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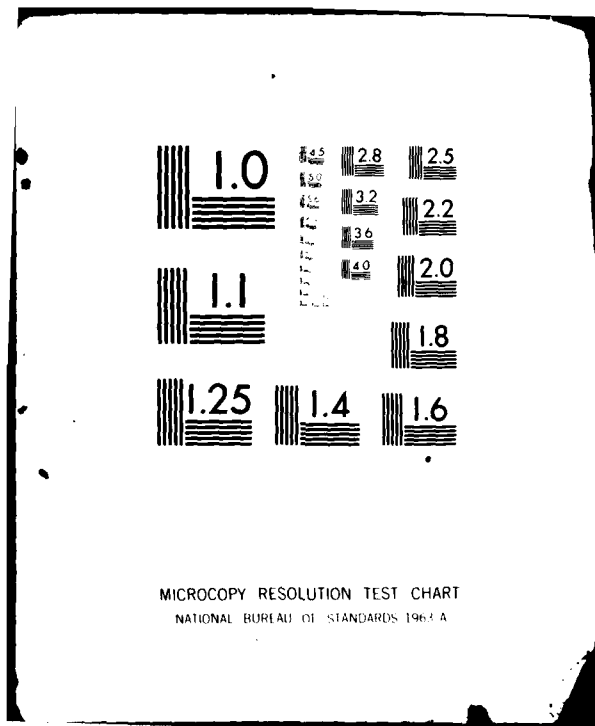
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By

G. Nagasubramanian, Bob Lee Wheeler, F.-R. Fan and

A. J. Bard

Prepared for Publication

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Department of Chemistry  
Austin, Texas 78712

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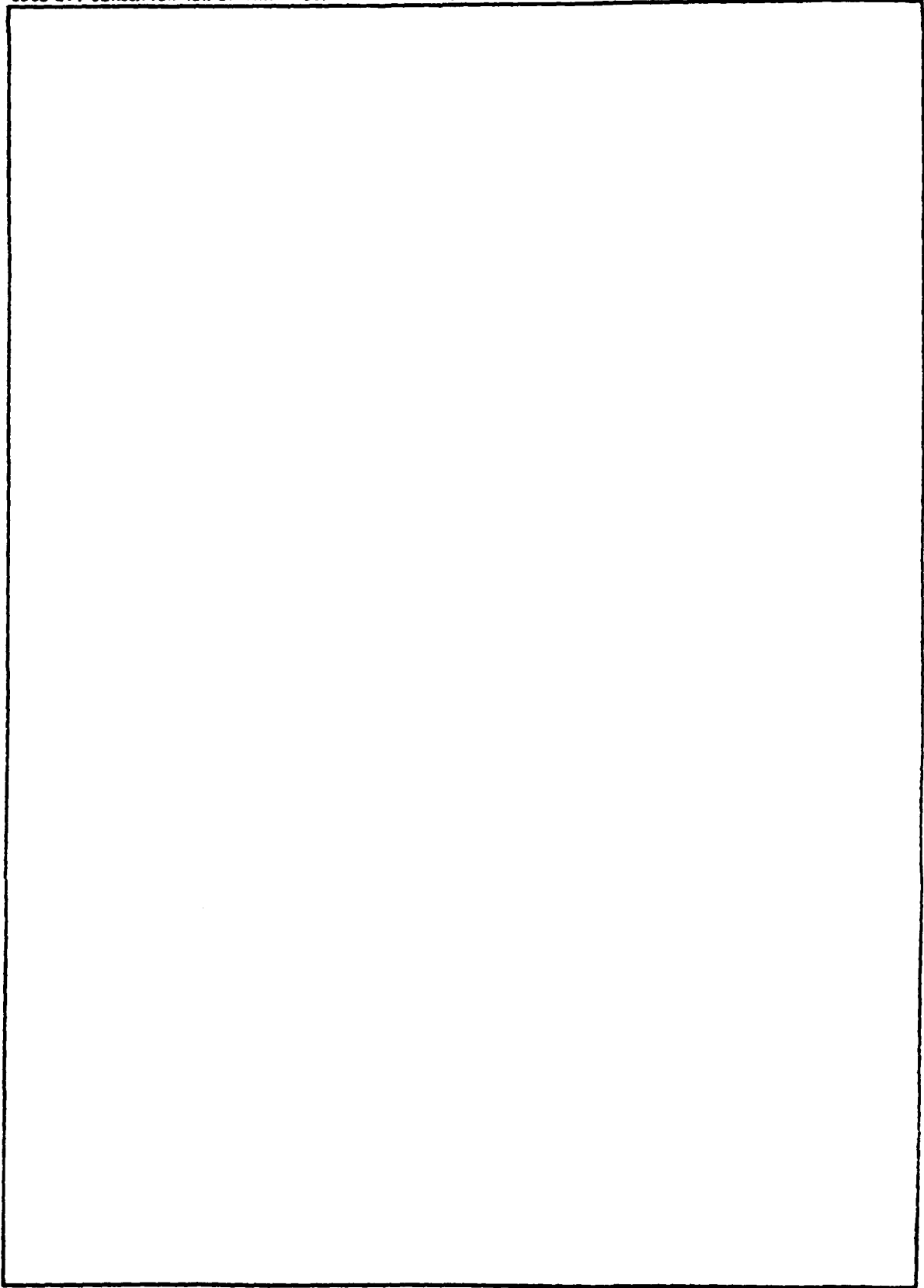
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FERMI LEVEL PINNING AT SEMICONDUCTOR ELECTRODES.

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ABSTRACT

The shift in  $V_{fb}$  of several single crystal semiconductors (p-Si, p-GaAs, n-TiO<sub>2</sub>, n-CdS, n-MoSe<sub>2</sub>, n- and p-WSe<sub>2</sub>) was investigated in MeCN containing a number of redox couples. While  $V_{fb}$  shifts with  $V_{redox}$  for p-Si, p-GaAs, n-TiO<sub>2</sub> and n-CdS for the layered compounds (n-MoSe<sub>2</sub>, n- and p-WSe<sub>2</sub>) was essentially independent of  $V_{redox}$ .

