

I. EXAMINATION OF POTENTIAL GEOCHEMICAL CONTAMINANTS IN LEG 48 MATERIAL

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INTRODUCTION

In Brest, France, prior to the beginning of DSDP Leg 48, opportunity was taken to examine potential sources of geochemical sample contamination. Obvious problems associated with sampling operations, e.g., the cleanliness of core tubes, were discussed with shipboard personnel, and some procedural changes were made during the subsequent collection of Leg 48 material. During the leg, the shipboard geochemist also made a collection of various lubricants, greases, and other fluids that could be encountered during the normal procedures of sample collection and subsequent shipboard processing.

Analysis of these samples was undertaken by a combination of column chromatographic separation procedures, followed by analysis of the fraction equivalent to the "saturate alkane content" by capillary column gas chromatography. The object of these analyses was not to obtain detailed compositional information on the various potential contaminants, but to provide a number of "fingerprint" chromatograms which could be used to access whether these materials were present in the samples examined.

EXPERIMENTAL METHODS AND RESULTS

A list of the samples examined is given in Table 1 together with an indication of their "apparent saturate alkane" contents. These data were obtained by gravimetric determination of the material eluted from silica gel by *n*-heptane, using the same chromatographic technique employed for the analysis of the sediment extracts (Doran et al., this volume). The fraction is by no means entirely composed of saturate alkanes, but the values give an indication of the proportion of contaminant that could be carried through to the stage at which significant hydrocarbon analyses are made.

Figures 1-11 show the individual chromatograms of the "apparent SAC fractions" obtained by splitless injection on an SE30/SCOT column, using conditions identical with those employed for the examination of the sediment extracts (*ibid.*). Identification of *n*-alkanes and pristane and phytane were based solely upon retention time and comparison with standards, but were not confirmed by GC-MS.

CONCLUSIONS AND RECOMMENDATIONS

1. The large quantity of contaminants present onboard which could infiltrate the saturate alkane chromatographic fraction of sediment extracts dictates that every caution must be taken to minimize the contamination.

TABLE 1
Results of Analysis by Silica Gel Chromatography
of Materials Selected as Possible Contaminants

Sample	"n + p" (%)
Lubricant for deck machinery	50.9
Electrical sealant used to seal end caps	~ 0.1
Diesel fuel	31.5
Silicone spray rock saw lubricant	5.2
Fluid for hydraulic gear	69.0
Lubricant for ship and rig machinery	48.7
Grease gun grease	48.5
Fluid for hydraulic gear on heave compensator	~ 0.1
WD-40 occasional lubricant for rock saw	30.60
Pipe corrosion inhibitor	1.5
Pipe dope	24.9

Note: The fraction described as "n + p" is the liquid chromatographic fraction dating from a silica gel column where the saturate fraction from a sediment extract would elute.

2. Analyses of the saturate alkane fractions from the Leg 48 samples (*ibid.*) do not suggest that significant contamination from the various potential sources examined was present; it was impossible, in most cases, to establish whether or not contamination could have been present in low concentrations. Unfortunately, no specific marker compounds were present in high enough concentrations to enable specific contaminants to be readily recognized solely by gas chromatographic techniques.

3. It is impractical to exclude potential contaminants of this type from shipboard operations involving geochemical samples, and it is unlikely that many of the potential contaminants could be reformulated to contain specific marker compounds. It is recommended, however, that all possible sources of contamination of organic geochemical samples be constantly monitored and controlled. Whenever possible, more background examinations of this type should be undertaken and much more detailed analyses of potential contaminants obtained by GC-MS techniques. From such studies a library of gas chromatographic and gas chromatographic/mass spectrometric data could be compiled for improvement of future contamination assessments.

