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CHARACTERIZATION OF PASSIVE FILMS USING
INFRARED AND RAMAN SPECTROSCOPY

TECHNICAL REPORT NUMBER 2

CONTRACT NUMBER: N00014-76-c-0889

SUBMITTED TO:

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OFFICE OF NAVAL RESEARCH
METALLURGY PROGRAM - CODE 471

PREPARED BY:

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KINGSTON, RHODE ISLAND 02881

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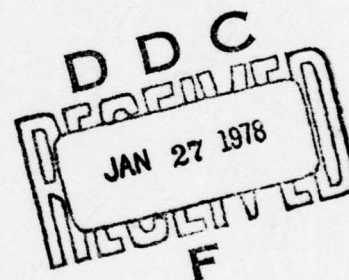
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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) This report discusses research at the University of Rhode Island using infrared (IR) and Raman spectroscopy as the primary investigative tools to study passive film formation. Equipment adapted for this purpose is described. Results of studies on lead and iron are presented. The results are analyzed by comparison with thermodynamic data. A tentative new version of Pourbaix's potential - pH diagram for lead in nil-chloride aqueous solutions is presented.		

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INTRODUCTION

The purpose of this project is to develop and demonstrate the use of the complementary techniques of infrared (IR) and Raman spectroscopy for studying passive films. These two techniques provide structural information, similar to that provided by X-ray or electron scattering, but have the advantage of not requiring long range atomic order (crystallinity). The information provided tends to be from thinner surface layers, and they have been used in catalysis and other surface studies to identify and analyze monomolecular surface layers (1). Raman spectroscopy can be used for in situ analysis of passive films in aqueous solutions (2,3). Water is a relatively poor Raman scatterer, which allows the in situ analysis. Unfortunately similarly weak Raman scattering effects are to be expected in some metal oxide and hydroxide systems. Fortunately, IR spectroscopy is highly sensitive to these structures so the two techniques used together can reveal information not available by other methods.

The overall goal of this research is to provide information on passive films to include the effects of alloying additions and anions on the corrosion behavior of structural alloys. Primary emphasis is to be placed on ferrous alloys because of their engineering importance.

Efforts during the past year have involved technique (equipment) development, studies of the passivity of lead in aqueous solution, and preliminary efforts to study iron oxides. Each of these efforts is discussed below.

EQUIPMENT

IR Spectroscopy:

Figure 1 is a schematic drawing of the infrared sample arrangement used in this study. It is a standard double beam, multiple specular reflectance setup such as was first described by Hannah (4). Infrared spectroscopy is normally performed in the transmission mode so most published spectra are from transmission, not reflectance, studies (5). Several papers have discussed the optimum incident angle and number of reflections for IR reflection work (6,7), but these conditions must still be experimentally determined for each different sample. Polarization of the incident light, or of the light prior to entering the monochromator, is very important and the optimal polarization setting must also be determined for each sample. The effect of polarization on a spectrum is an indication of the long-range orientation of the surface film. Figure 2 shows two spectra of the same lead oxide film with two different polarizations. The difference in the relative band intensities indicates that the oxide molecules have some order.

The IR light path shown in Figure 1 is not a series of simple reflections. The light is absorbed at each reflection off of the sample (M_3 in Figure 1). Infrared radiation is absorbed by the passive film to some extent and reflected at the film-metal substrate interface. The strength of the absorption depends, among other things, on wavelength and angle of incidence. The spectrum obtained is a combination reflection-absorption spectrum. Unequivocal analysis techniques

for these spectra, which may be markedly different from transmission spectra, are not available. Some of the uncertainty in analysis is removed by proper utilization of the reference sample (M_3' in Figure 1).

Despite the difficulties described above, infrared spectroscopy is the structural analysis tool most sensitive to thin surface layers (1). Some researchers have used conventional IR transmission spectroscopy to analyze surface films (8) but this technique involves mechanical or chemical removal of the film from the metal substrate, and thus is not amenable to truly thin films. A more promising method is the reflection-absorption one we have used. Others have used similar techniques for analysis of corrosion product layers with success (9,10).

Use of powdered substrates to study surface reactions is also possible. These have the advantage of maximizing the surface area exposed to the infrared beam, thus improving the sensitivity of the method. There are, however, serious drawbacks. For infrared transmission work metal powder absorbs light much too strongly and must be dispersed in a nonreactive and relatively transparent matrix (alumina or silica). For reflectance spectra the problem of analyzing diffuse reflection results is much more difficult than for specular reflection. The powder technique has not been used to date at the University of Rhode Island primarily because of the difficulty of defining the electrochemical exposure conditions of a porous sample. The problem of fabricating alloyed powder particles (each individual particle having the same composition) also exists. It is anticipated that initial efforts to fabricate and expose reactive powder samples will be undertaken during 1978.

The multiple specular reflectance technique requires a large, flat sample. Figures 3-6 show the special sample holder fabricated to hold these samples. Metallurgical preparation techniques used in standard electrochemical studies (11) have been found adequate for infrared spectroscopic studies.

Raman Spectroscopy:

Figure 7 is a schematic of the Raman spectrometer used in this investigation. The laser furnishes the intense monochromatic light needed to excite the molecules of the sample.

The sample cell used is shown in Figure 8. It is similar to other electrochemical cells except for the flat bottom and positioning of the sample. The metal sample is tilted to the optimum angle, approximately 20° for most samples, for collecting scattered light in the monochromator while admitting a minimum of reflected light.

This cell is used to obtain in situ Raman spectra of electrode surfaces in controlled electrochemical environments. After the completion of exposure in solution samples are dried and Raman spectra of the surfaces recorded. The dry sample and in-situ spectra have, in all cases, been similar with somewhat greater spectral intensity observed from the dry surface as shown in Figure 9. Replicate samples exposed in a standard electrochemical cell and first analyzed by infrared spectroscopy were also analyzed by the Raman technique. Both IR and Raman spectra were used to identify the surface films. It is to be noted that all films investigated to date have been metal oxides. It is to be expected that differences between in situ and removed-from-environment spectra may be observed when this investigation is extended to systems where water of hydration or adsorbed water is important.

LEAD

Lead was chosen as the metal for initial studies in this investigation because:

1. lead oxides are predicted to be good Raman scatterers (2);
2. the passive films formed on lead in many aqueous environments are thick and can be analyzed by X-ray diffraction to confirm infrared and Raman spectral results; and
3. lead has a number of common valence states, thus a number of different oxides may form on lead surfaces. Some lead oxides exist as two allotropes.

These latter conditions are analogous to those which would be expected for iron, chromium, and iron-chromium alloys where thin, possibly amorphous, passive films would make confirmation by X-ray or electron diffraction impossible.

Figure 10 is a simplified version of the potential - pH diagram published by Pourbaix (12). It was used to select conditions for a series of exposures intended to produce different passive films on lead substrates. The exposures selected and the results are summarized in Table 1. Figures 2 and 11 show IR spectra of lead oxide films formed under different exposure conditions. Figure 12 shows the infrared spectrum of lead exposed in a carbonate buffer. The observed bands can all be ascribed to basic lead carbonate. The PbO_2 on the surface, which was detected by X-ray diffraction, is not detected by either IR or Raman spectroscopy. Similar effects have been reported in infrared spectra of iron exposed in borate buffers (10). Problems of this nature have necessitated careful choice of buffers to avoid interference by buffer species, a problem for any experimental technique.

Infrared absorption spectra of many metal oxides are available (13), but few Raman spectra of oxides have been published. Of the lead oxides, only spectra of the two forms of PbO can be found in the literature (14). It was necessary to record the spectrum of pure Pb_3O_4 shown in Figure 13 in order to use Raman spectroscopy to identify this compound. A typical surface film spectrum is shown in Figure 14 where it is compared with the spectrum of the pure compound.

Figure 15 is a Pourbaix diagram for lead in nil-chloride solutions. The lines have been redrawn from Figure 10 to reflect the latest thermodynamic data available from the National Bureau of Standards. Figure 15 is in closer agreement with the experimental results of this study. Discrepancies between those species predicted in the diagram and those determined experimentally have not been explained by thermodynamics. Scanning electron microscopy investigations to confirm the spectral results are underway.

The effects of anions on the passive films formed on lead are also being investigated. Experimental Pourbaix diagram investigations for chloride-, sulfate-, and carbonate-containing solutions are underway.

IRON

Few published Raman spectra are available for iron oxides. This situation necessitated the synthesis of a series of iron oxides and the recording of their Raman spectra. Figures 16 and 17 show the spectra of allotropes of FeOOH and Fe_2O_3 . Figure 18 shows the Raman spectrum of Fe_3O_4 in a powder sample and the spectrum of the surface of Armco iron exposed to boiling water. Poling has published the infrared spectrum of an Fe_3O_4 film formed under these conditions (15), and the IR spectrum obtained in our investigation confirms his results. Figure 19 is the IR spectrum of an Armco iron surface after air exposure at 220°C . The presence of several metal oxide allotropes is indicated.

Oxide films on iron are substantially thinner than those formed on lead. This makes confirmation of experimental results by X-ray or electron diffraction more difficult. For this reason primary emphasis during recent months has been shifted to studies of lead surfaces. Further studies on iron are underway. It is anticipated that high-temperature exposures of ferrous alloys will provide thick films amenable to X-ray diffraction confirmation of the infrared and Raman spectra obtained. These will be used for comparison with spectra obtained from low-temperature exposures in aqueous solutions.

TABLE 1

LEAD EXPOSURES CONDUCTED IN NIL-CHLORIDE SOLUTIONS

pH*	Potential (Volts SHE)	Time of Exposure	Spectrum Observed
7	-0.60	18 hr	tetragonal PbO
	-0.10	3 hr	tetragonal PbO
	+0.60	18 hr	tetragonal PbO
	+1.10	17 hr	buffer
10	-0.76	17 hr	tetragonal PbO
	-0.27	45 min	tetragonal PbO
	-0.27	18 hr	tetragonal PbO
	+0.24	17 hr	tetragonal PbO
	+0.46	22 hr	tetragonal PbO
	+0.51	17 hr	tetragonal PbO
	+0.90	18 hr	buffer

* Phosphate buffer used for pH7 solution, carbonate buffer used for pH10 solution.

