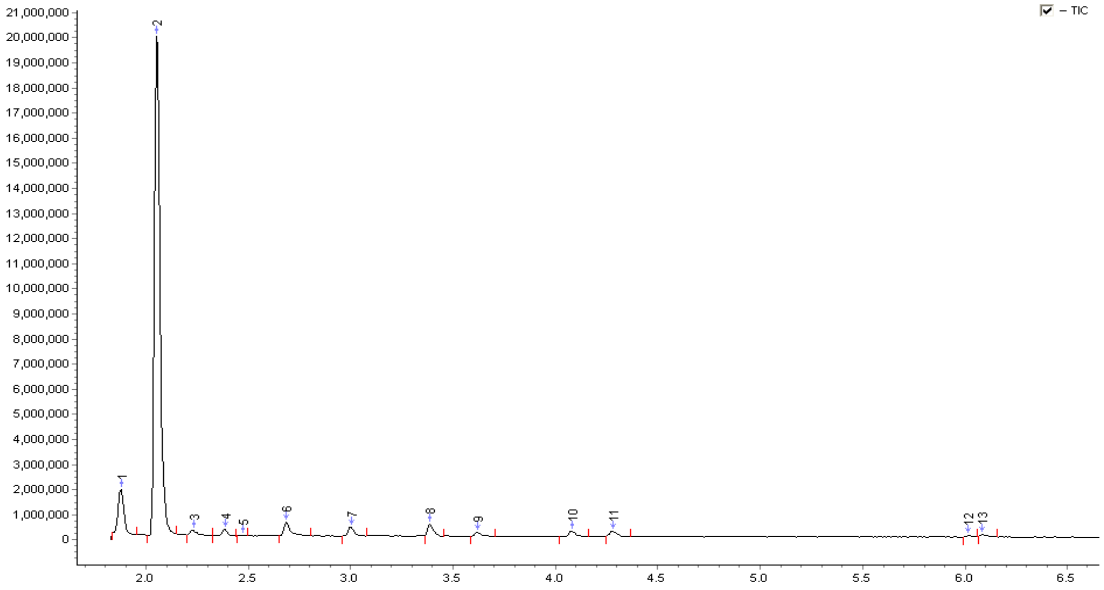

Figure 1 TIC chromatogram of cement retarder

Table 1 Analysis result of volatile components in cement retarder

Peak No	**英文名称 Compound**	**CAS 号 CAS No.**
1	Acetaldehyde	75-07-0
2	Ethanol	64-17-5
3	Acetone	67-64-1
4	Dimethyl sulfide	75-18-3
5	Acetic acid,methyl ester	79-20-9
6	Propanal,2-methyl-	78-84-2
7	2,3-Butanedione	431-03-8
8	Ethyl Acetate	141-78-6
9	1-Propanol,2-methyl-	78-83-1
10	Butanal,3-methyl-	590-86-3
11	Butanal,2-methyl-	96-17-3
12	1-Butanol,3-methyl-	123-51-3
13	1-Butanol,2-methyl-	1565-80-6

