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DESULFURIZING HYDROCARBONS WITH LEAD OXIDE-CLAY MIXTURES

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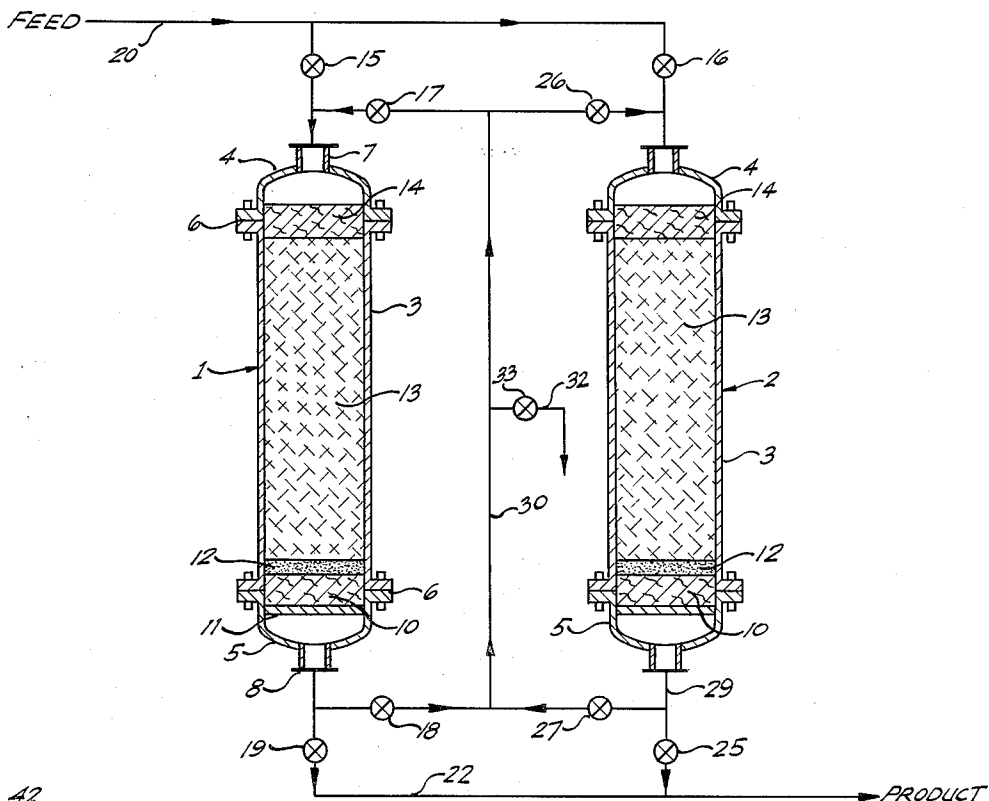


FIG. 1

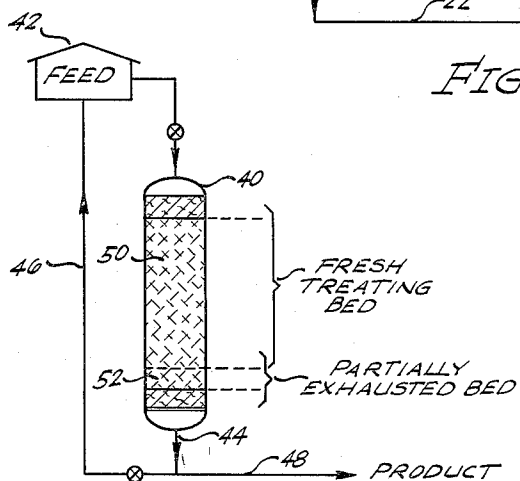


FIG. 2

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**DESULFURIZING HYDROCARBONS WITH LEAD
OXIDE-CLAY MIXTURES**

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This invention relates to the sweetening of mercaptan-contaminated hydrocarbons, and especially light hydrocarbons such as propane, butane, pentane and light naphtha fractions. The new process utilizes as the sweetening agent, a bed of dry, or substantially dry, lead oxide (litharge) mixed with an adsorbent clay such as fuller's earth. The contacting is carried out in such manner, more particularly described hereinafter, as to counteract the normal tendency of the clay-lead oxide treating agent to increase the corrosivity of the feed. In a preferred modification, the feed may be sweetened and simultaneously rendered non-corrosive.

