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Analysis of Bearing Deposits

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This technical report has been reviewed and is approved for publication. Publication of this report does not constitute Air Force approval of the report's findings or conclusions. It is published only for the exchange and stimulation of ideas.

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Deposits found in life-tested bearing assemblies were analyzed by electron spectroscopy for chemical analysis, by ion microprobe mass spectrometry, by infrared spectroscopy and by solvent analyses. These analyses showed the deposits to be one or more of the following compounds: ferric oxide, naphthenic acid, lead naphthenate, Apiezon C and alkali metal oxides or sulfates. On one of the bearings, the residue on the balls was of a different composition than that found in the raceways.

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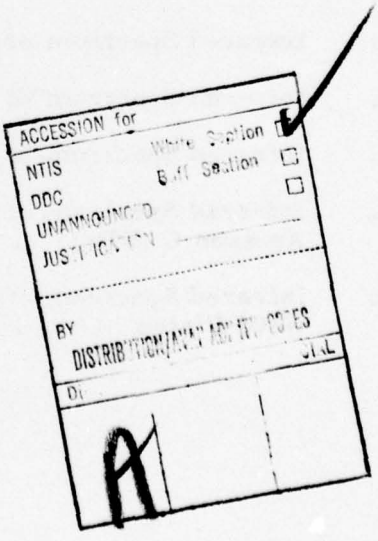

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I. INTRODUCTION

Three bearings received from the Aerospace Program Office (henceforth designated as 5A, 6B, and 008) were subjected to chemical analysis to determine the composition of surface deposits that resulted from life tests. These bearing assemblies had been lubricated with Apiezon C/lead naphthenate and tested under various preloads (60-1500 lb), rpm (50-105), and temperatures (30-90°C) until torque failure.

Bearing assembly 6B had heavy deposits of brown material of undetermined composition adhering to the balls and raceways (Figs. 1 and 2). The inside edges of the phenolic retainer slots also were noticeably darkened to the point of being black in some localized areas. Bearing assembly 5A was similar in appearance to bearing assembly 6B, but had noticeably less brown adherent. The third bearing assembly, 008, was relatively free of adherent and differed from the other two assemblies in that the balls were coated with small amounts of off-white particulate matter. In addition, small amounts of brown, black and white material were present in the 008 bearing raceways.

Bearing assemblies 5A and 6B were of a 4-in. bore and contained 21 balls of 9/16-in. diameter. Bearing assembly 008 had similar dimensions but had only 16 balls.

II. DISCUSSION

A. ANALYTICAL TECHNIQUES

Solvent analysis, infrared spectroscopy, electron spectroscopy for chemical analysis (ESCA) and ion microprobe mass analysis (IMMA) techniques were used to determine the chemical identity of the deposits on the various bearings.

Solvent analyses allow one to determine chemical identity by noting solubility characteristics.

Infrared analyses are useful for determining chemical bonding characteristics of materials (Ref. 1). This structural information is often sufficient in conjunction with other data to obtain chemical identity of an unknown material. Spectra of known compounds are generally taken also for the purpose of identifying unknown compounds that have very complex spectra.

ESCA, a relatively new surface analytical technique, allows the determination of elemental composition and element valency of materials (Ref. 2). X-rays are used to generate photoelectrons with kinetic energies characteristic of the elements in a material. Energy analysis of the electrons permits determination of the elements and their oxidation state. The ESCA technique is sensitive to the outermost surface of a sample; depths are limited to 0-50 Å. Thus, contamination of samples must be minimized during sample preparation and handling. Fingerprints and foreign debris such as dust and atmospheric hydrocarbons will generally mask the "true" composition of the surfaces, and will make spectral interpretation more difficult.

IMMA is particularly useful for elemental composition. Charged ions, usually O^- or O_2^+, are focused onto a spot ($\approx$1 μm dia.) or are rastered across a relatively large surface ($\approx$40 μm x 50 μm). Sample material is

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sputtered off in the form of secondary ions. These secondary ions are then analyzed in a mass spectrometer. Depth compositional profiles may also be obtained simply by sputtering away material in one place for a prolonged period of time and monitoring the mass of the secondary ions.

B. INSTRUMENTATION

A Perkin Elmer infrared grating spectrophotometer (Model 467), equipped with a KBr disk assembly holder and a sample holder for internal reflection spectroscopy, was used to obtain the infrared absorption spectra. Infrared absorption spectra were taken across the energy range 2.5 to 40 μm in the dual beam mode.

ESCA measurements were made on the ESCA-36 GCA/McPherson Electron Spectrometer. Specially constructed holders were used within the sample chamber to accommodate the balls. The x-ray excitation energy was set at 1253.6 eV (Mg K_α irradiation), and the x-ray power was set at 300 W. A turbomolecular pump reduced the pressure within the sample chamber to 10^{-6} - 10^{-7} Torr during analyses.

IMMA experiments were made on an Analytical Research Laboratories, Inc. spectrometer equipped with a Digital Equipment Corporation PDP11/05 computer. A 20 keV, 3-10 nA O_2^+ or O^- beam was used in this study. The sample chamber was pumped down to the 10^{-7} Torr range for analysis by a combination of sorption and ion pumps.

C. EXPERIMENTAL PROCEDURES AND RESULTS

1. Solvent Analysis

This analysis was limited to the balls of bearing 5A and 6B and to the 6B raceways.

Two balls from bearing 6B were soaked for several days and then given a one-hour ultrasonic agitation at room temperature, in either distilled water, ethanol, benzene, carbon tetrachloride or n-heptane. Each

solvent was tested on independent sets of balls. The brown adherent was neither soluble nor removable as a suspension in any of the organic solvents. The distilled water only partially removed the adherent.

A dilute solution of nitric acid appeared to remove the 6B adherent, but the balls and the solution turned black indicating chemical degradation of the ball surfaces.

A 10% solution of sodium hydroxide proved to be the reagent that would selectively remove all the brown adherent coating without apparent damage to either the 5A or 6B balls or to the adherent (Fig. 3).

The adherent on a single ball could be quantitatively removed as a fine particulate suspension in one hour by ultrasonic agitation in 5 cc of the 10% NaOH solution. After the ball was removed (now shiny) the solution was centrifuged and the precipitate washed in distilled water to remove all alkali. The material was then dried by washing in absolute ethanol and heating in an oven (80°C) overnight. This dried material was then subjected to infrared analysis (see Subsection C-2).

The total material removed from two balls of bearing 6B by the NaOH treatment weighed 1.3 mg (dry). No attempt was made to determine the quantity of material on any other ball or raceway.

The material in the raceways of bearing 6B was quite different from that found on the balls as it appeared to be readily removable by n-heptane. Only a small area in the raceway was tested so the remainder of the adherent would be available for other analyses.

2. Infrared Analysis

a. Inorganic Standards

Table 1 lists the inorganic compounds analyzed as standards for Figs. 4 through 15. They were analyzed as received in the transmission or reflection mode. For measurement in the transmission mode, the compounds were

Table 1. Method, Inorganic Compounds, Source, Purity and Infrared Spectrum Number for Standards

Method	Compound	Source	Purity (%)	Infrared Figure
KBr disk	KBr	Baker	99.8	4
Salt flat	NaCl	Wilks	----	5
Internal Reflection	KRS-5 (TlBrI)	Wilks	----	6
KBr disk	PbO_2 (black)	Baker	96.2	7
KBr disk	PbO (yellow)	Baker	99.0	8
KBr disk	Pb_3O_4 (red)	Baker	98.0	9
KBr disk	$PbCO_3$	Matheson	----	10
KBr disk	$2PbCO_3 \cdot Pb(OH)_2$	BDH	----	11
Internal Reflection	Pb metal foil	Alpha Ventron	99.9995	12
Internal Reflection	$PbCl_2$	prepared in-house*	Unknown	13
Internal Reflection	$PbSO_4$	prepared in-house*	Unknown	14
KBr disk	Fe_2O_3	ROC/RIC	99.5	15

*Precipitate from the reaction of lead naphthenate with the corresponding acid (i.e., HCl or H_2SO_4)

intimately mixed with potassium bromide (infrared transparent) and pulverized in a mortar and pestle. A potassium bromide disk containing the inorganic compound was then formed for infrared measurements by pressing in a screw press. Alternatively, the compounds were simply placed against a KRS-5 plate for internal reflection infrared measurements (Ref. 3).

b. Organic Standards

An unlubricated phenolic retainer (grade LBB) obtained from Synthane Taylor Incorporated was powdered by filing, and the filings were incorporated in a KBr pellet. The exact composition and cure was not known. However, the infrared absorption of this material had characteristic bands at ~3360, 2910, 1625, 1440, 1060, 620 and 350 cm^{-1} (Fig. 16).

Only bands at 2930, 2860, 1460 and 1380 cm^{-1} characteristic of the Apiezon C were found in the infrared spectrum of a 2% lead naphthenate solution in Apiezon C (Fig. 17). This spectrum was obtained by spreading a thin layer of the lubricant (prepared at Aerospace) across the surface of a KBr disk.

A good infrared spectrum of the lubricant additive, lead naphthenate was determined by forming a thin layer of the material between two sodium chloride flats and measuring the transmission spectrum. Characteristic bands were found at 2970-2860, 1530, 1460 and 1400 cm^{-1} (Fig. 18). When using KBr or KRS-5 (thallium bromoiodide) as a support, additional bands at 710 and 430 cm^{-1} were noted. In addition, the peaks at 1530, 1460 and 1400 cm^{-1} were considerably broadened (Figs. 19 through 21).

c. Material from Bearing Assemblies

(1) Balls

Potassium bromide was added to the centrifuge tubes containing the dried adherent from balls 5A and 6B (see Subsection C-1), and intimately mixed within the tubes. The adherent could then be quantitatively removed for KBr pellet formation and for subsequent infrared analysis. Spectra for

the 5A and 6B adherents are shown in Figs. 22 and 23. The main features in each spectrum were similar; major peaks were observed at 3440, 555, 470 and 330 cm^{-1}. An infrared spectrum of the adherent on the balls of bearing 008 was not obtained due to the extremely small amount of material present.

(2) Raceways

In contrast to the difficulties encountered in removing material from the balls, the deposits in the raceways could be removed by simply abrading them with KBr. (In the case of the balls, the abrasion techniques proved to be ineffective in removing the adherent.) Infrared absorptions for material in the outer race of bearing 6B were found to be at 3440, 2940, 2860, 1675, 1460, 1380, 575, 480 and 350 cm^{-1} (Fig. 24). Material from the inner race of bearing 008 had infrared adsorptions at 2930, 2860, 1730, 1540, 1460, 1405, 1160 and 1030 cm^{-1}. Note that for the 008 material, no absorption occurred in the 600-200 cm^{-1} region (Fig. 25).

The 005 raceways, the 6B inner race and the 008 outer race were not analyzed.

(3) Retainer Slots

Only the 6B bearing was analyzed. The KBr abrasion technique was used in this instance also to effect deposit removal. The infrared spectrum of this deposit is shown in Fig. 26. Band positions and band shapes were found to be almost the same as those found for the 6B outer race material (Fig. 24 versus Fig. 26).

d. Reaction Studies

(1) Extraction of Lubricant

The lubricant remaining in the phenolic 6B retainer was extracted by overnight soaking in n-heptane. The solvent was stripped and an infrared spectrum of the remaining material was taken. In addition to those bands found for unused lubricant (Fig. 17) a band at 1715 cm^{-1} appeared (Fig. 27).

(2) Formation of Naphthenic Acid

A synthetic sample of naphthenic acid was prepared by reacting lead naphthenate with one of the strong acids, HCl or H_2SO_4. The most significant feature of the infrared spectrum of naphthenic acid is the band at 1705 cm^{-1}. The rest of the spectrum is similar to that of lead naphthenate (Figs. 28 and 29 versus Fig. 18).

(3) Reaction of Sodium Hydroxide with Phenolic

Powdered phenolic (see Subsection C-2b) exposed to the same sodium hydroxide processing treatment as given the balls gave an infrared spectrum virtually identical to the original phenolic (Fig. 30 versus Fig. 16).

(4) Thermal Degradation of Phenolic

Powdered phenolic (see Subsection C-2b) was arbitrarily heated to 200°C for one hour to test if these conditions were sufficient to cause chemical changes. The heated phenolic differed from the unheated phenolic only in the appearance of a new band at 1725 cm^{-1} (Fig. 31 versus Fig. 16).

(5) Thermal Stability of Lubricant

The lubricant was heated to 200°C for one hour. Although these heating conditions were somewhat arbitrary, it was felt that any chemical reaction (as opposed to thermal degradation, i.e., pyrolysis) would be observable by inspection of the infrared spectra. The infrared spectrum of the heated lubricant was the same as that recorded for the unheated lubricant (Fig. 32 versus Fig. 17).

(6) Reaction of the Phenolic with Lubricant

Using the same reasoning as in (5), powdered phenolic and lubricant were heated together at 200°C for one hour, to note possible chemical reactions. The infrared spectrum of this heated mixture was merely the composite of those spectra found from heating the individual components (Fig. 33 versus Fig. 32 plus Fig. 31).

(7) Reaction of Phenolic Monomers with Lead Naphthenate

Heated mixtures of phenol and phenolic or formaldehyde and phenolic had infrared spectra that were merely composites of the individual components (Figs. 34 through 36). The heating conditions (10 minutes at 100°C) were as high as the solvents would allow. Higher temperatures or longer reaction times resulted in significant evaporative losses.

