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FUEL CELL FUEL STUDIES

Final Technical Report

by

William J. Asher
Carl E. Heath

December 1968

U. S. Army Mobility Equipment Research and Development Center
Fort Belvoir, Virginia

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13. ABSTRACT

The objective of this contract (DA-44-009-AMC-1484[T]) was to develop a means for sulfur removal from military fuel, such as JP-4, so the purified fuel could be used in reformer fuel cells. It was preferable that the purification processing be adaptable to minaturization so that each fuel cell could have a purifier. Work has centered on processing with a discardable chemical sulfur adsorbent and cyclic adsorption on molecular sieves using either ammonia or the new Hybrid desorption to cyclically remove the impurities from the sieve. The chemical adsorbent could be used either by itself or as a final clean-up treatment for the sieve product.

The Esso proprietary chemical adsorbent was operable without the complication of water or hydrogen introduction with the feed found necessary by previous workers (2). However, the sulfur capacity was no better than obtained in the earlier study.

In processing using cyclic adsorption on molecular sieves, ammonia desorption proved effective over a wide variety of processing requirements. However, it was not effective processing high disulfide sulfur content feeds. The disulfide caused a deposit buildup on the sieve, which rapidly destroyed adsorption performance. Only removal of disulfides with separate processing would make the ammonia desorption operable with all feeds. This complication

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is not recommended. Disulfides are generally not found in the fuel types studied but they do occur in fuels from refineries where older treating processes are used. Because of this disadvantage, the alternate technique of Hybrid desorption would be favored.

The new Hybrid desorption is a simultaneous carbon dioxide displacement and burning with air of the impurities adsorbed on molecular sieves. Although considerable difficulty was encountered in finding a molecular sieve stable with the necessary carbon dioxide-water mixture between 700 and 950°F, $Ba^{+2}Y$ has been demonstrated stable for 417 hours and is projected to be effective for at least 1000 hours. The necessary temperature rise of all of the sieve charge when the impurities are being burned off was achieved using a thin wall adiabatic sieve case. Successful operation was demonstrated on feeds containing up to the maximum allowable 4000 ppm sulfur. Sulfur levels of all feeds were reduced to 50 ppm or below. This low level can be removed with a small quantity of chemical adsorbent. A unit design for operating 1000 hours with the maximum allowable sulfur content (4000 ppm) was made using the preferred combination of molecular sieves with Hybrid desorption followed by a small quantity of chemical adsorbent. The purified fuel yield was a good 92 weight percent and the projected weight about 10 pounds per kilowatt of fuel cell power. This weight is a substantial improvement compared to 330 pounds per kilowatt required for the same purification using only the chemical adsorbent of the earlier study (2,9). The Hybrid desorption runs made in this study were intended to demonstrate the concept. It is felt that considerable room for optimization and weight reduction remains.

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FINAL TECHNICAL REPORT

Period Covered 10 January 1966 - 9 November 1968

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William J. Asher
Carl E. Heath

December 1968

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Fort Belvoir, Virginia

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FOREWORD

These studies were authorized by the U. S. Army Mobility Equipment Research and Development Center. This work was done under contract DA-44-009-AMC-1484(T). The DA Project/Task/Work Unit Number of this work is 1T662705A012 02 016EF. This report covers the work of the entire contract period 10 January 1966 through 9 November 1968. The work in the fifth report period - 10 January 1968 through 9 November 1968 - is reported in detail. The entire study was aimed at the development of a process for purifying standard military fuel for use in hydrocarbon consuming fuel cells.

The authors acknowledge the skilled work of John F. Phillips, and John J. Mulligan, Senior Research Technicians, in conducting the experimental programs.

SUMMARY

The objective of this contract (DA-44-009-AMC-1484[T]) was to develop a means for sulfur removal from military fuel, such as JP-4, so the purified fuel could be used in reformer fuel cells. It was preferable that the purification processing be adaptable to minaturization so that each fuel cell could have a purifier. Work has centered on processing with a discardable chemical sulfur adsorbent and cyclic adsorption on molecular sieves using either ammonia or the new Hybrid desorption to cyclically remove the impurities from the sieve. The chemical adsorbent could be used either by itself or as a final clean-up treatment for the sieve product.

The Esso proprietary chemical adsorbent was operable without the complication of water or hydrogen introduction with the feed found necessary by previous workers (2). However, the sulfur capacity was no better than obtained in the earlier study.

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The new Hybrid desorption is a simultaneous carbon dioxide displacement and burning with air of the impurities adsorbed on molecular sieves. Although considerable difficulty was encountered in finding a molecular sieve stable with the necessary carbon dioxide-water mixture between 700 and 950°F, $Ba^{+2}Y$ has been demonstrated stable for 417 hours and is projected to be effective for at least 1000 hours. The necessary temperature rise of all of the sieve charge when the impurities are being burned off was achieved using a thin wall, adiabatic sieve case. Successful operation was demonstrated on feeds containing up to the maximum allowable 4000 ppm sulfur. Sulfur levels of all feeds were reduced to 50 ppm or below. This low level can be removed with a small quantity of chemical adsorbent. A unit design for operating 1000 hours with the maximum allowable sulfur content (4000 ppm) was made using the preferred combination of molecular sieves with hybrid desorption followed by a small chemical adsorbent. The purified fuel yield was a high 92 weight percent of the total fuel. The purifier projected weight was about 10 pounds per kilowatt of fuel cell power. This weight is a substantial improvement compared to 330 pounds per kilowatt required for the same degree of purification using only the chemical adsorbent of the earlier study (2,9). The hybrid desorption runs made in this study were intended to demonstrate the concept. It is felt that considerable room for optimization and weight reduction remains.

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FUEL CELL FUEL PURIFICATION

1. Introduction

The objective of this contract was to purify military fuels (such as JP-4 fuel) for use in fuel cells. Initially the purification was to be done for direct hydrocarbon fuel cells in preference to indirect, reforming fuel cells. It is highly desirable that the purifying processes for either type of fuel cell be adaptable to miniaturization. This would be essential if each fuel cell were to have a fuel purifying unit attached. Purifying for the direct hydrocarbon fuel cell is a difficult task as many things have to be removed. Aromatics, naphthenes, olefins, and sulfur compounds have all been found to decrease the performance of some direct hydrocarbon fuel cells. Purification would probably require three processes; catalytic dehydrogenation of naphthenes to aromatics, cyclic molecular sieve adsorption to remove the aromatics and most of the sulfur, and a final chemical adsorbent for removing a small quantity of sulfur in the adsorption effluent. In contrast, purification for a reformer fuel cell is simpler as only the sulfur compounds have to be removed. This would probably require only two processes; a cyclic molecular sieve adsorption followed by a final chemical adsorbent.

The Energy Conversion Research Division of U.S. Army Mobility Equipment Research and Development Center, Fort Belvior, Virginia monitored the progress of this program as well as the competitive position of the direct hydrocarbon fuel cell vs. the reforming fuel cell. After this program was initiated, the performance of the reforming fuel cell improved to a level better than the direct hydrocarbon fuel cell. This combined with the necessary 60 to 75 percent yield loss on removal of the naphthene and aromatics (1) for the direct hydrocarbon fuel cell lead The Energy Conversion Research Division to change the objective of this study to purification for reforming fuel cells. Therefore, work in the later portion of this contract has been directed at sulfur removal.

Cyclic adsorption on molecular sieves is the key process used in this study. Single adsorptions on chemical adsorbents followed by discarding the adsorbent have been demonstrated in another study (2). The advantage of molecular sieve processing is that the adsorbent is cyclically reused after each desorption. As the adsorption and desorption steps are typically short (10 to 30 minutes each) the molecular sieve would be used many times in 1000 hours of operation. Thus, even with a lower sulfur loading for each adsorption, much less adsorbent is required. This reduction in the weight of adsorbent required in comparison to single adsorption on a chemical adsorbent is the primary advantage of cyclic adsorption. The demonstrated chemical adsorbent (2) handling the military fuel with the maximum allowed sulfur content (0.4 weight percent) for 1000 hours would require 330 pounds of chemical adsorbent per kilowatt of power. Thus, the potential for weight reduction is large.

Although the substitution of a cyclic molecular sieve process for the single step chemical adsorbent would reduce the weight, the optimum processing would probably consist of a cyclic molecular process followed by a very small chemical adsorbent unit. Operation of a molecular sieve unit to reduce the highest allowable sulfur content fuel (4000 wt. ppm) down to less than 10 ppm should be possible; however as the operation conditions are made more severe to achieve this 400 to 1 reduction factor the yield of purified fuel goes down. A better balance of yield and weight would probably be obtained by operating the molecular sieve process to sufficiently remove the sulfur from most feeds, and for very high sulfur content feeds allow a very small chemical adsorption unit to remove the small fraction of feed sulfur in the molecular sieve product. Toward this end of combining the cyclic molecular sieve process with a chemical adsorbent, a proprietary chemical adsorbent, that showed initial sulfur capacity in excess of those previously demonstrated (2), was tested in this reporting period.

The major effort throughout the program has been directed at the development of the key cyclic molecular sieve adsorption process. The first objective of the program was the development of molecular sieves with good adsorption characteristics for both aromatic and sulfur compounds. Many different cationic forms of the anionic X and Y type molecular sieve frameworks were prepared and tested. Several were found which had partially good adsorption characteristics (1). Therefore, throughout most of the program the adsorption on molecular sieves was not a problem. The effective and efficient desorption of the adsorbed impurities has been the problem to overcome.

The desorption of the molecular sieves has been conducted using two basic methods:

- Ammonia displacement
- Hybrid desorption

Ammonia displacement functions by the ammonia being passed over the molecular sieve and adsorbing on the sieve, replacing the previously adsorbed impurities which are then swept out of the bed. After this desorption step the impurity containing feed is reintroduced for the next adsorption step. The impurities are then adsorbed replacing the ammonia which goes out the exit of the bed with purified fuel. Esso Research and Engineering Company has considerable experience, both laboratory and commercial, with this ammonia displacement cycle (3,4). This, of course, means that many of the general problems for this specific application had been solved before this program was initiated. The ammonia displacement does however have two disadvantages. An inventory of the recirculated ammonia must be carried with the processing unit and small amounts of additional ammonia must be added at maintenance intervals to make up for losses. Secondly, there is always the possibility of detection of the unit's location if a malfunction releases ammonia.

Hybrid desorption is the new process invented during this program. It is a carbon dioxide displacement with simultaneous burning. Carbon dioxide is less strongly adsorbed than ammonia. As a result it will not replace all of the adsorbed impurities. However, it was found early in the program that it would desorb most of the impurities (5). By adding a small amount of air to the desorbing carbon dioxide stream it was found that the remaining impurities could be burned off quite rapidly. The rapid 15 to 30 minute times to complete the burning is in contrast to the several hours time normally required for burning catalysts or molecular sieves (6). As this was a new process, there have been many problems developing it. These problems of an undeveloped process have been the major disadvantage of Hybrid desorption. It has two major advantages however. Since carbon dioxide is a waste product of the fuel cell there is no need for either a substantial inventory of carbon dioxide or periodic resupply. Secondly, there is, of course, no danger of exposing the unit's location if a malfunction releases carbon dioxide.

Prior to this final report period the ammonia desorption method was tested under many of the conditions to be expected in field units. All of the tests showed successful operation. The first tests showed only moderate reduction in impurity adsorption when the sieve was loaded with ammonia from a previous desorption (7). Stable cyclic operation was demonstrated with a natural JP-4 feed containing about 85 ppm sulfur (5). A synthetic JP-4 feed with the sulfur level increased to 1000 ppm with the addition of benzothiophene was also run in successful pilot plant cyclic operation (8). In addition, tests were made of potentially detrimental conditions that would occur in field units with a small inventory of recirculated ammonia. The ammonia would become saturated at ambient temperature and pressure with both water brought in with the feed and the feed hydrocarbon itself. Water in the ammonia showed no detrimental effects and feed in the ammonia showed only a moderate decrease in performance (8).

At the start of this final report period only one basic question remained about the applicability of the ammonia desorption method. The question was the operability of the process with all the sulfur type compounds that might be found in military fuels. Company funded studies had shown this process to be operable on a wide variety of refinery streams containing a wide variety sulfur compounds. However, there is one type sulfur compound that is not present at substantial concentrations in refinery streams but might be present at high concentrations in some finished products. This type sulfur compound is the disulfide. Disulfide compounds are formed from mercaptan sulfur compounds by the now outdated sweetening processes such as Doctor and Hypochlorite. These processes were commonly used as a final treating step to reduce the very odoriferous mercaptan content to specification levels. This, of course, created products with very high disulfide contents. High disulfide contents would not be expected in fuels processed in North America or Western Europe where the sweetening process has been replaced with more modern technology. However, these disulfide forming sweetening processes are still used in some less developed parts of the world and fuels processed by them could be purchased for military use. In view of this and the fact that disulfide is the least stable of the sulfur compounds it was decided to check the operation of the ammonia desorption with disulfide compounds in the final report period to check for any possible complication.

Prior to this final report period the new Hybrid desorption method had shown promise, but many problems had come to light. Although physically adsorbed carbon dioxide interfered less with impurity adsorption than did the physically adsorbed ammonia, carbon dioxide chemisorbed on one of the sieves ($Ca^{+2}Y$) at normal adsorption temperature (700°F). This drastically reduced the impurity adsorption performance (7). Unfortunately, chemisorption became a major problem at the 950°F peak temperature required for Hybrid desorption (5). Two of the best molecular sieves ($Ca^{+2}X$ and $Sr^{+2}X$)

chemisorbed carbon dioxide, drastically lowering the impurity adsorption performance (8). In addition to stability in the presence of carbon dioxide the molecular sieves had to be stable to a combination of carbon dioxide and the water generated by the burning in Hybrid desorption. This was also a major problem as both of the sieves tested lost substantial impurity adsorption performance (8). Separate from the problems of molecular sieve stability, standard packed bed geometry and construction gave problems. The molecular sieve near the wall of a packed bed in an uninsulated standard wall thickness pipe remained too near the adsorption temperature when the 950°F burning front went through the bed. As a result, the sieve near the wall was not effectively desorbed (5).

In this final reporting period, work on Hybrid desorption was done in three areas. First was testing additional forms of molecular sieves for stability to carbon dioxide at 950°F and water-carbon dioxide mixtures. Second was the design, fabrication, and testing of a new type adiabatic sieve case to circumvent the low sieve temperature near the wall. And finally, was the testing of this new desorption technique with feeds of different sulfur contents and types.

2. Experimental Investigation

The chemical adsorbent was evaluated in the Dynamic Testing Unit. It is essentially twelve adsorbent cases in an oven with means for heating feed streams to the temperature of the adsorbent. Provisions are, of course, made for pumping the feed through the adsorbent cases and condensing and collecting the adsorbent effluent. This unit is more completely described in Appendix 2 of an earlier report (1). The adsorbent cases were loaded with two different particle sizes of the proprietary chemical adsorbent. Runs were made at space velocities typical of expected field use (about 2 w/w/hr) so the flow dynamics would be realistic. Space velocity is defined as the weight fuel per weight of adsorbent passed over the adsorbent per hour (w/w/hr.). The sulfur content of the feed was increased from an expected 10-50 ppm of a variety of sulfur compound types to 400 ppm with benzothiophene. Since benzothiphene is the most difficult sulfur type to remove with the chemical adsorbent, this provided a severe test. The increase to 400 ppm sulfur reduced the time required for the test.

The evaluation of the effectiveness of ammonia displacement with disulfide compounds was performed in actual cyclic operation using a pilot unit. This pilot unit was fully described in Appendix A-3 of an earlier report (7). For this evaluation, the sulfur concentration of the standard feed for this study CK 6979 (complete analysis in Appendix A-2 of an earlier report [7]) was increased to 1000 ppm with dinormalbutyl disulfide. A CITE fuel from the Far East was also provided by U.S. Army Mobility Equipment Research and Development Center, Fort Belvior, Virginia for evaluation. The molecular sieve used in these tests was the $Ca^{+2}X$ which gave very good performance in all previous tests.

In the Hybrid desorption program several sieves ($Ba^{+2}Y$, $Mg^{+2}Y$, $Mn^{+2}Y$ and $Sr^{+2}Y$) were tested for tolerance to carbon dioxide-water mixtures at 700 and 950°F. These sieves were all prepared using the standard general ion exchange procedure given in Appendix A-1 of an earlier report (7) using 2.0 normal solutions and two ion exchanges. The testing of these sieves was done in the Dynamic Testing Unit described in Appendix 2 of an earlier report (1). Each of the sieve forms was exposed to three different condition sets. The base case was 700°F exposure to carbon dioxide saturated with water at 80°F for only one hour before the feed was passed over the bed. This was just long enough to attain equilibrium carbon dioxide and water loading on the sieve. To test for carbon dioxide-water interaction degrading the sieve with time, carbon dioxide saturated with water at 80°F was passed over the sieves at 700°F for 409 hours. To test for tolerance to the complete environment, each form of sieve was exposed to the carbon dioxide-water mixture at 700°F for 409 hours and 950°F for 8 hours. When feed was passed over the sieve beds the adsorption effluents were collected in cuts and analyzed by ultra violet absorption for aromatic concentration. Although sulfur removal rather than aromatic removal was to be the function of these sieves, any sieve damage reducing sulfur removal would also reduce aromatic removal. Analysis for aromatic removal was used to monitor because it is much less expensive.

To circumvent the problem of the molecular sieve adjacent to the walls of a conventional sieve case remaining too near the adsorption temperature when the Hybrid burning wave went through the bed, a special adiabatic sieve case was designed and fabricated. There were two basic problems with the conventional sieve case. First the wall of standard thickness pipe had a very large heat capacity. The enthalpy generated by the impurity burning in the center of the bed increased the sieve temperature but the enthalpy generated near the wall had to raise the temperature of both the sieve and the wall resulting in a much lower temperature. Secondly the conventional sieve case had no insulation between the wall and the furnace which was providing an adsorption temperature environment. As a result the limited temperature increase which would occur tended to be dissipated to the furnace. In the new adiabatic sieve case, the wall heat capacity relative to the sieve was drastically reduced by going to thin wall (0.20 inches) tubing 1.5 inches in diameter. The heat loss to the furnace was reduced by a 0.28 inch layer of 20/40 mesh perlite insulation between the outside sieve case wall and a retaining tube. This entire assembly was mounted in the electrically heated furnace held at the adsorption temperature. It is envisioned that such an assembly - complete with insulation - would be mounted in a fuel-fired furnace of a field unit.

During the pilot plant operation, temperatures were monitored at the 3, 9, and 15 inch depths of molecular sieve both at the center of the bed and at the wall. Center temperatures were measured with 1/16-inch diameter sheathed thermocouples coming in the bottom of the case. Wall temperatures of the case were measured with small couples welded on the outside of the 0.20 inch tubing. Detailed drawings of the adiabatic case are shown in Appendix 1.

Tests of Hybrid desorption with this adiabatic bed were made in the cyclic pilot unit previously discussed. In addition to checking the operability of the process in the new sieve case, performance with several feeds were tested. A standard JP-4 feed (CK 6978 analysis Appendix A-2 of an earlier report [7]), this feed with benzothiophene added to increase the sulfur content to 4000 ppm, and a CITE from the Far East (supplied by U. S. Army Mobility Equipment Research and Development Center) were all used.

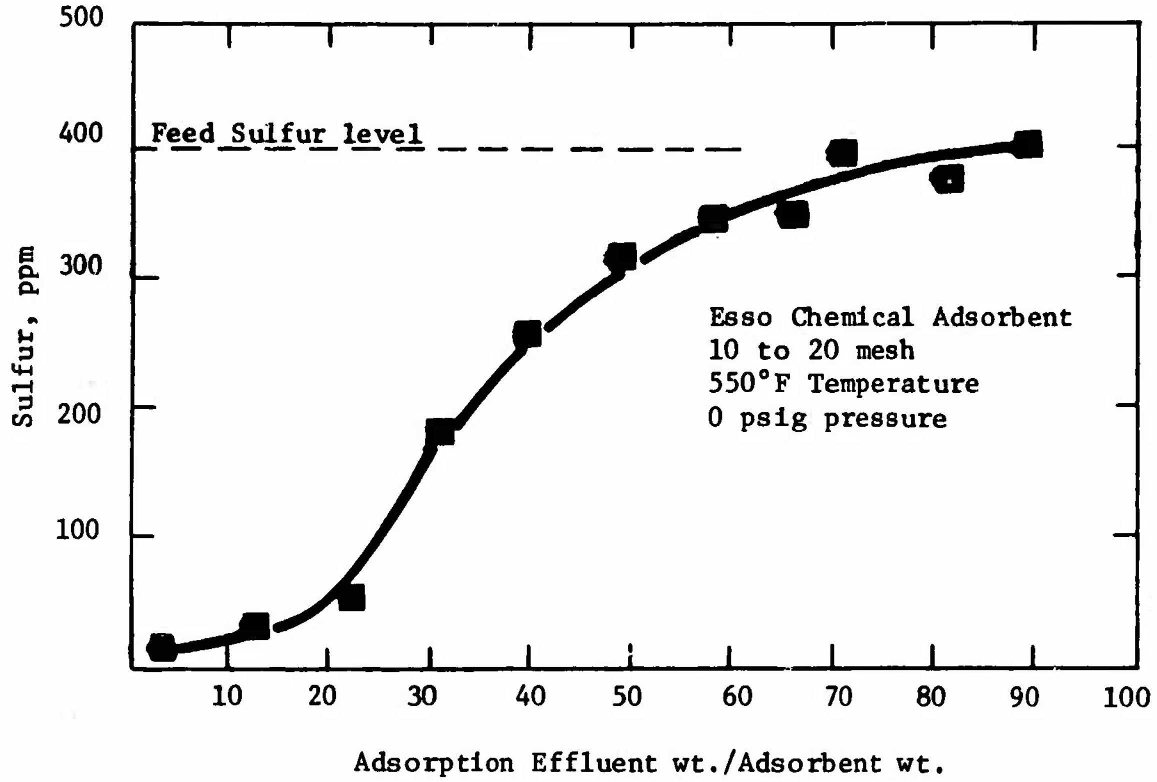
3. Results and Discussion

3.1. Sulfur Removal with a Chemical Adsorbent

Chemical adsorbents can be used to remove all of the sulfur in the fuel as done in a previous study (2,9) or as a final cleanup after the fuel has been processed with cyclic molecular sieve adsorption. A company-funded program had uncovered a chemical adsorbent which gave very high sulfur loadings with some compounds. Additional company work on the proprietary chemical adsorbent showed that thiophenic type sulfur was the most difficult to remove. In this program the simulant benzothiophene was selected because it is believed to be the most difficult sulfur compound to remove. It is both the highest boiling and the most resonance stabilized of the sulfur compounds to be expected in JP-4 or CITE fuels. The standard 85 ppm sulfur JP-4 fuel was increased to 400 ppm sulfur for the tests. Two widely separated temperatures (350 and 500°F) and pressures (0 and 200 psig) were used in four experiments with 1/8 inch diameter pills. These tests were run using simpler conditions than prior workers who found it necessary to add either water or hydrogen to the feed to get successful operation (2,9). The adsorbent containers did not plug with carbon under these conditions as they did in the earlier study (9), but the sulfur removal performance was not outstanding either (see Appendix 2). However, the similarity of the performance under the very different conditions was striking. The experiments run at 0 psig pressure had all vapor in the space surrounding the chemical adsorbent particles while the experiments at 200 psig had mixed liquid and vapor in this space. This combined with the small performance difference using 350 and 500°F temperatures strongly suggests that the performance limiting step was sulfur compound diffusion through liquid condensed in the pores of the chemical adsorbent. If diffusion in pores was the limiting step, reduction in chemical adsorbent particle size should improve sulfur removal performance. Test with the chemical adsorbent partical size reduced to 10 to 20 mesh did give the improved performance shown below in Figure 1.

