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(54) Method of making silicone-containing lubricating oils.

(57) The properties and performance of a lubricating oil containing silicones, and particularly the stability of the silicone molecules in suspension in the oil, are very significantly improved if during the addition of the silicones to the oil the mixture is subjected to a particular heat treatment and/or an electromagnetic energy field, particularly a strong magnetic field. In a preferred method of manufacturing the silicone oil, the silicones, preferably a dimethyl silicone fluid, (up to 20%) is added to a mixture of an oil (up to 40%), and a silicone solvent, preferably perchloroethylene, (remainder) in the presence of a magnetic field to form a silicone-rich carrier, and the carrier is subsequently added, as a small proportion, to a base oil, for example an SAE 30 paraffinic oil and again subjected to a magnetic field. The silicone oil produced should contain, by volume, not more than 1% silicone, preferably 0.2% and not more than 4% solvent, preferably 0.4% and is suitable for use as a two stroke oil, an upper cylinder lubricant, or an engine sump oil.

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- 1 -

"Improvements Relating to the Manufacture of
Silicone Oils"

This invention relates to the incorporation of silicone into lubricating oils to make what are referred to as silicone oils.

5. Mineral oils containing silicone are known, and have very much better lubricating properties than oils without silicone. Such silicone oils may be used with great effect as ordinary engine sump oils or as additives for sump oils, but are particularly suitable for use as upper cylinder
10. lubricants and two stroke oils, especially in modern high power to weight ratio two stroke engines which can operate at speeds up to 20,000 rpm. So far, however, there have been problems with the stability of such oils, the silicone tending
15. to separate too readily from the oil and, in some cases, the mixture breaking down under the extreme conditions encountered in the cylinders with the production of substances which are deleterious to the operation of the engine.
20. We have found that if during the manufacture of a silicone oil a particular heating cycle is followed or the mixture is subjected to a particular magnetic field, the stability of the silicone oil

will be a paraffinic mineral oil, and may comprise a mixture of different paraffinic mineral oils.

- For best results, the oil or oils used in the method in accordance with the invention should be substantially free of water (less than 30 parts per million by weight), and should preferably have a sulphur content which is less than 1%, preferably less than 0.3%, by weight. Most standard SAE 30 paraffinic oils which are on sale should be satisfactory from this point of view, particularly in respect of the low water contents.
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- The silicone which is used in the method in accordance with the invention preferably has a molecular chain length which substantially matches that of the oil, or in other words the viscosities of the silicone and the oil are preferably of the same order as each other, at least over the normal ambient and operating temperature range. Provided this requirement can be met, phenyl silicones, homologeous series silicones or halogenated silicones may be used, but preferably the silicone used in the invention is a dimethyl silicone. Particularly good results have been achieved with a half and half mixture of a dimethyl silicone fluid having a nominal viscosity of 300 centistokes at 25°C and a dimethyl silicone fluid having a nominal viscosity of 50 centistokes at 25°C.
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- The solvent, when used, is preferably perchloroethylene, which may be of an industrial or analytical grade, but other common aliphatic solvents or aromatic solvents may be used, such as carbon tetrachloride, chloroform, ethylene dichloride, trichlorethylene, benzene, toluene, xylene, diethyl ether, di-isopropyl ether, or white spirit. To a lesser extent, cyclohexane,
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- and preferably the carrier oil comprises at least a portion which is the same as the base oil. In the preferred case where the base oil is a standard SAE 30 paraffinic mineral oil, the carrier oil is
5. preferably a mixture of paraffinic technical white oil and a paraffinic mineral oil which is identical to the base oil, preferably in the ratio of 4:1.

