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Demetallation of Used Oil to Facilitate Its  
Utilization as a Fuel

J. H. Kang and A. R. Tarrer, Chemical Engineering Department,  
Auburn University, Auburn, AL 36849

Joe Kaminski, The Office of the Assistant Secretary of Defense

Jim Parrish, Defense Reutilization and Marketing Service (DRMS)

Edward R. Bates, U. S. Environmental Protection Agency

## NOTICE

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**ABSTRACT:** Used oil invariably contains soluble and insoluble metal-bearing compounds. The presence of these compounds complicates the recycling of used oil as a fuel, by causing boiler maintenance problems and by making it difficult to comply with particulate emission standards and RCRA regulations. Demetallating a used oil prior to its burning minimizes maintenance problems and facilitates meeting the regulatory requirements controlling its burning. Different physical and chemical methods can be used to demetallate used oils. In this work, a chemical demetallation agent was used to convert entrained non-filterable metals into a form which could be effectively removed by filtration. Two different types of chemical demetallation agents were used: metallic borohydrides ( $\text{NaBH}_4$  and  $\text{KBH}_4$ ) and diammonium phosphate (DAP). The activity and selectivity of these demetallation agents under different reaction conditions in various types and sizes of reactors (thereby varying mass transfer rates) were compared. Several types of used oils having different metal contents were examined.

It was observed in this study that metallic borohydrides were highly selective with regard to the types of metals they removed. The accompanying sludge formed also made the separation operation inefficient. On the other hand, results obtained with DAP were extremely favorable. The presence of water during the reaction was found to play an important role as a carrier for the DAP, aiding in its dispersion and reducing the importance of mass transfer. This study which also evaluated different separation techniques found the use of both sedimentation and filtration to be practical, with filtration rates being significantly accelerated by the addition of low percentages of No. 2 fuel oil.

**KEY WORDS:** Waste oil, demetallation, ash content, lead content, metallic borohydrides, diammonium phosphate, lead reduction.

## Introduction

In the United States, automotive and other oil-consuming industries generate about 1.1 billion gallons of used oil each year [1]. This voluminous amount constitutes a serious waste oil disposal problem. Recent Environmental Protection Agency (EPA) regulations and growing public concern over hazardous wastes make development of a viable alternative to land-filling very desirable. The objective of this work was to develop a practical and feasible waste oil demetallation process to remove the ash-forming contaminants, especially organolead compounds, from the oil so that the demetallated used oil would qualify as a specification-grade fuel oil [2].

Waste oil, although contaminated, has a high energy value. A major operation in reclaiming waste oil is the removal of water and various ash-forming impurities that remain dispersed in the used oil. The ash-forming impurities consists of very fine carbon particles, organometallic particulates (e.g., organolead compounds), and inorganic materials (e.g., dust).

Reuse of an untreated used oil having a high concentration of lead and other metallic contaminants may lead to serious air pollution and boiler maintenance problems. To qualify for minimal regulatory requirements, i.e., specification-grade fuel oil requirements under EPA regulations, the lead content of a waste oil to be recycled as a fuel in a commercial boiler can not exceed 100 ppm. Since most of the used oil from the automotive industry contains several times the proposed EPA limit, it is imperative that an economic process be developed to reduce the lead content of the oil to an acceptable level so that the reclaimed waste oil can safely be reused as a fuel oil without endangering public health or the environment.

Waste oils were demetallated using two types of demetallating reagents: metallic borohydrides and diammonium phosphate (DAP). A detailed parametric

study was undertaken to map out the process variables so as to identify the most efficient demetallation conditions. An evaluation of solid-liquid separation techniques was also done to design an efficient solids removal operation for future pilot plant studies.

#### **Experimental Procedure**

**Materials** -- All used oil was collected from the Auburn University Waste Oil Reprocessing Pilot Plant. The demetallating reagents used were sodium borohydride, potassium borohydride, sodium borohydride aqueous solution, and diammonium phosphate (DAP). The phase transfer catalyst used was tri-n-butyl-methyl ammonium chloride.

**Equipment** -- Three types of reactors were used. They were: an autoclave (3785 ml and 300 ml), a microreactor (45 ml), and a distillation flask (300 ml). The autoclave was equipped with a turbine agitator, a cooling coil, baffles, and a thermowell, all made of 316 stainless steel. The autoclave was heated by an electric furnace and agitation was provided by a magne drive system. The tubing bomb microreactor (TBMR) was constructed of 316 stainless steel tubing (1.9 cm O.D. with 0.165 cm wall thickness and a length of 20.3 cm). Agitation was provided by a shaker assembly as shown in Figure 1. For a typical run, the TBMR was immersed in a preheated fluidized sand bath. As for the distillation flask, the reaction mixture was agitated using a magnetic stirrer, and the reactor was heated in a constant temperature paraffin oil bath.

**Analysis** -- The experimental results were verified in accordance with an EPA approved quality assurance project plan. The properties of the demetallated used oil were evaluated to assess its potential as an EPA specification-grade fuel oil using the following test methods: water content, ASTM D4006-81 (Water in crude oil by distillation); ash content, a modification of ASTM

D482-80 (Ash from petroleum products); lead content, standard atomic absorption (AA) analysis.

In each ash analysis, an oil specimen (about 5 g) in a preweighed crucible was dried first on a hot plate and then heated in a Model F-6000 Thermolyne muffler furnace at a rate of 373K (100°C) per hour. Once the temperature reached 873K (600°C), the crucible was allowed to stay in the furnace for an additional 12 hours. Then, the crucible was removed and cooled in a desiccator. The weight of ash remaining in the crucible was determined, and its percent concentration in the original oil sample was computed.

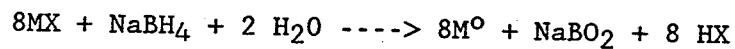
In each lead analysis, the ash was dissolved in an acid solution of 10 ml of HNO<sub>3</sub> solution (1:1) and 2 ml of concentrated HCl. The lead content was determined using AA spectroscopy. A lead working standard was prepared by serial dilution of a lead standard solution (1000 ± 1% ppm) supplied by Fisher Scientific. A blind-spiked oil sample was occasionally prepared to verify the results. An organic lead standard (lead cyclohexanebutyric acid, [C<sub>6</sub>H<sub>11</sub>(CH<sub>2</sub>)<sub>3</sub>COO]<sub>2</sub> Pb) was used for this purpose.

## Results and Discussion

### *Use of Metal Borohydride As A Demetalling Reagent*

The properties of metal borohydrides, such as NaBH<sub>4</sub>, KBH<sub>4</sub>, and SWS solution (consisting of 12 wt% NaBH<sub>4</sub>, 40 wt% NaOH, 48 wt% water), are described in detail in technical literature published by Morton Thiokol, Inc., Ventron Division [2,3].

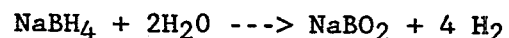
Metallic borohydrides (e.g. sodium borohydride) are strong reducing agents. The following reaction is typical of the metal reduction that occurs with sodium borohydride [2].



where M = the metal (valence 1<sup>+</sup>)  
X = the anion

In this chemical reaction, stoichiometrically, sodium borohydride ( $\text{NaBH}_4$ ) has 8 reducing equivalents per molecule. Based on bench-scale experimental results, Morton Thiokol, Inc. reported that a stabilized water solution (i.e., SWS solution) was an effective reagent for removing lead from used crankcase oil [4]. It should be noted that sodium borohydride ( $\text{NaBH}_4$ ) degrades in the presence of water or acid via hydrolysis to liberate hydrogen and a salt ( $\text{NaBO}_2$ ) when the pH of the solution is low. A representative chemical reaction is as follows:

Water Hydrolysis:



A parametric study was done to investigate the lead reduction efficiency of two Morton Thiokol products: Venpure powders ( $\text{NaBH}_4$  and  $\text{KBH}_4$ ) and SWS solution. A tubing bomb microreactor (TBMR) and the reaction conditions listed in Table 1 were used in this study. A concentrated NaOH aqueous solution (50 wt%) was usually added to maintain a high pH and thus a low hydrolysis activity of the metal borohydrides.

