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THE USE OF A MICROARC ATOMIZER FOR SAMPLE INTRODUCTION INTO AN --ETC(U)
SEP 82   J P KEILSOHN, R D DEUTSCH, G M HIEFTJE   N00014-76-C-0838
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The use of a microarc atomizer as a device for microsample introduction into the ICP is described. The microarc, a high-voltage, low-current, pulsating dc discharge, efficiently vaporizes desolvated, discrete microvolumes of sample solution into the plasma. The microarc-ICP combination exhibits relative detection limits and working curves comparable to those of other methods of sample introduction but functions with sample volumes in the 0.5 - 1.0 μL range. Matrix and ionization interference effects are absent or trivially overcome. Computer control of the instrument adds simplicity of operation.

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THE USE OF A MICROARC ATOMIZER FOR SAMPLE INTRODUCTION INTO AN INDUCTIVELY COUPLED PLASMA

by

J. P. Keilsohn, R. D. Deutsch, and G. M. Hieftje

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Bloomington, Indiana 47405

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INTRODUCTION

The inductively coupled plasma (ICP) has shown itself to be an excellent source for atomic emission spectroscopy. The number of papers published since its inception illustrates the utility of the technique.[1] Much of the ICP research today involves the use of smaller plasma torches,[2-5] lower rf powers[6] and argon flow rates,[2-6] different plasma gases,[6-8] and novel methods of sample introduction,[9-16] all in an effort to improve sensitivity and precision and to reduce costs.

Many types of sample-introduction techniques have been explored for use with the ICP. Prominent among these techniques are pneumatic nebulization (based on Meinhard,[10] ultrasonic,[10] cross-flow[11] or Babington[12-14] devices) and electrothermal methods (based on tantalum-filament[15] and graphite-furnace[16] systems). Unfortunately, these devices require rather large volumes of sample solution for reliable operation. This required volume ranges from tens of milliliters for pneumatic nebulizers to 100 microliters in the case of the tantalum filament vaporizer.[15]

In the present study, we explore the use of a "microarc" for sample solution vaporization and introduction into the ICP. The microarc enables the use of sample volumes between 0.1 - 10 μL and operates especially conveniently on volumes in the 0.5 - 2 μL range.

The microarc is essentially a high-voltage, low-current, atmospheric-pressure, pulsating dc discharge which atomizes individual samples of metal salts rapidly and completely.[17] The arc combines high temperature and ion sputtering to vaporize even refractory samples, so that matrix interferences are minimized.[17,18] These advantages accrue partially because the microarc separates in time the processes of solvent evaporation, sample decomposition, and atomic vapor production,[17-19] so each process can be optimized in efficiency.

The microarc was originally developed for use with a microwave-induced plasma,[17,18] where the ability to separate temporally the process of sample desolvation from other atomization events is a great asset. The fundamental operating characteristics of the microarc have been examined and described elsewhere.[19]

EXPERIMENTAL

Apparatus. A block diagram of the overall experimental system is shown in Figure 1. The microarc electrode holders and arc stand, which have been modified from those previously described,[17,18] are constructed entirely of glass and are shown in cut-away view in Figure 2. The microarc sample electrode (cathode, A in Figure 2) is constructed from a loop of 0.25 mm (AWG No. 30) tungsten wire which has been heat-sealed into a piece of glass tubing (6 mm o.d.). The glass tubing allows the electrode to be adjusted in the arc stand much more easily than previous arrangements[17-19] and adds rigidity to the electrodes.

The microarc anode is constructed from a stainless-steel wire which is affixed with epoxy into a piece of glass tubing (6 mm o.d., B in Figure 2). A 24-gauge syringe needle with the end sealed provides a readily available source of stainless steel wire. Because of the difficulty of heat-sealing stainless steel wire into a glass tube, the glass tube was first constricted by fire polishing, the anode wire slipped through it, and epoxied in place. The anode wire extended 20 mm past the end of the glass tube.

The microarc stand was also constructed of glass. The two electrodes are held in place by an o-ring and 'Ace Thred' (Ace Glass, Inc., Vineland, NJ) fitting affixed to the end of 15 mm (o.d.) glass tubing. The two holders meet at a 90° angle; this arrangement permits easy alignment and replacement of the electrodes. The all-glass construction provides convenient viewing of the arc and electrodes during sample placement and microarc operation. Routine cleaning of the arc stand is also simplified by the all-glass construction.

The sample gas flows over the microarc electrodes and carries vaporized sample rapidly into the ICP through the sample tube of the plasma torch. A rubber septum (not shown in Figure 2) is attached to the arc stand at a right angle to the plane of the electrodes to allow the sample to be applied to the cathode without disruption of the sample-gas flow.

Desolvation of sample solution deposited on the cathode is accomplished by ohmic heating of the cathode wire with a constant-current supply as previously described.[17] The power supply for the microarc discharge itself is the same as previously described[17] and operates at 1500 volts and 20 milliamps. For the present work the mechanical relay in the arc power supply was replaced by a solid-state device to eliminate electrical noise generated by the relay contacts. The arc power supply was controlled by a laboratory-constructed microarc timer, which enables the arc operating duration to be varied from 0.1 - 999.9 seconds in 0.1 sec increments. In operation, the desired arc time can be controlled either manually by a group of thumbwheel switches on the front panel of the arc timer or remotely through a parallel connector on the back panel of the timer. The starting of the timer (and therefore of the microarc) can also be controlled either from a front-panel push button or remotely. Operationally, the arc timer consists simply of a programmed relay which in turn controls the on-off relay in the microarc power supply.

