

Automated, High-Performance Direct Mercury Analysis

- USEPA 7473
- ASTM D 6722-01
- ASTM D 7623-10
- UOP 1009-15
- JIS K0102



**Thermal Decomposition
Mercury Analyzer**



A Rigaku
Company

MA-3000 Direct Combustion Hg Analyzer



AUTOMATED DIRECT MERCURY ANALYSIS

The applications and demand for thermal decomposition mercury measurements are expanding due to the amendment of laws and regulations regarding mercury measurement. In response to this trend, Nippon Instruments Corporation developed the MA-3000 automated thermal decomposition mercury analyzer to meet the needs of tomorrow with best in class sensitivity, reliability and flexibility.

WHY USE THERMAL DECOMPOSITION ANALYSIS?

Reducing vaporization mercury measurement requires acid pretreatment, which makes accurate measurements of some samples quite difficult due to the influence of interferences or to mercury vaporizing during acid decomposition. Thus it requires expertise to monitor and review pretreatment conditions.

Thermal decomposition mercury analysis does not require any such troublesome acid pretreatment, and therefore lets inexperienced users easily perform measurements. In addition, this analysis method is earth-friendly because no acidic effluent is discharged.

NIPPON INSTRUMENTS CORPORATION

Since being established in 1978, Nippon Instruments Corporation has specialized in analyzing mercury. Efforts to control mercury and prevent the spread of any damage to human health resulting from mercury have just commenced on a global basis. This has led to an increasing awareness of the significance of accurately measuring and controlling mercury. Nippon Instruments Corporation is committed to making a contribution to customers through mercury measurements as indicated by our corporate philosophy to "Contribute to the Development of Human Society through Advances Made in Scientific Technology".

COMPACT, BENCHTOP Hg ANALYZER

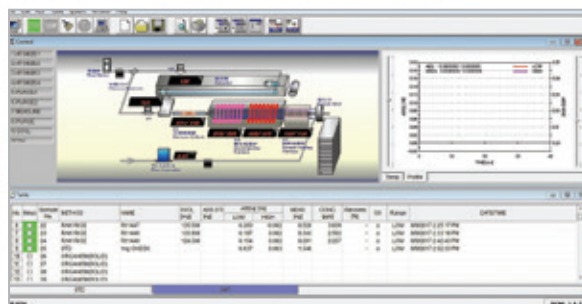
Sensitivity. MA-3000 has the lowest detection limit and practical quantitation limit available for a commercial direct combustion mercury analyzer. For a 200 mg real world sample, this would equate to low parts per trillion detection limits. The unique optical system achieves a seamless low to high measurement range without cross-range interference. With an ultra wide dynamic range, MA-3000 reliably covers more types of applications and sample matrices.

Reliability. The unique optical system achieves seamless low to high measurements without cross-range interference. With an ultra wide dynamic range, MA-3000 reliably covers more types of applications and sample matrices. The high-capacity automatic sample changer is controlled by Microsoft Windows based software with numerous advanced features and 21 CFR part 11 compliance.

Flexibility. For maximum flexibility, the analyzer is optionally available with accessories for Reduction Vaporization and Gas Tube Desorption analysis, allowing a single analyzer for multiple analytical techniques.

INTUITIVE WINDOWS® BASED SOFTWARE

Suitable for non-technical operators, MA-3000 software runs under the Windows operating system and is 21 CFR Part 11 compliant.



VISCIOUS HYDROCARBONS AND RESIDUUMS

By purchasing the MA-3000 in combination with the petro-pyrolysis mercury analyzer PE-1000 for the analysis of light petroleum fractions, a wider range of petro samples – including heavy oils, sludges and wastewater – may be analyzed. This can significantly increase operational efficiency.

REDUCE WORKFLOW BOTTLENECKS

For certain complete unknowns, measured with the reducing vaporization technique, there can be a need to determine decomposition conditions. These extra steps can create measurement bottlenecks. Therefore, the direct combustion approach, as employed by the MA-3000, is an ideal complementary technique for handling all the solid, non-aqueous liquids and particulate-laden liquid matrices, where chemical preparation for such matrices is more complicated and tedious.



