ADI 2045VA Process Analyzer

Trace metal and plating bath analysis

Metrohm
Process Analytics

Voltammetric analysis goes online

Voltammetric trace analysis (VA) is used for determining the type and amount of electrochemically active substances in a liquid sample on the basis of a current-voltage relationship. These can be inorganic or organic ions, or even neutral organic compounds. This electrochemical technique is mostly used for trace metal analysis as well as quality control of plating baths.

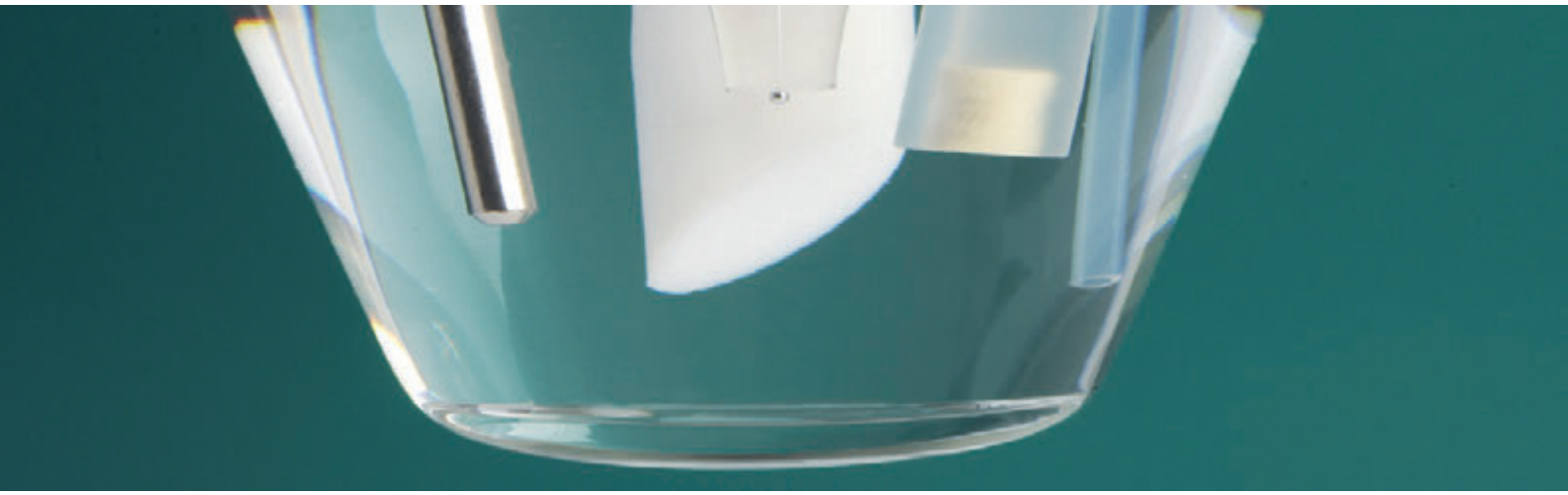
Voltammetric analyzers, developed based on many years of laboratory experience, are installed online in many industries for fast and efficient process control, which an in-house laboratory cannot provide.

Sensitive, accurate trace analysis

The importance of voltammetry is based on its high degree of accuracy and sensitivity, and the possibility of **speciation analysis**. Voltammetry is often used for supplementing and validating spectroscopic methods and is characterized by:

- Low equipment costs
- Comparably low investment and operating costs
- Short analysis times

The infrastructure needed for a voltammetric measurement system requires hardly any expenditure. All it needs is electricity and in some cases a supply of nitrogen. Ventilation, flammable gases and associated safety requirements are no issues. In the case of the Multi-Mode Electrode pro (MME), the mercury used is contained in a hermetically sealed reservoir for safe handling.



Online analyzers from Metrohm Process Analytics

Metrohm Process Analytics is the brand name representing the well-known Applikon wet chemistry process analyzers as well as the Metrohm NIRSystems instruments for process analysis. Under this brand name we offer analytical systems for titration, spectroscopy, electrochemistry, photometry, ion chromatography, TOC, as well as ion selective measurements.

Metrohm Applikon B.V. has over 40 years of experience with online measurements in the chemical industry, wastewater treatment plants, power plants, refineries, semiconductor manufacturers, and more. All of the analyzers are used directly in the process and are able to run 24 hours a day to continuously monitor the components of interest, keeping processes running smoothly.

