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# Applications of stripping voltammetric techniques in food analysis

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**Abstract** Stripping voltammetric techniques are powerful analytical tools that are becoming widely used in various chemical analysis fields. Hence, the objective of this survey is to give a general overview on the scope of the applicability of stripping voltammetric methods in food industries. The applications discussed include recent studies on the utilization of these electroanalytical methods in determination of food contaminants (toxic metals, pesticide, fertilizers and veterinary drugs residuals), trace essential elements, food additive dyes and other organic compounds of biological significance. Tables that give method summaries referenced to the original work are provided.

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## 1. Introduction

The science of food and nutritional analysis has developed rapidly in recent years. Measuring the quality, safety, nutrition and stability of food products is the primary concern of analytical chemists who are working within food processing industry and in academic and governmental food analytical laboratories. In fact, food scientists obtained this valuable information via the application of various modern instrumental techniques which analyzed the foodstuffs composition, appearance, texture, flavor and shelf life. The importance and awareness of

nutrition in the public health issues has resulted in increased demands for knowledge of the nutrient content of the foods.

Accordingly, there has been a growing tendency to introduce more accurate, reproducible and objective procedures for the evaluation of quality in foods in place of the old fashion, slow and relative inadequate methods in food analysis. The fundamental aspects of food analytical techniques in terms of their application, theory, instrumentation and experimental methodology were described in some analytical chemistry references (Meyers, 2000; Worsfold et al., 2005), several food analysis monographs (Tunick and Palumbo, 1998; Pomeranz and Meloan, 1994; Stewart and Whitaker, 1984; Baltes et al., 1982; Pearson, 1976) and article reviews for the applications of some analytical technique for food analysis such as ICP-MS (Cubadda, 2004), supercritical fluid chromatography (Anklam et al., 1998), infrared spectroscopy (Cropper and Hamer, 1949), chemiluminescent (Navas and Jimenez, 1996) and biosensors (Mello and Kubota, 2002).

Although chromatographic and spectroscopic techniques are traditionally highly desirable for the analysis of food and agricultural products, however, recent data about instrumental

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methods of food analysis revealed that new analytical techniques such as electrophoresis, flow injection analysis, bioassays and stripping voltammetric technique had been increasingly employed in foodstuffs analysis over the last two decades. In the field of food analysis stripping voltammetric techniques were primarily used in the determination and study of food additives (preservatives, color agents, artificial sweeteners) and contaminants (toxic metals, pesticides, residual veterinary drugs).

However, stripping voltammetric methods had been proved to be exceedingly effective in analyzing some molecules of high biological significance (i.e. vitamins, aromatic flavors, alkaloids and natural organic acids), which indicates the quality of the food product and its nutritional value. Currently, stripping voltammetric technique has become widely adopted for monitoring of various food components and contaminants. Thus the aim of this paper is to give a general overview of the reliability and possibility for using these electroanalytical methods in food analysis.

## 2. Principles of stripping voltammetry

The technique of stripping voltammetry (SV) has been receiving considerable attention since it is the most sensitive electroanalytical technique and over the last decade it has evolved into a very versatile and powerful analytical technique. In recent years it has become a widely used method for food analysis. This electrochemical method encompasses a variety of electroanalytical procedures having a common characteristic initial step. In all these procedures; the analyte of interest is accumulated on a working electrode by controlled potential electrolysis. After a short rest period, this preconcentration step is followed by the stripping step, which involves the dissolution of the deposit when a linear ramp is applied to the electrode. Thus, a detectable current is produced at the electrode surface following the oxidation or reduction of the analyte at a characteristic potential.

By careful interpretation of the resulted peak shape current–potential voltammogram recorded during the stripping step, important and desired analytical information is readily obtained. The peak potential (position of  $E_p$ ) is characteristic of the given substance and thus it can be used for qualitative identification, whereas the peak current  $I_p$  is proportional to the concentration of the corresponding analyte in the test solution. This analytical quantitative information can be obtained from the height or area of the stripping voltammetric peak. Since stripping curves/peaks for various analytes occur at characteristic potentials, hence several species can often be determined simultaneously. A comprehensive treatment and discussion of the principles, instrumentation and applications of stripping voltammetry can be found in several monographs (Wang, 1985; Vydra et al., 1976; Brainina and Neyman, 1993; Brainina, 1974; Wang, 1994).