It is an object of this invention to provide an inexpensive sweetening process based on ambient temperature, solid-liquid contacting techniques. Another object is to provide methods for utilizing clay-lead oxide mixtures for sweetening without increasing the corrosivity of the feed. A more specific object is to obtain both sweetening and elimination of corrosive sulfur in a single contacting process. Other objects will become apparent from the following more detailed description.

It is known that mercaptans will react with certain lead compounds to give lead mercaptides. The well-known doctor sweetening process is based upon the reaction of aqueous sodium plumbite with sour hydrocarbons to form lead mercaptides, followed by oxidation of the lead mercaptides with free sulfur to give lead sulfide and the more innocuous alkyl disulfides. However, the standard doctor sweetening process is disadvantageous in that it requires the use of large volumes of expensive reagents, careful control of quantities, and a plurality of treating stages, all of which render the process expensive.

In attempting to carry out a simpler sweetening process by contacting sour hydrocarbons with dry lead oxide-clay mixtures, I have found that sweetening may be obtained, but for unknown reasons the resulting product is much more corrosive than the original feed. In some cases an initially non-corrosive feed is rendered highly corrosive by the treatment. "Corrosiveness" is defined in analytical terms herein as the tendency of a hydrocarbon fraction to discolor a copper strip by the formation of copper sulfide thereon under standard conditions. Corrosiveness is in some cases more undesirable than mercaptan sulfur, since it commonly results in corrosion of storage tanks, pipelines, and valves, and also leads to the formation of undesirable deposits in internal combustion engines. Mercaptan sulfur is ordinarily non-corrosive; corrosive sulfur is thought to comprise for the most part, dissolved free sulfur, hydrogen sulfide, and/or carbonyl sulfide.

In further exploring the clay-lead oxide sweetening process, I have unexpectedly found that when the contacting with sour hydrocarbon is continued for a sufficient length of time, the corrosivity of the product decreases and eventually vanishes. Thus, whereas the initial product is sweet but highly corrosive, the product recovered after several hours of contacting is not only sweet, but non-corrosive to the copper strip test. Presumably some unknown material is present in the fresh bed which either itself contributes corrosive sulfur, or converts non-corrosive sulfur in the feed to corrosive sulfur. In any event, after a few hours on-stream, this material is apparently destroyed or neutralized before the treating capacity of

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the bed has been exhausted. After this point is reached, a feed which is initially sour and corrosive may be rendered both sweet and non-corrosive.

To give best effect to this discovery, I have devised a dual-bed treating process which permits continuous uninterrupted operation, to give a continuously sweet and non-corrosive product, while at the same time permitting periodic renewal of exhausted treating beds. This process may be best understood with reference to FIGURE 1 of the accompanying drawing which illustrates one suitable form of apparatus.

The principal units of apparatus comprise two identical cylindrical reactors 1 and 2 which may be constructed of mild steel, stainless steel, or other suitable structural materials. Each unit comprises a cylindrical shell 3, and upper and lower cup-shaped closure members 4 and 5. The closure members 4 and 5 are removably attached to cylindrical shell 3 by means of annular flanges 6, which are secured together in conventional manner by means of bolts or other suitable means. Each reactor is also equipped with an upper liquid inlet port 7 and product outlet port 8. The appropriate fluid transfer lines, as well as the necessary flow-regulating valves, are indicated schematically and their operation will be described more in detail in connection with the operation of the process.

To illustrate the operation of the process from an initial start-up, each reactor, 1 and 2, is charged with fresh treating beds as follows: First a shallow layer 10 of gravel or other coarse aggregate is placed in the lower section of the reactors, resting upon a perforated supporting plate 11. On top of gravel layer 10, a layer of finer filtering material 12 is deposited to prevent the washing out of lead oxide. Filter layer 12 may consist for example of glass wool, asbestos or other equivalent. The active treating bed 13 is then packed in above filter bed 12, and extends almost to the top of the reactor. Finally, another layer of gravel or aggregate 14 may be placed on top of the treating bed.

