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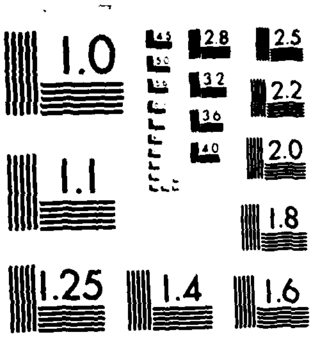

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A new method is described for the introduction of solid samples and liquid-sample residues into the inductively coupled plasma. Unlike earlier such methods, the new technique requires no separate power supply but rather draws its energy from the inductively coupled discharge itself. Termed a radio-frequency arc, the new sampling source carries a current of several amperes from the conventional plasma fireball to a grounded support. Under the tested conditions, the resulting high-frequency arc draws approximately

20. Abstract (continued)

140 watts from the main discharge and is shown to be adequate to volatilize conductive solids directly or others such as powders or liquid residues which can be placed into or on an inert conductive electrode. In its present form, the sampler operates in an apparently thermal fashion and causes fractional distillation of elements in a sample. Capabilities and limitations of the present arrangement are critically assessed and directions for future work are offered.

OFFICE OF NAVAL RESEARCH
Contract N14-76-C-0838
Task No. NR 051-622

SAMPLE INTRODUCTION INTO THE INDUCTIVELY COUPLED PLASMA BY A RADIO-FREQUENCY ARC

by

P. B. Farnsworth and G. M. Hieftje

Prepared for Publication
in
ANALYTICAL CHEMISTRY

Indiana University
Department of Chemistry
Bloomington, Indiana 47405

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The inductively coupled plasma (ICP) has over the past decade developed into a powerful tool for multielement analysis by atomic emission spectrometry (AES) (1-3). Despite its widespread use, ICP-AES remains limited in routine applications by sample introduction methods that require large volumes of aqueous solutions. Considerable impetus has existed and continues for the development of ICP sample introduction techniques capable of accepting either solid samples or very small quantities of solutions. Previous efforts in this direction have encompassed a wide variety of techniques, including direct aspiration of powders into the plasma (4-8), spark or arc sampling (9-11), laser vaporization (12,13), aspiration of microliter quantities of solution (14,15), electrothermal atomization (16-20), and direct insertion of a graphite-cup sample holder into the base of the ICP discharge (21-24).

In this preliminary communication we describe a new method for the analysis of solids and microsamples by ICP-AES. The new method parallels the direct insertion methods mentioned above; however instead of the sample being transported to the plasma, the plasma is brought to the sample. Initial experiments described here indicate that the method is capable of sampling a wide range of elements in solid form and can be applied to solution microsampling as well.

EXPERIMENTAL SECTION

The new technique is based on the discovery that a grounded conductor, placed below the sample tube of a modified ICP torch, can attract a stable arc filament from the base of the ICP discharge. The torch and sample holder assembly used in this preliminary study is illustrated in Figure 1. The quartz torch differs from conventional ICP torches in that its overall length has been reduced to 8.5 cm and that the 4-mm i.d. central sample tube is flared at the base to form a small bell jar. The sample gas is introduced tangentially into this bell jar, which is clamped with a nylon ring to a water-cooled copper base. A viton O-ring provides a gas-tight seal between the bell jar and the base. A boron-nitride shield isolates the base thermally and electrically from the arc. Several different sample holders and configurations were used, each of which will be described in detail in later sections.

Power was supplied to the plasma by a 27.12 MHz, 2.5 kW rf generator (Model HFP-2500 D with model APCS-1 power control and AMN-2500E impedance matcher, Plasma-Therm Inc., Kresson, NJ). A conventional three-turn load coil was used. Argon flow rates were 18.0 L/min coolant gas, 0.9 L/min plasma gas, and 0.9 L/min sample gas. In early studies the radio-frequency (rf) arc was ignited simultaneously with the ICP by turning on all gas flows and applying a high-voltage pulse from a Tesla coil to a metal ring placed around the torch between the base of the plasma tube and the bell jar. For later experiments the plasma and the arc were ignited independently; the plasma was lit with the sample gas turned off and electrical contact between the copper base and ground broken by a solenoid-actuated, high-voltage switch. Once the plasma had stabilized, the arc could be lit by turning on the sample gas, grounding the base, and striking the Tesla coil a second time.