ICP. A conventional ICP torch (20 mm o.d.) operating from a 27.12 MHz power supply (Model 2500, Plasma-Therm, Kresson, NJ) was used for the present work. Operating conditions of the ICP are listed in Table 1. The plasma was imaged 1:1 onto the entrance slit of a monochromator (Model EU-700, GCA McPherson, Acton, MA) by means of a 5-cm diameter quartz lens (f = 10 mm). The dispersed radiation was detected by a photomultiplier tube (1P28, RCA, Lancaster, PA), converted to a proportional voltage and amplified (Model 244, Keithley Instruments, Inc., Cleveland, OH).

Reagents. Stock solutions were prepared as described by Dean and Rains.[20] All salts and acids were reagent-grade and water used in dilution was distilled-deionized.

Data Collection and Control. A MINC-11 Laboratory Computer (Digital Equipment Corp., Maynard, MA) was used for all experimental control and data collection. The computer system was operated under MINC BASIC (Digital Equipment Corp.), chosen for its simplicity in generating graphical representations of collected data on the terminal screen. The role of the computer can best be understood from the instrument block diagram (Figure 1) and from a description of a typical data-collection sequence.

Once the control program is started, the computer prompts the operator via a CRT for identifying information, such as sample name and desired element and wavelength. The time period desired for microarc operation is also entered by the operator, who then places the sample onto the sample cathode using a microliter syringe (Model 7101N or 7105N, Hamilton Co., Reno, NV). The computer is informed of the sample volume and, for the determination of working curves or detection limits, the sample concentration.

The computer then waits a period of time (usually 30 seconds) for the sample to be desolvated by the heated sample electrode. The desired arc "on" time (usually 5 seconds) is loaded by the computer into the microarc timer through its Digital Out module. The timer is started and, at the same time, data collection is begun by the computer. Data are collected using the MINC Pre-amplifier and Analog-to-Digital Converter modules during the entire period of time that the arc is on.

The starting of the microarc timer results in automatic striking of the microarc by means of a high-voltage pulse supplied by the arc power unit.[17] The sample is then sputtered from the cathode surface by the microarc, destroying the sample matrix and liberating atomic vapor. The atomic vapor is swept through the ICP torch sample tube and into the plasma by the sample gas passing over the arc.

After the resulting atomic emission peak is recorded, the collected data are displayed on the terminal screen in a graphic (time vs. emission intensity) form. After a few seconds for arc cooling and data manipulation, the arc is restruck, following the same procedure as during sample vaporization. However, no sample is added to the electrode for this second sequence, so a background time-profile is obtained. A typical set of signal and background curves is shown in Figure 3 for a 1 ng (1 μL of 1 μg mL^{-1}) sample of copper.

It was found previously[17,18] that the difference in areas between the signal and background curves in Figure 3 should be proportional to the sample mass. The signal area (integral) is found by summing the data points for the analyte (curve A of Figure 3) and subtracting the sum of the background data points (curve B of Figure 3). The computer saves all the recorded data on its floppy disk and calculates detection limits and working curves using the collected data.

RESULTS AND DISCUSSION

Plasma Viewing Region. The imaging optics employed in this work permit only a 1-cm vertical portion of the plasma to be viewed at any one time. This limitation necessitated the determination of the optimal viewing height and sample-gas flow rate before the analytical utility of the ICP-microarc combination could be evaluated.

Figure 4 shows the effect of sample-gas flow rate on the observed signal from 10 ng copper at three different viewing heights in the ICP. Each measurement plotted in Figure 4 is the mean of ten successive determinations. The maximum signal (analyte minus background integrals) was found to be in the region from -2.5 mm to 7.5 mm, referenced to the top of the plasma load coil and at a sample-gas flow rate of 1 L/min. The spatial behavior of Figure 4 is similar to that reported by Blades and Horlick[21,22] for the ICP. The effect of flow rate most likely results from a combination of analyte diffusion and dilution effects, with diffusion predominating at lower gas-flow rates and dilution at higher rates. The optimal conditions in Figure 4 (1 L/min Ar flow and -2.5 to 7.5 mm viewing region) were employed for all later determinations. Interestingly, the same optimal viewing region was found using a pneumatic nebulizer operating at the same gas-flow rate.

Detection Limits and Working Curves. Detection limits were determined at the 95% confidence level from the signal-to-noise ratio.[23] In turn, the signal was defined as the integral (area) of the sample trace minus the integral of the background trace; the noise level was calculated as the standard deviation of the areas of ten background traces. No blank was needed; not surprisingly, it was found that the desolvated blank (usually water) resulted in the same signal as generated by the bare electrode itself. Detection limits were computed by extrapolation to a signal-to-noise ratio of 2.262 ($\alpha = 0.05$, $N = 10$).[23]

Table II lists detection limits for a number of elements using the microarc-ICP combination. The values are presented in terms of both analyte mass and concentration to simplify comparison to other work.[15,24] From these results, the microarc-ICP system is capable of concentration detection limits which are comparable to those of methods which require much larger sample volumes and sample masses. The poor detection limit for iron results from a high background noise level at the analyte line.

Linear calibration curves for the microarc-ICP system range over 5-6 orders of magnitude for most elements. A typical set of calibration curves is shown in Figure 5.

