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TECHNICAL REPORT NO. 33

A NEW METHOD FOR TRACE METAL DETERMINATIONS BY MONITORING
MODULATED MAGNETICALLY INDUCED OPTICAL ROTATION IN ATOMIC VAPOURS

by

Curtis A. Monnig and Gary M. Hieftje

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The rotation of plane-polarised radiation by atoms within a magnetic field can be used for sensitive and selective trace-metal detection (1-30). In this technique, plane-polarised resonance radiation is directed through an atom cloud confined within a magnetic field. The magnetically induced birefringence and dichroism in the atomic vapour rotates the plane of polarisation of the transmitted beam with respect to the incident beam. The magnitude of this rotation depends upon the number of analyte atoms in the path of the beam, the strength of the magnetic field, and factors which vary with the element under investigation. With a constant magnetic field, trace-level determinations of many elements can be performed by monitoring the polarisation rotation induced by the analyte vapour and comparing it to the optical rotation produced by the vapour from a set of standard solutions.

In practice, the induced polarisation rotation is almost never measured directly. Instead, the atom reservoir is usually placed between two crossed polarisers and the transmission of resonance radiation through the polariser pair is monitored. When no analyte vapour is present, radiation which is transmitted by the first polariser is blocked by the second. However, when a sample is atomised, the plane of polarisation of the transmitted radiation is partially rotated by the analyte vapour and a small fraction can pass through the second polariser (the analyser). Unfortunately, this optical arrangement has several drawbacks. At low concentrations, the intensity transmitted by the analyser is proportional to the square of the analyte atomic vapour concentration in the atomiser. In turn, this quadratic response causes the signal to drop very rapidly with concentration, so detection limits suffer. It has been shown that the signal can be linearised by offsetting one of the polarisers a small angle from the crossed configuration (28). Although this approach improves the signal-to-noise characteristics of the instrument (24), the selection of the optimum offset angle is dependent upon a number of complex instrument parameters (31).

Another liability of this conventional measurement approach is its susceptibility to background scattering and absorption. This problem arises just as in atomic absorption spectrometry and is most troublesome when an electrothermal atomiser is employed. Several methods for background-correcting an AMORS signal have been proposed (11,32). On the whole, these schemes are conceptually simple and can provide satisfactory correction under many circumstances. However, they must satisfy the same instrumental and temporal-sampling criteria as are required for atomic absorption spectroscopy (33,34) and are often costly and complex.

One way to overcome many of the problems listed above is to measure directly the rotation of the plane of polarisation of the transmitted radiation. Instrumentally, this approach can be implemented by periodically sweeping (modulating) the polarisation of the incident beam and employing phase-sensitive detection (35). In such an instrument, an input optical element periodically rotates the plane of polarisation of incident radiation and an output polariser blocks radiation not polarised at a selected angle. As a result, signals similar to those in Figure 1 are observed. The upper curve in

Figure 1 shows the signal observed when no analyte is present in the optical path. When the analyte vapour is introduced, an additional polarisation rotation is induced by the atomic species and produces a small phase shift (ϕ) in the signal. Additionally, a reduction in the intensity of the transmitted radiation occurs from atomic and molecular absorption as well as from signal loss caused by scattering. Importantly, a direct phase measurement provides a signal which is linear with analyte concentration and relatively independent of interferences which affect the intensity striking the detector (lamp drift, nonspecific absorption, scattering, etc.). Unfortunately, an earlier test of polarisation modulation in AMORS suffered from a rather low modulation frequency, limited largely by the use of a mechanically driven modulating element (35). As a result, signal detection options were limited, and rapid, transient sample vapour pulses from a electrothermal atomiser could not be faithfully followed.