In a typical run, the TBMR was charged with 20 g of waste oil, the specified amounts of 50 wt% NaOH aqueous solution and VenPure product. The reactor was then sealed. Subsequently, the TBMR was attached to the vertical shaft of the agitation assembly, as shown in Fig. 1, and agitated at ambient temperature for 3 minutes at 860 rpm before lowering it into the preheated fluidized sand bath. The heat-up time was about 60 seconds. At the end of the chosen reaction time, the TBMR was removed from the sand bath, immediately quenched in cold tap water, and checked for any leaks. The gases were released slowly from the bomb under a hood and the liquid product was then collected and vacuum filtered. The filtrate (or product oil) was collected for ash and lead analyses.

With 0.65 wt% SWS and 1.2 wt% NaOH solutions, the lead content of the Type B oil was markedly reduced from 480 to 70 ppm (Run 1) as shown in Table 2. In other words, an 85.4% lead reduction was achieved. This is well within the allowable maximum lead content of 100 ppm for specification grade used oil set by EPA. However, a high sludge production (16 wt%) resulted from this run.

Since metal borohydrides are expensive, the charge of SWS solution (containing 12 wt% sodium borohydride) was reduced to 0.43 wt% in Run 2. Also, the charge of NaOH concentrated solution was reduced from 1.2 wt% to 0.8 wt%. The result was that the lead content of the oil decreased by only about 50%, to 240 ppm. The ash content of the product oil was 0.58 wt%. The amount of sludge production was about the same as that from Run 1.

In order to reduce the sludge production, in Run 3, only 0.05 wt%  $\text{NaBH}_4$  powder was used and no NaOH was added. The amount of lead reduction was only 10.4 percent; however, little sludge was produced (2 wt%). To enhance the demetallation activity, 0.8 wt% of 50 wt% NaOH was added in Run 4 and the other reactions conditions were kept the same. The lead removal efficiency increased from 10.4% for Run 3 to 37.5% for Run 4.

To improve the solubility of  $\text{NaBH}_4$  in the oil phase, 0.10 wt% of phase transfer catalyst (tri-n-butyl-methyl ammonium chloride) was also added to the oil (Run 5). Comparing the results of Run 5 with those of Run 3 (in both runs no NaOH was added), the addition of the phase transfer catalyst did result in an increase in lead removal efficiency from 10.4 to 27.1%. With 0.8 wt% NaOH solution added in Run 6, the lead reduction efficiency improved slightly more to 31.3 wt%. On the whole, there seemed to be a trade-off between using either SWS or  $\text{NaBH}_4$  powder as a demetalling reagent; the former performed better in terms of lead reduction efficiency; whereas the latter produced less



sludge.

To determine the reproducibility of the demetallation experiment, Run 1 was duplicated by Run 7, and the results were essentially the same for both runs, confirming the accuracy of the experiment.

Since waste oil is derived from various sources, it is plausible that the lead removal efficiency using a demetalling reagent might respond differently to different types of waste oil. Thus, a different oil, viz., Type A oil (lead content = 660 ppm, ash content = 0.59%) was used in Runs 8-12.

In Run 8, the reaction conditions were almost the same as those in Run 1 only a different oil was used. The lead content of Type A oil was reduced by about 24% to 500 ppm; whereas for Type B oil the lead content decreased by 85% to 70 ppm (Table 3).

When the charge of SWS was doubled (Run 9), the lead content of Type A oil was reduced by about 58% (from 660 ppm to 280 ppm) as shown in Table 4. SWS contained 12 wt% sodium borohydride, which implied that the sodium borohydride concentration in the oil in Run 9 was 1,560 ppm. A sodium borohydride concentration of this level would not be economically feasible for reclaiming waste oil.

As mentioned earlier, the runs with caustic and SWS solution were characterized by high sludge production. A gel-like product was occasionally observed, which was difficult to filter, making the determination of sludge content very difficult. The gel formation was probably due to the presence of sodium hydroxide which acts to saponify fatty acids (e.g. detergents) present in the used oil. In order to inhibit the gel formation, sodium borohydride was added in a powder form (Runs 10 and 11) instead of in a solution; NaOH was still introduced as a 50% aqueous solution to maintain the activity of sodium borohydride.

In Run 10, 0.15 wt% (i.e., 1500 ppm)  $\text{NaBH}_4$  was added, which was approximately the same amount of  $\text{NaBH}_4$  added in Run 9; the lead reduction efficiency was slightly lower than that of Run 9. This was probably due to the greater solubility of  $\text{NaBH}_4$  in Run 9. In Run 11, a phase transfer catalyst (tri-n-butyl methyl ammonium chloride) was added, and the lead content of Type A oil was reduced by about 73% to 180 ppm.

When potassium borohydride ( $\text{KBH}_4$ ) was used in Run 12, the lead reduction was 59% (i.e., from 660 to 270 ppm). Thus the final lead content was still above the 100 ppm allowable maximum limit.

Examination of the ash content of the product oil (shown in Tables 2 and 4) reveals that, for both types of waste oil, the ash content of the product oil was not markedly decreased by metal borohydride treatment, although the lead content was selectively reduced in some cases. This might be attributed to the formation of other fine salts which could not be separated by regular filtration. A major disadvantage of using metal borohydrides as a demetalling reagent is that the pH must be kept high for the borohydrides to be stable, and this results in a very viscous (gel) product.

In the previous runs, the reaction temperature used was  $110^\circ\text{C}$ . When the reaction temperature was increased from  $110^\circ\text{C}$  to  $150^\circ\text{C}$ , keeping the other conditions the same as those used in Run 1, the product was a gel, and as a result, a poor lead reduction efficiency was obtained (Runs 13 and 14, Table 5).

*Investigation of Gel Formation* -- Several experiments were conducted in which a cotton seed oil or a non-detergent virgin motor oil was reacted with either SWS solution or  $\text{NaBH}_4$  powder to confirm that saponification does occur when fatty acids are present.

A cotton seed oil (20g) was blended with 1.2 wt% NaOH in 50% aqueous

solution, and then reacted with 0.65 wt% SWS in a TBMR (Run 15). The reaction was conducted under reaction conditions given in Table 1 (similar to those used for the TBMR demetallation runs). The reaction product was collected in a 2.5 cm O.D. x 15 cm length test tube. The sludge produced was about 35 volume % (Table 6). This sludge was probably the salt resulting from the saponification of fatty acids, in the cotton seed oil, with NaOH. The cotton seed oil was reacted with 0.05 wt%  $\text{NaBH}_4$  powder in the absence of sodium hydroxide (Run 16). In this case no sludge was formed, confirming that the sludge (gel) was due to the reaction of fatty acids with NaOH. The amount of  $\text{NaBH}_4$  was doubled, and still, there was no sludge formed (Run 17). The effect of NaOH on non-detergent motor oil was observed by reacting an SAE-30 non-detergent motor oil with SWS (Run 18). After the reaction, two layers of liquid were formed. The top layer was reddish brown and the bottom layer was bluish green in color. A trace amount of suspended solids was also observed. In summary, the gel product formed while treating used oil with borohydrides with NaOH present was probably due to the saponification of fatty acids.

*Effect of Caustic* -- In an attempt to reduce the amount of sludge production, two reactions (Runs 19 and 20, Table 7) were performed in which 1.5 wt%  $\text{NH}_4\text{OH}$  was added instead of NaOH, using the reaction conditions shown in Table 1. Also, to help stabilize the  $\text{NH}_4\text{OH}$ , 25 psig of  $\text{NH}_3$  was used (Run 19). As compared to a 0.8 wt% NaOH addition (Run 4), the amounts of lead reduction and sludge production were about the same.

*Effect of Water* -- As noted earlier, the reducing ability of metallic borohydrides can be hampered substantially by water hydrolysis. Since the waste oils obtained were used as-received (which had a water content of about 5 volume %), the poor performance for those runs with metallic borohydrides, in terms of lead reduction efficiency and ash content remaining in the oil,

may possibly be attributed to the presence of water during the reaction. To investigate the effect of water on lead removal, a sufficient amount of Type A oil (containing 5 vol % water) was blended with 0.25 wt% and 0.50 wt% NaOH solutions (50 wt% concentrated solution) respectively. Water along with some light ends were then distilled off via atmospheric distillation. The residual oils were termed as Type C and Type D, respectively. The properties of Type C & D oils are given in Table 8.

In Run 21, 20 g of dry Type C oil was reacted with 0.9 wt% of SWS solution in a TBMR under reaction conditions given in Table 1. As shown in Table 9, the lead content of the Type C oil was successfully reduced from 700 to 100 ppm; i.e. about 86% lead reduction was achieved. This suggests that a much better demetalling performance can be achieved with a dry oil (e.g., type C) than with a wet oil (e.g. Type A).