Due to the close collaboration with Metrohm AG, a world leader in ion analysis, Metrohm Applikon has access to a large database of applications for laboratory-based measurements and instrument technology that can be used as a basis for online applications.

Metrohm Process Analytics takes the famous Metrohm laboratory solutions a step further. Fully automated, online customizable analyzers facilitate process monitoring across a wide array of applications. Drawing on our core competencies in a wide range of analytical techniques, we leverage our applications knowledge to create reliable solutions for process analysis that optimize efficiency, decrease chemical consumption and create a safer work environment.

Basis of Voltammetric Analysis

Voltammetry is an electrochemical technique in which a current as a result of an electron transfer is measured. A potential that varies in time is applied to an electrode, a redox reaction will take place of which the current is then measured.

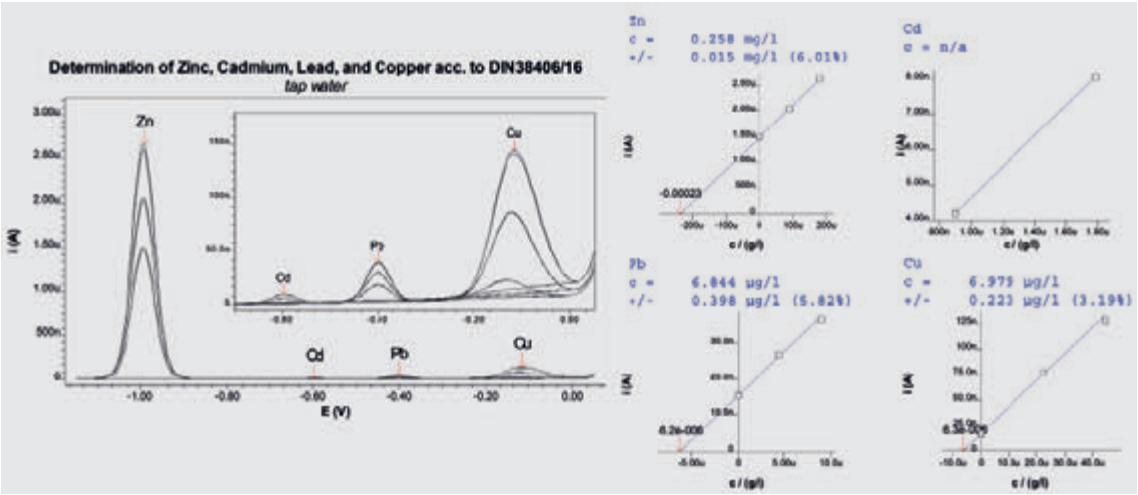
Stripping voltammetry

By applying a discrete potential over a period of time, metal(s) are deposited on the electrode. In a second step the potential is swept and the metal is stripped off the electrode surface. The currents measured during the sweep are directly related to the metal concentration in solution.

Due to the fact that a kind of preconcentration step (the deposition of the metal) is used in this process, cathodic and anodic stripping voltammetry (CSV and ASV) are capable of achieving very low detection limits.

Easily measure chemical speciation

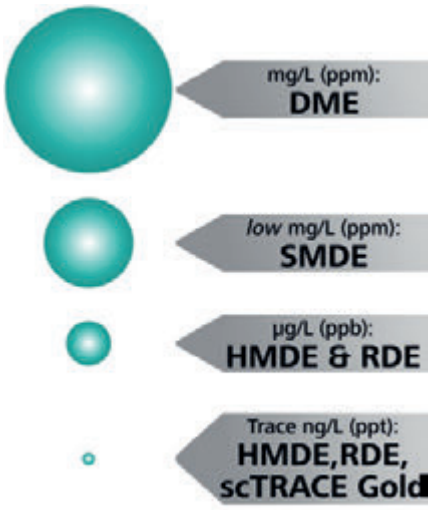
Voltammetry allows to distinguish between free and bound metal ions or between their different oxidation states as well as to specify the biological availability of heavy metals. This makes voltammetry into an **essential tool** for environmental analysis. Comparable insights can only be gained by spectroscopy after complicated separation of the metal species.