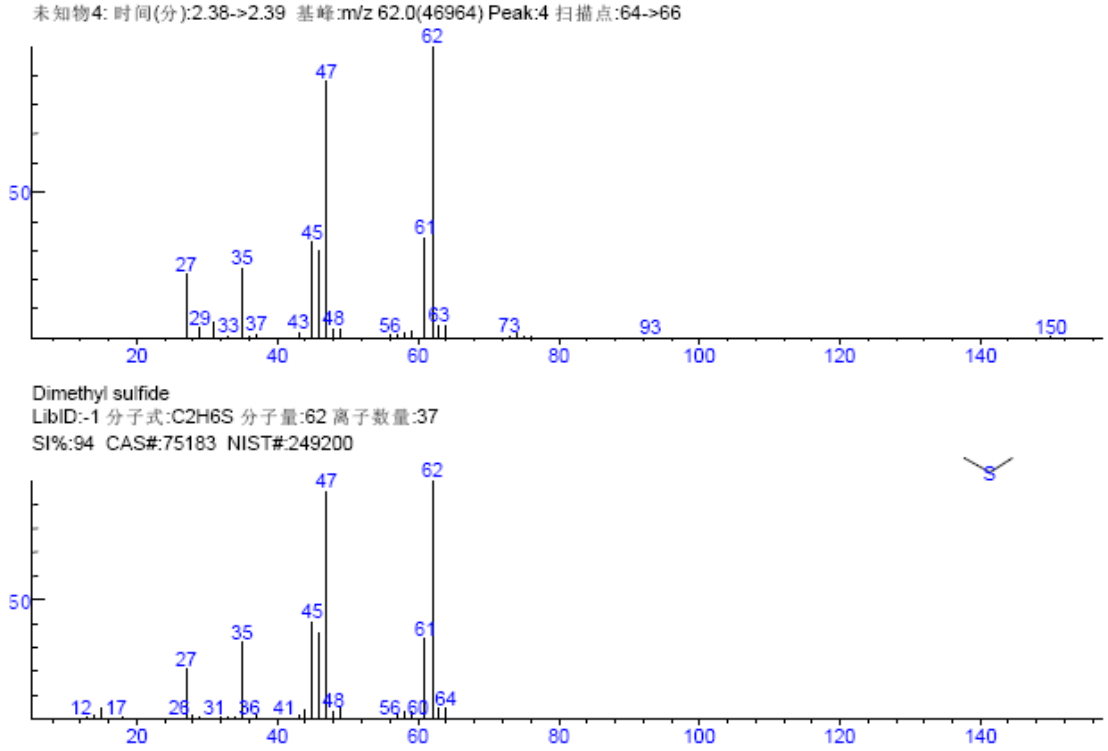

Figure 2 Mass spectra and search result of peak No. 4

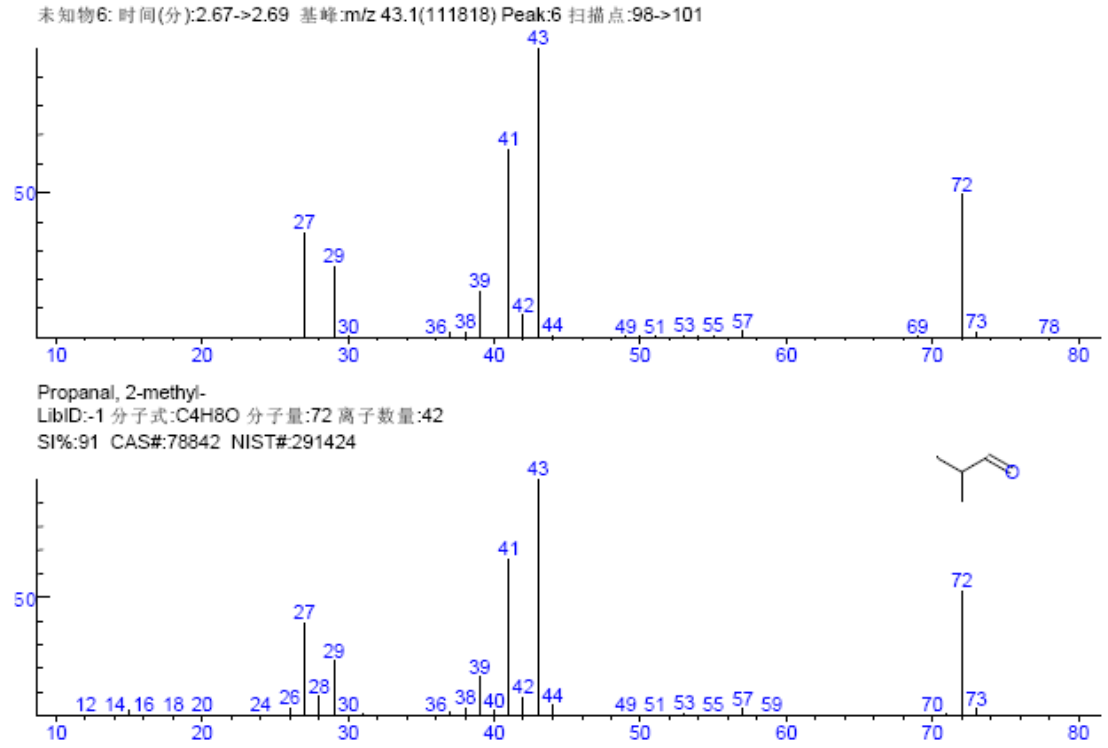

Figure 3 Mass spectra and search result of peak No. 6

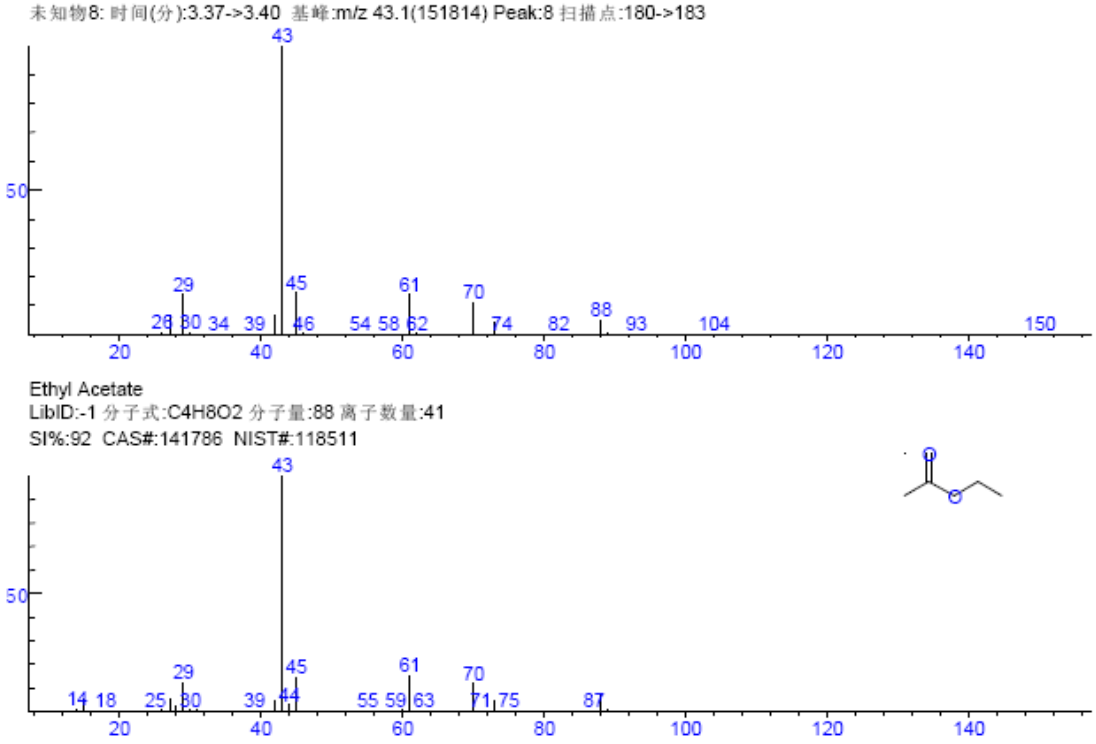

Figure 4 Mass spectra and search result of peak No. 8