With both reactors charged with fresh treating beds, it is next desired to pass the feed through one reactor until that reactor is exhausted of its corrosion-additive propensities. To accomplish this a first contacting sequence is established as follows:

Contacting sequence 1.—Valves 15 and 19 are opened and valves 16, 17, 18 and 25 are closed. The feed then flows in through line 20, valve 15, reactor inlet 7, reactor 1, reactor outlet 8, valve 19 and product outlet line 22. This sequence is continued until tests indicate that the product is non-corrosive to the copper strip test. The initially obtained corrosive product may be recycled to feed storage not shown, and again treated to remove its corrosiveness subsequently in the process.

When the product from reactor 1 becomes non-corrosive, it has been found that passing it through reactor 2, which contains a virgin bed of treating material, will again render it corrosive. It is hence necessary to condition the bed in reactor 2 so as to eliminate its corrosion-additive function. To accomplish this, a second contacting sequence is initiated as follows:

Contacting sequence 2.—Valves 15, 18, 25 and 26 are closed, and valves 16, 27, 17 and 19 are opened, whereby the feed flows through line 20, valve 16, reactor 2, line 29, valve 27, line 30, valve 17, reactor 1, valve 19, and product outlet line 22. During the initial phases of this treating sequence, the product in line 30 is periodically sampled through line 32 by opening valve 33, and tested for corrosivity. The product so sampled is initially corrosive, and hence the necessity for passing it through reactor 1. The treatment in the partially exhausted bed in reactor 1 removes the corrosivity imparted in reactor 2,

and the final product is both sweet and non-corrosive. At this point the start-up operations are complete and continuous production of sweet, non-corrosive product may be indefinitely maintained.

Contacting sequence 2 is continued until the product sampled from line 32 becomes sour to the doctor test. When this occurs, it is an indication that the bed in reactor 2 is exhausted. If it is desired to maintain continuous, uninterrupted operation, a third contacting sequence is initiated as follows:

Contacting sequence 3 (optional).—Valves 16, 17, 18 and 25 are closed, and valves 15 and 19 are opened, whereby the feed flow is exclusively through reactor 1, while the product in line 22 continues sweet and non-corrosive. While this flow is being maintained, the closure members 4 and 5 of reactor 2 are removed, the spent bed is discharged, and a fresh bed of lead oxide and clay is added. It is preferable to accomplish the renewal of the bed in reactor 2 before reactor 1 is exhausted, in order that sufficient treating capacity in reactor 1 will remain to effect a clean-up of the initial corrosion to be added by the new bed in reactor 2 when it is placed on-stream. If continuous operation is not necessary, reactor 1 may of course be simply shut down while reactor 2 is recharged. In this event, the fourth contacting sequence immediately follows the second.

Contacting sequence 4.—To initiate the new bed in reactor 2, valves 15, 26, 25 and 18 are closed, and valves 16, 27, 17 and 19 are opened, whereby the product flows first through reactor 2 then through reactor 1, and into line 22. This operation is continued until the effluent from reactor 2, which may be removed for sampling through line 32, becomes non-corrosive. At this point, or shortly thereafter, it is preferable to switch the feed flow so that it will contact the bed in reactor 1 ahead of the bed in reactor 2, rather than to complete the utilization of the relatively new bed in reactor 2. This provides for smooth, continuous operation, which otherwise might not be obtained if reactor 1 is used merely for stand-by, and/or clean-up treatment. In such an event reactor 2 would be continually recharged, and the eventual exhaustion of reactor 1 might upset the cycle of operations. Hence, after the initial upstream treatment of a new bed, it is preferable to place it downstream so that the oldest bed will be next exhausted. This changeover is effected by initiating a fifth treating sequence.

Contacting sequence 5.—Valves 17, 16, 19 and 27 are closed, and valves 15, 18, 26 and 25 are opened, whereby feed flows first through reactor 1, then line 30, then reactor 2, and into line 22 via valve 25. This flow sequence is maintained until the product sampled through line 32 becomes sour, indicating exhaustion of the bed in reactor 1. At this point, if continuous operation is desired, cycle 6 is initiated.