HOW IT WORKS

A weighed sample, in a boat, is automatically introduced into MA-3000. As oxygen flow begins, the decomposition furnace temperature is increased in stages; first to dry the sample, then to decompose it. Evolved gases are carried through a heated catalyst to produce free mercury. Combustion products are swept through a gold amalgamation trap where elemental mercury is concentrated. The trap is then heated to release the mercury into a carrier gas which transports it into the atomic absorption spectrometer where mercury is simultaneously measured in long and short paths.

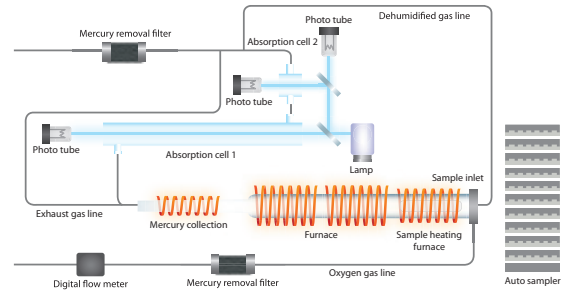


Figure 1. Schematic of thermal decomposition method

The NIC MA-3000 works on the principle of cold vapor atomic absorption spectroscopy, where monochromatic light at a wavelength of 253.7 nm is attenuated by mercury vapor in a measurement cell according to the Beer-Lambert Law: Absorbance is equal to the molar absorptivity times the concentration times the path length. In the simplest case, since epsilon and path length are constant, absorbance is proportional to concentration.

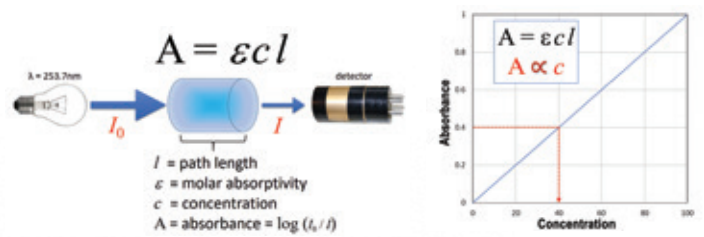
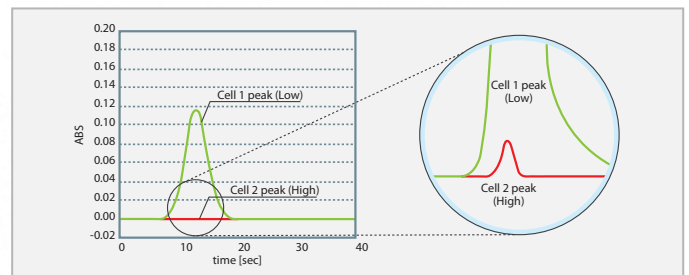
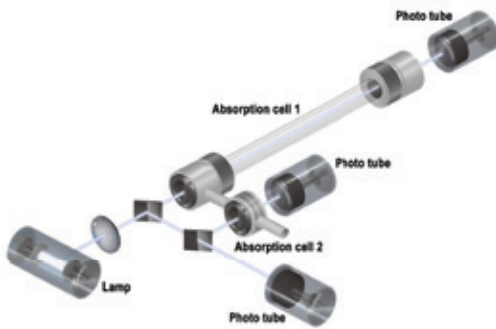


Figure 2. Cold vapor atomic absorption spectroscopy

DUAL-CELL TRI-BEAM

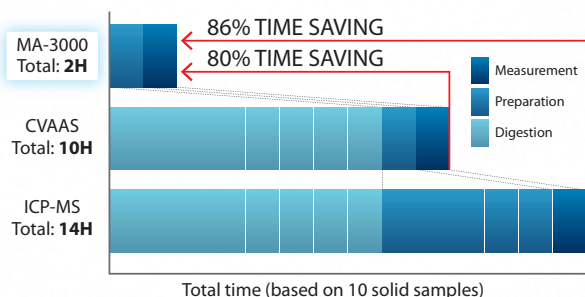
The patented dual-cell tri-beam detector (JP 5596995) achieves a seamless low to high measurement range without cross-range interference. With a dynamic range of up to 70,000 ng, or up to 1000 ppm, MA-3000 reliably covers a wide variety of applications and sample matrices.



