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METHOD FOR SELECTIVELY REMOVING BASIC NITROGEN COMPOUNDS
FROM LUBE OILS USING TRANSITION METAL HALIDES AND
TRANSITION METAL TETRAFLUOROBORATES

A method is disclosed for the selective removal of basic nitrogen compounds (BNC) from natural and synthetic hydrocarbon feedstocks, preferably petroleum feedstocks, most preferably lube and transformer oils, which method, according to the present invention, comprises mixing the feedstock under conditions of agitation and heating to a temperature in the range of from 25 to 120°C with a nonaqueous solution of either (A) an anhydrous nonpolymeric halide, other than chloride, of a metal selected from the metals of Group IVb*, Group Vb, Group VIb, Group VIIb, the non-noble Group VIII metals, copper, zinc, cadmium, and mercury, or (B) an anhydrous nonpolymeric tetrafluoroborate of a metal selected from the metals of Group IVb, Group Vb, Group VIb, Group VIIb, the non-noble Group VIII metals, copper, zinc, cadmium and mercury, which metal halide or metal tetrafluoroborate is complexed with the nonaqueous polar solvent whereby the BNC exchange with the complexed nonaqueous polar solvent and themselves become complexed with the metal halide or tetrafluoroborate, and separating a feedstock of reduced BNC content from the complex of the nonaqueous polar solvent with either the metal halide or metal tetrafluoroborate with which the BNC removed from the feedstock are now complexed by their exchange with the polar solvent. The preferred halide is bromide, and the preferred polar solvent is methanol. The oil may then be decanted to separate it from the metal halide- or fluoroborate-BNC complexes. The separated oil (e.g. the decantate) may be washed with a polar solvent, which preferably includes water, and then preferably dried. The basic nitrogen compound-metal halide or metal tetrafluoroborate complex dissolves in the polar solvent, and that which is in the oil is removed by the polar solvent wash. The preferred polar solvent for the wash step is

*Periodic Table according to "The Handbook of Chemistry and Physics", published by the Chemical Rubber Publishing Company, Cleveland, Ohio, USA.

water. By the practice of this method, the basic nitrogen compound content of the oil is reduced by at least 90%.

The anhydrous nonpolymeric metal halide- or metal tetrafluoroborate-nonaqueous polar solvent complex can be used as such, or it can be impregnated onto a support material such as silica, alumina, silica-alumina, faujasite, kaolin, carbon, zeolite, coal, vermiculite, etc., and used as supported basic nitrogen compound complexation compositions. These supported materials can be regenerated after use by washing with polar solvents. They recover essentially all of their complexation ability.

The reaction can be described in terms of the following formula:



wherein M is the metal component selected from Group IVb, Group Vb, Group VIb, Group VIIb, the non-noble (iron group) metals of Group VIII, copper, zinc, cadmium, mercury; X is a halide, other than chloride, or is the anhydrous nonpolymeric tetrafluoroborate; n is the number of X atoms satisfying the valence requirements of the metal at the oxidation state employed; Q is the complexed nonaqueous polar solvent; z is the number of nonaqueous polar solvent molecules and BNC is basic nitrogen compounds.

The metal M is preferably selected from the group consisting of nickel, chromium, vanadium, zinc, copper, manganese, iron, cobalt, titanium, molybdenum, cadmium, and mercury. The preferred halide is bromide. The most preferred metal bromides are chromium tribromide, nickel dibromide, vanadium dibromide, zinc dibromide, and the copper, manganese, iron and cobalt bromides. These metal bromides are preferably complexed with a nonaqueous polar solvent selected from the group consisting of methanol, ethanol, acetone, acetonitrile, most preferably methanol.