Among the techniques employing a preconcentration step, the first to be developed has been anodic stripping voltammetry (ASV), which mainly applied to trace analysis of heavy metal ions using a hanging mercury drop electrode. The basis of ASV for metals is the electrolytic dissolution of a metal which previously had been deposited on a mercury electrode. The preconcentration is achieved by cathodic deposition at a controlled time and potential. In the following measurement step, the potential is scanned anodically and linearly. During the anodic

scan procedure the amalgamated metals are stripped out of the electrode, reoxidized and dissolved back to the bulk of the sample solution. Then the resulting anodic current is recorded as a function of the applied voltage. The cathodic stripping voltammetric technique (CSV) is possible when an anodic reaction can form an insoluble compound (e.g. mercury salts or complexes) with the electrode material. In this technique, the oxidation of the analyte is used for its preconcentration as an insoluble film on the electrode, subsequently, the concentrated analyte is reduced and measured during a negative going scan. Thus it is sometimes known as the “mirror image” of ASV technique. Quantification is accomplished by measuring the height of the resulting reduction peak.

Responding to the need to apply this sensitive method to those substances that cannot be preconcentrated by electrolysis at the electrode surface, alternative principles have been proposed for the accumulation of the analyte via the utilization of the adsorption phenomena. In the adsorptive stripping voltammetry (AdSV) accumulation of the analyte occurs by physical adsorption rather than by electrolytic deposition. However, depending on the oxidation–reduction properties of the accumulated analyte, it is determined by scanning the potential in appropriate negative or positive direction. Many organic molecules have a strong tendency to be adsorbed from aqueous solution onto a mercury surface. Additionally, many inorganic cations have been determined by AdSV technique after their complexation with surface-active complex agents.

It is worth mentioning that although mercury working electrodes such as hanging mercury drop electrode (HMDE) and mercury film electrode (MFE) were extensively applied in the past, nonetheless they become increasingly disallowed and banned in some countries due to health and environmental considerations. Alternatively, environmentally friendly electrodes and chemically modified electrodes are becoming the focus of substantial recent research studies and have received great attention in electroanalysis. Most popular schemes used to substitute mercury electrodes include carbon paste electrodes (Shams and Torabi, 2006), polymer film electrode (Shelton and Chambers, 1991), surface-bound crown ethers electrodes (Ijeri and Srivastava, 2001), carbon nanotube electrodes (Gong et al., 2005), boron-doped diamond electrodes (Spataru et al., 2007), bismuth film electrodes (Baldo et al., 2003) and screen printed electrodes (Crew et al., 2008).

In many respects, the sampling and preparation of a sample are critical steps in any technique for food analysis. Accordingly, the pretreatment of food samples before chemical analysis varies greatly depending on the type of food sample or the type of the analyte determined. However, it is more often to use mixtures of strong acids for samples decomposition (wet ashing) or microwave digestion for elemental analysis. On the other hand, liquid–liquid extractions or solid-phase extractions are frequently required for the determination of organic analytes.

In general, stripping voltammetric techniques have had enjoyed the reputation of being highly sensitive, selective and in many cases where it has been commercialized, fairly inexpensive as well as fairly easy to run. Additionally, the high accuracy and precision with the possibility of portable analysis with easy to operate and low cost instrumentation have led to the rapid expanding application of stripping voltammetry in food analysis as testified by the numerous publication on this subject in the last few years.

### 3. Stripping voltammetric determination of contaminants

Chemical contaminants in food are of great concern to the consumer, food manufacturers and regulatory authorities. Contamination of food with chemicals may arise from environmental pollutants and industrial chemicals during food processing and packaging or from the use of agrochemicals and veterinary drugs in food production.

