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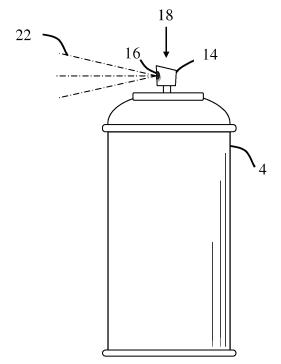
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## (54) Lubricant dispenser

(57) A lubricant dispenser is disclosed that comprises a container and a lubricant composition, the lubricant composition comprising a propellant and a salt of a fatty acid carboxylate and, optionally, a dispersant. The carboxylate is preferably stearate or palmitate. The dispenser is preferably an aerosol lubricant dispenser. The dispenser is useful in dispensing non-toxic and environmentally benign lubricant. Also disclosed is a method of making such a lubricant dispenser.



#### Description

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[0001] The present invention relates to lubricant dispensers and methods of making lubricant dispensers.

**[0002]** Lubricants for general use are known and may be liquids (for example, oils) or solids (for example PTFE, graphite, or molybdenum disulphide). Lubricant dispensers are known, including lubricant spray dispensers which dispense oil-based lubricants or dispersions of solids from pressurized containers. The pressurized containers may contain aerosol propellants.

[0003] US-A-2007/0027037 discloses an aerosol dispensing container that contains an aerosol propellant and a polytetrafluoroethylene (PTFE) lubricant mixture.

**[0004]** Unfortunately, there are drawbacks to known dispensers. Such drawbacks may include cost, staining, odour, loss of lubricant at the site of action, potential to be harmful to the environment and potential toxicity. This reduces the use of lubricant dispensers in many applications for which they would be useful, for example in domestic situations or in the food industry.

[0005] It is an aim of the present case to address these problems.

**[0006]** The present invention accordingly provides, in a first aspect, a lubricant dispenser comprising a container and a lubricant composition, characterised in that the lubricant composition comprises a propellant (preferably an aerosol propellant), and a salt of a fatty acid carboxylate.

[0007] Surprisingly, the inventor has discovered that such a lubricant dispenser may dispense lubricant that is highly penetrating, non-staining and reduces the tendency for a lubricated surface to attract dust or debris (unlike lubricating oils). Furthermore, such a dispenser dispenses lubricant that tends to stay at the site of application (unlike lubricating oils which tend to run away under gravity or to easily wipe away) and that does not dissolve in water or in many organic solvents. Finally, such a dispenser may be used in many applications because salts of a fatty acid carboxylates are generally environmentally benign, non-toxic and may be food contact approved.

[0008] Usually, the lubricant composition will further comprise a dispersant.

**[0009]** Usually, the dispersant will comprise a solvent. Examples of suitable solvents include  $C_4$  to  $C_6$  hydrocarbons (for example butane, pentane or hexane, of which pentane is preferred) a  $C_2$  to  $C_4$  alcohol (for example ethanol, propanol, (n-propanol or isoproposanol or a mixture), butanol (a single isomer or mixture of two or more)) or mixtures thereof. The dispersant is advantageous because it improves the dispersion (and hence flow) of the salt of a fatty acid carboxylate in the container and provides better mixing.

[0010] It is preferred if the salt of a fatty acid carboxylate is a particulate having a average particle size of 0.1  $\mu$ m to 10  $\mu$ m, preferably 0.2  $\mu$ m to 7  $\mu$ m, more preferably 0.4  $\mu$ m to 6  $\mu$ m, most preferably around 1 $\pm$ 0.5  $\mu$ m. The particle size is preferably below 10  $\mu$ m because this improves penetration of the particles into small gaps and improves contact between particles of the lubricant and surfaces to be lubricated. Furthermore, low particle size improves dispersion (including the stability of dispersion) of the lubricant particles in the container. Particle size may conveniently be measured by laser diffraction using a particle sizer (for example, a Mastersizer 3000 laser diffraction instrument with HydroMV accessory from Malvern Instruments Ltd). Generally particle sizers may use the Mie theory of particle sizing, especially those models intended for particles of irregular shape or particle size may be determined by inspection using transmission or scanning electron microscopy (SEM or TEM) or more conveniently by particle sizing software analysing SEM or TEM images.