Any oil which can be benefited by the removal of basic nitrogen compounds can be treated by the method of the instant invention. Natural and synthetic hydrocarbon feedstocks, such as those derived from coal, tar sands or oil shale, etc., can thus be processed. Typical of feedstocks which are processed are the petroleum oils destined

1 for use as lubricating or transformer oils wherein the
2 presence of basic nitrogen compounds is known to be a
3 major cause of reduced oxidative stability. These oils
4 need not be pretreated prior to this BNC removal process,
5 since the process functions effectively in the presence of
6 a broad spectrum of contaminants, including, for example,
7 and not by way of limitation, N-methyl pyrrolidone, acids,
8 ionic species, phenols, sulfates, etc. Polar compounds,
9 other than BNC, also contribute to the oxidative instabi-
10 lity of the oils and these too can be removed by use of
11 the metal solution complexes wherein the polar compounds
12 complex to the metal halides or metal tetrafluoroborates.

13 There is no limit on the amount of basic nitro-
14 gen compounds which can be efficiently removed by the in-
15 stant process. Any quantity can be removed provided an
16 effective concentration of metal halide or metal tetra-
17 fluoroborate material is employed. Determination of what
18 constitutes an effective concentration of material is
19 readily achieved by reference to Formula I above once the
20 metal and its oxidation state are substituted into the for-
21 mula. Depending upon the metal selected and its oxidation
22 state as used, from 2 to 6 basic nitrogen compound mole-
23 cules can be removed by complexation to one metal halide
24 or metal tetrafluoroborate molecule. Preferably, a stoi-
25 chiometric amount of metal halide or metal tetrafluorobo-
26 rate (as determined by the metal oxidation state) is em-
27 ployed.

28 As previously stated, the metal halide or metal
29 tetrafluoroborate is employed in this process in the form
30 of a complex with a nonaqueous polar solvent. The con-
31 centration of metal halide or metal tetrafluoroborate,
32 which may be effectively employed in the chosen solvent
33 depends upon the choice of metal and is limited solely by
34 the solubility of the metal halide or metal tetrafluoro-
35 borate in the solvent. Typically, this ranges anywhere
36 from about 0.1 gram material or less per milliliter sol-
37 vent to 1.0 gram material or more per milliliter solvent.

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1 Higher concentrations of metal materials or greater volumes
2 of complex solution are employed for oils more highly con-
3 taminated with basic nitrogen compounds.

4 The oils and metal solution complexes are mixed
5 so as to obtain high surface contact between the oils and
6 the metal solution complexes. This is typically achieved
7 by mixing under conditions of agitation so as to insure
8 complete mixing of the components and the resultant ex-
9 change of BNC with the polar solvent on the metal halide
10 or metal tetrafluoroborate. This agitation can be achieved
11 by any of a variety of standard methods including mechan-
12 ical stirring and bubbling gases, preferably inert gases
13 such as N_2 through the oil-metal solution complex combina-
14 tion.

15 These oil-metal solution complex combinations
16 are subjected to mild heating on the order of a temperature
17 between about 25 to 120°C, preferably 50 to 100°C, most
18 preferably 50-80°C. This heating is employed so as to
19 facilitate the exchange of the BNC for the polar solvent in
20 the metal halide or metal tetrafluoroborate as shown in
21 Formula I.

22 The oil and metal solution complex are mixed and
23 heated for a time sufficient to insure substantially com-
24 plete exchange of the BNC and the polar solvent moiety.
25 The oil is then decanted. The decantate is washed with
26 polar solvent or water to remove any metal halide or metal
27 tetrafluoroborate-BNC complex remaining in the oil. These
28 complexes are soluble in the polar solvent or water. The
29 wash solvent may be employed at any convenient temperature,
30 preferably between 0-20°C. The volume of wash solvent is
31 also, any convenient volume, typically 1-5 volumes was
32 solvent per volume decantate.

33 The oil is dried under any convenient condition.
34 The oil is found to have had its basic nitrogen compound
35 content reduced by at least 90% by the practice of the in-
36 stant process.

37 It must be noted that when the metal halide or

1 metal tetrafluoroborate materials complexed with the polar
2 solvents are described, they are identified as being an-
3 hydrous, nonpolymeric materials; and the polar solvent is
4 identified as being any polar solvent except water. Poly-
5 meric materials are to be avoided since their exchangeable
6 sites are very limited and difficult to gain access to.
7 Further, polymeric metal halides are relatively insoluble
8 in the solvents employed in the instant invention. Simi-
9 larly, the presence of water at the exchange site in place
10 of other polar solvents is to be avoided since water is
11 exchanged only with extreme difficulty and only at tempera-
12 tures high enough to adversely effect the quality of the
13 oil and/or decompose the metal halide (see Table 7).