If struck to a thermally stable electrode, the arc has no apparent effect on the ICP discharge itself except to lower the power delivered to the fireball. A rough measure of the amount of power being siphoned from the plasma by the arc was obtained by measuring the continuum intensity at 300 nm with the arc on at a power of 1.5 kW. The plasma

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was then re-lit with the arc off and the power was reduced until the continuum intensity at 300 nm matched that measured earlier with the arc on. The match occurred after a reduction in rf power of 140 watts.

The arc was further characterized by measuring with an A.C. current probe (Tektronix model no. 6022) and oscilloscope the current passing through the lead connecting the copper base to ground. The oscilloscope trace revealed the current to be nearly sinusoidal at 27 MHz and to vary with applied rf power as shown in Figure 2. The difference in Figure 2 between the current measured with a tungsten-pin electrode and that passed by a copper-cylinder sample is probably due to thermionic emission and the difference in temperature reached by the two electrodes. The 0.8-mm tungsten pin had poor thermal contact with the copper heat sink and glowed white hot. In contrast, the 6-mm copper cylinder remained relatively cool. Thermal emission of electrons from the tungsten would lower the impedance of the arc channel and raise the current. A surprising result of the current measurements was the discovery of a substantial current that is present even when the arc is not lit (cf. Figure 2). This current presumably results from capacitive coupling between the copper torch base and the water inlet block on the high-voltage side of the load coil.

RESULTS AND DISCUSSION

The effectiveness of the rf arc as a sampling device was tested in preliminary experiments on three different types of samples: metal alloys, non-conducting powders, and microliter quantities of solutions. Two different detection schemes were used. In all cases the plasma was imaged with a quartz lens at unit magnification onto the entrance slit of a 0.35 m monochromator (Model EU-700, GCA McPherson, Acton, MA). For the alloys, the elemental line of interest was located with the aid of a hollow-cathode lamp. The output of a 1P28A photomultiplier tube, operated at 800-900 V, was then integrated at each wavelength for a series of one-second intervals by a digitizing gated integrator (model 720 VAIADC, Keithley Instruments, Cleveland, OH). The digitized intensities were stored in a laboratory computer (MINC-11/03, Digital Equipment Corp., Maynard, MA). For the powders and solutions the photomultiplier was replaced by a cooled 1024-element photodiode array (model 1024 S, Reticon Inc., Sunnyvale, CA) operated as described by Horlick (25).

Aluminum and stainless-steel alloys of the nominal composition shown in Table I were prepared in the form of 3.2-mm diameter cylinders, 3.2 mm in length. At a plasma forward power of 1.5 kW, the samples melted within a few seconds of arc ignition. The time-dependent emission curves from two samples of 2024 aluminum are shown in Figure 3. From Figure 3, sampling of alloys by the arc appears predominantly thermal after the first few seconds; the elements appear to distill from the sample in order of their volatility.

The thermal behavior illustrated in Figure 3 hinders the use of this new arc sampling technique for quantitative analysis. Problems such sampling might cause include: 1) the introduction of unusually large fluxes of matrix or minor-constituent (< 1%) elements into the plasma, a situation which could result in nonlinearities in plasma emission, severe line broadening and background shifts (26); 2) delayed sampling of nonvolatile components with attendantly long analysis times;

3) the likely need for total sample consumption; and 4) irreproducible transient signals.

The first of these problems is particularly evident in the time dependence of Mg emission shown in Figure 3. The peak photocurrent in trace b is greater than 400 μA (monochromator slits -- 25 μm x 3 mm), even though Mg comprises only 1.5% of the ~ 70 mg sample. The sample introduction rate corresponds approximately to that which would be achieved by aspirating with an efficient pneumatic nebulizer a 4% Mg sample solution into the ICP. Figure 4 illustrates the fourth problem; the time-dependent emission from Cr in 304 stainless steel, sampled by the RF arc, is extremely erratic and does not reach a steady level even after 15 minutes.

Because of the above limitations, the rf arc does not in its present form appear to be suitable for the general analysis of solid alloys. It might, however, be useful for the determination of trace impurities in relatively pure or consistent alloy matrices.

The next sample type studied, non-conducting powders, required a different sampling arrangement. For such samples, the best results to date have been obtained with the sample packed into small graphite cups. The cups had the same outer dimensions as the metal cylinders described earlier (3.2 mm dia.), but had a 1.6-mm diameter cavity drilled in one end. This configuration proved effective for sampling of inorganic materials, but was unsuitable for samples with high organic content. Organic-based samples extinguished the rf arc shortly after its ignition.

