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Journal of Chromatography A, 669 (1994) 139–150

JOURNAL OF CHROMATOGRAPHY A

Rapid screening of rolling mill oils using high-temperature capillary gas chromatography

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(First received October 8th, 1993; revised manuscript received January 18th, 1994)

Abstract

The application of gas chromatography to rapid screening of rolling mill oils is investigated. A specially manufactured, high-temperature glass capillary column is used to separate typical rolling oil analytes such as fatty acids, mono-, di- and triglycerides, synthetic esters and mineral oil in one analysis. The column has a long life and meets the specifications set by ASTM D2887 for simulated distillation of mineral oils. Reliable and unique chromatograms are obtained to fingerprint a number of rolling mill oils. Information gathered can be correlated to the chemical composition and physical properties of commercial oils. A standard gas chromatograph, equipped with a split/splitless injector, gives sufficient reproducibility to detect significant changes in the composition of oils. The method is under evaluation for routine screening of batches of oils before release to the rolling mills.

1. Introduction

The steel industry is increasingly being compelled to manufacture and supply products to stringent quality control standards, utilizing environmentally friendly processes. In the field of rolling mill oil technology the surface quality of the rolled product, production costs, production volume and the environmental impact are critical in the selection of lubricants. A constant demand exists for larger reductions, better load-carrying and friction-reducing performance, higher mill speeds, lower loads and reduced staining tendencies. To satisfy this demand, rolling mill lubricants underwent significant development in recent years, but associated analytical control methods did not develop to the same degree. When considering analytical methods, it is important to realize the complicated nature of rolling oils. They may contain mineral oil, additives and whole esters (triglycerides, triacyl glycerols, TAGs), partial esters (diglycerides or monoglycerides), synthetic esters and free fatty acids which can be mixtures of saturated and unsaturated compounds with varying chain lengths. Rolling oil formulations are traditionally based on natural glycerides due to their excellent lubricating performances, but they tend to yield high levels of carbon deposits on the steel surface after annealing. Synthetic esters based on *e.g.* trimethylol propane (TMP), neopentylglycol (NPG) and pentaerythritol, have been developed for better steel surface cleanliness.

The complexity of the sample, therefore, con-

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0021-9673/94/$07.00
SSDI 0021-9673(94)00055-E

tributes to the difficulty in developing a chemical screening method [1,2]. Rolling oil formulation is also in the hands of long-established suppliers who regard rolling oil know-how as a trade secret. They formulate and manufacture the oils according to the type of mill and method of application, resulting in a large range of different products with compositions unknown to the steel industry. This report considers the investigation of a rapid analytical method for screening rolling oils, such as hot rolling oils, sheet (cold) rolling oils, tin plate rolling oils and process oils such as pickler (after pickling) oils. The existing needs for such a method include:

Laboratory evaluation of new formulations. Because of the time and costs involved in full-scale mill tests, and the potential for product loss in the case of failure, there is an urgent need to develop more modern laboratory test methods for the evaluation of new products. The fact is, however, that laboratory evaluations are commonly considered only as a screening tool. The ultimate performance of a rolling oil can only be evaluated on the mills and it is doubtful whether key characteristics, or even the staining tendencies, can be predicted by any means other than plant trials. Useful information can, however, be obtained from a chemical analysis of a candidate oil by comparison with that of an oil with known in-plant performance.

Constant analysis and evaluation for conformity to specification. Every shipment of rolling oil must be inspected and laboratory tested prior to being released for use. A fast screening method for chemical composition is required to serve as a very discriminative acceptance test [2].

Studies on rolling oil degradation. The TAGs in the fat can decompose during storage to form free fatty acids. Degradation products include free fatty acids, aldehydes and ketones [3]. Polymerization or hydrolysis can also occur during electrostatic application. These factors can affect the lubricating performance of an oil as well as consumption and product quality. Chemical analysis can easily reveal the relative amount and nature of any decomposition.

Several traditional physical and chemical laboratory tests, listed in Table 1, are available to determine pertinent properties of process and rolling oils. These tests regard the oil as a simple substance, resulting in the control method being based on an indirect evaluation, and not on chemical analysis. More advanced methodology and instrumental analytical techniques have also been reported for comprehensive research purposes. Tanikawa and Fujioka [1] reported an approach for the clarification of the lubrication performance of cold rolling oil using the analytical methods of HPLC, gel permeation chromatography, GC, IR and GC–MS. These methods were utilised to investigate reaction products such as free and iron–soap-forming fatty acids, and to determine their carbon number and degree of unsaturation. Tusset and Hancart [2]