14 The anhydrous, nonpolymeric metal halides used
15 in the instant invention are prepared by the electro-
16 chemical technique explained in detail in "Electrochemical
17 Preparation of Anhydrous Halides of Transition Metals
18 (Mn-Zn)", by J. J. Habeeb, L. Neilson and D. G. Tuck,
19 Inorganic Chemistry 17(2), 306 (1978).

20 Essentially, the anhydrous, nonpolymeric metal
21 halides are prepared by preparing a solution of nonaqueous
22 polar solvent and halogen, immersing a cathode of a material
23 such as platinum, and an anode made of the desired metal in
24 the solution and applying a current. The reaction is typi-
25 cally carried out under an inert atmosphere such as nitro-
26 gen. After the reaction is stopped, the excess halogen is
27 vented. Metal halide-polar solvent complexes are quite
28 stable if stored under inert atmospheres.

29 The anhydrous, nonpolymeric metal halide and
30 metal tetrafluoroborate-polar solvent complexes can be
31 employed as such in the instant process, or they can be
32 deposited on a suitable inorganic refractory oxide or car-
33 bonaceous support and used as a regenerable supported BNC
34 complexation material. Typical support materials include
35 silica, alumina, natural and synthetic zeolites. carbon,
36 faujasite, callcite, coal, etc.; preferably silica, alumina,
37 and zeolites; most preferably silica and alumina. These

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1 supported complexes are prepared by mixing the chosen
2 support with a volume of metal halide or metal tetrafluoro-
3 borate-nonaqueous polar solvent complex, heating the com-
4 bination at from 50 to 100°C under an inert atmosphere,
5 followed by drying at from 75 to 125°C. The heating and
6 drying steps can be accomplished as a single step. Care
7 is taken not to drive off the complex polar solvent mole-
8 cules. The dried combination is cooled, preferably in an
9 inert atmosphere or under vacuum. The combination metal
10 loading is not critical but will have a typical metal
11 loading range of from 0.5 to 10% metal. Again, the higher
12 the concentration, the more BNC can be removed employing
13 a given volume of supported complex.

14 When these supported materials are used, the oil
15 is contacted with them as by pouring and the BNC are re-
16 moved by exchange with the nonaqueous polar solvent com-
17 plexed with the metal halide or metal tetrafluoroborate.

18 After the theoretical maximum volume of oil has
19 been passed through the supported complex, oil passage is
20 terminated; and the support complex regenerated by washing
21 with acetone or any polar solvent (except water) at tem-
22 peratures between about 25 to 75°C, preferably about 50°C.
23 Supported complexes which are thus regenerated recover
24 essentially all of their ability to remove BNC.

25 The following Examples are presented so as to
26 help describe the invention and are not presented by way
27 of limitation.

28 EXAMPLES

29 Example 1 - Removal of Compounds Containing Basic Nitrogen 30 From Lube Oils by Chromium Tribromide.

31 150 g of a refined transformer oil containing
32 46 ppm basic nitrogen was mixed with 8 g of methanol con-
33 taining approximately 300 mg of chromium tribromide. The
34 solution mixture was heated at 75°C for 15 minutes with
35 nitrogen gas bubbling (or stirring) at a rate of 50 cc/
36 minute to ensure complete mixing. The oil sample was then
37 decanted. The decantate was washed with cold water and

1 dried by heating to 105°C with a nitrogen flow of 250 cc/
2 minute. The chromium complex had been extracted into the
3 water layer.

4 The dried decanted oil had a basic nitrogen con-
5 tent of less than 4 ppm, a reduction of more than 90%.
6 The benefit of removing these basic nitrogen components
7 with CrBr_3 is shown by the fact that the Rotary Bomb Life
8 (ASTM D2112) with 0.06 wt.% 2,6-ditertiarybutyl-para-cresol
9 increased from 179 minutes for the untreated oil to 282
10 minutes for the treated oil.