To further minimize the use of SWS solution, the charge of SWS solution was reduced to 0.4 wt% in Run 22. As a result, about 74% lead reduction was obtained.

In Runs 23 and 24, 0.1 wt% of a phase transfer catalyst (tri-n-butyl-methyl ammonium chloride) was added to see if the demetalling performance could be improved. Compared to run 21 (without catalyst), catalyst addition (e.g., Run 23) did not improve the results.

To study the effect of NaOH charge on lead reduction, Type D oil was used in Runs 25 and 26. The difference between Type C and Type D oil was the amount of NaOH in the oil; the latter had twice the amount of NaOH than the former (~1250 ppm). In comparison to Run 22, with the same charge of SWS solution, Run 26 gave a lower lead reduction efficiency.

On the whole, Table 9 shows that the presence of water in the oil has an adverse effect on demetallation when metallic borohydrides are used. This can

be avoided by distilling off the water before the demetallation reaction.

It should be noted that the results presented so far were obtained for a 45 ml tubing bomb microreactor (TBMR). Knowing that these bench-scale demetallation results were to be incorporated in the scale-up and design of the Auburn University Waste Oil Reprocessing Pilot Plant, experiments were extended to a larger batch reactor, i.e., a 3785 ml autoclave, to simulate actual operating conditions as much as possible.

As mentioned above, water can decrease the demetalling activity of metallic borohydrides. Therefore, the as-received Type A oil, containing about 5 vol.% of water, was blended with 0.25 wt% NaOH aqueous solution (50 wt%) in the following runs and then distilled to a specified temperature under atmospheric pressure. The residual oil was assumed to be moisture-free. The moisture-free oil (or dry oil) was then treated with metallic borohydride products in the one-gallon autoclave. The results are given in Table 10 and discussed below:

In Run AS1, 890 g of dry oil (lead content = 700 ppm) was reacted with 0.9 wt% of SWS solution in the 3785 ml autoclave using the reaction conditions shown in Table 11. For Run AS1, only a 37% lead reduction efficiency was obtained. The poor demetalling performance was probably due to the low distillation cut-off temperature (129°C), which resulted in a certain amount of water being left behind in the residual oil. In other words, the residual oil obtained in Run AS1 was not dry enough.

Thus, in Run AS2, the distillation cut-off temperature was further raised to 175°C. The oil and SWS solution charges to the autoclave were 900 g and 0.91 wt%, respectively, which were about the same as in Run AS1. With the same reaction conditions, the lead content of the oil was markedly reduced from 700 ppm to 25 ppm; i.e., a 96% lead reduction was obtained compared to 37% in Run AS1.

Although a promising result was obtained in Run AS2, the amount of SWS solution (containing 12 wt%  $\text{NaBH}_4$ ) used was considerable, and hence, uneconomical. Thus Run AS2 was duplicated by Run AS3 except that the SWS solution charge was halved. As a result, only a 40% lead reduction was obtained; this suggested that more SWS solution was required, if a better demetalling performance was to be achieved.

To compare the demetalling effectiveness of SWS solution and  $\text{NaBH}_4$  powder, Run AS4 was performed in which the same amount of  $\text{NaBH}_4$  was used as in Run AS1 (i.e. 0.10 wt%  $\text{NaBH}_4$  power) and 0.20 wt% phase transfer catalyst (tri-n-butyl-methyl ammonium chloride) were reacted with 545 g of dry oil in the autoclave. Coincidentally, the same level of lead reduction was obtained. However, with  $\text{NaBH}_4$  powder (Run AS4) the product oil had a lower ash content as compared to Run AS1 with SWS solution; also, less sludge was obtained in Run AS4.

To study the demetalling ability of  $\text{KBH}_4$  powder, Run AS4 was duplicated (Run AS5) except that  $\text{KBH}_4$  powder was used. As shown in Table 10, the lead reduction efficiency increased from 37% to 51%, but the ash content of the oil was not altered significantly.

Since  $\text{KBH}_4$  powder is more expensive than  $\text{NaBH}_4$  powder, Run AS4 was duplicated again in Run AS6, in which the  $\text{NaBH}_4$  charge was increased from 0.10 to 0.15 wt% while maintaining the rest of conditions the same as in Run AS4 to determine whether a better lead reduction efficiency could be obtained. It was found that the lead reduction efficiency increased from 37 to 71%, but the ash content of the product oil did not change appreciably, as shown in Table 10. Although the amount of  $\text{NaBH}_4$  used can be further increased to improve lead reduction performance, it is uneconomical to do so when the operating costs for reprocessing waste oil are considered. Therefore, another demetalling method was explored in an effort to seek the most economical process.

## *Use of Diammonium Phosphate As a Demetallation Reagent*

As previously described, the major problems in using metallic borohydrides as demetalling reagents were: (1) a soap-like sludge was usually formed after the reaction, which was very difficult to filter; (2) a caustic oil was produced (this could result in corrosion problems); and (3) the treated oil after filtration usually had a higher ash content than the original waste oil. Because of these problems, another demetallation process, which had been proposed by the Phillips Petroleum Company as a pretreatment step in converting waste oil into a lubricating oil was investigated [1,5]. In this process, waste oil was reacted with a demetalling reagent, diammonium phosphate (DAP) (both with and without the addition of excess water). Different types of reactors were used in this investigation to permit dominant mechanisms over different operational ranges to be identified.

In the bench scale experiments, three types of reactors were used: (1) A tubing bomb microreactor (TBMR) as described previously; (2) A 500 ml distillation flask equipped with a magnetic stirrer; and (3) A 3785 ml autoclave reactor. The TBMR was operated under closed conditions, whereas the distillation was done under open conditions; i.e. the flask was open to the atmosphere to distill off water and trace amounts of light components.

### *TBMR Reaction Studies*

*Effect of Reaction Time and Temperature* -- The variation in lead content of the DAP-treated oil with reaction time was observed as shown in Figure 2. The demetallation experiments were done at 150°C. From an initial value of about 210 ppm, the lead concentration of the product oil decreased with increasing reaction time to a final value of 16.5 ppm for the 90 minute run at an 800 cpm agitation rate. From this figure, it can also be deduced that agitation has a significant effect on the lead content of the oil. For example, for a 45 minute reaction time, when the agitation rate was decreased

from 800 to 400 cpm, the lead content of the treated oil increased from 70 to 125 ppm. An increase in agitation is thought to increase the interfacial contact area for reaction between the oil and aqueous reactants.

To investigate the effect of reaction time on other types of oil at various reaction temperatures, several experiments were performed in which 20g of Type B oil and 8 wt% DAP (in 40 wt% aqueous solution) were reacted in a tubing bomb microreactor at temperatures of 200°C and 250°C, for 15 to 60 minutes. The results are shown in Figure 3. With a reaction temperature of 200°C, the lead content of type B oil was reduced from 480 to 230 ppm for the 60 minutes run, i.e., a 52% lead reduction. As temperature was increased to 250°C, the lead content was further reduced to about 30 ppm (i.e. a 94% lead reduction). Consequently, it can be inferred from Figures 2 and 3 that either a higher reaction temperature or a longer reaction time or both facilitate oil demetallation, as seen in Figure 4.

*Distillation Flask Reaction Studies* -- Run DF1 was performed in which 60 g of Type A oil (lead content = 660 ppm, water content = 5%) and 8 wt% DAP powder were mixed and heated in the 300 ml distillation flask to 150°C and the reaction was allowed to continue for an additional hour. The heat-up time was about 70 min. During the reaction, water and trace amounts of light components were distilled off. A notable result was obtained in this run (DF1) i.e. the lead content of the waste oil was successfully decreased from 660 to 24 ppm (Table 12). In other words, a 96% lead reduction efficiency was obtained. More promisingly, the ash content of the oil was also reduced to 0.02 wt% which is much lower compared to that from metallic borohydride runs (~0.5 wt%).

One may speculate that the lead reduction obtained in Run DF1 was due to the high reaction temperature and good separation by filtration. Miller found that some part of lead can be removed just by filtration [5]. Run DF2 was



thus performed in which oil A alone, without adding any DAP, was heated in a distillation flask for the same period of time as in Run DF1. The result was that only a 3% lead reduction was obtained, indicating that DAP plays a very important role in demetallation. In addition, without DAP, the ash content of the waste oil was not altered appreciably. In an attempt to reduce the DAP charge, Run DF1 was duplicated by Run DF3 except that the amount of DAP used was cut from 8 to 2 wt%. Again, a similar lead reduction (97%) was obtained.

