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2,604,449

GREASES AND COMPOUNDS FOR MAKING
SAME AND OTHER COMPOSITIONS

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14 Claims. (Cl. 252—33.6)

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This invention relates to greases and other com-
pounds prepared from amic acids and salts of
same. It also relates to such amic acids and
derivatives thereof, including such salts.

Ordinary lubricating greases as now commer-
cially produced are made by the addition of soap
or a mixture of soaps to lubricating oils and/or
other petroleum fractions. The soap or soaps
serve to produce gelation of the mixture. The
gelled mass may vary in consistency from a
solid brick to a viscous oil of a slightly greater
viscosity than the original oil. The usual greases
are made up of calcium or soda or calcium and
soda soaps in petroleum oil or other petroleum
substance. Instead of soda, potassium or lithium
soaps may be used alone or in combination with
calcium, barium or strontium soaps. These cal-
cium, soda and potassium greases have well-
known disadvantages. For example, when using
sodium or potassium soaps, the resultant greases
are not resistant to water and, where used in
wet places, form emulsions which run off the
parts being lubricated. The lime soap grease and
the soda and potassium soap greases have poor
operating qualities at low temperature, each pro-
ducing high starting torques at such low tem-
peratures, for example, at -20°F . On the other
hand, when the calcium greases are heated up
to the melting point, the water of gelation, the
so-called "tie" water, is boiled off if the tempera-
ture remains at such point for any considerable
time. Thereafter, when the grease cools, the
soap tends to separate from the oil since it lacks
the "tie" water. When the sodium and potas-
sium greases are heated up and cooled, they tend
to set to a much heavier mass which is unstable
to shearing forces. The calcium greases, further-
more, have relatively low dropping points. Calci-
um and sodium and potassium greases have little
rust preventive action. The barium and stron-
tium greases require at least half again as much
soap per equivalent gelling efficiency as the so-
dium greases. Barium and strontium greases
are water resistant and resist temperatures up
to about 275°F ., but gradually harden so as to
lose their efficiency as lubricants. The ordinary
lithium greases have resistance to water, but
they do not function satisfactorily in applica-
tions where ambient temperatures exceed 350°F .

In addition, all of the greases heretofore made
and mentioned above or proposed, except that in
the copending application of Bryant and Frost,
Serial No. 37,534, filed July 8, 1948, now Patent No.
2,594,286, have the disadvantage that they are
made from fats and fatty acids which have con-

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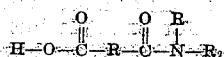
siderable variation in their constitution as pro-
cureable on the market from time to time and
hence cause considerable variation in the greases
produced therefrom.

The principal object of the present invention,
accordingly, is to produce greases and other com-
pounds prepared from salts of amic acids which
shall overcome at least some of these disadvan-
tages and which shall have superior characteris-
tics to those found in the hitherto known greases
mentioned above. Another object of the inven-
tion is to provide a suitable base for produc-
ing such greases. Another object of the inven-
tion is to provide a series of new and useful com-
pounds consisting of amic acids, derivatives and
compounds thereof.

The invention accordingly comprises the novel
products, the specific embodiments of which are
described hereinafter by way of example and in
accordance with which we now prefer to practice
the invention.

We have found in accordance with our inven-
tion that a composition containing a proportion
of an alkali metal salt or a polyvalent metal salt
of amic acid having the formula

(A)



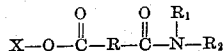
where R is selected from the group consisting of
methylene groups having a carbon chain length
of 1 to 8 carbon atoms and the group



R₁ is an aliphatic radical of a carbon chain
length of 4 to 18 carbon atoms, and R₂ is selected
from the group consisting of hydrogen and an
aliphatic radical of a carbon chain length of 4
to 18 carbon atoms, mixed with a proportion of
petroleum substance such as various petroleum
oils, petrolatum or like substances, produces use-
ful lubricating characteristics. When the salt
of amic acid (or grease base) given in the formula
is present in the proportion of about 6 to about
17% of the grease, it forms a smooth, semi-solid
mass at room temperature. Compounds con-
taining more than 17% range upwardly to an ex-
tremely hard consistency. The salt of amic acid
(or grease base) may be contained in other pro-
portions, say, from 0.5% to 30% of the composi-
tion for varying purposes. With 0.5% composi-
tion using a fluid petroleum oil such as cylinder
oil, the composition is smooth and somewhat
thickened with respect to the cylinder oil but is
still fluid.

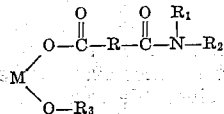
According to the present invention, the use of the amic acid derivatives produces a substantially more uniform grease base. It is true that there may be some variation in the content of the amines employed to produce the amic acids, but in general it is possible to secure amines and other constituents which are approximately 98% pure and we prefer to use such compounds in making up our greases and other compounds in order to avoid the difficulties encountered in the use of natural fats and fatty acids as heretofore employed. The salts of amic acid prepared by us in accordance with our invention include the following:

(B)



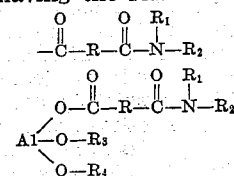
where X is an alkali metal, and R, R₁ and R₂ are as above defined.

(C)

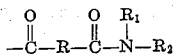


where M is an alkaline earth metal; R, R₁ and R₂ are as above defined, and R₃ is selected from the group consisting of hydrogen and an amic acid radical having the formula

(D)



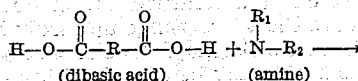
where R, R₁ and R₂ are as above defined, and R₃ and R₄ are each selected from the group consisting of hydrogen and an amic acid radical having the formula



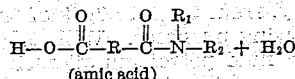
The substances prepared by us in accordance with our invention may be either condensations of a primary amine with a dibasic acid or they may be condensations of a secondary amine with a dibasic acid. For example but without limitation, as indicated below, we have produced in accordance with our invention, N-mono-lauryl adipamic acid prepared from a primary amine, and N-di-lauryl adipamic acid prepared from a secondary amine. It is not necessary, of course, that the radicals, R₁ and R₂, as given in the above formulae, should be identical. They may be the same or they may be radicals of different chain lengths within the definitions given therefor. Thus, we recognize compounds such as N-lauryl-palmityl-adipamic acid and similar substituted acids where R₁ and R₂ are of different chain lengths as falling under the formulae above given.

The amic acids of our invention result from the condensation of an amine with a dibasic acid in stoichiometrical proportions so as to amidate only one of the available carboxyl groups in accordance with the following general equations:

(E)



(F)



where R, R₁ and R₂ are as hereinabove defined.

We may employ in accordance with our invention the following dibasic acids falling under the above Formula E: Itaconic acid, adipic acid (hexanedioic acid), succinic acid (butanedioic acid), azelaic acid (nonanedioic acid), sebacic acid (decanedioic acid), and malonic acid (propanedioic acid), and others.

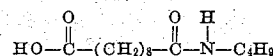
Illustrative of the amines which may be either primary or secondary amines are the following: Mono-n-butylamine, mono-n-hexylamine, mono-n-octylamine, mono-n-decylamine, mono-laurylamine, mono-myristylamine, mono-palmitylamine, mono-stearylamine, mono-linoleylamine, mono-oleylamine, di-octylamine, di-laurylamine, di-palmitylamine and di-stearylamine. These amines, as procurable, may contain a mixture of amines, which will react and appear in the final product. Thus, lauryl amine may contain small quantities of other amines such as octylamine, but we prefer to keep the quantity of such impurities limited to a small amount.

The following are examples illustrative of our invention of (I) Amic acids, (II) Salts of such acids and greases made therefrom, and (III) Mixed amic acids and greases therefrom; including processes for making each of Groups I, II and III. It is to be understood that these examples together with the examples of the dibasic acids and amines given above are illustrative and the invention is not to be considered as limited thereto except as indicated in the appended claims.

The molecular weights of the amic acids which are given below as theoretical are the molecular weights calculated from the reacting weights of the commercial ingredients which as heretofore stated may contain small amounts of impurities. To the extent that these impurities occur, the stated theoretical molecular weights of the compounds differ from the theoretical molecular weights of compounds of 100% purity.

(I) AMIC ACIDS

1. N-mono-n-butyl-sebacamic acid



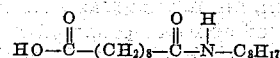
Parts

Mono-n-butylamine 73 (26.4%)
Sebacic acid 203 (73.6%)

The sebacic acid was heated to its melting point (approximately 270° F.). The butylamine was added slowly, with no agitation, the temperature being permitted to rise to 290-310° F. after all of the amine had been added. After two (2) hours at 290-310° F., acidity checking revealed the end of the reaction. The loss in weight, as water, was determined as a cross-check on the end point of the reaction.