#### 3.1. Determination of toxic metals in food

Over the years, stripping voltammetric techniques have been shown to possess a great potential for the determination of trace and ultratrace concentrations of toxic metals such as mercury (Sancho et al., 2001), lead (Satzger et al., 1983), cadmium (Gajan et al., 1982), arsenic (Sancho et al., 1998), antimony (Clinio and Torsi, 2004) and uranium (Abo-Maali and Elhady, 1999). The simultaneous determination of ultratrace levels of lead and cadmium in selected agriculture crops (over 1700 crop samples) by differential pulse anodic stripping voltammetry was reported by Stazger and his colleagues (Satzger et al., 1982). A collaborative study carried out via 13 laboratories for the direct determination of lead in evaporated milk and apple juice by anodic stripping voltammetry was also reported in the literature (Zink et al., 1983). Similarly, 20 laboratories participated in a collaborative study for the anodic stripping voltammetric determination of lead and cadmium in various food commodities (i.e. beans, beef, fish, infant formula apple juice and cereal) (Capar et al., 1982). In addition, Golimowski et al. published a comparative study of the levels of cadmium and lead toxic metals in 36 wine samples by anodic stripping voltammetry (Golimowski et al., 1979).

Data on the ultratrace background levels of lead and cadmium in raw agricultural crops by differential pulse anodic stripping voltammetry was reported by Stazger and his team (Satzger et al., 1984). Furthermore, stripping voltammetric techniques, especially ASV method, were successfully utilized for the determination of these toxic metals content in various

foodstuffs such as leafy vegetables (Matloob, 2003), wheat and rice (Ogorevc et al., 1987), common table salt (Ali, 1999), liver and fish (Adelaju and Bond, 1983), infant formulas (Esteve et al., 1994) and canned soft drinks (Sabry and Wahbi, 1999). Additionally electroanalytical data on the stripping voltammetric determination of toxic metals in food commodities are listed in Table 1.

#### 3.2. Determination of pesticides residuals in food

Pesticides including fungicides, herbicides and insecticides are routinely used in most food production to control pests (e.g. insect, fungi, rodents, worms and herbs) that would otherwise destroy or reduce the food production. The problems associated with pesticides are that they are very often nonspecific and may therefore be toxic to organisms that are not pests. In addition, pesticides may also be non biodegradable so that they persist in the environment and may accumulate in living organisms. Stripping voltammetric technique proved to be highly effective in analyzing various pesticides molecules found as contaminants in foodstuffs, soil (Mainisankar et al., 2005) and tap water (Ibrahim et al., 2001) and irrigation water (Pedrero et al., 1993). The adsorptive stripping voltammetric (AdSV) technique has been used to develop a method for the determination of fenthion pesticide in olive oil after the extraction of fenthion by solid-liquid extraction procedure using silica cartridge (Diaz et al., 2008). AdSV method has also been applied successfully to the determination of the trace concentration of pyridafenthion insecticide in wine sample (Sampedro et al., 1998) and phosalone and carbophos insecticides in potato and tomato (Ulakhovich et al., 1998).

Similarly, the anodic stripping voltammetric (ASV) technique has been used for the ultratrace measurements of ziram (a dithiocarbamate fungicide) in rice samples using a static mercury drop electrode (Mathew et al., 1996). Pedrero et al. reported a sensitive adsorptive stripping voltammetric procedures for dinoseb herbicides at nanomolar levels in spiked apple juice (Pedrero et al., 1994) and potatoes (Pedrero

**Table 1** Applications of stripping voltammetry in the determination of toxic metals in food samples.

Toxic metals	Stripping voltammetric technique	Working electrode	Food sample	References
Hg(II)	ASV	Gold film electrode	Table salt	Okcu et al. (2008)
Pb(II)	ASV	HMDE	Cow's milk	Inam and Somer (2000)
Cd(II)	CSV	HMDE	Meat, egg	Meryan et al. (1998)
As(III)	CSV	HMDE	Oyster tissue	Bin Othman et al. (1984)
Sb(III)	ASV	HMDE	Wheat, maize	Clinio and Torsi (2004)
U(VI)	CSV	Glassy carbon electrode	Sugar	Abo-Maali and Elhady (1999)
Hg(II)	ASV	Modified graphite electrode	Alcohol drinks	Zakharova et al. (1996)
As(III)	ASV	Modified glassy carbon electrode	Alcohol and alcohol free drinks	Zakharova et al. (1998)
Pb(II), Cd(II)	ASV	Mercury microelectrode	Honey	Sanna et al. (2000)
Pb(II)	ASV	Modified silver electrode	Canned juice, stewed fruit	Fomintseva et al. (1997)
Pb(II), Cd(II)	ASV	Thick-film modified electrode	Wines	Brainina et al. (2004)
Pb(II), Cd(II)	ASV	Modified glassy carbon electrode	Honey, apple drink	Buckova et al. (1996)
Cd(II), Pb(II)	ASV	Modified glassy carbon electrode	Beer	Gutierrez et al. (1999)
Pb(II), Cd(II)	ASV	Modified glassy carbon electrode	Edible oils	Wahdat et al. (1998)
Pb(II), Cd(II)	ASV	Modified ultra microelectrode	Alcohol drink	Matysik et al. (1994)
As(III)	CSV	HMDE	Tobacco leaves	Kowalska et al. (1999)
Pb (II)	ASV	Thick-film epoxy graphite composite	Canned fruit, drinks and juices	Faller et al. (1997)