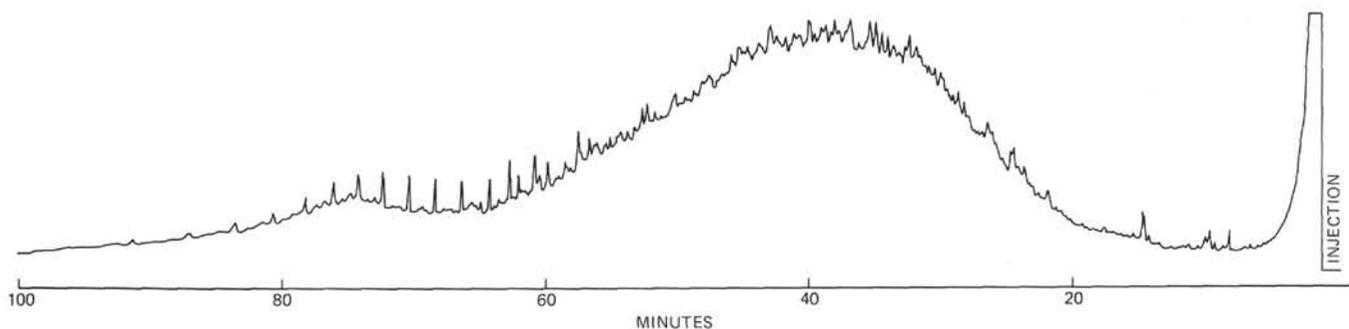


Figure 1. Gas chromatogram of lubricant for deck machinery.

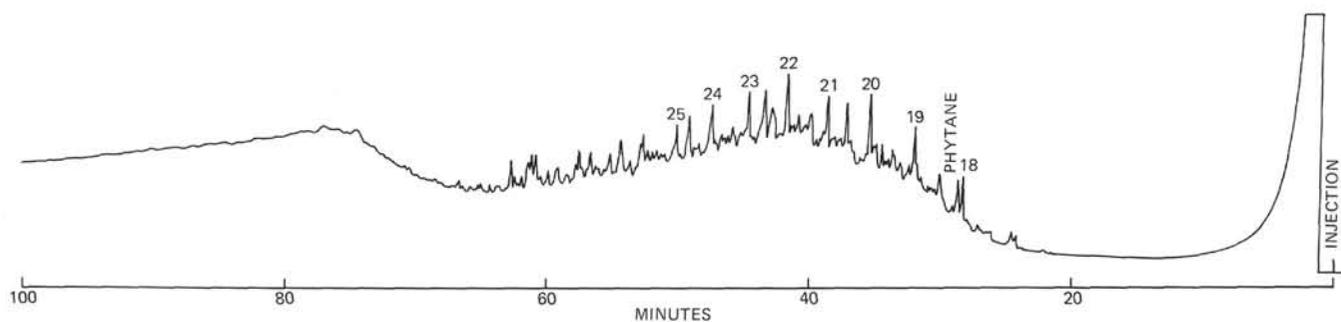


Figure 2. Gas chromatogram of electrical sealant used to seal end caps.

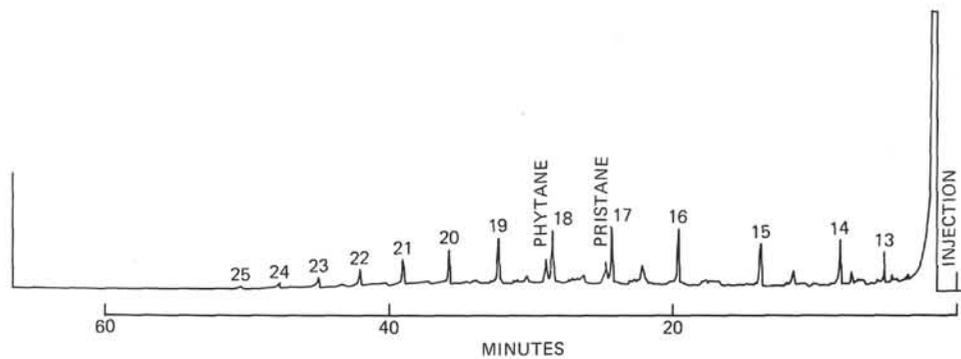


Figure 3. Gas chromatogram of diesel fuel.