Test Data	Theoretical	Actual
Water loss.....percent.....	6.53	6.61
Acidity—calculated as oleic acid.....do.....	109.3	110.0
Melting point.....°C.....		80.0
Nitrogen.....percent.....	5.43	5.38
Mean molecular weight.....	258	256.1
Color.....		White
Odor.....		Bland

2. N-mono-n-octyl-sebacamic acid



Parts

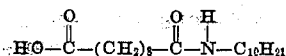
Mono-n-octylamine 129 (38.85%)
Sebacic acid 203 (61.15%)

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The process was carried out as in Example 1, but the reaction was complete in 2½ hours, as indicated by the acidity test.

Test Data	Theoretical	Actual
Water loss.....percent.....	5.42	5.60
Acidity—calculated as oleic acid.....do.....	89.80	89.95
Melting point.....°C.....		88.9
Nitrogen.....percent.....	4.46	4.41
Mean molecular weight.....	314	313.8
Color.....		White
Odor.....		Bland

3. N-mono-decyl-sebacamic acid

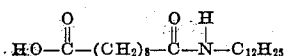


	Parts
Decylamine.....	157 (43.6%)
Sebacic acid.....	203 (56.4%)

The batch was processed as for Example 1, except that the reaction time was three (3) hours.

Test Data	Theoretical	Actual
Water loss.....percent.....	5.0	5.2
Acidity—calculated as oleic acid.....do.....	82.4	82.9
Melting point.....°C.....		94.5
Nitrogen.....percent.....	4.09	3.98
Mean molecular weight.....	342	340
Color.....		White
Odor.....		Spicy

4. N-mono-lauryl-sebacamic acid

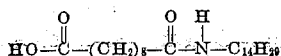


	Parts
Laurylamine.....	185 (47.7%)
Sebacic acid.....	203 (52.3%)

The batch was processed as for Example 1, except that the reaction time was four (4) hours.

Test Data	Theoretical	Actual
Water loss.....percent.....	4.64	4.70
Acidity—calculated as oleic acid.....do.....	76.2	76.7
Melting point.....°C.....		93.5
Nitrogen.....percent.....	3.78	3.70
Mean molecular weight.....	370	368
Color.....		White
Odor.....		Spicy

5. N-mono-myristyl-sebacamic acid

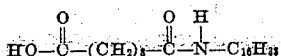


	Parts
Myristylamine.....	213 (51.2%)
Sebacic acid.....	203 (48.8%)

The batch was made up exactly as for Example 4.

Test Data	Theoretical	Actual
Water loss.....percent.....	4.33	4.41
Acidity—calculated as oleic acid.....do.....	70.8	71.3
Melting point.....°C.....		103.5
Nitrogen.....percent.....	3.51	3.47
Mean molecular weight.....	398	395
Color.....		White
Odor.....		Spicy

6. N-mono-palmityl-sebacamic acid



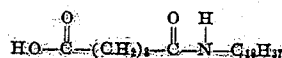
	Parts
Palmitylamine.....	242 (54.3%)
Sebacic acid.....	203 (45.7%)

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The batch was made up exactly as for Example 4.

Test Data	Theoretical	Actual
Water loss.....percent.....	4.05	4.20
Acidity—calculated as oleic acid.....do.....	88.1	88.6
Melting point.....°C.....		109.0
Nitrogen.....percent.....	3.28	3.22
Mean molecular weight.....	427	424
Color.....		White
Odor.....		Spicy

7. N-mono-stearyl-sebacamic acid

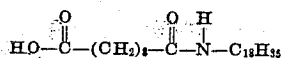


	Parts
Mono-stearylamine.....	270 (57%)
Sebacic acid.....	203 (43%)

The batch was processed as for Example 1, except that the reaction time was 5½ hours.

Test Data	Theoretical	Actual
Water loss.....percent.....	3.8	3.95
Acidity—calculated as oleic acid.....do.....	61.9	62.0
Melting point.....°C.....		114.5
Nitrogen.....percent.....	3.07	3.01
Mean molecular weight.....	456	454
Color.....		White
Odor.....		Spicy

8. N-mono-oleyl-sebacamic acid



	Parts
Mono-oleylamine.....	268 (56.8%)
Sebacic acid.....	203 (43.2%)

The batch was processed exactly as for Example 4.

Test Data	Theoretical	Actual
Water loss.....percent.....	3.82	3.90
Acidity—calculated as oleic acid.....do.....	62.2	62.7
Melting point.....°C.....		99.2
Nitrogen.....percent.....	3.05	3.06
Mean molecular weight.....	453	450
Color.....		Golden
Odor.....		Brown
		Spicy

9. N-dilauryl-sebacamic acid



	Parts
Sec. laurylamine.....	356 (63.7%)
Sebacic acid.....	203 (36.3%)

The batch was processed as for Example 1, except that the reaction time was six (6) hours.

Test Data	Theoretical	Actual
Water loss.....percent.....	3.22	3.18
Acidity—calculated as oleic acid.....do.....	62.2	62.4
Melting point.....°C.....		81.1
Nitrogen.....percent.....	2.59	2.56
Mean molecular weight.....	541	538
Color.....		Tan
Odor.....		Soapy

10. N-dipalmityl-sebacamic acid



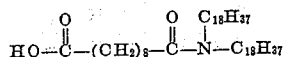
	Parts
Sec. palmitylamine.....	465 (69.6%)
Sebacic acid.....	203 (30.4%)

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The batch was processed exactly as for Example 9.

Test data	Theoretical	Actual
Water loss.....percent.....	2.69	2.63
Acidity—calculated as oleic acid.....do.....	43.4	43.7
Melting point.....°C.....	2.15	87.6
Nitrogen.....percent.....	2.15	2.12
Mean molecular weight.....	650	645
Color.....		Cream
Odor.....		Bland

11. *N*-distearyl-sebacamic acid



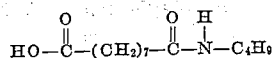
Parts

Sec. stearylamine.....521 (71.9%)
Sebacic acid.....203 (28.1%)

The batch was made exactly as in Example 9.

Test Data	Theoretical	Actual
Water loss.....percent.....	2.49	2.44
Acidity—calculated as oleic acid.....do.....	40.0	40.3
Melting point.....°C.....	1.98	89.5
Nitrogen.....percent.....	1.98	1.92
Mean molecular weight.....	706	700
Color.....		Cream
Odor.....		Bland

12. *N*-mono-*n*-butyl-azelaämic acid



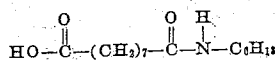
Parts

Mono-*n*-butylamine.....73 (27.3%)
Azelaic acid.....190 (72.2%)

The batch was processed exactly as in Example 1.

Test Data	Theoretical	Actual
Water loss.....percent.....	6.34	6.30
Acidity—calculated as oleic acid.....do.....	115.0	115.4
Melting point.....°C.....	5.72	22
Nitrogen.....percent.....	5.72	5.66
Mean molecular weight.....	245	244.2
Color.....		Amber viscous fluid
Odor.....		Bland

13. *N*-mono-*n*-hexyl-azelaämic acid



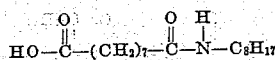
Parts

Mono-*n*-hexylamine.....101 (34.7%)
Azelaic acid.....190 (65.3%)

The batch was processed exactly as in Example 1.

Test Data	Theoretical	Actual
Water loss.....percent.....	6.19	6.24
Acidity—calculated as oleic acid.....do.....	104.0	103.7
Melting point.....°C.....	5.07	81.1
Nitrogen.....percent.....	5.07	5.12
Mean molecular weight.....	271	271.5
Color.....		White
Odor.....		Spicy

14. *N*-mono-*n*-octyl-azelaämic acid



Parts

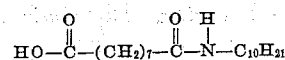
Mono-*n*-octylamine.....130 (40.7%)
Azelaic acid.....190 (59.3%)

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The batch was processed exactly as in Example 3.

Test Data	Theoretical	Actual
Water loss.....percent.....	5.62	5.57
Acidity—calculated as oleic acid.....do.....	93.3	93.7
Melting point.....°C.....	4.64	84.3
Nitrogen.....percent.....	4.64	4.59
Mean molecular weight.....	302	301
Color.....		White
Odor.....		Spicy

15. *N*-mono decyl-azelaämic acid



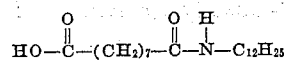
Parts

Mono-*n*-decylamine.....160 (45.7%)
Azelaic acid.....190 (54.3%)

The batch was processed exactly as in Example 3.

Test Data	Theoretical	Actual
Water loss.....percent.....	5.14	5.0
Acidity—calculated as oleic acid.....do.....	84.9	85.23
Melting point.....°C.....	4.22	85.7
Nitrogen.....percent.....	4.22	4.14
Mean molecular weight.....	332	331
Color.....		Cream
Odor.....		Spicy

16. *N*-mono lauryl-azelaämic acid



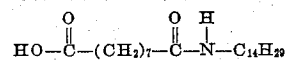
Parts

Monolaurylamine.....188 (49.8%)
Azelaic acid.....190 (50.2%)

The batch was processed exactly as in Example 4.