Example of a determination with the Computrace of heavy metals Zn, Cd, Pb, and Cu present in tap water according to DIN 38406 Part 16. Voltammograms (left) and standard addition curves (right) are shown.

Typical Detection limits		
Antimony	Sb(III)/Sb(V)	200 ppt
Arsenic	As(III)/As(V)	100 ppt
Bismuth	Bi	500 ppt
Cadmium	Cd	50 ppt
Chromium	Cr(III)/Cr(VI)	25 ppt
Cobalt	Co	50 ppt
Copper	Cu	50 ppt
Iron	Fe(II)/Fe(III)	50 ppt
Lead	Pb	50 ppt
Manganese	Mn	2 ppb
Mercury	Hg	100 ppt
Molybdenum	Mo(IV)/Mo(VI)	50 ppt
Nickel	Ni	50 ppt
Platinum	Pt	0.1 ppt
Rhodium	Rh	0.1 ppt
Selenium	Se(IV)/Se(VI)	300 ppt
Thallium	Tl	50 ppt
Tungsten	W	200 ppt
Uranium	U	25 ppt
Zinc	Zn	50 ppt

Concentration range of ADI 2045VA electrodes



ADI 2045VA Process Analyzer

Metrohm is the **world market leader** in voltammetry. Hardly any other method is better suited to detect even the smallest traces of heavy metals in water. The Metrohm Applikon ADI 2045VA analyzer is an online Process Analyzer based on the principle of voltammetric analysis, which has routinely been applied in the lab for several decades.

The ADI 2045VA system is built in the same rugged housing as our other established and proven multipurpose analyzers. Its analytical heart is the 797 Computrace VA system of Metrohm, the most well-known laboratory VA system.

The advantage of this setup is that the same methods can be used as are being developed and used in the lab.

Working Electrodes

- Multi-Mode Electrode pro (MME)
- Rotating Disk Electrodes (RDE)
- scTRACE Gold sensor

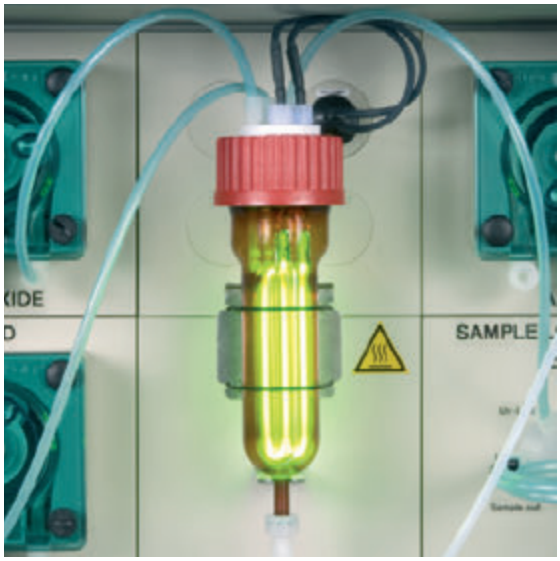
Flexibility enables many different applications

Depending on the application the so-called wet part can be configured choosing the right modules like sampling valves, tubing pumps, dosinos, valves, and more.

Depending on the application, the number of burettes may change, or a sample digester (UV or thermal) may or may not be present.

Suitable for a variety of industries

- Semiconductor production
- Mining applications
- Water quality control



Digestion Capabilities: UV or Thermal

When performing metal analysis, often a part of the metals are bound in (organic) compounds. A digester module is used to release these metals by exposing the sample to UV radiation, possibly in combination with an oxidizer and acid. Now all metals are determined in a subsequent VA analysis, and the amount of free vs. total metals can be calculated.

Eliminate interfering substances

Interfering compounds such as peroxide are simply destructed to inactive molecules like water and carbon dioxide.

Additionally, a thermal digester module is available for instances where the UV digester is not sufficient.

Operation

The system is controlled by an Industrial PC (IPC) in combination with a programmable logic controller (PLC). The PLC controls all external and wet part devices, while the Computrace and Dosino burettes are driven directly by the IPC.

The analyzer includes a touch screen display, making operation easy and intuitive. An optional keyboard and pointing device can be connected via a USB port. The operation bar gives a number of options while the status bar shows system status at every moment. The user is informed about relevant events, such as result limit alarms, by means of a pop-up window that can be acknowledged. All external and wet part devices can be manually operated for maintenance purposes.