In the present paper, a new instrument is described which employs an electro-mechanically driven optical element (a photoelastic modulator) to rotate the plane of the polarised radiation at a high fixed frequency (42 kHz). When this modulator is placed with the atom reservoir between a pair of crossed polarisers and radiation is passed through the optical system, rotation of the radiation caused by analyte atoms appears as a phase shift in the transmitted signal. As we shall demonstrate, monitoring changes in the signal phase provides linear calibration curves with excellent detection limits, and freedom from the deleterious effects of background absorption, light scattering, and source-intensity drift.

EXPERIMENTAL

Instrumentation

Figure 2 shows a schematic diagram of the instrument used in this study. Lens L_1 focuses the hollow-cathode lamp radiation into the center of the graphite-furnace assembly. Two polarisers (P_1 and P_2) select specific polarisation orientations of the beam. Above 300 nm, polymeric-film linear polarisers (Oriel Inc., Model 27340) were used, whereas below 300 nm, calcite prism polarisers (Oriel Inc., Model 2540-1) were employed because of their superior performance in the deep ultraviolet. Polarisation modulation was accomplished by a photoelastic modulator (PEM) containing a fused-silica optical element and operated at a 42.04 kHz modulation frequency (Hinds International, Model PEM-80 Series II FSA). The PEM is was selected as the polarisation modulation optical element in this instrument because of its large optical aperture (> 4 cm), high optical activity (90° rotation possible), broad spectral range (180 to 2000 nm), convenient modulation frequency, and commercial availability. Lens L_2 images the furnace onto a circular aperture at the entrance slit of the monochromator; the aperture serves to block furnace emission while transmitting the hollow-cathode lamp radiation.

The atom reservoir used for these experiments is a carbon rod atomiser similar in design to a

Varian CRA-90 graphite furnace. Although CRA-90 minitube furnaces were used directly, the furnace-head dimensions differ slightly from the commercially available workhead to allow the poles of the magnet access to the furnace. An argon sheath gas (4.0 L/min) was used to prevent oxidation of the furnace during atomisation. A Varian CRA-90 power supply provided power for the carbon furnace.

A uniform longitudinal magnetic field must be produced within the analytical volume of the atomiser for reproducible AMORS signals to be obtained. This field was generated by a permanent magnet designed and constructed in our laboratory. The assembly consisted of two samarium-cobalt permanent magnets (Magnetic Sales and Manufacturing, Inc., Model 18 ring magnets) secured to a low-carbon steel yoke. The magnetic field experienced by the atomic vapour could be altered by varying the pole-gap distance of the magnet and ranged typically from 1.2 to 5.2 kG over a pole gap distance of 25 to 10 mm, respectively.

The monochromator (GCA/McPherson EU 700-56) employed 250 μm entrance and exit slits (0.5 nm bandpass); the photomultiplier tube (Hamamatsu R928) was operated at a bias voltage of -800 V. The current from the photomultiplier tube was converted to a proportional voltage by a high-speed current-to-voltage amplifier (10^5 V/1 A) constructed locally. After being passed through a bandpass filter adjusted for maximum transmission at 84.08 kHz (Krohn-Hite, Model 3342), the modulated signal was sent to a phase meter (Wavetek, Model 750) which was referenced to the 2F output (84.08 kHz) from the PEM driver. The voltage output from this phase meter was recorded by a digital storage oscilloscope (Nicolet Inc., Model 3091) which subsequently transferred the data to an IBM-XT laboratory computer for data processing and display.

Analytical Reagents

All chemicals used were of analytical-reagent grade. Stock solutions of lithium and potassium were prepared by dissolving the respective chloride salts in deionized water. Magnesium solutions were prepared by dissolving the pure metal in the minimum amount of hydrochloric acid. Test solutions were prepared by appropriate dilution of the stock solutions and were acidified with sub-boiled nitric acid just prior to their use. High-precision micropipets (Rainin, Inc.) were used for preparation of these solutions and for dispensing the sample into the graphite furnace.