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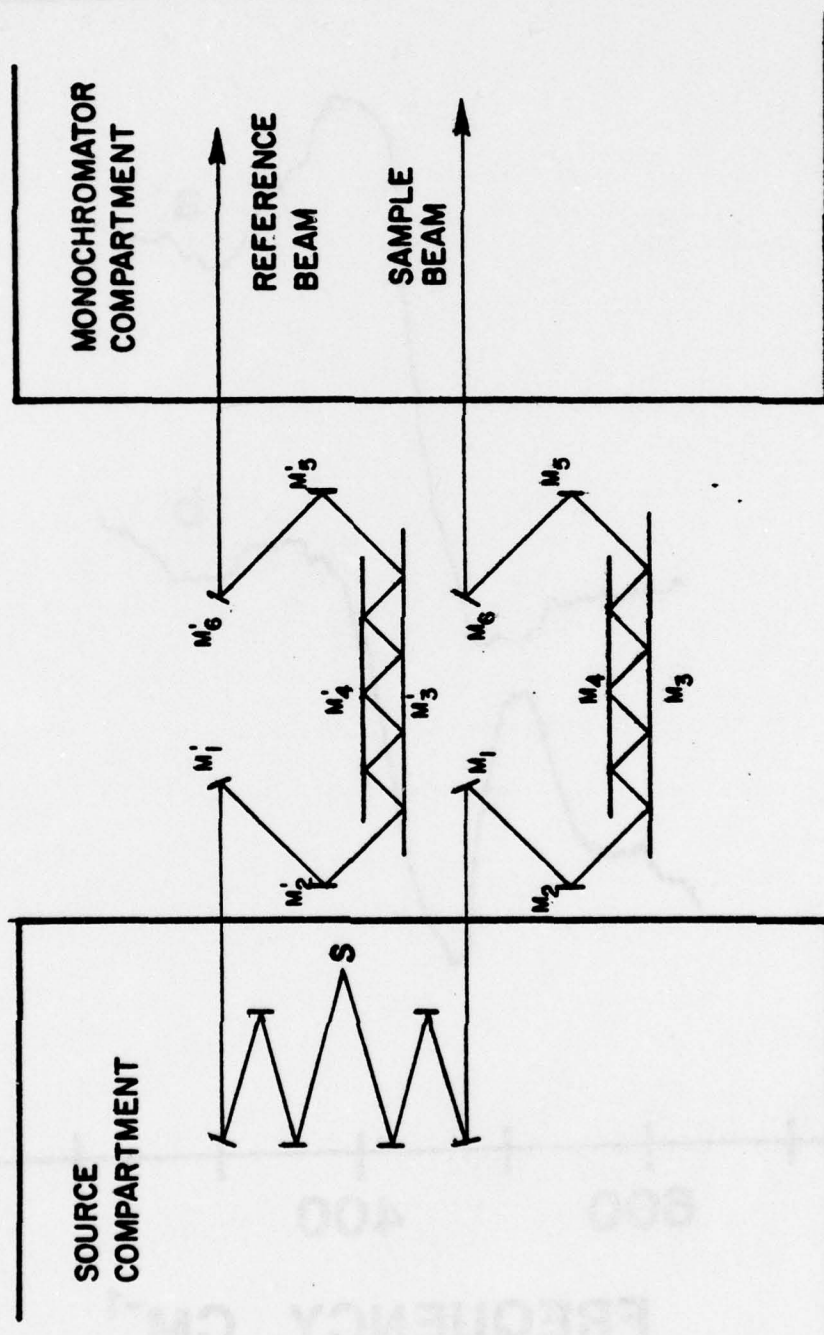
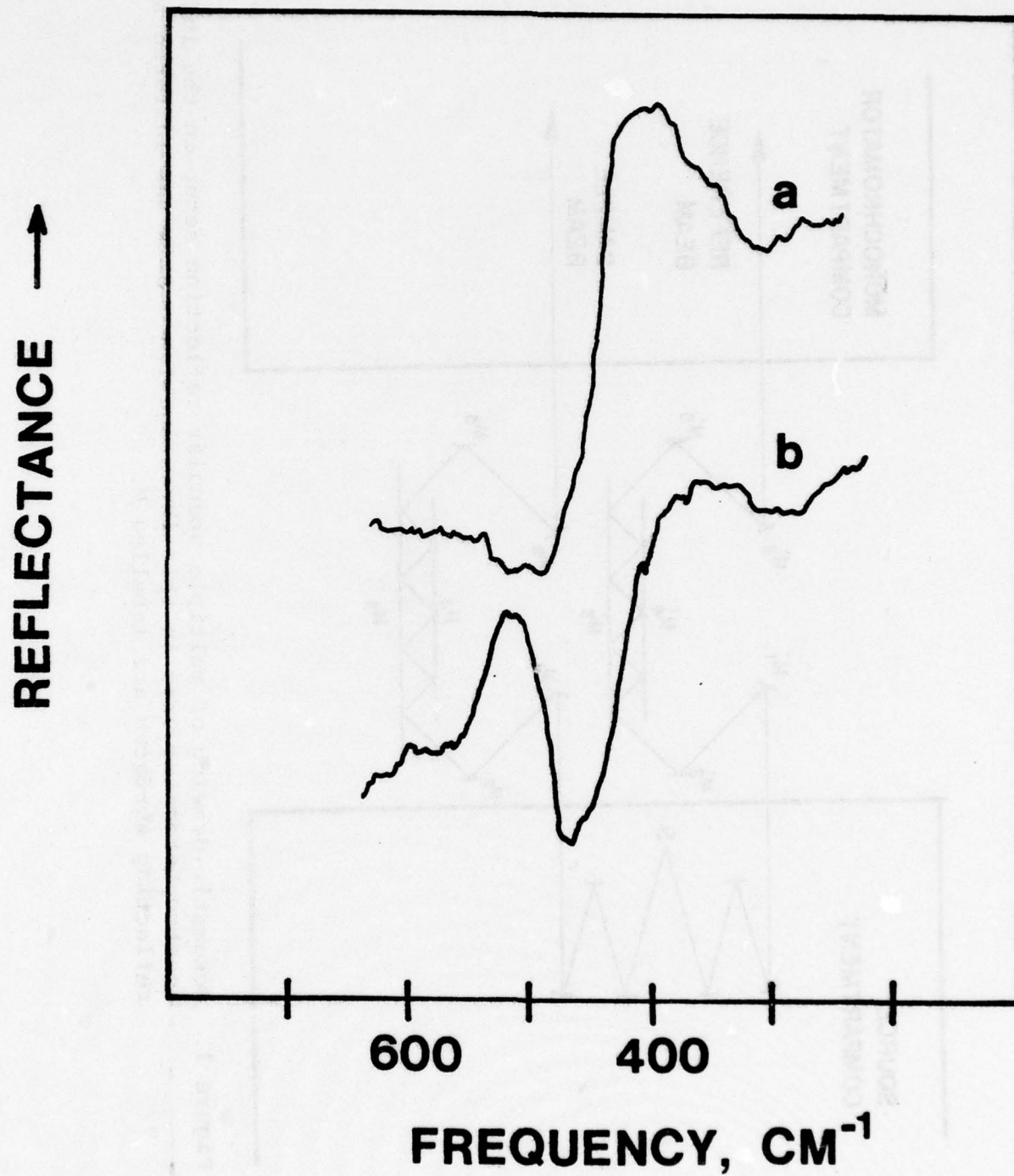


Figure 1. Schematic drawing of multiple specular reflection setup in the infrared spectrophotometer used in this investigation. S is the infrared source, reflecting surfaces are labelled M.

Figure 2. (a) Infrared reflection-absorption spectrum of a lead surface after 17 hour exposure in pH 10 solution at +0.24v (vs. SHE), (b) the same surface with a different setting of the polarizer