Interferences. To examine the susceptibility of the microarc-ICP combination to interferences, two classical interference systems were examined: that of sodium on calcium and that of phosphate on calcium. Both studies were conducted using 10 $\mu g\ mL^{-1}$ calcium solutions with added amounts of sodium (as NaCl) or phosphate (as H_3PO_4). The results of these studies are shown in Figures 6 and 7.

The flatness of the calcium-phosphate curves (cf. Figure 6) indicates the freedom from matrix interferences which the microarc-ICP arrangement provides; this finding is consistent with that shown from previous work in a microwave-induced plasma.[17,18] In contrast, some effect of added sodium on calcium emission exists (cf. Figure 7) but the influence flattens out at approximately a two-to-one ratio of sodium to calcium. This behavior is similar to that observed with other ICP sample-introduction devices, but the flattening of the interference curve (cf. Figure 7) occurs here at a much lower sodium-to-calcium ratio.[3,5] Several alternatives might exist to eliminate this interference: the first would be to add excess sodium to all samples and standards; the other possible solution would be to optimize the viewing region for minimization of this interference[21,22] rather than for maximization of signal-to-noise ratio.

Precision. At elemental concentrations at least twenty times the detection limit, the microarc-ICP combination exhibits a relative standard deviation of from 1 to 5% depending on the element. Many factors appear to affect this precision, although the most important is the purity of the sample-gas argon. Bottled argon decreases the stability of the microarc more than liquid argon. The sample electrode material and shape also affect precision although the reason for this influence is not yet clear.[19]

Determination of Several Elements in NBS SRM 1571 Orchard Leaves. To assess the practical utility of the microarc-ICP combination, the determination of several elements in a "real" sample was undertaken, Table III lists the determined values for three elements along with the corresponding certified values for NBS SRM 1571. Digestion of the sample was by standard methods,[25] with nothing additional being added to the sample. All concentrations were determined from calibration curves. The results indicate that the microarc-ICP combination performs with good sensitivity and accuracy in the routine analysis of a biological material.

CONCLUSIONS

The use of the microarc atomizer as a sample introduction device for the ICP is attractive for small sample volumes, since little sensitivity and precision are lost. Yet, microliter sample sizes can be used. The microarc-ICP combination should be well suited for analyses where only small volumes of sample are available, such as biological applications, but where high sensitivity is desirable or necessary.

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Table I. General Operating Conditions for the Microarc-ICP

rf power	1.25 kW
gas flows, L min^{-1}	
coolant	17.5
plasma	0.4
nebulizer (a)	1.0
Monochromator entrance and exit slits	50 μm x 5 mm
PMT supply voltage	800 V
Time constant of detection system	30 ms

(a) See text for discussion

Table II. Detection Limits Obtained with the Microarc-ICP Combination

Element	Wavelength nm	Microarc		ICP with Tantalum Filament Vaporization[a] pg	Conventional[b] ICP ng/ml
		pg	ng/ml		
Ba (II)	455.4	0.11	0.22	0.03	0.87
Ca (II)	393.6	0.06	0.06	-	0.13
Cd (II)	226.5	47.	15.	600.	2.3
Cu (I)	324.7	3.9	1.9	-	3.6
Fe (II)	259.9	44.	29.	-	4.1
Li (I)	670.7	0.87	0.87	-	1.9
Mg (II)	279.5	0.4	0.4	-	0.1
Na (I)	589.0	3.6	3.6	-	19.
Ni (II)	221.6	2.3	3.6	-	6.7
Pb (I)	405.7	280.	280.	300.	180.
Zn (I)	213.8	0.12	0.08	-	1.2

[a] Ref. 15

[b] Ref. 24

Table III. Analysis of NBS Standard 1571 Orchard Leaves

Element	Wavelength (nm)	Present Study (wt %)	Certified Value (wt %)
Ca	393.6	2.10	2.09 ± 0.03
Mg	279.5	0.61	0.62 ± 0.02
Fe	259.9	296[a]	300 ± 20[a]

[a] μg/g

Figure Captions

Figure 1. Block diagram of the Microarc-ICP Instrument. PMT - photomultiplier tube; H.V.P.S. - high-voltage PMT power supply; i-to-V amp - current-to-voltage converter. See text for details.

Figure 2. Microarc stand, Electrode holders, and Electrodes.

A - sample cathode loop (tungsten)
B - Anode (stainless steel)

Figure 3. Typical emission-time curves for the microarc-ICP combination obtained using 1 ng (1 μL of 1 μg/mL) copper (324.7 nm).

A - Sample emission curve
B - Background emission curve

Figure 4. Effect of viewing region and nebulizer flow rate on copper emission (10 ng sample). Heights measured relative to the top of the ICP load coil.

0 — +3.5 mm to +13.5 mm
+ — -2.5 mm to +7.5 mm
* — -10 mm to 0 mm

Figure 5. Typical working curves for Microarc-ICP System. Note: curves for Cu and Fe are nearly congruent.