Procedures

The hollow cathode lamp was allowed to warm up for 30 minutes, during which time the polarisers were adjusted for maximum extinction (crossed configuration). The first polariser was oriented so its axis was 45° to that of the PEM crystal. The dispensed sample volume was 10 μL . Atomiser dry, ash, and atomisation temperatures were those recommended by Varian (36). The magnetic field used for the determination of each element was selected from the value reported by Kitagawa *et al.* (17) which gave the maximum signal and which was within the scanning range of our magnet. Three replicate

determinations of each sample were performed to overcome variations introduced in pipetting the sample.

RESULTS AND DISCUSSION

A lithium calibration curve obtained with the new instrument is shown in Figure 3. The smallest mass of lithium which could be reproducibly detected with this instrument ($S/N = 3$) under standard analysis conditions was 1 pg. For comparison, the characteristic mass of lithium in a Varian CRA-90 graphite furnace atomic absorption instrument is 2 pg (36). The useful analytical region of the calibration curve extends currently from 1 pg up to a mass of approximately 80 pg of lithium. Above this level, the limited slew rate of the current phase meter prevents accurate measurement of the phase shift.

Table 1 shows detection limits for several elements obtained with this instrument. In general, these detection limits are comparable to those reported for graphite-furnace atomic absorption analysis with a Varian CRA-90 atomiser. The slightly higher than expected detection limit for magnesium is the result of increased baseline noise, caused by the decreased optical throughput of the calcite prism polarisers.

The influence of changes in the amplitude of the modulated signal on the output of the phase meter is shown in Figure 4. Because the instrument measures the phase of the signal and not its amplitude, changes in amplitude ideally should not affect the analytical measurement. In practice, most phase meters exhibit a limited input signal range over which the output remains independent of signal amplitude. The upper trace in Figure 4 shows the temporal response of the instrument when 25 pg of lithium was atomised. In the lower trace, 25 pg of lithium is again atomised under identical conditions except that a neutral density filter (50% transmission) has been inserted into the optical path. Within the reproducibility of the measurement, the integrated areas under these curves are identical. This finding implies that attenuation of transmitted radiation by molecular absorption, scattering, or light-source drift will have little effect on the accuracy of the measurement. Of course, the precision of the measurement would be expected to degrade under these conditions since the signal-to-noise ratio of the modulated signal is decreased.

Several factors have been shown dramatically to affect the sensitivity of this new technique. Chief among these factors is the spectral profile of the radiation source. In a previous communication, Kitagawa *et al.* (37) report that spectral broadening of the source used in their AMORS instrument resulted in significantly higher levels of observed polarisation rotation. Similar results were obtained here. Figure 5 shows the effect of hollow-cathode lamp current on the observed phase-angle change during the atomisation of 25 pg of Li. As the lamp is overdriven (maximum recommended operating current is 15 mA), the spectral line broadens so it overlaps more strongly with the Zeeman-split

absorption profile that produce the optical rotation. This observation suggests that detection limits might be improved by using a radiation source which is spectrally broader and more intense than a typical hollow cathode lamp. However, recent studies with pulsed hollow-cathode discharges suggest that extensive broadening of the lamp spectral profile might eventually decrease the optical rotation that is observed (35). Of course, the magnetic field would be expected to play a complex role in determining the optimum spectral width of the lamp.

Figure 6 illustrates the effect of the PEM retardation amplitude on the observed change in the signal phase. In curve C, the half-wave retardation wavelength is set to the wavelength being monitored (670.78 nm). When 10 pg of lithium is atomised, only a small deflection is observed. In curve B, the half-wave retardation wavelength is reduced to 445 nm. For a given angular rotation induced by the atomic vapour, it will appear as a larger phase shift when the optical retardation is varied over a smaller range (a lower half-wave retardation wavelength). This trend continues when in curve A the half-wave retardation wavelength is further reduced to 220 nm. Clearly, this trend should continue until the rotation induced by the PEM equals the rotation produced by the atomic vapour (180° phase shift). However, as the optical retardation modulation range is reduced, the amplitude of the signal at the detector is decreased; in turn, it becomes more difficult to accurately monitor the phase of the signal. Under what conditions each of these effects dominates the signal-to-noise ratio of the measurement is an area currently being explored in our laboratory.