40 **[0011]** Generally, the salt of a fatty acid carboxylate may be of formula:

Mn+ (RCOO-)<sub>n</sub>

wherein M is a cationic species, R is a  $C_7$  to  $C_{25}$  aliphatic chain and n is 1, 2 or 3. M may be ammonium, or an alkali metal cationic species. However, M is preferably a divalent metallic cationic species, more preferably Zn, Fe, or an alkaline earth or trivalent Al or Fe.

**[0012]** It is thus preferred if the metal of the salt of the fatty acid carboxylate or M in the formula above is selected from the group consisting of Mg, Ca, Zn, Al and Fe. It is most preferred if the metal or M is Ca or Mg.

**[0013]** Usually R will be a  $C_7$  to  $C_{25}$  alkyl chain (i.e. the fatty acid carboxylate is a saturated fatty acid carboxylate). Examples of suitable saturated fatty acid carboxylates are wherein the fatty acid carboxylate has an alkyl chain and is selected from the group consisting of caprylate ( $C_7$ ), caprate ( $C_9$ ), laurate ( $C_{11}$ ), myristate ( $C_{13}$ ), palmitate ( $C_{15}$ ), stearate ( $C_{17}$ ), arachidate ( $C_{19}$ ), behenate ( $C_{21}$ ), lignocerate ( $C_{23}$ ) and cerotate ( $C_{25}$ ).

**[0014]** The more preferred salt of a fatty acid carboxylate is selected from the group consisting of magnesium stearate, calcium stearate, zinc stearate, aluminium stearate, iron stearate, magnesium palmitate, calcium palmitate, zinc palmitate, aluminium palmitate, iron palmitate and mixtures thereof. The most preferred salt of a fatty acid carboxylate is selected from magnesium stearate, calcium stearate, magnesium palmitate, calcium palmitate and mixtures thereof.

**[0015]** The propellant may comprise a  $C_1$  to  $C_4$  hydrocarbon (which may be selected from the group consisting of methane, ethane, propane, butane, and mixtures thereof), or may be selected from the group consisting of hydrofluor-

oalkane (HFA), nitrous oxide, and carbon dioxide. Hence, the propellant may comprise a liquefied propellant. HFA, if used, may be selected from the group consisting of 1,1,1,2-tetrafluoroethane (HFA 134a), 1,1,1,2,3,3,3 heptafluoropropane (HFA 227) and mixtures thereof.

**[0016]** The lubricant dispenser will usually comprise a pressure resistant vessel (container) and a dispensing valve with a nozzle. Because the propellant is usually an aerosol propellant, the lubricant when dispensed is usually in the form of an aerosol (i.e. a fluid substance/mixture in the pressurized container that when dispensed from the pressurized container is released as a suspension of fine solid particles in a vapour). Generally, the aerosol dispenses in a mist-type pattern.

**[0017]** The lubricant dispenser may be packaged with an applicator tube. The applicator tube enables the lubricant to be better directed from the nozzle. The lubricant dispenser is preferably manually operable.

**[0018]** It is preferred if the lubricant dispenser is made by filling (usually pressure filling through the dispensing valve, but in some circumstances cold filling may be used) the container with the lubricant composition.

**[0019]** Thus, in a second aspect, the present invention provides, a method of making a lubricant dispenser, the method comprising providing a container, providing a lubricant composition comprising a propellant, and a salt of a fatty acid carboxylate, and filling the container with the lubricant composition. Usually, the method with further comprise providing a dispensing valve with a nozzle and sealing the container.

**[0020]** So that the present specification may be more completely understood, reference is made to the accompanying drawings in which:

Figure 1 illustrates a partial cross-section through a lubricant dispenser according to an exemplary embodiment.

Figure 2 illustrates the lubricant dispenser dispensing lubricant from the nozzle.

Figure 3 illustrates the lubricant dispenser with applicator tube dispensing lubricant.