11 Example 2 - Removal of Compounds Containing Basic Nitrogen
12 From Lube Oils by Nickel Dibromide.

13 175 g of oil was mixed with 10 ml of methanol
14 containing 300 mg of nickel dibromide. The solution mix-
15 ture was heated to 80°C for 15 minutes with stirring and
16 nitrogen gas bubbling at a rate of 100 cc/minute. The oil
17 sample was then decanted. The decantate was washed with
18 cold water and dried by heating to 120°C with nitrogen flow
19 of 300 cc/minute. The nickel complex, a yellow solid, was
20 collected and washed for identification. The degree of
21 basic nitrogen compound removal is presented in Table 1.

22 TABLE 1

Oil	Basic Nitrogen in ppm	
	Before Treatment	After Treatment
60 Neutral	46	3
600 Neutral	81	9
Light Raw Distillate	111	6

29 Example 3 - Removal of Compounds Containing Basic Nitrogen
30 From Lube Oils by Methanolated Vanadium Dibro-
31 mide.

32 175 g of oil was mixed with 15 ml of methanol
33 containing 300 mg of vanadium dibromide. The solution
34 mixture was heated to 80°C for 15 minutes with stirring and
35 nitrogen gas bubbling at a rate of 100 cc/minute. The oil
36 sample was then decanted. The decantate was washed with
37 cold water and dried by heating to 170°C with nitrogen flow

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1 of 400 cc/min. The vanadium complex, a thick dark brown
2 solid, was treated with water to obtain pure basic nitro-
3 gen containing compounds for further investigations. The
4 degree of basic nitrogen compound removal is presented in
5 Table 2.

6 TABLE 2

7	8	9	Oil	Basic Nitrogen in ppm	
				Before	After
				<u>Treatment</u>	<u>Treatment</u>
10			60 Neutral	46	3
11			600 Neutral	81	7
12			Light Raw Distillate	111	1

13 The basic process essentially is the injection of
14 vanadium bromide - methanol solution into oil followed by
15 decantation of the oil and then water washing to remove the
16 vanadium bromide complexes of basic nitrogen compounds.

17 The process could also be applied to upgrade heavy crudes.

18 Example 4 - Removal of Compounds Containing Basic Nitrogen
19 From Lube Oils by Zinc Dibromide and Copper
20 Bromides.

21 Zinc dibromide in methanol and copper bromides
22 (a mixture of Cu(I) and Cu(II) bromides) in methanol are
23 powerful agents for the removal of these basic nitrogen
24 compounds from a wide variety of lube oils--namely, 60
25 neutrals, 600 neutrals and raw distillates--by forming
26 water soluble complexes of zinc bromide and copper bromides.

27 In a typical experiment, 175 g of oil was mixed
28 with 5-fold excess of metal bromides in methanol to basic
29 nitrogen compounds. The solution mixture was heated to
30 80°C for 10 minutes with stirring and nitrogen gas bubbling
31 at a rate of 200 cc/minute. The oil sample was then decan-
32 ted. The decantate was washed with warm water (60°C) and
33 dried by heating to 120°C with nitrogen flow of 700 cc/
34 minute. The metal complexes, either as thick dark brown
35 oil stuck to the walls of the reaction vessel or as brown
36 solid. In both cases, metal complexes were collected for
37 identification. The degree of basic nitrogen compound
38 removal is presented in Table 3.

TABLE 3

	<u>Oil</u>	<u>Basic Nitrogen Content of Oils in ppm</u>		
		<u>Before Treatment</u>	<u>Treated With ZnBr₂</u>	<u>Treated With Copper Bromides</u>
1				
2				
3				
4				
5	60 Neutral	46	1	4
6	600 Neutral	81	3	6
7	Light Raw Distillate	111	1	9

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1 The benefit of removing basic nitrogen compo-
2 nents with ZnBr_2 is shown by the fact that the Rotary Bomb
3 Life (ASTM D2112) with 0.06 wt.% 2,6-ditertiarybutyl-para-
4 cresol increased from 179 minutes for the untreated 60
5 neutral oil to 263 minutes for the treated oil and with
6 0.3 wt.% 2,6-ditertiarybutyl-para-cresol increased from
7 49 minutes for the untreated light Raw Distillates to 173
8 minutes for the treated oil.