Test Data	Theoretical	Actual
Water loss.....percent.....	4.77	4.78
Acidity—calculated as oleic acid.....do.....	78.3	78.5
Melting point.....°C.....	3.89	82.2
Nitrogen.....percent.....	3.89	3.83
Mean molecular weight.....	360	359
Color.....		Light Brown
Odor.....		Spicy

17. *N*-mono myristyl-azelaämic acid



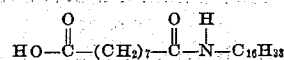
Parts

Monomyristylamine.....215 (53.2%)
Azelaic acid.....190 (46.8%)

The batch was processed exactly as in Example 4.

Test Data	Theoretical	Actual
Water loss.....percent.....	4.45	4.49
Acidity—calculated as oleic acid.....do.....	72.8	73.1
Melting point.....°C.....	3.61	83.6
Nitrogen.....percent.....	3.61	3.55
Mean molecular weight.....	387	385.5
Color.....		Cream
Odor.....		Bland

18. *N*-mono palmityl-azelaämic acid



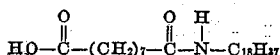
Parts

Monopalmitylamine.....245 (56.3%)
Azelaic acid.....190 (43.7%)

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The batch was processed as in Example 1, except that the reaction time was four and one-half (4½) hours.

Test Data	Theoretical	Actual
Water loss.....percent.....	4.14	4.02
Acidity—calculated as oleic acid.....do.....	67.7	67.9
Melting point.....°C.....		91.2
Nitrogen.....percent.....	3.36	3.40
Mean molecular weight.....	417	416
Color.....		Cream
Odor.....		Bland

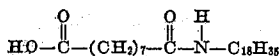
19. *N*-mono stearyl-azelaämic acid

Parts

Monostearylamine.....270 (58.7%)
Azelaic acid.....190 (41.3%)

The batch was processed exactly as in Example 7.

Test Data	Theoretical	Actual
Water loss.....percent.....	3.92	3.87
Acidity—calculated as oleic acid.....do.....	63.9	64.2
Melting point.....°C.....		105.0
Nitrogen.....percent.....	3.17	3.11
Mean molecular weight.....	452	440
Color.....		Light Brown
Odor.....		Spicy

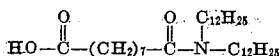
20. *N*-mono oleyl-azelaämic acid

Parts

Monoöleylamine.....267 (58.3%)
Azelaic acid.....190 (41.7%)

The batch was processed exactly as in Example 4.

Test Data	Theoretical	Actual
Water loss.....percent.....	3.94	3.86
Acidity—calculated as oleic acid.....do.....	64.2	64.5
Melting point.....°C.....		78.2
Nitrogen.....percent.....	3.19	3.20
Mean molecular weight.....	439	437
Color.....		Deep amber
Odor.....		Spicy

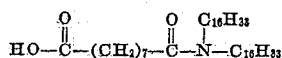
21. *N*-dilauryl-azelaämic acid

Parts

Sec. laurylamine.....356 (65.2%)
Azelaic acid.....190 (34.8%)

The batch was processed as in Example 9.

Test Data	Theoretical	Actual
Water loss.....percent.....	3.30	3.24
Acidity—calculated as oleic acid.....do.....	53.3	53.7
Melting point.....°C.....		70.6
Nitrogen.....percent.....	2.65	2.59
Mean molecular weight.....	528	525
Color.....		Brown
Odor.....		Soapy

22. *N*-dipalmityl-azelaämic acid

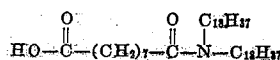
Parts

Sec. palmitylamine.....465 (71.0%)
Azelaic acid.....190 (29.0%)

10

The batch was processed as in Example 9.

Test Data	Theoretical	Actual
Water loss.....per cent.....	2.75	2.79
Acidity—calculated as oleic acid.....do.....	44.3	44.5
Melting point.....°C.....		72.4
Nitrogen.....per cent.....	2.20	2.17
Mean molecular weight.....	637	634
Color.....		Tan
Odor.....		Bland

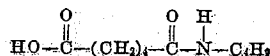
23. *N*-distearyl-azelaämic acid

Parts

Sec. stearylamine.....521 (73.3%)
Azelaic acid.....190 (26.7%)

The batch was made exactly as in Example 9.

Test Data	Theoretical	Actual
Water loss.....per cent.....	2.53	2.60
Acidity—calculated as oleic acid.....do.....	40.7	40.9
Melting point.....°C.....		76.6
Nitrogen.....per cent.....	2.02	2.00
Mean molecular weight.....	693	690
Color.....		Tan
Odor.....		Bland

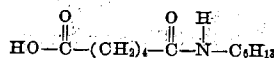
24. *N*-mono-*n*-butyl-adipamic acid

Parts

Mono-*n*-butylamine.....73 (33.1%)
Adipic acid.....148 (66.9%)

The batch was processed exactly as in Example 1.

Test Data	Theoretical	Actual
Water loss.....per cent.....	8.15	8.01
Acidity—calculated as oleic acid.....do.....	139.0	139.6
Melting point.....°C.....		93.3
Nitrogen.....per cent.....	6.89	6.80
Mean molecular weight.....	203	202
Color.....		White
Odor.....		Bland

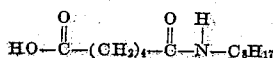
25. *N*-mono-*n*-hexyl-adipamic acid

Parts

Mono-*n*-hexylamine.....101 (40.6%)
Adipic acid.....148 (59.4%)

The batch was made exactly as in Example 1.

Test Data	Theoretical	Actual
Water loss.....per cent.....	7.23	7.33
Acidity—calculated as oleic acid.....do.....	122.0	122.4
Melting point.....°C.....		104.7
Nitrogen.....per cent.....	6.06	5.94
Mean molecular weight.....	231	230
Color.....		White
Odor.....		Spicy

26. *N*-mono-*n*-octyl-adipamic acid

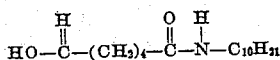
Parts

Mono-*n*-octylamine.....130 (46.8%)
Adipic acid.....148 (53.2%)

The batch was made exactly as in Example 3.

Test Data	Theoretical	Actual
Water loss.....per cent.	6.48	6.52
Acidity—calculated as oleic acid.....do.	108.3	108.7
Melting point.....°C.		115.6
Nitrogen.....per cent.	5.38	5.30
Mean molecular weight.....	260	259
Color.....		White
Odor.....		Spicy

27. *N*-mono decyl-adipamic acid



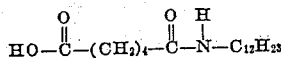
Parts

Mono-*n*-decylamine 160 (51.9%)
Adipic acid..... 148 (48.1%)

The batch was processed exactly as in Example 3.

Test Data	Theoretical	Actual
Water loss.....per cent.	5.84	5.92
Acidity—calculated as oleic acid.....do.	97.3	97.7
Melting point.....°C.		105.0
Nitrogen.....per cent.	4.83	4.77
Mean molecular weight.....	290	288.5
Color.....		White
Odor.....		Spicy

28. *N*-monolauryl-adipamic acid



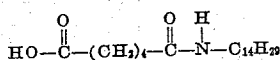
Parts

Laurylamine 188 (55.9%)
Adipic acid..... 148 (44.1%)

The batch was processed exactly as in Example 4.

Test Data	Theoretical	Actual
Water loss.....Per Cent.	5.36	5.42
Acidity—calculated as oleic acid.....do.	88.7	89.0
Melting point.....°C.		96.7
Nitrogen.....Per Cent.	4.40	4.42
Mean molecular weight.....	318	316.5
Color.....		White
Odor.....		Spicy

29. *N*-monomyristyl-adipamic acid



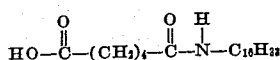
Parts

Myristylamine 215 (59.2%)
Adipic acid..... 148 (40.8%)

The batch was processed exactly as in Example 4.

Test Data	Theoretical	Actual
Water loss.....Per Cent.	4.96	5.01
Acidity—calculated as oleic acid.....do.	81.7	81.9
Melting point.....°C.		89.0
Nitrogen.....Per Cent.	4.06	3.94
Mean molecular weight.....	345	344
Color.....		White
Odor.....		Bland

30. *N*-monopalmityl-adipamic acid



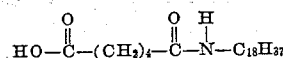
Parts

Palmitylamine 245 (62.4%)
Adipic acid..... 148 (37.6%)

The batch was processed exactly as in Example 4.

Test Data	Theoretical	Actual
Water loss.....percent.	4.58	4.63
Acidity—calculated as oleic acid.....do.	75.2	75.5
Melting point.....°C.		82.0
Nitrogen.....percent.	3.73	3.70
Mean molecular weight.....	375	373
Color.....		White
Odor.....		Bland

31. *N*-monostearyl-adipamic acid



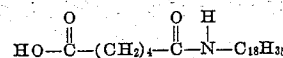
Parts

Stearylamine 270 (64.6%)
Adipic acid..... 148 (35.4%)

The batch was processed exactly as in Example 7.