3. ESCA Analysis

Representative balls from each of the bearing assemblies were analyzed for surface composition by mounting them directly in the ESCA sample chamber. No pretreatment was given to the balls; they were analyzed as received. The ESCA spectra revealed only the presence of carbon, oxygen and sodium. No other elements were detected. All the balls (5A, 6B and 008) had similar ESCA spectra.

The black/brown residue from the outer race of bearing 6B was removed with a spatula and placed on an ESCA sample support made of etched aluminum (type 1100). A similar procedure was used for the outer raceway deposits of bearing 008. In both instances, ESCA spectra confirmed the presence of lead, carbon, sodium and oxygen. Surprisingly, the lead binding energies, even after correcting for sample charging effects, were 0.75 eV higher than that previously measured for any known lead compound including lead naphthenate (Fig. 37). A very small amount of sulfur, present as sulfate (Fig. 38) and magnesium (oxidation state not determined), were also found by analysis of the ESCA spectra of the 008 raceway residue.

4. IMMA Analysis

All the IMMA analyses were made on a sample area of 40 μm x 50 μm. A 6B ball was analyzed in two areas: in an area appearing as a "clean" band around the ball and in an area of brown deposits (Figs. 39 and 40). The most significant difference in the two spectra was in the relative abundance of magnesium and calcium. The deposits were much richer in these elements. Note that several peaks have the same designation; the multiplicity of peaks is due to isotope effects on the mass of a given specie.

The ion mass spectrum of the 6B raceway deposits (Fig. 41) was similar but not identical to that found for the ball deposits.

An analysis of the deposits on the ball surfaces of bearing 5A and 008 revealed little difference in the elements detected (Figs. 42 and 43). However, the amount of iron and chromium detected on the 008 ball surface relative to calcium was considerably less than for the 5A ball surface.

D. DISCUSSION OF RESULTS

The extremely low amounts of material found in the various bearings precluded a complete analysis of material in each raceway and ball surface. Thus, some analysis of the individual bearings was confined to either deposits on the balls or to deposits in the raceways.

A priori, it was concluded that the brown material found on the 6B ball surfaces was of phenolic origin. This erroneous conclusion was based on the color of the deposit and on the solubility characteristics of the material. However, it became apparent from IMMA and infrared spectra that the 6B ball adherent was mainly iron oxide, Fe_2O_3 (a brownish-red material). Note that the broad band at 3440 cm^{-1} and the bands and relative intensities at 560, 475 and 330 cm^{-1} for the 6B ball adherents match fairly well those found for Fe_2O_3 (Fig. 15 versus Fig. 23). The band positions for the 6B adherents in the 600-250 cm^{-1} region are shifted to slightly lower energies relative to those of Fe_2O_3 by about 20 cm^{-1}. It is felt that this shift is minor. The iron oxide on the ball surface may have a different crystal structure than the Fe_2O_3 standard, and this difference could readily account for the observed shift. Furthermore, this region of the infrared is quite sensitive to small changes in material composition. The lack of any bands in the 700-200 cm^{-1} region for heated Apiezon C/lead naphthenate (Fig. 32) and the lack of matching bands for Pb metal (from possible reduction of lead naphthenate), PbO, PbO_2, Pb_3O_4 (oxidized lead naphthenate), Apiezon C, $PbCO_3$, $PbCO_3 \cdot Pb(OH)_2$, i.e., precipitated lead carbonate, and lead naphthenate indicate that these

compounds are minor constituents, if at all present. Although the NaOH removal technique may have lost some lead naphthenate during processing, this seems unlikely as direct analysis of the ball surfaces by ESCA revealed no lead while the IMMA gave only a small lead signal [Fe^{56} and Pb^{208} IMMA signal sensitivities are roughly equal (Ref. 4)].

The coating thickness of the Fe_2O_3 from two balls of bearing 6B was calculated from the total weight of Fe_2O_3 removed, a Fe_2O_3 density of 5.24 gm/cm^3 and the balls' surface area. The average thickness was found to be $\sim 0.2\ \mu m$.

Infrared bands at 2940, 2860, 1460 and 1380 cm^{-1} found for material from the 6B retainer slots and from the outer raceway match exactly those of the 2% lead naphthenate in Apiezon C lubricant (Fig. 17). The absorption at 3440 cm^{-1} and in the 700-200 cm^{-1} region are identical to those found for Fe_2O_3 (i.e., 3440, 580, 485, 350 cm^{-1}) in position and in relative intensities. The broad band at 1675 cm^{-1} was not positively identified, although the onset of the peak at 1700 cm^{-1} is consistent with naphthenic acid.

Lubricant removed from the 6B phenolic retainer by n-heptane reinforces the above infrared assignment at 1675 cm^{-1} as being due to naphthenic acid. In addition to infrared bands characteristic of Apiezon C (2930, 2860, 1460 and 1380 cm^{-1}), a fairly strong band at 1715 cm^{-1} was observed for the removed lubricant. The band width and band position of naphthenic acid is in good agreement with this band (Fig. 27 versus Fig. 28). Note, however, that the characteristic bands in the 700-200 cm^{-1} region, attributable to Fe_2O_3, were absent. This is expected as Fe_2O_3 is not soluble in the extracting solvent, n-heptane.

The brown material adhering to the 5A ball surfaces was also analyzed as Fe_2O_3 (Fig. 22 versus Fig. 15). A downfield shift in the infrared spectrum of about 20 cm^{-1} relative to the Fe_2O_3 standard, similar to that found for the 6B bearing, was again observed.

Residue from the 008 bearing inner race had infrared bands at 2930, 2860, 1730, 1540, 1460, 1405, 1160 and 1030 cm^{-1} which were consistent with a mixture of lead naphthenate (bands at 2970-2860, 1540, 1460 and 1405 cm^{-1}) and possibly naphthenic acid (band at 1705 cm^{-1}) (Figs. 18, 28 versus Fig. 25). The naphthenic acid band is situated upfield from the absorption peak of the naphthenic acid standard by 25 cm^{-1}. A peak shift of this magnitude for the same compound is expected in view of the fact that the support used in obtaining the spectra was different and that the naphthenic acid was not pure. The weak bands at 1160 and 1030 cm^{-1} were not identified.

ESCA analysis of the various balls and raceways were consistent with the infrared results. The presence of carbon, oxygen and sodium on the ball surfaces was expected. Carbon is always detected on surfaces by ESCA. The oxygen signal was no doubt from the oxygen in the Fe_2O_3 and the sodium signal was probably due to sodium in fingerprints left on the ball surfaces from previous handling. The inability to detect iron was not surprising since this element has low sensitivity of detection. Furthermore, the relatively high noise level in the ESCA spectra for these samples would have obscured any low signal.

The weak sulfate spectra obtained from bearing 008 provides a clue as to how naphthenic acid formed. Chemical reaction between lead naphthenate and a proton source such as H_2SO_4 (residual catalyst left in the phenolic from its manufacture) to form naphthenic acid and $PbSO_4$ may have occurred.

Some caution must be exercised in interpreting the IMMA results. Due to charging effects, the 5A and 008 ball surfaces were probed with an $^{18}O^-$ beam instead of by a $^{18}O_2^+$ beam as was the case for the 6B system. Spectral comparisons of the 6B surfaces with the 5A or 008 surfaces are thus not possible. Different element sensitivities exist for the two modes of excitation. In addition, since the sputtering yields also depend on the matrix of the sample, spectral comparisons of two samples using the same excitation beam are

semi-quantitative at best. Only with samples having similar matrices (i.e., a pure compound containing various trace elements) can the technique be quantitative.

The most significant findings using the IMMA technique were: (a) lead was found on the 6B and 5A ball deposits and was below the detection limit in the case of the 008 ball deposits, and (2) the 6B ball deposits were rich in magnesium and calcium relative to the clean area of the balls, and the iron and chromium content of the 008 ball deposits were low relative to the 5A ball deposits.

III. CONCLUSIONS

Two important findings in regard to the analyses of the 6B bearing system were made. First, material adhering to the balls was different in composition from that found in the raceways, and second, naphthenic acid was detected in the lubricant remaining in the phenolic retainer and on the raceways.

Iron oxide as Fe_2O_3 appeared to be the major component on the 6B ball surfaces, whereas on the raceways iron oxide was mixed with about equal parts of naphthenic acid and lubricant. The differences in solubility characteristics, in the ESCA spectra, and in the infrared spectra between the ball adherent and the raceway residue support the first significant finding. The second finding was based primarily on infrared analyses, although some additional supporting evidence was found in the ESCA spectra of residue from the 008 raceway.

Lead was found on the 6B ball surfaces by IMMA and on the 6B raceways by ESCA. The valency of lead found in the latter case appeared to be in the +2 oxidation state.

The detection of various alkali and alkaline earth metals (e.g., sodium, potassium, calcium, magnesium, etc.) may also indicate that oxides (e.g., CaO) or ferrites (e.g., $CaO \cdot Fe_2O_3$) were present on the 6B ball surfaces.

Since analysis of the 5A bearing assembly was restricted to only the balls, our conclusions are necessarily limited. However, it can be stated that the material on these ball surfaces was similar in composition to that found on the 6B ball surfaces.

In contrast to the 5A and 6B systems, however, the residues in the 008 bearing assembly were quite different in composition. Visually, the characteristic red-brown color of Fe_2O_3 was absent on the ball surfaces. One might suspect that the white particulate matter on these ball surfaces was an oxide or a sulfate salt of lead or of another metal. Unfortunately, the extreme low quantities of material allowed only elemental identification. Support for

these suggested salts can be found from the color of the deposits and from the IMMA and ESCA results. Again, in contrast to the other bearing assemblies, the residue on 008 raceways was composed of near equal mixtures of naphthenic acid and lead naphthenate. Both Fe_2O_3 and Apiezon C are conspicuously absent. Loss of Apiezon C from the raceway and selective concentration of the additive appear to have taken place. One possible mechanism by which Apiezon C could be lost from the raceway would be by the process of absorption by the retainer. Such a mechanism has been observed for incompletely filled porous nylon reservoirs in contact with lubricant films on smooth surfaces (Ref. 5). Evaporation of the Apiezon C from the bearing assembly could also account for some loss. However, why Apiezon C loss occurred only in the 008 bearing cannot be answered.

The exact mechanism by which Fe_2O_3 forms on the ball surfaces of the 5A and 6B bearings is beyond the scope of the present report. However, one might note that iron oxides can form on lubricated iron surfaces under an imposed load (Ref. 6). It was suggested that oxygen within the oil (dissolved oxygen or that from the oxygen-containing additives, such as lead naphthenate) reacted with the iron to form the iron oxide. Presumably, this reaction was catalyzed by the generation of localized areas of hot metallic iron formed during the wear process. Why the 5A and 6B ball surfaces became coated with firmly bound Fe_2O_3 and appeared relatively free of lubricant, whereas their raceways had lubricant containing naphthenic acid and suspended Fe_2O_3 and had no adhering Fe_2O_3, could be explained by the phenomenon known as dewetting. This dewetting process would imply that prior to or during the test, the surfaces of the balls and raceways had developed different chemical compositions. The consequence of the surface chemical change of the balls was to inhibit wetting by the lubricant thereby giving rise to the noted absence of oil coverage. Dewetting apparently was not induced on the raceways.

In summary then, we have shown that (1) the additive/lubricant ratio does not necessarily remain constant during bearing operation, (2) lead naphthenate can degrade to naphthenic acid during bearing operation, and (3) bearing surfaces can oxidize during bearing operation.