*Effect of Reactor Configuration on Lead Reduction* -- Table 12 shows that satisfactory demetallation results under mild conditions can be obtained using a distillation flask as a reactor. It should be noted that the reactor was open to the atmosphere. To study the effect of the reactor configuration on the demetallation performance, a tubing bomb microreactor (TBMR) was used as a closed system reactor. The mass transfer effect for this type of reactor was also studied. A detailed discussion of the experimental results is given below.

As shown in Table 12, with 2 wt% DAP, 97% lead reduction could be obtained (Run DF3). The reaction time for this run was 1 hr. To determine the effect of the reactor configuration on lead removal, Run D1 was performed in which 20 g of the same type of oil used in Run DF3 and 2 wt% DAP were charged to a TBMR and reacted for 1 hr under a closed system. Only a 70% lead reduction was obtained in Run D1 compared to 97% in Run DF3.

In Run D2, three steel balls (1/4" diameter) were added to the TBMR in order to increase the mixing effect and interfacial contact area while maintaining the rest of conditions the same as in Run D1. The lead reduction efficiency increased from 70 to 77%. When six steel balls were added in Run D3, the lead reduction efficiency increased further to 81%.

To study the effect of water presence on lead reduction, Run D3 was duplicated by D4 in which dry oil (obtained by distilling off water from Type

A oil) was used instead of wet oil (i.e. Type A oil). As shown in Table 13, the lead reduction efficiency decreased from 81 to 78%. This difference was attributed to a lower interfacial contact area available for the dry oil. It should be noted that this difference was small because of the excellent dispersion obtained in this type of reactor; a much larger difference was observed later, using a reactor (autoclave) that did not have as good particle dispersion properties.

The inconsistent results (Table 12 & 13) obtained from the two types of reactors (distillation flask and TBMR) may predominantly be due to the difference in heat-up time; i.e., for a 150°C demetallation run, the distillation flask had a much longer heat-up time (75 min) than the TBMR (1 min). In other words, some demetallation may have occurred during the heat-up.

To study heat-up effects, Run D5 was performed to duplicate Run 1 (see Table 14) except that the TBMR was preheated in the fluidized sand bath (see Figure 1) so that the total heat-up time from room temperature to 150°C was about 75 min. (It should be noted that in a typical TBMR run, the sand bath alone was preheated to 150°C before the TBMR was immersed; the heat-up time was then about 1 minute.) Table 14 shows that, with the same heat-up time as that for distillation (Table 12), a satisfactory result was obtained from Run D5; i.e. 91% lead reduction could be achieved from a TBMR run. In comparison with the lead reduction obtained from the distillation run, (91% versus 97% lead reduction), this result was acceptable for a reactor with a different configuration and mixing pattern.

As discussed above, with 75 minutes of preheating time and an additional hour for reaction (i.e. total reaction time, 2 hr), 91% lead reaction could be obtained in Run D5. It would be interesting to see what the lead reduction efficiency would be with a 2 hr reaction time, 160°C reaction temperature and

a regular TBMR 1 minute heat-up time. Run D8 was thus performed and the results given in Table 14 show that about the same level of lead reduction as Run D5 was obtained.

It was thus concluded that with a total reaction time of 2 hrs at 160°C, a desirable demetallation result could be achieved for various types of reactor configurations (distillation flask or tubing bomb microreactor). This agreement is mostly due to the fact that thermodynamic equilibrium (= complete lead removable) was achieved under these reaction conditions.

Since the distillation flask was used for the evaporation of water and light components, so that the hydroxyl phosphate droplets could shrink to facilitate removal of lead particulates, an open-type reactor such as a distillation flask (Run DF3) may be more suitable for oil demetallation as compared to a closed reactor, such as TBMR (Run D5).

To further evaluate the effectiveness of DAP demetallation with respect to the configuration of the reactor used (i.e., the importance of mass transfer), a certain amount of Type A oil (lead content = 660 ppm, water content = 5 vol%) and 1.5-2.0 wt% DAP were reacted in 45 ml, 300 ml, and 3,785 ml (i.e., 1 gallon) autoclaves, at 160°C respectively. The 45 ml microautoclave, as a matter of fact, was the TBMR (tubing bomb microreactor). The results are given in Table 15.

One should note the main difference between a TBMR and a regular autoclave in terms of agitation and heating; the former was vertically agitated; whereas the latter was horizontally stirred. The mass transfer rates in the TBMR are generally better than those in the 300 ml and 3,785 ml autoclaves [7,8]. Also, the heat-up time varied a great deal; for example, the heat-up time for a TBMR heated by a preheated fluidized sand bath was about 1 minute, whereas, for a 300 ml or a 3785 ml autoclave (heated by a electrical furnace from ambient temperature) the heat-up time was approximately 2 hours.

Table 15 shows that regardless of the reactor configuration, above 90 percent lead reduction was obtained using DAP as a demetalling reagent under mild reaction conditions for a reaction time of about 2 hours. The amount of solids produced was less than 1 wt%, excluding the DAP added. These observations implied that DAP is more suitable for used oil demetallation than sodium borohydride.

*Effect of Water* -- As described earlier, water present at the early stage of a demetallation reaction can facilitate lead removal by making more surface contact area available for the DAP.

A comparative study was carried out using a 1-gallon autoclave. The effect of water on ash and lead removal was investigated using the reaction conditions given in Table 16. Unlike the earlier studies with a TBMR (Table 13), the dispersion of the DAP was probably much less in this reactor (a stirred autoclave).

In Run AD1, with dry oil obtained by distilling off water from Type A oil, and 4 wt% DAP, a 74% lead reduction was obtained as shown in Table 17; the ash content of the product oil from this run was 0.310 wt%.

To discern the effect of water on the demetalling performance, Run AD1 was duplicated by AD2, except that a wet oil (i.e., Type A oil) was used. As a result, 99.7% lead reduction was achieved and the ash content of the product oil was very low (0.020 wt%). This observed increase in lead removal efficiency that resulted with the addition of water was significantly larger than that observed before with a TBMR and was felt to be due to the difference in the mass transfer characteristics of the reactors; namely, the degree to which the DAP was dispersed.

In Run AD3, the DAP charge was reduced from 4.0 to 1.5 wt% to reduce the operating cost and sludge generation and one part of the top of the autoclave was opened to vent water and the lights to facilitate lead removal. In so

doing, the lead reduction was 98.8% and ash content was 0.030 wt%. It should be noted that for all the DAP runs, the sludge generation, excluding the DAP added, was no more than 1 wt%. A duplicate run was done in AD4 and similar results were obtained as evidenced in Table 17.

Examination of the demetallation results (Runs AD1-AD4) for Type A oil as shown in Table 17 reveal that: 1) much better lead and ash removal could be achieved when used oil contained a certain amount of water (apparently, the water aids in dispersing the DAP during initial period of reaction); 2) total metal contaminant content (i.e. ash content) was successfully reduced to an acceptable level ( $< 0.1$  wt%); 3) a good quality of product oil could be produced with only 1.5 wt% diammonium phosphate (DAP) under mild reaction conditions; and 4) reproducibility of the DAP demetallation experiment seemed to be acceptable.

Other types of used oils (Type B & E) were also investigated using the same operating conditions as Run AD3. The lead contents for Type B and Type E oils were 480 ppm and 180 ppm, respectively and the ash contents were 0.52 wt% and 0.65 wt%, respectively.

For Type B oil, a 96% lead reduction was obtained in Run AD5 while a 99% lead reduction was obtained for Type E oil in Run AD6. The ash content of the product oil from both the runs was very low. Table 17 also shows that with 1.5 wt% DAP treatment, used oil (e.g. Type A, B, or E) seemed to be fairly well demetallated.

*Liquid-Solid Separation Studies* -- The objective of these studies was to evaluate the responses in terms of separation ease to the addition of filter aid and the use of a diluent (No. 2 fuel oil). As has been done throughout this work, both gravity sedimentation and filtration were studied for removing solids.

Two DAP demetallation runs (Runs AD8 and AD9) were performed in a 3785 ml

autoclave to study the effects of filter aid and diluent on the settling time and filtration rates. The response of ash and lead content of the DAP-treated oil to the settling time were obtained. The effects of dilution with No. 2 fuel oil on settling time and filtration rate were also studied. In Run AD8, 1 wt% filter aid (diatomaceous earth) was added to the reactor with 700 g of Type A oil and 2 wt% DAP before the DAP reaction, whereas, in Run AD9, the same amount of the filter aid was added after the DAP reaction.