Sampling and emission from a non-conducting inorganic powder, NBS coal fly ash (SRM 1633a) were studied in detail. Spectra were integrated from a 3-mg sample of the fly ash for periods of either 9.2 or

55.2-seconds by summing 10 or 60 0.92-second integrals from the photodiode array. The 9.2-second integrals produced higher signal-to-background ratios than the 55.2-second integrals, indicating that emission from the studied elements is most intense during the first few seconds of the rf arc "burn". This observation was confirmed by monitoring on an oscilloscope the video output from the diode array as the array was repetitively read during a burn.

Analysis of the spectra from the coal fly-ash sample was complicated by the detailed spectral background generated by the large amounts of Fe, S, and Ti which the sample contains. This complicated background, combined with the limited resolution of the spectrometer (FWHM > 0.5 Å), obscured large spectral regions and made positive identification of several sample constituents impossible. Despite this limitation, a range of elements was identified, and produced easily detectable signals even in the spectral regions where the photodiode array is relatively insensitive (< 300 nm). Table II summarizes the results of a qualitative analysis (9.2-second burn) of the fly-ash sample spectrum.

The results in Table II indicate that the rf arc is capable of sampling both volatile and refractory elements in the course of a 9.2-second burn. This observation and the fact that the fly-ash sample is not totally consumed even after several minutes suggest that processes other than the thermal distillation observed for the metal alloys are involved in the sampling of the fly-ash powder.

Clearly, the new method appears to be well suited for the qualitative analysis of powdered inorganic samples. Before it can be used quantitatively, however, more development is required. In this preliminary study, the sampling of the fly ash was erratic. Moreover, because the sample was not totally consumed during the integration period, the emission results were not quantitatively reproducible. For example, the run-to-run variation in the intensity of the Zn 213.8 nm line exceeded 40%.

Using the rf arc for sampling microliter quantities of solution required further modification of the sample-containment device. For each run in these liquid-sampling experiments a 1-μl droplet containing 100 ng of Cd was placed on the flat tip of a length of 0.9 mm HB Pentel graphite pencil lead, employed as a convenient electrode. The sample droplet was then desolvated by directing at it for 30 sec the output of a heat gun. A second 1 μl droplet, containing 100 ng of Zn, was next placed on the tip of the pencil lead and similarly desolvated. The pencil lead was then mounted in place of the tungsten pin used for previous samples, so that it was anchored in the torch base and extended above the top of the boron-nitride pedestal into the bell jar chamber (cf. Figure 1).

The time-dependent emission from the two solution-borne elements was monitored during a single burn by recording 32 successive 1.85-second integrals from the photodiode array. The results, plotted in Figure 5, suggest that both elements are dislodged when the arc is struck, either by sputtering or mechanical shock. The first readout of the array after arc ignition registers the most intense Zn and Cd lines. What follows seems to be thermal evolution similar to that observed with the direct-sample-insertion techniques (21). Both zinc and cadmium appear to have nearly volatilized completely after 40 seconds. Presumably, zinc emission remaining after 40 seconds arises from zinc in the pencil-lead electrode. A subsequent burn of a pencil-lead electrode without any added sample solution produced zinc emission of intensity comparable to that in Figure 5.

Because liquid samples are totally consumed in the present procedure, the rf-arc measurements are more reproducible than those from powders or alloys. Measurements of intensities for four runs, each obtained by averaging thirty 1.85-second integrals from the photodiode array, yielded a relative standard deviation of 9.8% for the Cd 214.4-nm line and 19% for the Zn 213.9-nm line. The difference in precision between the two elements can be attributed to irreproducibility in the erosion of the zinc-containing pencil-lead substrate. Much of the remaining imprecision in the intensities can be attributed to factors other than sampling. For example, droplets were delivered with a microliter syringe with an estimated precision of 2-5%. In addition, the present rf-arc configuration requires the ICP torch to be removed and replaced after each burn. Small differences in torch position can have large effects on the observed emission.

The 100-ng solution samples produced spectra with high signal-to-noise ratios. For example, the signal-to-noise ratio for the Cd 214.4 nm peak was greater than 250. No effort was made in these measurements to optimize the viewing region in the plasma for sensitivity. It should be possible, with some optimization and the use of a photomultiplier tube, to detect subnanogram quantities of these (and probably other) elements.

Although the rf arc as a sampling device is clearly not a universal solution to the problem of introducing solids or small samples into the ICP, it does show promise for certain samples. It is particularly appealing in that it is inexpensive and simple. It requires no external power source and can be easily incorporated as an accessory into an existing ICP system. These preliminary results warrant a detailed study to further probe its capabilities and limitations and to explore its applicability to a wider range of sample types.

ACKNOWLEDGEMENT

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P. B. Farnsworth, Department of Chemistry, Brigham Young University, Provo, Utah 84602.