et al., 1991). Moreover, there are several published papers dealing with the application of AdSV technique in the determination of some herbicides molecules such as desmetrne in apple juice (Pedrero et al., 2001), thiram in strawberries (Shubietah et al., 1995), ioxynil in potato (Shaidarova et al., 1998) and terbacil and lenacil in fruit samples (Thriveni et al., 2007).

### 3.3. Determination of veterinary drugs residuals in food

Veterinary drugs are used to treat animals for bacterial infections, parasitic infections, to enhance growth, to control fertility and reproduction or to alter the behavior. There is some possibility that a residual of these veterinary drugs find its way to human diet via the cattle and poultry products such as milk, meat and dairy. Within each of these drugs types there are different classes and methods for residue analysis. Stripping voltammetric methods are very well known established as an analytical technique for the determination of numerous of pharmaceutical drugs in general (Alghamdi, 2002; Abu Zuhri and Voelter, 1998; Gratteri et al., 1992; Vire et al., 1989) and some veterinary drug. Quantitative analysis of three antibiotic fluoroquinolones veterinary drugs in bird feedstuffs was carried out using linear sweep stripping voltammetry (LSSV) method Ni et al., 2006. In addition, Ghoneim et al. had used both differential pulse AdSV and square wave AdSV techniques for the determination of anthelmintic veterinary drug nitroxiol (Ghoneim and Elries, 2006).

### 3.4. Determination of fertilizers residuals in food

Fertilizers are any substance that added to soil in order to increase its productivity and they can be of natural origin or more frequently they can be made up of synthetic chemicals, particularly nitrates and phosphates. As in the case of pesticides and veterinary drugs, residual fertilizers may find its way to the food products. Also, there some health as well as environmental impacts caused by the build up of high fertilizer concentrations. Gunaghan and colleagues had reported a sensitive anodic stripping voltammetric procedure for trace nitrite fertilizer in meat and food products (Guanghan et al., 1997). Also, whereas similar anodic voltammetric approach but with different chemically modified electrode has been developed for the electroanalytical determination of nitrite in food samples such as sausages and pickled vegetables (Santos et al., 2009).

## 4. Stripping voltammetric determination of trace essential elements

Essential elements are any elements required by living organisms to ensure normal growth, development maintenance. The trace essential elements usually occurred in living tissues at low concentration levels and thus their requirements are much less. The most important elements are Fe, Mn, Zn, Cu, I, Co, Se, Mo, Cr and Si and each element may fulfill one or more of a variety of metabolic roles. The determination of food minerals and essential elements can be done using various instrumental techniques. Among them, stripping voltammetric electroanalytical methods stands out because they are fast, sensitive and inexpensive while still being adequately precise. In fact, there have been many reviews devoted to empha-

size and illustrate the wide spectrum and scope of stripping voltammetric applications and its potentialities in the analysis of metal ions (Abu Zuhri and Voelter, 1998; Zaitsev et al., 1999; Brainina, 2000).