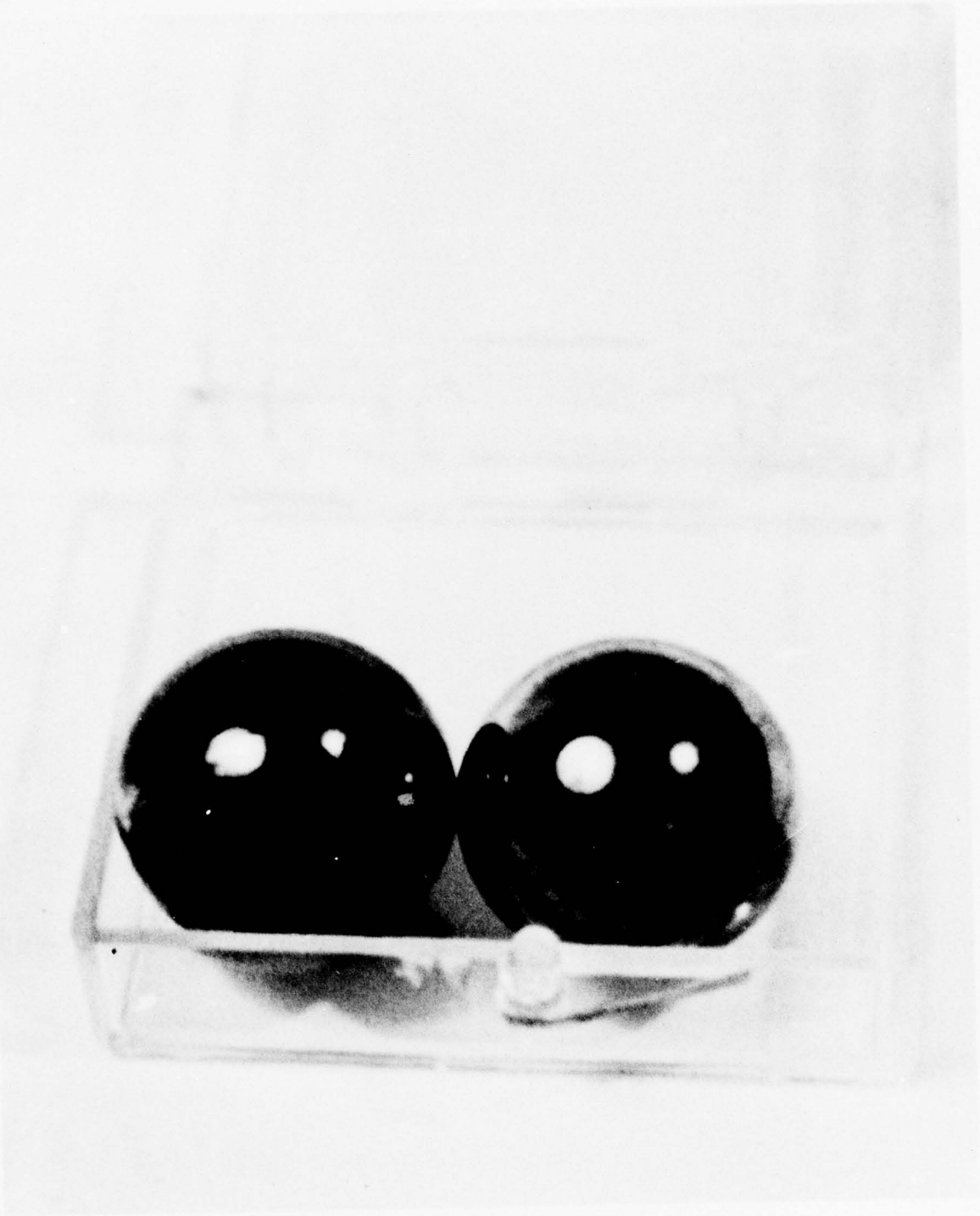

Fig. 1 - Balls from bearing 6B: left ball, as received; right ball, after deposit removal by NaOH

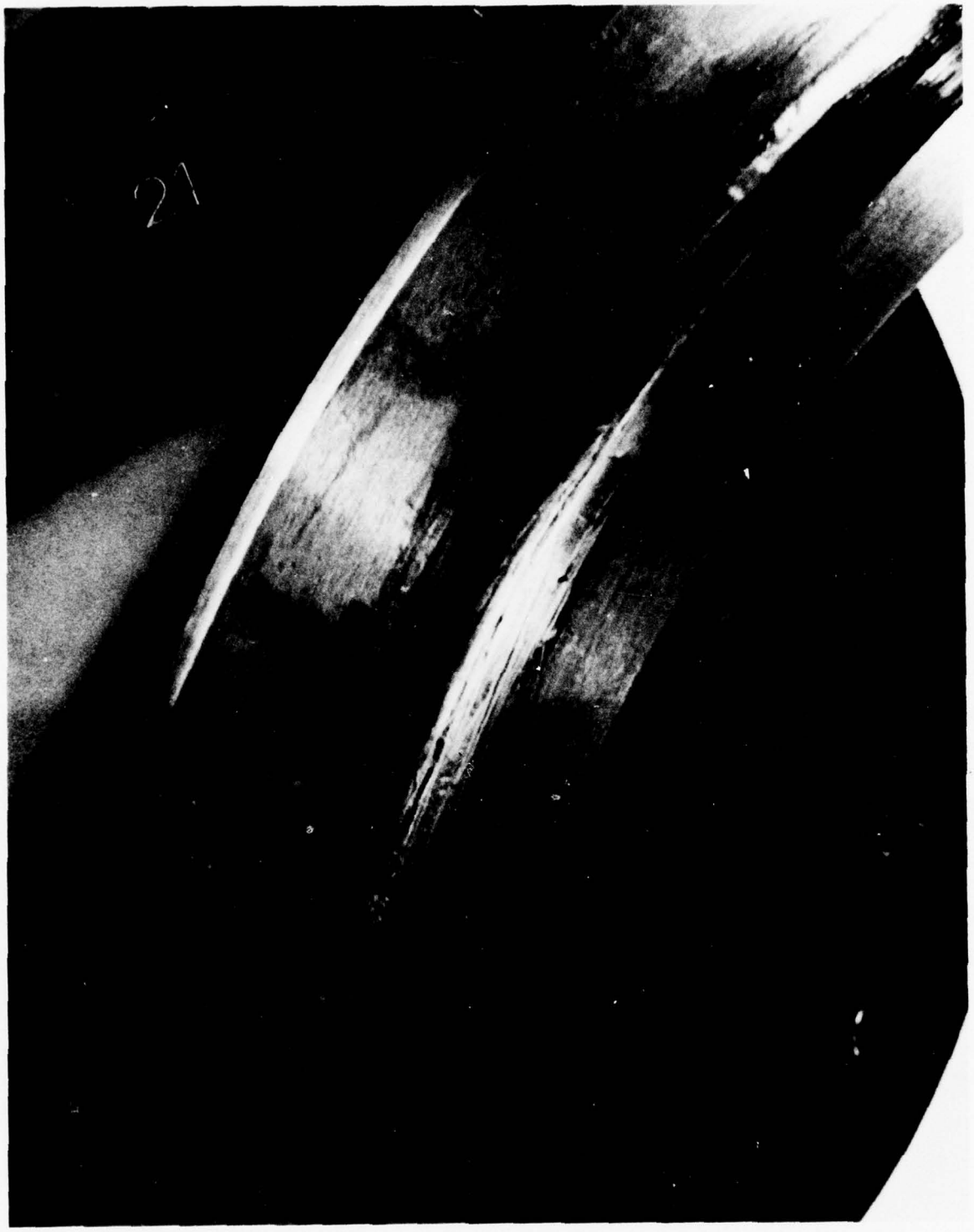

Fig. 2 - Outer raceway of bearing 6B showing brown deposits

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Fig. 3 - Removal of ball deposits by NaOH solution

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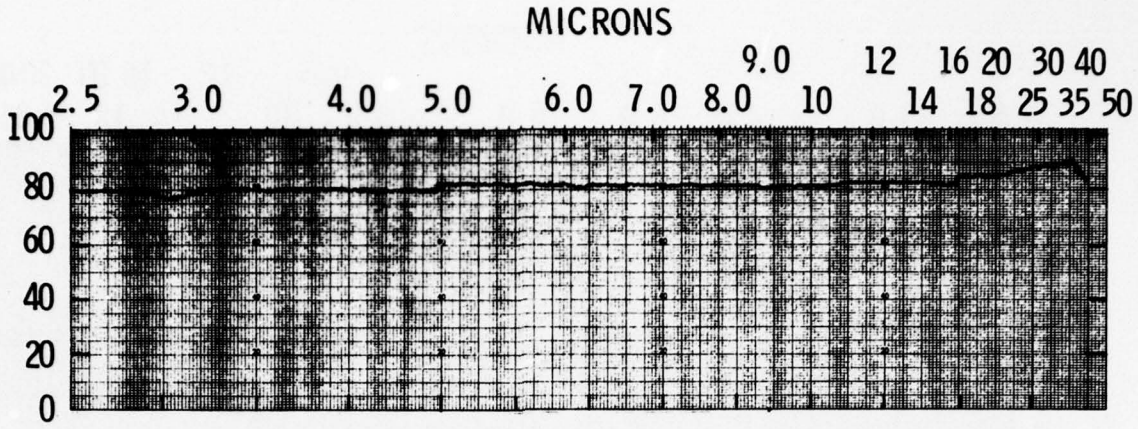

Fig. 4. Infrared Spectrum of KBr

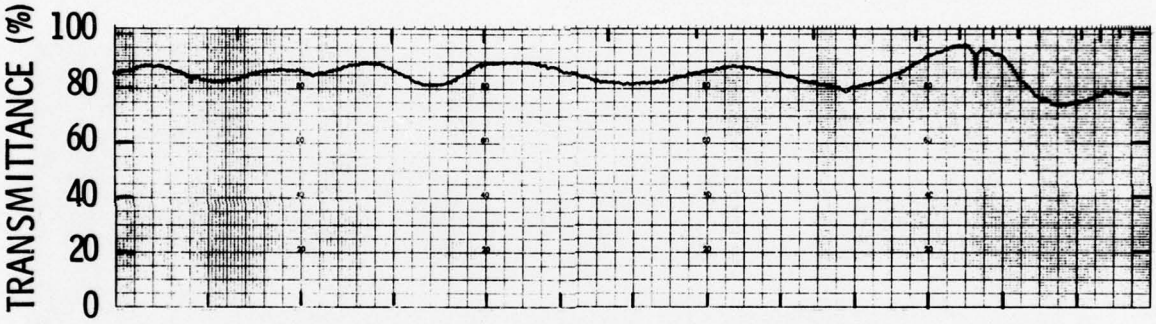

Fig. 5. Infrared Spectrum of NaCl

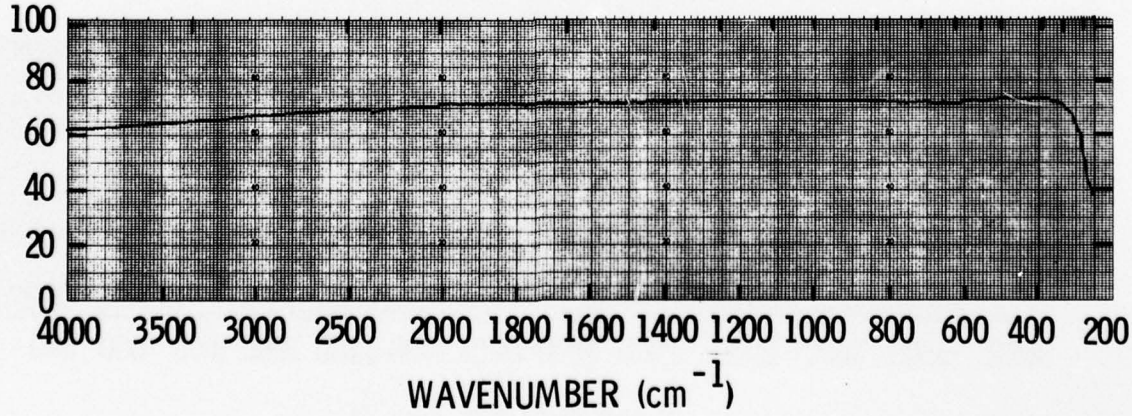

Fig. 6. Infrared Spectrum of Blank KRS-5 Plates

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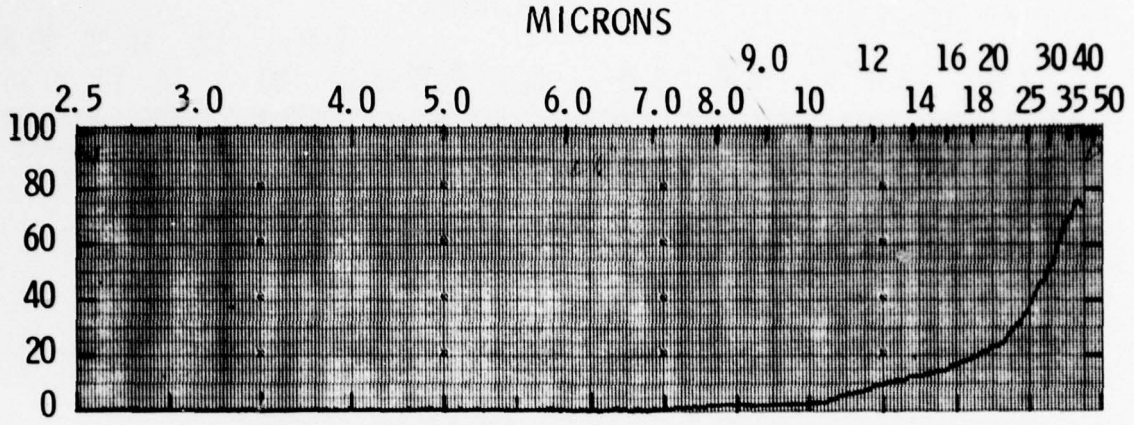

Fig. 7. Infrared Spectrum of PbO_2 (black powder)

Fig. 8. Infrared Spectrum of PbO (yellow powder)

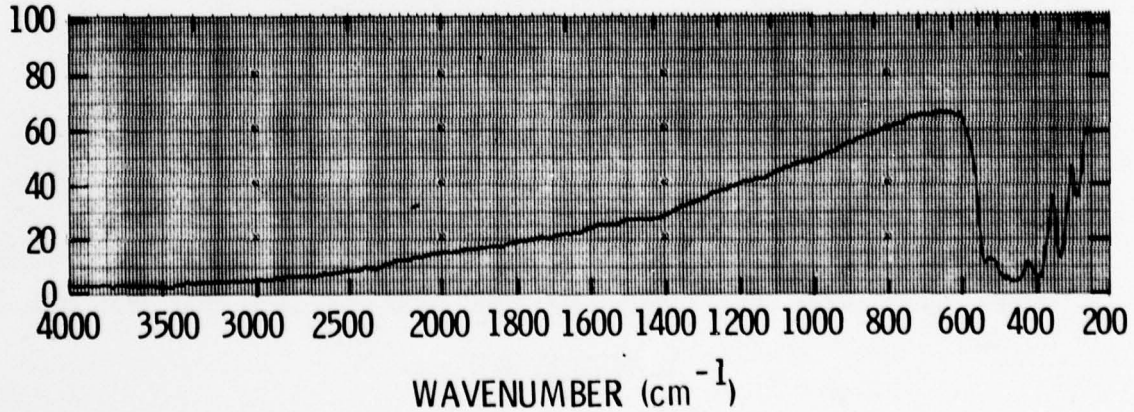

Fig. 9. Infrared Spectrum of Pb_3O_4 (red powder)