MA-3000 peak profile

PERFORMANCE EXAMPLES

A proprietary patented (JP 5001419) combustion tube, with a robust catalyst formulation, extends analysis applications to include samples with complicated matrices, including those with halogens and high selenium. This design delivers exceptional long term stability, low operating cost and minimal maintenance. The data shown illustrates the excellent comparability to other mercury methods as well as the analysis precision for repeated measurements.



Results of standard reference materials

Sample name	Certified values (Total Hg)	N	AV (mg/kg)	CV (%)	Recovery (%)
Coal (NIST 1632c)	0.0901~0.0975 mg/kg	5	0.0954	2.8	99
Dogfish liver (DOLT-4)	2.36~2.80 mg/kg	5	2.59	3.2	100

Results of different samples

Sample	Sample name	Sample size (mg)	Results		STD addition	
			AVE	CV (%)	Recovery (%)	CV (%)
Salt	NaCl (for experiment use)	190~220	3.81 µg/kg	5.3	102	1.2
High Se containing sample (Se is contained 34~46 wt%)	NIST 1515* +SeO ₂	50~60	0.0453 mg/kg	1.9	*NIST 1515 0.044±0.004 mg/kg	

MA-3000 measures samples without tedious and cumbersome acid/alkali pretreatment. It uses environmental-friendly technique that eliminates hazardous operation. Typical measuring time is as little as 5 minutes. Total running cost saving compared to CV-AAS and ICP-MS is 80~86%.

MA-3000

Key Features and Benefits

■ LOWEST DETECTION LIMITS

MA-3000 has the lowest detection limit, and practical quantitation limit, available for a direct (combustion) mercury analyzer. Method Detection Limit (MDL) is less than 0.001 ng (< 1 pg).

■ ADVANCED OPTICS FOR WIDEST DYNAMIC RANGE

Unique dual sequential cell, triple beam detector optics (Patented JP 5596995) achieve a seamless low to high measurement range without cross-range interference. With a dynamic range of up to 70,000 ng, or up to 1000 ppm, MA-3000 reliably covers a wide variety of applications and sample matrices, offering unmatched versatility.

■ SUPERIOR SENSITIVITY AND STABILITY

Employing a high-quality thermally stabilized Hg-discharge lamp that emits a very strong, stable line emission at 253.7 nm eliminates the need for any intensity-robbing optical filtering of the source. Three photomultiplier tube detectors provide high sensitivity high range, low range and reference measurements, ensuring ultra stable operation. Other systems use inexpensive 50% transmittance mirrors to redirect the light source, but this reduces the intensity, lowering sensitivity. Instead, MA-3000 uses high quality optical gratings to redirect the full intensity through the sample cell and to the reference detector, for superior sensitivity.

■ ADVANCED AUTOMATION WITH 100-POSITION AUTOSAMPLER

Unique multi-tier automatic sample changer is typically configured with ten trays each holding ten ceramic sample boats. Boat capacity is up to 1,500 mg or 1,600 μ L of sample. Operation is as simple as weighing out a sample into a boat and loading it into a tray and then into the instrument.



■ REAL TIME CONTINUOUS DIAGNOSTICS

MA-3000 software continuously monitors key diagnostics, such as all heater temps, flow rates, valve actuations, and voltages. This allows the software to automatically prompt the user if there is an issue, and it also allows for quick and easy troubleshooting for maximum uptime and reliability.

■ SPACE EFFICIENT BENCHTOP DESIGN

Engineered for modern laboratory design, the MA-3000 is 50% smaller than the previous model. It can run on any standard single-phase wall power, from 100 to 240 VAC (50/60 Hz).