Result display, trend graph

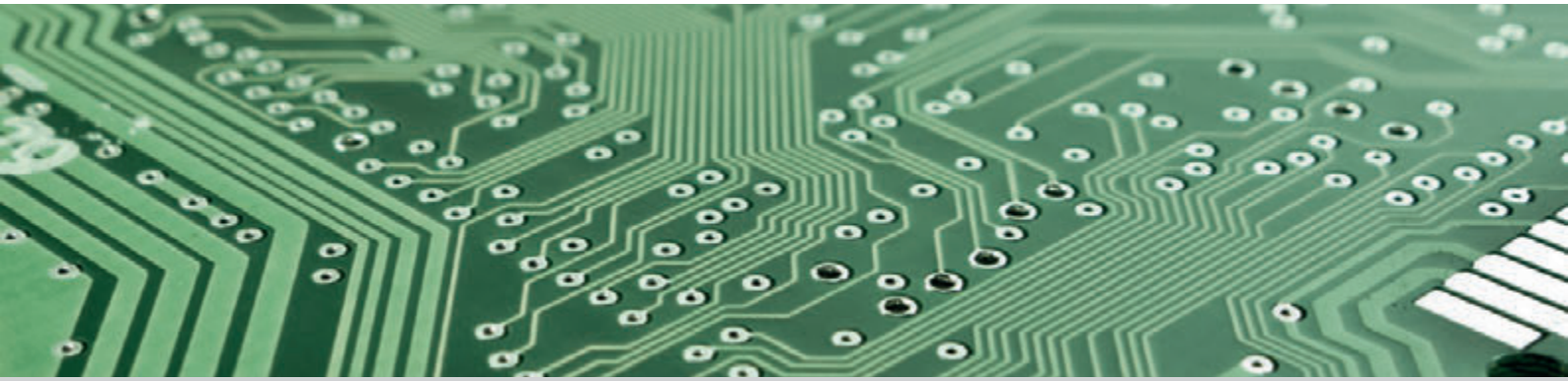
Results are presented in a list or in a graphical view. The appearance can be modified, i.e. scales, colors and pointers to indicate set point and alarm limits in a trend graph and bar graph.

Events can be logged for later inspection
• Occurred result alarms
• Device warnings and errors
• Control actions
• Configuration changes, etc.

Relevant system information, like analysis results, logbook events, system configuration info, action lists and data can be exported in XML-format on a removable memory stick connected to the easily accessible USB port on the front of the analyzer cabinet.

Standards and Accepted Methods for VA

Parameter	Standard	Matrix
Zn, Cd, Pb, Cu, Tl, Ni, Co	DIN 38406-16	Drinking and wastewater
CN^-	Sample preparation acc. to DIN 38405-13	Drinking and wastewater
U^{VI}	DIN 38406-17	Ground-, raw-, and drinking water
EDTA, NTA	DIN 38413-5	Wastewater, sludge
Copper content	EN 15376, ASTM D 4806, ASTM D 5798	Bioethanol
Fumaric acid	AOAC 968.16	Fruit and vegetable juices
Saccharin	SLB 41-2.5	Fruit and vegetable juices
Pb	AOAC 979.13, AOAC 979.17	Dairy, Fruit and vegetable juices



Working Electrodes for the ADI 2045VA

MME, Multi-Mode Electrode pro

As a working electrode for the system, small mecury drops are formed at the end of a capillary. The Multi-Mode Electrode pro has a hermetically sealed mercury reservoir of 6 mL and is ideally suited for (heavy) metal analysis. This reliable electrode for polarography and voltammetry can be operated as DME, SMDE or HMDE.

Key points of the Multi-Mode Electrode pro

- New electrode surface for every measurement
- Enough mercury for > 10,000 analyses, or 200 days
- Collection of mercury for safe disposal
- 10 years of measurement at 50 analyses per day requires 100 mL mercury in total

Metrohm Multi-Mode Electrode pro with Hg reservoir and capillary

Safety always comes first

During and after the analysis, mercury is collected on the bottom of the analysis vessel and is then drained off into a mercury trap. It is always kept under liquid, so no mercury vapor is allowed to escape and the safety risk for the user is thus minimized. The mercury trap can be emptied on occasion, so that the collected mercury can be disposed of correctly.