Table 1
Rolling oil tests

Test	Description
Saponification value	Number of –COO– groups (including–COOH groups) per unit weight of rolling oil (according to ASTM D94 [14])
Acid value	Number of –COOH groups per unit mass of rolling oil (according to ASTM D974 [15])
ESI	Emulsion stability index
SI	Spreadability index
BOI	Burn-off index
Viscosity	Viscosity of undiluted oil (according to ASTM D445, 40°C [16])
Density	According to ASTM D1217, 40°C [17]
Falex test	Failure load [p.s.i. at 40°C, 1 p.s.i. = 6894.76 Pa)]
Chloride test	According to ASTM D1317 [18]
Sulphur test	According to ASTM D1552 [19]
Flash point	According to ASTM D93 [20]

reported a new method for the direct analysis of triglycerides, additives and mineral oil content. This method involves GC, MS, IR, Fourier transform IR, an electrothermal atomic absorption unit and tedious sample preparation techniques such as liquid–liquid extraction, silica gel column chromatography, and chemical conversion (derivatization) of polar analytes. While the traditional methods provide insufficient information to suit modern technology, the above-mentioned techniques are aimed at research programmes which require complete chemical analysis. A rapid screening method does not require such detailed analysis, but should rather be fast, reliable, inexpensive and simple to perform. In this report the use of GC for such a method was investigated. Although Tusset and Hancart mention the application of GC as a fast screening method in their paper, no attention or information is given regarding important aspects of the technique such as column bleed, column stability, column fouling due to highly polar additives, long-term column performance, losses due to the injection technique and repeatability. This report considers the evaluation of an non-polar glass capillary column for screening rolling oils and to compare chromatograms of samples to those of reference standards and calibration graphs. The injection technique is also investigated. The method is intended to be fast, uncomplicated and easily adaptable by a works laboratory, employing inexperienced chromatographers. The scope of the method includes analytes such as fatty acids, mono-, di- and triglycerides and mineral oil.

Most GC columns developed for fatty acid or glyceride separations are aimed at maximum resolution according to the degree of unsaturation and a quantitative analysis of all individual components in the mixture, which requires a column with sufficient polarity. This application, however, only requires separation by carbon number, for which an non-polar column is suitable. As most rolling oils contain a large quantity of mineral oil, the column was also intended to be able to determine the boiling point distribution of the petroleum fractions. The standard test method for simulation distillation (SIMDIS) analysis and certain column requirements are described in ASTM D2887 [4]. Column resolution, as measured on the n-C_{16}/n-C_{18} pair, is required to be between 3 and 8, and the hydrocarbon with the lowest boiling point to have a retention time of at least 1 min. The stationary phase is also required to be stable at high temperatures with minimum bleed. The glass capillary column used in this study was, therefore, evaluated for its stability at the high temperature (up to 400°C) at which the TAGs and mineral oil elute.

The most important aspect of a GC analysis is probably the quantitative injection of the TAGs, for which on-column injection is the preferred technique [5,6]. Incomplete sample transfer is associated with the range of boiling points dealt with, and sample decomposition [7]. It has been found that although on-column injection can greatly limit these losses, a certain degree of decomposition or discrimination is unavoidable. Hinshaw and Seferovic [8] compared split and splitless programmed-temperature (PTV) injection to cold on-column and hot (classical) split injection. The PTV injector gave results comparable to cold on-column injection but, due to strong discrimination and decomposition, the hot split (conventional) injector was the least suitable for TAG analysis. It is, however, also important to consider column fouling components, such as highly polar additives present in the oil. A direct injection technique is not suitable in cases where these substances have not been removed prior to injection, as it can lead to fouling and rapid column deterioration. If a limited degree of sample loss is not critical, a conventional hot split injector, fitted with a sleeve containing silanized glass wool, can solve this problem. Bannon *et al.* [9] suggested (a) high-speed injection to overcome needle discrimination, (b) higher injection temperature, diluted sample solution and small sample volume to achieve rapid vaporization, and (c) improved insert design to achieve good mixing. Although their study was focused only on fatty acid methyl esters, the principles should also apply to heavier components. For the study in this report it was deemed possible to utilize a conventional hot

split injector for rolling oil analysis and to limit decomposition or discrimination by adhering to the recommendations of Bannon *et al.* It has been shown that the recovery of TAGs depends not only on the injection technique, but also on column quality, carrier gas flow-rate, the molecular mass of the component and amount of sample injected [5]. Although absolute quantitation is not required in our application, these effects were kept in mind to ensure sufficient reproducibility, to enable the detection of small changes in oil composition.

2. Experimental

2.1. Capillary gas chromatography system

Experimental investigations were performed using a Siemens Sichromat capillary gas chromatograph equipped with a conventional hot split/splitless injector and a flame ionization detection (FID) system.

2.2. Capillary column

A high-temperature glass capillary column was manufactured according to the method of Blum and Aichholz [10] in the university laboratory of one of the authors. The column was 12 m × 0.3 mm I.D. and had a stationary phase film thickness of 0.2 μm. The stationary phase was PS089 (Petrarch Systems, Bristol, USA), an OH-terminated, 5% phenyl-, 95% polydimethylsiloxane [10]. The column was tailored for high-temperature applications up to 400°–450°C [10], with a stationary phase film thickness that gives a compromise between low elution temperatures and high column loadability. Flexible fused-silica end sections ("legs") were attached [11] to the glass capillary to allow convenient coupling of the column to the gas chromatograph.