Figure 1

Sulfur Removal With Esso Chemical Adsorbent



Integration of the performance obtained shows a sulfur loading on the adsorbent of about 1 weight percent when the sulfur level in the product reaches 5 percent of the feed sulfur level. This sulfur loading can be compared with a 1.3 weight percent sulfur obtained in a previous study (9). However the previous study used the more favorable but additionally complicated technique of including 3.5 moles of water for every mole of carbon in the fuel passed over the chemical adsorbent. Furthermore, in the previous study only 43 percent of the sulfur was thiophenic rather than the 79 percent used in this study, and thiophene rather than the more difficult to remove benzothiophene was used in the previous study. Thus, the proprietary chemical adsorbent requires no water addition to function. Its performance on more difficult to remove sulfur compounds was somewhat lower than the performance of the earlier study. On a comparable feed, the proprietary adsorbent might give close to previous performance (9) but without the need for water addition.

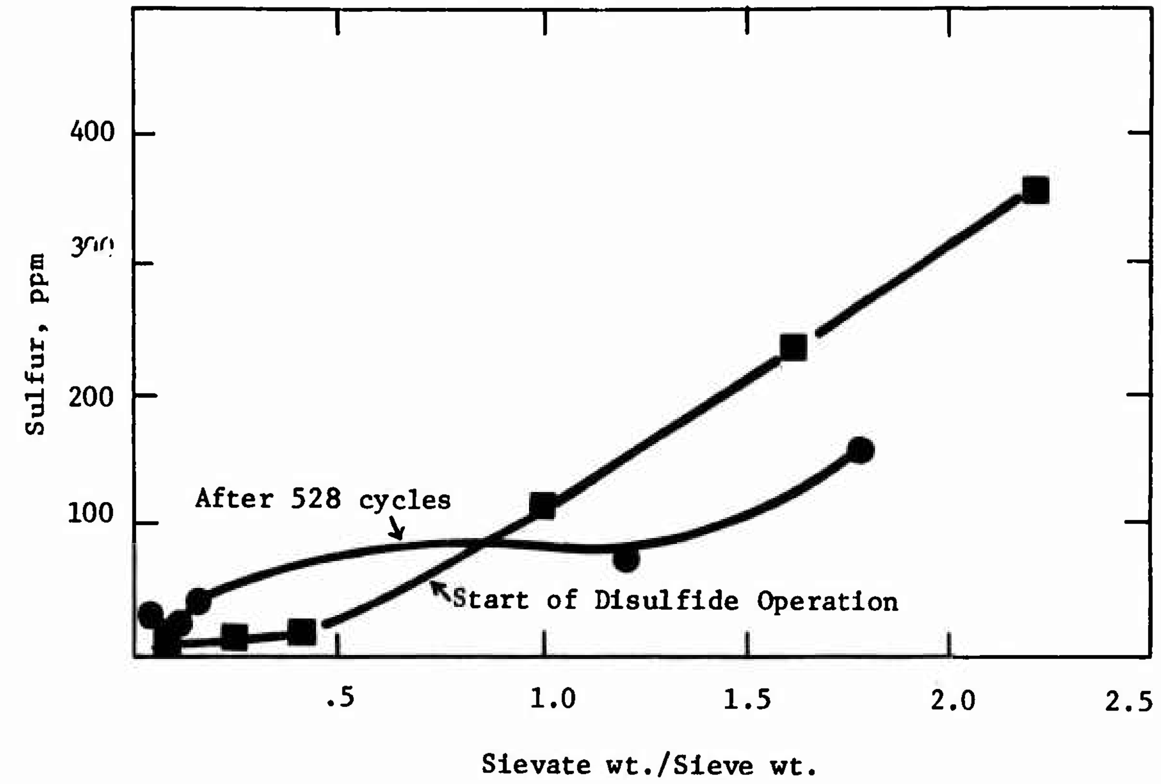
The proprietary chemical adsorbent operating with water in the feed, as in the earlier study (9), might substantially improve the performance. As has been mentioned, the proprietary chemical adsorbent appears to be limited by diffusion through liquid condensed in the adsorbent pores. Including 3.5 moles of water per mole of carbon in the feed would substantially reduce the feed partial pressure and therefore the quantity of condensation in the adsorbent pores. However, it is doubtful that the improvement could reach the 2.5 weight percent sulfur on the adsorbent achieved in the previous study (2) by the further complication of hydrogen recycle in addition to the water inclusion.

3.2. Disulfide Type Sulfur Removal Using Molecular Sieves with Ammonia Desorption

In view of the lower chemical stability of disulfides compared to other sulfur compounds, disulfides were tested in the cyclic molecular sieve process using ammonia for desorption to determine if there were any problems. Dinormal butyl disulfide was selected as the model compound and the sulfur level of the standard 85 ppm JP-4 feed (CK 6979) was increased to 1000 ppm with this disulfide. The performance was evaluated in cyclic operation using the type molecular sieve ($Ca^{+2}X$) and the conditions used successfully with other feeds (5,8). The sulfur levels in the adsorption effluent were satisfactory, averaging about 25 ppm at about 0.35 w/w adsorption effluent processed per cycle (see Appendix 3). However, under stable cyclic operation where no sulfur compound breakdown or permieat and fouling accumulation on the sieve occurs, the combined sulfur content in all the leaving liquid streams must equal the sulfur content of the entering feed. In this operation the combined sulfur content of leaving streams was an ominous 10 percent of that brought into the unit with the feed. Although mass spectrometry was not able to detect either hydrogen sulfide or mercaptans in the gases leaving the unit, odor indicated that there were some sulfur compounds in these gases. It was feared that sulfur compounds might be building up on the molecular sieves lowering the adsorption performance. After 528 cycles the sievate sulfur content vs. quantity was measured and compared with that at the start of the disulfide operation. Figure 2 below shows that the sulfur level at the start of an adsorption had increased compared to the start of the operation and that something had changed the characteristics of the molecular sieve.

Figure 2

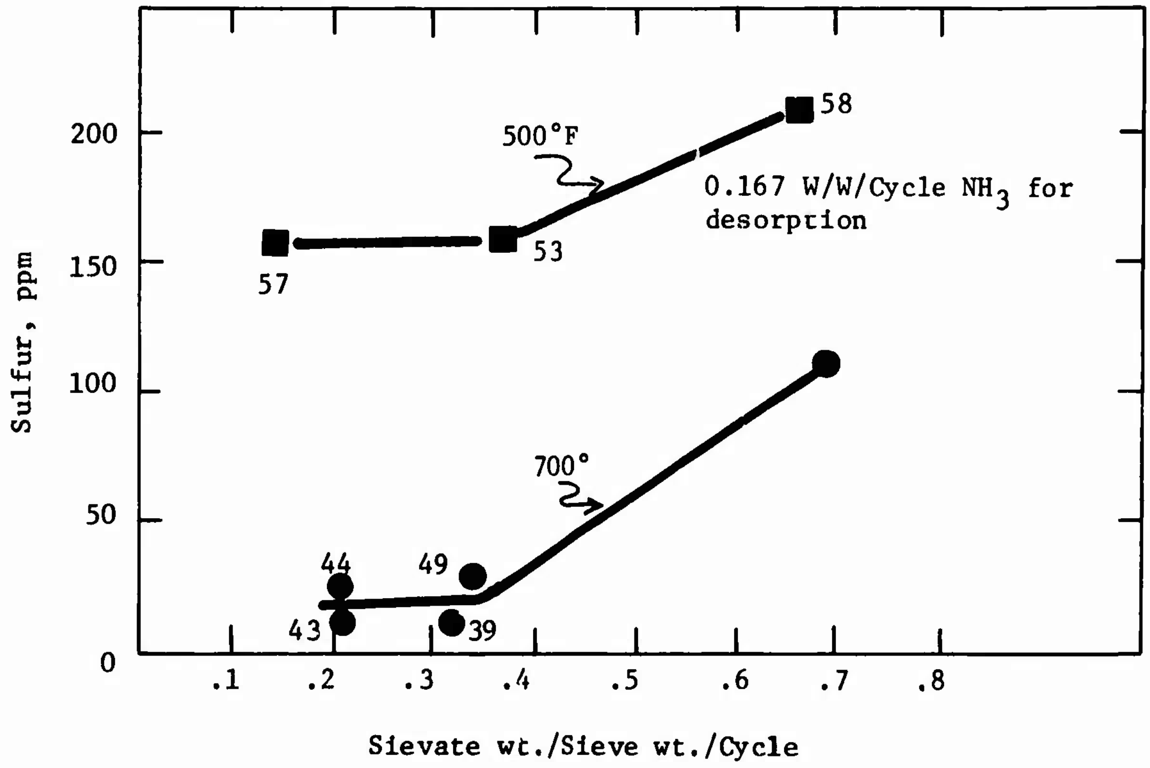
Operation With Disulfides Changes Sieve Performance



It was decided to lower the operating temperature from 700 to 500°F to see if the disulfide decomposition rate could be lowered thereby reducing the accumulation of unwanted sulfur compounds on the molecular sieve. The disulfide decomposition rate was lowered as indicated by the recovery of the feed sulfur in the liquid products increasing from 10 to 40 percent. However, the sulfur contents of the sievate product increased from 25 to over 150 ppm as shown below in Figure 3. (More detail in Appendix 3)

Figure 3

Lower Temperature Yields Higher Sulfur Contents



Whether the increase in product sulfur concentration was caused by a reduction in the disulfide decomposition rate or the onset of adsorption rate limitation was academic. Lower temperatures were not the method to obtain satisfactory operation.

A very high disulfide content (2100 ppm) fuel was then used with a new batch of molecular sieve with checked performance, in cyclic ammonia desorption operation. This high disulfide CITE fuel, obtained in the Far East, and supplied for this program by U.S. Army Mobility Equipment Research and Development Center, gave disastrous performance. (See Appendix 4 condition 8 through 15.) The sievate product, in contrast to completely clear sievates previously obtained in this and company studies, was bright yellow. The vapor space over the sievate and desorbed products was an even more intense yellow; additionally white and yellow crystals formed on container walls in the vapor space. The odor of hydrogen sulfide from the products overwhelmed the ammonia odor. The sievate products were high in sulfur content (40 to 130 ppm) and even contained elemental sulfur. Completing the disastrous performance was a decline in total hydrocarbon adsorption capacity from .065 to .044 w/w/cycle in only 7 cycles of operation. This indicated a blocking of adsorption sites on the sieve.

The source of these problems is nearly certainly the decomposition of some of the less stable disulfide compounds at the adsorption temperature, where the sieve may have a catalytic action, to form elemental sulfur and hydrogen sulfide. The high concentration of hydrogen sulfide formed can react with the ammonia to form the solids ammonium hydrosulfide [$(NH_4)HS$, white] and ammonium monosulfide [$(NH_4)_2S$, yellow]. These compounds explain the crystals in the products. It is probable that these or related compounds condensing on the highly polar surface of the sieves caused the adsorption performance decline.

Thus, the ammonia desorption method is not suitable for processing fuels with high disulfide concentration. Additional processing would be required to remove the disulfides before the other sulfur compounds could be successfully removed by a cyclic molecular sieve process using ammonia for desorption.

3.3. Hybrid Desorption

There was no sieve stable under Hybrid desorption conditions - carbon dioxide and water exposure at elevated temperature - at the start of this report period. However, mechanisms were hypothesized for the observed sieve degradations which suggested routes to circumvent the problems. Degradation of X-type sieve with time had been found on exposure to carbon dioxide and water at 700°F. The mechanism proposed for this degradation is as follows. The adsorbed carbon dioxide and water can combine making carbonic acid. This acid is, of course, partially ionized so there are H^+ ions in the adsorbed fluid. These H^+ ions can replace some of the cations on the sieve making the H^+ form of X-type sieve which is unstable (10). The crystalline lattice of this unstable sieve collapses to an ammorphous mass with nearly no adsorption capacity. Although H^+ ions, leading to the collapse, would be present in very small concentration at any time, the continual resupply of carbon dioxide and water to form more H^+ ions could, with time, produce the total destruction of the sieve. If this hypothesis is correct using Y-type molecular sieves which are stable in the H^+ form (10) should eliminate the crystal destruction solving this problem.

The other problem of molecular sieve stability was the chemisorption of carbon dioxide at elevated temperature drastically lowering the impurity adsorption. An example of carbon dioxide chemisorption at ambient temperature on one particular sieve was found in the open literature (11). Examination of the infrared adsorption of the chemisorbed species indicated that it was similar to a carbonate structure. The fact that only one of the several sieves tested chemisorbed the carbon dioxide lead the authors(11) to hypothesize a geometric mechanism as follows. Since the carbon dioxide would first physically adsorb on the cations, only a cation of the proper radius would allow proper alignment of the adsorbed carbon dioxide and oxygen in the molecular sieve framework for the chemisorption to occur. In this study some cationic forms of sieves which were carbon dioxide stable at ambient temperature were found to chemisorb at 700°F (7). Still other forms of sieve stable at 700°F chemisorbed at 950°F (8). The interpretation of these results were the range of cation radius allowing proper alignment for chemisorption broadened as increasing temperature increased the vibrational amplitude of all the ions and atoms in the molecular seive crystals. Thus we postulate that a sieve resistant to chemisorption might be found by studying a wide range cation radii, as some could be outside the range allowing chemisorption. Based on range of ionic radius and reduction potentials required to prevent cation reduction to the metal on hydrocarbon contact found earlier in this study (1), the cations listed below in Table 1 were selected for study.

Table 1

Cations Selected for Stability to CO_2 Tests

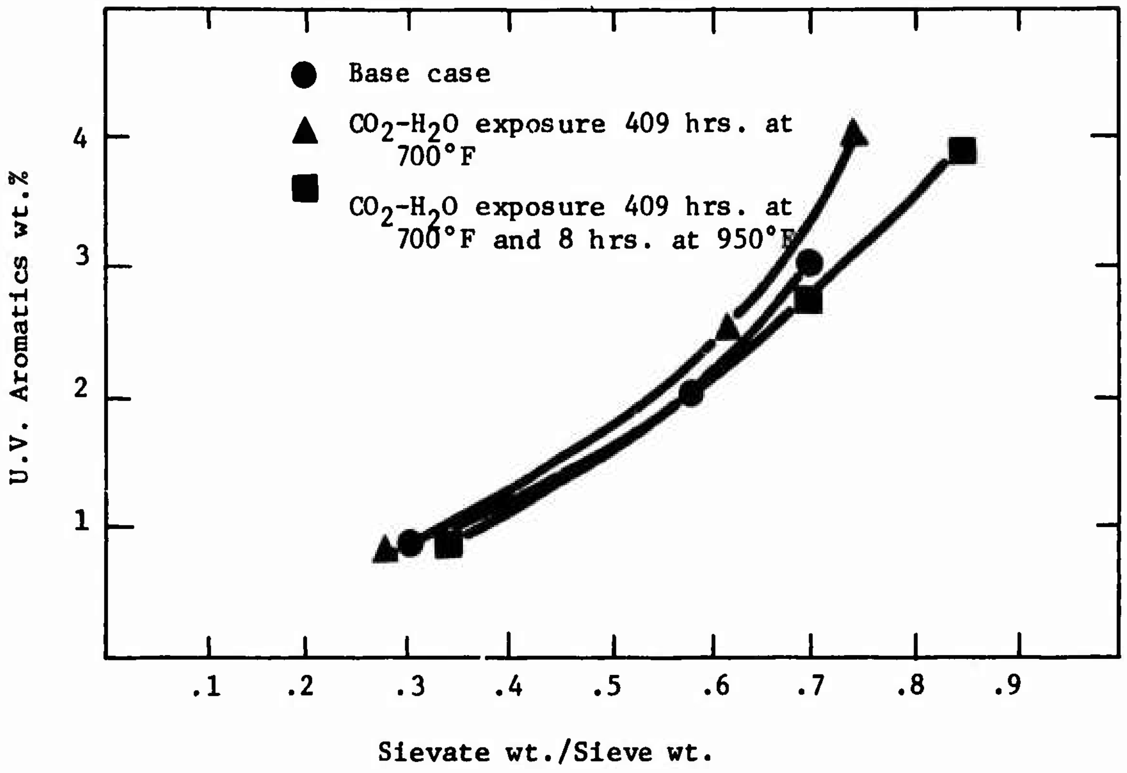
Cation	Ionic Radius Å
Mg^{+2}	0.66
Mn^{+2}	0.80
Sr^{+2}	1.12
Ba^{+2}	1.34

These cations were used on the Y-type molecular sieve framework to circumvent the carbon dioxide-water degradation.

Conditions were chosen to test the sieves selected so that positive results would indicate a probable useful life of 1000 hours. Tests for the carbon dioxide-water effect on degradation were run with carbon dioxide saturated with water at 80°F passing over the sieve at 700°F. The lower 700°F adsorption temperature, rather than the 950°F peak desorption temperature, was selected for this test because it would maximize the quantity of adsorbed water which should maximize degradation. This test was arbitrarily terminated after 409 hours. Tests for carbon dioxide chemisorption at 950° F were run a much shorter 8 hours, since the sieve spends a very small fraction of the total time at 950°F. Water was included with the carbon dioxide in these higher temperature tests on the chance that it might be detrimental. Negative results were obtained with three of the sieves tested (see Appendix 5). However, the largest cation (Ba^{+2}) on the Y sieve gave very good results (see Figure 4).

Figure 4

Hybrid Desorption Conditions Do Not Damage Ba^{+2} Y Sieve



The adsorption performance after the exposure to the potentially damaging conditions for a total of 417 hours is within experimental error of that for the base case sieve. This indicates that the Ba^{+}Y sieve would be expected to have a useful life under Hybrid desorption conditons of at least 1000 hours.

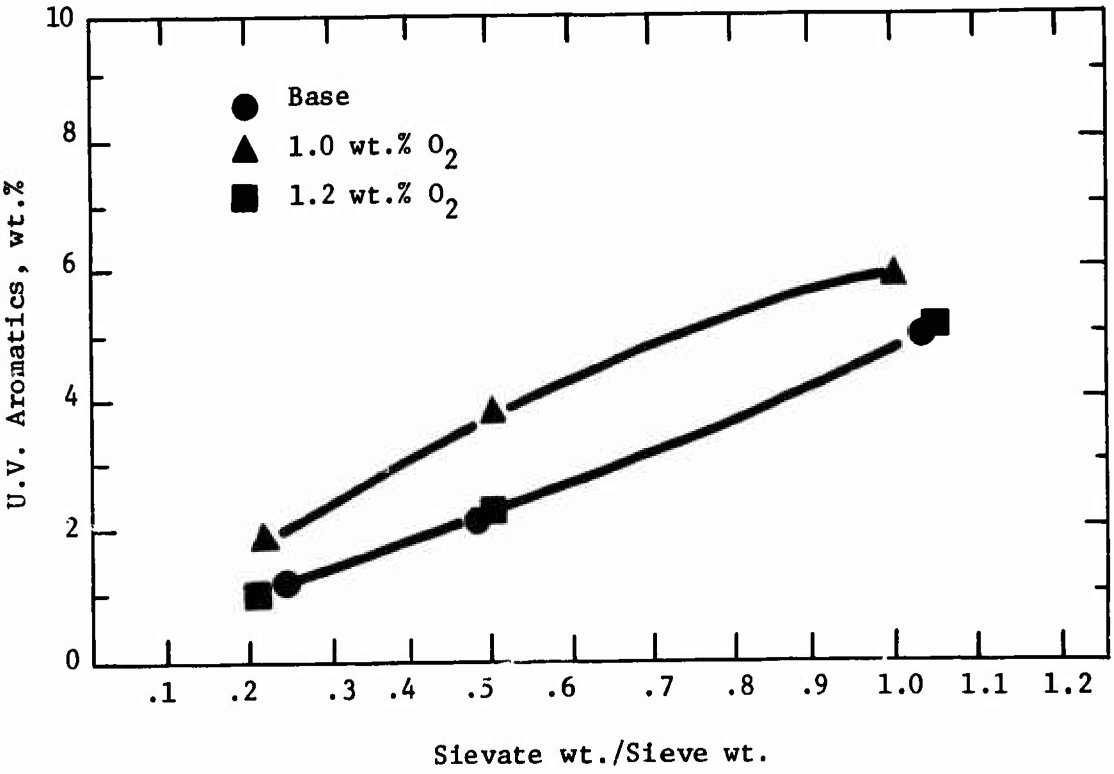
In addition to finding a molecular sieve stable to Hybrid desorption conditions, it was necessary to devise a sieve case which would allow all of the sieve to reach 950°F when the temperature wave moves through the sieve bed. As will be recalled, the heat capacity of the thick walls of a standard sieve case and heat losses to the surroundings prevented the sieve adjacent to the walls from increasing to the required temperature (see Figure A-5 of an earlier report [5]). The new thin wall adiabatic sieve case (see Appendix 1) was used to evaluate the stable Ba^{+2}Y sieve in actual operation. The first Hybrid desorption was performed with 1.475 weight percent oxygen in the carbon dioxide. The heat retention in the bed was better than expected producing wall temperature rises between 215 and 400°F above the 700°F base temperature. These compare with a 30°F rise using a standard sieve case. The temperature rise in the center of the bed however was 370 to 600°F - too much. The sieve reached a 1380°F maximum temperature for a short time, which is high enough to cause sieve thermal decomposition on prolonged exposure. Some small damage was apparently done to the sieve, as after intervening runs with lower oxygen content, returning to 1.475 weight percent oxygen produced center temperature rises of only

300 to 450°F (see Appendix 6). This indicates that the adsorption performance of the sieve was somewhat damaged by the high temperature of the first desorption, and that slightly better performance would have been obtained throughout the program if the maximum temperature had been held to say 1200°F.