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### Introduction.

For the simplest model for semiconductor photoelectrochemical (PEC) cells, the flatband potential of the semiconductor,  $V_{fb}$ , remains fixed while the metal or carbon counter electrode attains the potential of the redox couple in the solution,  $V_{redox}$ . Under these conditions, the open circuit photovoltage,  $V_{oc}$ , under moderate irradiation intensities, is  $|V_{redox} - V_{fb}|$  and  $V_{oc}$  is predicted to vary linearly with a unity slope with  $V_{redox}$ . A corollary of this model is that photoreduction at a p-type semiconductor of species with  $V_{redox}$  corresponding to an energy level above the conduction band edge (or photooxidation at an n-type material for a couple below the valence band edge) would not occur. However, such "ideal" behavior is rarely found. For example, studies of PEC cells with low bandgap ( $E_g$ ) materials such as Si, GaAs, and CdTe with aqueous and nonaqueous solutions containing a number of redox couples whose  $V_{redox}$  values span a potential regime wider than the  $E_g$  of the semiconductor exhibit an essentially constant output photovoltage (1-3). Moreover, photoreductions at potentials well above the conduction band edge have been observed. A striking case is the photoinjection of electrons from p-GaAs or p-Si in liquid ammonia, where the energy level for such a process is well above the dark  $V_{fb}$  value in the original solution (4).

To account for this departure from ideal behavior, the concept of Fermi level pinning (FLP) was proposed, in which the extent of band bending in the semiconductor space charge region remains constant with different redox couples (1-3,5). This model of the semiconductor/liquid junction follows closely that originally proposed for semiconductor/metal junctions (Schottky barriers), where the photovoltage is often found to be independent of the work function of the metal (6). This FLP has been ascribed to the presence

of a high density of surface or interface states. As described in a number of theoretical papers on these junctions (5,7) the Fermi level is pinned at the energy levels of these states, and changes in potential across the semiconductor/solution interface will cause mainly a change in the potential drop across the Helmholtz layer. This change in potential drop across the Helmholtz layer implies that the band edges are not fixed, but vary with  $V_{\text{redox}}$ ; i.e.,  $V_{\text{fb}}$  tracks  $V_{\text{redox}}$ . Such a shift in  $V_{\text{fb}}$  with  $V_{\text{redox}}$  provides evidence for FLP and is the main subject of this work.

#### Experimental.

The single crystals of n-TiO<sub>2</sub>, n-CdS, n- and p-WSe<sub>2</sub>, n-MoSe<sub>2</sub>, p-Si and p-GaAs were polished and mounted as electrodes as described elsewhere (8-14). The following etchants were used for the different electrodes:

n-TiO<sub>2</sub>. Conc. HNO<sub>3</sub> for 1 min followed by HF for 10 sec then rinsed with double distilled water. This procedure was repeated twice.

n-CdS. Conc. HCl for 40 sec then rinsed with double distilled water.

n- or p- type WSe<sub>2</sub>; n-MoSe<sub>2</sub>. 6 M HCl for 30 sec then rinsed with distilled water. This procedure was repeated twice.

p-Si. HNO<sub>3</sub>:CH<sub>3</sub>COOH:HF (3:1:3) containing a drop of Br<sub>2</sub> for 30 sec followed by pure HF etching for 5 sec, then rinsed with double distilled water.

p-GaAs. H<sub>2</sub>SO<sub>4</sub>:H<sub>2</sub>O<sub>2</sub>(30%):H<sub>2</sub>O (3:1:1 by volume) solution for 50 sec.

The instruments employed and the purification of the solvent and the redox couples are described elsewhere (8-14). An aqueous saturated calomel electrode (SCE) introduced directly into the PEC cell and separated from it by a medium porosity glass frit was used as a reference electrode. All the potentials are expressed against this aqueous SCE unless otherwise specified. The capacitance was measured using a lock-in amplifier technique with digital data acquisition employing a Digital Equipment Corp. MINC-11



computer. The C-V data was processed in the computer and plots of  $1/C^2$  vs.  $V$  plotted on a Houston Instruments Model DMP5 Digital Plotter. A block diagram of the apparatus is shown in Fig. 1.

#### Results and Discussion.

The determination of  $V_{fb}$  from capacitance (C)-potential (V) data via Schottky-Mott (S-M) plots of  $1/C^2$  vs.  $V$  is well documented in the literature (16). Since the S-M plots pertain to the space charge capacitance,  $C_{sc}$ , identification of C with  $C_{sc}$  requires that surface states do not contribute to the capacitance of the measured value of C. This will be the case when the measurement frequency,  $f$ , is sufficiently high with respect to the time constant associated with the charge exchange between surface states and the bulk of the semiconductor. Under these conditions  $C = C_{sc}$  and the surface states do not contribute even when they are effective in pinning the Fermi level and in equilibration with the d-c solution redox potentials. The working frequency range was selected from a plot of C vs.  $f$  at a given potential. A typical plot for p-Si is shown in Fig. 2. For example with p-Si for frequencies above 4 kHz the capacitance becomes essentially independent of  $f$  and we assume  $C = C_{sc}$  under these conditions. The  $V_{fb}$  measured in such frequency regimes for various semiconductors in the presence of various redox couples are given in Table 1. For n-TiO<sub>2</sub>, some effect of frequency on C was found for frequencies of 0.1, 0.2 and 0.5 kHz (Fig. 3), but the S-M plots extrapolated to essentially the same value for  $V_{fb}$ . Such behavior has been noted and discussed by Comes, et al. (15).