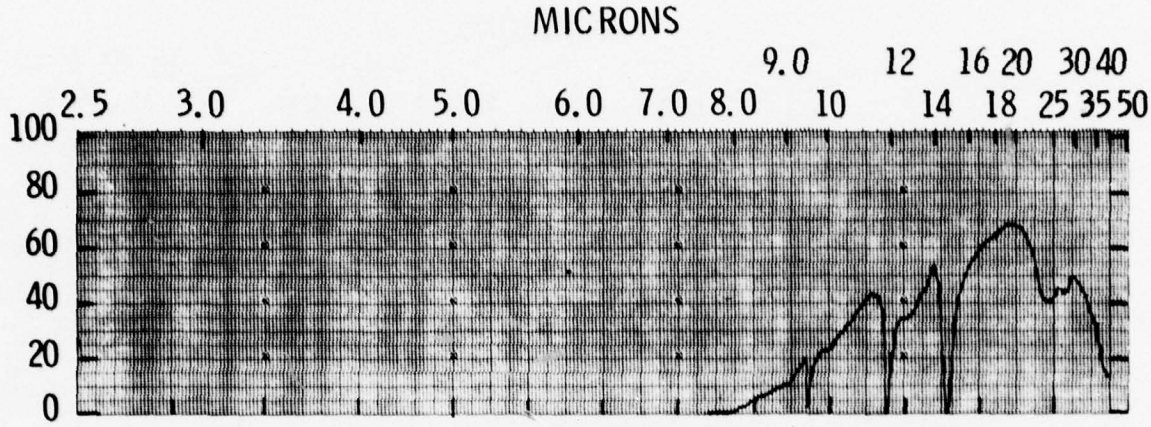

Fig. 10. Infrared Spectrum of $PbCO_3$ in 1200-250 cm^{-1} Region

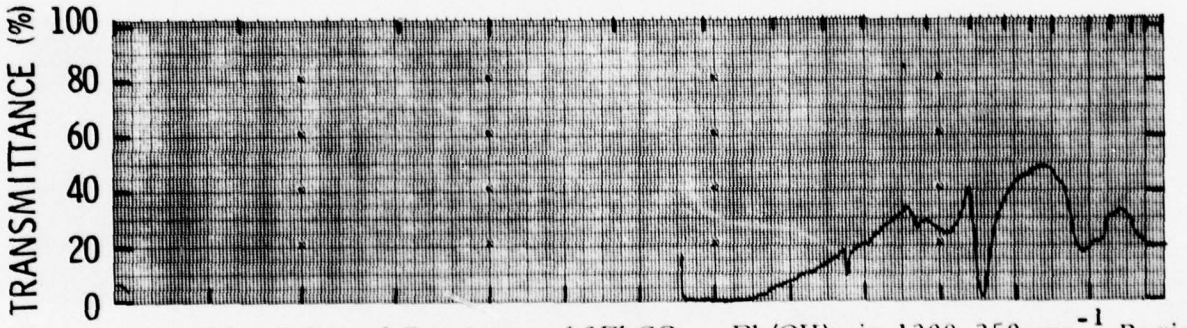

Fig. 11. Infrared Spectrum of $2PbCO_3 \cdot Pb(OH)_2$ in 1200-250 cm^{-1} Region

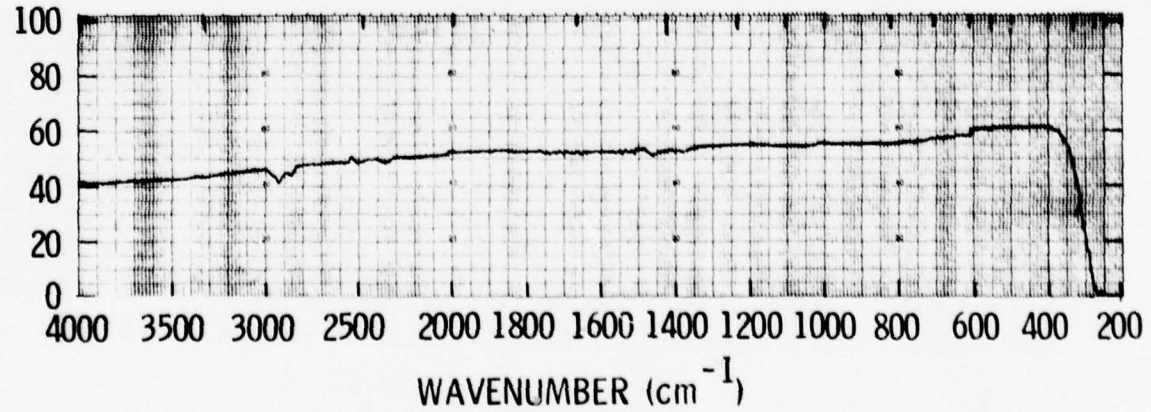

Fig. 12. Infrared Spectrum of Lead Foil

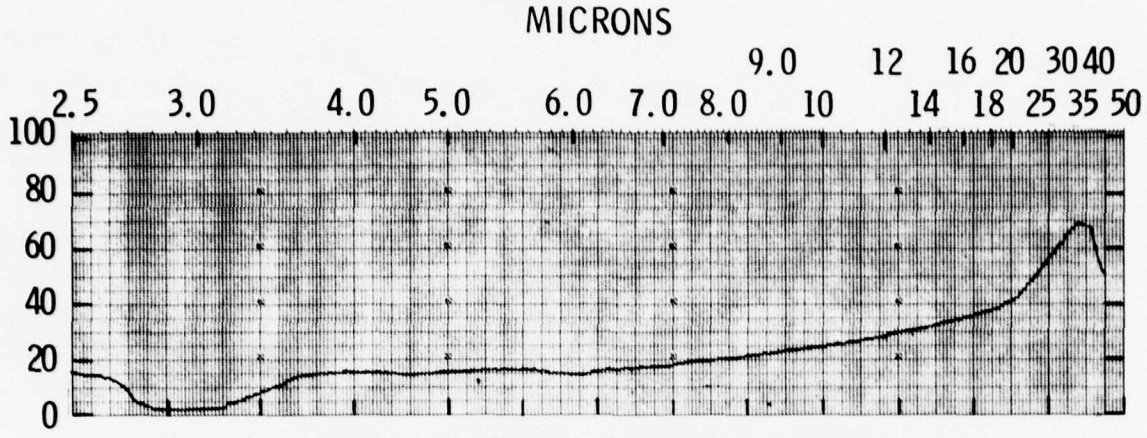

Fig. 13. Infrared Spectrum of $PbCl_2$

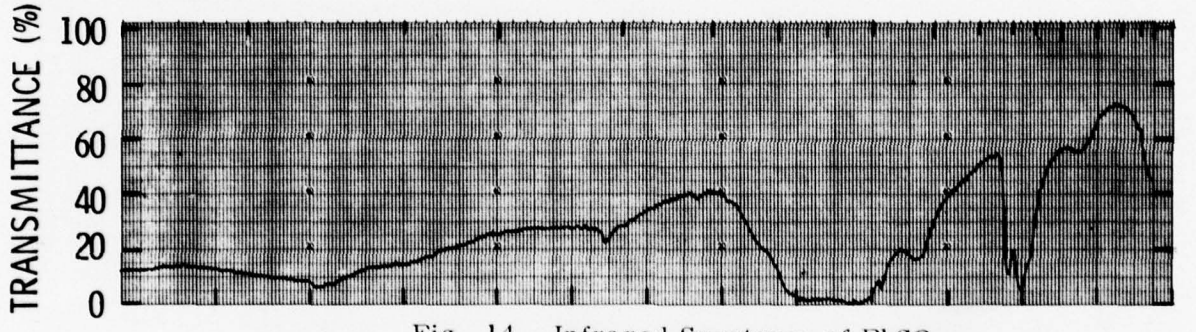

Fig. 14. Infrared Spectrum of $PbSO_4$

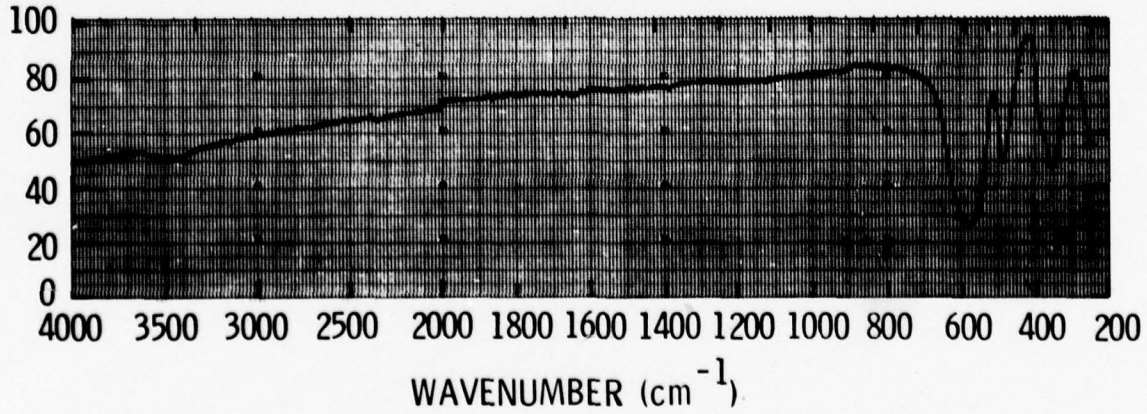

Fig. 15. Infrared Spectrum of Fe_2O_3

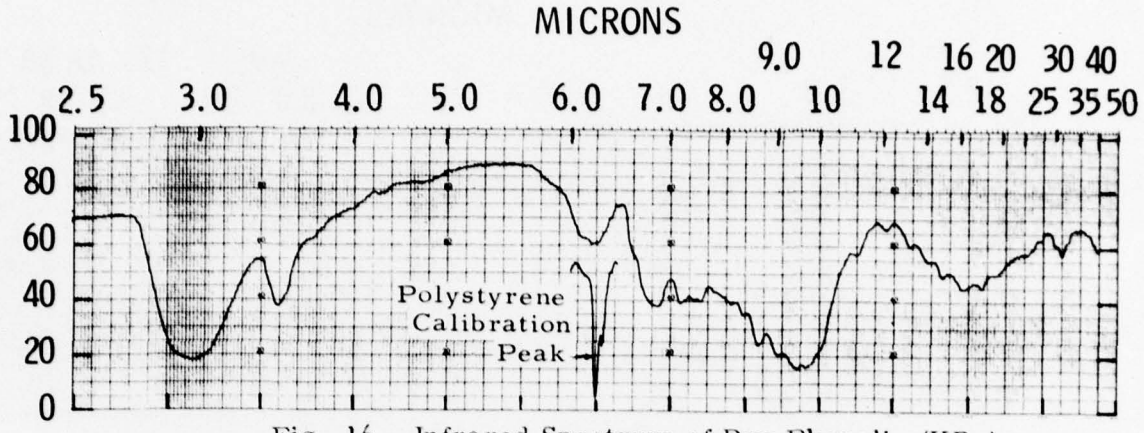

Fig. 16. Infrared Spectrum of Dry Phenolic (KBr)

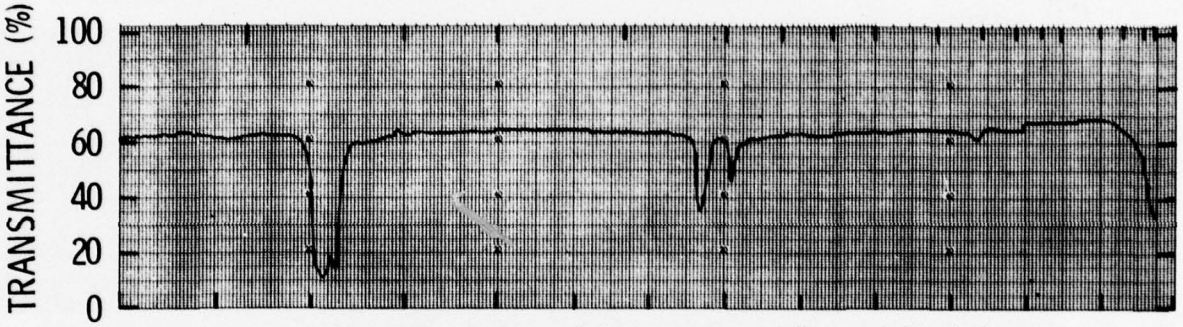

Fig. 17. Infrared Spectrum of 2% Lead Naphthenate in Apiezon C (KBr)