The first few hybrid desorptions were followed by burns where the entire sieve case was elevated to 950°F by an external heater. This technique was found earlier in this study (5) to assure the sieve was returned to a standard condition before a subsequent experiment. Eliminating this step of externally heating the sieve allowed the evaluation of the completeness of Hybrid desorption using different oxygen contents in the carbon dioxide. Lower oxygen concentrations, of course, produce lower peak temperatures passing through the bed which are less likely to give complete desorption. As seen below in Figure 5, 1.0 percent oxygen was not high enough to return the sieve to the standard condition; however, 1.2 percent oxygen did return the sieve to base level of performance.

Figure 5

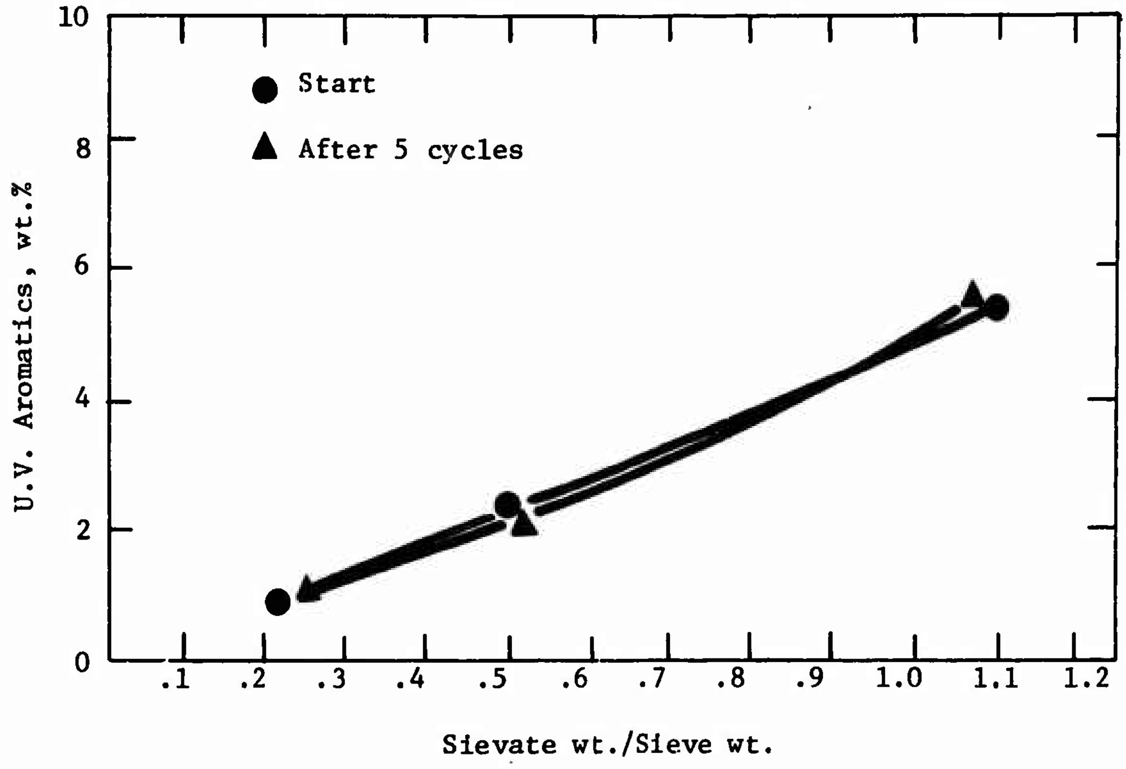
Hybrid Desorption Performance With Different Oxygen Concentrations



To move beyond what might be a borderline operability situation the oxygen concentration was increased from 1.2 to 1.475 percent. No adsorption performance decline was shown after five hybrid desorptions without the step artifically increasing the temperature. This is shown in Figure 6 below.

Figure 6

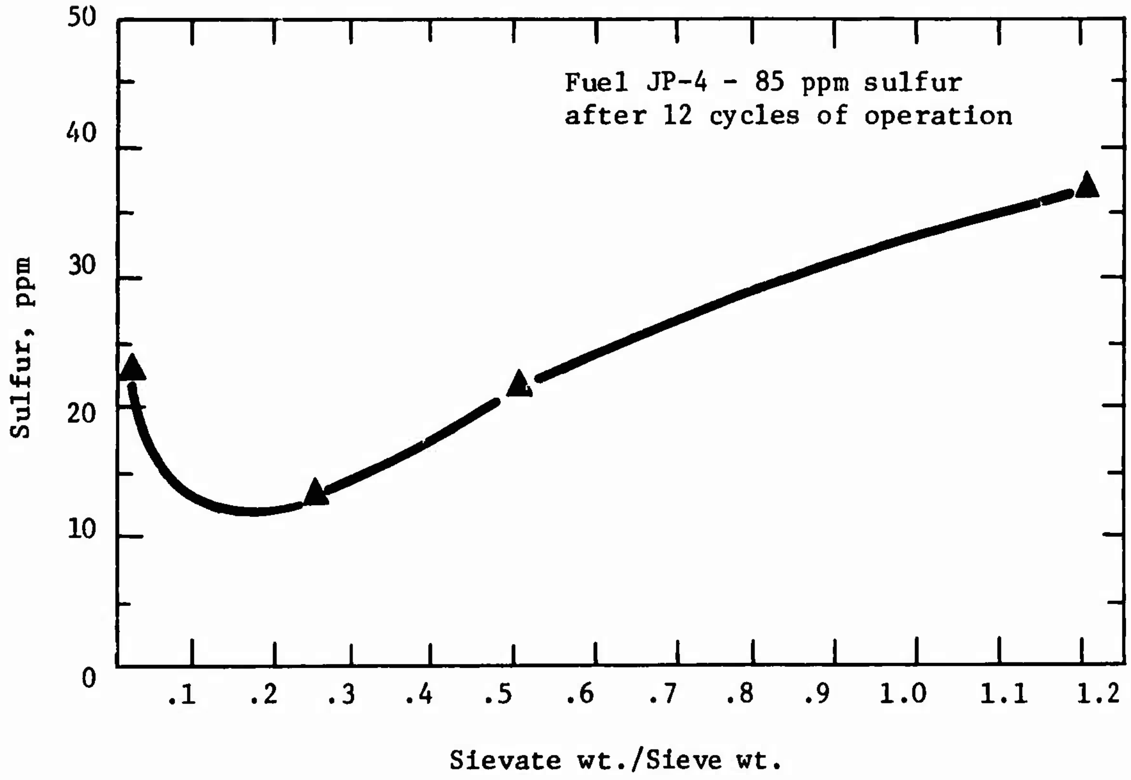
Stable Cyclic Operation with Hybrid Desorption



The good sulfur removal from the standard 85 ppm sulfur feed after the cyclic operation is shown in Figure 7.

Figure 7

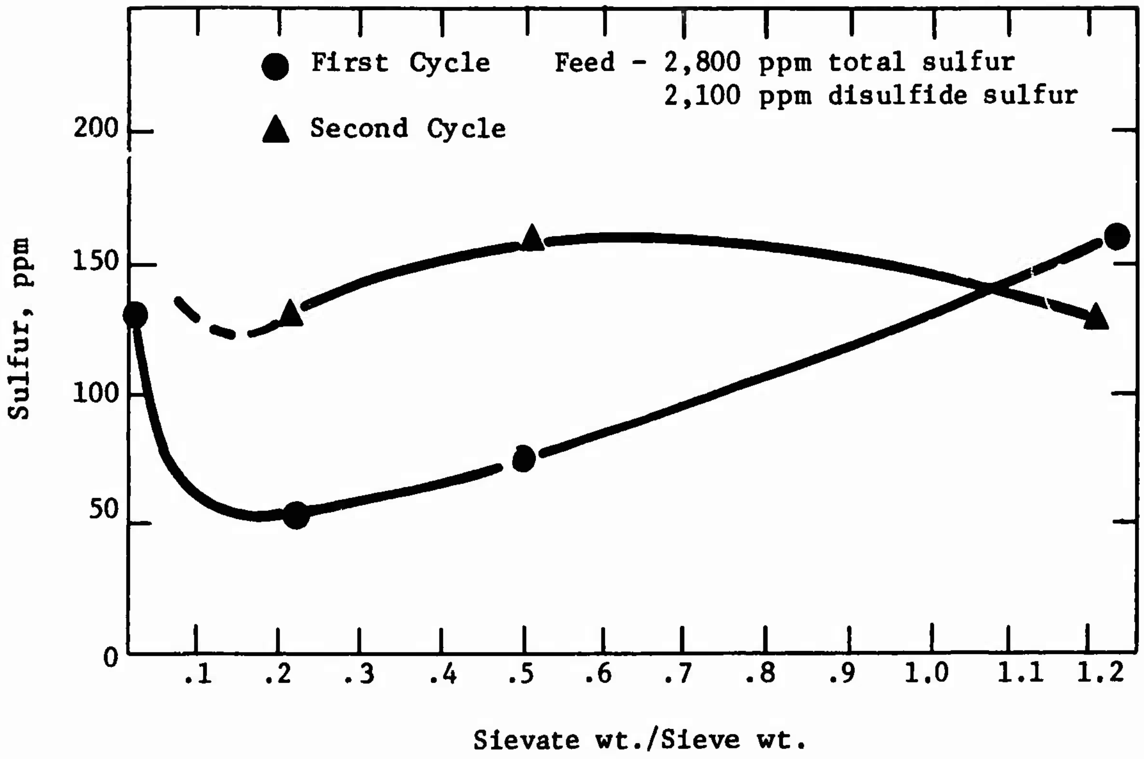
Sulfur Removal with Hybrid Desorption in Cyclic Operation



Next the CITE fuel with the very high disulfide content, (2,100 ppm) which proved to be the nemesis of the ammonia desorption method was tried with hybrid desorption. The very good sulfur removal down to 500 ppm sulfur with this 2,800 ppm total sulfur feed was obtained on the first cycle. (See Appendix 9 for Sulfur Analytical methods) Somewhat poorer sulfur removal was obtained after a hybrid desorption with a wall temperature rise in one area to only 895°F. (See Appendix 6) This indicates slightly higher oxygen contents will be required for best performance with this fuel. See Figure 8.

Figure 8

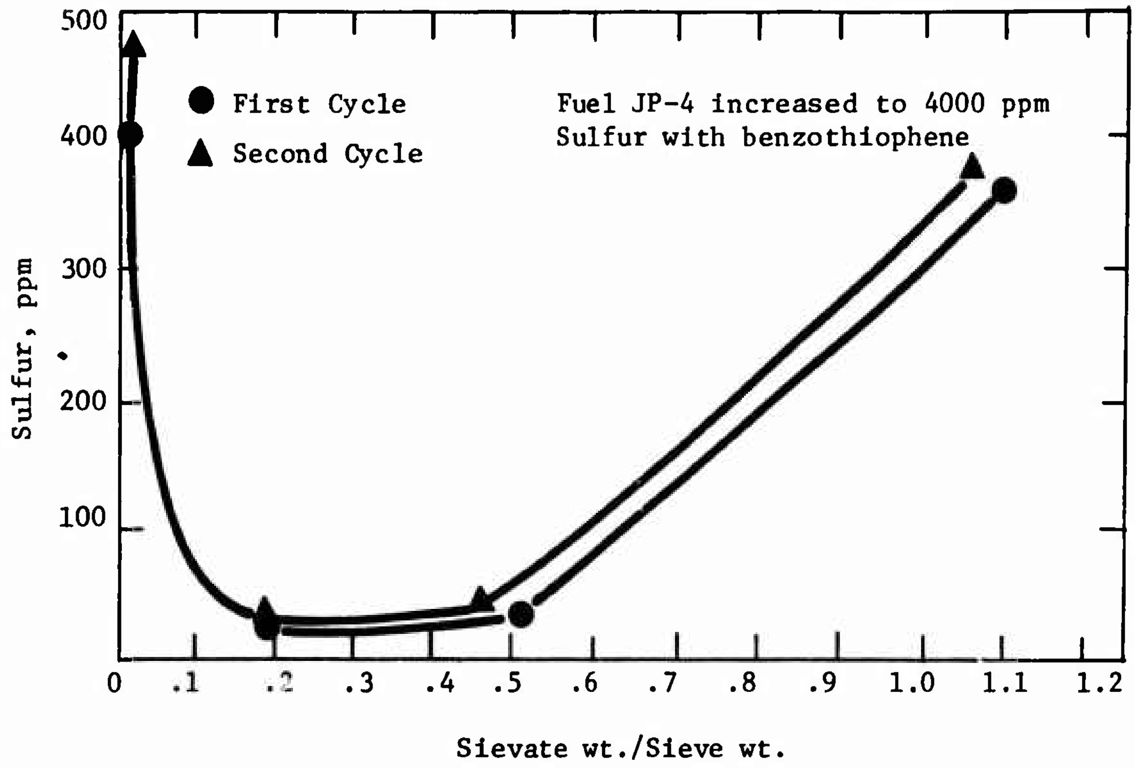
Sulfur Removal From a
High Disulfide Content CITE



Finally, Hybrid desorption was used with a feed increased to maximum allowable 4000 ppm sulfur content with benzothiophene. The excellent adsorption performance down to 20 ppm sulfur both initially and after a hybrid desorption are shown in Figure 9.

Figure 9

Sulfur Removal From 4000 ppm Sulfur Fuel



The performance characteristics of Hybrid desorption as used in all the proceeding examples suggest different conditions should lead to substantial improvements. For example each deviation above 700°F adsorption temperature decreased and deviation below 700°F increased the adsorption of aromatic and sulfur compounds (see Appendix 6). In addition to this indication that lower adsorption temperature would improve performance, several other areas should benefit from lower adsorption temperature rises in Hybrid desorption before reaching a temperature which might damage the sieve. This would allow Hybrid desorption with higher oxygen contents which would complete the desorption in less time. Additionally the sieve has some catalytic activity after a Hybrid desorption which can have negative effects. For example, the higher sulfur contents of the first sievate collected vs. the sulfur content after 0.2 w/w of sievate had been collected (Figure 7, 8, 9) is probably caused by the initial catalytic activity. Initially, the sieve probably breaks down the least stable sulfur compounds to either low molecular weight sulfur compounds or hydrogen sulfide, both of which are poorly adsorbed by the sieve and go out of the bed with the sievate product. The catalytic activity of the sieve stops after about 0.2 w/w of feed is passed over it as the most active sites would be expected to be covered with a carbonaceous deposit. Fortunately the initial catalytic activity generates sulfur compounds which are picked up at higher capacity by chemical adsorbents than typical feed sulfur compounds. However, these generated sulfur compounds to constitute an additional load for the chemical adsorbent which it would be desirable to minimize. Lower adsorption temperatures would minimize the reaction rate of the less stable sulfur compound minimizing initial sulfur contents and the load on the chemical adsorbent. Additionally, a lower adsorption temperature would lower the formation rate of the carbonaceous deposit. Since the carbonaceous deposit formed from a less stable fuel can be difficult to burn, minimizing the deposit should be helpful in establishing one set of not too severe hybrid desorption conditions for all fuels. Finally, it will be recalled that the temperature had been more moderate, say 1200°F, it is probable that all of the results of this program would have been somewhat better than measured.

Although the Hybrid desorption cyclic molecular sieve process is not optimized, a preliminary unit design was made based on the demonstrated conditions and performance. The requirements for the design were the purification of military fuel of maximum sulfur content (4000 ppm) for 1000 hours with less than 2 ppm of sulfur in the product. A flow diagram of this unit is shown in Appendix 7. It is considerably less complex than the flow diagram for ammonia desorption shown in an earlier report (8). The process basis used with the Hybrid desorption flow diagram for performance projections is given in Appendix 8. The yield of purified fuel would be about 92 weight percent. During the Hybrid desorption about 2.3 weight percent of the feed is burned in the beds. An additional 5.4 weight percent is desorbed. This desorbed fuel is somewhat more than would be needed to maintain temperature in a well insulated fuel purification unit and some of it could be used in the reforming unit. The projected weight for the fuel purification unit for a 3.75 KW reformer-fuel cell is below 40 pounds or about 10 pounds per kilowatt. This 10 lb/kw is substantially below the 330 lb/kw required to do the same purification using only the previously demonstrated (2) chemical adsorbent.

4. Conclusions and Recommendations

The difficulty of purifying military fuels, such as JP-4 for use in fuel cells varies widely with what must be removed. The removal of naphthene, aromatic, and sulfur compounds for direct hydrocarbon fuel cells sensitive to all three is quite difficult. The necessary yield debit for this removal is about 60 to 75 percent. Furthermore, the removal of naphthenes requires the use of an additional dehydrogenation process. As most of the yield debit and the additional process is required for napthene removal, this increases the incentive to make any future fuel cell systems naphthene tolerant.

Aromatic and sulfur compounds can be removed with cyclic adsorption on molecular sieves. The removal of only sulfur compounds with molecular sieves is a less severe requirement and can be accomplished with a higher yield of purified fuel. Sulfur-only removal can also be done with a disposable chemical adsorbent as demonstrated in an earlier study (2). However, the preferred processing is the removal of most of the sulfur compound with a cyclic molecular sieve process followed by a final sulfur removal with a chemical adsorbent.

The Esso proprietary chemical adsorbent for sulfur used in this study functioned without the complicating presence of either water or hydrogen which were required in a previous study (2). The weight of sulfur adsorbed on this proprietary chemical adsorbent was about 1.0 weight percent or about the same as obtained in the previous study where water alone was added to the feed. However, it is doubtful that the Esso chemical adsorbent could reach the 2.5 weight percent sulfur adsorbed in the previous study with both water and hydrogen present with any step short of the addition of these two streams. Unless the simplification of eliminating these two steams takes on additional importance for some specific application it is recommended that the adsorbent demonstrated in the earlier study (2) be used for the final cleanup step.

Cyclic processing with molecular sieves uses two basic steps. They are adsorption of impurities on the molecular sieve and desorption of the impurities, readying the sieve for the next adsorption step. The adsorption of aromatic and sulfur impurities proved not to be a problem as many sieves were found early in the program to have good adsorption characteristics. The desorption of the impurities has been the area that required most of the effort. Three basic methods were used for desorbing the molecular sieve.

- Ammonia displacement
- Carbon dioxide displacement
- Hybrid desorption

The ammonia displacement method functioned very well under a wide variety of operating requirements. However, it does not function with feeds of very high disulfide content. The disulfides decomposed yielding hydrogen sulfide which in turn formed ammonium sulfide type compounds on the sieve unacceptably lowering the adsorption performance. Military fuels can contain high concentration of disulfides. The only way these fuels could be successfully purified using ammonia displacement is by first removing the disulfide compound in separate processing. Although processing removing the disulfides using catalytic conversion at low or perhaps no hydrogen pressure could probably be developed, it is not recommended that this more complicated processing be developed.

Desorption by carbon dioxide displacement was found ineffective. The carbon dioxide could not remove all of the adsorbed impurities. Further pursuit of simple carbon dioxide displacement is not recommended.

The recommended Hybrid desorption is an outgrowth of carbon dioxide displacement. Hybrid desorption is a simultaneous carbon dioxide displacement and burning with oxygen from air. Although there was considerable difficulty in finding a sieve stable in the carbon dioxide-water environment required, one has been found - $Ba^{+2}Y$. This molecular sieve showed no measurable decline in adsorption performance after 417 hours exposure to the environment. It is expected that this sieve will give good performance for at least 1000 hours operation.

Successful operation of Hybrid desorption has been demonstrated in a thin wall, adiabatic sieve case with 700°F temperatures for the environment, the inlet fuel, and the inlet carbon dioxide-air mixture. The same temperature for the environment and these streams was required to make the process sufficiently simple. The process has been demonstrated with fuels containing up to the maximum allowed 4000 ppm sulfur content.

A unit design was made for removal of 4000 ppm sulfur for 1000 hours using Hybrid desorption followed by a chemical adsorbent to remove the small quantity of sulfur in the molecular sieve product. The yield of purified fuel from this process was a very good 92 weight percent. The material desorbed from the sieve including the impurities is about 5.4 weight percent of the fuel feed to the unit. This is more than sufficient to maintain the temperature of a well insulated unit. The projected weight of the unit is about 10 pounds per kilowatt; a substantial advantage versus the 330 pounds per kilowatt required for the same purification using the previously developed (2) chemical adsorbent alone. The hybrid desorption followed by the small quantity of chemical adsorbent is the purification process recommended particularly where the fuels may have high sulfur contents and where it is desirable to lengthen the time between fuel purifier shutdowns.

As Hybrid desorption performance is believed to be substantially non-optimized, it is recommended that further pilot unit type studies aimed at a closer approach to optimum be undertaken before prototype units are built.

