RAPID MERCURY ANALYSIS FOR THE FIELD: 
METHOD DEVELOPMENT AND APPLICATION TO NATURAL GAS 
UTILITY SITES 

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Abstract. A new technique based on traditional concepts has been developed for rapid, on-site 
analysis of mercury in environmental media. In this method, mercury is analyzed by integration of 
thermal decomposition, amalgamation, and atomic absorption spectrometry (TDA-AAS). Sample 
preparation and analysis are essentially integrated into a single instrumental system; solid samples 
can be analyzed directly, without chemical pre-treatment, in an analysis time of approximately 5 
minutes per sample. A wide range of standard reference material has been analyzed by TDA-AAS. 
Agreement with the certified values at the 95% confidence interval for all matrices tested validates 
this technique. Subsequent to validation, TDA-AAS has been used in a series of field studies in 
conjunction with remediation of mercury-contaminated soil at natural gas utility sites. Reasonable 
agreement has been demonstrated between TDA-AAS on-site results and laboratory results using 
conventional mercury analysis techniques. Independent laboratory confirmation of the field data is 
not required as TDA-AAS demonstrates lab-quality results on-site. This field technique has been 
shown to surpass traditional laboratory methods in terms of both precision and detection limits. A 
method for the United States Environmental Protection Agency (US EPA), Method 7473, has been 
developed and validated based on TDA-AAS methodology (US EPA, 1998).

Keywords: amalgamation, atomic absorption spectrometry, field analysis, mercury, thermal decom-
position

1. Introduction

A great deal of concern over mercury (Hg) pollution has been generated since the 
Minamata Bay tragedy of Japan in the 1950’s, where a total of 111 poisonings 
and 43 deaths resulted from consumption of Hg-contaminated seafood (Manahan, 
1994). The US EPA recognizes the environmental health threat posed by this heavy 
metal pollutant and accordingly, it is regulated by the Resource Conservation and 
Recovery Act (RCRA). A 1996 estimate of the annual anthropogenic emission of 
Hg was more than 216,000 kg (Johnson, 1997). A variety of sources, including 
power, pharmaceutical, pesticide, and mining industries, have contributed to Hg 
contamination of the environment. The natural gas utility industry has also added to 
the Hg problem by their use of Hg in monitoring and control instrumentation. 
Spills from damaged manometers, which may contain up to 4.5 kg of Hg each, have
left discrete but numerous polluted areas (Charlton et al., 1994). Due to the large
number of metering sites, the industry has recognized a need for rapid field analysis
for site characterization and/or remediation of Hg-contaminated soil (Foust and
Gui, 1997).

There are several analytical techniques applicable for the determination of mer-
cury in solid samples. Despite advances in analysis, cold vapor atomic absorption
spectrometry (CV-AAS), which was originally developed in 1968 by Hatch and Ott
(1968), remains the most commonly used method. With CV-AAS, solid samples
must be digested prior to analysis. The need for sample preparation has restricted
CV-AAS to use in the laboratory or a sophisticated mobile lab. While there are
techniques available for field analysis of Hg, problems limit their practicality in a
field setting. Static headspace analysis (SHSA) (Kriger and Turner, 1995), anodic
stripping voltammetry (ASV) (Foust and Gui, 1997), and enzyme-linked immuno-
osorbent assays (ELISA) (Bruce and Richards, 1998) have been used for on-site
analysis of Hg. These methods use highly corrosive acids or oxidizing solutions and
require ‘laborious sample preparation’ (Solc et al., 1997). In addition, the ELISA
method for Hg only provides semi-quantitative results. Hg testing in the field has
also been performed using a transportable energy-dispersive x-ray fluorescence
spectrometer. While this method is quantitative, disadvantages include greatly ex-
pensive instrumentation and relatively poor detection limits ($\approx 5 \text{ mg kg}^{-1}$) (Solc
et al., 1997). Other techniques, such as those which employ a gold film sensor for
mercury vapor analysis, ‘Jerome mercury vapor analyzer’, (Solc et al., 1997) and
those based on colorimetric ligand exchange reactions (Cherian and Gupta, 1990),
have been employed in the field, but for qualitative screening purposes only.

A method has been developed which addresses the problems associated with
many existing field techniques. This method, based on thermal decomposition,
amalgamation, and atomic absorption spectrometry (TDA-AAS), provides quant-
itive results on-site. Solid samples can be analyzed directly by TDA-AAS as
sample preparation is essentially integrated into the instrumental system. The error-
prone and labor-intensive steps associated with a discrete sample preparation stage
are eliminated. Hazardous chemicals for sample decomposition and/or extraction
are not needed, and therefore no waste is generated. In comparison with other field
techniques, the costs associated with TDA-AAS are reasonable.

Thermal decomposition, a technique in which samples are simply heated to
extreme temperatures (> 700 °C) for Hg release and subsequent analysis, is not
a new concept. This technique was introduced by Vaughn and McCarthy in the
1960’s for Hg analysis of ores and other geological materials (1966, 1964). Proce-
dures using thermal decomposition were first reported in the mainstream analytical
journals in the late 1960’s and early 1970’s (Aston and Riley, 1972; Kosta and
Byrne, 1969; Rook et al., 1972). Despite its simplicity, thermal decomposition
was not an accepted technique because of reported problems with reproducibility
and interferences. Almost 30 years later, this approach to Hg analysis has been
integrated into a dedicated instrumental system. The drawbacks associated with