- In preparing the silicone-rich carrier the heating of the components in the absence of oxygen
10. is important to prevent oxidation of the silicone. There are a number of possible ways of doing this, but the preferred method, at least when the boiling point of the solvent is below 160°C (as is the case with the preferred solvent perchloroethylene)
15. is not to add the silicone until the carrier oil and the solvent have been mixed and raised to the boiling point of the solvent in a suitable vessel. After allowing the oxygen to be driven from the vessel by the boiling solvent, the silicone is
20. then injected directly into the mixture in the vessel, and boiling of the mixture is maintained for a short while before sealing the vessel and allowing the contents to cool slowly. The silicone-rich carrier is preferably added to and mixed with
25. the base oil at a temperature of 60°C , this step and the subsequent cooling of the final mixture taking place in the absence of oxygen for the same reason as mentioned above.

- Preferably however, the stabilisation of the
30. silicone suspension in the method in accordance with the invention is carried out using an electromagnetic energy field rather than the relatively complicated heat treatment described above. A magnetic field has been found to be particularly

equivalent to the silicone-rich carriers hereinbefore described. In other words the additive would comprise, by volume, not more than 20% silicone, not more than 40% oil, and solvent as the remainder, it being the

5. intention for such an additive to be added to sump oil in an amount of from 1 to 5% by volume of the sump oil. The manufacture of the additive would be exactly the same as for the silicone-rich carriers described earlier. In this case the oil will preferably
10. be a mixture of paraffinic technical white oil and an SAE 30 paraffinic mineral oil, preferably in the ratio of 4 : 1. Generally it is thought that the silicone and solvent contents will be less for a diesel engine sump oil additive than for a petrol
15. engine sump oil additive.

Three examples in which the method in accordance with the invention was carried out to form a silicone oil suitable for use as a two stroke oil, an upper cylinder lubricant, or an engine oil will now be described.

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Example I

- A dimethyl silicone mixture was made consisting of 50% dimethyl silicone fluid obtainable from ICI as F111/50 (having a nominal viscosity of 50 centistokes at 25°C), and 50% dimethyl silicone fluid
25. obtainable from ICI as F111/300 (having a nominal viscosity of 300 centistokes at 25°C). In addition, a carrier oil mixture was made consisting of, by volume, 80% paraffinic technical white oil, and
30. 20% of a standard SAE 30 paraffinic oil. This oil was obtained as SAE 30 Castrol 110A, and had a water content less than 30 parts per million by weight, and a sulphur content less than 0.3% by weight.

oil containing, by volume, 0.2% silicone, 3.5% perchloroethylene, 1.04% technical white oil, and the SAE 30 paraffinic oil as the remainder.

5. Example II

As in Example I, the first stage was to make a silicone-rich carrier. This carrier had exactly the same composition as that in Example I, and again the carrier oil mixture and the perchloroethylene were mixed together before the dimethyl silicone mixture was added. In this case however, the mixture of the carrier oil and the perchloroethylene was placed, at room temperature, in a test tube within a coil comprising between 5,000 and 6,000 turns of SWG 34 copper wire, and the coil energised by a 28 volt d/c supply to generate an electromagnetic field within the coil. The dimethyl silicone mixture was then added to and mixed with the contents of the test tube in the presence of this relatively weak magnetic field and the mixture subjected to the field for about 5 minutes.

After that, some of the silicone-rich carrier so formed was added to and vigorously mixed with a quantity of a base lubricating oil in the form of a standard SAE 30 paraffinic oil (obtained as SAE 30 Castrol 110.), the silicone-rich carrier being added in an amount which was 5% by volume of the total mixture. In contrast to Example I however, the silicone-rich carrier was added to the base oil at room temperature and the mixture was subjected to the same magnetic field which was used in the formation of the silicone-rich carrier. The mixture was subjected to the magnetic field for about 5 minutes, and the result was a high quality silicone oil having the same composition as that produced in Example I.

have been carried out using the oils made in Examples I to III, and these are described and reported as follows.