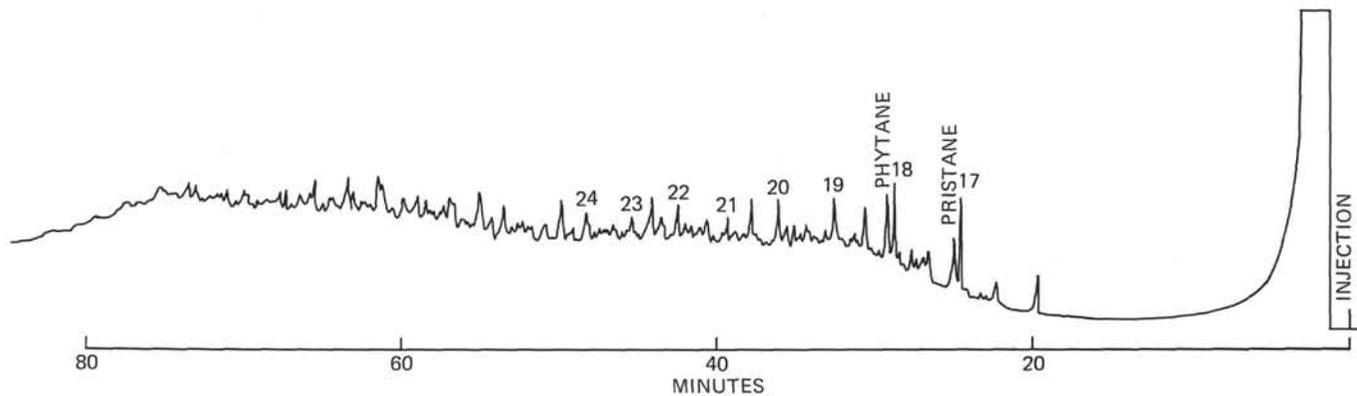


Figure 4. Gas chromatogram of silicone spray rock saw lubricant.

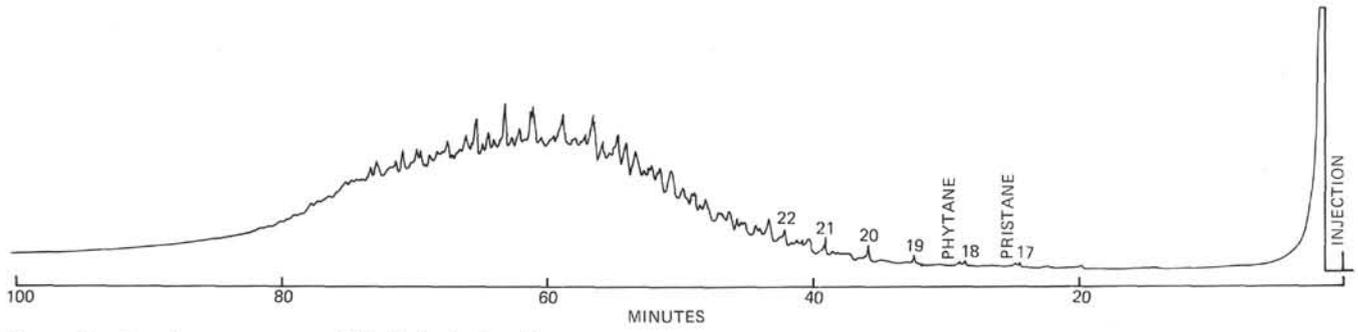


Figure 5. Gas chromatogram of fluid for hydraulic gear.