In general,  $V_{fb}$  values could be determined from S-M plots with a precision of at least  $\pm 0.1$  V (15,16). The effect of changes in  $V_{redox}$  was studied by addition of both the reduced and oxidized form of the redox couple to the solution. Typical plots of  $V_{fb}$  vs.  $V_{redox}$  are shown in Figs. 4-8. In all cases the dark current was below  $5 \mu A/cm^2$  over the potential

range of the S-M plots in the presence of redox couples.

Except for the layered compounds (as shown in Fig. 8),  $V_{fb}$  shifts with  $V_{redox}$ , suggesting the importance of FLP. With n-TiO<sub>2</sub> (Fig. 4) a strong effect of  $V_{redox}$  is noted, even for couples with energy levels considerably above half the bandgap. However, in most cases the slope of the  $V_{fb}$  vs.  $V_{redox}$  curve is less than one. This has been discussed recently for semiconductor/liquid junctions (17) and has been ascribed to partial FLP with a change in potential applied across the interface partly dropping across the space charge region and partly across the Helmholtz layer.

The effect of surface states and the onset of FLP for a given semiconductor may depend upon the nature of the surface pretreatment (3b) and the nature of the solvent and electrolyte. For example, Gomes, et al. (15), briefly noted that no effect of redox couple was found on the  $V_{fb}$  derived from S-M plots for molten NaOH etched TiO<sub>2</sub> crystals in aqueous solutions. The effect of etching and chemical pretreatment of the surface as well as solvent on the S-M plots and extent of FLP is currently under investigation.

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TABLE I.  $V_{(fb)}$  OF VARIOUS SEMICONDUCTORS IN ACETONITRILE CONTAINING DIFFERENT REDOX COUPLES<sup>a</sup>

electrolyte	Blank	TMPD <sup>0/+</sup>	Fe(CP) <sub>2</sub> <sup>0/+</sup>	10-MP <sup>0/+</sup>	κ <sub>ub</sub> <sup>0/+</sup>	Per <sup>0/+</sup>	Th <sup>0/+</sup>	CHR <sup>0/+</sup>	OX <sup>0/-</sup>	MV <sup>2+/1+</sup>	BQ <sup>0/-</sup>	ΔQ <sup>0/-</sup>	Ru(bpy) <sub>3</sub> <sup>2+/+</sup>	PhNO <sub>2</sub> <sup>0/-</sup>	Ph(CN) <sub>2</sub> <sup>0/-</sup>
redox		0.27	0.53	0.79	0.94	1.14	1.26	1.56	-0.2	-0.43	-0.56	-0.96	-1.27	-1.33	-1.72
Electrodes <sup>b</sup>															
n-TiO <sub>2</sub>	-0.0	-1.0	-0.82	-0.7	-0.42	-0.18		-0.13							
n-Se <sub>2</sub>	-0.3	-0.4		-0.4			-0.4								
n-MoSe <sub>2</sub>	0.1	-0.12		0.0			-0.1	-0.11							
n-CdS	-0.9	-0.8	-0.7	-0.4			-0.2								
n-Se <sub>2</sub>	0.78									0.75	0.62		0.8	0.75	
n-GaAs	0.06										-0.06	-0.14		-0.33	-0.46
n-Si	0.15								-0.02		-0.12	-0.44	-0.52	-0.53	

<sup>a</sup>Abbreviations: AQ = anthraquinone; BQ = benzoquinone; CHR = chrysenes; Fe(CP)<sub>2</sub> = ferrocene; 10-MP = 10-methyl phenothiazine; MV = methyl viologen 4,4'-dimethyl bipyridinium; OX = oxazine; PhNO<sub>2</sub> = nitrobenzene; Per = perylene; Th(CN)<sub>2</sub> = phthalonitrile; Rub = rubrene; bpy = 2,2'-bipyridine; TMPD = N,N,N',N'-tetramethyl-p-phenylenediamine; Th = thianthrene

<sup>b</sup>Doping level

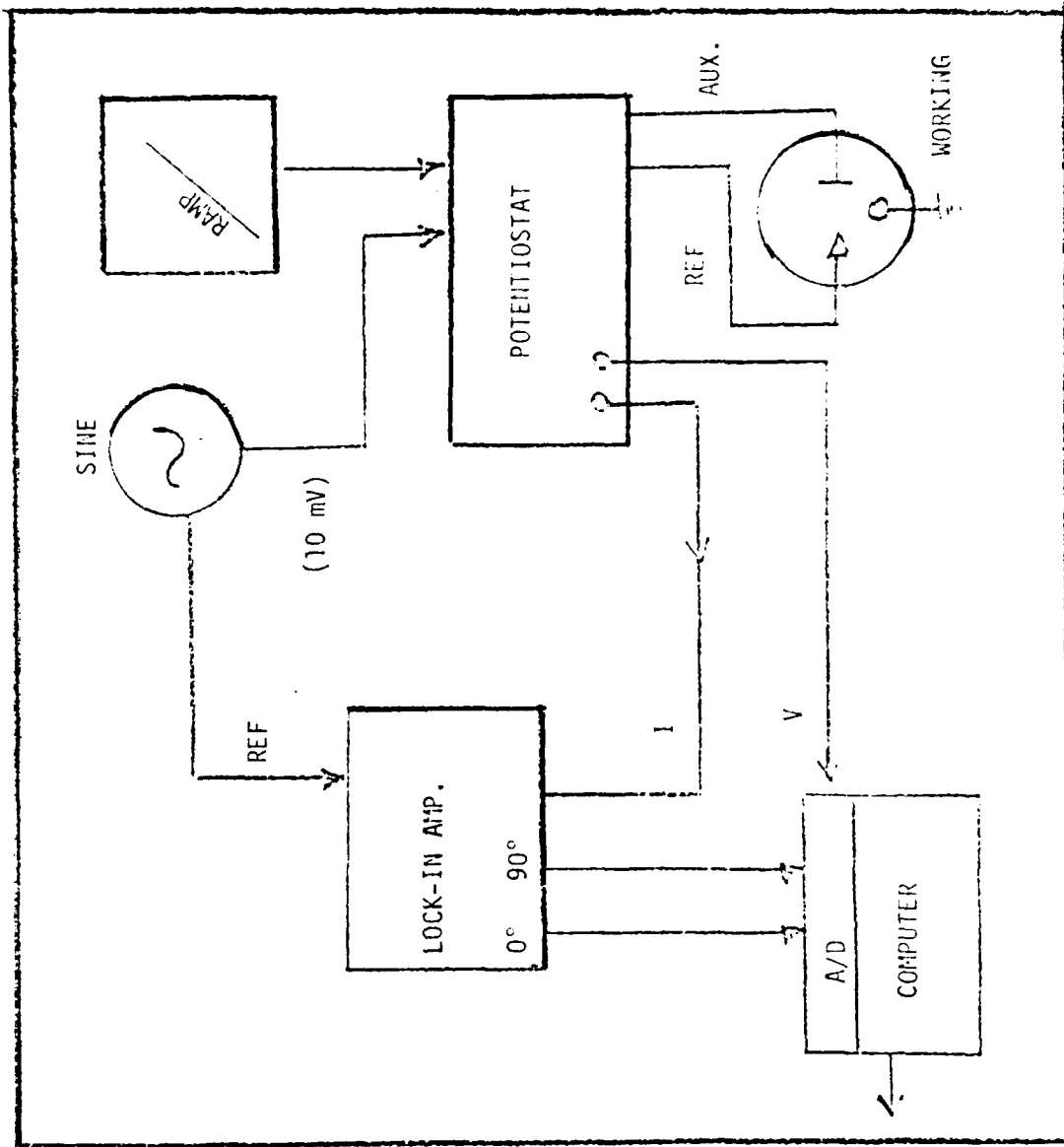
- (i) n-TiO<sub>2</sub> ~ 10<sup>19</sup>/cm<sup>3</sup>
- (ii) Layer type compounds ~ 10<sup>15</sup>/cm<sup>3</sup>
- (iii) n-CdS ~ 10<sup>17</sup>/cm<sup>3</sup>