**[0021]** Figure 1 shows a lubricant dispenser 2 comprising a metallic pressure resistant container 4 having a main body 8 and a top portion 6. The lubricant dispenser 2 also comprises a dispensing valve 13 with actuating button 14 and nozzle 16. The main body 8 of the pressure resistant container 4 contains a lubricant composition 10.

**[0022]** The lubricant composition 10 comprises propellant HFA134a, n-pentane as dispersant and magnesium stearate (2.5 wt % in n-pentane) as lubricant. Above the lubricant composition 10 is head space 12 containing vapourised propellant and providing a sufficient pressure reservoir so that, when dispensed through nozzle 16 of dispensing valve 13, the lubricant composition 10 forms an aerosol mist.

**[0023]** Figure 2 shows the lubricant dispenser 2 dispensing lubricant. When a user pushes down on the actuating button 14 in direction 18 the dispensing valve 13 is opened, releasing lubricant composition from the pressurized container 4. The lubricant composition is dispensed in a mist 22 from the nozzle 16.

**[0024]** Figure 3 shows the lubricant dispenser 2 dispensing lubricant with the use of an applicator tube 20. The applicator tube 20 is attached to nozzle 16 of the dispensing valve 13. As described in relation to Figure 2, when a user pushes down on the actuating button 14 in direction 18 dispensing valve 13 is opened releasing lubricant composition from the pressurized container 4. The lubricant composition is dispensed in a mist 22 from the end of applicator tube 22.

[0025] The invention is further illustrated by the Examples described below.

#### Example 1

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**[0026]** An oil-based lubricant product packaged in an aerosol canister with a dispensing valve was obtained and actuated until empty. After full de-pressurisation, the concave base of the can, close to the side wall, was punctured and the can was rinsed out with three aliquots of heptane.

[0027] Sub-micron magnesium stearate was prepared by adding magnesium stearate (10 g) to dehydrated ethanol (400 g) and high shear mixed using a Silverson mixer for 1 minute. The resultant dispersion was then added to the product vessel of an Avestin C50 high pressure homogeniser and processed in recirculation mode for 30 minutes at a pressure of 20,000 psig (138 MPa). Sub-micron magnesium stearate was dispersed in ethanol dispersion (60 ml) and was injected into the canister *via* the hole in the base and the can was chilled in dry ice. HFA (30 ml) propellant (1,1,1,2-tetrafluoroethane, HFA134a) was cooled in dry ice and injected into the can. The can was sealed using a self tapping screw and an EPDM rubber diaphragm. The canister was placed in a warm water bath at 30 °C to check for leakage. No leakage was apparent.

<sup>55</sup> **[0028]** The lubricant was sprayed *via* an applicator tube on to several squeaking door hinges and was found to be effective immediately at eliminating the squeaking.

#### Example 2

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[0029] Magnesium stearate (Merck, Partek grade, vegetable sourced) (7.5 g) was dispersed in n-pentane (283 g) by shaking for 20 seconds. The dispersion was microfluidized for 60 minutes using a Microfluidics M110-P microfluidizer with stirring in recirculation mode and using a 50 micron (JR20Z) diamond interaction chamber (IXC). The pressure was set at 20,000 psi (138 MPa) and the chiller at -5 °C. The particle size was sub-micron with a median size of about 0.5  $\mu$ m to 2  $\mu$ m and generally uniform as shown by TEM analysis.

**[0030]** The aerosol formulation was prepared by adding 105 ml of the sub-micron dispersion to an impact extruded aluminium aerosol canister (1 inch (2.54 cm) orifice, 175 ml brimful capacity). The can was then placed into a cryobath at -60 °C for 30 minutes after which time chilled HFA134a (20 ml) was added. The 1 inch valve with diptube was then crimped in place (LI98 1 inch (2.5 cm) valves (with butyl L133 dip tubes trimmed to the correct length) (Lindal) with ST300 + TI30 0.33 mm nozzles).

[0031] The sealed canisters were heated in a water bath at 50 °C for 3 minutes to check for integrity and leaks. None of the canisters leaked. The canisters were each fitted with a button actuator and an applicator tube (trimmed to 10cm). [0032] The canisters from this experiment produced a very rapidly evaporating spray which produced a dry coating within seconds of application.