* - Cu (I) 324.7 nm
X - Fe (II) 259.9 nm
+ - Mg (II) 279.5 nm
0 - Na (I) 589.0 nm

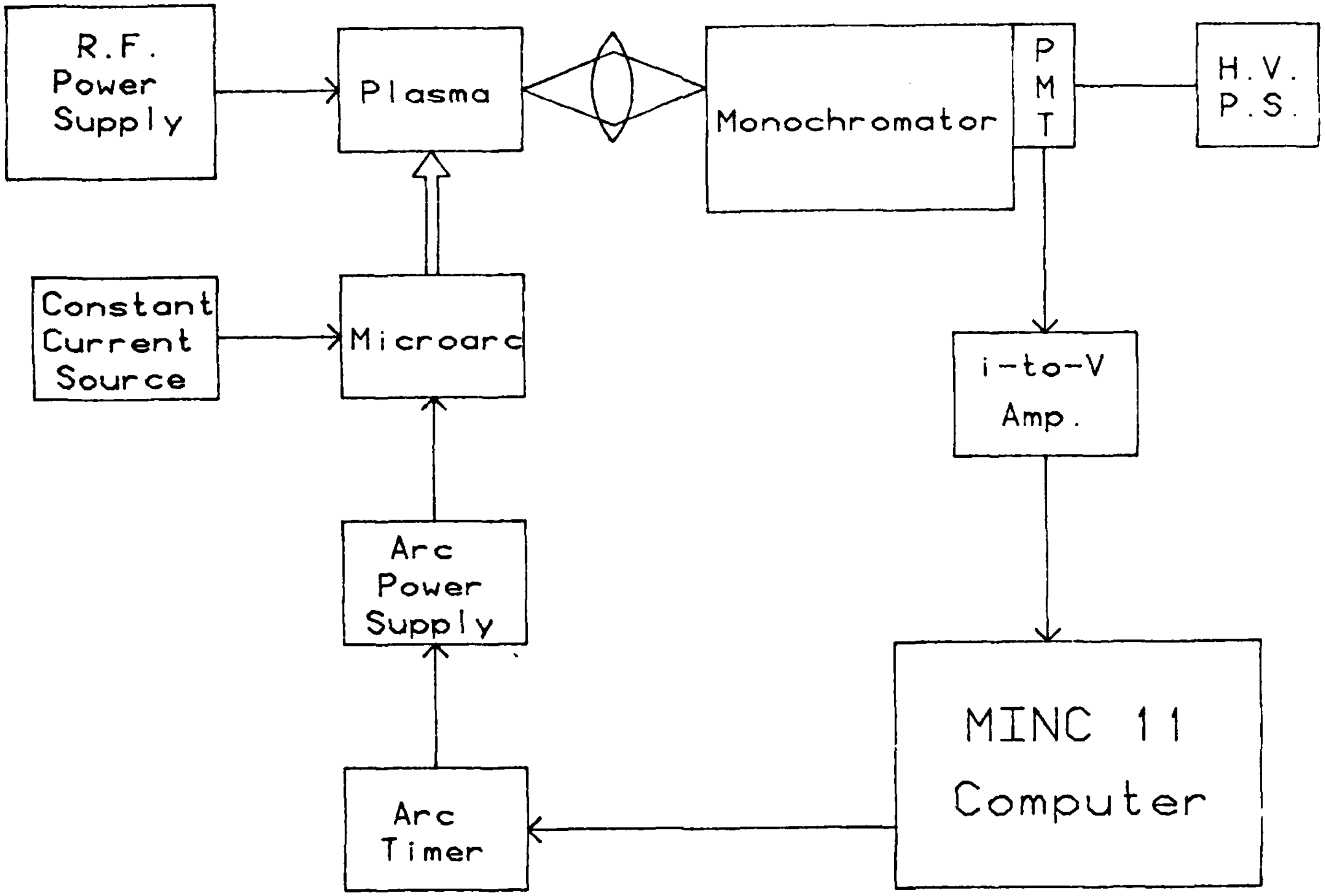
All working curves have been extrapolated to include the detection limit.

Figure 6. Phosphate Interference on 10 $\mu g\ mL^{-1}$ Calcium. Ca (II) - 393.4 nm.

Figure 7. Sodium Interference on 10 $\mu g\ mL^{-1}$ Calcium.