5. Test 1

Two clear glass jars were set up, one jar containing 500ccs of the silicone oil produced in Example II, and the other jar containing 500ccs of a silicone oil of identical composition and manufactured in exactly the same manner except that the silicone-rich carrier and the final mixture were not subjected to a magnetic field or any other treatment. Then, without disturbing the jars or their contents in any way, the nature and appearance of the two oils were observed over a number of days. After the first day the control oil (i.e. the oil with no magnetic treatment) appeared homogeneous except for thin horizontal clear regions, or striae, near the upper surface. This condition remained substantially the same until the fifth day when large particles (silicones) began depositing on the bottom of the jar. By the sixth day there was a fairly heavy deposit of silicones at the bottom of the jar and the striae were more pronounced, this trend continuing through to the ninth day. In contrast, throughout the whole nine day period the silicone oil produced in accordance with Example II remained a completely homogeneous suspension, exhibiting no striae or deposition. During this period the ambient temperature around the jars ranged between 10 and 12°C, but on the tenth day the temperature suddenly increased to 16°C, following which some of the heavy silicone

pressure, and are well known in the oil industry for determining the performance of an oil. The results of the tests are given in the following table:-

	Wear	Seizure Under Pressure	Weld Point
5. Base Oil Only	2.432mm	65kg	130kg
10. Control Oil (Silicone oil with no treatment)	1.569mm	75kg	135kg
15. Example II (Silicone oil with magnetic field treatment)	1.545mm	80kg	140kg

These results show very clearly that simply mixing silicones with a base lubricating oil very much improves the properties of the oil, and more importantly that by making the silicone oil in accordance with the invention the properties of the oil are improved still further quite appreciably.

Test 3

25. Two motor cars whose petrol consumption had been measured over a long period of time using the recommended standard petrol and oil were then run using the silicone oil produced in Example I as an upper cylinder lubricant (15 to 30 ccs per gallon of petrol)

30. and using the silicone-rich carrier of Example I as a sump and gear box oil additive (5ccs per pint), and the average petrol consumption measured over a period of about seven months. In the first motor car, a VW Golf, the average petrol consumption before

5. In the case of one commercially available synthetic two stroke oil the reduction in the average diameter of the piston thrust face was 60 microns, and in the case of another commercially available synthetic two stroke oil the reduction was 25 microns, both very large amounts of wear in the relatively short duration of the tests. In addition, the piston thrust face was scratched in each case, although in general the engine was fairly clean apart from light carbon streaks on the engine bore and spark plug with the first oil.
10. In contrast, when using the silicone oil produced in Example I as the two stroke oil, no reduction in the average diameter of the piston thrust face was measured, indicating zero wear over the duration of the test. Furthermore there appeared a bright mirror finish on the piston surfaces, although the rest of the engine was perhaps not quite as clean as after using the synthetic oils. The spark plug however was a uniform grey colour, which is good.
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Test 5

25. The 21cc Ohlsson and Rice two stroke engine was run powering the Champ generator, and the engine surface temperature was measured at different power output levels using a calibrated thermistor bolted to the engine cylinder head. The test was carried out a number of times using different two stroke oils in the petrol used to fuel the engine (2% oil in the mixture), and the results are shown in the accompanying graphs which plot engine surface temperature (degrees centigrade) against generator power output (current in amps). It was found that the engine would often run at
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during its manufacture.

In the graph of Figure 3, the curve x—x represents a fuel containing 2% of a commercially available synthetic two stroke oil different from those used in the tests of Figure 1;

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the curve ■—■ represents a fuel containing 2% of a two stroke silicone oil in accordance with the invention and consisting of a half and half mixture of the two oils used in the tests of Figure 2; and,

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the curve ○—○ represents a fuel containing 2% of a two stroke silicone oil which is the same as that represented by ■—■ in Figure 2 except that the base oil used in forming the silicone oil contained 0.2% of an aliphatic chlorinated wax (obtained from ICI as Cereclor 42).