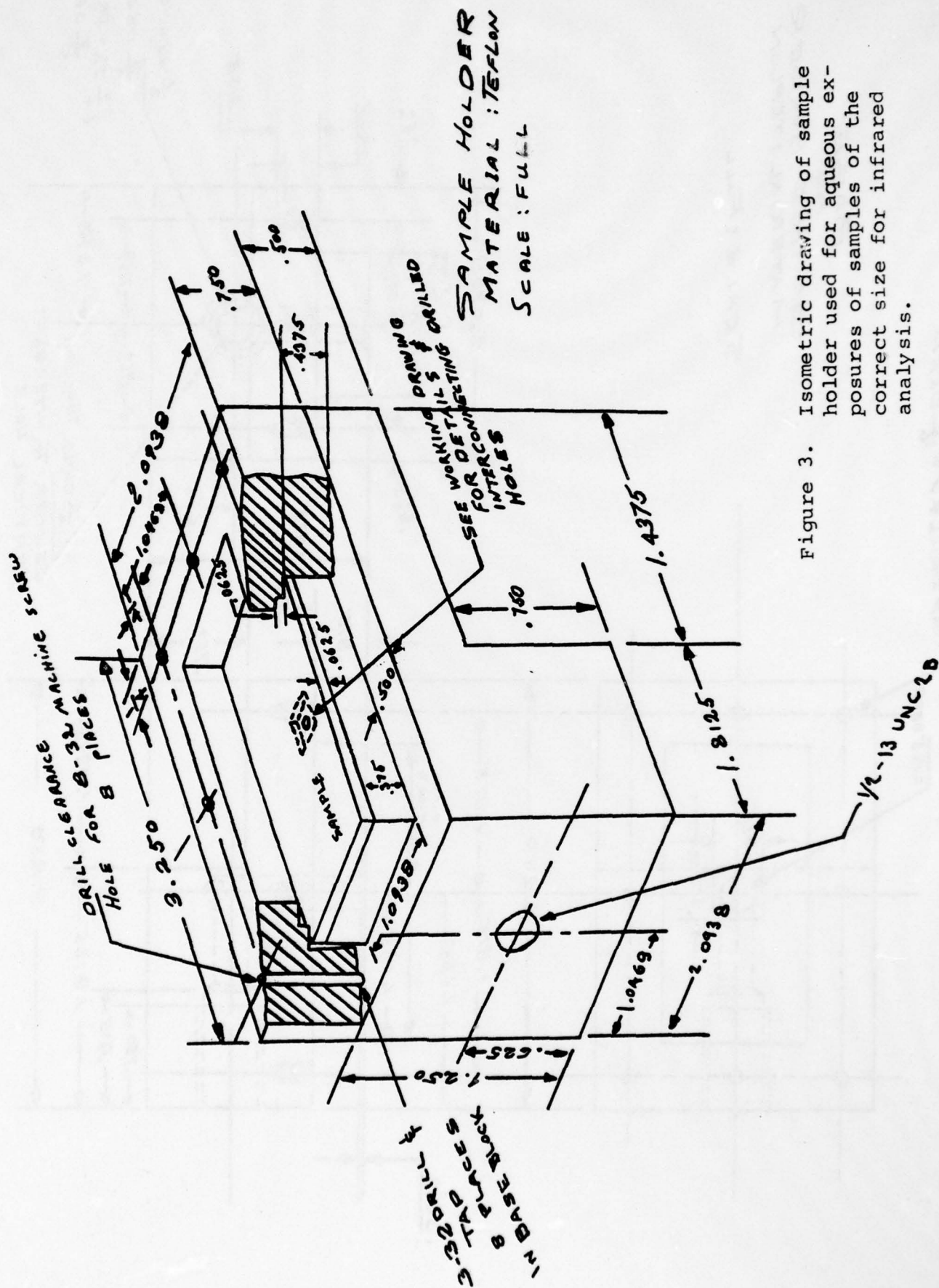


Figure 4. Base for sample holder

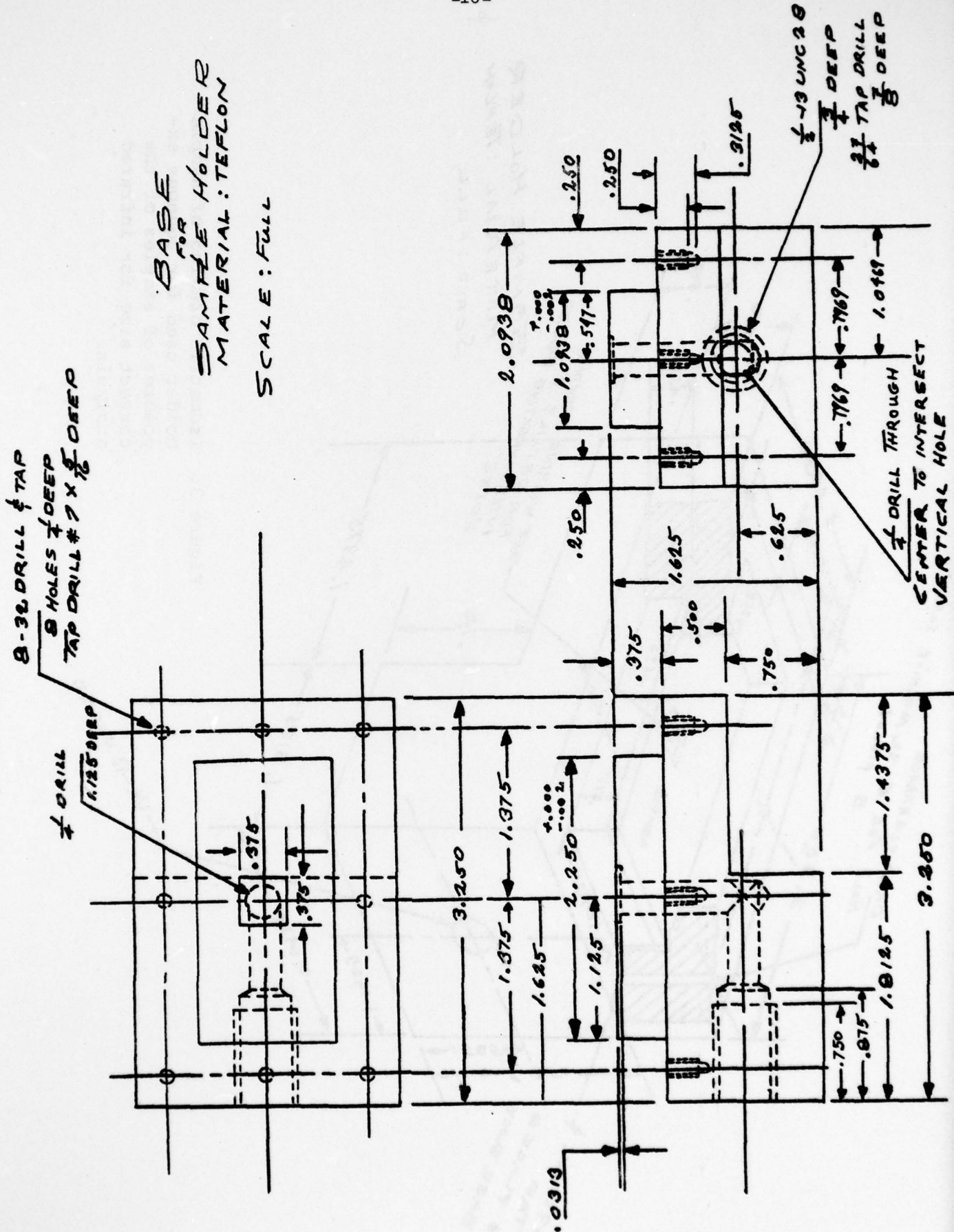
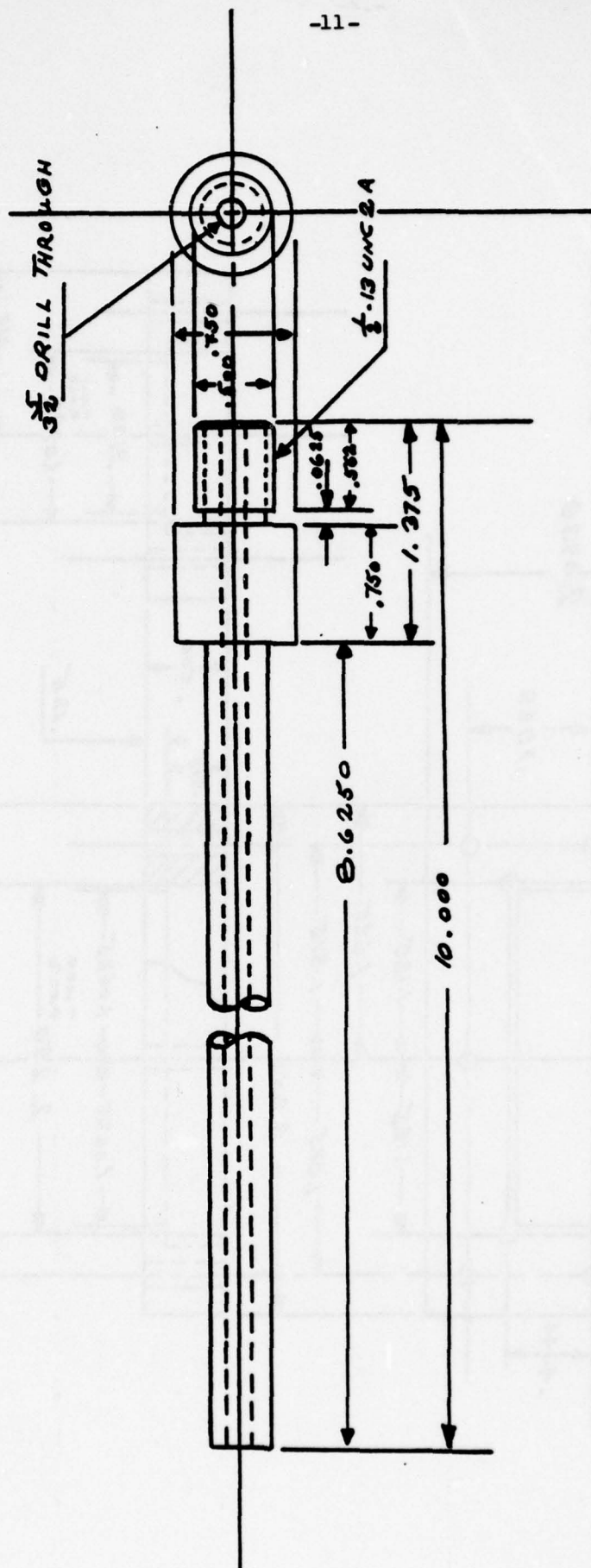