The scTRACE Gold can detect trace amounts of As and Hg in water

scTRACE Gold sensor

Anodic stripping voltammetry (ASV) using the scTRACE Gold sensor offers a straightforward, highly affordable alternative to the spectroscopic determination of arsenic, distinguishing between As(total) and As(III). The limit of detection is significantly lower than international maximum allowable limits.

- Also suitable for trace inorganic mercury detection

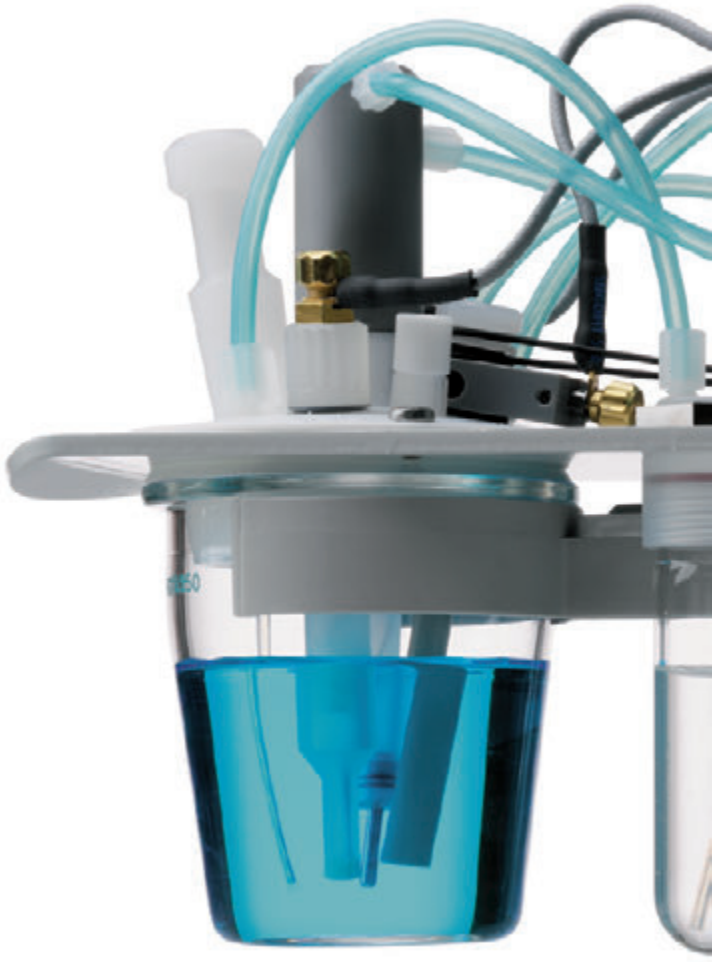
Many different important applications

Online use of voltammetric analysis is of importance in many industries. In Zinc mining for example, the technique can be applied during **smelting**, **purification**, **electrolysis** or in the **control of the process water**.

Example applications for the ADI 2045VA

- Mining for Au, Cu, Zn, and other metals
- Electroplating bath quality
- Water quality control (drinking, river, surface, and waste water)
- Monitoring trace metal concentrations

Cyclic Voltammetric Stripping



Cell with electrodes for CVS analysis of a Cu bath

Production control of electroplating baths

Cyclic Voltammetric Stripping Analysis (CVS) and Cyclic Pulse Voltammetric Stripping Analysis (CPVS) are widespread methods in the electroplating industry for the determination of organic additives in electroplating baths.

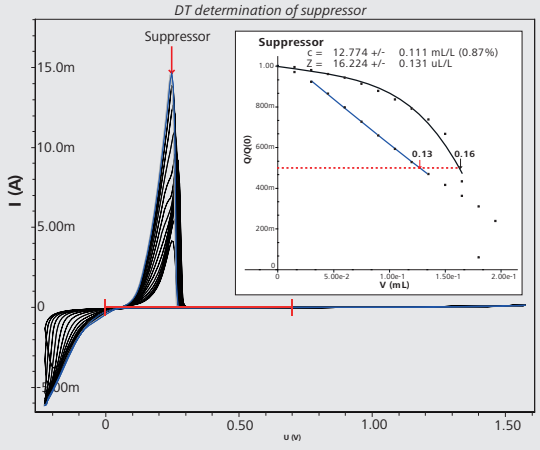
Rotating Disk Electrode (RDE)

The RDE induces a flux of analyte to the electrode by rotating in a controlled manner during experiments. Therefore the boundary layer is continually refreshed from the bulk sample. For many technical coatings, particularly in the manufacture of PCBs and semiconductor components, this method is an essential part of production control.