Contacting sequence 6 (optional).—Valves 15, 26, 27 and 19 are closed and valves 16 and 25 are opened, whereby the feed flows exclusively through reactor 2, and the product in line 22 continues sweet and non-corrosive while the bed in reactor 1 is being renewed. To condition the new bed in reactor 1, a seventh contacting sequence is initiated.

Contacting sequence 7.—Valves 17, 16, 19 and 27 are closed, and valves 15, 18, 26 and 25 are opened, whereby the feed flows through reactor 1, line 30, reactor 2, and into product line 22, and continues sweet and non-corrosive. This flow sequence continues until the product sampled through line 32 becomes non-corrosive. At this point, a complete cycle of regeneration and conditioning has been accomplished, and the valves are hence adjusted for passage first through reactor 2 and then reactor 1 as in sequence 2 above. This cycle of operations may be continued indefinitely without interruption of product flow to obtain a continuously sweet and non-corrosive product.

The above description is not intended to be limiting in scope; obviously many variations may be made without departing from the scope of the invention.

One such variation, which is advantageous from the standpoint of initial cost and simplicity of operation (especially in small operations), consists in using a single treating unit, and when such unit becomes exhausted, renewing or regenerating only the major upstream portion of the bed, and leaving downstreamwardly, near the product outlet, a minor portion of the old bed. This mode of operation is illustrated in FIGURE 2, and is most advantageous where the feed contains not more than about 0.5 gram-atom of corrosive sulfur for each gram-atom of mercaptan sulfur.

To initiate such an operation, the treating unit 40 may be charged with a fresh bed of clay-lead oxide, and the initially corrosive effluent therefrom recycled to feed storage at 42, via lines 44 and 46 for later treatment to remove the corrosive sulfur. As soon as the effluent becomes noncorrosive (and sweet), product is collected from lines 44 and 48 until tests indicate a break-through of mercaptan sulfur. At this point, the downstream portion of the bed is still capable of removing corrosive sulfur, but will not remove mercaptan sulfur.

To regenerate the exhausted bed, it is desirable to replace the major upstream portion thereof with fresh material 50, but to maintain a minor portion 52 of the old bed in place at the effluent end of the reactor. The proportion of the old bed to be retained may vary from about 1% or less, to about 20% of the total. Ordinarily, no more of the old bed is retained than is required to absorb the corrosive sulfur contained in the initial portions of feed traversing the fresh portion of the bed. This amount may vary depending upon specific characteristics of feed, lead oxide charge and the type of clay used.

As an example of this operation, a stream of liquid butane-pentane containing 30 parts per million of mercaptan sulfur and 6 parts per million of elemental sulfur is passed downwardly through a fresh bed of 75% fuller's earth-25% lead oxide (litharge) at a liquid hourly space velocity of about 2. The initial sweet product is found to be more corrosive than the feed, and is hence recycled to the feed storage. After about 18 hours, the corrosion number of the effluent drops to zero, and a sweet and non-corrosive product is then collected for about 60 days, after which the product begins to test sour to the doctor test.

The flow of feed is then terminated and the entire bed is dumped. About 10% thereof, preferably selected mainly from the midsection of the old bed (e.g. the 80% section located above the lower 10% and below the top 10%) is dumped back into the bottom of the reactor. The upper 90% portion is then refilled with fresh clay-lead oxide mixture. The regenerated bed is then capable of producing sweet noncorrosive product for about 55 days from the outset.

In this manner, the regenerations may be continued indefinitely without danger of building up totally exhausted clay-lead oxide mixture (i.e. mixture which is exhausted of both mercaptan-removing and corrosive sulfur-removing capacity) at the bottom of the bed. Thus, a continuously sweet and noncorrosive product is obtained.

The treating beds employed herein preferably consist of mixtures of commercial litharge (PbO), and any suitable adsorbent clay. Suitable clays include for example fuller's earth, bauxite, bentonite and the like. Preferably, the clay should have a slightly alkaline reaction, though this does not appear to be essential. The preferred material is Attapulugus clay, which is a slightly alkaline fuller's earth consisting essentially of hydrous magnesium-aluminum silicate. Other materials such as diatomaceous earth may be employed. The clay is preferably employed in weight-excess of the lead oxide, but any proportions

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which provide a suitably porous bed may be utilized. Preferably, the clay should comprise between about 40% and 90% by weight of the bed, the remainder being lead oxide, and the two components should be intimately admixed by suitable agitation.