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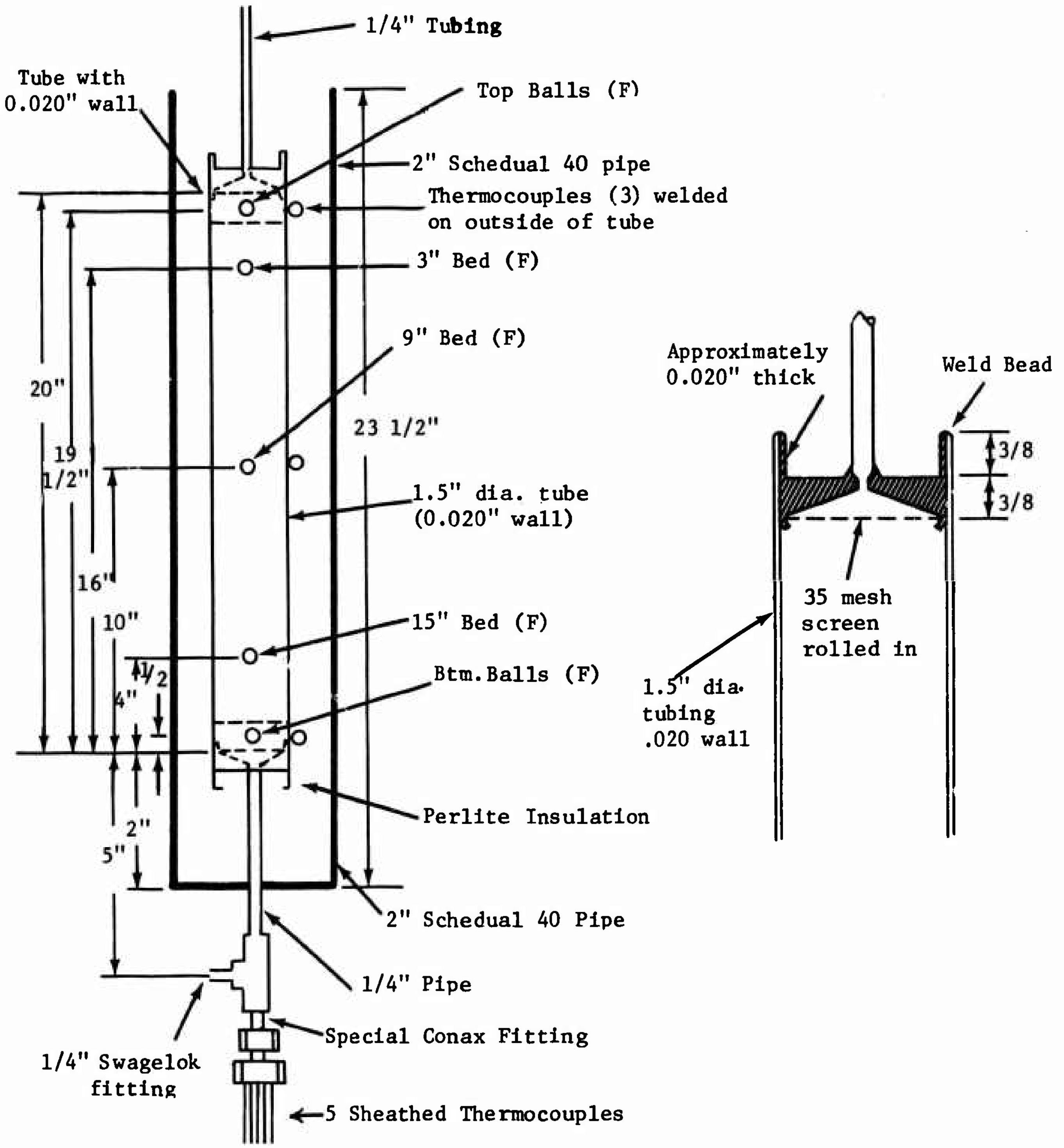
Appendix 1

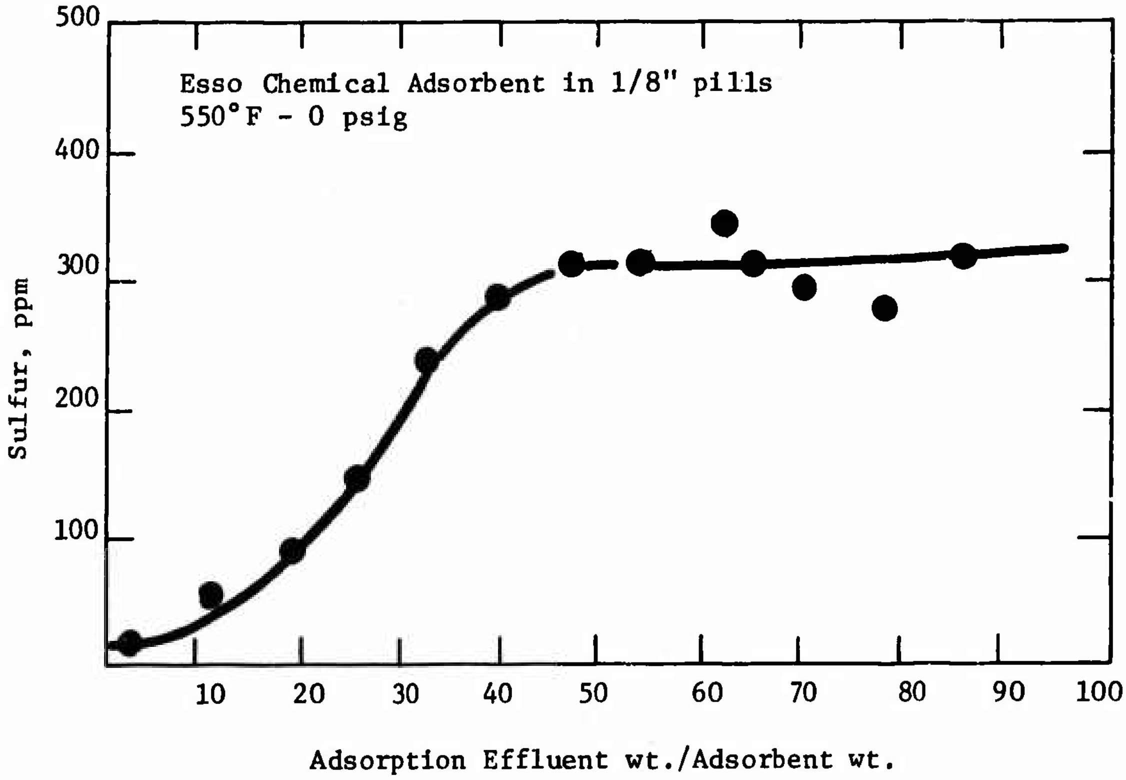
Adiabatic Sieve Case

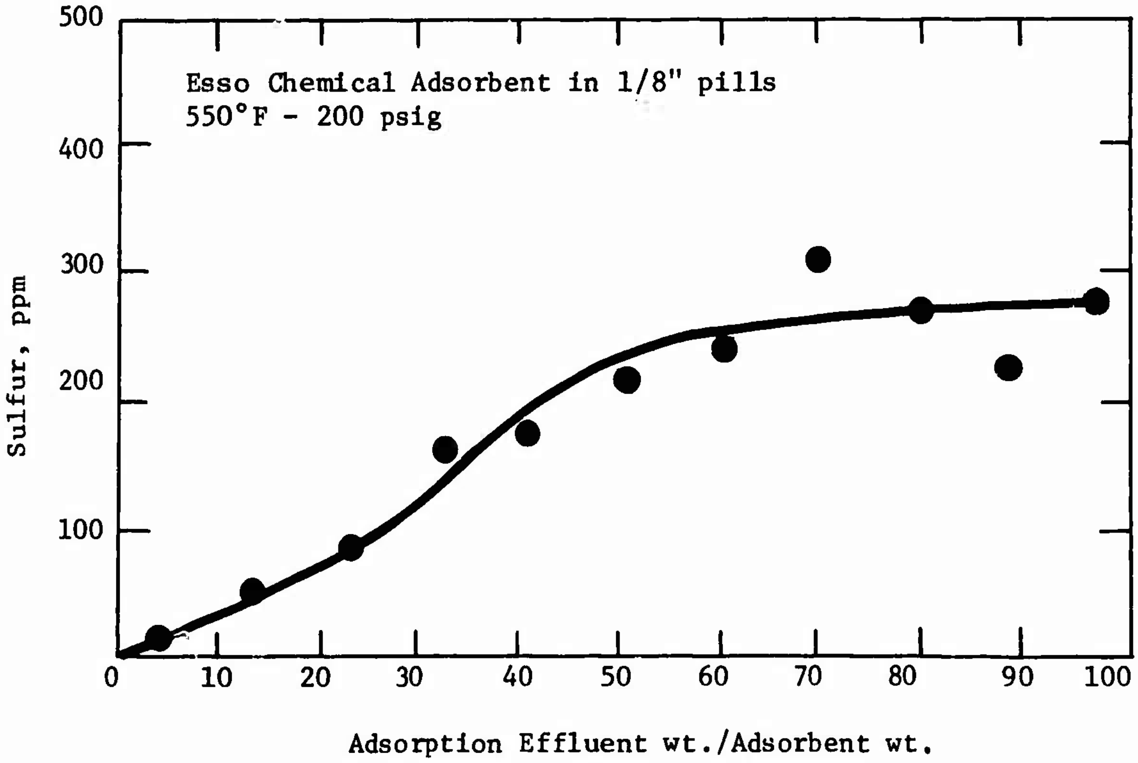
18 inch molecular sieve bed depth



Appendix 2

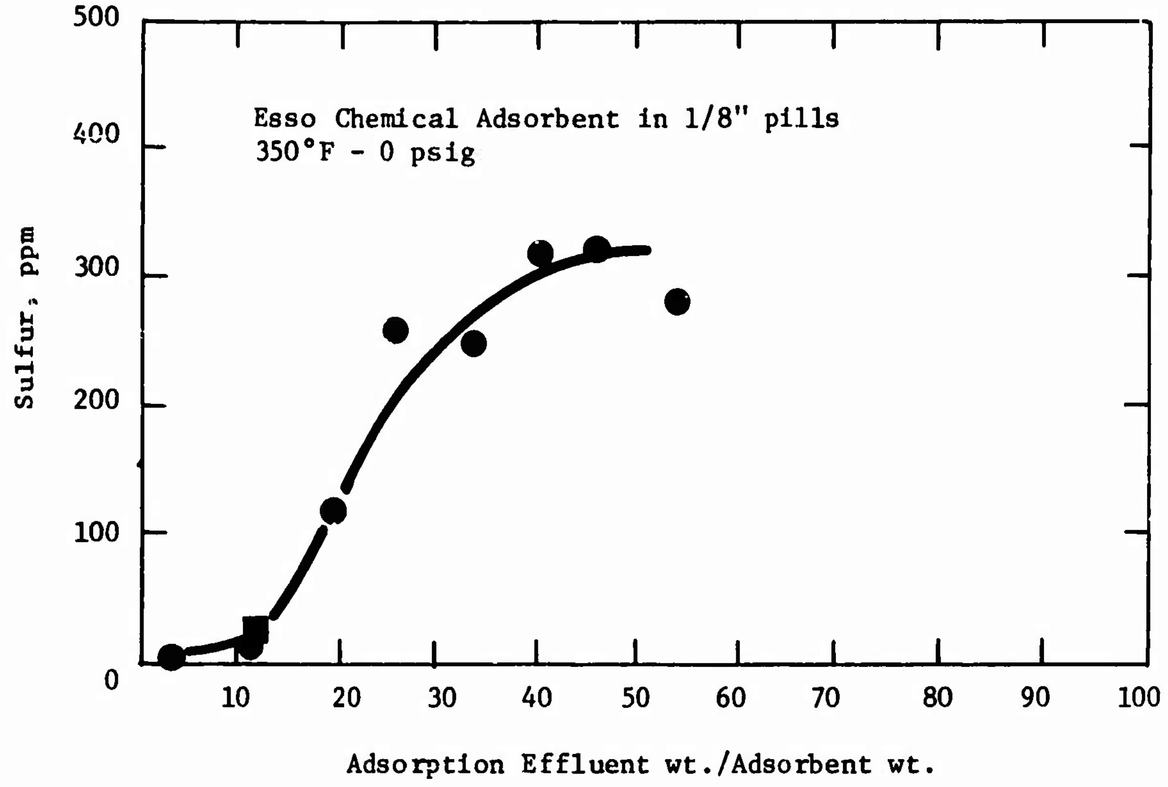
Chemical Adsorbent Performance

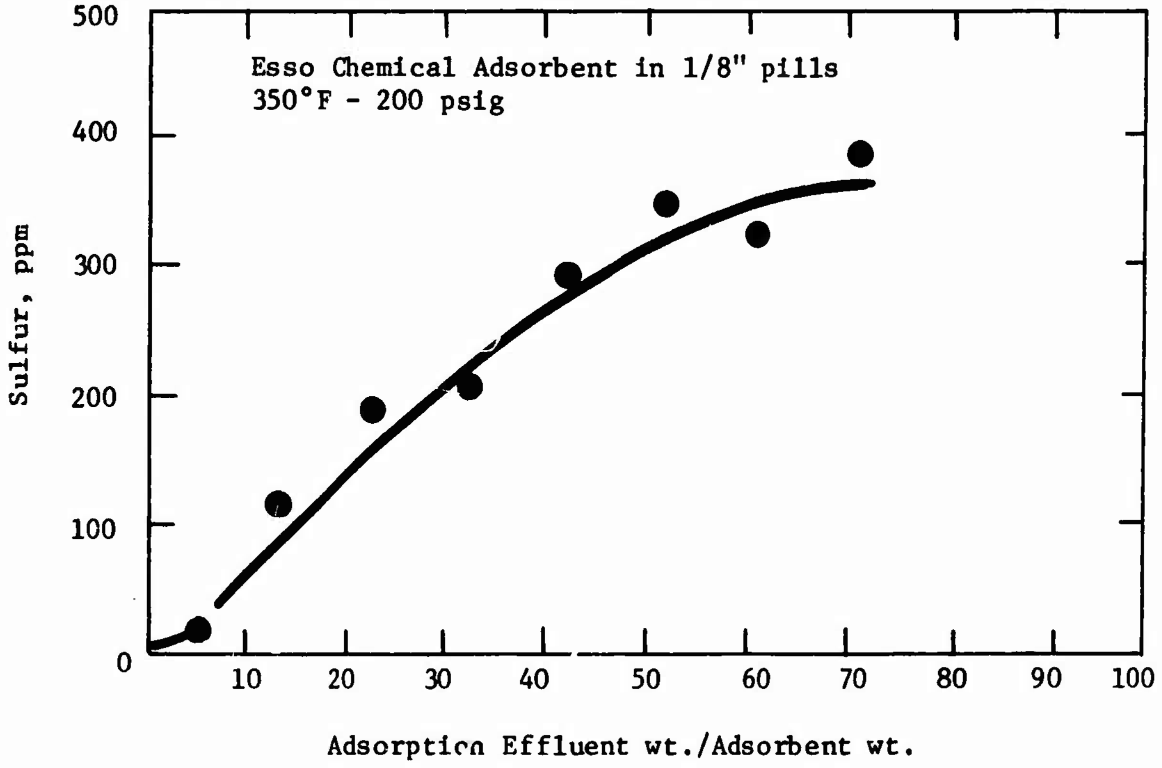




Appendix 2 (Con't)

Chemical Adsorbent Performance





APPENDIX 3

CYCLIC OPERATION WITH AMMONIA DESORPTION

Run 966 - 107
Sieve Used Ca^{+2}X(2N, 2, O, L)

Condition - Period	8					9				10			
Time, Hrs	10					8				8			
No. of Cycles in Period	60					48				48			
Sub-Period	A	B	C	D	E	A	B	C	D	A	B	C	D
Time, Hrs	2	2	2	2	2	2	2	2	2	2	2	2	2
No. of Cycles in Sub-Period	12	12	12	12	12	12	12	12	12	12	12	12	12
Cut No.													
Time of Cut (Min)													
Unit Data													
Adsorption													
Temp (°F)						716	709	709	711	712	713	713	711
Pressure (psig)						.25				.25			
Time (Min)						5				5			
Feed Used						CK 6978				CK 6978			
Total Feed Rate, From Feed (W/W/Hr)	4.289					4.489				4.685			
Total Feed Rate, From Product (W/W/Hr)	4.36	4.34	4.33	4.33	4.39	4.28	4.39	4.38	4.37	4.48	4.41	4.44	4.50
Total Feed Per Cycle From Feed (W/W/Cycle)	.1787					.3741				.3904			
Total Feed Per Cycle From Product (W/W/Cycle)	.3633	.3617	.3605	.3608	.3658	.3563	.3656	.3654	.3645	.3732	.3678	.3697	.3754
Sievate Per Cut (W/W)													
Sievate Per Cycle (W/W/Cycle)	.3025	.3025	.3020	.3020	.3035	.2954	.3015	.3020	.3018	.3265	.3071	.3066	.3102
Fresh Feed Per Cycle						.3392				.3542			
Fresh Feed Rate (W/W/Hr)						4.070				4.685			
Desorption													
Temp (°F)						709	705	709	709	713	706	707	712
Pressure (psig)						1.0				.75			
NH$_3$ Rate (W/W/Hr)	2.02					2.02				2.02			
Desorbate 1													
Time (Sec)	30					30				30			
W/W/Cycle NH$_3$	.0167					.0167				.0167			
W/W/Cycle	.0415	.0454	.0443	.0452	.0483	.0457	.0471	.0433	.0428	.0333	.0428	.0436	.0414
Desorbate 2													
Time (Sec)	270					270				270			
W/W/Cycle NH$_3$	.1515					.1515				.1515			
W/W/Cycle	.0193	.0138	.0142	.0136	.0140	.0152	.0170	.0201	.0199	.0134	.0179	.0195	.0238
Total Desorbate (W/W/Cycle)	.0608	.0592	.0585	.0588	.0623	.0609	.0641	.0634	.0627	.0467	.0607	.0631	.0652
Total NH$_3$ (W/W/Cycle)	.1682					.1682				.1682			
Recovery	96.6					97.04				95.18			
Analysis													
Sievate													
Sulfur (Lamp, ppm)				1.9/1.0 *	.8			4.4	5.3			8.2/7.6/ 5.9	7.2
U.V. Aromatics													
Benzene	.0354	.0317	.0326	.0309	.0332	.486	.667						
Bz, T, Xy								.913	.962		1.397	1.602	1.6
Feed Dist. Aromatics										6.51			
Total Aromatics	.0354	.0317	.0326	.0309	.0332	.486	.667	.913	.962	6.51	1.397	1.602	1.6
Desorbate I													
Sulfur (Lamp, ppm)													
U.V. Aromatics													
Benzene													
Bz, T, Xy													
Feed Dist. Aromatics													
Total Aromatics	(ABCD - 11.71 Used in Run 966-107-9)					154.8	(ABCD - 12.46 Used in Run 966-107-10)			(ABCD - 12.31 Used in Run 966-107-11)			
Desorbate 2													
Sulfur (Lamp, ppm)					327				329				362
U.V. Aromatics													
Benzene													
Bz, T, Xy													
Feed Dist. Aromatics					175.7				180.1				133.6 /120.7
Total Aromatics					175.7				180.1				133.6 /120.7
Total Feed, Sulfur (Lamp)	103					103				98			

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* 1.9/1.0 indicates multiple analysis on the same sample.

APPENDIX 3 (CONT.)

CYCLIC OPERATION WITH AMMONIA DESORPTION

Run 966 - 107

Sieve Used $Ca^{+2}X$(2H, 2, O, L)

Condition - Period	11				12				13			
Time, Hrs	8				8				8			
No. of Cycles in Period	48				48				48			
Sub-Period	A	B	C	D	A	B	C	D	A	B	C	D
Time, Hrs	2	2	2	2	2	2	2	2	2	2	2	2
No. of Cycles in Sub-Period	12	12	12	12	12	12	12	12	12	12	12	12
Cut No.												
Time of Cut (Min)												
Unit Data												
Adsorption												
Temp (°F)	711	709	711	712	705	710	711	707	712	713	710	712
Pressure (psig)	.25	.25	.50		.25				.25			
Time (Min)	5				5				5			
Feed Used	CK 6978				CK 6978				CK 6978			
Total Feed Rate, From Feed (W/W/Hr)	4.663				4.600				4.645			
Total Feed Rate, From Product (W/W/Hr)	4.51	4.50	4.48	4.48	4.34	4.40	4.32	4.19	4.44	4.51	4.40	4.04
Total Feed Per Cycle From Feed (W/W/Cycle)	.3886				.3833				.3871			
Total Feed Per Cycle From Product (W/W/Cycle)	.3762	.3747	.3730	.3729	.3618	.3669	.3599	.3494	.3696	.3759	.3666	.3368
Sievate Per Cut (W/W)												
Sievate Per Cycle (W/W/Cycle)	.3117	.3081	.3096	.3102	.3081	.3091	.3100	.2947	.3147	.3178	.3127	.2813
Fresh Feed Per Cycle	.3563				.3506				.3533			
Fresh Feed Rate (W/W/Hr)	4.276				4.207				4.240			
Desorption												
Temp (°F)	704	709	715	711	711	709	709	707	704	704	710	710
Pressure (psig)					.75				1.0			
NH_3 Rate (W/W/Hr)	2.02				2.02				2.02			
Desorbate 1												
Time (Sec)	30				30				60			
W/W/Cycle NH_3	.0167				.0167				.0335			
W/W/Cycle	.0416	.0430	.0396	.0395	.0417	.0446	.0384	.0431	.0449	.0481	.0466	.0494
Desorbate 2												
Time (Sec)	270				270				240			
W/W/Cycle NH_3	.1515				.1515				.1345			
W/W/Cycle	.0229	.0236	.0238	.0232	.0120	.0132	.0115	.0116	.0100	.0100	.0073	.0061
Total Desorbate (W W/Cycle)	.0645	.0666	.0634	.0627	.0537	.0578	.0499	.0547	.0549	.0581	.0539	.0555
Total NH_3 (W/W/Cycle)	.1682				.1682				.1680			
Recovery	96.29				93.83				93.6			
Analysis												
Sievate												
Sulfur (Lamp, ppm)			11.3	14.2			12.6/10.7	100/104			14.9	8.0
U.V. Aromatics												
Benzene	1.58	1.64	1.62	1.60	1.66	1.66	1.72	1.57	1.95	1.99	1.93	1.70
Bz, T, Xy	1.58	1.64	1.62	1.60	1.66	1.66	1.72	1.57	1.95	1.99	1.93	1.70
Feed Dist. Aromatics												
Total Aromatics	1.58	1.64	1.62	1.60	1.66	1.66	1.72	1.57	1.95	1.99	1.93	1.70
Desorbate 1												
Sulfur (Lamp, ppm)												
U.V. Aromatics												
Benzene												
Bz, T, Xy												
Feed Dist. Aromatics	(ABCD - 15.16 Used in Run 966-107-12)				(ABCD - 14.56 Used in Run 966-107-13)				(ABCD - 18.91 Used in Run 966-107-14)			
Total Aromatics												
Desorbate 2												
Sulfur (Lamp, ppm)				35.6				350				
U.V. Aromatics												
Benzene												
Bz, T, Xy												
Feed Dist. Aromatics				182.2				199.7				157.65
Total Aromatics				182.2				199.7				157.65
Total Feed, Sulfur (Lamp)	110/107				113				103/100			

APPENDIX 3 (CONT.)

