Instrument Decision Criteria for Explosives Chemistry Analysis

Table of Contents

Instrument Decision Criteria for Explosives Chemistry Analysis

1 INTRODUCTION

This document provides general decision criteria for data generated by instruments used in explosives chemistry analysis. This document does not address identification requirements for specific products or classes of materials, but does provide guidelines for the acceptance of data to be used for qualitative identifications.

2 SCOPE

This procedure applies to caseworking personnel conducting work in explosives chemistry analysis.

3 PROCEDURE

The following criteria will be met for a qualitative identification to be made. The identity of a material will be confirmed by comparison to a reference or known material, if available. Reference or known materials may be run concurrently with an unknown sample or may be previously analyzed on the instrument under the same parameters. All results will be verified using orthogonal techniques or alternate methods, if available.

When a reference or known material is not available or when only reference data (e.g., from scientific literature, publications, or an instrument library) is used, a material may be reported as "consistent with" a substance.

3.1 Chromatography

Peaks should show good chromatographic characteristics with reasonable peak shape, width, and resolution.

The retention time of the peak of interest should be within ±2% for gas chromatography (GC) and ±5% for liquid chromatography (LC) when compared to a contemporaneously analyzed reference or known material.

The baseline signal-to-noise ratio (SNR) for an analyte will be greater than three to one (3:1) to be considered a peak. The noise within a chromatogram will be identified as a region within the data that contains a representative baseline (a signal consisting of consistent height and no identifiable peaks). Peak-to-peak variation (maximum minus the minimum) will be selected for noise calculation. The analyte of interest must have a peak that is three times or greater than the calculated noise. Refer to the Explosives Level 4 Documents for instrument software parameters and calculations.

The peak area for an analyte will be at least ten times greater than the peak area of any carryover or system peaks which may have been present in preceding analyses (e.g., blanks or negative controls) to be considered present within a sample.

When utilizing peak area to intercompare samples or controls, check the integrated area of the peak to ensure that it is only calculated for the analyte peak of interest (i.e., not including smaller baseline peaks before or after the analyte peak).

3.2 Mass Spectrometry (MS)

A mass tolerance of ±0.5 m/z (mass-to-charge) for low resolution instruments (e.g. quadrupole, ion trap mass spectrometers) will be used when comparing an analyte of interest with that of a contemporaneously analyzed reference or known material. Extracted ion chromatograms may be used to facilitate detection of a target analyte prior to comparison of the mass spectrum with that of a reference or known material.

3.2.1 Accurate Mass Spectrometry

Ions in an unknown measured accurate mass spectrum should be ±0.005 m/z of the ions in the known measured accurate mass spectrum, or the theoretical m/z (i.e., exact mass). Any isotope of a molecular (or pseudo-molecular) ion may be considered diagnostic if it meets the ±0.005 m/z criterion. One additional adduct ion, beyond the pseudo-molecular ion, may also be considered diagnostic if it meets the ±0.005 m/z criterion.

When using the ultra performance liquid chromatograph with mass spectrometer (UPLC/MS) for accurate mass analysis, the target analytes should be observed within the range below from their expected monoisotopic masses:

Redacted

3.3 X-ray Diffraction (XRD)

The diffraction patterns from the questioned component should compare favorably to the corresponding reference or known material.

If the identity of an unknown material is matched through a library search, a reference or known material may be analyzed for comparison, if available. Results should be confirmed through orthogonal techniques such as Fourier transform infrared spectroscopy (FTIR), Raman spectroscopy, scanning electron microscopy with energy dispersive X-ray spectroscopy (SEM/EDS), or MS, when possible.

3.4 SEM/EDS

Peaks in the EDS spectrum should exhibit a Gaussian peak shape. Elemental assignments made by the software should be verified by the individual conducting the exam. Spectral artifacts, such as sum peaks and escape peaks, can occur with high intensity peaks and should be considered when verifying elemental assignments.

Escape peaks occur when a Si X-ray produced by fluorescence escapes the detectors resulting in an artificial peak at an energy (keV) equal to the parent peak minus 1.74keV (Kα for Si).

Sum peaks occur when two X-rays from a sample hit the detector simultaneously resulting in an artificial peak at an energy of the sum of those X-rays.

3.5 Other Tests

The results of all tests (e.g., visual inspections, FTIR, Raman, pH) should compare favorably to the corresponding reference or known material. Spectral subtraction may be useful for assessing mixtures.

4 LIMITATIONS

These criteria are not meant to be all-inclusive. Known limitations for specific analytes will be documented in the applicable subdiscipline technical procedures.

5 REVISION HISTORY

Revision	Issued	Changes
00	09/30/2022	Original document issued.