O - Ca (I) 422.6 nm

★ - Ca (II) 393.4 nm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Microarc
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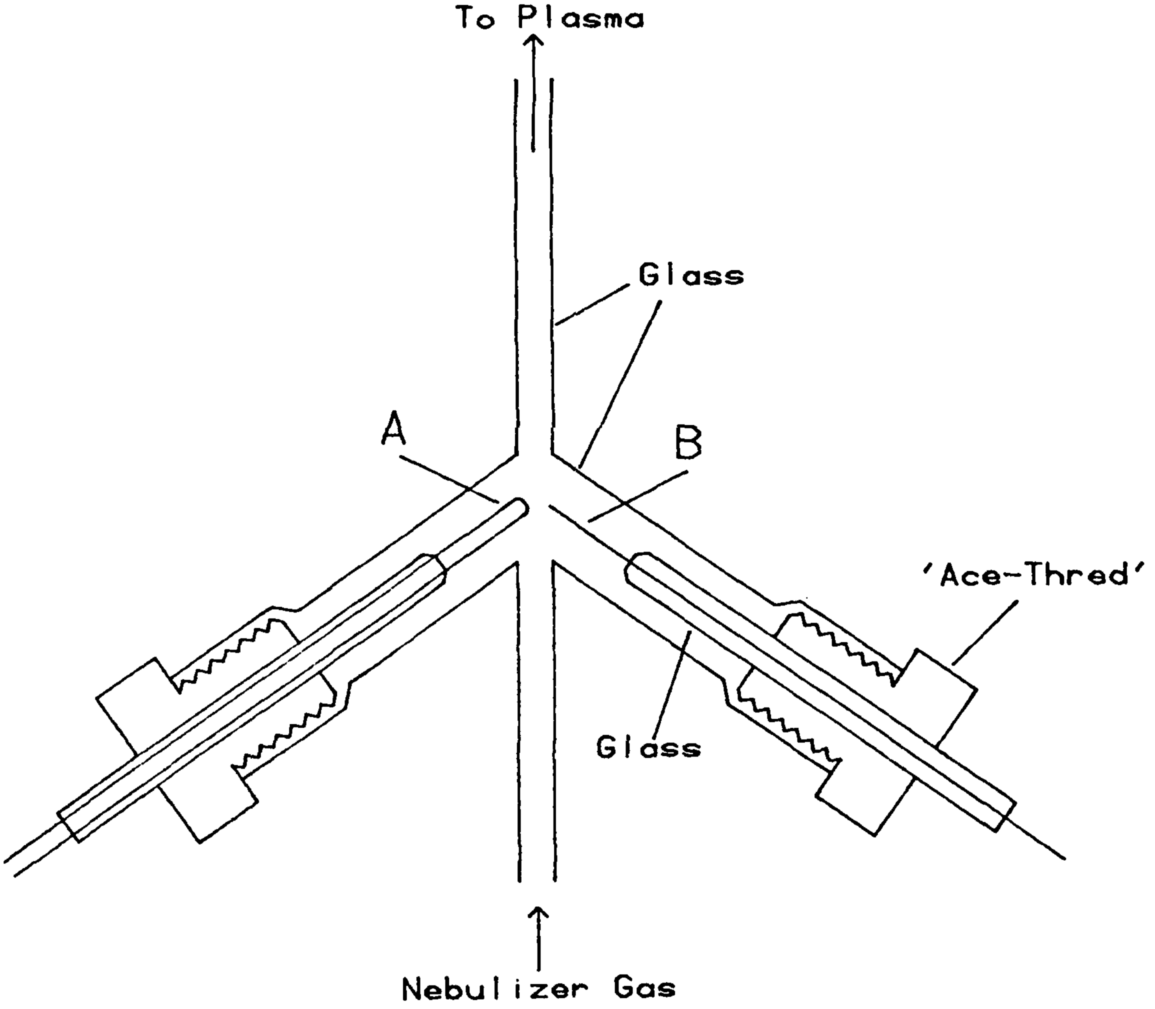