#### Example 3: MGS aerosol canisters

[0033] Magnesium stearate (Fisher USP BP EP grade) (30 g) was dispersed in pentane (mixed isomers, HPLC grade 283 g) by shaking for 20 seconds. The dispersion was microfluidized for 50 minutes using a Microfluidics M110-P Microfluidizer with stirring in recirculation mode. The pressure was set at 138 MPa and the chiller at -5 °C. Other settings: auxiliary process module (APM) 200 micron; IXC: JR20Z 50 micron diamond. The magnesium stearate/pentane dispersion was mixed with more pentane to a 1:40 solid weight: weight of dispersion ratio (i.e. 1 g magnesium stearate to 41 g total weight pentane and magnesium stearate) (MGP dispersion). The particle size was sub-micron with a median size of about 0.5 µm to 2 µm and generally uniform as shown by TEM analysis. The MGP dispersion was much more stable to precipitation after shaking than a dispersion that had not been microfluidization.

[0034] MGP dispersion (130 ml) was poured into each of seven 210 ml capacity plain welded aerosol canisters. The canisters were chilled to -28 °C with moisture excluded.

[0035] H134a (25 ml) was added to each canister. The canisters were each immediately sealed by crimping LI98 1 inch (2.5 cm) valves (with butyl L133 dip tubes trimmed to the correct length) (Lindal) with ST300 + TI30 0.33 mm nozzles. The canisters were each fitted with a button actuator and an over cap and provided with an applicator tube.

**[0036]** The MGS aerosol produced a very uniform coating when sprayed directly from the nozzle and the coating has barrier properties on metal substrate by providing some degree of corrosion protection. The spray when directed from the tube applicator was found to be effective for targeted lubrication applications, for example, seized switches and squeaking door hinges.

[0037] A number of evaluation tests were conducted as described below.

Gas hob gas control knob lubrication

**[0038]** A gas hob burner knob had become stiff to operate and was almost impossible to turn due to build up of hardened grime/fats around the shaft not accessible for cleaning. Previously an oil-based spray lubricant had been used to lubricate it but the effect only lasted a few days. MGS aerosol was sprayed *via* the applicator tube around the shaft while turning the knob. The knob became far easier to turn and remained so for several weeks.

Cycle chain lubrication

**[0039]** A bicycle chain was dirty and did not move freely. The chain and all sprockets and their bearings were sprayed by MGS aerosol *via* the applicator tube. The chain moved easily immediately and remained free-running for several weeks. The effect of previous treatment with an oil-based spray lubricant lasted less than a week.

Cooker light switch lubrication

**[0040]** A soiled light switch on a cooker extraction unit was difficult to switch on and off. MGS aerosol was sprayed *via* the applicator tube on to the switch and the switch was well lubricated.

Waste pipe fitting to new lavatory pan

**[0041]** A lavatory pan was fitted inside the rubber seal of a plastic waste pipe and was difficult to insert without some form of lubrication. The rubber seal contacting areas were sprayed with MGS aerosol *via* the applicator tube. After MGS aerosol application the rubber gasket slid easily over the lavatory pan outlet and the difficulty in fitting was resolved.

Jammed torch switch

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[0042] A map reading magnifying glass with integral LED light had been left inside a car and the switch for the light had become jammed. The switch was sprayed with MGS aerosol *via* the applicator tube. The switch was freed up and remained free and smoothly functioning.

Rusty wood saw

- [0043] A wood saw left in damp conditions had developed rusted regions on the blade above the cutting zone which made sawing difficult due to increased friction. MGS aerosol was applied to both sides of the rusted blade via the spray nozzle. The lubricated areas of the blade could be readily seen due to the even, white deposit. The saw became easier to use after the MGS aerosol application.
- 20 Shower curtain rail

**[0044]** A shower curtain had become difficult to pull across due to build up of friction between the curtain hooks and the rail. The top of the rail was sprayed with MGS aerosol *via* the applicator tube. The curtain operated more freely.