Figure 5. Support tube for sample holder



SAMPLE HOLDER TUBE
MATERIAL: TEFLON

SCALE: FULL

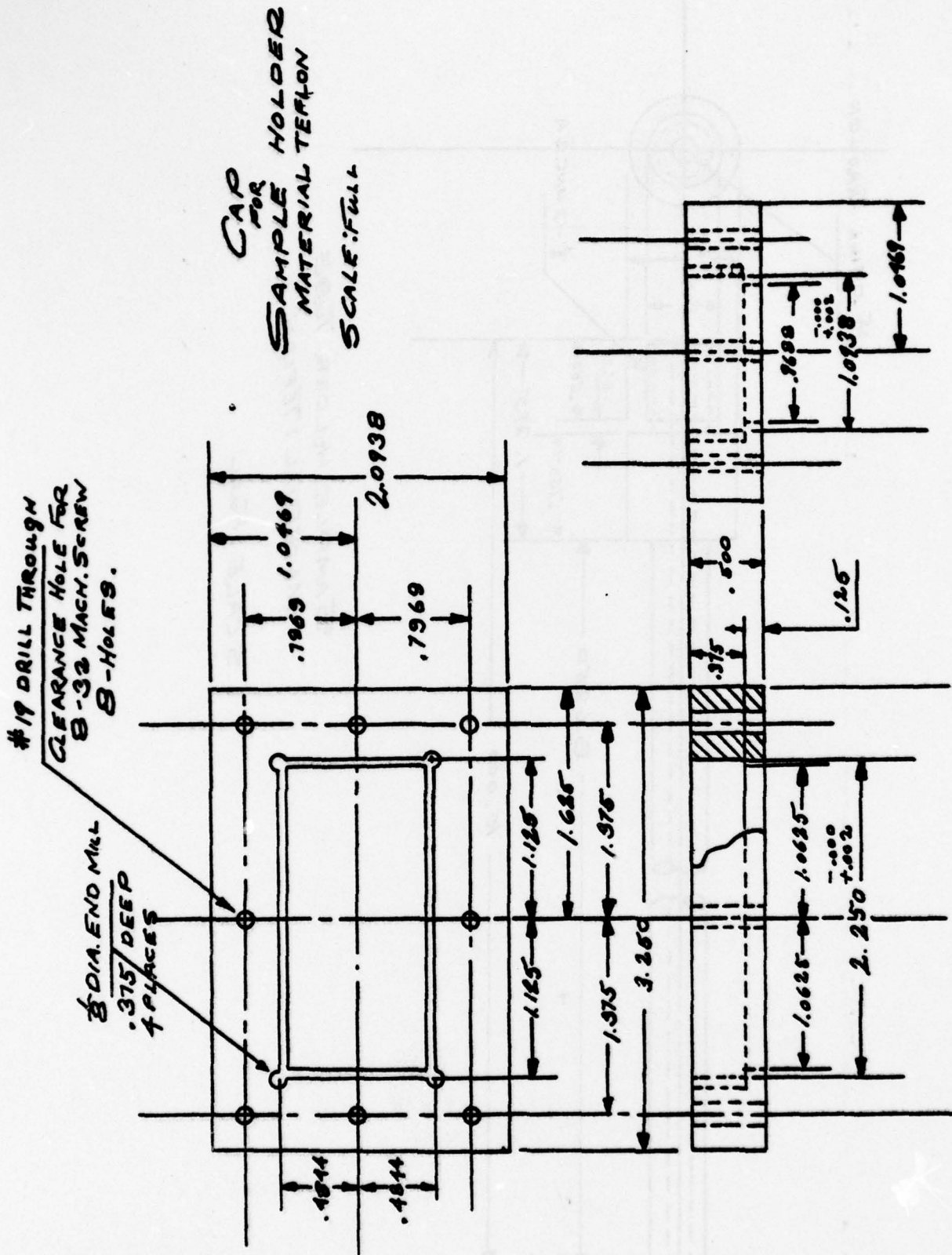
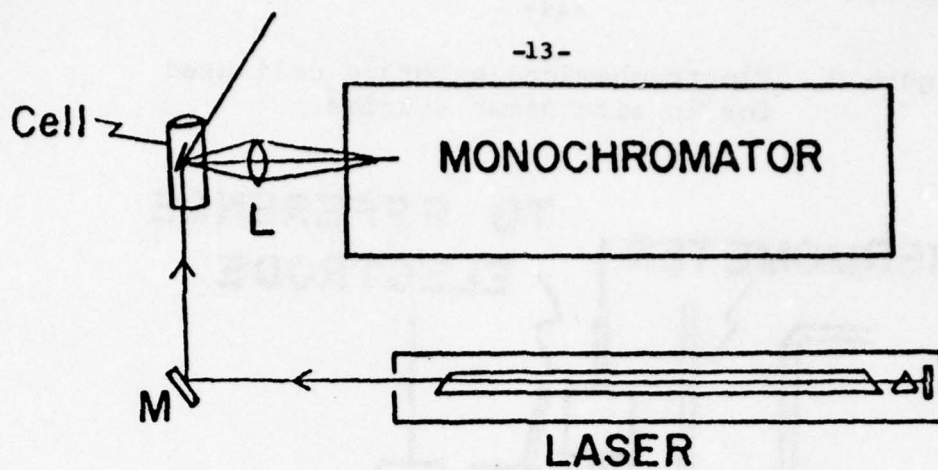
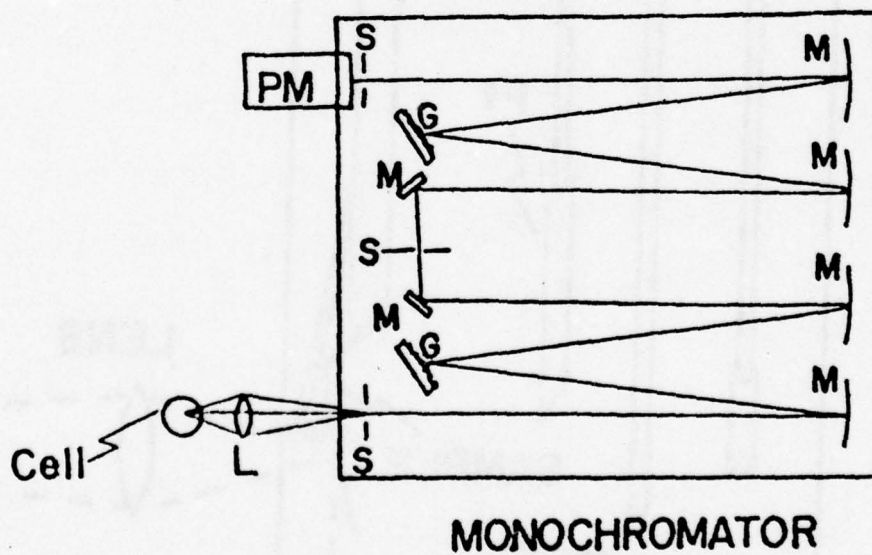


Figure 6. Sample holder cap



SIDE VIEW



TOP VIEW

Figure 7. Optical schematic of Raman spectrometer

Figure 8. Electrochemical exposure cell used for in situ Raman studies

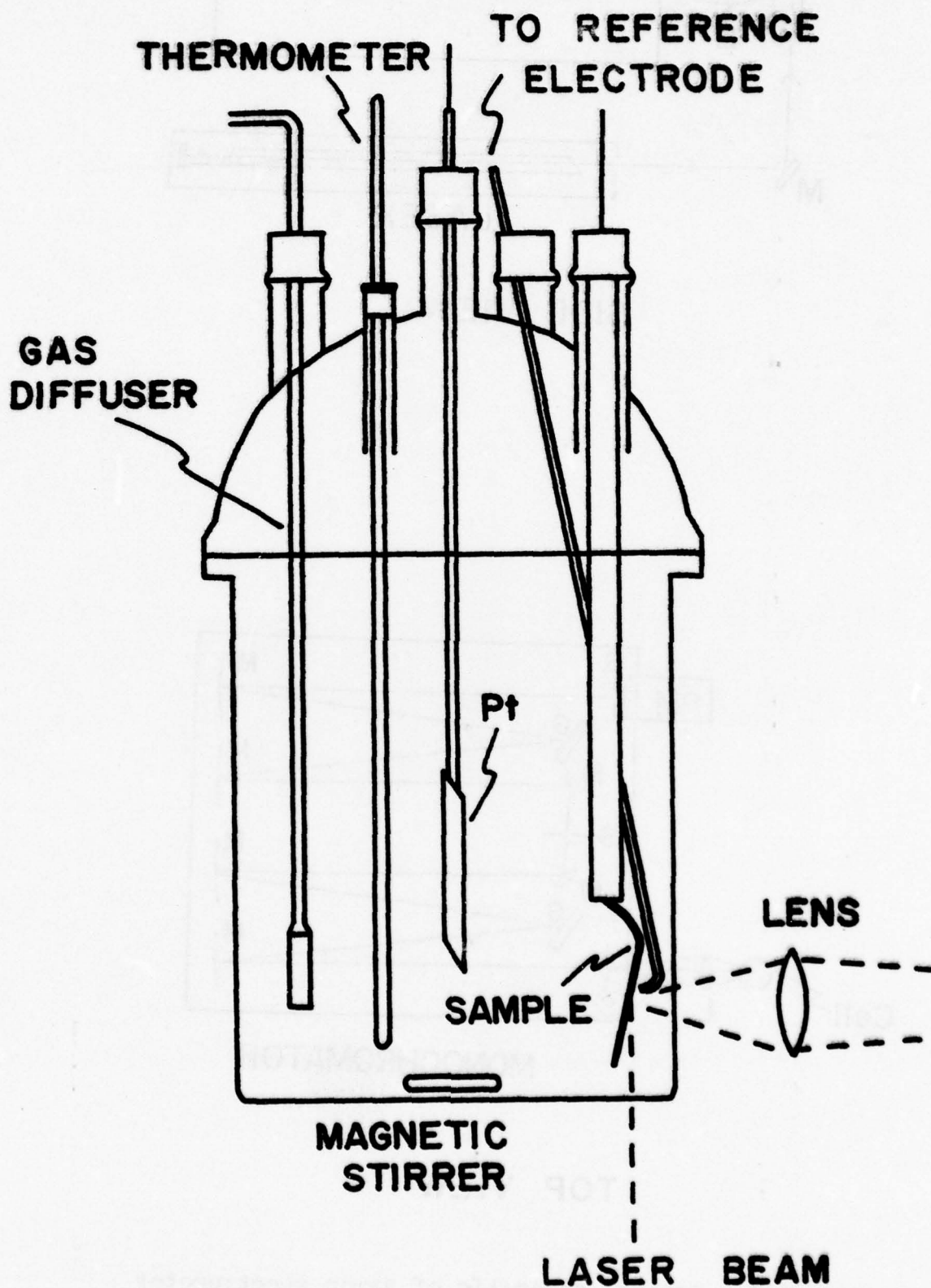


Figure 9. (a) In situ Raman spectrum of tetragonal PbO film formed on a lead surface after 2 hours at -0.60v (vs. SHE) in pH 7 buffer solution, (b) Raman spectrum of the same sample after completion of 3-1/2 hours of exposure and 30 minutes drying at room temperature