The reaction in the treating beds is quite rapid at ordinary atmospheric temperatures, and hence space velocities may range between about 0.5 and 50 liquid volumes per volume of bed per hour, and preferably between about 1 and 20 volumes. The sweetening reactions are substantially more rapid in the case of lower-boiling hydrocarbons, and hence elevated temperatures may be employed when treating higher-boiling materials such as gasoline or kerosene. For low-boiling materials, e.g. propane or light naphthas, preferred temperatures may range between about 50° and 150° F.; for heavier materials up to kerosene, temperatures of about 100° to 200° F. may be preferred.

As indicated, substantially any hydrocarbon feed may be treated herein. The process appears best adapted for the treatment of light hydrocarbons such as propane, butane, pentane, hexane and the like, as well as light gasolines. The high-boiling feeds such as kerosene, especially those containing aromatic hydrocarbons, may tend to dissolve lead mercaptides from the treating bed, and hence suitable precautions must be observed in treating such stocks. Preferably, the feed is treated in liquid phase, but vapor phase operations are also contemplated. Preferably, the feed should contain, or be adjusted to contain, not more than about 0.5 gram atom of corrosive sulfur for each gram atom of mercaptan sulfur. The usual feeds will be found to contain between about 2-20 p.p.m. of elemental sulfur, and about 5-100 p.p.m. of mercaptan sulfur.

When any of the treating beds described herein become totally exhausted it is in most cases economically feasible to discard the exhausted bed and replace it with a new one. However, where considerations of cost are important, the exhausted beds may be regenerated by stripping out the adsorbed hydrocarbons, drying, and oxidizing the bed with air or oxygen at temperatures between about 600° and 1500° F. for several hours. The exhausted beds comprise mainly lead mercaptides with perhaps some lead sulfides, depending upon the nature of the feedstock, and especially the ratio of mercaptan sulfur to corrosive sulfur in the feed.

The doctor test employed herein is well-known, and involves shaking a sample of the hydrocarbon with aqueous sodium plumbite, followed by the addition of a pinch of elemental sulfur to the hydrocarbon phase. A resulting dark color is a positive reaction indicating a sour product, whereas if no coloration develops the product is sweet, i.e. contains less than about 3-4 p.p.m. of mercaptan sulfur.

The copper strip corrosion test employed herein is a standard A.P.I. method, which involves heating a 40 ml. sample of the feed with a ½" x 3" strip of clean, polished copper for 3 hours at 50° C. The strip is then removed and compared with a fresh strip. The degree of corrosivity is indicated on the following scale:

No change.....	0
Slight discoloration.....	1
Brown or peacock shade.....	2
Steel gray.....	3
Black, not scaled.....	4
Black scale.....	5

The following examples will serve to illustrate some of the critical features of the process, but are not intended to be limiting in scope.

Example I

A dual-unit treating plant similar to that illustrated in the drawing was constructed comprising two treating beds, each made up of 600 pounds of 15-30 mesh Attapulgu

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fuller's earth, and 200 pounds of commercial litharge intimately admixed with each other.

It was found that when a liquid propane stream containing 10 parts per million of mercaptan sulfur and 5 parts per million of elemental sulfur was contacted in one of the reactors at atmospheric temperatures, the product after one hour of treatment was doctor sweet, but gave a copper strip corrosion number of 5, whereas the initial feed showed a copper strip corrosion number of only 1. After the contacting had been continued for 24 hours at 750 barrels per day, it was observed that the product was still doctor sweet, but the corrosion number had diminished to zero, indicating no corrosive sulfur.

The flow of feed was then adjusted to pass first through the virgin bed in the second reactor, and then through the partially exhausted bed. The initial product from the virgin bed was again observed to be highly corrosive, while the final product from the second, partially exhausted bed was found to be doctor sweet and had a corrosion number of zero. This clearly illustrates the ability of a partially exhausted bed to eliminate corrosive materials resulting from treatment in a fresh bed.