Table 18 shows that an oil of better quality was obtained by adding 1 wt% filter aid before the DAP demetallation (e.g. Run 8) if gravity sedimentation was adopted; for example, in Run AD8, after 24 hour of settling at 60°C without any diluents, the lead content of the product oil (i.e., top-layer oil) was greatly reduced from 660 to 20 ppm (i.e., 97% lead reduction), whereas, in Run 9, with the addition of 1 wt% filter aid after the DAP reaction, the lead content of the product oil was reduced from 660 to 46 ppm (i.e., 93% lead reduction). A higher ash content for the product oil was also obtained when the filter aid was added after the reaction (e.g., Run AD9) as seen in Table 18. When filtration was used, both runs produced a similar oil quality. In comparison with gravity sedimentation, the solids removal efficiency with filtration was higher. However, with gravity sedimentation, the product oil had acceptable ash and lead contents, and this method does have its advantages in lower operation and maintenance costs.

With filtration a good solids-removing efficiency was obtained; however the filtration rates were very low for DAP-treated used oil. In order to evaluate the use of a diluent (No. 2 fuel oil) to improve filtration rates, another DAP demetallation run was carried out in a 1000 ml distillation flask under the reaction conditions shown in Table 12 (Run DF3). After the reaction, the reaction product including spent DAP was evenly divided into four fractions; a specified amount of filter aid or No. 2 fuel oil or both

were thoroughly blended in three of the four fractions. Viscosity of each fraction was first measured using a Cannon-Manning viscometer before filtration (using the house vacuum). The filtration rate for each fraction was determined, and so was the viscosity of the filtrate from each fraction. The results showed that the viscosity of the product oil drastically decreased from 90 to 33 cP at 25°C when diluted with 20% No. 2 fuel oil by weight of total liquid as shown in Table 19 (No. 2 fuel oil has a viscosity of 3.5 cP at 25°C). The filtration rate, after dilution, approximately doubled (from 11.5 to 25.0 ml/min). With the addition of 1% by weight of the total liquid, the filtration rate was further increased from 25.0 to 34.0 cm<sup>3</sup>/min; however, the viscosity of the resulting liquid increased from 32.8 to 38.4 cP due to the presence of extra solids (i.e., filter aid).

#### Conclusion

Used oil treated with NaBH<sub>4</sub> aqueous solution (i.e., SWS solution) was characterized by high sludge production and ash content. High sludge production could be circumvented by substituting SWS solution with NaBH<sub>4</sub> or KBH<sub>4</sub> powder. However, the lead reduction efficiency decreased. Demetallation performance with metal borohydride treatment could be greatly improved by distilling off water contained in the used oil before the reaction.

As for DAP demetallation, water present (about 5 wt%) during the early stages of the demetallation reaction was found to be beneficial. This beneficial effect was felt to be due to the additional interfacial contact area available for DAP particles to react with oil. Overall, mass transfer (DAP dispersion) was observed to be important; however, by extending the reaction time ( $\approx$  2 hr), even with mild reaction conditions, thermodynamic equilibrium ( $\approx$  100% lead removal) could be approached, thereby minimizing the significance of any mass transfer related parameters.

In liquid/solid separation, a better quality of oil was obtained by

filtration as compared to gravity sedimentation. (Nevertheless, an EPA specification-grade oil was produced using gravity sedimentation.) The oil filtration rate was doubled by dilution with 20 wt% of No. 2 fuel oil.

#### Acknowledgements

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Table 1--Reaction conditions for typical TBMR runs

Demetallation reagent	Metallic Borohydrides	DAP
Reaction temperature, °C	110, 150	150-160
Heat-up time, sec	60	60
Reaction time, hr	2.0	0.5-2.0
Agitation rate, cpm	860	860

Table 2--Demetallation results using metal borohydrides (for type B oil)

<u>Run No.</u>	1	7	2	3	4	5	6
.50% NaOH Added to the Oil <sup>a</sup> , wt%	1.2	1.2	0.8	0	0.8	0	0.8
SWS, wt%	0.65	0.65	0.43	0	0	0	0
NaBH <sub>4</sub> Powder, wt%	0	0	0	0.05	0.05	0.05	0.05
Phase Transfer Catalyst <sup>b</sup> , wt%	0	0	0	0	0	0.1	0.1
<u>After Reaction:</u>							
Sludge Production, wt%	16	16	15	2	-	6	7
<u>Product Oil Analysis:</u>							
Ash Content, wt%	0.36	0.36	0.58	-	0.77	0.40	0.53
Lead Content, ppm	70	69	240	430	300	350	330
Lead Reduction, %	85.4	85.6	50.0	10.4	37.5	27.1	31.3

<sup>a</sup>Oil charge - 20g Oil Type: B (lead content = 480 ppm, ash content = 0.52 wt% water content = 1.8 vol%).

<sup>b</sup>Tri-n-butyl-methyl ammonium chloride.

Table 3--Effect of oil type on demetallation

Run No.	1	8
Oil Type	B	A
Lead Content, ppm	480	660
Oil Charge, g	20	20
5% NaOH Added to the Oil, wt%	1.2	1.2
SWS, wt%	0.65	0.65

Product Oil Analysis:

Ash Content, wt%	0.36	0.57
Lead Content, ppm	70	500
Lead Reduction, %	85.4	24.2

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Table 4--Demetallation results using metal borohydrides (for type A oil)

Run No.	8	9	10	11	12
50% NaOH. Added to the oil <sup>a</sup> , wt%	1.2	1.2	1.8	1.8	1.8
SWS, wt%	0.65	1.3	0	0	0
KBH <sub>4</sub> Powder, wt%	0	0	0	0	0.15
NaBH <sub>4</sub> Powder, wt%	0	0	0.15	0.15	0
Phase Transfer Catalyst <sup>b</sup> , wt%	0	0	0	0.10	0.35

Product Oil Analysis:

Ash Content, wt%	-	0.62	0.56	0.50	0.68
Lead Content, ppm	500	280	300	180	270
Lead Reduction, %	24.2	57.6	54.5	72.7	59.1

<sup>a</sup>Oil charge - 20 g Oil Type: B (lead content - 660 ppm, ash content - 0.59%, water content - 5 volume %).

<sup>b</sup>Tri-n-butyl-methyl ammonium chloride.

Table 5--Effect of reaction temperature on sludge production

Run No.	1	13	14
Oil Type	B	B	B
Reaction Temp., °C	110	150	150
50% NaOH, wt%	1.2	1.2	1.2
SWS in the oil, wt%	0.65	0.65	0.65
<u>After Reaction:</u>			
Sludge Production, wt%	15.5	100% (Gel)	100% (Gel)
<u>Product Oil Analysis:</u>			
Lead Content, ppm	70	400	-
Lead Reduction, %	85.4	16.7	-

Table 6--Effect of caustic solution on gel formation

Run No. Oil Type	15 Cotton Seed Oil <sup>a</sup>	16 Cotton Seed Oil	17 Cotton Seed Oil	18 Motor Oil <sup>b</sup>
50% NaOH in the oil, wt%	1.2	0	0	1.2
SWS, wt%	0.65	0	0	0.65
NaBH <sub>4</sub> Powder, wt%	0	0.05	0.10	0
<u>After Reaction:</u>				
Sludge Production, Vol%	35	0	0	trace <sup>c</sup>

<sup>a</sup>Made from winterized cotton seed oil with TBHQ and citric acid in propylene glycol to retard oxidation with dimethylpolysiloxane added as an antifoaming agent. Made by Lou Ana Foods, Inc., Opelousas, LA 70570.

<sup>b</sup>GRC, G-100 Non-Detergent Motor Oil, SAE-30, made from Gurley Refining Company, Memphis, Tennessee 38101.

<sup>c</sup>Two phases were formed with the top layer in reddish-brown and the bottom layer in dark bluish-green with trace amount of suspended particle.