2.3. Calibrating standards and samples

The following reference standards were used:

(i) A boiling point standard was prepared, from a mixture of *n*-alkanes with boiling points as indicated in Table 2, for the distillation curve.

Table 2
Contents and boiling points of the *n*-alkane mixture

Peak	Carbon number	Boiling point (°C)
1	17	302
2	18	316
3	22	369
4	24	391
5	26	412
6	28	431
7	32	466
8	34	481
9	36	496
10	38	509
11	40	522
12	42	534
13	44	545

Values as reported in ASTM D2887 [4].

(ii) A fatty acid methyl ester (FAME) mixture, "AOCS animal and vegetable reference mixture No RMS-6" of which the composition is given in Table 3, was obtained from Supelco.

(iii) Malaysian palm oil containing free fatty acids (C_{14}, C_{16}, C_{18}), diglycerides (D_{32}, D_{34} and D_{36}) and triglycerides (T_{46}, T_{48}, T_{50}, T_{52}, T_{54} and T_{56}). Nomenclature is according to Geeraert and Sandra [12].

(iv) A rolling oil base containing beef tallow, obtained from a supplier.

Typical cold rolling, hot rolling and pickler oil formulations, from a variety of suppliers, were used to obtain comparative chromatograms.

Table 3
Composition of the RMS-6 standard

Peak	Component	Concentration (%, w/w)
1	Methyl myristate (C14:0)	2
2	Methyl palmitate (C16:0)	30
3	Methyl palmitoleate (C16:1)	3
4	Methyl stearate (C18:0)	14
5	Methyl oleate (C18:1)	41
6	Methyl linoleate (C18:2)	7
7	Methyl linolenate (C18:3)	3

Nomenclature according to Geeraert and Sandra [12].

2.4. Chromatographic conditions

From the results given in earlier papers [5–9] describing the dependence of results on various parameters, the following conditions were chosen:

The injection technique. The rationale in using a conventional hot split injector is that it is the most practical sample introduction technique, it prevents column fouling and a limited degree of discrimination or decomposition will not adversely affect the quality of information that can be obtained from a typical chromatogram. The injector was operated at a temperature of 325°C. Extensive tests have shown that significant TAG decomposition occurs at injection temperatures above 325°C. On the other hand, vaporisation was found to be incomplete at lower temperatures. It was also found necessary to use the injector in the splitless mode as TAG losses, due to splitter discrimination, were very evident. The split valve was opened 60 s after injection. An investigation of the exact degree of discrimination or decomposition was not considered important. Although it is accepted that a certain degree of TAG decomposition takes place during injection, the conventional hot split injector, used in the splitless mode, was found to be a suitable injection technique for this application.

Type of carrier gas and flow-rate. Helium was used as a carrier gas in this application. A high flow-rate was used to ensure elution at the lowest possible temperatures, keeping in mind the increased viscosity of the carrier gas at high temperatures. A linear velocity of 1.2 m s^{-1} was measured at a 100°C (initial temperature) and 0.7 m s^{-1} at 375°C (final temperature). As our gas chromatograph is only equipped with constant carrier pressure control, it was not possible to optimize flow for maximum resolution over the wide temperature range.

Sample introduction. A plunger-in-barrel type syringe should be avoided as the high-molecular-mass components, such as triglycerides, can be entrained inside the syringe needle during injection. Bannon *et al.* [9] recommended high-speed injection to overcome needle discrimination, but in the case of this study a Pressure Lok mini-injector was used as an alternative solution to this problem.

The small injection volumes of diluted samples also resulted in improved vaporization. In the case of oils with a low viscosity a five-fold dilution of the sample, *i.e.* in hexane, toluene, acetone or mixtures thereof, is recommended. Carry-over of high-carbon-number components, due to syringe contamination, was limited by leaving the syringe needle in the injector for approximately 10 min after each injection. Carry-over was further limited with a septum purge flow of 20 cm^3 min^{-1}.

Thorough mixing of the vaporized sample with the carrier gas. A modified Jennings Cup type of injector insert has been reported to yield optimum FAME recoveries when using a splitter [9]. A glass detector insert (internal diameter of 1.5 mm) with a plug of quartz wool, was chosen for this application. A high flow-rate was necessary as discrimination of sample components with higher carbon number is apparently affected by the linear velocity of the carrier gas in the injector [5].

Oven temperature. The initial temperature was 100°C, then heated to 375°C with a constant rise of 8°C min^{-1}.

Detector temperature. The FID system was operated at 390°C to prevent TAG condensation in the detector sleeve.