25 UPVC door lock

[0045] A UPVC double-glazed door lock had become stiff. MGS aerosol was sprayed directly into the lock using the applicator tube. The lock became easy to operate and remained so.

**[0046]** Lubricant dispensers as claimed in the specification may include any feature described herein separately or in combination with any other feature(s), if necessary with appropriate modification of other features, as would be readily apparent to the skilled person.

#### **Claims**

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- 1. A lubricant dispenser comprising a container and a lubricant composition, **characterised in that** the lubricant composition comprises a propellant and a salt of a fatty acid carboxylate.
- 2. A lubricant dispenser as claimed in claim 1, wherein the lubricant composition further comprises a dispersant.

3. A lubricant dispenser as claimed in either claim 1 or claim 2, wherein the dispersant comprises a solvent.

**4.** A lubricant dispenser as claimed in claim 3, wherein the solvent comprises a  $C_4$  to  $C_6$  hydrocarbon, preferably a  $C_4$ 

to C<sub>6</sub> alkane.

- **5.** A lubricant dispenser as claimed in claim 3, wherein the solvent comprises a C<sub>1</sub> to C<sub>4</sub> alcohol, preferably a C<sub>2</sub> to C<sub>3</sub> alcohol.
- 6. A lubricant dispenser as claimed in any one of the preceding claims, wherein the salt of a fatty acid carboxylate is a particulate having a average particle size of 0.1 μm to 10 μm, preferably 0.2 μm to 7 μm, more preferably 0.4 μm to 6 μm.
  - 7. A lubricant dispenser as claimed in any one of the preceding claims, wherein the salt of a fatty acid carboxylate is of formula:

Mn+ (RCOO-)<sub>n</sub>

wherein M is a cationic species, R is a  $C_7$  to  $C_{25}$  aliphatic chain and n is 1, 2 or 3.

- 8. A lubricant dispenser as claimed in claim 7, wherein M is selected from the group consisting of Mg, Ca, Zn, Al and Fe.
- **9.** A lubricant dispenser as claimed in either claim 7 or claim 8, wherein R is a  $C_7$  to  $C_{25}$  alkyl chain.
- 10. A lubricant dispenser as claimed in any one of the preceding claims, wherein the fatty acid carboxylate is selected from the group consisting of caprylate (C<sub>7</sub>), caprate (C<sub>9</sub>), laurate (C<sub>11</sub>), myristate (C<sub>13</sub>), palmitate (C<sub>15</sub>), stearate (C<sub>17</sub>), arachidate (C<sub>19</sub>), behenate (C<sub>21</sub>), lignocerate (C<sub>23</sub>) and cerotate (C<sub>25</sub>).
- 11. A lubricant dispenser as claimed in any one of the preceding claims, wherein the salt of a fatty acid carboxylate is selected from the group consisting of magnesium stearate, calcium stearate, zinc stearate, aluminium stearate, iron stearate, magnesium palmitate, calcium palmitate, zinc palmitate, aluminium palmitate, iron palmitate and mixtures thereof.
  - **12.** A lubricant dispenser as claimed in any one of the preceding claims, wherein the propellant comprises a C<sub>1</sub> to C<sub>4</sub> hydrocarbon or is selected from the group consisting of hydrofluoroalkane, nitrous oxide, and carbon dioxide.
    - 13. A lubricant dispenser as claimed in any claim 12, wherein the  $C_1$  to  $C_4$  hydrocarbon is selected from the group consisting of methane, ethane, propane, butane, and mixtures thereof.
- 20 14. A lubricant dispenser as claimed in any one of the preceding claims, further comprising a dispensing valve and a nozzle.
  - **15.** A method of making a lubricant dispenser, the method comprising providing a container, providing a lubricant composition comprising a propellant, and a salt of a fatty acid carboxylate, and filling the container with the lubricant composition.