G. M. Hieftje, Department of Chemistry, Indiana University, Bloomington, Indiana 47405.

Table I. Nominal Composition of Several Alloys Used in Preliminary rf-arc Experiments

Alloy	Concentrations of Alloying Elements(%)	
2024 Al	Cu	4.5
	Mn	0.6
	Mg	1.5
6061 Al	Cu	0.25
	Si	0.6
	Mg	1.0
	Cr	0.2
304 Stainless Steel	C	0.15
	Mn	2.0
	P	0.045
	S	0.03
	Cr	18-20
	Ni	8-12

Table II. Qualitative Analysis of Spectrum from a 3-mg Sample of NBS SRM 1633 A (Coal Fly Ash), Volatilized by the rf Arc into a 1.5 kW ICP[a]

Detected		
Element	Concentration in Sample	Line(s) (nm)
Si	22.8%	many
Al	14%[c]	many
Fe	9.40%	many
K	1.88%	766.49
		769.90
Ca	1.11%	393.37
		396.85
Ti	0.8%[c]	many
Mg	0.455%	279.55
		280.27
Na	0.17%	589.59
Ba	0.15%[c]	230.43
		233.53
V	300 ppm[c]	310.23
		311.07
Zn	220 ppm	213.86
		206.2
Cr	196 ppm	283.56
		205.55
As	145 ppm	193.66
Sr	130 ppm	407.77
		421.55
Ni	127 ppm	232.00
Cu	118 ppm	327.40

Not Detected	
Element	Concentration
Mn	190 ppm[b,c]
Ce	180 ppm[c]
Rb	131 ppm
Pb	72.4 ppm[b]
Ga	58 ppm[c]
Co	46 ppm[c]
Mo	29 ppm[c]
Th	24.7 ppm
Be	12 ppm[b,c]
U	10.2 ppm
Hf	7.6 ppm[c]
Sb	7 ppm[c]
Tl	5.7 ppm
Eu	4 ppm[c]
Cd	1 ppm
Hg	0.16 ppm

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[a] All measurements made 20-22 mm above load coil.

[b] Spectral interferences from other elements on all strong lines.

[c] These concentrations not certified; all others are.

FIGURE CAPTIONS

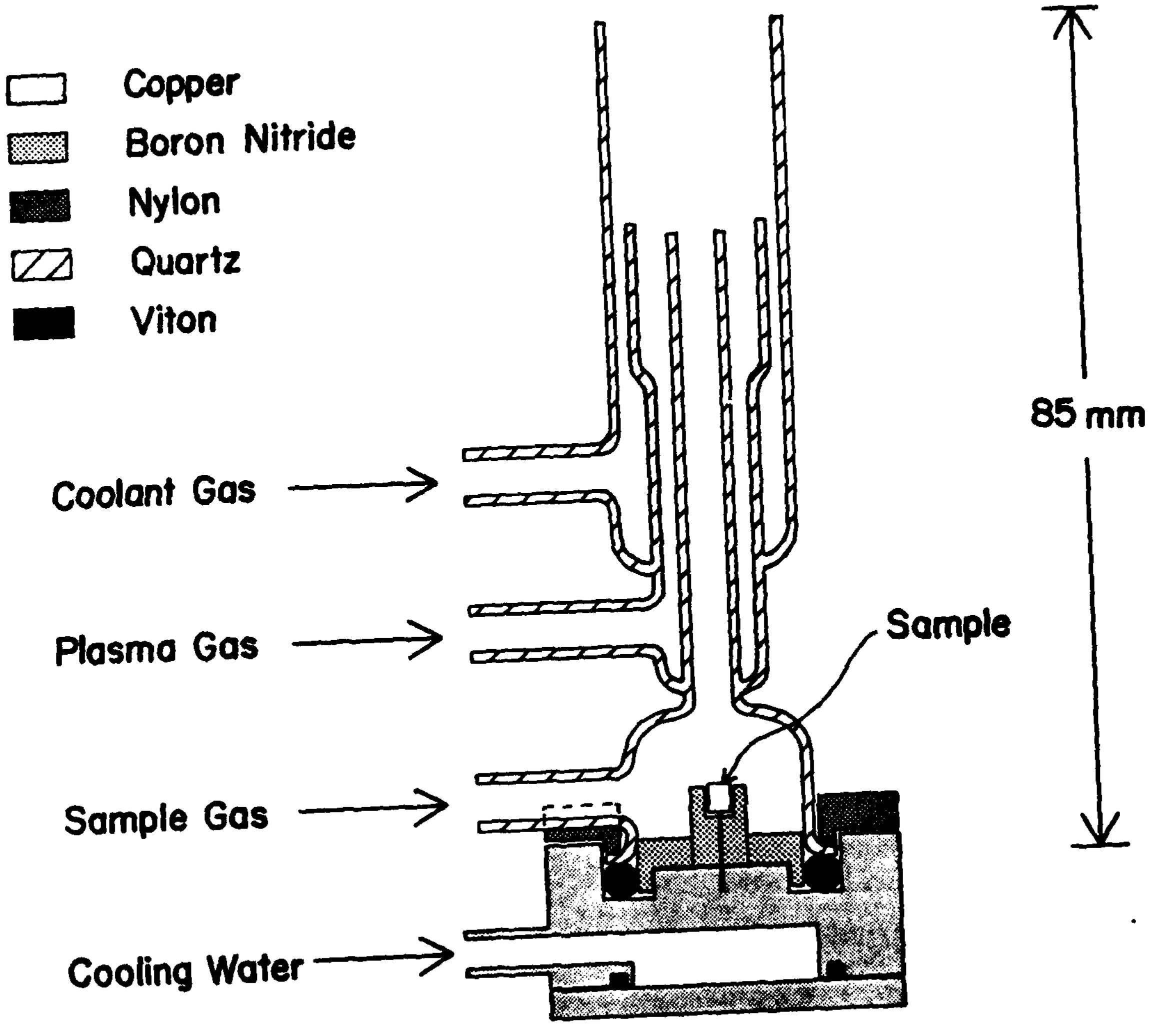
Figure 1. Schematic diagram of torch and sample-stand assembly used in the rf-arc sampling experiments.