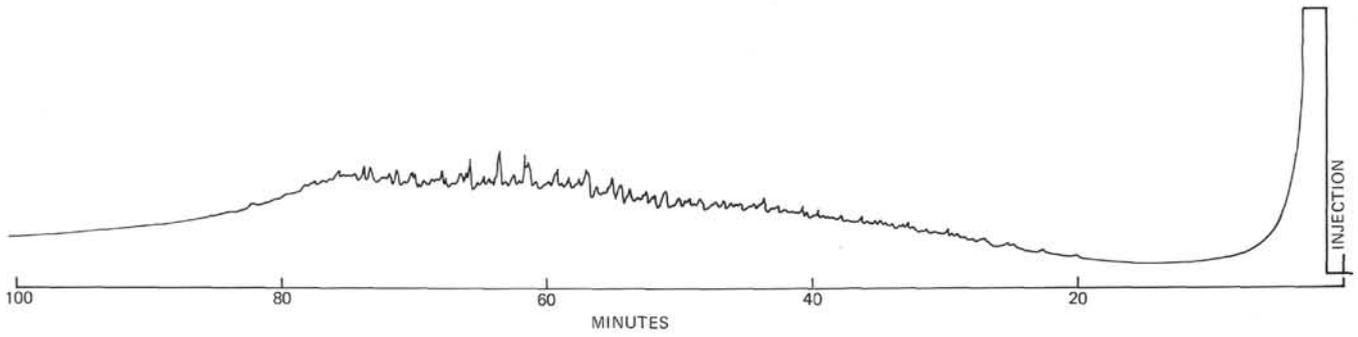


Figure 6. Gas chromatogram of lubricant for ship and rig machinery.

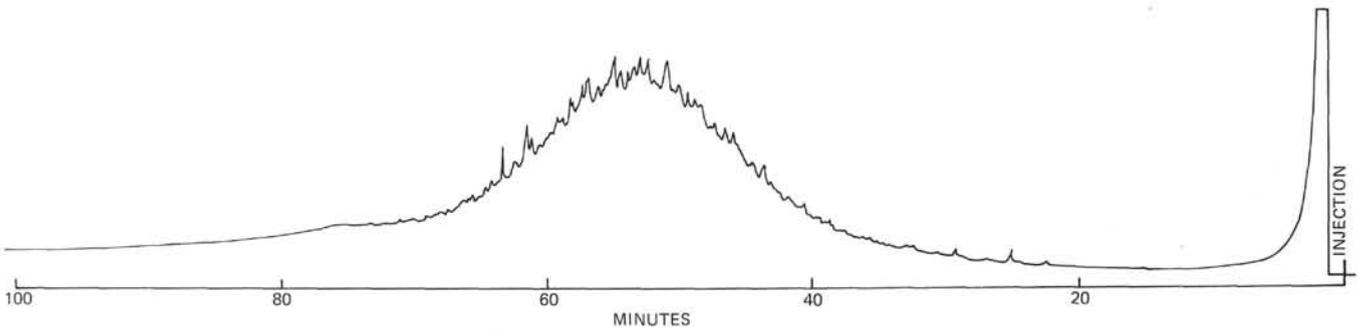


Figure 7. Gas chromatogram of grease gun grease.

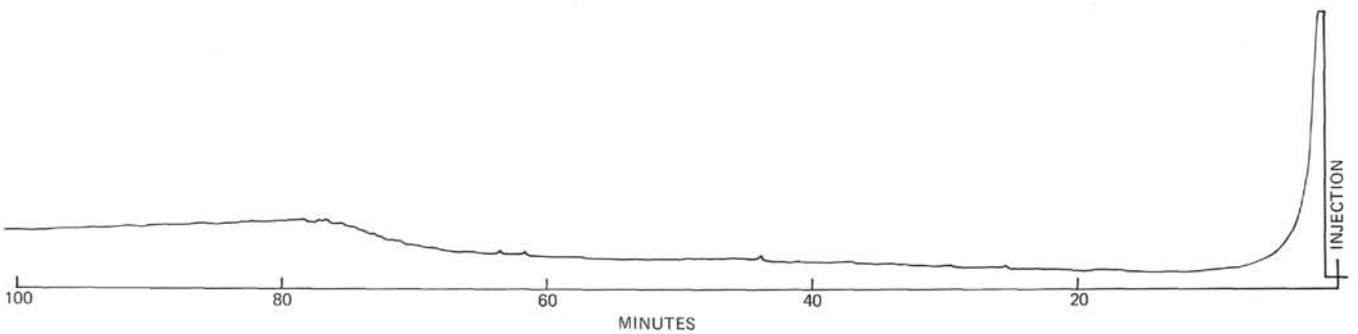


Figure 8. Gas chromatogram of fluid for hydraulic gear on heave compensator.