9 The basic process for ZnBr_2 and copper bromide
10 treating is envisaged to be injection of the metal bromide-
11 methanol solution into the oil followed by water washing
12 to remove metal bromide basic nitrogen complexes.

13 Example 5 - Removal of Compounds Containing Basic Nitrogen
14 From Lube Oils by Manganese, Iron, and Cobalt
15 Bromides.

16 In a typical experiment, 200 g of oil was mixed
17 with a 5-fold excess of a metal bromide in methanol rela-
18 tive to basic nitrogen compound content. The mixture was
19 heated to 80°C for 10 minutes with stirring and nitrogen
20 gas bubbling at a rate of 200 cc/minute. The oil was then
21 decanted. The decantate was washed with warm water (60°C)
22 and dried by heating to 120°C with nitrogen flow of 700
23 cc/minute. The metal complexes, thick, dark brown (green
24 in the case of cobalt bromide) oils stuck to the walls of
25 the reaction vessel, were collected for identification.
26 The degree of basic nitrogen compound removal is presented
27 in Table 4.

19

TABLE 4

	Oil	Basic Nitrogen Content of Oils in ppm			
		Before Treatment	Treated With $MnBr_2$	Treated With $FeBr_2$	Treated With $CoBr_2$
1					
2					
3					
4					
5	60 Neutral	46	3	4	1
6	600 Neutral	81	6	3	2
7	Light Raw Distillate	111	2	7	3

20

1 Example 6 - Methanolated Transition Metal Tetrafluoro-
2 borates are Basic Nitrogen Scavengers.

3 In a typical experiment, 200 g of oil is mixed
4 with 5-fold excess of metal tetrafluoroborate in methanol
5 relative to basic nitrogen compounds. The solution mix-
6 ture was heated to 80°C for 10 minutes with stirring and
7 nitrogen gas bubbling at a rate of 250 cc/minute. The oil
8 sample was decanted and then filtered. The metal complexes,
9 a thick, dark brown oil, were stuck to the walls of the
10 reaction vessel. The degree to basic nitrogen compound
11 removal is presented in Table 5.

21

TABLE 5

Basic Nitrogen Content of Oils in ppm

	<u>Oil</u>	<u>Before Treatment</u>	<u>After Treatment</u>
1			
2			
3			
4			
5	100 Neutral	25	1
6	Light Raw Distillate	110	4

22

1 Example 7 - Removal of Compounds Containing Basic Nitrogen
2 From Lube Oils by Silica Gel Impregnated with
3 Ti(III), V(II), Cr(III), Mo(II), Mn(II),
4 Fe(II), Co(II), Ni(II), Cu(II) and Zn(II)
5 Bromides.

6 Preparation of the impregnated silica gel was
7 performed as follows. Methanolated complexes of the men-
8 tioned transition metal bromides were prepared by electro-
9 lysis. Samples of these complexes containing 1 to 3 g of
10 metal were mixed with silica gel (different grades) and
11 heated to 70°C under nitrogen followed by drying at 100°C.
12 The new absorbent was cooled in vacuum.

13 In a typical experiment, a 20 cm long and 2.5 cm
14 diameter column was packed with 75 g impregnated silica
15 gel. An unimpregnated silica gel column of the same dimen-
16 sions was also used for comparison. Oil was poured through
17 each of the columns which were kept at 80-90°C. The col-
18 umns need not be maintained at these high temperatures,
19 effective operation being achieved at lower temperatures.
20 Higher temperatures were used to facilitate the rate of
21 flow of high viscosity oils through the column.

22 Silica gel impregnated with the above-mentioned
23 transition metal bromides are regenerated by washing with
24 acetone at 50°C after oil has been recovered. The degree
25 of basic nitrogen compound removal is presented in Table 6.