3. Results and discussion

3.1. Analysis of standard samples

Calibration data and chromatograms obtained for the reference standards are shown in Figs. 1 to 6. The calibration for the boiling range distribution of the petroleum fraction of the oil (SIMDIS analysis) is illustrated in Fig. 1. By analysing the mixture of known hydrocarbons, boiling point temperatures are assigned to the time axis. A typical plot of boiling point *vs.* elution time is shown in Fig. 2. The boiling range distribution of the mineral oil component of rolling oil can be determined from this plot. This method is applicable to petroleum fractions

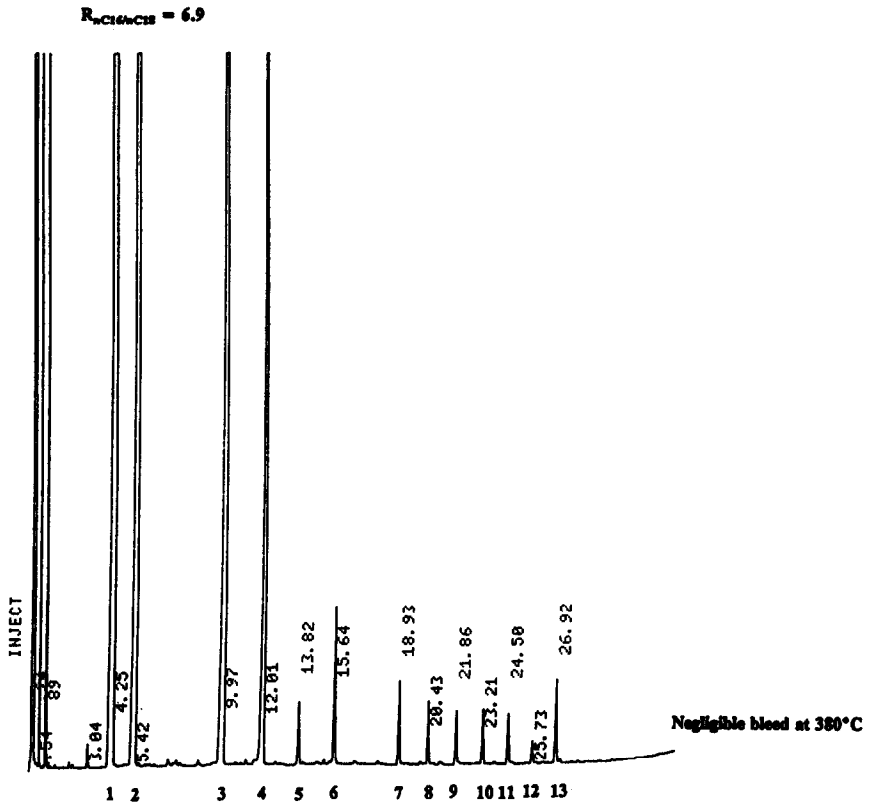

Fig. 1. Chromatogram of the *n*-alkane mixture. GC column and conditions as described in text. Peak identification according to Table 2. Peak 13 elutes at 26.92 min. Full scale is 1024 × 1 pA.

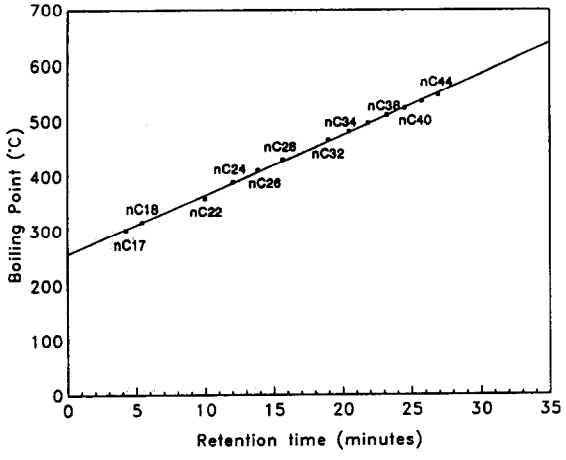

Fig. 2. Calibration curve for the *n*-alkane mixture.

having a maximum upper boiling point limit of 538°C. The column was found to perform well within the requirements of the standard ASTM test method [4]. A resolution of 6.9 was measured on the n-C_{16}/n-C_{18} pair, as compared to a required resolution of between 3 and 8. Baseline stability was acceptable at the operating temperature of 380°C. With a maximum column temperature of 450°C, and n-C_{44} already eluting at approximately 340°C, the boiling range covered could be extended appreciably if required. The resolution obtained for the fatty acid methyl ester standard, is demonstrated in Fig. 3. Separation of four out of the seven components, including separation of components according to degree of unsaturation, was achieved. The excellent resolution achieved for the complex mixture of partial and whole esters, as naturally occurring components in palm oil, is demonstrated in