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The ambient temperature throughout the tests was 11°C. As can be seen from the graph the engine temperatures at relatively low loads were much the same for the synthetic oil and the silicone oil mixture, but at higher loads were much the same for the synthetic oil and the engine would not actually produce maximum power. When run using the silicone oil containing Cereclor 42 the engine ran very cool at low loads, perhaps too cool since a fluffy deposit was found on the sparking plug. In all other cases using a silicone oil in accordance with the invention the sparking plug ended in very good condition, being a matt grey colour with no deposits. In general however, the reduction of the operating temperature of an engine, which the use of silicone two stroke oils produced in accordance with the invention appears to do, is beneficial in that there tends to be less wear in the engine and there is less

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- 19 -

C L A I M S

1. A method of making a silicone oil, in which a suspension of silicone in a lubricating oil is formed and the stability of the suspension is improved by subjecting the mixture to an energy field which is thermal and/or electromagnetic in nature.
5. 2. A method according to claim 1, in which the oil is a paraffinic mineral oil having a water content of less than 30 parts per million by weight and a sulphur content of less than 1% by weight.
10. 3. A method according to claim 1 or claim 2, in which the silicone is a dimethyl silicone having a molecular chain length which substantially matches that of the oil.
15. 4. A method according to any one of claims 1 to 3, in which the suspension is formed by mixing together the silicone, the oil, and a solvent in which the silicone is at least partly soluble.
20. 5. A method according to claim 4, in which the silicone oil produced is intended for use as an engine sump oil, a two stroke oil, or an upper cylinder lubricant, and comprises, by volume, not more than 1% silicone, not more than 4% solvent, and oil as the remainder.

- 21 -

- and the solvent first being mixed with a carrier oil and heated to between 100°C and 160°C in the absence of oxygen to form a silicone-rich carrier comprising, by volume, not more than 20% silicone, not more than 40% carrier oil, and solvent as the remainder, and the silicone-rich carrier subsequently being added to and mixed with a base oil at a temperature between 25°C and 125°C and the mixture, which forms the silicone oil, allowed to cool in the absence of oxygen.
5. 13. A method according to claim 12, in which the solvent is perchloroethylene and is heated with the carrier oil to at least 127°C before the silicone is added in the absence of oxygen to form the silicone-rich carrier.
10. 14. A method according to claim 12 or claim 13, in which the silicone-rich carrier is added to the base oil at a temperature of 60°C.
15. 15. A method according to claim 9 or any one of claims 12 to 14, in which the silicone-rich carrier comprises 4% silicone, 26% carrier oil, and 70% solvent, and is added to the base oil in an amount which is 5% by volume of the mixture thereof.
20. 16. A method according to any one of claims 9 to 15, in which the base oil is an SAE 30 paraffinic mineral oil having a water content of less than 30 parts per million by weight and a sulphur content of less than 1% by weight, and the carrier oil is a mixture of paraffinic technical white oil and a paraffinic mineral oil identical to the base oil.
25. 17. A method according to claim 16, in which the paraffinic technical white oil constitutes 80%, by volume, of the carrier oil.
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Fig.1.