Table 7--Effect of  $\text{NH}_4\text{OH}$  on sludge production

Run No.	4	19	20
50% NaOH, wt%	0.8	0	0
$\text{NH}_4\text{OH}$ , wt%	0	1.5	1.5
$\text{NaBH}_4$ Powder, wt%	0.05	0.05	0.05
$\text{NH}_3$ , psig	0	25	0
<u>Before Reaction:</u>			
Lead Content, ppm	480		
Ash Content, wt%	0.52		
<u>After Reaction:</u>			
Sludge Production, wt%	4.3	3.4	3.2
<u>Product Oil Analysis:</u>			
Ash Content, wt%	0.77	0.31	0.37
Lead Content, ppm	300	260	400
Lead Reduction, %	37.5	45.8	16.7

Note:

Oil type: B  
Oil charge: 20 g  
Reactor: 45 ml TBMR  
Agitation: 860 cpm



Table 8--*Properties of type oils used*

Oil Type	A	B	C	D	E
Water content, vol.%	5.0	1.8	0	0	0.8
Ash content, wt.%	0.59	0.52	0.75	1.02	0.65
Lead Content, ppm	660	480	700	640	180

Table 9--Effect of water on demetallation with SWS

Run no.	9	21	22	23	24	25	26
Oil <sup>a</sup> type	A	C	C	C	C	D	D
50% NaOH, wt%	1.2	-	-	-	-	-	-
SWS, wt%	1.3	0.90	0.40	0.90	0.25	0.35	0.40
Phase transfer catalyst <sup>b</sup> , wt%	0	0	0	0.1	0.1	0	0

Product Oil<sup>c</sup> Analysis:

Ash content, wt%	0.62	0.53	0.68	0.63	0.77	0.93	0.69
Lead content, ppm	280	100	180	120	300	280	220
Lead reduction, %	57.6	85.7	74.3	82.9	57.1	56.3	65.6

<sup>a</sup>Oil charge = 20 g

<sup>b</sup>Tri-n-butyl-methyl ammonium chloride.

<sup>c</sup>Sludge production in all the runs was high (about 15-20 wt%).

Table 10--Results from autoclave (3785 ml) runs using metallic borohydrides

Run No.	AS1	AS2	AS3	AS4	AS5	AS6
<u>Atmospheric Distillation Conditions</u> <u>used for oil preparation:</u>						
Type A <sup>a</sup> oil charge, g	890	1000	1000	600	600	600
50% NaOH, wt%	0.25	0.25	0.25	0.25	0.25	0.25
Distillation cut point, °C	129	175	175	150	150	150
<u>Reaction Mixture:</u>						
Residue <sup>b</sup> from distillation, g	890	900	900	545	545	545
SWS, wt%	0.90	0.91	0.46	0	0	0
NaBH <sub>4</sub> Powder, wt%	0	0	0	0.10	0	0.15
KBH <sub>4</sub> Powder, wt%	0	0	0	0	0.10	0
Phase transfer catalyst <sup>c</sup> , wt%	0	0	0	0.20	0.20	0.20
<u>Product Oil<sup>d</sup> Analysis:</u>						
Ash content, wt%	0.74	0.50	0.51	0.57	0.56	0.53
Lead content, wt%	440	25	420	440	345	200
Lead reduction, %	37.1	96.4	40.0	37.1	50.7	71.4

<sup>a</sup>Water content = 5.0%  
 Lead content = 660 ppm  
 Ash content = 0.59%

<sup>b</sup>Lead content = 700 ppm  
 Ash content = 0.86%

<sup>c</sup>tri-n-butyl-methyl ammonium chloride

<sup>d</sup>Sludge production in all the runs was high (about 15-20 wt%).

Table 11--Reaction conditions for autoclave (3785 ml) runs

Demetallation Reagent	Metallic Borohydrides	DAP
Reaction Temperature, °C	110	160
Heat-Up Time, hr	1.5	2
Reaction Time, hr	2	1
Agitation Rate, rpm	2000	2000

Table 12--Demetallation study with DAP using distillation flask

<u>Run No.</u>	DF2	DF3	DF1
Type A Oil <sup>a</sup> charge, g	60	60	60
Water content in the oil, vol %	5	5	5
Percent DAP added	0	2	8

Product Oil<sup>b</sup> Analyses

Lead Content, ppm	640	18	24
Lead reduction, %	3	97	96
Ash content, wt%	0.47	0.06	0.02

<sup>a</sup>Type A oil:

Lead Content - 660 ppm

Ash Content - 0.59 % (no filtration)

Water Content - 5 vol%

<sup>b</sup>Reaction Conditions:

Reaction temp, °C 150

Heat-up time, min 75

Reaction time, hr 1

Reaction Pressure - Atmospheric

<sup>c</sup>Less than 1 wt% solids (excluding DAP added) was produced in all the runs.

Table 13--Effect of DAP dispersion (steel ball addition) for TBMR runs

<u>Run No.</u>	D1	D2	D3	D4
No. of Steel Balls Added	0	3	6	6 <sup>a</sup>
<u>Product Oil<sup>b</sup> Analyses:</u>				
Lead Content, ppm	200	153	127	156
Lead Reduction, %	70	77	81	78

TBMR Reaction Conditions:

Reaction Temperature	150°C
Reaction Time	1
Agitation	780 cpm
Type A oil charge	20 g
DAP added	2 wt%

<sup>a</sup>Dry Oil (lead content = 700 ppm)

<sup>b</sup>Less than 1 wt% solids (excluding DAP added) was produced in all the runs.

Table 14--Effect of heating time and reaction temperature on demetallation

Run No.	D1	D5	D8	D9 <sup>a</sup>	D10
<u>TBMR Reaction Conditions<sup>b</sup>:</u>					
Reaction Temp, °C	150	150	160	160	200
Heat-Up Time, min	1	75	1	1	1
Reaction Time, hr	1	1	2	2	1
Agitation, cpm	800	800	800	800	800
<u>Product Oil<sup>c</sup> Analyses:</u>					
Lead Content, ppm	200	62	64	81	76
Lead Reduction, %	70	91	90	88	88

<sup>a</sup>Dry Oil (lead content = 700 ppm) was used.

<sup>b</sup>Oil type: A  
DAP charge: 2 wt%  
No. of steel balls used: 0

<sup>c</sup>Less than 1 wt% solids (excluding DAP added) was produced in all the runs.

Table 15--Effect of reactor configuration on demetallation

<u>Run No.</u>	D8	AD7	AD2
Autoclave Size, ml	45	300	3,785 (1 gal)
Oil Charge <sup>a</sup> , g	20	60	554
DAP Added, wt%	2	2	1.5
<u>Reaction Conditions:</u>			
Reaction Temp, °C	160	160	160
Heat-Up Time, hr	0	2	2
Reaction Time, hr	2	1	1
Reaction Pressure, psi	N/A	150	atmosphere
Agitation, rpm	800	2000	2000
<u>Product Oil<sup>b</sup> Analysis:</u>			
Lead Content, ppm	64	21	2
Lead Reduction, %	90.0	96.8	99.7

<sup>a</sup>Oil used: type A

<sup>b</sup>Less than 1 wt% solids (excluding DAP added) was produced in all the runs.



Table 16--Reaction conditions for autoclave (3785 ml) run using DAP

Temperature	- 160°C
Heat-Up Time	- 2 hr
Time	- 1 hr
Agitation	- 2000 rpm

Table 17--Demetallation with DAP using autoclave (3785 ml)

<u>Run No</u> <sup>a</sup>	AD1	AD2	AD3	AD4	AD5	AD6	AD7
Oil Type	A <sup>b</sup>	A	A	A	B	E	A <sup>b</sup>
Oil Charge, g	541	554	554	554	554	554	560
Percent DAP Added, %	4.0	4.0	1.5	1.5	1.5	1.5	1.5
<u>Product Oil</u> <sup>c</sup> Analyses:							
Lead Content, ppm	180	0	0	2	18	2	580
Lead Reduction, %	74.3	100	100	99.7	96.3	98.9	17.1
Ash Content, wt%	0.310	0.020	0.030	0.026	0.016	0.036	0.464

<sup>a</sup>Runs AD1 And AD2: The autoclave was completely sealed.

Runs AD3-7: One port of the autoclave was intentionally opened  
to allow the water vapor to escape in an effort to  
simulate the distillation process.