■ 21 CFR PART 11 WINDOWS BASED SOFTWARE

Running under the Windows operating system, the MA-3000's modern software is intuitive to use and provides a real-time graphical representation of the measurement status. The intuitive graphical interface (GUI) comes with numerous beneficial features and 21 CFR Part 11 compliance to improve laboratory productivity.

■ SUPERIOR CATALYST AND GOLD AMALGAMATOR

MA-3000's unique combustion tube catalyst (Patented JP 5001419) and gold traps have such long lifetimes and superior designs that have better tolerance to difficult matrices (containing halogenated and selenium compounds). Extremely stable calibration curves generated for each catalyst will typically continue with the same level of response right up until it must be replaced (9–12 months later, depending on usage).

■ APPLICATIONS

Thermal decomposition: Sediment, soil, food, biological tissue, blood, urine, ore, coal, plastics, crude oil, wastewater, and more.

■ TEST METHODS

USEPA 7473; ASTM D 6722-01; ASTM D 7623-10; UOP 1009-15; JIS K0102.

■ OPTIONAL ATTACHMENTS ADD VERSATILITY

Both automatic reduction vaporization (SC-5 and RD-5) and gas analysis attachments are available to make your MA-3000 even more productive.

■ BEST-IN-CLASS-ENGINEERING

In summary, better optics, better components, and a better design are the hallmarks of all Nippon Instrument's direct combustion mercury analyzers dating back to the 1970s. Being dedicated to only mercury analysis makes NIC the first choice for your mercury analysis needs.



CORK STOPPERS | MA-3A-CA-101

In a 2014 study (Lopes, C.B., et al. *Environ Sci Pollut Res* (2014) 21: 2108), it was shown that stopper-derived cork is an effective biosorbent towards bivalent mercury at environmentally relevant concentrations and conditions.



COAL | MA-3A-EG-001

Since mercury occurs naturally in coal and other fossil fuels, when these fuels are burned for energy, the mercury becomes volatilized and airborne into the atmosphere.



CRUDE OIL | MA-3A-EG-101

While the low mercury average levels found in crude oil (ca. 3.5 µg/kg) do not seem to represent an environmental hazard, the refining process tends to concentrate and collect the mercury components and direct the emissions to air release, petroleum products and waste products.



SHALE OIL | MA-3A-EG-103

Research has shown that many shale deposits are high in mercury content and that significant quantities of mercury can be released during oil shale processing.



SEDIMENT | MA-3A-EN-001

Sediment mercury is mercury that has become embedded into the bottom substrates of aquatic ecosystems.



ATMOSPHERIC AIR | MA-3A-EN-201

Atmospheric elemental mercury, although present only in trace amounts, has been established as a significant source of Hg to aquatic environments.



DRIED KELP | MA-3A-FD-002

Kelps have been found to be highly absorptive of mercury from aqueous solutions. Also, high levels of mercury in kelp supplements have been widely reported.



HIJIKI (SEAWEED) | MA-3A-FD-004

Edible seaweeds of all kinds contain 1–50 parts per billion (ppb) of mercury.



SHARK LIVER OIL | MA-3A-FD-010

Bioaccumulation in shark liver oil carries over into human populations, where it can result in mercury poisoning.



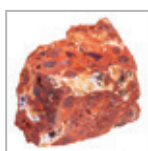
TUNA LEAN | MA-3A-FD-013

Fish and shellfish concentrate mercury in their bodies, often in the form of methylmercury, a highly toxic organic compound of mercury.



COPPER ORE | MA-3A-IN-003

Mercury occurs in elemental form as a natural amalgam in native metals like copper.



BAUXITE | MA-3A-IN-001

The mercury content of bauxite ore can vary significantly, in the range of 20–2000 ppm.



SILICA | MA-3A-IN-004

Silica mining and production can be contaminated with naturally occurring forms of mercury found in the abiotic environment, including: metallic mercury, mercuric sulfide, and the salts mercuric chloride and mercurous chloride.