Example II

A stream of liquid pentane containing 40 parts per million of mercaptan sulfur and 5 parts per million of elemental sulfur was contacted with a fresh 75% fuller's earth-25% lead oxide bed at atmospheric temperature, and at a liquid hourly space velocity of about 1.9. The initial product was sweet, but had a copper strip corrosion number of about 4, whereas the feed had a corrosion number of 1. The product continued to test highly corrosive for several hours. After operating for 24 hours, the corrosion number of the product dropped to zero, and the product was still sweet to the doctor test. This treatment was then continuously employed for 65 days, at which point the product became sour to the doctor test, indicating exhaustion of the bed.

Example III

A blend of light California straight-run and thermally cracked naphthas boiling between about 200° and 350° F. and containing 50 parts per million of mercaptan sulfur, and having a copper strip corrosion number of 4 was treated in a fresh bed as described in Example II, and the product after 8 hours was found to be doctor sweet but had a corrosion number of about 5. Continued treatment for another 24 hours gave at the conclusion thereof, a product of corrosion number zero and negative doctor test.

This application is a continuation-in-part of my prior application Serial Number 614,518, filed October 8, 1956, now abandoned.

The foregoing disclosure of this invention is not to be considered as limiting since many variations may be made by those skilled in the art without departing from the spirit or scope of the following claims.

I claim:

1. In a process for removing corrosive sulfur and mercaptan sulfur from a hydrocarbon feed containing the same, wherein said feed is treated in a contacting column with a bed of clay-lead oxide mixture, the improved method of operating said process to obtain a regenerative operation producing a continuously sweet and noncorrosive product, which comprises (1) first passing said feed through a fresh bed of clay-lead oxide mixture until the mercaptan-removing capacity of said bed is substantially exhausted and then terminating the passage of feed therethrough before the corrosive sulfur-removing capacity of said bed has been exhausted, (2) removing from the contacting column at least the upstreamward major portion of said exhausted bed, (3) refilling the upstreamward major portion of said contacting column with fresh clay-lead oxide mixture while retaining at the downstream extremity thereof a minor portion of said exhausted bed, (4) then resuming the passage of feed through the re-

generated bed to obtain a sweet and noncorrosive product.

2. A process as defined in claim 1 wherein said clay-lead oxide mixture is composed of a major proportion of alkaline fuller's earth, and a minor proportion of commercial litharge.

3. A process as defined in claim 1 wherein said hydrocarbon feed contains at least two gram-atoms of mercaptan sulfur per gram-atom of corrosive sulfur.

4. A process as defined in claim 1 wherein the retained minor portion of said exhausted bed is selected mainly from the 80% midsection of said exhausted bed.

5. A method for regenerating a clay-lead oxide treating bed which has been previously exhausted of its mercaptan-removing capacity by contact with a sulfur-containing hydrocarbon feed but is not yet exhausted of its corrosive sulfur-removing capacity, which comprises replacing the upstreamwardly-located major portion of said exhausted bed with fresh clay-lead oxide mixture, and retaining a minor portion of said exhausted bed at the downstream extremity.

6. A process as defined in claim 5 wherein the retained minor portion of said exhausted bed comprises about 1-20% thereof, and wherein said retained minor portion is selected mainly from the 80% mid-section of said exhausted bed.

7. A process as defined in claim 5 wherein said clay-lead oxide mixture is composed of a major proportion of alkaline fuller's earth, and a minor proportion of commercial litharge.

8. A process for sweetening a sour corrosive hydrocarbon feedstock to obtain a sweetened product of reduced corrosivity, which comprises providing two beds A and B of solid lead oxide-clay mixture, bed A being virgin and bed B being partially exhausted as a result of previous contacting with feedstock for a period of time such that the initially corrosive effluent therefrom has become non-corrosive but is still sweet, establishing a first contacting sequence wherein said feed is passed first through bed A and then through bed B, continuing said first contacting sequence until the initially corrosive effluent from bed A becomes non-corrosive and bed A is partially exhausted as above defined, then establishing a second contacting sequence wherein said feed is passed first through bed B and then through bed A, continuing said second contacting sequence until the initially sweet effluent from bed B becomes sour and bed B is exhausted, then replacing bed B with a new bed C of lead oxide-clay mixture, and reestablishing said first contacting sequence with the feed passing first through said new bed C then through partially exhausted bed A, and recovering a sweetened, non-corrosive product from each of said contacting sequences.