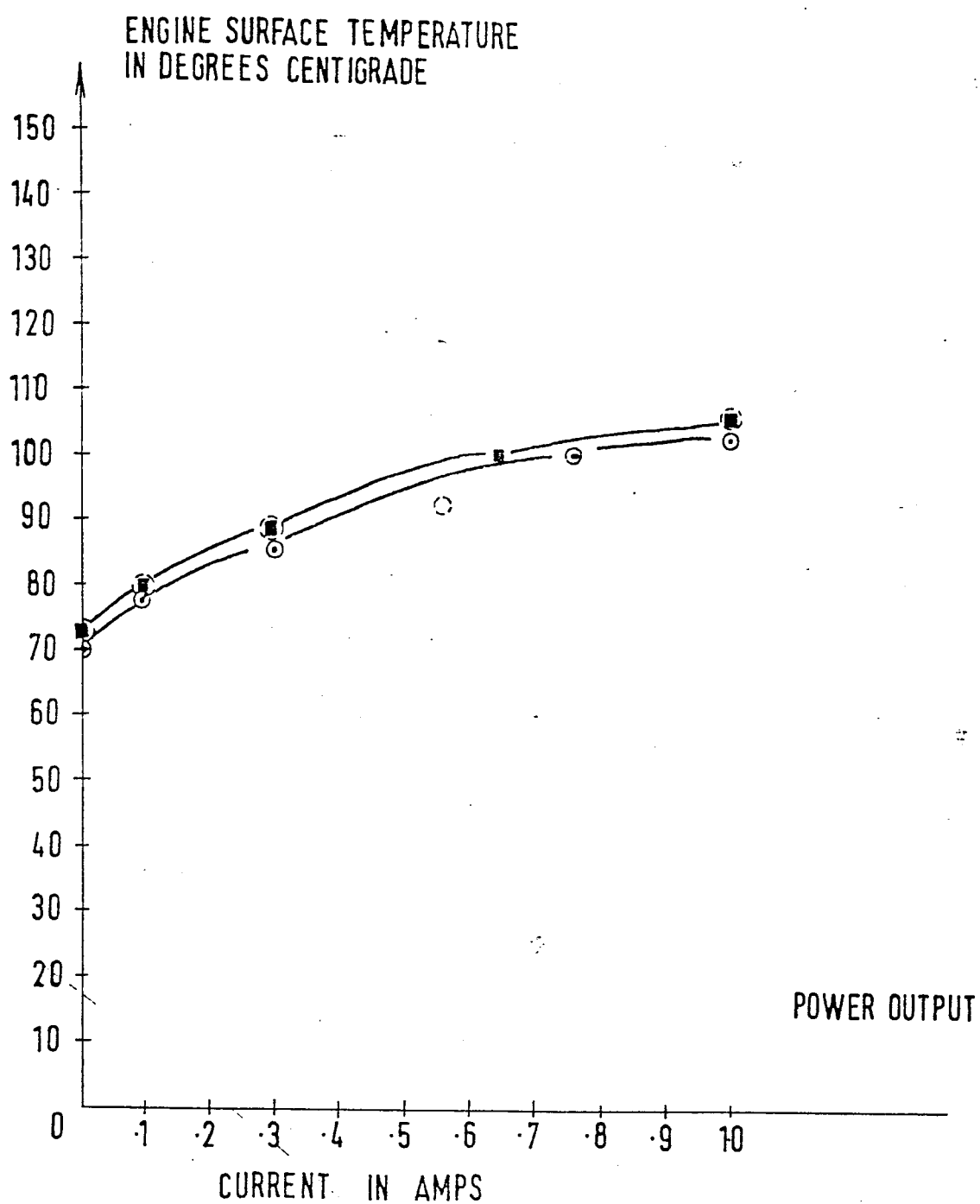


Fig.2.

