Instrument Parameters

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Instrument Parameters

1 INTRODUCTION

This document provides parameters for commonly used instruments in General Chemistry analyses and is to be used in conjunction with General Chemistry, Instrument Operation and Systems Support (IOSS), and Metallurgy technical procedures. Any specific instrument parameters or decision criteria in individual technical procedures will override those set forth in this document.

2 SCOPE

This document applies to CU personnel that are qualified and authorized to examine General Chemistry evidence.

3 EQUIPMENT

- Fourier Transform Infrared (FTIR) spectrophotometer with Attenuated Total Reflectance (ATR), transmission, or microscope attachments
- Raman spectrophotometer with microscope and/or bench capabilities
- Time-of-flight mass spectrometer with direct analysis in real time ionization source (DART/TOFMS)
- Gas chromatograph/mass spectrometer (GC/MS) equipped with an electron impact ionization source and a 30 meter DB-5 column (or equivalent)
- GC/MS equipped with a chemical ionization source and a 30 meter DB-5 column (or equivalent)
- GC/MS equipped an electron impact ionization source and a headspace autosampler (HS-GC/MS) and a 30 meter DB-624 column (or equivalent)
- Gas chromatograph equipped with a flame ionization detector (GC-FID), a headspace autosampler (HS-GC-FID) and a 30 meter Restek Rtx-BAC-2 column (or equivalent)
- Gas chromatograph equipped with a nitrogen/phosphorus detector (GC-NPD), a headspace autosampler (HS-GC-NPD) and a 30 meter Restek Rt-QS-Bond column (or equivalent)
- GC-NPD equipped with a 30 meter Rtx-1701 column (or equivalent)
- Gas chromatograph equipped with an electron capture detector (GC-ECD) and a 30 meter Rtx-CLPest column (or equivalent)
- High-temperature GC-FID equipped with a 15 meter Zebron “Inferno” ZB-1HT column (or equivalent)
- Liquid chromatography system with appropriate column coupled to a mass spectrometer (LC/MS) with electrospray ionization (ESI) or atmospheric pressure chemical ionization (APCI) (e.g., Thermo LTQ, Thermo LTQ OrbiTrap XL, Thermo Exactive OrbitTrap)
- Ion chromatograph for anions
- Ion chromatograph for cations
- Scanning electron microscope with energy dispersive X-ray spectrometer (SEM/EDS)
- X-ray powder diffractometer (XRD)

- Thermo QUANT'X X-ray Fluorescence Spectrometer (XRF)
- Bruker M4 Tornado Micro X-ray Fluorescence Spectrometer (micro-XRF)
- Ultraviolet-Visible spectrophotometer (UV-Vis)

4 INSTRUMENT PARAMETERS

The following instrument parameters are not intended to be prescriptive nor exhaustive. Minor modifications to the conditions may be used as needed and without authorization, provided the same parameters (or similar parameters for some techniques, e.g. SEM/EDS) are used for all applicable solvent blanks, control samples, and questioned items; and the Positive Control(s) provide acceptable data. The utilized parameters will be recorded and retained with the case notes.

4.1 FTIR

	ATR	Transmission	Microscope
No. of scans	32	32	128
Resolution	4	4	4
Final format	% Reflectance	% Transmittance	% Transmittance
Correction	None	None	None

4.2 Raman

	Microscope and Bench
Collect exposure time (sec)	10.0
Preview exposure time (sec)	1.0
Sample exposures	4
Background exposures	4
Final format	Shifted spectrum (cm^{-1})
Correction	White light
Cosmic ray threshold	Low

4.3 UV/Vis

Wavelength scan mode	
Start wavelength	800 nm
End wavelength	220 nm
Integration time	0.25 sec
Data interval	0.5 nm
Scan speed	120 nm/min

4.4 SEM/EDS

SEM Settings (record the following SEM parameters)	
Accelerating voltage	(typically 25.0 kV)
Imaging mode	(SEI or BEI, at minimum)
Magnification	
Working distance	(~13 mm ideal for EDS)
EDS Settings	
Adjust settings to achieve sufficient count rate and collection time	Impacted by combination of above SEM settings as well as beam current (i.e., "spot size"), amp time, and detector type (SiLi vs. SDD)

4.5 XRF

Refer to METAL-410, METAL-411, and METAL-412 for XRF parameters.