- (iv) p-GaAs ~ 10<sup>15</sup>/cm<sup>3</sup>
- (v) p-Si ~ 10<sup>15</sup>/cm<sup>3</sup>

## FIGURE CAPTIONS

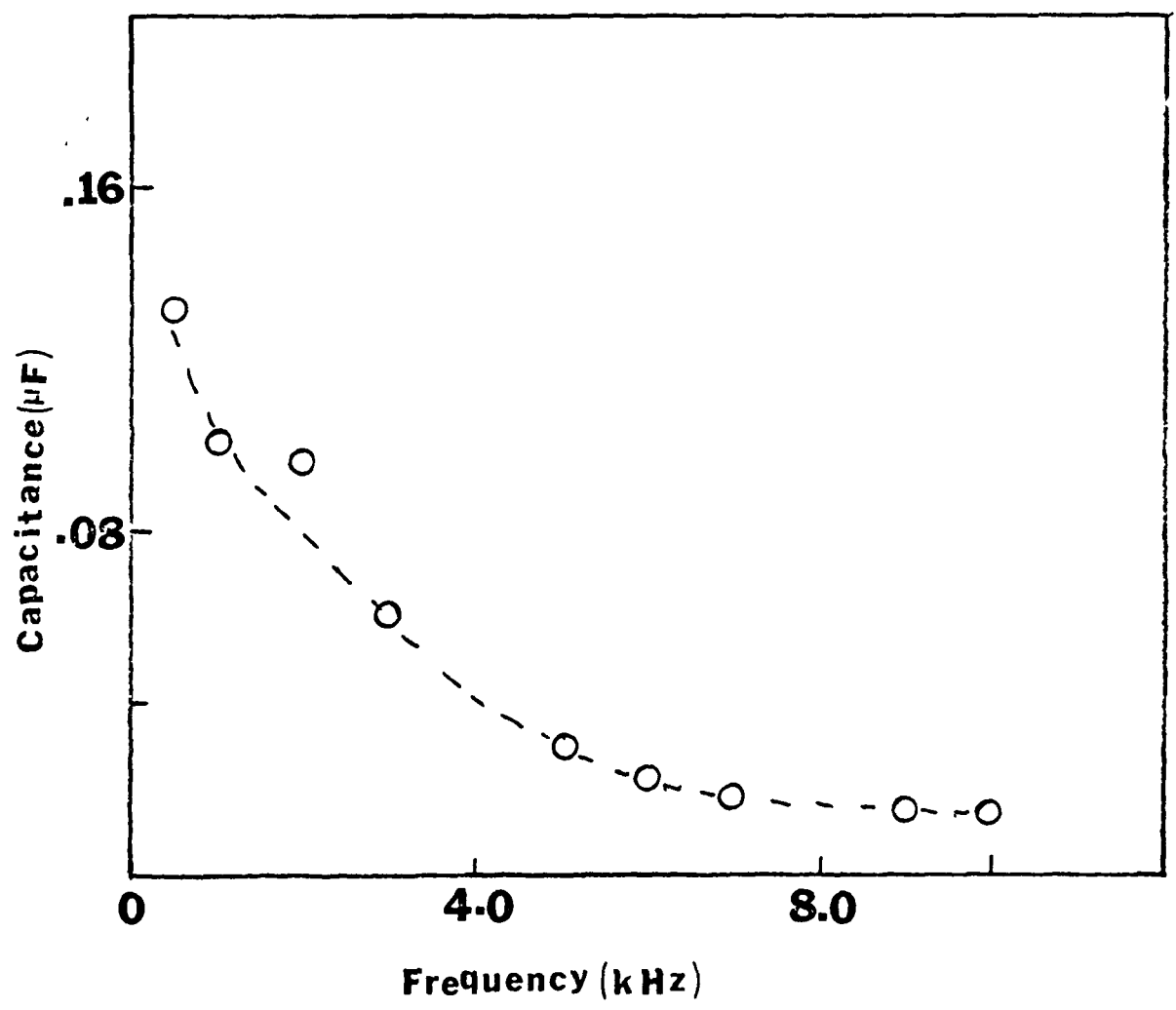
- Figure 1 Block diagram of the set-up used for measuring capacitance at different frequencies.
- Figure 2 C-f plot of p-Si in MeCN containing TBAP (0.1 M) only.
- Figure 3 Schottky-Mott plots for n-TiO<sub>2</sub> contacting MeCN, containing TBAP only (0.1 M),  $f = 0.1, 0.2$  and  $0.5$  kHz.
- Figure 4 Plot of  $V_{fb}$  as a function of  $V_{redox}$  for n-TiO<sub>2</sub>/MeCN, containing a number of redox couples. Supporting electrolyte 0.1 M TBAP. For abbreviations, see Table 1.
- Figure 5 Plot of variation of  $V_{fb}$  with  $V_{redox}$  for n-CdS/MeCN interface. Supporting electrolyte 0.1 M TBAP. For abbreviations see Table 1.
- Figure 6 Plot of variation of  $V_{fb}$  with  $V_{redox}$  for p-Si/MeCN interface. Supporting electrolyte 0.1 M TBAP. For abbreviations see Table 1.
- Figure 7 Plot of variation of  $V_{fb}$  with  $V_{redox}$  for p-GaAs/MeCN interface. Supporting electrolyte 0.1 M TBAP. For abbreviations see Table 1.
- Figure 8 Plot of variation of  $V_{fb}$  with  $V_{redox}$  for p-WSe<sub>2</sub>/MeCN interface. Supporting electrolyte 0.1 M TBAP. For abbreviations see Table 1.

