



Direct mercury determination in coal

ASTM D6722
EPA 7473

INTRODUCTION

Fossil coal contains toxic trace elements such as mercury, which is released into the air during coal combustion at plants and pollute the environment. In order to manage the stack gas cleaning process effectively, it is necessary to measure the mercury content in coal, as well as in stack gas, liquid and solid wastes.

The use of RA-915 series mercury analyzer equipped with the thermal decomposition facility provides fast direct determination of mercury in coal without chemical digestion of sample and intermediate amalgamation/sorption steps. Conventional methods of mercury determination in coal using atomic absorption spectrometry (ASTM D6414, ISO 15237, GB/T 16659, IS 12041) involve preliminary digestion of the sample that takes from 0.5 to 8 hours depending on the digestion conditions and consumes significant amount of reagents.

Other procedures for coal analysis (ASTM D6722, EPA 7473, DL/T 2296) involve thermal decomposition of the sample combined with amalgamation on a sorbent trap and catalytic conversion. Lumex Instruments proposes the simplified procedure of direct analysis without sample digestion and amalgamation.



MEASUREMENT METHOD

This method for mercury determination in coal is based on the direct thermal decomposition of a sample and subsequent mercury determination by differential atomic absorption spectroscopy. The interference from remaining impurity compounds is eliminated due to the high selectivity of the mercury analyzer with Zeeman background correction.

This approach does not involve preconcentration on a gold trap and «cooling step», thereby eliminating ensuing problems. The use of Zeeman background correction combined with a «dry» converter provides the highest sensitivity with no interferences from the sample matrix. The ambient air is used as a carrier gas, so that no cylinders with oxygen, argon, or other compressed gases are required.

ANALYSIS FEATURES

The proposed method of analysis by thermal decomposition has the following advantages compared to the common two-stage mercury determination (digestion + AAS or combustion + amalgamation + AAS):

- no laborious sample pretreatment with wet chemistry;
- direct mercury determination without preliminary accumulation on a gold /sorbent trap;
- low limit of detection, high selectivity;
- wide dynamic measurement range: more than 5 orders of magnitude;
- no «memory» effect;
- monitoring of the nonselective absorption during measurements;
- high analysis throughput (1–3 minutes per sample);
- no need for reagents and compressed carrier gas from a gas cylinder;
- low running cost;
- calibration and QA/QC with the certified SRM of any composition;
- possibility of mercury thermospeciation (the gradual heating of the sample for detection of mercury thermospecies in coal).

MEASUREMENT RANGE

Detection limit: **1 ppb (1 µg/kg)**.

Upper limit of the measurement range: **500 ppm (500 mg/kg)**.

MEASUREMENT PROCEDURE

Depending on the expected concentration of mercury in the sample, an appropriate heating mode is selected in the software (either slow or fast heating). The homogenized coal sample (50–500 mg) is placed into a sample boat and is inserted into the thermal decomposition chamber of the analyzer. The sample is heated at a temperature of 200–800°C depending on the selected operation mode. The mercury compounds are evaporated and partially dissociated, forming elemental mercury. All the gaseous products formed are transported into the second chamber of the atomizer by a carrier gas (Hg-free ambient air). Mercury compounds are totally dissociated. Downstream from the atomizer the gas flow enters the heated analytical cell, and the mercury atoms are detected.

EQUIPMENT AND REAGENTS

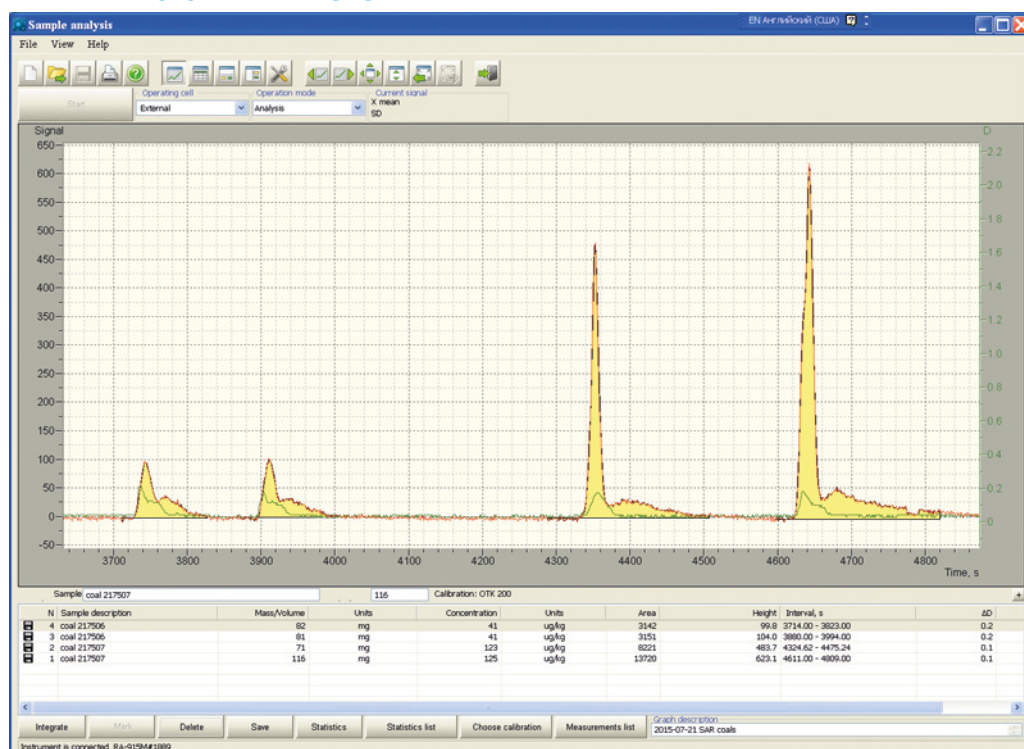
The following equipment and materials are used for analysis:

- RA-915M mercury analyzer combined with PYRO-915+ attachment;

or

- RA-915 Lab mercury analyzer;
- PC with Windows® and RAPID software;
- any solid or liquid certified SRM of mercury;
- Lumex Instruments kit, order № 0300003293.

EXAMPLES OF ANALYSIS



Samples:

coal sample 217506 ($m_1 = 82$ mg, $C_1 = 41$ ppb; $m_2 = 81$ mg, $C_2 = 41$ ppb), $C_{av} = 41$, RSD = 0%

coal sample 217507 ($m_1 = 71$ mg, $C_1 = 123$ ppb; $m_2 = 116$ mg, $C_2 = 125$ ppb), $C_{av} = 124$, RSD = 1%

