twenty-five per cent. of each size, should contain by calculation 39.835 per cent. metallic iron. Calculating the amount of iron in the first (dry) sample, which is a very poor one, having been spread out thin with a spatula, we find the sample would yield 39.699 per cent. iron, thus causing an error of 0.136 per cent. While this sampling error is notable, it must be remembered that this error would be increased very much if the quartations were carried further, as was shown by the preliminary work. Taking the average results dry we find an error of 0.022 per cent. Taking the average results moist we find an error of 0.006. The error in the percentage of iron ranges from 0.136 per cent. to 0.006 per cent., the greater error coming from a sample made dry and the least from one made moist. In the average results there is a difference of 0.016 per cent. in favor of moist sampling. This fact agrees with the result of an investigation on the moist sampling (with alcohol) of cast-iron borings published by one of the writers some years ago (*Trans. A. I. M. E.*, **14**, 760). The results may be briefly recapitulated as follows:

(1) It is more accurate to sample moist than to sample dry.

(2) Moist sampling is preferable because of neatness of separation and absence of dust.

(3) It is better to mix dry, then moisten, and mix thoroughly again before quartating.

(4) It is far better to flatten out the sample by simple pressure than to spread it out by means of a spatula.

THE ANALYSIS OF LUBRICATING OILS CONTAINING "BLOWN" RAPE-SEED AND "BLOWN" COTTON-SEED OILS.

By Thos. B. Stillman, Ph.D.

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RAPE-SEED oil has long been the standard oil in Europe for lubrication. Its constancy of viscosity at varying temperatures, its non-liability to acidity as compared with other seed oils, and its low cold test, unite in producing the results required of a good lubricant. It, however, is no exception to the rule that vegetable and animal oils suffer partial decomposition when subjected to high temperature produced by friction,

with a result that fatty acids are liberated and corrosion of bearings produced.

The substitution of mineral oils in varying proportions with rape-seed oil has reduced this tendency, this reduction being determined by the percentages of mineral oil present, as the latter liberates no free acids.

It is a peculiar fact, however, that a mineral oil alone does not give as satisfactory results in lubrication (especially cylinder lubrication[1]) as does a mixture of mineral and vegetable or mineral and animal oils, one of the primary causes being that the viscosity of mineral oils rapidly diminishes at high temperatures, whereas the reduction of viscosity of vegetable and animal oils is very much less.

If it were not for this peculiarity between these two classes of oils, mineral lubricating oils could easily supplant (on the score of cheapness) all other oils used in lubrication.

The admixture of oils then being required for the better class of lubricants, it follows that in England where rape-seed oil has been the standard, its use should be continued in compounded oils.

The proportion of rape-seed oil added to mineral oil varies from five to twenty per cent. Where the mineral oil is a clear paraffin oil twenty per cent. of the seed oil is used; where the mineral oil is a dark, heavy oil, five per cent. is generally added.

The separation and estimation of the rape-seed oil in these mixtures presents no difficulty to the analytical chemist when no other seed oil is present, since the saponification of the seed oil, the separation of the fatty acids and recognition of the same are a part of the usual chemical work of this character. The recognition of the constituents of a mixed lubricating oil by analysis is a very different problem from giving a formula by which the mixture can be made. This is evidenced as follows:

Suppose the analysis shows

Rape-seed oil, 20 per cent.
Paraffin oil, 80 per cent.

Paraffin oil varies in specific gravity from 0.875 to 0.921, and it is essential to include in the report of the analysis not only

[1] *The Railroad and Engineering Journal*, **64**, 73-126.

the amount of the paraffin oil but also the gravity, since paraffin oil of gravity 0.875 is a very different product from that of 0.921 gravity, the former selling at seven and one-half cents and the latter at twenty-three cents per gallon. This determination can be made by taking the gravity of the original mixed oil (0.912), then knowing by analysis that twenty per cent. is rape-seed oil (gravity 0.918), the gravity of the eighty per cent. of paraffin oil is easily calculated. Thus:

x = specific gravity of rape-seed oil (0.918)
y = specific gravity of paraffin oil
x = 20 per cent. or $\frac{1}{5}$
y = 80 per cent. or $\frac{4}{5}$

Then $\frac{1}{5}x + \frac{4}{5}y = 0.912$
$0.183 + \frac{4}{5}y = 0.912$
$\frac{4}{5}y = 0.729$
$y = 0.910$

The mixture being composed, therefore, of
Paraffin oil (sp. gr. 0.910), 80 per cent.
Rape-seed oil (sp. gr. 0.918) 20 per cent.

The direct determination by analysis from the ether solution of the mineral oil in the mixture does not give an oil of the same specific gravity as the mineral had before it was mixed with the seed oil. This can be accounted for by the volatilization of a portion of the lighter hydrocarbons of the mineral oil when the ether is expelled during the analysis. For this reason the determination of the percentage of seed oil and the calculation of the mineral oil offers less liability to failure than finding the mineral oil directly.

The introduction of "blown" rape-seed oil instead of the normal rape-seed oil complicates the investigation and renders the use of the formula above given valueless. Rape-seed oil has a gravity of 0.915 to 0.920. Rape-seed oil "blown" has a gravity of from 0.930 to 0.960.

Two difficulties are immediately presented: (1) The chemical analysis does not indicate whether the rape-seed oil is "blown" or not; (2) The use of the formula given without the correct gravity of the "blown" oil would give false results regarding the paraffin oil. To overcome this difficulty some synthetical work is required.