9. A process as defined in claim 8 wherein said contacting beds A, B, and C are composed of a major proportion of an alkaline fuller's earth, and a minor proportion of commercial litharge.

10. A method for sweetening a sour hydrocarbon feedstock to obtain a non-corrosive product therefrom, which comprises passing a stream of said feedstock through a bed of clay and lead oxide to obtain an initially corrosive product, periodically sampling the effluent therefrom and determining its corrosivity, recycling the corrosive, sweetened product initially obtained and blending it with a larger quantity of untreated feedstock in storage until the effluent becomes non-corrosive, and then discontinuing the recycle of effluent to feedstock storage and collecting a sweetened non-corrosive product.

11. A continuous process for treating a sour, corrosive hydrocarbon feedstock to obtain a sweetened product of reduced corrosivity which comprises providing two beds

A and B of solid lead oxide-clay mixture, bed A being virgin and bed B being partially exhausted as a result of previous contacting with feedstock for a period of time such that the initially corrosive effluent therefrom has

5 become non-corrosive but is still sweet, establishing a first contacting sequence wherein said feed is passed first through bed A and then through bed B, continuing said first contacting sequence until the initially corrosive effluent from bed A becomes non-corrosive and bed A is partially exhausted as above defined, then establishing a second contacting sequence wherein said feed is passed first through bed B and then through bed A, continuing said second contacting sequence until the initially sweet effluent from bed B becomes sour and bed B is exhausted, establishing a third contacting sequence wherein said feed is passed only through bed A, and during said third contacting sequence replacing bed B with a new bed C of lead oxide-clay mixture, reestablishing said first contacting sequence with the feed passing first through said new bed C, then through partially exhausted bed A, and recovering a sweetened non-corrosive product from each of said contacting sequences.

12. A process as defined in claim 11 wherein said hydrocarbon feedstock is a lower paraffin hydrocarbon.

13. A process as defined in claim 11 wherein said hydrocarbon feedstock is a light gasoline fraction.

14. A process as defined in claim 11 wherein said hydrocarbon feedstock contains at least two gram-atoms of mercaptan sulfur per gram-atom of corrosive sulfur.

15. A method for treating a sour, corrosive hydrocarbon stream to remove both mercaptan sulfur and corrosive sulfur therefrom, which comprises first pretreating a fresh bed of clay-lead oxide mixture with a sour hydrocarbon stream until the originally corrosive effluent therefrom has become non-corrosive, terminating said pretreatment before the mercaptan-absorbing capacity of said bed has been depleted, then contacting said sour, corrosive hydrocarbon stream with the pretreated bed and recovering a sweet, non-corrosive product, and further treating said originally corrosive effluent to remove corrosive sulfur.

16. A method for treating a sour, corrosive hydrocarbon stream to remove both mercaptan sulfur and corrosive sulfur therefrom, which comprises first pretreating a fresh bed of clay-lead oxide mixture with a sour hydrocarbon stream until the originally corrosive effluent therefrom has become non-corrosive, terminating said pretreatment before the mercaptan-absorbing capacity of said bed has been depleted, then contacting said sour, corrosive hydrocarbon stream with the pretreated bed and recovering a sweet, non-corrosive product.

17. A method for treating a sour, corrosive hydrocarbon stream to remove both mercaptan sulfur and corrosive sulfur therefrom, which comprises first pretreating a fresh bed of clay-lead oxide mixture with a sour hydrocarbon to obtain an initially corrosive product, continuing said pretreatment until the hydrocarbon product is non-corrosive, then, before the corrosive-sulfur removing capacity of said pretreated bed is exhausted, contacting said sour, corrosive hydrocarbon stream with a bed of clay-lead oxide including, at least at the downstream end thereof, said pretreated bed and recovering from said contacting a sweet non-corrosive product.

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