Fig 4



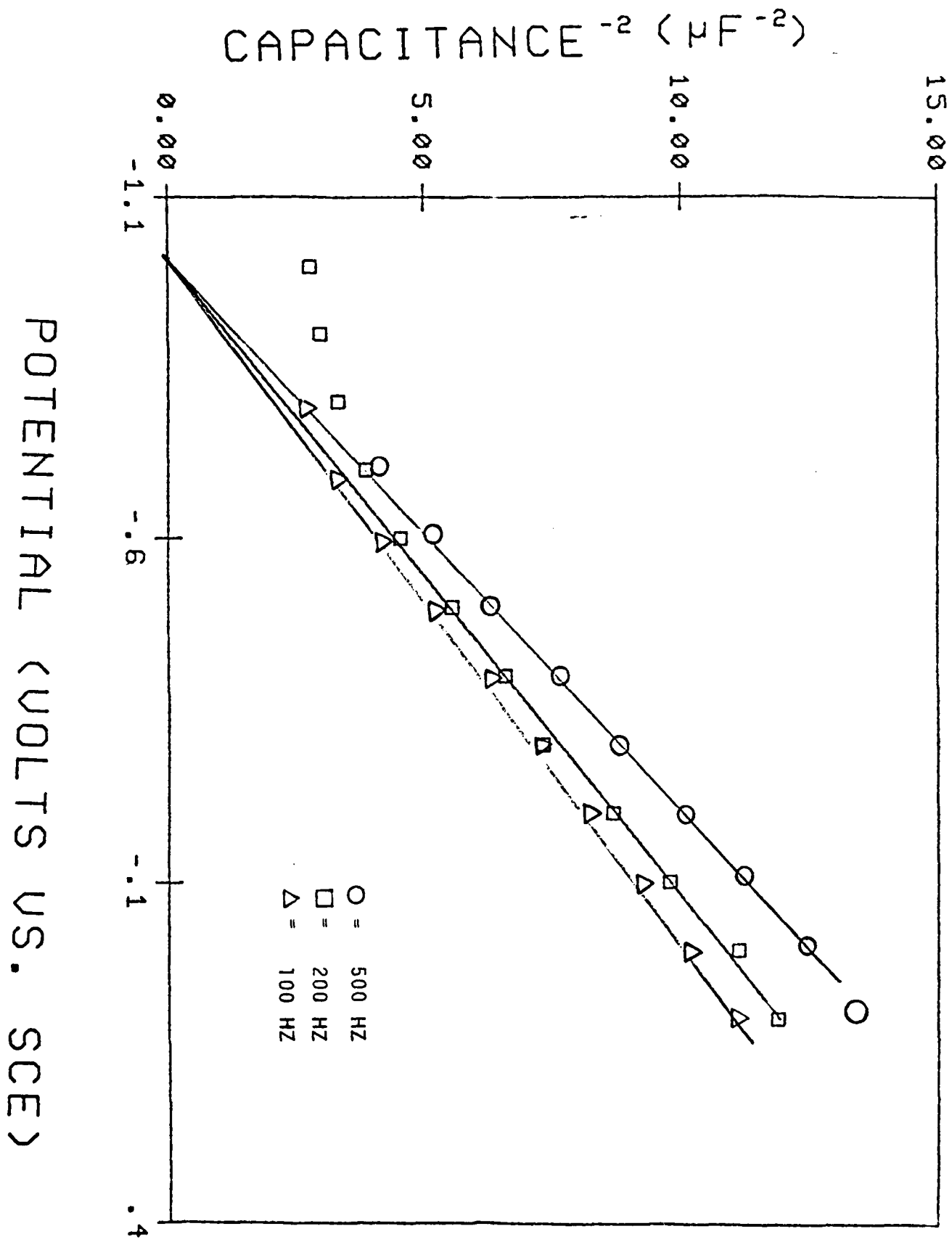
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P-SI/MECN, TBAP