CYCLIC OPERATION WITH AMMONIA DESORPTION

Run 966 - 107

Sieve Used $Ca^{+2}X(2N, 2, O, L)$

Condition - Period	14				15				16				
Time, Hrs	8				8				10				
No. of Cycles in Period	48				47				60				
Sub-Period	A	B	C	D	A	B	C	D	A	B	C	D	E
Time, Hrs	2	2	2	2	2	2	2	2	2	2	2	2	2
No. of Cycles in Sub-Period	12	12	12	12	12	11	12	12	12	12	12	12	12
Cut No.													
Time of Cut (Min)													
Unit Data													
Adsorption													
Temp (°F)	709	712	710	712	708	707	706	710	707	722	725	707	709
Pressure (psig)	.25				.25				.25				
Time (Min)	5				5				5				
Feed Used	CK 6978				CK 6978				CK 6978				
Total Feed Rate, From Feed (W/W/Hr)	4.175				5.202				4.271				
Total Feed Rate, From Product (W/W/Hr)	4.44	4.42	3.54	3.35	4.38	5.20	4.57	4.60	2.966	4.33	4.00	3.16	4.81
Total Feed Per Cycle From Feed (W/W/Cycle)	.3479				.4335				.3559				
Total Feed Per Cycle From Product (W/W/Cycle)	.3698	.3684	.2947	.2788	.3649	.4335	.3811	.3834	.2472	.3606	.3336	.2630	.4006
Sievate Per Cut (W/W)													
Sievate Per Cycle (W/W/Cycle)	.3153	.3138	.2404	.2272	.2959	.3639	.3295	.3328	.2015	.3107	.2850	.2154	.3519
Fresh Feed Per Cycle	.3138				.3908				.3221				
Fresh Feed Rate (W/W/Hr)	3.766				4.690				3.865				
Desorption													
Temp (°F)	710	710	712	710	711	710	717	711	718	712	714	711	700
Pressure (psig)	.25		1.25		1.0	1.5		.5	.50		.52		.5
NH_3 Rate (W/W/Hr)	2.02				2.02				2.02				
Desorbate 1													
Time (Sec)	60				60				60				
W/W/Cycle NH_3	.0335				.0335				.0335				
W/W/Cycle	.0476	.0482	.0486	.0454	.0506	.0497	.0454	.0448	.0409	.0438	.0427	.0420	.0425
Desorbate 2													
Time (Sec)	240				250				240				
W/W/Cycle NH_3	.1345				.1345				.1345				
W/W/Cycle	.0069	.0064	.0057	.0062	.0184	.0199	.0062	.0058	.0048	.0061	.0059	.0056	.0062
Total Desorbate (W/W/Cycle)	.0545	.0546	.0543	.0516	.0690	.0696	.0516	.0506	.0457	.0499	.0486	.0476	.0487
Total NH_3 (W/W/Cycle)	.1680				.1680				.1680				
Recovery	95.02				89.93				90.22				
Analysis													
Sievate													
Sulfur (Lamp, ppm)			9.8/9.2/ 8.0				18.1					19.5/15.5/ 12.6	24.5/25.2
U.V. Aromatics													
Benzene													
Bz, T, Xy	2.54	2.80	1.47	1.15	2.32	3.22	3.32	3.47	2.71	2.81	2.78	1.91	
Feed Dist. Aromatics													3.39
Total Aromatics	2.54	2.80	1.47	1.15	2.32	3.22	3.32	3.47	2.71	2.81	2.78	1.91	3.39
Desorbate 1													
Sulfur (Lamp, ppm)													
U.V. Aromatics													
Benzene													
Bz, T, Xy													
Feed Dist. Aromatics													
Total Aromatics													
Desorbate 2													
Sulfur (Lamp, ppm)													482
U.V Aromatics													
Benzene													
Bz, T, Xy													
Feed Dist. Aromatics				126.1				134.23					136.03
Total Aromatics				126.1				134.23					136.03
Vent Line													
Benzene													
Bz, T, Xy													
Feed Dist. Aromatics											(CDE 111.11)		
Total Aromatics													
Total Feed, Sulfur (Lamp)	114												

APPENDIX 3 (CONT.)

CYCLIC OPERATION WITH AMMONIA DESORPTION

Run 966 - 107

Sieve Used Ca^{+2}X(2N, 2, 0, L)

Condition - Period	17		18		19		20		21		22		23	
Time, Hrs	8		8		8		8		8		8		8	
No. of Cycles in Period	48		48		48		48		48		48		48	
Sub-Period	A	B	A	B	A	B	A	B	A	B	A	B	A	B
Time, Hrs	4	4	4	4	4	4	4	4	4	4	4	4	4	4
No. of Cycles in Sub-Period	24	24	24	24	24	24	24	24	24	24	24	24	24	24
Cut No.														
Time of Cut (Min)														
Unit Data														
Adsorption														
Temp (°F)	725	709	708	709	707	709	709	707	709	704	706	708	707	712
Pressure (psig)	.25		.25		.25		.25		.25		.25		.25	
Time (Min)	5		5		5		5		5		5		5	
Feed Used	CK 6978		CK 6978		CK 6978		CK 6978		CK 6978		CK 6979		CK 6979	
Total Feed Rate, From Feed (W/W/Hr)	4.902		5.192		4.795		4.612		4.908		4.762		4.762	
Total Feed Rate, From Product (W/W/Hr)	4.537	4.4568	4.57	4.63	4.62	4.58	4.57	4.07	4.54	4.57	4.55	4.57	4.61	4.59
Total Feed Per Cycle From Feed (W/W/Cycle)	4.085		.4327		.3996		.3843		.4090		.3968		.3970	
Total Feed Per Cycle From Product (W/W/Cycle)	.3781	.3714	.3807	.3857	.3848	.3819	.3807	.3388	.3784	.3806	.3795	.3808	.3839	.3821
Sievate Per Cut (W/W)														
Sievate Per Cycle (W/W/Cycle)	.3282	.3239	.3318	.3361	.3361	.3344	.3338	.2954	.3325	.3338	.3353	.3354	.3360	.3359
Fresh Feed Per Cycle	.3724		.3944		.3638		.3512		.3759		.3636		.3651	
Fresh Feed Rate (W/W/Hr)	4.469		4.733		4.366		4.214		4.511		4.363		4.381	
Desorption														
Temp (°F)	712	703	712	711	714	711	712	716	702	712	721	714	714	713
Pressure (psig)	.5		.5		.5		.5		.5		.5		.5	
NH$_3$ Rate (W/W/Hr)	2.02		2.02		2.02		2.02		2.02		2.02		2.02	
Desorbate 1														
Time (Sec)	60		60		60		60		60		60		60	
W/W/Cycle NH$_3$	.0335		.0335		.0335		.0335		.0335		.0335		.0335	
W/W/Cycle	.0428	.0419	.0425	.0426	.0421	.0409	.0404	.0373	.0399	.0404	.0379	.0395	.0406	.0392
Desorbate 2														
Time (Sec)	240		240		240		240		240		240		240	
W/W/Cycle NH$_3$	.1345		.1345		.1345		.1345		.1345		.1345		.1345	
W/W/Cycle	.0070	.0056	.0063	.0070	.0065	.0066	.0065	.0061	.0060	.0064	.0063	.0059	.0073	0070
Total Desorbate (W/W/Cycle)	.0499	.0475	.0488	.0496	.0486	.0475	.0469	.0434	.0459	.0468	.0442	.0454	.0479	.0462
Total NH$_3$ (W/W/Cycle)	.1680		.1680		.1680		.1680		.1680		.1680		.1680	
Recovery	91.76		88.58		95.94		93.65		92.83		95.84		96.49	
Analysis														
Sievate														
Sulfur (Lamp, ppm)		23.2/20.9		27.8/29.3		23.8		27		28.4		28.8		27.3
U.V. Aromatics														
Benzene														
Bz, T, Xy														
Feed Dist. Aromatics	3.42	2.87	3.14		3.09	3.09	3.21	3.15	3.21	2.94	3.36	3.21	3.18	3.15
Total Aromatics	3.42	2.87	3.14		3.09	3.09	3.21	3.15	3.21	2.94	3.36	3.21	3.18	3.15
Desorbate 1														
Sulfur (Lamp, ppm)														
U.V. Aromatics														
Benzene														
Bz, T, Xy														
Feed Dist. Aromatics														
Total Aromatics														
Desorbate 2														
Sulfur (Lamp, ppm)		682		532		496		512		560		596		643
U.V. Aromatics														
Benzene														
Bz, T, Xy														
Feed Dist. Aromatics		144.14		114.1	140.5	133.0		153.2		180.18		125.22		134.53
Total Aromatics		144.14		114.1	140.5	133.0		153.2		180.18		125.22		134.53
Vent Line														
Benzene														
Bz, T, Xy														
Feed Dist. Aromatics	A+B 111.11													
Total Aromatics														
Total Feed, Sulfur (Lamp)														

APPENDIX 3 (CONT.)

CYCLIC OPERATION WITH AMMONIA DESORPTION

Run 966 - 107

Sieve Used Ca^{+2}X(2N, 2, 0, L)

Condition - Period	24		25		26		27		28		29	
Time, Hrs	8		8		8		8		64		64	
No. of Cycles in Period	48		48		48		48		8		8	
Sub-Period	A	B	A	B	A	B	A	B	A	B	A	B
Time, Hrs	4	4	4	4	4	4	4	4	4	4	4	4
No. of Cycles in Sub-Period	24	24	24	24	24	24	24	24	32	32	32	32
Cut No.												
Time of Cut (Min)												
Unit Data												
Adsorption												
Temp (°F)	70370	3708	706	708	709	709	697	699	708	704	701	710
Pressure (psig)	0		0		0		0		0		0	
Time (Min)	5		5		5		5		2.5		2.5	
Feed Used	CK 6979 + Thiophene to 1000 ppm S						CK 6979 + Thiophene to 1000 ppm S					
Total Feed Rate, From Feed (W/W/Hr)	4.8364		4.6363		4.6974		4.6943		4.378		4.80	
Total Feed Rate, From Product (W/W/Hr)	4.4892	4.5462	4.3392	4.5036	4.4676	4.5216	4.494	4.5036	3.7392	4.5312	4.5360	4.5528
Total Feed Per Cycle From Feed (W/W/Cycle)	.4030		.3862		.3912		.3910		.1824		.2000	
Total Feed Per Cycle From Product (W/W/Cycle)	.3741	.3789	.3616	.3753	.3723	.3768	.3745	.3753	.1558	.1888	.1890	.1897
Sievate Per Cut (W/W)												
Sievate Per Cycle (W/W/Cycle)	.3305	.3356	.3191	.3344	.3293	.3326	.3305	.3318	.1153	.1481	.1478	.1488
Fresh Feed Per Cycle												
Fresh Feed Rate (W/W/Hr)												
Desorption												
Temp (°F)	712	712	712	714	726	718	718	714	717	717	718	714
Pressure (psig)	.25		.25		1.25		1.25		.50		.50	
NH_3 Rate (W/W/Hr)	2.0171		2.0171		4.0342		4.0342		2.0171		2.0171	
Desorbate 1												
Time (Sec)	30		30		15		15		30		30	
W/W/Cycle NH_3	.0168		.0168		.0084		.0084		.0168		.0168	
W/W/Cycle	.0330	.0327	.0324	.0323	.0311	.0318	.0311	.0312	.0311	.0309	.0311	.0312
Desorbate 2												
Time (Sec)	270		270		285		285		270		270	
W/W/Cycle NH_3	.1512		.1512		.1596		.1596		.1512		.1512	
W/W/Cycle	.0106	.0106	.0101	.0086	.0119	.0124	.0129	.0123	.0094	.0098	.0101	.0097
Total Desorbate (W/W/Cycle)	.0436	.0433	.0425	.0409	.0430	.0442	.0440	.0435	.0405	.0407	.0412	.0409
Total NH3 (W/W/Cycle)	.1680		.1680		.1680		.1680		.1680		.1680	
Recovery	93.5		95.39		95.7		95.88		94.52		94.63	
Analysis												
Sievate												
Sulfur (Lamp, ppm)	739	688	720	780	790	750	790	660	690	680	680	7101
U.V. Aromatics												
Benzene												
Bz, T, Xy	.9565	1.335										
Feed Dist. Aromatics				1.216		0.748		0.7687		0.3033		0.1741
Total Aromatics	.9565	1.335		1.216		0.748		0.7687		0.3033		0.1741
Desorbate 1												
Sulfur (Lamp, ppm)		1226		1600		1660		1600		1200		1400
U.V. Aromatics												
Benzene												
Bz, T, Xy												
Feed Dist. Aromatics		57.96		44.44		38.43		39.03		26.42		29.12
Total Aromatics		57.96		44.44		38.43		39,03		26.42		29.12
Desorbate 2												
Sulfur (Lamp, ppm)		531		457		700		900		700		900
U.V. Aromatics												
Benzene												
Bz, T, Xy												
Feed Dist. Aromatics		130.63		127.61		112.01		113.51		103.60		175.67
Total Aromatics		130.63		127.61		112.01		113.51		103.60		175.67
Sulfur % Recovery		73.33		87.52		84.91		76.50		84.73		84.46
Total Feed, Sulfur (Lamp)												

APPENDIX 3 (CONT.)

CYCLIC OPERATION WITH AMMONIA DESORPTION

Run 966-107

Sieve Used $Ca^{+2}X$(2B, 2, 0, L)

Condition - Period	30											
Time, Hrs	2.5											
No. of Cycles in Period	1											
Sub-Period												
Time, Hrs												
No. of Cycles in Sub-Period												
Cut No.	1	2	3	4	5	6	7	8	9	10	11	12
Time of Cut (Min)	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
Unit Data												
Adsorption												
Temp (°F)	699											
Pressure (psig)	0											
Time (Min)	30											
Feed Used	CK 6979 + Benzothiophene to 1000 ppm S											
Total Feed Rate, From Feed (W/W/Hr)	4.4498											
Total Feed Rate, From Product (W/W/Hr)	3.8976											
Total Feed Per Cycle From Feed (W/W/Cycle)	2.2249											
Total Feed Per Cycle From Product (W/W/Cycle)	1.9488											
Sievate Per Cut (W/W)	.150	.1564	.1705	.1738	.1880	.1503	.1414	.1784	.1797	.1656	.2011	.1723
Sievate Per Cycle (W/W/Cycle)	1.9092											
Fresh Feed Per Cycle												
Fresh Feed Rate (W/W/Hr)												
Desorption												
Temp (°F)	708											
Pressure (psig)	5											
NH_3 Rate (W/W/Hr)	2.0171											
Desorbate 1												
Time (Sec)	30											
W/W/Cycle NH_3	.0168											
W/W/Cycle	.0311											
Desorbate 2												
Time (Sec)	7200											
W/W/Cycle NH_3	4.0342											
W/W/Cycle	.0085											
Total Desorbate (W/W/Cycle)	.0396											
Total NH_3 (W/W/Cycle)	4.0510											
Recovery	87.62											
Analysis												
Sievate												
Sulfur (Lamp, ppm)	6.5	2.8	6.3	89		320			1500	875		888
U.V. Aromatics												
Benzene												
Bz, T, Xy		0.568	1.57									
Feed Dist. Aromatics						6.60			8.76			11.29
Total Aromatics		0.568	1.57			6.60			8.76			11.29
Desorbate 1												
Sulfur (Lamp, ppm)												
U.V. Aromatics												
Benzene												
Bz, T, Xy												
Feed Dist. Aromatics					80.48							
Total Aromatics												
Desorbate 2												
Sulfur (Lamp, ppm)												
U.V. Aromatics												
Benzene												
Bz, T, Xy												
Feed Dist. Aromatics												
Total Aromatics												
Total Feed, Sulfur (Lamp)												

APPENDIX 3 (CONT.)

CYCLIC OPERATION WITH AMMONIA DESORPTION

Run 966 - 107
Sieve Used $Ca^{+2}X$(2N, 2, O, L)

Condition - Period	31		32		33		34		35		36	
Time, Hrs	8		8		8		8		8		6	
No. of Cycles in Period	48		50		48		48		64		64	
Sub-Period	A	B	A	B	A	B	A	B	A	B	A	B
Time, Hrs	4	4	4	4	4	4	4	4	4	4	4	2
No. of Cycles in Sub-Period	24	24	25	25	24	24	24	24	32	32	32	32
Cut No.												
Time of Cut (Min)												
Unit Data												
Adsorption												
Temp (°F)	698	706	705	703	699	705	701	713	730	703	724	696
Pressure (psig)	0		0		0		0		0		0	
Time (Min)	5		5		5		5		2.3		2.5	
Feed Used	CK 6979 + Benzothiophene to 1000 ppm S											
Total Feed Rate, From Feed (W/W/Hr)	4.7096		5.196		4.9052		4.9052		488		4.7402	
Total Feed Rate, From Product (W/W/Hr)	4.1064	4.1484	3.9924	3.9996	4.2324	4.2084	4.1940	4.2240	3.9144	4.0344	3.8784	3.8304
Total Feed Per Cycle From Feed (W/W/Cycle)	.3924		.4330		.4087		.4087		.2034		.1575	
Total Feed Per Cycle From Product (W/W/Cycle)	.3422	.3457	.3327	.3333	.3527	.3507	.3495	.3520	.1631	.1681	.1616	.1596
Sievate Per Cut (W/W)												
Sievate Per Cycle (W/W/Cycle)	.3002	.3030	.2921	.2916	.3122	.3091	.3070	.3107	.1267	.1287	.1237	.1216
Fresh Feed Per Cycle												
Fresh Feed Rate (W/W/Hr)												
Desorption												
Temp (°F)	707	712	704	721	709	710	712	714	708	710	711	712
Pressure (psig)			25	.125	1.35		1.35		.5		.5	
NH_3 Rate (W/W/Hr)	2.0171		2.0171		4.0342		4.0342		2.0171		2.0171	
Desorbate 1												
Time (Sec)	30		30		15		15		30		30	
W/W/Cycle NH_3	.0168		.0168		.0168		.0168		.0168		.0168	
W/W/Cycle	.0322	.0319	.0307	.0306	.0304	.0306	.0308	.0306	.0287	.0308	.0300	.0297
Desorbate 2												
Time (Sec)	270		270		285		285		270		270	
W/W/Cycle NH_3	.1512		.1512		.3193		.3193		.1512		.1512	
W/W/Cycle	.0098	.0108	.0099	.0111	.0101	.0110	.0117	.0107	.0077	.0086	.0099	.0083
Total Desorbate (W/W/Cycle)	.0420	.0427	.0406	.0417	.0405	.0416	.0425	.0413	.0364	.0394	.0379	.0380
Total NH_3 (W/W/Cycle)	.1680		.1680		.3361		.3361		.1680		.1680	
Recovery	87.7		76.94		86.07		85.83		81.47		81.41	
Analysis												
Sievate												
Sulfur (Lamp, ppm)		32.6		46.9		34		31.0		12.4		4.9
U.V. Aromatics												
Benzene												
Bz, T, Xy		1.51		2.04		2.35		1.55		0.652		0.401
Feed Dist. Aromatics												
Total Aromatics		1.51		2.04		2.35		1.55		0.652		0.401
Desorbate 1												
Sulfur (Lamp, ppm)		3700		2400		3400		2200		1500		1600
U.V. Aromatics												
Benzene												
Bz, T, Xy												
Feed Dist. Aromatics		52.55		37.53		50.45		65.16		36.93		64.56
Total Aromatics		52.55		37.53		50.45		65.16		36.93		64.56
Desorbate 2												
Sulfur (Lamp, ppm)		7800		12700		10400		9600		6600		7300
U.V. Aromatics												
Benzene												
Bz, T, Xy												
Feed Dist. Aromatics		154.65		147.1		159.15		163.66		94.50		97.79
Total Aromatics		154.65		174.1		159.15		163.66		94.50		97.79
Sulfur % Recovery		57.62		56.15		59.70		46.85		54.80		58.66
Total Feed, Sulfur (Lamp)												

APPENDIX 3 (CONT.)

CYCLIC OPERATION WITH AMMONIA DESORPTION

Run 966 - 107
Sieve Used Ca^{+2}X(2N, 2, 0, L)

Condition - Period	37											
Time, Hrs	30											
No. of Cycles in Period												
Sub-Period												
Time, Hrs												
No. of Cycles in Sub-Period												
Cut No.	1	2	3	4	5	6	7	8	9	10	11	12
Time of Cut (Min)	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
Unit Data												
Adsorption												
Temp (°F)	696											
Pressure (psig)	0											
Time (Min)	30											
Feed Used	CK 6979 + Benzothiophene to 1000 ppm S											
Total Feed Rate, From Feed (W/W/Hr)	6.6210											
Total Feed Rate, From Product (W/W/Hr)	4.8476											
Total Feed Per Cycle From Feed (W/W/Cycle)	3.3105											
Total Feed Per Cycle From Product (W/W/Cycle)	2.4238											
Sievate Per Cut (W/W)	.1764	.1941	.1984	.1998	.1910	.2053	.1937	.2013	.2017	.1982	.2029	.2090
Sievate Per Cycle (W/W/Cycle)												
Fresh Feed Per Cycle												
Fresh Feed Rate (W/W/Hr)												
Desorption												
Temp (°F)												
Pressure (psig)	25											
NH_3 Rate (W/W/Hr)	2.0171											
Desorbate 1												
Time (Sec)	30											
W/W/Cycle NH_3	.0168											
W/W/Cycle	.0503											
Desorbate 2												
Time (Sec)	7200											
W/W/Cycle NH_3	4.0342											
W/W/Cycle	.0014											
Total Desorbate (W/W/Cycle)	.0517											
Total NH_3 (W/W/Cycle)	4.0510											
Recovery	83.5											
Analysis												
Sievate												
Sulfur (Lamp, ppm)	3.4	23.7	137			1600			408			819
U.V. Aromatics												
Benzene												
Bz, T, Xy	0.496											
Feed Dist. Aromatics		1.936	4.44			15.10			11.11			12.01
Total Aromatics	0.496	1.936	4.44			15.10			11.11			12.01
Desorbate 1												
Sulfur (Lamp, ppm)												
U.V. Aromatics												
Benzene												
Bz, T, Xy												
Feed Dist. Aromatics												
Total Aromatics												
Desorbate 2												
Sulfur (Lamp, ppm)												
U.V. Aromatics												
Benzene												
Bz, T, Xy												
Feed Dist. Aromatics												
Total Aromatics												
Total Feed, Sulfur (Lamp)												

APPENDIX 3 (CONT.)