4.6 XRD

	Miniflex 6G
X-ray source	40 kV, 15 mA
Scan mode	1D(scan)
Scan speed	20 deg/min
Step width	0.02 deg
Scan range	5-80 deg

4.7 GC/MS

4.7.1 Electron Impact (EI)

GC Settings	
Injection mode	Split or Splitless
Injection volume	1 uL
Inlet temperature	250 °C
Oven program	60 °C for 2 min, 35 °C /min to 260 °C for 15 min
Full Scan MS Settings	
Polarity	Positive
Solvent delay	3.00° min
Scan range	43 to 400 *m/z*

4.7.2 Chemical Ionization (CI)

GC Settings	
Injection mode	Split or Splitless
Injection volume	1 uL
Inlet temperature	250 °C
Oven program	60 °C for 2 min, 35 °C /min to 260 °C for 15 min
Full Scan MS Settings	
Polarity	Positive or Negative
Solvent delay	3.00 min
Reagent gas	Methane
Scan range	100 to 400 *m/z*

4.8 GC-ECD

GC Settings	
Injection mode	Splitless
Injection volume	1 uL
Inlet temperature	230 °C
Oven program	125 °C for 1 min, 7 °C /min to 280 °C for 22 min
ECD Settings	
Temperature	300 °C
Makeup gas	Nitrogen
Makeup flow	30 mL/min

4.9 GC-NPD

GC Settings	
Injection mode	Split
Split ratio	15:1
Injection volume	1 uL
Inlet temperature	250 °C
Oven program	125 °C for 1 min, 7 °C /min to 280 °C for 22 min
NPD Settings	
Temperature	250 °C
Offset	10
Makeup gas	Nitrogen
Makeup flow	30 mL/min
Air flow	60 mL/min
Hydrogen flow	2 mL/min

4.10 High Temperature GC-FID

Oven program	55 °C for 2 min, 30 °C /min to 100 °C for 0 min, 15 °C /min to 400 °C for 3.5 min
PTV Inlet	
Injection mode	Splitless
Injection volume	1 uL
Initial temperature	55 °C
Ramp	500 °C to 400 °C for 10 min
Pressure	7 psi
Total flow	34 mL/min
FID Settings	
Temperature	420 °C
Mode	Constant makeup flow
Hydrogen flow	40.0 mL/min
Air flow	450.0 mL/min
Makeup flow	30.0 mL/min

4.11 Headspace GC/MS (EI)

GC Settings	
Injection mode	Split
Split ratio	10:1
Inlet temperature	150 °C
Oven program	50 °C for 3 min, 10 °C /min to 250 °C for 21.5 min
Headspace Autosampler Settings (Maestro)	
Incubation time	30 min
Incubation temperature	80 °C
Syringe	2.5 mL-HS
Syringe temperature	90 °C
Injection volume	1000 uL
Full Scan MS Settings	
Polarity	Positive
Solvent delay	1.75 min
Scan range	27 to 400 *m/z*

4.12 Headspace GC-FID

GC Settings	
Injection mode	Splitless
Oven program	50 °C for 3 min, 10 °C /min to 250 °C for 21.5 min
Headspace Autosampler Settings (Maestro)	
Incubation time	30 min
Incubation temperature	80 °C
Syringe	2.5 mL-HS
Syringe temperature	90 °C
Injection volume	1000 uL
FID Settings	
Heater temperature	250 °C
H_2 flow	40 mL/min
Air flow	400 mL/min

4.13 Headspace GC-NPD

GC Settings	
Back SS Inlet N2	
Injection Mode	Split
Split ratio	10:1
Inlet temperature	110 °C
Oven program	110 °C for 0 min, 4 °C /min to 130 °C for 10 min
Headspace Autosampler Settings (Maestro)	
Incubation time	5 min
Incubation temperature	45 °C
Syringe	2.5 mL-HS
Syringe temperature	55 °C
Injection volume	250 uL
NPD Settings	
Maximum bead voltage	4.095 V
Dry bead	Yes
Heater	225 °C

4.14 LC/MS

LC Settings	
Column dimensions	Length 150 mm, Diameter 2.1 mm
Particle size	5 um
Column oven temperature	30 °C
Flow rate	0.3 mL/min
MS Settings	
Ionization mode	ESI
Sheath gas	20
Aux gas	10
Sweep gas	5
Capillary temperature	275 °C
Scan mode	Full
Scan range	100-650 *m/z*
High Resolution MS Settings	
Resolving power	30,000

4.15 DART/TOFMS

DART Source Settings	
Polarity	Positive or Negative
Source gas	Helium
Temperature	400 °C

TOFMS Settings	
Polarity	Same as DART Source
Ion guide RF (or equivalent)	800 V for 80-800 *m/z* scan range, 500 V for 50-500 *m/z* scan range