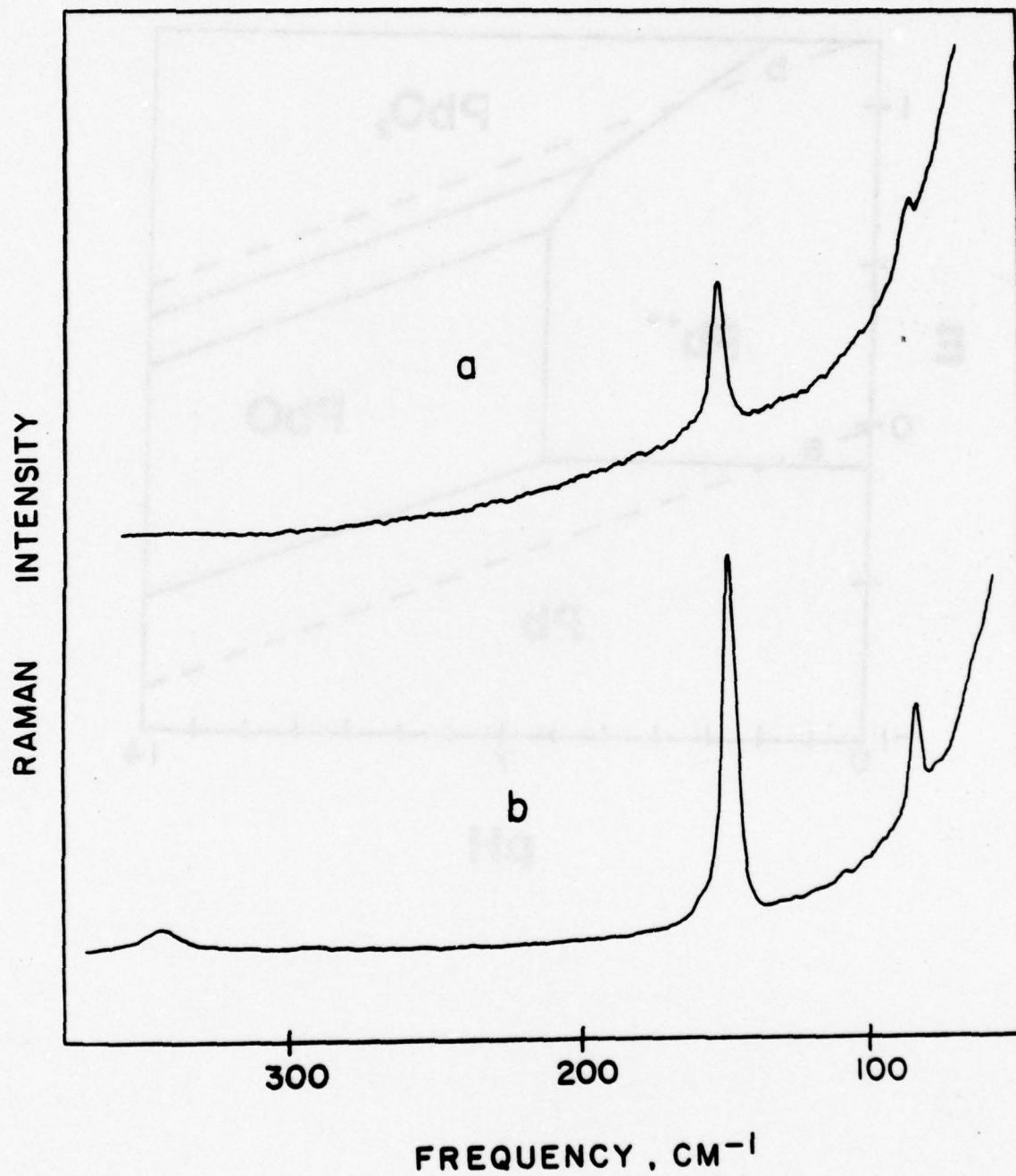


Figure 10. Simplified potential-pH (Pourbaix) diagram for lead in water, assuming no anion effects (Reference 12)

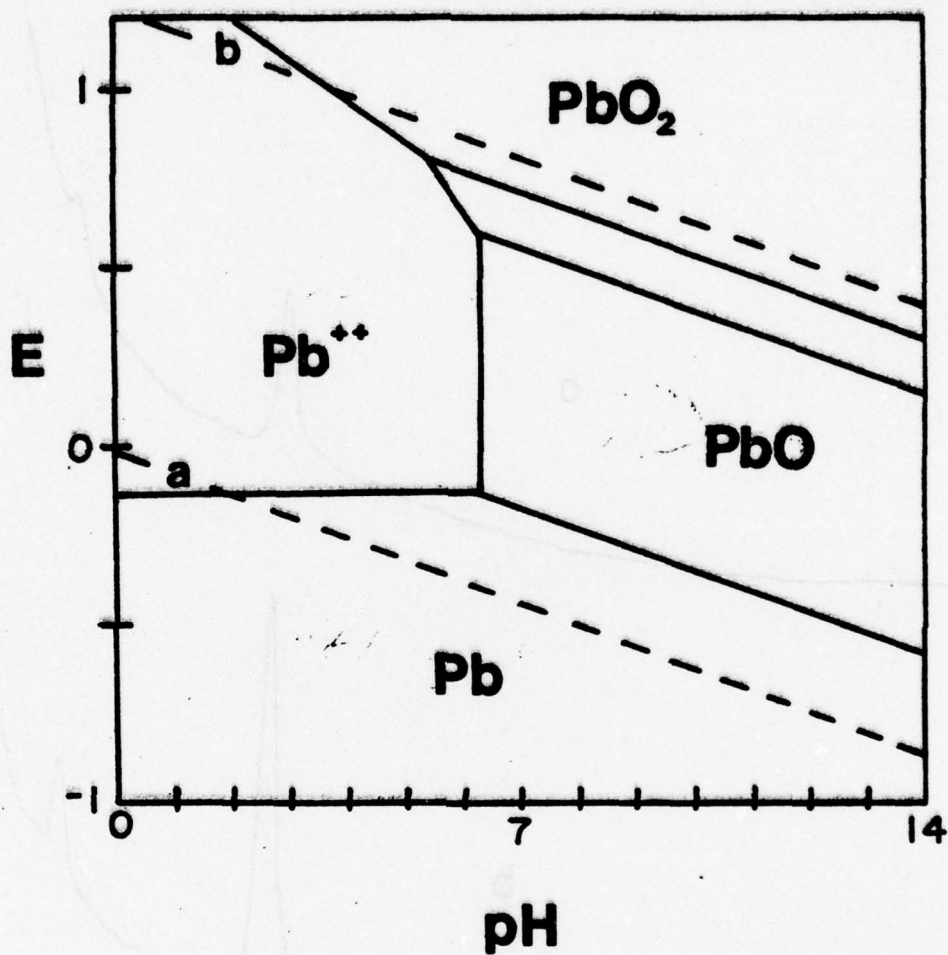


Figure 11. Infrared spectrum of orthorhombic PbO surface film on lead metal. The sample was exposed to 0.1M chloride, pH 7 buffer solution at +0.59v (vs. SHE) for 17 hours.

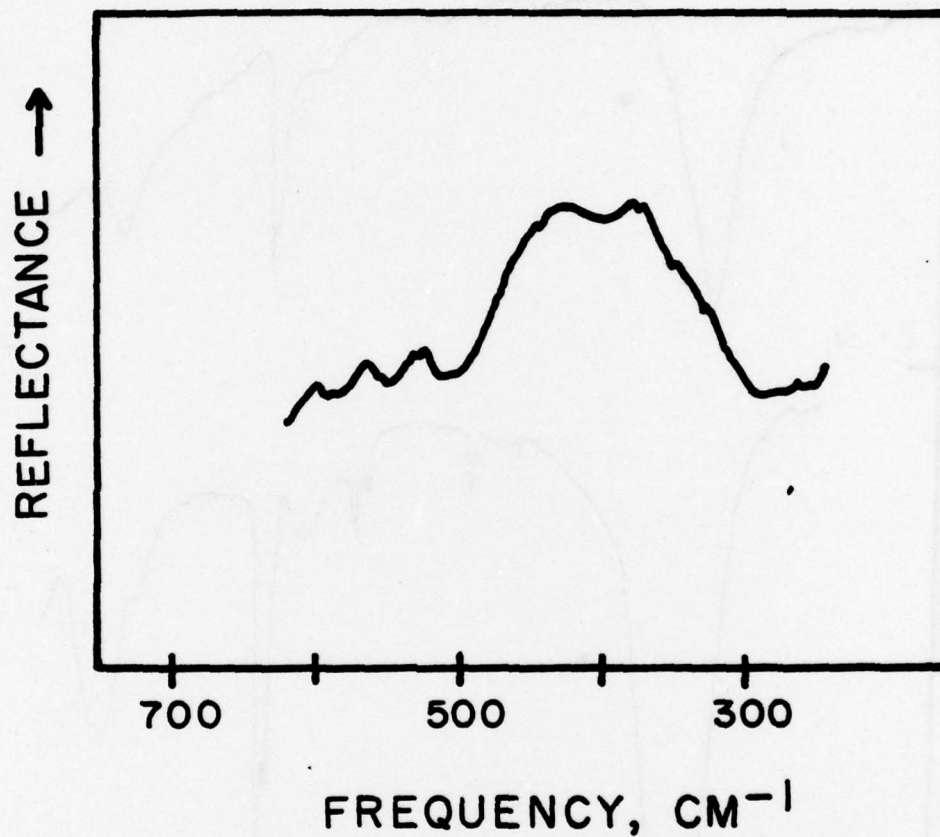


Figure 12. Infrared spectra of basic lead carbonate (a) film on lead which had been exposed 18 hours at +0.90v (vs. SHE) in pH 10 carbonate buffer, (b) reagent grade $(\text{PbCO}_3)_2 \cdot \text{Pb}(\text{OH})_2$ in KBr pellet