Suppose the specific gravity of the mixed oil is 0.922 and the analysis shows twenty per cent. of rape-seed oil. It will be necessary then to produce a mixture in these proportions that will duplicate the original sample. A check upon this will be the viscosity of the original sample as compared with the one to be made by formula: Thus:

The original oil has a gravity of 0.922, contains (by analysis) twenty per cent. of rape-seed oil, and has a viscosity at 100° F. of 335 seconds (Pennsylvania Railroad Pipette).

First.—Make a mixture of paraffin oil (sp. gr. 0.910), generally used in this character of lubricant, eighty per cent., and rape-seed oil ("unblown") twenty per cent. The viscosity is 165 seconds, showing that this mixture can not be used in place of the original oil.

Second.—Make a mixture of paraffin oil (sp. gr. 0.910) and rape-seed oil (partially blown, sp. gr. 0.930) in the same proportions as above. The resulting viscosity is 267 seconds, showing that the compound is still lacking in viscosity.

Third.—Make a mixture of paraffin oil (sp. gr. 0.910) eighty parts, and rape-seed oil, "blown," (sp. gr. 0.960), twenty parts. The viscosity is 332 seconds.

This now fulfills the conditions required and the synthetical sample agrees with the original in gravity, composition and viscosity.

The use of "blown" rape-seed oil is being gradually replaced by "blown" cotton-seed oil. The latter, which has had but a limited use in lubrication, owing to its liability to acidity, has been greatly improved by this process of "blowing," which is nearly complete oxidation of the oil under comparatively high temperature.

This largely prevents the occurrence of the acidity in the oil, and thus the main objection to its use in lubrication disappears. It is much cheaper than rape-seed oil, since it costs forty cents per gallon, to seventy cents per gallon for the latter. The chemical reactions of the two oils are very similar, and careful analytical work is required that the chemist be not misled.

The following table of comparisons will indicate this:

SPECIFIC GRAVITY.

Cotton-seed oil	0.920 to 0.925
Rape-seed oil	0.915 to 0.920
"Blown" cotton-seed oil	0.930 to 0.960
"Blown" rape-seed oil	0.930 to 0.960

VISCOSITY (PENNSYLVANIA RAILROAD PIPETTE) AT 100° F.

	Seconds.
Cotton-seed oil (sp. gr. 0.925)	165
Rape-seed oil (sp. gr. 0.918)	210
"Blown" cotton-seed oil (sp. gr. 0.960)	2143
"Blown" rape-seed oil (sp. gr. 0.960)	2160

HEIDENREICH'S TEST.

	Before stirring.	After stirring.
Cotton-seed oil	Faint reddish brown.	Brown.
Rape-seed oil	Yellow brown.	Brown.

MASSIE'S TEST.

Cotton-seed oil	Orange red.
Rape-seed oil	Orange.

IODINE ABSORPTION.

Cotton-seed oil	104 to 114
"Blown" cotton-seed oil	93 to 103
Rape-seed oil	102 to 108
"Blown" rape-seed oil	94 to 100

In the comparison of the two oils, when not mixed with a mineral oil, the above tests can be used. The conditions are altered, however, when either one or both are so mixed, since these tests apply only to the pure oils and not to those reduced with large percentages of mineral oil. After the separation of the seed oil from the mineral oil by saponification the identification of the seed oil depends upon the reactions of the fatty acids obtained, and a careful examination and comparison of these reactions shows that the melting points have the greatest difference and thus become a means of recognition.

Thus, the fatty acids from rape-seed oil melt at 20° C., and from cotton-seed oil at 30° C. Hence, if upon analysis of a lubricating oil under above conditions, the fatty acids obtained show a melting point of 20° C. the seed oil can be pronounced rape-seed oil

If the melting point is between these limits, say 23° C., the seed oils are present in a mixture, the proportions of which can be determined by the following formula:

w_1 = proportion of rape-seed oil
w_2 = proportion of cotton-seed oil
w_3 = weight of mixture (20 per cent.)
t_1 = temperature of melting point fatty acids of rape-seed oil.
t_2 = temperature of melting point fatty acids of cotton-seed oil.
t_3 = temperature of melting point of mixed fatty acids.

Then $w_1 = w_3 \frac{t_3 - t_2}{t_1 - t_2}$

$w_2 = w_3 \frac{t_3 - t_1}{t_2 - t_1}$

Inserting the values:

$w_1 = 20 \frac{23 - 30}{20 - 30} = 14$ per cent.

$w_2 = 20 \frac{23 - 20}{30 - 20} = 6$ per cent.

Or,

Paraffin oil	80 per cent.
Rape-seed oil.....	14 per cent.
Cotton-seed oil.....	6 per cent.
Total	100 per cent.

By synthetical work upon these proportions, with comparison of viscosities of the sample submitted with the product, the result will be not only a correct analysis, but a working formula can be given by which a manufacturer can duplicate the original oil.

STEVENS INSTITUTE OF TECHNOLOGY,
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AN AUTOMATIC EXTRACTOR.

BY W. D. HORNE.

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IN the analysis of commercial fertilizers soluble phosphoric acid is separated by repeated washings of the mass with small quantities of water. Two grams of the sample are treated on a ribbed filter with successive portions of water, about ten cc. at a time, until at least 250 cc. have run through, each addition being deferred until the preceding has passed through.

Feeling that time might be saved by having this washing done automatically led to the device of a simple apparatus which mechanically delivers the required quantity of water at measured intervals in a gentle stream upon the material on the filter, stirring it up and leaching out the soluble matter without requiring any attention after the start.