Test Data	Theoretical	Actual
Water loss.....percent.	4.31	4.41
Acidity—calculated as oleic acid.....do.	70.5	70.7
Melting point.....°C.		81.4
Nitrogen.....percent.	3.50	3.47
Mean molecular weight.....	400	399
Color.....		Tan
Odor.....		Bland

32. *N*-monooleyl-adipamic acid



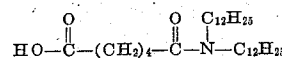
Parts

Oleylamine 267 (64.4%)
Adipic acid..... 148 (35.6%)

The batch was processed exactly as in Example 7.

Test Data	Theoretical	Actual
Water loss.....percent.	4.34	4.50
Acidity—calculated as oleic acid.....do.	71.0	71.3
Melting point.....°C.		76.2
Nitrogen.....percent.	3.53	3.50
Mean molecular weight.....	397	396
Color.....		Opaque Amber
Odor.....		Bland

33. *N*-dilauryl-adipamic acid



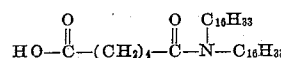
Parts

Sec. laurylamine 356 (70.65%)
Adipic acid..... 148 (29.35%)

The batch was processed exactly as in Example 9.

Test Data	Theoretical	Actual
Water loss.....percent.	3.57	3.61
Acidity—calculated as oleic acid.....do.	58.0	58.3
Melting point.....°C.		81.1
Nitrogen.....percent.	2.88	2.81
Mean molecular weight.....	486	484
Color.....		Tan
Odor.....		Soapy

34. *N*-dipalmityl-adipamic acid



Parts

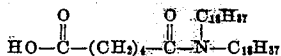
Sec. palmitylamine 465 (75.8%)
Adipic acid..... 148 (24.2%)

13

The batch was processed exactly as in Example 9.

Test Data	Theoretical	Actual
Water loss.....percent	2.94	3.02
Acidity—calculated as oleic acid.....do.	47.3	47.6
Melting point.....°C		58.3
Nitrogen.....percent	2.35	2.31
Mean molecular weight.....	595	593
Color.....		Tan
Odor.....		Soapy

35. N-distearyl-adipamic acid



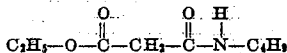
Parts

Sec. stearylamine 521 (77.5%)
 Adipic acid 148 (22.5%)

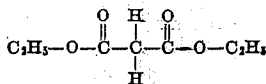
The batch was processed exactly as in Example 9.

Test Data	Theoretical	Actual
Water loss.....percent	2.69	2.78
Acidity—calculated as oleic acid.....do.	43.3	43.6
Melting point.....°C		97.8
Nitrogen.....percent	2.15	2.05
Mean molecular weight.....	651	648
Color.....		Tan
Odor.....		Soapy

36. N-mono-n-butyl-malonamic ester



Since malonic acid is unstable in normal atmospheres, we used malonic ester,



as the source of malonic radical. Instead of water being evolved, ethanol is the reaction product.

Parts

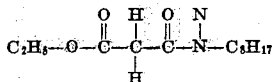
Mono-n-butylamine 73 (31.3%)
 Malonic ester 160 (68.7%)

The malonic ester was heated to 270° F. The mono-n-butylamine was added slowly and the temperature was permitted to rise to 290–310° F., after all the amine had been added. After four (4) hours, at 290–310° F., saponification equivalency and weight loss indicated the end of the reaction.

Test Data	Theoretical	Actual
Ethanol loss.....percent	19.7	20.1
Saponification equivalent—calculated as oleic acid.....percent	151	152
Mean molecular weight.....	187	185
Nitrogen.....percent	7.48	7.40
Appearance.....		(¹)
Odor.....		Spicy

¹ Amorphous opaque white solid.

37. N-mono-n-octyl-malonamic ester



Parts

Mono-n-octylamine 130 (44.7%)
 Malonic ester 160 (55.3%)

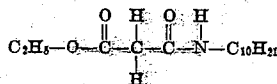
14

The batch was processed exactly as in Example 36.

Test Data	Theoretical	Actual
Ethanol loss.....percent	15.85	15.80
Saponification equivalent—calculated as oleic acid.....percent	115.7	115.3
Mean molecular weight.....	244	245
Nitrogen.....percent	5.73	5.70
Appearance.....		(¹)
Odor.....		Spicy

¹ Amorphous opaque white solid.

38. N-mono-decyl-malonamic ester



Parts

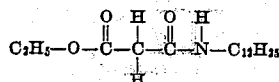
Mono-decylamine 160 (50%)
 Malonic ester 160 (50%)

The batch was processed exactly as in Example 36, except the reaction time, which was four (4) hours.

Test Data	Theoretical	Actual
Ethanol loss.....percent	14.4	14.9
Saponification equivalent—calculated as oleic acid.....percent	103.6	103.9
Mean molecular weight.....	274	272
Nitrogen.....percent	5.12	5.04
Appearance.....		(¹)
Odor.....		Spicy

¹ Amorphous opaque cream colored solid.

39. N-mono-lauryl-malonamic ester



Parts

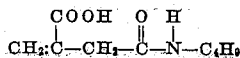
Mono-laurylamine 188 (54%)
 Malonic ester 160 (46%)

The batch was made exactly as in Example 38.

Test Data	Theoretical	Actual
Ethanol loss.....percent	13.2	13.6
Saponification equivalent—calculated as oleic acid.....percent	93.6	93.9
Mean molecular weight.....	302	300
Nitrogen.....percent	4.04	4.01
Appearance.....		(¹)
Odor.....		Spicy

¹ Amorphous opaque cream-colored solid.

40. N-mono-n-butyl-itaconamic acid



Parts

Mono n-butylamine 73 (36%)
 Itaconic acid 130 (64%)

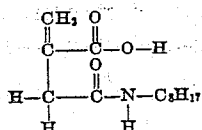
The butylamine and itaconic acid were added to a vessel together, and the temperature was slowly raised to 260° F. The amidation began at 215° F. with copious evolution of water. The temperature was maintained at 260–280° F. until the reaction was complete, which was evidenced

by cessation of evolution of water, after forty (40) minutes.

Test Data	Theoretical	Actual
Water loss.....percent.....	8.87	9.15
Acidity—calculated as oleic acid.....do.....	152.5	153.0
Melting point.....° C.....		(1)
Nitrogen.....percent.....	7.56	7.53
Mean molecular weight.....	185	184
Color.....		Straw
Odor.....		Bland

1 Viscous liquid with -11° C. freezing point.

41. N-mono-n-octyl-itaconamic acid

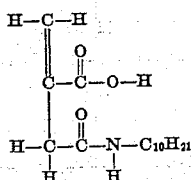


	Parts
Mono-n-octylamine	130 (50%)
Itaconic acid	130 (50%)

The batch was made as in Example 40, except that the reaction time was one (1) hour.

Test Data	Theoretical	Actual
Water loss.....percent.....	6.93	6.99
Acidity—calculated as oleic acid.....do.....	116.5	116.9
Melting point.....° C.....		34.4
Nitrogen.....percent.....	5.78	57.1
Mean molecular weight.....	242	241
Color.....		Yellow
Odor.....		Spicy

42. N-mono-decyl-itaconamic acid

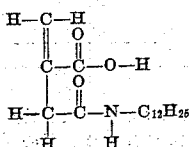


	Parts
Monodecylamine	160 (55.2%)
Itaconic acid	130 (44.8%)

The batch was processed as in Example 40, except that the reaction time was 80 minutes.

Test Data	Theoretical	Actual
Water loss.....percent.....	6.21	6.40
Acidity—calculated as oleic acid.....do.....	103.7	104.0
Melting point.....° C.....		36.7
Nitrogen.....percent.....	5.15	5.11
Mean molecular weight.....	272	271.5
Color.....		Yellow
Odor.....		Spicy

43. N-mono lauryl-itaconamic acid

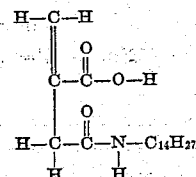


	Parts
Mono laurylamine	188 (59.2%)
Itaconic acid	130 (40.8%)

The batch was made exactly as in Example 42.

Test Data	Theoretical	Actual
5 Water loss.....percent.....	5.66	5.71
Acidity—calculated as oleic acid.....do.....	94.0	94.3
Melting point.....° C.....		40.0
Nitrogen.....percent.....	4.67	4.64
Mean molecular weight.....	300	299
Color.....		Cream
Odor.....		Spicy

44. N-monomyristyl-itaconamic acid

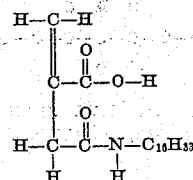


	Parts
Myristylamine	215 (62.3%)
Itaconic acid	130 (37.7%)

The batch was made exactly as in Example 42.