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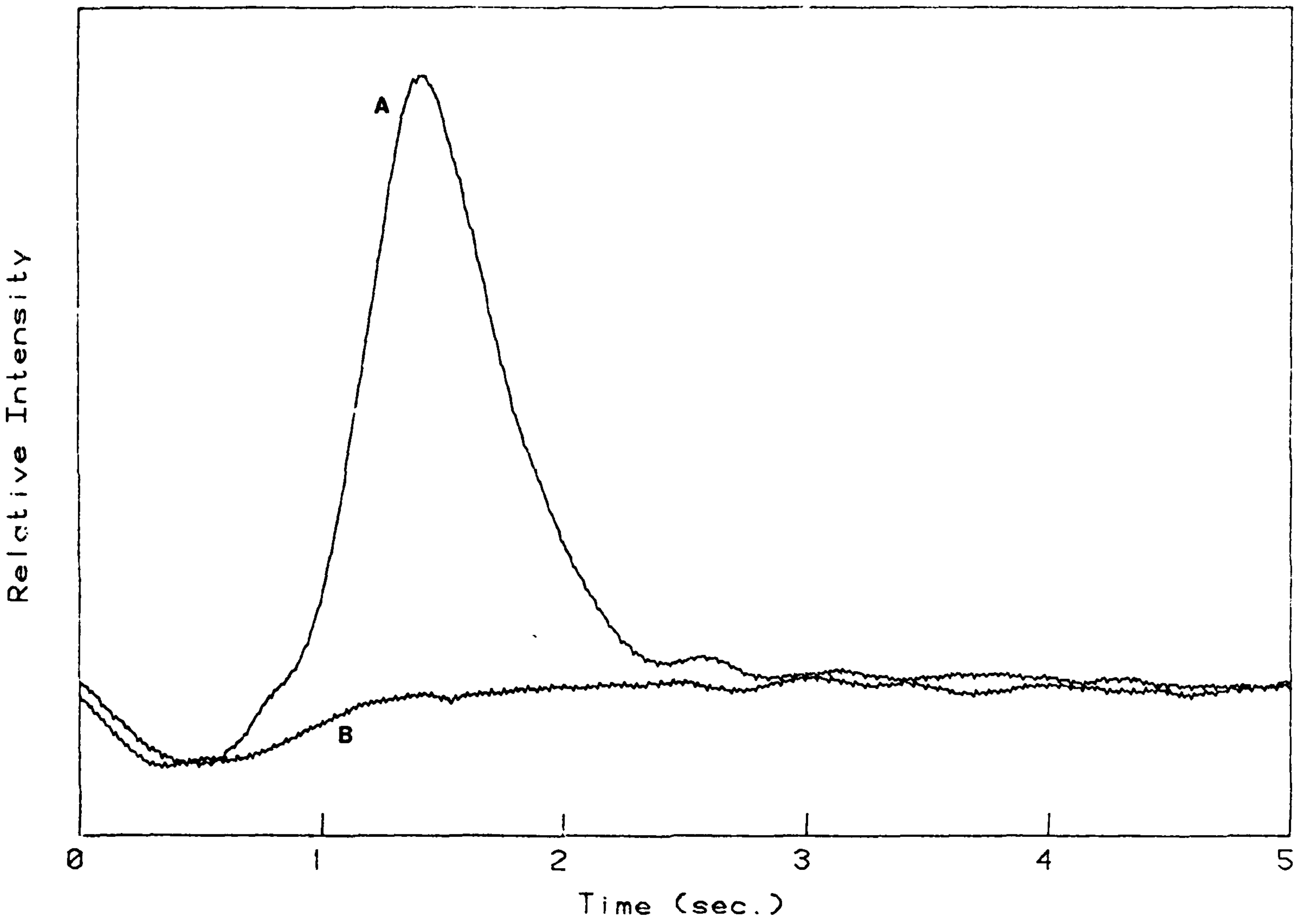
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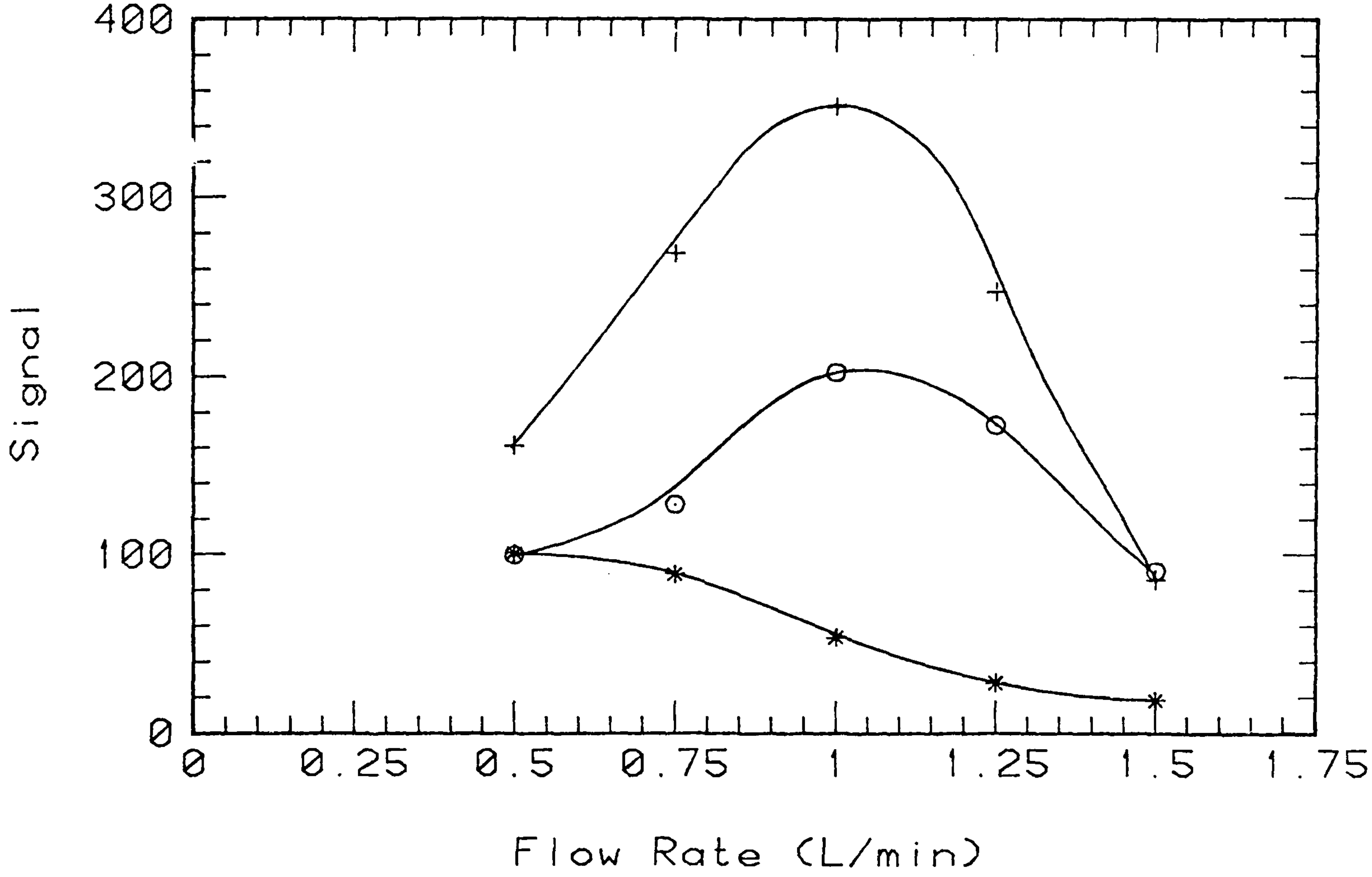