<sup>b</sup>Dry Oil: Lead Content = 700 ppm

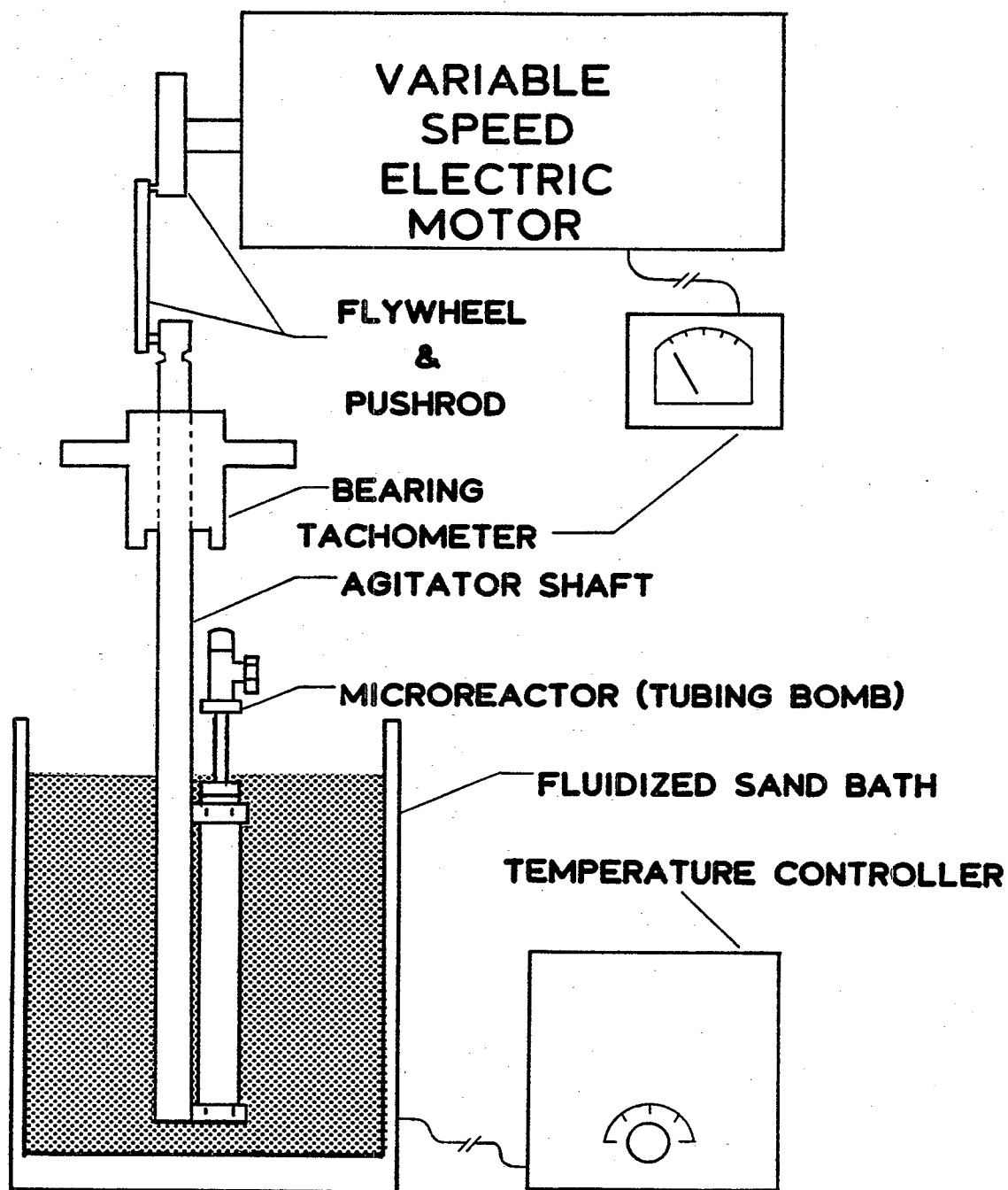
<sup>c</sup>Less than 1 wt% solids (exlcuding DAP added) was produced in all runs

Table 18--Effect of filter aid and/or diluent on oil quality

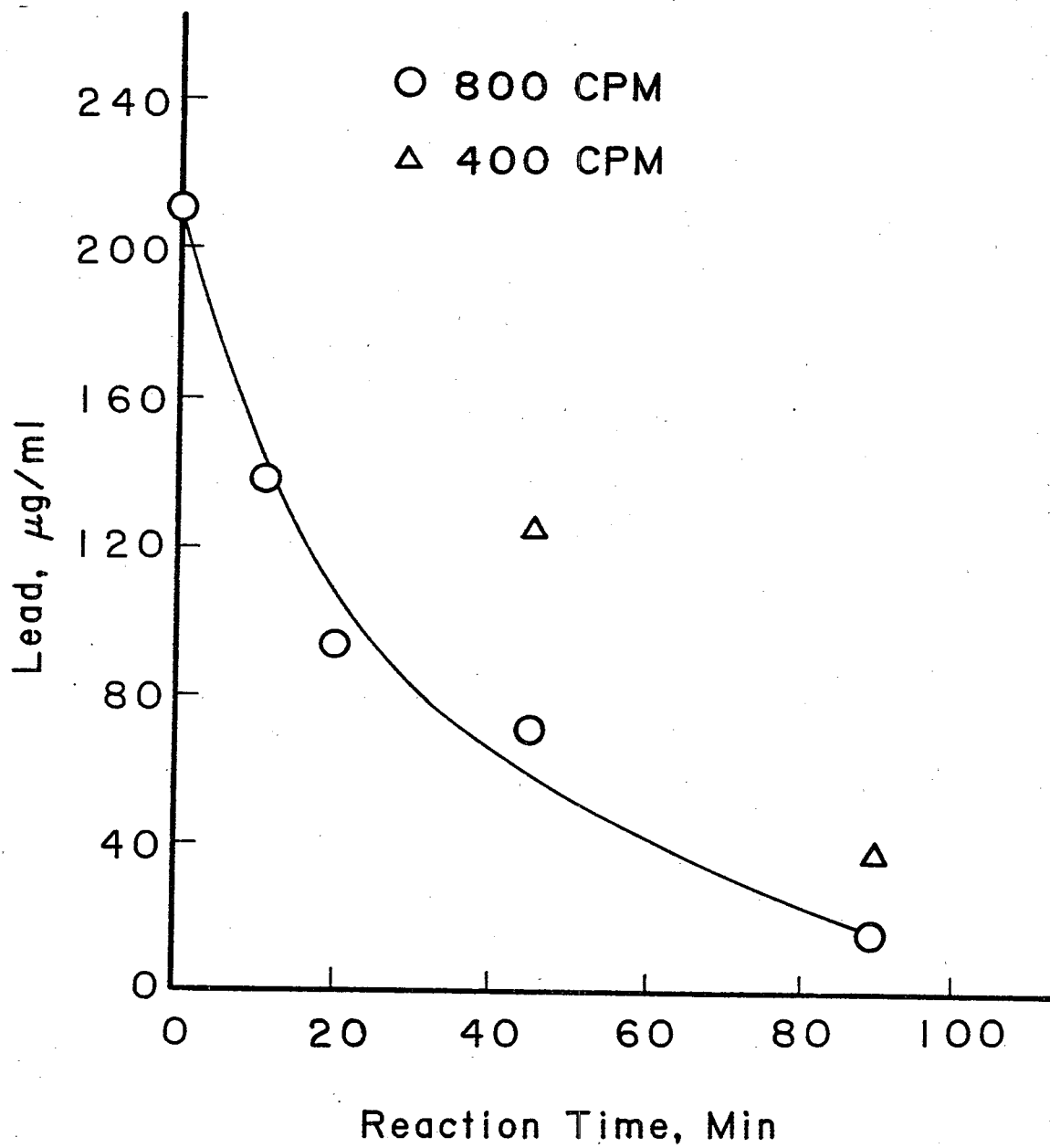
Run No.	AD 8		AD 9	
I.- Sedimentation in 60°C: Water bath for 24 hr	Ash Content, wt%	Pb Content, ppm	Ash Content, wt%	Pb Content, ppm
Oil only			0.066	52
Oil+1 wt% filter aid	0.046	20	0.124	46
Oil+20 wt% No. 2 fuel oil			0.050	36
Oil+1 wt% filter aid+ 20 wt% No. 2 oil	0.012	10	0.066	32
II. Vacuum Filtration:				
Oil only			0.012	4
Oil+1 wt% filter aid	0.012	0	0.014	6
Oil+20 wt% No. 2 fuel oil			0.004	2
Oil+1 wt% filter aid+20 wt% No. 2 fuel oil	0.008	0	0.002	4

Table 19--Effect of filter aid and diluent on  
filtration rate and oil viscosity