Test Data	Theoretical	Actual
30 Water loss.....percent.....	5.22	5.40
Acidity—calculated as oleic acid.....do.....	86.2	86.4
Melting point.....° C.....		48.7
Nitrogen.....percent.....	4.28	4.22
Mean molecular weight.....	327	326
Color.....		Cream
Odor.....		Spicy

45. N-mono palmityl-itaconamic acid

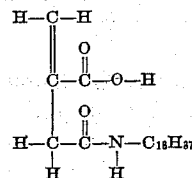


	Parts
Mono palmitylamine	245 (65.3%)
Itaconic acid	130 (34.7%)

The batch was made exactly as in Example 42.

Test Data	Theoretical	Actual
55 Water loss.....percent.....	4.8	5.1
Acidity—calculated as oleic acid.....do.....	79.0	79.7
Melting point.....° C.....		59.2
Nitrogen.....percent.....	3.92	3.87
Mean molecular weight.....	357	354
Color.....		Cream
Odor.....		Spicy

46. N-mono stearyl-itaconamic acid



	Parts
Mono stearylamine	270 (67.5%)
Itaconic acid	130 (32.5%)

17

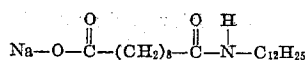
The batch was processed the same as in Example 42, except that the reaction time was two (2) hours.

Test Data	Theoretical	Actual
Water loss.....percent	4.5	4.9
Acidity—calculated as oleic acid.....do	73.8	74.0
Melting point.....° C		68.6
Nitrogen.....percent	3.67	3.61
Mean molecular weight.....	382	381
Color.....		Tan
Odor.....		Spicy

(II) SALTS OF SUCH ACIDS AND GREASES MADE THEREFROM

The alkali, alkaline earth, and other polyvalent metal salts of the above amic acids are employed with oils to form greases in accordance with our invention. Below are listed examples of products utilizing the above types of ingredients.

47. N-lauryl-sodium sebacamate grease



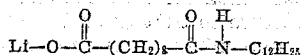
	Per cent
N-lauryl sebacamate acid.....	10
Sodium hydroxide—flake.....	1
200/100° F. S. S. U. Coastal pale oil.....	89
	100
Salt content.....	10.6

The N-lauryl sebacamic acid and oil are added to a kettle and heated to about 400–440° F. The sodium hydroxide is dissolved in three times its weight of water and is slowly added to the heated sebacamic acid and oil in the kettle. Reaction commences. The addition of the alkali solution is controlled so that the temperature is about 435° F. when all of the alkali has been completely added; the grease is cooled to about 120° F. maximum without disturbing, that is, without raking, stirring or otherwise agitating it. The grease is then milled, that is, passed over rollers, kettle stirred, or passed through a homogenizer or mill until the structure is substantially homogeneous.

The above grease had the following properties:

Penetration ASTM (American Society for Testing Materials) D-217-47T.....	296
Dropping point (D.P.) ASTM-566-42.....	379° F.
Water tolerance Chrysler.....	10%
Color.....	Straw

48. N-lauryl-lithium sebacamate grease



	Per cent
N-lauryl-sebacamic acid.....	10
Lithium hydroxide monohydrate.....	1
100/100° F. S. S. U. Coastal pale oil.....	89
	100
Salt content.....	10.13

The batch was made exactly as in Example 47. The finished grease had the following properties:

Penetration ASTM D-217-47T.....	320
D./P. ASTM D-566-42.....	409° F.
Water tolerance Chrysler.....	Less than 10%

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49. N-lauryl-lithium sebacamate grease

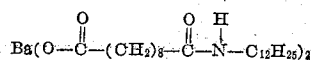
	Per cent
N-lauryl-sebacamic acid.....	10
Lithium hydroxide monohydrate.....	1
3500/100° F. S. S. U. Residual black oil.....	89
	100

Salt content..... 10.13

The batch was made exactly as in Example 47. The finished grease had the following properties:

Penetration ASTM D-217-47T.....	288
D./P. ASTM D-566-42.....	410° F.
C. R. C. wheel bearing grease test.....	Fair to good
Rust prevention—10% salt spray.....	O. K. after 72 hours

50. N-lauryl-barium sebacamate grease



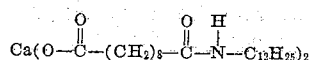
	Per cent
N-lauryl-sebacamic acid.....	11.44
Barium hydroxide octahydrate.....	4.28
200/210° F. S. S. U. Mid-Continent semi-bright stock.....	84.28
	100.00

Salt content..... 13.25

The batch was made exactly as in Example 47 except that crystals of alkali were used. The finished grease had the following properties:

Penetration ASTM D-217-47T.....	295
D./P. ASTM D-566-42.....	355° F.
C. R. C. wheel bearing grease test.....	Good
Water tolerance Chrysler.....	Less than 10%

51. N-lauryl-calcium sebacamate grease

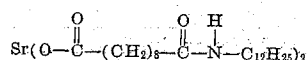


	Per cent
N-lauryl-sebacamic acid.....	10
Calcium hydroxide—100% 200-mesh.....	1
200/210° F. S. S. U. Mid-Continent semi-bright stock.....	89
	100
Salt content.....	10.62

The batch was processed as in Example 47, except that the lime was made up as a 1:3 slurry in oil. The finished grease had the following properties:

Penetration ASTM D-217-47T.....	298
D./P. ASTM D-566-42.....	294° F.

52. N-lauryl-strontium sebacamate grease



	Per cent
N-lauryl sebacamic acid.....	10.0
Strontium hydroxide octahydrate.....	3.1
3500/100° F. S. S. U. Residual black oil.....	86.9
	100.0
Salt content.....	11.05

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The batch was processed as in Example 50. The finished grease had the following properties:

Penetration ASTM D-217-47T	311
D./P. ASTM D-566-42	310° F.

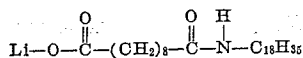
53. *N-lauryl-lithium sebacamate grease*

	Per cent
N-lauryl sebacamic acid	10
Lithium hydroxide monohydrate	1
100/100° F. S. S. U. solvent extracted Mid-Continent pale oil	89
	100

Salt content	10.13
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The batch was made exactly as in Example 47. The finished grease had the following properties:

Penetration ASTM D-217-47T	332
D./P. ASTM D-566-42	396° F.

54. *N-oleyl-lithium sebacamate grease*

	Per cent
N-oleyl-sebacamic acid	10
Lithium hydroxide monohydrate	.75
100/100° F. Coastal pale oil	89.25
	100.00

Salt content	10.02
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The batch was made as in Example 47. The finished grease had the following properties:

Penetration ASTM D-217-47T	316
D./P. ASTM D-566-42	405° F.

55. *N-oleyl-lithium sebacamate grease*

	Per cent
N-oleyl-sebacamic acid	10
Lithium hydroxide monohydrate	.75
300/100° F. S. S. U. Tech. white oil	89.25
	100.00

Salt content	10.02
--------------	-------

The batch was made as in Example 47. The finished grease had the following properties:

Penetration ASTM D-217-47T	327
D./P. ASTM D-566-42	395° F.

56. *N-lauryl-sodium sebacamate grease*

	Per cent
N-lauryl-sebacamic acid	10
Sodium hydroxide	1
100/100° F. S. S. U. Coastal pale oil	89
	100

Salt content	10.6
--------------	------

The batch was made as in Example 47. The finished grease had the following properties:

Penetration ASTM D-217-47T	270
D./P. ASTM D-566-42	360° F.
Water tolerance Chrysler	70%

57. *N-lauryl-lithium sebacamate grease*

	Per cent
N-oleyl-sebacamic acid	10
Lithium hydroxide monohydrate	1
200/100° F. S. S. U. Mid-Continent pale oil	44.5
180/210° F. S. S. U. Mid-Continent bright stock	44.5
	100.0

Salt content	10.13
--------------	-------

20

The batch was made as in Example 47. The finished grease had the following properties:

Penetration ASTM D-217-47T	298
D./P. ASTM D-566-42	427° F.
Water tolerance Chrysler	10%

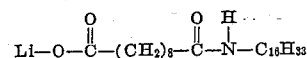
58. *N-lauryl-sodium sebacamate grease*

	Per cent
N-lauryl-sebacamic acid	15.00
Sodium hydroxide	1.50
180/210° F. S. S. U. Mid-Continent bright stock	41.75
200/100° F. S. S. U. Mid-Continent pale oil	41.75
	100.00

Salt content	15.9
--------------	------

The batch was made as in Example 47. The finished grease had the following properties:

Penetration ASTM D-217-47T	245
D./P. ASTM D-566-42	°F. 375
Water tolerance Chrysler	per cent. 65

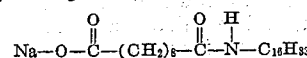
59. *N-palmityl-lithium sebacamate grease*

	Per cent
N-palmityl-sebacamic acid	10.0
Lithium hydroxide monohydrate	0.9
3500/100° F. S. S. U. Residual black oil	89.1
	100.0

Salt content	10.16
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The batch was made as in Example 47. The finished grease had the following properties:

Penetration ASTM D-217-47T	240
D./P. ASTM D-566-42	°F. 410

60. *N-palmityl-sodium sebacamate grease*

	Per cent
N-palmityl-sebacamic acid	10.0
Sodium hydroxide	0.9
3500/100° F. S. S. U. Residual black oil	89.1
	100.0

Salt content	10.51
--------------	-------

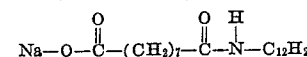
The batch was made as in Example 47. The finished grease had the following properties:

Penetration ASTM D-217-47T	245
D./P. ASTM D-566-42	°F. 371

61. *N-palmityl-sodium sebacamate grease*

The grease of Example 60 was diluted further with Residual black oil 3500/100° F. S. S. U. at room temperature so that the salt content in the formula was about 2.9%. The properties of this composition were:

Penetration ASTM D-217-47T	406
Dropping point ASTM D-566-42	°F. 367

62. *N-lauryl-sodium azelaämate grease*

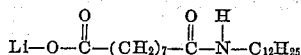
	Per cent
N-lauryl-azelaämic acid	6
Sodium hydroxide	0.67
300/100° F. S. S. U. Coastal pale oil	93.33
	100.00

Salt content	6.42
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The batch was made as in Example 47. The finished grease had the following properties:

Penetration ASTM D-217-47T	341
D./P. ASTM D-566-42	°F 345

63. *N-lauryl-lithium azelaämate grease*

	Per cent
N-lauryl-azelaämic acid	6
Lithium hydroxide monohydrate	0.67
300/100° F. S. S. U. Coastal pale oil	93.33
	100.00

Salt content 6.1

The batch was made as in Example 47. The finished grease had the following properties:

Penetration ASTM D-217-47T	330
D./P. ASTM D-566-42	°F 395

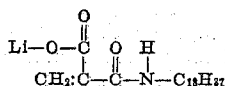
64. *N-lauryl-lithium sebacamate grease*

	Per cent
N-lauryl-sebacamic acid	4.0
Lithium hydroxide monohydrate	0.4
150/210° F. S. S. U. Residual black oil	57.3
100/100° F. S. S. U. Coastal pale oil	38.3
	100.0

Salt content 4.05

The batch was made as in Example 47. The finished grease had the following properties:

Penetration ASTM D-217-47T	348
D./P. ASTM D-566-42	°F 395

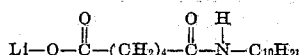
65. *N-stearyl lithium itaconamate grease*

	Per cent
N-stearyl-itaconamic acid	10.0
Lithium hydroxide monohydrate	1.1
75/210° F. S. S. U. Mid-Continent bright stock	88.9
	100.0

Salt content 10.23

The batch was made as in Example 47. The finished grease had the following properties:

Penetration ASTM D-217-47T	340
D./P. ASTM D-566-42	°F 370

66. *N-decyl-lithium adipamate grease*

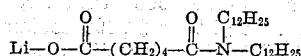
	Per cent
N-decyl-adipamic acid	10.0
Lithium hydroxide monohydrate	1.45
75/210° F. S. S. U. Mid-Continent bright stock	88.55
	100.00

Salt content 9.46

The batch was made as in Example 47. The finished grease had the following properties:

Penetration ASTM D-217-47T	326
D./P. ASTM D-566-42	°F 391

22

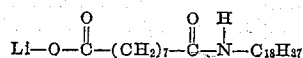
67. *N-dilauryl-lithium adipamate grease*

	Per cent
N-dilauryl-adipamic acid	10.0
Lithium hydroxide monohydrate	0.87
300/100° F. Mid-Continent pale oil	89.13
	100.00

Salt content 10.21

The batch was made as in Example 47. The finished grease had the following properties:

Penetration ASTM D-217-47T	310
D./P. ASTM D-566-42	°F 411
C. R. C. wheel bearing grease test	Good

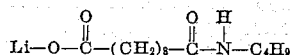
68. *N-stearyl-lithium azelaämate grease*

	Per cent
N-stearyl-azelaämic acid	10.0
Lithium hydroxide monohydrate	0.93
75/210° F. Coastal bright stock	89.07
	100.00

Salt content 10.2

The batch was made as in Example 47. The finished grease had the following properties:

Penetration ASTM D-217-47T	295
D./P. ASTM D-566-42	°F 416

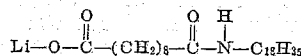
69. *N-butyl-lithium sebacamate grease*

	Per cent
N-butyl-sebacamic acid	10.0
Lithium hydroxide monohydrate	1.63
600/100° F. Mid-Continent lube distillate	88.37
	100.00

Salt content 10.38

The batch was made as in Example 47. The finished grease had the following properties:

Penetration ASTM D-217-47T	316
D./P. ASTM D-566-42	°F 394

70. *N-oleyl-lithium sebacamate grease*

	Per cent
N-oleyl sebacamic acid	1.0
Lithium hydroxide monohydrate	0.09
100/100° F. S. S. U. Coastal pale oil	8.9
300/100° F. S. S. U. Coastal pale oil	90.01
	100.00

Salt content 1.02

The batch was made as in Example 47. The finished grease was fluid and had the following properties:

Viscosity (Gardiner Mobilometer at 77° F.) ¹	1-100-1000
Pour point	°F -15

¹ This test is made with a series of disks numbered from 1 to 3 with arbitrary weights, the viscosity being measured by disk movement through the material tested and being represented in seconds.

The above Gardner Mobilometer viscosity is designated as "1-100-1000" which represents #1 disk, 100 gm. weight, and 1000 seconds at 77° F. Gardner Mobilometer viscosities hereinafter referred to are simply designated similarly to the expression quoted above.

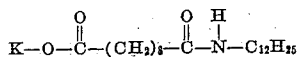
71. N-lauryl-lithium sebacamate grease

	Per cent
N-lauryl-sebacamic acid	30
Lithium hydroxide monohydrate	2.3
300/100° F. Mid-Continent neutral	67.7
	100.0
Salt content	30.39

The batch was made as in Example 47. The finished grease had the following properties:

Penetration ASTM D-217-47T	107
D./P. ASTM D-566-42	°F. 471
Water tolerance Chrysler	Nil

72. N-lauryl-potassium sebacamate grease



	Per cent
N-lauryl-sebacamic acid	10.0
Potassium hydroxide	1.5
75/210° F. S. S. U. Coastal bright stock	88.5
	100.0
Salt content	11.06

The batch was made as in Example 47. The finished grease had the following properties:

Penetration ASTM D-217-47T	342
D./P. ASTM D-566-42	°F. 379
Water tolerance Chrysler	per cent. 30

73. N-lauryl-lithium sebacamate grease

	Per cent
N-lauryl-sebacamic acid	10
Lithium hydroxide	1
180/210° F. S. S. U. Coastal cylinder stock	87
Lead naphthenate (30% lead)	2
	100
Salt content	10.13

The 180/210° F. S. S. U. Coastal cylinder stock and N-lauryl sebacamic acid were heated to 400° F. with constant agitation. After the acid had completely melted and dispersed in the oil, the neutralization with the lithium hydroxide (15-20%) solution in water was begun. The neutralization was carried out and the grease processed, without lead naphthenate, as described in Example 47.

After the grease had cooled to room temperature, it was melted to a smooth, homogeneous consistency, after which the lead naphthenate was added and blended in. It must be noted that the lead naphthenate should not be included in the high temperature phase operation in this grease formulation.

The physical properties of the resultant grease were:

Penetration—unworked	245
Penetration ASTM D-217-47T	280
Penetration—5000 strokes	320
D./P. ASTM D-566-42	°F. 405
Chrysler basket D./P.	°F. 330
Water tolerance Chrysler	per cent. 10
Rust preventive test (10% salt spray)	
O. K. after 72 hours	75

74. N-lauryl-lithium sebacamate grease

	Per cent
N-lauryl-sebacamic acid	5.0
Lithium hydroxide monohydrate	0.5
180/210° F. S. S. U. Coastal cylinder stock	92.5
Lead naphthenate (30% lead)	2.0
	100.0
Salt content	5.06

The batch was made exactly as in Example 73. The physical properties of the resultant grease were:

Penetration—unworked	302
Penetration ASTM D-217-47T	304
Penetration—5000 strokes	346
D./P. ASTM D-566-42	°F. 397
Chrysler basket D./P.	°F. 310
Water tolerance Chrysler	per cent. 10

75. N-lauryl-lithium sebacamate grease

	Per cent
N-lauryl-sebacamic acid	2.0
Lithium hydroxide monohydrate	0.2
Sulfurized lard oil (10% sulfur)	10.0
300/100° F. S. S. U. Coastal pale oil	87.8
	100.0
Salt content	2.05

It is to be noted that due to the sulfur in the sulfurized lard oil, that material can not be included in the high temperature operation in the manufacture of these greases, so that the N-lauryl sebacamic acid and the mineral oil are heated to 400° F. and processed as in Example 47.