23

TABLE 6

1	TABLE 6																
2	<u>Column (I)</u>																
3	<u>Silica Gel (Blank)</u>																
4	No. of ml of oil	50	100	150	200	300	400	500									
5	Basic nitrogen (ppm) in Recovered Oil	<2	<2	-5	14	16	21	25									
6																	
7	<u>Column (II)</u>																
8	<u>Impregnated Silica Gel with CrBr₃</u>																
9	No. of ml of oil	250	500	1000	2000	3000	4000										
10	Basic nitrogen (ppm) in Recovered Oil	<1	<1	<1	<2	<2	<2	<2									
11																	
12	<u>Column (III): Column (II) - Regenerated</u>																
13	No. of ml of oil	100	400	600	800	1200	1500										
14	Basic nitrogen (ppm) in Recovered Oil	<2	<2	<2	<2	2	2	2									
15																	

24

1 Similar results may be obtained using the other metal
2 bromide complexes. The benefit of removing basic nitrogen
3 components by this method is shown by the fact that Rotary
4 Bomb Life (ASTM D2112) with 0.3 wt.% 2,6-ditertiarybutyl-
5 para-cresol increased from 49 minutes for the untreated
6 light Raw Distillates (110 ppm BNC) to 238 minutes after
7 treatment (2 ppm BNC).

8 This method has an excellent potential in lube
9 oil processing due to the fact that impregnated silica gel
10 beds can be efficiently regenerated and reused for an
11 indefinite number of cycles.

12 Example 8

13 In a typical experiment, 200 g of oil (60 neutral)
14 containing 46 ppm BNC) was mixed with 400 mg $TiCl_4$ in
15 methanol. The mixture was heated to 80°C for 10 minutes
16 with stirring and nitrogen gas bubbling at a rate of
17 200 cc/minute. The oil was decanted. The decantate was
18 washed with warm water (60°C) and dried by heating to 120°C
19 with nitrogen gas flow of 700 cc/minute. BNC concentration
20 before treatment was 46 ppm while after treatment BNC con-
21 centration was 7 ppm. However, the Rotary Bomb Life
22 (ASTM D2112) with 0.06 wt.% 2,6-ditertiarybutyl-para- cresol
23 decreased from 200 minutes before treatment to 86 minutes
24 after treatment indicating that $TiCl_4$, although removing
25 BNC from oils, has deleterious effect on the treated oil.
26 This may be due to the easy reduction of $TiCl_4$ to $TiCl_3$
27 and the production of the highly reactive chlorine atom
28 which acts as strong oxidizing agent in addition to Ti(iv)
29 which is an oxidizing agent when it reduces to Ti(iii).

30 Elemental sulphur concentration was decreased
31 from 0.24 wt.% before treatment to 0.1 wt.% after treat-
32 ment. This indicates that $TiCl_4$ is non-selective coordina-
33 ting compound. Removal of naturally occurring antioxidant
34 sulphur compounds will also have a detrimental effect on
35 oil.

36 On treating the oil with $FeCl_3$ using exactly the
37 same quantities of materials and procedure, similar results

1 were observed.

Before Treatment			After Treatment (FeCl ₃)		
<u>BNC</u>	<u>S%</u>	<u>RBOT min</u>	<u>BNC</u>	<u>S%</u>	<u>RBOT min</u>
46	0.24	200	5	0.12	132

6 Example 9

7 In a typical experiment, 175 g of oil was mixed
8 with 5-fold excess of metal bromides in water relative to
9 basic nitrogen compounds content. The mixture was heated
10 to (75-80°C) for 10 minutes with stirring and nitrogen gas
11 bubbling at a rate of 200 cc/minute. The oil was decanted.
12 The decantate was washed with warm water (60°C) and dried
13 by heating to 120°C with nitrogen flow of 700 cc/minutes.
14 Results are summarized in Table 7 which show that whereas
15 BNC content dramatically decreases when using a nonaqueous
16 solvent, only minimal reduction in BNC content is achieved
17 when using water as the solvent.