4.16 Ion Chromatography

4.16.1 Cations

	Waters	**Dionex**
Mobile phase	3.0 mM HNO_3/0.1 mM EDTA	20 mM methanesulfonic acid
Pump mode	Isocratic	Isocratic
Flow rate	1.0 mL/min	1.0 mL/min
Column	Waters IC-Pak C M/D	Dionex IonPac CS12A
Column temperature	Ambient	30 °C
Injection volume	10 uL	25 uL
Acquire time	15 min	15 min
Detector	Waters 432 conductivity detector	Suppressed conductivity detector

4.16.2 Anions

	Dionex (KOH)	Dionex (K_2CO_3)
Mobile phase	20 to 80 mM potassium hydroxide	10 mM potassium carbonate
Pump mode	Multi-step gradient 20 mM at 0 min 20 mM at 2 min 30 mM at 9 min 80 mM at 13 min 80 mM at 21 min 20 mM at 21.1 min 20 mM at 25 min	Isocratic
Flow rate	1.0 mL/min	1.5 mL/min
Column	Dionex IonPac AS19 with IonPac AG19 guard	Dionex IonPac AS22 with IonPac AG22 guard
Column temperature	30 °C	30 °C
Injection volume	25 uL	25 uL
Acquire time	25 min	16 min
Detector	Suppressed conductivity detector	Suppressed conductivity detector

5 LIMITATIONS

The following criteria are used as guidelines in determining the acceptability of data. Any specific criteria in individual General Chemistry technical procedures will override the below criteria.

5.1 Chromatography

- When compared to a contemporaneously analyzed reference material or Positive Control, the retention time of a GC peak should be ± 0.15 min (9 seconds) for a peak with a retention time < 7.50 min or ± 2% for a peak with a retention time ≥ 7.50 min.
- The retention time of a LC peak should be within ± 5% of a contemporaneously analyzed reference material or Positive Control.
- The signal intensity for a peak should be at least 10x greater than the intensity from any carryover peak which is present in the preceding blank or the Negative Control.

5.2 Mass Spectrometry

- The mass spectrum of the analyte of interest should compare favorably with that of a contemporaneously analyzed reference material or Positive Control. See GENCHEM-303 for further guidance.
- DART/TOFMS peaks of interest should be within ± 0.005 *m/z* of a contemporaneously analyzed reference material or Positive Control, and/or the theoretical accurate mass value of the ion of interest.

5.3 SEM/EDS and XRF

The instrument software and KLM reference lines are used to label any peaks that are present in a collected spectrum using the following systematic approach:

- Label any high-energy peaks and major peaks. High-energy peaks are less likely to have overlapping peaks when compared to low-energy peaks. If a major peak is present, then a complete family of peaks can generally be identified.
- Major peaks may give rise to spectral artifacts, such as sum peaks and escape peaks. Check the spectrum for the presence of these artifacts and label any that are identified. Keep in mind that sum peaks may arise from more than 1 element if multiple major peaks are present.
- Attempt to label any remaining, lower intensity peaks. This may require adjustment of the vertical scale to reveal the necessary detail. Inconsistent peak ratios (when compared to reference lines) or asymmetric peaks may indicate peak overlap. Weak intensities and/or peak overlap may prevent identification of an element within the spectrum.
- The presence of an element may be considered unequivocal only when a characteristic, unique set of lines is produced, or when a single peak occurs at an energy where it cannot be mistaken for another element or a spectral artifact. Unequivocal identification may not be possible if an element is present at a low concentration and/or if its lines overlap with signals from other elements present in the sample.
- Unequivocal elemental assignments are represented by labeling the peak with the elemental symbol.
- Probable (i.e., not unequivocal) elemental assignments are represented by labeling the peak with parentheses around the elemental symbol.
- Escape peaks may be represented by the addition of "- Si" to the elemental symbol (e.g. Cl – Si), or simply by annotating as "Escape" or similar.
- Sum peaks are represented by the "+" sign (e.g., C + O).
- Possible peaks that are too weak to assess are labeled as "?" or similar.
- Additional information may be included with peak labels (e.g., series and shell).

5.4 All Other Data

- Data from the analyte of interest should compare favorably with that of a contemporaneously analyzed reference material, Positive Control, and/or library entry.
- The signal intensity for a peak should be at least 10x greater than the intensity of a corresponding peak which is present in a preceding blank or the Negative Control.

6 REVISION HISTORY

Revision	Issued	Changes
02	05/06/2022	Revised to match new format requirements. Added content to Section 3.4. Removed Miniflex II and Miniflex 600 from Section 3.6, added Miniflex 6G and updated parameters.
03	06/30/2023	Removed split ratio from Section 4.12. Revised Section 4.13 to reflect change to a split-splitless inlet. Removed first bullet and revised GC retention time criteria in Section 5.1. Changed reference standard to reference material throughout.