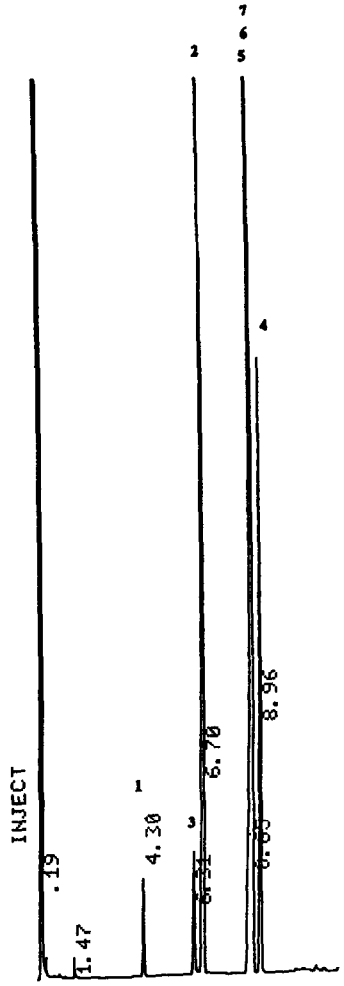

Fig. 3. Chromatogram of the RMS-6 fatty acid standard. GC column and conditions as described in text. Peak identification according to Table 3. Peak 4 elutes at 8.96 minutes. Full scale is 1024 × 1 pA.

Fig. 4. The di- and triglycerides eluted according to their carbon number and were identified accordingly. Excellent baseline stability and low column bleed were achieved at the high elution temperature required for eluting the TAGs. A typical carbon number calibration curve for the fatty acids, diglycerides and triglycerides is shown in Fig. 5. The use of the method to calibrate the instrument for identifying the components found in beef tallow, is illustrated in Fig. 6. This calibration can be used together with the

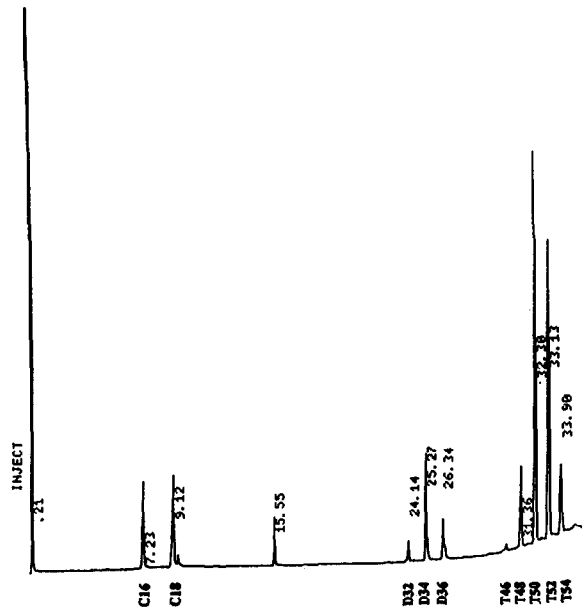

Fig. 4. Chromatogram of palm oil. GC column and conditions as described in text. Peak identification shown on the chromatogram. Peak T_{54} elutes at 33.90 min. Full scale is 1024 × 1 pA.

palm oil calibration to identify the TAG carbon number in cold rolling oil formulations. The method is not only useful to identify the tallow components but also to evaluate the tallow quality. It is evident from Fig. 6 that this specific

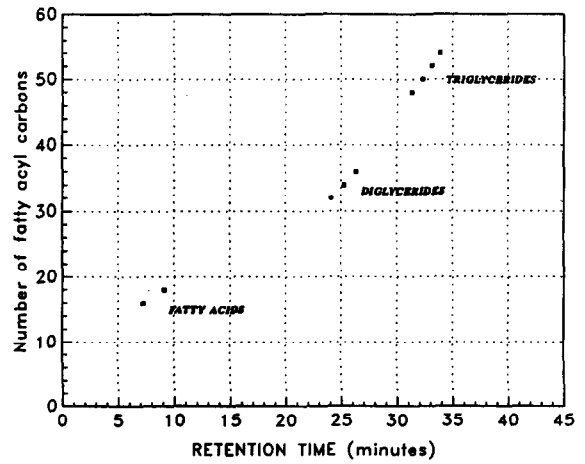

Fig. 5. Calibration curve for fatty acids, diglycerides and triglycerides.

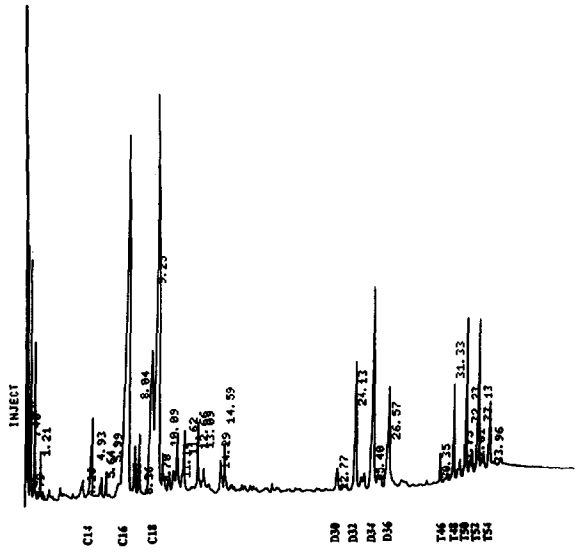

Fig. 6. Chromatogram of a rolling oil base containing beef tallow. GC column and conditions as described in text. Peak identification shown on the chromatogram. Peak T_{54} elutes at 33.96 min. Full scale is 1024 × 1 pA.