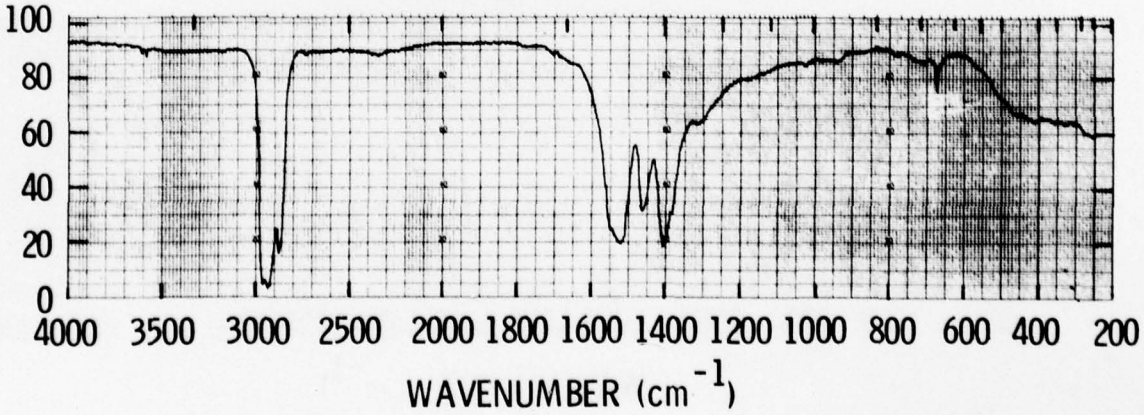

Fig. 18. Infrared Spectrum of Lead Naphthenate, Mull Between NaCl Plates

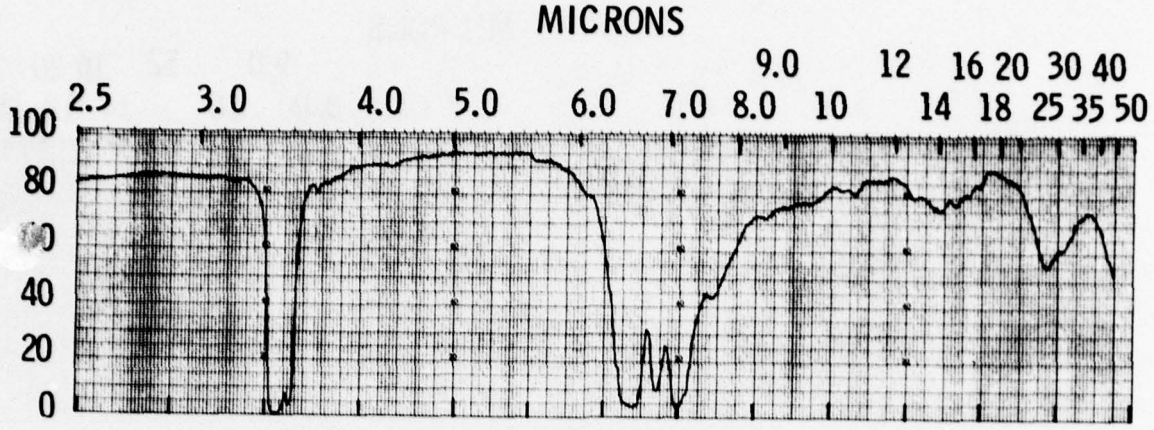

Fig. 19. Infrared Spectrum of Lead Naphthenate (KRS-5 mull)

Fig. 20. Infrared Spectrum of Lead Naphthenate on Surface of KBr Pellet

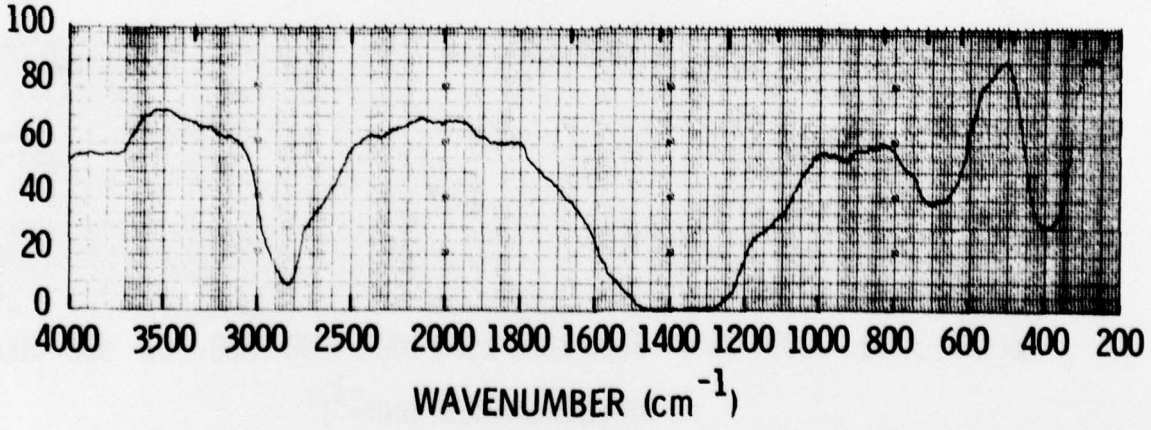

Fig. 21. Infrared Spectrum of Lead Naphthenate Intimately Mixed with the KBr Pellet

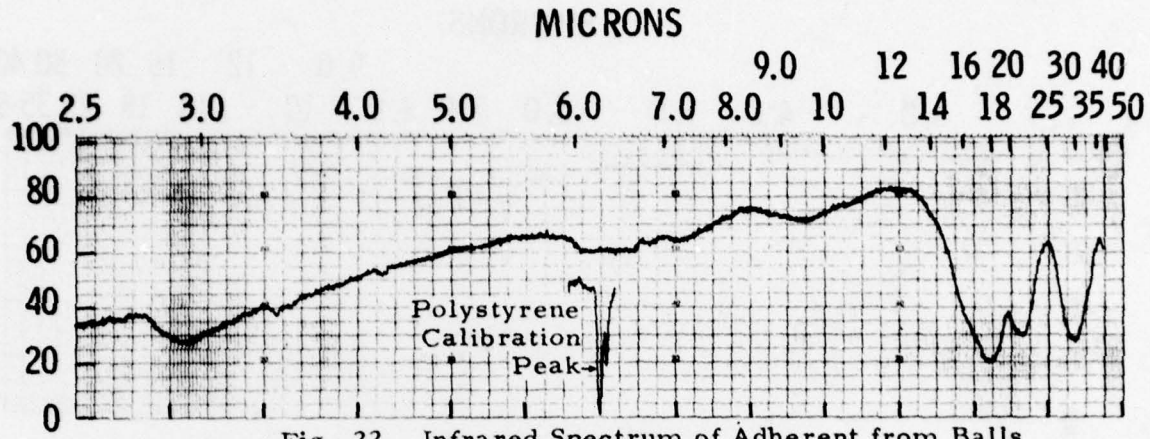

Fig. 22. Infrared Spectrum of Adherent from Balls of Bearing 5A (KBr)

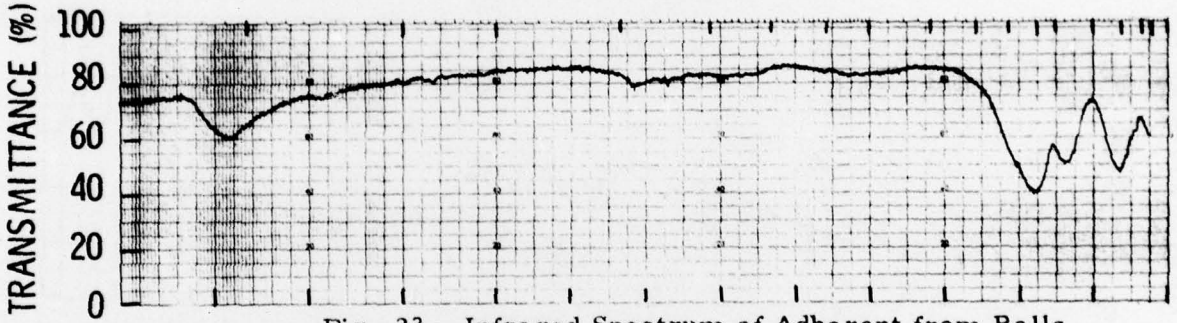

Fig. 23. Infrared Spectrum of Adherent from Balls of Bearing 6B (KBr)

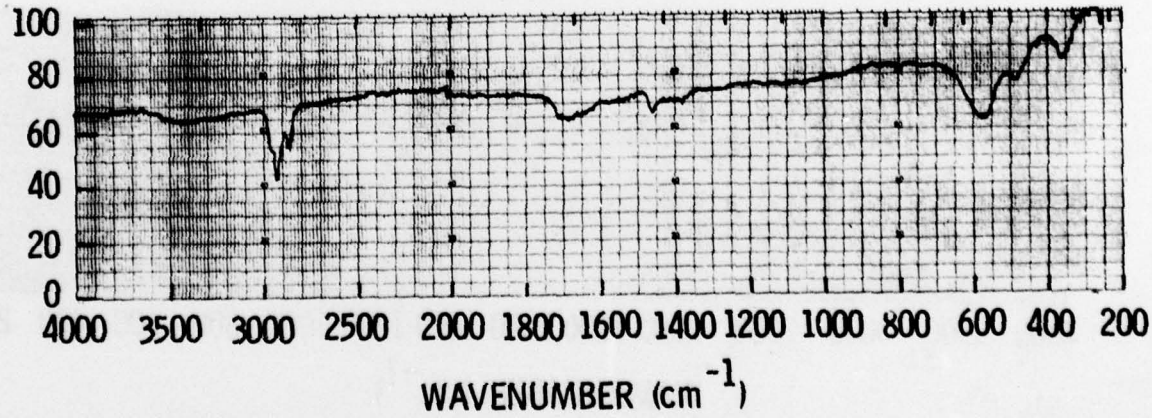

Fig. 24. Infrared Spectrum of Residue from Outer Race of Bearing 6B (KBr)

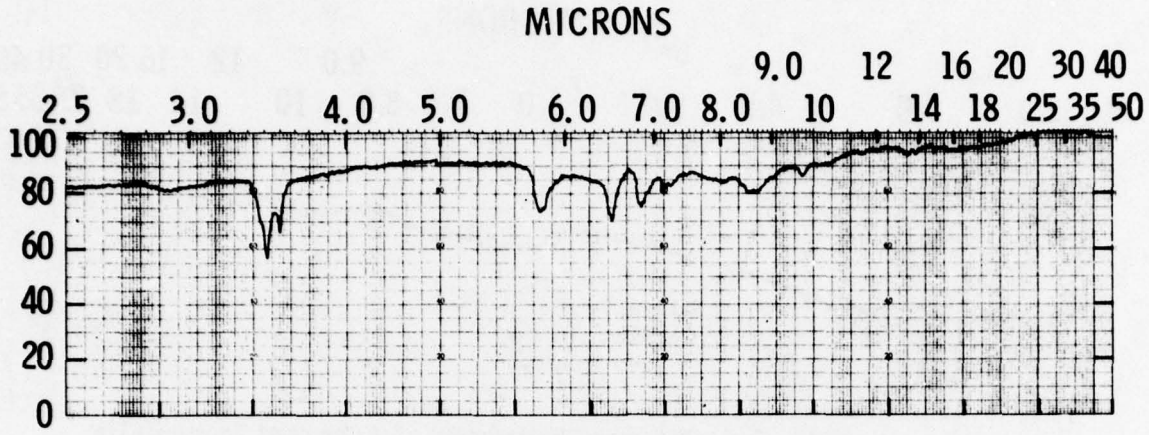

Fig. 25. Infrared Spectrum of Residue from Inner Race of Bearing 008 (KBr)

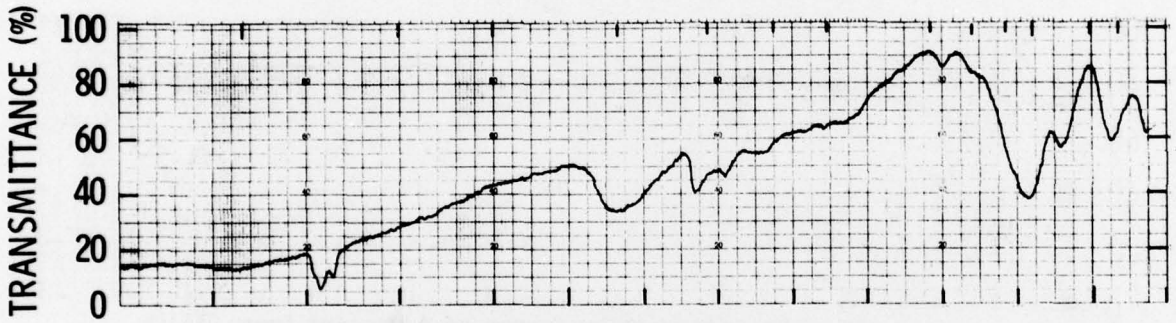

Fig. 26. Infrared Spectrum of Residue from Retainer Slots of Bearing 6B (KBr)

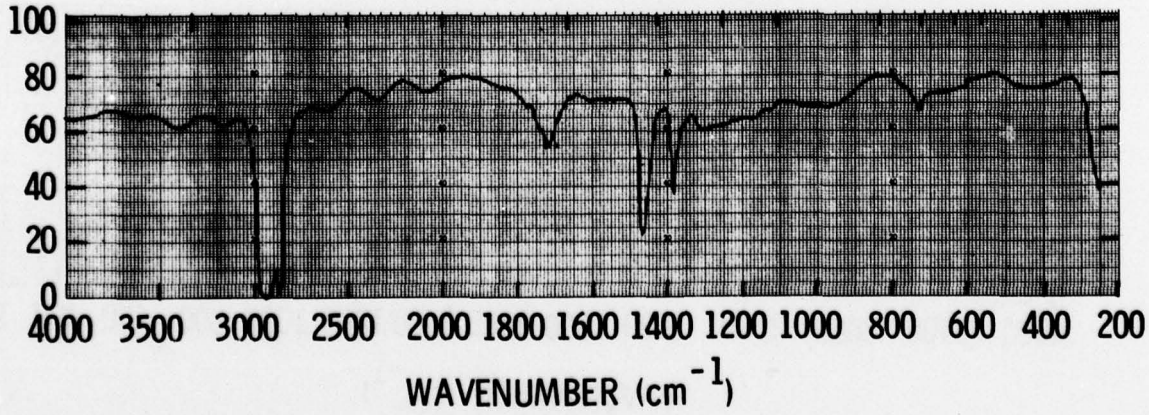

Fig. 27. Infrared Spectrum of Material Leached from Bearing 6B Phenolic Retainer by n-heptane; Solvent Removed (KRS-5)