Figure 2. Radio-frequency current from torch base to ground as a function of applied rf power for an arc to a tungsten pin (0.8-mm diam.), an arc to a cooled copper cylinder (6-mm diam.) and no arc.

Figure 3. Time-dependent emission from a) Cu 324.8 nm and b) Mg 279.5 nm lines from solid 2024 aluminum alloy sampled by the rf arc into the ICP. See text for experimental conditions.

Figure 4. Time-dependent ICP emission from Cr 283.6 nm line volatilized from 304 stainless steel by the rf arc. Experimental conditions as for Figure 3.

Figure 5. Time-dependent emission from Zn 213.8 and Cd 214.4 nm lines. (100 ng solution-based samples placed on tip of 0.9 mm pencil lead, observation zone 15-17 mm above load coil.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Sample
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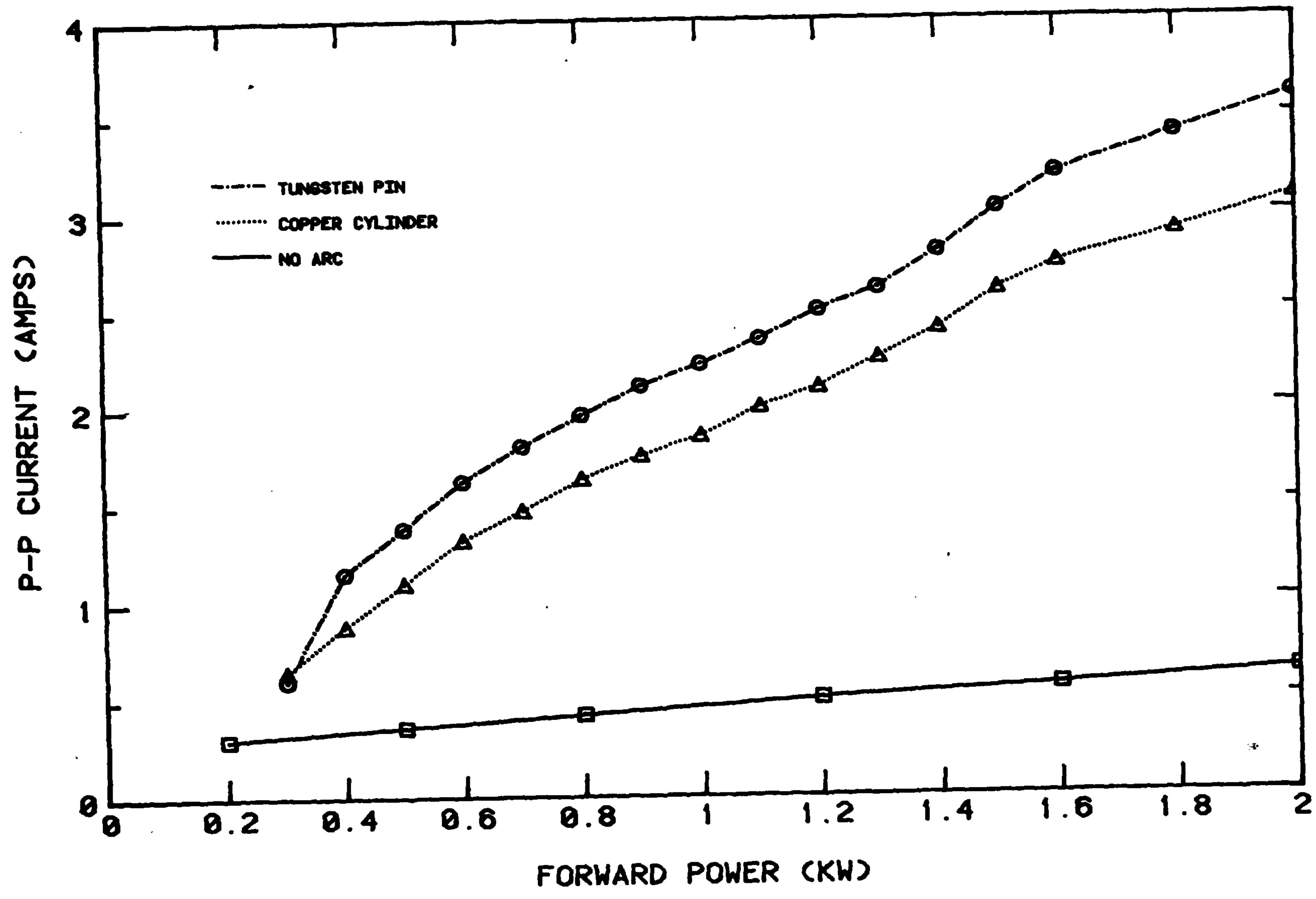