SULFIDE | MA-3A-IN-007

When rain falls on sulfide ore waste, sulfuric acid is produced. Sulfuric acid leaches out metals and chemicals from the waste and creates acid mine drainage, which contaminates lakes, rivers, and groundwater with mercury and mercury compounds.



SLUDGE | MA-3A-IN-008

Sludge is typically a soup-like material containing significant quantities of interstitial water and is often contaminated with heavy metals including mercury.



CALCIUM FLUORIDE | MA-3A-IN-009

Fluorite (the mineral form of calcium fluoride) can be contaminated with mercury and processing and use of fluorite can lead to environmental mercury contamination.



WHOLE BLOOD | MA-3A-SC-007

Elevated mercury in blood usually indicates exposure to organic mercury (usually in the form of methylmercury) or recent exposure to a high level of elemental mercury vapor.



Specifications		
Mercury detecting section	Measurement principle	Non-dispersive dual-cell tri-beam CVAAS PATENTED (JP NO. 5596995)
	Wavelength	253.7 nm
	Detectors	Photo tubes (Reference-background; Absorption cell 1; Absorption cell 2)
	Detection limit	< 1 pg (< 0.001 ng)
	Maximum measurement range	70,000 ng ¹
	Measuring time	Approx. 5 minutes
Sample decomposition section	Combustion tube	Quartz (filled with catalyst) PATENTED (JP NO. 5001419)
	Sample boat	Ceramic 39 × 11 × 7.2 (mm) (standard) / 39 × 11 × 11.5 (mm) large (optional)
	Maximum decomposition temperature	Up to 1000°C
Auto sample changer section	Sample position	100 positions
	Sample boat capacity	Solid: Up to 1,500 mg Liquid: Up to 1,600 µL (depends on sample matrices/boat type)
Data processing system (PC-Win / MA-3000) 21 CFR Part II compliance	OS	Windows 7, 8, and 10
	Display	Peak profile, calibration, raw data, statistics, flow graphics and status
	Device control	Analysis condition (temperature, decomposition time); blank check; standard check
	Data processing	Calibration curve (linear and cubic fits); peak height and peak area tabulation; simultaneous LOW and HIGH concentration tabulation; unit selection; concentration conversion; baseline correction; statistics (mean value; standard deviation; CV%)
	Printing	Memo; analysis condition; calibration curve; measured and statistical data; peak profiles
Dimensions and utility	Dimensions	430 W × 770 D × 470 H (mm)
	Weight	55 kg
	Power supply	AC100~240 V, 15A (MAX)
	Power capacity	1.5 KVA
	Gas	Oxygen (>90% purity), 0.1~0.29 MPa (or compressed purified air – depending on the application)

¹ Calibration curve: Cubic fits, Flow rate : 0.5L/min



Accessories	
Ceramic sample boat, large capacity ceramic sample boat, nickel sample boat, combustion tube (pre-packed with catalysts), mercury collector tube, Teflon® joint set	

Options
For maximum flexibility, the analyzer is optionally available with accessories for reduction vaporization (left side, SC-5 autosampler on top and RD-5 auto reagent dispenser on bottom) and gas tube desorption (right side, RH-MA3) analysis, allowing a single analyzer for multiple analytical techniques.



S-MA manual reducing vaporization attachment (5 mL)



MB-1 mercury vapor calibration box

Reduction vaporization attachment	Auto reagent dispenser	RD-5
	Auto sample changer	SC-5 (5 mL: 30 positions)
	Manual attachment	S-MA (5 mL)
	Detection limit	1 ppt (5 mL)
	Maximum measurement range	Up to 100 ppb (5 mL)
Gas analysis attachment	Heating unit for mercury collector tube	RH-MA3
	Detection limit	0.001 ng
	Maximum measurement range	Up to 1000 ng



Nippon Instruments Corporation

A Rigaku Company

Identify the right mercury analyzer for you
<https://www.hg-nic.com>



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