CONCLUSIONS

Although the data presented in this communication represent only our initial efforts to explore this new method of detection for AMORS signals, it seems clear that the instrument possesses several significant attributes for trace metal analysis. First, the instrumentation is compact, can be simple to operate, and generates a signal that varies linearly with analyte concentration. Furthermore, it offers improved freedom from the effects of molecular absorption, scattering, and source-intensity drift.

Practically, several questions still need to be answered before the analytical utility of this method of signal detection can be fully ascertained. Of fundamental importance is the effect of source and transmission noise on the accuracy of the phase measurements. To a large extent, the importance of these noise sources will depend upon the manner in which phase detection is carried out. The analytical characteristics of the current instrument are limited by the temporal response and noise characteristics of the detection electronics. Consequently, further optimisation of this circuit must be performed to realise the full analytical potential of the technique. These development efforts are currently under way in our laboratory.

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TABLE 1.

Detection Limits for Several Elements Obtained with the New
Polarisation-Modulated AMORS Instrument

Element	Wavelength (nm)	Magnetic Field (kG)	Detection Limit at S/N = 3 (pg)	GFAAS Characteristic Mass (pg)*
Li	670.7	3.0	1.0	2.0
K	589.0	2.2	0.9	1.0
Mg	285.2	3.9	5	0.06

* From Reference 36

FIGURE CAPTIONS

- Figure 1. Illustration of the temporal response of the photomultiplier signal when the polarisation is sinusoidally modulated. The upper curve shows the expected signal under normal operating conditions. The lower trace is the expected signal when analyte vapour is interacting with the light beam. The letter A designates loss of signal amplitude by atomic absorption, background scattering and absorption, and light-source drift. The Greek letter ϕ represents the phase shift induced by the polarisation rotation.
- Figure 2. Schematic diagram of the AMORS instrument. Abbreviations represent: H.V. Power Supply, high voltage power supply; P_1 and P_2 , polarisers; L_1 and L_2 , plano-convex lenses (f.l. 15 cm); PEM, photoelastic modulator; B, magnetic field; PMT, photomultiplier tube; I-V Amplifier, current-to-voltage amplifier
- Figure 3. Calibration curve for lithium. Signal monitored at 670.7 nm.
- Figure 4. Temporal response of the instrument during the atomisation of 25 pg of Li. Curve A - atomisation under normal analysis conditions. Curve B. - atomisation with neutral-density filter (O.D. 0.3) in optical path.
- Figure 5. Temporal response of the instrument for the atomisation of 25 pg of lithium as a function of hollow-cathode lamp current (i). Recommended maximum lamp current is 15 mA.
- Figure 6. Temporal response of the instrument for the atomisation of 25 pg of lithium as a function of the photoelastic modulator half-wave retardation wavelength. Curve A. half-wave retardation 220 nm, Curve B. half-wave retardation 445 nm, Curve C. half-wave retardation 670 nm.

