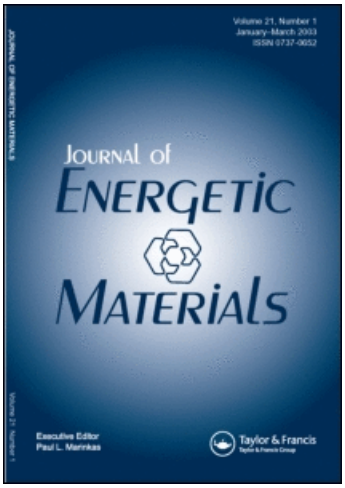

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QUANTITATIVE ANALYSIS OF TIN AND LEAD IN TDI COMBUSTION CATALYST

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ABSTRACT

A convenient method for quantitative analysis of tin and lead in 2,4-tolylene diisocyanate (TDI) combustion catalyst, which contains Tin, lead and their compounds or complexes, is described. The method consists of decomposition of samples by sodium hydroxide fusion, extraction with hydrochloric acid and complexation by EDTA, followed by titration with zinc chloride standard titration solution, which give rise to a precise content of tin and lead respectively in one conical flask.

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INTRODUCTION

Lead stannate TDI combustion catalyst has been recently developed as an important combustion catalyst used in rocket machine propellants. It is composed of carbon black, lead, lead oxides, tin dioxide and tin complexes. The tin dioxide, therein exists in β–SnO_2 and is hardly dissolved in any mineral acid even aqua regia. Previous methods[1-3] of determining β–SnO_2 consisted of fusion of samples with reductive metals and sodium hydroxide or carbonate, then extraction with hydrochloric acid and determination by iodimetry. Application of these methods to the lead stannate catalyst has been proved to be difficult because of ① an addition of reductive metal, such as zinc, will interfere with the analysis of lead; ② stannous cation is sensitive to oxygen and requires thoroughly protection during the titration. Establishment of a new analytical method to determine lead and tin precisely becomes an important subject for the research and application of this catalyst. Here we describe our method which can analyse the precise content of lead and tin in one conical flask with rapid and simple operation.

PROCEDURE

All the materials here used are analytical grade.

1. Weigh 1 g sodium hydroxide and put into the bottom of silver crucible, weigh 0.4 g sample (precision 0.0001 g) and put evenly into the crucible over the sodium hdroxide and cover with 1 g sodium hydroxide, cover the silver crucible then place into muffle furnace. Decompose at a temperature 700℃ for 1 h, wait for the crucible to be cool, place the crucible in a beaker. Add 50 ml of 1 + 1 hydrochloric acid, heat until the sample dissolve completely. Transfer the solution into a 200 ml volumetric flask, rinse the crucible and beaker 3 times with 1 + 1 hydrochloric acid, combine the solution and dilute with 1 + 1 hydrochloric acid to the graduation..

2. Pipette accurately 20 ml above solution and deliver it into a 250 ml conical flask, add 10.0 ml of 0.5000 mol / L EDTA standard solution, adjust the pH value at 2～3 with solid sodium hydroxide, add 10 ml of 40% hexamethylene tetramine solution, heat the solution until boiling.

3. Add 50 ml H_2O and 2～3 drops of xylenol orange indicater, titrate the excess EDTA with standard titration solution of zinc chloride. Record the volume V_1 at first end point where the yellow colour of the solution changes to red. Add 1.5 g of ammonium fluoride and heat again to about 40℃. Continue to titrate the solution with zinc chloride standard solution until the yellow colour of the solution changes again to red, record the volume of standard solution V_2.

DISCUSSION

Selection of Decomposing Condition

Lead stannate TDI combustion catalyst contains lead and tin, which both need to be determined accurately. The sample is decomposed with sodium hydroxide at high temperature (700℃) to give rise to carbon dioxide, lead oxide and tin dioxide, which is a stable state for tin to assure the precision of the determination and avoid troublesome protecting.

When dissolving the fusion residue, concentration of hydrochloric acid is important because an excess of chlorion shall avoid forming precipitate of lead chloride. Here 1 + 1 hydrochloric acid is shown to be suitable.

Selection of Titrating Condition

Adjust the pH value with solid sodium hydroxide to avoid forming a bulky volume of the solution because the sample solution contains a large excess of hydrochloric acid. Hexamethylene-tetramine is chosen as a buffer to control the pH at 5～6.

The excess EDTA can be titrated either by lead nitrate standard titration solution or by zinc chloride standard titration solution. Here zinc chloride standard solution is applied because lead nitrate will result slurry solution due to precipitate of lead chloride, which will affect the precision of analysis.

RESULTS

Calculation

The percentage of SnO_2 and Pb in the sample shall be calculated according following equations:

$$SnO_2\% = \frac{M_{Zn}(V_2 - V_1) \times 150.69 \times 10}{m \times 1000} \times 100$$

$$Pb\% = \frac{M_E V_E - M_{Zn} V_2 \times 207.2 \times 10}{m \times 1000} \times 100$$

where

M_E —— the concentration of EDTA standard solution, mol / L;

V_E —— The volume of EDTA standard solution used, ml;

M_{Zn} —— the concentration of $ZnCl_2$ standard titration solution;

V_1 —— the comsuption volume of $ZnCl_2$ standard titration solution at first end point;

V_2 —— the comsuption volume of $ZnCl_2$ standard titration solution at second end point;

m —— mass of the sample;

207.2 —— Pb atomic weight;

150.69 —— SnO_2 molecular weight;

10 —— 10–fold diluting.

Precision

The parallel datum from the same sample

Pb: 56.03, 55.94, 55.82, 56.09, 56.14, 55.95, 56.08, 56.06

Average value: 56.01

Standard deviation: 0.1039

Coefficient of variation: 0.18%

SnO_2 : 33.93, 34.02, 34.12, 34.65, 34.10, 34.12, 34.43, 34.04

Average value: 34.18

Standard deviation: 0.2404

Coefficient of variation: 0.7%

Recovery

$Pb(NO_3)_2$:	Add (g)	0.1032	0.1541
	Found (g)	0.1034	0.1536
	Recovery (%)	100.19	99.67
SnO_2:	Add (g)	0.0513	0.1042
	Found (g)	0.0501	0.1034
	Recovery (%)	97.66	99.23

CONCLUSION

The method described above is a precise and easy operating technique for quantitative analysis of lead and tin in the TDI combustion catalyst. It is very important from both research and manufacture of this new type of catalyst point of view. Moreover, the method is also applicable to other energetic materials which contain lead and tin compounds as combustion catalyst.

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