CYCLIC OPERATION WITH AMMONIA DESORPTION

Run 966 - 107

Sieve Used Ca^{+2}X(2N, 2, 0, L)

Condition - Period	38											
Time, Hrs	2.51											
No. of Cycles in Period	1											
Sub-Period												
Time, Hrs												
No. of Cycles in Sub-Period												
Cut No.	1	2	3	4	5	6	7	8	9	10	11	12
Time of Cut (Min)	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
Unit Data												
Adsorption												
Temp (°F)	666											
Pressure (psig)	0											
Time (Min)	30											
Feed Used	Ck. 6979 + Di normal-Butyl Disulfide to 1000 ppm S											
Total Feed Rate, From Feed (W/W/Hr)	5.409											
Total Feed Rate, From Product (W/W/Hr)	4.795											
Total Feed Per Cycle From Feed (W/W/Cycle)	2.705											
Total Feed Per Cycle From Product (W/W/Cycle)	2.3974											
Sievate Per Cut (W/W)	.1443	.1962	.1931	.2256	.1812	.1987	.2027	.1992	.2029	.2041	.2133	.1956
Sievate Per Cycle (W/W/Cycle)												
Fresh Feed Per Cycle												
Fresh Feed Rate (W/W/Hr)												
Desorption												
Temp (°F)												
Pressure (psig)	0											
NH_3 Rate (W/W/Hr)	2.0											
Desorbate 1												
Time (Sec)	30											
W/W/Cycle NH_3	.0167											
W/W/Cycle	.02995											
Desorbate 2												
Time (Sec)	7200											
W/W/Cycle NH_3	4.0											
W/W/Cycle	.01064											
Total Desorbate (W/W/Cycle)	.0406											
Total NH_3 (W/W/Cycle)	4.017											
Recovery, %	92.95											
Analysis												
Sievate												
Sulfur (Lamp, ppm)	1.9	8.8	12			116			242			323
U.V. Aromatics												
Benzene												
Bz, T. X	0.708	1.65	3.98									
Feed Dist. Aromatics						6.66			9.42			8.76
Total Aromatics	*0.708*	*1.65*	*3.98*			*6.66*			*9.42*			*8.76*
Desorbate 1												
Benzene												
Bz, T, Xy												
Feed Dist. Aromatics	60.06											
Total Aromatics	*60.06*											
Desorbate 2												
Sulfur (Lamp, ppm)												
U.V. Aromatics												
Benzene												
Bz, T. Xy												
Feed Dist. Aromatics												
Total Aromatics												
Sulfur % Recovery												
Total Feed, Sulfur (Lamp)												

APPENDIX 3 (CONT.)

CYCLIC OPERATION WITH AMMONIA DESORPTION

Run 966 - 107

Sieve Used $Ca^{+2}X(2N, 2, 0, L)$

Condition - Period	39		40		41		42		43		44	
Time, Hrs	8		8		8		8		8		8	
No. of Cycles in Period	48		48		48		48		48		48	
Sub-Period	A	B	A	B	A	B	A	B	A	B	A	B
Time, Hrs	4	4	4	4	4	4	4	4	4	4	4	4
No. of Cycles in Sub-Period	24	24	24	24	24	24	24	24	24	24	24	24
Cut no.												
Time of Cut (Min)												
Unit Data												
Adsorption												
Temp (°F)	691	691	702	702	707	741	712	706	410	710	716	723
Pressure (psig)	0											
Time (Min)	5.0								2.5			
Feed Used	Ck. 6979 + Di-normal-Butyl Disulfide to 1000 ppm S											
Total Feed Rate, From Feed (W/W/Hr)	4.954		4.940		4.914		4.850		6.735		6.570	
Total Feed Rate, From Product (W/W/Hr)	4.549	4.371	4.443	4.199	4.745	4.631	4.672	4.682	6.250	6.228	6.295	6.283
Total Feed Per Cycle From Feed (W/W/Cycle)	0.4128		0.4116		0.4095		0.4042		0.2806		.2738	
Total Feed Per Cycle From Product (W/W/Cycle)	0.37912	0.36425	0.3704	0.3499	0.3954	0.3859	0.3893	0.3902	0.2604	0.2595	0.2623	0.2618
Sievate Per Cut (W/W)												
Sievate Per Cycle (W/W/Cycle)	0.33869	0.323	0.3299	0.3103	0.357	0.3479	0.3499	0.3499	0.2144	0.2117	0.2144	0.2144
Fresh Feed Per Cycle												
Fresh Feed Rate (W/W/Hr)												
Desorption												
Temp (°F)	710	710	719	703	713	705	711	707	716	726	706	714
Pressure (psig)	0.5				1.5				0.5			
NH_3 Rate (W/W/Hr)	2.0				4.07				2.0			
Desorbate 1												
Time (Sec)	30				15				30			
W/W/Cycle NH_3	.0167				.0170				.0167			
W/W/Cycle	.03140	.03155	.0309	.0304	.0287	.02824	.0287	.0277	0.0362	0.0368	0.03686	0.03687
Desorbate 2												
Temp (Sec)	230				285				270			
W/W/Cycle NH_3	0.15				0.322				0.15			
W/W/Cycle	.00903	.0097	.0096	.0092	.0097	.00977	.0107	.0126	.00977	.01097	0.01106	0.01048
Total Desorbate (W/W/Cycle)	.04043	.04125	.0405	.0396	.03838	.03801	.0394	.0403	.0460	.0478	.0479	.0474
Total NH_3 (W/W/Cycle)	0.1667				0.3392				0.1667			
Recovery %	90.38		92.44		95.40		96.43		92.64		95.73	
Analysis												
Sievate												
Sulfur (Lamp, ppm)		16.1		46		13		34		15.4		22
Desorbate 1												
Sulfur (Lamp, ppm)		924		861		259		526		271		229
Desorbate 2												
Sulfur (Lamp, ppm)		396		443		668		495		451		346
Total Feed												
Sulfur (Lamp, ppm)												
Sulfur % Recovery		10.92		13.28		5.33		9.51		8.01		7.25
Analysis - U.V. Aromatics												
Sievate												
Benzene												
Bz, T, Xy		1.81		2.4		2.03		1.99		0.9		1.07
Feed Dist. Aromatics												
Total Aromatics		1.81		2.4		2.03		1.99		0.9		1.07
Desorbate 1												
Benzene												
Bz, T, Xy												
Feed Dist. Aromatics		52.8		47.4		50.4		45.94		37.83		37.8
Total Aromatics		52.8		47.4		50.4		45.94		37.83		37.8
Desorbate 2												
Benzene												
Bz, T, Xy												
Feed Dist. Aromatics		123.7		129.7		193.7		146.6		150.2		106.3
Total Aromatics		123.7		129.7		193.7		146.6		150.2		106.3

APPENDIX 3 (CONT.)

CYCLIC OPERATION WITH AMMONIA DESORPTION

Run 966 - 107

Sieve Used $Ca^{+2}X(2N, 2, 0, L)$

Condition - Period	45		46		47		48		49		53	
Time, Hrs.	8		8		8		8		8		8	
No. of Cycles in Period	32		32		48		48		48		48	
Sub-Period	A	B	A	B	A	B	A	B	A	B	A	B
Time, Hrs.	4											
No. of Cycles in Sub-Period	16	16	16	16	24	24	24	24	24	24	24	24
Cut No.												
Time of Cut (Min)												
Unit Data												
Adsorption												
Temp (°F)	712	717	717	715	715	724	723	724	653	721	492	497
Pressure (psig)	0											
Time (Min)	10.0				5.0							
Feed Used	Ck. 6979 + Di-normal-Butyl Disulfide to 1000 ppm S											
Total Feed Rate, From Feed (W/W/Hr)	4.57		4.524		4.905		4.944		4.776		4.893	
Total Feed Rate, From Product (W/W/Hr)	4.526	4.381	4.503	4.336	4.668	4.574	4.686	4.700	4.686	4.476	4.711	4.736
Total Feed Per Cycle, From Feed (W/W/Cycle)	0.7616		0.754		0.4088		0.412		0.3980		.4078	
Total Feed Per Cycle, From Product (W/W/Cycle)	.7544	.7301	.7505	.7227	.3890	.3812	.3905	.3917	.3905	.3730	.3926	.3947
Sievate Per Cut (W/W)												
Sievate Per Cycle (W/W/Cycle)	0.7167	0.6921	0.7128	0.6842	0.3543	0.3461	0.3555	0.3587	.3542	.3382	.3597	.3621
Fresh Feed Per Cycle												
Fresh Feed Rate (W/W/Hr)												
Desorption												
Temp (°F)	720	712	711	711	700	710	718	718	708	721	318	521
Pressure (psig)	0.5				3.5	3.0	3.5		.75	--	0.5	
NH_3 Rate (W/W/Hr)	2.0				8.51				2.0			
Desorbate 1												
Time (Sec)	3.0				7.5				30			
W/W/Cycle NH_3	.0167				.0177				.0167			
W/W/Cycle	.03041	.02947	.02943	.02914	.0226	.0232	.02375	.02364	.02829	.02676	.02714	.02649
Desorbate 2												
Time (Sec.)	270				292.5				270			
W/W/Cycle NH_3	0.15				0.6914				0.15			
W/W/Cycle	.00733	.00855	.00825	.00935	.01206	.01189	.0112	.00932	.00802	.00805	.00633	.00613
Total Desorbate (W/W/Cycle)	.0377	.0380	.03768	.03849	.03466	.03511	.03495	.03296	.03631	.03481	.03347	.03262
Total NH_3 (W/W/Cycle)	0.1667				.7091				0.1667			
Recovery, %	97.44		97.68		94.22		94.91		95.91		96.55	
Analysis												
Sievate												
Sulfur (Lamp, ppm)		113				25.6		9.0		23.5		158
Desorbate 1												
Sulfur (Lamp, ppm)		625				810		741		542		3000(.3%)
Desorbate 2												
Sulfur (Lamp, ppm)		327				738		520		487		566
Total Feed, Sulfur (Lamp)												
Sulfur % Recovery		15.45				10.36		7.34		6.24		39.27
U. V. Aromatics												
Sievate												
Benzene		.4										
Bz, T, Xy		4.84		4.35		2.17		2.4		2.66		3.54
Feed Dist. Aromatics												
Total Aromatics		4.84		4.35		2.17		2.4		2.66		3.54
Desorbate 1												
Benzene												
Bz, T, Xy												
Feed Dist. Aromatics		63.06		57.0		54.05		48.0		39.9		85.28
Total Aromatics		63.06		57.0		54.05		48.0		39.9		85.28
Desorbate 2												
Benzene												
Bz, T, Xy												
Feed Dist. Aromatics		177.2		192.1		150.1		157.9		138.1		162.2
Total Aromatics		177.2		192.1		150.1		157.9		138.1		162.2

APPENDIX 3 (CONT.)

CYCLIC OPERATION WITH AMMONIA DESORPTION

Run 966 - 107

Sieve Used $Ca^{+2}X$(2N, 2, 0, L)

Condition, Period	50											
Time, Hrs	16.51											
No. of Cycles in Period	1											
Sub-Period												
Time, Hrs.												
No. of Cycles in Sub-Period												
Cut No.	1	2	3	4	5	6	7	8	9	10	11	12
Time of Cut (Min)	2.5											
Unit Data												
Adsorption												
Temp (°F)	720											
Pressure (Psig)												
Time (Min)	30											
Feed Used	Ck. 6979 + Di-normal-Butyl Disulfide to 1000 ppm S											
Total Feed Rate, From Feed (W/W/Hr)	4.32											
Total Feed Rate, From Product (W/W/Hr)	3.928											
Total Feed Per Cycle, From Feed (W/W/Cycle)	2.16											
Total Feed Per Cycle, From Product (W/W/Cycle)	1.9641											
Sievate Per Cut (W/W)	.0550	.0458	.1039	.1496	.1740	.2259	.2114	.1754	.1907	.1895	.1956	.2183
Sievate Per Cycle (W/W/Cycle)												
Fresh Feed Per Cycle												
Fresh Feed Rate (W/W/Hr)												
Desorption												
Temp (°F)	714											
Pressure (psig)	75											
NH_3 Rate (W/W/Hr)	2.0											
Desorbate 1												
Time (Sec)	30											
W/W/Cycle NH_3	.0167											
W/W/Cycle	.02897											
Desorbate 2												
Time (Sec)	57,600											
W/W/Cycle NH_3	1408											
W/W/Cycle												
Total Desorbate (W/W/Cycle)	.02897											
Total NH_3 (W/W/Cycle)	1409.0167											
Recovery, %	90.78											
Analysis												
Sievate												
Sulfur (Lamp, ppm)	26	11.0	29			80			76			165
U. V. Aromatics												
Benzene												
Bz, T. Xy		0.73	1.68									
Feed Dist. Aromatics	1.29					6.51			22.97			9.15
Total Aromatics	1.29	0.73	1.68			6.51			22.97			9.15
Desorbate 1												
Benzene												
Bz, T, Xy												
Feed Dist. Aromatics					75.97							
Total Aromatics					75.97							
Desorbate 2												
Benzene												
Bz, T, Xy												
Feed Dist. Aromatics												
Total Aromatics												

APPENDIX 3 (CONT.)

CYCLIC OPERATION WITH AMMONIA DESCRIPTION

Run 966 - 107

Sieve Used $Ca^{+2}X(2N, 2, 0, L)$

Condition - Period	51											
Time, Hrs.	16.51											
No. of Cycles in Period	1											
Sub-Period												
Time, Hrs.												
No. of Cycles in Sub-Period												
Cut No.	1	2		4	5	6	7	8	9	10	11	12
Time of Cut (Min)	2.5											
Unit Data												
Adsorption												
Temp (°F)	709											
Pressure (Psig)	0											
Time (Min)	30											
Feed Used	Ck. 6979 + Di-normal-Butyl Disulfide to 1000 ppm S											
Total Feed Rate, From Feed (W/W/Hr)	5.232											
Total Feed Rate, From Product (W/W/Hr)	4.839											
Total Feed Per Cycle From Feed (W/W/Cycle)	2.616											
Total Feed Per Cycle From Product (W/W/Cycle)	2.4194											
Sievate Per Cut (W/W)	.1594	.1848	.1955	.2004	.2127	.2121	.1866	.2056	.2011	.1958	.2041	.2336
Sievate Per Cycle (W/W/Cycle)												
Fresh Feed Per Cycle												
Fresh Feed Rate (W/W/Hr)												
Desorption												
Temp (°F)	715											
Pressure (psig)	.75											
NH_3 Rate	2.0											
Desorbate 1												
Time (Sec)	30											
W/W/Cycle NH_3	.0167											
W/W/Cycle	.0277											
Desorbate 2												
Temp (Sec)	57,600											
W/W/Cycle NH_3	1408											
W/W/Cycle												
Total Desorbate (W/W/Cycle)	.0277											
Total NH_3 (W/W/Cycle)	1408.0167											
Recovery, %	92.50											
Analysis												
Sievate												
Sulfur (Lamp, ppm)	344	122	82			70			77			121
U.V. Aromatics												
Benzene												
Bz, T, Xy	1.3	2.68	5.43									
Feed Dist. Aromatics						12.31			8.58			9.00
Total Aromatics	1.3	2.68	5.43			12.31			8.58			9.00
Desorbate 1												
Benzene												
Bz, T, Xy												
Feed Dist. Aromatics					63.96							
Total Aromatics					63.96							
Desorbate 2												
Benzene												
Bz, T, Xy												
Feed Dist. Aromatics												
Total Aromatics												

APPENDIX 3 (CONT.)

CYCLIC OPERATION WITH AMMONIA DESORPTION

Run 966 - 107

Sieve Used $Ca^{+2}X$(2N, 2, 0, L)

Condition - Period	52											
Time, Hrs.	16.51											
No. of Cycles in Period	1											
Sub-Period												
Time, Hrs.												
No. of Cycles in Sub-Period												
Cut No.	1	2	3	4	5	6	7	8	9	10	11	12
Time of Cut (Min)	2.5											
Unit Data												
Adsorption												
Temp (°F)	495											
Pressure (psig)	0											
Time (Min)	30											
Feed Used	Ck. 6979 + Di-normal-Butyl Disulfide to 1000 ppm S											
Total Feed Rate, From Feed (W/W/Hr)	5.33											
Total Feed Rate, From Product (W/W/Hr)	4.758											
Total Feed Per Cycle From Feed (W/W/Cycle)	2.665											
Total Feed Per Cycle From Product (W/W/Cycle)	2.379											
Sievate Per Cut (W/W)	.1402	.1787	.1864	.1991	.2266	.2194	.1848	.1838	.1815	.2139	.2101	.2289
Sievate Per Cycle (W/W/Cycle)												
Fresh Feed Per Cycle												
Fresh Feed Rate (W/W/Hr)												
Desorption												
Temp (°F)	508											
Pressure (psig)	.5											
NH_3 Rate (W/W/Hr)	2.0											
Desorbate 1												
Time (Sec)	30											
W/W/Cycle NH_3	.0167											
W/W/Cycle	.0240											
Desorbate 2												
Time (Sec)	57,600											
W/W/Cycle NH_3	1408											
W/W/Cycle	.00158											
Total Desorbate (W/W/Cycle)	.0256											
Total NH_3 (W/W/Cycle)	1408.0167											
Recovery, %	89.29											
Analysis												
Sievate												
Sulfur (Lamp, ppm)	245	72	202			191			348			368
U.V. Aromatics												
Benzene												
Bz, T, Xy	0.975	1.92	3.76									
Feed Dist. Aromatics						5.25			6.84			7.68
Total Aromatics	0.975	1.92	3.76			5.25			6.84			7.68
Desorbate 1												
Benzene												
Bz, T, Xy												
Feed Dist. Aromatics	133.6											
Total Aromatics	133.6											
Desorbate 2												
Benzene												
Bz, T, Xy												
Feed Dist. Aromatics												
Total Aromatics												

APPENDIX 3 (CONT.)

CYCLIC OPERATION WITH AMMONIA DESORPTION

Run 966 - 107

Sieve Used $Ca^{+2}X(2N, 2, 0, L)$

Condition - Period	54		55		56		57		58	
Time, Hrs	8									
No. of Cycles in Period	48						64		32	
Sub-Period	A	B	A	B	A	B	A	B	A	B
Time, Hrs	4									
No. of Cycles in Sub-Period	24						32		16	
Cut No.										
Time of Cut (Min)										
Unit Data										
Adsorption										
Temp (°F)	500	497	499	498	503	502	492	494	504	
Pressure (psig)	0				.25		0			
Time (Min)	5.0						2.5		10.0	
Feed Used	Ck. 6979 + Di-normal-Butyl Disulfide to 1000 ppm S									
Total Feed Rate, From Feed (W/W/Hr)	4.706		4.776		4.825		4.621		4.272	
Total Feed Rate, From Product (W/W/Hr)	4.705	4.452	4.522	4.630	4.459	4.201	4.522	4.130	4.155	4.168
Total Feed Per Cycle From Feed (W/W/Cycle)	0.3921		0.398		0.4021		.1925		0.712	
Total Feed Per Cycle From Product (W/W/Cycle)	.3921	.3710	.3768	.3858	.3716	.3501	.1884	.1721	.6925	.6946
Sievate Per Cut (W/W)										
Sievate Per Cycle (W/W/Cycle)	.3607	.3409	.3479	.3559	.3460	.3224	0.1629	0.1469	.6624	.6664
Fresh Feed Per Cycle										
Fresh Feed Rate (W/W/Hr)										
Desorption										
Temp (°F)	515	513	516	514	519	518	515	511	522	516
Pressure (psig)		0.5	1.5		4.0		.75			
NH_3 Rate (W/W/Hr)	2.0		4.07		8.51		.75			
Desorbate 1										
Time (Sec)	30		15		7.5		30			
W/W/Cycle NH_3	.0167		.0170		.0177		.0167			
W/W/Cycle	.02586	.02472	.02276	.02257	.0199	.0186	.0214	.0205	.02548	.02419
Desorbate 2										
Time (Sec)	270		285		292.5		270			
W/W/Cycle NH_3	0.15		0.322		0.6914		0.15			
W/W/Cycle	.00558	.00537	.00617	.00729	.00573	.0091	.0041	.0047	.00466	.004003
Total Desorbate (W/W/Cycle)	.03144	.03009	.0289	.0299	.02564	.0277	.0255	.0252	.03014	.02819
Total NH_3 (W/W/Cycle)	0.1667		0.3392		0.7091		0.1667			
Recovery, %	97.31		95.79		89.75		93.65		97.40	
Analysis										
Sievate										
Sulfur (Lamp, ppm)		266		85		321		159		210
Desorbate 1										
Sulfur (Lamp, ppm)		2200		2200		2800		2000		1667
Desorbate 2										
Sulfur (Lamp, ppm)		689		486		679		840		1141
Total Feed, Sulfur (Lamp)										
Sulfur % Recovery		43.35		23.96		49.54		43.70		31.96
U.V. Aromatics - Sievate										
Benzene										
Bz, T, Xy		3.29		3.42				1.68		4.88
Feed Dist. Aromatics						2.55				
Total Aromatics		3.29		3.42		2.55		1.68		4.88
Desorbate 1										
Benzene										
Bz, T, Xy										
Feed Dist. Aromatics		54.0		48.9		75.9		30.6		67.5
Total Aromatics		54.0		48.9		75.9		30.6		67.5
Desorbate 2										
Benzene										
Bz, T, Xy										
Feed Dist. Aromatics		148.0		162.7		148.6		114.1		178.0
Total Aromatics		148.0		162.7		148.6		114.1		178.0

APPENDIX 4

CYCLIC OPERATION WITH AMMONIA DESORPTION SECOND SIEVE CHARGE

Run 966 - 108

Sieve Used $Ca^{+2}X$(2N, 2, 0, L)

Condition - Period	1											
Time, Hr.	2.51											
No. of Cycles in Period	1											
Sub-Period												
Time, Hrs.												
No. of Cycles in Sub-Period												
Cut No.	1	2	3	4	5	6	7	8	9	10	11	12
Time of the Cut (Min)	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
Unit Data												
Adsorption												
Temp (°F)	699											
Pressure (psig)	0											
Time (Min)	30											
Feed (Used)	Ck. 6979											
Total Feed Rate, From Feed (W/W/Hr)	4.556											
Total Feed Rate, From Product (W/W/Hr)	3.9552											
Total Feed Per Cycle From Feed (W/W/Cycle)	2.278											
Total Feed Per Cycle From Product (W/W/Cycle)	1.9776											
Sievate Per Cut (W/W)	.0642	.1545	.1824	.1545	.1801	.1830	.1841	.1557	.1636	.1784	.1811	.1960
Sievate Per Cycle (W/W/Cycle)												
Fresh Feed Per Cycle												
Fresh Feed Rate (W/W/Hr)												
Desorption												
Temp (°F)	715											
Pressure (psig)												
NH_3 Rate (W/W/Hr)	1.85											
Desorbate 1												
Time (Sec)	30											
W/W/Cycle NH_3	.0154											
W/W/Cycle	.0284											
Desorbate 2												
Time (Sec)	7200											
W/W/Cycle NH_3	3.70											
W/W/Cycle	.0489											
Total Desorbate (W/W/Cycle)	.0773											
Total NH_3 (W/W/Cycle)	3.724											
Recovery, %	87.46											
Analysis												
Sievate												
Sulfur (Lamp, ppm)	502(1)	1139 1036	14.3 12.7	578		264 354	54.5	300	27.4 18.6		14.6	167 125
U.V. Aromatics												
Benzene			.010									
Bz, T, Xy						1.91						
Feed Dist. Aromatics	.03	.021							4.50			5.34
Total Aromatics	.03	.021	.010			1.91			4.50			5.34
Desorbate 1												
Sulfur (Lamp, ppm)												
U.V. Aromatics												
Benzene												
Bz, T Xy												
Feed Dist. Aromatics	44.14											
Total Aromatics												
Desorbate 2												
Sulfur (Lamp, ppm)												
U.V. Aromatics												
Benzene												
Bz, T, Xy												
Feed Dist. Aromatica	145.6											
Total Aromatics												
Total Feed, Sulfur (Lamp)												

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(1) Sulfur in lines from earlier run caused the erratic sulfur contents in these first cycle sievate cuts.