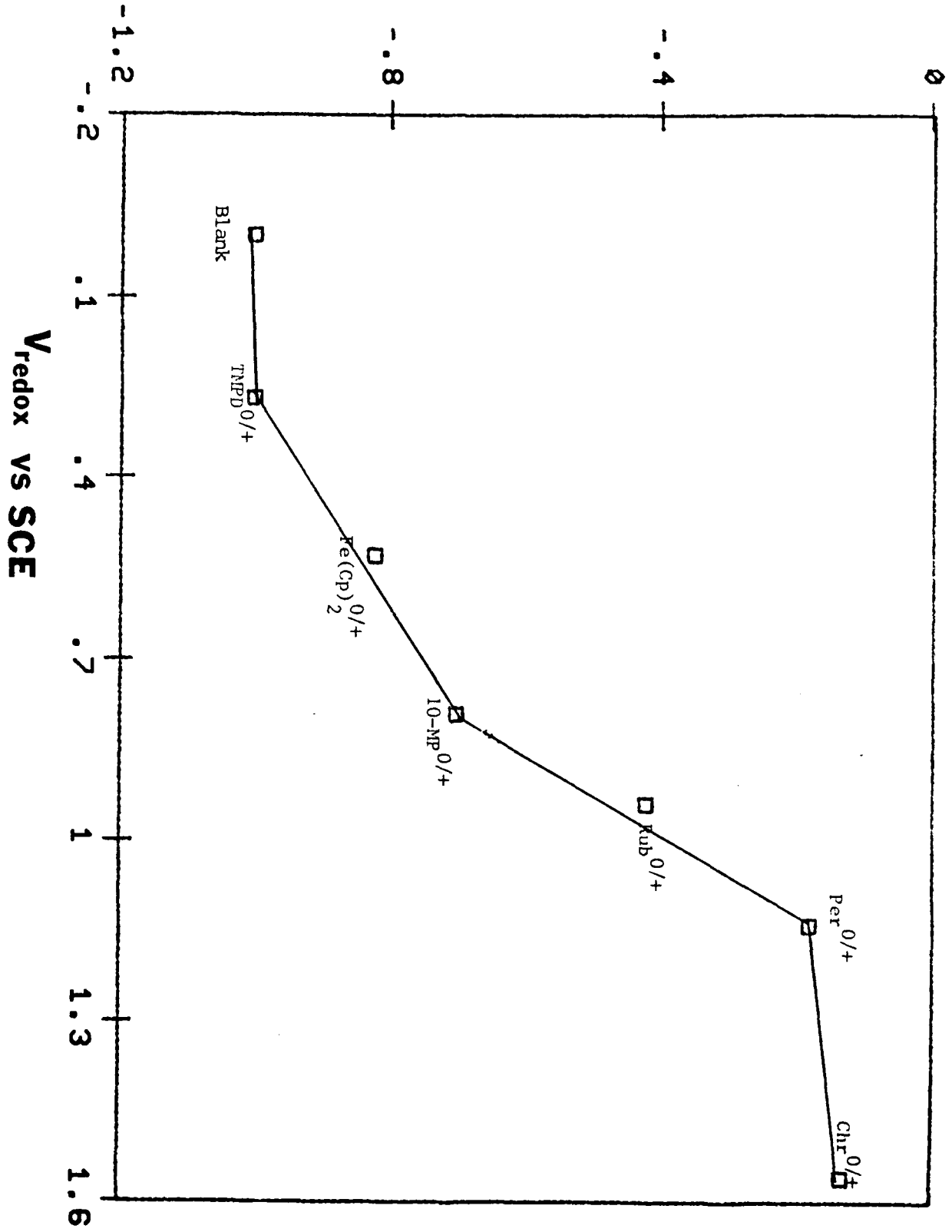
N-TiO<sub>2</sub>/MECN, TBAP

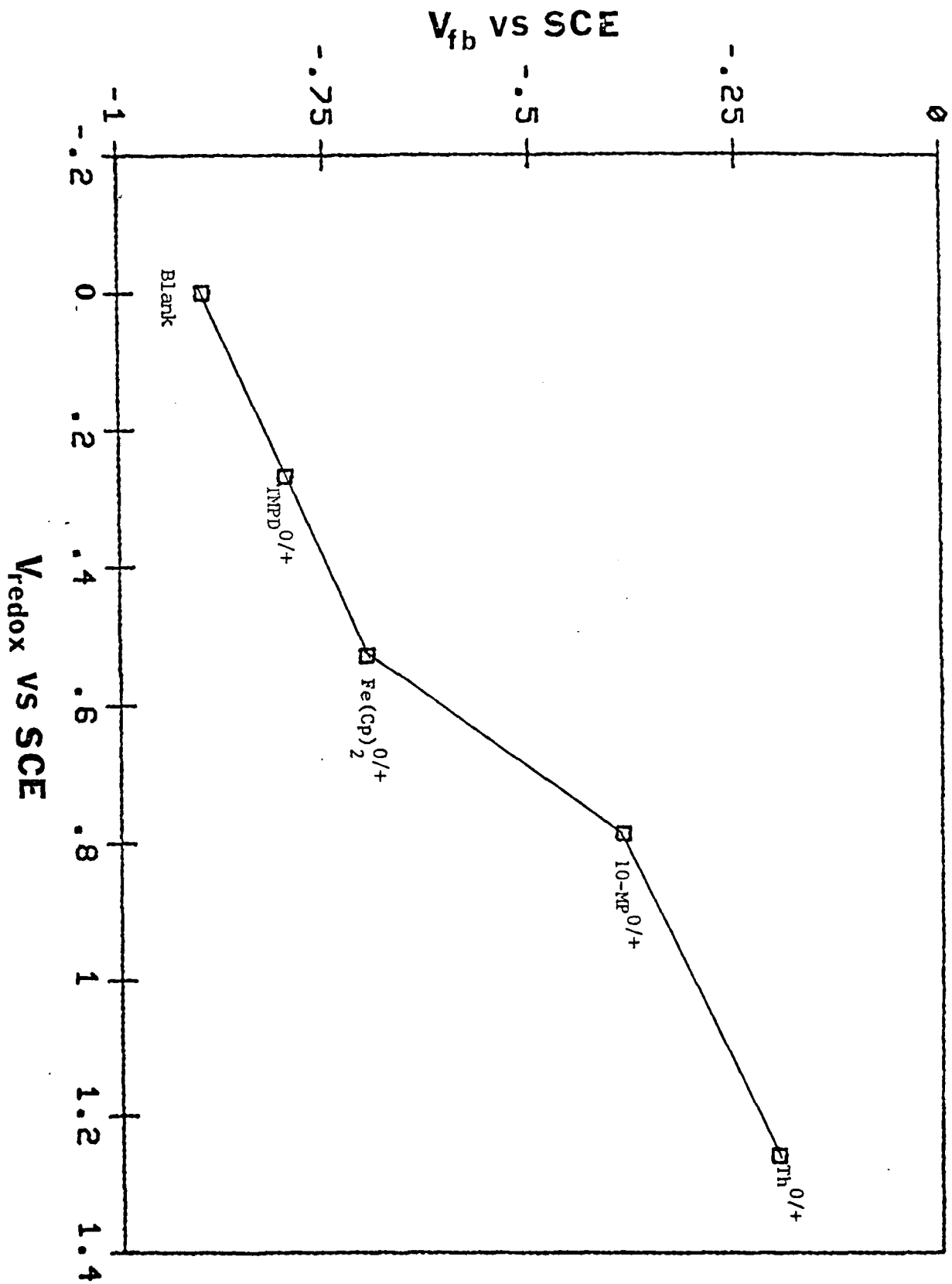