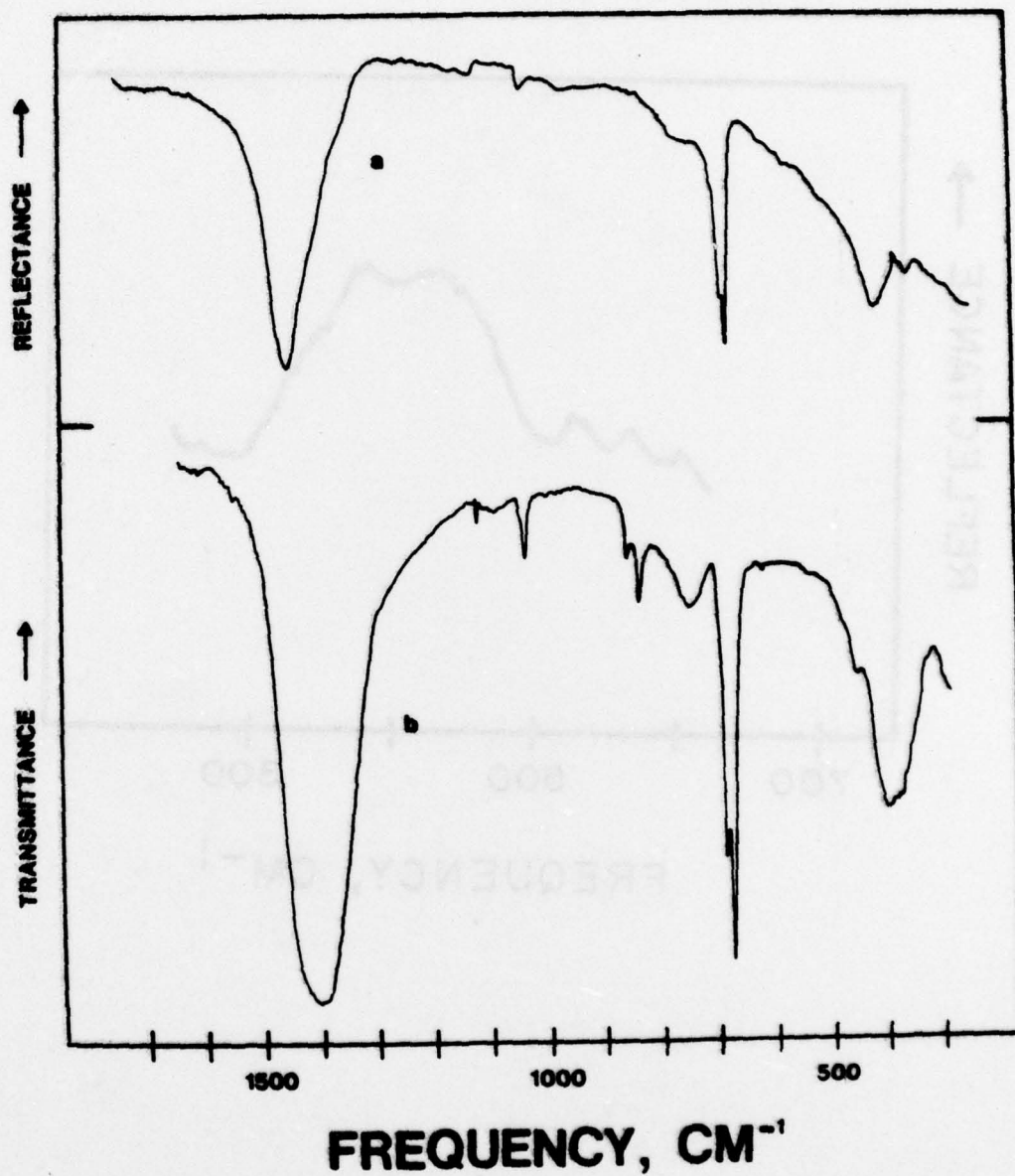


Figure 13. Raman spectrum of Pb_3O_4

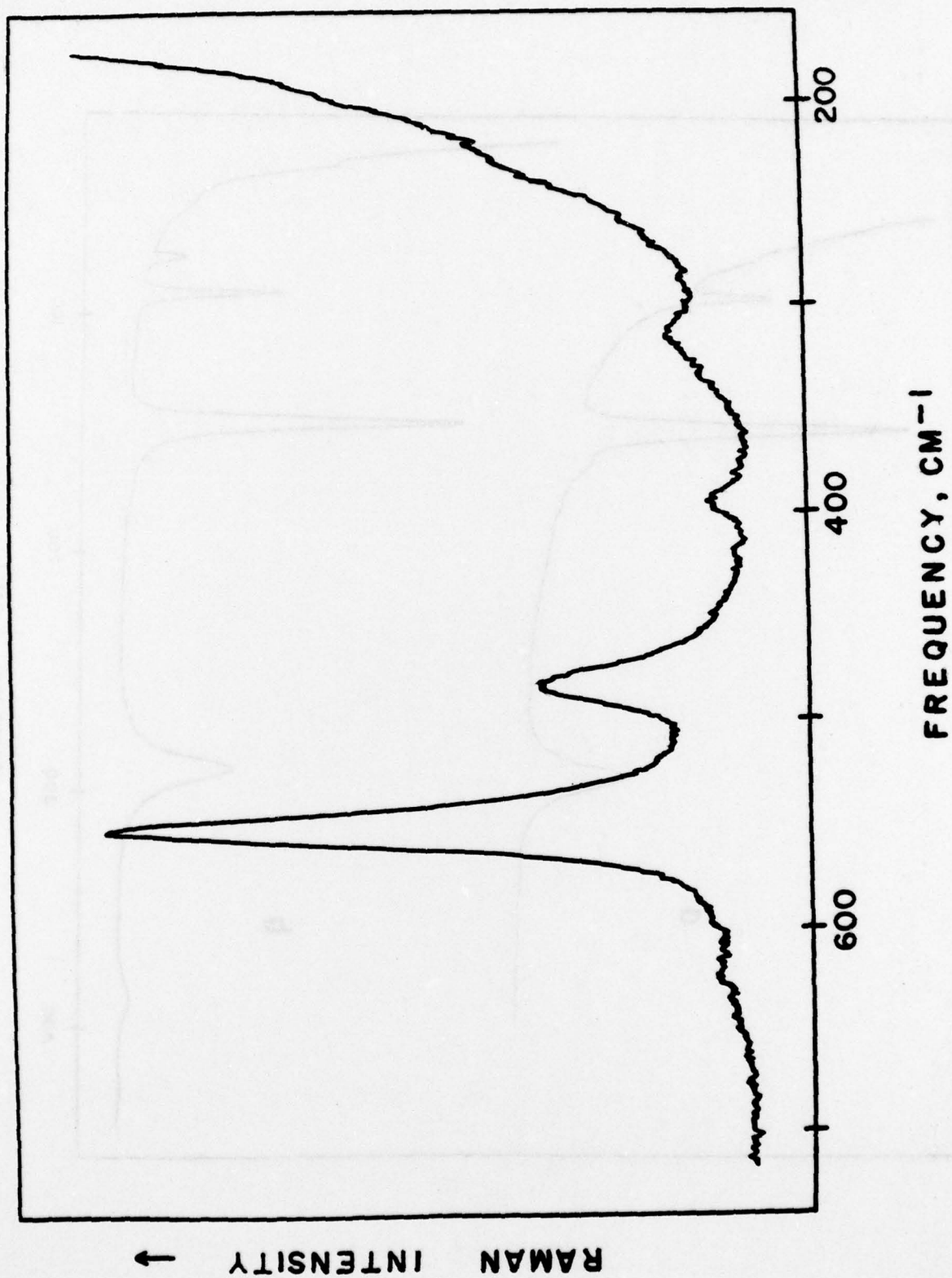


Figure 14. Raman spectra of (a) orthorhombic PbO film formed by 18 hour exposure of lead at +0.59v (vs. SHE) in 0.1M chloride, pH 7 solution, (b) orthorhombic PbO powder