After the grease had been melted to a homogeneous mass, the sulfurized lard oil was blended into the grease.

The resultant grease had the following properties:

Viscosity (Gardner Mobilometer at 77° F.)	2-50-(40 to 50)
Almen test carries	20 lb. lever arm load
Falex test (500 lb. step test)	3000 lbs. gauge
Falex test (100 lb. step test)	2700 lbs. gauge
Water tolerance Chrysler	10%
Heat stability test:	

Since the material is wholly fluid, standard dropping points as a measurement of heat or temperature resistance could not be applied. In this case, then, Gardner Mobilometer tests were run at varying temperatures:

At 77° F. actual run was	43 seconds
At 100° F. actual run was	42 seconds
At 125° F. actual run was	42 seconds
At 150° F. actual run was	40 seconds
At 200° F. actual run was	40 seconds

25

76. *N*-lauryl-lithium sebacamate grease

	Per cent
N-lauryl sebacamic acid	0.5
Lithium hydroxide monohydrate	0.05
180/210° F. S. S. U. Coastal cylinder stock	69.60
100/100° F. S. S. U. Coastal pale oil	29.85
	100.00

Salt content 5

The batch was made as in Example 47. The resultant grease had the following properties:

Viscosity (Gardner Mobilometer at 77° F.) 2-50-(30 to 40)

Water tolerance Chrysler .. 10%

Heat stability test (Gardner Mobilometer):

At 77° F. actual run was 36 seconds

At 100° F. actual run was 36 seconds

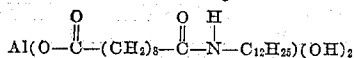
At 125° F. actual run was 40 seconds

At 150° F. actual run was 39 seconds

At 200° F. actual run was 39 seconds

Rust preventive test (10% salt spray) O. K. after 72 hrs.

The heat stability test indicated that the grease does not thin out rapidly as the temperature is elevated under ordinary operating conditions.

77. *Aluminum-mono-N*-lauryl sebacamate grease

392 parts of the sodium salt of the *N*-lauryl sebacamic acid were dissolved in 1000 parts of water at room temperature. To this solution were added 80 parts of sodium hydroxide. When the above solution was homogeneous, it was heated to boiling, about 212° F. A solution made up of 171 parts of aluminum sulfate nonahydrate dissolved in 500 parts of water was slowly added to the first solution. Boiling was maintained and, after all of the solution had been added, the boiling was continued for 30 minutes. The salt which had risen to the top of the aqueous portion was separated from the aqueous portion by siphoning off the aqueous portion. Fresh water was added to the salt and reboiled. This separation and reboiling was continued with fresh water until the aqueous portion was neutral to phenolphthalein. The salt was then dried and powdered.

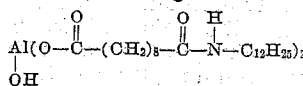
A mixture of the aluminum salt and oil to make the grease was prepared in the following manner:

	Per cent
Aluminum mono- <i>N</i> -lauryl sebacamate	10
300/100° F. S. S. U. Coastal pale oil	90
	100

The powdered aluminum mono-*N*-lauryl sebacamate was mixed into the oil to make a pasty slurry. The mass was heated, with continuous agitation to the point where the mass turned from a pasty mass to a clear solution (about 375° F.). The mass was permitted to cool to room temperature without agitation. When cool, the grease was milled out to a homogeneous mass. The finished grease had the following properties:

Penetration ASTM D-217-47T	346
D./P. ASTM D-566-42	°F. 316
Water tolerance Chrysler	per cent. 25

26

78. *Aluminum-di-N*-lauryl sebacamate grease

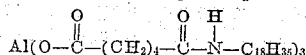
To 392 parts of sodium *N*-lauryl sebacamate (prepared as hereinbelow, Example 80) dissolved in 1000 parts of water was added 126 parts of aluminum sulfate nonahydrate dissolved in 500 parts of water. The mass was heated to boiling. The precipitate formed was purified by subsequent washings with boiling distilled water until washings were neutral to phenolphthalein. The washed precipitate was dried and powdered.

A mixture of the aluminum salt and oil to make the grease was prepared in the following manner:

	Per cent
Aluminum di- <i>N</i> -lauryl sebacamate	10
300/100° F. S. S. U. Mid-Continent pale oil	90
	100

The powdered aluminum di-*N*-lauryl sebacamate and the oil were mixed in a kettle to a pasty consistency and were then gradually heated to about 440° F. with continuous agitation. The mass changes from a pasty opaque mass to a transparent fluid after heating for about 20 minutes. The mass was allowed to cool to room temperature without agitation. When cool, the mass was milled out smooth by agitating in the kettle. The resulting product was a smooth, adhesive, transparent grease. The grease had the following properties:

Penetration ASTM D-217-47T	310
D./P. ASTM D-566-42	394° F.
Water tolerance Chrysler	15%
Color	Golden Yellow
C. R. C. wheel bearing grease test	Fair
Rust preventive (10% salt spray)	O. K. after 72 hours

79. *Aluminum tri-N*-oleyl adipamate grease

420 parts of sodium *N*-oleyl adipamate (prepared as hereinbelow, Example 80) were dissolved in 1000 parts of water and the whole was brought to a boil. To the hot salt solution was slowly added the hot solution of 57 parts of aluminum sulfate nonahydrate in 300 parts of water. Since there were three (3) parts of sodium salt in excess, the reaction mixture was alkaline to phenolphthalein. The aluminum salt was purified by several washings of boiling water, until the washings were neutral to phenolphthalein. The salt was then dried and powdered.

A mixture of the aluminum salt and oil to make the grease was prepared in the following manner:

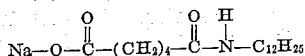
	Per cent
Aluminum <i>N</i> -oleyl adipamate	10
300/100° F. S. S. U. Coastal pale oil	90
	100

The mixture was processed as in Example 77. The finished grease had the following properties:

Penetration ASTM D-217-47T	330
D./P. ASTM D-566-42	°F. 344
Water tolerance Chrysler	per cent. 15

The following procedure was used to make the sodium salts of the amic acids in their separate form. These sodium salts were used in the preparation of the aluminum salts above.

80. Process for making alkali metal and alkaline earth metal salts of amic acids



370 parts of the N-lauryl sebacamic acid were dissolved in 1000 parts of ethanol. To this alcoholic solution was added, dropwise, an alcoholic solution of 40 parts of sodium hydroxide in 300 parts of ethanol. This was insufficient alkali to give color indication with phenolphthalein so a slight excess of 2 parts of NaOH in 15 parts of alcohol was added and was found sufficient to give the phenolphthalein color test. The phenolphthalein color indication was just discharged by introduction of carbon dioxide gas into the solution. Then the alcohol was distilled off, and the anhydrous salt (sodium N-lauryl sebacamate) was pulverized at -10°F .

A similar process to the above may be employed for producing the other alkali metal salts of the various amic acids.

The alkaline earth metal salts of the various amic acids may be made in a similar manner to the aluminum salts made above, namely, by combining the alkali metal salt of the amic acid in water solution with the appropriate alkaline earth chloride or other water-soluble alkaline earth salt. Purification is accomplished in the same manner as stated for the aluminum salts above.

(III) MIXED AMIC ACIDS AND GREASES THEREFROM

It has been found in accordance with our invention that by using mixtures of one or more different salts of amic acids, we may vary the properties thereof and of the greases therefrom advantageously. For example, but without limitation, we may improve the water tolerance characteristic of a grease by using a grease ingredient having an exceptional water tolerance, such as but without limitation, a salt of N-stearyl sebacamic acid; or rust protection may be improved by using a salt of N-oleyl sebacamic acid; or shear stability may be improved by using a salt of N-lauryl sebacamic acid; or dropping point may be improved by using a salt of N-octyl sebacamic acid. These various salts of amic acids mentioned in this paragraph may be combined with one another or with other herein mentioned amic acid salts to give the properties desired.

We have found that there are available commercially certain amines which contain varying proportions of primary and secondary amines.

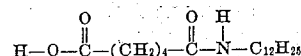
Illustrative of the mixed amines, but without limitation, is a technical lauryl amine of the following constituency:

	Per cent
Primary amines	85
Octyl amine	per cent. 8
Decyl amine	do. 9
Dodecyl (lauryl) amine	do. 47
Tetradecylamine	do. 18
Hexadecylamine	do. 8
Octadecylamine	do. 5
Octadecenylamine	do. 5
Mixed secondary amines	15

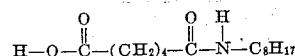
This mixed amine will produce, of course, a mixture of the corresponding amic acids with the dibasic acid selected.

By test, it was determined that the reacting (equivalent) weight of the technical lauryl amine is 235. This weight was then used in determining the reaction mixtures of the acid and amine in the preparation of amic acids as follows:

81. N-Tech. lauryl-adipamic acid



mixed with



and other like compounds of different hydrocarbon chain lengths in the amine group.