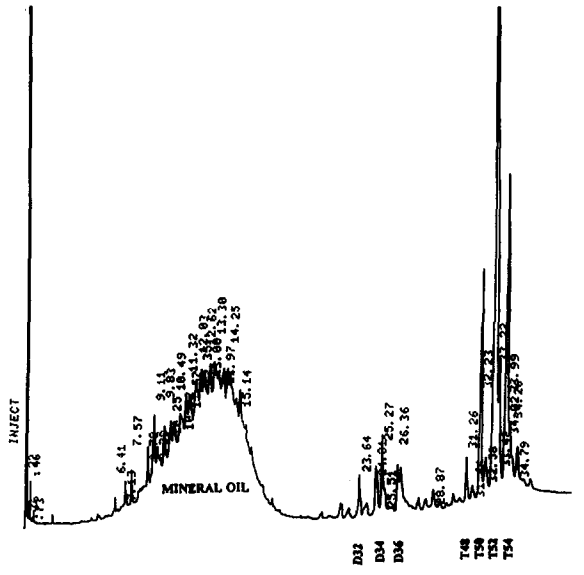

Fig. 7. Chromatogram of a typical Pickler oil. GC column and conditions as described in text. Peak identification shown on the chromatogram. Peak T_{54} elutes at 33.99 min. Full scale is 1024 × 1 pA.

rolling oil base is of poor quality as it contains a large quantity of free fatty acids.

3.2. Analysis of rolling and process oil

Pickler oils

Pickler oils [13] are specialized rolling oils which are specifically formulated to protect the metal sheets against corrosion during the few days which elapse between the pickling and rolling processes. These oils are required to volatilize completely during annealing and therefore consists mainly of mineral oil with a low carbon residue (low burn-off index). Enhanced product cleanliness and decreased carbon edge formation is usually achieved by limiting the fat and free fatty acid content to the amount required to give sufficient lubricity to the mineral oil. Traditional control methods rely on physical tests for viscosity, carbon residue, saponification value and free fatty acid content. The chromatogram in Fig. 7 illustrates the use of gas chromatography as an alternative method to identify the mineral oil, fats and free fatty acid content of such an oil. The chromatogram reveals mineral oil, as a major component with a boiling range of 317 to 459°C, a relative small amount of fat and no free fatty acids. The established boiling range can possibly be used as an indication of the oil viscosity.

Cold rolling oils

As the lubricant is the lifeblood of a cold rolling operation [13], the oil involved directly affects the rolling mill productivity, as well as surface quality and cleanliness. The formulations for sheet and tinplate rolling oils are traditionally based on vegetable or animal fatty materials, either individually or in various combinations. As a result of natural variations it is difficult to perform quality tests on these oils, especially with the traditional methods depicted in Table 1. The advantage of GC analysis is that the individual components in the oil can be monitored, as shown in Fig. 8. The chromatogram reveals a mineral oil boiling range of 216 to 483°C, free fatty acids, carbon numbers of the fat constituents, as well as relative concentrations of the several classes of the components. The occurrence of T_{50}, T_{52}, T_{54} and T_{56}, and the ratios between them, was reminiscent of the trace

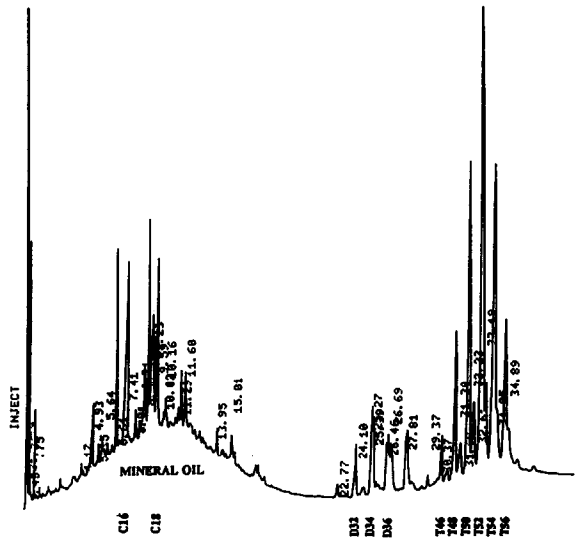

Fig. 8. Chromatogram of a typical cold rolling oil. GC column and conditions as described in text. Peak identification shown on the chromatogram. Peak T_{56} elutes at 34.89 min. Full scale is 1024 × 1 pA.

obtained for a palm oil. Cold rolling oils can, however, also be composed of synthetic materials, which have an improved performance over the traditional products based on natural fats, and which are also easier to control. The chromatogram of a rolling oil based on synthetics (according to the supplier) is shown in Fig. 9, as an example of the wide application range of the method. It is evident that the range of synthetic esters in the mixture has a distinctly different profile from that of the TAG profile of a natural fat as in Fig. 8. The boiling range of the mineral oil was determined as smaller than 302°C and larger than 548°C. Besides synthetic mixtures, a further variation of cold rolling oils are those for conventional tin plate, which requires the best lubrication and high plate-out performance. Beef tallow plus a nominal amount of free fatty acids is normally used for this purpose. A typical chromatogram of such an oil is shown in Fig. 10. No mineral oil was detected, but the oil