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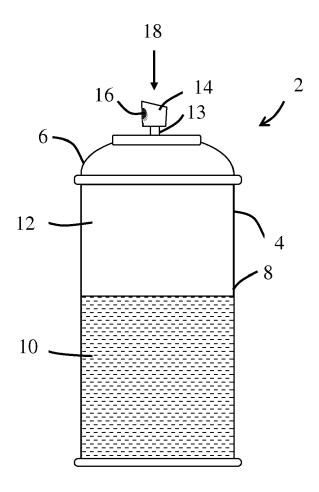


Figure 1

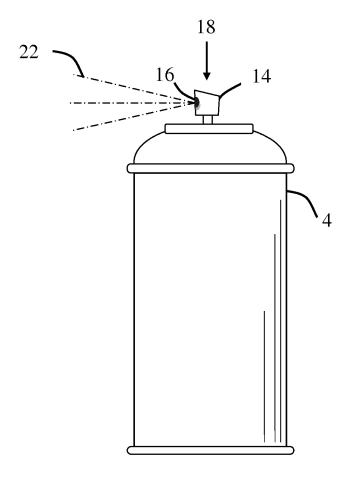


Figure 2

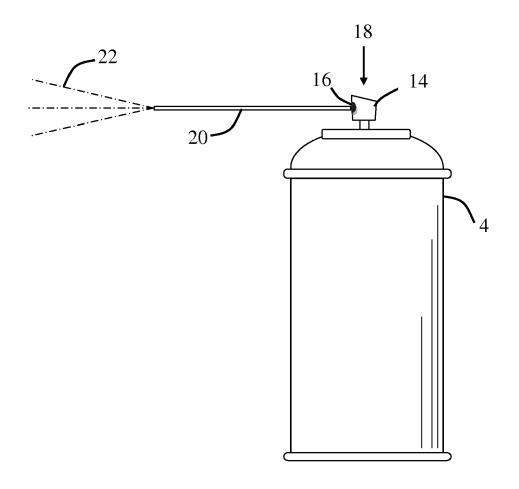


Figure 3



## **EUROPEAN SEARCH REPORT**

Application Number EP 14 17 3640

0-4	Citation of document with in	ndication, where appropriate,	Relevant	CLASSIFICATION OF TH
Category	of relevant pass		to claim	APPLICATION (IPC)
X	US 3 304 230 A (BEF 14 February 1967 (1 * column 4, lines 6		1-4,7,9, 10,12-15	INV. C10M105/24
Х	AL) 18 December 200	 IMANN ROBERT L [US] ET D1 (2001-12-18) L-15; examples 37,42 *	1-15	ADD. C10N10/02 C10N10/06 C10N10/16
X	US 4 443 348 A (WRI 17 April 1984 (1984 * column 3, lines 5 examples 1-2 *		1-15	
Х	US 3 461 079 A (GOL 12 August 1969 (196		1-4, 7-10,12, 14,15	
	* examples I,II *			
				TECHNICAL FIELDS SEARCHED (IPC)
				C10M
				C10N
	The present search report has Place of search Munich	been drawn up for all claims  Date of completion of the search  29 September 2014	4 Ber	Examiner trand, Samuel
C	ATEGORY OF CITED DOCUMENTS	T : theory or principle	underlying the ir	nvention
X : part Y : part docu A : tech	icularly relevant if taken alone icularly relevant if combined with anot iment of the same category nological background written disclosure	E : earlier patent doc after the filing date	ument, but publis e I the application r other reasons	hed on, or

## ANNEX TO THE EUROPEAN SEARCH REPORT ON EUROPEAN PATENT APPLICATION NO.

EP 14 17 3640

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Patent family

29-09-2014

Publication

1	0

Patent document

	cited in search report		date		member(s)	date
15	US 3304230	A	14-02-1967	BE DE FR GB US	643756 A 1492102 A1 3220 M 1040731 A 3304230 A	13-08-1964 30-04-1969 29-09-2014 01-09-1966 14-02-1967
20	US 6331509	B1	18-12-2001	US US	6331509 B1 2002098989 A1	18-12-2001 25-07-2002
	US 4443348	A	17-04-1984	JP JP US	H029079 B2 S5978295 A 4443348 A	28-02-1990 07-05-1984 17-04-1984
25	US 3461079	Α	12-08-1969	NONE		

Publication

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#### REFERENCES CITED IN THE DESCRIPTION

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## Patent documents cited in the description

US 20070027037 A [0003]