	Parts
Tech. laurylamine	235 (61.7%)
Adipic acid	146 (38.3%)

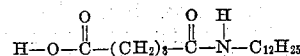
The amic acid was made in the same way as provided in Example 4.

The amic acid had the following properties:

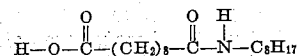
Test Data	Theoretical	Actual
Yield	95.3	94.9
Water loss	4.7	5.1
Nitrogen	3.86	3.80
Mean molecular weight	365	365
Acidity—calculated as oleic acid	77.7	77.35
Melting point		124
Appearance		(1)
Odor		Soapy

¹ Amorphous creamy white solid.

82. N-Tech. lauryl-sebacamic acid



mixed with



and other like compounds of different hydrocarbon chain lengths in the amine group.

	Parts
Tech. laurylamine	235 (53.6%)
Sebacic acid	203 (46.4%)

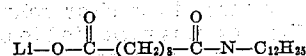
The amic acid was made in the same way as provided in Example 4.

The amic acid had the following properties:

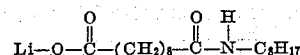
Test Data	Theoretical	Actual
Yield	95.8	95.6
Water loss	4.2	4.4
Nitrogen	3.33	3.29
Acidity—calculated as oleic acid	67.2	67.4
Mean molecular weight	420	418
Melting point		109° C.
Appearance		(1)
Odor		Soapy

¹ Light tan amorphous solid.

83. Lithium-N-Tech. lauryl sebacamate grease



mixed with



and other like compounds of different hydrocarbon chain lengths in the amine group.

N-Tech. lauryl sebacamic acid.....	Per cent	
Lithium hydroxide monohydrate.....		10
600/100° F. S. S. U. Coastal cylinder oil		1
blend		39

Salt content..... 100

The batch was made exactly as in Example 47. The finished grease had the following properties:

Penetration ASTM D-217-47T	276
Penetration—unworked	276
Penetration—5000 strokes	324
D./P. ASTM D-566-42	416° F.
Chrysler basket D./P.	367° F.
Water tolerance Chrysler	15%
C. R. C. wheel bearing grease test ...	O. K.
Rust preventive (10% salt spray) ...	O. K. after 72 hours
G. E. ball bearing test (212° F. at 3450 R. P. M.)	O. K. after 200 hours, no sign of failure

The properties represent a combination of properties resulting from the use of the mixture of amines in the formation of the grease base, which accordingly is a mixture of salts of the various amic acids made from such amines.

Lubricating greases made with compounds in accordance with our invention are superior to the greases made with the natural fatty materials in that the dropping points of the greases made in accordance with our invention are from 50-75° F. higher than the best natural fat greases of equivalent gelator content. Also, the amic acid compound greases, because of the polarity of the gelator are rust preventives and will actually displace water from metal surfaces, and will resist water after the surface has once been treated with the lubricant. In addition, natural fatty materials are mixtures of substances, some of which do not form greases. These non-grease forming substances are present in varying amounts and accordingly change the greases. In the greases of our invention all ingredients employed form greases and there is substantially no non-grease forming material.

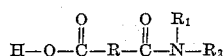
When properly compounded, our amic acid compound greases can be used at operating temperatures much nearer their ASTM dropping points than can the natural fat greases of equivalent gelator content.

The resistance to shear of our amic acid greases is much greater than the natural fat greases of equivalent gelator content.

The control of the manufacture of the greases of our invention is easier than in the manufacture of natural fat greases.

What we claim is:

1. As a new composition, a mixture containing a substantial proportion of a gelation agent and a petroleum lubricant, said gelation agent being selected from the group consisting of the alkali metal salt of an amic acid having the formula

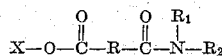


and a polyvalent metal salt of said amic acid, where R is selected from the group consisting of methylene groups having a carbon chain length of 1 to 8 carbon atoms and the group



R₁ is an aliphatic radical of a carbon chain length of 4 to 18 carbon atoms, and R₂ is selected from the group consisting of hydrogen and an aliphatic radical of a carbon chain length of 4 to 18 carbon atoms.

2. As a new composition, a mixture containing a substantial proportion of a gelation agent and a petroleum lubricant, said gelation agent being a compound having the formula

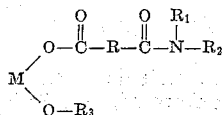


where X is an alkali metal, R is selected from the group consisting of methylene groups having a carbon chain length of 1 to 8 carbon atoms and the group



20 R₁ is an aliphatic radical of a carbon chain length of 4 to 18 carbon atoms, and R₂ is selected from the group consisting of hydrogen and an aliphatic radical of a carbon chain length of 4 to 18 carbon atoms.

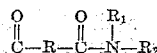
25 3. As a new composition, a mixture containing a substantial proportion of a gelation agent and a petroleum lubricant, said gelation agent being a compound having the formula



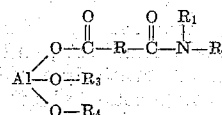
where M is an alkaline earth metal; R is selected from the group consisting of methylene groups having a carbon chain of 1 to 8 carbon atoms and the group



40 R₂ is an aliphatic radical of a carbon chain length of 4 to 18 carbon atoms, and R₃ is selected from the group consisting of hydrogen and an amic acid radical having the formula



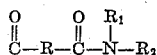
45 4. As a new composition, a mixture containing a substantial proportion of a gelation agent and a petroleum lubricant, said gelation agent being a compound having the formula



where R is selected from the group consisting of methylene groups having a carbon chain of 1 to 8 carbon atoms and the group



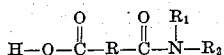
50 R₁ is an aliphatic radical of a carbon chain length of 4 to 18 carbon atoms; R₂ is selected from the group consisting of hydrogen and an aliphatic radical of a carbon chain length of 4 to 18 carbon atoms, and R₃ and R₄ are each selected from the group consisting of hydrogen and an amic acid radical having the formula



55 5. A grease having about 0.5% to 30% grease base of the total weight of the grease, said grease base being selected from the group consisting of

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an alkali metal salt and a polyvalent metal salt of an amic acid having the formula

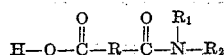


where R is selected from the group consisting of methylene groups having a carbon chain length of 1 to 8 carbon atoms and the group



R₁ is an aliphatic radical of a carbon chain length of 4 to 18 carbon atoms, and R₂ is selected from the group consisting of hydrogen and an aliphatic radical of a carbon chain length of 4 to 18 carbon atoms, and the remainder of the grease being composed principally of petroleum oil.

6. A grease having about 6 to about 17% of grease base, said grease base being selected from the group consisting of an alkali metal salt and a polyvalent metal salt of an amic acid having the formula



where R is selected from the group consisting of methylene groups having a carbon chain length of 1 to 8 carbon atoms and the group



R₁ is an aliphatic radical of a carbon chain length of 4 to 18 carbon atoms, and R₂ is selected from the group consisting of hydrogen and an aliphatic radical of a carbon chain length of 4 to 18 carbon atoms, and the remainder of the grease being composed principally of petroleum oil.

7. A grease composed of about 0.5 to 30% of N-lauryl.alkali metal sebacamate and the remainder being composed substantially of mineral oil.

8. A grease composed of about 10 to 13% of N-lauryl.alkaline earth metal sebacamate and the remainder being composed substantially of mineral oil.

9. A grease composed of about 1 to 10% of N-oleyl.alkali metal sebacamate and the remainder being composed substantially of mineral oil.

10. A grease composed of about 10% of N-palmityl.alkali metal sebacamate and the remainder being composed substantially of mineral oil.

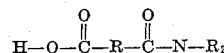
11. A grease composed of about 6% of N-lauryl.alkali metal azelaamate, and the remainder being composed substantially of mineral oil.

12. A grease composed of about 10% of aluminum di-N-lauryl sebacamate and the remainder being composed substantially of mineral oil.

13. A composition containing a substantial proportion of a gelation agent and a petroleum lubricant, said gelation agent being a mixture of at

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least two substances selected from the group consisting of the alkali metal salt of an amic acid having the formula

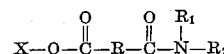


and a polyvalent metal salt of said amic acid, where R is selected from the group consisting of methylene groups having a carbon chain length of 1 to 8 carbon atoms and the group



R₁ is an aliphatic radical of a carbon chain length of 4 to 18 carbon atoms, and R is selected from the group consisting of hydrogen and an aliphatic radical of a carbon chain length of 4 to 18 carbon atoms.

14. A composition containing a substantial proportion of a gelation agent and a petroleum lubricant, said gelation agent being a mixture of at least two substances selected from the group consisting of an amic acid salt having the formula



where X is an alkali metal, R is selected from the group consisting of methylene groups having a carbon chain length of 1 to 8 carbon atoms and the group



R₁ is an aliphatic radical of a carbon chain length of 4 to 18 carbon atoms, and R₂ is selected from the group consisting of hydrogen and an aliphatic radical of a carbon chain length of 4 to 18 carbon atoms, the difference between the substances of the mixture being constituted by a different chain length in the aliphatic radicals attached to the N atom.

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