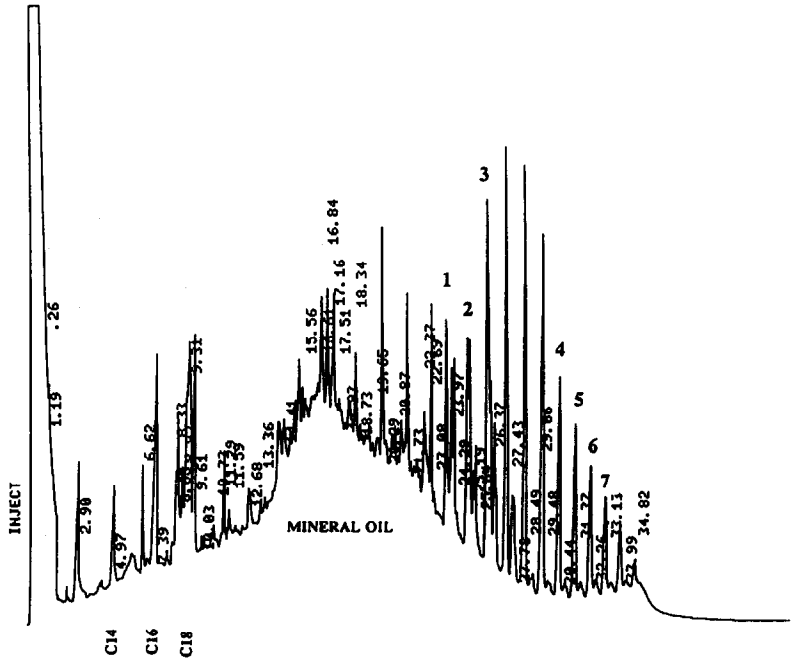

Fig. 9. Chromatogram of a cold rolling oil based on synthetic esters. GC column and conditions as described in text. Peak identification shown on the chromatogram. Peak T_{54} elutes at 33.99 min. Full scale is 1024 × 1 pA.

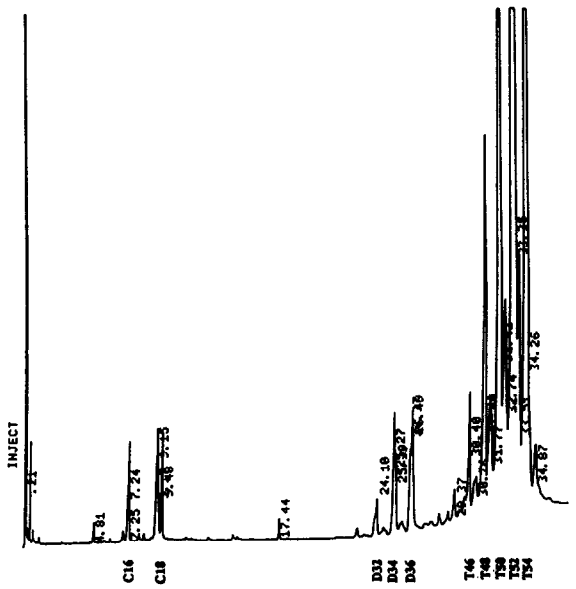

Fig. 10. Chromatogram of a plate out performance oil. GC column and conditions as described in text. Peak identification shown on the chromatogram. Peak T_{54} elutes at 34.26 min. Full scale is 1024 × 1 pA.

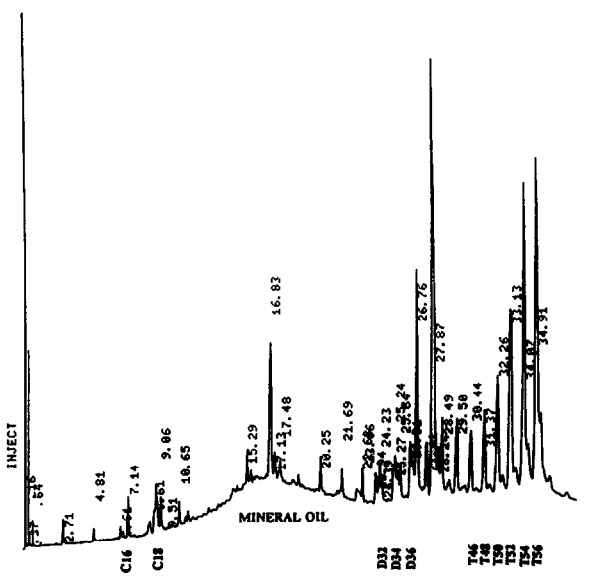

Fig. 11. Chromatogram of a hot rolling oil based on mineral and natural oils. GC column and conditions as described in text. Peak identification shown on the chromatogram. Peak T_{56} elutes at 34.91 min. Full scale is 1024 × 1 pA.

contained a large amount of T_{46}–T_{54} TAGs, as evident from the figure.