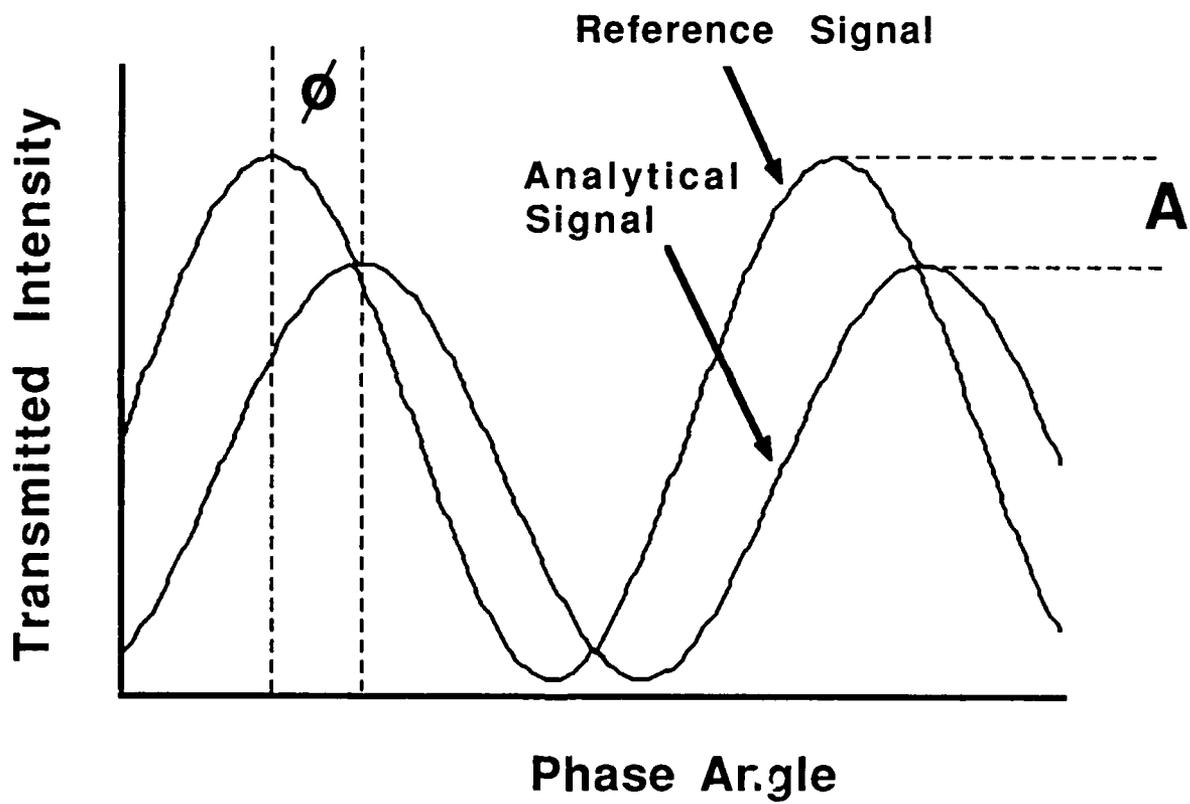


Fig. 1

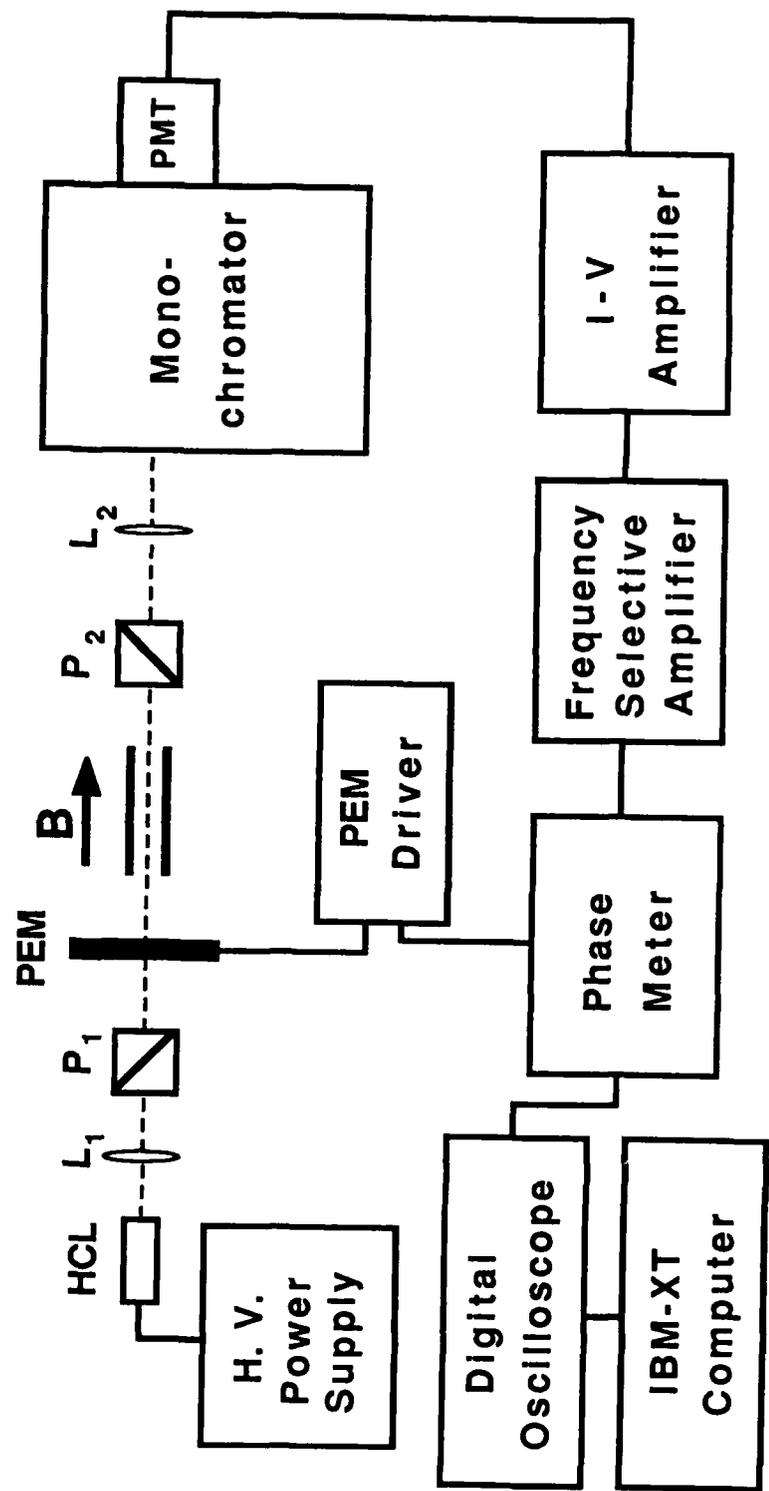