A study of the square wave AdSV determination of iron essential element in olive oils has been reported (Galeno et al., 2003). Cathodic stripping voltammetry determination of trace amounts of selenium in tuna fish sample (Lambert and Turoczy, 2000) and in rapeseed oils and seed (Blades et al., 1976) has been made. Giroussi et al. had demonstrated the applicability of AdSV technique for the determination of trace levels of cobalt in vegetable animal foodstuffs (Giroussi et al., 1995) and milk powder (Koops and Klomp, 1991). The usefulness of AdSV method in the determination of nickel in infant formulas has been described by Barbera et al. (1994). Aluminum content in samples of commercial tea leaves and coffee beans was determined by adsorptive stripping voltammetry (Stryjewska et al., 1993). An electroanalytical method has been developed for the determination of copper in cow's liver tissue by differential pulse cathodic stripping voltammetry (Safavi and Shams, 1999). Gao and Siwo had reported AdSV procedure for the determination of molybdenum essential element in plant foodstuffs (Gao and Siwo, 1996).

Similarly, thallium element has been analyzed in olive leaves by Zhou and Neeb via the application of AdSV technique (Zhou and Neeb, 1994). A sensitive and selective method has been reported for the determination of ultratrace concentration of tin by AdSV on a glassy carbon mercury film electrode. The developed stripping voltammetric procedure was applied for the determination of tin in fruit juices (Adelolujo and Pablo, 1992). Finally, Yang and colleagues published a paper illustrated the application of cathodic stripping voltammetric approach in the determination of iodine mineral in table salt, laver and eggs (Yang et al., 1991). Summary of representative examples demonstrating the applicability of stripping voltammetric techniques for the determination of several essential elements are given in Table 2.

## 5. Stripping voltammetric determination of food additive dyes

Food additives are substances added to the food during its manufacture or processing in order to improve its flavor, color, texture, stability and shelf life and these additives are usually present in minute quantities. Natural (from vegetable, animal or mineral origin) or synthetic food dyes provide food with an attractive appearance. On the other hand, use of dyes or chemical impurities in colorant agents can have undesirable side effects. Although food additive dyes traditionally have been analyzed by spectrophotometric and/or chromatographic methods, however, they are also suited to be analyzed by electrochemical methods, most recently using stripping voltammetric techniques. In fact, Fogg (1998) and his colleagues (Zima et al., 1999) have reviewed the utilization of stripping voltammetric techniques for the determination of several synthetic dyes usually used in textile industry.

However, in the field of artificial colorants permitted for use in foodstuffs, Fogg and his colleagues once again effectively employed stripping voltammetric method for the determination of various food additive dyes. They demonstrated that 16 food synthetic coloring matters can adsorb strongly on a hanging mercury drop electrode and to be amenable to determination by differential pulse adsorptive stripping voltammetry.



**Table 2** Applications of stripping voltammetric techniques in the determination of essential elements in foodstuff products.

Essential elements	Stripping voltammetric technique	Working electrode	Food sample	References
Cu(II)	AdSV	HMDE	Rice, vegetables	Guanghan et al. (2004)
Zn(II)	ASV	Bismuth film electrode	Milk	Guo et al. (2005)
Se(IV)	CSV	HMDE	Rice	Panigate and Falcicola (2007)
Mo(VI)	AdSV	HMDE	Plant foodstuffs	Gao and Siow (1996a)
Fe(II)	AdSV	HMDE	Apple, potato	Gao and Siow (1996b)
Al(III)	AdSV	HMDE	Tea leaves, coffee beans, citrus leaves	Stryjewska et al. (1993)
Cr(III)	AdSV	HMDE	Peach leaf	Jiang and Cai (1991)
Ni(II)	AdSV	Glassy carbon mercury film	Berries	Wang et al. (1994)
Se(IV)	CSV	HMDE	Food supplements	Holak and Specchio (1994)
Co(II)	AdSV	HMDE	Wine, mussel	Bebeki and Voulgropoulos (1992)
Sn(IV)	AdSV	HMDE	Canned food	Li et al. (2005)
Ni(II)	AdSV	HMDE	Oil, vegetables, milk	Tripathi et al. (1994)
Se(IV)	CSV	HMDE	Garlic	Inam and Somer (1999)
Mo(VI)	AdSV	Glassy carbon mercury film	Plant and animal tissues	Adeloju and Pablo (1995)
Se(IV)	CSV	HMDE	Cow's milk	Inam and Somer (2000)
Ni(II)	AdSV	HMDE	Milk	Zhang et al. (1991)
Mo(VI)	AdSV	HMDE	Plants	Karakaplan and Henze (1993)
Ni(II), Co(II)	CSV	HMDE	Beet sugar	Sancho et al. (2000a)
Cu(II)	ASV	Nafion coated mercury film	Beer	Gutierrez et al. (1999)
Ni(II)	CSV	HMDE	Canned vegetables	Meryan et al. (1997)
Zn(II)	AdSV	HMDE	Instant coffee	Alghamdi (submitted for publication)

**Table 3** Applications of stripping voltammetric techniques in the determination of some additive dyes in foodstuff products.