Hot rolling mill oils

The presence of triglycerides in hot rolling oil is undesirable as acrolein (an unpleasant, toxic gas) is evolved due to the pyrolysis of glycerol.

$$CH_2OH \cdot CHOH \cdot CH_2OH \rightarrow$$
$$CH_2{:}CH \cdot CHO + 2H_2O$$

Triglycerides are, however, not the only source of acrolein, and the absence of this gas at high temperatures should be confirmed by means of pyrolytic or other methods.

A qualitative chemical analysis is necessary as a fast screening method, to reveal the presence of triglycerides in newly developed products. A chromatogram of a typical hot rolling oil is shown in Fig. 11. The profile of the TAGs found in this specific hot rolling oil formulation is reminiscent of sunflower oil. This was established by comparing the test chromatogram with a standard chromatogram of pure sunflower oil. It was also possible to distinguish between free fatty acids and fatty acid methyl esters. The fatty acid methyl ester peaks are symmetrically shaped, while the free fatty acid peaks elute with a considerable degree of peak fronting. The characteristic fronting profile is due to the well known non-linear partition isotherm of the polar acid in the non-polar stationary phase. The boiling range of the mineral oil found in this mixture was determined as 361 to 537°C.

3.3. The precision of the method

One of the intended uses of the method is to study rolling oil stability, and for this purpose repeatability is important. The overall repeatability of the method depends on the repeatability of the injection technique and the stability of the column. The precision for the set of optimum parameters was assessed by determining the relative standard determination (R.S.D.) of the absolute and relative[a] heights of

[a] The % height relative to the sum of the peak heights of all the peaks listed in Table 4.

Table 4
Reproducibility of an analysis of cold rolling oil ranging from C_{16} to T_{52}, peak numbers 1–7 corresponding to Fig. 9

	C_{14}	C_{16}	C_{18}	1	2	3	4	5	6	7
Average peak height ($n = 10$)	288	634	616	547	543	932	457	328	218	150
S.D.	29	70.2	71.0	36.8	33.1	57.6	52.5	37.4	29.4	20.6
R.S.D. (%)	10.1	11.1	11.5	6.7	6.1	6.2	11.5	11.4	13.5	13.7
Average relative peak height[a]	6.1	13.4	12.9	11.6	11.6	19.8	9.7	7.0	4.6	3.2
S.D.	0.61	1.37	1.16	0.34	0.36	0.63	0.98	0.71	0.55	0.38
R.S.D. (%)	9.92	10.3	9.0	2.9	3.2	3.2	10.1	10.1	12.0	12.0

[a] % Height relative to the sum of the peak heights of all the above-mentioned components.

typical rolling oil components with ten consecutive injections. The synthetic mixture shown in Fig. 9 was chosen for this purpose. The R.S.D.s of both the absolute peak heights and relative % heights of each component are given in Table 4. Considering the small injection volume of 0.01 μl, the results illustrate that an acceptable precision is achieved with this method. The method would, therefore, allow the detection of significant changes in rolling or process oil composition.

4. Conclusions

The quality of information gleaned from the GC traces was considered to be adequate for screening or control purposes, and could be correlated with the reference standards. The method allows the following information to be obtained from the chromatograms: (i) identification of free fatty acids, (ii) identification of partial esters according to carbon number, (iii) identification of whole esters according to TAG carbon number, (iv) simulated distillation analysis of the petroleum oil fraction (boiling point distribution) and (v) distinction between natural fats and products based on synthetic esters in some cases, by comparing the di- and triglyceride profiles to those determined for animal and plant fats. Typical natural and animal fat profiles are available in literature.

By combining the advantages of the microvolume Pressure Lok syringe with the recommended conditions of Bannon *et al.* [9], thermal decomposition, discrimination and precision problems were largely overcome. The method was found to be reproducible (and therefore sensitive towards changes in chemical composition of samples), fast (in comparison to traditional methods), simple and easy to maintain.

The column was stable at high temperature, performed within the requirements of ASTM D2887 for a SIMDIS analysis, and was able to separate various fat components according to carbon number. A long column lifetime is expected, as approximately 150 injections were made at the time of this report. During this time no deterioration in column performance was observed. The investigation also covered a variety of formulations including fifteen different types of oil, from six different suppliers.

It must, however, be emphasized that this application is only suitable as a fast qualitative screening method for major chemical composition. Small quantities of additives, such as nonionic emulsifiers, cannot be detected. The type and amount of emulsifier is critical for good rolling oil performance. The ultimate performance of a rolling oil can therefore only be evaluated during a full scale mill test.

5. Acknowledgements

The advice of J.J. Joubert and the assistance of E.M. Louw are gratefully acknowledged, and S.P.G. Luyt is thanked for proofreading the manuscript. The authors also wish to thank the management of ISCOR for granting permission for the paper to be published.

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