Fig. 2

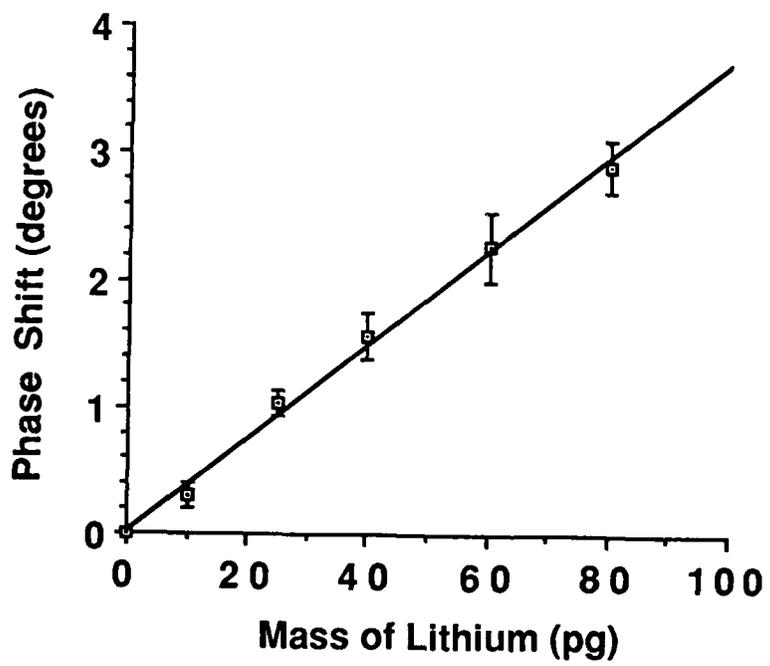


Fig. 3