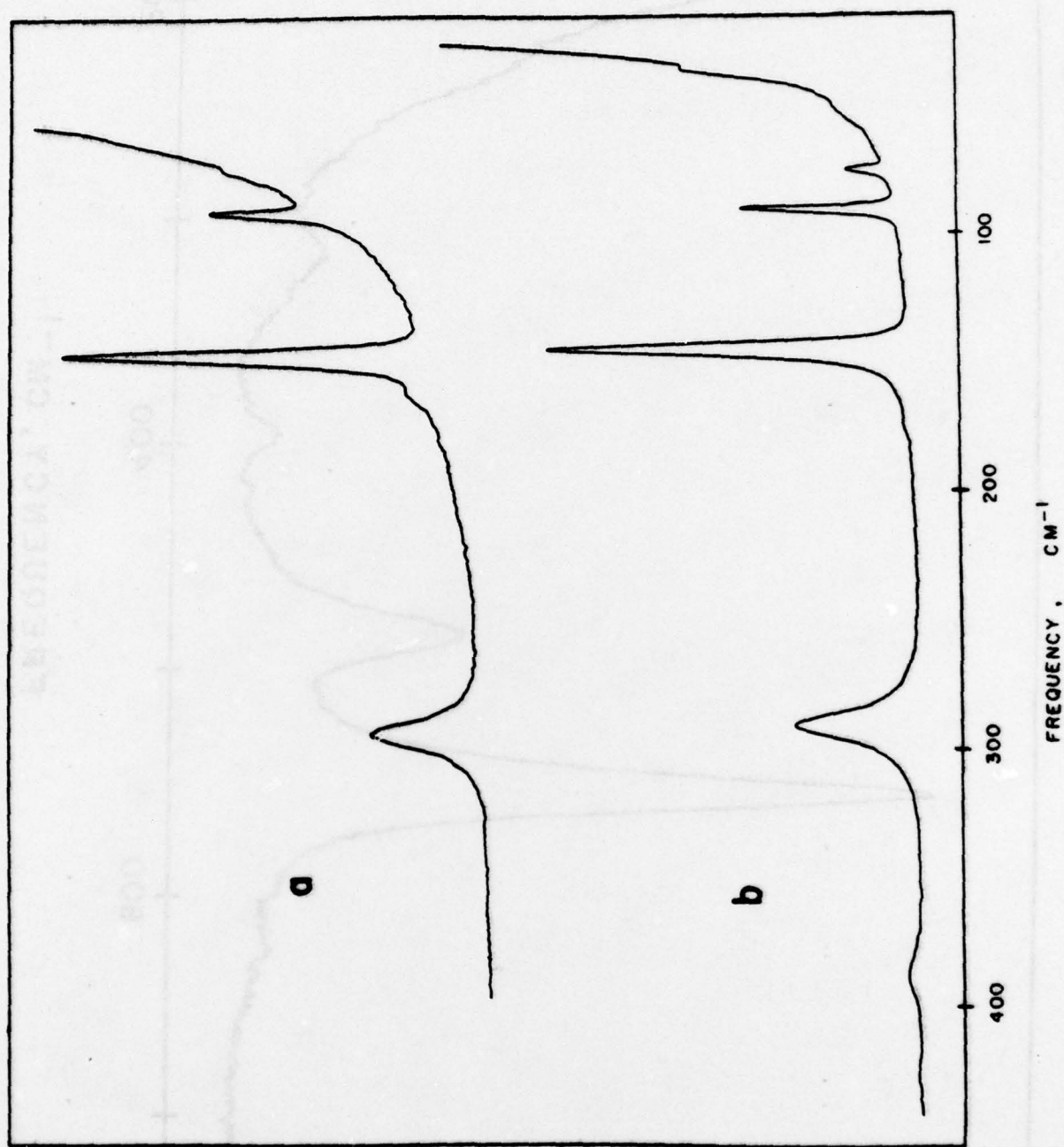


Figure 15. Potential-pH (Pourbaix) diagram calculated for lead in aqueous solution assuming no anion effects. Thermodynamic data used for the calculations were supplied by the Thermodynamics Data Center, National Bureau of Standards. Experimental points represented by o indicate tetragonal PbO was found, w indicates α -PbO₂.