APPENDIX 4 (CONT.)

CYCLIC OPERATION WITH AMMONIA DESORPTION SECOND SIEVE CHARGE

Run 966 - 108

Sieve Used $Ca^{+2}X(2N, 2, 0, L)$

Condition - Period	2		3		4	
Time, Hr.	8		8		8	
No. of Cycles in Period	48		48		32	
Sub-Period	A	B	A	B	A	B
Time, Hrs.	4	4	4	4	4	4
No. of Cycles in Sub-Period	24	24	24	24	16	16
Cut. No.						
Time of Cut (Min)						
Unit Data						
Adsorption						
Temp (°F)	704	710	712	711	705	702
Pressure (psig)	0		0		0	
Time (Min)	5		5		10	
Feed (Used)	Ck. 6979					
Total Feed Rate, From Feed (W/W/Hr)	4.3778		4.3693		4.3018	
Total Feed Rate, From Product (W/W/Hr)	4.0884	4.2024	4.208	4.2384	4.1886	4.2132
Total Feed Per Cycle From Feed (W/W/Cycle)	.3648		.3641		.6373	
Total Feed Per Cycle From Product (W/W/Cycle)	.3407	.3502	.3507	.3532	.6981	.7022
Sievate Per Cut (W/W)						
Sievate Per Cycle (W/W/Cycle)	.2730	.2777	.2770	.2888	.6212	.6264
Fresh Feed Per Cycle						
Fresh Feed Rate (W/W/Hr)						
Desorption						
Temp (°F)	709	714	707	708	705	704
Pressure (psig)	1.0		1.0		1.0	
NH_3 Rate (W/W/Hr)	1.8545		1.8545		1.8545	
Desorbate 1						
Time (Sec)	30		30		30	
W/W/Cycle NH_3	.01545		.01545		.01545	
W/W/Cycle	.0349	.0478	.0461	.0406	.0440	.0439
Desorbate 2						
Time (Sec)	270		270		270	
W/W/Cycle NH_3	.1391		.1391		.1391	
W/W/Cycle	.0328	.0247	.0276	.0238	.0329	.0319
Total Desorbate (W/W/Cycle)	.0677	.0725	.0737	.0644	.0769	.0758
Total NH_3 (W/W/Cycle)	.1546		.1546		.1546	
Recovery, %	94.69		96.73		97.65	
Analysis						
Sievate						
Sulfur (Lamp, ppm)		0.2		<0.2		12
U.V. Aromatics						
Benzene		0.52				
Bz, T, Xy				.591		1 01
Feed Dist. Aromatics						
Total Aromatics						
Desorbate 1						
Sulfur (Lamp, ppm)		185		111		230
U.V. Aromatics						
Benzene						
Bz, T, Xy		29.12				
Feed Dist. Aromatics		25.82		30.03		51.95
Total Aromatics						
Desorbate 2						
Sulfur (Lamp, ppm)		173		60		396
U.V. Aromatics						
Benzene						
Bz, T, Xy						
Feed Dist. Aromatics		69.66		78.67		111.11
Total Aromatics						
Total Feed, Sulfur (Lamp)						

APPENDIX 4 (CONT.)

CYCLIC OPERATION WITH AMMONIA DESORPTION SECOND SIEVE CHARGE

Run 966 - 108

Sieve Used Ca^{+2} X(2N, 2, 0, L)

Condition - Period	5											
Time, Hr.	2.51											
No. of Cycles in Period	1											
Sub-Period												
Time, Hrs.												
No. of Cycles in Sub-Period												
Cut No.	1	2	3	4	5	6	7	8	9	10	11	12
Time of Cut (Min)	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
Unit Data												
Adsorption												
Temp. (°F)	712											
Pressure (psig)	40											
Time (Min)	30											
Feed (Used)	Ck. 6979 + Benzothiophene to 1000 ppm											
Total Feed Rate, From Feed (W/W/Hr)	4.4091											
Total Feed Rate, From Product (W/W/Hr)	3.9300											
Total Feed Per Cycle From Feed (W/W/Cycle)	2.2045											
Total Feed Per Cycle From Product (W/W/Cycle)	1.9650											
Sievate Per Cut (W/W)	.0580	.1532	.1699	.1864	.1784	.1770	.1814	.1669	.1743	.2036	.1767	.1392
Sievate Per Cycle (W/W/Cycle)												
Fresh Feed Per Cycle												
Fresh Feed Rate (W/W/Hr)												
Desorption												
Temp (°F)	713											
Pressure (psig)	1.0											
NH_3 Rate (W/W/Hr)	1.8545											
Desorbate 1												
Time (Sec)	30											
W/W/Cycle NH_3	.01545											
W/W/Cycle	.6570											
Desorbate 2												
Time (Sec)	7200											
W/W/Cycle NH_3	3.709											
W/W/Cycle	0.459											
Total Desorbate (W/W/Cycle)	.1029											
Total NH_3 (W/W/Cycle)	3.724											
Recovery,	89.13											
Analysis												
Sievate												
Sulfur (Lamp, ppm)		11.3	0.2			<0.2			266			577
U.V. Aromatics												
Benzene			1.58									
Bz, T, Xy						2.47						
Feed Dist. Armoatics	0.80	1.11							3.54			7.35
Total Aromatics	0.80	1.11	1.58			2.47			3.54			7.35
Desorbate 1												
Sulfur (Lamp, ppm)												
U.V. Aromatics												
Benzene												
Bz, T, Xy												
Feed Dist. Aromatics	63.06											
Total Aromatics												
Desorbate 2												
Sulfur (Lamp, ppm)												
U.V. Aromatics												
Benzene												
Bz, T, Xy												
Feed Dist. Aromatics	247.75											
Total Aromatics												
Total Feed, Sulfur (Lamp)												

APPENDIX 4 (CONT.)

CYCLIC OPERATION WITH AMMONIA DESORPTION SECOND SIEVE CHARGE

Run 966 - 108

Sieve Used $Ca^{+2}X$(2N, 2, 0, L)

Condition - Period	6		7	
Time, Hr.	8			
No. of Cycles in Period	48		48	
Sub-Period	A	B	A	B
Time, Hrs.	4	4	4	4
No. of Cycles in Sub-Period	24	24	24	24
Cut No.				
Time of Cut (Min)				
Unit Data				
Adsorption				
Temp (°F)	708	707	706	709
Pressure (psig)	0		0	
Time (Min)	5		5	
Feed (Used)	Ck. 6979 + Benzothiophene 1000 ppm		Ck. 6979 + Benzothiophene 1000 ppm	
Total Feed Rate, From Feed (W/W/Hr)	4.4346		4.810	
Total Feed Rate, From Product (W/W/Hr)	4.2108	4.2696	5.0748	4.2768
Total Feed Per Cycle From Feed (W/W/Cycle)	.3696		.4008	
Total Feed Per Cycle From Product (W/W/Cycle)	.3509	.3558	.4229	.3564
Sievate Per Cut (W/W)				
Sievate Per Cycle (W/W/Cycle)	.2771	.2824	.3483	.2827
Fresh Feed Per Cycle				
Fresh Feed Rate (W/W/Hr)				
Desorption				
Temp (°F)	707	704	704	707
Pressure (psig)	1.0		1.0	
NH_3 Rate (W/W/Hr)	1.8545		1.8545	
Desorbate 1				
Time (Sec)	30		30	
W/W/Cycle NH_3	.01545		.01545	
W/W/Cycle	.0462	.0460	.04616	.0459
Desorbate 2				
Time (Sec)	270		270	
W/W/Cycle NH_3	.1391		.1391	
W/W/Cycle	.0276	.0274	.0284	.0278
Total Desorbate (W/W/Cycle)	.0738	.0734	.0746	.0737
Total NH_3 (W/W/Cycle)	.1545		.1545	
Recovery, %	95.63		97.20	
Analysis				
Sievate				
Sulfur (Lamp, ppm)		<0.2		4.8
U.V. Aromatics				
Benzene		0.06		0.70
Bz, T, Xy				
Feed Dist. Aromatics				
Total Aromatics				
Desorbate 1				
Sulfur (Lamp, ppm)		1,834		2,361
U.V. Aromatics				
Benzene				
Bz, T, Xy				
Feed Dist. Aromatics		26.42		26.12
Total Aromatics				
Desorbate 2				
Sulfur (Lamp, ppm)		4,854		6,700
U.V. Aromatics				
Benzene				
Bz, T, Xy				
Feed Dist. Aromatics		86.18		88.28
Total Aromatics				
Total Feed, Sulfur (Lamp)				

NDIX 4 (CONT.)

CYCLIC OPERATION WITH AMMONIA DESORPTION SECOND SIEVE CHARGE

Run 966 - 108

Sieve Used $Ca^{+2}X$(2M, 2, 0, L)

Condition - Period	B											
Time, Hr.	2.51											
No. of Cycles in Period	1											
Sub-Period												
Time, Hrs.												
No. of Cycles in Sub-Period												
Cut No.	1	2	3	4	5	6	7	8	9	10	11	12
Time of Cut (Min)	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
Unit Data												
Adsorption												
Temp (°F)	711											
Pressure (psig)	0											
Time (Min)	30											
Feed (Used)	Ft. Belvoir CITE Batch #2											
Total Feed Rate, From Feed (W/W/Hr)	4.445											
Total Feed Rate, From Product (W/W/Hr)	3.9794											
Total Feed Per Cycle From Feed (W/W/Cycle)	2.2273											
Total Feed Per Cycle From Product (W/W/Cycle)	1.9897											
Sievate Per Cut (W/W)	.0114	.1386	.1625	.1714	.1988	.1902	.2275	.1352	.1880	.1826	.1827	.2008
Sievate Per Cycle (W/W/Cycle)												
Fresh Feed Per Cycle												
Fresh Feed Rate (W/W/Hr)												
Desorption												
Temp (°F)	722											
Pressure (psig)	1.0											
NH_3 Rate (W/W/Hr)	1.8545											
Desorbate 1												
Time (Sec)	30											
W/W/Cycle NH_3	.01545											
W/W/Cycle	.0460											
Desorbate 2												
Time (Sec)	7200											
W/W/Cycle NH_3	3.709											
W/W/Cycle	.0273											
Total Desorbate (W/W/Cycle)	.0733											
Total NH_3 (W/W/Cycle)	3.724											
Recovery, %	89.33											
Analysis												
Sievate												
Sulfur (Lamp, ppm)		37.4	241			312			1,730			1,497
U.V. Aromatics												
Benzene	.452	.161	1.90			15.90			20.06			25.57
Bz, T, Xy												
Feed Dist. Aromatics												
Total Aromatics	.452	.161	1.9			15.90			21.96			25.57
Desorbate 1												
Sulfur (Lamp, ppm)	82.6											
U.V. Aromatics												
Benzene												
Bz, T, Xy												
Feed Dist. Aromatics												
Total Aromatics												
Desorbate 2												
Sulfur (Lamp, ppm)	259											
U.V. Aromatics												
Benzene												
Bz, T, Xy												
Feed Dist. Aromatics												
Total Aromatics												
Total Feed, Sulfur (Lamp)												

APPENDIX 4 (CONT.)

CYCLIC OPERATION WITH AMMONIA DESORPTION SECOND SIEVE CHARGE

Run 966 - 108

Sieve Used $Ca^{+2}X$(2N, 2, 0, L)

Condition - Period	9		10		11		12		13	
Time, Hr.	8		8		8		8		8	
No. of Cycles in Period	48		48		64		64		32	
Sub-Period	A	B	A	B	A	B	A	B	A	B
Time, Hrs.	4	4	4	4	4	4	4	4	4	4
No. of Cycles in Sub-Period	24	24	24	24	32	32	32	32	16	16
Cut No.										
Time of Cut (Min)										
Unit Data										
Adsorption										
Temp (°F)	709	708	707	706	707	720	711	710	703	707
Pressure (psig)	0		0		0		0		0	
Time (Min)	5		5		2½		2½		10	
Feed (Used)	Ft. Belvoir CITE Batch #2									
Total Feed Rate, From Feed (W/W/Hr)	4.744		4.5483		4.5213		4.5000		4.0611	
Total Feed Rate, From Product (W/W/Hr)	4.2204	4.1952	4.2540	4.3136	4.2120	4.2168	4.1640	4.1856	4.2042	3.6900
Total Feed Per Cycle From Feed (W/W/Cycle)	.3954		.3790		.1884		.1875		.6768	
Total Feed Per Cycle From Product (W/W/Cycle)	.3517	.3496	.3545	.3595	.1755	.1757	.1735	.1744	.7007	.6150
Sievate Per Cut (W/W)										
Sievate Per Cycle (W/W/Cycle)	.2845	.2869	.2939	.3000	.1208	.1198	.1193	.1204	.6452	.5648
Fresh Feed Per Cycle										
Fresh Feed Rate (W/W/Hr)										
Desorption										
Temp (°F)	716	704	704	703	708	707	711	707	706	704
Pressure	1.0		1.0		1.0		1.0		1.0	
NH_3 Rate (W/W/Hr)	1.8545		1.8545		1.8545		1.8545		1.8545	
Desorbate 1										
Time (Sec)	30		30		30		30		30	
W/W/Cycle NH_3	.01545		.01545		.01545		.01545		.01545	
W/W/Cycle	.0431	.0405	.04078	.0395	.0371	.0367	.03663	.03617	.03844	.03636
Desorbate 2										
Time (Sec)	270		270		270		270		270	
W/W/Cycle NH_3	.1391		.1391		.1391		.1391		.1391	
W/W/Cycle	.0241	.0222	.01986	.01997	.0176	.0192	.01760	.01783	.01701	.01388
Total Desorbate (W/W/Cycle)	.0672	.0627	.0606	.0595	.0547	.0559	.05423	.05400	05545	.05024
Total NH_3 (W/W/Cycle)	.1546		.1546		.1546		.1546		.1546	
Recovery, %	88.69		94.21		93.23		92.80		97.19	
Analsyis										
Sievate										
Sulfur (Lamp, ppm)		80		119		43.1		42		37
U.V. Aromatics										
Benzene						0.65		8.95		
Bz, T, Xy										
Feed Dist. Aromatics		8.59		9.11						38.68
Total Aromatics										
Desorbate 1										
Sulfur (Lamp, ppm)		402		544		215.4		289		287
U.V. Aromatics										
Benzene										
Bz, T, Xy										
Feed Dist. Aromatics		94.42		85.24		57.66		75.41		99.57
Total Aromatics										
Desorbate 2										
Sulfur (Lamp, ppm)		289		95.9		36.5		386		470
U.V. Aromatics										
Benzene										
Bz, T, Xy										
Feed Dist. Aromatics		149.83		165.57		112.91		200.0		184.59
Total Aromatics										
Total Feed, Sulfur (Lamp)										

APPENDIX 4 (CONT.)

CYCLIC OPERATION WITH AMMONIA DESORPTION SECOND SIEVE CHARGE

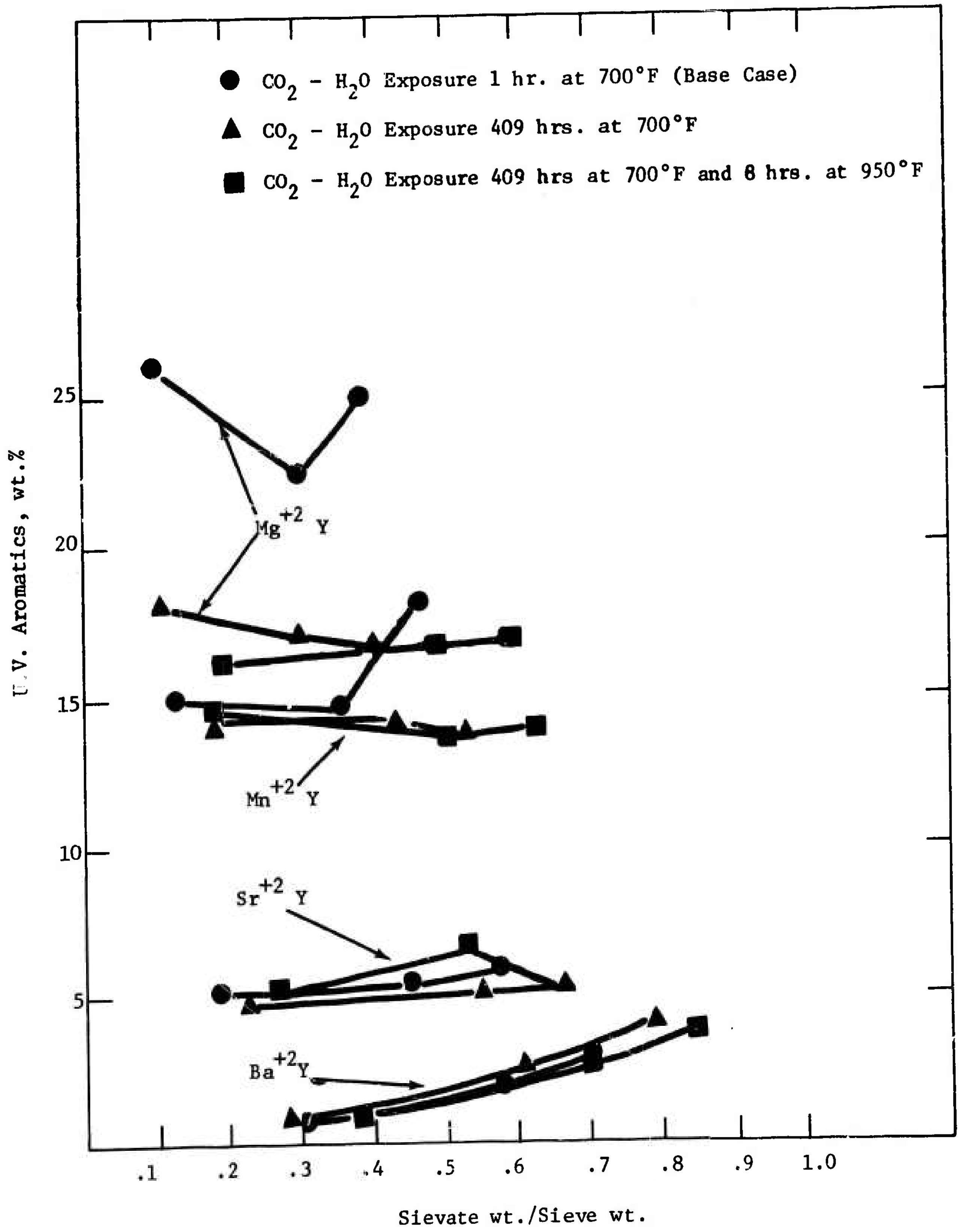
Run 966 - 108

Sieve Used $Ca^{+2}X$(2N, 2, 0, L)