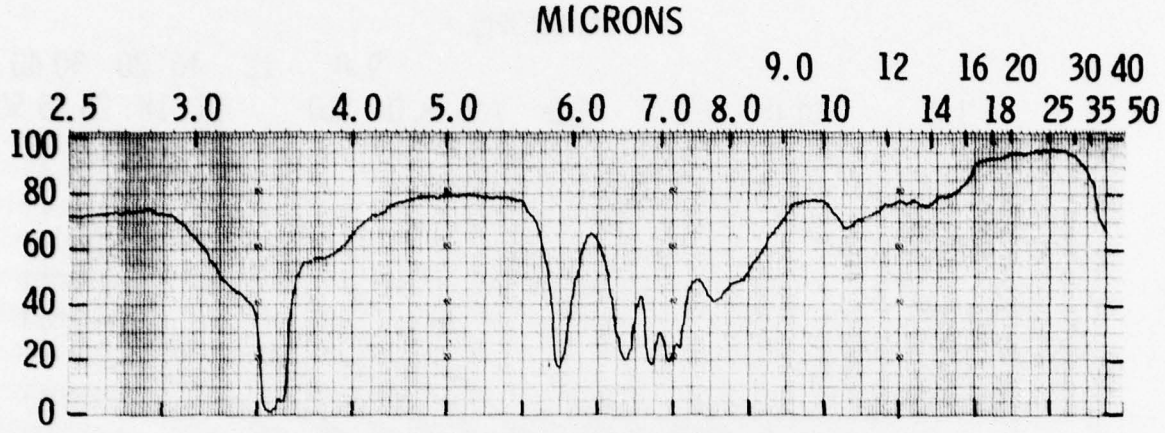

Fig. 28. Infrared Spectrum of Oil Layer Resulting from Reaction of Lead Naphthenate + 8% HCl. Mixture Heated at 100°C for 10 min (KRS-5)

Fig. 29. Infrared Spectrum of Oil Layer Resulting from Reaction of Lead Naphthenate + 10% H_2SO_4. Mixture Heated at 100°C for 10 min (KRS-5)

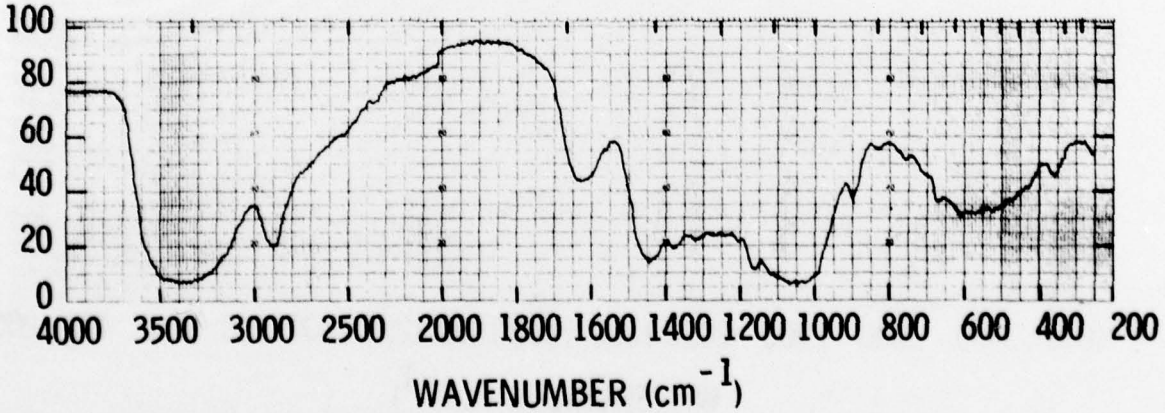

Fig. 30. Infrared Spectrum of Phenolic Treated with Sodium Hydroxide (KBr)

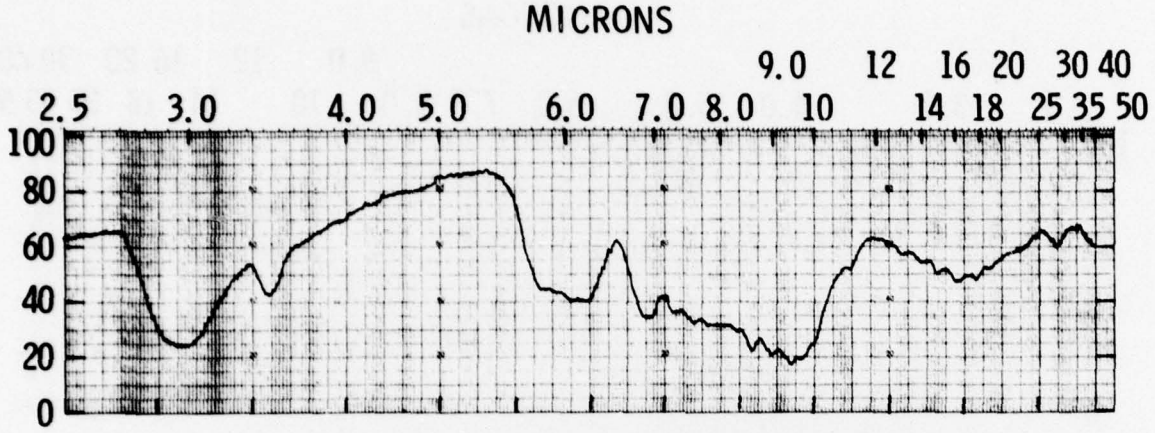

Fig. 31. Infrared Spectrum of Phenolic Heated at 200°C for One Hour (KBr)

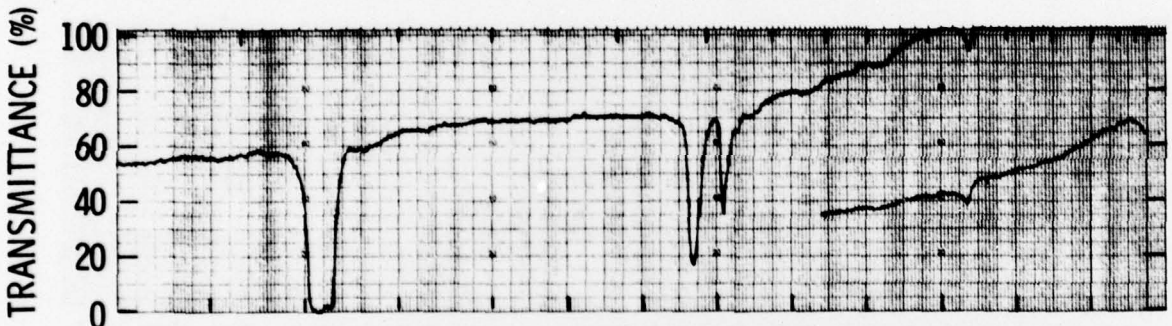

Fig. 32. Infrared Spectrum of 2% Lead Naphthenate in Apiezon C After Heating at 200°C for One Hour (KBr)

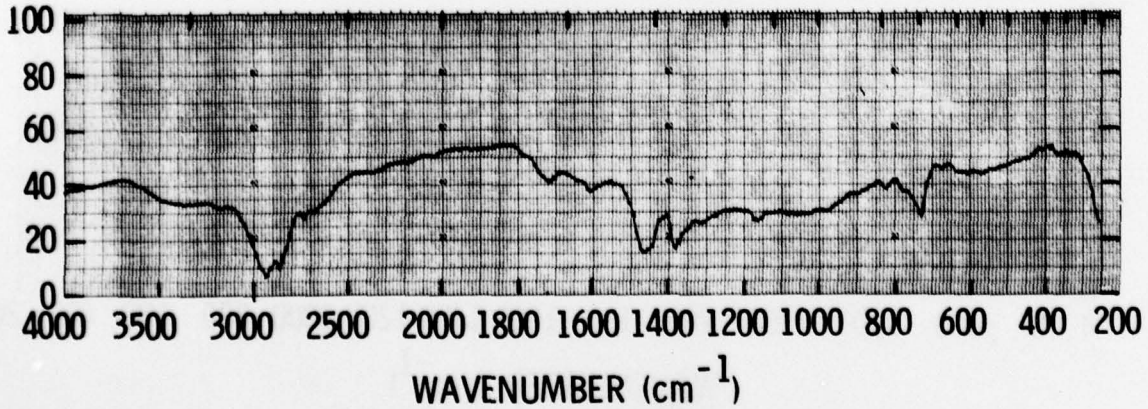

Fig. 33. Infrared Spectrum of Dry Phenolic Mixed with Apiezon C/2% Lead Naphthenate and Heated at 200°C for One Hour (KRS-5 mull)

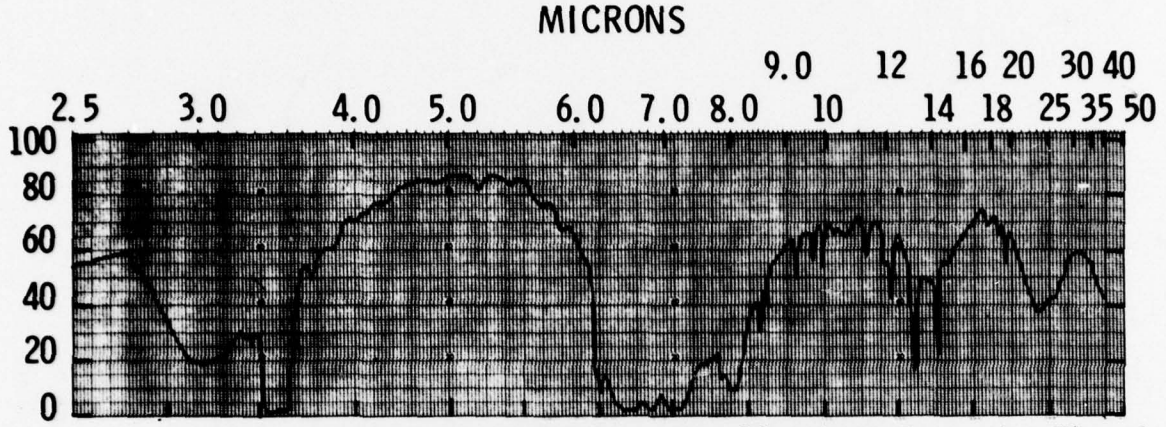

Fig. 34. Infrared Spectrum of Products Resulting from Contacting Phenol and Lead Naphthenate at 100°C for 10 min (KRS-5)

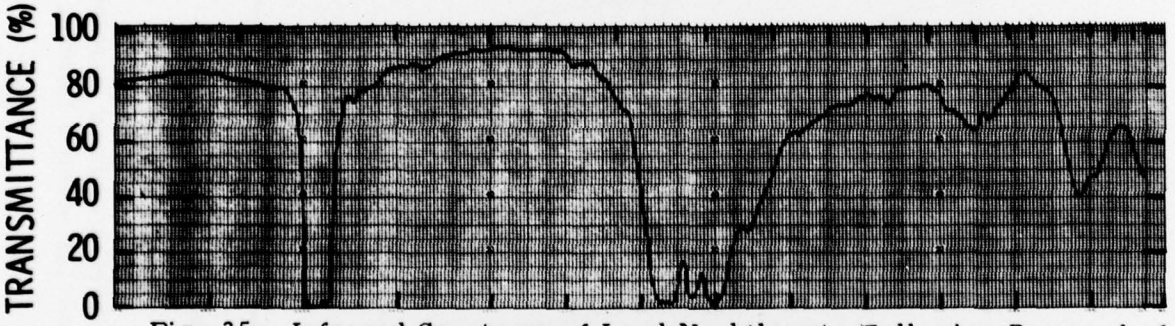

Fig. 35. Infrared Spectrum of Lead Naphthenate Following Removal of Phenol (KRS-5)

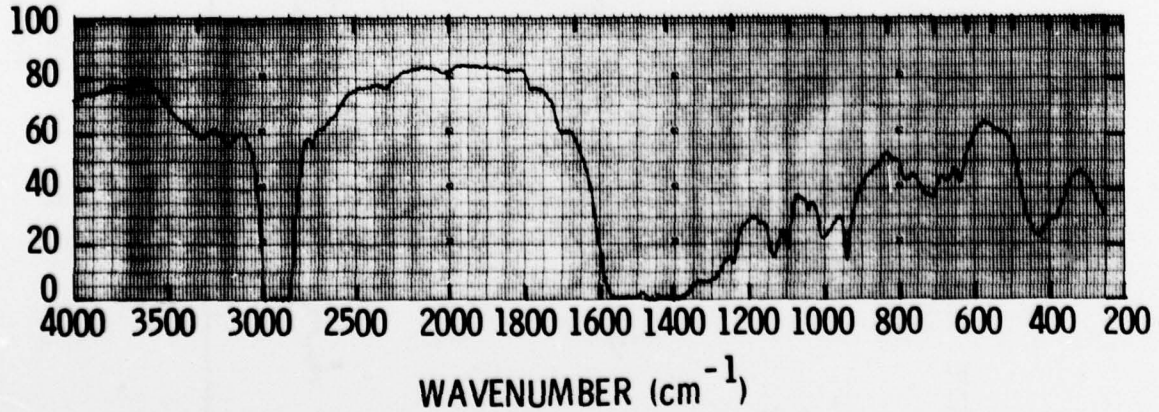

Fig. 36. Infrared Spectrum of Products Resulting from Contacting Formaldehyde and Lead Naphthenate at 100°C for 10 min (KRS-5)