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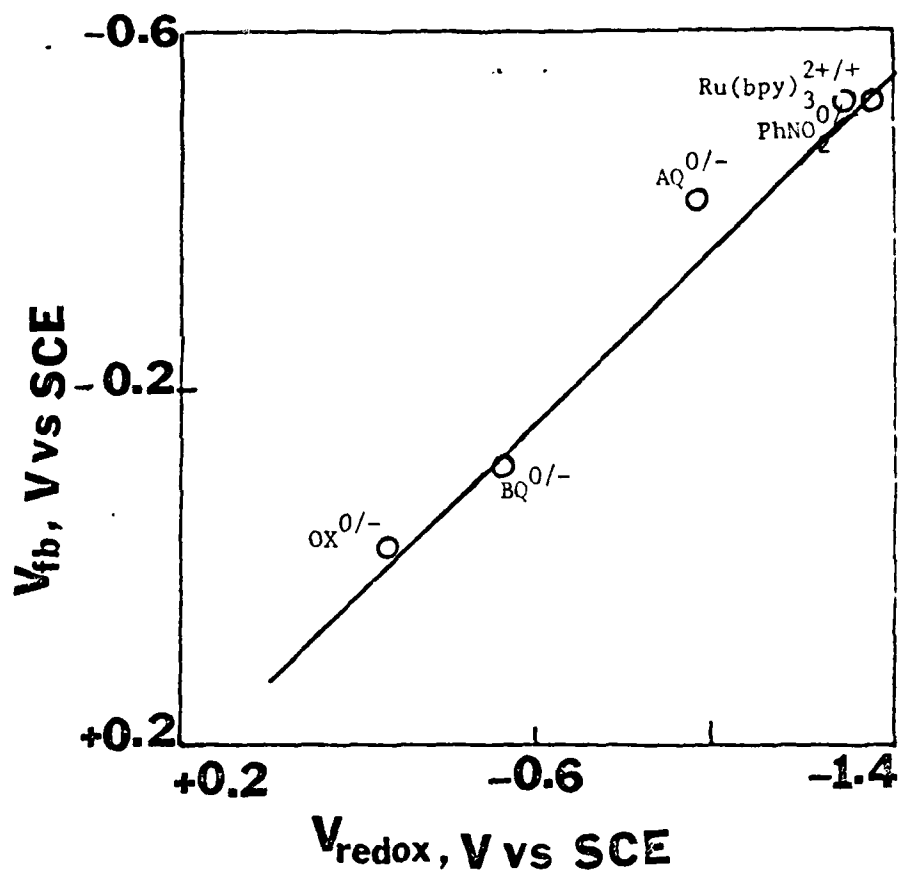