Food dye	E number	Stripping voltammetric technique	Food sample	References
Sunset yellow	E110	AdSV	Soft drinks	Nevado et al. (1997a)
Tartrazine	E100	AdSV	Soft drinks	Nevado et al. (1997b)
Amaranth	E123	AdSV	Fizzy drinks	Alghamdi (2005a)
Allura red	E129	AdSV	Sweets	Alghamdi (2005b)
Quinoline yellow	E104	AdSV	Refreshing drinks	Nevado et al. (1997)
Carmoisine	E122	AdSV	Synthetic mixture	Castrillejo et al. (1990)
Ponceau 4R	E124	AdSV	Synthetic mixture	Castrillejo et al. (1990)
Carmine	E120	AdSV	Ice cream, soft drinks	Alghamdi (in press)
Erythrosine B	E127	AdSV	Ice cream, soft drinks	Alghamdi et al. (in preparation)
Brilliant blue FCF	E133	CSV	Commercial food products	Florian et al. (2002)

try (Fogg et al., 1986). A quantitative chemometric approach was used by Ni et al. for the simultaneous adsorptive voltammetric analysis of mixed colorants solution of some food dyes namely amaranth, sunset yellow, tartrazine and ponceau 4R (Ni et al., 1996) and the proposed method was applied for the determination of these four food dyes in some food samples such as drink, candy, and juice (Ni et al., 1997). Finally, Table 3 summaries the analytical results obtained from the reported articles published by several research workers who investigated the stripping voltammetric behavior and properties of several food additive dyes.

## 6. Stripping voltammetric determination of other organic molecules

Many other organic compounds some of them are of biological significance can also be measured in foodstuffs samples using stripping voltammetric technique. Application of stripping voltammetric techniques to the determination of several

vitamins in pharmaceutical (Alghamdi, 2002), clinical (Killard et al., 2000) and food samples is being extensively explored. Vitamin determination in food analysis is very important in efforts to minimize nutrient losses before and after food processing or to use vitamin as food additives such as antioxidants. A sensitive and selective method for the determination of vitamin C (ascorbic acid) using stripping voltammetry on a glassy carbon electrode has been reported by Lu et al. (1994). The proposed method was successfully employed for the determination of vitamin C in fruit and vegetable juices. Phytohormones are plant hormones that regulate plant growth and usually occur in plant at extremely low concentration levels. Blanco et al. developed a cathodic stripping voltammetric method to quantify minute amounts of three cytokinins plant hormones in apples (Blanco et al., 2000). Similarly, Hernandez and colleagues published an electrochemical method based on adsorptive stripping voltammetry for the study and determination of kinetin (growth regulator in plant) in apple extracts (Hernandez et al., 1997).

A cathodic stripping voltammetric method has been used by Reichart and Obendorf for the determination of naringin (flavonoid compound found in some citrus fruits) in grapefruit juice (Reichart and Obendorf, 1998). Likewise, saponin molecules (phtyo-chemical which is found in various vegetables) were also determined in sugar juices from sugar refining processes by stripping voltammetry (Sancho et al., 2000b). Finally, Rortela et al. had described an adsorptive stripping voltammetric method for the determination of the flavor enhancer inosinic acid in dehydrated soup samples (Portela et al., 1994).

## 7. Conclusion

Stripping voltammetric electrochemical techniques are widely applicable to food sample analysis and they can highly compete with traditional analytical methods which are currently used in this field such as spectrophotometry and chromatography. The range of analysts that can be determined and monitored using stripping voltammetric techniques has also expanded beyond the classical and simple metal ions voltammetric analysis. It is likely that in the near future stripping voltammetric techniques will definitely emerge as one of the powerful instrumental techniques adopted for food quality monitoring since these electroanalytical techniques can be easily automated or miniaturized for portable instrumentation suitable for on-site analysis.

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