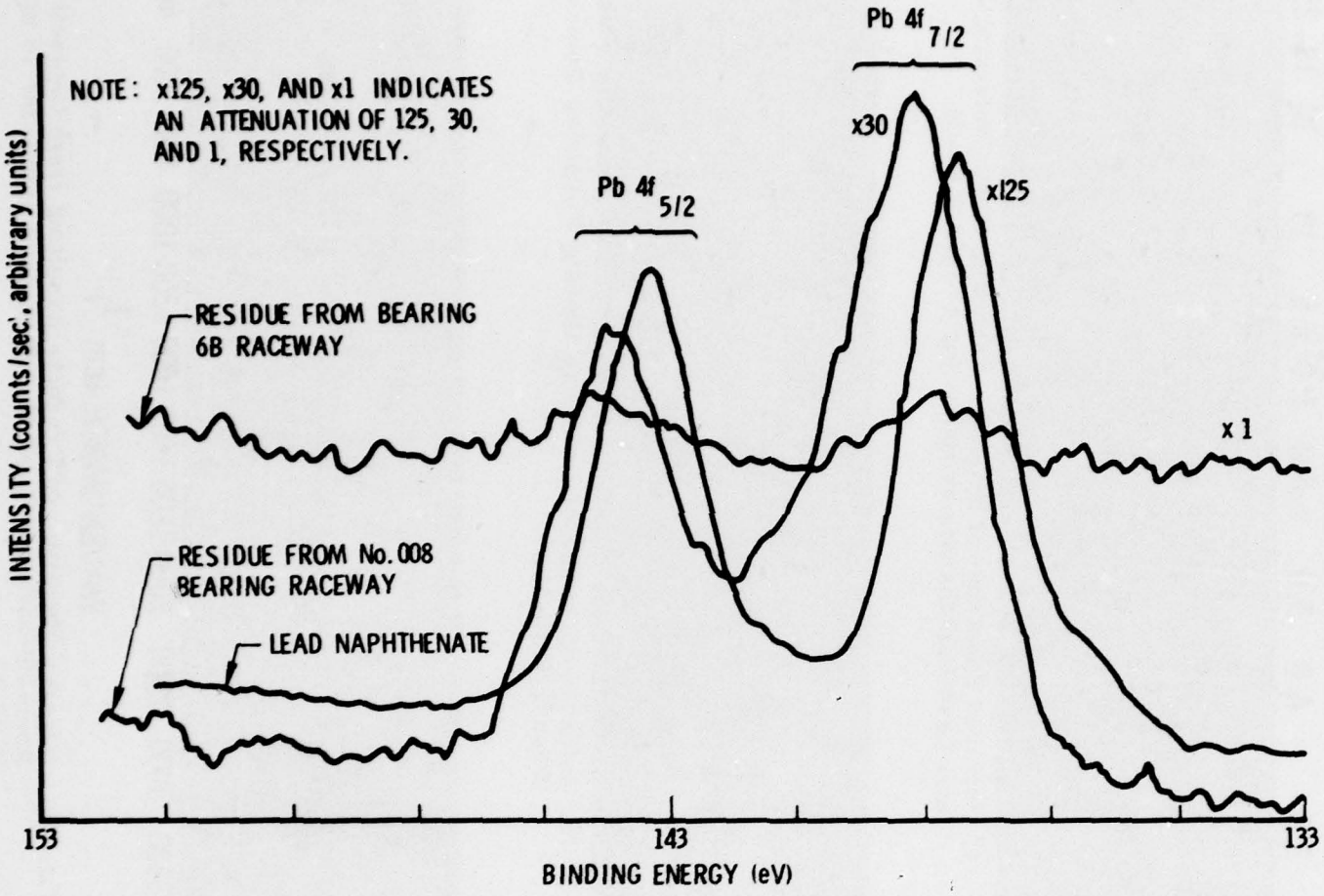

Fig. 37. ESCA Lead Spectra of Lead Naphthenate and of Deposits from Life-tested Bearing Assemblies

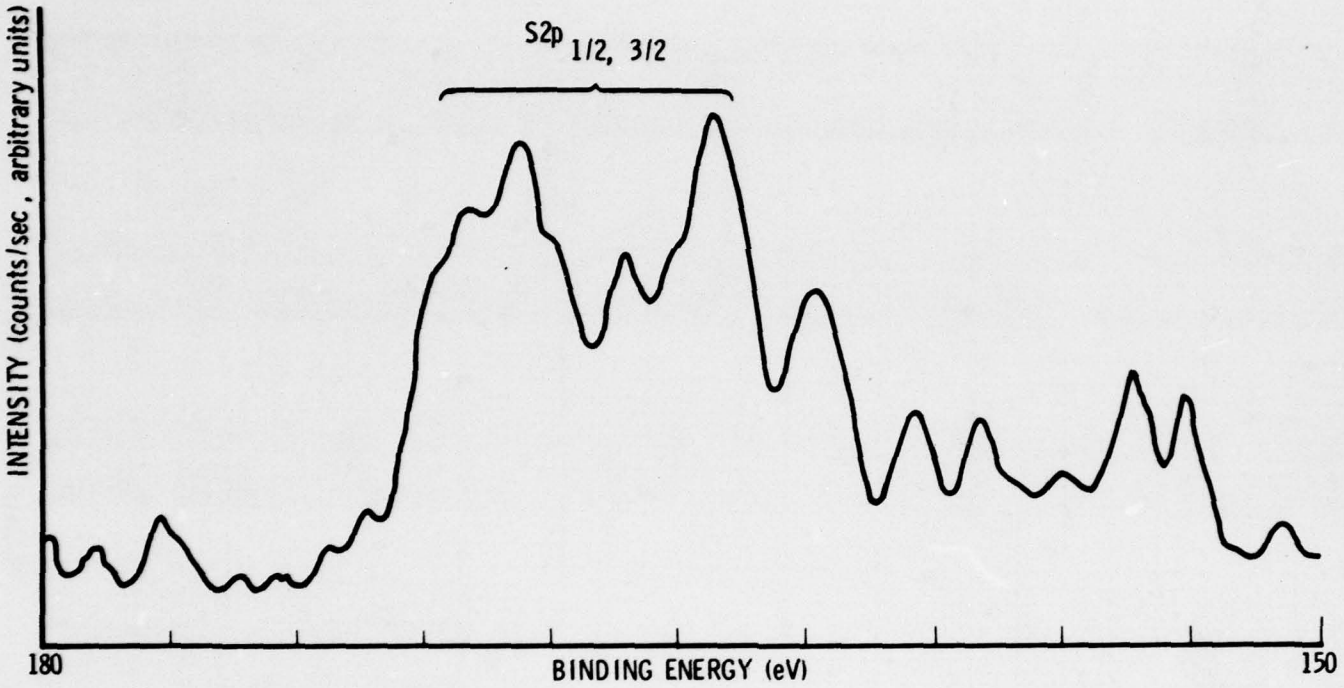

Fig. 38. ESCA Sulfur Spectrum of Deposit from Outer Raceway of Bearing 008

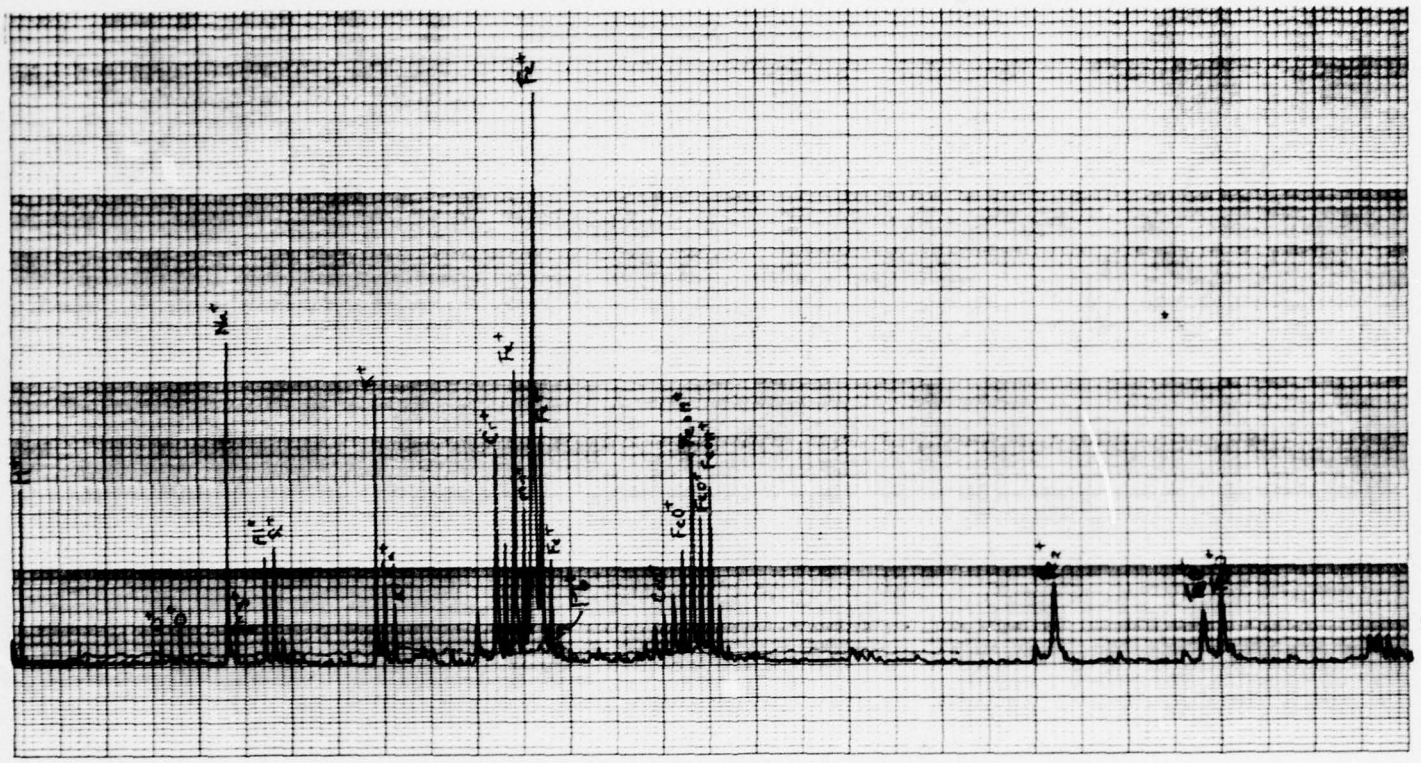

Fig. 39. IMMA Spectrum Using O_2^+ Excitation of 6B Ball Surface in an Area Free from Deposits

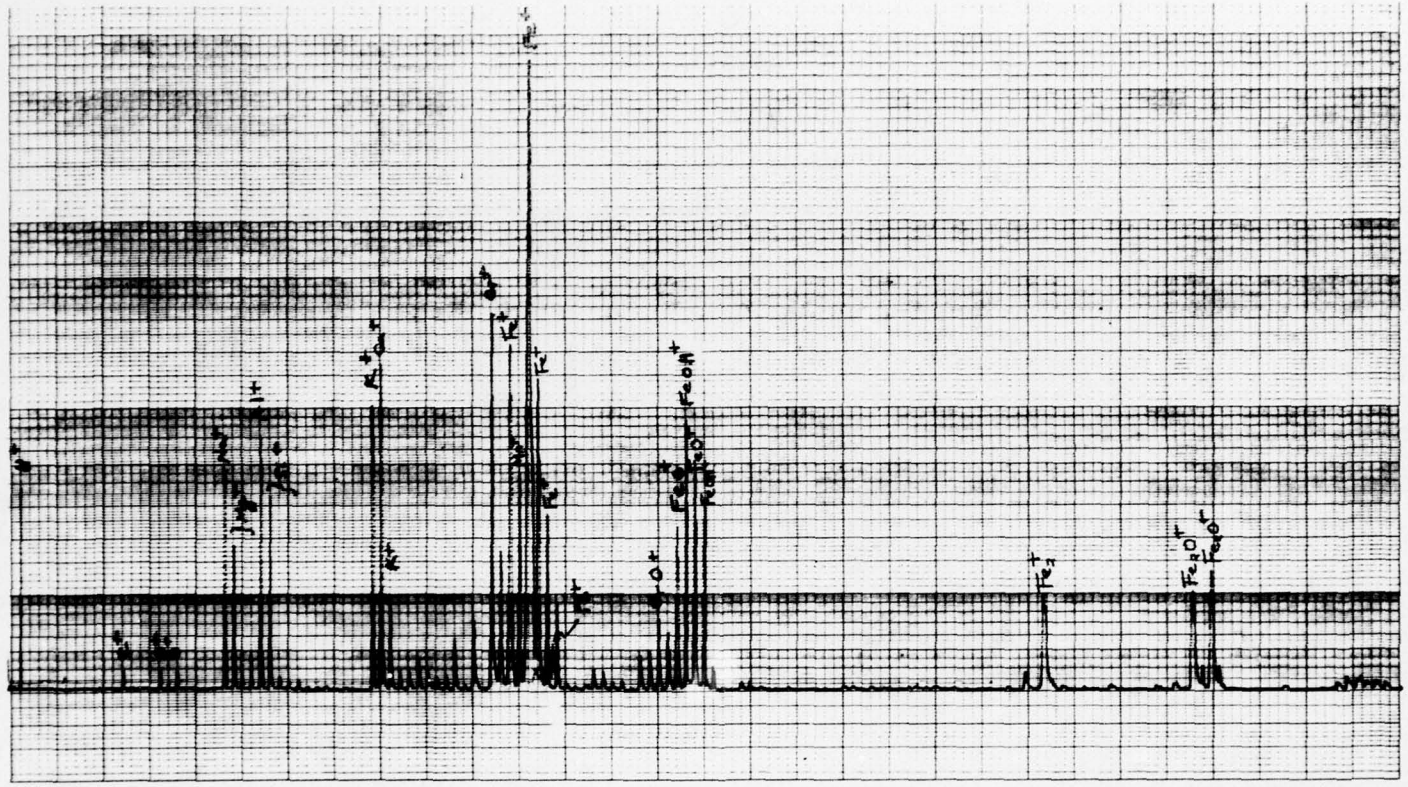

Fig. 40. IMMA Spectrum Using O_2^+ Excitation of 6B Ball Surface in an Area Covered with Deposits