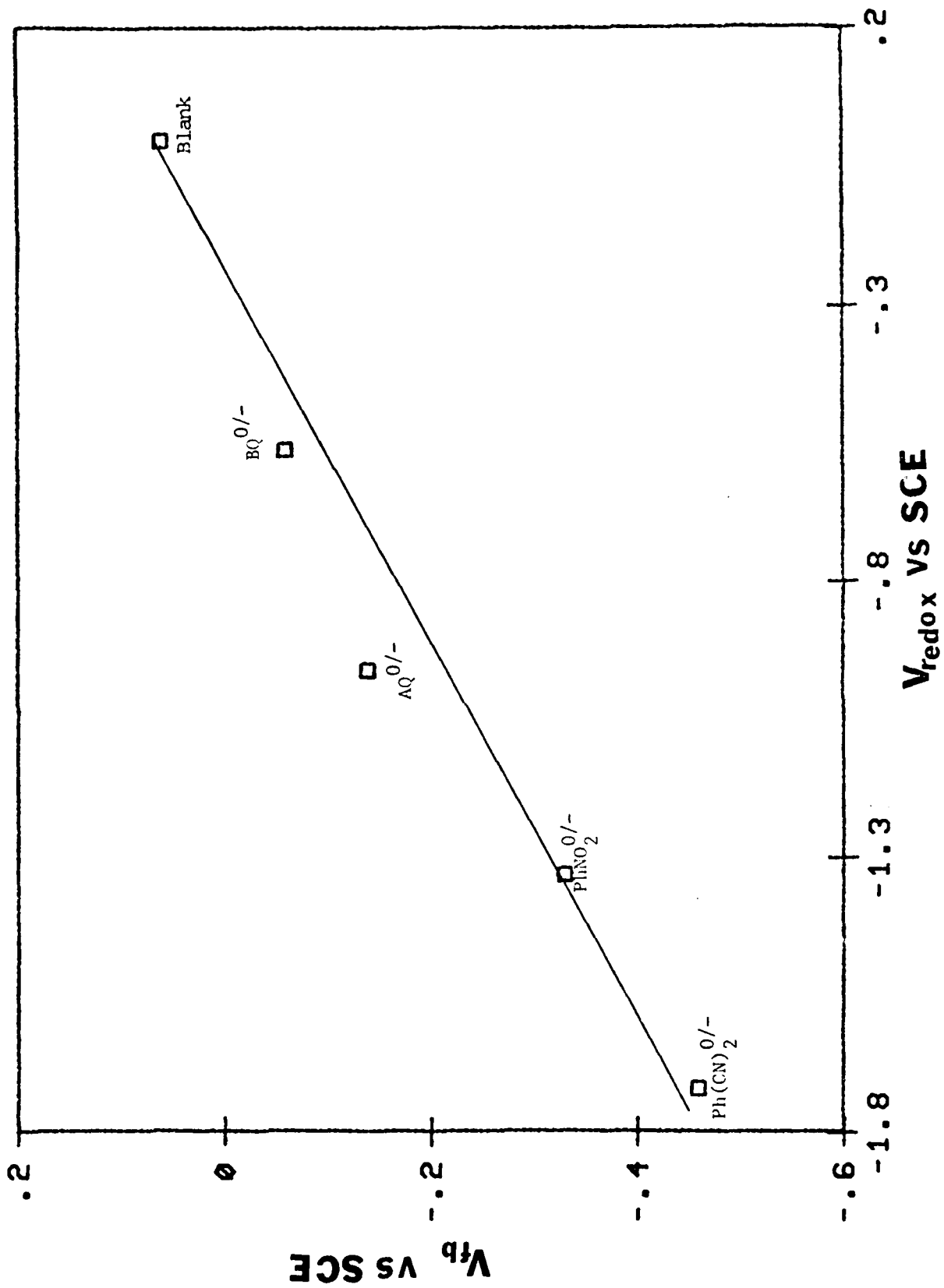


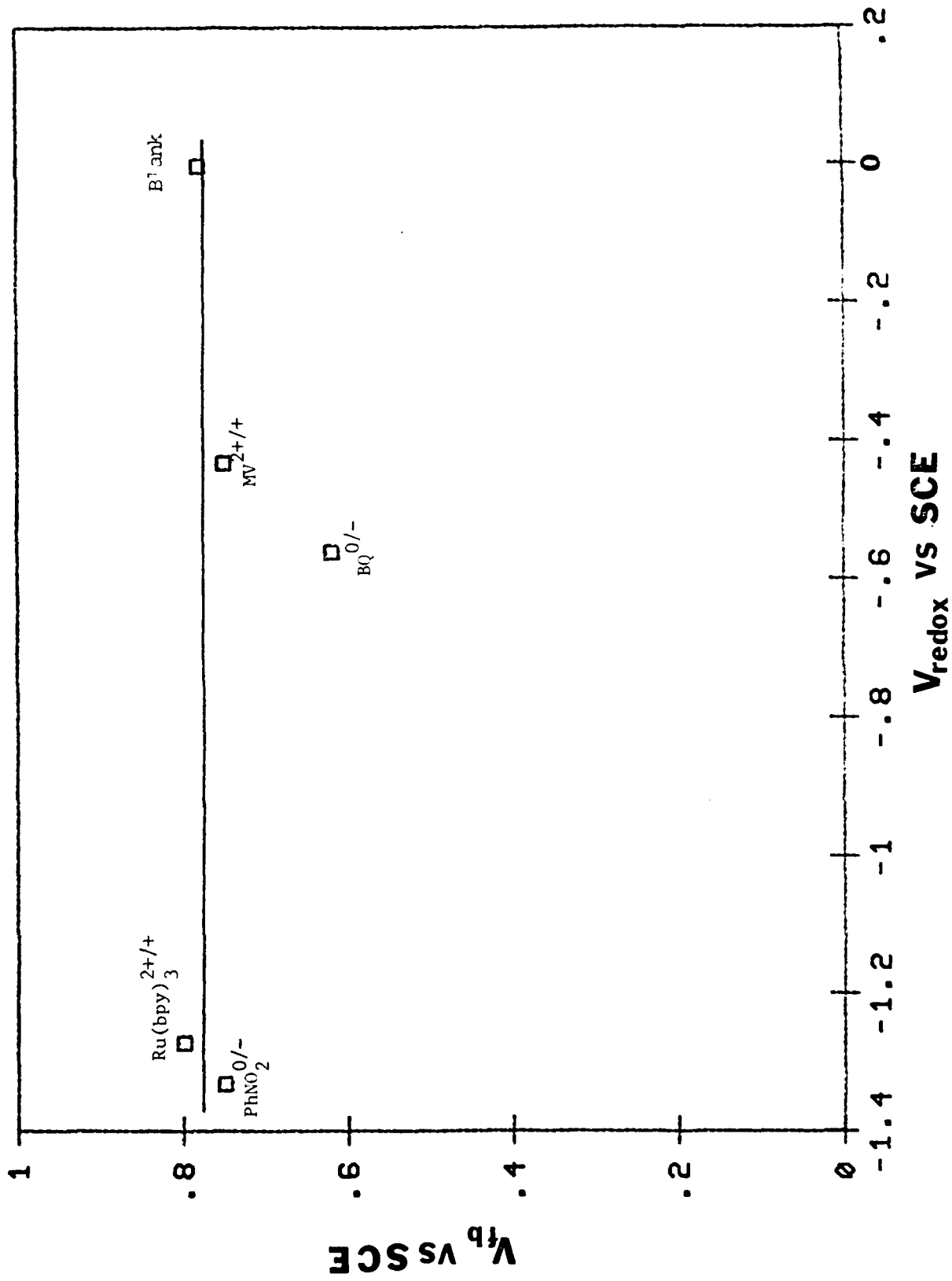
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