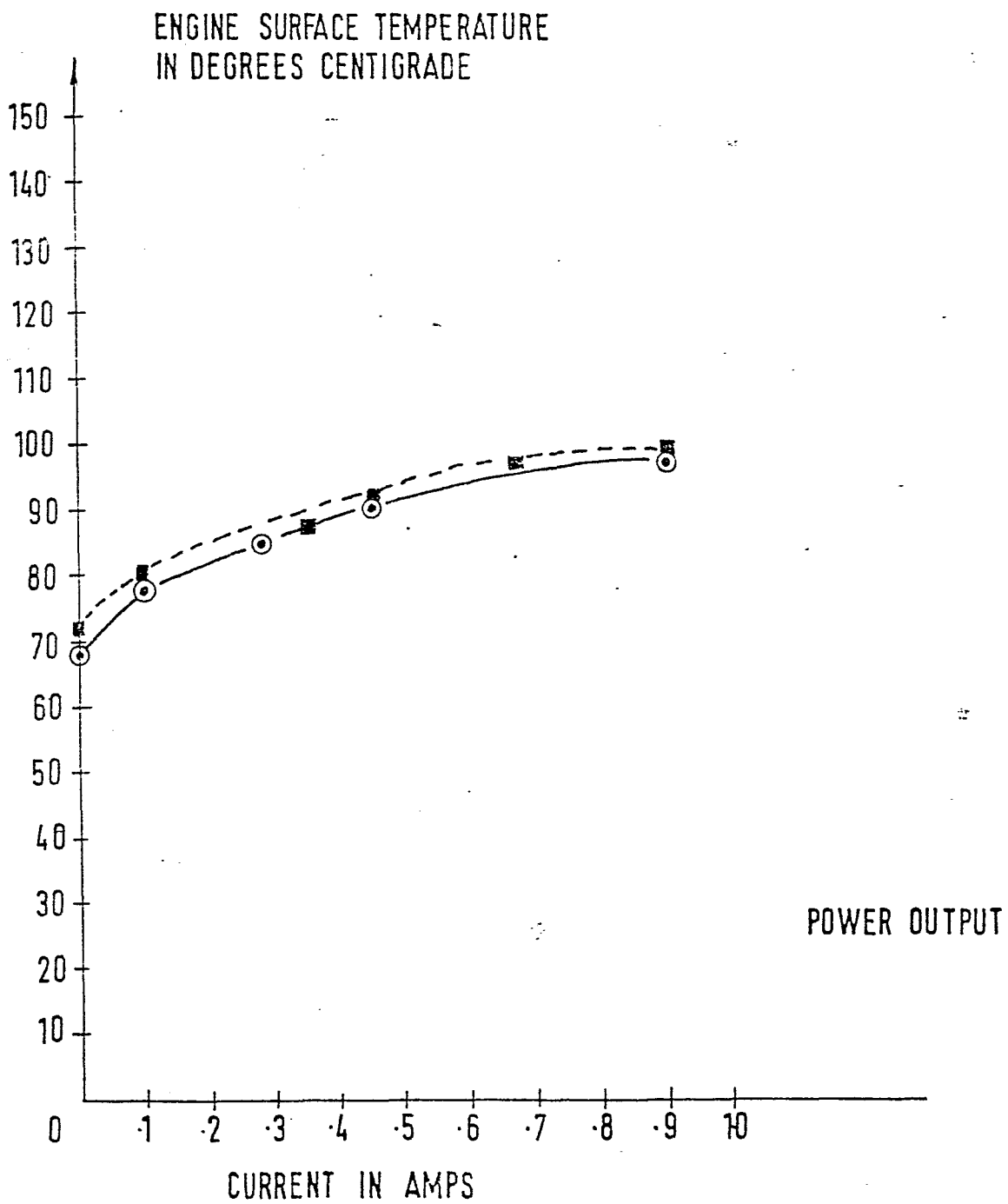
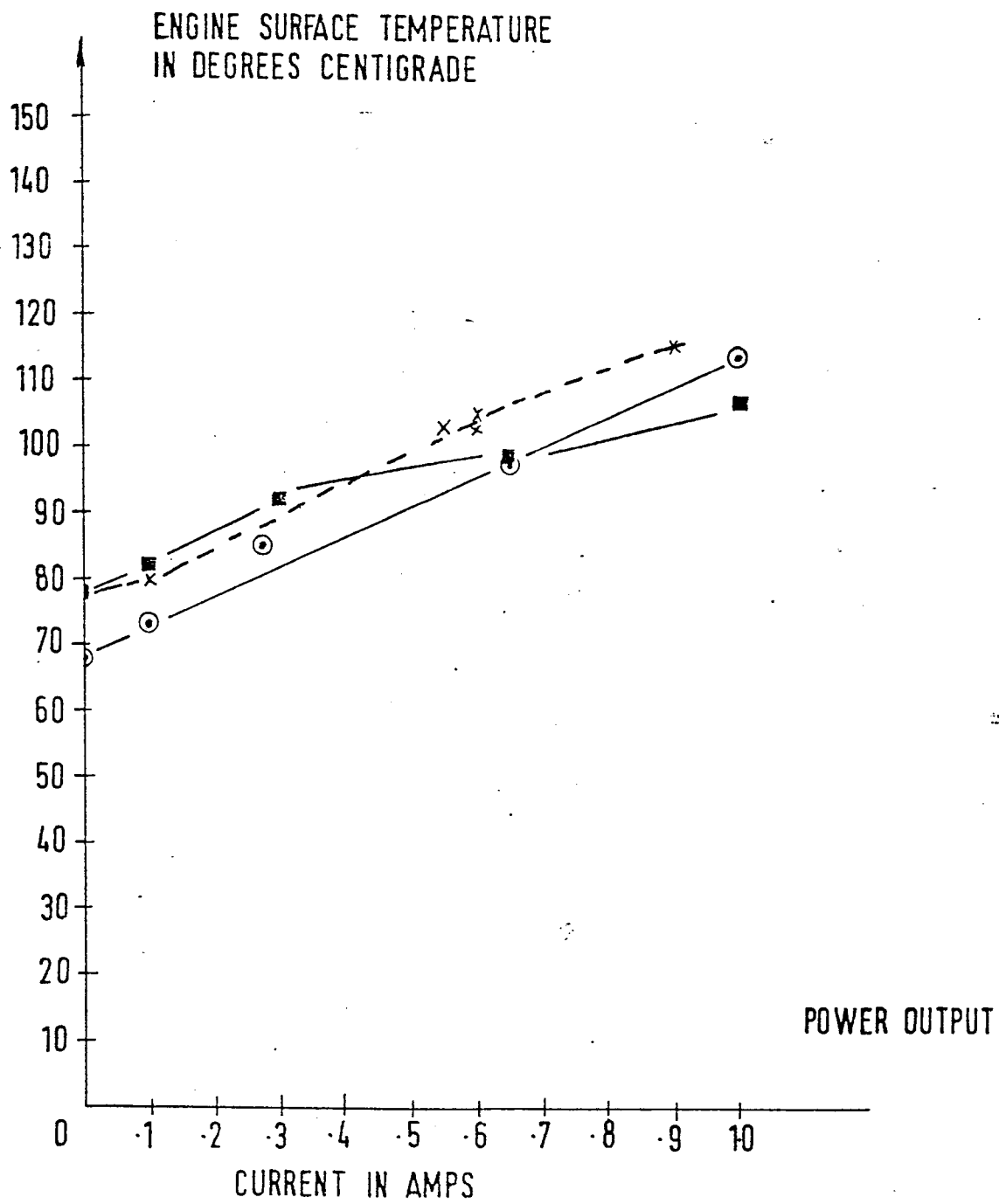