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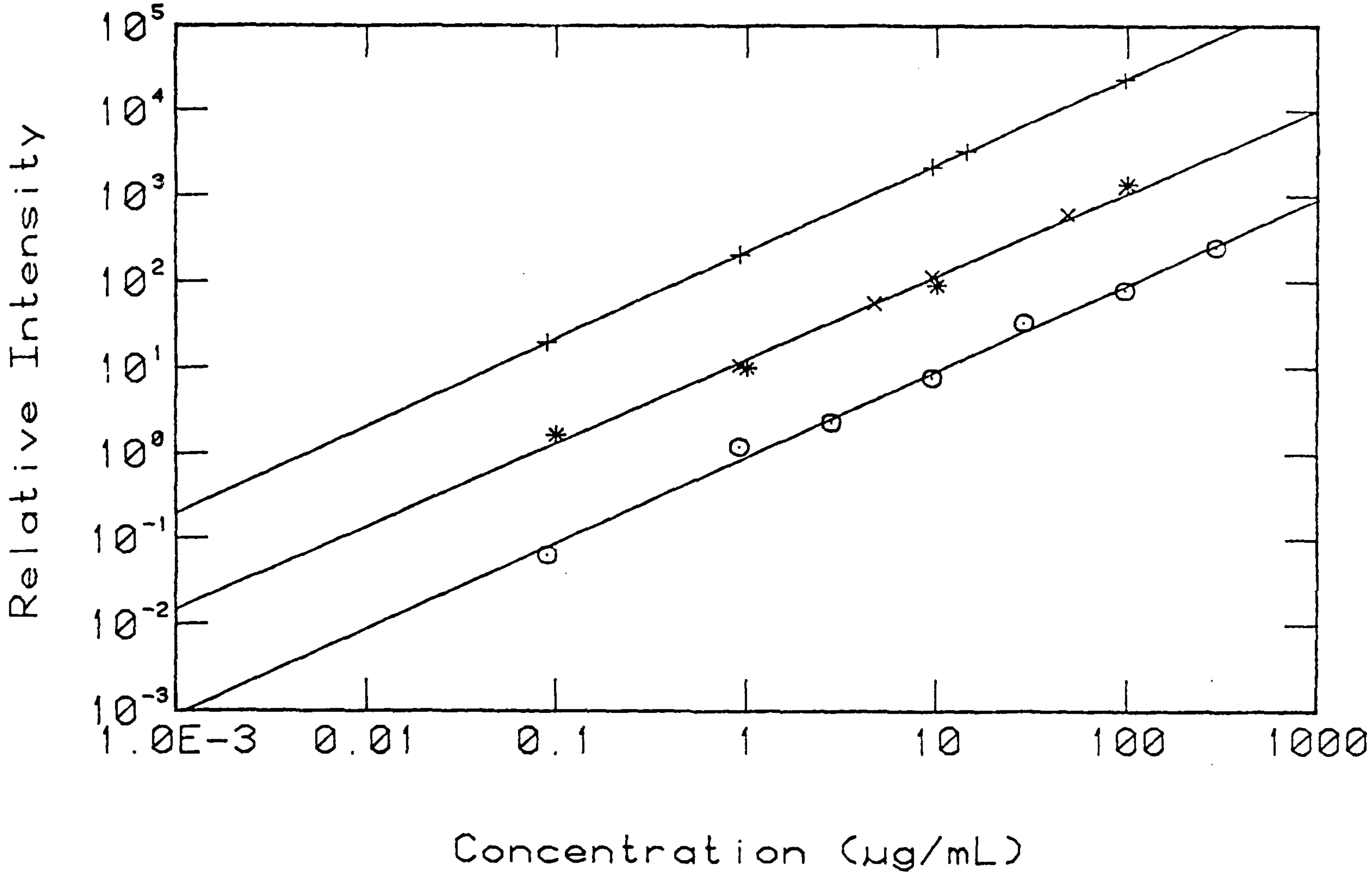
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