- Solid state platinum electrode, no mercury
- Organic additive determinations under process circumstances
- Accurately measures brighteners, levelers, and suppressors
- Concentrations in mL additive per L bath liquid

Determination of suppressor in acid copper with calibration curve

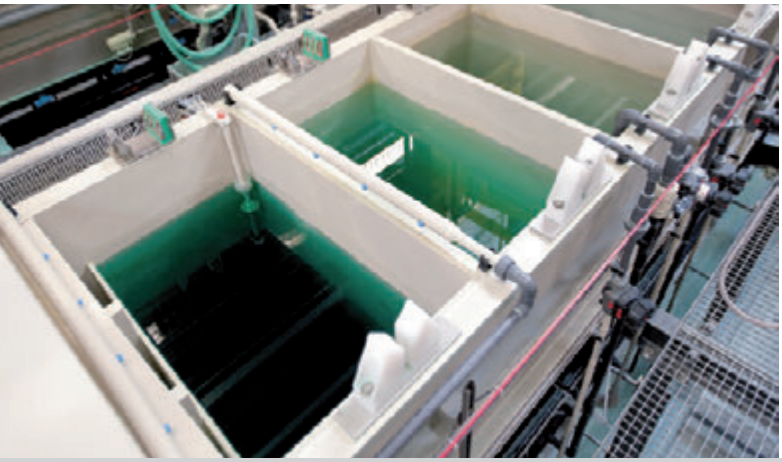
DT determination of suppressor



With CVS or CPVS methods, the concentration of **organic additives** can be determined accurately. This guarantees continuous and interference-free operation of the plating bath. In particular, the accuracy of the analytical results has helped the method to become well accepted in the electroplating industry.

The most important application fields for CVS

- Acidic copper baths
- Tin-lead baths and tin baths
- Alkaline zinc baths



Precise measurements even in saline conditions

The particular challenge for analysis in the electroplating sector are the large quantities of salts typically found in electroplating samples. For such samples, many measuring techniques can at best provide unsatisfactory results; others cannot provide any results at all.

Here, electrochemical analysis is able to demonstrate its strengths; the disadvantage for other methods is transformed into a big advantage: large amounts of salt equal good conductivity, which is a prerequisite for electrochemical analysis.

Technical information

Dimensions (H x W x D)	870 x 700 x 500 mm
Weight	85 kg
Power supply	100–120 V / 200–240 V
Power consumption	50–60 Hz / 600 W
IP protection	IP65 / NEMA4
Ambient temperature	5–40 °C

Other specifications

Applied Analysis methods

- DP (Differential Pulse)
- SQW (Square Wave)
- CVS (Cyclic Voltammetric Stripping)
- CPVS (Cyclic Pulse Voltammetric Stripping)

Sampling and sample streams

Sampling	batchwise
Sample frequency	programmable
Analysis time	typically 15–30 minutes
Sample streams	maximum 4
Sample volume	0.2–50 mL
Sample temperature	5–45 °C
Sample pressure	0–2 BarG / 0–28 PSIG (without preconditioner)

Connectivity

Discrete I/O:

Analog Result Output	4–20 mA, max 16 ea.
Digital Remote Control	24 VDC, Start, Stop, Continue, Sample Override 1, 2, 3, 4.
Digital Detector Input	24 VDC, max 12 ea. (Leak, Reagent Level, etc,)
Digital Relay Output	2 A, Power On, Sequence Running, Warning, Error, Result Alarm 1, 2, 3, 4, Service

Serial ports:

Ethernet	for remote control by Windows functionality
RS232	remote control by Windows functionality
USB	for keyboard, pointing device and removable memory media

General

Touch screen operation	
Housing material:	Electronics cabinet: Zinc plated steel, epoxy coated Wet part door: Polystyrene, epoxy coated

Maintenance

Weekly	visual inspection
Monthly	reagents supply
Biannual	maintenance check

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Subject to modifications
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