Fig.3.





European Patent
Office

EUROPEAN SEARCH REPORT

0006000

Application number

EP 79 30 0996

DOCUMENTS CONSIDERED TO BE RELEVANT			CLASSIFICATION OF THE APPLICATION (Int. Cl. ³)
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	
X	<u>US - A - 3 024 194</u> (E.S. FRANCIS et al.) * Column 7, line 3 - column 9, line 59; column 10, line 58 - column 11, line 38; column 6, lines 30-41; column 1, line 69 - column 2, line 68 * --	1-6	C 10 M 1/50 F 01 M 3/04
A	<u>GB - A - 1 208 022</u> (MONSANTO)		
A	<u>GB - A - 600 693</u> (SHELL)		
A	<u>US - A - 3 275 559</u> (C.J. HENRY) ----		TECHNICAL FIELDS SEARCHED (Int.Cl. ³) C 10 M 1/50
			CATEGORY OF CITED DOCUMENTS
			X: particularly relevant A: technological background O: non-written disclosure P: intermediate document T: theory or principle underlying the invention E: conflicting application D: document cited in the application L: citation for other reasons
			&: member of the same patent family, corresponding document
The present search report has been drawn up for all claims			
Place of search	Date of completion of the search	Examiner	
The Hague	06-09-1979	RO TSAERT	