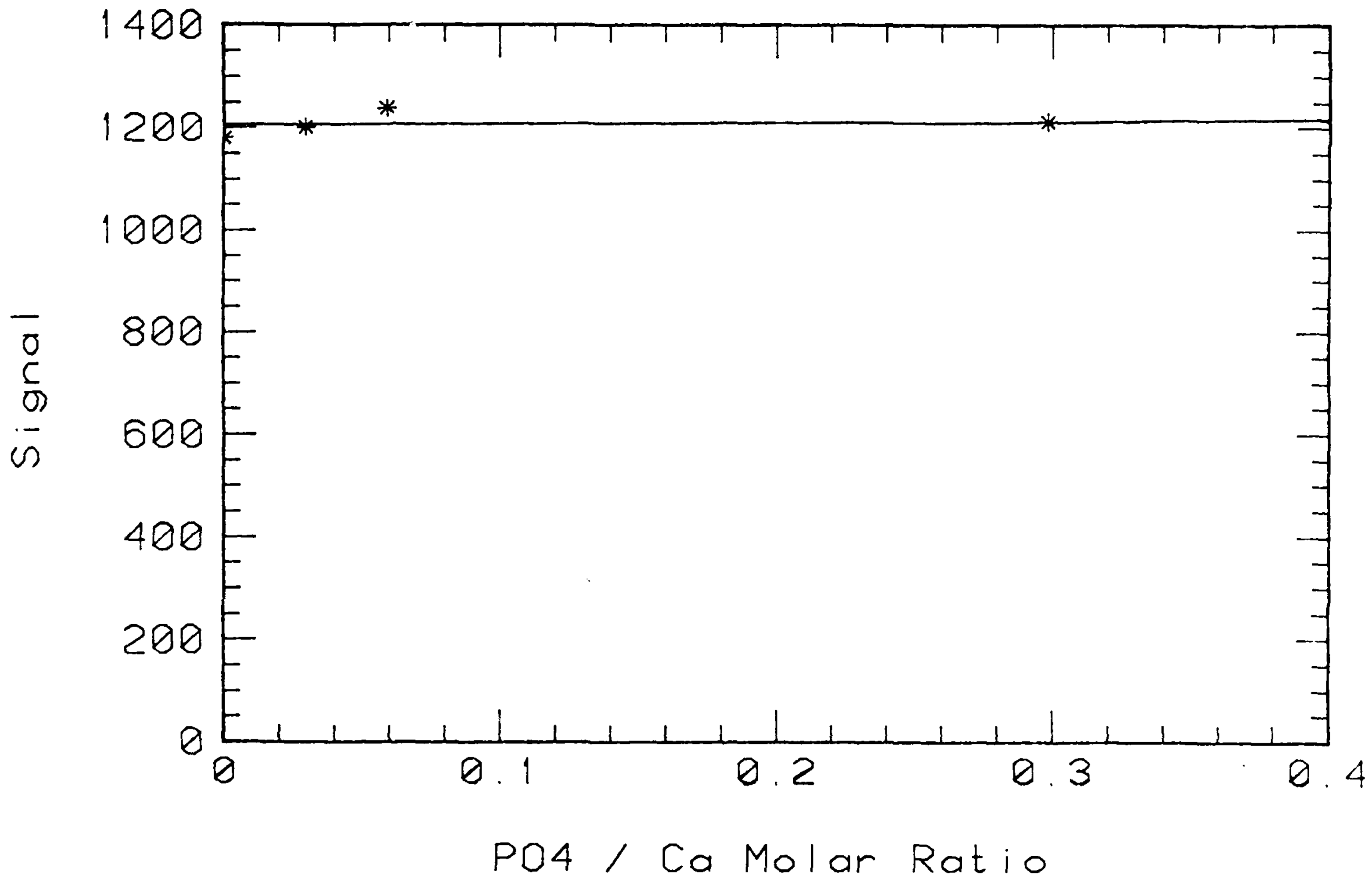
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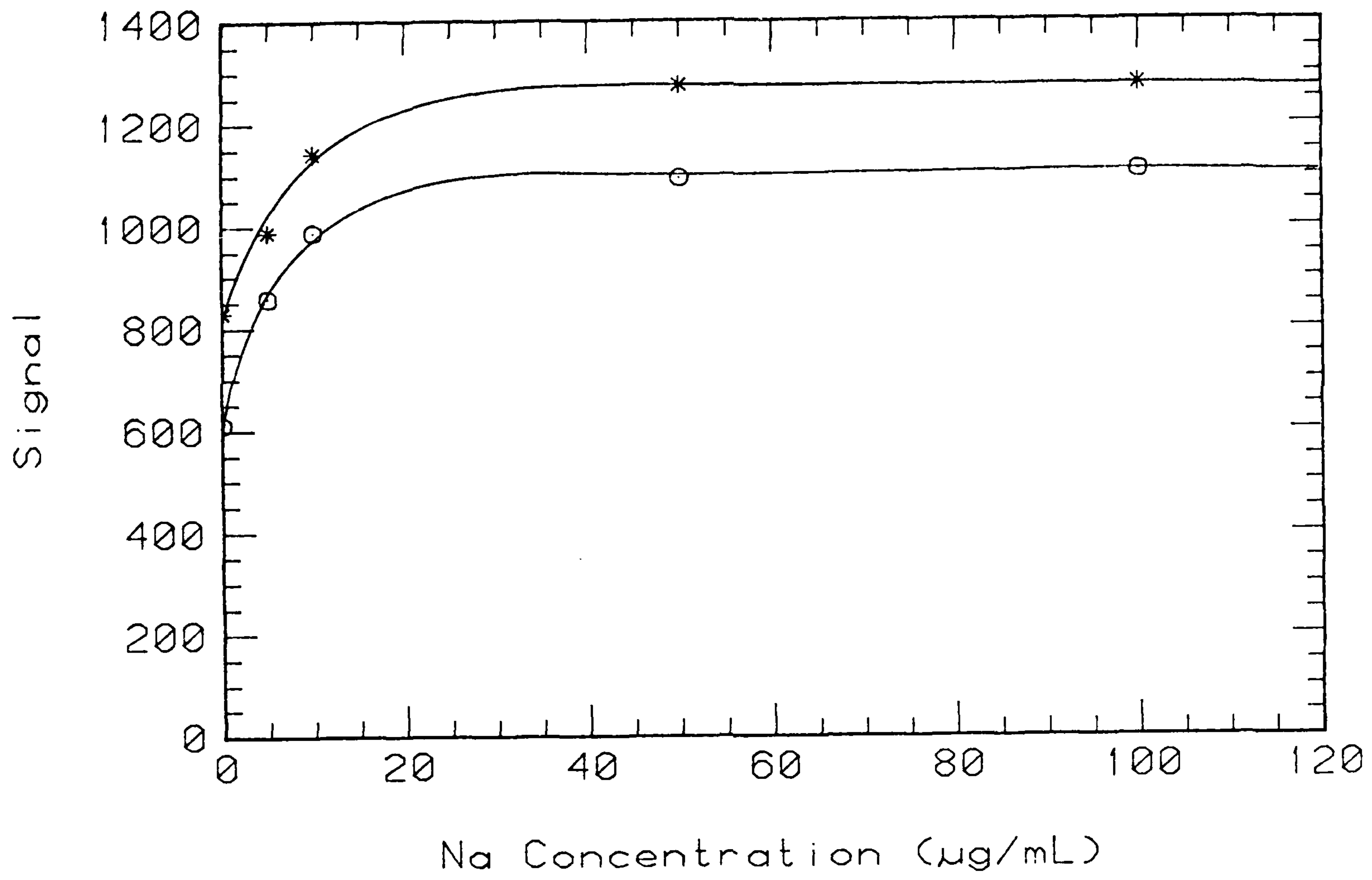
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