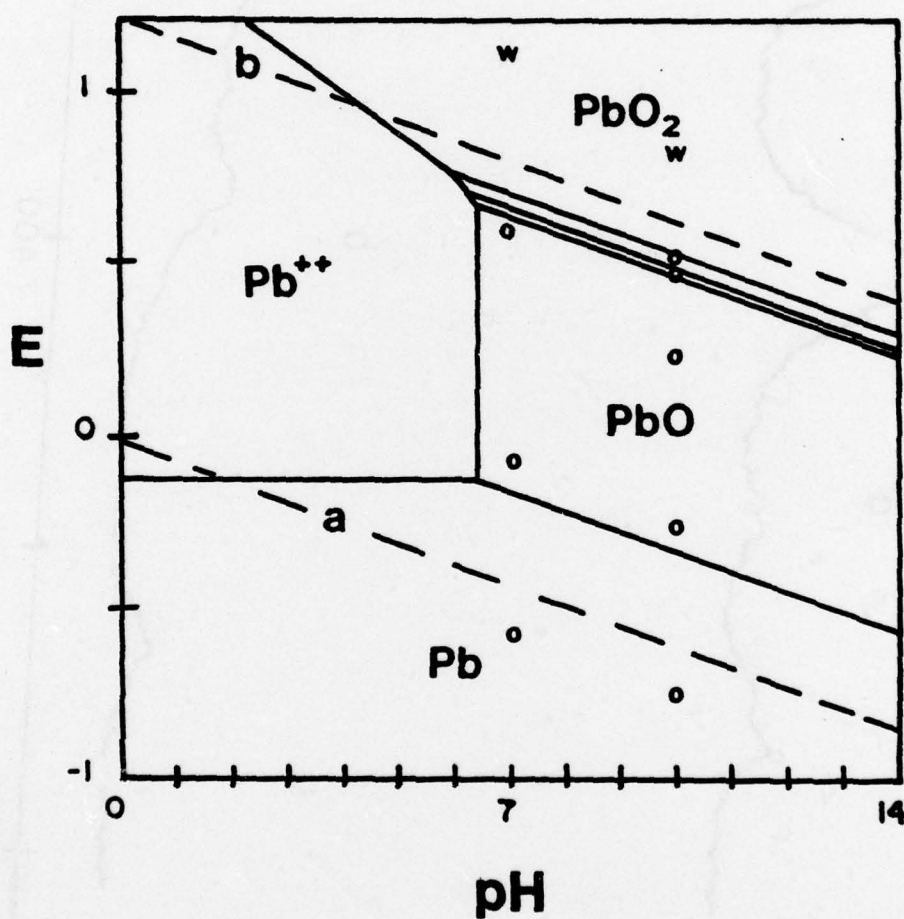


Figure 16. Raman spectra of (a) α -FeOOH, and (b) γ -FeOOH

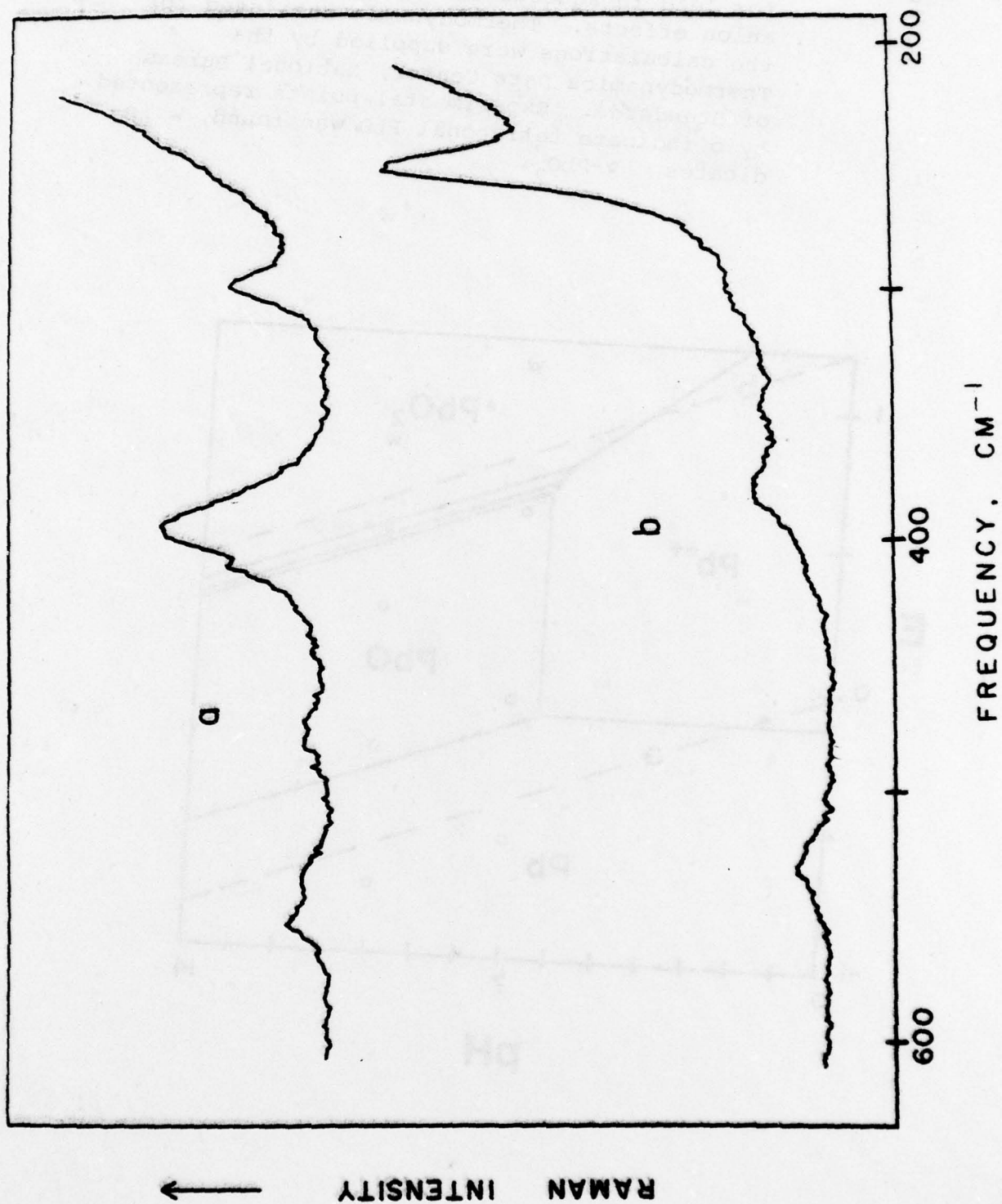


Figure 17. Raman spectra of (a) α -Fe₂O₃, and (b) γ -Fe₂O₃

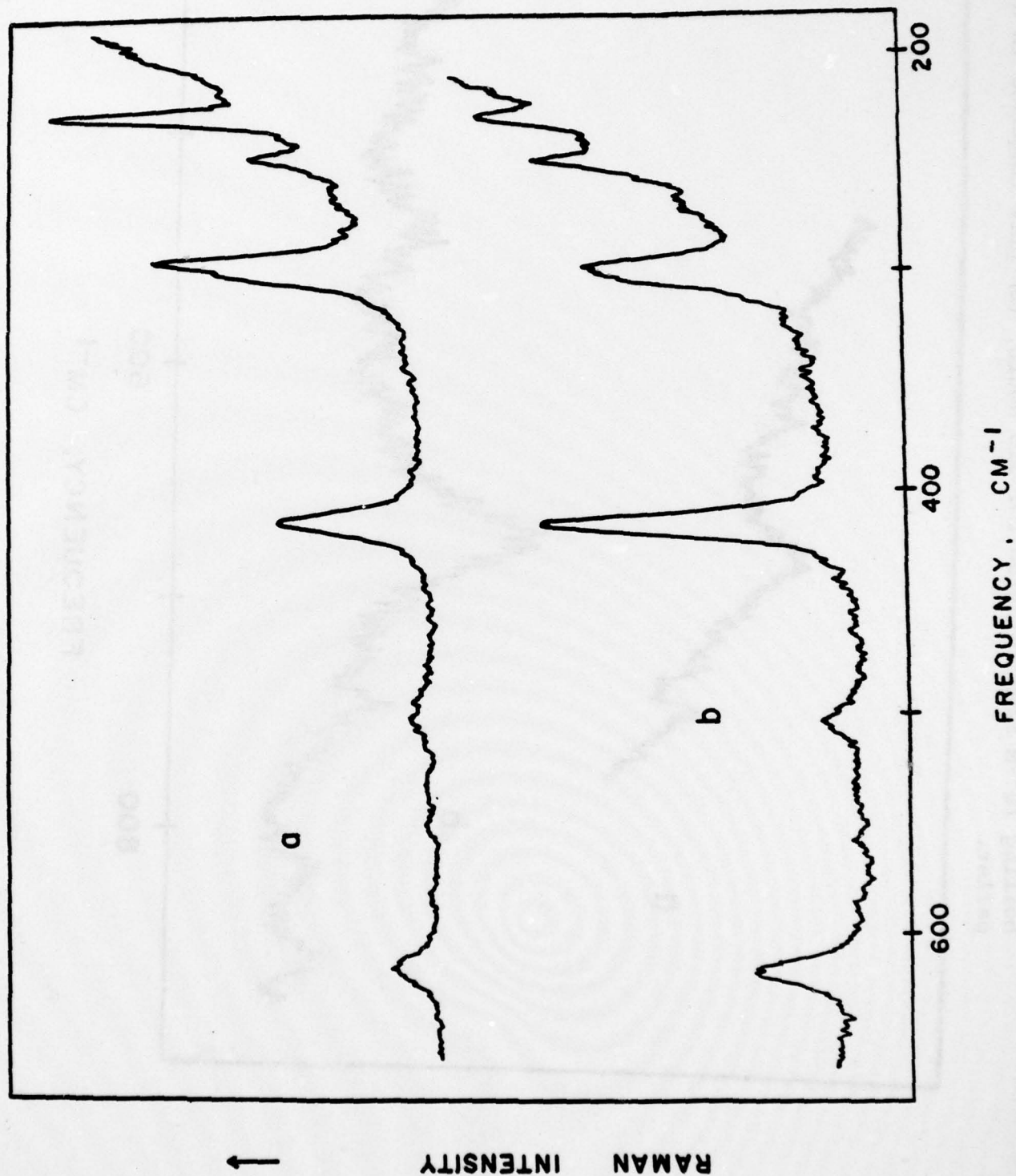


Figure 18. Raman spectra of Fe_3O_4 : (a) surface of Armco iron which had been in boiling 10-5M NaOH solution for 115 hours, (b) pure compound in a KBr pellet.

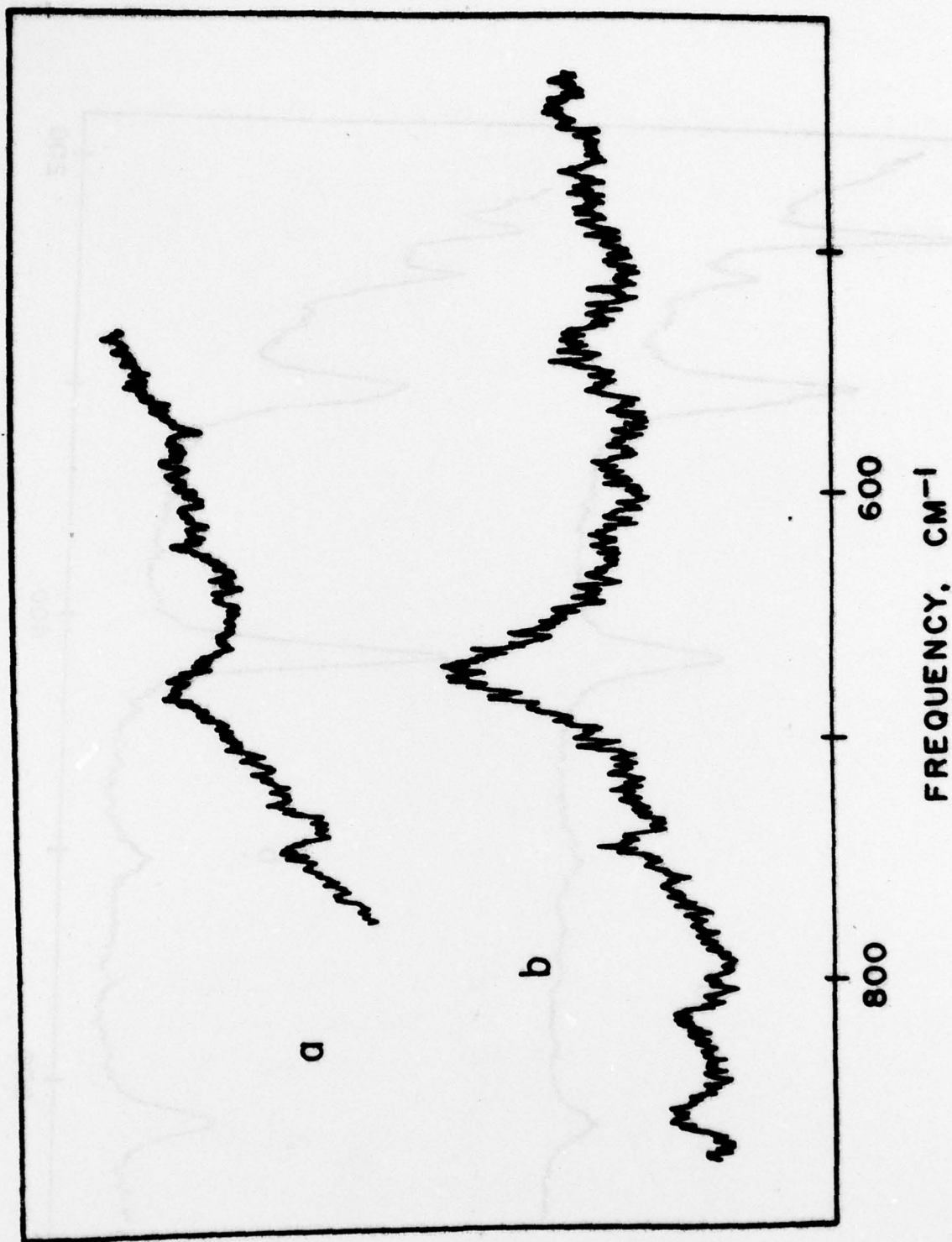
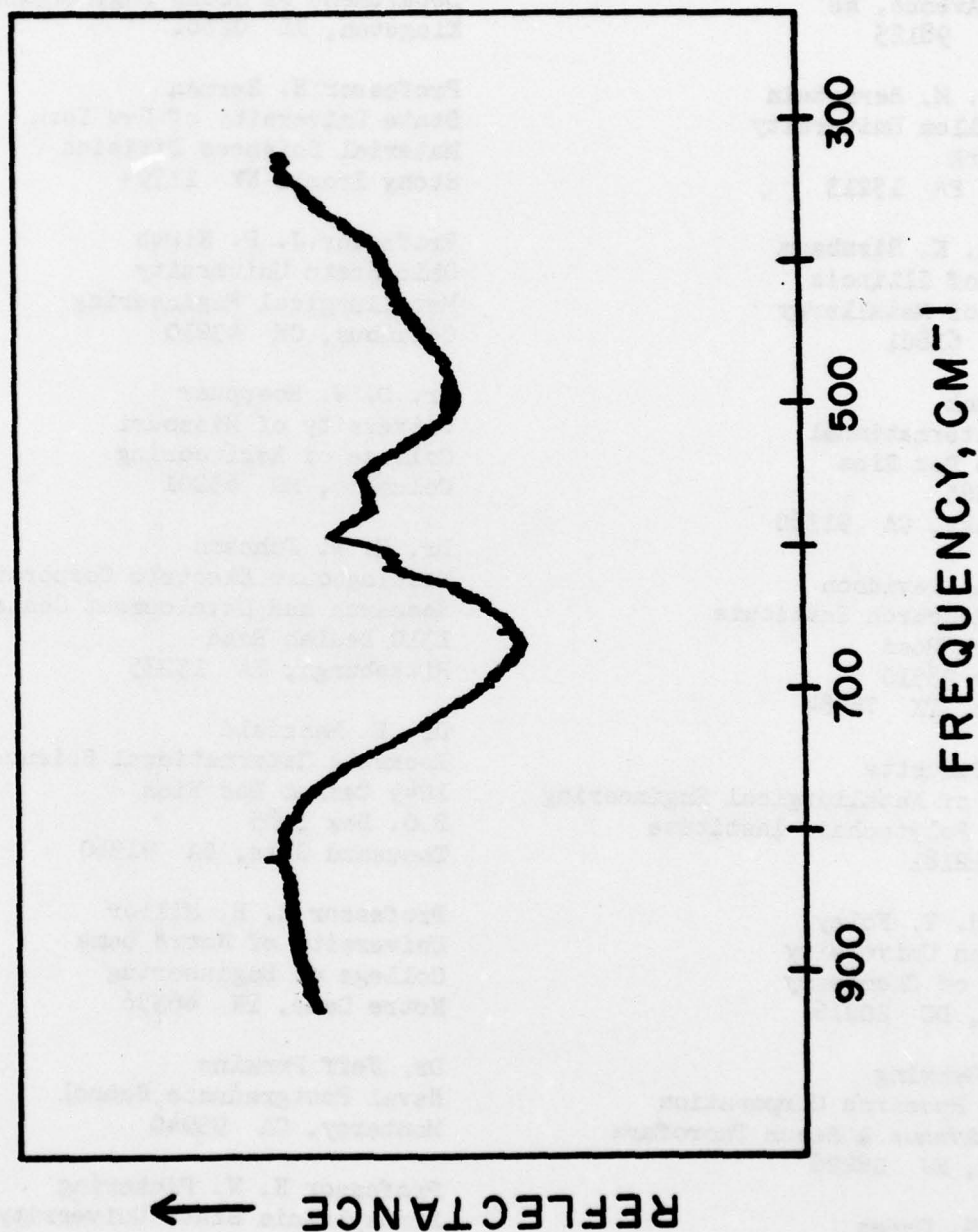


Figure 19. Infrared spectrum of mixed oxide coating formed on Armco iron by air oxidation at 220°C.



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