23

	Example	Oil	Before Treatment			After Treatment			Metal Complex (Solvent)	Temperature
			BN(ppm)	S%	BN(ppm)	S%	BN(ppm)	S%		
1	1	60N	46	0.24	4	0.24			CrBr ₃ (Methanol)	75°C
2	2	60N	46	0.24	3	0.24			NiBr ₃ (Methanol)	80°C
3	1	60N	17.5	0.21	1.0	0.21			CrBr ₃ (Methanol)	75-80°C
4	5	600N	81	0.6	6.0	0.6			MnBr ₂ (Methanol)	80°C
5	4	600N	81	0.6	3	0.6			ZnBr ₂ (Methanol)	80°C
6	6	100N	21	0.33	~2	0.33			Zn(BF ₄) ₂ (Methanol)	80°C
7	6	Hydrofined Light Raw Distillate	103	0.29	~2	0.29			Zn(BF ₄) ₂ (Methanol)	75-80°C
8	1	Hydrofined Light Raw Distillate	103	0.29	~3	--			CrBr ₃ (Methanol)	75-80°C
9	9	60N	46	0.24	34	--			CrBr ₃ (H ₂ O)	75-80°C
10	9	60N	46	0.24	29	--			FeBr ₂ (H ₂ O)	75-80°C
11	9	Hydrofined LRD	103	0.29	81	~			CrBr ₃ (H ₂ O)	75-80°C
12	9	60N	46	0.24	21	--			CrBr ₃ (H ₂ O)	140-145°C



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CLAIMS

1. A method for removing basic nitrogen compounds (BNC) from a natural or synthetic hydrocarbon feedstock comprising mixing the feedstock under conditions of agitation and heating to a temperature in the range of from 25 to 120°C with a nonaqueous solution of either (A) an anhydrous nonpolymeric halide, other than chloride, of a metal selected from the metals of Group IVb, Group Vb, Group VIb, Group VIIb, the non-noble Group VIII metals, copper, zinc, cadmium, and mercury, or (B) an anhydrous nonpolymeric tetrafluoroborate of a metal selected from the metals of Group IVb, Group Vb, Group VIb, Group VIIb, the non-noble Group VIII metals, copper, zinc, cadmium and mercury, which metal halide or metal tetrafluoroborate is complexed with the nonaqueous polar solvent whereby the BNC exchange with the complexed nonaqueous polar solvent and themselves become complexed with the metal halide or tetrafluoroborate, and separating a feedstock of reduced BNC content from the complex of the nonaqueous polar solvent with either the metal halide or metal tetrafluoroborate with which the BNC removed from the feedstock are now complexed by their exchange with the polar solvent.

2. The method of claim 1 comprising the step of washing the separated feedstock with polar solvent and drying.

3. The method of claim 2 wherein the polar solvent wash employs water as the polar solvent.

4. The method of any one of claims 1 to 3 wherein the nonaqueous polar solvent is selected from methanol, ethanol, acetone and acetonitrile.

5. The method of any one of claims 1 to 4 wherein the complex of the nonaqueous polar solvent with either the metal halide or the metal tetrafluoroborate is impregnated onto a support material.



28

6. The method of claim 5 wherein the supported complex of nonaqueous polar solvent with either the metal halide or the metal tetrafluoroborate is regenerated after use by washing with nonaqueous polar solvent at a temperature of from 25 to 75°C.

7. The method of any one of claims 1 to 6 wherein the metal is selected from nickel, chromium, vanadium, zinc, copper, manganese, iron, cobalt, titanium, molybdenum, cadmium and mercury.

8. The method of any one of claims 1 to 7 wherein the metal halide is a metal bromide.

9. The method of claim 8 wherein the metal bromide is selected from chromium tribromide, nickel dibromide, vanadium dibromide, zinc dibromide and the copper, manganese, iron and cobalt bromides.

10. The method of any one of claims 1 to 9 wherein the heating is conducted at a temperature in the range of from 50 to 100°C.

11. A method according to any one of claims 1 to 10 substantially as hereinbefore described.

12. A method as in claim 11 substantially as described with reference to the examples.

13. A hydrocarbon feedstock of reduced basic nitrogen compound content obtained by the method of any one of claims 1 to 12.