Condition - Period	14		15	
Time, Hr.	8		8	
No. of Cycles in Period	48		48	
Sub-Period	A	B	A	B
Time, Hrs.	4	4	4	4
No. of Cycles in Sub-Period	24	24	24	24
Cut No.				
Time of Cut (Min)				
Unit Data				
Adsorption				
Temp (°F)	709	709	603	603
Pressure (psig)	0		0	
Time (Min)	5		5	
Feed (Used)	Ft. Belvoir CITE Batch #2			
Total Feed Rate, From Feed (W/W/Hr)	4.5369		4.6079	
Total Feed Rate, From Product (W/W/Hr)	4.2400	4.2708	4.2552	4.2396
Total Feed Per Cycle From Feed (W/W/Cycle)	.3781		.3840	
Total Feed Per Cycle From Product (W/W/Cycle)	.3533	.3559	.3546	.3533
Sievate Per Cut (W/W)				
Sievate Per Cycle (W/W/Cycle)	.3033	.3034	.3086	.3106
Fresh Feed Per Cycle				
Fresh Feed Rate (W/W/Hr)				
Desorption				
Temp (°F)	705	704	610	612
Pressure (psig)	2.0		2.0	
NH_3 Rate (W/W/Hr)	3.7704		3.7704	
Desorbate 1				
Time (Sec)	15		15	
W/W/Cycle NH_3	.01571		.01571	
W/W/Cycle	.03336	.03235	.02944	.0264
Desorbate 2				
Time (Sec)	285		285	
W/W/Cycle NH_3	.2985		.2985	
W/W/Cycle	.01669	.02022	.01652	.01634
Total Desorbate (W/W/Cycle)	.05005	.05257	.04596	.04274
Total NH_3 (W/W/Cycle)	.3142		.3142	
Recovery, %	93.80		92.17	
Analysis				
Sievate				
Sulfur (Lamp, ppm)		134		
U.V. Aromatics				
Benzene				
Bz, T, Xy				
Feed Dist. Aromatics		10.23		
Total Aromatics				
Desorbate 1				
Sulfur (Lamp, ppm)		196		352
U.V. Aromatics				
Benzene				
Bz, T, Xy				
Feed Dist. Aromatics		156.72		
Total Aromatics				
Desorbate 2				
Sulfur (Lamp, ppm)		506		
U.V. Aromatics				
Benzene				
Bz, T, Xy				
Feed Dist. Aromatics		180.32		
Total Aromatics				
Total Feed, Sulfur (Lamp)				

Appendix 5

Sieve Stability Under Hybrid Desorption Conditions



APPENDIX 6

HYBRID DESORPTION PERFORMANCE IN AN ADIABATIC SIEVE CASE

Run No. 966-112
Sieve Used Ba^{+2}Y(N, 2, 0, L)
Conditions During Adsorption
Temp. - 700°F
Press. - 0 psig
Feed Rate - 3.0 w/w/hr
Time - 22 minutes
During Hybrid Desorption
CO_2 Rate - 8.1 w/w/hr
D-1 - 1 minute
D-2 Required to Complete Burn

Condition Period	Feed Used	2nd and(1) 3rd Burns Used	Air Rate (w/w/hr)	Oxygen Content (Wt.%)	Base Temp. (°F)	Center T at Peak (°F)			Wall T at Peak (°F)			Time Peak Temp. 3" to 15" (Min.)	Extrapolated Total Time (Min.)	Hydrocarbon Burned (w/w)	Hydrocarbon Desorbed D-1 (w/w)	Hydrocarbon Desorbed D-2 (w/w)	Total Hydrocarbon Desorbed (w/w)	Hydrocarbon Desorbed and Burned (w/w)	Aromatic Adsorption Wt.% Aromatic by U.V.		Sulfur Adsorption ppm Sulfur	
						3"	9"	15"	3"	9"	15"								at 0.2 w/w Sievate	at 1.0 w/w Sievate	at 0.2 w/w Sievate	at 1.0 w/w Sievate
1	JP-4 CK6978	Yes	.611	1.475	718	368	565	660	215	342	400	10	15	.0093	.0165	.0404	.0569	.0750	0.7(3)	5.5		
2	JP-4 CK6978	Yes	.320	0.80	729	125	142	146	91	138	95	37	55-1/2	.0181	.0087	.0096	.0183	.0364	1.2	5.2	13.5	15.5
3	JP-4 CK6978	Yes	.417	1.00	734	175	269	228	125	166	158	24	36	.0153	.0180	.0120	.0300	.0453	1.4	5.4		
4	JP-4 CK6978	No	.417	1.00	705	210	220	253	135	168	178	20	30	.0128	.0180	.0108	.0288	.0416	0.7	4.5		
5	JP-4 CK6978	No	.417	1.00	706	200	238	237	140	162	170	24	36	.0153	.0183	.0093	.0276	.0429	1.8	5.8		
6	JP-4 CK6978	Yes	.417	1.00	716	189	211	208	130	144	145	28	42	.0179	.0177	.0051	.0228	.0407	2.6(4)	6.2	13	25.5
7	JP-4 CK6978	No	.490	1.20	700	218	298	307	150	198	205	16	24	.0120	.0174	.0153	.0327	.0447	0.4(3)	4.3		
8	JP-4 CK6978	No	.611	1.475	700	301	439	458	205	272	295	12	18	.0112	.0177	.0195	.0372	.0484	0.8	5.0	11	32
9	JP-4 CK6978	No	.611	1.475	690	277	487	515	220	305	320	11	16-1/2	.0103	.0183	.0200	.0383	.0486	0.8	4.7		
10(2) a	JP-4 CK6978	No	.611	1.475	696	296	398	413	204	253	268	13	19-1/2	.0122	.0156	.0135	.0291	.0413	--	--		
b	JP-4 CK6978	No	.611	1.475	695	326	398	405	223	253	262	16	24	.0150	.0183	.0150	.0333	.0483	--	--		
c	JP-4 CK6978	No	.611	1.475	699	307	428	493	215	280	310	16	24	.0150	.0186	.0186	.0372	.0522	--	--		
11	JP-4 CK6978	No	.611	1.475	701	313	416	420	220	262	257	14	21	.0130	.0186	.0132	.0318	.0449	2.3(4)	5.8	10.5	30.5
12	JP-4 CK6978	No	.611	1.475	702	309	416	432	210	262	275	13	19-1/2	.0122	.0177	.0177	.0354	.0476	0.8	5.0	12	33.2
13	CITE, Ft. Belvoir	No	.611	1.475	698	285	350	343	197	243	240	19	28-1/2	.0178	.0153	.0180	.0333	.05111	9.0	20.0	50	135
14	CITE, Ft. Belvoir	No	.611	1.475	698	265	350	342	189	238	244	21	31-1/2	.0196	.0147	.0162	.0309	.0505	11.3	20.7	130	145
15	4000 ppm Benzothiophene Sulfur	No	.611	1.475	703	285	363	362	194	257	246	15	22-1/2	.0140	.0177	.0183	.0200	.0340	2.4	17.0	20.7	285
16	4000 ppm Benzothiophene Sulfur	No	.611	1.475	705	271	330	321	186	217	224	16	24	.0150	.0159	.0096	.0255	.0405	3.4	18.8	20	318
17	JP-4 CK6978	No	.611	1.475	705	282	337	325	192	220	223	17	25-1/2	.0159	.0165	.0072	.0237	.0390	3.2	8.8	32.5	59.5

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(1) 2nd Burn of same O_2 concentration as first burn, but with a 950°F base temperature
3rd burn at 21 wt.% O_2 and 950 base temperature.

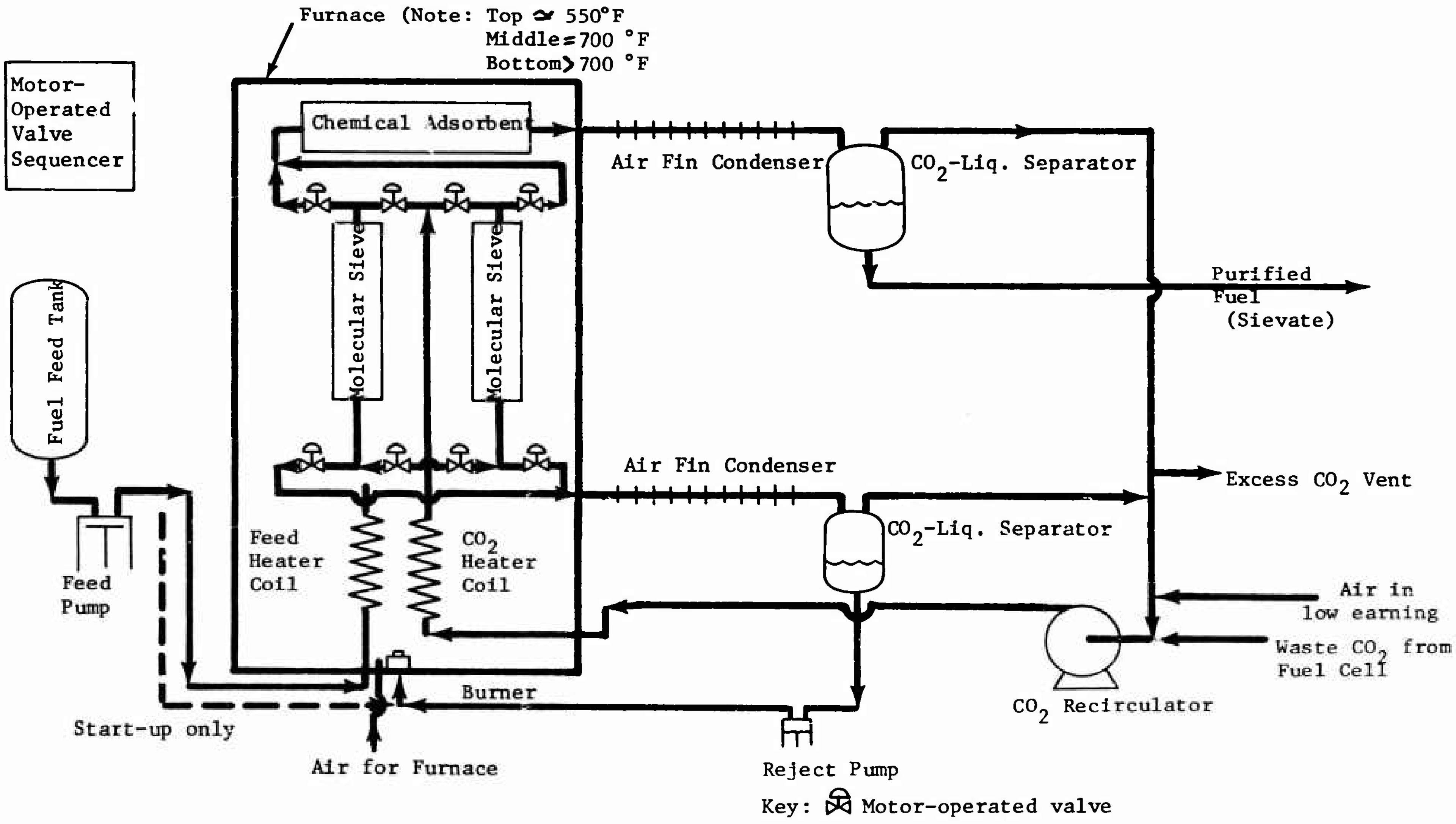
(2) Three cycles of adsorption and desorption were used in this condition period.

(3) Adsorption temperature below 700°F.

(4) Adsorption temperature above 700°F.

Appendix 7

Flow Diagram for Fuel/Purifier Hybrid Desorption Fuel Purifier



APPENDIX 8

HYBRID DESORPTION PROCESS BASIS

The following process basis was derived from the data for processing the maximum allowable 4000 ppm sulfur fuel for 1000 hours. These conditions and performance are believed to systematically away from optimum, however this basis represents demonstrated operation. It was used in projecting the performance and weight of a field unit.

Sieve Bed

- $Ba^{+2}Y$ (2N, 2, 0, L) sieve to be used
- 1.5 inch diameter, 0.20 inch wall tubing sieve case
- 0.28 inch perlite insulation outside tubing
- 18 inches minimum bed length
- Environment temperature equal to inlet gas temperatures

Adsorption Step

- 700°F inlet vapor temperature
- 0 psig outlet pressure
- 20 minute step time
- 0.650 w/w/cycle feed to bed
- 0.600 w/w/cycle sievate out of bed

Hybrid Desorption Step

- 700°F inlet gas temperature
- 0 psig outlet pressure
- 10 w/w/hr CO_2 rate into bed
- 1.475 wt. % oxygen content from air in inlet stream
- 20 minute step time
- 0.015 w/w/cycle of adsorbed material burned in bed
- 0.035 w/w/cycle desorbed from bed

The above process basis was used to size a purifier for 3.75 kw reformer-fuel cell requiring 1.745 lb/hr of purified fuel. The purifier resulting had 0.97 lb. of sieve in each of the two required sieve beds. Molecular sieve cost is typically 1 to 2 dollars a pound. Assuming the sievate sulfur content is the maximum 50 ppm observed with one fuel in this study, 8.75 lb of Esso chemical adsorbent would be required for 1000 hours operation. This adsorbent would be expected to cost about 3 dollars a pound. If the final clean up of the sievate was done in the reformer the chemical adsorbent weight could be reduced. In the reformer the added complications of inclusion of water and hydrogen with the sievate is more readily accomplished. With these two streams added and using a previously demonstrated chemical adsorbent (2), the required chemical adsorbent drops from 8.75 to 3.53 pounds.

APPENDIX 9

SULFUR ANALYSIS

A. High Sulfur Content

Analysis of samples which were high in sulfur (greater than 1000 ppm) were done with the conventional Lamp method - ASTMD 1266. The samples which had these high sulfur contents were usually a feed to the purification process or on initial desorbate to be recycled to the process feed.

B. Low Sulfur Content

Analysis of samples with low sulfur content (less than 1000 ppm) were done by a microcoutrometry method. The products of the purification process, which were typically below 50 ppm, were analyzed by this method. The determination of sulfur by microcoutrometry as it has been evolved by the Analytical Research Division of Esso Research and Engineering Company is given below. This method was designated 60.23 T in November of 1968 by the Analytical Research Division.

Determination of Sulfur by Microcoulometry

Introduction

The method discussed here is a rapid and precise method for determining sulfur in middle distillate fuel oils, solvents, alcohol, lubricating oil base stocks and other hydrocarbons free of metallo-organic compounds. This is a microcoulometric method based on Faraday's Law of Coulometry. It makes use of the Dohrman microcoulometric system apparatus and the combustion tube designed by Drushel and Ellerbe.

Scope

This method is applicable to sulfur levels below 1 ppm to 20%. However, the presence of chlorine and nitrogen in concentrations in excess of 1% and 0.1% respectively may cause interference by liberating iodine from the potassium iodide in the electrolyte by the action of the oxides of nitrogen and chlorine. This can be overcome by the addition of a few milligrams of sodium azide to the electrolyte. It can be used to analyze middle distillate fuel oils, solvents, alcohol, lubricating oil base stocks, and other hydrocarbons free of metallo-organic compounds.

Summary of Method

The sample is diluted with a suitable solvent and a microliter of this is burned in a combustion furnace with oxygen, using helium or argon as a carrier gas. The products of combustion (which include sulfur dioxide when sulfur is present in the samples) are swept into a microcoulometric cell containing an electrolyte and a monitored quantity of iodine. The sulfur dioxide reacts with the titrant (I_3^-) according to the equation:

$$I_3^- + SO_2 + H_2O \longrightarrow SO_3 + 3I^- + 2H$$

As the titrant ion is being consumed, the sensor/reference electrode pair detects this change and sends a signal to the amplifier which in turn sends the voltage back to the generator electrode pair to regenerate the consumed titrant in the cell. The reaction at the generator anode is:

$$3I^- \longrightarrow I_3^- + 2e^-$$

The current flow (measured in microcoulombs) required to regenerate the titrant I_3^- is recorded as a peak on a strip chart, the area of which is proportional to the sulfur content of the sample.

Precision

(From a preliminary cooperative program)

Repeatability: Duplicate results obtained by the same operator should be considered suspect if they differ by more than .03 at the 0.4% level.

Reproducibility: Duplicate results obtained by operators in different laboratories should be considered suspect if they differ by more than .08 at the 0.4% level.

Apparatus

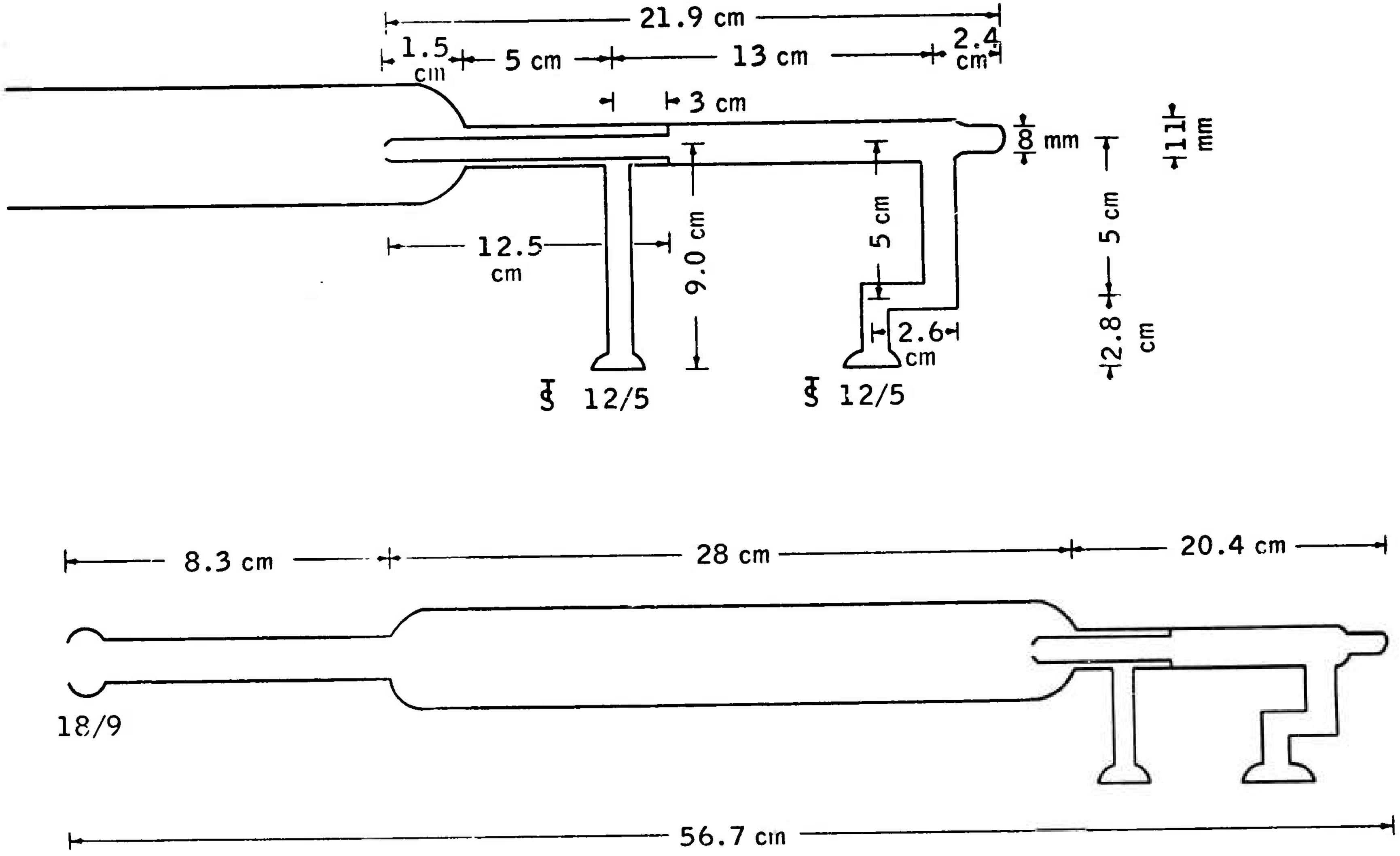
The Dohrmann Recording Microcoulometric titrating system includes items (1) through (10) and can be purchased from Dohrmann Instruments, Co., Analytical Instruments Division, 1062 Linda Vista Ave., Mountain View, California 94040.

(1) Model #C-200-AR Microcoulometer, with magnetic stirrer, power cable, recorder cable and duplex cabinet to accommodate R-100/R-200 recorder.

(2) S-200 Pyrolysis Furnace with power cord, titration cell shelf, § 12/5 pyrex socket.

(3) Model No. R-100-1 Recorder; potentiometric recorder, 0.1 mv/in., 0.5 sec. pen speed, 500K ohms impedance; with power cable and cabinet. A Disc Integrator Model 242-5 should be installed for correct interpretation of coulometric data.

Figure 1



SULFUR COMBUSTION TUBE - COULOMETRIC METHOD

(4) T-300-P Titration Cell

(5) Adapter Tube, Part No. 523733; adapts the § 18/9 ball joint to § 12/5.

(6) Clamp, Thomas #12; clamps the combustion tube to the titration cell.

(7) Clamp, Thomas #18; clamps the scrubber tube to the combustion tube.

(8) Flowmeter, Part No. 523420

(9) Flowmeter, Part No. 523430

(10) Coulometer/Recorder Analyzer

(11) Syringe - 10 μl capacity, available from Precision Sampling Company, P.O. Box 15119, Baton Rouge, Louisiana 70815. (The manufacturer cited here is highly recommended; a high quality syringe is needed.)

(12) Combustion Tube - Figure 1, dimensions of the inlet surface may have to be altered in accordance with the design of the block heater.

Reagent

(1) Electrolyte - Dissolve 0.5 g. potassium iodide and 0.4 ml. of glacial acetic acid in 1000 ml. of distilled water. Store in a brown bottle or in a dark place.

(2) Reactant Gas - Oxygen

(3) Carrier Gas - Either helium or argon. Although nitrogen gas can be used as a carrier, it must be tested for sulfur contamination.

NOTE 1: The baseline should not be more than 1 or 2% of the full scale (0.01 to 0.02 mv) above the true zero at a RANGE OHMS setting of 010.

(4) Solvent for Sample Dilution - Isooctane, toluene or any suitable solvent that is relatively free of sulfur. The solvent should be run as a blank when standards containing only a few ppm. of sulfur are used for calibration.

(5) Organic Sulfur Standard - An organic sulfur compound that has had its sulfur content established by a reliable sulfur method. Phenyl sulfide is satisfactory as a sulfur standard.

(6) High Sulfur Standard Solution - Accurately weigh into a 100 ml. volumetric flask the required quantity of the standard sulfur compound to give a concentration of about 130-150 μg sulfur/ml. Dilute to the mark with the solvent described as Reagent (4).

(7) Iodine, resublimed, Fisher Scientific Co.; I-37 is satisfactory.