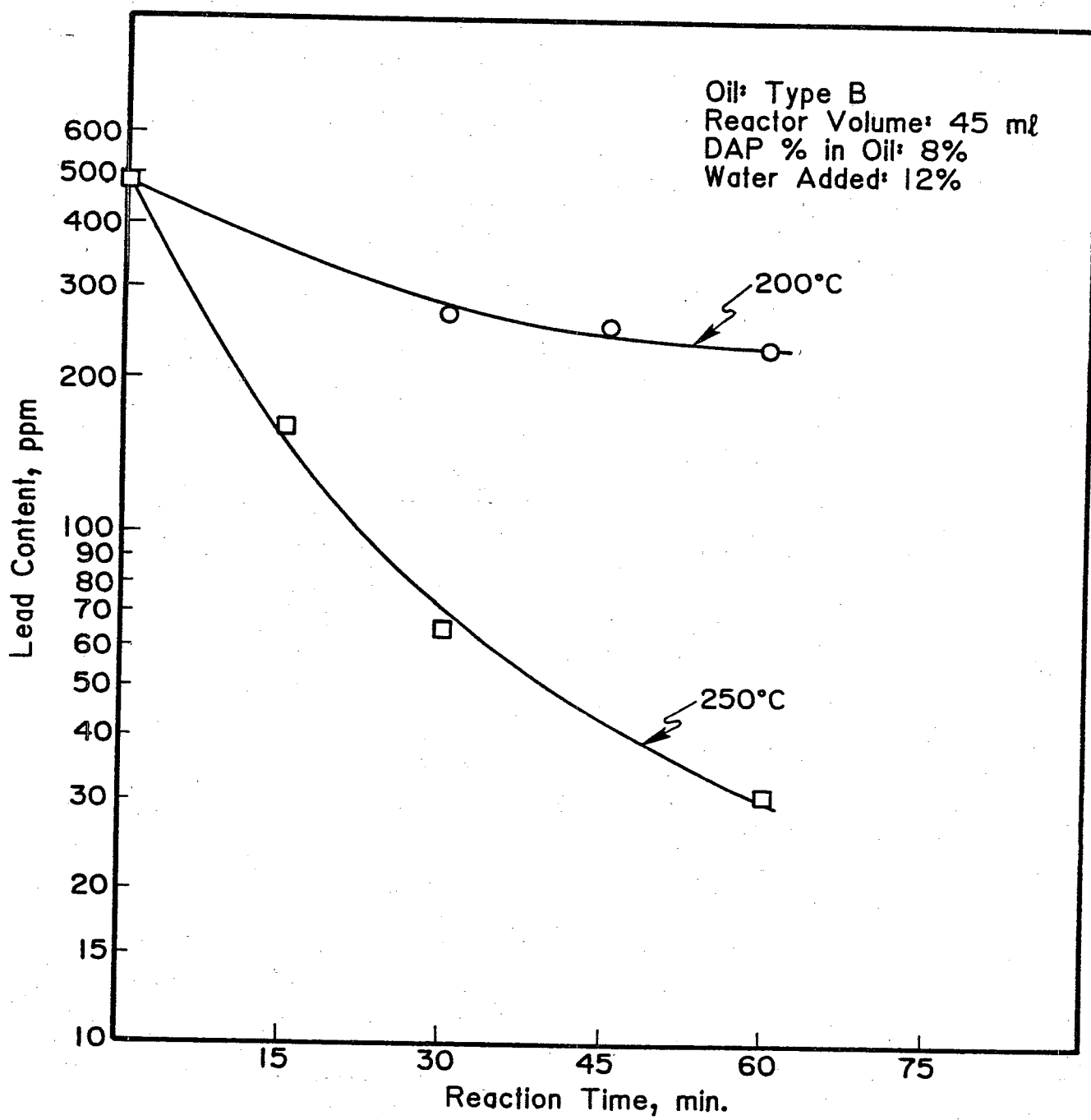
	Viscosity at 25°C (cP)	Filtration Rate (ml/min)	Filtrate Viscosity at 25°C (cP)
Oil Only	89.9	11.5	87.7
Oil + 1 wt% Filter Aid	95.5	12.0	87.3
Oil + 20 wt% No. 2 Fuel Oil	32.8	25.0	37.3
Oil + 20 wt% No. 2 Fuel Oil + 1 wt% Filter Oil	38.4	34.0	37.6



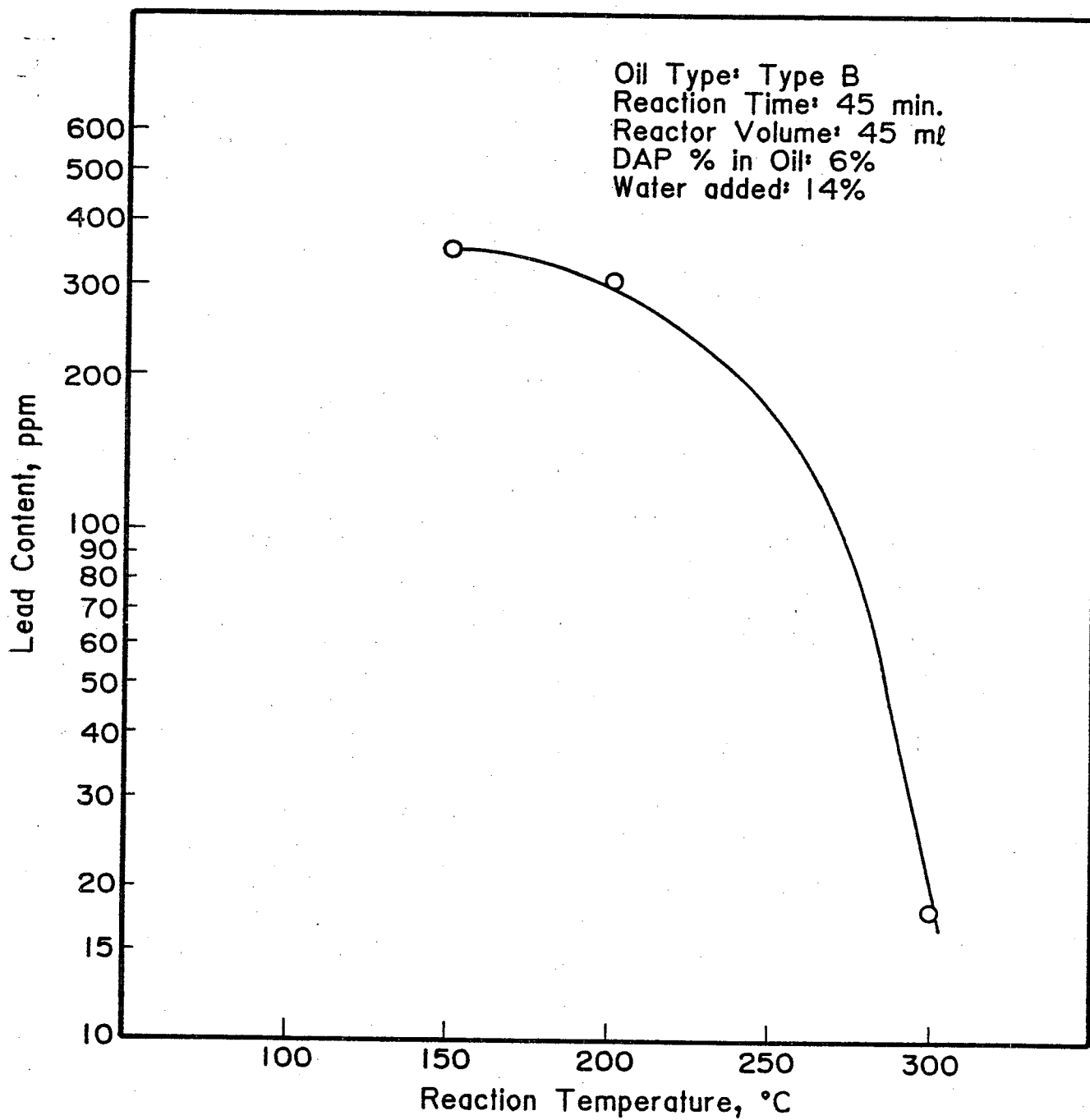
Schematic diagram of tubing bomb microreactor agitation assembly.



Effect of reaction time and agitation rate on lead removal.



Effect of reaction time on lead removal at various temperatures.



Effect of reaction temperature on lead removal for type B oil.