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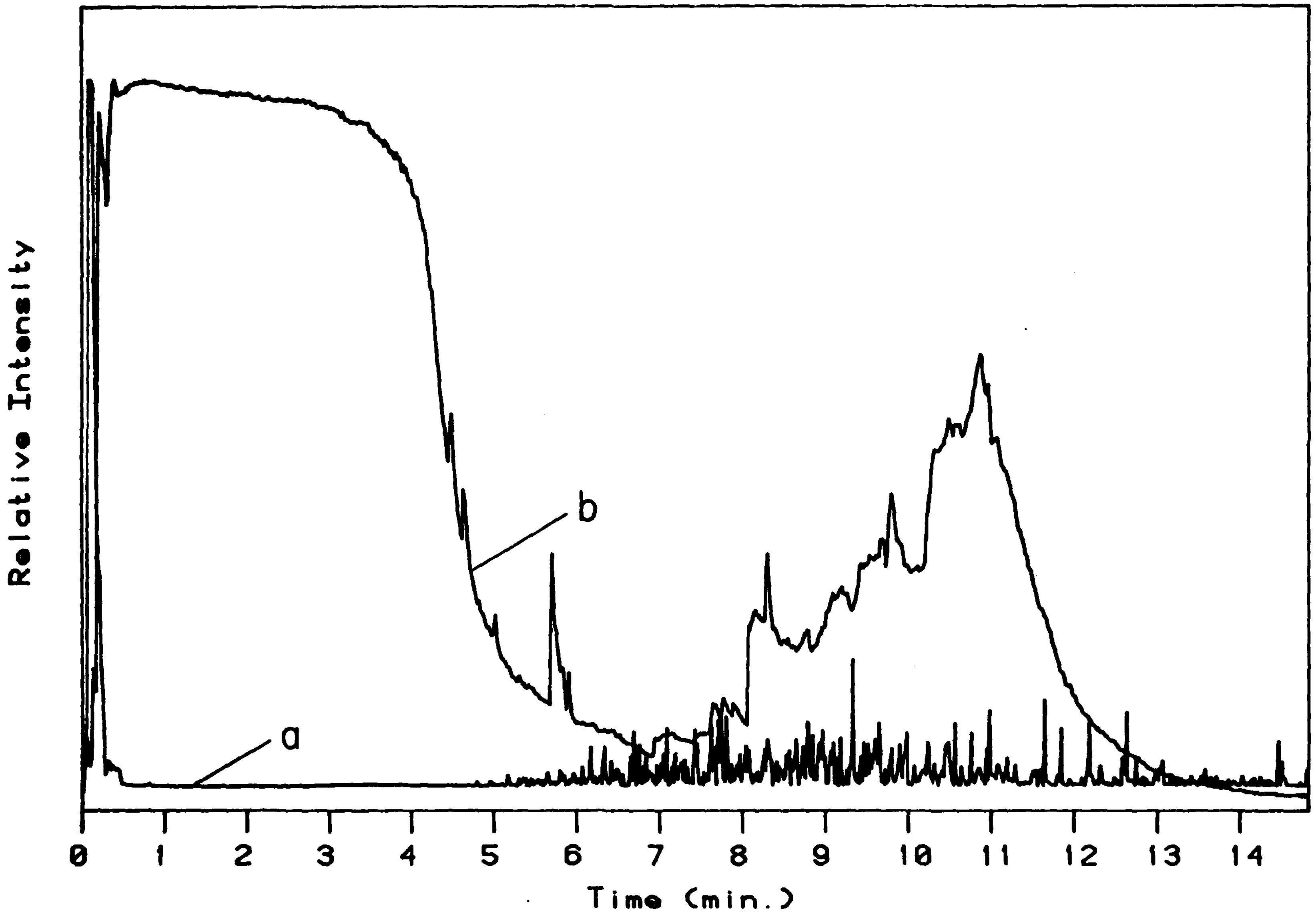
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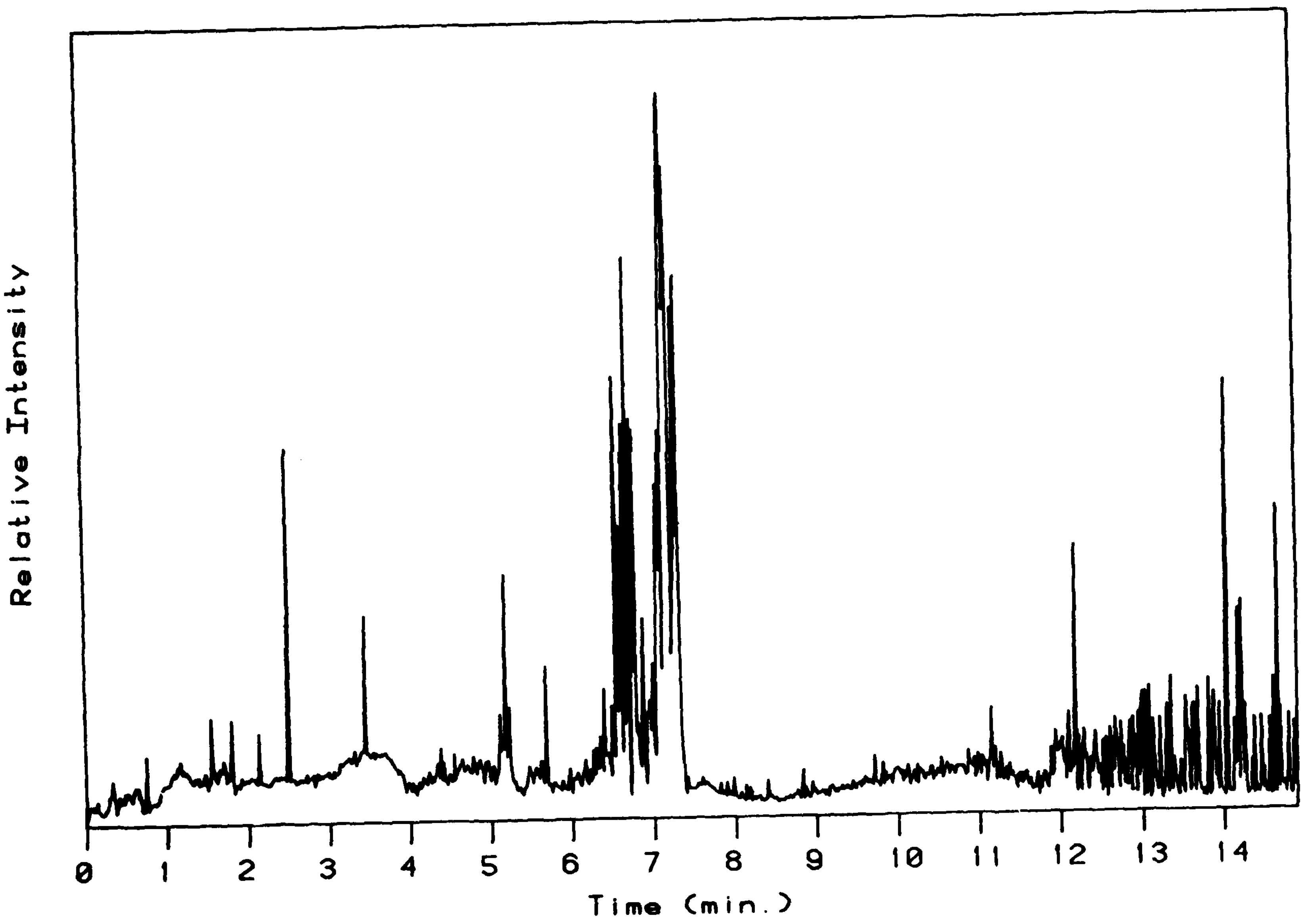
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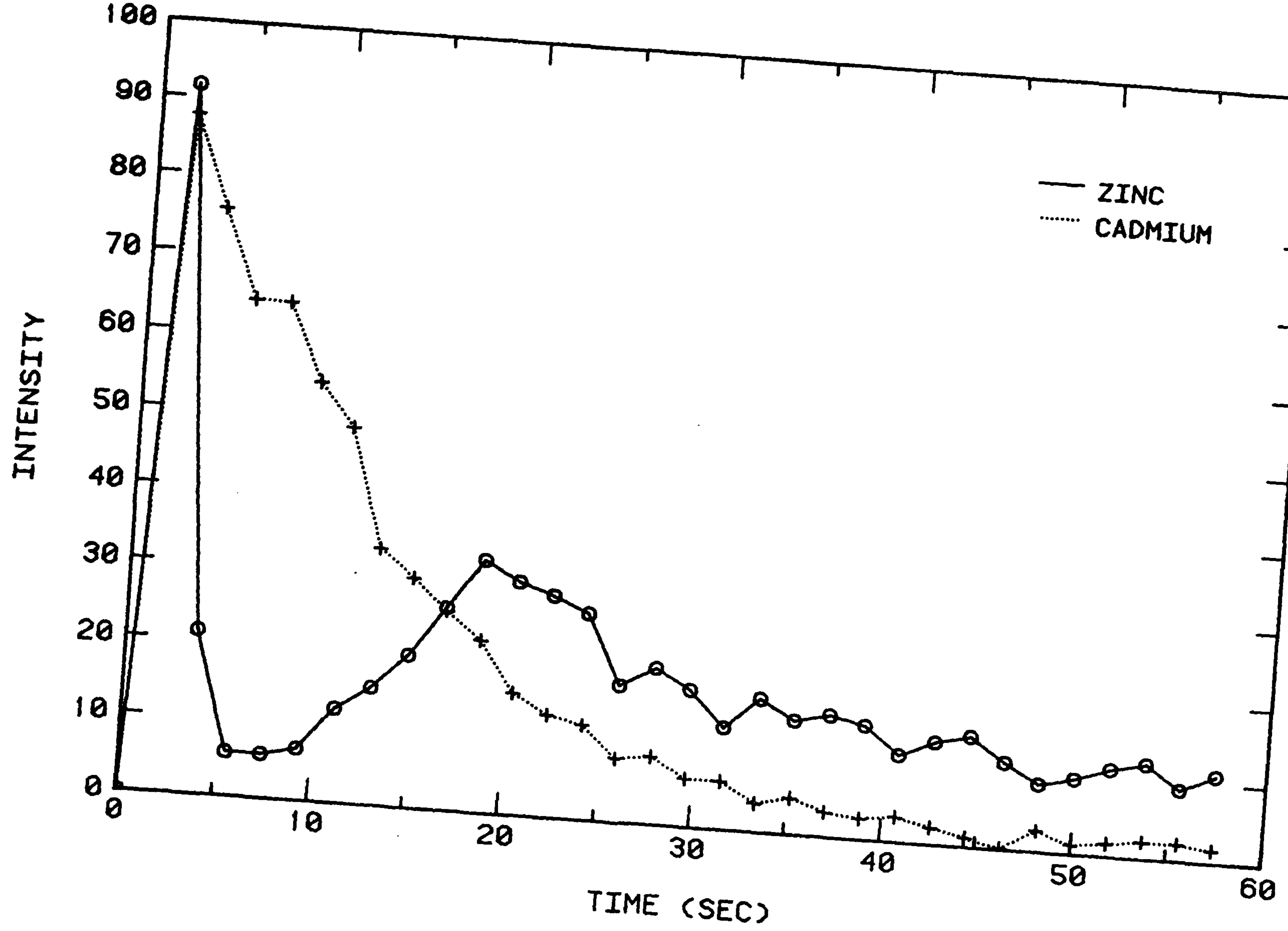
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