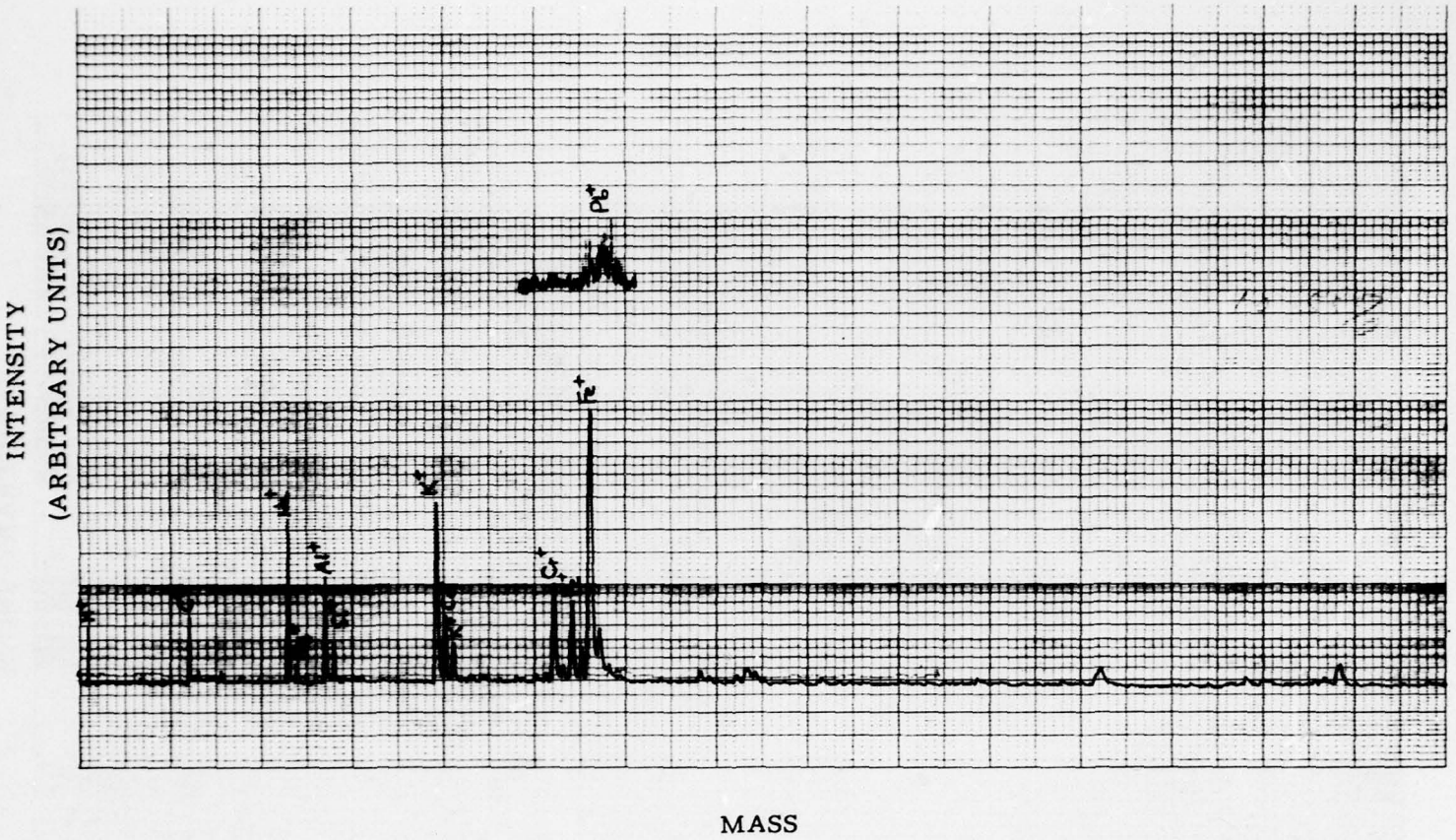

Fig. 41. IMMA Spectrum Using O_2^+ Excitation of 6B Raceway Deposits

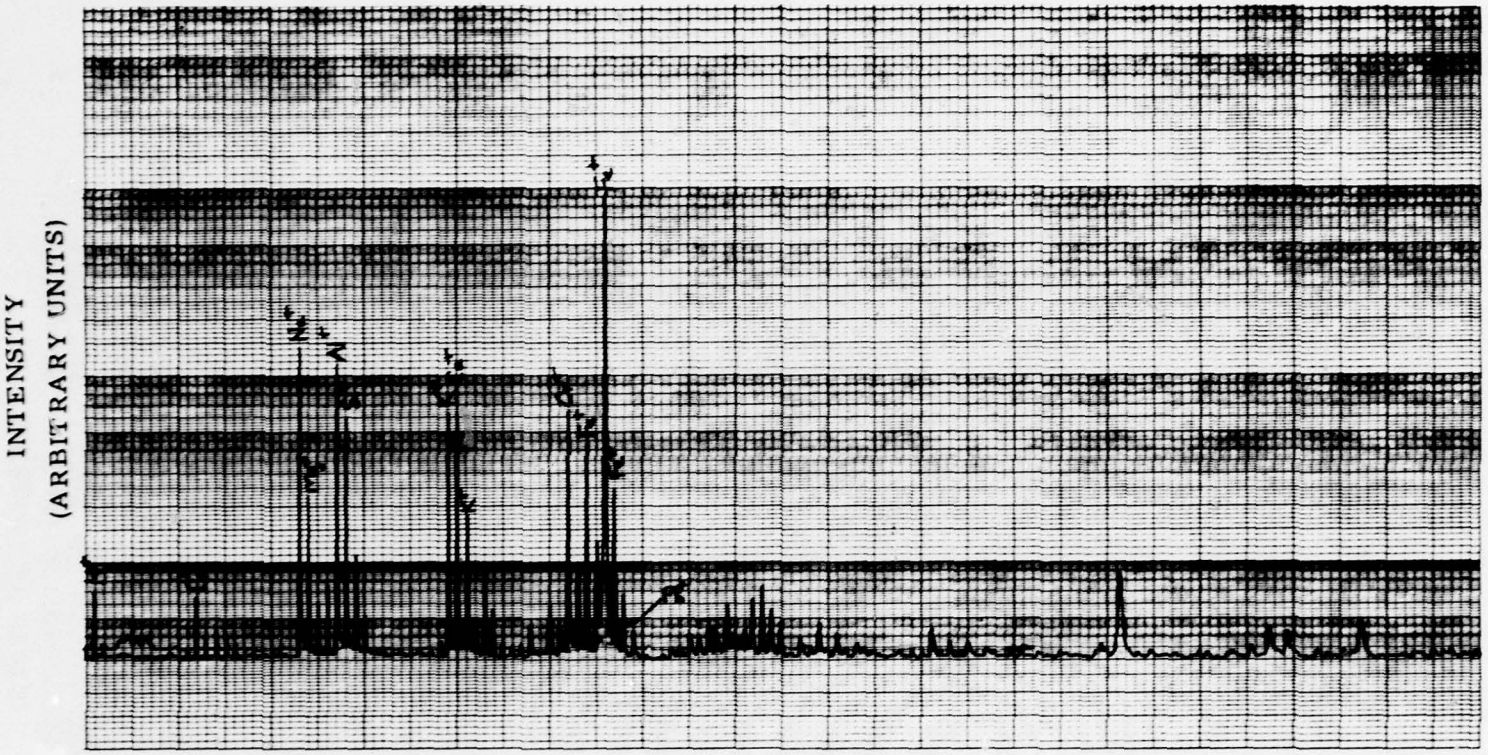

Fig. 42. IMMA Spectrum Using O^- Excitation of Deposit on 5A Ball Surfaces

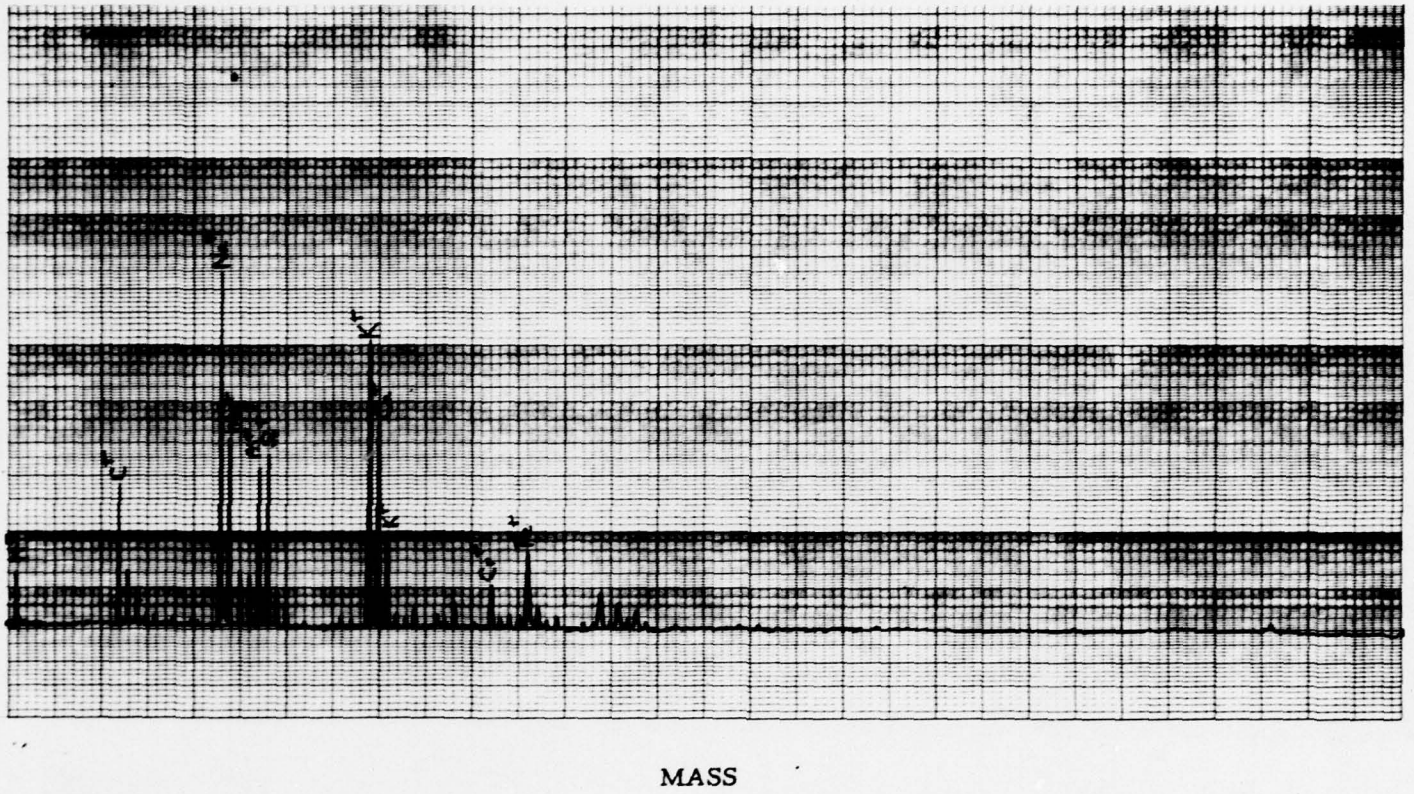

Fig. 43. IMMA Spectrum Using O^- Excitation of Deposit on 008 Ball Surfaces

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4. Personal communication with N. Marquez, Material Science Laboratory, Ivan A. Getting Laboratories, The Aerospace Corporation, El Segundo, California.

5. Fote, A.A., R.A. Slade and S. Feuerstein, "Surface Transport of Oil in the Presence of Nylasint and Micro-Well Reservoirs," Report No. TR-0076(6270-30)-7, The Aerospace Corporation, El Segundo, California, 19 July 1976.

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