Fig 4

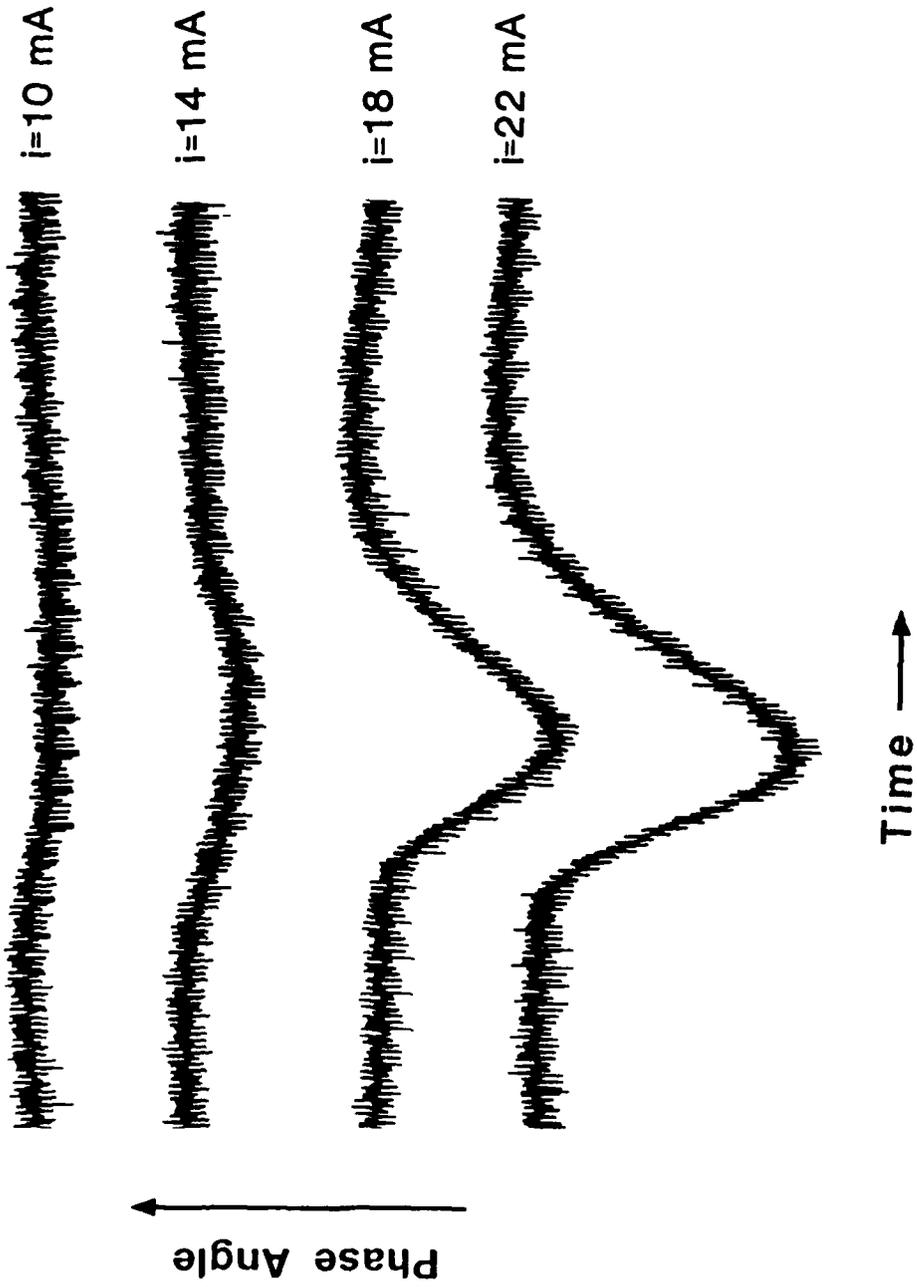


Fig. 5

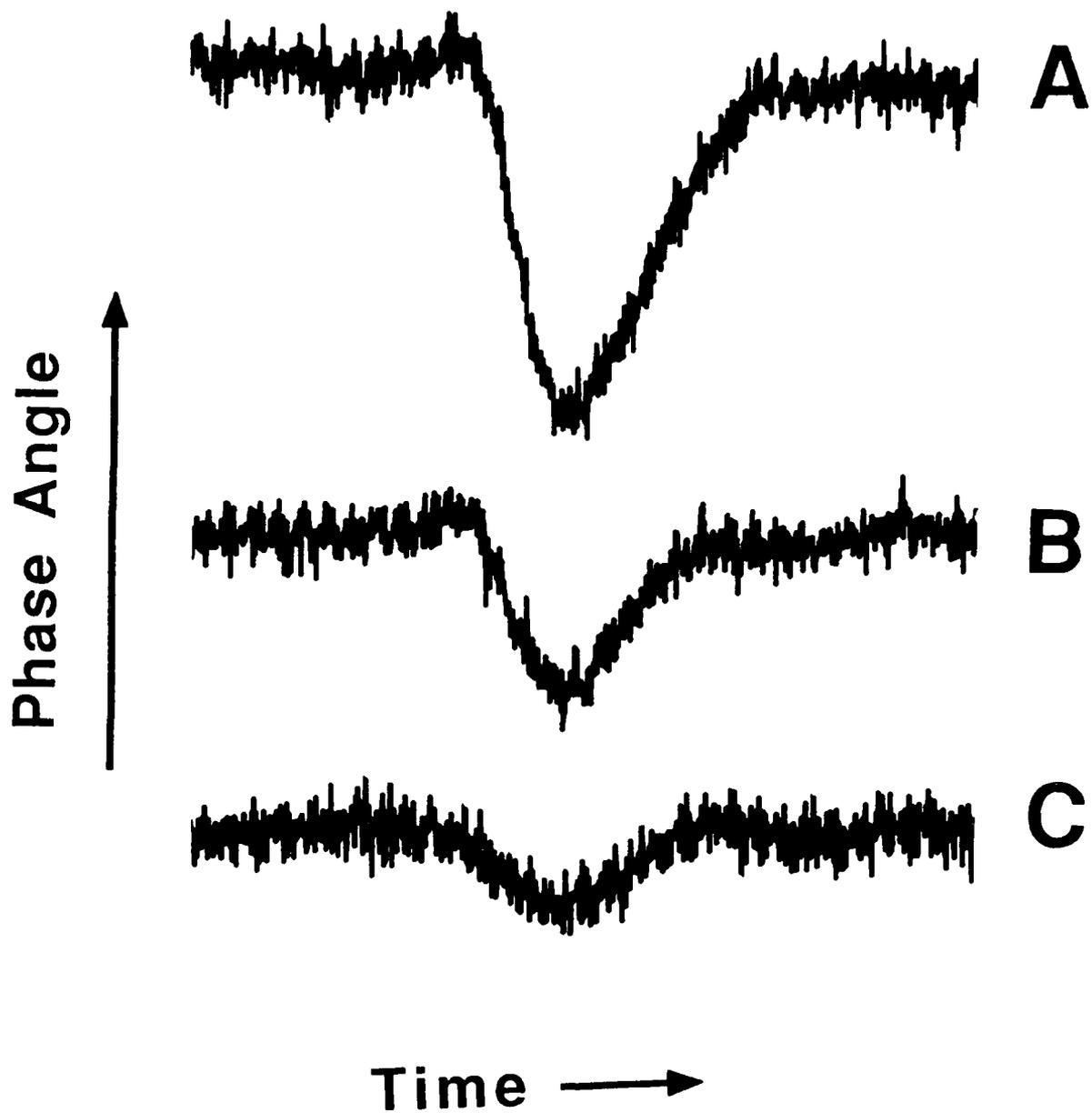


Fig. 6

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