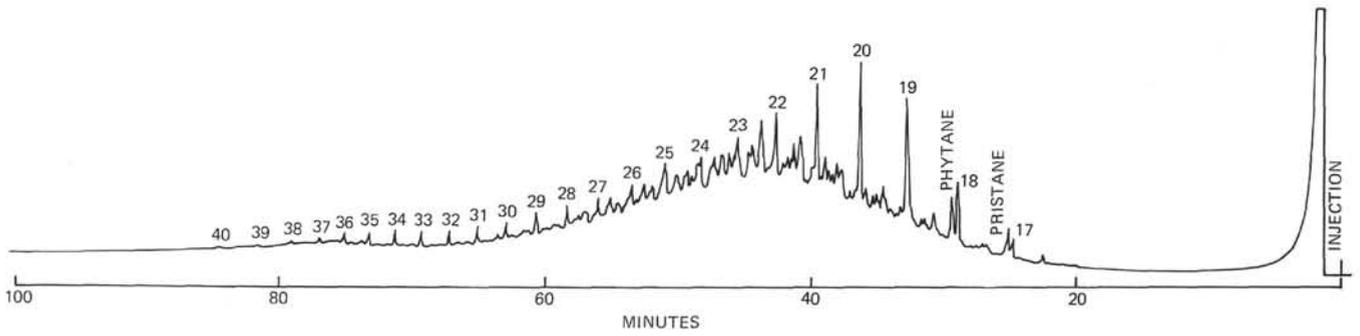


Figure 9. Gas chromatogram of WD-40 occasional lubricant for rock saw.

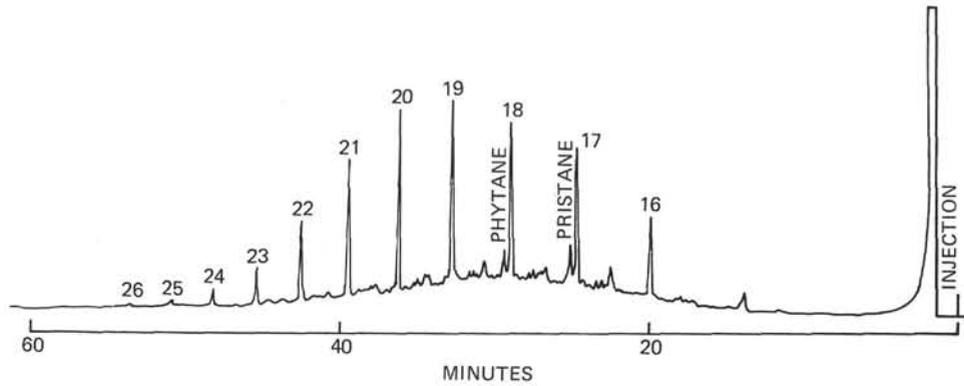


Figure 10. Gas chromatogram of pipe corrosion inhibitor.

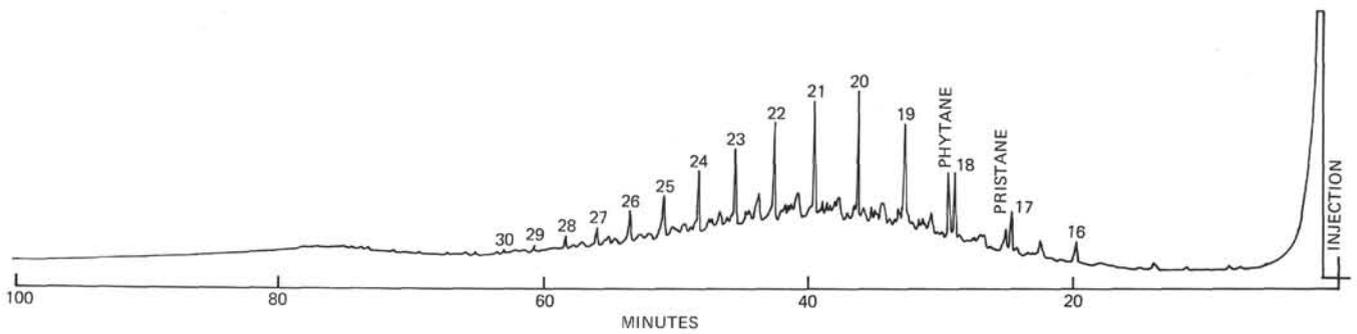


Figure 11. Gas chromatogram of pipe dope.